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Francisco Carlos Robles Hernandez

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Improvement in Functional Characteristics of Al-Si Cast Components Through the Utilization of a Novel Electromagnetic Treatment of Liquid Melts

By

Francisco Carlos Robles Hernández

A Dissertation

Submitted to the Faculty of Graduate Studies and Research Through Engineering Materials in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy at the University of Windsor

Windsor Ontario, Canada, 2004

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In the present dissertation the study of Electromagnetic Stirring (ES) and Electromagnetic Stirring and Vibration (ESV) techniques in several Al-Si hypo and hypereutectic alloys were conducted. Five ES and ESV workstations were designed and tested in order to develop a technology capable of conducting melt treatments on Al-Si hypereutectic alloys at temperatures above liquidus. The 390 series alloys were selected because they have shown comparable wear characteristics to cast iron (currently used for cylinder liner applications) three times higher thermal conductivity, the difference in thermal expansion coefficient thermal between the Al-Si alloys was <6%, stability and metallurgical compatibility with the 319 alloy.

The ES technique was effectively used in a semi-solid state to refine the microstructure of the 319 alloy used to cast engine blocks. For instance the ES treatment was capable of reducing the grain of the 319 alloy size by 600%, and partially eliminates the dendrites. However, the ES treatment had negligible effect on Al-Si eutectic, Mg and Cu enriched phases and was limited to the semi-solid state and for hypoeutectic alloys.

The ESV technique was successfully implemented to modify the primary Si particles in Al-Si hypereutectic (390.1(I), 390.1(II), 393.2(I) and 393.2(II)) alloys at temperatures as high as ~100°C above liquidus. The effectiveness of the ESV melt treatment is higher as the temperature gets closer to liquidus. For the first time the application of ESV treatments were able to modify the microstructure of Al-Si hypereutectic alloys from the liquid state. A novel algorithm known as the “Image Analysis Based Si Modification Level” to determine the effect of the ESV melt treatment on the microstructure.

Thermal analysis shows that the liquidus temperature increases up to 15°C as a function of the ESV melt treatment conditions and alloy composition. Therefore, thermal analysis can be used as on-line technique to predict this SiML. Additionally, this SiML, Vickers microhardness and Brinell hardness presented similar behaviour as a function of the ESV melt treatment conditions. The SiML increased from 4.3 to 7.7 and the microhardness increased up to 35% in heat treated test samples, while Brinell hardness increased 25%. Using multiple regression analysis a correlation between the ESV melt treatment conditions, the results of TA, SiML and Vickers and Brinell hardness was established. With multiple regression analysis, several algorithms were developed to predict the SiML or mechanical properties based on the ESV melt
treatment conditions or the TA results. These algorithms are a powerful tool for on line
determination of the SiML of the mechanical properties of ESV treated castings.

The tensile properties of the ESV treated melts were determined in test bars cast in the
Stahl mould. The ESV melt treated test samples presented an improvement of ~20% in as cast
conditions and ~30% after the heat treatment. A fractography analysis of the test bars was
conducted and was found that the primary Si and the shrinkage pores are responsible for the
lower mechanical properties of the test bars cast with untreated melt. Additionally, the
improvement of the tensile properties of the ESV melt treated samples was due to the elimination
of the porosity and the highly refined Si due to the melt treatment.

The ESV treated melts were utilized to cast prototype pistons and cylinder liners. The
cylinder liners were used to produce prototype V6 engine blocks. The prototype engine blocks
were cast at NEMAK of Canada Corporation with cast in prototype Al-Si cylinder liners. The
mechanical characteristics and the microstructure were evaluated following Ford’s Materials
Specifications for cylinder liners and regular production engine blocks in the bulkhead sections.
Soundness (porosity) and mechanical properties in the V6 prototype engine blocks were assessed
in comparison to the regular production engine block. The porosity measurements indicated that
the prototype engine blocks presented a 40% less porosity when Al-Si cylinder liners are used.
This resulted in a 12% increase of the tensile properties of the bulkheads.
CO-AUTHORSHIP STATEMENT

The IRC Staff (Drs. W. Kasprzak, J. H. Sokolowski, and M. Kasprzak) and students (F. C. Robles Hernández and J. Ohl) developed the Electromagnetic Stirring (ES) Platform utilized for the Preliminary experiments of this Ph.D. dissertation [1, 21]. The ES platform proved to be very successful for the 319 melt treatment in the semi-solid state [4, 5, 16].

Intended melt treatments of liquid alloy were proven to be not feasible using the ES platform [4]. Therefore, on the request of Dr. Sokolowski, the electrical expert, Dr. M. Kasprzak, with some involvement by J. Ohl, designed and built four (4) Electromagnetic Stirring and Vibration (ESV) Platforms. These platforms were tested extensively in the IRC’s Light Metals Laboratory. Part of the metallurgical testing and studies carried out by the author are presented in this dissertation [3, 4, 6].

The above-mentioned testing platforms allowed for in-depth metallurgical and electrical analysis of possible advantages and disadvantages associated with an individual platform [21]. In order to overcome some of the limiting characteristics of the previously mentioned platforms a novel Electromagnetic, Thermal and Chemical Treatment (ETCT) Platform was invented [6]. The ETCT was invented by Drs. M, Kasprzak, W. Kasprzak, W. T. Kierkus and J. H. Sokolowski, and students F. C. Robles Hernández and J. Ohl. The main component of the ETCT platform is a dual conductor electromagnetic coil [6]. This platform was successfully utilized in this dissertation for the statistically designed experiments for the electromagnetic treatments of various Al-Si alloys in the liquid state. Based on this novel platform’s performance, the authors of this invention are considering patenting it. Therefore, intricate details of this platform are not revealed in this dissertation.

In order to perform the metallurgical test of the electromagnetically treated melt for casting prototype pistons and cylinder liners, permanent die casting technology was developed. The moulds, gating and riser systems, coatings and casting process thermal parameters (i.e. initial mould temperature) were designed by the author in collaboration with Drs. W. Kasprzak and J. H. Sokolowski [1, 2].

Initially, the electromagnetically treated melt in a simple shape crucible exhibited macro size pores located in the upper part of the crucible. With the assistance of Dr. W. Kasprzak, the author
designed and optimized a new crucible with the “riser” that controls the integrity of very slowly solidified test samples. The author also carried out extensive experiments, including all the experiments in the present Ph.D. dissertation with this crucible [2]. This crucible led the author to the idea of utilizing directional solidification system (developed by D. Yousif et. al. [3]) for the analysis of electromagnetically treated melt’s sensitivity to the temperature gradient.

An integral part of the new crucible was the pouring system which controls cleanliness (e.g. insoluble oxide films) of the melt and consequently of the cast components. Dr. W. Kasprzak and the author designed and tested the siphon pouring system [2].

The author claims the original ideas outlined below to be his intellectual property which were generated in the scope of this dissertation.

- An idea for thermal protection of the electric motor leading to the development of the robust and reliable Electromagnetic Stirring Platform.
- An idea of manufacturing of the dual conductor coil that is a critical component of the ETCT platform.
- The design of the electromagnetic cores for the two-coil-workstation with a ferromagnetic core and the two-core-two-coil workstation.
- Full design regarding the steel mould geometry for the prototype defect free cylinder liner and piston components.
- An idea to utilize a water-cooled plate for metallurgical analysis of the controlled directional solidification process of the electromagnetically treated melt.
DEDICATION

I dedicate this dissertation to my wife (Luz de Maria Flores) and to my mother (Martha Hernández Ramírez) because they are the most important part of my life.

To my sister (Karla Karyna Robles Hernández) and to my brothers (Juan Marcos Robles Hernández) and to my Mother-in-law (Anna Flores) for their friendship, generosity and continuous support.

Este trabajo de investigación es resultado del esfuerzo de todos los mexicanos que de alguna u otra forma contribuyeron con mi educación. Al mismo tiempo quiero dedicar este trabajo a todos los mexicanos que no han tenido acceso a la educación y que en muchas ocasiones son relegados de la sociedad por estúpidos que no pueden ver más allá del espacio pequeño que les rodea y que sin duda ellos son ignorantes. Mi más sincero agradecimiento al sistema educativo mexicano que me proporcionó educación del más alto nivel además de haber sido laica y gratuita y que posteriormente me dio los medios para obtener este grado académico. Un eterno agradecimiento a México y a todos los mexicanos en donde quiera que estén.
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I am grateful to “Consejo Nacional de Ciencia y Tecnologia (CONACyT),” Mexico for their invaluable support during my studies.

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I acknowledge the help of the Technical Support Centre (TSC) at the University of Windsor in particular the late M. Enns. Thanks to J. Robinson for his help with the Scanning Electron Microscopy (SEM) and sample preparation.

I would like to thank my wife Luz de Maria Flores for her friendship, support and for her patience during this dissertation.

The friendship and support of family members M. Hernández Ramírez, K. K. Robles Hernández, J. M. Robles Hernández and A. Flores, and my friends A. Mitrasinovic, Dr. G. Plascencia Barrera, Dr. J. Tiburcio Baez, J. Santos Leon, M. A. Alvaréz Lara, A. Reyes Trejo, A. Suaste, Dr. D. Jaramillo Vigueras, Dr. J. Guerrero Paz, Dr. O. Coreño, L. Gonzalez Reyes, Dr. J. J. Cruz Rivera, D. Szablewski, D. Yousif and R. Francis.
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<th>Definition</th>
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<tr>
<td>AAA</td>
<td>American aluminum association</td>
</tr>
<tr>
<td>AA</td>
<td>Artificial aging</td>
</tr>
<tr>
<td>A</td>
<td>Area of the test sample through out the heat is released</td>
</tr>
<tr>
<td>$a_{fs}$</td>
<td>Apparent fraction solid of the low temperature eutectic reactions</td>
</tr>
<tr>
<td>$a_{fs}^{Mg2Si}$</td>
<td>Apparent fraction solid of the Mg2Si eutectic</td>
</tr>
<tr>
<td>$a_{fs}^{AlNi}$</td>
<td>Apparent fraction solid of the AlNi eutectic</td>
</tr>
<tr>
<td>$a_{fs}^{Cu}$</td>
<td>Apparent fraction solid of the Al-Cu eutectics</td>
</tr>
<tr>
<td>AITAP</td>
<td>Aluminum thermal analysis platform</td>
</tr>
<tr>
<td>ASM</td>
<td>American Foundrymen’s Society</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society of Testing Materials (Standards)</td>
</tr>
<tr>
<td>$B_0$</td>
<td>Direct magnetic field</td>
</tr>
<tr>
<td>$B(t)$</td>
<td>Variable magnetic field</td>
</tr>
<tr>
<td>$Bi$</td>
<td>Biot number</td>
</tr>
<tr>
<td>BL</td>
<td>Baseline or baseline equation</td>
</tr>
<tr>
<td>$C_P$</td>
<td>Heat capacity at a constant pressure</td>
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<tr>
<td>CR</td>
<td>Cooling rate</td>
</tr>
<tr>
<td>CCS</td>
<td>Controlled current source</td>
</tr>
<tr>
<td>$d$</td>
<td>Diameter of a particle</td>
</tr>
<tr>
<td>DCP</td>
<td>Dendrite coherency point</td>
</tr>
<tr>
<td>$dT/dt$</td>
<td>First derivative of the temperature in function of time</td>
</tr>
<tr>
<td>$dT_{0}^{dc}/dt$</td>
<td>First derivative of the temperature in function of time for the zero curve or baseline</td>
</tr>
<tr>
<td>EAP Grade</td>
<td>Si grade based on the Essex Aluminum Plant standard</td>
</tr>
<tr>
<td>Eq., Eqs.</td>
<td>Equation(s)</td>
</tr>
<tr>
<td>ES</td>
<td>Electromagnetic stirring</td>
</tr>
<tr>
<td>ESV</td>
<td>Electromagnetic stirring and vibration</td>
</tr>
<tr>
<td>ETCT</td>
<td>Electromagnetic, thermal and chemical treatment</td>
</tr>
<tr>
<td>$F_L$</td>
<td>Lorentz forces (vibration forces)</td>
</tr>
<tr>
<td>$F$</td>
<td>Frequency of the AC current in Hz</td>
</tr>
<tr>
<td>$f_s$</td>
<td>Fractions solid</td>
</tr>
<tr>
<td>g</td>
<td>Gravity constant (9.81 m/s²)</td>
</tr>
<tr>
<td>HB</td>
<td>Brinell hardness</td>
</tr>
<tr>
<td>HRB, HRC</td>
<td>Rockwell hardness (B and C respectively)</td>
</tr>
<tr>
<td>HC</td>
<td>Homogeneity Coefficient</td>
</tr>
<tr>
<td>h, k, l</td>
<td>Miller index</td>
</tr>
<tr>
<td>HT</td>
<td>Heat treatment (solution treatment and artificial aging (T6))</td>
</tr>
<tr>
<td>I</td>
<td>Intensity of the AC or DC current(s) applied</td>
</tr>
<tr>
<td>$I_{AC}$</td>
<td>Alternating current</td>
</tr>
<tr>
<td>$I_{DC}$</td>
<td>Direct current</td>
</tr>
<tr>
<td>$I_{AC/DC}$</td>
<td>Alternating and direct currents</td>
</tr>
<tr>
<td>IRC</td>
<td>Industrial research chair</td>
</tr>
<tr>
<td>J</td>
<td>Electrical current density</td>
</tr>
<tr>
<td>K</td>
<td>Temperature indicated in the absolute scale (Kelvin degrees)</td>
</tr>
<tr>
<td>L</td>
<td>Length</td>
</tr>
<tr>
<td>LMT</td>
<td>Low mass thermocouple</td>
</tr>
<tr>
<td>Acronym</td>
<td>Description</td>
</tr>
<tr>
<td>----------</td>
<td>--------------------------------------------------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>LOM</td>
<td>Light optical microscope</td>
</tr>
<tr>
<td>MMRI</td>
<td>McMaster Manufacturing Research Institute</td>
</tr>
<tr>
<td>N</td>
<td>Frequency of the vibration created by the interaction of the AC and DC fields</td>
</tr>
<tr>
<td>NCE</td>
<td>Network of Centre of Excellence</td>
</tr>
<tr>
<td>Q</td>
<td>Heat</td>
</tr>
<tr>
<td>r</td>
<td>Radial direction, indicating the release of the heat in solidifying test samples</td>
</tr>
<tr>
<td>RDF</td>
<td>Radial distribution function</td>
</tr>
<tr>
<td>S_A</td>
<td>Standard deviation of selected test sample</td>
</tr>
<tr>
<td>SDAS</td>
<td>Secondary dendrite arm spacing</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>SEM/BSE</td>
<td>Scanning electron microscopy conducted using Backscattering Electrons mode</td>
</tr>
<tr>
<td>SEB/BSE</td>
<td>Scanning electron microscopy conducted using Secondary Electrons mode</td>
</tr>
<tr>
<td>SEM/EDX</td>
<td>Scanning electron microscope/energy dispersive X-Ray</td>
</tr>
<tr>
<td>Si_EQ</td>
<td>Silicon equivalency</td>
</tr>
<tr>
<td>Si_ML</td>
<td>Silicon modification level</td>
</tr>
<tr>
<td>S(Q)</td>
<td>Structure factor</td>
</tr>
<tr>
<td>S_R</td>
<td>Lowest measured standard deviation among all the test samples</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscope</td>
</tr>
<tr>
<td>t</td>
<td>Time of the ESV melt treatment</td>
</tr>
<tr>
<td>T</td>
<td>Temperature</td>
</tr>
<tr>
<td>T_LIQ</td>
<td>Is the time recorded by thermal analysis at the liquidus temperature</td>
</tr>
<tr>
<td>T_PRI-Si</td>
<td>Primary Si nucleation temperature</td>
</tr>
<tr>
<td>T_PRI-Si_MIN</td>
<td>Primary Si undercooling</td>
</tr>
<tr>
<td>T_PRI-Si_G</td>
<td>Primary Si growth</td>
</tr>
<tr>
<td>T_M-Si</td>
<td>Al-Si eutectic nucleation temperature</td>
</tr>
<tr>
<td>T_M-Si_MIN</td>
<td>Al-Si eutectic minimum (undercooling) temperature</td>
</tr>
<tr>
<td>T_M-Si_E</td>
<td>Al-Si eutectic growth temperature</td>
</tr>
<tr>
<td>T_Mg-Si</td>
<td>Temperature of nucleation for the Mg2Si eutectic</td>
</tr>
<tr>
<td>T_3Ni</td>
<td>Temperature of nucleation for the Al3Ni eutectic</td>
</tr>
<tr>
<td>T_Mg2Si+Al-Cu</td>
<td>Temperature of nucleation for the Al2Cu and Al-Cu fine eutectics</td>
</tr>
<tr>
<td>T_SOL</td>
<td>Is the time recorded by thermal analysis at the solidus temperature</td>
</tr>
<tr>
<td>T_SOL</td>
<td>Solidus temperature</td>
</tr>
<tr>
<td>T_ESV</td>
<td>Temperature of the ESV melt treatment</td>
</tr>
<tr>
<td>T_Pouring</td>
<td>Pouring Temperature</td>
</tr>
<tr>
<td>T_C</td>
<td>Temperature in the centre of the test sample</td>
</tr>
<tr>
<td>T_e</td>
<td>Temperature close to the wall of the test sample</td>
</tr>
<tr>
<td>Total Area</td>
<td>Area between the first derivative and baseline</td>
</tr>
<tr>
<td>T(r)</td>
<td>Thermocouple located in the centre of the test sample</td>
</tr>
<tr>
<td>T(R)</td>
<td>Thermocouple located in the wall of the test sample</td>
</tr>
<tr>
<td>T_Range</td>
<td>Range of temperatures where the ESV melt treatment was applied</td>
</tr>
<tr>
<td>U</td>
<td>Heat transmission coefficient</td>
</tr>
<tr>
<td>UMSA</td>
<td>Universal metallurgical simulator and analyzer</td>
</tr>
<tr>
<td>V</td>
<td>Volume of the test sample</td>
</tr>
<tr>
<td>z, h</td>
<td>Height</td>
</tr>
<tr>
<td>u, v, k</td>
<td>Unit vectors</td>
</tr>
<tr>
<td>Symbol</td>
<td>Definition</td>
</tr>
<tr>
<td>--------</td>
<td>------------</td>
</tr>
<tr>
<td>1D</td>
<td>Release of the heat in one dimension (usually the radial one)</td>
</tr>
<tr>
<td>2D</td>
<td>Release of the heat in two dimensions (usually the radial and horizontal)</td>
</tr>
<tr>
<td>°C</td>
<td>Temperature in degrees Celsius or in the centigrade scale</td>
</tr>
<tr>
<td>λ</td>
<td>Thermal conductivity</td>
</tr>
<tr>
<td>ΔT</td>
<td>Temperature range</td>
</tr>
<tr>
<td>ρₜ</td>
<td>Density of a droplet</td>
</tr>
<tr>
<td>ρₚ</td>
<td>Density of the fluid</td>
</tr>
<tr>
<td>ν</td>
<td>Settling velocity</td>
</tr>
<tr>
<td>σ₀, Yₕ</td>
<td>Yield stress or yield strength</td>
</tr>
<tr>
<td>μᵥ₁₀₀₀</td>
<td>Vickers hardness measurements conducted with a load of 1000g</td>
</tr>
<tr>
<td>μᵥ₂₅</td>
<td>Vickers microhardness measurements conducted with a load of 25g</td>
</tr>
<tr>
<td>ṅ, η</td>
<td>Viscosity</td>
</tr>
</tbody>
</table>
CHAPTER 1. INTRODUCTION

Since one in six jobs in North America is linked to the automotive industry, its sustainable growth is vital to the Canadian economy. With increased globalization and recent downsizing of corporate research resources, it has become increasingly difficult for the Canadian automotive light casting industry to stay competitive. They must therefore with the help of associated universities develop and manufacture light weight, structurally safe and low cost components including engine blocks and pistons. In order to achieve these goals, novel lightweight materials, innovative and more efficient processing technologies must be developed and implemented by global aluminum suppliers like the NEMAK of Canada Corporation.

The NSERC/Ford-NEMAK/University of Windsor Industrial Research Chair (IRC) in Light Metals Casting Technology and the AUTO21 NCE C7 Group have made tremendous strides in addressing these issues. This dissertation is an integral part of these two programs.

At the present time a vast majority of global engine block manufacturers are using cast iron cylinder liners. Therefore, many potential advances in engine block and gasoline engine design as well as their casting technologies can not be realized. In order to address some of the above mentioned goals including the conversion of cast iron liners to Al-Si hypereutectic alloy(s) the improvement of the existing piston characteristics is proposed, such as lighter, higher thermal conductivity, more thermally stable, more similar thermal expansion coefficient, etc.

To date, the use of grain refiners (Ti, B and TiB) and silicon modifiers (Sr and Na for hypoeutectic, and P and rare earth elements for hypereutectic) for Al-Si alloys have been the main compounds used to improve microstructural characteristics of the castings. Castings are usually heat treated for two reasons; to release the residual stresses (die casting) and to dissolve solute atoms (Cu, Ni, Mg, etc.) within the matrix that form precipitation hardening [26]. Additions of proper amounts of grain refiners and silicon modifiers as well as controlled heat treatments usually improve the service characteristics of casting components, including the mechanical properties. The main disadvantage of chemical treatments, predominantly the use of modifiers (i.e. Sr for eutectic Si) promotes the formation of porosity [26], which is detrimental to the cast component’s durability.
Since the 1960's the application of mechanical or electromagnetic stirring techniques have been used to improve casting characteristics [27 - 34]. To date, Electromagnetic Stirring (ES), mechanical stirring (MS), ultrasonic stirring treatment (UST) and Electromagnetic Stirring and Vibration (ESV) have been used mostly for continuous casting of ferrous and non-ferrous alloys. ES and ESV techniques are widely accepted for its improve homogeneity of castings, reduction of porosity, improve mechanical properties, improved melt fluidity, refinement of the microstructure and more equiaxed, lower micro and macrosegregation, to mention just a few [35 - 73]. Unfortunately, the application of these techniques have been limited to the semi-solid state; this is due to the fact that the refining occurs during the early stages of nucleation and growth of the precipitating phases.

During the feasibility studies the IRC developed an ES work station for the W319 Al-Si treatment. Using this workstation proved to be very successful for the refinement of the alpha aluminum grain size (expressed by the density) and the secondary dendrite arm spacing (SDAS). The reduction in grain density was from 3 to 94 grains/cm² and the SDAS from 74 μm to 66 μm. Since the Al-Si hypoeutectic Si alloys do not present the Si agglomerates in liquid state, the ES treatment is effective only in semi-solid [16].

The feasibility studies gave an impetus to explore more powerful electromagnetic treatment(s) of Al-Si alloy melts. Four electromagnetic stirring and vibration (ESV) workstations were designed and built by the IRC team and utilized in this dissertation. The “optimum” workstation known as the Electromagnetic, Thermal and Chemical Treatment (ETCT) Platform was utilized for the main experiments of this dissertation. In the Intellectual Property and Claims section is described the author’s contribution to this development.

This dissertation is focused on the development of a melt treatment technology capable of refining the microstructure and modify of phases such as primary and eutectic Si present in Al-Si hypereutectic alloys in a liquid state [6]. The reasons for the effectiveness of the ESV melt treatments at temperatures above liquidus will be demonstrated scientifically throughout this dissertation. At the inception of this work the hypothesis that Al-Si hypereutectic melts are composed of two immiscible liquids was formulated. One of these liquids is a liquid solution containing all the elements of the alloy, including Si and the other has Si enriched regions (Si agglomerates). Further analysis of the ESV treated melts confirmed the presence of Si enriched agglomerates (light optical microscopy and image analysis).
The Al-Si hypereutectic alloys were selected based on their wear resistance and acceptance for piston and cylinder liner applications [22 - 25]. During the course of this research the conversion of the cast iron cylinder liners to the ESV melt treated Al-Si hypereutectic alloys was proposed [4]. The conversion of these materials will simplify the casting and heat treatment processes, machining operations, and improve the engine heat transfer by creating a metallurgical interface instead of the "mechanical" interface (the present situation). Other benefits will be the considerable engine block weight reduction and its improved compactness by possibly minimizing the bridge thickness or even eliminating it [4].

The ESV melt treatments were conducted using statistically designed experiments to determine the effect of the different experimental parameters on the modification and refinement of the microstructure as well as on the mechanical properties. The ESV melt treatment independent variables used to carry out the melt treatments were temperature (T in °C), frequency (f in Hz), electrical current (I<sub>ac/dc</sub> in A), time (t in s), hydrogen level (H<sub>2</sub> in mL H<sub>2</sub>/100 g of Al) and cooling rate (in °C/s). Statistically designed experiments were used to assess the effect of the independent variables on the structure and mechanical properties and the modifications and refinement of the ESV melt treated casting. The test samples structural characteristics were analyzed using statistical tools [74], which allowed for the statistically valid correlation between the ESV melt treatment conditions, thermal analysis and the mechanical properties.

During the solidification of the reference (untreated and solidified under natural heat exchange conditions) and ESV melt treated test samples thermal analysis was conducted. These results were used to assess the relationship between the melt treatment parameters and the solidification characteristics of the test samples. The as cast and heat treated test samples were analyzed using light optical microscopy and image analysis. In order to determine the ESV melt treatment effect on the microstructure a novel procedure to measure the phase modification was developed. The previously-mentioned procedure is known as the Image Analysis Based Silicon Modification Level (SiML) and can be used for all 3XX.X alloys. Additionally, mechanical properties (tensile test, micro hardness and Brinell hardness (BH)) were evaluated. The correlation between thermal analysis, SiML and mechanical properties was carried out by developing mathematical equations as a function of independent variables using single and multiple regressions analysis.
Chapter 1. Introduction

The ESV treated melts were used to cast prototype pistons and cylinder liners for industrial applications using steel gravity die-casting moulds that were designed and built under the scope of this research. Prototype cylinder liners with different amounts of Si were cast using untreated and ESV treated melts. The cylinder liners were used to cast prototype 2.5L V6 engine blocks at NEMAK Engineering Centre. The characteristics of the regular production and prototype engine blocks were compared to estimate the advantages of substituting cast iron for Al-Si hypereutectic cylinder liners.

1.1. Scientific and Technical Objectives

The following list contains specific topics with their respective objectives related to the main areas of this dissertation:

1) To provide a comprehensive and concise literature review on modification and refinement mechanisms and industrial technologies for Al-Si hypereutectic alloys in liquid and semi-solid states (Chapter 2).
   a) To understand the atomic level of liquid Al-Si melt structures.
   b) To study the available techniques (including patented ones) for microstructure refinement and phase modification of Al-Si alloys by using mechanical and electromagnetic means.
   c) To provide examples of potential applications of the Al-Si hypereutectic alloys for the automotive industry.

2) To review the negative effects of cast in cast iron cylinder liners used by the Ford Motor Company in Al alloys (i.e. the W319 engine blocks) (Chapter 3).
   a) To conduct a series of calorimetric tests to determine the differences in the thermal expansion coefficients between cast iron and Al-Si hypo and hypereutectic alloys.
   b) To determine the nature of the detrimental interface created between the cast iron and the Al from the W319 alloy.
   c) To justify the scientific and technical advantages of substituting cast iron for Al-Si cylinder liners in the regular production of W319 alloy engine blocks cast at the NEMAK Windsor Aluminum Plant.
3) To develop a state-of-the-art technology capable of performing microstructure refinement and phase modification of castable Al-Si wear resistant alloys to substitute cast iron in cylinder liners applications (Chapter 5).
   a) To develop and test ES and ESV treatment technologies in semi-solid and liquid (melt) states to better understand the microstructure refining and phase modification mechanisms for applications in Al-Si alloys.
   b) To use different Al-Si compositions to determine a potential alloy suitable for the substitution of cast iron in cylinder liner applications.
   c) To statistically design experiments using the DOE procedure in order to identify the effect of the ESV melt treatment conditions on microstructure and mechanical properties of test samples and prototype pistons and cylinder liners.

4) To establish the statistical correlation between ES and ESV melt treatment conditions and the image analysis with thermal analysis for on-line determination of the level of phase modification of Al-Si alloys (Chapter 5).
   a) To use thermal analysis techniques for characterization of electromagnetically treated Al-Si hypereutectic alloy melts.
   b) To utilize the cooling curve algorithms [176, 183, 184] to precisely determine characteristic temperatures, fraction solids and apparent fraction solids of the phases present in reference and ES/ESV melt treated test samples. To determine the effect of the ESV melt treatment on the cooling curve characteristics.

5) To perform metallurgical characterization of the microstructure of Al-Si hypereutectic alloys in liquid, semi-solid and solid states for reference and ESV melt treated melts (Chapter 5).
   a) To demonstrate that the Al-Si hypereutectic alloys in liquid states are composed of two immiscible liquids. These two liquids were the key factor for the development of an effective ESV melt treatment technology for applications in liquid state.
   b) To conduct a microstructural characterization of the Al-Si hypereutectic alloys including the assessment of the solidification sequence and fraction solid.
   c) To utilize the SiML algorithm to assess the level of modification of Si in Al-Si hypo and hypereutectic alloys.
6) To statistically correlate ESV melt treatment conditions, thermal analysis results and Si modification with mechanical (hardness and/or strength) and physical properties (thermal expansion coefficient) for on-line applications (Chapter 5).

   a) To relate the ESV melt treatment conditions with thermal analysis and SiML data for the establishment of mathematical models able to predict the SiML and mechanical properties based on ESV melt treatment conditions and thermal analysis results.

7) To cast prototype pistons and cylinder liners (Chapter 5)

   a) To design permanent moulds for gravity die casting to cast the prototype pistons and cylinder liners.

   b) To cast prototype V6 engine blocks using cast in cast iron and ESV melt treated Al-Si cylinder liners.

   c) To conduct a metallurgical characterization (including mechanical properties) of the V6 regular production and prototype engine blocks with the aim to justify the substitution of cast iron cylinder liners for Al-Si ESV melt treated cylinder liners for a new generation of engine blocks.
CHAPTER 2. LITERATURE REVIEW

2. Introduction to the Literature Review

For this dissertation a world wide literature review including patents was conducted to support the technical and scientific achievements through the utilization of the ES and ESV melt treatment techniques. In the present work there are several aspects, such as the application of melt treatments in a liquid state, which provide the novelty required for Ph.D. research. This literature review consists of the metallurgical aspects of Al-Si hypo and hypereutectic alloys, thermal analysis, microstructure refinement techniques, and potential applications for Al-Si alloys in the automotive industry. The importance of studying Al-Si hypereutectic alloys in this research is based on the fact, that they are ideal candidates to substitute cast iron for cylinder liners applications; this is due to their mechanical and physical characteristics. It is important to mention that the available literature on Al-Si hypereutectic alloys is limited compared to Al-Si hypoeutectic alloys.

2.1. Metallurgy of Aluminum Silicon Alloys

Aluminum Silicon (Al-Si) alloys are the most important among cast alloys and have wide-spread applications, especially in the aerospace and automotive industries [26, 184]. One dominant group of Al-Si foundry alloys contain between 5 and 25 wt% Si; the most important group is the one which contains Mg, Ni and Cu additions. The microstructure of the Al-Si alloys consist of a primary phase (α-Al or Si for hypo and hypereutectic respectively), Al-Si eutectic and Mg, Fe, Ni and Cu enriched phases [76, 121 - 128]. Al-Si castings constitute from 80 to 90% of the total Al castings produced world wide [26, 81, 82]. Fe is the main impurity and most undesirable element for Al-Si alloys because it forms detrimental brittle crystals (intermetallics) [81]. Mn is added to transform the detrimental Fe enriched intermetallics into chinese script phases, which are less harmful.

To facilitate the identification of Al alloys the American Aluminum Association (AAA) classified these alloys as a function of their alloy chemical composition. For the most abundant
element, after Al, a particular family is assigned. The rest of the elements can be found in the alloys with a range of compositions of several other elements; i.e. Al-Si alloys constitute the 3XX.X family [82]. Some alloys investigated in this dissertation do not have an AAA designation because they were custom made hybrids at the University of Windsor; therefore, their designation was assigned based on their chemical composition.

An Al-Si binary system is composed by a eutectic reaction, which takes place at ~12.3 wt% Si at a temperature of 577°C (Figure 2.1). All of the Al-Si compositions with less than 12.3 wt% Si are known as hypoeutectic, with more than 12.3 wt% Si hypereutectic and 12.3 wt% Si eutectic. Si imparts fluidity, wear resistance, low density, high thermal conductivity, low thermal expansion, and good retention of physical and mechanical properties at high temperature [81].

![Figure 2.1. Section of the binary Al-Si phase diagram [81].](image)

Due to the mechanical properties, including wear resistance, and physical characteristics (fluidity, relatively low density and more similar thermal expansion coefficient with 319 Alloy) of the Al-Si hypereutectic alloys made them the ideal candidate to substitute cast iron cylinder liners on Al-Si engine blocks [23, 25, 143 - 155, 159, 208]. Al-Si hypereutectic alloys are mainly used for die casting applications. For example pistons, cylinder liners and engine blocks. To date Al-Si hypereutectic alloys have been successfully implemented for engine blocks. Unfortunately, their application is restricted to selected luxurious and expensive vehicles [143, 144, 155 - 160].
2.1.1. Chemical Compositions of Aluminum Silicon Alloys

2.1.1.1. Silicon

Silicon improves the metal castability and reduces metal shrinkage during solidification. The natural morphology of the eutectic Si is acicular or plate-like and polyhedral for the primary Si present in Al-Si hypereutectic alloys. However, due to the skewed couple zone effect (non-equilibrium solidification), primary Si particles can be found in hypoeutectic compositions with more than 9 wt% Si that were solidified out of the equilibrium conditions [126, 169]. These morphologies are modified into a less harmful geometry using chemical additions or by solution treatment [160].

2.1.1.2. Copper

Copper in Al-Si alloys such as the 319 improves as-cast and high temperature strength properties. However, it reduces the ductility. Copper is added as hardening strengtheners. In general the hardness of Al-Si alloys is acceptable, but the application of heat treatments promotes the precipitation of strengthening Cu enriched phases that allow the full development of the hardness and strength of Al-Si alloys [160, 210, 217]. Often Al-Si castings are over aged to provide a compromise between strength and dimensional stability in service. Cu enriched phases may take several forms depending on the alloy’s chemical composition, solidification and heat treatment conditions [160].

2.1.1.3. Magnesium

Magnesium is an element that is usually added as a precipitation hardener. The application of solution treatments (T5 or T6) promote the formation of a supersaturate solid solution with silicon, which after quenching (if applied) form Mg$_x$Si$_y$ rich particles. The Mg$_x$Si$_y$ particles undergo a second transformation by changing into Mg$_2$Si during natural or artificial aging [119, 132]. Under controlled precipitation conditions the Mg$_2$Si is homogeneously distributed resulting in improved mechanical properties [119]. Armstrong [120] reported that
certain advantages can be obtained when using strontium in combination with higher magnesium alloys. Gupta et al [129, 130] found that additions of magnesium from 0.07 wt% to 0.6 wt% have a negligible effect on mechanical properties of the alloy, either in the as-cast or in the T5 condition. However, the grain size increases with the Mg content after a 0.30% level is exceeded [132]. Mg additions (>0.6 wt%) increase the ultimate tensile strength (UTS) and fatigue resistance but reduce the ductility [131].

2.1.1.4. Nickel

Nickel is added in some Al-Si alloys (such as the 339) to increase tensile strength, yield strength at temperatures in the range of 250 to 375°C, but reduces elongation at temperatures below 50°C. The increase in strength is mainly due to the precipitation of the Al3Ni intermetallic [77, 97]. Additions of less than 0.3 wt% Ni have a negligible effect on the mechanical properties, but between 0.3 to 0.5 wt% Ni the yield strength improves by up to 50 MPa [137]. In fact, Ni has similar strengthening effects as Cu in Al-Si alloys, but in comparison Ni is more costly. However, for pistons that are only artificially aged additions of up to 2 wt% are commonly used to increase the amount of strengthening elements within the matrix [143].

2.1.1.5. Zinc

Additions of zinc in the range of 1.0 wt% to 4 wt% slightly improve tensile properties in heat treated (T5 or T6) Al-Si castings, without affecting ductility. However, less than 1 wt% Zn did not affect the mechanical properties of the Al-Si alloys [50, 51]. Mackay et. al. [96] found that by increasing the levels of Zn up to 15 wt% in Al-Si hypoeutectic alloys superior mechanical properties can be attained.

2.1.1.6. Iron

Fe is considered the principal impurity element of Al-Si alloys, which is undesirable because it forms brittle AlFe intermetallic crystals [185 - 187]. Ideally, the amount of Fe should be as low as possible. For example, in primary alloys (356 and 357) it should be up to 0.2 wt%.
Chapter 2. Literature Review

Fe. In recycled alloys (319, 390) it should contain up to 1 wt% Fe [97]. In Al-Si casting Fe reacts with aluminum and silicon to form the stoichiometry intermetallic Al$_5$FeSi ($\beta$-phase), with a plate like morphology (needle), that reduces mechanical properties and increases machining costs [97].

2.1.1.7. Manganese

Manganese has very low solubility in Al and combines with the remaining Fe and Si. The combination of these four (4) elements results in the formation of an Al$_{13}$(Mn, Fe)$_3$Si$_2$ intermetallic [97, 98]. The Al$_{13}$(Mn, Fe)$_3$Si$_2$ is known as chinese script, but it can also be blocky or dendritic [97]. A ratio between 1:2 (Mn to Fe respectively) is usually recommended to bring the Fe $\beta$-phase into an $\alpha$-phase, which is less hazardous than the $\beta$-phase [115 - 117].

2.2. Metallurgy of the Al-Si Hypereutectic Alloys

In this dissertation the 390 and 393 alloys and their metallurgical characteristics were studied carefully and are presented in the following sections. Table 2.1 provides the results of characterization using thermal analysis and scanning electron microscopy and energy dispersive X-ray (SEM/EDX) techniques [76]. The results presented in the above-mentioned table correspond to the most complete characterization found in the literature for the 390 alloy. These results will be used to support the findings of the present research.

2.2.1. Structure of Liquid Pure Metals and Al-Si Hypereutectic Melts

Characterization of melts using X-Ray Diffraction, synchrotron radiation, electron and neutron beams had demonstrated that metals in liquid state have short range ordered structures. The results of characterization conducted using the above-mentioned techniques provide the possibility to experimentally determine the structure factor ($S(Q)$) of melts as well as the radial atomic distribution function. The structure factor is defined by Eq. 2.1, which is a very important relation in X-ray crystallography since it permits calculation of the intensity of any $hkl$ reflection from a knowledge of the atomic positions [200]. The short range atomic arrangement in highly
pure elements is reduced as the superheat temperature increases. By measuring the electrical resistance and magnetic susceptibility a correlation with the size of the atomic arrangements can be established. Also, gamma ray absorption spectroscopy was used to determine differences in the density of liquid elements, to measure the nanometric “agglomerates” that held short to medium range atomic arrangements [121].

Table 2.1. Chemical composition and solidification reactions detected by thermal analysis and SEM/EDX in the 390 Al-Si alloy solidified under natural heat exchange conditions [76].

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Element in wt%</th>
<th>Nucleation Reactions</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Si</td>
<td>Fe</td>
<td>Cu</td>
</tr>
<tr>
<td>390^1</td>
<td>15</td>
<td>0.3</td>
<td>5.5</td>
</tr>
<tr>
<td>390^2</td>
<td>17.45</td>
<td>0.74</td>
<td>4.81</td>
</tr>
<tr>
<td>1</td>
<td>390^1</td>
<td>Formation of Primary Silicon</td>
<td>636^1</td>
</tr>
<tr>
<td>2</td>
<td>390^1</td>
<td>Development of Dendritic Network</td>
<td>561^1</td>
</tr>
<tr>
<td>3</td>
<td>390^1</td>
<td>Liq. → Al + Si + Al_{13}FeSi</td>
<td>575^1</td>
</tr>
<tr>
<td>3a</td>
<td>390^2</td>
<td>Liq. → Al + Si + Al_{15}(Mn,Fe)<em>{3}Si</em>{2}</td>
<td>573^2</td>
</tr>
<tr>
<td>3b</td>
<td>390^2</td>
<td>Liq. → Al + Si + Al_{6}FeSi</td>
<td>575^2</td>
</tr>
<tr>
<td>4</td>
<td>390^1</td>
<td>Liq. → Al + Si + Mg_{2}Si</td>
<td>555^1^2</td>
</tr>
<tr>
<td>5</td>
<td>390^1</td>
<td>Liq. + Mg_{2}Si → Al + Si + Al_{2}Cu + Al_{5}Mg_{8}Cu_{2}Si_{6}</td>
<td>512^1^2</td>
</tr>
<tr>
<td>6</td>
<td>390^1</td>
<td>Liq. → Al + Al_{2}Cu + Al_{5}Mg_{8}Cu_{2}Si_{6} + Si</td>
<td>507^1^2</td>
</tr>
</tbody>
</table>

\[ S(Q)_{hkl} = \sum_{N=1}^{N} f_Se^{-2\pi(\beta h_x+\beta k_x+\beta l_x)} \]  
Eq. 2.1

where: \( S(Q) \) is the structure factor,  
\( f \) is the atomic scattering factor,  
\( hkl \) are the Miller indices and  
\( uvw \) are the fraction coordinates (unit vectors).

The X-ray diffraction results of the structure factor of pure Al and Cu are presented in Figure 2.2 [219]. Figure 2.2 shows the presence of secondary reflections (in front of the main reflection), with a characteristic structure factor similar to those of pure liquid Cu and Al (Figure 2.2a and b respectively). These reflections show that there not only exist short range order
structures in liquid state but also medium-range order structures. The secondary reflections are attributed to the clustering or agglomeration of atoms in the liquid state [219].

![Graph showing total structure factors of liquid Cu and Al](image)

**Figure 2.2.** Total structure factors ($S(Q)$) of liquid a) Cu and b) Al [219].

In binary or multi-element solutions in a liquid state the short range atomic arrangement occurs differently and is classified according to the appearance of its phase diagrams (solid solutions, with intermetallic compounds, eutectics and with gaps of immiscibility). For example, a closer examination of the concentration of solid solutions is non consistent with the idea of an ideal solution because they present non uniform electrical resistance. Liquid alloys with intermetallic compounds show regions with maximum electrical resistance that are presumably atomic arrangements with compositions similar to the intermetallics. In eutectic compositions micro heterogeneity regions can be observed (i.e. the Al-Si eutectic at temperatures of 700°C present Si rich regions of ~4.5 nm that are reduced to 0 nm at temperatures of >1000°C). Alloying systems with immiscibility gaps also show the above-mentioned heterogeneity regions, but its presence was only detected at temperatures close to liquidus ($\pm 3^\circ$C) [121].

Si is a semi-conductor at normal temperatures and pressures, but becomes metallic in a liquid state. The structure of molten Si presents local order similar to that found in liquid white tin. However, the studies conducted in liquid Al-Si hypereutectic alloys are very limited [125]. In the following text an analysis of the mid and short order Si atoms in Al-Si liquid alloys is presented and discussed.
Chapter 2. Literature Review

The X-ray diffraction technique was used to characterize high purity Al-Si eutectic and hypereutectic compositions in a liquid state present (at temperatures from 620 to 1075°C). The diffractograms present the main peak (reflection) with a structure factor ($S(Q)$) similar to pure Al (26.8 nm$^{-1}$) [125] and pre-reflection of Si for different Si contents and at temperatures as high as 1075°C (Figure 2.3a and b respectively). The pre-reflection had an $S(Q)$ from 1.4 to 1.6 to nm$^{-1}$, which was identified as Si agglomerations with medium-range order. However, the diffractograms made at room temperature did not present the Si pre-reflection. The Si agglomerations were presumably in the form of Si$_4$ tetrahedrons and their characteristic bonding distance of Si – Si can be determined using the super imposed principle developed by Cervink [122]. W. Wang et. al [122] calculated the Si$_4$ tetrahedron distances using the Cervink method (the cube diagonal) and confirmed that the pre-reflection observed in X-ray diffraction at the different temperatures corresponded to the $S(Q)$ of the tetrahedral Si$_4$.

In other research Bletry et. al. [123] used a geometrical model to explain the structure of a binary liquid of amorphous alloy based on the first neighbour parameter and its atomic radius. The results of this analysis concluded that Si in an Al-Si alloy tends to segregate. The segregation of the Si atoms at high temperatures takes place in the micro-scale due to the strong bonding interaction of the Si – Si atoms. The segregation of the Si – Si atoms promotes the nucleation of primary Si particles. A similar phenomenon was observed by the segregation of C (graphite) in cast iron ingots and in the Na-Sn liquid melts, where for high concentrations of Sn aggregated tetrahedral covalent bonds of Sn atoms were identified [123].

The effect of Si modifiers such as Sb was studied in the liquid Al-12.5 wt% Si (an almost eutectic composition) using X-ray diffraction. These results showed that Sb promotes the agglomeration of Si – Si covalent bonds that result in the multiplication of Si – Si agglomerates (Figure 2.3b). The Si – Si agglomerations at temperatures below liquidus act as nuclei for primary Si particles, therefore by multiplying them a refining effect of the resultant primary Si particles is observed. The nucleated primary Si particles under the above-mentioned conditions presented different shapes and sizes in a solid state when compared to the primary Si particles of a reference melt. Numerical simulations show that in pure liquid Si, 25-30% of the atoms have covalent (Si – Si) bonds [125].

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Chapter 2. Literature Review

Figure 2.3. a) Structure factor \( (S(Q)) \) of the Al-Si alloys with different compositions at 670°C b) radial distribution function of the Al-12.5 wt% Si alloys with and without Sb at various temperatures [117, 122, 128, 219].

The analysis of the nearest neighbouring atomic distances and the average atomic densities of Al-Si binary alloys of various hypo- and hypereutectic compositions at different superheat temperatures was measured by W. Wang et al. [127]. The nearest neighbour atomic distance changes slightly with the content of Si, which means that the melt maintains the structure of pure Al. The density of Al-Si hypo- and hypereutectic alloys in a liquid state increases with the concentration of Si; this implies that some of the free volume in pure Al (interstitial sites) is occupied by Si atoms. For Al-Si concentrations close or above the eutectic composition, agglomerations of Si\(_4\) covalent bonds are promoted (tetrahedral); therefore, more abundant Si\(_4\) agglomerates were observed. Because Si has a low coordination number (4), the formation of Si\(_4\) agglomerates increased the amount of “insoluble” Si resulting in the reduction of the density of the liquid Al-Si melts close to the liquidus temperatures. Conversely, by increasing the superheat temperature the density of the Al-Si hypereutectic compositions increases because the Si – Si bonds decompose into Si atoms that diffuse into the free volume of the Al enriched melt [127].
Chapter 2. Literature Review

Figure 2.4. Total structure factor ($S(Q)$) of the liquid melts for the a) Al-Cu alloys at 700°C, b) Al-Fe alloys at 1550°C, c) Al$_{80}$Ni$_{20}$ alloy at different temperatures and d) Al$_{90}$Ni$_{10}$ alloy at different temperatures [219].

Xiufang et. al. [219] found that different alloy compositions present the pre reflection indicating the evidence of the medium range order agglomerations. Figure 2.4 shows the X-ray diffraction results of the determination of the $S(Q)$ for different compositions of the Al-Cu at 700°C, Al-Fe at 1550°C and Al-Ni at different compositions and temperatures.

Figure 2.4 shows that the intensity of the pre reflection depends not only on composition but also at the temperature at which the X-ray diffraction was conducted. The pre reflection in the tested alloys is the evidence of a possible formation of chemical compounds or attributed to the
agglomeration of heteroatoms. According to the Al-Fe phase diagram there are intermetallics with nominal compositions of FeAl₂, Fe₃Al5 and FeAl₃; from which Fe₂Al₅ has the highest melting point (1171°C). Based on quantum mechanics calculations it was determined that Al and Fe atoms have very strong attractions in solid state (Figure 2.4b). Therefore, it can be concluded that the atomic distribution of Fe and Al in liquid state remains similar at low degrees of superheating. Comparable to the Al-Fe alloys, Al-Ni alloys forms the NiAl₃ and Ni₂Al₃ compounds and there is a strong interactions at temperatures close to liquidus (Figure 2.4c).

2.2.2. Solidification Pathway of the Al-Si Hypereutectic Alloys

The solidification of Al-Si hypereutectic alloys begins with the precipitation of the primary Si particles at the respective liquidus temperature of the particular composition. The second phase precipitated is a small volume fraction of a dendritic Al phase (α-Al halo). Quasi-dendritic Al crystals nucleate in the vicinity of the primary Si crystals or any other inoculants and grow together with the primary Si crystals. The dendrites become coherent before the precipitation of the Al-Si eutectic (Dendrite Coherency Point (DCP)). The fraction solid at the DCP is low and occurs in a long period. This long coherency time is related to the large amount of latent heat released from the precipitation of Si crystals since the latent heat to melt Si is considerably high. The high latent heat combined with the primary Si particles morphology (polyhedral) are the reason for this alloy’s good fluidity. The Al dendrites that form surrounding the primary Si particles grow very fast (in comparison to Si) and start to interact with each other very early by temporarily bridging with each, however, significant strength is not developed. At the coherency point the strength increases rapidly in step like increments up to the eutectic temperature [77].

The α-Al halo formation is an effect of the presence of temperature gradients due to the solidification of Al-Si hypereutectic alloys under non equilibrium conditions (high cooling rate). The above-mentioned undercooling increases together with the amount of Si and is more visible in a cooling curve, for example, in compositions beyond the eutectic. It was estimated and confirmed experimentally that Si particles that grew at a rate of 40 μm/s or lower, solidified far from the coupled eutectic; therefore, the α-Al halo was not developed. However, other researchers found that the critical growth rate for the α-Al halo formation can vary from 80 to 100 μm/s [126].
During the formation of the Al-Si eutectic phases, the Al5FeSi phase also precipitates. Mg and Cu enriched phases nucleate at lower temperatures than the Al-Si eutectic. Additions of Si result in the precipitation of larger amounts of primary Si particles and the delay of the coherency point. Also, the primary Si particles become coarser, but this does not affect the rest of the precipitation reactions [77].

2.2.3. Thermal Analysis of Al-Si Hypereutectic Alloys

Thermal analysis is a powerful tool that has been successfully used to determine characteristic temperatures, fraction solids and latent heats during the solidification of castings under natural heat exchange conditions. Thermal analysis has been applied effectively for Al-Si hypoeutectic alloys (see Table 2.1) [10, 41, 76, 136, 177 - 183, 191 - 199]. The IRC in Metal Casting Technology is one of the world leaders in characterization of Al-Si hypoeutectic and Mg alloys. Some of the IRC’s contributions in thermal analysis are the development of a unique algorithm to determine the baseline and fraction solid using the Newtonian method to determine the first derivative [182, 183]. Additionally, the IRC has used thermal analysis for on line prediction of the level of microstructure modification of the W319 alloy using chemical and electromagnetic stirring modification techniques [16, 198, 199] as well as novel heat treatments and the development of casting processes [13, 168 - 171]. The present project is the beginning of the IRC’s involvement in the thermal analysis of Al-Si hypereutectic alloys combined with the effect of melt treatments on cooling curves. Griffiths et. al. [39, 41] reported thermal analysis results for ES applications, but unfortunately, the thermal analysis results lack the required high accuracy.

Unfortunately, Al-Si hypereutectic alloys have not been extensively studied using thermal analysis. For instance, Bäkerud et. al. [76, 78] have reported the most complete characterization using thermal analysis and SEM/EDX on two 390 Al-Si hypereutectic alloys. However, they did not provide a detailed description of the methodology used to determine the first derivative. Additionally, there is not a clear understanding of the assessment of the fraction solid curve, since the algorithm to assess the baseline is not provided. A summary of the solidification cooling curve analysis and reactions of the 390 Al-Si hypereutectic alloys is presented in Figure 2.5 and Table 2.1 respectively.
Figure 2.5. Thermal analysis data collected in an Al-Si 390 alloy test samples solidified at a cooling rate of 0.5°C/s. a) Cooling curve and its first derivative (dT/dt), b) calculated relative rate of solidification and fraction solid versus time, c) temperature versus fraction solid and d) relative rate of phase formation versus fraction solid [76]. Note: reverse order between the dependent and independent variables in figures c and d.

Nafisi et. al. [78] used thermal analysis as a tool to determine the optimum amount of eutectic and primary Si modifiers (Sr and/or P respectively) for a required level of microstructure modification. They reported that the liquidus temperature is affected by the refining effect of the Si modifiers, which is in agreement with the findings of Djurdjevic et. al. [176]. Additions of modifiers above the required level cause the lowering of the liquidus temperatures, therefore the amount of modifiers that correspond to the highest liquidus temperature is considered the ideal to reach the maximum level of modification [78]. Apelian et. al. [79] determined using thermal analysis that a depression of ≥5.5°C of the liquidus temperature correspond to a the chemical modification of > 90% of Si in the 356 Al-Si alloy, [79].

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2.3. Structure Modification Techniques for Al-Si Alloys

2.3.1. Chemical Grain Refinement

The use of grain refiners for Al-Si alloys significantly improves the strength, ductility, castability, machinability and fatigue performance of Al-Si sand castings. The utilization of grain refiners mainly allows for the elimination of undesirable large dendritic grain structures. The most usual grain refiners for Al-Si alloys are the Al-Ti and Al-Ti-B usually added into the melt in the form of master alloy rods or wires prior to casting. Therefore an ideal master alloy for grain refinement must have a high reactivity. Kinetics analysis of the dissolution of grains refiner master alloys was performed by the IRC in Light Metal Casting Technology showing that some grain refiners have dissolution rates as low as ~4 to 6 s [118]. Large holding times after the addition of the grain refinement have undesirable effects due to the decomposition of the TiAl3. Once the TiAl3 is decomposed it can not be easily reactivated resulting in a lower refining effect and sedimentation [133]. The presence of Boron in the Al-Ti master alloys improves the refinement ability of the TiAl3 particles. Unfortunately, the refinement effect using Al-Ti-B enhances the pore formation as folded oxides [48]. Therefore, excessive amounts of Al-Ti-B master alloys are detrimental to the mechanical properties.

2.3.2. Chemical Modification of Si in Al-Si Hypereutectic Alloys

Phosphorous (P) is usually added into the Al-Si hypereutectic alloys as a primary Si modifier. P is usually added in the form of Al - P, Al - Cu - P or Cu - 8 wt% P Al - Fe - P master alloys [100, 101]. The Al - Cu - P [95, 100] and/or Cu - P [77] master alloys are known as the best master alloys to modify primary Si. Additions of P transform the primary Si from the star-shaped into polyhedral and the number of primary Si particles is increased up to three (3) times. Therefore, the neighbouring distance between particles is reduced ~50% [100]. The refinement effect is accomplished due to the formation of the high melting point Al - P compound that acts as a heterogeneous nucleus, and permits Al - P/primary Si epitaxial growth [78, 95, 100]. Additions of inoculants such as Ca create a more effective refinement of primary Si particles and reduces the incubation time [102, 103, 184]. Refinement of primary Si improves yield and tensile strengths, wear resistance and elongation [95, 104].

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Chang et. al. [105] supports the theory that the nucleation of the primary Si particles takes place on the Al - P compound. This compound forms at temperatures as high as 982°C which is well above the liquidus temperature of most Al-Si alloys. The AlP compound has a cubic crystalline structure with a lattice parameter of 5.45 Å that is very similar to the diamond like crystalline structure of the primary Si (a = 5.43 Å) and that has the effect of a heterogeneous nuclei. The Al-Ce compound plays a similar role as the Al-P to nucleate the primary Si particles, but, Al-Ce has an orthorhombic structure with lattice parameter $a=9.27\text{Å}$, $b=7.68\text{Å}$ and $c=5.75\text{Å}$. Therefore, primary Si particles grow epitaxial to the “c” direction of the Al-Ce [78, 105].

Combinations of Si modifiers (Sr + P) were investigated as an alternative method for chemical modification of Al-Si hypereutectic alloys [95]. As a result the microstructure of the Al-14 wt% Si alloy presented a microstructure comparable to the eutectic composition. Using 50 ppm of P and Sr in the Al-18 wt% Si the opposite effect was observed (primary Si particles became coarser). Additions of more than 300 ppm of Sr have no significant effect in either case. The refinement of the primary Si particles with P and Sr had an effect on yield strength, tensile strength and elongation; however there was not specific combination that increased the three properties at the same time [95].

Additions of rare earth elements such as Ce, La, Nd and Pr also have a refining effect on Al-Si hypereutectic alloys [105 - 107]. The morphology of the primary Si particles has a dependency not only in the master alloy additions but also on the silicon content. Additions of up to 3 wt% of a master alloy containing La, Ce, Nd, Pr and Pa showed successfully results in refining of the microstructure of Al-Si hypereutectic alloys containing up to 20 wt% Si. In fact, an addition of ~1wt% of rare earth elements is enough to create this morphology transformation; however, a substantial refinement is achieved using master alloy additions of > 2 wt%. Also the morphology of eutectic Si is affected and changes from plate like to refine fibrous. However, the effect of La as a primary Si modifier is limited to binary Al-Si compositions, because in the presence of copper, the $\text{Al}_7\text{Si}_7\text{Cu}_2\text{La}_{1.5}$ intermetallic is formed and has a negative effect; such as the increase of two dimensional defects (twins or stacking faults) [107].
2.3.3. Thermal Modification of Al-Si Alloys

Two kinds of thermal modification are the most commonly applied for Al-Si alloys (superheating and varying cooling rate) and their combinations. Al-P is known as a high temperature nucleant for primary Si which at superheating temperatures of up to 500°C reduced in size. This resulted in a negligible effect on the microstructure. Vives et. al. [57] found that solidifying pure elements (such as Tin or Al) from different superheat temperatures changes only the orientation of the microstructure. On the other hand cooling rates from <0.01°C/s to 20°C/s reduce the interdendritic spacing from ~0.1 to ~0.02 μm respectively. The combination of the superheat temperatures (500°C) and cooling rate is effective only for cooling rates >20°C/s to refine the interdendritic spacing. Si phases (primary and eutectic) and grain size can be refined as the superheat temperature increases. Also, isothermal holdings at various superheat temperatures starting from 3°C up to 300°C above liquidus have an effect on reduction of interdendritic spacing and intermetallic compound size [108, 109]. Additionally, isothermal holdings at temperatures slightly below liquidus and rapidly cooling rates transformed the primary Si particles from blocky to petal shapes [110]. Bergsma et. al. [49] found that the effect of quenching 356 and 357 Al-Si alloys (cooling rate = from 15 to 30 °C/s) in liquid or semi-solid states has a similar refinement effect on the microstructure as melt stirring.

2.3.4. Effect of Melt Stirring Techniques on Microstructure Modification

Stirring techniques have been used since the 1960’s for the following purposes. Degassing, grain refinement, mixing, desulfuring iron, removing inclusions and passivation of iron. The most common stirring technologies are mechanical, ES and ESV [27 - 37]. ES and ESV techniques have been well accepted for melt treatments of ferrous and non-ferrous alloys. The rotation of the alloy in a semi-solid state creates shear stresses that break down dendrites, which from a new nucleus for a new grain. Resulting in smaller grain size and secondary dendrite arm spacing (SDAS) that improves feedability and reduces shrinkage porosity [27 - 73].
2.3.4.1. Mechanical and Ultrasonic Stirring

Spencer et. al. and Moon et. al. [111, 112] conducted research using Sn-15 wt% Pb and Al-6.5 wt% Si alloys respectively to measure shear stresses of the alloys at different fraction solids. They applied mechanical stirring in a semi-solid state and found that the shear stresses in mechanically stirred test samples were reduced hundreds of times as the intensity of the mechanical stirring increased. Castings solidified under these conditions were dendrite free and due to the rheological conditions during the solidification process are known as rheocasting.

The use of mechanical or magneto-hydrodynamic stirring and intensive ultrasonic treatment during solidification reduces or eliminates shrinkage, gas cavities and dendrite cells in a wide range of Al-Zn-Mg-Cu alloys (from hypo to hyper eutectic) [52]. This ultrasonic treatment created three (3) microstructure refining effects. Cavitation, which activates insoluble particles such as, oxides and intermetallics and fragmentation of Si particles as well as dendrites. The impact in mechanical properties of pure elements using this ultrasonic technique increases tensile strength 35% (from 54 to 72 MPa), hardness 14% (from HB 17.2 to 19.7) and elongation from 48 to 52%. Particularly the strength of the Al-Zn hypereutectic alloys increases moderately while hardness decreases but the plasticity increases up to 3.5 times [52].

Vives [56] designed technologies to produce sonic, ultrasonic stirring and mechanical vibrations using a piezoelectric transducer or a magneto-hydrodynamic resonator. The transducer and resonator were connected to a rod that is immersed in the solidifying Al alloy inducing waves with frequencies of up to 25 kHz. This ultrasonic treatment increases fluidity, refines the grain, depresses dendritic and columnar zones and can be used for degassing purposes at high frequencies due to the cavitation phenomena that collapses gas bubbles. The degassing conditions took place by way of a cyclic compressive mechanism of the gas bubbles (hydrogen) present in the solidifying Al alloy. The cyclic compressive mechanism creates cavitation that imploded the gas bubbles pressurizing them up to a few thousand atmospheres. The imploded gas bubbles reach a critical pressure that forces them to collapse and projects them upwards.
2.3.4.2. Introduction to Electromagnetic Stirring (ES) of Semi-Solid Alloys

ES is a technique that has been used for the refinement of the microstructure of different ferrous and non-ferrous alloys in a semi-solid state [29 - 60]. Due to the complexity in the developments of technologies for ES the authors in many cases protect their inventions with patents [61]. For more detailed information about the patents the Master's thesis of J. Ohl entitled “Development of a Novel System Used for Electromagnetic Treatment of Aluminum Alloys during the Solidification Process” can be referred at [21].

The basic principle of the ES process is to rotate the melt within a magnetic field, consequently a Lorentz force develops as a result of the interaction of fluid motion and the magnetic field producing electromagnetic stirring. Therefore, the solidification process combines both centrifugal and electromagnetic fields [21]. The rotation of the liquid alloy creates centrifugal, gravitational and Lorencial Forces [42]. Eq. 2.1 shows the Lorentz forces created during the ES treatment. At the industrial level the most common apparatus used for electromagnetic stirring are linear and rotary stirrers, which are fundamentally an electric motor that uses a rotating magnetic field to spin a semi-solid alloy [50].

\[ F_L = J \times B \]  

Eq. 2.2

Where: \( F_L \) are the Lorentz forces (N/m\(^3\)), 
J is the current density, (in A/m\(^2\)) and 
B is the magnetic field intensity (in Tesla (T))

2.3.4.3. Examples of Electromagnetic Stirring Techniques

Application of the melt spinning technique has become the most popular for rapid solidification processes to produce advanced materials. Also the single-roll strip casting is a similar technology used for near-net-shaped thin strips (thickness = 1-10 mm) or ribbons (thickness <1 mm) of microcrystalline or amorphous materials. The planar-flow casting process consists of the injection of the alloy through a slit nozzle at the end of a pressurized crucible [75]. The intensity of the electromagnetic field and the coil designs (shape and location of the coils) permits the control of the meniscus and ingot surface for continuous casting and twin-roll casting. This is possible by manipulating the momentum balance of pressure and surface tension [75].

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In general the ES apparatus has been used for different applications, but in most cases the ES apparatus consists of a cooling system (outside of the mould, to avoid damage to the electromagnetic coil(s)), a heating system (to control the temperature in the liquid metal) and an electromagnetic field source (stator or magnet(s)). The electromagnetic source is commonly placed outside of the cooling system, that creates a magnetic field. Figure 2.6 presents three different apparatus used in ES for semi-solid melt treatments and composites production (Griffiths et al. [39], Vivès [45] and Sunh-Chul et. al. [47]).

Griffiths et al [39, 41] conducted research studies on Al alloys containing 8.5 wt% Si and 2.5 wt% Si that were superheated to 80°C above their respective liquidus temperatures. When the alloys reached a temperature of 40°C above liquidus they were water cooled (water rate: 1000 L/min) and different currents were applied into the coils (1, 2.5 and 5A) during their solidification. The apparatus used by Griffiths is shown in Figure 2.6a. Thermal analysis results show that the liquidus temperatures change as a function of the ES conditions (i.e. at 0 and 1 A of current applied into the coils during solidification, the liquidus temperature was observed at T = 630 °C, whereas for 2.5 and 5A of current the liquidus temperature dropped to 615°C). Thus a narrower solidification zone was observed. The analysis of the microstructure of the ES treated test samples shows an increase of the grain equiaxed zone from 26 and 60% for the ES treatments using 1 and 5A respectively. Also a reduction in macrosegregation in all directions was observed.

Zhang et. al. [42, 46] solidified Al-Si-Cu alloys in a rotating cylindrical mould with a hollow centre. The test samples in this apparatus are solidified in the presence of a DC magnetic field that can be induced in the centre of the test samples or outside. The rotating speed can vary from 1200 to 2000 rpm and the magnetic field intensity from 0 to 0.23T. The induction of the magnetic field from the centre of the test sample created non-rotational conditions of the liquid alloy; therefore, the refining effect of this type of melt treatment is almost negligible. Conversely, by inducing the magnetic field from the exterior of the mould the alloy was rotated and the refinement of the microstructure was more effective. For instance, Al-Si hypereutectic alloys solidified in the presence of magnetic fields larger than 0.1 T showed refinement of the primary Si particles and the Al-Si eutectic was transformed into a rod-like eutectic structure. Figure 2.7 shows micro and macrographs of the top view and in the profile zone for the different stirring conditions.
Desnai et al. [53] used a device similar to Griffiths' (Figure 2.6a) but without the cooling system. This research was conducted using multi-component Al alloys. Two main factors were identified as the most important in the modification of the microstructure, the effect of stirring intensity and the alloy composition. Relatively low AC currents (50A) applied during the ES treatments did not have an effect on the microstructure; hence the test sample had a similar microstructure as a reference test sample. Whereas a moderate current (350A) had the same effect as conventional ES techniques and a current of 600A resulted in vigorous agitation of the alloy and consequently, a highly refined microstructure. On the other hand, the alloy composition had an influence in the liquidus temperature and on the cooling rate. This research was carried
out using several types of Al alloys from the high purity Al (1050 AAA) to the 2XX, 3XX, 5XX, 6XX and 7XX AAA series of Al alloys.

Figure 2.7. Micro and macrostructures of Al-Cu alloy test samples cast under different conditions (a, b) 0.0T, (c, d) 0.1T, and (e, f) 0.23T [42, 46].

It is unusual that ES conditions are reported as a function of voltage. Most of the researchers used either current or magnetic flux intensity as main parameters. In fact, voltage and current are parameters that depend on each other in order to obey Ohm's law. Sunh-Chul et al. [47] reported ES parameters as a function of voltage. A schematic image of their apparatus is provided in Figure 2.6b. ES treatments applied using voltages of up to 160 V did present significant changes in the microstructure of the Al-7 wt% Si. However, by using 180V and 200V the resultant microstructure exhibited considerable reductions in grain size and more globular dendrites. The application of these melt treatments was conducted under isothermal conditions using a fraction solid of 0.2 and 0.5%. The analysis of the microstructure shows that the higher the fraction solid the more globular the microstructure and the presence of inclusions is reduced.
In the research conducted by Yang et. al. [54] the 356 Al alloy was ES treated during the solidification process. The 356 Al alloy was ES treated at constant fraction solids and cooling rates, but with different ES times and the rotation speeds (39, 95, 119, 142 and 178 s\(^{-1}\)). Using a rotation speed of 178 s\(^{-1}\) it was found that the microstructure became non-dendritic, with lower shrinkage porosity and the solidification time was reduced. The reduction in grain size was from 82 to 46 \(\mu m\) for the ES treated test samples at rotation speeds of 95 and 142 s\(^{-1}\) respectively. Short ES times (30s) had an almost negligible effect on the grain sizes.

Vives [60] developed an ES technology using permanent magnets that were placed azimuthally in a rotor of an electric motor. This ES technique is suitable for high temperature applications. The rotor with the magnets was introduced into a crucible and was made of stainless steel with a 22 kg capacity of Pb - 12 wt% Sb alloy (\(\rho = 10.44 \text{ g/cm}^3\)). The power was controlled by an autotransformer from 0 to 3000 W; the rotor had 3 pairs of poles. Two different types of permanent magnets were used one of them Recoma 20 and the other one Alnico 1550. The Recoma 20 magnet used had a 28 x 114 mm cross section and was 11 mm in height with 0.9 T of residual magnetism. The dimensions of the Alnico 1550 were 24 x 24 x 30 mm with 1.26 T of residual magnetism. Figure 2.8 shows the diagrams of the apparatus, the rotor with the magnets and the magnetic field distributions [60]. The semi-solid ES treatments conducted in the Pb - 12 wt% Sb was performed for temperatures 10°C below liquidus (286°C) and lower. The speed of the rotor varied from 0 to 3000 rpm. During the ES treatments was observed that fractions solids below 0.35 create a turbulent flow of the semi-solid Pb-12 wt% Sb and with higher fraction solids the flow of the alloy was more stable [60].

Comparing the apparatus designed by Zhang et. al. [42, 46] and Vives [60], both have the capabilities to induce the rotation of the melt from the centre of the test sample. Zhang et. al. [42, 46] reported that this type of system was non-rotational; conversely Vives [60] reported a rotational system with a considerable level of refinement of the microstructure. Two main reasons are the success of Vives [60] device, the application of the ES treatments in the presence of higher magnetic intensities (from \(~2.5\) to \(~4.5\) times higher) and the fact that this device rotates the rotor instead of the mould containing the liquid alloy.
2.3.4.4. Electromagnetic Stirring and Vibration

Vivès [35, 56 - 60] in several research publications showed the results of solidifying semi-solid ferrous and non-ferrous melts in the presence of combined AC and DC electromagnetic or electric fields. By allowing the interactions of the above-mentioned fields within the solidifying melt the collective effect of the fields is created. The collective effect of the combined AC and DC fields creates more severe stirring conditions by creating the interaction of the waves of different fields. These interactions create vibrations at low, high or variable frequencies.

*Working Principle*

This principle consists of the use of two magnetic fields, a stationary one \( (B_0) \) and a variable one \( (B) \) in the vicinity of the molten metal. Both the magnetic and the electric fields are parallel to the vertical axis of the ingot. The interaction of the magnetic fields makes a convex meniscus at the top of the liquid metal (Figure 2.6). The interaction of the magnetic fields produces two components one vertical (rotational) and the other radial (non-rotational). The
interaction of both components is responsible for vibration during the stirring process. Figure 2.9a shows a diagram of the electromagnetic fields used to produce stirring and vibration [35].

The work principle of this system consists of the simultaneous application of a stationary magnetic field \( B_0 \), and a sinusoidal electric current. The sinusoidal electric current can be low or high frequency (N) with a respective sinusoidal current of intensity \( i = I \sin t \) (Where \( I \) is the intensity of the current applied, \( t \) is time and \( i \) is the intensity of the current at the time \( t \)). Vivès [58] In this research a low frequency current applied perpendicularly to the direct magnetic field in a tank during the solidification of the alloy was used (Figure 2.9b).

The interaction between the external direct magnetic field \( (B_0) \) with the variable electrical current of density \( \text{j} \) gives rise to a vertical periodic vibration electromagnetic force \( (\text{j} \times B_0) \). The vibration force has a frequency \( 'N' \) that forces the molten solidifying alloy to vibrate. The vertical electromagnetic vibration force (expressed as pressure) covers the entire volume of the solidifying alloy and is given by:

\[
P = \frac{B_0 \cdot I \cdot L}{a} \sin \omega \cdot t \quad (\text{N/m}^2)
\]

\text{Eq. 2.3}

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Vives [35] combined AC and DC electromagnetic fields to produce sonic and ultrasonic vibration. Using currents varying between 0 and 165A a magnetic field intensity from 0 to 0.1T was applied to solidifying melts of the 2024 and 1050 Al melts. At the same time a variable electrical current between 0 and 465A was passed through the semi-solid alloys with liquidus temperatures of 660°C and 664°C, and solidus temperatures of 485°C and 635°C respectively. The ESV semi-solid treatments were applied at 15°C and 8°C below liquidus [35].

The profile of the ESV treated melt presented different characteristics as a function of the magnetic field intensities. A current of 216A turned the melt into a parabolic top edge. To understand the phenomenon taking place during the melt treatment, Vives [35] conducted experiments varying various parameters at the same time. Several parameters were varied at the same time, but their resultant values correspond to a constant dimensionless numbers such as the Hartman one. The Hartman (\(M\)) number involves viscosity (\(\eta\)), electric conductivity of the liquid (\(\sigma\)), an externally imposed magnetic field (\(B_0\)) and its height (\(z\)) and the temperature in the following relationship:

\[
B_0 H \left( \frac{\eta \sigma}{j} \right)^{\frac{1}{2}}.
\]

The purpose of this type of experiment was to demonstrate that a constant \(M\) can be achieved by using different intensities on the above-mentioned independent variables. This resulted in a more discrete methodology to conduct melt treatments allowing a more detailed analysis of the microstructure.

The reference test sample of the 1050 Al showed columnar grains, the ESV melt treated test sample of the same alloy presented quite the opposite microstructure. Using the ESV treatment the level of microstructure modification achieved was the highest in comparison with the effect of mechanically or ES treated test samples. However, the refinement was not as effective as for alloys with a higher amount of alloying elements [59].

The characteristics of the ESV device shown in Figure 2.10 consist of a crucible of 2 x 4 x 12 cm of stainless steel, a cooling and a heating system. The combination of adjustable cooling and heating systems serves to regulate the rate of heat extraction and consequently, the cooling rate. The cooling rate is the relationship between the temperature ranges the time from liquidus to solidus. Three (3) coils placed in parallel were used, one on top of the other. Through the central coil AC current with a frequency of 50 Hz was used to create a magnetic field along the sample. Both AC and DC currents were modulated from 0-3500A. For these currents the magnetic field...
varied in the range of 0 to 0.7T. The interaction of both magnetic fields created a current density of \( J = 4.38 \times 10^6 \text{ Am}^{-2} \), \( J \times B_o = 3.06 \times 10^9 \text{ Nm}^{-3} \), and a pressure of \( P = 122.5 \text{ kPa} \).

The microstructure results of the electromagnetic vibration showed the refinement of the structure even in the absence of cavitation (Figure 2.11). For this particular case experiments using 356 Al-alloy proved that the motion of the molten alloy was strong enough to keep the stirring metal pool at a constant temperature (isothermal). The electromagnetic pressure used had an amplitude of 0.35 bar. Once the test samples reached the liquidus temperature the above-mentioned pressure was applied for a period of ten (10) minutes and the cooling rate was 3.5\(^\circ\)C/min. A peak pressure of 116 kPa was imposed suddenly through a rapid increase of current intensity for a relatively short time period (15 s). The semi-solid alloy was rapidly cooled by water at a rate of 100 \(^\circ\)C min\(^{-1}\). This process yielded 500 grains in an area of 40\(\mu\)m\(^2\). In another electromagnetic vibration experiment the conditions were as follows; a negative pressure of 116 kPa was transmitted 50 times per second (Hz) for 12 min to the solidifying melt from the liquidus at a cooling rate of 4\(^\circ\)C/min. The result was a grain size of 50\(\mu\)m, which was smaller than that produced by the hydrodynamic effect in the absence of cavitation (see Figure 2.11) [56].

![Figure 2.10. The principle of producing electromagnetic vibration using a magneto dynamic resonator [56].](image-url)
2.4. Novel Engine Block Technologies Using Aluminum Silicon Alloys

2.4.1. Applications of Al-Si Alloys for Cylinder Liners

In 2002 the Ford Motor Company decided to utilize the cast iron AISI A4 for cast in cylinder liner applications of selected automotive engine blocks. The currently used cast iron cylinder liners have a chemical composition of 3-3.5 wt% C, 2-2.8 wt% Si, 0.5-1 wt% Mn, 0.1-0.4 wt% P, 0.12 wt% S max, 0.2-1.5 wt% Cu+Ni, 0.15 wt% Sb and $C_{Equivalent} = 4.45$ wt%. The hardness of the cast iron must be in the range of 205-273 HB or 90-104 HRB (ASTM E10). The A4 cast iron contains at least 50% of the type A graphite with grades between 4 and 7 and a maximum of 25% of graphite type D and E with grades between 4 and 8 (ASTM A247/D562). The matrix microstructure at the wear surface consists of fine lamellar pearlite with a maximum of 5% free ferrite and a maximum of 5% free carbides. If present, iron phosphide eutectic (steadite) must be fine, evenly distributed in a random pattern with no continuous network and shall not exceed 5% [139, 140]. These cylinder liners are used by the NEMAK of Canada Corporation at Windsor Aluminum Plant to cast engine blocks.
In order to develop low weight high performance materials, several Al-Si compositions including eutectic and higher Si composition (hypereutectic) alloys have been tested. Al-Si eutectic and hypereutectic alloys possess low thermal expansion, excellent castability and low density that make them attractive for automotive applications (cylinder liners, pistons and engine blocks) [139, 140]. However, there is still a lot of controversy between the roles of the primary Si particles present in Al-Si hypereutectic alloys. Primary Si particles increased the hardness of the alloy locally, thus its wear resistance; conversely, due to their brittleness they have the tendency to crack. The cracking of the primary Si particles exposes the soft Al matrix to severe wear conditions resulting in serious damage to the cylinder liners [142, 152]. Some investigations demonstrated that due to the brittleness of the primary Si present in Al-Si hypereutectic alloys; their wear resistance performance under certain experimental conditions was worse than Al-Si eutectic alloys for cylinder liner applications [139, 142].

The size of the primary Si particles is a key parameter to control the wear characteristics of Al-Si hypereutectic alloys [147]. Conversely, some researchers concluded that under different test conditions alloys with equal amounts of Si contents but coarser silicon particles demonstrated similar wear resistance as the alloys with finer primary Si particles [148 - 151].

Lasa et. al. [152] used different Al-Si alloys cast under different conditions and heat treatments to determine their wear resistance. The test samples were cast using permanent moulds, thixocast, squeeze cast and lost foam techniques. As primary silicon modifier phosphorous was added except for the permanent mould cast samples. The silicon content of the alloys varies from 12.5 wt% to 19.5 wt%. All samples cast in a permanent mould presented very coarse and heterogeneous distribution of the primary Si particles. On the other hand the primary Si particles of the thixocast are very fine and uniformly distributed. Figure 2.12 depicts microstructures of some of the test samples used in this research that were cast using different techniques; all microstructures were micrographed in as received conditions. The samples were heat treated following the T6 procedure; after the treatment the samples presented higher hardness and also the Si eutectic particles were slightly spheroidized [152].

As a result of the wear testing [151, 152] it was found that in all alloys the primary Si particles especially the biggest ones were damaged during the wear test. The damage to these particles is probably due to the collision with the abrasive hard particles from the disc. The heat treatment improved the wear resistance of the hypoeutectic test samples cast in a permanent
mould only. A rise in the temperature during the wear test makes the lubricant more fluid, thus a more extensive lubrication is observed. The speed of the disc affected the wear conditions even at a constant load [152].

The first successful attempt to eliminate cast iron cylinder liners was conducted by General Motors in the Vega 2300 vehicle. The engine block of the Vega 2300 was cast using an Al-Si hypereutectic alloy [155 - 158]. Al-Si hypereutectic alloys possess a combination of characteristics that make them these alloys highly desirable materials for combustion engine block applications [155]. More than thirty (30) experimental Al-Si hypereutectic alloys with different Si contents varying from 12 to 35 wt% Si were tested for the Vega engine blocks [158].

The results of the tests of all the experimental alloys showed that the optimal composition for the engine block was 16-18 wt% Si, 0.6-1.1 wt% Fe, 4-5 wt% Cu, 0.1 wt% Mn max, 0.45-0.65 wt% Mg, 0.1 wt% Zn max., 0.2 wt% Ti and no more than 0.2 wt% max. of others elements. This alloy is known by the AAA standard designation as the 390 Al-Si alloy [155]. Figure 2.13 shows the mechanical properties of the Vega’s alloy tested under different temperature conditions.

Figure 2.12. Microstructure of Al-Si hypereutectic alloys cast using different techniques (a, b) permanent mould, c) thixocast, d) lost foam and e) squeeze casting [152]. Note: in all cases coarse polyhedral primary Si particles.
Chapter 2. Literature Review

Figure 2.13. Mechanical properties of the Vega 2300 engine blocks' alloy tested under different temperature conditions a) testing at elevated temperatures after a holding of time of 1000 h b) testing after recovery at room temperature of the test samples exposed to specific temperatures for 1000 h and c) thermal conductivity of the alloy after different heat treatments [155 - 158].

After the machining of the engine block a special electrochemical treatment is applied to the bores. In this treatment a layer between 0.12 mm and 0.0025 mm in depth of Al is removed to expose the eutectic and primary Si particles, which in fact increases the wear resistance on the surface of the cylinder liner. At the same time the removal of the Al matrix created cavities that are filled with oil reducing the friction between the cylinder liner and the piston ring. The electrochemical treatment was performed in a sodium nitrate electrolyte passing a current of 62 A and 9 V between the anode (engine block) and the cathode for 9 s. Selected Vega engine blocks were analyzed. After running the engine blocks for 130,000 km, these engine blocks presented a thin (~0.04 mm) Fe enriched film in the bores. The thin Fe film was considered negligible allowing the engine block to be used for a longer time [156, 157].

A reduction of \( \frac{2}{3} \) of weight, a lower machining cost, less labour, increased thermal conductivity, a lower thermal expansion coefficient, increased wear resistance, high castability are some of the advantages of using this alloy for engine block applications [155 - 158].

As an alternative technique, the elimination of cast iron cylinder liners by coating the bores with ceramics or steels has already been tested at the laboratory level with promising results for industrial applications [159, 160]. The elimination of cast iron cylinder liners represents a
weight reduction in the engine block of up to 25% and increases the thermal conductivity. In order to lower the price of the Al-Si engines hypoeutectic alloy compositions can be utilized, however, some of the wear resistance is affected. Therefore, the application of ceramic sprayed into the thin films in cylinder bores seems to be a long-term solution that also increases the service life of the engine blocks [159].

Malaczynski et. al. [160] used diamond-like carbon coatings on the 390 alloys. They plated the surface of the bores of an Al-Si engine block, but there face two main limitations. The application of a plating process was very complicated and environmentally restrictive. Therefore, they studied diamond-like coatings, which were not precisely better because this process could not be applied in the automotive industry unless a mass production facility could be developed. Furthermore, this type of coatings can only be operated at temperatures below 220°C; otherwise there would be a risk of losing the original metallurgical properties. The thickness of the coating played an important role (i.e. lower thickness than 1.5 - 3 μm) and could be completely removed uncovering the 390 substrate. Conversely, is expected that a coating of more than 7 μm can cover the “life expectancy” of a modern combustion engine block (over tens of years or hundreds of thousands of miles). At temperatures of 400°C was observed the highest friction coefficient; nonetheless the coating withstood 11,000 cycles [160].

Edrisy [220] utilized two thermal spray deposition techniques to coat the bores of Al-Si engine blocks using the AISI 1020 plain carbon steel. These thermal spray deposition techniques were the Plasma Transfer Wire Arc (PTWA) deposition and the High Velocity Oxy-Fuel (HVOF) deposition. Using the HVOF deposition technique the AISI 1020 steel was doped with 2.5 wt% Al, which reduced the wear of the coatings. The wear rates of the HVOF (with the AISI 1020) were considerably higher in comparison to the PTWA deposition technique.

Using the HVOF deposition techniques the bores of a Corvette and a Saturn engine blocks were coated with AISI 1020-2.5 wt% Al and the wear mechanism were studied. It was found that the two engine blocks presented scuffing damage on the bore faces. The scuffing was presented in the form of tribolayers composed of nanocrystals of the deformed iron matrix with particles of inclusions. The delamination of the tribolayers was the principal source of material removal during scuffing, which was facilitated by crack formation at the FeAlO3 inclusions.
CHAPTER 3. PROJECT JUSTIFICATION

3.1. The Global and Canadian Automotive Industry; Present Status and Future Requirements

In 1974, the CAFE program was established by a single act of the USA Congress in the aftermath of the 1974 oil embargo. The CAFE program ordered automakers to increase fuel efficiency of the nation's cars and light trucks [161, 162]. Based on the U.S. Environmental Protection Agency's policy the fuel consumption in a vehicle should be reduced by ~ 53% and by ~ 20% in trucks [166]. From 1978 to 1991 the fuel efficiency in internal combustion engines increased by ~ 40% (from 8.5 to 11.9 Km/L of gasoline) keeping the engine's performance constant. However, since 1991 the fuel efficiency has not improved. Improving fuel economy is a worthy goal to reduce the dependence on oil imports and reduce emissions (mainly CO, Hydrocarbons, NOx and particulate matter) that contribute to global warming. All these atmospheric pollutants are associated with a wide range of health problems; including cancer and environmental risks [162, 166].

Safety, insurance fees and people's driving habits are opposite to the car's weight reduction. Heavier vehicles appear to be safer, particularly those over 4,000 pounds [165]. i.e. J. R. Haley [164] reported that from 1975 to 1990, 46,000 unnecessary deaths occurred in light weight cars that could have been avoid in larger or heavier cars. It is for this reason that car modifications should be weight neutral (low-friction lubricants, multi-valve overhead camshafts, variable valve timing, cylinder deactivation, high-speed automatic transmissions, and improved rolling resistance). By 1987 although fuel economy improved substantially, total gasoline consumption fell by only 2.5 percent in the U.S.A. [163]. This did not decrease the oil usage, merely held it steady.

Alternative technologies such as hybrid engines (with fuel efficiencies of up to 140 km/L on the city and 100 L/km on the highway) and fuel cells are still long term solutions. The costs of switching from today's petroleum/diesel fuel infrastructure to one that supports either hydrogen or electric cars would be enormous and would take decades to put into place [167]. A hybrid car is from $ 8000 to $ 10,000 (CAD) dollars more expensive than a regular vehicle and fuel cells are

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still under investigation. It will take many years for them to be implemented in regular consumption vehicles.

3.2. Metallurgical Limitations of Using Dissimilar Materials for Engine Block Production Using Cast-in Cylinder Liners

Improvements in advanced engine block components are one of the challenges for the Ford engineering community. The NSERC/Ford-NEMAK/University of Windsor Industrial Research Chair (IRC) in Light Metals Casting Technology investigated the bridge cracking problems in the cylinder of the 2.3L I4 engine block (Figure 3.1) manufactured at the Ford Cleveland Aluminum Casting Plant. These engine blocks were cast as prototypes to produce more compact engines with bridges (distance between cylinder liners) having a length of 4 mm. The bridges in a regular I4 2.5L engine cast are 8 mm in thickness. The cast V6 engines at Windsor Aluminum Plant have bridges of 14 and 9 mm respectively; this data is important since in this dissertation prototype V6 engine blocks were cast using Al-Si hypereutectic cylinder liners.

The prototype I4 2.5L engine blocks had several cracks along the bridges and bulkhead sections. The IRC conducted research on these engine blocks and the result of the metallurgical analysis indicated the following as the main sources for the occurrence of cracks [185, 187]:

- Solidification of the engine block with cast iron cylinder liners. The difference in thermal expansion/contraction coefficient between both materials ~ 42%.
- Geometry of the slab type riser.
- Utilized of heat treatment resulted in detrimental residual stresses
Chapter 3. Project Justification

Figure 3.1. Top deck of the engine block cast at Ford Cleveland aluminum casting plant. Arrows showing cast-in cast iron cylinder liners and the thin bridge (4 mm) regions where cracks occurred [185, 187].

3.2.1. Residual Stress Effect Due to the Solidification of Materials with Dissimilar Thermal Expansion Coefficients

Using the Universal Metallurgical Simulator and Analyzer (UMSA) the effect of remelting hybrid (W319 Al alloy and cast iron) test samples was studied during the solidification process. Hollow (rings) W319 Al alloy test samples were machined and a cast iron insert (extracted from the NEMAK cylinder liners) was pressed in to create the hybrid UMSA’s test samples. Also numerical simulations were carried out based on the dilatometry results and using the press fit stress analysis model to determine the intensity of the stresses [186].

The UMSA test samples with the cast iron inserts were re-melted in the UMSA and then solidified under natural heat exchange conditions where a series of micro-cracks developed during solidification. Also, the result of the numerical simulation indicates that the residual stresses in the 319 Al ring were up to 320 MPa, which is far higher that the yield strength of the 319 alloy. Consequently, the 319 alloy under these stress conditions should present cracks. Figure 3.2 presents SEM micrographs of the cracks observed in the metal matrix of the Al alloy as well as the possible detrimental effect due to the metallurgical reactions between molten Al and cast iron.
3.3. Interface Analysis between Cast Iron and the W319 Alloy

The IRC conducted research on the detrimental effect of using cast in cast iron cylinder liners on the V6 engines cast at Windsor Aluminum Plant using the W319 alloy. The cylinder liners prior to casting were cleaned with steel balls (shot blasting). The shot blasting left Fe rich residues on the surface of the cylinder liners; the Fe residue promoted the formation of AlFe brittle crystals that created a poor metallurgical interface with a high density of microcracks [187].

Another negative effect found in the production of the V6 engine blocks with cast iron cylinder liners was that the flow of the melt during the mould filling in some cases transported oxides (spinels). These spinels dragged zirconia sand to the cylinder liner sections. Once the spinels were in direct contact with the cylinder liner, they entrapped the Fe rich contaminants that acted as a barrier that prevented the formation of an interface without gaps between the liner and the W319 alloy (Figure 3.3) [187].

Figure 3.2. SEM/BSE micrographs of the interface of the W319 alloy test samples (1) and the cast iron (2) interface with numerous microcracks developed during the solidification (3) of the test samples due to the differences in thermal expansion coefficient at two magnifications a) 220 X and b) 1000 X. Note: iron rich layer interface developed during solidification (4) [186].

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3.4. Justification of this Research

In this Chapter an analysis of the disadvantages of using cast iron cylinder liners in Al engine blocks is presented. For instance, since 1970's the application of Al-Si hypereutectic alloys was used as a potential candidate to eliminate cast iron cylinder liners [158]. The use of Al-Si hypereutectic alloys offer up to a 15% reduction of the total weight of the engine block. This is considerable in the engine block, and is only ~2% of the overall weight of a car. By reducing the car's weight by only 2% this modification is considered weight neutral. The Car Average Fuel Economy (CAFE) standards suggest that weight neutral changes, which increased the fuel efficiency (i.e. fuel injection) keeping constant the weight of the vehicle, are the most suitable [161-167]. Al-Si cylinder liners are also appropriate in hybrid engines that in turn result in short and long term savings.
Al-Si hypereutectic alloys have demonstrated similar wear resistance in comparison to cast iron. It is for this reason that these alloys are the ideal lightweight candidate to substitute cast iron for liners applications. However, the presence of coarse primary Si particles is the main limitation of these alloys due to its brittleness. Every time a primary Si particle wears out and fractures it uncovers an α-Al enriched region, which is very soft and susceptible to wear. Therefore, in order to produce Al-Si hypereutectic cylinder liners, the undesirable primary Si particles need to be eliminated. For this reason in this dissertation was proposed the development of a novel melt treatment capable for the refinement of Si. This melt treatment needs to be effective in liquid state in order to be able to cast the cylinder liners using the treated melt. Additionally, it is expected that by refining the primary Si particles the homogeneity will be increased and the amount of Al-Si eutectic will be considerably increased. This will result in a dramatic improvement of the wear characteristics of the Al-Si hypereutectic alloys.

Al-Si hypereutectic alloys for cylinder liners present advantages for Al-Si engine block applications. Unfortunately these applications have been limited to luxury vehicles. For instance, the implementation of Al-Si cylinder liners can create a metallurgical interface with higher thermal conductivity, reduce residual stresses due to a more similar thermal expansion coefficient, to mention a few. The poor interface between the engine and the cast iron cylinder liner is due to the affinity of Al for Fe reactivity. Additionally cast iron cylinder liners are shot blasted using steel media. The difference in thermal expansion coefficient between the 319 Al alloy and cast iron is 42%, while between the 319 and 390 alloys it is only 8%; which allows to cast shorter engine blocks (~25% weight reduction) or to introduce larger cylinder liners that have the potential for a higher performance engine.
4.1. Introduction to the Experimental Procedures

The experimental work in this dissertation was divided into two main sections, the preliminary experiments and the statistically designed experiments. During the preliminary experiments several Al-Si hypo and hyper eutectic alloys were tested using ES and ESV techniques. In total one ES and four ESV workstations were designed, built and tested during the preliminary. During the preliminary experiments ~ 250 experiments were carried out under different conditions. The resulting test samples were characterized using thermal analysis and light optical microscopy (LOM). The characterization results were analyzed to determine the variables that had a significant effect on the microstructure modification of the ESV melt treated test samples. The rest of the variables were kept constant in order to decrease to a manageable number the testing parameters during the statistically designed experiments.

Fourteen melts of different compositions were selected to conduct the experimental part of this dissertation (Table 4.1). These alloys were also used for the development of the Si equivalency (Si_{EQ}) algorithms for Al-Si hypereutectic alloys [17, 18]. The Si equivalency algorithms were used to calculate liquidus temperature of all the alloys based on their chemical compositions; establishing a constant criterion allowing for the elimination of the liquidus temperature from the experimental variables. The ES or ESV treatments were conducted for the alloys shown in "bold" in Table 4.1. The chemical composition of all alloys was determined using an Optical Emission Spectrometer (OES) at the NEMAK Engineering Centre, and the results are presented in Table 4.1.

The industrial casting partners involved in this dissertation were the NEMAK Essex Aluminum Plant, the NEMAK Windsor Aluminum Plant, both located in Windsor, Canada and Karl Schmidt Unisa, Inc. (KUS) located in Fountain, IN USA. KUS and Essex Aluminum Plant were the suppliers of the 390.1(1) and 390.1(II) Al-Si hypereutectic alloys respectively and the W319 alloy was donated by Windsor Aluminum Plant. The casting operations conducted in the respective plants was carried out using a superheat temperature of 150 ±10°C above liquidus. The superheat temperature for the experimental alloys used on this dissertation was determined as 150°C above the calculated liquidus temperature using the Si equivalency algorithms.
4.2. Alloy Preparation

All fourteen alloys previously mentioned belong to the 3XX.X family of aluminum alloys based on the American Aluminum Association (AAA) Standard (Table 4.1). Except for the W319, 390.1(I) and 390.1(II) alloys, the rest were prepared at the University of Windsor. The amounts of "base alloy", pure Al (industrial grade), Al-50 wt% Si and Al-50 wt% Cu master alloys necessary to reach the chemical composition of every alloy shown in Table 4.1 was calculated using a computer program developed by R. Francis [10]. The base alloys were W319 and 390.1(I) for hypo and hypereutectic compositions respectively.

Table 4.1. Chemical composition (in wt%) of the investigated aluminum alloys and their calculated $T_{Liq}$ temperature using the Si equivalency algorithms [17, 18]. Note: all alloys were employed for the Si equivalency algorithm, however, only the alloys (in bold) were melt treated [17, 18]. No grain refiners (Ti, B), or Si modifiers (Sr, Na or P) were added to these alloys (< 10 ppm).

<table>
<thead>
<tr>
<th>AAA Designation</th>
<th>Si</th>
<th>Cu</th>
<th>Fe</th>
<th>Mg</th>
<th>Mn</th>
<th>Ni</th>
<th>$T_{Liq}$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Calculated</td>
</tr>
<tr>
<td>C355</td>
<td>4.85</td>
<td>1.03</td>
<td>0.09</td>
<td>0.14</td>
<td>0.01</td>
<td>0.05</td>
<td>626.8</td>
</tr>
<tr>
<td>308.0</td>
<td>4.89</td>
<td>3.85</td>
<td>0.09</td>
<td>0.16</td>
<td>0.01</td>
<td>0.02</td>
<td>622.3</td>
</tr>
<tr>
<td>328.0</td>
<td>7.00</td>
<td>0.96</td>
<td>0.29</td>
<td>0.21</td>
<td>0.01</td>
<td>0.01</td>
<td>611.3</td>
</tr>
<tr>
<td>W319</td>
<td>7.18</td>
<td>4.68</td>
<td>0.17</td>
<td>0.26</td>
<td>0.01</td>
<td>0.03</td>
<td>605.6</td>
</tr>
<tr>
<td>322.2(I)</td>
<td>9.12</td>
<td>1.18</td>
<td>0.18</td>
<td>0.28</td>
<td>0.01</td>
<td>0.05</td>
<td>596.7</td>
</tr>
<tr>
<td>322.2(II)</td>
<td>9.85</td>
<td>4.38</td>
<td>0.14</td>
<td>0.27</td>
<td>0.01</td>
<td>0.07</td>
<td>587.1</td>
</tr>
<tr>
<td>336.2</td>
<td>10.84</td>
<td>0.94</td>
<td>0.11</td>
<td>0.19</td>
<td>0.01</td>
<td>0.06</td>
<td>584.6</td>
</tr>
<tr>
<td>332.2</td>
<td>10.55</td>
<td>4.36</td>
<td>0.13</td>
<td>0.17</td>
<td>0.01</td>
<td>0.08</td>
<td>568.0</td>
</tr>
<tr>
<td>390.1(I)</td>
<td>13.14</td>
<td>4.11</td>
<td>0.51</td>
<td>0.99</td>
<td>0.21</td>
<td>2.19</td>
<td>600.2</td>
</tr>
<tr>
<td>390.1(II)</td>
<td>15.53</td>
<td>3.20</td>
<td>0.62</td>
<td>0.62</td>
<td>0.29</td>
<td>0.62</td>
<td>634.6</td>
</tr>
<tr>
<td>393.2(I)</td>
<td>25.0</td>
<td>5.64</td>
<td>0.64</td>
<td>0.09</td>
<td>0.15</td>
<td>0.1</td>
<td>756.9</td>
</tr>
<tr>
<td>393.2(II)</td>
<td>25.0</td>
<td>1.18</td>
<td>0.39</td>
<td>0.05</td>
<td>0.12</td>
<td>0.4</td>
<td>752.5</td>
</tr>
<tr>
<td>393.2(II)</td>
<td>24.16</td>
<td>2.46</td>
<td>0.93</td>
<td>0.17</td>
<td>0.36</td>
<td>1.25</td>
<td>738.4</td>
</tr>
<tr>
<td>393.2(III)</td>
<td>28.64</td>
<td>2.43</td>
<td>0.90</td>
<td>0.15</td>
<td>0.36</td>
<td>1.32</td>
<td>794.9</td>
</tr>
</tbody>
</table>

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4.3. Melting Procedures

4.3.1. Melt Preparation

All alloys were melted in a PHS 12 kW resistance furnace (model VTR40M) with a capacity of 12 kg of molten Al alloy. Once the alloy was melted and reached its superheat temperature, the melt was skimmed and degassed. The melt was degassed using a FOSECO Mobile Degassing Unit, by blowing Ar gas at a rate of 20 SCFH (Standard Cubic Feet per Hour), through a graphite impeller rotating at 120 RPM (Revolutions per Minute). After degassing for approximately 20 minutes the Hydrogen level was < 0.100 ± 0.005 mL H₂/100 g of Al. The Hydrogen level was measured with an AlSCAN analyzer. The overall view of the Melt Preparation Station is shown in Figure 4.1.

![Figure 4.1. Melt preparation station.](image)
4.4. Thermal Analysis Procedure

The thermal analysis of all the test samples was performed using two workstations. The first one is the Aluminum Thermal Analysis Platform (AITAP) and the second one is the Universal Metallurgical Simulator and Analyzer (UMSA). Both workstations include their own unique software and were these entire systems were fully designed by the IRC members. The two workstations contain of a K type thermocouple connected to a high speed National Instruments data acquisition system linked to a personal computer. Thermocouples are traceable to the National Institute of Calibration Standards and Technology (NIST). More details about the thermocouples for the two workstations are given in Section 4.4.1.

4.4.1. Preparation and Calibration of Thermocouples

Two types of K thermocouples were used for thermal analysis experiments; one was the commercial (Acrolab) 1.6 mm (outer diameter), and the other was a custom made, low mass thermocouple (LMT). The LMT was assembled using Omegatite® ceramic, 6 inches in length and 1.1 mm in outer diameter, chromel/alumel thermocouple wires 0.125 mm in diameter were inserted in the Omegatite® ceramic. The tips of both wires were welded using an electric arc and covered with OMEGA® high temperature cement (thin films) to avoid contact with the molten alloy. This thermocouple was characterized by very low thermal inertia and a low time constant. Both, the commercial and the custom made (in house) thermocouples have a maximum working temperature of 1260°C, well above the maximum temperature as reported in this research (1040°C).

The thermocouples were individually calibrated [10], in order to eliminate any bias error in temperature measurement. Calibration was performed using the Carbolite® furnace (Model EUROTHERM 902P). The Resistance Temperature Detector (RTD) probe was calibrated at an International Standards Organization certified laboratory (Acrolab) located in Windsor (ISO-10012). The average error in temperature measured in the range of 300-750 °C was less ± 0.6 °C. Figure 4.2 shows the graph of the temperatures of the Resistance Temperature Detector probe versus the experimental thermocouples (commercial and LMT). The commercial and LMT calibration equations were determined by linear regression against the Resistance Temperature
Detector probe and are shown in Table 4.2. This calibration equation was incorporated into the AITAP’s and UMSA’s software to assure the reliability of the cooling curves thermal characteristics.

Table 4.2. Calibration equations for the experimental thermocouples [10].

<table>
<thead>
<tr>
<th>Thermocouple type (Φ mm)</th>
<th>Equation ((T_{\text{measured}} = aT_{\text{RTD}} + b))</th>
<th>Standard Deviation (°C)</th>
<th>95% Confidence Limit, (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LMT 1.1 (1)</td>
<td>(0.992), (3.242)</td>
<td>0.0958</td>
<td>0.0337</td>
</tr>
<tr>
<td>LMT 1.1 (2)</td>
<td>(0.991), (1.660)</td>
<td>0.1869</td>
<td>0.0658</td>
</tr>
<tr>
<td>LMT 1.1 (3)</td>
<td>(0.992), (2.451)</td>
<td>0.1532</td>
<td>0.0479</td>
</tr>
<tr>
<td>Acrolab 1.6 (1)</td>
<td>(0.997), (0.329)</td>
<td>0.4358</td>
<td>0.1534</td>
</tr>
<tr>
<td>Acrolab 1.6 (2)</td>
<td>(0.993), (0.724)</td>
<td>0.4620</td>
<td>0.1626</td>
</tr>
<tr>
<td>Acrolab 1.6 (3)</td>
<td>(0.999), (-1.356)</td>
<td>0.6065</td>
<td>0.2135</td>
</tr>
</tbody>
</table>

Figure 4.2. Calibration lines for the Acrolab and low mass thermocouples [10].

4.4.2. Thermal Analysis Using AITAP

AITAP was used to record the temperature vs. solidification time (cooling curve) of the test samples. The cooling curves were recorded from the pouring temperature until the test sample reached 400°C. However, the pouring temperature was different for the different alloys.
and melt treatments (as shown in Section 4.3). The beginning of the recording of the cooling curve is ~50°C below the superheat temperature because of the heat lost during the transportation and pouring of the melt from the furnace into the experimental cup. AITAP was used for the experiments performed for the reference and melt treated test samples, including, the feasibility studies and the statistically designed experiments.

4.4.3. Thermal Analysis Using the Universal Metallurgical Simulator and Analyzer (UMSA)

The UMSA is a multi-task platform/simulator that can experimentally vary processing conditions and continuously quantify the effects of these changes on microscopic characteristics (Figure 4.3). The UMSA platform is being patented under PCT/CA02/01903, Canada, 2002 by scientists from the University of Windsor and the Silesian University of Technology, Poland. The instrument can perform metallurgical simulations on a micro and/or macroscale. The UMSA results were used to predict the metallurgical properties of the test samples [177-181, 185-187].

The UMSA merges several “conventional” instruments such as a melting station, heat treatment furnaces, a quenching station and a thermal analysis device into a single apparatus. UMSA can be utilized to simulate a variety of industrial metal casting and heat treatment processes to understand the phenomena taking place during these operations. Using UMSA the effect of variables such as temperature, time, heating, and cooling rates can be related to the micro structural and mechanical properties of the castings [168, 177, 179, 181]. Experimental data obtained with UMSA can be utilized to evaluate thermodynamic constants such as the specific heat and total heat of melting.

UMSA thermal analysis was performed in order to identify the characteristic temperature of the metallurgical reactions during the heating and cooling of the UMSA test samples (Figure 4.4). Thermal analysis of the alloys was conducted by heating the test samples Type I to 750 ±0.5°C (390.1(I) alloy) and to 850 ±0.5°C (393.2(I) and 393.2(II)) followed by cooling under natural heat exchange conditions. This thermal analysis was performed during the heating and cooling cycles for all the alloys under UMSA controlled conditions. The thermal signatures of the test samples were recorded and analyzed to obtain the First Derivative (dT/dt), base lines and
the fraction solid curves using the IRC standard procedures [182, 183]. This thermal analysis procedure was adapted for the Al-Si hypereutectic alloys used in this dissertation (Appendix I).

4.4.4. Preparation of the UMSA Samples

Advanced thermal analysis was performed using the UMSA platform (1kW power output) [181]. The test samples used in the UMSA platform were prepared in two different ways, the first procedure consists of the machining of a specific section of a casting; in the second procedure, molten alloy is poured into a steel split casting mould. A picture of the UMSA platform is shown in Figure 4.3 and a picture of the UMSA test sample is presented in Figure 4.4. In all cases the machining was performed by the Technical Support Centre at the University of Windsor.

![Figure 4.3. The 1kW output UMSA platform (patent PCT/CA02/01903) [171].](image)

The casting procedure for the poured UMSA test samples consists of several steps that are described as follows. Prior to the pouring of the molten alloy, the mould was coated with mica and then clamped. The mould was coated to avoid any chemical reaction and facilitate the
un-moulding operation after casting, while clamping sealed the mould to avoid leaks. For coating purposes a mica suspension was prepared with distilled water at a ratio of 1:5 in volume (mica:water). The above-mentioned suspension was applied with a nylon fleece brush of 25.4 mm in thickness on top of the mould allowing the formation of a thin layer of mica. After 10 minutes of drying in a stove a second mica layer was put on the mould. The final thickness of the mica layer was of 100 to 150 μm. Molten alloy (at its superheat temperature) was poured into permanent steel mould.

Figure 4.4. UMSA test sample. a) Macrograph of the test sample and b) schematic drawing and the test samples dimensions (mm).

Figure 4.5. a) Permanent steel split casting mould to cast UMSA’s test bars and b) the test bar [1].

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After the coatings the mould was clamped and dried out in a Lindberg 2000 Temperite Furnace for 24 hours at a temperature of 250°C. At this preheating temperature there were not found cold spots in the cast UMSA test bars. The cast UMSA test bars after casting were machined at the Technical Support Centre under the dimensions presented in Figure 4.4. Figure 4.5 shows the pictures of the steel mould and UMSA test bar.

4.5. **Design of the ESV Melt Treatment Cup**

A non-magnetic stainless steel (304) cup with a riser was designed for the ESV melt treatments [2]. This cup is called “The Standard ESV Cup”. Using a single cup for all the experimental variables such as geometry of the cup, mass and coatings were kept constant, thus eliminating the cup and coating as variables from the statistically designed experiments. The standard ESV cup was always coated with mica suspension following the procedure mentioned in Section 4.4.4. The preheating temperature of the standard ESV cup was $700 \pm 10^\circ$C for a period of two hours and after the first casting (or experiment) the cup was again preheated for at least 10 min. The standard ESV cup was designed with a riser to entrap gas porosity and residual segregation of primary Si.

For the thermal analysis procedure the standard ESV cup was shielded on top and/or on the bottom using ceramics to increase the riser’s efficiency, allowing a heat release in the radial direction. Figure 4.6a-b show pictures of the standard ESV cup and a section of the test sample after polishing. The mass of the cup was $\sim 260$g, with a wall thickness of 1 mm with a capacity of 500 mL which is equivalent to $1250 \pm 20$g of molten Al-Si alloy. With this cup test samples can be solidified under natural heat exchange conditions (reference) or can be forced to solidify directionally (air chilled test samples) using a Cu chilling plate.

4.5.1. **Cu Chilling Plate**

The directional solidification process was conducted to study the effect of a gradient of temperatures during the solidification of reference and ESV melt treated test samples. The directional solidification was carried out using the air chilling station (Cu plate) [3] in order to determine the effect of the temperature gradient on the solidified test samples structure. The Cu
plate was machined by the Technical Support Centre from a solid piece of high purity Cu (Figure 4.6c-d). This plate has an inlet that is connected into a compressed air line at a pressure of 600 kPa and a flow rate of ~1.5 Standard Cubic Meter per Minute. The directional solidification forced the release of the heat in the vertical direction out through the bottom of the test sample. Additionally, heat is also released due to the natural heat exchange conditions in the radial direction giving two dimensions of heat flow (2D) in the test samples. By forcing the test samples to solidify under these conditions, the homogeneity of the test samples was improved (Figure 4.6b) allowing for the use of these test samples for tensile testing. Note that only the test samples used for the statistically designed experiments were solidified using the Cu Plate.

Figure 4.6. a) The standard ESV cup [2] used for the statistically designed experiments, b) cross section of the test samples in as cast conditions, c) technical drawing with dimensions (mm) and d) Cu chill plate for sirectional solidification [3].

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4.5.2. Siphon Pouring Device

In order to pour the highest quality melts for tensile bars and/or automotive components a siphon device (see Figure 4.7) was designed for the standard ESV cup. With the siphon device, the residual primary Si macro-segregated and inclusions or oxides were avoided during pouring. This permits the pouring of the melt located into the centre and bottom of the standard ESV cup. The siphon device was machined by the Technical Support Centre from a piece of 304 stainless steel [2]. Prior to the first pouring, the device was coated with mica following the procedures mentioned in Section 4.4.4; and preheated to temperature of 700 ± 10°C for a period of at least two hours for the first casting and then only 10 minutes for the subsequent castings.

![Figure 4.7. a) Top view of the pouring device and b) pouring device together with the ESV cup [2].](image)

4.6. Al-Si Alloys Melt Treatments

The 250 test samples cast for the preliminary experiments (feasibility studies) were solidified in the presence of magnetic fields using the ES or ESV workstation(s). The following subsections provide an overview of the work stations as well as the casting operations and conditions of the test samples.
4.6.1. Electromagnetic Stirring (ES)

Figure 4.1 shows a schematic diagram of the Electromagnetic Stirring Workstation built using a stator of an electric motor that was adapted for high temperature operating conditions. Under the ES conditions, the molten metal works as a rotor. A W20 G3M Variac was used to supply the current under controlled conditions. Table 2 shows the electrical parameters and operating conditions of the ES workstation.

After the hydrogen level was measured, the W319 melt was poured into the non-magnetic cup without a riser. All test samples were solidified inside the ES workstation. The ES treated test samples were solidified in the presence of an AC currents varying from 2A to 8A during the solidification process. Both ends of the cup (top and bottom) were insulated to conduct the release of the heat in only one dimension (radial direction) [182]. Three test samples were cast per given electrical current condition. All ES treatments were carried out in the temperature range between 645°C and 560°C (~45°C above liquidus and ~40°C below liquidus).

Figure 4.8. Schematic drawing of the electromagnetic stirring workstation used for W319 alloy melt treatments [16].
Table 4.3. Electrical characteristics of the ES workstation [16].

<table>
<thead>
<tr>
<th>Device</th>
<th>Phases</th>
<th>Current</th>
<th>AC Voltage Range (V)</th>
<th>Frequency (Hz)</th>
<th>Power Factor</th>
<th>Rotation Speed (RPM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stator</td>
<td>3 AC</td>
<td>AC</td>
<td>230YY/460Y</td>
<td>60</td>
<td>0.7</td>
<td>1100</td>
</tr>
<tr>
<td>Variac</td>
<td>3 AC</td>
<td>230</td>
<td></td>
<td></td>
<td>N/A</td>
<td>N/A</td>
</tr>
</tbody>
</table>

4.6.2. Electromagnetic Stirring and Vibration (ESV)

The melt treatments conducted using the ESV technique were performed for two reasons, the first was to find all independent and dependent variables (preliminary experiments) and their effect on the microstructure modification and Si refinement. The second was carried out for the statistically designed experiments. Four distinct and unique ESV workstations were built based on the electrical configuration connection of the electromagnetic coils. Based on the electrical configuration connections a particular name was assigned to every workstation. The preliminary experiments carried out with the ESV workstations were performed under various conditions such as: different melt treatment and superheat temperatures, currents, times, H₂ levels, type of test cups, cooling rates, electrical configurations, weight of the test samples, with and without chemical modifiers, ceramic reinforcement particles and Ni coated graphite. Chapter 5 provides a summary of selected results of the microstructure of the ESV melt treated test samples.

1) Two-Coil Workstation in Parallel Connection

This workstation had the two electromagnetic coils placed on top of each other and connected in parallel one to the AC and the other one to the DC power supply respectively. The intention was to separate the coils and improve the cooling conditions of the electromagnetic coils. Ceramic spacers were placed between them. The test samples were placed inside the coils (Figure 4.9) [6, 21].

2) Two-Coil Workstation in Series Connection

This workstation used both coils in the same fashion, but they were connected in series passing both AC and DC in the same conductor at the same time. Once again the test samples were placed in the centre of the coils, with the aim to stir and vibrate the molten...
metal by the influence of the electromagnetic field. The picture of this system is the same as
the previous one (Figure 4.9) [6, 21].

3) Two-Coil Workstation in a Ferromagnetic Core

The coils in this workstation were placed horizontally beside each other in the vertical
direction. Through the centre of both coils a single ferromagnetic core was passed, the coils
were placed at the back of the core. This core had a letter “C” shape. The test samples were
placed in the opening of the In C. The electromagnetic coils were connected in parallel one
to the AC and the other one to DC electrical current respectively forcing the magnetic flux to
pass through the test sample from the end of the core to the beginning (Figure 4.10) [6, 21].

4) Two-Core-Coil Workstation

This workstation consists of two coils and two C type ferromagnetic cores. Each coil was
placed at the back of its respective core. Both cores were placed perpendicularly, allowing
the sample to be placed from the top in the centre of both openings. One coil was connected
to an AC source and the other one to a DC source (Figure 4.10) [6, 21].

All test samples cast in the workstations described above were poured using melts
prepared under the conditions mentioned in Section 4.1. The majority of the treated test samples
were poured using 390.1(I) and 390.1(II) Al-Si alloys. The author of this dissertation was
responsible for the metallurgical analysis and development of the scientific hypothesis of the
effect of the ESV melt treatments, which is presented in Chapters 5 and 6. Mr. J. Ohl conducted
the electrical analysis of the ESV workstations. It is fundamental to mention that both individuals
collaborated fully with another and openly sheared information and ideas about their respective
tasks. As an example the introduction of the ferromagnetic cores was based on the literature
review, the author brought the IRC attention. However, Mr J. Ohl due to his electrical
background was responsible to build the respective workstations. Also, it is important to clarify
that all workstations were built following extensive technical and scientific research carried out
by the IRC Interdisciplinary team [4].

For more detailed information about the electrical parameters of the workstations and the
ferromagnetic core characteristics please see, Mr. J. Ohl’s Master’s Thesis entitled “Development
of a novel system used for Electromagnetic Treatment of Aluminum Alloys during the
Solidification Process” [21]. In this thesis the results of the metallurgical analysis of the presented test samples was carried out by the author.

![Figure 4.9. ESV workstations showing a) an overall view of the two coil system used in parallel and series connections, b) top view of the workstation without coils [4].](image)

1) Sample’s Cooling System, 2) Thermocouple, 3) Ceramic Cover of the ESV Workstation, 4) Sample’s Insulation, 5) Coil’s Cooling System (1), 6) ESV Coil(s), 7) Ceramic Base, 8) Coil(s)’ Cooling System (2), 9) Steel Base, 10) Ceramic Insulation, 11) Pneumatic Lift, 12) Heating System (Resistance Furnace of 80 Ω) [4].

After extensive research conducted using two coil workstation it was found that except for the invention of the Two-Coil-two-Core workstation, the use of two coils is detrimental for ESV melt treatment applications. In personal communications among Dr. J. H. Sokolowski, Mr. J. Ohl and the author a “Dual” coil was designed and developed and was used to built the ETCT platform, which will be patented [6]. In the dual coil system both the AC and DC currents are forced to go through the same conductor, eliminating the detrimental effect of creating an AC current in the DC coil. The overview of the ETCT platform is shown in Figure 4.9.
4.7. **Statistical Design of Experiments**

The preliminary experiments (the results are presented in Chapter 5) using the ES and the four ESV workstations identified the independent variables that have a significant effect on the refinement of the microstructure and the modification level of phases such as primary and eutectic Si. Table 4.4 shows a list of independent and dependent variables recognized as parameters having an effect on the level of refinement of the microstructure. With all these variables it was impossible to perform the full factorial set of experiments. Therefore, the microstructure analysis of the test samples from the preliminary experiments was used to select only the variables that have strong effect on microstructure refinement. The selected variables were used to conduct statistically designed experiments and minimize the number of tests. Mr. Robin Francis assisted with the statistical designed experiments [7].
4.7.1. Selection of Independent Variables for the Statistically Designed Experiments

The feasibility studies were conducted to determine not only the independent variables, but also to assess the reasons that would allow for the elimination of as many variables as possible. The aim was to conduct an analysis of variables and at the same time minimize the number of experiments needed to demonstrate the effect of the ESV melt treatments (see Table 4.4).

a) The Si equivalency algorithms [18] were used to calculate the liquidus temperature of every experimental alloy. The superheat temperature was set for all alloys 150°C above liquidus (as used in industry).

b) All experiments were conducted in the ESV workstation with the dual coil (ETCT) [6].

c) The ESV melt treatments were conducted with a computer controlled power supply also known as the current control source [8] eliminating human error.

d) In order to minimize the variability under different experimental conditions, a standard ESV cup was designed for all the experiments and was always coated with mica.

e) Neither silicon modifiers nor grain refiners were added to the melt for the statistically designed experiments. The reason for this was that during the preliminary experiments it was found that combining the ESV treatment and chemical modifiers resulted in an increase in the level of porosity in the microstructure. Additionally, the level of microstructure modification achieved with the ESV melt treatment was similar or higher than with the additions of chemical modifiers.

f) Melt treatments applied at temperatures of 100°C above the liquidus or higher presented negligible effects in microstructure modification and refinement. Therefore, no melt treatment for the statistically designed experiments was carried out at temperatures higher than 80°C above liquidus.

g) During the preliminary experiments it was observed that the temperature of the test sample cast using the ESV standard cup and solidified under natural heat exchange conditions inside the coils drops ~40°C in three minutes. Three minutes was selected due to the limitations of the ESV workstations; longer times increase the temperature of the electromagnetic coils up to 100°C and they were designed for a maximum temperature of 160°C. Also, this time period was enough to refine most of the primary Si particles of the 390.1(I) alloy. (Note: ESV treatments over 3 minutes can be carried out ONLY when the electromagnetic coils initial temperature is ≤ 24°C (Table 4.7).)
h) Dissimilar intensities of the AC and DC currents resulted in unfavorable microstructure refinement and phase modification increasing the test sample’s porosity.

i) As mentioned in the literature review [47], voltage is a dependent variable that depends on the current and the electrical resistance of the test sample (Ohm’s law). Note: that the same alloy under the same melt treatment conditions has the same electrical resistance.

Table 4.4. List of independent and dependent variables related to the ESV melt treatments.

<table>
<thead>
<tr>
<th>Melt Characteristics</th>
<th>List of Independent Variables</th>
<th>List of Dependent Variables</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metal cleanness</td>
<td></td>
<td>Melt Characteristics</td>
</tr>
<tr>
<td>Chemical composition</td>
<td></td>
<td>Liquidus and Superheat</td>
</tr>
<tr>
<td>Master alloys</td>
<td></td>
<td>H₂</td>
</tr>
<tr>
<td>Additions</td>
<td></td>
<td>Fluidity of the melt</td>
</tr>
<tr>
<td>Silicon modifiers</td>
<td></td>
<td>Flowability of the melt</td>
</tr>
<tr>
<td>Grain refiners</td>
<td></td>
<td>Solidification Conditions</td>
</tr>
<tr>
<td>Casting temperature</td>
<td></td>
<td>Test sample’s cooling rate</td>
</tr>
<tr>
<td>Temperature</td>
<td></td>
<td>Low (ESV test sample)</td>
</tr>
<tr>
<td>Temperature of the</td>
<td></td>
<td>High (prototype piston or</td>
</tr>
<tr>
<td>ESV melt treatment</td>
<td></td>
<td>cylinder liner)</td>
</tr>
<tr>
<td>Isothermal holding</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ESV Treatment</td>
<td></td>
<td>Microstructure</td>
</tr>
<tr>
<td>Workstation (s)</td>
<td></td>
<td>Si modification level</td>
</tr>
<tr>
<td>Electromagnetic</td>
<td></td>
<td>Microstructure refinement</td>
</tr>
<tr>
<td>Stirring</td>
<td></td>
<td>(matrix)</td>
</tr>
<tr>
<td>Electromagnetic</td>
<td></td>
<td>Soundness (porosity)</td>
</tr>
<tr>
<td>Stirring and</td>
<td></td>
<td>SDAS</td>
</tr>
<tr>
<td>Vibration</td>
<td></td>
<td></td>
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<tr>
<td>Time of the ESV</td>
<td></td>
<td></td>
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<tr>
<td>Melt treatment</td>
<td></td>
<td></td>
</tr>
<tr>
<td>The Current Control</td>
<td></td>
<td></td>
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<tr>
<td>Source [8]</td>
<td></td>
<td></td>
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<tr>
<td>Current of the ESV</td>
<td></td>
<td></td>
</tr>
<tr>
<td>treatment (AC/DC)</td>
<td></td>
<td></td>
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<tr>
<td>Frequency of the AC</td>
<td></td>
<td></td>
</tr>
<tr>
<td>current</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Time of the ESV</td>
<td></td>
<td></td>
</tr>
<tr>
<td>melt treatment</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type of test sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ESV test sample</td>
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<td></td>
</tr>
<tr>
<td>Prototype piston</td>
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<tr>
<td>Prototype cylinder</td>
<td></td>
<td></td>
</tr>
<tr>
<td>liner</td>
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<tr>
<td>Tensile test bar</td>
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<tr>
<td>Fluidity Mould</td>
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<tr>
<td>Air cooling system</td>
<td></td>
<td></td>
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<tr>
<td>of the ESV workstation</td>
<td></td>
<td></td>
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<tr>
<td>ESV Melt Treatment</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Media</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Electromagnetic</td>
<td></td>
<td></td>
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<tr>
<td>stirring cup (mass</td>
<td></td>
<td></td>
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<tr>
<td>of the test sample)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Siphon Device</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type of Ceramic</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coating of the ESV</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cup</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solidification</td>
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<td></td>
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<tr>
<td>Conditions</td>
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<td></td>
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<tr>
<td>Air quenching using</td>
<td></td>
<td></td>
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<tr>
<td>the Cu plate</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test sample heat</td>
<td></td>
<td></td>
</tr>
<tr>
<td>exchange conditions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 dimension (Radial)</td>
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<td></td>
</tr>
<tr>
<td>2 dimension (Radial+</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Vertical)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Air cooling system</td>
<td></td>
<td></td>
</tr>
<tr>
<td>of the test sample</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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4.7.2. Experiments Designed Using the Fraction Factorial Method

The selection of experimental variables was based on the analysis of the microstructure of the representative test samples from the preliminary experiments [31 - 72]. In the literature there are no reports of the application of electromagnetic stirring at temperatures above the liquidus. Also, except for the experiments conducted by Suhn-Chul et. al. [47], voltage is not considered as an ES or ESV variable. The number of experimental variables for the ESV melt treatments was reduced to the following seven parameters:

i) AC Current Frequency (Hz),
ii) Electrical Current Intensity (AC and DC in A),
iii) ESV melt treatment temperature range, which in all cases was above liquidus (°C),
iv) Hydrogen level (mL H₂/100 g of Al),
v) Different times (t = s) for the ESV treatments under isothermal conditions
vi) Melt Si level (chemical composition)
vii) Cooling rate

Due to the difficulty of controlling 7 experimental variables for the statistically designed experiments they were conducted in 3 stages. In the first stage, 4 variables were selected (Table 4.5) and the full factorial set of experiments was conducted at two levels [7].

All test samples in Table 4.5 were analyzed using the image analysis procedure (Section 4.10.1) to determine the novel silicon modification level (SiML). The results of the SiML were statistically analyzed to determine the variables with the strongest effect on the microstructure refinements. As a result of the statistical analysis it was found that the temperature of the ESV melt treatment and AC current frequency are the variables with the strongest microstructure refinement and phase modification. Therefore, narrower ranges of ESV melt treatment temperatures and three frequencies were used to identify the ESV melt treatment conditions that lead to the highest level of modification for the second stage of statistically designed experiments (Table 4.6).
Table 4.5. Determination of the ESV melt treatment conditions using the ESV dual coil (ETCT) workstation with the 390.1(I) alloy in all cases.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>Frequency (f) (Hz)</th>
<th>Current AC/DC (A)</th>
<th>ESV Melt Treatment Temperature Range, (°C)</th>
<th>Degassing Conditions†</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>40</td>
<td>650-610</td>
<td>Yes</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>40</td>
<td>720-680</td>
<td>Yes</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>40</td>
<td>650-610</td>
<td>No</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>40</td>
<td>720-680</td>
<td>No</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>100</td>
<td>650-610</td>
<td>Yes</td>
</tr>
<tr>
<td>6</td>
<td>10</td>
<td>100</td>
<td>720-680</td>
<td>Yes</td>
</tr>
<tr>
<td>7</td>
<td>10</td>
<td>100</td>
<td>650-610</td>
<td>No</td>
</tr>
<tr>
<td>8</td>
<td>10</td>
<td>100</td>
<td>720-680</td>
<td>No</td>
</tr>
<tr>
<td>9</td>
<td>100</td>
<td>40</td>
<td>650-610</td>
<td>Yes</td>
</tr>
<tr>
<td>10</td>
<td>100</td>
<td>40</td>
<td>720-680</td>
<td>Yes</td>
</tr>
<tr>
<td>11</td>
<td>100</td>
<td>40</td>
<td>650-610</td>
<td>No</td>
</tr>
<tr>
<td>12</td>
<td>100</td>
<td>40</td>
<td>720-680</td>
<td>No</td>
</tr>
<tr>
<td>13</td>
<td>100</td>
<td>100</td>
<td>650-610</td>
<td>Yes</td>
</tr>
<tr>
<td>14</td>
<td>100</td>
<td>100</td>
<td>720-680</td>
<td>Yes</td>
</tr>
<tr>
<td>15</td>
<td>100</td>
<td>100</td>
<td>650-610</td>
<td>No</td>
</tr>
<tr>
<td>16</td>
<td>100</td>
<td>100</td>
<td>720-680</td>
<td>No</td>
</tr>
</tbody>
</table>

† Yes means that the hydrogen (H₂) level was < 0.100 mL H₂/100 g of Al and No means non-degassing and the H₂ level was > 0.100 mL H₂/100 g of Al.

Table 4.6. Experimental conditions for the second stage of the ESV melt treatments.

<table>
<thead>
<tr>
<th>Sample (390.1(I) Alloy) ID #</th>
<th>Frequency (Hz)</th>
<th>ESV Melt Treatment Temperature Range, (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>17</td>
<td>40</td>
<td>720-680</td>
</tr>
<tr>
<td>18</td>
<td>80</td>
<td>720-680</td>
</tr>
<tr>
<td>19</td>
<td>100</td>
<td>720-680</td>
</tr>
<tr>
<td>20</td>
<td>40</td>
<td>680-650</td>
</tr>
<tr>
<td>21</td>
<td>80</td>
<td>680-650</td>
</tr>
<tr>
<td>22</td>
<td>100</td>
<td>680-650</td>
</tr>
<tr>
<td>23</td>
<td>40</td>
<td>650-610</td>
</tr>
<tr>
<td>24</td>
<td>80</td>
<td>650-610</td>
</tr>
<tr>
<td>25</td>
<td>100</td>
<td>650-610</td>
</tr>
</tbody>
</table>

Note: all ESV melt treatments in Table 4.6 were performed with an I_{AC/DC} = 100 A, t = 3 min. with degassed 390.1(I) melt.

In the literature it has been reported that Al-Si alloys with up to 25 wt% Si had been utilized for cylinder liner applications [23 - 25, 120, 143]. Consequently, an alloy with 25 wt% of Si (393.1(I)) was used for the third stage of the statistically designed experiments. For this stage of experiments different electromagnetic treatment times under isothermal conditions were used to assess the effect of time and isothermal holding during the ESV melt treatments (Table 4.7).
Table 4.7. Experimental conditions for the third stage of experiments using different times and Si contents for ESV melt treatments conducted at isothermal temperatures. Using the ESV dual coil workstation (ETCT).

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>Isothermal Holding Time (s)</th>
<th>Si Content/Alloy wt% Si</th>
<th>Alloy 390.1(I)</th>
<th>Alloy 393.2(I)</th>
</tr>
</thead>
<tbody>
<tr>
<td>26</td>
<td>22.5</td>
<td>14.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>45</td>
<td>14.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28</td>
<td>90</td>
<td>14.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>180</td>
<td>14.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>360</td>
<td>14.5</td>
<td></td>
<td></td>
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<tr>
<td>31</td>
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<td>32</td>
<td>90</td>
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<tr>
<td>33</td>
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</tr>
<tr>
<td>34</td>
<td>360</td>
<td>25</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note: all ESV melt treatments in Table 4.7 were performed with an $I_{AC/DC} = 100$ A, $f = 100$ Hz, with degassed 390.1(I) and 393.2(I) melts at temperatures of 610 and 760 ±3°C respectively.

4.7.3. Casting Conditions of the Different Test Samples

All test samples were cast with masses of 1260 ±20g using the standard ESV cup and solidified under natural heat exchange conditions or on the Cu chilling plate. The same heat exchange conditions were used during the solidification of reference or ESV melt treated test samples. For all test samples an Acrolab K thermocouple connected to the AiTAP (previously calibrated) was placed in the centre of the test samples. The solidification conditions of the test samples is provided below:

1) Reference Test Samples

The standard ESV cup was preheated as previously indicated and natural heat exchange conditions were allowed during the solidification process. The 390.1(I) and 393.2(I) alloy melts were poured into the standard ESV cup and placed inside the dual coil (ETCT) through the entire solidification process. By covering the top and bottom of the standard ESV cup with ceramic pieces the release of the heat was mainly restricted to the radial direction (one dimension (1D)); resulting in the best conditions for thermal analysis [182, 183]. The cooling rate for the 390.1(I) alloy test samples solidified under this conditions was ~0.08°C/s.
2) Air Chilled (Directionally Solidified) Test Samples

The melt was poured in the preheated standard ESV cup and placed on the Cu chilling plate designed for directional solidification. Under directional solidification the heat was released through the bottom of the test sample (vertically) and radially due to natural convection conditions, therefore, the heat was released in 2D one radially and the other one vertically. This practice resulted inappropriate for thermal analysis due to the temperature gradient created along the solidifying test samples. The cooling rate in the centre of the test sample (location of the thermocouple), for the 390.1(I) alloy that solidified under these conditions was ~0.09°C/s.

3) ESV Treated Test Samples

The ESV melt treatments were conducted under the conditions given in Table 4.5, Table 4.6 and Table 4.7. The current control source was programmed using electrical conditions for the respective test samples given in the above-mentioned tables. Once the test samples reached the temperature of the treatment, the current control source supplied the variable AC and DC currents for specific times. After the ESV melt treatment was concluded the test samples was allowed to solidify inside the electromagnetic coils (heat released in one dimension), or removed from the coils and placed on the Cu plate for directional solidification (heat released in two dimensions). The cooling rate for the solidification conditions in 1D was 0.08°C/s and for 2D 0.09°C/s.

Cooling curves for the reference and ESV melt treated test samples were recorded using AlTAP during the entire solidification process. The recording of the cooling curves was conducted at a rate of 5 counts per second. Appendix II provides the details of the thermal analysis procedures used in this dissertation.

4.8. Casting of the Test Samples and Automotive Components

4.8.1. Test Samples for Prototype Pistons

The casting of pistons was performed using the 390.1(I) melt under either untreated (as-cast) or ESV melt treated conditions. For the reference prototype pistons the melt was directly transferred from the resistance furnace after degassing into the steel die cast mould (Figure 4.11).
Chapter 4. Experimental Procedures

The ESV treated conditions to cast the pistons were $I_{\text{AC/DC}} = 100\ A$, $f = 100\ Hz$ and $t = 3\ min$ in the range of temperatures from 650 to 610 ± 3°C, cooling rate = 1.3°C/s.

Figure 4.11. a) Steel die mould to cast prototype pistons, b) sketch of the piston and c) prototype piston. Note: all dimensions are in mm.

In order to keep the same solidification conditions for all prototype pistons the die was coated with mica and dried for 12 hours at a temperature of 250 ± 5°C. This temperature was selected based on the production line conditions at Essex Aluminum Plant to die-cast pistons. All prototype pistons were allowed to solidify and cool down inside the mould for 5 minutes and were then removed and placed on zirconia sand until they reached room temperature. A picture of the permanent mould, a sketch and a cast prototype piston are shown in Figure 4.11.
4.8.2. Test Samples for Prototype Cylinder Liners

The prototype cylinder liners were cast in a steel mould design based on the dimensions of the cast iron cylinder liner for the V6 engine block (Figure 4.12a). The 390.1(I) and 393.2(I) alloys were used to cast the reference and ESV treated prototype cylinder liners. The ESV melt treatment conditions were $I_{AC/DC} = 100 \text{ A}, f = 100 \text{ Hz}, t = 3 \text{ min.}$ in the range of temperatures from 650 to 610 ± 3°C, cooling rate = 1.6°C/s (see Table 4.7 samples ID# 28 and 33). Once again to keep the solidification conditions constant in all cases, the mould was coated with mica using the standard procedure described in Section 4.4.4 then preheated at 400 ± 5°C for 12 hours. For the next castings the mould was preheated to the same temperature for 30 min. The 400°C was the minimum temperature at which the mould was can be filled with reference melt poured at the superheat temperature. Following the pouring of the melt 5 min was allowed to solidify and cooled down the prototype cylinder liner. Figure 4.12 shows the steel mould for casting of the cylinder liners, a sketch of the cylinder liner and the prototype cylinder liner.

It is important to mention that both steel permanent moulds, for the prototype pistons and cylinder liners, were designed by the author with the assistance of S. Budinsky, M. Enns And A. Jenns from the Technical Support Centre. Both moulds were made of AISI 1020 plain carbon steel.

4.8.3. Casting of Tensile Bars for Monotonic Tensile Testing

The tensile test bars were cast using a Stahl mould with dimensions complying with the B108 ASTM standard [173]. The mould was coated prior to the first casting, only in the gripping sections of the tensile test sample, while the gauge sections were uncoated in all cases, in order to maximize the structure’s integrity. The mould was always preheated to a temperature of 400°C prior to the pouring operation. The mould temperature was monitored using a K type thermocouple that was inserted through the running system to the pouring well in the bottom of
the mould. Test bars were cast using 390.1(I) and 393.2(I), alloys that were poured for reference and ESV melt treated test bars. The reference test bars were poured using the 390.1(I) and 393.2(I) alloys at their respective superheat temperatures.

Figure 4.12. a) Picture of the steel mould to cast prototype cylinder liners, b) technical sketch of the cylinder liner and c) prototype cylinder liner. Note: all dimensions are in mm.

Casting of tensile test bars using the ESV treated melt was conducted for the 390.1(I) and 393.2(I) alloys using a $I_{AC/DC} = 100$ A, $f = 100$ Hz a $t = 3$ min $T_{ESV} = 610 \pm 2^\circ$C, $t = 3$ min for the 390.1(I) alloy and $T_{ESV} = 750 \pm 2^\circ$C, $t = 6$ min for the 393.2(I) alloy. After pouring these test bars, the samples were allowed to cool down in the Stahl mould for ~10 min.
Figure 4.13. a) Stahl mould, ASTM B108 used to cast tensile test bars and b) actual test bars.

Fifteen test bars for each casting condition described above were made using the melting procedures and degassing conditions mentioned in Section 4.2. The test bars were divided into three sets of five test samples in order to carry out the tensile test under different conditions. One set of test samples was tested after artificial aging and the last set after the T6 heat treatment (Table 4.8). Figure 4.13 shows a picture of the Stahl mould and test bars. The heat treatment and artificial aging conditions are provided in Table 4.8.
4.9. Prototype V6 Engine Blocks Using Al-Si Cylinder Liners

4.9.1. Cosworth Base Core for the V6 Prototype Engine Blocks

As cast prototype Al-Si cylinder liners were machined at the Technical Support Centre to the same dimensions as the cast iron cylinder liners used in the V6 engine block for regular production. The reference and ESV melt treated prototype cylinder liners were cast using the 390.1(I) and 393.2(I) alloys. Three prototype cylinder liners were held in a sand core as shown in Figure 4.14. The three prototype Al-Si cylinder liners for every sand core were cast with same alloy and under same conditions. From every set of three prototype cylinder liners one of them had machined external grooves similarly to the cast iron cylinder liners. The grooved prototype cylinder liner was placed in the centre of the sand core (Figure 4.14c and d). The main idea of the grooves is to increase the contact area between the engine block material and to break down the oxide layer formed on the surface of the liquid W319 melt during casting.

Five V6 engine blocks were cast for this dissertation, one of them was taken from the production line at Windsor Aluminum Plant, the other four were cast as prototypes using Al-Si cylinder liners. The Cosworth packages for the V6 engine blocks have two sand cores for the cylinder liners and every sand core holds three cylinder liners. For each the four prototype engine blocks, one of the sand cores with the cast iron cylinder liner was removed and replaced with a sand core with a prototype Al-Si cylinder liner (Figure 4.14 and Figure 4.15a).

Table 4.8. Summary of the heat treatment procedure conducted for all the V6 engine blocks and tensile bars. This procedure is similar to the one conducted at B & W for regular production V6 engine blocks. Note: the quenching operation at University of Windsor was performed in water for all the tensile test bars and bulk head sections.

<table>
<thead>
<tr>
<th>Thermal Sand Removal; Temperature and Duration</th>
<th>Solution Treatment; Temperature and Duration</th>
<th>Artificial Aging; Temperature and Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>495 ± 5°C 4.5 Hours</td>
<td>510 ± 5°C 8 Hours</td>
<td>250 ± 5°C 5 Hours</td>
</tr>
</tbody>
</table>
4.9.2. Assembly of Mould Packages and Pouring of the V6 Engine Blocks

Figure 4.15a shows a picture of the V6 mould package prior to the assembly of the cylinder liners and Figure 4.15b demonstrates the assembly of the prototype sand cores. The cylinder liners were identified with numbers from 1 to 6 alternately for each side of the engine block (Figure 4.15a). After the assembly of the sand core packages, they were transported on a conveyor (Figure 4.15c) to the “Tocco” station. The Tocco operation consists of the preheating of the cast iron cylinder liners using Cu induction heaters (Figure 4.15d) to avoid cold spots created between the cylinder liner and the molten W319 alloy during the filling operation. This operation was carried out for 20 seconds for the cylinder liners to reach a temperature of ~350°C.
Figure 4.15. Sand core assembly of the prototype V6 engine blocks using Al-Si cylinder liners. a) Engine block's sand core package, b) assembly of the Al-Si cylinder liners, c) transport of packages, d) tocco (liner preheating) operation e) fume extraction unit during the solidification procedure and d) prototype V6 engine block.
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Following the Tocco operation, the mould packages were transported to the electromagnetic pump that filled up the packages with W319 alloy. Once the mould packages were filled, they were rolled over 180° and then transported to the fume extraction unit where the engine blocks were allowed to solidify (Figure 4.15e). The pouring and rollover operations took place over a period of ~25 s. The solidification of the engine blocks was conducted under natural heat exchange conditions. Then the packages were sent to Windsor Aluminum Plant for sand removal in the thermal sand removal furnace (Section 4.9.3). Figure 4.15f shows a picture of a sand free prototype V6 engine block.

4.9.3. Thermal Sand Removal, Solution Treatment and Artificial Aging of the Prototype Engine Blocks

Following the casting operation, the engine blocks were subjected to a solution treatment which started in the thermal sand removal unit. This process consists of heating at 495 ± 5°C for 4.5 h, followed by quenching with compressed air to 250 °C in 4.5 min (cooling rate of 0.9°C/s). The production line engine block was shipped to B&W Heat Treat Limited, in Kitchener, Ontario, which is the facility that heat treats the production V6 engine blocks. The heat treatment performed at B&W was a T6 (solution, quench and then aged) treatment. Table 4.8 summarizes the heat treatment conditions. Only the bulkhead sections (Figure 4.17) of the prototype engine blocks were subjected to a similar heat treatment as the one applied at B&W facilities. This heat treatment was conducted at the University of Windsor. The Temperite 2000 Lindberg resistance furnace was used for the heat treatments and the temperature was controlled using the AITAP platform.

4.9.4. Porosity Evaluation of the Bulkhead Sections of the V6 Engine Blocks

The porosity evaluation of the bulkhead sections was carried out according to Ford’s Engineering Materials Specifications (WSE-M2AA151-A3). The same procedures (as suggested in these specifications) were used to evaluate the soundness of the regular production and the four
prototype engine blocks cast with Al-Si cylinder liners. A brief description of the different tests conducted in the bulkhead sections is provided below:

a) Preparation of the Analytical Test Sample for Image Analysis (Porosity and SDAS)
b) Evaluation of the porosity in test bars and bulkhead sections (X-Ray Radiography)

4.9.5. Analysis of the V6 Engine Block Microstructure Using Light Optical Microscopy

The light optical microscope was used to assess the Secondary Dendrite Arm Spacing (SDAS) of the bulkhead sections of production and prototype engine blocks on the polished surface after the thermal sand removal treatment. The WSE-M2A151-A3 specification requires that ten dendrites need to be measured in total for a single metallographic sample; however, in the present work twenty five dendrites were measured on every side of the bulkhead section and standard deviation was calculated. This procedure was performed at the University of Windsor using the Leica 550 DMR. Figure 4.16 shows a picture of a polished bulkhead section showing the areas where the SDAS measurements were conducted.

Porosity measurements were also performed in the same areas using the Clemex image analysis system (JS-2000) at the NEMAK's Engineering Centre. Both image analysis system were calibrated using the respective Leica or Clemex calibration standards before any structures features were measured. The porosity analysis was performed using 25 fields at 50X magnification, which covered an area of 72 mm². The Ford Engineering Materials Specification WSE-M2A151-A3 required an analytical area larger than 25 mm² and that no pore should be >750 µm in any dimension (Ferret diameter).
4.9.6. Preparation of the Bulkhead Sections for X-Ray Radiography

The bulkhead sections were then radiographed at the NEMAK's Engineering Centre using the ASTM E155. In the four prototype engine blocks the bulkhead sections were removed allowing the cylinder liners, both cast iron and Al-Si, to remain intact. Figure 4.17 shows the rear part of the V6 engine block before and after the bulkheads were separated; every bulkhead was labelled with a number from i to iv from the front to the back of the engine block. This labelling was conducted to identify possible differences in mechanical and metallographic characteristics (strength and SDAS) and correlate these to their respective porosity levels. Prior to the radiographic analysis, the X-Ray apparatus is calibrated using the E155 standard.

4.9.7. Extraction of Tensile Bars from the Bulkheads Sections

All bulkhead sections were used for Brinell hardness tests using the ASTM Standard, B557M-94. The average and standard deviation are reported in Chapter 5. The bulkhead sections of the prototype engine blocks were divided in half to extract one test bar from the side with the cast iron cylinder and another test bar from the Al-Si cylinder liner side (Figure 4.18). A set of test bars of prototype engine blocks were tested after the thermal sand removal procedure and another set of test bars after heat treated conditions. Note: all engine blocks underwent the thermal sand removal procedure; the regular production engine block was provided in as heat treated conditions, therefore, no data for as thermal sand removal conditions is available.
4.1. Metallographic Sample Preparation for Metallurgical Analysis

All tensile test samples, test bars and automotive components were prepared for metallographic observations, image analysis and metal matrix microhardness measurements. Metallographic test samples were ground with SiC abrasive paper (grit number 240 to 1200 in five steps using finer paper each time). Polishing stages use a sequence of diamond suspensions...
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of 6, 3 and 1 μm with the final polishing carried out using 0.05μm silica slurry suspension. All operations with abrasive paper and diamond suspension(s) were performed using the semi-automated BUEHLER machine operated at 240 RPM for periods of 3 to 5 minutes depending on the surface quality of the test samples. The final polishing was performed at 60 RPM for 20 min.

4.10.1. Phase Characterization Using Light Optical Microscopy (LOM)

Following the polishing of the test samples they were analyzed under an automated Leica DMR LOM. The microscope is equipped with image analysis Q550IW software. A experimental computer program was designed using the Q550IW software which is capable for the analysis of eutectic and primary Si phases present on hypo and hypereutectic Al-Si alloys. This program assesses the SiML in eight levels of modification. The standard procedure to determine the SiML was conducted based on morphological characteristics such as area and perimeter of primary Si particles. The details of the SiML procedures are provided in Chapter 5. The procedure to determine the SiML involves the statistical study of 25 fields at 200 X magnification evenly distributed along the polished surface of the different test samples and/or automotive components. The analytical area of every field was 440 by 350 μm. Figure 4.19 shows the procedure to determine of the SiML indicating the distances among the analytical fields for the different test samples (Table 4.9).
Table 4.9. X and Y Distances for image analysis to assess the SiML of the Different Test Samples.

<table>
<thead>
<tr>
<th>Test Sample</th>
<th>X (μm)</th>
<th>Y (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard ESV</td>
<td>22000</td>
<td>16000</td>
</tr>
<tr>
<td>UMSA</td>
<td>2800</td>
<td>2600</td>
</tr>
<tr>
<td>Prototype Piston</td>
<td>12000</td>
<td>4000</td>
</tr>
<tr>
<td>Prototype Cylinder Liner</td>
<td>22000</td>
<td>4000</td>
</tr>
</tbody>
</table>

Figure 4.19. Schematic drawing of the distribution of the analytical fields to perform the SiML analysis for the various test samples and automotive components cast using reference and ESV treated melts. Measurements were conducted at 200 x magnification. X & Y values are the distances between the centres of the analytical fields.

4.10.2. Phase Characterization Using Scanning Electron Microscopy (SEM)

Metallographic observations were made using a JEOL JSM 5800 Scanning Electron Microscope (SEM) in the Back Scattered Electron (BSE) and Secondary Electron (SE) modes at different magnifications. The qualitative and quantitative Energy Dispersive X-ray Microanalysis of the selected samples’ structural constituents was done using the Noran EDX Spectrometer. Selected test samples were deep-etched using an HF 4% solution for 30s prior to SEM observations to clearly observe silicon and Cu enriched phases. The observation of the fractured surfaces of the tensile bars was conducted with secondary electrons mode (SEM/SE) at different magnifications varying from 100X to 5000X, to identify the type of fracture as well as to analyze the phases present on the fracture surface.
4.11. UMSA Test Samples Quenching Methodology

To examine the progress of solidification reactions during the liquid and semi-solid stages, series of test samples were quenched from intermediate (target) temperatures (see Figure 4.20). These target temperatures were established based on their characteristic nucleation temperature that was determined using the first derivative of the cooling curve recorded with UMSA for the respective 390.1(I) and 393.2(I) alloys. The target temperature for quenching was in all cases 10 \( \pm 5^\circ \text{C} \) above the nucleation temperature of each reaction. Test samples of both alloys were quenched at various temperatures above the liquidus temperatures. The target temperatures were determined to be a stage of solidification at which either phase nucleation occurs or when a phase is in the process of growing.

Prior to the quenching experiments, the UMSA test sample was wrapped using an AISI 304 stainless steel foil with an approximate thickness of 0.025 mm. The foil was coated using boron nitride solution to avoid any chemical reactions between the foil and the molten aluminum, preventing any molten metal leak. The coated foil was dried for 24 hours in a stove at a temperature of 180\(^\circ\text{C}\).

![Illustration of the quenching setup and cooling curves showing the effect of rapid solidification (quenching) at the target temperature on the cooling curve.](image)

Figure 4.20. a) Illustration of the quenching setup and b) cooling curves showing the effect of rapid solidification (quenching) at the target temperature on the cooling curve.
Chapter 4. Experimental Procedures

The target temperatures at which the UMSA test samples of the 393.2(1) alloy were quenched were 800°C, 770°C, 730°C and 650°C. For the 390.1(1) alloy the target temperatures were 730°C, 690°C, 660°C, 620°C, 560°C, 554°C, 525°C and 500°C. The schematic of quench set up is illustrated in Figure 4.20. The quenching operation was conducted by keeping the test sample stationary within the coil using a specially designed quenching holder [169]. From the target temperature the sample holder along with the UMSA test sample itself and the thermocouple were rapidly immersed into a bath of 1.2 kg of water-15 wt% - salt (NaCl) solution. This solution was maintained at a constant temperature of all ~20°C.

4.12. Mechanical Testing

4.12.1. Metal Matrix Microhardness Measurements

The metal matrix Vickers microhardness measurements were conducted using a Buehler microhardness tester under a 25g load for 15s (μHV_{25}). The Buehler microhardness tester was calibrated by performing ten indentations on a 200 μHV_{25} calibration standard. The microhardness of the standard was found to be 196 ± 7 μHV_{25}. The Vickers microhardness was performed as outlined by the ASTM E-384 Specification. Twenty measurements were performed on all test samples cast for the statistically designed experiments, including automotive components using reference and ESV treated melts. Selected test samples were also tested after heat treatment (heat treatment conditions available in Table 4.8). The average, standard deviation and confidence limits for 95 and 99 % were calculated. The 20 indentations were made in 2 parallel straight lines along the length of the test sample as shown in Figure 4.21. Matrix microhardness measurements were chosen instead of macrohardness to evaluate the possible effect of the ESV melt treatment on the matrix strengthening mechanism due to dissolution of Cu-Fe-Mg-Ni-Si enriched phases. The Homogeneity Coefficient (HC) of the aforementioned test samples was determined using the following equation [168].

\[
HC = \frac{(S_A)^2 - (S_R)^2}{(S_R)^2} \quad [168]
\]

\((S_A)^2\) - Standard deviation of a selected reference test sample.
\((S_R)^2\) - Lowest standard deviation measured among the test samples.
HC - Homogeneity coefficient.
4.13. Tensile Testing

The tensile testing was conducted in the IRC’s laboratory following the procedure described in the ASTM E-8 standard. The testing was performed at room temperature using an Instron 8801 axial servohydraulic dynamic two column load frame. The static load was monitored using a dynamic load cell having a maximum capacity of 100 KN. A strain gauge extensometer of 25.4 mm was attached to the test bar for measuring sample elongation. The self calibration of the Instron system was performed prior to every test according to the procedure in the manual.

The tensile test bars were machined from the die cast test samples (Figure 4.13) and from the bulkhead sections (Figure 4.18). Sets of five test bars cast under same conditions were tested as-cast, artificial aging and solution treatment. The average value of 0.2% offset yield strength (YS or σy), Ultimate Tensile Strength (UTS) and Elongation [173] together with standard deviation and the 95% confidence limits were calculated.
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Figure 4.22. Schematic drawing of the tensile test bars and machined dimensions for the die cast test samples and the extracted test bars from the bulkhead sections (ASTM E-8). Note: all dimensions are in mm.

According to the ASTM standard, the Yield Strength was determined by using the offset method of 0.2%.
CHAPTER 5. RESULTS AND DISCUSSIONS

5.1. Preliminary Experiments

5.1.1. Results and Discussions on the Effect of Electromagnetic Stirring (ES)

The preliminary experiments were conducted using an ES technique for Al-Si hypoeutectic alloys (W319). For the results of the ES experiments three potential applications were anticipated and three limitations were revealed for the treatment of Al-Si hypereutectic alloys.

1) Partial elimination of the dendritic structure (Figure 5.1),
2) A reduction of the grain size up to ~6 times (Figure 5.3),
3) Ability to assess the determination of the level of modification by using thermal analysis (Figures 5.1 and 5.3),
4) This treatment is effective in semi-solid state only,
5) No refinement of the Al-Si and Al-Cu enriched phases occurred (Figure 5.2) and
6) There was no microstructure refinement of phase modification on the Al-Si hypereutectic alloys.

![Figure 5.1. Microstructure (50x) of the reference W319 alloy test sample under LOM a) reference test sample and an b) ES treated test sample with an $I_{AC} = 8$ A. All test samples were solidified at a cooling rate = 0.12°C/s. Note: changes in the α-al matrix morphology (#1) and the unmodified Al-Si eutectic (#2) and Cu enriched phases (#3).](image)

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Chapter 5. Results and Discussions

Figure 5.2. SEM/SE micrographs (1000 X) of deep etched W319 alloy test samples solidified at 0.12°C/s. a) Reference and b) ES treated (I_{AC} = 8A). Note: matrix (#1), Al-Si eutectic (#2), Cu enriched phases (blocky #3a, eutectic #3b and fine eutectic #3c).

Table 5.1. Characteristic temperatures of selected metallurgical reactions for the reference and ES treated test samples for the W319 alloy.

<table>
<thead>
<tr>
<th>AC Current (A)</th>
<th>Cooling Rate (°C/s)</th>
<th>Liquidus (°C)</th>
<th>Liquidus Undercooling (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td></td>
<td>601.8</td>
<td>1.62</td>
</tr>
<tr>
<td>2</td>
<td>0.11</td>
<td>604.2</td>
<td>0.95</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>604.8</td>
<td>0.74</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td>605.5</td>
<td>0.57</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>605.9</td>
<td>0.47</td>
</tr>
</tbody>
</table>

Figure 5.3. Effect of ES treatment conditions (AC current) on grain size and undercooling on the α-Al reaction of the investigated W319 alloy.
Based on these results, it was found that in order to develop a technology capable of refining the microstructure of the Al-Si hypereutectic alloys more effective refining methods need to be developed. The literature review indicated that the ESV technique could possibly refine Al-Si hypereutectic melts. Utilization of thermal analysis to assess the refinement of the microstructure could be useful for industrial applications.

5.2. Results and Discussions of the Preliminary Experiments Using Electromagnetic Stirring and Vibration (ESV)

The micrographs of the test samples presented in this section correspond to experiments conducted under different ESV melt treatment conditions. The general idea of these experiments was to understand the effect of independent variables (such as current, treatment temperature and time) but also the effect of ESV melt treatments while using various workstations. All these experiments were conducted using the 390.1(II) alloy. In fact, the following results are the most representative along with the preliminary experiment sections. There is the potential to patent the ESV workstation (ETCT) [6].

5.2.1. Results of the Preliminary Experiments Using the Electromagnetic Stirring and Vibration Workstations

The preliminary experiments conducted using the ESV workstations were performed under different electrical conditions as well as with ESV cups, times and temperatures. Selected examples of the results are showed below, which correspond to the best ESV melt treatment capabilities of each workstation. The values of the electrical parameters under which the test samples were ESV melt treated using the different workstations are presented in Table 5.2. The values provided for the electrical current with the electrical filter developed by Mr. J. Ohl [21] correspond to the reduction of the unwanted induced AC current on the DC electromagnetic coil (underlined data in Table 5.2).
The phase identification (see Figures 5.4 to 5.8) was conducted using LOM. In order to have an idea of the elements that compose the corresponding phases, a qualitative chemical analysis using SEM/EDX was performed. Using LOM and SEM/EDX four phases and porosity were identified in the 390.1(II) alloy and are listed in following:

1. Aluminum matrix.
2. Primary Si.
3. Eutectic Si.
4. Al-Cu-Mg-Ni-Cr-Mn-Fe enriched phases.
5. Porosity.

Table 5.2. Electrical parameters used for the ESV melt treatments conducted using the different workstations. Note: in all cases the frequency of the AC current was 60 Hz [6, 21].

<table>
<thead>
<tr>
<th>System</th>
<th>Current, (A)</th>
<th>Voltage, (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AC coil</td>
<td>DC coil</td>
</tr>
<tr>
<td>Two coils in parallel</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Two coils in parallel with filters</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Two coils in series</td>
<td>42</td>
<td>66</td>
</tr>
<tr>
<td>Two-coil system with a ferromagnetic core</td>
<td>70</td>
<td>100</td>
</tr>
<tr>
<td>Two-coil system with a ferromagnetic core with filters</td>
<td>80</td>
<td>80</td>
</tr>
<tr>
<td>Two-core-coil</td>
<td>80</td>
<td>80</td>
</tr>
</tbody>
</table>

5.2.1.1. Microstructure of the Reference Test Sample for the 390.1(II) Alloy

The reference test samples for the 390.1(II) alloy were allowed to solidify under natural heat exchange conditions inside the ESV coils. The same conditions were used for the ESV melt treated test samples. Figure 5.4 shows LOM micrographs of the 390.1(II) reference test sample where it was observed that the microstructure was similar to that of a regular piston taken from the regular production in the die casting process at Essex Aluminum Plant (Figure 5.54). The constituents of the 390.1(II) alloy were coarse. The reference test sample exhibited primary Si macrosegregation. Cu and Fe rich phases could be found together in the inter dendritic space.
5.2.1.2. Two-Coil Workstation in Parallel Connection

Figures 5.5a and 5.5b present LOM micrographs of a 390.1(I) ESV melt treated test sample using the two-coil workstation in parallel connection. The electrical parameters used to cast this particular test sample were $I_{AC} = 44 \text{ A}$, $I_{DC} = 66 \text{ A}$, $f = 60 \text{ Hz}$, $t = 3 \text{ min}$, $T_{\text{Range}} = 680 \text{ to } 640 \pm 2^\circ\text{C}$ using the AC filter. The microstructure characteristics of an ESV treated test sample under these conditions exhibited a high level of structure refinement and homogeneity. The identified phases in these test samples show an almost negligible amount of primary Si particles, isolated dendritic areas, a network of not fully refined Si eutectic particles and refined Cu and Fe phases. The primary Si particles were refined and solidified as Al-Si eutectic; therefore, the number of Si eutectic particles is higher while compared with the reference sample (Figure 5.4).
5.2.1.3. Results of the ESV Melt Treated Test Samples Using the Two-Coil Workstation in Series Connection

Figure 5.6 shows LOM micrographs of the 390.1(II) alloy ESV melt treated in the two-coil workstation connected in series using $I_{ACDC} = 100 \, A$, $f = 60 \, Hz$, $t = 3 \, min$, $T_{Range} = 680$ to $640 \pm 2^\circ C$ without the AC filter. The microstructure of this test sample presents a considerable level of modification mostly in the Al-Si eutectic, Cu and Fe enriched phases. The microstructure is less dendritic with an increased amount of Al-Si eutectic due to the refinement of the primary Si particles, which were transformed into eutectic. Cu enriched phases are highly refined, while Fe enriched ones can be observed with their typical Chinese script morphology.

Figure 5.6. LOM micrographs of the test sample treated in the Two-Coil system in series connection using $I_{ACDC} = 100 \, A$, $f = 60 \, Hz$, $t = 3 \, min$, $T_{Range} = 680$ to $640 \pm 2^\circ C$ without the AC filter at a) 50X and 200X magnification. Note: #1 a-Al Matrix, #2 the refined Al-Si eutectic, #3 refined Al-Cu-Ni enriched and #4 partially refined primary Si particles.

5.2.1.4. Results of the ESV Melt Treated Test Samples Using the Two-Coil Workstation with a Ferromagnetic Core

LOM of the 390.1(II) alloy test sample ESV melt treated in the two-coil with a ferromagnetic core under $I_{ACDC} = 80 \, A$, $f = 60 \, Hz$, $t = 3 \, min$, $T_{Range} = 680$ to $640 \pm 2^\circ C$ by using the AC filter are shown in Figure 5.7. The $I_{ACDC}$ cannot exceed the 80 A limit because, using the two power supplies and the ferromagnetic cores the resistance was low in the system; therefore, it
was impossible to go beyond this current level. The microstructure refinement of these test samples presents highly modified phases; most of the primary Si was transformed into Al-Si eutectic. However, there was found a section in the test sample containing fine primary Si particles. Moreover, was also found the so called fish bone Al-Si phase, which is in fact an "intermediate" phase between the primary and the eutectic Si [76, 114, 106].

![Micrographs of test sample 390.1(I) alloy ESV melt treated in the Two-Coil workstation.](image)

**Figure 5.7.** LOM Micrographs of the test sample 390.1(I) alloy ESV melt treated in the Two-Coil workstation with a ferromagnetic core using $I_{ACDC} = 80$ A, $f = 60$ Hz, $t = 3$ min, $T_{Range} = 680$ to $640 \pm 2^\circ$C with the AC filter at a) $50$ X and $200$ X magnification. Note: 1) α-Al matrix, 2) the father-like Al-Si eutectic and 3) refined Fe enriched phases.

### 5.2.1.5. Results of the ESV Melt Treated Test Samples Using the Two-Core-Two-Coil Workstation Connected in Series

The ESV melt treated 390.1(II) alloy in the two-core-coil workstation using an $I_{AC} = 80$ A, $f = 60$ Hz, $t = 3$ min, $T_{Range} = 680$ to $640 \pm 2^\circ$C without the AC filter are presented in Figure 5.8. Under the above-mentioned conditions the ESV melt treated test samples had the highest level of modification. The level of modification is such that using LOM micrographs at $50X$ magnification it was impossible to distinguish the phases. At $200X$ magnification the Mg, Ni and Cu enriched phases were identified. This microstructure is primary Si free, with isolated dendrites. The characteristic eutectic observed on the microstructure of this test sample is known as the "coral-like" and in this particular case each "coral finger" is smaller than $1 \mu m$. Using conventional melt treatments such as chemical, thermal, mechanical and electromagnetic treatment techniques yet been reported in the literature.
These workstations demonstrated two main characteristics that were ideal to justify this project; the possibility to modify Al-Si hypereutectic alloys at temperatures above liquidus and the possibility to cast components from the ESV treated melt. The level of microstructure modification of the two-coil-two-core workstation was found to be the highest; this workstation also offers the possibility to focus the electromagnetic field at a specific point. This allows to conduct ESV melt treatments without damaging the electromagnetic coils, which is beneficial. This workstation was a permanent workstation because the price of the ferromagnetic cores (>20,000 USD) exceeded the budget of the project. Table 5.3 describes the advantages and disadvantages of each workstation; and was presented in M. Sc. Thesis of Mr. J. Ohl [21].
Table 5.3. Advantages and disadvantages of the ESV melt treatment workstations [21].

<table>
<thead>
<tr>
<th>Method</th>
<th>Advantage of the method</th>
<th>Disadvantage of the method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Two-coil workstation in parallel</td>
<td>• Current sources available in the IRC’s laboratory</td>
<td>• Generation of the AC current component on a DC coil</td>
</tr>
<tr>
<td></td>
<td>• Possibility to change the direction of the DC field</td>
<td>• Close proximity to heat source (tested sample)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Electromagnetic field is uneven</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Mechanical vibration due to push-pull forces</td>
</tr>
<tr>
<td>Two-coil workstation in series</td>
<td>• More uniform electromagnetic field than in parallel configuration</td>
<td>• Substantially lower current flow through coils</td>
</tr>
<tr>
<td></td>
<td>• Weaker mechanical vibration due to push-pull forces</td>
<td>• Close proximity to heat source (tested sample)</td>
</tr>
<tr>
<td></td>
<td>• Elimination of air-coupling (generation of the AC component)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• More turns (two coils together)</td>
<td></td>
</tr>
<tr>
<td>Two-coil workstation with a ferromagnetic core</td>
<td>• Uniform electromagnetic field</td>
<td>• Very strong generation of the AC current component on a DC coil</td>
</tr>
<tr>
<td></td>
<td>• Removes the coil from close proximity to the test sample</td>
<td>• Mechanical vibration due to push-pull forces</td>
</tr>
<tr>
<td></td>
<td>• Creates an opportunity to add more turns, and hence, makes the magnetic flux density even higher</td>
<td></td>
</tr>
<tr>
<td>Two-core-two-coil workstation</td>
<td>• Uniform electromagnetic field</td>
<td>• Experiences some slight mechanical vibration similar to that of a transformer</td>
</tr>
<tr>
<td></td>
<td>• Complete elimination of the AC current on a DC coil</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Removes the coil from close proximity to the test sample</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Creates an opportunity to add more turns, and hence, makes the magnetic flux density even higher</td>
<td></td>
</tr>
</tbody>
</table>

5.3. Assessment of Liquidus and Superheat Temperatures of Al-Si Hypereutectic Alloys

For a binary Al alloy, the liquidus temperature for a given chemical composition can easily be derived with a high level of accuracy, from the Al-Si phase diagram. However, there are only a limited number of sources that report the relationship between liquidus temperature and chemical composition for a multi-component hypoeutectic aluminum alloy, that can be used for industrial applications [201, 17]. Moreover these equations do not address melt treatment or its cleanliness.
Recently, algorithms were developed, based on the Silicon Equivalency (SiEQ) method, which took into consideration the effect of alloying elements, impurities, dissolved gases (H2) in the alloy and melt treatments on the liquidus temperature for the hypoeutectic Al-Si alloy [199]. The SiEQ method was derived from the binary equilibrium phase diagrams, for Al - Xi (where Xi could be any element) systems that are predominantly the eutectic type. In most cases their liquidus temperature drops uniformly from the melting point of pure Al ($T_{\text{MELT, Al}} = 660.45°C$) to the temperature of their eutectic composition (for Al-Si it is 12.3 wt% Si) [204]. Using a similar approach as for the SiEQ algorithms developed by Djurdjevic et. al. [199] for Al-Si hypoeutectic alloys. In the present research the above-mentioned algorithms were used and corrected and a new SiEQ for Al-Si hypoeutectic alloys was made [18]. The following are the algorithms the liquidus temperature of a multicomponent Al-Si hypoeutectic (Eq. 5.1 [18, 199]) and hypereutectic (Eq. 5.2 [18]) alloy.

$$T_{LQ}^{Al-Si} \cdot \sum_{Xi} X_i = 660.452 - 6.11 \sum Si_{EQ}^{Xi} - 0.057 \sum (Si_{EQ}^{Xi})^2 \quad \text{(°C)}$$  \hspace{1cm} \text{Eq. 5.1}

$$T_{LQ}^{Al-Si} \cdot \sum_{Xi} X_i = 389.79 + 15.855Si - 0.0561(Si)^2 + 3.14 \sum Si_{EQ}^{Xi} + 0.057 \sum Si_{EQ}^{Xi}^2 \quad \text{(°C)}$$  \hspace{1cm} \text{Eq. 5.2}

where: $Si$ is the amount of Si on the specific alloy in wt%

$\sum Si_{EQ}^{Xi}$ is the corresponding Si equivalency contribution of all the minor and major elements to the specific composition of the alloy.

Figure 5.9 shows the actual liquidus lines of the Al-Si phase diagram (solid lines) [207] and the calculated liquidus lines (dashed lines) using Eq. 5.1 and Eq. 5.2 for hypo and hypereutectic alloys respectively. The liquidus temperatures for the chemistry presented in Table 4.1 were calculated using the SiEQ method. The liquidus temperatures for the alloys, used in this work were measured by with AITAP and plotted in Figure 5.9 as dots [17].

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5.4. Phase Identification of the Al-Si Hypereutectic Alloys

5.4.1. Characterization of As-Cast Al-Si Hypereutectic Test Samples Using SEM and LOM

As shown in Figure 5.9 the superheat temperature was set to a constant value for the specific compositions of the investigated alloys. It is important to consider that the reported values for each chemical composition in Table 4.1 are the average for a few OES analysis conducted in various ingots. Figure 5.9 shows the differences in composition for distinct ingots of the same alloy having similar liquidus temperatures.

Figure 5.9. Literature (solid liquidus line) [204], calculated (dashed line) [18] and measured liquidus temperatures (dots) on a “multicomponent Al-SiEQ phase diagram” using the SiEQ algorithms for experimental 3XX.X compositions of the AAA alloy designation [18].
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5.15. The qualitative chemical analysis of all phases was conducted by SEM/EDX the results are shown in Figure 5.12a to 5.12h.

![SEM/BSE micrographs](image)

**Figure 5.10.** SEM/BSE micrographs of the reference 390.1(I) alloy showing the phases with their respective identification at a) 100 X, (b and c) 500 X and d) 1000 X magnification.

There are several Fe-based stoichiometries that have a script type appearance and these are Al₈Fe₂Si, α-Al₁₃(Fe, Cr, Mn)₃Si₂, π-Al₁₃Mg₃FeSi₆ and the π-Al₁₃Mg₃Cu₂Si₆ phases [169]. Through the process of elimination based on the EDX spectrum (Figure 5.12b) the phases Al₈Fe₂Si, α-Al₁₃Fe₃Si₂, π-Al₁₃Mg₃Cu₂Si₆ and π-Al₁₃Mg₃FeSi₆ can be ruled out; since Mg Kα or Kβ reflections were absent in the EDX spectra. The presence of Cu is very weak in the EDX spectra thus it was probably due to the vicinity of the Fe rich phase with the Cu and/or Ni enriched phases. However, the Cr and Mn reflections were present, leading the identification of the α-Al-Fe-Si type as α-Al₁₃(Fe, Cr, Mn)₃Si₂ as most probable stoichiometry (Figure 5.11).
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Figure 5.11. SEM/BSE micrographs of the reference 393.2(I) alloy showing the Ni, Cu, Fe enriched phases (a, b) 500 X magnification.

The precipitation sequence of the Al-Si hypereutectic alloys commences at the liquidus temperature by the precipitation of the primary Si followed by the Al-Si eutectic. Neither phase is dendritic, therefore alloys such as 390.1(I), 390.1(II), 393.2(I) and 393.2(II) are dendrite free when they are allowed to solidify at equilibrium conditions [38, 76, 77, 126]. However, in most cases castings are forced to solidify under non-equilibrium conditions, resulting in the formation of a halo in the surrounding primary Si particles [76 - 80]. The haloes are impoverished Si zones rich in Al and are called α-Al haloes (Figures 5.13, 5.14 and 5.17). The α-Al halo has a similar composition to the α-Al dendrites with a tendency to form dendrites. Once the α-Al haloes are at the end of their precipitation stage, the nucleation of the α-Al dendrites and the Al-Si eutectic starts. With sufficient undercooling α-Al dendrites nucleate out of the couple zone and their coarsening path forms a skeleton together with the primary Si particles and the Al-Si eutectic (Figure 5.17) [169, 170].

As a result of the solidification of the test samples under non-equilibrium conditions, the Dendrite Coherency Point (DCP) occurs prior to the nucleation of the Al-Si eutectic. However, in most cases the dendrites present are isolated and they rarely touch each other. Figure 5.17 shows the typical LOM microstructures of the 390.1(I) and 393.2(I) alloy test samples solidified under different conditions showing in most cases that dendrites do not touch each other.

The 390.1(I) alloy used in this research has a Ni content between 2 and 2.5 wt% Ni which in most cases was detected by EDX together with Cu. Ni was identified forming the Al-Ni
eutectic, and the Al-Cu-Ni phases. The characterization and phase identification of the 390 alloy is presented by Bäckerud [76], however the level of Ni present is negligible, thus is not reported. Therefore, Ni enriched phases were not identified. Nonetheless, the reported [76] chemical composition of the 390.1 alloy has ~1.5 wt% Ni with 11 wt% Si and 1 wt% Cu, which is similar to the 390.1(I) alloy investigated in this research. For the 339.1 alloy two Ni enriched phases were identified, which are the Al3Ni and the Al3(Ni, Cu) that correspond with the EDX findings conducted on the 390.1(I) alloy (Figures 5.12c, d and f).

The Al-Cu enriched phases were identified by Bäckerud [76] as “complex eutectic reactions forming Al2Cu and other phases”. However, based on thermal analysis for the 390.1(I) and 393.2(I) alloys three characteristic reactions were identified, which correspond to the LOM and SEM phase identification of both the 390.1(I) and 393.2(I) alloys (Figures 5.10, 5.11, 5.13, 5.16 and 5.19). M. B. Djurdjevic et. al. [191] conducted thermal analysis together with phase identification and reported the stoichiometry of three Al-Cu enriched phases (Table 5.4). It is important to mention that his work was conducted using the W319 alloy, however, Bäckerud [76] and Dahle et. al. [38, 126] agree that after the nucleation of the Al-Si eutectic the solidification paths of Al-Si hypereutectic alloys are similar to the hypoeutectic ones.

Table 5.4. Stoichiometric composition of the Cu enriched phases found in the W319 alloy [13].

<table>
<thead>
<tr>
<th>Morphology of the Cu Enriched Phases</th>
<th>Stoichiometric Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>&quot;Blocky&quot;</td>
<td>Cu10.59Al4.09Si2.82Mg2.48</td>
</tr>
<tr>
<td>&quot;Eutectic&quot;</td>
<td>Cu7.27Al12.25Si10.47</td>
</tr>
<tr>
<td>&quot;Fine Eutectic&quot;</td>
<td>Cu4.14Al13.32Si1.72Mg0.81</td>
</tr>
</tbody>
</table>

The Ni and Cu were found in the EDX results because these elements (atomic numbers 28 and 29) have complete solubility in solid, semi-solid and liquid states; with the same crystalline structure (FCC), similar electro negativity, valency and lattice parameters ($a_{Cu} = 3.61$ Å and $a_{Ni} = 3.55$ Å). Based on the Hume-Rothery Empirical Rules, Cu and Ni form an ideal solution [192, 193], where atoms can freely interchange their positions within the lattice [16, 194]. Thus the reflections of Ni in the SEM/EDX spectra on the Al-Cu-Ni phase were a consequence of the high solubility of both elements.
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Figure 5.12. SEM/EDX spectra of the identified phases for the 390.1(1) Al Alloy. a) primary Si, b) Al15(Fe, Cr, Mn)3Si2, c) Al2Cu eutectic, d) Mg2Si. The SEM accelerating voltage was 15 kV and 2000 x magnification.

Primary and eutectic Si particles, are the main interest of this study because they affect the wear resistance of Al-Si alloys due to their high hardness; conversely, coarse particles of primary Si are brittle [79, 81]. During solidification the primary Si particles grow based on the layer growth mechanism; this mechanism consists on an ordered deposition of epitaxial layers of Si atoms [81]. Due to the similarity in atomic weight between Al and Si (13 and 14 g/mol respectively) SEM/EDX does not allow for an extensive characterization of the previously mentioned phases [192, 193]. Nonetheless, SEM observations of deep etched test samples permit the observation of three dimensional images of silicon (Figure 5.16).
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Figure 5.12. Continuation of the SEM/EDX spectra of the identified phases in the 390.1(I) Al alloy. e) Al$_3$NiCu blocky eutectic, f) Al-Cu fine eutectic, g) Al-Ni eutectic and h) Pb based. The SEM accelerating voltage was 15 kV and 2000 X magnification.

LOM images (in 2D) show the primary Si particles changing from polyhedral (usually rectangular or trapezoidal) to plate like (in 3D) for the 390.1(I) and 393.2 alloys respectively. Figures 5.13, 5.14 and 5.17 are the evidence that the Si content in the Al-Si hypereutectic alloys has an effect on the size of these particles [195]. The cooling rate is another factor that has an influence on the size and morphology of primary Si particles. By increasing the cooling rate from 0.08 to >1°C (test samples solidified under natural heat exchange conditions and die cast respectively) the plate like structure is transformed into the rectangular in the 393.2 alloy. Primary Si particles were also found in the form of a star shape or hollow plates (Figures 5.14 and 5.17). These types of primary Si were reported by H. Yi et. al. [106, 107] and J. E. Gruzleski [81]. Due to the brittleness of the primary Si particles in the case of friction (i.e. polishing or wear) they crack leaving the α-Al matrix exposed and reducing the wear resistance of these alloys. Figures 5.13 to 5.17 present LOM micrographs of the 390.1 and 393.2 test samples solidified at different cooling rate.
Pb based particles were observed at 500X magnification, however at 1,000X magnification their presence was more evident (Figure 5.10d). The Pb based particles are commonly found in Al-Si alloys and are a by-product of the secondary smelting process [169]. The respective SEM/EDX spectrum for the Pb particles is shown in Figure 5.12h.

Figure 5.13. Microstructures of the reference 390.1(1) Al-Si alloy test sample (cooling rate = 0.08°C/s) at a) 100 X, b, c) 200 X and d) 500 X magnification. Note all phases were characterized based on the SEM/EDX results.
Figure 5.14. Microstructure of the heterogeneous areas of a) primary Si and b) Al-Si eutectic of a reference test sample for the 393.2(I) alloy (cooling rate \(-0.16^\circ\text{C/s}\)) at 100 X mag.

Figure 5.15. LOM and SEM/BSE micrographs of a reference prototype cylinder liner cast with 390.1(I) (cooling rate \(1.6^\circ\text{C/s}\)) alloy at a) 50 X, b) 200 X, c) 100 X and d) 500 X magnification.
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Figure 5.16. SEM/SE micrographs of a reference test sample of the 390.1(I) alloy deep etched microstructures a) eutectic Si plates, b) primary Si particles [13 - 15].

Figure 5.17. LOM micrographs of a reference test samples for the 390.1(I) (a,b) and 393.2(I) (c,d) alloys solidified at (a,c), cooling rate ~ 0.08°C/s and 0.16°C, and (b,d) cooling rate ~1.6°C/s, 25 X magnification. Note the coarse primary Si particles and the structure heterogeneity in the 393.2(I) alloy.

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5.4.2. Metallurgical Characterization of ESV Melt Treated Test Samples for Al-Si Hypereutectic Alloys Using SEM and LOM Techniques

The microstructures of the ESV melt treated 390.1(I) alloy show the same phases as those in same alloy solidified using untreated melt. The ESV melt treatment conditions of these particular test samples were $I_{ac/dc} = 100 \text{A}$, $f = 100 \text{Hz}$, $t = 3 \text{ min}$ in temperature range of 650 to $610 \pm 2^\circ \text{C}$ with the ESV workstation (ETCT). After the ESV melt treatment the test samples were allowed to solidify under natural heat exchange conditions (cooling rate $\sim 0.08 \text{ °C/s}$). Figures 5.18a to 5.18d show the similarity with the 390.1(I) reference test sample (Section 5.4.1) and the presence of the $\alpha$-Al matrix. The presence of the $\alpha$-Al$_5$(Fe, Cr, Mn)$_3$Si$_2$ phase can be observed in Figure 5.18b. In the present micrograph the Chinese script structure is not evident, this is due to the fact that a micrograph is an image of the cross section of a test sample where the particles were cut in an arbitrary direction, which as a result only a section of this phase can be observed.

By using SEM/BSE the Cu and Ni enriched phases in the ESV melt treatment test samples were identified (Figures 5.18a to 5.18d). Pb particles were also found and can be observed in Figure 5.18b. The spectra of the identified phases using the SEM/EDX are similar to those in the respective reference test samples (Figures 5.12a to 5.12h).

The refined primary Si particles in the ESV melt treatment test samples were transformed from coarse polyhedral crystals into refined Al-Si eutectic. The stirring and vibration created during the ESV melt treatment break apart the Si agglomerates, therefore, the number of Si agglomerates increases. Every new Si agglomerate behaves as a nuclei resulting in the multiplication of the primary Si particles as a consequence a refined Si structure is created. Comparing Figure 5.13 with Figure 5.20a it can be observed that due to the effect of the ESV melt treatment the number of Al-Si eutectic particles was increased. SEM/BSE micrographs of deep etched test samples reveal the three dimensional morphology of the Al-Si eutectic as well as the Fe Chinese script phase (Figure 5.19). In this figure it can be observed that Al-Si eutectic "plates" were transformed by the ESV melt treatment into rounded particles with a coral like morphology (Figure 5.16).

The Mg, Ni and Cu enriched phases were also identified using LOM (Figure 5.20) and these observations confirm the SEM/BSE results. Due to the refinement of the microstructure of
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the ESV melt treated test samples, the full characterization of the Al-Cu phase was difficult using LOM or SEM, therefore, techniques such as TEM would be more ideal. Figure 5.20d presents the Mg$_2$Si phase also identified under LOM at a higher magnification (500 X).

![Image of micrographs](image.png)

**Figure 5.18.** SEM/BSE micrographs of the test sample for the 390.1(1) Alloy ESV melt treated using $I_{AC/DC} = 100$ A, $f = 100$ Hz, $T_{range} = 650$ to $610^\circ$C for 3 min, solidified under natural heat exchange conditions at a cooling rate ~ 0.08°C/s. Micrographs a) 100 X, (b and d) 500 X and c) 1000 X magnification.

Test samples for the 390.1(I) and 393.2(I) alloys were ESV melt treated and solidified at a cooling rate of cooling rate > 1°C/s. This cooling rate was the result of solidifying the test samples in the permanent moulds designed for automotive components in this dissertation for prototype pistons and cylinder liners. Figure 5.21a shows LOM with Al-Si eutectic particles highly refined, as well as the Mg, Ni and Cu enriched ones (390.1(I) alloy). In Figure 5.21b LOM of a test sample for the 393.2(I) alloy with isolated primary Si particles that are considerably refined and that have very refined dendrites are
presented. Due to the level of modification and the highly refined microstructure was not possible to make any further identification of the rest of the phases; this is in part due to the limit of the SEM-EDX spatial resolution (see Figure 5.22).

The microstructure of the ESV melt treated prototype pistons and cylinder liners appears primary Si free and the present eutectic particles are in the form of a coral-like structure (Figure 5.22). Isolated dendrites were also found using LOM at 50X and 200X magnification. Using SEM/BSE micrographs Mg, Ni, Cu enriched phases and Pb particles were identified at 100X magnification. The above-mentioned phases were observed homogeneously distributed and refined (Figure 5.22b). The Al$_{15}$(Fe, Cr, Mn)$_2$Si$_2$ phase has a blocky appearance and was found in the vicinity of the Al-Ni eutectic phase. The Al-Cu-Ni enriched phases were found in two eutectic morphologies one in the typical semi-blocky and the other one in a plate like, which due to the effect of the two dimensional image these particles appear to be identified in the form of needles.

SEM/BSE conducted in deep etched test samples show the presence of Al-Si eutectic in the form of a coral-like structure (Figure 5.23). Figures 5.23a to 5.23b are the evidence that the cooling rate and the ESV melt treatment combined together have a positive effect resulting in a significant refinement of the microstructure.

![Figure 5.19. SEM/BSE micrographs of a deep etched test sample of the 390.1(I) alloy ESV melt treated ($I_{ACDC} = 100$ A, $f = 100$ Hz, $T_{range} = 650^\circ$C to 610$^\circ$C for 3 min, solidified under natural heat exchange conditions at a cooling rate ~ 0.08$^\circ$C/s [13 - 15].](image-url)
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Figure 5.20. LOM micrographs of the ESV melt treated 390.1(I) alloy at $I_{AC/DC} = 100 \, A$, $f = 100 \, Hz$, $T_{Range} = 650-610 ^\circ C$ for 3 min, cooling rate $\sim 0.08 \, ^\circ C/s$ at a) 100X, b) 200X and (c,d) 500 X.

Figure 5.21. LOM of ESV melt treated a) 390.1(I) and b) 393.2(I) Al-Si alloys using $I_{AC/DC} = 100A$, $f = 100 \, Hz$, $T_{Range} = 650 - 610 \, ^\circ C$ for 3 min, cooling rate $\sim 1.6^\circ C/s$ cast in the die cast permanent mould for prototype cylinder liners at 100 X magnification.

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Figure 5.22. LOM and SEM/BSE micrographs of a prototype piston and or cylinder liner cast with the ESV melt treated 390.1(l) alloy using a $I_{ACDC} = 100$ A, $f = 100$ Hz, $t = 3$ min, and a $T_{ESV} = 610 \pm 2^\circ$C at a) 200 X, b) 100 X, c) 500 X and d) 2500 X magnification.

Figure 5.23. SEM/BSE micrographs of the test sample for the ESV melt treated 390.1(l) alloy using $I_{ACDC} = 100$ A, $f = 100$ Hz, $T_{range} = 650^\circ$C to 610$^\circ$C for 3 min cast in a steel permanent mould at a cooling rate $\sim 1.6^\circ$C/s [13 - 15].
5.5. Thermal Analysis of Al-Si Hypereutectic Alloys

Thermal analysis performed on Al-Si hypoeutectic alloys is well known. The IRC has reported and published several documents [182, 197-200]. Other researchers have also presented thermal analysis of Al-Si hypoeutectic alloys. Unfortunately, there is not as much information about thermal analysis for Al-Si hypereutectic alloys. Bäckerud et al. [76] was the consulted reference in order to compare the thermal analysis results of the Al-Si hypereutectic alloys. The effect of electromagnetic stirring on solidification was reported by Griffiths et al. [39, 41], but the resolution of their cooling curves does not allow for in-depth analysis of the characteristics.

In the present work thermal and characterization analysis was conducted for the 390.1(I), 393.2(I) and 393.2(II) alloys using the regular thermal analysis (AITAP) and the advanced thermal analysis (UMSA) platforms under similar conditions. For all the reference test samples the solidification procedure was conducted under natural heat exchange conditions. In this type of solidification process the release of the heat must be conducted in the radial (r) direction (1 dimension (1D)) to minimize the gradient of temperatures within the test sample. The algorithms developed by Dr. W. T. Kierkus et al. [182] for Al-Si hypoeutectic alloys were used and to conduct the cooling curve analysis for the 390.1(I) and 393.1(II) alloys used in this dissertation. These algorithms include the determination of the first derivative, baseline, integral(s) and fraction solid (Appendix I).

5.5.1. Thermal Analysis Procedure for Al-Si Hypereutectic Alloys

The cooling curve and first derivative of the reference test sample for the 390.1(I) alloy using UMSA was recorded during the heating and cooling cycles and these are presented in Figures 5.24a and b. In the calculated first derivative, eleven metallurgical reactions were identified, which are summarized in Table 5.5. A number was assigned to each reaction to facilitate its identification (see Table 5.5). The recorded temperatures during the heating cycle in UMSA were used for analysis, since during this process the heating source (electromagnetic coil) is introducing energy. This energy affects the accuracy to determine the temperatures, therefore, only the cooling cycle was considered to conduct the thermal analysis (Appendix I).
Figure 5.25 shows the characteristic cooling curve, first derivative and baseline for the 390.1(1) alloy recorded using AlTAP in the standard ESV cup solidified under natural heat exchange conditions (1D). The first derivative curve was determined with the aim to accurately identify the specific temperatures at which the solidification reactions take place in Al-Si hypereutectic alloys. The cooling rate is determined as follows:

$$\text{Cooling Rate} = \frac{(T_{LQ} - T_{SOL})}{(t_{SOL} - t_{LQ})} \quad \text{Eq. 5.3}$$

where $T_{LQ}$ is the liquidus temperature.  
$T_{SOL}$ is the solidus temperature. 
$t_{LQ}$ is the time recorded by thermal analysis at the liquidus temperature.  
$t_{SOL}$ is the time recorded by thermal analysis at the solidus temperature.

Table 5.5. List of reactions identified on the heating (UMSA) and cooling curves (UMSA and AlTAP) present in the test samples for the 390.1 and 393.2 alloys that were solidified under natural heat exchange conditions.

<table>
<thead>
<tr>
<th>Heating Cycle (Only UMSA)</th>
<th>Cooling Cycle (UMSA and AlTAP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ID. #</td>
<td>Description</td>
</tr>
<tr>
<td>1</td>
<td>Artificial aging temperature of (release of residual stresses [208]) and possible start of dissolution of soluble phase(s)</td>
</tr>
<tr>
<td>2</td>
<td>Beginning of the alloy melting process</td>
</tr>
<tr>
<td>3</td>
<td>Finish of the alloy melting process</td>
</tr>
<tr>
<td>7*</td>
<td>Nucleation of the Mg2Si eutectic phase</td>
</tr>
<tr>
<td>9*</td>
<td>Nucleation of the Al-Cu-Ni phase</td>
</tr>
<tr>
<td>11</td>
<td>End of the solidification (Solidus)</td>
</tr>
</tbody>
</table>

The effect of external factors (such as melt treatment) on Mg, Ni, Cu, Fe, Mn and Cr, enriched phases can be evaluated by determining the amount of these phases during the precipitation process. M. Djurdjevic et. al. [191] and R. I. MacKay [169] reported that by using thermal analysis techniques the apparent fraction solid could be used to conduct in depth analysis of the Mg, Ni, Cu enriched phases. For this reason, the thermal analysis algorithms (Appendix I) were applied to determine the corresponding baseline to the Al-Si eutectic reaction (Figure 5.26). This baseline represents a curve where no reactions take place during this solidification path except at the Al-Si eutectic, which presumably ends at the solidus temperature. Any change in the
amount of heat released due to reactions 7 to 10 is expressed in the first derivative as a peak(s).
The determination of the integral between the previously mentioned baseline and the first
derivative can be used to calculate the amount of precipitating phases. The corresponding
fraction between the integral of the entire solidification process and the respective fraction of the
Mg, Ni, Cu enriched phases is called the apparent fraction solid.

The value of the apparent fraction solid in a melt with a constant composition and similar
solidification conditions must be the same. It is for this reason, that any change in the apparent
fraction solid in a test sample of the same melt, but, distinct solidification conditions; can be
related to the amount of dissolved Mg, Ni, Cu, Fe, Mn and Cr enriched phases within the matrix
(Figure 5.26).

Figure 5.24. Temperature vs. time curves obtained using the UMSA platform during the heating
and cooling cycles of a remelted reference test sample for the 390.1(I) Alloy. Note: heat was released during the cooling cycle in the radial (r) direction (1D). Each number represents a reaction explained in Table 5.5.
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Figure 5.24b. Thermal signature obtained using the UMSA platform during the heating and cooling cycles of a remelted reference test sample for the 390.1(I) alloy. Note: the heat was released during the cooling cycle in the radial (r) direction (1D).

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Figure 5.25. Cooling, first derivative and baseline vs. time curves of a reference test sample of the 390.1(I) alloy recorded using AITAP. Note: heat was released radially (1D) at a cooling rate ~ 0.08 °C/s. Each number represents a reaction identified in Table 5.5.

Figure 5.26. Section of the cooling and first derivative curves with the respective Al-Si eutectic baseline of a reference test sample for the 390.1(I) alloy (cooling rate ~ 0.08 °C/s) used to determine the apparent fraction solid of the Mg, Ni, Cu enriched phases.

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The cooling curves and therefore the first derivatives of the 393.2(I) and 393.2(II) alloys were found to be similar to each other and different that that of the 390.1(I) alloy. Both 393.2 alloys have a considerably higher liquidus temperature (point #4), a larger time between liquidus and Al-Si eutectic reactions (point #5) due to the higher Si of the 393.2 alloys when compared to the 390.1(I) alloy (compare Figures 5.24b and 5.28). The results of thermal analysis using AITAP are presented in Figure 5.27 and show all the identified reactions in the 390.1(I) alloy. The identification numbers in Figures 5.27 and 5.28 are the same as for the 390.1(I) alloy and are presented in Table 5.5.

![Figure 5.27. Cooling, first derivative and baseline vs. time curves of a reference test sample for the 393.2(I) alloy recorded with AITAP. Note: the heat was released in the radial (r) direction (ID) at a cooling rate ~ 0.16 °C/s. Each number represents a reaction identified in Table 5.5.](image)

A summary of the temperatures and the fraction solids of all the solidification reactions identified using UMSA and AITAP for the 390.1(I), 393.2(I) and 393.2(II) alloys is presented in Table 5.6 - Table 5.9. Test samples of the 390.1(I) alloy in the above-mentioned tables were solidified using UMSA and AITAP under different conditions. The higher cooling rate using AITAP (cooling rate ~ 0.09 °C/s) corresponds to the test samples solidified in the Cu plate (air chilling), while the lower cooling rate is for the test samples solidified inside the electromagnetic coils (cooling rate ~ 0.08 °C/s).
Figure 5.28. Calculated first derivative vs. temperature of the 393.2(I) test sample. Note: the heat was released in the radial (r) direction (1D) at cooling rate ~ 0.08 °C/s.
Table 5.6. Summary of the temperatures, (°C) of the nucleation reactions for different alloys obtained using the UMSA platform.

<table>
<thead>
<tr>
<th>Solidification Reactions</th>
<th>390.1(I) CR=0.22°C/s</th>
<th>390.1(I) CR=0.67°C/s</th>
<th>393.2(I) CR=0.31°C/s</th>
<th>393.2(II) CR=0.37°C/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Si</td>
<td>600.5</td>
<td>600.8</td>
<td>737.0</td>
<td>796.1</td>
</tr>
<tr>
<td>Al-Si Eutectic Nucleation</td>
<td>562.0</td>
<td>563.5</td>
<td>573.6</td>
<td>564.2</td>
</tr>
<tr>
<td>Al-Si Eutectic Growth</td>
<td>558.3</td>
<td>560.9</td>
<td>573.1</td>
<td>574.3</td>
</tr>
<tr>
<td>DCP</td>
<td>557.7</td>
<td>562.6</td>
<td>570.9</td>
<td>573.7</td>
</tr>
<tr>
<td>Mg$_2$Si Eutectic</td>
<td>550.2</td>
<td>550.7</td>
<td>N/A</td>
<td>571.8</td>
</tr>
<tr>
<td>Al-Ni Eutectic</td>
<td>524.2</td>
<td>521.5</td>
<td>506.3</td>
<td>501.3</td>
</tr>
<tr>
<td>Al$_3$Cu Eutectic</td>
<td>502.8</td>
<td>N/A</td>
<td>499.9</td>
<td>488.5</td>
</tr>
<tr>
<td>Al-Ni-Cu + Al-Cu$_{f}$ + Pb</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Solidus</td>
<td>478.6</td>
<td>477.0</td>
<td>478.4</td>
<td>474.5</td>
</tr>
<tr>
<td>Solidification Range</td>
<td>122.0</td>
<td>123.7</td>
<td>259.0</td>
<td>321.6</td>
</tr>
</tbody>
</table>

Table 5.7. Fraction solids, (%) of the corresponding nucleation reactions for different alloys obtained using the UMSA platform.

<table>
<thead>
<tr>
<th>Solidification Reactions</th>
<th>390.1(I) CR=0.22°C/s</th>
<th>390.1(I) CR=0.67°C/s</th>
<th>393.2(I) CR=0.31°C/s</th>
<th>393.2(II) CR=0.37°C/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Si</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Al-Si Eutectic Nucleation</td>
<td>7.6</td>
<td>6.1</td>
<td>36.2</td>
<td>39.3</td>
</tr>
<tr>
<td>Al-Si Eutectic Growth</td>
<td>10.3</td>
<td>9.2</td>
<td>40.2</td>
<td>45.3</td>
</tr>
<tr>
<td>DCP</td>
<td>21.9</td>
<td>21.9</td>
<td>46.0</td>
<td>48.7</td>
</tr>
<tr>
<td>Mg$_2$Si Eutectic</td>
<td>65.0</td>
<td>66.5</td>
<td>N/A</td>
<td>56.5</td>
</tr>
<tr>
<td>Al-Ni Eutectic</td>
<td>89.8</td>
<td>89.7</td>
<td>98.5</td>
<td>98.9</td>
</tr>
<tr>
<td>Al$_3$Cu Eutectic</td>
<td>97.3</td>
<td>97.9</td>
<td>98.8</td>
<td>99.6</td>
</tr>
<tr>
<td>Solidus</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
</tr>
</tbody>
</table>

Table 5.8. Summary of temperatures, (°C) of the solidification reaction reactions for different alloys obtained using the AITAP platform.

<table>
<thead>
<tr>
<th>Solidification Reactions</th>
<th>390.1(I) CR=0.09°C/s</th>
<th>390.1(I) CR=0.08°C/s</th>
<th>393.2(I) CR=0.16°C/s</th>
<th>393.2(II) CR=0.17°C/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Si</td>
<td>600</td>
<td>599</td>
<td>740.7</td>
<td>793.9</td>
</tr>
<tr>
<td>Al-Si Eutectic Nucleation</td>
<td>566.0</td>
<td>564.0</td>
<td>577.5</td>
<td>580.6</td>
</tr>
<tr>
<td>Al-Si Eutectic Growth</td>
<td>563.0</td>
<td>561.5</td>
<td>569.6</td>
<td>571.2</td>
</tr>
<tr>
<td>DCP</td>
<td>560.0</td>
<td>557.0</td>
<td>568.4</td>
<td>568.6</td>
</tr>
<tr>
<td>Mg$_2$Si Eutectic</td>
<td>554.0</td>
<td>553.0</td>
<td>549.5</td>
<td>543.6</td>
</tr>
<tr>
<td>Al-Ni Eutectic</td>
<td>540.0</td>
<td>537.0</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Al$_3$Cu Eutectic</td>
<td>520.0</td>
<td>520.0</td>
<td>517.4</td>
<td>523.4</td>
</tr>
<tr>
<td>Al-Ni-Cu + Al-Cu$_{f}$ + Pb</td>
<td>505.0</td>
<td>504.0</td>
<td>498.5</td>
<td>500.5</td>
</tr>
<tr>
<td>Solidus</td>
<td>488.0</td>
<td>487.0</td>
<td>477.4</td>
<td>479.3</td>
</tr>
<tr>
<td>Solidification Range</td>
<td>112</td>
<td>112</td>
<td>263.3</td>
<td>314.6</td>
</tr>
</tbody>
</table>

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Table 5.9. Summary of fraction solids, (%) of the solidification reactions for different alloys obtained using the AITAP platform.

<table>
<thead>
<tr>
<th>Solidification Reactions</th>
<th>390.1(I) CR=0.09°C/s</th>
<th>390.1(I) CR=0.08°C/s</th>
<th>393.2(I) CR=0.31°C/s</th>
<th>393.2(II) CR=0.37°C/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Si</td>
<td>0</td>
<td>0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Al-Si Eutectic Nucleation</td>
<td>12.8</td>
<td>12.5</td>
<td>35.2</td>
<td>40.1</td>
</tr>
<tr>
<td>Al-Si Eutectic Growth</td>
<td>14.4</td>
<td>14.4</td>
<td>41.6</td>
<td>43.1</td>
</tr>
<tr>
<td>DCP</td>
<td>42.1</td>
<td>38.2</td>
<td>46.3</td>
<td>49.1</td>
</tr>
<tr>
<td>Mg$_2$Si Eutectic</td>
<td>55.7</td>
<td>54.4</td>
<td>N/A</td>
<td>55.8</td>
</tr>
<tr>
<td>Al-Ni Eutectic</td>
<td>76.6</td>
<td>76.8</td>
<td>97.5</td>
<td>98.1</td>
</tr>
<tr>
<td>Al$_2$Cu Eutectic</td>
<td>88.4</td>
<td>87.6</td>
<td>99.1</td>
<td>99.6</td>
</tr>
<tr>
<td>Al-Ni-Cu + Al-CuFb + Pb</td>
<td>94.8</td>
<td>94.7</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Solidus</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
</tr>
</tbody>
</table>

5.6. Identification of Phases in Al-Si Hypereutectic Alloys in the Liquid State and the Mushy Zone Using Rapid Solidification.

The solidification process for the 3XX.X Al-Si hypereutectic alloys can be observed in the cooling curves presented in Section 5.5. In this research a series of quenching experiments at different temperatures from the liquid state and at different fraction solids were conducted. The identification of the phases was carried out using LOM, SEM/BSE and SEM/EDX (Section 5.4).

During solidification, each phase grows at the expense of liquid instability at specific temperatures. Several attempts were made in order to reveal the nature of the mushy zone for Al-Si hypoeutectic alloys; but due to the existing limitations of the experimental equipment the determination of phases is still unclear for certain phases and alloys [76, 169, 170]. Nonetheless, applications of advanced thermal analysis using the UMSA platform are probably the most useful existing technology to clarify this solidification sequence. To date, Al-Si hypereutectic alloys have not been studied in such detail.

Al-Si hypereutectic alloys (such as 390.1 and 393.2) have primary Si in their solidification pathways a first precipitation phase; instead of the typical $\alpha$-Al dendrites characteristic of Al-Si hypoeutectic alloys. Primary Si solidifies and grows at low fraction solids and until the remaining pool of liquid metal reaches the Al-Si eutectic composition for near
equilibrium cooling rates. From this point forward, the solidification sequence is comparable to the Al-Si hypoeutectic alloys.

It has been reported that, prior to the beginning of solidification process (liquidus) of hypereutectic alloys there is an agglomeration of Si atoms in the “liquid” state [124, 125, 127, 128]. This agglomeration of atoms has an ordered arrangement but with a different crystalline structure than the solid primary and eutectic Si. The rapid solidification of these agglomerates becomes one of the main focuses of the present research because they are the main key to demonstrate experimentally that ESV melt treatments above liquidus can result in a considerable level of microstructure refinement. Currently, the only known technology that permits accurate and controlled rapid solidification of test samples from the specific temperatures is UMSA. Therefore, by using UMSA an interpretative methodology of solidification for the Al-Si hypereutectic alloys will be established (Section 5.6.1 and Figure 5.29 through Figure 5.39).

In Section 5.4 it was mentioned that the Al-Si hypereutectic alloys subjected to solidification under heat exchange conditions close to equilibrium, that the so called DCP is not present. However, by solidifying test samples at conditions far from the equilibrium they solidify following the skewed couple zone path [38, 126]. It is for this reason, that these test samples develop dendritic structures and haloes surrounding the primary Si particles. This phenomenon permits the determination of the **Secondary Dendrite Arm Spacing** (SDAS) in quenched samples.

5.6.1. Phases Identification Using Thermal Analysis in Quenched Test Samples of Al-Si Hypereutectic Alloy test Samples

Alloys 390.1(I) and 393.2(I) were quenched from temperatures as high as ~100°C above liquidus. During solidification in the mushy zone evolution the test samples were quenched at the temperatures of nucleation of every reaction. The 393.2(I) alloy was quenched at the following temperatures: 790°C, 770°C, 730°C and 600°C (Figure 5.29a). The 390.1(I) alloy was quenched at temperatures of 730°C, 690°C, 660°C, 630°C, 560°C, 554°C, 525°C, 500°C and at full solidification (<480°C) (Figure 5.29b). The above-mentioned temperatures correspond to ~10°C above the ESV melt treatment or nucleation temperatures.
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The target temperatures at which the test samples were quenched above and below liquidus for the 390.1(I) and 393.2(I) alloys are shown in Figure 5.29. This figure shows sections of the cooling curve (recorded by UMSA) before quenching and the rapid response of the thermocouple inside the test sample during the released of the heat. The fact that the test samples were forced to solidify in few seconds is used as an argument to say that the characteristic microstructure that exist at the specific quenching temperature was frozen. The target temperatures during the mushy zone evolution were carried out for all the solidification reaction (point #5 through #9) shown in Figure 5.25.

Table 5.10 summarizes the temperatures of the artificial aging [169], the beginning of melting and the liquidus temperature of the respective alloy. Both histograms show that the temperatures recorded in the cooling curves of all the exercised UMSA test samples were similar among them. This indicates that the chemical composition of all quenched test samples was similar, because differences in composition (i.e. Si content) have an effect in their respective solidification temperature.

Table 5.10. Summary of the recognized temperatures of the heating portion of the UMSA Thermal signature. The heating rate of both alloys was 0.53°C/s.

<table>
<thead>
<tr>
<th>Thermal Characteristics</th>
<th>390.1(I)</th>
<th>393.2(I)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent start of dissolution of soluble phase(s)</td>
<td>312.71 ± 0.5</td>
<td>342.74 ± 0.7</td>
</tr>
<tr>
<td>Minimum Solution Treatment Temperature (Point #1 Figure 5.24b)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Start (beginning) of the alloy melting process (Point #2 Figures 5.24b and 5.28)</td>
<td>494.16 ± 0.7</td>
<td>497.98 ± 0.6</td>
</tr>
<tr>
<td>Finish (end) of the alloy melting process (Point #3 Figures 5.24b and 5.28)</td>
<td>652.59 ± 0.8</td>
<td>762.32 ± 0.5</td>
</tr>
</tbody>
</table>
Figure 5.29. The cooling curves of the reference test samples recorded using UMSA a) 390.1(I) and b) 393.2(I) alloys with overlapping cooling curves for the quenching experiments.
5.6.2. Characterization of Rapidly Solidified Al-Si Hypereutectic Alloys Using LOM and SEM

The LOM micrographs shown in Figures 5.31 and 5.32 present the microstructure of the high temperature quenched test samples for the 393.2(I) and 390.1(I) alloys respectively. The
above-mentioned figures are the proof of the Si agglomerates present in molten Al-Si hypereutectic alloys (Section 5.6). The growth of these particles was measured using image analysis for the two different alloys. The image analysis results shown in Figure 5.34 and confirm that the growth of the Si particles of both alloys is linear as temperature decreases. At lower temperatures than the Al-Si eutectic the primary Si particles did not show further growth. Therefore it can be concluded that the primary Si particles stop their growth at the beginning of the Al-Si eutectic nucleation.

The fact that the quenching of the test samples at temperatures above liquidus demonstrated the presence of the Si agglomerates at temperatures above the liquidus in Al-Si hypereutectic alloys. Therefore, it can be said that the ESV melt treatments can affect the melt structure above the liquidus temperature, which consequently changes the solidifying behaviour of the Si phases. However, at elevated temperatures (>100°C above liquidus) the ESV melt treatment effect is significantly reduced; due to the fact that the Si agglomerates are small and once the ESV melt treatment is stopped they can continue their free growth. It is for this reason that the most effective ESV melt treatment takes place at temperatures as close as possible to the liquidus. This brings two key factors that affect the effectiveness of the ESV melt treatments to our attention.

The growth of the primary Si particles stops at the Al-Si eutectic temperature and the difference between liquidus and the Al-Si eutectic temperature is a function of the amount of Si are key factors in the effectiveness of the ESV treatments above liquidus. For instance, for the 390.1(I) alloy after the ESV melt treatment conducted at 610°C the linear growth of the primary Si particles (Figure 5.34) takes ends at the Al-Si eutectic (only ~50°C). In contrast for an ESV melt treated test sample in liquid state (750°C) of the 393.2(I) alloy; after the treatment the primary Si particles can growth for ~200°C. This results in coarser primary Si particles as the Si content increases. Therefore, the opportunity to grow primary Si particles in the 390.1(I) alloy is four times less than in the case of the 393.2(I) alloy. Nonetheless, based on this theory and the supporting evidence there is a good chance to improve the Si modification of the 393.2(I) alloy in semi-solid state, but this practice could possibly compromise fluidity.
The quenched test samples at the respective target temperatures below the liquidus temperature presents a characteristic feature (Figure 5.32) identified as a “crown”. This crown surrounds the solidified Si agglomerates and is orientated perpendicularly to the edges of the Si agglomerates. Furthermore, the frozen secondary phases form oriented quasi-dendritic “channels” that are extended beyond the crown. It is possible that the channels are directly related with the natural convection of the alloy and act as the transport of imbalances in composition with the supersaturated melt and the low concentration regions. The quasi-dendritic regions resulted from the effect of quenching of the test samples from the liquid stage.

Figure 5.31. Sequence of LOM micrographs of the 393.2(I) reference test samples quenched using the UMSA platform from different target temperatures above liquidus a) 800°C, b) 770°C, c) 730°C and d) below liquidus at 650°C. Micrographs taken at 500 X magnification.

The sections where the crowns appeared were identified as the sections where the α-Al halo took place, therefore these regions were transporting Si into the primary Si particles. At the
same time Mg, Fe, Mn, Cr, Ni and Cu enriched phases started to be segregated in the areas of the
\(\alpha\)-Al haloes together with the Al-Si eutectic. Therefore, the crown area becomes the \(\alpha\)-Al haloes
due to impoverished and segregated effects taking place in the neighbouring area to the Si particles (Figures 5.32 to 5.35).

The same growth direction as for Si is followed by the \(\alpha\)-Al dendrites or haloes, but there
are significant differences in the crystalline structure between these two phases. This does not
allow a coherent solidification between both elements. The FCC crystalline structure of Al is an
“\(\alpha\)l” copper type with a lattice parameter \(a\) of 4.0490 Å, while \(a\) for Si is 5.4282 Å with an
“A4” (diamond like) ordered crystalline structure [200]. Additionally, nucleation of Al and Si is
normal and layered (2D) [170]. In other words during nucleation of Al, the Al atoms have no
preferred locations to be attached, conversely the nucleation of Si atoms is carried out in an
ordered way. This results in a different rate of growth between the two types of nucleation; for
instance, the nucleation and growth of Al is considerably higher than the one for Si [170]. This is
the reason for the rapid increase in fraction solid from the Al-Si eutectic to the dendrite coherency
point (~10% to > 25%) in only 4°C (Table 5.8, Table 5.9).

The size of the primary Si particles for the 390.1(I) alloy reference test samples quenched
at a temperature of 560°C (Figure 5.33) were measured using image analysis. The size of these
particles shows no further growth from this point forward. These particles have the typical
polyhedral shape; also the above-mentioned crown becomes unclear, showing (Figure 5.33b) the
beginning of the formation of the \(\alpha\)-Al halo at the edge. Also the orientation of quasi-dendritic
regions starts to be unclear. This quenching temperature is very close to the Al-Si eutectic
reaction and because the quenching of the test samples does not take place instantly, some of the
remaining molten alloy has a similar composition to the eutectic solidified in the form of fine Al-
Si eutectic. However, the Al-Si eutectic had not yet been fully developed yet.

The test sample for the 390.1(I) alloy quenched at a temperature of 550°C that
corresponds to ~58% of fraction solid shows a clear definition of primary Si particles with their
\(\alpha\)-Al respective halo(s), Al-Si eutectic and coarse \(\alpha\)-Al dendrites. These test samples were
quenched at a temperature close to the DCP (see Figures 5.24 and 5.30), therefore, after this point
no apparent enlargement of the dendrites was supposed to occur; nonetheless, they continue
coarsening due to the interdendritic feeding. The DCP can be identified as the point where the semi-solid slurry becomes rigid, and there is no longer free flow of the remaining liquid; except for the interdendritic feeding. Additionally, the coarse Al-Si eutectic particles will become coarser during the remaining solidification process (up to the solidus point).

The quenching of the presently described test samples was at a temperature of 560°C, which is ~5°C above the Mg2Si eutectic phase (545 ± 0.7 °C). Using SEM/BSE the Mg2Si was successfully identified and is presented in Figure 5.36. In the vicinity to the Mg2Si eutectic particles, quenched Cu and Ni enriched regions were observed.

The 390.1(1) alloy reference test sample quenched from a temperature of 525°C was quenched close to the temperature of nucleation of the Ni and Cu enriched eutectics (points #7 - #10 in Figure 5.25). At 525°C the equivalent fraction solid was ~80%. The Al-Ni eutectic was identified together with the Al13(Fe, Cr, Mn)3Si2 phases (Figure 5.37). The exact nucleation temperature of Al13(Fe, Cr, Mn)3Si2 is unknown, but is somewhere between 550°C and 525°C.

The 390.1(1) alloy test sample of reference quenched at a temperature of 500°C contained a fraction solid of ~ 96%, with a SDAS of 28.2 ± 1.8 μm. At this point, the coarsening of the dendrites was almost negligible (Figure 5.38). LOM micrographs of this test sample reveal the Al-Cu-Ni, the Al12Cu and the Al-Cu fine eutectic phases. Furthermore, in order to differentiate the sequence of solidification SEM/BSE micrographs of the test samples in different areas were taken. Figure 5.39a shows SEM/BSE micrographs of the edge of the test sample, which is the area that solidified, first once in contact with the quenching media (H2O + 15 wt% NaCl). These micrographs show the Al-Ni-Cu eutectic phase and the Al12Cu and Al-Cu fine eutectic (Al-CuFE) and were frozen from their liquid state. In Figure 5.39b the Al12Cu was identified in the centre of the test sample, and this phase solidified following the Al-Cu-Ni eutectic. Therefore, the last phases to solidify were the Al-CuFE (FE indicates Fine Eutectic see Table 5.4) and the Pb particles (Figure 5.39b).
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Figure 5.32. LOM micrographs of the 390.1(1) UMSA test samples quenched from different target temperatures above liquidus a) 730°C, b) 660°C, and c) 620°C. micrographs at 500X magnification.

An analysis of the Mg, Ni and Cu enriched phases was performed by using the apparent fraction solid ($a_f^{Mg_2Si}$, $a_f^{AlNi}$, $a_f^{AlNiCu}$, and $a_f^{AlCu}$) that were determined using the methodology explained in Section 5.5 and Figure 5.2. It is important to understand the behaviour of these phases because they are rich in Cu, Ni and Mg and are the precipitation hardening elements. A reduction of the $a_f$ due to ESV melt treatment(s) is a possible indication that the ESV melt treatment allows for a more efficient dissolution of the Mg, Ni and Cu within the α-Al matrix. By reducing the $a_f$ of the above-mentioned phases a direct link between the thermal analysis, image analysis and mechanical properties can be developed. The results of this study are presented in the automotive components section of this dissertation (Pistons and Cylinder Liners). A summary of the phases observed as well as the identified phases during the solidification sequence of the 390.1(1) alloy is presented in Table 5.5. Table 5.6 to Table 5.9 contain the temperatures and
fraction solids of the different precipitation reactions recorded using the UMSA and AITAP platforms. It is important to note that the solidification sequence of the 390.1 and 393.2 alloys are very similar, except for the primary Si nucleation temperatures.

Figure 5.33. LOM micrographs of the 390.1(I) UMSA test samples quenched at 560°C (~40°C below liquidus) at a) 200 X and b) 500 X magnification.

Figure 5.34. Coarsening analysis of the Si agglomerates and primary Si particles as a function of the quenching temperature for the reference test samples for the 390.1(I) and 393.2(I) alloys. Note: the results presented in this figure correspond to the measurements of ten analytical fields using the image analysis system at 500 X magnification.
Figure 5.35. LOM micrographs of the 390.1(I) reference UMSA test sample quenched at 550°C (~50°C below liquidus) at a) 200 X and b) 500 X magnification.

Figure 5.36. SEM/BSE micrographs showing the Mg2Si surrounded by quenched Cu and Ni (390.1(I) alloy) quenched at 550°C at 1000 X magnification.

Figure 5.37. LOM micrographs of the 390.1(I) UMSA test samples quenched at 525°C a) 200 X and b) 500 X magnification.
Figure 5.38. LOM micrographs of the 390.1(I) UMSA test samples quenched at 500°C at a) 200 X and b) 500 X magnification.

Figure 5.39. SEM/BSE micrographs of the 390.1(I) UMSA reference test samples quenched at 500°C (1000 X magnification).
Table 5.11. Solidification sequence of the 390.1(1) alloy determined with advanced thermal analysis using UMSA in a series of quenching experiments and characterized with LOM and SEM/BSE techniques. This solidification sequence also applies to the 390.1(II) and 393.2 alloys except for the difference in liquidus temperatures (634 and 600°C respectively). S\textsubscript{SRAN} stands for the solidification Range.

<table>
<thead>
<tr>
<th>Summary of Reactions and Corresponding Characteristic Points</th>
<th>Temperature Range (°C)</th>
<th>Fraction Solid (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Reaction #1) ( L \rightarrow \text{Primary Si} ) ( T_{\text{PRI-SI NUC}} = 600.5°C ) ( f_s \text{PRI-SI NUC} = 0% )</td>
<td>( (T_{\text{PRI-SI NUC}} - T_{\text{AL-SIF NUC}} = \Delta T_{\text{PRI-SI SRAN}}) = 37.0 )</td>
<td>( f_s \text{PRI-SI NUC} - 0% = a_{\text{Al}} ) ( f_s \text{PRI-SI} = 6.9 )</td>
</tr>
<tr>
<td>(Reaction #2) ( L \rightarrow \text{Al-Si (Eutectic)} ) ( T_{\text{AI-SI ENUC}} = 563.5°C ) ( f_s \text{AI-SI ENUC} = 18.1% )</td>
<td>( (T_{\text{Mg2Si ENUC}} - T_{\text{AI-SI ENUC}} = \Delta T_{\text{AI-SI SRAN}}) = 11.8 )</td>
<td>( f_s \text{AI-SI ENUC} - f_s \text{Mg2Si ENUC} = 21.9 )</td>
</tr>
<tr>
<td>(Reaction #3) ( L \rightarrow \text{Al + Si} + \text{Al}\text{15(Fe,Cr,Mn)}\text{3Si2} )</td>
<td>Temperature for the ( \text{Al}\text{15(Fe,Cr,Mn)}\text{3Si2} ) somewhere between 563 and 525. Solidification ends before or at Solidus (( T_{\text{SOL}} ))</td>
<td>Between 18.1 ( f_s ) and 89.4 ( f_s )</td>
</tr>
<tr>
<td>(Reaction #4) ( L \rightarrow \text{Al + Si + Mg2Si} ) ( f_s \text{Mg2Si ENUC} = 97.4% )</td>
<td>( T_{\text{AI-NI ENUC}} - T_{\text{Mg2Si ENUC}} = \Delta T_{\text{AI-NI SRAN}} = 11.9 )</td>
<td>( f_s \text{AI-NI ENUC} - f_s \text{Mg2Si ENUC} = 21.9 )</td>
</tr>
<tr>
<td>(Reaction #5) ( L \rightarrow \text{Al + Si + Al-Ni} ) ( f_s \text{Al-NI ENUC} = 89.8% )</td>
<td>( T_{\text{AI-Cu FE,NUC}} - T_{\text{AI-NI ENUC}} = \Delta T_{\text{Al-Cu SRAN}} = 29.2 )</td>
<td>( f_s \text{AI-Cu FE,NUC} - f_s \text{Al-NI ENUC} = 8.0 )</td>
</tr>
<tr>
<td>(Reaction #6) ( L \rightarrow \text{Al + Si + Al-Cu} ) ( \text{(Fine Eutectic (FE))} ) ( f_s \text{Al-NI ENUC} = 97.5% )</td>
<td>( T_{\text{Al-Cu FE,NUC}} - T_{\text{Al-Cu FE,NUC}} = \Delta T_{\text{Al-Cu SRAN}} = 14.2 )</td>
<td>( f_s \text{Al-Cu FE,NUC} - f_s \text{Al-NI ENUC} = 7.8 )</td>
</tr>
<tr>
<td>(Reaction #6) ( L \rightarrow \text{Al + Si + Al-CuFE} + \text{PbParticles} ) ( f_s \text{Al-CuFE + Pb ENUC} = 100% ) Detected by AITAP</td>
<td>( T_{\text{SOL}} - T_{\text{Al-Cu FE,NUC}} = \Delta T_{\text{Al-Cu SRAN}} = 22.8 )</td>
<td>( f_s \text{SOL} - f_s \text{Al-Cu FE,NUC} = 2.6 )</td>
</tr>
</tbody>
</table>

As can be observed from Table 5.6 to Table 5.9 the temperatures as well as the fraction solids measured by either platform, UMSA or AITAP, are in good agreement. Additionally the number of phases detected using the thermal analysis correspond to the phases identified with metallography, except for the Pb particles. The reason for this is that Pb is presented in the 390.1(1) and 393.2(1) alloys in traces to the order of ppm, therefore, they are difficult to be detected using thermal analysis. The Al-Ni-Cu + Al-CuFE +Pb reaction was not visible in the
thermal signature obtained by UMSA. This is probably due to the fact that the cooling rate that was from \( \sim 2 \) up to \( \sim 17 \) times higher when using UMSA instead of AITAP; additionally these phases correspond to less than 3\% of the fraction solid, which makes their determination more difficult. It is important to mention that AITAP was used during the ESV melt treatment and UMSA during the remelting of the test samples.

5.6.3. Thermal Analysis of ESV Melt Treated 390.1(I) and 393.2(I) Al-Si Hypereutectic Alloys

The thermal analysis for the ESV melt treated test samples was conducted in a series of two experiments. In the first test samples were forced to solidify on an air quenched Cu plate, in the other, the test samples were solidified under natural heat exchange conditions. The heat was released radially \((r)\), this means through one dimension (1D). In the air quenched test samples the heat was released radially \((r)\) and vertically \((z)\), therefore in two dimensions (2D). Appendix I shows a model of the two kinds of solidification conditions.

The air quenched (heat exchange in 2D) test samples were used for the statistically designed experiments. The thermal analysis conducted on these test samples was limited to the determination of the liquidus and solidus temperatures. The identification of the rest of the nucleation reactions was unclear (Figure 5.40). The above-mentioned limitation is due to the gradient of temperatures formed within the test sample that promotes convection. Additionally, the microstructure of test samples solidified under 2D heat exchange conditions was influenced by the air quenched (directional) solidification.
Figure 5.40. Cooling, first derivative and baseline curves vs. time of a 390.1(I) Al-Si alloy test sample recorded with AITAP in an ESV melt treated test sample solidified under radial and vertical (2D) heat exchange conditions (cooling rate = 0.09 °C/s). a, b and c show the beginning and end of the ESV melt treatment and air quenching respectively.

In order to clearly identify the specific nucleation reactions, test samples under selected ESV melt treatment conditions were solidified under natural heat exchange (1D) conditions (Figure 5.41). The cooling curves and first derivatives of both, 1D and 2D, solidification conditions presented extra features such as the beginning and the end of the ESV melt treatment (identified as a and b in Figures 5.40 and 5.41) and air quenching. Appendix II presents the summary of all temperatures of the nucleation reactions of the ESV melt treated test samples solidified under 1D and 2D heat exchange conditions.

The effect of the ESV melt treatment in the liquidus temperature of the test samples solidified under radial and vertical heat exchange conditions (2D) is presented in Figure 5.42. The previously mentioned figure clearly shows the effect of frequency and current on the liquidus temperature. Additionally, the liquidus temperature was also influenced by the hydrogen level; undegassed melt test samples exhibited higher liquidus. The details about degassing conditions are provided in Section 4.3. The highest liquidus was recorded in the ESV melt treated test samples and a $I_{AC/DC} = 100$ A, $f = 100$ Hz, and $T_{ESV} = 610^\circ$C (390.1(I) alloy).
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Figure 5.41. Cooling, first derivative and baseline curves vs. time for a 390.1(1) Al-Si alloy test sample recorded with AITAP. The test sample was ESV melt treated and solidified under natural heat exchange conditions (heat released in 1D and a cooling rate = 0.09 °C/s). Note: a and b show the beginning and end of the ESV melt treatment.

The results of the temperature measurements for the test samples that were solidified under 1D or 2D heat exchange conditions are presented in Appendix II. The data in the previously mentioned tables was used to determine the effect of the independent ESV melt treatment variable, AC current frequency, temperature, time, AC and DC Currents, Melt Si level (chemical composition), \([H_2]\) level and isothermal holding, for the statistically designed experiments. Figure 5.42 and 5.43 show the effect of temperature, frequency, time and degassing conditions on liquidus temperatures; the results presented in these two figures correspond to the ESV melt treated test samples that were solidified on the Cu plate (air quenching). It is important to mention that the heat was released in a 2D direction (radially and vertically).
Figure 5.42. Effect of the ESV melt treatment on the liquidus temperature for the 390.1(l) alloy ESV melt treated test samples using the chilled Cu plate.

Figure 5.43. Effect of ESV melt treatment time and alloy composition on liquidus temperature. Note: experiments carried out using \( I_{ACDC} = 100 \, \text{A}, f = 100 \, \text{Hz}, T_{ESV} = 610 \) and 750°C ± 2 °C for the 390.1(l) and 393.2(l) alloys respectively. Test samples solidified on the chilling Cu plate (radial and vertical heat release).
The results of thermal analysis conducted on test samples that were solidified under natural heat exchange conditions (heat exchange in 1D) are presented in Figures 5.44 to 5.46. The above-mentioned figures confirm that the liquidus temperature is influenced by the ESV melt treatment (+7°C). The liquidus and the Al-Si eutectic nucleation temperatures as a function of the frequency (-5°C) of the ESV melt treatment are presented in Figure 5.44. This figure shows an increase in liquidus temperature as a function of the frequency of the ESV melt treatment; but the nucleation temperature of the Al-Si eutectic reaction decreases. As a result the time elapsed between the liquidus and the nucleation of the Al-Si eutectic increases with the frequency of the ESV melt treatments (Figure 5.41).

It is also important to mention that by allowing the heat to be released from the test sample only in the radial direction (1D), the nucleation temperatures of the Mg, Ni, Cu enriched phases can be detected (Figure 5.46). However, except for the Al-Ni eutectic reaction (~20°C) the difference in the nucleation temperature for the rest of the reactions is negligible. The nucleation temperature of the Al₂Cu phase was unclear in the cooling curves of the ESV melt treated test samples.

![Figure 5.44.](image)

Figure 5.44. Liquidus and Al-Si eutectic temperatures for the 390.1(I) alloy ESV melt treated test samples using different frequencies (f_{AC}) and solidified under natural heat exchange conditions (radial heat exchange (1D) cooling rate = 0.08 °C/s).
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Figure 5.45. Temperatures of identified reactions during the solidification process for the ESV melt treated test samples at 610°C using different frequencies and solidified under natural heat exchange conditions (cooling rate = 0.08°C/s).

Figure 5.46. Effect of the ESV treatment on the nucleation temperature of Mg, Ni, Cu, and Pb enriched phases of melt treated test samples solidified using radial (1D) heat exchange conditions.
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The partial integral to calculate the apparent fraction solid of the Mg, Ni and Cu enriched phases of the ESV melt treated test samples solidified under natural heat exchange conditions is presented in Appendix II (Table A2.11). It is important to note that the apparent fraction solid of all the previously mentioned test samples was similar; indicating that the amount of Mg, Ni and Cu enriched phases that precipitate during solidification were similar among the different ESV melt treatments and the reference test sample. Therefore, most likely the ESV melt treatment at temperatures above liquidus had no effect on the dissolution of the Mg, Ni and Cu enriched phases. Nonetheless, in Section 5.7, a homogeneity analysis is performed and a further analysis of these phases is conducted.

The possibility of developing precise mathematical models for on-line prediction of the level of solidifying microstructure modification and refinement depends on the accuracy of the thermal analysis platform. Also the algorithms for the analysis of the cooling curves play an important role. The calibration of the thermocouples presented in Chapter 4, shows that the margin of error in UMSA is < 0.2°C and ~ 0.6°C using Acrolab thermocouples. Therefore, their industrial application could be a key factor to on line determination of the level of microstructure modification on ESV melt treated castings. For instance, the difference in liquidus temperature of the ESV melt treated test samples was up to 13 °C, due to the high accuracy of the thermal analysis. This technology has the required potential for industrial applications.

5.7. Novel Approach to Determine the Level of Microstructural Modification Al-Si Hypereutectic Alloys

5.7.1. The Novel Image Analysis Based Silicon Modification Level (SiML) Algorithm

The aim of this section is to develop a novel analytical approach to determine the level of modification of Si in Al-Si hypo and hypereutectic alloys (3XX.X series). The currently available standards to determine the level of modification of Si particles were investigated. Two standard methods were found; one is the “AFS Standard” and the other one used by the Ford Motor Company and is known as the “Primary Si Size Determination” (Figure 5.47). The first method can only be used for Al-Si hypoeutectic alloys and the second one for Al-Si hypereutectic alloys.
In both methods the determination of the level of modification of Si is carried out by comparing micrographs of the test sample with the respective standards by simple observation, which can result in a method with a bias error. The matching of micrographs by simple comparison does not take the advantage of the analytical accuracy made possible by modern image analysis methods.

Furthermore, the *Primary Si Size Determination* standard for Al-Si hypereutectic alloys has as a main limitation that it only considers the diameter of the primary Si particles and it ignores the other characteristics of the Al-Si eutectic particles. Additionally, in this research the ESV melt treated test samples showed very large size distribution of the Si particles within a single analytical field as shown in Figure 5.48. Therefore, a standard that can be used to analyze all combination of sizes of Si particles is required. Thus a new algorithm to perform a standard analysis of the Si particles in Al-Si alloys was created. This algorithm will be referred as the *"Image Analysis Based Silicon Modification Level"* (SiML). It has the capabilities to evaluate the primary and eutectic Si particles separately or together. In this dissertation it was planned to relate the SiML to the thermal analysis results for its on-line determination.

![Figure 5.47. Micrographs used by Essex Aluminum Plant (EAP) and Ford Motor Company for the determination of the "Primary Si Size Determination". Note: All micrographs must be taken at a 100X magnification.](image-url)
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Figure 5.48. Micrographs showing the differences in size of Al-Si particles found on Al-Si hypereutectic alloys for distinct Si contents and magnifications a) 390.1(I) alloy at 500X magnification and b) 393.2(II) alloy at 50X magnification.

The SiML was developed by using the Leica DMR and Q550IW image analysis system using 100 micrographs of various Al-Si hypereutectic compositions at different levels of modification. These micrographs were observed at various magnifications varying from different 25 to 500X. Figure 5.48 shows LOM micrographs of the coarsest and finest silicon particles that were analyzed to develop the SiML methodology. The Si particles were measured by using the image analysis system to assess the following particle characteristic stereological parameters: Average Area, Average Perimeter, Average Length, Average Width, Average Roundness, Average Aspect Ratio, Average Equivalent Diameter and Number of Particles. Statistical analysis revealed that average area and average perimeter of the silicon particles have the most distinctive correlation with the SiML. Therefore, these two characteristic were selected as stereological parameters to assess the SiML. The micrographs were then ranked individually for each of the stereological parameters and divided into 8 groups corresponding to 8 Si modification levels. Figure 5.49 shows the average levels for each modification level.

The statistical analysis to assess the SiML revealed that Area and Aspect Ratio of the silicon particles could be used as main stereological parameters to distinguish the Al-Si eutectic from the primary Si particles. Figure 5.50 depicts the limits in area and aspect ratio of the two Si phases.

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The average area of the Si particles has an overlap between the Al-Si eutectic and primary Si particles. Between 0 and 50 \( \mu m^2 \) in area only Al-Si eutectic exist and over 1100 \( \mu m^2 \) only primary Si particles were observed. Moreover, 99.95% of the primary Si particles have an area of less than 500 \( \mu m^2 \) and was used as an upper limit for the Al-Si eutectic particles and lower limit for the primary Si ones. Based on this limit, separation of the Al-Si eutectic and primary Si particles can be conducted.

On the other hand, Si particles with area sizes between 50 and 500 \( \mu m \) need a further analysis to determine the stereological characteristics that permit their separation. For this reason and because is well known that primary Si particles are polyhedral while Al-Si eutectics are plate-like, so their aspect ratio was used as a stereological feature that can be used to separate them. The aspect ration of 99.5% of the Al-Si eutectic is 2.3 or larger, while 99.9% of the primary Si particles have an aspect ratio smaller than 2.3.

The 0.5% of the Al-Si eutectic particles fell within the range of primary Si particles and 0.1% of the primary Si ones which is in the range of the Al-Si eutectic particles. The statistical analysis showed that in every 2000 Si particles measured (eutectic and primary), only 2.5 Si eutectic particles were misidentified as primary Si ones, when judged by aspect ratio. On the other hand, in every 200 primary Si particles measured (~7 micrographs) only 1 of them was misidentified as Al-Si eutectic. Thus the error of this method was considered negligible and the number of particles that were not “properly” separated had insignificant influence the final results.
Figure 5.49. Effect of the stereological parameters a) average area, average perimeter, number of particles and b) average aspect ratio, length, width, roundness and average Equivalent Diameter as a function of the SiML.

The analysis of the other measured stereological parameters is as follows; the number of Si particles, has no symmetry below a SiML = 3. Length, Width, or Equivalent Diameter display
an inflexion point at SiML = 5 then change very little for small particles. This makes complicated
the development of a standard scale that can be utilized to evaluate the level of microstructural
modification of Si particles for various compositions or levels of modification (Figure 5.49).
Therefore, Area and Perimeter were selected to create the SiML standard because these two
parameters present a continuous and monotonic decline with the SiML. Next, a particular
analysis using linear and exponential regressions will be conducted for the perimeter and area
respectively (Figure 5.51).

![Graph](image)

**Figure 5.50. Stereological Limits of the Eutectic and Primary Si Particles Present on Al-Si
Hypereutectic Alloys with and without Modification**

From the general form of the relationship in Figure 5.49, the perimeter has been assigned
a linear relationship plotted against the SiML. Figure 5.52 shows the relationship chosen between
the SiML versus Area is exponential. For instance, by looking at Area of the Si particles with a
SiML smaller than 4 a rapid change can be observed, but for a SiML above 4, the response
approaches asymptotic, so it does not adequately separate the Si particles. In order to avoid this
limitation using Area as an analytical parameter for the purpose of this dissertation, the perimeter
was used to determine the SiML. In fact, a linear behaviour of the stereological parameter would
be the ideal case, since it attains the same level of confidence through the entire range of SiML.
However, the **Primary Si Size Determination Scale** (Ford's standard) is based as a function of the
diameter of the primary Si particles. The precise determination of the equivalent diameter of elongated Si particles is impractical, therefore, serious limitations of using the diameter of the Si particles as an analytical parameter exists (Figure 5.49). Image analysis makes the perimeter an easy parameter to be used for the determination of the SiML with the above-discussed advantages.

Figure 5.51. Regression analysis if the Si particles present in Al-Si 3XX alloys for the assessment of the Si Modification Level (SiML), using the results of the linear and exponential (for average perimeter and average area respectively) regressions for the statistical analysis of the monotonic stereological parameters in function of the SiML.

Based on the results of Figure 5.51 the regression equations for Perimeter and Area are shown as:

\[
\text{Perimeter} = -14.904(SiML) + 145.73 \quad R^2 = 0.99
\]

\[
\text{Area} = 1112.3(SiML)^{-1.536} \quad R^2 = 0.97
\]

Using the above equations, the Area and Perimeter of the Si particles present in the microstructure of an Al-Si alloy test sample can be estimated if the SiML is known. It is also possible to determine the SiML based on the average Area and Perimeter of the Si particles with the equations shown in Figure 5.52a and b:
Figure 5.52. Results of the regression analysis of stereological parameters of Si particles conducted for the assessment of the Image Analysis Base Si Modification Level (SiML) by using a) perimeter and b) area of the Si particles. Note: the linear behavior of perimeter to assess the SiML.

The equations presented in Figure 5.52a and b were used to determine the respective Area and Perimeter that correspond to each SiML. The results of these calculations are presented in Table 5.12. Based on the values determined for every SiML, the eight respective micrographs from the 100 micrographs analyzed were chosen for Perimeter and Area respectively. In Figure
5.53 only the micrographs that correspond to the respective regression conducted for the perimeter are shown, because this parameter attained the same response along all the Perimeter scale as a function of the SiML (Figure 5.53).

Table 5.12. Results of the SiML for the Al-Si 3XX.X alloys based on statistical analysis of the Si stereological characteristics. Note: data in this Table corresponds to the micrographs presented in Figure 5.53 and in the equation shown in Figure 5.57 [19].

<table>
<thead>
<tr>
<th>SiML</th>
<th>Area (μm²)</th>
<th>Perimeter Results (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>872.37</td>
<td>130.82</td>
</tr>
<tr>
<td>2</td>
<td>519.94</td>
<td>115.92</td>
</tr>
<tr>
<td>3</td>
<td>299.80</td>
<td>101.02</td>
</tr>
<tr>
<td>4</td>
<td>179.37</td>
<td>86.11</td>
</tr>
<tr>
<td>5</td>
<td>126.08</td>
<td>71.21</td>
</tr>
<tr>
<td>6</td>
<td>107.34</td>
<td>56.31</td>
</tr>
<tr>
<td>7</td>
<td>90.58</td>
<td>41.40</td>
</tr>
<tr>
<td>8</td>
<td>43.23</td>
<td>26.50</td>
</tr>
</tbody>
</table>

The two equations used for the determination of the SiML as a function of Perimeter and Area (Figure 5.52) were used to create a computer program that determines the SiML based on the Image Analysis results. Therefore, SiML is determined automatically as the Image Analysis is conducted. It is also important to mention that the SiML can be determined on three different ways using the equations presented in Figure 5.52, comparing the results of Image Analysis with the results presented in Table 5.12 and comparing the micrographs from Figure 5.53 with microstructure of an actual test sample. Furthermore, the assessment of the SiML can also be carried out by calculating the area fraction of the Si particles with a lower or higher aspect ratio than 2.3. In order to determine the SiML by comparison using the micrographs presented in Figure 5.53 it is important to print the micrographs of the same size of the above-mentioned figure at 200X magnification.

5.7.2. Assessment of the SiML in Ford’s Essex Aluminum Plant Permanent Die-Cast 390.1(II) Al-Si Alloy Pistons

Six Essex Aluminum Plant die-cast regular production pistons and six Optical Emmision Spectroscopy (OES) test samples were used to assess the SiML and the Primary Si Size
\textit{Determination.} The overall idea of this exercise was to present the SiML as a more reliable method, in comparison to the \textit{Primary Si Size Determination} to Essex Aluminum Plant Ford Motor Company. It is important to mention that Essex Aluminum Plant used to add 60 ppm of P as a Si modifier in the form of Cu-8 wt\% P master alloy for its regular production pistons. Figure 5.54 shows an actual Ford's die-cast piston and a micrograph of its respective microstructure. The summary of the results of the \textit{Primary Si Size} Determination and the SiML are presented in Table 5.13.

![Image of micrographs with labels: Level 1, Level 2, Level 3, Level 4, Level 5, Level 6, Level 7, Level 8, Magnification 200 X, 50 \textmu m]

\textbf{Figure 5.53. Calibration LOM micrographs to assess the SiML in the 3XX.X Al-Si alloys [19].}

In order to compare the spatial resolution of the SiML technique versus the Ford's method using the data from Table 5.13 was plotted in Figure 5.55. As can be observed the SiML is capable of distinguishing small differences in the primary Si while the Essex Aluminum Plant grading is a very rough estimation.
Figure 5.54. a) Ford's Essex Aluminum Plant die cast 390.1(II) regular production piston \( (T_{\text{Pouring}} = 750 \pm 10^\circ\text{C}) \) and b) a representative micrograph of the microstructure of the Essex Aluminum Plant piston (200 X magnification). Note: coarse primary Si particles (compared with the microstructures in Figure 5.48 and Figure 5.53) [20].

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Table 5.13. Image analysis results and comparison between the *Primary Si Size Determination* (Essex Aluminum Plant Grade) and the SiML [20].

<table>
<thead>
<tr>
<th>Test Sample Type</th>
<th>Average area of primary Si particles ($\mu m^2$)</th>
<th>Average length of primary Si particles ($\mu m$)</th>
<th>EAP Grade</th>
<th>SiML</th>
</tr>
</thead>
<tbody>
<tr>
<td>OES</td>
<td>314.1 ± 240.6</td>
<td>27.4 ± 10.7</td>
<td>3</td>
<td>1.89</td>
</tr>
<tr>
<td></td>
<td>465.2 ± 350.6</td>
<td>30.1 ± 12.5</td>
<td>3</td>
<td>2.20</td>
</tr>
<tr>
<td></td>
<td>498.2 ± 235.4</td>
<td>29.4 ± 9.8</td>
<td>3</td>
<td>2.05</td>
</tr>
<tr>
<td></td>
<td>524.6 ± 465.8</td>
<td>33.6 ± 23.4</td>
<td>4</td>
<td>1.96</td>
</tr>
<tr>
<td></td>
<td>568.9 ± 435.1</td>
<td>35.9 ± 18.7</td>
<td>4</td>
<td>1.84</td>
</tr>
<tr>
<td></td>
<td>607.7 ± 500.0</td>
<td>38.5 ± 21.5</td>
<td>4</td>
<td>1.75</td>
</tr>
<tr>
<td>Pistons</td>
<td>143.7 ± 88.5</td>
<td>17.8 ± 6.4</td>
<td>2</td>
<td>4.80</td>
</tr>
<tr>
<td></td>
<td>142.7 ± 88.2</td>
<td>17.6 ± 6.6</td>
<td>2</td>
<td>4.81</td>
</tr>
<tr>
<td></td>
<td>178.4 ± 125.9</td>
<td>20.1 ± 8.5</td>
<td>2</td>
<td>4.24</td>
</tr>
<tr>
<td></td>
<td>172.1 ± 74.0</td>
<td>18.9 ± 5.8</td>
<td>2</td>
<td>4.35</td>
</tr>
<tr>
<td></td>
<td>124.5 ± 192.4</td>
<td>16.7 ± 6.1</td>
<td>2</td>
<td>5.25</td>
</tr>
<tr>
<td></td>
<td>269.0 ± 194.5</td>
<td>23.5 ± 9.7</td>
<td>3</td>
<td>2.80</td>
</tr>
</tbody>
</table>

Figure 5.55. Comparison between the Essex Aluminum Plant grade and the SiML of the Ford’s die-cast pistons and OES test samples.
5.7.3. Assessment of the SiML for the Reference and ESV Melt Treated Test Samples for the Statistically Designed Experiments

Assessment of the SiML of the test samples cast for the statistically designed experiments was conducted following the procedures provided in Chapter 4. The SiML of the reference and ESV melt treated test samples was used to determine the effect of the ESV independent variables on the level of refinement of Si. Table A2.4 to Table A2.7 in Appendix II presents the results of the SiML for the respective test samples and the ESV melt treatment conditions. The results of the above-mentioned tables were plotted and are presented in Figures 5.53 to 5.56.

The SiML was also used for the determination of the Homogeneity Coefficient based on its standard deviation \((S_h)\) using Eq. 4.1. The homogeneity coefficient quantifies the macrosegregation of the primary Si particles. The \(S_h\) parameter in Eq. 4.1 is determined based on the most homogeneous test sample (lowest standard deviation). The homogeneity coefficient increases together with the heterogeneity of the test sample. Figure 5.56 shows histograms of selected test samples that were ESV melt treated at different conditions. Due to the gravity effect combined with the chilling of the bottom of the test samples the top centre of the ESV melt treated test samples solidifies last; however, the reference test samples present macrosegregation along the entire test sample. Additionally, Si has a lower density than Al and in order to obey Stokes' law (Eq. 5.4) travels upward. This resulted in primary Si macrosegregation to the top portion of the test sample.

\[
HC = \frac{(S_A)^2 - (S_h)^2}{(S_h)^2} \quad [168] \tag{4.1}
\]

\((S_A)^2\) – Standard deviation of a selected reference test sample.
\((S_h)^2\) – Lowest standard deviation measured among the test samples.
HC - Homogeneity coefficient.

Stokes’ law states that a dropped spherical object in a viscous but laminar fluid must travel up or down proportionally to its specific gravity with respect to the fluid, at a rate proportional to the square of the diameter of the particles and inversely proportional to the viscosity of the fluid (see Eq. 5.4). Therefore, the primary Si particles in a reference test sample can segregate to a higher degree and velocity than the refined Si particles present in the ESV melt.
Chapter 5. Results and Discussions

treated test samples. This results in a higher level of macrosegregation observed in the reference test sample in comparison to the ESV melt treated. However, ESV melt treated test samples using low current, frequency or high temperatures show the opposite behaviour (Figures 5.53a and 5.53b and Table A2.8).

\[
v = \frac{1}{18} \left( \frac{(\rho_s - \rho_f)gd^2}{\mu} \right) \quad \text{(cm/s)} \tag{Eq. 5.4}
\]

Where:
- \(v\) the settling velocity (cm/s)
- \(g\) the gravitational acceleration (cm/s²)
- \(\rho_s\) the density of the dropped object (g/cm³)
- \(\rho_f\) density of the fluid (g/cm³)
- \(d\) the diameter of the dropped object (cm)
- \(\mu\) the viscosity of the fluid (Dyne s/cm²)

The average diameter of the primary Si particles can be determined using the results of the image analysis. Using these image analysis results from the quenching experiments (Figure 5.34) and Eq. 5.4, the settling velocity of the primary Si particles was estimated. It was found that the primary Si particles of the reference test samples (SiML = 4.3) have a ~300% higher settling velocity in comparison to the ESV melt treated test sample with a SiML = 6.29. Therefore, in the reference test sample the tendency to segregate is considerably higher. For more detail about the settling velocities for the various ESV melt treated test samples under different ESV melt treatment conditions Table A2.8 in Appendix II can be consulted.

Figure 5.57 presents the combined effect of frequency and ESV temperature range on the SiML. This figure shows that the effectiveness of the ESV melt treatment increases with the frequency and decreases with the temperature of the treatment. The results of the SiML indicate that ESV melt treatments using low frequency and high temperature have the opposite effect on Si modification. Therefore, test samples ESV melt treated at low frequency and high temperature presented lower SiML in comparison to the reference test sample of the 390.1(I) alloy. In other words low intensity ESV melt treatments promote the coarsening of primary Si particles.

The effect of the ESV melt treatment on the Si modification is a logarithmic function of time (Figure 5.58) for both, 390.1(I) and 393.2(I), Al-Si alloys. This indicates that due to some industrial constraints (short treatment times in the continuous process) the SiML could be compromised. For instance, for the ESV melt treatment using the 390.1(I) alloy in the first 180 s the SiML went from 4.30 to 6.29; in the following 180 s the SiML increased to 6.38 only. In the
case where the extension of Eq. 5.6 was valid, then to reach the highest SiML (8) an additional ~23 min is required. In a similar exercise conducted for the 393.2(I) alloy the SiML went from 2.15 to 4.99 in the first 360 s and to 5.83 in the following 180 s and an additional ~40 min would be required to reach a SiML = 8 (Eq. 5.7). It was concluded that it is impractical to use large ESV melt treatment times to reach the highest SiML.
Chapter 5. Results and Discussions

Table 5.14. Values of the SiML for each analytical field.

<table>
<thead>
<tr>
<th>Position X/Y</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.7</td>
<td>4.1</td>
<td>1.4</td>
<td>4.2</td>
<td>0.9</td>
</tr>
<tr>
<td>B</td>
<td>3.4</td>
<td>3.7</td>
<td>4.4</td>
<td>3.8</td>
<td>4.1</td>
</tr>
<tr>
<td>C</td>
<td>4.5</td>
<td>4.2</td>
<td>4.3</td>
<td>2.7</td>
<td>5.8</td>
</tr>
<tr>
<td>D</td>
<td>5.0</td>
<td>5.9</td>
<td>6.0</td>
<td>3.5</td>
<td>5.0</td>
</tr>
<tr>
<td>E</td>
<td>5.1</td>
<td>5.5</td>
<td>5.4</td>
<td>5.1</td>
<td>5.9</td>
</tr>
</tbody>
</table>

Table 5.14. Values of the SiML for each analytical field.

<table>
<thead>
<tr>
<th>Position X/Y</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>5.8</td>
<td>4.6</td>
<td>3.9</td>
<td>5.8</td>
<td>3.7</td>
</tr>
<tr>
<td>B</td>
<td>4.8</td>
<td>6.1</td>
<td>5.0</td>
<td>3.1</td>
<td>6.6</td>
</tr>
<tr>
<td>C</td>
<td>5.0</td>
<td>5.6</td>
<td>1.0</td>
<td>5.3</td>
<td>5.1</td>
</tr>
<tr>
<td>D</td>
<td>5.5</td>
<td>5.1</td>
<td>5.4</td>
<td>5.7</td>
<td>5.2</td>
</tr>
<tr>
<td>E</td>
<td>5.6</td>
<td>5.8</td>
<td>5.5</td>
<td>5.8</td>
<td>5.4</td>
</tr>
</tbody>
</table>

Table 5.14. Values of the SiML for each analytical field.

<table>
<thead>
<tr>
<th>Position X/Y</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>6.5</td>
<td>6.1</td>
<td>4.3</td>
<td>3.9</td>
<td>5.9</td>
</tr>
<tr>
<td>B</td>
<td>5.7</td>
<td>5.5</td>
<td>5.2</td>
<td>4.2</td>
<td>5.7</td>
</tr>
<tr>
<td>C</td>
<td>6.3</td>
<td>5.3</td>
<td>5.6</td>
<td>4.7</td>
<td>5.7</td>
</tr>
<tr>
<td>D</td>
<td>6.6</td>
<td>6.7</td>
<td>4.9</td>
<td>5.8</td>
<td>6.3</td>
</tr>
<tr>
<td>E</td>
<td>7.3</td>
<td>5.5</td>
<td>6.8</td>
<td>6.7</td>
<td>7.1</td>
</tr>
</tbody>
</table>

Figure 5.56. Distribution of the SiML for selected 390.1(I) test samples at a) reference, b) 10 Hz and c) 40 Hz.
Table 5.14. Values of the SiML for each analytical field.

<table>
<thead>
<tr>
<th>Position X/Y</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>5.5</td>
<td>3.5</td>
<td>2.7</td>
<td>4.8</td>
<td>5.6</td>
</tr>
<tr>
<td>B</td>
<td>6.5</td>
<td>5.5</td>
<td>3.9</td>
<td>5.3</td>
<td>5.9</td>
</tr>
<tr>
<td>C</td>
<td>6.3</td>
<td>5.0</td>
<td>5.2</td>
<td>5.5</td>
<td>5.3</td>
</tr>
<tr>
<td>D</td>
<td>6.3</td>
<td>6.7</td>
<td>6.2</td>
<td>6.9</td>
<td>5.6</td>
</tr>
<tr>
<td>E</td>
<td>7.0</td>
<td>6.7</td>
<td>6.9</td>
<td>6.6</td>
<td>7.0</td>
</tr>
</tbody>
</table>

Figure 5.56. Continuation. d) 80 Hz and e) 100 Hz. Note: the higher SiML in the top centre of the histograms, which represents the primary Si macrosegregation.

Figure 5.57. SiML of the reference and ESV melt treated test samples using 390.1(l) alloy, different frequencies and temperatures. Note: high refinement matches with high $f$ and low $T$. 

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Figure 5.58 presents the respective regression curves for Eq. 5.6 and Eq. 5.7 for the 390.1(I) and 393.2(I) alloys respectively as a function of the time of the ESV melt treatments; with these the level of modification of different alloys can be determined for different times. It is important to note that Figure 5.58 corresponds to the obtained SiML results for the ESV melt treated test samples for the 390.1(I) and 393.2(I) alloys that were treated using an $I_{ACDC} = 100 \, A$, $f = 100 \, Hz$, $T_{ESV} = 610 \pm 2^\circ C$ and $750 \pm 2^\circ C$ respectively.

\[
SiML_{390.1(I) \text{ Alloy}} = 0.8376 Ln(t) + 1.7514 \quad (t \text{ in s}) \quad R^2 = 0.95 \quad \text{Eq. 5.5}
\]

\[
SiML_{393.2(I) \text{ Alloy}} = 1.3607 Ln(t) - 2.7819 \quad R^2 = 0.97 \quad \text{Eq. 5.6}
\]

where: $t$ time of the ESV melt treatment (s)

Note that Eq. 5.5 and Eq. 5.6 are not valid for shorter times than 22s and 45s respectively.

![Figure 5.58. Effect of the ESV treatment time under isothermal conditions on the silicon modification level (SiML) for the 390.1(I) and 393.2(I) alloys using an $I_{ACDC} = 100 \, A$ at $T_{ESV} = 610$ and $750 \pm 2^\circ C$ from the liquidus of the respective alloys. Note the logarithmic effect between time and SiML.](image)

Using the results presented in Figure 5.43 the regression equations were determined for the 390.1(I) and 393.2(I) alloys (Eqs. 5.7 and 5.8 respectively) that were ESV melt treated under

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different conditions. Figure 5.43 shows the effect of ESV melt treatment time on the liquidus temperatures of the respective alloys.

\[
T_{LQ(390.1(t))} = 3.3297Ln(t) + 591.37 \quad T_{LQ} \text{ in } ^\circ C; t \text{ in s} \quad R^2 = 0.95 \quad \text{Eq. 5.7}
\]

\[
T_{LQ(393.2(t))} = 6.2933Ln(t) + 716.61 \quad T_{LQ} \text{ in } ^\circ C; t \text{ in s} \quad R^2 = 0.97 \quad \text{Eq. 5.8}
\]

Eqs. 5.5 to 5.8 were obtained for the same test samples, but using different characterization technique; Eqs. 5.5 and 5.65 were determined with image analysis (SiML) and Eqs. 5.7 and 5.8 with thermal analysis. By looking at the four previously mentioned equations it can be concluded that the effect of time on the ESV melt treatments is logarithmic. Also, because Eqs. 5.5 and 5.7 as well as Eqs. 5.6 and 5.8 correspond to the same test samples they were combined in Eqs. 5.9 and 5.10 respectively. Eqs. 5.9 and 5.10 are very important because they can be implemented into the UMSA or AITAP software or into the thermal analysis system attached to the ESV workstation to predict in real time (on line) the level of Si modification during the solidification of castings. Therefore, they have the potential for industrial applications.

\[
SiML_{390.1(t)} = 0.251T_{LQ} - 146.9976 \quad T_{LQ} \text{ in } ^\circ C \quad \text{Eq. 5.9}
\]

\[
SiML_{393.2(t)} = 0.2162T_{LQ} - 155 \quad T_{LQ} \text{ in } ^\circ C \quad \text{Eq. 5.10}
\]

where \( T_{LQ} \) is the Liquidus Temperature

Note: Eq. 5.9 and Eq. 5.10 apply only to the test samples of the respective alloys solidified in the Cu plate (air quenched).

A similar analysis as for the ESV melt treatment time was conducted for the frequency (AC current) of the ESV melt treatments. Figure 5.59 shows the results of the SiML on the ESV melt treated test samples using different AC current frequencies. Eqs. 5.11 and 5.12 are the regression equations for the 390.1(I) alloy ESV melt treated using an \( I_{AC/DC} = 40 \) and 100 A respectively; the other ESV melt treatment parameters were a \( t = 180 \) s and \( T_{ESV} = 610 \pm 2^\circ C \). Note: no equations for the 393.2(I) alloy are presented because this alloy was ESV melt treated at different times only.
In the previous sets of equations a methodology to determine the SiML of ESV melt treated test samples was provided. By using these equations it was possible to determine the SiML as a function of the time, frequency and liquidus temperature. These findings had the potential for industrial applications to predict the level of Si modification during the recording of the cooling curves. Additionally, using Figure 5.44 and from Figures 5.54 to 5.57 the SiML could also be assessed for different ESV melt treatment conditions. All data used to assess the previously mentioned equations and graphs is available in Appendix II (Table A2.4 to Table A2.7).

\[ SiML_{AC/DC=40A} = 0.0153(f_{Hz}) + 4.4856 \quad f \text{ in Hz} \quad R^2 = 0.94 \quad \text{Eq. 5.11} \]

\[ SiML_{AC/DC=100A} = 0.0189(f_{Hz}) + 4.3737 \quad f \text{ in Hz} \quad R^2 = 0.99 \quad \text{Eq. 5.12} \]

Figure 5.59. Relationship between frequency \( (f_{Hz}) \) and SiML on the ESV melt treated test samples at different currents for 3 min at \( T_{ESV} = 610 \pm 2^\circ\text{C} \) and on the 390.1(I) Al-Si alloy. Note the linear effect between frequency and SiML.

In the literature, several researchers have reported that changes in liquidus temperature have an effect on microstructure characteristics such as grain size and SDAS [39, 41, 77, 78, 95, 16, 196 - 199]. Therefore, the study of the liquidus temperature as a function of the ESV melt treatment conditions is a key to the success of this research. Based on Figures 5.42 and 5.43 it
can be seen that the frequency, ESV treatment temperature, degassing conditions and current have an effect on the liquidus.

\[
\text{SiML} = -0.0074(T_{\text{ESV Treat}}) + 10.812 \\
R^2 = 0.95
\]

\[
\text{SiML} = -0.0169(T_{\text{ESV Treat}}) + 16.046 \\
R^2 = 0.95
\]

\[
\text{SiML} = -0.0117(T_{\text{ESV Treat}}) + 12.07 \\
R^2 = 0.99
\]

Figure 5.60. Relationship between the ESV melt treatment temperature and the SiML on the 390.1(I) alloy test samples using different frequencies. The ESV melt treatment conditions were \(I_{\text{AC/DC}} = 100\) A and \(t = 3\) min. Note: the SiML increases and lower the ESV melt treatment temperature and high frequency.

5.7.4. Statistical Determination of the Contribution of Independent Variables Used for the ESV Melt Treatments on the SiML

The results of the SiML of the ESV melt treated test samples solidified at low and high cooling rates using different heat exchange conditions (1D, 2D and steel die mould) were used to conduct a multiple regression analysis. The multiple regression analysis was carried out to correlate the independent variables to the SiML. The statistical evaluation was conducted to determine the contribution of independent variables on the SiML and also the possible interaction among independent variables. The data for the different SiML is provided in Appendix II in Table A2.4 to Table A2.7. The results of the statistical analysis for single regressions are presented Eqs. 5.4 – 5.12 and were determined using Microsoft Excel. Multiple regression analysis was conducted using Minitab and Microsoft Excel software [7].
Figure 5.61 summarizes the results of the statistical analysis using the Minitab software, where independent variables were statistically analyzed to determine their effect on SiML. In every case figure with an intersecting line indicates the interaction between the respective independent variables, which means that in order to achieve an optimum SiML both variables need to be controlled together. Parallel lines show no interaction between independent variables. For instance, the ESV melt treatment temperature and frequency have the strongest interaction among all independent variables. In order to reach the highest SiML using a low ESV melt treatment temperature or high frequency alone with their effect is almost negligible. However, combining a low ESV melt treatment temperature with a high frequency the effectiveness of the melt treatment is increased, thus a higher SiML is achieved.

The results of the statistical analysis conducted using data from Table 5.15 and Figure 5.61 confirms that the multiple regression analysis using the Minitab and Microsoft Excel software. For instance, comparing Figure 5.61 with Table 5.15 it can be observed that in both cases the contribution of every independent variable to the SiML is in good agreement. Eqs. 5.13 and 5.14 are the multiple regressions obtained with Microsoft Excel and they were obtained for all reference and ESV melt treated test samples that were solidified at low and high cooling rates. The low cooling rate (Eq. 5.13) corresponds to the test samples solidified in the standard ESV cup with a release of the heat radially and vertically respectively (1D and 2D). The high cooling rate (Eq. 5.14) was obtained in a steel die cast mould (prototype pistons and cylinder liners). The results of the SiML for the prototype pistons and cylinder liners are presented in Section 5.9.

Table 5.15. Results of the contribution of independent variables on the SiML used for statistically designed experiments (ESV melt treatments). The variables contribution was determined by multiple regressions with Minitab and Microsoft Excel software [7].

<table>
<thead>
<tr>
<th>Independent Variables</th>
<th>Effect on SiML</th>
<th>Minitab Software (Only high Cooling Rate)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Maximum Contribution, (SiML)</td>
<td>Low CR</td>
</tr>
<tr>
<td>Frequency, (Hz)</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>$T_{ESV}$, (°C)</td>
<td>0.9</td>
<td>0.9</td>
</tr>
<tr>
<td>Current ($I_{AC/DC}$)</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>Time, (s)</td>
<td>2.1</td>
<td>2.8</td>
</tr>
<tr>
<td>Composition, (wt% Si)</td>
<td>2.1</td>
<td>1.7</td>
</tr>
<tr>
<td>Degassing</td>
<td>0.6</td>
<td>0.4</td>
</tr>
<tr>
<td>Cooling Rate (°C/s)</td>
<td>0.1</td>
<td>2.0</td>
</tr>
<tr>
<td>Total (SiML)</td>
<td>6.4</td>
<td>8.3</td>
</tr>
</tbody>
</table>
Figure 5.61. Statistical analysis of the effect of independent variables used on microstructure of ESV melt treated test samples. Note: parallel lines indicate no interaction between analyzed independent variables [7].

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The mathematical models presented in this chapter were obtained with Microsoft Excel. Figure 5.62 shows the comparison of the measured SiML (using image analysis) for test samples and the predicted ones using Eqs. 5.13 and 5.14. It can be observed the relative low scatter between the determined and predicted values for the SiML. The average error of the multiple regression analysis obtained using Microsoft Excel was <0.5

\[
SiML = 16.402621 + 0.0032471(f_{Hz}) + 0.003876(I_{AC/DC})
- 0.014793(T_{ESV}) + 11.3785(H_2) + 0.00612855(t)
- 0.192356(\text{wt\%} \text{Si}) - 14.50403(CR)
\]
\[R^2 = 0.86 \quad \text{Eq. 5.13}\]

\[
SiML = 14.783726 + 0.0030816(f_{Hz}) + 0.0033109(I_{AC/DC})
- 0.013962(T_{ESV}) + 8.1178095(H_2) + 0.00606826(t)
- 0.19289(\text{wt\%} \text{Si}) + 1.3318574(CR)
\]
\[R^2 = 0.90 \quad \text{Eq. 5.14}\]

Where: $f_{Hz}$ is in the range of 10 to 100 Hz

$I_{AC/DC}$ is in the range of 40 to 100 A for both AC and DC currents

$T_{ESV}$ is in the range of 620 to 680°C for the 390.1(I) alloy and 750°C for the 393.2(I) alloys respectively

$H_2$ is in the rage of 0.065 to 1.5 mL H$_2$/100g of Al

$T$ is in the range of 20 to 360 s for the 390.1(I) alloy and from 20 to 480 for the 393.2(I) alloy

$\text{wt\%Si}$ is in the range of 14 to 26 $\text{wt\%Si}$

low CR is in the range of 0.08 to 0.09°C/s and high cooling rate up to 1.6°C/s

Note: the Eq. 5.13 covers the cooling rate range from 0.08 to 0.09 °C/s and for Eq. 5.14 the cooling rate range between 0.08 and 1.6°C/s. The effect of the $T_{ESV}$ parameter is negative in both equations, which indicates that the lower the temperature the more effective the ESV melt treatment.
Chapter 5. Results and Discussions

Figure 5.62. Relation between the SiML of the measured and predicted SiML using the multiple regression analysis. Note: relative low scatter among measured and predicted SiML; the standard error for both equations was <0.5.

High hydrogen levels \([H_2]\) promote undesirable porosity, however ESV melt treatments at different temperatures and frequencies apparently have no effect on porosity; however, \([H_2]\) and high AC/DC currents enhance porosity formation. ESV melt treatments conducted using low current \((I_{AC/DC})\), frequency, high temperature and un-degassed melt resulted in higher SiML. ESV melt treatments using high currents and degassed melt (Figure 5.61) have a negative effect.

The results presented in Section 5.7.3 correspond to the findings of the statistical analysis of the ESV melt treated test samples for the 390.1(I) and 393.2(I) alloys solidified at low and high cooling rate. An example of the combined effect of frequency and \(T_{ESV}\) is shown in Figures 5.54 and 5.57; where high frequencies and low \(T_{ESV}\) correspond to a high level of modification. Eqs. 5.11, 5.12 and Figure 5.59 show that frequency has a linear effect on SiML. Also, \(T_{ESV}\) has a linear affect on the Si modification (Figure 5.60). However, the effect of time on SiML for ESV melt treated test samples is logarithmic (Eqs. 5.5, 5.6 and Figure 5.58).

The results of Section 5.7.3 demonstrate that independent variables with a linear effect on SiML are more effective because their response is constant along the ESV melt treatment tested.
ranges. Logarithmic responses are asymptotic; therefore after certain point their effect becomes negligible.

5.7.5. Assessment of the SiML of Selected ESV Melt Treated Test Samples Solidified Under Natural Heat Exchange Conditions

To this point, the present results and analysis pertained to the results of the ESV melt treated test samples where the heat was released in 1D (radial) and 2D (radial and vertical). Therefore, in this particular section a comparison between the results of the ESV melt treatment test samples solidified in 1D and 2D (cooling rate = 0.08 to 0.09 °C/s) is conducted. The ESV melt treatment conditions used to conduct this series of experiments are shown in Table 5.16. These experiments were planned to demonstrate the effect of the directional solidification (heat released in 2D). AC current frequency and ESV melt treatment temperature were selected based on the effect of the ESV melt treatment for test samples solidified on the Cu Plate (Table 5.15).

TA results of the test samples solidified under natural heat exchange conditions are presented in Figure 5.41 and from Figures 5.44 to 5.46 and Table A2.9 to Table A2.11 (Appendix II). The ESV melt treatment conditions for these test samples were \( I_{ACDC} = 100 \) A for \( t = 3 \) min and a cooling rate = 0.08°C/s, using degassed melt, for different melt treatment temperature and frequency of the AC current. The test samples were analyzed using image analysis system to determine their SiML and were compared with the test samples solidified on the Cu plate (air quenched, heat released in 2D). Figure 5.63 shows the SiML as a function of the change in liquidus temperature for the different ESV melts treatment conditions using 390.1(I) alloy. Eq. 5.13 predicts the SiML as a function of the \( T_{LIQ} \) (compare Eqs. 5.9 and 5.10 with Eq. 5.13).

\[
SiML_{1D} = 0.2317(T_{LIQ}) - 134.52 \\
R^2 = 0.94 \\
\text{Eq. 5.15}
\]

where 1D Radial Heat Exchange (Natural Heat Exchange Conditions) and \( T_{LIQ} \) Liquidus Temperature
Figure 5.63. Relationship between liquidus temperature and SiML for ESV melt treated test samples for the 390.1(I) alloy solidified under natural heat exchange conditions (radial heat released (1D)). ESV melt treatment conditions are summarized in Table 5.16.

Evaluating Eqs. 5.9 and 5.15 for a hypothetical case to reach the highest level of Si Modification (SiML = 8) the resultant liquidus temperatures were 615.11°C and 617.53 respectively for the 390.1(I) alloy. The small difference in liquidus temperature and SiML for the test samples solidified under different heat exchange conditions can be due to the difference in cooling rate. Air quenched test samples solidified ~ 10% faster (from a cooling rate = 0.08 to 0.09 °C/s).

The release of the heat in the radial direction allowed the determination of the nucleation temperatures of the Mg, Ni, Cu enriched phases. As discussed in Section 5.5 in the case of test samples directionally solidified the precise determination of the temperatures of the above-mentioned phases was not possible. Using the methodology shown in Figure 5.26 the apparent fraction solid of the previously mentioned phases was determined.
Table 5.16. SiML for the 390.1(1) alloy ESV melt treated test samples solidified under natural heat exchange conditions (radial heat released (1D)).

<table>
<thead>
<tr>
<th>Test Sample ID #</th>
<th>SiML</th>
<th>Standard Deviation</th>
<th>Hom. Coeff. (HC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference Test Sample</td>
<td>4.5</td>
<td>3.9</td>
<td>26.3</td>
</tr>
<tr>
<td>$f=40 \text{ Hz}; T_{ESV} = 685^\circ \text{C}$</td>
<td>5.0</td>
<td>4.1</td>
<td>28.3</td>
</tr>
<tr>
<td>$f=40 \text{ Hz}; T_{ESV} = 650^\circ \text{C}$</td>
<td>4.9</td>
<td>3.9</td>
<td>25.8</td>
</tr>
<tr>
<td>$f=40 \text{ Hz}; T_{ESV} = 610^\circ \text{C}$</td>
<td>5.4</td>
<td>3.6</td>
<td>22.3</td>
</tr>
<tr>
<td>$f=80 \text{ Hz}; T_{ESV} = 610^\circ \text{C}$</td>
<td>5.7</td>
<td>3.0</td>
<td>14.9</td>
</tr>
<tr>
<td>$f=100 \text{ Hz}; T_{ESV} = 650^\circ \text{C}$</td>
<td>5.8</td>
<td>2.5</td>
<td>10.2</td>
</tr>
<tr>
<td>$f=100 \text{ Hz}; T_{ESV} = 610^\circ \text{C}$</td>
<td>6.1</td>
<td>2.2</td>
<td>7.8</td>
</tr>
</tbody>
</table>

5.7.6. The Effect of Remelting on the ESV Melt Treated Test Samples

In order to determine the effect of remelting (of the 390.1(1) alloy); reference and ESV melt treated test samples were remelted using the UMSA platform. Four reference and four ESV melt treated test samples with an average SiML = 4.30 ± 4.49 and 6.12 ± 1.23 respectively were observed. One of the test samples was analyzed using image analysis after machining, one was remelted once, another twice and the last one three times and then analyzed using image analysis. The data for thermal analysis for the reference and ESV melt treated test samples is summarized in Table 5.16 and Table A2.10 respectively. The remelted test samples were heated using the UMSA platform at ~750°C and held isothermally for 1 min; then solidified under natural heat exchange conditions (radial heat exchange (1D)). During the entire heating and cooling cycles the heating and cooling curves of the test samples were recorded (Figure 5.64). The representative LOM micrographs are shown in Figure 5.65.
Figure 5.64. Thermal signatures for the 390.1(I) alloy ESV melt treated test sample after the first and second remelting using the UMSA platform. The test samples were ESV melt treated using a $I_{ACDC} = 100$ A, $f = 100$ Hz, $t = 3$ min, and a $T_{ESV} = 610 \pm 2^\circ$C. The numbers correspond to reactions identified in Table 5.5.

Figure 5.65 shows that the reference test sample after the remelting procedure has the same similar SiML as the regular 390.1(I) samples that was solidified under natural heat exchange conditions. Conversely, the ESV melt treated test sample following the first remelting present some level of modification of the Al-Si particles. It is also clear that after the remelting some of the primary Si particles appear, but they are neither as coarse nor are there as many as in the reference test sample. The image analysis results and the calculated (using Eq. 5.13) SiML of the test samples after every remelting sequence are presented in Figure 5.66.

Figure 5.66 shows the results of the SiML on the ESV melt treated test samples remelted using the UMSA platform, which shows that after the first and second remelting sequence the SiML was reduced by 15 and 25% respectively. Therefore, 85 and 80 % of the modification of the Si particles remains (Figure 5.65). However, after the third remelting 80% of the Si modification was lost, which corresponds with the LOM observations (Compare Figures 5.62a to 5.62c with Figures 5.62d to 5.62f). The accuracy of Eq. 5.15 is presented in Figure 5.66 where the SiML was predicted in the remelted test samples based on the liquidus temperature of the remelted UMSA samples.
The thermal analysis characteristics (Figure 5.64), of the ESV melt treated test samples after the first and second remelting showed a decrease in the liquidus temperature. The numerical thermal analysis data of the as-cast and remelted reference and ESV melt treated test samples are presented in Table A2.12 and Table A2.13 (Appendix II). Beside the difference in liquidus in the ESV melt treated test samples after remelting an increased in the fraction solids was observed up to the DCP. This increment was ~ 33% and indicates that some of the primary Si particles were modified by the ESV melt treatment into Al-Si eutectic. This Al-Si eutectic was partially transformed after the three remelting sequences, however, in neither case were they totally transformed into primary Si (Figure 5.66). The identification of the Mg2Si eutectic was unclear in the thermal signature of the remelted ESV melt treated test sample; it is for this reason that its fraction solid could not be determined. The fraction solid at the Al2Cu eutectic is almost the same for all remelted and reference test samples, which confirmed that ESV melt treatments have a negligible effect on the modification of the Cu enriched phases. All numerical data is available in Table A2.12 and Table A2.13 (Appendix II).
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Reference as-cast

ESV melt treated as-cast

Reference after the second remelting

ESV melt treated after the second remelting

Reference after the third remelting

ESV melt treated after the third remelting

Figure 5.65. LOM micrographs for the 390.1(I) alloy of the (a-c) reference and (d-f) ESV melt treated test samples, as-cast (a, d), remelted twice (b, e) and remelted three times (c, f) remelting procedure using the UMSA platform. 200 X magnification.

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5.8. Analysis of the Thermal Stability of the 3XX.X Series of Al-Si Alloys

The main focus of this dissertation is the application of ESV treated melts into automotive applications, including pistons and cylinder liners. Al-Si cylinder liners and engine blocks are in most cases still a research and development topic. In the early stages of this research work several discussions were held between the IRC and Ford Motor Company piston group [12, 13 - 15] with the aim to study potential applications of ESV melt treatments on pistons. One of the main concerns for piston applications is the thermal stability of Al-Si hypereutectic alloys. In fact, because of the importance of thermal stability of the pistons they are not solution treated, but artificially aged only. Therefore, a study of the thermal stability of Al-Si alloys was carried out.
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Thermal stability is the permanent change in dimensions of castings upon a solution heat treatment and aging. It is important not to confuse this growth with the thermal expansion coefficient. Pistons must be thermally stable, otherwise in a running engine they can fail causing permanent damage of the engine. The most common failure causes are leaking of oil into the combustion chamber, or the collapse of the skirt [208, 210, 211]. Ford’s Piston Engineering Specification demands that the highest thermal stability correspond to a lower hardness during the artificial aging. In order to reach the previously mentioned conditions there is a compromise between temperature and time [210].

The dimensional stability of pistons is measured by heating the pistons at a temperature of 200°C for six hours followed by cooling and an additional heating for twelve hours at the same temperature [210]. Although most of the dimensional changes occur after the six hour period the eighteen hour assures that the full dimensional changes occur. The dimensions of the pistons subjected to this test are measured on the top land, skirt, bore diameter (D in cm), and at the bottom of the skirt before and after the heating process. The average dimensional change must be as follows (Eqs. 5.16 and 5.17):

\[
\text{Dimensional Change} = \frac{0.020* D}{100}, \quad \text{(cm)} \quad \text{Eq. 5.16}
\]

and the average maximum dimensional change must be less than \(\frac{0.025* D}{100}\) \text{Eq.5.17}

Exceeding these limits indicates that inadequate aging conditions were selected and/or the piston material is not suitable for this application.

The skirt collapse is another factor that must be taken into consideration for piston design. The skirt collapse is the phenomenon where the piston skirt takes a permanent dimensional stability. This phenomenon occurs when the residual stresses after the stress relief heat treatment act in the direction of the applied load on the skirt [210]. As a result the clearance between the piston skirt and the cylinder bore increases. In fact, the skirt collapse is a function of the artificial aging conditions and the dimensional stability. However, a direct test between the dimensional stability and skirt collapse is not possible because other factors affect the skirt collapse, such as piston design and skirt stiffness. The skirt collapse needs to be limited to less than 0.055% of the piston diameter, larger differences are directly related to an unsuitable heat treatment procedure.
The thermal expansion coefficient of several alloy compositions (Table 4.1) was determined using dilatometry (Figure 5.67). The dilatometer utilized was a Netzsch 402EP and the dilatometry analysis was conducted by heating the test samples from room temperature to 550°C and holding at this temperature for 10 min. Figure 5.67 presents the change in thermal expansion coefficient as a function of the Si<sub>EQ</sub> composition, which was used to assess a mathematical model to predict the thermal expansion coefficient at different temperatures as a function of the Si<sub>EQ</sub> composition (Eqs. 5.18 and 5.19). Using the Si<sub>EQ</sub> concept developed for Al-Si hypoeutectic [205] and hypereutectic [18] compositions, the thermal expansion coefficient of a wide variety of Al-Si alloys can be determined. The respectively equations of the regression analysis of the dilatometry results for 500°C and 400°C are Eqs. 5.18 and 5.19. The dilatometry studies were conducted together with S. Lattuca [9].

\[
\begin{align*}
TEC_{500°C} &= -0.0055(Si_{EQ})^4 + 0.22(Si_{EQ})^3 - 3.076(Si_{EQ})^2 + 16.62(Si_{EQ}) - 5.32 \quad R^2 = 0.92 \quad \text{Eq. 5.18} \\
TEC_{400°C} &= -0.005(Si_{EQ})^4 + 0.195(Si_{EQ})^3 - 2.56(Si_{EQ})^2 + 12.67(Si_{EQ}) + 3.15 \quad R^2 = 0.94 \quad \text{Eq. 5.19}
\end{align*}
\]

where: \( Si_{EQ} \) is the equivalent chemical composition [18, 205]

\( TEC \) is the Thermal Expansion Coefficient at the specified temperature in °C

The determination of the thermal expansion coefficient was conducted on different Al-Si hypo and hypereutectic and cast iron alloy temperatures ranging from 100°C to 500°C. The main idea is to compare the differences between the thermal expansion coefficient of cast iron and the 390.1(I), 393.2(I) alloys as this can be used as an argument to justify the substitution of cast iron for Al-Si hypereutectic alloys for cylinder liner applications. For this reason, dilatometry experiments were conducted at temperatures of 500°C and 400°C which are the critical temperatures where the residual stresses in the engine blocks start to be developed (Figure 5.68).

Test samples for the 390.1(I) and 390.1(II) alloys were obtained from the pistons provided by KUS and Essex Aluminum Plant respectively. Dilatometry analysis was carried out on ESV melt treated test samples for the 390.1(I) and 390.1(II) alloys. The artificial aging temperature used for the KUS and Essex Aluminum Plant pistons was between 260°C and 280°C, which is also the approximate service temperature of a running engine, therefore there was special interest in the expansion between 200°C and 260°C (Figures 5.66 and 5.67).
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Figure 5.67. Thermal expansion coefficient in percentage (%) at 400 and 500°C for the various Al-Si alloys tested.

Figure 5.68. Thermal expansion coefficients for the tested alloys at different temperatures. Note: test samples for the 390.1(I) and 390.1(II) alloys were tested in as-cast and ESV treated conditions ($I_{ACDC}=100A$, $f=100Hz$, $t=3min$, $T_{ESV}=610$ and $640°C$ respectively).

Figures 5.64, 5.65 and 5.66 shows that the thermal expansion coefficient is affected by the chemical composition as well as by the temperature. The thermal expansion coefficient of

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cast iron has the lowest and expansion coefficient and is \( \sim 42\% \) lower that the W319 alloy (Figure 5.69) at the service temperature of the engine (280°C). At the same temperature the difference between the W319 and the 390.1(I) was \( < 6\% \) for test samples in the as-cast and ESV melt treated conditions respectively. At lower temperatures the difference in thermal expansion coefficient between the W319 and the cast iron becomes more critical (~74% at a 100°C) for the integrity of the engine blocks that use cast-in cast iron cylinder liners. It is the main cause of the development of residual stresses in Al engine blocks with cast-in cast iron cylinder liners.

![Figure 5.69. Thermal expansion coefficient of several alloys at the maximum service temperature (280°C) conditions in a running engine. Note: 390.1(I) and 390.1(II) alloys were ESV melt treated at \( I_{ACDC} = 100A, f = 100 \text{ Hz}, t = 3 \text{ min}, T_{ESV} = 610, 640 \pm 2^\circ\text{C} \) for the respective alloys.](image)

5.9. Laboratory Casting of the ESV Melt Treated Automotive Components

Using the results of thermal analysis of the prototype pistons and cylinder liners the calculated cooling rates were 1.3 and 1.6 \(^\circ\text{C/s}\) respectively. This was estimated show Eq. 5.3. The SiML of the reference prototype pistons and/or cylinder liners resulted in the most refined microstructure (SiML = 5.06 and 5.85 respectively), among the test samples poured with the untreated melt. Figure 5.15 shows the representative micrographs of the prototype pistons and
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cylinder liners. The LOM micrographs of the automotive semi-components cast using the 390.1(I) alloy ESV melt treated are shown in Figure 5.22; the following were the ESV melt treatment conditions $I_{AC/DC} = 100$ A, $f = 100$ Hz, $t = 3$ min and $T_{ESV} = 610^\circ$C. The SiML of the prototype cylinder liner resulted in the highest (SiML = 7.66) in all the research conducted. The level of Si modification presented in ESV melt treated test samples cast in the steel die cast moulds is higher than any chemical, thermal or stirring treated melt reported in the literature for Al-Si hypereutectic alloys.

5.9.1. Prototype Pistons and Cylinder Liners

The casting of pistons and cylinder liners using untreated melts required pouring directly from the superheat temperatures (see Figure 5.9) in order to fill the moulds. These reference (untreated) prototype pistons and cylinder liners exhibited shrinkage porosity due to the poor feedability and fluidity of the melt. Figures 5.67 to 5.69 show pictures of an industrially cast piston from Essex Aluminum Plant and a prototype piston and cylinder liners cast with the ESV technology.

Fluidity measurements using the spiral pattern were performed for the 390.1(I) alloy at different temperatures with reference and ESV treated melts. The spirals were cast in sand cores prepared using the Cosworth process technology at NEMAK Engineering Centre (Chapter 4). Figure 5.73 presents the top views of representative spirals that show the increased fluidity of the ESV treated melt when compared to the reference melt. Figure 5.74 presents fluidity test patterns, which illustrate considerably higher fluidity of the ESV melt treated. This test illustrates that the ESV treated melts have a superior fluidity and therefore the superheating temperatures of the melt can be reduced to a few degrees above the liquidus temperature of the respective alloy.

Another advantage of the ESV treated melt is the increased feedability. Comparing Figures 5.67a and 5.67b it can be observed that the prototype piston was designed with a considerable smaller riser (~300%) in comparison with the Essex Aluminum Plant piston. The piston cast with the reference melt exhibits deep misruns that were not present in the prototype pistons cast using the ESV treated melt. A similar effect can be observed in Figure 5.72 for the cylinder liners cast using the reference and ESV treated melt. The lower pouring temperature and the riser’s reduction allow considerable savings of energy and an improved yield. Therefore, this
can be translated into cost savings and improved productivity. These advantages could be used for potential justification and to replace cast iron with Al-Si hypereutectic cylinder liners.

The image analysis results to determine the respective SiML of the prototype pistons and cylinder liners are presented in Table 5.17. The SiML was assessed by conducting the analysis of the SiML in 50 fields using three prototype pistons and three prototype cylinder liners. Table 5.17 shows the SiML, the standard deviation and the homogeneity coefficient for the test samples cast using the reference or ESV treated melt ($I_{ACDC} = 100 \text{ A, } f = 100 \text{ Hz, } T_{ESV} = 610 \text{ °C for 3 min}$) for the 390.1(I) alloy, $T_{ESV} = 750 \text{ °C for 6 min (for the 393.2(I) alloy)}. The SiML for the prototype cylinder liners was lower by ~ 15% than for the pistons. The standard deviation of the SiML was used to determine the HC of all the tested samples because this test sample is the most homogeneous one. Therefore, the HC of the prototype piston is equal to zero (0).

![Figure 5.70. Pictures of the a) industrially cast piston at Essex Aluminum Plant (poured at 750°C) and a b) prototype piston cast with the tested technology (poured at 610°C). Note: the considerably smaller riser in the prototype piston (390.1(I) alloy).](image)

It is useful to compare the results from this Section with the ones presented in Section 5.7, Figures 5.51 and 5.52, which correspond to the SiML conducted in the Essex Aluminum Plant cast pistons. It is important to note that the melt at Essex Aluminum Plant was chemically modified using 60 ppm of P by adding the Cu-8 wt% P master alloy.

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Figure 5.71. Top view of the prototype piston cast with the 390.1(I) alloy a) reference melt (pouring temperature 750°C), and b) ESV melt treated ($I_{AC/DC} = 100$ A, $f = 100$ Hz, $t = 3$ min, and a $T_{ESV \ & \ pouring} = 610 \pm 2°C$). Note: the misruns occurs due to the poor feedability of the untreated melt.

Figure 5.72. Top and front views of the prototype cylinder liners cast with the 390.1(I) alloy (a, b) reference melt (pouring temperature 750°C), and (c, d) ESV melt treated ($I_{AC/DC} = 100$ A, $f = 100$ Hz, $t = 3$ min, and a $T_{ESV} = 610 \pm 2°C$).
Figure 5.73. Top view of the spirals of the 390.1 alloy vast with a) reference, and b) ESV melt treated ($I_{AC/DC} = 100 \, A, f = 100 \, Hz$ and $t = 3 \, min$). In both cases the pouring temperature was 750°C. Note: the difference in the spiral length is ~40% (22.5 cm).

Figure 5.74. Fluidity results for the 390.1(1) alloy measurements using reference and ESV melt treatment melts ($I_{AC/DC} = 100 \, A, f = 100 \, Hz, t = 3 \, min$). Note: the increased fluidity of the ESV treated melt; at 610°C it is higher than the fluidity for the reference one.
Table 5.17. Results of the SiML for the prototype pistons and cylinder liners cast with 390.1(1) alloy reference and ESV treated melt \((I_{ACD}=100A, f=100Hz, t=3\) min, and \(T_{ESV}=610\)).

<table>
<thead>
<tr>
<th>Component/Treatment</th>
<th>SiML</th>
<th>Standard Deviation</th>
<th>Homogeneity Coeff. (HC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Piston Reference</td>
<td>5.06</td>
<td>1.87</td>
<td>5.2</td>
</tr>
<tr>
<td>ESV Melt Treated</td>
<td>7.56</td>
<td>0.76</td>
<td>0.0</td>
</tr>
<tr>
<td>Cylinder Reference</td>
<td>5.85</td>
<td>1.55</td>
<td>3.3</td>
</tr>
<tr>
<td>ESV Treated Melt</td>
<td>7.66</td>
<td>0.75</td>
<td>0.0</td>
</tr>
</tbody>
</table>

5.10. Vickers Microhardness and Brinell Hardness of the Selected Test Samples

The mechanical and physical characteristics of the currently used materials for pistons and cylinder liners were provided by KUS and by the Ford Motor Company (Table 5.18).

Table 5.18. Mechanical and physical characteristics of the 390.1(1) alloy for pistons and cast iron cylinder liners as specified by KUS and by the Ford Motor Company \([209 - 213]\).

<table>
<thead>
<tr>
<th>Properties</th>
<th>KUS</th>
<th>Ford Motor Company</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Piston</td>
<td>Cylinder Liner</td>
</tr>
<tr>
<td>Density (g/cm(^3))</td>
<td>2.77</td>
<td>2.73</td>
</tr>
<tr>
<td>Thermal Expansion Coefficient ((10^6 K^{-1}))</td>
<td>19</td>
<td>19</td>
</tr>
<tr>
<td>Thermal Conductivity (W m(^{-1}) K(^{-1}))</td>
<td>130</td>
<td>130</td>
</tr>
<tr>
<td>Ultimate Tensile Strength (MPa)</td>
<td>320</td>
<td>&gt; 180</td>
</tr>
<tr>
<td>Yield Strength (MPa)</td>
<td>300</td>
<td>&gt; 180</td>
</tr>
<tr>
<td>Hardness (HB)</td>
<td>87</td>
<td>90-130</td>
</tr>
</tbody>
</table>

* Data not available in Ford’s Materials Specifications, thus it was taken from the ASM Metals Handbook [212].

The Materials Specification provided by KUS and Ford did not present values for Metal Matrix Microhardness for pistons or cylinder liners. However, in this dissertation Vickers microhardness measurements were performed to prove that ESV melt treatments most likely have an effect on the distribution of the Mg, Ni and Cu enriched phases within the matrix. During the solution heat treatment Ni and Cu are dissolved more easily (faster) within the matrix resulting in a higher hardness.
5.10.1. Results of Vickers Microhardness

The microhardness measurements (Vickers ($\mu$HV$_{25}$)) were carried out based on the ASTM E92-82 standard [215]. This analysis was performed on selected test samples only that were cast using the reference melt and the ESV treated melt at different frequencies, temperatures and times. The selected ESV melt treated test samples for this analysis were solidified using the chill plate and under natural heat exchange conditions. Table A2.14 and Table A2.15 in Appendix II present the results of microhardness and standard deviation and 95% and 99% confidence limits.

The measured microhardness of the ESV melt treated test samples for the 390.1(I) alloy increases as a function of the frequency and decreases as the temperature of the treatment increases. However, the current (AC/DC) applied for the ESV treatments has a negligible effect on the microhardness. The microhardness was conducted on (see Appendix II Table A2.14 and Table A2.15) the test samples that were forced to solidify directionally (heat released in the radial and horizontal directions). The standard deviation of the test samples was in all cases very consistent and was kept below ±10 $\mu$HV, which was the result of the highly homogeneous ESV treated test samples structure. Figure 5.75 presents the results of microhardness of the reference and ESV melt treated test samples for the 390.1(I) Alloy.

Eqs. 5.20 and 5.21 were determined based on the results of the microhardness measurements of the ESV melt treated test samples for the 390.1(I) alloy using different frequencies. Eq. 5.20 corresponds to the test samples solidified on the Cu plate (cooling rate = 0.09°C/s) and Eq. 5.21 for the ones solidified under natural heat exchange conditions (cooling rate = 0.08°C/s).

$$
\mu HV_{\text{Cu Plate (Air Quenched)}} = 0.1416(f_{Hz}) + 81.478 \quad R^2 = 0.99 \quad \text{Eq. 5.20}
$$

$$
\mu HV_{\text{Natural Heat Exchange Cond.}} = 0.1219(f_{Hz}) + 83.572 \quad R^2 = 0.98 \quad \text{Eq. 5.21}
$$

Where $f_{Hz}$ is the frequency of the ESV melt treatments and is expressed in Hz. Both Eqs. are limited to ESV melt treatments using an $I_{AC/DC}$ = 100 A $t$ = 3 min, and a $T_{ESV}$ = 610 ±2°C.
Because Eqs. 5.20 and 5.21 correspond to ESV melt treated test samples under similar conditions, except for solidification, they were combined into a single equation (Eq. 5.22). The microhardness results for the ESV melt treated test samples are shown in Figure 5.75 including the 95% confidence limit and the predicted microhardness using Eq. 5.22. It is clear from Figure 5.75 that Eq. 5.22 can be used as a mathematical model to predict the metal matrix microhardness of the ESV melt treated test samples for the 390.1(I) alloy using different frequencies.

\[ \mu HV_{Low \text{ Cooling Rate}} = 0.1318(f_{Rc}) + 82.525 \]  

Eq. 5.22

The microhardness increments are in perfect accordance with the rise in SiML and in both cases were linear. Therefore, Eq. 5.20 and Eq. 5.21 can be combined with Eq. 5.12 to assess either the microhardness or the SiML as a function of the other. The respective equations are as follows:

\[ \mu HV_{Low \text{ Cooling Rate}} = 6.4497(SiML) + 55.3628 \]  

Eq. 5.23

\[ SiML = \frac{\mu HV_{Low \text{ Cooling Rate}} - 55.3628}{6.4497} \]  

Eq. 5.24

The SiML results presented in Section 5.7 and the microhardness clearly show that microstructure and mechanical properties of test samples cast with ESV treated melt are less sensible to low cooling rate (from 0.08 to 0.0.9°C/s). Comparing Eqs. 5.5 and 5.6 with Eqs. 5.20 and 5.21 respectively, it can be concluded that the differences in SiML and microhardness for for the abovementioned cooling rates are negligible. This is probably due to the fact that the highly refined Si reduces its segregation and more homogeneous microstructures are formed. Section 5.7.2 and Table A2.8 (Appendix II) shows that as the SiML increases, the macrosegregation decreases. This assumption is the result of the determination of the settling velocity of Si particles in a solidifying test sample using the Stoke’s law (Eq. 5.4). The respective values for SiML and the settling velocities of the Si particles can be consulted in Table A2.4 to Table A2.15 (Appendix II).
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5.10.2. Effect of Heat Treatment (T6) on Microhardness

Reference and ESV melt treated test samples for the 390.1(I) alloy were heat treated following the T6 heat treatment conditions using different solution treatment times. All test samples were artificially aged at 250°C for 5 hrs as indicated in Table 4.8. The ESV melt treatment conditions of the selected test sample were \( I_{ACDC} = 100 \text{ A}, f = 100 \text{ Hz}, t = 3 \text{ min}, T_{ESV} = 610 \pm 2°C \). Figure 5.76 presents the histogram of the microhardness for the heat treated test samples as a function of the solution treatment time. The solution treatment was conducted at a temperature of 500 ± 5°C for times between 30 and 240 min. After the solution treatment, the test samples were water quenched (to room temperature) and then artificially aged at a temperature of 250 ± 5°C for five hours. The temperatures of the heat treatments were the same as the ones used at NEMAK at Windsor Aluminum Plant or NEMAK Engineering Centre (data available in Chapter 4). The selected heat treatment conditions were the same as for the V6 engine blocks with cast in cast iron and prototype cylinder liners.

Figure 5.75. Metal matrix microhardness (Vickers) for the 390.1(1) alloy ESV melt treated at different frequencies and the predicted microhardness using Eq. 5.22. Note: high accuracy of Eq. 5.22 to predict microhardness for the Al-Si hypereutectic alloys.
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Figure 5.76. Effect of heat treatment on microhardness of test samples cast using reference and ESV treated melts ($I_{ac/dc} = 100$ A, $f = 100$ Hz, $t = 3$ min, and $a T_{ESV} = 610 \pm 2^\circ$C, cooling rate = $0.09 ^\circ$C/s). Note: significantly higher microhardness of the ESV treated samples.

Figure 5.76 shows that the microhardness of the ESV melt treated test sample increases with the solution treatment time. The ESV melt treated test samples exhibited a higher microhardness after casting and throughout the different solution treatments. For instance, the microhardness of the ESV melt treated test samples in as cast conditions was ~19% higher, and ~35% after 240 min of solution treatment. The reference test samples show an increase in microhardness as a function of the solution treatment time up to 120 min, then after (240 min) the value of microhardness slightly decreases.

The increased microhardness could be an effect of the higher homogeneity obtained on the ESV melt treated test samples. It is, the better distribution of the Ni and Cu enriched phases permit their easier dissolution within the matrix resulting in improved mechanical properties. However, due to the finely dispersed Ni and Cu enriched phases within the $\alpha$-Al matrix and the limitations of the EDX probe (due to the volume of interaction of the electron beam) it was not possible to quantify the amount of dissolved Ni and Cu in the matrix. It is proposed, for future work that in order to quantify the amount of dissolved Ni and Cu in the $\alpha$-Al matrix, if any, a characterization using transmission electron microscopy and X-ray diffraction needs to be conducted.
5.10.3. Results of Brinell Hardness

Brinell hardness measurements were conducted on ESV melt treated test samples, prototype pistons and cylinder liners for the 390.1(I) and 393.2(I) alloys respectively. The test samples solidified in the ESV standard cup were melt treated using different frequencies and a $I_{AC/DC} = 100 \text{ A}$, $T_{ESV} = 610 \pm 2^\circ C$, $t = 3 \text{ min}$ and a cooling rate $= 0.08 - 0.09^\circ C/s$. The melt to cast the prototype pistons and cylinder liners was ESV treated under the same conditions using a $f = 100 \text{ Hz}$ and for the 393.2(I) alloy at $t = 6 \text{ min}$, $T_{ESV} = 750^\circ C$ a cooling rate of 1.3 and 1.6°C/s for the prototype pistons and cylinder liners respectively. It is important to mention that all the test samples tested for micro or macrohardness were cast using degassed melt. Additionally, industrially cast pistons from Essex Aluminum Plant and KUS were also tested as-cast and artificially aged conditions (see Table 5.19 and Figure 5.78). The artificial aging was performed at the University of Windsor following Ford’s Materials Specifications and the KUS standard procedures (250°C for 5 h) [208, 211]. Brinell measurements were carried out at the NEMAK Engineering Centre using a load of 500 Kg (ASTM standard 10/ISO6506) [214].

Table 5.19. Brinell hardness of the die-cast automotive components (industrial and prototype pistons and cylinder liners) for the 390.1(I) and 393.2(I) alloys under as-cast and heat treatment conditions. Prototype pistons and cylinder liners were cast with reference and ESV treated melts under optimum conditions.

<table>
<thead>
<tr>
<th>Reference Melt</th>
<th>ESV Treated Melt</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Industrially Cast Piston (Essex Aluminum Plant)</strong></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>107 ± 3</td>
</tr>
<tr>
<td>Artificially Aged*</td>
<td>102± 5</td>
</tr>
<tr>
<td><strong>Industrially Cast Piston (KUS)</strong></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>122 ± 6</td>
</tr>
<tr>
<td>Artificially Aged*</td>
<td>119 ± 1</td>
</tr>
<tr>
<td><strong>Prototype Piston</strong></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>120 ± 4</td>
</tr>
<tr>
<td>Artificially Aged</td>
<td>117 ± 6</td>
</tr>
<tr>
<td><strong>Prototype Cylinder Liner (Different Alloys)</strong></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>390.1(I)</td>
</tr>
<tr>
<td></td>
<td>134 ± 3</td>
</tr>
<tr>
<td>Solution Treated and Artificially Aged (T6)</td>
<td>157 ± 4</td>
</tr>
</tbody>
</table>

* The industrially cast pistons were provided in as-cast conditions and were artificially aged at the University of Windsor using the KUS and Ford Motor Company Standard Procedures.
Eqs. 5.25 and 5.26 were determined with the regressions for the Brinell hardness of ESV melt treated test samples using different frequencies for the 390.1(I) alloy. The test samples were solidified on the Cu plate and under natural heat exchange conditions for the respective alloys. The accuracy of Eqs. 5.25 and 5.26 was determined by comparing them with the actual Brinell hardness results.

\[
HB_{\text{Cu Plate (CR = 0.09°C/s)}} = 0.3129(f_{Hz}) + 110.21 \quad R^2 = 0.93 \quad \text{Eq. 5.25}
\]

\[
HB_{\text{NHEC (CR = 0.08°C/s)}} = 0.336(f_{Hz}) + 105.12 \quad R^2 = 0.94 \quad \text{Eq. 5.26}
\]

Eqs. 5.25 and 5.26 were combined into a single equation (Eq. 5.27) for the prediction of the Brinell hardness for ESV melt treated test samples. This is a further argument to conclude that test samples cast with ESV treated melt are less sensible to cooling rate. Figure 5.77 shows the results of the Brinell hardness for the ESV melt treated test samples using different frequencies.

![Figure 5.77](image)

Figure 5.77. Brinell macrohardness for the 390.1(I) alloy ESV melt treated at different frequencies and solidified on the Cu plate and under natural heat exchange conditions (cooling rate = 0.09 and 0.08°C/s respectively). Also the predicted data obtained using Eq. 5.27 is presented. Note: high accuracy of Eq. 5.27 to predict brinell macrohardness for the ESV melt treated test samples solidified at a low cooling rate. Also, it is evident that ESV treated melts have low sensibility to low cooling rate.
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\[ H_{B_{\text{Low Cooling Rate}}} = 0.3243 \left( f_{Hz} \right) + 107.67 \quad \text{Eq. 5.27} \]

By combining Eq. 5.12 and Eq. 5.27, the assessment of the corresponding Brinell Hardness can be predicted as a function of the SiML for the ESV melt treated test samples (Eq. 5.28). The opposite procedure (prediction of the SiML based on hardness) can be assessed using Eq. 5.29.

\[ H_B = 17.01 \left( \text{SiML} \right) + 31.67 \quad \text{Eq. 5.28} \]

\[ \text{SiML} = 0.0583 \left( H_B \right) - 1.8622 \quad \text{Eq. 5.29} \]

The statistical evaluation of both equations resulted in an error of less than 7.5%. Therefore, either equation can be used as a valid algorithm to determine the Brinell hardness of the ESV melt treated test samples for the 390.1(I) alloy as a function of its SiML and vice versa.

Figure 5.78 shows the effect on Brinell hardness as a function of the ESV melt treatment times. The results presented in Figure 5.78 correspond to the 390.1(I) alloy test samples solidified in the Cu Plate (cooling rate = 0.09°C/s). The Brinell hardness results are also in accordance with the SiML where the effect of the ESV melt treatment time is logarithmic (compare Figures 5.43 and 5.55 with Figure 5.75). The prediction of the Brinell hardness as a function of the ESV time for the 390.1(I) alloy can be assessed using Eq. 5.30.

\[ H_{B_{\text{Cu Plate (CR = 0.09°C/s)}}} = 12.95 \ln \left( t \right) + 73.431 \quad R^2 = 0.93 \quad \text{Eq. 5.30} \]

Combining Eq. 5.12 and Eq. 5.29 the SiML and Brinell hardness for test samples solidified on the Cu plate can be determined (Eq. 5.31 and Eq. 5.32).

\[ \text{SiML}_{390.1(I) \text{ Alloy @ CR = 0.09°C/s}} = 0.0647 \left( H_B \right) - 2.998 \quad \text{Eq. 5.31} \]

\[ H_{B_{390.1(I) \text{ Alloy @ CR = 0.09°C/s}}} = 15.461 \left( \text{SiML} \right) + 46.35 \quad \text{Eq. 5.32} \]

The margin of error for Eq. 5.31 and Eq. 5.32 was < 3%.
Eqs. 5.31 and 5.32 are of great importance because the hardness (Vickers or Brinell) can be determined as a function of the SiML and/or vice versa. Unfortunately, the validation of these algorithms was not conducted in the present research for test samples chemically or thermally modified. However, using the results of Table 5.13 and Eq. 5.23, Eq. 5.29 and Eq. 5.32 the error obtained was < 7%; the validation of these equations could be a topic for future work.

By combining Eqs. 5.30 and 5.31 with Eq. 5.9 the Vickers microhardness and Brinell hardness can be determined using the thermal analysis data, as shown in Eqs. 5.33 and 5.34. Using the data from Table A2.1 - Table A2.3 the respective values of liquidus temperatures for the selected test samples were used to determine their micro and macrohardness. The statistical analysis of the predicted liquidus showed that Eqs. 5.33 and 5.34 have a 95% accuracy.

\[
\mu HV_{\text{Low Cooling Rate 390.1(l)}} = 1.4944(T_{LQ}) - 812.2508 \quad \text{Eq. 5.33}
\]

\[
HB_{\text{Low Cooling Rate 390.1(l)}} = 3.5811(T_{LQ}) - 2032.7975 \quad \text{Eq. 5.34}
\]
Application of Eqs. 5.33 and 5.34 in UMSA or AITAP software should be considered in any future work and implemented in the ESV workstation (ETCT) software for its commercialization. These algorithms could be the key to on-line prediction of the mechanical properties for ESV treated melts. It is also important to mention that the algorithms developed during the course of this dissertation are valid for the present results, however, in future they can be modified for other applications.

Table 5.20. Results of the Brinell hardness measurements for test samples cast using reference and ESV treated ($I_{ACDC} = 100\, \text{A}, f = 100\, \text{Hz}, t = 3\, \text{min}, \text{and} a T_{ESV} = 610 \pm 2^\circ\text{C}$) melts. Test samples solidified under different heat exchange conditions.

<table>
<thead>
<tr>
<th>ESV Melt Treatment Conditions</th>
<th>Reference</th>
<th>$f = 40, \text{Hz}$</th>
<th>$f = 60, \text{Hz}$</th>
<th>$f = 80, \text{Hz}$</th>
<th>$f = 100, \text{Hz}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test Samples Solidified in the Cu Plate (Air Quenched)</td>
<td>Brinell Hardness (HB)</td>
<td>113 ± 5.2</td>
<td>111 ± 2.9</td>
<td>124 ± 6.8</td>
<td>123 ± 6.7</td>
</tr>
<tr>
<td>Test Samples Solidified Under Natural Heat Exchange Conditions</td>
<td>Brinell Hardness (HB)</td>
<td>108 ± 4.7</td>
<td>109 ± 5.0</td>
<td>N/A</td>
<td>136 ± 6.2</td>
</tr>
</tbody>
</table>

* The Brinell hardness measurements were measured in the centre of the test sample and were solidified in the ESV Standard Cup, in the vicinity of the thermocouple.

5.10.4. Multiple Regression Analysis for Micro and Macrohardness

An evaluation of the statistically designed experiments was conducted to determine a single equation capable of assessing the microhardness and macrohardness based on the ESV melt treatment conditions (Eqs. 5.35 and 5.36). The correlation analysis of the independent variables and their contribution to microhardness and macrohardness was performed (Table A2.14). The multiple regression analysis was conducted using Microsoft Excel. The parameters with the strongest effect are time, cooling rate and chemical composition on the ESV melt treated test samples, but this only applies for test samples solidified at high cooling rates. This also corresponds to the SiML results. Therefore, the modification of Si has an effect on the mechanical properties of the Al-Si hypereutectic alloys.
\[ \mu HV = 433.72857 - 0.075095(I_{AC/DC}) + 0.147857(f_H) \\
- 0.184444(t) - 0.525238(T_{ESV}) - 113.333(CR) \quad R^2 = 92.3 \quad \text{Eq. 5.35} \]

\[ HB = 296.073 - 0.1258121(I_{AC/DC}) + 0.3026606(f_H) \\
+ 0.0791449(t) - 0.524797(T_{ESV}) + 16.23971(CR) \quad R^2 = 88.8 \quad \text{Eq. 5.36} \]

Eqs. 5.35 and 5.36 are only applicable for the results of this particular dissertation and specifically for the ESV workstation using the dual coil [6]. However, both equations can be expanded for more general applications by changing the electrical current \( I_{AC/DC} \) parameter for the strength of the magnetic field, geometry of the electromagnetic coils, the deep of penetration of the AC current and by determining the pressure or Lorentz forces \( F_L \). A similar approach can also be followed with Eqs. 5.13 and 5.14. This will also be considered as a topic for the future work.

### 5.11. Integrity of the Tensile Bars and Prototype V6 Engine Blocks

#### 5.11.1. Introduction

Assessment of the mechanical characteristics of the V6 engine blocks using cast iron or Al-Si cylinder liners was discussed. All engine blocks were cast using the low pressure Precision Sand Casting Cosworth Process. The main goal was to measure the engine blocks characteristics and then compare them against the Ford Engineering Materials Specification WSE-M2A151-A3 [216]. The substitution of cast iron with Al-Si cylinder liners and the elimination of the “Tocco” for the Al-Si hypereutectic cylinder liners are novel ideas that were discussed in this dissertation. The cast in prototype Al-Si cylinder liners were made with 390.1(I) and 393.2(I) alloys using reference and ESV treated melts. The ESV melt treatment conditions to cast the cylinder liners were \( I_{AC/DC} = 100 \, \text{A}, f = 100 \, \text{Hz}, \) for the 390.1(I) alloy \( T_{ESV} = 610 \pm 2^\circ\text{C} \), \( t = 3 \, \text{min} \) and for the 393.2(I) alloy \( T_{ESV} = 750 \pm 2^\circ\text{C} \), \( t = 6 \, \text{min} \).

The tensile characteristics of the prototype pistons and cylinder liners were measured by casting tensile test bars into a Stahl mould using reference and ESV treated melts [173]. The tensile test bars were cast using the 390.1(I) and 393.2(I) alloys with reference and ESV treated melt. The ESV melt treatments were conducted at different temperatures. The tensile test bars
were tested in as cast, artificially aged and heat treated conditions in batches of five bars per condition. The machining of the respective tensile test bars was performed after the heat treatment by the Technical Support Centre. The results of the tensile properties were compared with the Ford Materials Specifications for pistons and cylinder liners [209, 210].

5.11.2. Assessment of Porosity in the Tensile Test Bars and Bulkhead Sections of the W319 V6 Engine Block Using the ASTM E 155 Standard

The porosity was measured using X-ray radiography and the microstructure characteristics of the bulkhead sections of the V6 engine blocks were conducted at NEMAK Engineering Centre. Both methods were used to assess the gas and shrinkage porosity of the bulkheads for regular production and prototype V6 engine blocks. Radiographic results are presented since this is the quality control method used at Windsor Aluminum Plant and at NEMAK Engineering Centre for assessment of the bulkhead soundness. The metallographic analysis of the bulkhead sections was used since fine pores are not detected by X-ray radiography. Table 5.21 presents the results of radiographic analysis of the bulkhead sections of a regular, prototype engine block and for selected tensile test bars. Figure 5.79 shows an example of a radiograph of a bulkhead sections where the metallographic determination of porosity was made. The dashed lines in Figure 5.79 present the section where the porosity measurements were conducted using metallographic techniques.

The level of porosity in the regular production engines was considerably high in comparison to the prototype engine blocks (Figure 5.80). This reduction in porosity is the effect created by the absence of the “tocco”, which resulted in a higher cooling rate of the bulkhead section and the better feedability. The porosity in both sides of the production engine is comparable, while in the prototype engines lower porosity is evident in the left side (cast iron cylinder liners). In most cases bulkhead 2 and 3 had the higher porosity, which applies for both, regular production and prototype engines. The slightly lower cooling rate in the left side of the engine block probably enhanced or promoted the interdendritic feeding during solidification, hence lower porosity. Figure 5.80 shows the average area porosity (%) of the V6 regular production and prototype engine blocks (Table A2.16 Appendix IV). R. I. MacKay [169] proved ~ 70% reduction in porosity resulted in ~ 40% improvement in the high cycle fatigue performance.
Table 5.21. The radiographic results of the bulkhead sections of the V6 engines and tensile test bars.
Refer to Chapter 4 for the layout of the radiographs for the V6 engine blocks.

<table>
<thead>
<tr>
<th>Radiographed Component</th>
<th>Bulkhead Section of the Engine Block</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regular Production Engine Block</td>
<td></td>
<td>P 1,2</td>
<td>P1</td>
<td>P1</td>
<td>P1</td>
</tr>
<tr>
<td>Prototype Engine Block (ESV Treat. Al-Si Cylinder Liner)</td>
<td></td>
<td>P1</td>
<td>P1</td>
<td>P1</td>
<td>P1</td>
</tr>
</tbody>
</table>

Selected Tensile Test Bar(s)

<table>
<thead>
<tr>
<th>390.1(i) Alloy</th>
<th>As-Cast</th>
<th>Artificially Aged</th>
<th>Heat Treated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>G5</td>
<td>S3</td>
<td>S4</td>
</tr>
<tr>
<td>ESV Treated @ 610°C</td>
<td>S3</td>
<td>S3</td>
<td>S4</td>
</tr>
<tr>
<td>ESV Treated @ 640°C</td>
<td>S4,5</td>
<td>S3,4</td>
<td>S1,4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>393.2(i) Alloy</th>
<th>As-Cast</th>
<th>Artificially Aged</th>
<th>Heat Treated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>G1</td>
<td>S3</td>
<td>S3</td>
</tr>
<tr>
<td>ESV Treated @ 750°C</td>
<td>S2</td>
<td>S2,4</td>
<td>S3</td>
</tr>
</tbody>
</table>

G: Gas Level 1 through 7 (Beyond 4 is critical)
P: Porosity Level 1 through 7 (Beyond 4 is critical)
S: Shrinkage Level 1 through 7 (Beyond 4 is critical)

Note:
G signifies pores that are oval to circular in shape and originate from either gas pore ($H_2$), which nucleate and growth fairly early on during solidification or is the result of a pocket of air, which becomes encapsulated by an oxide skin during turbulent filling.
P signifies highly dispersed porosity, which results from the related feeding conditions during solidification.
S signifies localized feeding issues, which may have resulted from a hot spot condition or latent heat generated hot spot.

Figure 5.79. Radiograph of a representative bulkhead section of a V6 engine block. The dashed lines show the respective areas where the porosity and SDAS analysis was carried out. Note low porosity in the bulkhead sections.
Figure 5.80. Results of the assessment of porosity in the bulkhead sections of the V6 regular production and prototype and engine blocks. Ref. indicates that the prototype cylinder liner was cast with the reference and ESV for prototype cylinder liners cast using ESV treated melt. Note: low porosity in the prototype engine blocks, in comparison to a regular production engine block.

5.11.3. Assessment of the Secondary Dendrite Arm Spacing (SDAS) in the Bulkhead Sections of the V6 Engine Blocks

The SDAS measurements were conducted on the bulkhead sections of all tested engine blocks in the segments shown in Figure 5.81. The Ford Engineering Materials Specification WSE-M2A151-A3 indicates that the average value of SDAS should be 50 μm in critical areas and its average in any area should not exceed 60 μm [216]. The image analysis results indicate that the SDAS in the prototype engine is slightly smaller; however, as seen in Figure 5.81, the average value of SDAS in all cases exceeds the specified value. The SDAS in all engine blocks (Figure 5.81) was similar, which creates an apparent contradiction between porosity and SDAS; because if the porosity was reduced by the chilling effect of the cylinder liners, the SDAS is supposed to present a similar effect. However, this could be a consequence of using a combined cast iron (preheated) and prototype Al-Si hypereutectic cylinder liner. For the future work it is proposed
that prototype engine blocks be cast using only Al-Si cylinder liners to conduct the high cycle fatigue tests.

5.11.4. Results of the Monotonic Mechanical Testing of the Cast Tensile Test Bars and V6 Engine Blocks

A total of seventy five (75) test bars were cast in reference and ESV melt treatment conditions using the 390.1(I) and 393.2(I) alloys. Sets of five tensile test bars were tested in as cast, artificially aged and heat treated conditions for a total of fifteen for every casting condition. The results of the yield stress (0.2% offset), the ultimate tensile strength, the modulus of elasticity, and the elastic and total elongation are shown in Table A2.17 and Table A2.18 (Appendix II) for the tensile test bars and the V6 engine blocks respectively. The conducted heat treatment for both the tensile test bars and the engines was performed following the TSR operation. The artificial aging was conducted following the Ford’s Materials Specification for pistons [208].

Figures 5.79 to 5.81 show the considerable improvements in mechanical properties between the tensile test bars cast with reference and ESV treated melts. The ESV melt treatment was performed using an $I_{AC/DC} = 100 \text{ A}, f = 100 \text{ Hz}, t = 3 \text{ min}, T_{ESV} = 610$ or $650 \pm 2^\circ\text{C}$ for the 390.1(I) alloy and for the 393.2(I) alloy it was treated for $t = 6 \text{ min}$ and $T_{ESV} = 750 \pm 2^\circ\text{C}$. From the above-mentioned figures it can be observed that the main improvements were in tensile strength and UTS. Also, the elongation of the 390.1(I) alloys was improved, however, the effect of the ESV melt treatment for the 393.2(I) alloy is unclear. Presumably, the unclear effect of the ESV melt treatment for the 393.2(I) alloy could be the reason of the inconsistency in soundness of the test bars by using the Stahl mould (Table 5.21). The test bars cast with melt ESV treated melt at $T_{ESV} = 610 \pm 2^\circ\text{C}$ exhibited the highest mechanical properties including elongation ($0.6 \pm 0.04 \Delta L/L$), in fact, the 390.1(I) alloys exhibited higher mechanical properties compared with the 393.2(I) alloy.
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Table 5.21 shows that the porosity of the tensile test bars is considerably different for all the experimental conditions. This is a consequence of the Stahl mould that introduces considerable bias, which is a common problem reported by the industry. Therefore, it is proposed that for future work the tensile test bars be extracted directly from the test samples cast in the standard ESV cup that were solidified directionally (Cu plate); using the same procedure as X. Chen [168].
Figure 5.82. Summary of the results of the monotonic tensile tests conducted on the test bars for the 390.1(l) alloy cast into the Stahl mould reference and ESV treated melts in as-cast and heat treated conditions. Note: the increment in tensile and ultimate tensile strengths.

![Graph showing tensile test results](image)

Figure 5.83. Summary of the results of the monotonic tensile test conducted on the test bars for the 393.2(l) alloy cast into the Stahl mould using reference and ESV treated melts in as-cast and heat treated conditions.

![Graph showing tensile test results](image)

Figure 5.82 shows the positive effect of the ESV melt treatment on mechanical properties of the 390.1(l) alloy. The improvement in mechanical properties corresponds to a ~20% in as-
cast and ~30% in heat treated conditions between the reference and the ESV melt treated test samples at a $T_{ESV} = 610 \pm 2^\circ$C. Figure 5.84 presents (Table A2.17 Appendix II) the results of the tensile test of the reference and ESV melt treated test samples of the 393.2(I) alloy. This particular alloy also presents improvements on tensile and UTS in ESV melt treated condition, however, the improvement (< 10%) in mechanical properties is not as prominent. Therefore, the 390.1(I) alloy has a higher tensile and UTS than the 393.2(I) alloy; this could be more suitable for pistons and cylinder liner applications.

Figure 5.84 shows the results of the tensile properties of the bulkhead sections of the regular production engine block and a representative example of the prototype engine block. The results of all the tested engine blocks are presented in Table A2.18 available in Appendix II. In Figure 5.84 it can be observed that for the regular production engine block only one set of data is provided, which is the average of the tensile strength of all bulkheads for this particular engine block. The regular production engine was provided after the heat treatment and artificial aging and it is for this reason that no data about artificial aging or as cast conditions is provided.

Figure 5.84 clearly shows the advantage of using of Al-Si hypereutectic cylinder liners in the V6 engine block, showing an average increase of the yield strength and UTS of ~ 10% for engine blocks containing only 50% of prototype cylinder liners. Figure 5.85 presents the tensile test results for particular bulkheads from the regular production and prototype engine blocks. The improvement in mechanical properties of the prototype engine block by compared to the regular production is clear from this figure. In future prototype engine blocks should be cast using only Al-Si cylinder liners. This will clarify the effect of Al-Si hypereutectic cylinder liners on mechanical characteristics and soundness of the engine blocks. The difference in mechanical properties among the prototype engines between the different ESV melt treatments and compositions was as expected (see Table A2.18 Appendix II).
Figure 5.84. Summary of the average tensile test results for the bulkheads sections of the regular production and prototype W319 alloy engine blocks in as-cast and heat treated conditions. Note: the mechanical properties of the heat treated regular production engine block are similar to the as-cast prototype engine block.

Since the difference in SDAS is negligible among the bulkhead sections of all engine blocks; the improvement in mechanical properties must be due to other reasons. Figure 5.80 and Table A2.16 (Appendix II) shows the results of porosity for the bulkheads for the regular production and prototype engines; it is evident the lower porosity in the bulkheads of the prototype engine blocks. Therefore, by increasing the number or volume of the pores the applied stresses are amplified at the tips of micro-cracks or voids acting as stress concentrators. The magnitude of this amplification depends on micro-crack orientations, geometry and dimensions [217].

The stress concentration of a gas pore (in this case it is a perfect sphere) is 3 at its interface. Therefore, a gas pore has the capacity to increase the nominal stress locally more than three times and it decreases by increasing the distance away from the pore. The stress fields of nearly the pores create high stress values. So, clusters of pores are very detrimental. The fact that the porosity in the bulkhead sections was considerably reduced in the prototype engine blocks resulted in a rise in the yield strength and UTS (see Figures 5.81 and 5.82). Shrinkage porosity has a more detrimental effect in comparison to gas porosity; this is due to its intricate morphology.
that acts as a more effective stress concentrator. Figure 5.85 clearly shows that by using 50% Al-Si cylinder liners in prototype engine blocks the mechanical properties are considerably better.

![Mechanical Properties of Cast Iron and Al-Si Cylinder Liners](image)

Figure 5.85. Tensile test characteristics of the bulkhead sections of the heat treated V6 a) prototype (with Al-Si cylinder liners) and b) regular production engine blocks. Note: $\sigma_y$ stands for yield stress.

5.11.5. Metallographic Analysis of the Tensile Test Bars and Bulkhead Sections of the V6 Engine Blocks

Figure 5.86 shows SEM/SE fractographs of the 390.1(I) alloy cast with the reference melt in as-cast and heat treated conditions at various magnifications. There is a significant difference between the propagation of dislocations and the distribution of the stresses of Al and Si; for instance, Al is ductile, while Si is brittle. Therefore, the movement of dislocations is restricted to the slip planes of Al and primary Si particles acts as barrier where dislocations pile up (Figure 5.87). The fracture of these test bars occurred in areas with a high concentration of primary Si particles or in the presence of gas or shrinkage pores.
The fracture of brittle particles present in a ductile matrix consists (i.e. eutectic and primary Si in the α-Al matrix) of three stages. The first is the pile up of dislocations along the slip planes of the α-Al matrix, followed by the build up of shear stresses to nucleate a microcrack. Finally, an increase of stress is observed that results in the formation and propagation of a microcrack. The brittle fracture of Si is characterized by an abrupt separation of the crystallographic planes (cleavage), without the evidence of plastic deformation. On the other hand, the α-Al from the matrix that at the temperature of the test is ductile exhibits the typical dimples indicating a considerable degree of plastic deformation [217]. Therefore, the primary Si particles act as stress concentrators that rapidly build up the stresses ending in catastrophic failure. This effect can be clearly observed in Figures 5.83 and 5.84 where the primary Si particles raised the stresses and most likely initiated the fracture of the test bar(s).

The reference test bars for the 390.1(1) alloy contain cavities (gas + shrink pores) with highly dendritic edges (Figure 5.86). Due to the intricate morphology of the dendrites combined with the size of the pore, they become a perfect stress concentrator that easily builds the stresses up enough to act as a crack initiator. The lack in soundness (porosity) of the reference test bars combined with the presence of coarse primary Si are the main reasons for the lower mechanical properties.

The SEM/BSE fracture analysis of the test bars cast with the ESV treated melt for the 390.1(1) alloy is presented in Figure 5.88. The ESV treatment was performed using an $I_{AC/DC} = 100 \text{ A}$, $f = 100 \text{ Hz}$, at $T = 610^\circ \text{C}$ and $t = 3 \text{ min}$. These micrographs show highly refined primary Si particles dispersed in the Al-matrix. Within the entire fracture face no porosity or dendrites were found. Moreover, the matrix surrounding the Si particles present a considerable level of plastic deformation. The higher homogeneity, refinement of the Si particles, the absence of pores and dendrites resulted in the higher yield stress and UTS of these samples.

The 393.2(1) tensile test bars fractured from the reference and ESV treated melts were observed using the SEM/SE technique; the respective micrographs are presented in Figure 5.89. The reference test bars show coarse primary Si particles in the vicinity of the edge of the test bar, the stress concentrated in these particles was high enough that they were most likely the crack initiators. The tensile test bars were cast using the pouring device (shown in Chapter 4) that eliminates the presence of oxide films originating in the treated melt. However, some of these
oxides were found in the reference test samples of the 393.2(I) alloy that could be the result of the turbulent flow created during the pouring of the melt into the Stahl mould.

On the other hand, the ESV melt treated test samples showed considerably smaller primary Si particles, nonetheless, they are still very coarse and are similar in size to the reference tensile bars cast with the 390.1(I) alloy. The ESV treatment was performed using an $I_{AC/DC} = 100\, \text{A}$, $f = 100\, \text{Hz}$, at $T = 750\, ^\circ\text{C}$ and $t = 6\, \text{min}$. The presence of Al, Cu and Ni phases is evident and located in an area where some level of deformation is observed within the matrix. Due to the presence of the relatively coarse primary Si particles the difference in mechanical properties is not as evident when compared to the 390.1(I) reference and ESV treated test bars.

The fractures observed within the tested test bars of the V6 regular production bulkhead sections of the engine block are presented in Figure 5.90. The appearance of plastic deformation was observed, with the presence of Al-Ni-Cu enriched phases that were cracked and reveal a ductile appearance. The Al-Si eutectic particles normal to the application of the stress are observed in Figure 5.90a, these cracked particles have the typical cleavage appearance. Also dendrites were detected among the fractured surface of the tensile test bars due to shrinkage. Using X-Ray radiography this fine porosity was not detected (Table 5.21). Nonetheless, the lowest level of porosity (due to lack of feeding) was assigned during the evaluation using LOM/IA (Figure 5.90). The regions of the dendrite arms that were fractured show a typical fibrous or appearance that is commonly found in ductile fractures containing “dimples” [217].

The fractographs of the bulkheads section of the test bars corresponding to the V6 prototype engine block are presented in Figure 5.91. The morphology of the fracture of these test bars is similar to the one presented for the regular production engine; however, in this case shrinkage pores were not found. This is one of the reasons for the improved mechanical properties in the bulkhead sections of the prototype engine blocks. All prototype engine blocks presented reduced level of porosity in comparison to the regular production engine blocks (Table 5.21 and Table A2.18).
Figure 5.86. SEM/SE micrographs of two 390.1(I) reference tensile test bars cast and presenting the possible location of the crack initiation (a, b and c) concentration of primary Si particles and (d, e and f) shrinkage porosity.
Figure 5.87. Model of microcrack formation at a pile up of edge dislocations around a primary Si particle (barrier) in Al-Si alloys.

Figure 5.88. SEM/SE fractographs of the 390.1(I) test bars cast with the ESV treated melt \(I_{ACDC} = 100\, \text{A}, f = 100\, \text{Hz}, \text{at } T_{ESV} = 610\, \text{°C}\) for 3 min) at various magnifications. Dashed lines and arrows indicate the origin of the crack initiation. Note: observed refined Si particles on this test sample in comparison to the 390.1(I) reference sample.
Figure 5.89. SEM/SE fractographs of the surfaces for the 393.2(1) reference (a, b and c) and (d, e and f) ESV treated test bars. The dashed lines indicate the possible location for the crack initiation. The ESV treatment was performed using an $I_{AC/DC} = 100\, \text{A}, f = 100\, \text{Hz}$, at $T_{ESV} = 750^\circ\text{C}$ and $t = 6\, \text{min}$. 

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Chapter 5. Results and Discussions

Figure 5.90. SEM/SE fractographs of tensile test samples extracted from the bulkhead sections of the W319 V6 regular production engine block, cast at the NEMAK Engineering Centre with cast-in cast iron cylinder liners. Note: interconnected shrinkage porosity and the highly deformed dendrites with striations showing a ductile fracture of the α-Al dendrites with typical dimples (high magnification).
Figure 5.91. SEM/SE fractographs of tensile test samples extracted from the bulkhead sections of the W319 V6 prototype engine block, cast at the NEMAK Engineering Centre using 390.1(l) cylinder liners. Note: the highly deformed α-Al regions showing a ductile fracture with typical dimples (high magnification).

5.12. General Discussions of the Experimental Results

5.12.1. ES and ESV Melt Treatments for the Al-Si Hypo and Hypereutectic Alloys

Using ES or ESV melt treatment methodologies for Al-Si hypo and hypereutectic alloys various degrees of refinement and/or modifications can be assessed in semi-solid and liquid states.
Chapter 5. Results and Discussions

In the following a description of the microstructure and effectiveness of the ES or ESV treated test samples is given.

The ES technology has the capabilities to significantly refine the microstructure of the W319 Al-Si hypoeutectic alloy in the semi-solid state only. The refinement of the microstructure is the result of the shearing forces that breakdown the solidifying α-Al dendrites, which consequently increases significantly the nucleation sites. The microstructure of the ES treated test samples presented partial elimination of the dendritic structure and a grain size reduction of ~600% for the W319 alloy in comparison to the reference test samples. The morphology of the Al-Si eutectic, Mg and Cu enriched phases was not affected by the ES melt treatment. Thermal analysis demonstrated that this technique can be used successfully for the on-line determination of the level of microstructure refinement based on the cooling curve characteristics.

The combined effect of stirring and vibration created a much more powerful technology that was capable to modify the primary and eutectic Si in Al-Si hypereutectic alloys. This treatment is efficient in both semi-solid and liquid states. The vibration effect was the result of the interaction of combined AC and DC electromagnetic fields that were induced into the liquid melt. In order to develop a unique technology for stirring and vibration a novel “Dual” coil was designed under the scope of this project. The dual coil has the capabilities to pass through a combination of direct (DC) and variable (AC) currents in the same conductor. The additional use of the controlled current source [8] was a key instrument in eliminating the detrimental AC component created on the DC coil, when two parallel coils are used for stirring and vibration. The AC current created in the DC coil are the consequence of the Eddy currents characteristic of an AC electromagnetic field. Therefore, the dual coil was used to build an ESV workstation (ETCT) that will be patented.

5.12.2. The SiEQ Methodology

The newly developed SiEQ methodology was used to determine the liquidus temperature and the thermal expansion coefficient of multi-component Al-Si alloys. The determination of the liquidus temperature allowed the elimination of the superheat temperature as an independent variable for the ESV melt treatments. Additionally, the assessment of the thermal expansion
coefficient using the Si$_{eq}$ methodology at various temperatures represents a unique procedure that can be widely used at the industrial level.

5.12.3. The Characterization of the Al-Si Hypereutectic Alloys Using Thermal Analysis and Microscopy

There is a good agreement between the results of characterization of the ESV melt treatments using these three techniques: optical microscopy, SEM/EDX and thermal analysis presented. The identification of the Fe and Pb enriched phases was not possible by thermal analysis because Fe enriched phases nucleate during the Al-Si eutectic reaction and the Pb content is considerably lower, which creates difficulties with its identification. The precipitation of the Fe enriched phases can take place together within more than one phase; therefore, in the first derivative this phase was not identified due to a possible convoluted peak. In addition, the amount of Fe is significantly smaller and this further created difficulties with its proper identification. As a result of the quenching experiments a range of temperatures were proposed where the Fe enriched phase(s) would precipitate.

It is important to mention that the first derivative of the cooling curves of the Al-Si hypereutectic alloys presents a non-monotonic signal in the section prior to the beginning of solidification (temperatures above liquidus). This non-monotonic behaviour can be due to the nucleation and growth of the Si agglomerates (identified as Si$_4$ in the literature). This hypothesis was confirmed while conducting a series of quenching experiments from temperatures as high as 100°C above liquidus. In the microstructure of the quenched test samples frozen Si agglomerates were found with a similar morphology to the primary Si particles. These Si agglomerates grow linearly from the liquid state to the Al-Si eutectic reaction, where the primary Si stops its coarsening and this is the beginning of the nucleation of the Al-Si eutectic reaction. In the presence of stirring and vibration the Si agglomerates were modified.

Thermal analysis was utilized to characterize the Mg, Ni and Cu enriched phases. This characterization was conducted using the baseline method between the Al-Si eutectic reaction and the solidus temperature, in order to determine the apparent fraction solid of the above-mentioned phases. The apparent fraction solid is proportional to the precipitating fraction of the respective phase. Furthermore, the analyzed cooling curves of the ESV melt treated test samples that were
solidified under natural heat exchange conditions presented similar apparent fraction solids in comparison to the reference test sample(s). This can be translated in that the amount of the precipitating phase is similar, however, the level of refinement and homogeneity of the ESV melt treated test samples was considerably superior and increases with the intensity of the ESV melt treatment.

The analyzed microstructures of the reference and ESV melt treated test samples for the Al-Si hypereutectic alloys presented a wide size distribution of Si particles. Therefore, in the present project the development of an unbiased standard that can be used to determine the effectiveness of the ESV melt treatment was proposed. This standard is known as the “Image Analysis Based Si Modification Level” (SiML), which overcomes the limitations of the currently used standards (“AFS Standard” and the “Ford primary Si grade”). Using the SiML the Al-Si eutectic as well as the primary Si particles can be analyzed separately or together.

The results of the characterization of the microstructure using image analysis indicates that the SiML presented a direct relationship between the ESV melt treatment conditions and thermal analysis. Using the results of thermal analysis and the ESV melt treatment conditions several equations were developed to predict the SiML as a function of the frequency of the AC current, time and current. Additionally, using multiple regression analysis the development of two equations to predict the SiML as a function of the independent variables used for the ESV melt treatment were developed. The above-mentioned equations have the potential for implementation in an industrial environment. The independent variables are:

1) AC current frequency (in Hz),
2) Electrical current intensity (AC and DC in A)
3) ESV melt treatment temperature (in °C)
4) Hydrogen level (in mL H2/100 g of Al),
5) Time of the ESV melt treatment
6) Melt Si level and
7) Cooling rate

The statistically designed experiments also determined the effect of each of the above-mentioned independent variables used for the ESV melt treatments. As a result it was determined that time, chemical composition and $T_{ESV}$ have the strongest effect in the modification of Si for test samples solidified at low cooling rates (cooling rate = 0.08°C/s). It is important to mention that cooling rates from 1.3 to 1.6°C/s were associated with higher SiML which resulted in a further improvement in the mechanical properties of the ESV melt treated castings. Therefore, the Al-Si hypereutectic treated melts are ideal for die casting applications.
5.12.4. Novel Applications of the ESV Treated Melts

A series of remelting experiments for the ESV melt treated test samples were conducted using the 390.1(1) alloy using both reference and ESV melt treated test samples. The remelting operation was conducted at 750°C for at least three min. followed by solidification under natural heat exchange conditions. The microstructure of the ESV melt treated test samples exhibited a considerably higher level of modification of the Si particles after two remeltings. This indicates that it is worthwhile to consider the use of the ESV melt treatment technology at the industrial level in the production of ingots or in continuous casting to produce rods, slabs, etc. that need to be further remelted, thixocast or rheocast.

5.12.5. The Thermal Expansion Coefficient and Thermal Stability of Reference and ESV Melt Treated Al-Si Hypereutectic Castings

The thermal expansion coefficient of the Al-Si hypereutectic was measured in cast iron and several Al-Si hypo and hypereutectic alloys in reference and ESV melt treated castings. As a result of this analysis it was found that the cast iron presented a considerably lower thermal expansion coefficient in comparison to the Al-Si hypoeutectic alloys such as 319; currently used for engine block production at NEMAK. The difference in the thermal expansion coefficient (~42%) resulted in the creation of residual stresses in 319 engine blocks cast using cast-in cast iron cylinder liners. The excessive residual stresses prevented further advancement of engine blocks design with cast-in cast iron cylinder liners. In contrast, Al-Si hypereutectic alloys presented almost the same thermal expansion coefficient as the W319 (<6% difference). The eventual implementation of Al-Si hypereutectic alloys could be beneficial in the development of shorter engine blocks or the production of a new generation of higher performance engine blocks using cylinder liners with a larger diameter.

5.12.6. The Effect of the ESV Melt Treatment on the Melt Treatment Characteristics
The ESV treated melts revealed that characteristics such as fluidity and feedability can be considerably improved by using ESV melt treatments for Al-Si hypereutectic alloys. The improved fluidity (~40%) allows the reduction of the casting temperature to the liquidus one, therefore, the required superheat temperature can be considerably reduced for industrial applications. This can result in energy and cost savings, thus improved productivity. The improved feedability (~300%) of the ESV treated melt permitted the production of prototype pistons with considerably small risers and also the elimination of misruns. This can reduce drastically the amount of recycled material due to the remelting of the risers and feeder systems.

5.12.7. Effect of the ESV Melt Treatment on Mechanical Properties

The ESV treated melts showed significant improvements in Vickers metal matrix micro hardness (\( \mu \)HV) and Brinell macro hardness (HB). The higher the SiML the more the improvement in the micro and macro hardness. It was observed that as the frequency of the AC current increases and as the temperature of the treatment is closer to liquidus the micro and macro hardness was improved. The rise in the metal matrix microhardness was \(~19\%\) for castings in the as-cast condition and up to \(~35\%\) after the T6 heat treatment with respect to test samples cast using an untreated melt. Additionally, the increase in Brinell hardness was \(~20\%\) for the as-cast material and \(~25\%\) for the T6 heat treated test samples in comparison to the reference test samples.

ESV melt treated test samples showed improvements in Yield Strength (\( Y_S \)) and Ultimate Tensile Strength (UTS). The tensile test bars were cast using the Stahl mould at temperatures as low as liquidus (610 and 750°C for the 390.1(I) and 393.2(I) alloys respectively). The tensile test was conducted in the test bar in the as-cast and heat-treated conditions for the reference and ESV treated melts. The T6 heat treatment further improved the tensile properties of the ESV melt treated castings. More detailed information is provided below.

The fractography analysis of the tested bars revealed two main reasons for the lower mechanical properties of the reference test samples. The first one was the agglomeration of coarse primary Si particles and the second one was the reduced soundness of the castings (shrinkage and gas porosity). The fractographs of the ESV melt treated test samples for the 390.1(I) alloy showed primary Si and porosity free structures. Therefore, the refinement of the
primary Si particles into eutectic type ones and the improved feedability of the ESV treated melt resulted in castings with considerably higher mechanical properties. These characteristics were considered ideal for a material that will eventually be a substitute for cast iron for cylinder liner applications. Additionally, the mechanical properties of the ESV melt treated 390.1(l) alloy are higher in comparison to the minimum requirements for pistons and cylinder liner applications based on Ford’s Engineering Materials Specification. This makes the Al-Si hypereutectic casting under ESV melt treated conditions suitable for industrial application in pistons and cylinder liners.

5.12.8. The Production of V6 Prototype Engine Blocks Using Al-Si Cylinder Liners

The characterization of the prototype V6 engine blocks using cast-in Al-Si hypereutectic cylinder liners showed the following advantages: the elimination of the preheating operation (tocco) of the cast iron cylinder liners, a slight reduction in SDAS, and a reduction of ~ 40% porosity in the bulkhead sections. The reduced porosity in the bulkhead sections resulted in a ~15% improvement in tensile properties for the prototype engine blocks. The fracture analysis of the test bars extracted from the regular production engine blocks showed interconnected shrinkage pores. Shrinkage pores act as stress concentrators. For instance, a spherical pore has the capacity to sustain the stresses up to three times and as the shape of the pore becomes more intricate (which is the case with the shrinkage pores). The prototype V6 engine blocks presented shrinkage free bulkhead sections that resulted in superior mechanical properties.
CHAPTER 6. CONCLUSIONS

1. An extensive literature review (including papers, patents, books and IRC reports) was conducted and revealed the following:

1.1. Al-Si hypereutectic alloy(s) can be used for the replacement of existing cast-in cast iron cylinder liners used in aluminum engine blocks due to their mechanical and physical characteristics [25, 143, 151, 155 - 160]. The present methods (chemical modification of silicon and semi-solid melt processing) for the necessary structure improvement of the Al-Si cylinder liners and pistons and mechanical characteristics are not sufficient or optimum for their application in the regular production of engine blocks (Sections 2.3 and 2.4) [28 - 73].

1.2. Al-Si hypereutectic liquid melts have atomic structures with short and medium ranges order at temperatures above the liquidus (Section 2.2.1) [117, 121 - 128]. Therefore, the presently known techniques (i.e. melt thermal and ultrasonic treatments) could potentially be considered for in-depth research and development of industrially viable technologies (Section 2.3). However, these treatments have many limitations.

1.3. The Electromagnetic Stirring and Vibration (ESV) treatment of various alloy industrial melts is conducted in the temperature range between the liquidus and the solidus temperature [28, 35, 37, 43, 45, 56 - 60, 65]. To date this treatment does not take advantage of the liquid melt structures. Therefore, feasibility studies were conducted utilizing the ESV treatment for Al-Si liquid melts for cast components like cylinder liners and pistons (Sections 2.2, 2.4).

2. The literature and the present studies revealed the following metallurgical and physical differences between the W319 alloy (engine block’s alloy) and the cast iron cylinder liners as main limitations to reduce or eliminate the bridges between cylinder liners [185 - 187].

2.1. The substitution of cast iron for Al-Si hypereutectic alloys for cylinder liner applications represents a weight neutral replacement, which is most desirable [161 - 167]. At the same time this will allow for several important technological improvements in the metal casting industry resulting in improved productivity.

2.2. Cast iron cylinder liners are not “fused” with the aluminum block, therefore a mechanical interface with a considerable amount of detrimental gaps is formed (Section 3.3 and Figure 3.2) [187]. In addition, if the cylinder liner cleanliness (iron fines) prior
to casting is not preserved, a very dense network of AlFe brittle crystals is formed weakening the interface (Section 3.4 and Figure 3.3).

2.3. Cast iron cylinder liners require precise pre-heating synchronized with the casting of the engine block’s operation. This and several other technological factors associated with the cast iron cylinder liners make them undesirable for the progressive metal casting industry and limits the development of higher performance engine blocks [187].

3. The preliminary experiments demonstrate various degrees of effectiveness of the ES and ESV melt treatments for Al-Si hypo and hypereutectic alloys in semi-solid and liquid states (Sections 5.1 to 5.3).

3.1. The ES technology has the capabilities to significantly refine the microstructure of the W319 Al-Si hypoeutectic alloy in the semi-solid state only (Section 5.1) [2]. The ES of the solidifying melt introduced dendrite arm shearing (increasing the number of nuclei) that resulted in a partial elimination of the dendritic structure and consequently a reduction in the grain size ~ 600%, (Figures 5.1 and 5.3). However, the ES treatment has a negligible effect on the Al-Si eutectic, Mg and Cu enriched phases morphology (Figure 5.2). Thermal analysis was successfully used as an on-line tool for the determination of the effect of the ES treatment on the microstructure (Table 5.1).

3.2. The combined stirring and vibration effect created by the ESV melt treatment was able to modify the primary and eutectic Si in Al-Si hypereutectic alloys in the semi-solid and liquid states (Section 5.2, Figures 5.4 to 5.8).

3.3. Using two electromagnetic coils for ESV melt treatments, one for direct (DC) and another for variable (AC) currents, was detrimental because an AC current component is created in the DC coil; this results in a significant lost of the AC current. This phenomenon was not observed for the two-core-two-coil workstation where each coil was placed around a ferromagnetic core (Table 5.2, 5.3 and Section 5.2). However, the ferromagnetic cores are too expensive for eventual industrial applications.

3.4. A dual conductor electromagnetic coil was invented, using this coil it was possible to pass AC and DC currents through the same conductor. Also the controlled current source eliminates the detrimental AC component on the DC coil. This electromagnetic coil was considerably cheaper in comparison to the electromagnetic cores and was used to design the ESV workstation (ETCT) [6] (Sections 5.3 to 5.11).
4. The application of the newly developed Si\textsubscript{EQ} methodology for determination of the liquidus temperature of multicomponent Al-Si alloys (Section 5.3 and Eq. 5.1 and 5.3 and Figure 5.9) allowed for elimination of the superheat temperature as an independent variable for the ESV melt treatments for the studied hypereutectic alloys.

5. The results of characterization using light optical microscopy, SEM/EDX and thermal analysis are presented in Sections 5.4, 5.5 and 5.6 and the following conclusions can be made:

   5.1. The results of microstructural characterization were in agreement with thermal analysis using AITAP and UMSA platforms (Sections 5.4, 5.5, Table 5.5, Figures 5.10 to 5.23 and 5.24 to 5.28). The detected phases using thermal analysis were then identified with LOM and SEM for the reference and ESV melt treated test samples (Tables 5.6 to 5.9).

   5.2. The microstructure of the quenched test samples for the 390.1(1) and the 393.2(1) Al-Si hypereutectic alloys (Figures 5.31 and 5.32) showed the immiscible Si agglomerates in the liquid melts, corresponding to the reported Si\textsubscript{4} phase in the literature [121, 125].

   5.3. The Si\textsubscript{4} agglomerates are Si enriched regions corresponding to the preliminary stage of nucleation of the primary Si particles. These agglomerates presented susceptibility to the ESV melt treatments, resulting in a considerable Si modification that was observed in the microstructure and monitored with thermal analysis (Figures 5.14 to 5.23).

   5.4. The Si\textsubscript{4} agglomerates growth linearly from the liquid state to the Al-Si eutectic approximately (Figure 5.34). While at this temperature the Al-Si eutectic nucleates ($T_{Al-Si_{Nuc}} \sim 557^\circ C$) and grows up to the solidus temperature ($T_{Solid} \sim 480^\circ C$) [169, 183, 191]. Therefore the determination of the apparent fraction solid of the Mg, Ni and Cu enriched phases using the baseline method was feasible (Figure 5.26 and Table A2.11).

6. The currently used methods for characterization of the Si morphology for the Al-Si alloys [19, 20, 198, 199] were not adequate for this project. Therefore, a novel and reliable SiML algorithm for comprehensive quantification of the effect of the ESV melt treatments for the Al-Si alloys to assess the level Si modification was developed (Figures 5.47 to 5.50).

   6.1. Using the results of the characterization of the microstructure a multiple regression analysis was conducted to develop two equations (Eqs. 5.13 and 5.14) that predict the SiML as a function of the independent variables ($f, T_{ESV}, I_{AC/DC}$, chemical composition, degassing and cooling rate). Equations 5.13 and 5.14 have the potential to predict the SiML in an industrial environment.
6.2. The effect of the independent variables used for the statistically designed experiments (Table 5.15) showed that time and chemical composition have the strongest effect to modify Si; it is to reach higher SiML. However, combining ESV melt treatments with cooling rates from 1.3 to 1.6°C/s the SiML is further improved as well as the mechanical properties. Therefore, ESV treated melts can be ideal for die casting applications (i.e. pistons and cylinder liners).

7. ESV melt treated test samples that were remelted twice exhibited a considerably higher SiML, therefore, the ESV technology could be considered to manufacture pre-treated ingots for thixocasting or rheocasting applications (Section 5.75, Figures 5.61-5.63).

8. Studies of the thermal expansion coefficient showed the following:
8.1. Eqs. 5.18 and 5.19 showed that the thermal expansion coefficient can be precisely determined using the SiEQ algorithm methodology for multi-component Al-Si alloys and various temperatures.

8.2. Al-Si hypereutectic ESV treated test samples have a slightly lower thermal expansion coefficient in comparison to the ones cast using the reference melt (Figure 5.66).

8.3. The Al-Si hypereutectic alloys have a similar TEC as the W319 alloy (Figure 5.65). Therefore, it can be predicted that by replacing the cast iron for Al-Si hypereutectic cylinder liners for cast-in applications in engine blocks, the residual stresses are reduced and will allow shortening of the bridges to manufacture considerably more compact engine blocks [186].

9. The effect of the ESV melt treatments on melt characteristics revealed that:
9.1. The fluidity of the ESV treated melt is considerably higher in comparison to the reference melt (Figure 5.70). Therefore, for industrial applications the pouring temperature of the cast components can be significantly reduced resulting in energy savings and improved productivity.

9.2. The achieved reduction in the riser weight of the prototype pistons (~300%) demonstrated the superior feedability of the ESV treated melt. Therefore, substantial savings can be achieved by eliminating defects like misruns and reducing the amount of remelted materials from risers and feeder systems (Figures 5.68 and 5.69).
9.3. The use of Al-Si hypereutectic alloys for cylinder liner applications can improve the thermal conductivity of the engine block by up to ~3 times (from 46 to 130 W/mK) (Table 5.18).

10. Mechanical testing data of the ESV melt treated test samples showed the following significant improvements:

10.1. The Vickers metal matrix microhardness (μHV) and the Brinell macro hardness (HB) results indicate that the strengthening of ESV melt treated castings increases with the higher SiML (Table A2.14 and A2.15). The micro and macro hardness rises with the frequency, time and cooling rate of the ESV melt treatment but decreases as the temperature increases.

10.2. The ESV melt treated test samples of the 390.1(I) alloy in as cast conditions showed a ~19% higher metal matrix microhardness than the reference ones (Figure 5.72). However, the T6 heat treatment further increases the metal matrix microhardness up to ~35% for the ESV melt treated (390.1(I) alloy) in compared to the heat treated test samples cast with the reference melt.

10.3. In as-cast conditions the Brinell hardness was ~20% higher for the ESV melt treated test sample for the 390.1(I) alloy comparing with the reference test sample (Figure 5.74 and Table 5.19). After the T6 heat treatment the Brinell hardness is ~25% higher for the ESV melt treated test sample.

10.4. Eqs. 5.25 to 5.36 relate the ESV melt treatment conditions to the α-Al matrix microhardness and the Brinell hardness resulting in mathematical models that can be used for industrial application. For instance, predicted ESV melt treatment conditions can be used to achieve the desired hardness.

10.5. Eqs. 5.33 and 5.34 can be utilized to determine on line (using A1TAP) the microhardness or macrohardness of the ESV melt treated castings since these properties are statistically related to the liquidus post electromagnetic treatment temperature.

11. The effect of ESV melt treatments on Yield Strength (Y_S) and Ultimate Tensile Strength (UTS) on test bars cast using the Stahl mould [173] revealed that:

11.1. The test bars cast with ESV treated melt at temperatures as low as liquidus (610 and 750°C for the 390.1(I) and 393.2(I) alloys respectively) exhibited the highest mechanical properties (Figures 5.79 and 5.80). These properties were further improved within the
heat treated test samples. Resulting the test samples for the 390.1(I) alloy ESV melt treated and heat treated with the highest mechanical properties (Figure 5.79).

11.2. Two main reasons for the lower mechanical properties of the reference test samples were found; the agglomeration of coarse primary Si particles and the shrinkage porosity (Figures 5.83 and 5.84). These two features were not observed in ESV melt treated test samples for the 390.1(I) alloy (Figure 5.85).

11.3. The 393.2 alloy was less susceptible to the effect of the ESV melt treatments as the modification of the primary Si particles is concerned. This resulted in lower improvement of the mechanical characteristics of the 393.2(I) test samples. Therefore, it can be concluded that the size of the primary Si particles plays an important role in the tensile properties of the Al-Si hypereutectic alloys (Figure 5.84).

11.4. Based on the previous hypothesis it can be concluded that the ideal alloy chemical composition for piston and cylinder liner applications must be primary Si free. Therefore, the 390.1(I) alloy test samples, in the ESV melt treatment conditions, are the best for above-mentioned automotive applications (Figures 5.79 and 5.80). The mechanical characteristics of the ESV melt treated 390.1(I) alloy are similar to those required for pistons and cylinder liners based on Ford's Engineering Materials Specification (Table 5.18) [209 – 211, 216].

12. The prototype V6 engine blocks cast with non-preheated Al-Si cylinder liners showed the following advantages in comparison to the regular production engine blocks using preheated (tocco) cast iron cylinder liners:

12.1. The elimination of the “tocco” operation for the cast-in prototype Al-Si cylinder liners exhibited a slight reduction in SDAS, and a considerable reduction of the porosity of the bulkhead sections.

12.2. The reduced porosity of the bulkhead sections resulted in a considerable increase in tensile properties for the prototype engine blocks (Figures 5.81 and 5.82).

12.3. The fractography analysis conducted on the test bars extracted from the bulkhead sections of the regular production V6 engine blocks showed interconnected shrinkage pores. The shrinkage pores were the crack initiators because they acted as stress concentrators (Figure 5.87). In contrast, due to the shrinkage free test bars extracted from the bulkhead sections of the prototype V6 engine blocks their mechanical properties were superior.
CHAPTER 7. FUTURE WORK

Using the algorithms developed in this dissertation based on ESV melt treatment conditions, thermal analysis, image analysis and mechanical properties it is proposed to create a computer program for the on line determination of the SiML and hardness of ESV melt treated castings. This software can be implemented in the ESV workstation (ETCT) that will be eventually patented and commercialized.

It is propose a methodology to include the presence of the Si4 agglomerates in liquid state in the Al-Si phase diagram. This procedure can be performed using thermodynamic data and the results of the conducted quenching experiments.

To perform thermal, chemical and combined melt treatments or powder metallurgy using the 390.1(l) alloy to reach the different SiML to prove if Eqs. 5.22, 5.23, 5.27 and 5.28 can be used for other applications.

The test samples solidified directionally can be utilized to confirm the results of the mechanical testing using the Stahl Mould.

Casting of prototype V6 engine blocks with only ESV melt treated Al-Si cylinder liners. These prototype engine blocks could be cast using ESV melt treated Al-Si cylinder liners of larger diameter or shorter bridges following the cast-in cylinder liners procedure. These engines are recommended to be dynotested and to investigate the wear resistance of the prototype ESV melt treated Al-Si cylinder liners.
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the use of at least one magnetic field which differs from the field of confinement. Assignee: Aluminum


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APPENDIX I. THERMAL ANALYSIS

A2.1. Heat Exchange Conditions and Thermal Analysis Procedures

In order to achieve high accuracy using thermal analysis it is important to establish heat exchange conditions that will comply with the model utilized for the baseline. For the experimental portion of this dissertation the test samples were solidified under two different conditions. In the first one the test samples were solidified under natural heat exchange conditions by solidifying the test sample inside the Dual coil of the ETCT workstation (Figure A1.1a). In the second procedure the entire solidification process was carried out on top of the chilling device (Cu plate), forcing the test sample to solidify directionally (Figure A1.1b). The data gathered from a test sample poured into the ESV standard cup was recorded using a Low Mass Thermocouple (LMT) located in the centre of the test sample (Tc in Figure A1.1). The cooling curves were recorded using AITAP at a rate of five readings per second.

In the solidification of test samples under natural heat exchange conditions the heat is released only in the radial direction (one dimension (1D)). The assessment of the heat released in one dimension is achieve by insulating the top and bottom of the test samples with ceramics (Figure A1.1a). The main idea of allowing the heat to be released in only radially (1D) is to minimize the gradient of temperature between the centre and the wall of the test sample. The gradient of temperatures can be measured using two thermocouples, one located in the centre of the test samples and the other one in the vicinity of the wall. The results of several investigations conducted by the IRC shows that the gradient of temperatures (\(\Delta T = T(r) - T(R)\)) was in all cases \(\leq 6^\circ C\); which was considered negligible for the purpose of thermal analysis. Using the above-mentioned criteria’s the IRC developed novel algorithms (presented in the following Sections) to determined the first derivative and the baseline and fraction solid curves based on the recorded cooling curves [182, 183]. Additionally, the IRC utilized successfully the above-mentioned conditions on several research investigations conducted using various alloys [16, 169, 172, 175 - 183, 191, 197].

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Figure A1.1. Diagrams of test samples solidified under a) natural heat exchange conditions (heat released radially (1D)) and b) directionally using the Cu chilling plate (heat released radially and vertically (2D)).

Figures A1.2, and A1.3 show the cooling, first derivative, baseline and fraction solid curves of the 390.1(1) Al-Si hypereutectic alloy solidified using the heat released in 1D and 2D conditions. The cooling curves were recorded using only one thermocouple located in the centre of the test sample. The respective first derivative, baseline and fractions solid were determined using the algorithms developed by Dr. W. T. Kierkus et. al. [182, 183]. This research shows the results of reference and ESV melt treated test samples solidified in 1D conditions using the 390.1(I) and 393.2(I) alloys (Figures 5.25, 5.27 and 5.40). It is evident that allowing the heat to be release only in the radial direction the characterization of all the reactions that occur during solidification of the studied alloys can be assess.

Additionally, thermal analysis conducted on test samples for the 390.1(I) and 393.2(I) alloys solidified directionally was investigated. Directional solidification (Figure A1.1b) creates a two dimensional temperature gradient (radially and vertically). Figure A1.1b shows a schematic drawing of the conditions under which the test sample was solidified. Figure A1.4 shows the cooling curve, first derivative and baseline curves of a reference test sample of the 390.1(I) alloy solidified directionally. In this figure it is evident the lack of accuracy to determine the nucleation
of solidifying reactions created due to the effect of a “double” temperature gradient (heat released in 2D).

Figure A1.2. Cooling, first derivative and baseline curves of the 390.1(1) alloy solidified under natural heat exchange conditions (radial heat exchange (1D)).

Figure A1.3. First derivative, baseline and fraction solid curves of the 390.1(1) alloy solidified under natural heat exchange conditions (radial heat exchange (1D)).
Appendix I. Thermal Analysis

Figure A1.4. Cooling, first derivative and baseline curves of the 390.1(1) alloy solidified directionally on the Cu chilling plate (radial and vertical heat exchange (2D)). Note: the poor accuracy to determine the nucleation temperature of Mg, Ni and Cu phases.

The analysis presented on this report is the Newtonian model. To use this baseline calculation procedure the characteristics of the Thermal System (TS), sample and cup, has to be considered. The TS has to comply with the Newtonian cooling model. It is for this reason that the temperature gradient must be held as uniform as possible in both, radial (r) and vertical (Z), directions during the solidification process. These conditions are mostly found when a thin walled cup is used during the experimentation as well as insulated top and bottom (see Figure A1.1); the heat is released through the radial (r) direction (1 dimension (1D)). The heat can also be released for the same model in both directions, vertical (Z) and radial (r), and it is known as two dimensional solidification (2D). In this dissertation test samples were solidified under both conditions; for instance, 2D was used for the statistical designed experiments (using AITAP) and 1D for the rest of the experiments (using AITAP and UMSA).

In addition, the rate of cooling, due to the heat loss from the cup to the surroundings, should be limited by the Biot Modulus (Bi<0.1 Eq. A2.1) for the system, which depends on the “dimensions”, “overall heat transfer coefficient” and thermal conductivity of the tested material.
Appendix I. Thermal Analysis

\[ Bi = \frac{hL}{\lambda_b} \tag{Eq. A2.1} \]

Where: \( Bi \) is the dimension less Biot number,
\( h \) is the overall heat transfer coefficient,
\( L \) is the characteristic length and
\( \lambda_b \) is the thermal conductivity of the body

During the solidification the thermal capacitance of the system should be temperature independent. Under these assumptions the energy balance equation can be written as follows;

\[ \frac{dT_c}{dt} = \left( -\frac{UA}{\rho C_p V} \right) (T_c - T_w) + \frac{1}{\rho C_p V} \frac{dQ_l}{dt} \tag{Eq. A2.2} \]

Where: \( T_c \) and \( T_w \) are temperatures at the centre and wall of the test sample respectively,
\( t \) is time,
\( r \) is the density of the molten alloy
\( C_p \) is the heat capacity at a constant pressure,
\( U \) is the heat transmission coefficient and
\( A \) and \( V \) are the area through the heat is released and volume of the test samples respectively

The solution of this equation for \( T_c(t) \) represents the cooling curve. In cases where no metallurgical reactions occur, when \( Q_l = 0 \) and when:

\[ T_c \geq T_{c,liq} \text{ and } T_c \leq T_{c,SOl} \tag{Eq. A2.3} \]

Eq. A2.2 can be reduced into Eq. A2.4, as follows:

\[ \frac{dT_c}{dt} = \left( -\frac{UA}{\rho C_p V} \right) (T_c - T_\infty) \tag{Eq. A2.4} \]

Solving Eq. A2.4 will determine the time dependent \( T_c \) before solidification has started and after it has finished, as stated in Eq. A2.3. It is logical that the heat transmission coefficient “\( U \)”, time and temperature dependent, obey the same continuous function before and after the solidification. Also the term \( \rho C_p \) is time and temperature dependent, however its variation is too small that it can be treated as a constant. Based on the measured \( T_c \) and the calculated \( dT_c/dt \), the
Appendix I. Thermal Analysis

U(T_c) function can be calculated if the right side of Eq. A2.4 is fit in polynomial terms of T_c by using a standard regression method.

\[
\frac{dT_c}{dt} = \sum_{i=0}^{n} a_i(T_c)^n
\]

Eq. A2.5

Therefore the values of dT_c/dt can be determined for all recorded values of T_c, based on the baseline equation (BL):

\[
\left( \frac{dT_{c,\text{zero}}}{dt} \right) = BL(T_c)
\]

Eq. A2.6

This equation is clearly dependent on time because T_c is time dependent. Figures A1.2 and A1.3 show the calculated first derivative, baseline and fractions solid of a reference test samples of the 390.1(I) alloy. The above-mentioned test sample was solidified under natural heat exchange conditions (radial heat released (1D)). The thermal analysis was conducted in the ESV standard Cup under the conditions provided in Chapter 4.

The fraction solid is calculated based on the integral under the curve between the first derivative and the baseline. Eq. A2.7 represents the area under the curve.

\[
\text{Total Area} = \int_{\text{sol}}^{\text{iq}} \frac{dT_c}{dt} dt - \int_{\text{sol}}^{\text{iq}} \frac{dT_{c,\text{zero}}}{dt} dt
\]

Eq. A2.7

The fraction integral is determined by calculating the portion of the integral from liquidus to the specific time (i) and temperature (i) characteristic point or reaction under analysis. This portion of the integral is then used to determine the corresponding fraction solid. Also, the determination of the apparent fraction solid of the Mg, Ni and Cu enriched phases is carried out by the regression of the Al-Si eutectic reaction and the solidus point; divided by the integral of the total area. Eq. A2.8 shows the calculation for the area after liquidus for the time i.
Appendix I. Thermal Analysis

\[ \text{Area}_{t_{\text{Liq}} < t < t_{\text{Sol}} \text{ and } T_{\text{Liq}} > T_{\text{Sol}}} = \int_{t_{\text{Liq}}}^{t_{\text{Sol}}} \frac{dT_C}{dt} \, dt + \int_{t_{\text{Liq}}}^{t_{\text{Sol}}} \frac{dT_{C,\text{zero}}}{dt} \, dt \text{ or } \sum_{t_{T=1_{\text{Liq}}}}^{t_{T_{\text{Sol}}}} \frac{dT_C}{dt} - \sum_{t_{T=1_{\text{Liq}}}}^{t_{T_{\text{Sol}}}} \frac{dT_{C,\text{zero}}}{dt} \]  

Eq. A2.8

Finally, the fraction solid calculated by the fraction of the area for the time and temperature “i” divided by the total area (integral), which is expressed by Eq. A2.9.

\[ f_S = \frac{\text{Area}_{t_{\text{Liq}} < t < t_{\text{Sol}} \text{ and } T_{\text{Liq}} > T_{\text{Sol}}}}{\text{Total Area}} \]  

Eq. A2.9

An example of the determination of the fraction solid curve for a representative test sample of the 390.1(I) Alloy is presented in Figure A1.3; this test sample was solidified under natural heat exchange conditions close to equilibrium conditions (C.R. = 0.08°C/s).

A2.2. Experimental Results of the Different Thermal Analysis Performed on Al-Si Hypereutectic Alloys

Heat exchange conditions are very important to perform a proper thermal analysis. Therefore, it is important to understand them in order to perform a proper thermal analysis. Figure A1.1 shows schematic diagrams of the two heat exchange conditions used in the present dissertation to conduct thermal analysis. These diagrams show a curve indicating the temperature gradient (at the centre (T(r)) and at the wall (T(R)) of the test samples. For the case of the heat released only in the radial direction (1D) the gradient of temperatures (ΔT) between the centre and the wall of the test samples must be as low as possible and no more than ΔT < 6°C. The top and bottom of the sample are insulated using ceramics that avoid any loose of heat in the axial direction (Figure A1.1a). For the release of the heat in two dimensions (2D), the ceramic located at the bottom of the test sample is removed and the test sample is placed on top of a Cu plate that is air cooled (Figure A1.1b); where the gradient of temperature between centre and wall of the test sample must be ΔT > 6°C.
A2.2.2. Determination of the Characteristic Reactions that Occur During the Solidification of Al-Si Hypereutectic Alloys

In following a description of the interpretative methodology of the cooling curve is provided. This method will provide the characteristic temperatures for the specific reactions (points) on the different cooling curves. Once these points have been determined, using the fraction solid algorithm, the corresponding fraction solid can be estimated.

A2.2.3. Determination of the Characteristic Points that Occur During the Solidification of Al-Si Hypereutectic Alloys

Figures A1.5 to A1.7 show a schematic representation of the determination of the characteristic points. These Figures show sections of the cooling curve and its respective portion of the first derivative. The first derivative curve is presented with the aim to enhance the characteristic points of the cooling curve, thus simplifying the identification of these points. A table containing all the characteristic points together with its definition is presented following every Figure. The common method to identify the characteristic temperatures is the extension of regression lines corresponding to the segments where the first derivative changes its slope. This procedure is valid for the following characteristic points: $T_{\text{PRI-Si}}^\text{MIN}$, $T_{\text{Al-Si}}^\text{E,NUC}$, $T_{\text{MgSi}}^\text{E,NUC}$, $T_{\text{AlNi}}^\text{E,NUC}$, $T_{\text{AlNi-Cu}}^\text{E,NUC}$, and $T_{\text{AlCu}^\text{FE}}^\text{E,NUC}$. On the other hand, $T_{\text{PRI-Si}}^\text{G}$, $T_{\text{Al-Si}}^\text{E,MIN}$ and $T_{\text{Al-Si}}^\text{E,G}$ are determined at the zero value of the first derivative.

The undercooling of the primary Si nucleation ($T_{\text{PRI-Si}}^\text{MIN}$) of the 390 alloys (hypereutectic Al-Si systems) is not as profound as the one presented for hypoeutectic Al-Si alloys systems such as the 319 alloys. Therefore, the characteristic points $T_{\text{PRI-Si}}^\text{MIN}$ and $T_{\text{PRI-Si}}^\text{G}$ are not significant enough to be considered as key point to identify any microstructural changes using thermal analysis.
Figure A1.5. Determination of the characteristic points, $T_{PRI-Si}^{NUC}$, $T_{PRI-Si}^{MIN}$ and $T_{PRI-Si}^{G}$, during the solidification of the 390.1(I) alloy under natural heat exchange conditions.

Table A1.1. Definition of the characteristic points associated with the primary Si solidification of the 390.1(I) alloy.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Name</th>
<th>Detection Algorithm</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{PRI-Si}^{NUC}$</td>
<td>Primary Si nucleation (Liquidus)</td>
<td>This point corresponds to the intersection of the two local regression lines, slopes, of the first derivative (Figure A1.6).</td>
<td>At this point the primary Si particles start to precipitate.</td>
</tr>
<tr>
<td>$T_{PRI-Si}^{MIN}$</td>
<td>Primary Si Undercooling</td>
<td>This point is determined by the intersection of the first derivative curve with the first intersection with the zero value of the first derivative (Figure A1.6).</td>
<td>At this point, the nucleated primary Si particles have grown to beyond a critical size and the latent heat release balances the heat extracted from the test sample.</td>
</tr>
<tr>
<td>$T_{PRI-Si}^{G}$</td>
<td>Primary Si Growth</td>
<td>Second intersection of the first derivative with zero (Figure 4a).</td>
<td>Maximum point of recalescence.</td>
</tr>
</tbody>
</table>
Appendix I. Thermal Analysis

Figure A1.6. Determination of the characteristic points, $T_{E, NUC}^{Al-Si}$, $T_{MIN}^{Al-Si}$ and $T_{G}^{Al-Si}$, found during the solidification of Al-Si hypereutectic alloys.

Table A1.2. Definition of the characteristic points associated with the Al-Si eutectic solidification of the 390 alloy.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Name</th>
<th>Detection Algorithm</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{E, NUC}^{Al-Si}$</td>
<td>Al-Si eutectic nucleation temperature</td>
<td>This point corresponds to the intersection of two slopes when the first derivative changes (Figure 4b).</td>
<td>At this point the Al-Si eutectic phase starts to precipitate.</td>
</tr>
<tr>
<td>$T_{MIN}^{Al-Si}$</td>
<td>Al-Si eutectic minimum temperature.</td>
<td>This point corresponds to the local intersection of the first derivative with the zero level (Figure 4b).</td>
<td>At this temperature the nucleated Al-Si particles try to reach a critical radius, releasing the enthalpy of transformation absorbed during the melting process, thus Temperature increases.</td>
</tr>
<tr>
<td>$T_{G}^{Al-Si}$</td>
<td>Al-Si eutectic Growth</td>
<td>Second intersection of the first derivative with zero (Figure 4a).</td>
<td>At this point the growth of the Al-Si eutectic occurs.</td>
</tr>
</tbody>
</table>
Figure A1.7. Determination of the characteristic points, $T_{Mg2Si}$, $T_{AlNi}$, $T_{Al-Ni-Cu}$, and $T_{Al2Cu+Al-CuFe}$ found during the solidification of Al-Si hypereutectic alloys. Note: FE in the $T_{Al2Cu+Al-CuFe}$ reaction means Fine Eutectic.

Table A1.3. Definition of the characteristic points associated with the precipitation of Mg, Cu, Ni, Mn, Fe and Si rich phase on the 390 alloy.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Name</th>
<th>Detection Algorithm</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{Mg2Si}$</td>
<td>Mg$_2$Si, Al$_3$Ni, Al-Cu-Ni, Al$_2$Cu and Al-CuFine</td>
<td>All reactions where identified using SEM/EDX analysis and correspond to the Mg$_2$Si and three Al-Mg-Fe-Si-Cu rich phases. The above mentioned reactions are identified by the intersection of their respective slopes. (Figure 4c).</td>
<td>Temperature at which the precipitation of the Mg$_2$Si and Cu-Ni rich phase begins.</td>
</tr>
<tr>
<td>$T_{AlNi}$</td>
<td>Eutectic Eutectic phases</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$T_{Al-Ni-Cu}$</td>
<td>Eutectic</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$T_{Al2Cu+Al-CuFe}$</td>
<td>(THREE)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$T_{SOL}$</td>
<td>Solidus</td>
<td>Intersection of the two local slopes (Figure 4c)</td>
<td>End of solidification.</td>
</tr>
</tbody>
</table>
APPENDIX II. SUMMARY OF THE DATA COLLECTED FOR THIS DISSERTATION

Table A2.1. Summary of the cooling curve characteristics of the ESV melt treated test samples for the 390.1(I) alloy, at different temperature range(s) (ΔT), current(s) \( I_{AC/DC} \), frequencies(s) \( f \), with non-negassed (N) and degassed (Y) melts and a time \( t \) of 3 min. Note: all data presented in the following Table was recorded with AITAP.

<table>
<thead>
<tr>
<th>Sample I.D. #</th>
<th>ESV Melt Treatment Conditions</th>
<th>Cooling Curve Characteristic, (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( f ) (Hz)</td>
<td>( I_{AC/DC} ) (A)</td>
</tr>
<tr>
<td>1</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>6</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>7</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>8</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>9</td>
<td>100</td>
<td>40</td>
</tr>
<tr>
<td>10</td>
<td>100</td>
<td>40</td>
</tr>
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<td>11</td>
<td>100</td>
<td>40</td>
</tr>
<tr>
<td>12</td>
<td>100</td>
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<tr>
<td>15</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>16</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>
Appendix II. Summary of the Data Collected for this Dissertation

Table A2.2. Summary of the cooling curve characteristics of the ESV melt treated test samples for the 390.1(I) alloy at different temperature ranges (AT), at a current(s) $I_{AC/DC} = 100$ A, frequencies ($f$), and a time of ($t$) = 3 min for the degassed melt.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>ESV Melt Treatment Conditions</th>
<th>Nucleation Temperatures of the Phases, (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$f$ (Hz)</td>
<td>$\Delta T_{Range}$ (°C)</td>
</tr>
<tr>
<td>17</td>
<td>40</td>
<td>720-680</td>
</tr>
<tr>
<td>18</td>
<td>80</td>
<td>720-680</td>
</tr>
<tr>
<td>19</td>
<td>100</td>
<td>720-680</td>
</tr>
<tr>
<td>20</td>
<td>40</td>
<td>680-650</td>
</tr>
<tr>
<td>21</td>
<td>80</td>
<td>680-650</td>
</tr>
<tr>
<td>22</td>
<td>100</td>
<td>680-650</td>
</tr>
<tr>
<td>23</td>
<td>40</td>
<td>650-610</td>
</tr>
<tr>
<td>24</td>
<td>80</td>
<td>650-610</td>
</tr>
<tr>
<td>25</td>
<td>100</td>
<td>650-610</td>
</tr>
</tbody>
</table>

Table A2.3. Summary of the cooling curve characteristics of the ESV melt treated test samples isothermally treated for different compositions, using a $I_{AC/DC} = 100$ A, frequencies ($f$) = 100 Hz, for various times with degassed melt.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>Isothermal Time (s)</th>
<th>Alloy Type (AA)</th>
<th>$T_{LIQ}$</th>
<th>$T_{Al-Si}^{NUC}$</th>
<th>$T_{DCP}^{NUC}$</th>
<th>$T_{DCP}^{MIN}$</th>
<th>$T_{DCP}^{G}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>27</td>
<td>45</td>
<td>390.1(I)</td>
<td>601.9</td>
<td>572.2</td>
<td>560.0</td>
<td>557.5</td>
<td>560.2</td>
</tr>
<tr>
<td>28</td>
<td>90</td>
<td>390.1(I)</td>
<td>605.9</td>
<td>571.9</td>
<td>562.2</td>
<td>560.4</td>
<td>562.6</td>
</tr>
<tr>
<td>29</td>
<td>180</td>
<td>390.1(I)</td>
<td>609.9</td>
<td>569.7</td>
<td>558.6</td>
<td>556.6</td>
<td>557.0</td>
</tr>
<tr>
<td>30</td>
<td>360</td>
<td>390.1(I)</td>
<td>610.3</td>
<td>571.3</td>
<td>559.4</td>
<td>558.1</td>
<td>560.1</td>
</tr>
<tr>
<td>31</td>
<td>45</td>
<td>393.2(I)</td>
<td>740.9</td>
<td>577.2</td>
<td>569.9</td>
<td>568.4</td>
<td>570.6</td>
</tr>
<tr>
<td>32</td>
<td>90</td>
<td>393.2(I)</td>
<td>742.9</td>
<td>574.5</td>
<td>570.4</td>
<td>568.7</td>
<td>568.8</td>
</tr>
<tr>
<td>33</td>
<td>180</td>
<td>393.2(I)</td>
<td>754.9</td>
<td>577.5</td>
<td>570.3</td>
<td>569.9</td>
<td>571.1</td>
</tr>
<tr>
<td>34</td>
<td>360</td>
<td>393.2(I)</td>
<td>752.8</td>
<td>580.9</td>
<td>570.7</td>
<td>569.7</td>
<td>571.9</td>
</tr>
<tr>
<td>35</td>
<td>480</td>
<td>393.2(I)</td>
<td>755.0</td>
<td>580.0</td>
<td>570.3</td>
<td>568.7</td>
<td>572.13</td>
</tr>
</tbody>
</table>
### Table A2.4. Summary of the Si modification level (SiML) results for the reference test samples for the 390.1(I) and 393.2(I) alloys having different hydrogen levels.

<table>
<thead>
<tr>
<th>Test Sample</th>
<th>Alloy</th>
<th>Degassed Melt</th>
<th>Undegassed Melt</th>
<th>Hom. Coef. (HC)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>SiML Standard Deviation</td>
<td>SiML Standard Deviation</td>
<td></td>
</tr>
<tr>
<td>Reference</td>
<td>390.1(I)</td>
<td>4.30</td>
<td>3.75</td>
<td>25.4</td>
</tr>
<tr>
<td></td>
<td>393.2(II)</td>
<td>1.84</td>
<td>4.36</td>
<td>37.4</td>
</tr>
</tbody>
</table>

### Table A2.5. Summary of the SiML for the 390.1(I) alloy ESV melt treated for \( t = 3 \) min under different currents, frequencies, temperature ranges and hydrogen levels.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>ESV Melt Treatment Conditions</th>
<th>SiML</th>
<th>Standard Deviation</th>
<th>Hom. Coef. (HC)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( f )</td>
<td>( I_{AC/DC} )</td>
<td>( \Delta T_{Range} )</td>
<td>Degass.</td>
</tr>
<tr>
<td>1 10 40 650-610 Yes</td>
<td>5.21</td>
<td>3.77</td>
<td>24.3</td>
<td></td>
</tr>
<tr>
<td>2 10 40 720-680 Yes</td>
<td>4.72</td>
<td>4.04</td>
<td>28.0</td>
<td></td>
</tr>
<tr>
<td>3 10 40 650-610 No</td>
<td>5.71</td>
<td>3.85</td>
<td>25.4</td>
<td></td>
</tr>
<tr>
<td>4 10 40 720-680 No</td>
<td>5.06</td>
<td>3.31</td>
<td>18.5</td>
<td></td>
</tr>
<tr>
<td>5 10 100 650-610 Yes</td>
<td>4.59</td>
<td>3.54</td>
<td>21.3</td>
<td></td>
</tr>
<tr>
<td>6 10 100 720-680 Yes</td>
<td>5.25</td>
<td>3.28</td>
<td>18.1</td>
<td></td>
</tr>
<tr>
<td>7 10 100 650-610 No</td>
<td>5.06</td>
<td>2.7</td>
<td>12.0</td>
<td></td>
</tr>
<tr>
<td>8 10 100 720-680 No</td>
<td>5.3</td>
<td>3.28</td>
<td>18.1</td>
<td></td>
</tr>
<tr>
<td>9 100 40 650-610 Yes</td>
<td>6.1</td>
<td>2.06</td>
<td>6.5</td>
<td></td>
</tr>
<tr>
<td>10 100 40 720-680 Yes</td>
<td>4.64</td>
<td>3.68</td>
<td>23.1</td>
<td></td>
</tr>
<tr>
<td>11 100 40 650-610 No</td>
<td>5.71</td>
<td>2.82</td>
<td>13.1</td>
<td></td>
</tr>
<tr>
<td>12 100 40 720-680 No</td>
<td>5.02</td>
<td>3.58</td>
<td>21.8</td>
<td></td>
</tr>
<tr>
<td>13 100 100 650-610 Yes</td>
<td>6.29</td>
<td>1.87</td>
<td>5.2</td>
<td></td>
</tr>
<tr>
<td>14 100 100 720-680 Yes</td>
<td>4.12</td>
<td>4.49</td>
<td>34.8</td>
<td></td>
</tr>
<tr>
<td>15 100 100 650-610 No</td>
<td>6.39</td>
<td>1.73</td>
<td>4.3</td>
<td></td>
</tr>
<tr>
<td>16 100 100 720-680 No</td>
<td>5.41</td>
<td>2.87</td>
<td>13.6</td>
<td></td>
</tr>
</tbody>
</table>
Table A2.6. Summary of the SiML for the ESV melt treated test samples under different frequencies and temperature ranges. Note all results presented correspond to the ESV melt treated test samples for the 390.1(1) Alloy, \( I_{AC/DC} = 100 \, \text{A} \) and \( t = 3 \, \text{min} \) cast with the degassed melt (<0.100 mL \( \text{H}_2/100 \, \text{g Al} \)).

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>ESV Melt Treatment Conditions</th>
<th>SiML</th>
<th>Standard Deviation</th>
<th>Hom. Coef. (HC)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( f ) (Hz)</td>
<td>( \Delta T_{Range} ) (°C)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>40</td>
<td>720-680</td>
<td>4.12</td>
<td>4.40</td>
</tr>
<tr>
<td>18</td>
<td>80</td>
<td>720-680</td>
<td>4.50</td>
<td>3.47</td>
</tr>
<tr>
<td>19</td>
<td>100</td>
<td>720-680</td>
<td>4.94</td>
<td>4.49</td>
</tr>
<tr>
<td>20</td>
<td>40</td>
<td>680-650</td>
<td>4.81</td>
<td>4.01</td>
</tr>
<tr>
<td>21</td>
<td>80</td>
<td>680-650</td>
<td>5.02</td>
<td>4.15</td>
</tr>
<tr>
<td>22</td>
<td>100</td>
<td>680-650</td>
<td>5.83</td>
<td>2.66</td>
</tr>
<tr>
<td>23</td>
<td>40</td>
<td>650-610</td>
<td>5.69</td>
<td>2.69</td>
</tr>
<tr>
<td>24</td>
<td>80</td>
<td>650-610</td>
<td>5.76</td>
<td>2.82</td>
</tr>
<tr>
<td>25</td>
<td>100</td>
<td>650-610</td>
<td>6.28</td>
<td>1.95</td>
</tr>
</tbody>
</table>

Table A2.7. Summary of the SiML for the ESV melt treated samples for different 3XX.X alloy compositions using \( I_{AC/DC} = 100 \, \text{A}, f = 100 \, \text{Hz} \), for variable times for the degassed melt.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>Isothermal Holding Time (s)</th>
<th>Alloy Type (AA)</th>
<th>SiML</th>
<th>Standard Deviation</th>
<th>Hom. Coef. (HC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>26</td>
<td>22.5</td>
<td>390.1(1)</td>
<td>4.57</td>
<td>3.34</td>
<td>18.8</td>
</tr>
<tr>
<td>27</td>
<td>45</td>
<td>390.1(1)</td>
<td>4.69</td>
<td>3.23</td>
<td>17.5</td>
</tr>
<tr>
<td>28</td>
<td>90</td>
<td>390.1(1)</td>
<td>5.38</td>
<td>2.55</td>
<td>10.6</td>
</tr>
<tr>
<td>29</td>
<td>180</td>
<td>390.1(1)</td>
<td>6.29</td>
<td>1.82</td>
<td>4.9</td>
</tr>
<tr>
<td>30</td>
<td>360</td>
<td>390.1(1)</td>
<td>6.38</td>
<td>1.58</td>
<td>3.4</td>
</tr>
<tr>
<td>31</td>
<td>45</td>
<td>393.2(1)</td>
<td>2.15</td>
<td>3.25</td>
<td>32.8</td>
</tr>
<tr>
<td>32</td>
<td>90</td>
<td>393.2(1)</td>
<td>3.68</td>
<td>3.55</td>
<td>17.8</td>
</tr>
<tr>
<td>33</td>
<td>180</td>
<td>393.2(1)</td>
<td>4.38</td>
<td>2.35</td>
<td>21.4</td>
</tr>
<tr>
<td>34</td>
<td>360</td>
<td>393.2(1)</td>
<td>4.99</td>
<td>2.12</td>
<td>8.8</td>
</tr>
<tr>
<td>35</td>
<td>540</td>
<td>393.2(1)</td>
<td>5.83</td>
<td>2.36</td>
<td>7.0</td>
</tr>
</tbody>
</table>
Appendix II. Summary of the Data Collected for this Dissertation

Table A2.8. Results for the determination of the influence of the macrosegregation of the primary Si particles on the liquid 390.1(I) alloy laminar or turbulent flow.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>ESV Melt Treatment Conditions</th>
<th>Settling Velocity (cm/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$f$ (Hz)</td>
<td>$\Delta T_{\text{Range}}$ (°C)</td>
</tr>
<tr>
<td>Reference</td>
<td>0</td>
<td>N/A</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>650-610</td>
</tr>
<tr>
<td>23</td>
<td>40</td>
<td>650-610</td>
</tr>
<tr>
<td>24</td>
<td>80</td>
<td>650-610</td>
</tr>
<tr>
<td>25</td>
<td>100</td>
<td>650-610</td>
</tr>
</tbody>
</table>

Table A2.9. Summary of the conditions for the ESV melt treated test samples of the 390.1(I) alloy solidified under natural heat exchange conditions followed by the ESV treatment.

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>Current (A)</th>
<th>Frequency (Hz)</th>
<th>Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AC</td>
<td>DC</td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>100</td>
<td>40</td>
<td>685</td>
</tr>
<tr>
<td>36</td>
<td>100</td>
<td>40</td>
<td>650</td>
</tr>
<tr>
<td>37</td>
<td>100</td>
<td>40</td>
<td>610</td>
</tr>
<tr>
<td>38</td>
<td>100</td>
<td>80</td>
<td>610</td>
</tr>
<tr>
<td>39</td>
<td>100</td>
<td>100</td>
<td>610</td>
</tr>
<tr>
<td>40</td>
<td>100</td>
<td>100</td>
<td>650</td>
</tr>
</tbody>
</table>

Table A2.10. Temperatures for the identified reactions during solidification for the 390.1(I) alloy ESV melt treated and solidified under natural heat exchange conditions.

<table>
<thead>
<tr>
<th>Test Sample ID #</th>
<th>Liquidus (°C)</th>
<th>Al-Si Eutectic (°C)</th>
<th>Mg$_2$Si Eutectic (°C)</th>
<th>Al-Ni Eutectic (°C)</th>
<th>Al-Ni-Cu + Al-Cu$_{FE}$ ± Pb (°C)</th>
<th>Solidus (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$f = 40$ Hz $T_{\text{ESV}} = 685^\circ$C</td>
<td>602.1 ± 0.1</td>
<td>563.9 ± 0.9</td>
<td>551.9 ± 1.7</td>
<td>522.3 ± 1.2</td>
<td>500.5 ± 0.9</td>
<td>474.7 ± 0.6</td>
</tr>
<tr>
<td>$f = 40$ Hz $T_{\text{ESV}} = 650^\circ$C</td>
<td>602.2 ± 0.5</td>
<td>559.8 ± 0.8</td>
<td>548.8 ± 1.8</td>
<td>521.8 ± 0.3</td>
<td>498.7 ± 0.4</td>
<td>474.8 ± 0.6</td>
</tr>
<tr>
<td>$f = 40$ Hz $T_{\text{ESV}} = 610^\circ$C</td>
<td>602.3 ± 0.6</td>
<td>559.9 ± 1.0</td>
<td>548.9 ± 0.9</td>
<td>521.1 ± 1.2</td>
<td>498.8 ± 0.7</td>
<td>475.9 ± 0.7</td>
</tr>
<tr>
<td>$f = 80$ Hz $T_{\text{ESV}} = 610^\circ$C</td>
<td>603.3 ± 0.2</td>
<td>560.1 ± 0.6</td>
<td>548.3 ± 0.6</td>
<td>521.2 ± 0.6</td>
<td>498.9 ± 1.0</td>
<td>475.6 ± 0.5</td>
</tr>
<tr>
<td>$f = 100$ Hz $T_{\text{ESV}} = 650^\circ$C</td>
<td>605.7 ± 0.7</td>
<td>560.0 ± 0.9</td>
<td>548.9 ± 0.5</td>
<td>523.8 ± 0.7</td>
<td>500.2 ± 1.2</td>
<td>475.6 ± 0.4</td>
</tr>
<tr>
<td>$f = 100$ Hz $T_{\text{ESV}} = 610^\circ$C</td>
<td>607.8 ± 0.5</td>
<td>557.7 ± 0.6</td>
<td>548.4 ± 0.8</td>
<td>523.8 ± 0.2</td>
<td>499.2 ± 0.3</td>
<td>474.8 ± 0.9</td>
</tr>
</tbody>
</table>

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Table A2.11. Evaluation of the integrals between the fraction solids and baselines for the reference and ESV treated test samples solidified under natural heat exchange conditions.

<table>
<thead>
<tr>
<th>Test Sample ID #</th>
<th>Overall Integral (a. u.)</th>
<th>Mg, Ni, Cu, Pb Enriched Phases</th>
<th>Partial Integral (a. u.)</th>
<th>Apparent Fraction Solid, (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference Test Sample</td>
<td>394.4</td>
<td>45.8</td>
<td>13.4</td>
<td></td>
</tr>
<tr>
<td>$f = 40$ Hz; $T_{ESV} = 685^\circ$C</td>
<td>404.4</td>
<td>51.2</td>
<td>12.7</td>
<td></td>
</tr>
<tr>
<td>$f = 40$ Hz; $T_{ESV} = 650^\circ$C</td>
<td>394.5</td>
<td>52.8</td>
<td>13.4</td>
<td></td>
</tr>
<tr>
<td>$f = 40$ Hz; $T_{ESV} = 610^\circ$C</td>
<td>391.6</td>
<td>55.7</td>
<td>15.0</td>
<td></td>
</tr>
<tr>
<td>$f = 80$ Hz; $T_{ESV} = 610^\circ$C</td>
<td>418.74</td>
<td>55.23</td>
<td>13.19</td>
<td></td>
</tr>
<tr>
<td>$f = 100$ Hz; $T_{ESV} = 650^\circ$C</td>
<td>411.59</td>
<td>60.15</td>
<td>14.61</td>
<td></td>
</tr>
<tr>
<td>$f = 100$ Hz; $T_{ESV} = 610^\circ$C</td>
<td>399.64</td>
<td>59.07</td>
<td>14.80</td>
<td></td>
</tr>
</tbody>
</table>

Table A2.12. Summary of the temperatures for the precipitation reactions of the remelted 390.1(1) reference test samples using UMSA and solidified at natural heat exchange conditions.

<table>
<thead>
<tr>
<th>Solidification Reaction</th>
<th>1st Remelting</th>
<th>2nd Remelting</th>
<th>3rd Remelting</th>
<th>Average Fraction Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Si</td>
<td>600.73</td>
<td>600.53</td>
<td>600.63</td>
<td>0.00</td>
</tr>
<tr>
<td>Al-Si Eutectic</td>
<td>564.98</td>
<td>564.98</td>
<td>564.98</td>
<td>4.64</td>
</tr>
<tr>
<td>Al-Si Eutectic</td>
<td>560.43</td>
<td>560.93</td>
<td>560.68</td>
<td>5.13</td>
</tr>
<tr>
<td>DCP</td>
<td>562.57</td>
<td>562.34</td>
<td>562.46</td>
<td>22.53</td>
</tr>
<tr>
<td>Mg$_2$Si Eutectic</td>
<td>551.37</td>
<td>551.44</td>
<td>551.41</td>
<td>62.45</td>
</tr>
<tr>
<td>Al-Ni Eutectic</td>
<td>525.12</td>
<td>523.11</td>
<td>524.12</td>
<td>89.28</td>
</tr>
<tr>
<td>Al$_2$Cu Eutectic</td>
<td>503.45</td>
<td>503.45</td>
<td>N/A</td>
<td>97.40</td>
</tr>
<tr>
<td>Al-Ni-Cu + Al-Cu$_{FE}$ + Pb</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Solidus</td>
<td>480.70</td>
<td>477.54</td>
<td>479.12</td>
<td>100.00</td>
</tr>
<tr>
<td>Solidification Range</td>
<td>120.20</td>
<td>122.99</td>
<td>121.60</td>
<td></td>
</tr>
</tbody>
</table>
Table A2.13. Summary of the temperatures for the precipitation reactions of the remelted 390.1(I) ESV melt treated ($I_{ACDC} = 100$ A, $f = 100$ Hz, $t = 3$ min, and a $T_{ESV} = 610 \pm 2$°C) test samples using UMSA and solidified under natural heat exchange conditions.

<table>
<thead>
<tr>
<th>Solidification Reaction</th>
<th>UMSA of the Reference Test Sample of the 390.1(I) Alloy</th>
<th>1st Remelting</th>
<th>2nd Remelting</th>
<th>3rd Remelting</th>
<th>Average Fraction Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Si</td>
<td></td>
<td>604.46</td>
<td>604.78</td>
<td>601.28</td>
<td>0.00</td>
</tr>
<tr>
<td>Al-Si Eutectic</td>
<td></td>
<td>563.03</td>
<td>563.32</td>
<td>562.49</td>
<td>5.37</td>
</tr>
<tr>
<td>Al-Si Eutectic</td>
<td></td>
<td>558.99</td>
<td>559.28</td>
<td>558.45</td>
<td>9.54</td>
</tr>
<tr>
<td>DCP</td>
<td></td>
<td>558.42</td>
<td>558.71</td>
<td>557.88</td>
<td>15.15</td>
</tr>
<tr>
<td>Mg$_2$Si Eutectic</td>
<td></td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Al-Ni Eutectic</td>
<td></td>
<td>520.24</td>
<td>520.51</td>
<td>519.74</td>
<td>87.62</td>
</tr>
<tr>
<td>Al,Cu Eutectic</td>
<td></td>
<td>502.77</td>
<td>503.03</td>
<td>502.29</td>
<td>97.81</td>
</tr>
<tr>
<td>Al-Ni-Cu + Al,Cu$_{Fe}$ + Pb</td>
<td></td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Solidus</td>
<td></td>
<td>478.23</td>
<td>478.48</td>
<td>477.77</td>
<td>100.00</td>
</tr>
<tr>
<td>Solidification Range</td>
<td></td>
<td>124.84</td>
<td>124.91</td>
<td>124.72</td>
<td></td>
</tr>
</tbody>
</table>

Table A2.14. Summary of the microhardness for the ESV melt treated test samples using different frequencies, temperatures and times. Test samples were directionally solidified on the Cu chilling plate; the ESV conditions for these test samples were $I_{ACDC} = 100$ A, $f = 100$ Hz, $t = 3$ min, and a $T_{ESV} = 610 \pm 2$°C, except where indicated.

<table>
<thead>
<tr>
<th>ESV Melt Treatment Conditions</th>
<th>Reference $f = 40$ Hz</th>
<th>$f = 80$ Hz</th>
<th>$f = 100$ Hz</th>
<th>$T = 650$°C</th>
<th>$t = 90$ s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microhardness ($\mu$HV$_{25}$)</td>
<td>80.99</td>
<td>87.98</td>
<td>92.77</td>
<td>95.34</td>
<td>81.33</td>
</tr>
<tr>
<td>Standard Dev.</td>
<td>4.08</td>
<td>5.62</td>
<td>5.07</td>
<td>7.76</td>
<td>4.22</td>
</tr>
<tr>
<td>Homo. Coeff.</td>
<td>0.00</td>
<td>-0.90</td>
<td>-0.54</td>
<td>-2.62</td>
<td>-0.07</td>
</tr>
<tr>
<td>Confidence Limit (95%)</td>
<td>1.79</td>
<td>2.46</td>
<td>2.22</td>
<td>3.40</td>
<td>1.85</td>
</tr>
<tr>
<td>Confidence Limit (99%)</td>
<td>2.35</td>
<td>3.24</td>
<td>2.92</td>
<td>4.47</td>
<td>2.43</td>
</tr>
</tbody>
</table>

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Table A2.15. Summary of the microhardness for the ESV melt treated test samples using different frequencies. Test samples were solidified under natural heat exchange conditions. The ESV conditions for the test samples were $I_{ACDC} = 100$ A, $t = 3$ min, and $T_{ESV} = 610 \pm 2^\circ$C.

<table>
<thead>
<tr>
<th>ESV Melt Treatment Conditions</th>
<th>Reference</th>
<th>$f = 40$ Hz</th>
<th>$f = 80$ Hz</th>
<th>$f = 100$ Hz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microhardness ($\mu$HV$_{25}$)</td>
<td>83.38</td>
<td>89.06</td>
<td>92.45</td>
<td>96.21</td>
</tr>
<tr>
<td>Standard Dev.</td>
<td>6.28</td>
<td>5.79</td>
<td>7.44</td>
<td>9.68</td>
</tr>
<tr>
<td>Horn. Coeff.</td>
<td>0.00</td>
<td>0.15</td>
<td>-0.40</td>
<td>-1.37</td>
</tr>
<tr>
<td>Confidence Limit (95%)</td>
<td>2.75</td>
<td>2.54</td>
<td>3.26</td>
<td>4.24</td>
</tr>
<tr>
<td>Confidence Limit (99%)</td>
<td>3.62</td>
<td>3.33</td>
<td>4.29</td>
<td>5.57</td>
</tr>
</tbody>
</table>

Table A2.16. Mean area fraction of porosity (%) of the bulkhead sections of the W319 V6 engine blocks for the regular production and prototype engine blocks cast with Al-Si cylinder liners (reference and ESV melt treated).

<table>
<thead>
<tr>
<th>Engine Block</th>
<th>Bulkhead #1</th>
<th>Bulkhead #2</th>
<th>Bulkhead #3</th>
<th>Bulkhead #4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regular Production Engine Block</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Left</td>
<td>$0.25 \pm 0.3$</td>
<td>$0.52 \pm 0.65$</td>
<td>$0.68 \pm 0.22$</td>
<td>$0.24 \pm 0.15$</td>
</tr>
<tr>
<td>Right</td>
<td>$0.26 \pm 0.19$</td>
<td>$0.56 \pm 0.32$</td>
<td>$0.59 \pm 0.2$</td>
<td>$0.22 \pm 0.1$</td>
</tr>
<tr>
<td>Prototype Engine Block 390.1(I)REF$^*$</td>
<td>Left</td>
<td>$0.14 \pm 0.09$</td>
<td>$0.12 \pm 0.04$</td>
<td>$0.16 \pm 0.07$</td>
</tr>
<tr>
<td>Right</td>
<td>$0.13 \pm 0.07$</td>
<td>$0.25 \pm 0.12$</td>
<td>$0.21 \pm 0.11$</td>
<td>$0.12 \pm 0.04$</td>
</tr>
<tr>
<td>Prototype Engine Block 390.1(I)ESV$^*$</td>
<td>Left</td>
<td>$0.16 \pm 0.1$</td>
<td>$0.10 \pm 0.09$</td>
<td>$0.13 \pm 0.11$</td>
</tr>
<tr>
<td>Right</td>
<td>$0.17 \pm 0.06$</td>
<td>$0.23 \pm 0.08$</td>
<td>$0.22 \pm 0.11$</td>
<td>$0.13 \pm 0.05$</td>
</tr>
<tr>
<td>Prototype Engine Block 393.2(I)REF$^*$</td>
<td>Left</td>
<td>$0.14 \pm 0.06$</td>
<td>$0.16 \pm 0.09$</td>
<td>$0.24 \pm 0.12$</td>
</tr>
<tr>
<td>Right</td>
<td>$0.14 \pm 0.12$</td>
<td>$0.21 \pm 0.08$</td>
<td>$0.18 \pm 0.08$</td>
<td>$0.16 \pm 0.12$</td>
</tr>
<tr>
<td>Prototype Engine Block 393.2(I)ESV$^*$</td>
<td>Left</td>
<td>$0.13 \pm 0.06$</td>
<td>$0.12 \pm 0.04$</td>
<td>$0.15 \pm 0.08$</td>
</tr>
<tr>
<td>Right</td>
<td>$0.17 \pm 0.05$</td>
<td>$0.20 \pm 0.13$</td>
<td>$0.23 \pm 0.11$</td>
<td>$0.14 \pm 0.08$</td>
</tr>
</tbody>
</table>

$^*$ REF or ESV indicates where the cast-in Al-Si hypereutectic alloy cylinder liners were cast with reference or ESV treated melt.
**Appendix II. Summary of the Data Collected for this Dissertation**

Table A2.17. Summary of the results of the mean values of the monotonic tensile test bars cast with 390.1(I) and 393.2(I) alloys using reference and ESV treated melts as-cast, artificially aged and heat treatment conditions.

<table>
<thead>
<tr>
<th></th>
<th>Tensile Strength (MPa)</th>
<th>Elongation (AL/L)</th>
<th>Elasticity Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2% Yield</td>
<td>UTS</td>
<td>0.2% Yield</td>
</tr>
<tr>
<td>390.1(I) Alloy Reference</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>167 ± 6</td>
<td>180 ± 12</td>
<td>0.3 ± 0.002</td>
</tr>
<tr>
<td>AA</td>
<td>185 ± 9</td>
<td>191 ± 7</td>
<td>0.4 ± 0.005</td>
</tr>
<tr>
<td>Heat Treat.</td>
<td>213 ± 10</td>
<td>228 ± 8</td>
<td>0.4 ± 0.001</td>
</tr>
<tr>
<td>390.1(I) Alloy ESV Melt Treated at 610°C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>191 ± 7</td>
<td>215 ± 9</td>
<td>0.4 ± 0.002</td>
</tr>
<tr>
<td>AA</td>
<td>201 ± 9</td>
<td>225 ± 6</td>
<td>0.4 ± 0.01</td>
</tr>
<tr>
<td>Heat Treat.</td>
<td>246 ± 11</td>
<td>291 ± 12</td>
<td>0.5 ± 0.001</td>
</tr>
<tr>
<td>390.1(I) Alloy ESV Melt Treated at 650°C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>184 ± 12</td>
<td>197 ± 6</td>
<td>0.4 ± 0.003</td>
</tr>
<tr>
<td>AA</td>
<td>183 ± 10</td>
<td>194 ± 9</td>
<td>0.4 ± 0.004</td>
</tr>
<tr>
<td>Heat Treat.</td>
<td>232 ± 8</td>
<td>247 ± 7</td>
<td>0.3 ± 0.002</td>
</tr>
<tr>
<td>393.2(I) Alloy Reference</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>172 ± 9</td>
<td>210 ± 6</td>
<td>0.3 ± 0.0001</td>
</tr>
<tr>
<td>AA</td>
<td>180 ± 12</td>
<td>212 ± 8</td>
<td>0.3 ± 0.004</td>
</tr>
<tr>
<td>Heat Treat.</td>
<td>194 ± 14</td>
<td>221 ± 9</td>
<td>0.3 ± 0.001</td>
</tr>
<tr>
<td>390.1(I) Alloy ESV Melt Treated at 750°C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-Cast</td>
<td>191 ± 7</td>
<td>219 ± 8</td>
<td>0.4 ± 0.002</td>
</tr>
<tr>
<td>AA</td>
<td>191 ± 8</td>
<td>221 ± 12</td>
<td>0.3 ± 0.004</td>
</tr>
<tr>
<td>Heat Treat.</td>
<td>195 ± 10</td>
<td>233 ± 7</td>
<td>0.4 ± 0.001</td>
</tr>
</tbody>
</table>
Table A2.18. Summary of the results of the monotonic tensile tests for the regular production and prototype V6 engine blocks in as-cast and heat treated conditions.

<table>
<thead>
<tr>
<th>Engine Block Characteristics</th>
<th>SDAS (µm)</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation (MPa)</th>
<th>Modulus of Elasticity (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0.2% Yield</td>
<td>UTS</td>
<td>0.2% Yield</td>
</tr>
<tr>
<td>Engine With Only Cast Iron Cylinder Liners (Heat Treated and Artificially Aged)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cast Iron As-Cast</td>
<td>59 to 63</td>
<td>229 ±10</td>
<td>243 ± 8</td>
<td>0.46 ± 0.04</td>
</tr>
<tr>
<td>Cast Iron Side As-Cast</td>
<td>58 to 62</td>
<td>220 ± 6</td>
<td>246 ± 7</td>
<td>0.5 ± 0.003</td>
</tr>
<tr>
<td>Cast Iron Heat Treated</td>
<td></td>
<td>248 ± 7</td>
<td>273 ± 9</td>
<td>0.5 ± 0.005</td>
</tr>
<tr>
<td>Al-Si Side As-Cast</td>
<td>53 to 59</td>
<td>228 ± 8</td>
<td>252 ± 13</td>
<td>0.5 ± 0.001</td>
</tr>
<tr>
<td>Al-Si Side Heat Treated</td>
<td></td>
<td>250 ± 7</td>
<td>272 ± 8</td>
<td>0.5 ± 0.004</td>
</tr>
<tr>
<td>Engine With the 390.1(I) Al-Si Alloy and Cast Iron Cylinder Liners</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cast Iron Side As-Cast</td>
<td>58 to 62</td>
<td>220 ± 6</td>
<td>225 ± 12</td>
<td>0.5 ± 0.001</td>
</tr>
<tr>
<td>Cast Iron Heat Treated</td>
<td></td>
<td>237 ± 8</td>
<td>262 ± 7</td>
<td>0.5 ± 0.002</td>
</tr>
<tr>
<td>Al-Si Side As-Cast</td>
<td>55 to 62</td>
<td>222 ± 9</td>
<td>245 ± 14</td>
<td>0.5 ± 0.008</td>
</tr>
<tr>
<td>Al-Si Side Heat Treated</td>
<td></td>
<td>246 ± 7</td>
<td>268 ± 10</td>
<td>0.5 ± 0.004</td>
</tr>
<tr>
<td>Engine With ESV Melt Treated 390.1(I) Al-Si Alloy and Cast Iron Cylinder Liners</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cast Iron Side As-Cast</td>
<td>54 to 60</td>
<td>226 ± 12</td>
<td>269 ± 10</td>
<td>0.5 ± 0.002</td>
</tr>
<tr>
<td>Cast Iron Heat Treated</td>
<td></td>
<td>237 ± 8</td>
<td>262 ± 7</td>
<td>0.5 ± 0.002</td>
</tr>
<tr>
<td>Al-Si Side As-Cast</td>
<td>55 to 60</td>
<td>213 ± 11</td>
<td>251 ± 8</td>
<td>0.5 ± 0.002</td>
</tr>
</tbody>
</table>
VITA AUCTORIS

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