Segregation in aluminum-silicon alloys.

Larry Stewart Ball
University of Windsor

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SEGREGATION IN ALUMINUM-SILICON ALLOYS

by

LARRY STEWART BALL

A Thesis
Submitted to the Faculty of Graduate Studies through the Department of Engineering Materials in Partial Fulfillment of the Requirements for the Degree of Master of Applied Science at University of Windsor

Windsor, Ontario
1969
UNIVERSITY OF WINDSOR
FACULTY OF GRADUATE STUDIES

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance, a thesis entitled, "Segregation in Aluminum-Silicon Alloys" submitted by Larry Stewart Ball in partial fulfillment of the requirements for the degree of Master of Applied Science.

Supervisor

Oct. 3, 1969

Date
ABSTRACT

Macrosegregation in ingots of aluminum (rich)-silicon alloys is investigated. The ingots were unidirectionally solidified under conditions of slow and fast cooling (corresponding to an applied pressure of 50 psi and normal atmospheric pressure). Samples from the ingots were analyzed for silicon content from which segregation profiles were constructed. The amount of chill face (maximum) segregation was only 25 per cent of that predicted by theory. It was concluded that this was largely due to thermal convective mixing of the interdendritic liquid. The density variation with composition is relatively small for aluminum-silicon alloys, so that the gravity effect counteracting thermal convective mixing is small. The convective mixing dilutes the enriched liquid feeding the solidification contraction volumes, thus reducing the degree of segregation.
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INTRODUCTION

The mechanical properties of alloy castings, and to a lesser extent wrought alloys, are influenced greatly by the solidification process; for the casting structure produced on solidification tends to be preserved and cannot be removed by any practical amount of heat treatment or mechanical working. Thus defects and inhomogeneities such as porosity, segregation, or excessive and non-uniform grain size must be controlled during solidification or be accepted in the final structure. These defects or inhomogeneities resulting from solidification can have deleterious effects on the mechanical properties and corrosion resistance of the casting. For example, chemical inhomogeneities resulting from segregation effects have a particularly pronounced influence on the fatigue strength and corrosion resistance of castings. It is therefore necessary, if we wish to produce sound castings, to have some understanding of the solidification and segregation phenomena which can occur in an alloy system.

An investigation of macrosegregation in aluminum-silicon alloy ingots was undertaken primarily for two reasons: (1) the obvious commercial importance of aluminum-silicon casting alloys. The aluminum casting industry is based largely on the aluminum-silicon alloys and their many variations. This is because of the excellent castability of the aluminum-silicon alloys, castability being defined as the property which deter-
mines the ease with which castings and ingots of sound internal structure can be produced. The high castability of commercial binary aluminum-silicon alloys can be attributed to the large volume of low-melting point eutectic which results from the addition of only a few weight per cent silicon. All eutectic liquids generally exhibit good fluidity, i.e., the ability to flow readily (particularly at temperatures slightly above the melting point), and this property of high fluidity of the eutectic liquid results in the good casting characteristics shown by the aluminum-silicon alloys.

(2) There are no reported investigations of macrosegregation occurring in aluminum-silicon alloy castings. Macrosegregation in castings has been the source of many foundry problems in a number of alloy systems, notably the aluminum-copper alloys. In view of the importance of the aluminum-silicon alloys to the non-ferrous casting industry, a study of the solidification segregation in the aluminum-silicon system is of practical as well as academic importance.

The experimental solidification of the alloys was carried out in a permanent mold under conditions of unidirectional solidification. This technique eliminated many of the complicating variables which are present in normal foundry processes. It was desired to relate the measured amount of segregation quantitatively to that predicted using the inverse segregation theory first developed by Kirkaldy and Youdelis, and successfully applied to quantitatively account for the segregation observed in a number of alloy systems. The mechanism proposed for the inverse segregation phenomena is one of flow-
back of enriched liquid through interdendritic channels to compensate for volume changes accompanying the solidification process.

II HISTORICAL
A. Theory

The various macrosegregation types encountered in solidification of binary alloys are all based on a common microsegregation mechanism, and so a brief consideration of the microsegregation mechanism is necessary. The macrosegregation (i.e. the difference in an incremental volume between the actual solute concentration $C$ and the mean solute concentration $C_o$) is a second order effect resulting from a combination of the microsegregation mechanism and the flow of liquid through interdendritic channels to compensate for the volume changes accompanying the liquid-solid transformation.

1. Microsegregation

The basis for the microsegregation mechanism is the formation of a solid phase differing in composition from that of the liquid phase. Consider the directional solidification of a binary alloy, of concentration $C_o$, which has a typical binary eutectic phase diagram as shown in Figure 1. The equilibrium relationship between the solid-liquid phases is defined by the equilibrium distribution coefficient $k_o = \frac{C_s}{C_l}$ (where $C_s$ and $C_l$ are the solute concentrations in the solid and liquid constituents respectively). For reasonable solidification rates it has been shown that local equilibrium exists between
Figure 1. Partial binary eutectic diagram

Figure 2. Solute distribution after directional solidification.

(1) Complete convective mixing in the liquid
(2) Mixing by diffusion only
(3) Partial mixing in the liquid
the solid and liquid phases at the solid-liquid interface. The final solute distribution in the solid is determined by the degree of homogenization in the liquid ahead of the advancing liquid-solid interface. This is justified because solute diffusion in the solid is of the order of $10^4$ less than that in the liquid, and thus can have no significant effect on the final solute distribution in the solid.

The degree of homogenization of the liquid is dependent on the amount of convective mixing and diffusion of the solute in the liquid. Figure 2 illustrates the possible solute distributions that may be obtained in directionally solidified ingots, providing a planar interface is maintained. If complete liquid homogenization is attained through vigorous convective mixing then the distribution will be that of curve (1). The equation for curve (1) was first derived by Pfann$^2$ and is as follows:

$$C_s = k_0 C_0 (1-g)^{k_0-1}$$  \hspace{1cm} (1)

where $g$ represents the fraction solidified. If there is no convective mixing in the liquid and homogenization in the liquid occurs by diffusion only, then the resulting distribution will be that shown by curve (2). The significant characteristic for this case is the macroscopically uniform (steady state) central region in the ingot. Tiller et al$^3$ and Smith et al$^4$ have derived mathematical expressions for the solute concentration during the initial and final transient portions of the curve. For the more important initial transient, the solute
distribution in the solid is given by:

$$C_s = C_0 \left\{ (1-k_0) \left[ 1 - \exp \left( \frac{-k_o R x}{D} \right) \right] + k_0 \right\}$$

(2)

where $R$ is the solidification rate, $D$ is the liquid diffusion coefficient, and $x$ is the distance from the original point of solidification.

For intermediate cases where mixing is partially by convection and partially by diffusion, curve (3) is obtained. In this case, Pfann's equation may still be used to calculate the solute distribution in the solid, but $k_0$ is replaced by $k_E$, the effective distribution coefficient. Burton et al have derived an expression for $k_E$, viz.;

$$k_E = \frac{k_0}{k_0 + (1-k_0) \exp \left( \frac{-R S}{D} \right)}$$

(3)

where $S$ is the effective thickness of the interfacial liquid undisturbed by convective mixing.

In general, for solidification rates less than 1 cm/hr the segregation distribution will approximate curve (1), while for rates in excess of 10 cm/hr, the segregation approaches that given by curve (2). Between 1 and 10 cm/hr, the segregation distribution will vary between the two extremes (i.e. curve (3)).

2. **Macrosegregation**

For the most rapid cooling rates that are generally used in the solidification of binary alloy ingots or castings, a condition of instability known as constitutional supercooling results at the solid-liquid interface. Because of the concentration gradient that develops ahead of an advancing solid-
liquid interface, the equilibrium solidification temperature $T_e$, of the liquid ahead of the interface, increases in the manner shown in Figure 3. $T_a$, the actual temperature gradient in the liquid, which is controlled by heat flow rates, will increase approximately linearly and can drop below $T_e$. The alloy is then said to be constitutionally supercooled, with the quantity $(T_e - T_a)$ representing the amount of supercooling. Under conditions of constitutional supercooling dendrite development results. A schematic perspective view of growing dendrites is shown in Figure 4. During the growth of dendrites the interdendritic liquid is enriched by the mechanisms of microsegregation discussed previously. Acting alone, the microsegregation mechanism could not result in any macrosegregation because the composition of the solute-depleted dendrites and the solute-enriched liquid average to give the mean alloy concentration. The macrosegregation that occurs in ingots is a combination of microsegregation and the flow of interdendritic liquid, the latter to compensate for volume changes in the liquid-solid zone accompanying solidification. The majority of alloys undergo contraction on solidification, so the solute-enriched interdendritic liquid feeds downwards towards the chill face, increasing the concentration above the mean alloy concentration. This positive segregation is frequently referred to as 'inverse segregation' because of earlier assumptions that predicted the first regions to solidify in an ingot should be lower in solute content. The opposite effect was found in most chill-cast alloys and hence the name 'inverse segregation' was coined. In the
Figure 3. Variation in liquid concentration and temperature ahead of an advancing interface.

Figure 4: Perspective view of growing dendrites.
case of bismuth-rich alloys, which expand on solidification, a negative or 'normal' segregation results from the same mechanism.

Another macrosegregation phenomena which causes much more serious problems in practice is termed surface exudations. Almost invariably the chill faces of a directionally solidified ingot show a surface layer of eutectic constituent in the form of either massive sweat beads or a thin continuous layer. The massive sweat beads occur when the ingot reheats during the early stages of solidification. Apparently as soon as the ingot base solidifies it withdraws slightly from the chill face due to thermal contraction, and thus good thermal contact between the ingot and the chill face is lost. This results in the reheating of the solid shell by the high-temperature liquid alloy, and if the reheating temperature exceeds the eutectic temperature, the interdendritic eutectic constituent remelts and is forced out onto the ingot surface through the interdendritic channels by expansion pressure, where it solidifies in the form of massive sweat beads. However, in pressure cast ingots, where reheating is generally avoided, the chill surfaces of the ingots may still show a thin uniform layer of eutectic constituent. In this latter case the exudation is the result of surface tension forces, which draw the eutectic liquid (before complete solidification) under the base of the primary dendrites.

3. Mathematical Formulation of Inverse Segregation

The derivation of the equations describing the inverse segregation mechanism to which the reader is referred for detailed consideration, has been given by Kirkaldy and Youde is...
and Youdelis. Only the assumptions made in the derivation, and the principal equations will be given here.

The assumptions on which the derivation is based are as follows:

1. there is complete flowback of interdendritic liquid so that no shrinkage porosity occurs.
2. local equilibrium exists at the solid-liquid interface.
3. there is negligible solid diffusion, i.e. essentially complete coring occurs in the primary solid crystals.
4. the interdendritic liquid is homogeneous and in equilibrium with the solid.

In a binary alloy of a eutectic system, the segregation at any point in the ingot is given by

$$
\Delta C = C - c_o = \frac{m_1}{a_E} \frac{C_l}{E} + m_s \frac{C_s}{E} - c_o
$$

where $C = \text{mean solute concentration at any point in the solidified ingot}$

$c_o = \text{initial liquid solute concentration}$

$m_{1E} = \text{mass of liquid at eutectic temperature}$

$m_{sE} = \text{mass of solid at eutectic temperature}$

$C_{sE} = \text{mean solute concentration in the cored primary crystals at the eutectic temperature}$

$a_E = \text{contraction coefficient}$

$C_s = \text{mean solute concentration in the cored primary crystals}$
By definition \( a_E = \frac{v_s}{v_l} \), where \( v_s \) and \( v_l \) are the specific volumes of the eutectic solid and liquid respectively. At concentrations other than the eutectic \( a \) is defined by

\[
a = \frac{v_s}{v_l} + \frac{C_l - C_s}{v_l} \frac{dV_l}{dC_l}
\]

For the case of maximum segregation occurring at the chill face the mass terms in the segregation equation are derived relatively easily, for there is no flow of residual liquid past the chill face. At points farther in the ingot, the segregation mechanism is more complicated, for residual liquid is flowing through the solidification zone to feed the contraction occurring within, and this has a diluting effect which decreases the segregation. We shall consider only the case of maximum chill face segregation for which the fundamental differential equations for the calculation of the mass terms are as follows:

\[
\frac{dm_l}{m_l} = -\frac{a}{A} \frac{dc_l}{c_l} \quad \quad (6)
\]

\[
\frac{dm_l}{m_l} = -adm_s 
\]

and (for the cored primary crystals),

\[
d(m_s C_s) = c_s \frac{dm_s}{m_s} = \frac{(1-A)}{A} m_l \frac{dc_l}{c_l}
\]

where \( A = \frac{C_l - C_s}{C_l} \)

On integration, the mass terms for the cored crystals are given by:
\[ m_{s_{i+1}} = m_{s_i} + \frac{m_{\ell_i} - m_{\ell_{i+1}}}{a_i} \] (9)

and

\[ m_{\ell_{i+1}} = m_{\ell_i} \left[ \frac{c_{\ell_{i+1}}}{c_{\ell_i}} \right]^{-a_i} \frac{A_i}{A_{i+1}} \] (10)

The solute content in the cored primary crystals is given by:

\[ m_{s_{i+1}} C_{s_{i+1}} = m_{s_i} C_{s_i} + \frac{(1 - A_i)}{(A_i - a_i)} m_{\ell_i} C_{\ell_i} \left[ \left( \frac{c_{\ell_{i+1}}}{c_{\ell_i}} \right) \frac{A_i}{A_{i+1}} - 1 \right] \] (11)

The subscripts \(i\) and \(i+1\) refer to the \(i\)th and \((i+1)\)th terms respectively in the stepwise integration of the equations, which is necessary since \(a\) and \(A\) are usually functions of solute concentration. The summation of terms in equations (9), (10) and (11), is taken to the eutectic temperature to obtain the mass terms \(m_{\ell_E}, m_{s_E}\) and \(m_{s_E} C_{s_E}\) in the equation for the calculation of the segregation.

**B. PRACTICAL CONSIDERATIONS IN ALUMINUM-SILICON ALLOYS**

The microstructure of the solid aluminum-silicon eutectic constituent can be modified by the addition of quite small amounts (in the order of 0.01%) of sodium. In the unmodified alloy the silicon phase takes the form of needles or plates which are sources of stress risers. The addition of the sodium modifies the plate-like silicon phase to spherical particles, thereby improving the tensile strength of the alloy. A somewhat similar effect can be obtained by sufficiently rapid cooling, such as is
observed in die castings. For example, if we consider an aluminum-8 per cent silicon alloy the tensile strength increases from 18,000 psi to 23,000 psi and the ductility from 5 per cent to 10 per cent elongation when the alloy is cast in the modified versus unmodified condition. 

In cases where a modified structure occurs it is observed that final solidification is terminated a few degrees below the equilibrium eutectic temperature. This observation has been made from the measurement of thermal arrests and has occurred in both modified alloys as well as rapidly cooled castings. When supercooling occurs in a system, it is generally assumed that the metastable equilibrium relationships are represented by the extension of the liquidus and solidus curves. For aluminum-rich alloys supercooling will result in a shift of the eutectic point to a lower temperature and a higher silicon concentration. The change in the eutectic point must be considered when calculating the macrosegregation. For example, in equation (4) the mass and concentration terms are all dependent on the eutectic concentration. The terms $A$ and $a$ are functions of the liquidus composition, and would be altered by a change in the position of the liquidus.
III EXPERIMENTAL

A. Materials

Super-purity aluminum was obtained from the Aluminum Company of Canada Limited. The analysis was as follows:
99.992 per cent Al, 0.002 per cent Cu, 0.002 per cent Fe,
0.002 per cent Si and 0.002 per cent Mg. Fused lumps of silicon of purity 99.7 per cent were supplied by British Drug Houses Limited.

B. Apparatus

The solidification experiments were carried out using two different techniques. The first group of ingots were cast in a permanent mold and solidified under pressure to circumvent the exudation problem. The second group of ingots were cast in a permanent mold in air under normal atmospheric conditions. In the former case a slower cooling rate was obtained because of the peculiar construction of the mold, while in the latter a more rapid solidification rate resulted. It was felt that the faster solidification rate (for the second group of ingots) might preclude the possibility of large thermal convective mixing effects. In both cases the ingots were solidified unidirectionally by impinging a water stream onto the base plate of the mold.

1. Pressure-casting Apparatus (Slow Cool)

Figure 5 is a schematic diagram of the pressure-casting solidification apparatus. Pressures of 5 atmospheres could be maintained at temperatures up to \(800^\circ C\).

The ingots were solidified in a stainless steel mold coated with a thin layer of alundum cement (to prevent any
Figure 5. Schematic Diagram of Solidification Apparatus For Casting Under Pressure.
liquid metal-mold reaction). The mold was set upon a polished steel base plate 2 inches diameter by 3/8 inches thickness. The dimensions of the mold were 1.80 inches inside diameter by 3 inches in height. To ensure good contact between mold and base plate a length of stainless steel tubing was placed on top of the mold and then tightened down by means of the steel cap which screwed onto the pressure assembly.

The pressure tight assembly surrounding the mold consisted of a 2 inch diameter cast iron pipe length with threaded ends, onto which steel caps were screwed on both the top and bottom of the assembly. A pressure tight seal was obtained at the bottom junction by using Aremco Products number 510 refractory cement. For the top cap a less permanent method was used. A copper cooling coil was utilized to keep the temperature low enough that a pressure tight seal could be maintained with teflon tape.

The mold top was designed to allow for the entrance of argon gas, thermocouples and the melt itself. The melt inlet tube consisted of a 1/2 inch diameter steel pipe which was threaded on the top end and welded to the cap. Teflon tape was employed to maintain a pressure tight seal when a threaded cap was screwed onto the inlet tube. A 1/4 inch diameter copper tube was silver soldered to the mold cap to provide a connection to an argon gas cylinder, and by means of a valve, to a mechanical vacuum pump. An entrance was made for three thermocouples by drilling a 1/2 inch diameter hole in the assembly cap, filling it with a cold mounting polyester resin, and then drilling holes in the hardened plastic through which the ceramic thermo-
couple protection tubes were passed. A seal around the thermo-
couple protection tubes was made using the Aremco refractory
cement mentioned previously. The space around the thermocouple
wires was sealed by forcing some of the cement approximately
1/4 inch down the protection tube and allowing it to harden.

The molten metal was poured into the mold from a
bottom-pouring graphite crucible (shown in Figure 6) in order
to minimize gas absorption by exposure of the molten stream to
the atmosphere. The crucible was machined from graphite and
was approximately 2 1/2 inches inside diameter by 3 1/2 inches
in height. A graphite plug situated in the center of the cru-
cible was removed when it was desired to pour the molten metal.
The molten metal was cast into the mold by pouring it through
a 10 inch length of graphite tubing (shown in Figure 6), which
was preheated to 1400°F before pouring.

The furnace surrounding the mold was constructed by
winding Kanthal Ribbon (3/16" by 0.010") around a sillimanite
core, and then embedding the windings in alundum cement. Re-
fractory bricks were stacked around the furnace core to provide
insulation. Power was supplied from 120 volt, 15 ampere mains
through a variac autotransformer which allowed the voltage to
be adjusted from 0-140 volts. This arrangement enabled tempera-
tures up to 800°C to be maintained with ease.

The ingots were solidified by directing a jet of
water against the bottom cap of the pressure assembly. The
water was supplied through a 1/2 inch diameter copper pipe.
The pressure was adjusted until the free stream of water
attained a height of roughly one inch in a vertical direction.
The steel base plate, on which the ingots were solidified, was highly polished on both sides to insure good thermal contact for cooling.

Temperature measurements were made at three positions in the ingot during solidification. 30 gauge chromel-alumel wires were used to make the thermocouples. The lengths of the wires extending into the ingot were sheathed in 1/16 inch outside diameter ceramic protection tubes. The thermocouples were positioned as follows: number (1) in contact with the ingot base, number (2) a distance of 3/4 inches up from the chill plate, and number (3) a distance of 1 1/2 inches up from the chill plate. The recordings of the temperature changes were made using a 2-channel model D11A/U Westronics recorder and a single channel Bausch and Lomb recorder. The Bausch and Lomb recorder was set for a range of from 0-100 millivolts full scale. The first channel of the Westronics recorder was set at 0-1000°C full scale, and the second channel at 0-30 millivolts full scale.

2. Atmospheric Pressure Casting Apparatus (Fast Cool)

A schematic sketch of the apparatus is shown in Figure 6. The mold consisted of a refractory brick, 4 inches square by 2 1/2 inches in height, with a two inch diameter hole drilled in the center. The hole was lined with a layer of alundum cement with a slight downward taper to facilitate the removal of the ingot from the mold. The brick was placed on a polished steel chill plate 3/16 of an inch thick. The water supply for cooling was the same as that described previously for use in the pressure-
Figure 6. Sketch of Apparatus for Solidification Under Atmospheric Pressure.
casting assembly. The melt was poured into the mold using the bottom-pouring crucible sketched in Figure 6.

C. Procedure

1. Alloy Preparation

The super-purity aluminum was cleaned in an aqueous solution of 10 per cent sodium hydroxide. Approximately 250 grams of the cleaned aluminum was melted in fireclay crucibles lined with a coating of alundum cement to prevent the molten aluminum from absorbing silica from the walls of the crucible. The required amount of silicon was then placed in the bottom of a cylindrical graphite crucible over which the molten aluminum was poured. The crucible was then transferred to the Phillips high frequency induction furnace. A water cooled copper coil, supplying 6 to 7 kilowatts of power, was used to heat the crucible and the melt. The power was applied for ten minutes, allowing the solid silicon to alloy with the melt and also providing a significant stirring action to homogenize the alloy. The molten alloy was then held at 1400°F in a muffle furnace for 12 hours to insure complete assimilation of the silicon before solidification. Analyses of the ingots showed no measurable loss of silicon in the preparation of the ingots.

2. Solidification

The ingots were remelted in an electric muffle furnace at 1400°F. Since molten aluminum has a marked tendency to dissolve hydrogen gas from the moisture in the air (as much as 17 per cent by volume), it was necessary to degas the melt by bubbling argon gas through it. This method of degassing
provides an unsaturated gas interface into which the dissolved gas is absorbed and carried to the surface. The bubbling was accomplished by immersing a stainless steel tube into the melt and flowing argon through it at a controlled rate. The tube was removed intermittently to prevent attack by the molten alloy.

Each alloy was degassed twice prior to casting. For the first degassing the crucible containing the molten alloy was removed from the muffle furnace and degassing carried out until the alloy had solidified. The ingot was then remelted at 1400°F under quiescent conditions so that the surface oxide film remained intact. The metal was then transferred to the bottom-pouring graphite crucible (see Figure 6), where it was degassed again just prior to pouring. For the pressure cast ingots the assembly was evacuated to $10^{-2}$ mm with a mechanical vacuum pump, filled with argon at atmospheric pressure, and then raised to a temperature of 750°C. After the desired temperature was attained the graphite pouring tube, preheated to prevent premature solidification during pouring, was placed into position (shown in Figure 5). The melt was released into the mold with a constant superheat of about 50°C. The mold was re-evacuated and argon admitted until a positive pressure of 50 psi was attained. The furnace power was then cut off and the cooling water applied. During solidification the temperatures of the thermocouples were continuously recorded.

The ingots cast under atmospheric pressure were poured directly from the bottom-pouring crucible with a constant superheat of 50°C. Temperature measurements were made at a position in the firebrick to insure that solidification did not take
place prematurely. Immediately after pouring the cooling water was applied.

3. Sampling

To obtain a representative sample for determination of the mean alloy composition, the swarf from a hack saw cut down the centre of the ingot was collected. Since the segregation in the ingot varied in the vertical direction, this method is the best means for determining the mean composition. One half of the sectioned ingot was used for metallographic examination while further samples for chemical analysis were sectioned from the remaining half. The samples taken from the chill face were lathe cut turnings, 0.050 inches in thickness, taken parallel to the ingot base. Four samples were machined progressively from the base of the ingot so that the segregation at the chill face could be determined from an extrapolation of the segregation values (or their averaging) near the chill face. The samples at various heights in the ingot were obtained by collecting the swarf from the hack saw cuts made parallel to the ingot base. The thickness of each cut was approximately 0.035 inches.

4. Analysis

The samples collected from the lathe and saw cuts were analyzed by Chicago Spectro Service Laboratories, Inc. for silicon content. The analysis was carried out by a wet chemical technique and the results reported to the nearest one-hundredth of a per cent.
5. Metallography

One half of each ingot was rough polished on silicon carbide paper to 320 grit size and etched with Tucker's etch for macrostructure examination.

Microscropic examination for exudations was carried out on sections adjacent to the chill face. It was essential to maintain good edge preservation on the sample to observe the extent of surface exudations. Mounting in a 'short glass fibre' (diallyl phthalate) type of thermoplastic mounting medium achieved the desired results.

IV RESULTS

A. Metallographic

The macrostructures of three typical slow-cooled ingots representing alloys of 3, 6 and 9 weight per cent silicon are shown in Figures 7 to 9. The microstructures of the dendritic growth patterns at the chill faces of the ingots are shown in Figures 13 to 15.

The macrostructures of the fast-cooled ingots (3, 8 and 9 weight per cent silicon) are shown in Figures 10 to 12; the microstructures of the chill faces are shown in Figures 16 to 18.

B. Segregation Analysis

The analytical results for the slow-cool and fast-cool ingots, as reported by Chicago Spectro Service Laboratory, Inc., are given in Appendix I. For the slow-cool ingots eight samples were taken throughout the length of the ingot. However,
Figure 7. Macrostructure of Al-3% Si Ingot Solidified under pressure of 50 psi (XI.5).

Figure 8. Macrostructure of Al-6% Si Ingot Solidified Under Pressure of 50 psi (XI.5).

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Figure 9. Macrostructure of Al-9% Si Ingot Solidified Under Pressure of 50 psi (XI.5).

Figure 10. Macrostructure of Al-3% Si Ingot Solidified Under Atmospheric Pressure (XI.2).

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Figure 11. Macrostructure of Al-8% Si Ingot Solidified Under Atmospheric Pressure (X1.8).

Figure 12. Macrostructure of Al-9% Si Ingot Solidified Under Atmospheric Pressure (X1.5).
Figure 13. Microstructure of Base of Al-3% Si Ingot Solidified Under Pressure of 50 psi (X75).

Figure 14. Microstructure of Base of Al-6% Si Ingot Solidified Under Pressure of 50 psi (X75).
Figure 15. Microstructure of Base of Al-9% Si Ingot Solidified Under Pressure of 50 psi (X75).

Figure 16. Microstructure of Base of Al-3% Si Ingot Solidified Under Atmospheric Pressure (X75).

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Figure 17. Microstructure of Base of Al-8% Si Ingot Solidified Under Atmospheric Pressure (X75).

Figure 18. Microstructure of Base of Al-9% Si Ingot Solidified Under Atmospheric Pressure (X75).

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Figure 19-a. Constitutional - Specific Volume Relationships for Al-Si System.
for the fast-cool ingots, only four samples adjacent to the chill face were analyzed. The maximum segregation values are listed in Appendix II. These values were obtained by computing the average silicon composition of the four samples machined progressively from the chill face, and therefore they represent the mean composition at 0.010 inches from the chill face.

The experimental results are plotted in Figure 19, along with the theoretical maximum segregation curve as a function of silicon content. The theoretical curve was determined using a computer program (see Appendix III) adapted from that of Cahoon and Youdelis. The constitutional-specific volume data required for the calculations are shown in Figure 19-a.

V DISCUSSION

The effect of the faster cooling rate was to refine the columnar grain structure as is evident in comparing Figures 7 to 9 with Figures 10 to 12. This is to be expected, as higher solidification rates generally increase the nucleation rate and result in finer grain structures in castings. The columnar grain structure indicates that solidification was unidirectional as assumed in the theory. The photomacrographs exhibit negligible porosity indicating that essentially complete feeding of the contraction volumes takes place during solidification.

The most notable feature in the photomicrographs (Figures 13 to 18) is the absence of massive exudations of silicon-rich (eutectic) constituent at the chill face. As discussed earlier, exudations are the result of a mechanism that is not part of the solidification process resulting in inverse segre-
gation, and if present, corrections are necessary to obtain the true maximum inverse segregation values for the ingots. The absence of exudations indicates that no reheating interrupted the solidification process in the early stages. Figure 20 shows a typical exudation microstructure that does occur in aluminum-silicon ingots when reheating is present.

It is evident, from the positional variation of the segregation throughout the ingots (see Appendix I), that the maximum chill face segregation values cannot be determined by extrapolating the composition curves to the chill faces. To obtain a value for the maximum segregation occurring at the chill face, four samples taken progressively from the chill face were averaged. Since the composition curves are virtually flat in the vicinity of the chill face, and since no massive surface exudations were observed on the chill faces of both the slow and fast-cooled ingots, no extrapolation or exudation corrections were necessary for the chill face segregation values. Figure 19 shows that the experimental maximum chill face segregation values for both the slow and fast-cool ingots are approximately 25 per cent of those predicted by theory. Also, it is noted that there is one value of maximum segregation that is negative. This is inconsistent with the other results, and is most probably due to an error in the analysis. Also, positional variation of segregation exhibits considerable scatter (e.g. slow-cool ingots). These results indicate that some mechanism not accounted for in the proposed inverse segregation theory is operating during solidification of aluminum-silicon alloys, or that the constitutional-specific volume data used...
Figure 20. Microstructure of Base of Al-3% Si Ingot Showing Massive Surface Exudations (X100).
Figure 22 - Maximum Segregation Curves Corresponding to Changes in Constitutional - Specific Volume Relationships
In the calculation is in error.

In an attempt to account for the discrepancy between theory and experiment, several changes were made in the equilibrium phase diagram and specific volume data, and the theoretical segregation recalculated. The changes made were as follows:

1. Changes in the eutectic point and phase diagram as a possible result of supercooling
2. Changes in the position of the aluminum-rich liquidus curve, and
3. Changes in the specific volume curves.

The various changes in the phase diagram and specific volume data that were considered are shown in Figure 21 and the maximum segregation curves corresponding to the changes are given in Figure 22. Each of the three cases will be discussed briefly.

1. Supercooling

It has been noted previously that aluminum-silicon eutectic alloys show appreciable supercooling for rapid solidification rates. It is generally accepted that for supercooling the temperature-composition relationships for the alloy system are represented by the extension of the liquidus and solidus curves. In Figure 21 the solidus and liquidus lines have been extended to correspond to a supercooling of 25°C and 50°C. The corresponding specific volume data is obtained by extrapolating the equilibrium curves as shown in Figure 21. The data from the modified constitutional-specific volume curves were fed into the computer for calculation of the maximum segregation.
From Figure 22 it is evident that supercooling cannot account for the difference in maximum segregation.

(2) Change in position of the liquidus curve

If the position of the liquidus curve for the aluminum-silicon system as reported in the literature is in error, there will be corresponding errors in the calculated theoretical segregation results. To determine the effect of a change in the position of the liquidus curves on segregation, the maximum segregation values were calculated for the hypothetical liquidus curves (A) and (B) (shown in Figure 21), without altering the positions of the specific volume curves. It is noted that only when the liquidus curve is lowered that a corresponding decrease occurs in segregation. However, the segregation curve is lowered by only 20 per cent for the correspondingly large drop in the liquidus curve, an error which is not considered probable in the determination of the phase diagram.

(3) Change in specific volume data

The liquid specific volume curve was altered to the positions (1) and (2) (shown in Figure 21). These changes were considered because the only source of data for the specific volume curves was the work of Edwards. Because of the experimental difficulties attendant in determining the specific volume of (high temperature) liquids, and because of the lack of any corroborative evidence, for the specific volume data, there is a possibility of error in the data reported by Edwards. The changes result in a decrease in maximum segregation of about 25 per cent for position (1), and for position (2) a slight negative segregation occurs for silicon concentra-
tions above 8 weight per cent.

The various changes in the segregation curves corresponding to the changes in the constitutional and specific volume data are shown in Figure 22. It is evident that none of the above changes satisfactorily account for the difference between theoretical predictions and the measured experimental values for maximum segregation. Thus it must be concluded that some mechanism is operative in the solidification process other than the singular mechanism of flowback of enriched liquid along interdendritic channels to feed the solidification shrinkage volumes. One possibility which deserves a more thorough consideration is the role of convective mixing in the solidification process.

**Convective Mixing**

Convective mixing in a solidifying ingot results from density differences due to temperature gradients and composition variations in the liquid at the solid-liquid interface. Density differences resulting from the rejection of solute at a solid-liquid interface occur on a microscopic scale, i.e. over a distance approximately equal to the interdendritic spacing. On the other hand thermal gradients occur over distances comparable with the size of the ingot itself. The inverse segregation theory assumes complete diffusional mixing in the interdendritic spaces at planes of constant height above the ingot base, i.e. on isothermal planes in the solid-liquid region. It is assumed in the theory that no appreciable convective mixing occurs throughout the mushy zone, i.e. there is a
concentration gradient in the liquid along the length of the solid-liquid zone. The concentration is maximum at the bottom of the zone, and decreases to the mean alloy concentration \( C_q \) at the top of the zone (dendrite tips) as shown in Figure 23. This is a valid assumption provided the density of the enriched liquid is higher than that of the more dilute liquid alloy, so that gravity forces operate to greatly minimize, if not effectively prevent, (vertical) convective mixing. When this criteria is applied to the aluminum-silicon system the maximum density change that occurs is only 4 per cent, and even this is in some doubt. This is considerably less than the corresponding values of 35 per cent and 150 per cent for the aluminum-copper and aluminum-zinc systems. Since the inverse segregation theory has accurately predicted the quantitative segregation occurring in these latter systems, it would appear that this is a key factor for its failure in the case of aluminum-silicon. For an explanation as to why the enriched interdendritic liquid does not preferentially move to the bottom of the solid-liquid zone, one must examine further the role of convective mixing in the solidification process.

The effects of convective mixing have been studied by several investigators. Cole and Winegard\(^{15}\) measured large and rapid temperature oscillations ahead of the solid-liquid interface in horizontal melts of tin-lead alloys, for which the temperature gradients were relatively small (1°C per cm to 12°C per cm). This provides direct evidence of convective mixing in the melt, resulting from even the relatively small density changes accompanying the temperature differences. The
Figure 23. Average solid-liquid distribution in solidification zone

(a) morphology
(b) schematic liquid concentration distribution
composition effect was negligible since lead was present in small impurity concentrations (0.002 to 0.01 wt. per cent). No convective mixing was detected at temperature gradients less than 1°C per cm. They concluded that thermal convection is of greater significance than convection arising from concentration gradients in alloys of low impurity concentrations. MacAuley and Weinberg have reported appreciable convective flow in pure liquid tin when temperature gradients as small as 0.2°C per cm are present. The preceding studies emphasize the importance of thermal convection during the solidification process in alloy systems.

As indicated above there is very little density increase with silicon concentration increase for aluminum-silicon alloys (liquid). Thus there is a relatively small gravity effect acting to counteract convective mixing in the mushy zone due to temperature differences. As a result there is a strong tendency for convective mixing to operate during the solidification process. In this situation very little or no enriched liquid preferentially moves to the lower solidifying regions of the ingot, but rather there is a tendency for homogenization of the liquid throughout the solidification zone. This action accounts for the low experimental values of maximum chill face segregation; and for the rather scattered segregation values throughout the ingot.
VI. SUMMARY AND CONCLUSIONS

(1) The amount of inverse segregation at the chill face in ingots of aluminum (rich)-silicon alloys was only 25 per cent of that predicted by theory.

(2) The discrepancy between experiment and theory cannot be attributed to possible errors in constitutional diagram or specific volume data for the aluminum-silicon system.

(3) The variation of the liquid specific volume with alloy concentration is relatively small for the aluminum-silicon system compared to the aluminum-copper and aluminum-zinc systems. For composition variations from pure (liquid) aluminum up to the eutectic points, the density increases approximately 4% for the aluminum-silicon system, compared to 35% and 150% for the aluminum-copper and aluminum-zinc systems respectively. The latter two systems exhibit a large segregation tendency in agreement with theory.

(4) It is proposed that for aluminum-silicon alloys convective mixing due to thermal gradients is considerable in the solid-liquid region, since gravity forces are relatively small. The convective mixing dilutes the enriched liquid that flows back to feed contraction volumes, and thus decreases the maximum segregation that would be possible if no convective mixing occurred.

(5) The results for the slow and fast-cooled ingots are essentially identical indicating that thermal convective mixing is occurring continually during solidification as an integral part of the process.
REFERENCES

8 Archer, R. S. and Kempf, L.W., Trans. AIME, 73, 587, (1926).
APPENDIX I

RESULTS OF SILICON ANALYSES
Table 1. Silicon Concentrations for Slow-Cool Ingots (Solidified Under Pressure of 50 psi).

A. Ingot #22

<table>
<thead>
<tr>
<th>Distance from Chill Face (in inches)</th>
<th>Wt. per cent Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.025</td>
<td>3.28</td>
</tr>
<tr>
<td>0.075</td>
<td>2.88</td>
</tr>
<tr>
<td>0.125</td>
<td>2.92</td>
</tr>
<tr>
<td>0.175</td>
<td>3.04</td>
</tr>
<tr>
<td>0.55</td>
<td>3.24</td>
</tr>
<tr>
<td>1.02</td>
<td>2.94</td>
</tr>
<tr>
<td>1.60</td>
<td>2.96</td>
</tr>
<tr>
<td>2.20</td>
<td>3.08</td>
</tr>
</tbody>
</table>

Average Composition: 2.98

B. Ingot #24

<table>
<thead>
<tr>
<th>Distance from Chill Face (in inches)</th>
<th>Wt. per cent Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.025</td>
<td>6.00</td>
</tr>
<tr>
<td>0.075</td>
<td>6.02</td>
</tr>
<tr>
<td>0.125</td>
<td>5.94</td>
</tr>
<tr>
<td>0.175</td>
<td>5.94</td>
</tr>
<tr>
<td>0.48</td>
<td>5.86</td>
</tr>
<tr>
<td>1.11</td>
<td>5.96</td>
</tr>
<tr>
<td>1.71</td>
<td>5.90</td>
</tr>
</tbody>
</table>

Average Composition: 5.92

C. Ingot #23

<table>
<thead>
<tr>
<th>Distance from Chill Face (in inches)</th>
<th>Wt. per cent Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.025</td>
<td>8.70</td>
</tr>
<tr>
<td>0.075</td>
<td>8.80</td>
</tr>
<tr>
<td>0.125</td>
<td>8.72</td>
</tr>
<tr>
<td>0.175</td>
<td>8.70</td>
</tr>
<tr>
<td>0.53</td>
<td>8.66</td>
</tr>
<tr>
<td>1.01</td>
<td>8.80</td>
</tr>
<tr>
<td>1.51</td>
<td>8.70</td>
</tr>
<tr>
<td>2.12</td>
<td>8.76</td>
</tr>
</tbody>
</table>

Average Composition: 8.69
Table 2. Silicon Concentrations for Fast-Cool Ingots (Solidified Under Atmospheric Pressure).

<table>
<thead>
<tr>
<th>Ingot Number</th>
<th>Average Composition (Wt. % Si)</th>
<th>Compositions at Various Distances (in inches) from Chill Face</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0.025  0.075  0.125  0.175</td>
</tr>
<tr>
<td>B7</td>
<td>2.89</td>
<td>2.96   2.92   2.88    2.90</td>
</tr>
<tr>
<td>B8</td>
<td>8.72</td>
<td>8.76   8.70   8.74    8.78</td>
</tr>
<tr>
<td>B10</td>
<td>7.79</td>
<td>7.84   7.76   7.62    7.74</td>
</tr>
</tbody>
</table>

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APPENDIX II

CHILL FACE SEGREGATION RESULTS
Table 3. Chill Face (Maximum) Segregation Values for Slow-Cool and Fast-Cool Ingots.

<table>
<thead>
<tr>
<th>Ingot Number</th>
<th>Average Composition</th>
<th>Chill Face Concentration (avg. of 4 samples)</th>
<th>Segregation (Wt. % Si)</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>2.98</td>
<td>3.03</td>
<td>0.05</td>
</tr>
<tr>
<td>24</td>
<td>5.92</td>
<td>5.98</td>
<td>0.06</td>
</tr>
<tr>
<td>23</td>
<td>8.69</td>
<td>8.73</td>
<td>0.04</td>
</tr>
<tr>
<td>B7</td>
<td>2.89</td>
<td>2.92</td>
<td>0.03</td>
</tr>
<tr>
<td>B10</td>
<td>7.79</td>
<td>7.74</td>
<td>-0.05</td>
</tr>
<tr>
<td>B8</td>
<td>8.72</td>
<td>8.75</td>
<td>0.03</td>
</tr>
</tbody>
</table>
APPENDIX III

COMPUTER PROGRAM FOR MAXIMUM SEGREGATION
CHANGES IN NOTATION TO FACILITATE PROGRAMMING

<table>
<thead>
<tr>
<th>Original Notation</th>
<th>Program Notation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$m_{l_1}$</td>
<td>GL(I)</td>
</tr>
<tr>
<td>$m_{s_1}$</td>
<td>GS(I)</td>
</tr>
<tr>
<td>$a_{i}$</td>
<td>P(I)</td>
</tr>
<tr>
<td>$A_{i}$</td>
<td>A(I)</td>
</tr>
<tr>
<td>$a_E$</td>
<td>AE</td>
</tr>
<tr>
<td>$c_{L_1}$</td>
<td>CE</td>
</tr>
<tr>
<td>$dv_{1}/dc_{l}$</td>
<td>DVL</td>
</tr>
<tr>
<td>$c_{l_1}$</td>
<td>CL(I)</td>
</tr>
<tr>
<td>$c_{si}$</td>
<td>CS(I)</td>
</tr>
<tr>
<td>$v_{l_1}$</td>
<td>VL(I)</td>
</tr>
<tr>
<td>$v_{s_1}$</td>
<td>VS(I)</td>
</tr>
</tbody>
</table>

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DIMENSION CL(15), CS(15), VL(15), VS(15)
DIMENSION P(15), A(15), CLC(15), GL(15), B(15), D(15)
DIMENSION AX(20), BX(20), CX(20), AY(20), BY(20), CY(20)
DIMENSION AZ(20), BZ(20), CZ(20)

2 READ 1, N, AE, CE, H
PUNCH 1, N, AE, CE, H

77 READ 3, (CL(I), CS(I), VL(I), VS(I), I=1, N)
PUNCH 3, (CL(I), CS(I), VL(I), VS(I), I=1, N)
PUNCH 103
I=1
JK=1

50 BX(JK)=(VL(I+1)-VL(I))*(CL(I+2)**2-CL(I)**2)
BX(JK)=BX(JK)-(VL(I+2)-VL(I))*(CL(I+1)**2-CL(I)**2)
DIV1=(CL(I+2)-CL(I))*(CL(I+2)-CL(I+1))*(CL(I+1)-CL(I))
BX(JK)=BX(JK)/DIV1
AX(JK)=(VL(I+1)-AX(JK)*CL(I+1)**2-BX(JK)*CL(I))
BY(JK)=(VS(I+1)-BY(JK)**2-CL(I)**2)
BY(JK)=BY(JK)/DIV1
AY(JK)=AY(JK)/DIV2
CX(JK)=VL(I)-AX(JK)*CL(I+1)**2-CX(JK)

51 IJ=0
JK=1
MI=M-1
IF (M) 51, 51, 50

54 AM=CLU-CL(N)
IF (AM) 62, 53, 53

62 VLU=AX(JK)*CLU**2+BX(JK)*CLU+CX(JK)
CSU=AY(JK)*CLU**2+BY(JK)*CLU+CY(JK)
VSU=AZ(JK)*CLU**2+BZ(JK)*CLU+CZ(JK)
IJ=IJ+1
A(IJ)=(CLU-CSU)/CLU
P(IJ)=VSU/VLU+(2.*AX(JK)*CLU+BX(JK))*(CLU-CSU)/VLU
PUNCH 100, IJ, A(IJ), IJ, P(IJ)
CLU=CLU+H
GO TO 54

53 JK=JK+1
I=I+2
GO TO 54
55 DO 56 I=1,N
   BI=(A(I)-P(I))/A(I)
56 D(I)=-P(I)/A(I)

PUNCH 103
DO 57 I=1,IJ
   BI=I
57 CLC(I)=CLC(I)+BI*H
   JJ=IJ-1
   J=1

60 GS=0.0
   CSMS=0.0
   DO 58 I=J,JJ
      GL(J)=1.0
      GL(I+1)=GL(I)*(CLC(I+1)/CLC(I))**D(I+1)
      GS=GS+((GL(I)-GL(I+1))/P(I+1))
   CSMS1=((1.-A(I+1))/(A(I+1)-P(I+1)))*CLC(I)*GL(I)
58 CSMS=CSMS+CSMS1*(((CLC(I+1)/CLC(I))**B(I+1))-1.0)
   SEG=(((CE*GL(IJ)/AE)+CSMS)/(GL(IJ)/AE+GS))-CLC(J)
   SEG=SEG*100.
   CLP=CLC(J)*100.
   PUNCH 101,CLP,SEG
   PUNCH 102,CLP,SEG
   PUNCH 103
   J=J+1
   M+J-IJ
   IF (M) 60,2,2

76 CALL EXIT

1 FORMAT (I5,3F10.5)
3 FORMAT (4F10.5)
100 FORMAT (3X,1HAI3,1H=F9.5,3X1HPI3,1H=F9.5)
101 FORMAT (3X,9HAV. CONC.=F8.3,5H PCT.,3X,5HSEG.=F7.3,5H PCT.)
102 FORMAT (3X,4HMLE=F9.5,3X,4HMSE=F9.5,3X,6HCMSSE=F9.5)
103 FORMAT (1H )

END
VITA AUCTORIS

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