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Micro-mechanisms of Surface Defects Induced on Aluminum Alloys during Plastic Deformation at Elevated Temperatures

By

Olufisayo A. Gali

A Dissertation
Submitted to the Faculty of Graduate Studies through the Department of Mechanical, Automotive & Materials Engineering in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy at the University of Windsor

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I. Co-Authorship Declaration

I hereby declare that this thesis incorporates documentation of research performed by the author under the supervision of Dr. A.R. Riahi and Dr. J.A. Hunter. The design of the experiments and the collection of electron images in Chapter 1 were performed jointly by Mr. Q. Zhao (University of Windsor) and the author. The interpretation of the images and further analysis were carried out by the author himself. Dr. M. Shafiei (Novelis Global Research & Technology Center) assisted with the provisions of samples and an industrial perspective during discussions of the results analyzed by the author.

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ABSTRACT

Near-surface deformed layers developed on aluminum alloys significantly influence the corrosion and tribological behavior as well as reduce the surface quality of the rolled aluminum. The evolution of the near-surface microstructures induced on magnesium containing aluminum alloys during thermomechanical processing has been investigated with the aim generating an understanding of the influence of individual forming parameters on its evolution and examine the microstructure of the roll coating induced on the mating steel roll through material transfer during rolling. The micro-mechanisms related to the various features of near-surface microstructure developed during tribological conditions of the simulated hot rolling process were identified.

Thermomechanical processing experiments were performed with the aid of hot rolling (operating temperature: 550 to 460 °C, 4, 10 and 20 rolling pass schedules) and hot forming (operating temperature: 350 to 545 °C, strain rate: $4 \times 10^{-2} \text{s}^{-1}$) tribo-simulators. The surface, near-surface features and material transfer induced during the elevated temperature plastic deformation were examined and characterized employing optical interferometry, SEM/EDS, FIB and TEM.

Near-surface features characterized on the rolled aluminum alloys included; cracks, fractured intermetallic particles, aluminum nano-particles, oxide decorated grain boundaries, rolled-in oxides, shingles and blisters. These features were related to various individual rolling parameters which included, the work roll roughness, which induced the formation of shingles, rolling marks and were responsible for the redistribution of surface oxide and the enhancements of the
depth of the near-surface damage. The enhanced stresses and strains experienced during rolling were related to the formation and propagation of cracks, the nanocrystalline structure of the near-surface layers and aluminum nano-particles.

The mechanism of the evolution of the near-surface microstructure were determined to include grain boundary sliding which induced the cracks at the surface and subsurface of the alloy, magnesium diffusion to free surfaces, crack propagation from shear stresses and the shear strains inducing the nanocrystalline grain structure, the formation of shingles by the shear deformation of micro-wedges induced by the work roll grooves, and the deformation of this oxide covered micro-wedges inducing the rolled-in oxides. Magnesium diffusion to free surfaces was identified as inducing crack healing due to the formation of MgO within cracks and was responsible for the oxide decorated grain boundaries.

An examination of the roll coating revealed a complex layered microstructure that was induced through tribo-chemical and mechanical entrapment mechanisms. The microstructure of the roll coating suggested that the work roll material and the rolled aluminum alloy were essential in determining its composition and structure.

Subsequent hot forming processes revealed the rich oxide-layer of the near-surface microstructure was beneficial for reducing the coefficient of friction during tribological contact with the steel die. Damage to the microstructure include cracks induced from grain boundary sliding of near-surface grains and the formation of oxide fibres within cracks of the near-surface deformed layers.
DEDICATION

To the giants, who were kind enough to lift me up

The light bearers, who continued to light my way

The oceans of enlightenment, who let me drink from their wisdom

I am humbled and truly grateful,

To my late grandparents Mr. & Mrs. F.O.C. Akinpelu,

Your boundless love continues to sustain us

AND

To my nephews, who continue to bring meaning to all my endeavours,

FINALLY

To my every growing family, both foreign and domestic

Thank you for making it all worth it
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NOMENCLATURE

\( \mu \) \hspace{1cm} \text{COF}

\( A_0 \) \hspace{1cm} \text{Contact Area}

G \hspace{1cm} \text{Torque}

P \hspace{1cm} \text{Rolling force}

R \hspace{1cm} \text{Radius of the work roll}

Tm \hspace{1cm} \text{Melting Temperature}

ACOM-TEM \hspace{1cm} \text{Automated crystallographic orientation mapping - transmission electron microscope}

ASL \hspace{1cm} \text{Altered Surface Layers}

BSE \hspace{1cm} \text{Backscattered Electron}

CGBS \hspace{1cm} \text{Cooperative Grain Boundary Sliding}

COF \hspace{1cm} \text{Coefficient of Friction}

DLC \hspace{1cm} \text{Diamond-like-carbon}

EELS \hspace{1cm} \text{Electron Energy Loss Spectroscopy}

EDS \hspace{1cm} \text{Energy Dispersive X-Ray Spectroscopy}

EPMA \hspace{1cm} \text{Electron Probe Micro-Analyzer}

ESEM \hspace{1cm} \text{Environmental Scanning Electron Microscope}

FEM \hspace{1cm} \text{Field Emission Microscopy}

FCC \hspace{1cm} \text{Face Centre Cubic}

FFC \hspace{1cm} \text{Filiform Corrosion}

FIB \hspace{1cm} \text{Focused Ion Beam}
GBS  Grain Boundary Sliding
HR-SEM  High Resolution Scanning Electron Microscopy
HRTEM  High Resolution Transmission Electron Microscopy
MML  Mechanically Mixed Layers
NH-DLC  Non-Hydrogenated Diamond-Like-Carbon
NSM  Near-Surface Microstructure
PVD  Physical Vapour Deposition
RD  Rolling Direction
SAED  Selected Area Electron Diffraction
SEM  Scanning Electron Microscopy
SPD  Severe Plastic Deformation
STEM  Scanning Transmission Electron Microscopy
TEM  Transmission Electron Microscopy
TR  Total Reflectance
VSI  Vertical Scanning Interferometry
XPS  X-ray Photoelectron Spectroscopy
CHAPTER 1
INTRODUCTION

1.1 GENERAL OVERVIEW

Aluminum alloys have continued to find wide-spread commercial application in numerous industries as the wide range of available aluminum alloys and their vastly varied mechanical properties allow these alloys to fulfil the lightweight and strength requirements of manufacturers [1–5]. For example, aluminum alloys of the 1000 (Al), 3000 (Al-Mn), and 5000 (Al-Mg) series are widely used in the packaging industry, 6000 (Al-Mg-Si) series in construction, 5000 and 6000 series in the automotive, 2000 (Al-Cu) and 7000 (Al-Zn) series in aerospace, and the 5000 series in marine industries [1–6]. In fact, aluminum alloys are considered the most heavily used non-ferrous metals in the world, substituting for steel sheets in the automotive and aerospace industries [7–9]. Aluminum alloys also offer excellent corrosion resistance, weldability, recycling potential, high strength, high ductility, and good formability [9–11]. Due to their low density, high strength to weight ratios, and low thermal expansion, aluminum alloys are employed in areas where weight reduction is of critical concern [4,5,11,12]. The increased interest of the automotive industry in aluminum alloys, not just as body parts but extending their application to the engine block, has led to the increase in the demand for high quality aluminum sheets [13].

The properties of aluminum alloys are not just influenced by their alloying elements, but for wrought alloys the thermomechanical processing route is also of great importance [1,8]. The thermomechanical processing of aluminum alloys improve their
metallurgical properties and allow for the production of simple to complex product shapes [8,14,15]. These processes take advantage of the lower flow stress, work hardening, and springback, as well as the increased ductility, toughness, and formability experienced by aluminum alloys at elevated temperatures [16–19]. Elevated temperature forming processes, which include rolling, forming, stamping and extrusion, can be classified into hot working, which are forming operations above 0.6 Tm, where Tm is the melting temperature; and warm working, forming operations between 0.3 – 0.5 Tm [8,20]. During forming processes, the forming tools are used to apply compressive stresses on the aluminum blanks to produce sheets or the final product parts. These processes, therefore critically influence the surface appearance of aluminum sheet products [8]. The quality of processed aluminum surface is a crucial requirement for the customers, and consequently for the aluminum industry as well [14,21]. In the aluminum processing industry it is believed that the surface quality of the aluminum sheets are determined during the initial processing step namely hot rolling [22].

The thermomechanical processing of aluminum begins with rolling, which is used for the production of aluminum sheets, plates, bars, and strips, which are used in subsequent forming processes for the fabrication of parts (Fig. 1.1) [8,10,23]. The rolling stages of aluminum after casting include homogenization, hot rolling, and subsequent cold rolling, which is preceded by annealing as shown in the rolling processing route depicted in Figure 1.2 [24,25]. The hot rolling process is performed in stages: (i) breakdown rolling, which occurs on the as-cast ingot and reduces it to slab thickness, and (ii) tandem mill rolling, which reduces the slab to the final thickness [26]. During the heat treatments that precede rolling and occur between rolling stages, as well as during rolling
contact, the composition of the aluminum surface is constantly being altered [11,22]. This is the result of the diffusion of alloying elements, and reactions between the alloy surface with the surrounding atmosphere, including the absorption of components from the metal working fluids and contaminants [11,22]. Oxide layers and scales are formed on the aluminum surface during the various heat treatments. The thickness of these oxide layers vary with the heat treatment time and temperatures [27,28]. The thickness of the oxide layers is reduced during the rolling process and are noteworthy because they can induce surface defects and roughening of the aluminum surface [27,28]. However, the thin ubiquitous Al$_2$O$_3$ oxide film, which forms on aluminum alloys, even at room temperatures, has been described as being a protective layer that improves the corrosion resistance of the bulk alloy [3]. The roll surface is also known to be covered with oxide layers, which influence the performance of the roll [28,29]. The fracture and deformation of the oxide layer is known to be induced by the high contact pressures experienced during the rolling contact of the oxide covered surfaces of the roll and aluminum stock [27,30,31]. The rolling contact pressures are not constant, occurring for only a limited time that depends on the forming operation, in rolling it is 0.01 seconds, but continues to increase during the rolling process for that short time [20,27,32]. Damage and fracture to the oxide layers on the aluminum alloy, which occur as cracks, is influenced by the oxide layer thickness [27]. These spaces between the cracks in the oxide layer lead to the exposure and extrusion of fresh metal of the aluminum bulk [10,27].

As the heated aluminum stock is much softer than the steel roll, the intimate conformity of the roll and stock surfaces that occurs during hot rolling results in the material transfer of the softer aluminum to the roll surface [27,28,30,33,34]. The buildup
of aluminum transfer to the surface of forming tools is believed to be detrimental to the forming process, but in the case of aluminum rolling, it is believed to improve the biting characteristics required to draw the aluminum stock into the roll bite [8,27,30]. However, the effectiveness of the roll coating is influenced by its thickness as excessive aluminum transfer is still undesired and is removed from the work roll with nylon bristle brushes [30]. The thickness of the roll coating is influenced by lubrication. Typically lubrication is applied in all forming processes to control material transfer by limiting direct contact between the tool and the workpiece, control the temperature of the tools, and minimize friction and wear in the roll bite [10,28,30,35,36]. Lubrication for elevated temperature forming process vary from solid lubricants, like graphite and boron nitride (BN) used in hot forming, to emulsions used in rolling [37,38]. When used in elevated temperatures rolling operations, emulsions offer lubrication and act as coolants, fire retardants, and corrosion inhibitors [39–42].

Rolling lubricants are oil-in-water emulsions composed of mineral oil, water, oiliness agents, and emulsifier [10,22,41,42]. The working mechanism involved in these oil-in-water emulsion lubricants have been described as exceedingly complex [38]. For instance, during hot rolling, only oil is introduced into the roll bite, while water, which is used for cooling, evaporates to form steam pockets. Thus, the performance of the lubricant is dependent on both the oil composition and the properties of the emulsion [30,40,41]. Any changes to the base oil, additive type or content would alter emulsion properties, like surface wetting, lubrication, and emulsification [36,43]. The oil drawn into the roll bite coats and is then absorbed on both the roll and aluminum stock surfaces generating a low-friction low-shear-strength film due to the interactions between the
dispersed oil droplets and the contacting surfaces \[10,13,27,39,40\]. The spread of the oil on the roll and the aluminum stock in the inlet zone seen in Figure 1.3 is called “plate-out” \[40\]. The oil drawn into the roll bite between the roll and stock surfaces is under pressure, which is controlled by the plasticity of the aluminum stock. The stock begins to deform once the pressure in the oil exceeds the yield pressure of the aluminum alloy \[10,40,44,45\]. The thickness of the oil film and surface roughness (roughness height and distribution) determine the lubrication regime of the forming process \[13,28,38,45,46\]. Three lubrication regimes have been identified: boundary lubrication, mixed lubrication and hydrodynamic lubrication regime \[45\]. Rolling occurs in the mixed lubrication regime, close to the boundary regime, where there is some asperity contact between the tool and the stock surface for the production of bright rolled surfaces \[27,45,47\]. The mixed lubrication regime offers a compromise of reasonable friction levels and good surface finish as friction is too high within the boundary lubrication regime \[27,47\].

The surface roughness of both roll and stock surfaces influence the lubricant capture and delivery within the contact zone \[48\]. Examination of the used rolling lubricants have revealed that they contain metal oxide and debris from the aluminum stock \[27,28\]. This is due to the chemical interaction between aluminum surface with the lubricant and its additives to form complex soaps during rolling \[2,27,30,33\]. As a result of its distinct influence in the aluminum rolling process and its reaction with aluminum surface, the rolling lubricant is considered to have a critical influence on the surface quality of the rolled aluminum surface \[10,22,36,41,49\]. Damage to the work roll is also observed during rolling due to adhesion, abrasion, oxidation, and thermomechanical fatigue. This can lead to the need for the rolls to be removed from the mill for regrinding.
The lubricant is also employed to protect the work roll from this damage [39]. Rolls are centrifugally cast and possess a microstructure that includes a tempered martensitic matrix along with a network of hard primary carbides that influence the wear resistance of the roll and the lubricant distribution [51]. Because of the intimate conformity of the roll with the hot aluminum stock, any damage to the roll surface profile is continually printed on the continuous virgin aluminum surfaces produced during the rolling process [21,30].

The main parameters that influence rolling are summarized as the roll diameter, the deformation resistance of the rolled metal, and the friction between the rolls, which is required to draw in the rolled metal [23,32,52]. Friction and roll pressure are two of the compressive forces involved in rolling [23]. Rolling load is also known as the separating force as it equals the force exerted on the rolls by the metal trying to force the rolls apart. The rolling load is related to the diameter of the rolls, which induces greater deformation when it is increased and influence the material transfer [23,53,54]. The contact length is described as the portion of the roll and rolled metal in contact at any time during rolling, while the angle of contact, also known as the roll bite, is the angle between the entrance plane and centre of the rolls, which is also related to the diameter of the rolls [23,53]. The aspect ratio is the relationship between the projected length of contact and the mean thickness of the rolled metal and influences friction during rolling. The forward slip is the relationship between the speed of the roll and stock during rolling. It is related to the friction during rolling and is often used in its calculation [23,53–56].

The aluminum rolling process can be thus be described as a complex tribological system that includes the aluminum alloy, the lubricant film, the rolls and the oxides
covering the contacting surfaces (Fig. 1.4) [21,57]. The tribological conditions at the interface of the roll, or any forming tool and the aluminum alloy, are vital for the success of the forming operation [28,30]. This is especially true at elevated temperatures, where aluminum alloys can display superplastic behaviour at low strain rates but where surface quality can be reduced due to wear and enhanced plastic deformation [15,17,19,21]. The understanding and control of the tribological conditions and interactions between the aluminum alloy, lubricant, and forming tool are required to take full advantage of aluminum alloys and their elevated temperature forming processes [4,58].

Figure 1.1 Typical processing steps involved in the thermomechanical processing of aluminum alloys [59]

Figure 1.2 Typical process route for an aluminum can body stock [34]
Figure 1.3 Inlet zone of rolling region displaying on film of aluminum stock and roll [40]

Figure 1.4 The strip rolling process and a realistic toolpiece/workpiece interface [57]
1.2 SCOPE OF RESEARCH

The interest in the tribology of elevated temperature metal forming of aluminum stems not just from the advancement of our understanding, but from an economical sense. It determines the tool life, surface quality of the final product, and the productivity of the forming process [4,10,58,60]. The control and understanding of the tribological conditions at the interface of the forming tool and the workpiece is deemed crucial to maximizing the advantage of the formability of the workpiece and for an advancement and optimization of the forming process [4,28,58]. This is particularly the case with aluminum forming as aluminum has the critical drawback of poor resistance to seizure and galling because it easily adheres to the steel forming tool, leading to surface damage of both the tool and aluminum alloy [19,58,61]. The high production rates required in the rolling industry are influenced by the aluminum adhesion to the tool surface, especially since it has a detrimental effect on surface quality, rolling speeds and the ability to achieve high reductions [19,21,60]. The roll speed and reduction during rolling are closely linked to the coefficient of friction (COF), which must fall within a specific operating regime. This is because the COF can neither be too low, to avoid skidding, nor too high, to avoid poor surface quality [60]. Lubrication is thus applied to control friction and reduce adhesion, but the aluminum on steel contact is a particularly challenging tribosystem to lubricate, especially under the high pressures applied during aluminum forming processes, where lubricants can squeeze out [35,61]. The characteristics and tribological behavior of emulsions used during hot rolling is influenced by the type and concentration of the additives, oil film thickness formed in the roll bite, and the stability of the emulsions at high temperatures [38,60].
Interestingly, aluminum adhesion to steel and friction are also reduced by the Al$_2$O$_3$ film formed on the aluminum surface, as well as the tribolayer induced at the interface of the surfaces that are in sliding contact through material transfer [12,35,61]. The formation of tribolayers, their thickness, and composition are influenced by the forming temperature and lubricant employed [35]. Material transfer has been proposed to occur at rough surface features, which include grinding marks and protruding carbides, and defects on the tool and as it builds up, it modifies the tool surface [62]. The friction and wear mechanism transition that can be observed during forming processes have been associated with the transition between metal/metal, metal/oxide and oxide/oxide contact [12]. The understanding of the material transfer, which makes up the roll coating developed during the rolling process, can produce relevant knowledge transferable to other forming process like extrusion [63].

During forming processes, the surface and subsurface regions of the aluminum alloy are subjected to varying conditions and treatments from the bulk of the metal [64]. Subsurface plastic deformation, which occurs during these processes, is dependent on the tool surface roughness and can induce damage to the subsurface region of the metal alloy [62,65]. The plastic deformation of the aluminum alloy during processes, like rolling, induces strain in the rolled alloy, which is highest at the surface of the alloy and decreases gradually with increasing distance from the surface [66]. Micro-plastic processes, like the concentration of shearing strains at the subsurface due to high contact pressures, can lead to damage of the mating surfaces [47]. Various nanocrystalline microstructures are thus induced in the aluminum alloy subsurface during elevated forming process in the form of the tribolayer, however, the precise relationships between
the microstructure induced in these layers and the forming conditions remain uncertain [67]. One such tribolayer is the near-surface deformed layer, also referred to as the disturbed layer [67–70]. It is induced on aluminum alloys during high shear deformation processes like rolling and mechanical grinding [69,70]. The microstructure of this near-surface layer (NSM) consists of a surface layer, of continuous metal oxides, and a subsurface layer of ultra-fine grains decorated with oxide particles [34,64,67].

The tribological aspects of elevated temperature forming process is complex and influenced by several variables, such as materials, surface films, lubricants, contact pressure, and temperature [12,31]. Due to the intricate conditions at the roll/stock interface, tribochemical interactions occur as the metal is deformed by the tool and influences the surface quality of the rolled aluminum surface. Thus, examining the tribological interactions at rolling interface would prove insightful [12,71,72]. To that end, various tribometers have been developed and used to simulate various forming processes, however, there are not many tribometers for elevated forming process, and those that do exist are inappropriate in their representation of actual forming conditions [12,62]. It is well accepted that the ideal way of simulating the conditions of rolling is through rolling-sliding wear tests [51]. However, the tribological setups that have been used for simulating metal rolling have included disc-on-disc as well as pin-on-disc tests [30,62]. These do not capture the surface expansion experienced by the stock during the large plastic straining in metal forming, or the continuous formation of fresh stock surfaces that are formed and are in constant contact with the forming tool. Consequently, the processes miss several key events that are expected to occur [12,21,30,62].
Most aluminum forming processes involve contact with harder forming tools under high pressures at elevated temperatures [73]. Ideally, through the aluminum forming procedures, from rolling to the final forming process, the surface quality of the aluminum alloy should be such that it can go through each processing step without further processing [74]. Unfortunately, this is not the case as defects are continually induced on the aluminum surface and the aluminum surfaces need treatments at the risk of carrying these defects between forming processing steps. The tribological study of elevated forming processes would provide insight into the forming conditions that induce these defects. However, the tribological conditions of the forming process are difficult to simulate [75].

This thesis aims to systematically investigate the influence of various rolling conditions on the development of surface defects on the aluminium alloy with particular focus on the microstructural features of the tribolayer formed during hot rolling. It further examines how these tribolayers developed during rolling influence subsequent hot forming processes. It is based on work performed with tribo-simulators that were designed to emulate the tribological surface deformation experienced by the aluminum alloy during the hot rolling and forming processes. These tribo-simulators permit the altering of various forming parameters independently, including temperature, applied load, lubrication, tool and stock/strip surface conditions, and strain rate. As such, the effect of individual forming parameters on the development of surface defects and features can be examined. The rolling tribo-simulator possesses a roll-on-block configuration and can be used to measure the torque and rolling force during contact between the roll surface and the work piece surface at each rolling pass. The torque (G),
rolling force (P) and work roll radius (R) can be used to calculate the coefficient of friction (COF). A schematic diagram of the rolling tribo-simulator along with samples of torque and rolling force data acquired during rolling are displayed in Figure 1.5. The forming tribo-simulator possesses a pin-on-strip configuration and is capable of simulating the sliding contact during the forming process while measuring the COF during contact between the pin and the metallic strip being plastically deformed. A schematic diagram of the forming tribo-simulator with a sample of the COF data obtained during a test at room temperature is shown in Figure 1.6.

Thus, this dissertation is structured by first presenting, in Chapter 1, an introduction to a general overview of aluminum forming and our motivation of this research (Section 1.1). The scope of the dissertation is featured in Section 1.2, while Section 1.3 displays the desired objectives of this work. Section 1.4 contains a general literature survey of the work already performed in this field. Further chapters of this dissertation also contain studies that have been published in peer-reviewed journals. Chapter 2 examines the formation of micro-blisters observed on the surface of Al-Mg alloys during hot rolling. The novelty of these micro-blisters is that they were observed after the first hot rolling pass. The structure of the blisters was analysed and a mechanism for their formation was proposed based on the microstructure. Chapter 3 presents work into the development of micro-cracks on the Al-Mn surface region. The development of these cracks was studied through four rolling passes to examine how each pass affected their propagation. In Chapter 4, the oxide development within micro-cracks of Al-Mg alloys is examined in relation to the effect of hot rolling passes. This work aids the study of the effect of rolling stresses and strains on oxide development within cracks of the
near-surface. Chapter 5 contains work on the characterization of the near-surface defect features that had developed on the Al-Mn alloys through a rolling schedule of 10 passes. The effect of rolling passes, stress and strains on the development of the near-surface features was examined. Chapter 6 studies the effect of work roll roughness on near-surface damage. This work displays the role work roll roughness, and rolling stresses and strains play in near-surface microstructure development. Chapter 7 systematically examines the influence of the increase in the surface roughness of the work roll has on the features of the near-surface microstructure. This chapter also looks into the development of shingles on the surface of the aluminum alloy and how they are influenced by the work roll. Chapter 8 includes work with physical vapor deposition (PVD) coated work rolls and examines the effect of these coatings on mitigating aluminum adhesion and friction reduction. Chapter 9 presents work on the roll coating built up on a work roll during the hot rolling of Al-Mg alloy. Here, the microstructure of the roll coating developed on a polished roll is examined. Chapter 10 examines the influence of the tribolayer (near-surface microstructure) on subsequent forming processes in relation to friction and adhesion. This work also examines the surface features induced on the tribolayer during the elevated temperature forming and the influence of temperature on these features, adhesion and friction. Finally, Chapter 11 complies an overall summary and conclusions of this dissertation.

1.3 OBJECTIVES

Near-surface microstructures are induced on aluminum alloys during thermomechanical process, while the forming tools plagued by the buildup of aluminum...
adhesion to their surfaces. Section 1.4 goes into details with regards these near-surface microstructures as well as the buildup of material transfer on the forming tool surface, although they have been touched on in the previous sections. An in-depth understanding of the tribological conditions responsible for both the near-surface microstructure and aluminum adhesion buildup has long been outstanding. This is due, in part, to the lack of tribometers that effectively simulate elevated forming processes and the lack of control of individual parameters to better understand their influence on the NSM features. This thesis, therefore, has the following general objectives:

- to determine of the micromechanisms responsible for the formation of the surface and subsurface features of near-surface microstructure (tribolayer) induced on the aluminum alloys during hot rolling using laboratory scale tribological simulations of hot rolling.

- to develop a deeper understanding of the influence individual rolling parameters have on the formation and evolution of the surface defects and near-surface microstructures induced on aluminum alloys.

The work carried out in this thesis also extends to the material transfer developed on the forming tool, in this case, the roll during the hot rolling process. The material transfer in the form of the roll coating developed on the roll has significant influence on the surface appearance of the rolled aluminum and is believed to be essential for rolling. However, its microstructure at any stage of the rolling process is still unknown. This information is essential if its influence on the development of the near-surface microstructure is to be properly ascertained. Therefore this thesis objectives also included:
- the characterization of the microstructure of the roll coating during the initiation of its build-up on the work roll.
- examining of the influence of PVD coatings on the work roll with the aim of reducing material transfer to the work roll surface.

As stated earlier, an understanding of the near-surface microstructure (tribolayer) behavior during subsequent forming process and its influence on the tribological conditions during elevated temperature forming are areas where little research has been done. Therefore this present work extended it objectives to include:

- the study of the effect of the tribolayer (near-surface microstructure) developed during the rolling process (hot and cold rolling) on subsequent elevated temperature forming processes.

Figure 1.5 Schematic diagram of the rolling tribo-simulator displaying the possible motion directions for the work roll and work piece including the sample torque and rolling force data obtained during a rolling pass.
1.4 LITERATURE REVIEW

1.4.1 Surface features and defects induced on aluminum alloys during elevated temperature forming

Rolled aluminum sheets typically have their surfaces covered with oxides and rolled in lubricants [34,64,76]. The typical surfaces of these sheets are a reflection of work roll surface and have also been characterized as being covered with grooves, gorges, rolling ridges, shingles, and cracks that occurs transverse to the rolling direction shown in Figure 1.7 [47,67,76,77]. Some of these surface features are believed to be defects that could lead to the rejection of the rolled material due to the reduced surface quality and could even cause mill stoppage [78]. Surface defects on rolled or formed
products in general are thus considered a serious issue and various factors have been considered as being responsible for inducing their occurrence [78,79].

Grooves and rolling ridges, which dominate the rolled aluminum surface, are imprinted by the work roll topography during rolling [76,80]. Surface defects, like transverse cracks, identified not only on aluminum alloys but also steel, are also known as cross hatches and have had limited research into their manifestation [76,79]. This is due to the general belief that they are initially formed within the surface oxide of the aluminum alloy, although the continued propagation of these cracks determine if the rolled aluminum sheets are acceptable or rejected [45,76,79,81]. These cracks have been observed to induce the delamination of the near-surface region [67]. Gjonnes et al. [76] originally suggested their initiation to be due to the shear fracture or cracking of the oxide layer, which occurs while rolling ridges are being imprinted on the alloy surface. Bazhin et al. [81], however, associates these cracks with the oxide layer breakdown induced by external mechanical damage or the presence of internal defects. Le et al. [45], alternatively, attributes the fracture of the oxide layer to the surface bending and stretching of the aluminum surface during the rolling process. They also noted that these micro-cracks are sites for fresh metal extrusion to the alloy surface [45]. Fishkis et al. [64] argue that the occurrence of transverse cracks within the early hot rolling passes are due to the lower ductility of the surface layer, material transfer, and the shear stress reversals in the roll bite, which are then covered with thin layers of aluminum on subsequent passes. Cracks observed on the 5000 series of aluminum alloys have been found to be magnesium-rich at the contour of the cracks during rolling and cold drawing [82]. In steel, where extensive transverse crack research has been carried out, crack
healing has been observed and is related to the thermal and stress distribution around the cracks [79]. Meanwhile, the mechanism of crack evolution in steel during rolling has been determined to be primarily affected by the crack open-angle [79]. In aluminum alloys, cracks have been found to occur along grain boundaries, and some research has observed that intergranular cracks initiate under different types of loading [83,84]. Cracks and cavities have been related to the sliding of grain boundaries and grain boundary cavitation. However, these observation have been observed under conditions for stress corrosion, though a few researchers have reported similar cracks under high temperature deformation of Al-Mg alloys [58,83–86].

Shingles have long been considered to be a result of the interaction between the aluminum alloy and the rough work roll surfaces and result in the roughening of the aluminum alloy surface [34,56,64,68]. They have been described as tongues lying on the surface of the aluminum alloy, with the protruding tip of the tongue pointing in an opposing direction to rolling [80]. An interface between the tip of the shingle and the aluminum alloy surface has been reported to occur frequently [76,80]. The overlapping of different interfaces can induce laminates within the surface, and their manifestation has been connected to the existence of a highly deformed near-surface layer on aluminum alloys [67,68]. Shingles are one of the predominant features of cold and hot rolled aluminum alloys, so much so that their surfaces have frequently been described as having a shingled surface appearance [34,67,68,76,80]. Shingles have been associated with other features of the NSM; holes and cavities on the aluminum alloy surface, which they have been proposed to cover; grooves, which they occur behind; microcracks, at the edges and shoulders of shingles; and the entrapment of lubricant residues [34,67,68,76,80]. It has
been suggested that shingles are formed: by the aluminum on the surface being pushed backwards and smeared during rolling; the deformation or smearing of steps, peaks or protruding features on the surface of the aluminum alloy; material transfer and back transfer; and the plastic deformation of micro-wedges formed by the work roll grooves [56,64,68,76,80]. The formation and length of shingles are reportedly influenced by forward slip [56,67,68,76]. Riahi et al. [56] suggest that shingles are more pronounced on the aluminum alloy surface when forward slip is increased and when the number of rolling passes increased. The manifestation of shingles has also been related to the work roll topography, with work by Liu et al. reporting high population densities of shingles when high aspect ratio rolling was coupled with increased rolling speed and rough but worn roll surfaces [67]. When the aspect ratio and rolling speed were lowered and coupled with a freshly ground roll surface, lower population densities of shingles were observed [67]. Gjonnes [76,80], however, suggests that the shingle size and occurrence are related to the surface morphology of the work rolls.

The surface of the worked aluminum alloy is also influenced by the material transfer from its surface built-up on the forming tool surface [34,42,49,75]. The material transfer adhered to the tool surface can be transferred back to the aluminum surface during the forming process [34,75,87]. In the rolling process, this occurs when the roll coating becomes unstable [87–89]. The back transfer from the forming tool introduces pickup defects on the formed aluminum surfaces, which are visible to the naked eye [42,75,76,87,90]. During the extrusion process, they terminate in a fleck of aluminum debris, known as the pickup deposit [87,90]. In the extrusion process, these defects are noted to be influenced by temperature, inclusions in the cast billet, and the die deflection.
Alternatively, in rolling, they have been related to the carbide spacing on the work roll and the defects in the roll coating [76,90,91]. The control of carbide spacing has been suggested to be an effective prevention method for pickup defects [91].

Blisters are another surface defect that have observed on aluminum products formed at elevated temperatures [92,93]. Blisters, also referred to as core blisters, are described as raised spots on the surface of the rolled or formed product and are deemed to have a detrimental effect, not just to the surface quality, but also the mechanical and physical properties [92,94]. Blistering occurs when the oxide layer formed during a process, like hot rolling, detaches from the base metal and then swells [95]. Blister formation is thus based on gas diffusion and the separation of free surfaces where large pressures is built up during lubrication breakdown [90,92,94,96]. Blisters have been observed to break open during rolling, which produces defects that resemble a gouge or subsurface lamination [94]. In steel, blisters have been linked to CO and CO$_2$ gas arising from the decarburization of steel. It is related to two mechanisms: (i) the growth stress resulting from scale formation, and (ii) gas generation from steel, which occurs at the interface with the scale [95]. However, blistering in aluminum alloys is linked to hydrogen present in the ingot, which causes macroscopic defects and enhances void formation [96,97]. During rolling, the hydrogen gas pressure deforms the surface of the sheet and form a blister, especially when the strength of the material is reduced during annealing [94,96]. Blisters, which often occur parallel to the rolling direction, are formed during the heating of the alloy and are linked to internal defects in the ingot [60,94,96]. The outward diffusion of magnesium during the heating of aluminum alloys has also been observed to result in the formation and growth of voids at the alloy/oxide interface,
which facilitate the delamination of the oxide layer, thus inducing blister formation [98].
Therefore, it is apparent that the oxides covering the aluminum surface have a significant influence on the surface appearance and defects on the aluminum alloys.

![Secondary electron micrograph displaying the surface features of a rolled aluminum alloy](image)

**Figure 1.7** Secondary electron micrograph displaying the surface features of a rolled aluminum alloy [99]

### 1.4.2 Surface oxide layers

The aluminum sheet metal surface has been observed to be covered with oxides and hydroxides coated with a thin organic film [100]. The composition of the oxide layers on aluminum alloys are dependent on the alloying elements, temperatures, and the processing conditions [25,100,101]. It is therefore critical to characterize the oxide layers developed at varying stages of thermal treatment or elevated temperature forming in order to determine the oxide reaction with any reactive specie or contacting surfaces [25].
Aluminum alloys typically possess oxide layer between 2 – 7 nm thick at room temperature, which inhibits chemical interaction of the substrate with gases and moisture, yet permits the deformation of the substrate without rupture [81,102,103]. This oxide layer (Al₂O₃) is self-healing at room temperature, and during the initial stages of oxidation during heat treatment, where oxide thickness continues to grow, reaching upwards of 200 nm [27,81,102]. The increase in the Al₂O₃ thickness induces the reduction of the adhesive strength of the oxide with the substrate leading to scale failure from the compressive stresses in the oxide, which is caused by temperature changes and growth stresses [3,81,104]. The properties of the oxide are also temperature dependent; below 500 °C, Al₂O₃ is hard and brittle, while at higher temperatures and pressures the oxide is considered ductile and is able to sustain a low friction regime if the oxide is not damaged, thus allowing metal to metal contact [4,27,47]. At low temperatures, the Al₂O₃ is amorphous and oxidation is a result of the thermal diffusion of aluminum or oxygen inducing a continuous oxide layer [105,106]. At temperatures exceeding 425 °C, the oxidation rate accelerates through the nucleation and growth of crystalline γ-Al₂O₃ at the oxide/metal interface. This is due to the inward diffusion of oxygen through the amorphous oxide layer [5,101,105,106]. The transition from amorphous to crystalline is thus governed by temperature and the thickness of the oxide film, but is also influenced by the magnesium content of the aluminum alloy [3,106,107].

The addition of magnesium to aluminum alters the oxidation mechanism of aluminum alloys, accelerating their oxidation rate [106,108,109]. Magnesium concentrations in commercial Al-Mg alloys are typically within the range of 0.5 – 12 at.%, but even at impurity levels, magnesium’s influence on the oxide formation on
aluminium alloys is distinct, enhancing γ-Al₂O₃ nucleation [5,106,108]. At higher concentrations, magnesium suppresses γ-Al₂O₃ formation, promoting the nucleation and growth of MgAl₂O₄ and crystalline MgO, either by the reduction of γ-Al₂O₃ at the oxide/metal interface, or by direct growth at the alloy surface [81,101,106]. The increase in magnesium content has been reported to induce the formation of more complex Al-Mg mixed oxides like Mg doped Al₂O₃ [3]. The formation of magnesium-rich oxides in aluminum alloys have been linked to the higher affinity magnesium has for oxygen in comparison to aluminum and its faster diffusion rate in aluminum than the self-diffusion rate of aluminum [110–112]. Magnesium diffusion is also enhanced by defects and voids within the alloy and oxide layer [98,112]. The reduction of amorphous γ-Al₂O₃ to metallic aluminum is dependent on the thickness of the γ-Al₂O₃ layer and the rate of magnesium diffusion [24,106]. The magnesium-rich oxide layer that forms on Al-Mg alloys at elevated temperatures is induced by magnesium diffusion through the Al₂O₃ layer. This results in the magnesium depletion in the bulk alloy and the reduction of the oxide film’s protective nature [98,101,102,106]. This occurs because the incorporation of magnesium in the surface oxide increases the electrochemical activity of the alloy surface [113,114]. The oxide is therefore susceptible to hydrolysis and possess reduced corrosion resistance [101,102,105,111]. Magnesium also aids the disruption of the continuous amorphous Al₂O₃ on the aluminum alloy surface through the formation of a spinel that permits the rapid exchange of oxygen and metal ions [101,105]. Magnesium diffusion through the existing oxide layer occurs via two mechanisms: (i) the outward diffusion of magnesium ions, and (ii) magnesium vapour transfer from oxide defects and voids [19,98].
A schematic model of the oxide growth on Al-Mg alloys during heat treatments is displayed in Figure 1.8 [115]. After heat treatment at elevated temperatures, the oxide layer on Al-Mg alloys consist of an outer MgO layer, a MgAl$_2$O$_4$ spinel layer, and an Al$_2$O$_3$ layer adjacent to the aluminum surface (Figure 1.8) [19,115]. There is no sharp transition from the outermost MgO layer to the inner Al$_2$O$_3$ layer; rather, there is a formation of mixed intermediate layers consisting of MgO + MgAl$_2$O$_4$ and MgAl$_2$O$_4$ spinel above an Al$_2$O$_3$ + MgAl$_2$O$_4$ layer before the Al$_2$O$_3$ layer [19]. The magnesium-rich oxides on aluminum alloys have been described as non-uniform, dark piles or islands, on the alloy surface that induce an oxide-related roughness and are comprised of fine crystallites composed of MgO [4,24,98]. As heat treatments continue, the magnesium-rich oxide layers become continuous and cover the alloy surface [115]. Field et al. [101] observed crystalline MgO possessing a platelet morphology at the oxide/metal interface of aluminum alloys with 3 wt.% magnesium, while Overtfelt et al. [4] and Bazhin et al. [81] reported the presence of MgO with a loose granular structure, and Holub et al. [24] described the MgO on the aluminum surface as friable and powdery. Brock and Heine have been cited as establishing the platelet morphology of magnesium oxides for Al-3%Mg alloys [64]. The composition, amount, thickness, and oxide-related roughness of the magnesium-rich oxide layer is dependent on the magnesium content, temperature, alloy grain size, and the atmosphere [3,4,98,105,113]. When the magnesium concentration is low, the magnesium-rich oxides are closer to the metal/oxide interface; however, at high magnesium concentrations, the magnesium-rich oxides occur closer to the air/oxide interface [112]. The occurrence of an MgAl$_2$O$_4$ spinel or an γ-Al$_2$O$_3$ layer, though, can impede the rate of growth of the oxide layer as magnesium diffusion through
oxides, even MgO, is slower than in the metal [19,110,111]. Despite this, magnesium diffusion is dominant in the oxide layer during its formation [101,111]. During heating in a vacuum, magnesium diffusion in aluminum leads to weight loss of the aluminum alloy due to magnesium evaporation rather than oxidation [110,115]. In air, the surface oxide layer helps to inhibit the evaporation of magnesium [111].

During rolling, the oxide layers developed on the Al-Mg alloy are enriched with magnesium, metallic aluminum, from Al₂O₃ reduction by magnesium, and lubricant residues [116]. The formation of these oxides on the Al-Mg alloys during rolling affect the interaction between the roll and the aluminum slab, as well as the surface quality of the hot rolled products [117]. The surface oxides are incorporated into the surface region when it is folded to form shingles and can also lead to other undesirable effects like increased pickup defects [76,115]. On rolled aluminum sheets, the buildup of MgO and MgAl₂O₄ is known as staining and products with this buildup referred to as “dirty metal of inorganic origin” or simply “dirty metal” [24]. The name arises from the gray-to-black color or “dirty” appearance of the rolled aluminum surface thought to be induced by the defects in the magnesium-rich oxide layers [24]. The surface of rolled Al-Mg alloys are completely covered with MgO within a few minutes at 600 °C [115]. MgO produced during rolling was proposed to be spread across the surface of the aluminum alloy due to transfer and result in the formation of roll debris [24]. In addition, the distribution of magnesium across the rolled aluminum surface has been related to the roughness of the sample and the rolling marks on the sample [112]. In the aluminum rolling industry, fluroborates and various volatile fluorides are used to reduce the magnesium oxide concentration and corrosion of the finished and semi-finished Al-Mg alloys [25]. The
effectiveness of ammonium fluroborate (NH$_4$BF$_4$) treatments, applied before heat treatments, have been observed to be dependent on the magnesium concentration at the surface and within the bulk aluminum alloy, with better performances being observed with alloys that had higher magnesium bulk concentrations [25]. The MgO formation inhibition mechanism of NH$_4$BF$_4$ has been linked to the formation of an aluminum fluoride layer close to the Al$_2$O$_3$ layer [25]. The oxides covering the aluminum surface are the last barrier of protection against galling or scuffing when the lubricant film fails [47].

Figure 1.8 Schematic model of growth of oxide on Al-Mg alloys during heat treatment [115]
1.4.3 Tribolayers

The formation of tribolayers on the surface of aluminum alloys, which commonly occurs during sliding contact, has been described as being composed of oxides and possessing a nanocrystalline structure [31,118–120]. They have been described as mechanically mixed layers (MML) containing particles from the sliding counterface [120–122]. Tribolayers have been observed in elevated forming operations, such as rolling, forging, and stamping [12]. They have been reported as reducing shear stresses, wear, and friction during sliding contact acting as solid lubricants depending on their composition [12,35,123]. The wear and friction reduction properties of these layers has been attributed to their nanocrystalline structure and their ability to supress metal to metal contact [31]. The formation and retention of these layers, their composition, area fraction, thickness, and hardness are regarded as being influenced by the nature of the sliding surfaces, the temperature, environment, humidity, and the lubrication conditions [35,120,123,121]. The oxygen concentration of these layers has been related to temperature [31]. While, their formation increases the hardness of the alloy surface [120]. The performance of the tribolayers is dependent on their stability and thickness, with wear rate reduction observed with the increase in stability and thickness [120,124,125].

Rynio et al. studied on the evolution of tribolayers at elevated temperatures and found that the tribolayer structure and formation was governed by the type of plasticity observed during severe plastic deformation (SPD) processes [31]. Studies on the room temperature local nanoscale deformation of tribolayers formed on Al-Mg alloys revealed that the initial deformation strains were accommodated by the plastic deformation within the tribolayer, while progressive strains were accommodated by subgrain rotation [118].
Further deformation was observed to lead to material removal as a result of the penetration of cracks that nucleated around the subgrains [118]. The delamination of the oxide-rich tribolayers was reported by Edrisy et al. [119] as the primary source of material removal during the scuffing of automobile aluminum combustion engines. Additionally, Elmadagli et al. [122] noted that wear during the dry sliding of Al-Si alloys progressed by the formation and breakdown of tribolayers. They reported the breakdown of the tribolayers in the form of crack propagation and spallation leading to the detachment of tribolayers and its formation as being initiated by transfer from the counterface [122]. Lu et al. [125] observed that magnesium additions to aluminum alloys improved the tribolayers, as tribolayers produced from magnesium free aluminum alloys were coarse, loose, and inhomogeneous. They reported fine, dense, and homogenous microstructures for tribolayers formed with aluminum alloys containing magnesium, which improved the load bearing capacity of the tribolayer [125]. Riahi et al [120], Varga et al. [121] and Stoyanov et al. [124] have observed tribolayers with layered structures. Varga et al. [121] suggested three different layers could be formed during tribological contact: (i) transfer layers built up without oxidation occurring, (ii) mechanically mixed layers with no oxides, and (iii) composite layer buildup with oxides. Tribolayers are preferentially formed with ductile materials, and their formation and coverage is enhanced by increases in temperature [121,126].

1.4.4 Near-surface deformed layers

Metal forming and machining processes which induce severe strain, like rolling, and mechanical grinding, onto aluminum alloy workpieces can transform the surface
microstructure to form tribolayers referred to as near-surface deformed layers [58,69,127–129]. Thermomechanical processes are particularly known to generate damage within the near-surface region of aluminium sheets, such that the near-surface deformed layers are thought of as a generic or ubiquitous on rolled aluminum sheets [65,70,130,131]. These layers, which have been referred to as the subsurface layer, surface layer, Beilby layer, rolled-in layers, and altered surface layers (ASL), are induced because of the high levels of shear strain imposed on the aluminum alloy surface/near-surface region during metal processing [58,86,128,129,132–134]. It has also been referred to as the disturbed layer, the white layer or the flow zone [68]. The near-surface deformed layer has been of considerable interest to the aluminum industry due to its altered electrochemical, microstructural, and optical properties, which vary from the bulk alloy (Fig. 1.9), as well as its influence on the joining, welding, and corrosion behavior of the alloy [65,99,135,136]. The reduced corrosion resistance of the aluminum alloy has been a significant concern, especially with respect to filiform corrosion (FFC), which influences the cosmetic appearance and quality of aluminum alloys [131,137]. Filiform corrosion occurs on metals surfaces with organic coating due to the highly electrochemically active nature of the alloy surface. It was the preferential susceptibility of these surfaces to filiform corrosion that led to their discovery by Leth-Olsen [34,64,65,117,131,137,138]. Their discovery and the further work of Fishkis and Lin, who first presented a schematic representation of the near-surface microstructure of a hot rolled aluminum alloy (Fig. 1.10), can be credited as inciting the increased in-depth studies on the near-surface deformed layers [34,70].
The near-surface deformed layer has neither a well-defined structure nor composition [56,86,136]. However, in magnesium containing alloys, it is rich in magnesium due to in part to magnesium diffusion through to the alloy surface as discussed earlier [34,56,132,139]. The thickness of these layers on the aluminum alloy are non-uniform, varying between 0.5 – 8.0 µm, but this thickness is largely dependent on the thermomechanical history of the sheet metal [56,58,65,86,132,136]. The microstructure of the near-surface deformed layer or the near-surface microstructure (NSM) has been characterized as being composed of an ultrafine grain structure with a high population density of oxide particles (which have been described as being rolled-in and decorating the grain boundaries), fragmented intermetallic particles, dispersoids, voids, and subsurface cracks [69,99,128,129,136,138,140,141]. This subsurface layer, as seen in the schematic in Figure 1.10, is covered by a surface layer of continuous metal oxide phases consisting of Al₂O₃, MgO and MgAl₂O₄ [34,56,135,138]. In rolling, the oxide type is dependent on the stage of the rolling process with MgO observed as the dominant oxide at the end of the rolling process [34,64,117]. The rolled-in oxide and fragmented intermetallic particles have been proposed to pin the grain boundaries, preventing recrystallization from occurring within this tribolayer during heat treatments, resulting in the stabilized nanocrystalline grain structure [134,141,142]. The near-surface microstructure has also been revealed to be incorporated with aluminium carbides and amorphous carbon or lubricant particle from the rolled-in lubricant [70,99,132].

Studies on the rolled-in oxides and the presence of carbon in the near-surface region have revealed that they decrease the optical total reflectance (TR) of aluminum alloys [141]. The rolled-in oxides and lubricants have been attributed by several authors
to local plastic deformation (ploughing) of the aluminum surface, by the asperities on the work roll surface, and re-welding of the transferred surface back to the aluminum alloy surface, resulting in the mixing of the oxides and lubricant residues [69,99,143]. This phenomenon is also thought to be the explanation for the sharp transition that has been observed from the damaged NSM to the bulk microstructure [69]. Premendra et al. [141] proposed that lubricant entrapment within the near surface region was not only due to the rolling-in of the oxide and lubricant, but was also related to the entrapment of lubricant in the surface defects like holes, which are subsequently covered as the surface is folded over forming shingles [132]. The degree of oxide and lubricant incorporation varies along the alloy surface and is dependent on the severity of the roll and aluminum surface interaction [99]. Therefore, the incorporation of oxide and lubricant particles in the near-surface layer is considered a complex process related to the rolling processing parameters [99].

The amount of near-surface damage is considered to be highest during the early hot rolling passes [64,69,134]. The incorporation of oxide particles into the near-surface region also occurs during the hot rolling stage [134]. On subsequent rolling passes, as the alloy cools down, there is less severe damage within the near-surface region, which leads to the reduction of the damaged layer as it is distributed over the larger surface area, however, it becomes more distinct with these subsequent passes [69,134,141]. The plastic deformation imposed on the alloy surface due to unidirectional shear strain during rolling results in strain distribution with the alloy, the highest strain is observed at the surface of the alloy and reduces progressively as the distance from the surface increases [66,135]. The NSM had previously been reported to be the result of the combination of large shear
deformation along with repeated fracturing and the re-welding of the aluminum surface during the rolling process [134]. Initial studies proposed that the near-surface damage formed during rolling occurred through a three step process [64]:

1. The formation of surface depression (holes) by plowing, adhesive wear, delamination wear, or transverse surface cracking

2. The filling of the holes with the wear debris consisting of metal and oxide fines mixed with lubricants

3. The covering of holes with thin metal layers during the continuing rolling process leading to a shingled surface appearance.

Zhou et al. [68] credited that the generation of the near-surface layer as the result of a combination of large strain plastic deformation and mechanical alloying induced during the tribological interaction of the roll and the aluminum surface. They also related the nanocrystalline structure of the NSM to the severe strain processing from severe plastic deformation [66,68]. However, the mechanisms involved in the formation and evolution of the near-surface deformed layer are still subject to discussion [135].

Li et al. [135] revealed two classes of deformed layer, type A and type B, as displayed in Figure 1.11. As schematically represented in Figure 1.12, type A and type B are characterized by ultrafine grains. However, while type A, typically formed during hot rolling, possessed oxide decorated grain boundaries, type B, generated during hot and cold rolling, does not [68,135]. Further research by Zhou et al. [68] identified the multilayer structure of the near-surface layer, which has been characterized by an outermost layer comprised of the ultrafine grains next to an inner transition region characterized by mirobands of elongated grains aligned parallel to the rolling direction,
and lies on the bulk microstructure (Fig 1.12). Although the precise mechanism for the formation of these microbands was found to be unclear, it was suggested that this transition region could be related to the reduced shear strain within this region of the near-surface, as the strain gradient distribution within the near-surface displayed the highest shear stress at the surface and least at the bulk of the alloy [68]. Similar multilayer near-surface microstructures have also been reported by Tzedaki et al. [132], with grains elongated in the transverse direction observed closer to the bulk alloy and randomly oriented smaller grains close to the alloy surface. The occurrence of shingles on the rolled aluminum surface has been related to the presence of a near-surface deformed layer by researchers like Liu et al [67], who proposed that the thickness of the near-surface layer is directly related to the occurrence of shingles, cracks, and distribution of coarse intermetallic particles. Holes and cracks at the near-surface region have been proposed to be enhanced and introduced into the subsurface by the presence of shingles and debris on the alloy surface, with holes developing into subsurface cracks [34,64,132]. Tzedaki et al. [132] worked on lithographic printing sheets and revealed the cracks at the near-surface region were surrounded by ultrafine grains with possible grain overlapping. Magnesium segregation towards the surface of cracks was observed to form MgO on the subsurface crack faces [132]. These oxides within the near-surface were exposed to be both amorphous and crystalline in structure [132].

The corrosion susceptibility of the near-surface deformed layer has been linked with the enhanced secondary precipitation of intermetallic particles as well as the preferential nucleation and growth of dispersoids in the near-surface layer during heat treatment [69,130,138,142]. These intermetallic particles are rich in iron and manganese,
and their fracture during hot rolling increases the density of cathodic sites within the near-surface region [69,130,138]. Large plastic deformation on the aluminum alloy surface during rolling also results in the redistribution of intermetallic particles, while enhanced secondary precipitation of these particles occurs at elevated temperatures [130,134]. The differences in the intermetallic particle distribution within the NSM is believed to induce the electrochemical reactivity observed in the near-surface deformed layer [69,134]. The accelerated intermetallic particle precipitation within the near-surface deformed layer occurs because its nanocrystalline grain structure provides a high population of grain boundaries, which are energy favorable nucleation areas and high-rate diffusion paths [99,129]. The large concentration of defects with the near-surface region from the deformation during forming and machining processes significantly enhances the mobility of alloying atoms [68,99,129]. The precise mechanisms responsible for the electrochemical activation of the near-surface layer for certain Al-Mn alloys are still in question as it is unconfirmed if the precipitation of the manganese-rich dispersoids or the preferential grain boundary precipitation of precipitates is the responsible mechanism [70].

In aluminum rolling, the thickness, composition, and morphology of the grains and the formation of the near-surface deformed layer are influenced by the tribological conditions at the rolls and aluminum slab interface [86,139]. These tribological conditions included parameters such as rolling aspect ratio, rolling speed, temperature, lubrication, and roll roughness [86,139]. The exact relationship of the rolling processing conditions and the development of the NSM are yet to be defined [68]. Riahi et al. [56] report increased thickness of the near-surface deformed layer with higher forward slip.
They concluded that during the initial rolling stages, the forward slip and the number of rolling passes influenced the changes in morphology, thickness, and composition of the near-surface defects on the rolled aluminum [56]. The thickness of the near-surface layers have also been correlated to the surface morphology of the work roll, in particular the height of the surface asperities, noting thinner layers with newly ground work roll surfaces [67,70]. There is limited systematic investigations into the influence of rolling conditions on the structure and thickness of the near-surface layers due to the difficulty of laboratory simulations of the rolling process and obtaining commercial inter-pass samples [34,70]. This near-surface layer is removed by post production treatments, like anodizing and caustic etching, which significantly increase the commercial cost and material loss of aluminum processing [65,141].

Figure 1.9 Surface layer on hot rolled aluminum alloy [144]
Figure 1.10 Schematic illustration of near-surface microstructure on hot rolled product [144]
Figure 1.11 Transmission electron micrograph of ultramicrotomed section of the surface/near-surface region of an AA3005 aluminum alloy showing the near-surface deformed layer with 5 hot rolling passes [68]
Figure 1.12 Schematic diagrams showing the microstructure of near-surface deformed layer introduced by rolling: (a) type A and (b) type B [68]

1.4.5 Near-surface deformed layer influence on subsequent forming processes

The near-surface deformed layer developed during the aluminum rolling process is meant to have considerable impact on the tribological properties, wear characteristics, and mechanical properties of the alloy of subsequent sheet forming processes and the surface quality of the final products [68,99,140]. However, outside the considerable work done to understand the corrosion behavior and the general knowledge of its effect reducing adhesion, which affects welding and joining, few studies have been conducted to gain insights on its influence on subsequent forming processes, like forming, stamping or drawing. The friction and wear characteristics are meant to be directly related to the near-surface microstructure, which is in direct contact with the forming tools [140]. The ductility of the near-surface layer is thought to be reduced due to the presence of high population densities of brittle aluminum and magnesium oxide particles, lubricant
residue, and subsurface cracks and voids [64,68,99]. This could result in cracking within the near-surface region and metal transfer during forming processes [64,99]. The dominant MgO surface layer covering the near-surface deformed layer has been found to act as a solid lubricant, reducing the coefficient of friction (COF) during sliding contact [4,14,19,24]. Riahi et al. [19] have displayed that the adhesion tendency of aluminum alloys are also influenced by the thickness of the MgO layer at the alloy surface. They observed that an existing MgO layer on the surface of aluminum alloys lowered the junction strength formed between the surfaces in contact; thus, adhesion decreased as the surface oxide thickness increased, which corresponded to the increase in magnesium content of aluminum alloys [19].

A study by Wang et al. [140] on the effect of sheet forming on cold rolled an AlFeSi alloy showed the near-surface layer delaminating from the alloy surface and yielding flake-like debris. The delamination was attributed to the wear from sliding contact. Wang et al. [140] proposed a model for the delamination of the near-surface deformed layer during sheet forming displayed in Figure 1.13. They suggest that as the near-surface deformed layer displays reduced ductility, it provides favorable sites for the nucleation of subsurface cracks during sheet forming, which tend to propagate during forming processes [140]. Such sites include voids and oxide decorated grain boundaries. The propagation of these cracks, roughly parallel to the alloy surface, resulted in delamination. This indicates that the interface would be a favourable path for crack propagation [140].

Das et al. [58,86] performed experiments with a novel hot forming simulator, taking advantage of the superplastic behaviour of on Al-Mg alloys at high temperature
and low strain rates. They found that the COF varies with temperature increases in relation to damage to the near-surface layer during plastic deformation in the temperature range of 420 °C – 545 °C. They also observed damage within the tribolayer during forming, in the form of cracks within the surface oxide at lower temperatures, and low average surface roughness [58]. At higher temperatures, a change in the composition and mechanical properties of the near-surface deformed layer was detected. Within the cracks of the oxide-rich near-surface layers, they observed thin fibrous structures, as known as oxide fibres or ligaments, with a length of 1.5 – 2.5 µm and diameter of 0.1 – 0.2 µm (Fig. 1.14) [58,86]. They proposed that the plastic deformation of the bulk Al-Mg grains imposed plastic strain on the oxide-rich near-surface deformed layers due to GBS, which formed cracks within the near-surface deformed layer and stretched the oxide fibres, over the sliding grains, across the separating crack faces [58,86]. The oxide fibres displayed superplastic behavior, which was attributed to their nanocrystalline structure of the oxides. Their analysis of these nanocrystalline structure revealed they were composed of MgO and MgAl₂O₄ grains [86]. The formation of the oxide fibres has been described as being assisted by dynamic oxidation from the reaction of atmospheric oxygen with the outward diffusion of magnesium during high temperature plastic deformation [58,86]. Figure 1.15 illustrates the damage features induced on the near-surface layer during lower (420 °C) and higher (545 °C) temperature plastic deformation, while the schematic in Figure 1.16 illuminates the influence of the GBS on the deformation of the oxide fibres [58,86]. The damage to the near-surface deformed layer during plastic deformation was therefore assumed to be sensitive to the operating bulk deformation mechanisms [58]. It was found that the increase in forming temperature was accompanied by an increase in
the ductility of the near-surface layer, and the increase in COF was due to the increase in surface roughness during plastic deformation [58].

Similar experiments performed by Gali et al. [127], which employed the same hot forming tribo-simulator, revealed that the surface features generated on the near-surface deformed layer during plastic deformation at various temperatures influenced the friction changes observed with temperature increase. They reported surface damage to the brittle near-surface deformed layers at low temperatures in the form of cracks, while at higher temperatures, they found increased crack damage at the grain boundaries and an increasingly ductile near-surface deformed layer [127]. The surface damage resulted in two plateaus of surface roughness: the first with low roughness was observed below 300 °C, and the other, due to roughening induced by grain boundary sliding (GBS) as previously reported by Das et al. [86], above 300 °C [127]. The wear of the near-surface layer and COF during tribological contact was also related to the surface damage induced by plastic deformation; however, no wear of the near-surface layer nor material transfer to the tool surface was observed at 150 °C [127]. They observed oxide fibres within the cracks induced in the near-surface deformed layers from temperatures as low as 250 °C, illustrating: (i) the variation of the oxide fibre structures, from rib-like membranes to individual identified fibres; (ii) variation in depths, occurring closer to the surface with temperature increase; (iii) variation in diameters and lengths, which decreased and increased respectively as the temperature increased (Figure 1.17) [127]. They also found that the increased ductility of the near-surface deformed layer at elevated temperatures assisted the formation of fibrous structures [127]. They reported the increased material transfer observed on the tool surface with forming temperature increase, with material
transfer buildup observed from 300 °C [127]. Remarkingly, tribological contact would be initially be between the near-surface deformed layer and the forming tool, they conclude that a ductile surface layer that conformed to the protrusions induced by the sliding of the near-surface grains would lead to a high tendency of material transfer and adhesion to the tool [127].

Figure 1.13 Schematic diagrams showing the delamination process of the near-surface layer (a) crack initiation (b) crack propagation (c) fracture [140].
Figure 1.14 Examination of the Al-Mg plastically deformed at sample at 545 °C and 4×10^4 s^{-1} displaying (a) secondary electron image of oxide cracks which follow the underlying Al-Mg grain boundaries and oxide fibres are stretched across the faces of these cracks, (b) FIB cross section taken along the axis of tensile axis, showing the surface offsets due to GBS of Al-Mg grains and the corresponding fibres formed, and (c) TEM micrograph displaying the fibre sample formation at the surface step formed by the sliding grains [86].
Figure 1.15 Schematic representation of two types of surface damage features at two temperatures: (a) cracks appeared on the oxide layer at 420 °C and (b) superplastic oxide ligaments, triggered by surface offset due to GBS, observed at 545 °C [58].
Figure 1.16 Schematics showing (a) GBS of bulk Al-Mg grains lead to the formation of steps at the surface and (b) surface step formed at the grain boundary due to relative sliding between grain A and grain B of Al-Mg and the tribolayer above the grains stretched over this step generating a fibrous structure [86].
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Figure 1.17 Surface morphology of the Al-Mg sample deformed under $4 \times 10^{-2}$ s$^{-1}$ strain rate showing cracks and fibres at (a) 150 °C, (b) 200 °C, (c) 250 °C, (d) 300 °C, (e) 350 °C, (f) 400 °C and (g) 450 °C [127].

1.4.6 Aluminum transfer in elevated temperature forming

Aluminum transfer to steel tools, at elevated temperatures and contact pressures, is a common phenomenon that occurs during the metalworking processes [87]. The transfer modifies both the tool and the formed aluminum surfaces and also influences the
tribological conditions between the mating surfaces during the forming process [34,45,62]. The type of transfer film induced on the tool surface has been observed to either positively or detrimentally influence the tribological system [45]. Material transfer that builds up and strongly adheres on the tool surface (Fig. 1.18) can result in galling or seizure of the aluminum sheet, leading to damage to the tool and rejection of the formed product, while “slurry” transfer films have been reported to result in low friction [45,145]. The type of transfer layer formed on the tool surface is mostly influenced by the deformation conditions, lubrication and the surface condition of mating alloys [45,47,146]. Interestingly, material transfer is increased at higher work tool surface roughness, and is also know to increasing tool roughness [145,147]. Adhesion and material transfer are also influenced by temperature, surface composition, contact pressure and the sliding velocity [145,147,148].

In the metal forming industry, the initiation of material transfer is difficult to avoid; thus, control of the buildup and type of material transfer is encouraged [45,62,145]. While adhesion and material transfer are believed to be detrimental in other typical forming processes, in the aluminum rolling industry, the development of a transfer layer is actually believed to be beneficial and is encouraged [71,87]. The usefulness of the roll coating is limited due to the occurrence of galling, and it is dependent on its thickness; however, the critical thickness at which roll coatings becomes detrimental is still uncertain [47,87]. This transfer of aluminum to the work roll is referred to as “pickup”, while the transfer layer is called a “roll coating” and is assumed to act as a protective layer that aids in controlling friction. Its absence is thought to lead to refusal of the slabs (Fig. 1.19) [27,71,87]. The material transfer that makes up the roll coating occur
through various suggested mechanisms, including abrasion, adhesion, and delamination [67,70,139,143]. Yet material transfer during the rolling process can also be related to the mechanical, chemical, and thermal interfacial interactions that occur between the roll and the aluminum slab [73].

The roll coating has been described as non-uniform, patchy, continuous or discontinuous, and occurs as streaks aligned in the roll direction [63,87,88]. This coating initiates as individual isolated lumps streaked out in the rolling direction on the work roll surface, but after several hundred impressions a continuous roll coating is developed [89]. The roll coating continues to grow, increasing in amount and continuity as rolling progresses [88]. Physically, the roll coating induces a visible color change in the work roll surface, which differs with the rolling stage [10,87]. The roll coatings developed on the rolls of the reversing mill have been visually described as shiny grey in color, while the hot tandem mill roll coatings are said to be dark black and on the cold rolling mills the roll coatings appear bluish black [87].

However, the appearance of the roll coating has been thought of as an indication of the thickness of the coatings built-up on the work roll [63]. The formation and thickness of the roll coatings has been observed to be dependent, not just on the surface roughness, but also on the topography of the roll as carbides protruding from the roll surface increase the rate of formation of the roll coating [47,73]. After comparing the performances of steel and ceramic work rolls, Howes et al. [63,73] conclude that the roll material had a distinct influence on the development of the roll coatings. They observed that the roll material could influence the development of a continuous roll coating [73]. They went on to suggest that the amount of aluminum transferred that makes up the roll
coating was related to the aluminum wetting characteristics and thermal conductivity of the work roll material [63,73]. Scamans et al. [70] observed that the rate of aluminum transfer during rolling was increased when worn, but rough roll surfaces where coupled with high aspect ratios. Aluminum is generally adhered to the rougher regions of the work roll surface due to the entrapment [87,89]. The surface temperature and number of aluminum sheets rolled have also been reported to influence the thickness and appearance of the roll coating [42,87].

The roll coating has been identified through x-ray photoelectron spectroscopy (XPS) to be mainly comprised of Al₂O₃ and small amounts of aluminum metal [22,42,89]. Treverton et al. [72] state that the roll coating was suspected to be separated from the work roll surface by a relatively featureless film of high aluminum metal content. Yoshida et al. [42] and Smith et al. [89] identified small particles that were oxidized as well as larger particles that made-up the roll that had remained un-oxidized but were coated with oxide. However, the composition of roll coatings developed on commercial purity aluminum have been reported to include aluminum, oxygen, and carbon [42,49,149]. The carbon content of the roll coating is generally accepted to be due to oil residues polymerized at elevated temperatures during rolling [89,149]. Analysis has determined that the carbon in the roll coating might also be a result of metallic soaps that are formed from the reaction of the lubricant with un-oxidized aluminum [10,27,45]. The lubricant comes in contact with the fresh un-oxidized aluminum surface through the cracks in the surface oxide layer on the aluminum slab produced during its deformation [45,146]. The additives of the emulsion can also react with the aluminum surface to induce the tribochemical wear, causing the aluminum to dissolve into the lubricant to
produce metallic soaps [21,150]. The complex organometallic soaps contained in the roll coating due to these reactions have been suggested to be a blend of $\text{Al(RCO}_2\text{)}_2$(OH)$_2$, $\text{Al(RCO}_2\text{)}_2$(OH)$_2$, and $\text{Al(RCO}_2\text{)}_3$ [21,151]. It has been suggested that these organometallic soaps alter the physical behaviour of the roll coatings, influence the friction conditions and reduce further adhesion during rolling [36,45,146].

Howes et al. [73] explored the unlubricated rolling of aluminum enabled by the use of ceramic roll materials and found that the roll coating developed without extensive oxidation. Howes et al. [63] went on to describe the material transfer making up the roll coating under unlubricated conditions to be composed predominately of aluminum metal, and also noticed they were thicker and more continuous than roll coatings developed under lubricated conditions [88,152]. Emulsions, which act as coolants, reduce material transfer as they reduce the aluminum surface temperature due to their water content, with the water concentration playing a critical role in determining the cooling rate [87,88]. The color of the roll coatings have also been related to the production of a polymerized lubricant film, formed from oil adhered strongly to the roll surface, and its thickness: the thicker the film, the darker the color [87]. Therefore, the rolling lubricant and its chemistry have been determined to have a distinct influence on the roll coating formation and composition [42,43].

The emulsions, which offer lubrication during the rolling of aluminum, influence the thickness of the roll coating such that the thickness of the roll coating has been used as an evaluation of the emulsions performance [42,45,47,87]. Yoshida et al. [42,49] observed that the nature of the emulsion, used or fresh, influenced the thickness of the roll coating, while Budd et al. [43] showed that the correlation between the lubricant
composition and the roll coating thickness and distribution. Budd et al. [43] detected lower and more uniform roll coatings with the increase in the base oil viscosity and a decrease in the distribution and coating thickness with the increase in both acid chain length and content. They also noted the detrimental effect of primary alcohols to the roll coating thickness and distribution [43]. Base oil type and concentration, and the oil particle size in the emulsion also influence the roll coating thickness, with naphthenic emulsions inducing thicker roll coatings than paraffinic emulsions [22]. While oleic acid has been reported to disrupt the transfer and buildup of uniform roll coatings, its performance has been observed to be nullified by triethanol amine [22]. The distribution of emulsion on the roll surface has also been noticed to influence of uniformity of the roll coating with the thickest coatings observed where lubrication conditions were worst [71]. In addition, the inclusions of additives has been observed to increase the rate at which the roll coating builds up [71].

The emulsion likewise influences the aluminum oxide-to-metal ratios of the roll coating [22,49]. Yoshida et al. [22,49] stated that the oxide-to-metal ratio and the roll coating thickness increased with the quantity of oil adhered to the surface of the work roll [149]. Based on this analysis they proposed that the mechanism of roll coating formation to include the polymerization of lubricant on the roll surface and the accumulating of aluminum wear debris entrapped in the polymer forming the streaks [22]. Tripathi [87] independently proposed two roll coating buildup mechanisms, relating each one to the rolling speeds, highlighting that the lubrication required at both speeds would be different. He proposed that at low speeds of rolling, a micromechanical interlocking mechanism occurred, which involved the keying of plastically deformed metal in the
rougher profiles of the work rolls [87]. At higher speeds, a tribochemical mechanism became dominant, which was consistent with the mechanism proposed by Yoshida et al. [22]. This process involves the wear of both the roll and the rolled aluminum surface producing debris particles which are trapped in the polymeric film generated by the lubricant [87]. Additionally, Tripathi [87] indicated that the concentration of the wear particles generated, which themselves depend on the lubrication condition, would influence the thickness and appearance of the roll coating. Hui et al. [21] discusses a similar mechanical interlocking mechanism caused by the work roll asperities, as they plough into the rolled aluminum surface and form an adhesive layer that tears away or separates when the local area of the aluminum surface adheres severely to the roll surface depending on the shear strength of the interface.

The work roll surface is imprinted on the rolled aluminum surface, such that the rolled aluminum surface is a reflection of the work roll; consequently, the roll coating would influence the surface roughness of the rolled aluminum [34,73]. Discontinuous and patchy roll coatings could thus result in the roughening of the rolled aluminum strips while defects in the coating would be repeatedly imprinted on the rolled aluminum surface [72,73]. The thin alloy layers that transfer to the work roll to make up the roll coating are oxidized and can easily be transferred back and re-welded to the aluminum surface [67,99,135]. The roll coating buildup is thus a dynamic process with this transfer and back transfer occurring constantly during the hot rolling of aluminum and has been referred to by Smith et al. as a two-way transfer mechanism [71,89]. The back-transfer from the roll coating produce distinct surface disturbances on the aluminum alloy, which are visible to the naked eye and are referred to as “pickup defects” [34,64,87,89]. The
pickup defects endure subsequent rolling stages, reducing the surface quality of the final rolled products [89]. Although the roll coating discontinuously heals the losses induced by back transfer, these defects generated in the roll coating can introduce numerous pickup defects on the rolled aluminum surface before the coating is healed [89]. At this stage, the pickup defects are believed to be a result of an unstable roll coating [88]. Therefore, the roll coating would significantly influence not only the surface appearance of the rolled aluminum alloy, but also the evolution of the near-surface layers [34,42,72]. As roll coatings are believed to be essential for aluminum rolling, it has been suggested that a smooth and fine roll coating is required to obtain aluminum sheets with high surface quality [26,49,87]. In the aluminum rolling industry, a critical function of the emulsion is the development of stable and uniform roll coatings, while steel wire or abrasive-impregnated nylon work roll brushes are applied to the work roll surfaces during rolling to maintain a stable and uniform roll coatings [26].

Figure 1.18 Adhesive galling results on the tool steel and sheet metal contact [153]
Figure 1.19 Image showing material transferred (roll coating) from the stock surface to the work roll after the two-pass rolling [34].

1.5 SUMMARY OF LITERATURE REVIEW

As research into the near-surface deformed layers continues, a greater understanding of their structure and formation is developed, which leads to improved aluminum rolling practices. Work on aluminum alloys provided by the aluminum rolling industry or prepared in the laboratory with pilot mills have revealed the two types of near-surface microstructures and the effect of coupled rolling parameters, such as aspect ratio and roll topography [34,64,67–70]. The evolution of the oxide layers on the surface of aluminum alloys and the effect of rolling passes and magnesium concentration has on these oxide layers has also been rigorously researched [34,64,106,113]. A more comprehensive understanding of the relation of corrosion to the NSM has been derived
through research on the precipitation of intermetallic particles and dispersoids [69,130,134,138,142]. Studies on the NSM during subsequent forming processes have shown the delamination of the NSM during sheet forming and the formation of oxide fibres with damaged areas of the NSM during hot forming [58,86,127,140]. However, there is only a limited understanding of the role of individual rolling or forming parameters play in the development and evolution of the NSM and the influence the NSM has on subsequent forming processes in terms of friction and material adhesion, especially from a tribological point of view. Funds are constantly expended on the removal of these layers, but a better understanding would better aid the control of the NSM formation through rolling and subsequent forming.

The study of the growth of the roll coating through continued material transfer between the roll and the rolled aluminum slab could very likely be relevant to the developments in other aluminum processes such as forming and extrusion [73]. Yet there has been limited research in this area, and the majority of the work on roll coatings has been carried out on commercial purity aluminum alloys. Most of the published work carried out on roll coatings have either been performed by Alcan International in the UK, or Maruzen Oil in Japan, all before the late 1990’s. None of the existing work takes into consideration the effect magnesium additions in aluminum alloys. During the rolling of Al-Mg alloys, the MgO on the alloy surface would be transferred to the work roll surface during passes [139]. In fact, the magnesium-rich oxide layer would come in contact with the work roll first and as a result, the initial roll surface would be covered with this layer, which can act as a barrier to further adhesion of more aluminum to the roll surface [56].
Consequently, a more thorough understanding of the roll coating structure, the evolution of the near-surface layers developed on aluminum alloys, and their influence on subsequent forming processes would aid in developing strategies for improving the formed aluminum surface, thereby reducing the financial cost of aluminum metalworking [71]. This understanding can best be achieved through a methodical investigation of the tribological conditions at the rolling interface and the influence of various individual rolling parameters have on these generic roll and aluminum slab features. This is the planned focus of this thesis.

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CHAPTER 2

THE FORMATION OF MICRO-BLISTERS ON AL-MG ALLOY SURFACES DURING HOT ROLLING

2.1 INTRODUCTION

The oxide-rich near-surface microstructures formed on aluminum alloys during the rolling process affects the tribological conditions between the work roll, lubricant and the work piece and thus, the surface quality of the rolled product [1, 2]. It is therefore critical to have a good understanding of the influence these oxide rich near-surface layers and lubrication have on the tribological factors that affect rolling, such as friction and heat transfer [1, 2]. The composition of the oxide-rich layers are affected by the migration of alloying elements, the atmosphere rolling takes place in and the absorption of the components from lubricants [3]. The oxide type is also dependent on the stage and temperature of the rolling process, with MgO, Al$_2$O$_3$, and Al$_2$MgO$_4$ found at the early stages of rolling of Al-Mg alloys [1, 4]. The growth of the MgO in Al-Mg alloys has been associated with the outward diffusion of magnesium and magnesium vapor transfer through oxide defects and voids [5, 6]. MgO growth is enhanced by the high porosity of the initial MgO surface layer, through which magnesium permeates and either reacts with oxygen or reduces Al$_2$O$_3$ to aluminum [6, 7]. Magnesium diffusion has been reported to be enhanced in the presence of moisture at temperatures above 525ºC due to the formation of OH$^-$ ions [8]. Hence, the characterization of the oxide-rich near-surface microstructures at different stages of hot rolling is vital in determining their interaction with any reactive compounds [3].
The surface oxides on aluminum alloys are self-healing at room temperatures and in the initial oxidation stages [9]. However, as film thickness increases with temperature, cracks appear and further oxidation can occur [9]. In fact, internal oxidation is known to occur in Al-Mg alloys at temperatures above 350 ºC, forming a magnesium-rich oxide surface film [6, 9]. During heating to high temperatures, the outward diffusion of magnesium in aluminum alloys with high magnesium content has been reported to cause blisters due to the formation of voids at the alloy-oxide interface [5]. The growth of these voids facilitates the loss of oxide adhesion and the formation of localized blistered regions [5]. Blisters have also been observed to occur during hot rolling and extrusion of aluminum alloys, due to escaping gases [10, 11]. During hot rolling the oxides on the aluminum alloy can transfer to the work roll through adhesion and abrasion [1].

The hot rolling of aluminum alloys require lubricants for the prevention of material transfer to the roll and control of friction [12]. The lubricant therefore plays a significant role in the tribological interactions at the roll/work piece interface [13]. Emulsions have widely been the lubricant of choice for metal forming operations as they provide both lubrication and cooling [12, 13]. The mechanisms by which emulsions operate at high temperature are very complex, with their tribological behavior being affected by the formation of vapor films, as well as the thickness and the stability of the emulsion [13, 14]. Emulsions do not always distribute enough lubricant into the work piece/ work roll interface [13]. This kind of inadequate lubrication can lead to metal to metal contact and aluminum ‘pick-up’ [12, 14].

The tribological interaction between the work roll, emulsion and the evolving oxide-rich near-surface layers of the aluminum work piece contribute in determining the
surface defects that may be induced during hot rolling. While research has been focused on the defects that appear at the final stages of rolling, these defects are usually carried from the initial hot rolling passes. Further research is necessary to understand the development of surface defects that occur at the initial stage of rolling and thereby determine their contribution to the near surface features observed on the final rolled products. Investigations of the formation of these defects at the initial rolling stages would therefore provide insight into the evolution of the oxide-rich near-surface layers.

This study uses a unique rolling tribo-simulator to investigate the development of surface defects (i.e. micro-blisters) on an Al-Mg alloy during the first hot rolling pass. The rolling conditions that promote the formation of micro-blisters were examined and the micro-blisters were characterized using advanced electron optics techniques.

2.2 EXPERIMENTAL PROCEDURE

Rolling experiments were carried out with the use of a tribo-simulator with a roll-on-block configuration previously described in [15]. The tribo-simulator set-up simulates the tribological surface deformation of a work piece during rolling. The work roll was machined from AISI 440C steel to a diameter of 21 mm. An Al-4.5% Mg alloy, machined into blocks 10 mm wide, 30 mm thick and 95 mm long, was used as the work piece. The surfaces of the work roll and the aluminum specimens were polished to a surface roughness (Ra) of 0.015 µm and 0.020 µm so as to exclude the effects of roll roughness. Optical interferometry was used to analyze the polished AISI 440C steel surface using a WYKO NT1100 in the vertical scanning interferometry, VSI, mode. The surface texture analysis of the AISI 440C roll (Fig. 2.1(a)) displayed the presence of
carbides protruding from the surface. The height of carbide protrusions was measured at several areas to determine their average height as $0.065 \pm 0.006$ µm. Surface profilometry was also performed on an AISI 52100 steel roll, a typical work roll material, which is displayed in Figure 2.1(b) as a comparison. No carbides were observed protruding from the AISI 52100 surface.

Rolling was performed at a temperature of 550 °C with a forward slip of 7% for one pass and a contact pressure of 128 MPa. The Al-Mg specimens were rolled under low (flow rate of 0.2 ml/sec) and high (flow rate of 0.3 ml/sec) lubrication conditions. The lubricant used was an oil-in-water emulsion and was applied continuously at a temperature of 50 °C during the test. It was prepared by shearing the neat oil in water at a 4 % (v/v) concentration, using a homogenizer at 15,000 rpm for 5 minutes. The oil was composed of a paraffinic base oil, a synthetic ester boundary additive and a non-ionic emulsifier.

The specimen surfaces were examined with an FEI Quanta 200 FEG environmental scanning electron microscope (ESEM) under high vacuum and also an FEI Magellan 400 high resolution SEM (HR-SEM). The near surface microstructure of the alloy was examined using a ZEISS NVision 40 Cross Beam workstation (focus ion beam (FIB)) using gallium ions operated at low beam currents with an operating voltage of 30kV. The sample surface was protected by depositing a thin layer of carbon. Cross-sectional trenches were ion milled using the FIB H-bar method. The sample prepared by using the lift-out method was examined using an FEI Titan 80-300 LB transmission electron microscope (TEM).
Figure 2.1 Surface profilometry images displaying the surface topographies of (a) the AISI 440C steel work roll surface with protruding carbides and (b) AISI 52100 steel work roll surface.
2.3 EXPERIMENTAL RESULTS

2.3.1 Surface Analysis of AISI 440C Work Roll

Figure 2.2(a) shows an SEM image of the AISI 440C steel work roll used to hot roll the Al-Mg alloy for 1 pass. Carbides of various sizes were observed on the face of the steel work roll. Material transfer to the work roll surface was dispersed randomly but was also observed piled-up on the faces and edges of several carbides. EDS mapping of the work roll surface (Fig. 2.2(b)) revealed that the material transfer on the carbides was composed of aluminum and magnesium, with several areas being prominently covered in magnesium. The maps associated chromium and carbon as the elemental composition of the carbides and also identified oxygen covered the surfaces of all the carbides regardless of the presence of material transfer.
Figure 2.2 (a) SEM images of the AISI 440C steel work roll surface after 1 hot rolling pass displaying material transfer from the Al-Mg sample and (b) EDS maps of AISI 440C steel work roll displaying composition of carbides and material transfer. Cyan represents chromium, red represents carbon, blue represents magnesium, magenta represents aluminum and green represents oxygen.

2.3.2 Surface Analysis of Al-Mg Work Piece

Micro-blisters were observed covering the surface of the Al-Mg alloy rolled with the AISI 440C work roll after one hot rolling pass under low lubrication flow, as displayed in Figure 2.3(a). The micro-blisters though were absent on the alloy surface when the Al-Mg specimens were rolled with high lubrication flow under the same rolling conditions (Fig. 2.3(b)). Surface analysis of the Al-Mg specimens rolled under high and low lubrication flows revealed that they both possessed darkened material pile-ups dispersed at areas rich in nano-cracks on the surface (Fig. 2.3). Electron dispersive spectrometry (EDS) exposed these darkened piles to be rich in Mg and O. No micro-
blisters were observed on the surface of the Al-Mg alloy hot rolled with an AISI 52100 work roll (with the surface profile displayed in Figure 2.1(b)) under low and high lubrication flows as displayed in Figures 2.3(c) and (d), respectively. Surface features of these rolled Al-Mg samples were similar to those observed for samples hot rolled with AISI 440C under high lubrication flow but they displayed less darkened material piles.

The micro-blisters occurring on the surfaces of the Al-Mg alloy hot rolled with the AISI 440C work roll under low lubrication flow were examined using a high resolution SEM (HRSEM) which revealed their hemispherical shape with a similar surface texture to the surface oxide covering the alloy (Fig 2.4(a)). The diameter of individual blisters was measured to be $1.14 \pm 0.19 \mu m$, although not all the blisters observed were perfectly spherical. A close examination of individual blister surfaces (Fig 2.4(b)) exposed that they had nano-scale bumps of about $50 \pm 20 nm$ in diameter covering the blister surface. Damaged blisters were also detected on the Al-Mg alloy surface (Fig 2.4(c)) and they revealed the hollow structure of the blisters and the smooth inner surface of the blister shell. The damage to micro-blisters was in the form of the collapse or fracture of the micro-blister shell. There was evidence of oxide scales from the collapsed shell within the damaged blister. The surface under the blister appeared to be depressed (Fig 2.4(c)). The damaged blisters therefore provided crevices that can be quickly filled by lubricant or oxide debris.

Al-Mg surfaces hot rolled with the AISI 440C work roll under high lubrication flow (Fig. 2.3(b)) showed no evidence of micro-blisters. Blisters were also not observed when the Al-Mg surfaces were hot rolled with the AISI 52100 work roll which possessed no carbide protrusions from its surface under low (Fig. 2.3(c)) and high (Fig. 2.3(d))
lubrication flows. This suggests that the occurrence of blisters was influenced by the presence of the carbide protrusions on the steel roll surface and was eliminated when lubrication flow was increased, highlighting these as factors that facilitate its manifestation.
Figure 2.3 SEM images of the surface of Al-Mg alloy after 1 hot rolling pass showing (a) blisters on sample rolled with AISI 440C under low lubrication, (b) magnesium rich piles on sample rolled with AISI 440C under high lubrication, (c) magnesium rich piles on sample rolled with AISI 52100 under low lubrication, and (d) magnesium rich piles on sample rolled with AISI 52100 under high lubrication.
Figure 2.4 HR-SEM images of the blisters formed on Al-Mg alloy surface after 1 hot rolling pass under low lubrication showing (a) surface texture of blister and oxide-rich near surface layer (b) nano-scale bumps on the blister surface and (c) damaged blister revealing debris inside the hollow shell.

2.3.3 Subsurface Analysis of Micro-blisters

Cross-sectional FIB microscopy analysis displayed in Figure 5(a) confirmed the micro-blisters possessed a shell structure with a hollow centre. The Al-Mg alloy surface was covered with an oxide-rich layer with a thickness of about 86 ± 10 nm. A difference in height observed between the surface beneath the blister and the alloy surface induced a crater-like appearance under the blister. The depth of this depression was measured to be 0.12 ± 0.05 µm. The surface beneath the blister was smooth in appearance, as were the inner walls of the blister shell. TEM analysis revealed the nanocrystalline structure of the micro-blister shell (Fig. 5(b)). The micro-blister shell was observed to possess an uneven thickness (76 ± 30 nm) with larger thickness occurring at the crown and base of the shell from the TEM image displayed in Figure 5(b) A high resolution EDS map (Fig 5(c)) was used to determine that the shell was comprised of an outer layer, rich in magnesium and
oxygen, and an inner layer having a high concentration of aluminum. Selected area electron diffraction (SAED) analysis of the blister shell (Fig 5(d)) confirmed the nanocrystalline structure of the shell layers, and was used to identify MgO (111), (200) planes, Al₂O₃ (113), (018) planes and Al (311) plane. TEM analysis also revealed several high porosity MgO-rich pockets within the oxide-rich surface layer.
Figure 2.5 Cross section of blister displaying (a) surface features within the blister (b) transmission electron micrograph image of the blister, (c) EDS map overlay showing elemental composition of blister shell from the region within the insert box in (b) with elements Al (green), O (red) and Mg (blue) and (d) SAED pattern of the region shown in (c)
2.4 DISCUSSION

The structure of the shell of the hollow micro-blisters on Al-Mg alloys after 1 hot rolling pass under low lubrication flow is nanocrystalline and comprised of an inner layer of Al/Al₂O₃ and an outer layer of MgO (Fig. 5). The micro-blisters structure indicates a possible mechanism responsible for its formation. The porosity of the oxide-rich near surface layer along with cracks within this layer allow for the permeation of water vapour from the emulsion through the oxide-rich layer to the sub-surface aluminum. The penetrated water vapour then reacts with the aluminum to produce hydrogen and Al₂O₃ as the most stable products at the interface between the oxide-rich near-surface layer and the alloy according to the equation [16, 17]:

\[
2\text{Al} + 3\text{H}_2\text{O} = \text{Al}_2\text{O}_3 + 3\text{H}_2
\]  

(1)

It is possible for a similar reaction between magnesium and water vapour to produce hydrogen as magnesium is more reactive than aluminum. However, there is no evidence in the blister structure at this time to substantiate this reaction. The hydrogen production reaction between aluminum and water vapour can be facilitated by Cr₂O₃ covering the carbides on the steel roll surface, which can act as a catalyst [17]. The oxidation of chromium carbides to form Cr₂O₃ which inhibits further oxidation of carbides has been reported in literature [18, 19]. This is inferred from the oxygen map in Figure 2(b). As seen in the same Figure, oxygen was coincident on all carbides regardless of the presence of aluminum and magnesium transfer on the carbide surfaces. The increase in the porosity at the interface induced by the hydrogen gas generation allows for the delamination of the oxide-rich near-surface layer consisting of the freshly formed
Al₂O₃ below the previously existing MgO-rich oxide surface layer. This layer could adhere to the carbide surface protruding from the AISI 440C steel roll (Fig. 1(a)) which would apply a compressive stress onto the alloy surface during rolling as indicated by the presence of depressed craters beneath the blister (Fig. 5(a)).

Oxide transfer from the aluminum alloy surface to the carbide surface does not occur for all the carbides (Fig. 2(b)). A comparison of the average depth of the crater beneath the blister (0.12 ± 0.05 µm) and the average height of the carbide protrusions (0.065 ± 0.006 µm) reinforces this postulation. Carbide protrusion heights were measured using surface profiles obtained and ranged between 0.04 – 0.154 µm, while the depth of the craters beneath blisters ranged between 0.087 – 0.142 µm below the oxide-rich surface layer covering the alloy surface. We consider the minimum crater depth beneath the blister as the minimum height required for a carbide protrusion to cause the formation of a blister and the crater beneath the blister. An analysis of carbide protrusion heights covering the AISI 440C work roll showed that the percentage of the total carbide protrusions with a measured height over 0.087 µm was 44%. Therefore, less than half of the carbides covering the surface of the steel work roll would initiate blister formation.

The adhesion of the oxide-rich near-surface layer to the carbide surfaces in combination with the pressure from the hydrogen gas evolving beneath the oxide-rich surface layer initiate the formation of blisters. The evolution of hydrogen gas causing the formation of blisters and bubbles beneath an oxidized aluminum shell has been previously reported [11, 16, 20]. The aluminum nanocrystals observed in the EDS map of the blister shell and the SAED analysis in Figures 5(c) and (d), can be explained by magnesium diffusion in Al-Mg alloys. The diffusion of magnesium from the bulk alloy
through the $\text{Al}_2\text{O}_3$ layer of the blister shell reduces this oxide to metallic aluminum according to the equation [5-7, 21];

$$3\text{Mg} + \text{Al}_2\text{O}_3 = 3\text{MgO} + 2\text{Al} \quad (2)$$

The magnesium diffusion through the blister shell allows for enrichment of the outer MgO layer; as such, no necking occurs in the shell due to the applied strain (Fig. 5(a) and (b)). The diffusion of magnesium to the surface of Al-Mg alloys at high temperatures has been reported to cause blisters on these alloys [5]. The formation of the blisters can therefore be attributed to the evolution of the hydrogen gas and magnesium vapour [5, 6] at the oxide-rich near-surface / bulk interface, accompanied by the adhesion of the oxide-rich near-surface layer to the carbide surfaces. A schematic of this mechanism is displayed in Figure 6.

The proposed mechanism for micro-blister formation highlights the importance of sufficient lubrication in the mitigation of blister formation as this would reduce adhesion of the Al-Mg oxide-rich near-surface layer to the carbide particle protrusions. This was observed on Al-Mg surfaces hot rolled with increased lubrication flow (Fig. 3(b)). Low lubrication flow would hinder the proper distribution of lubricant during hot rolling and would increase the possibility of lubricant break down at high temperatures. The increased possibility of lubricant break down would in turn reduce the load bearing capacity of the lubricant at the regions of lubricant break down and increase the likelihood of material adhesion to the protruding carbide surfaces which enhance micro-blister formation. This study reinforces the need for proper lubrication during hot rolling, especially during the early stages of rolling when surface defects are likely to occur.
Figure 2.6 Schematic for the mechanism of micro-blisters formation on Al-Mg alloys during hot rolling.
2.5 CONCLUSIONS

Micro-blisters induced on an Al-Mg alloy by an AISI 440C steel work roll during a hot rolling regime of one pass at 550 °C with a forward slip of 7% under a low lubrication flow were studied. The steel work roll was polished, exposing the carbide particles protruding from the steel surface. The following conclusions were drawn:

1. Micro-blisters with a diameter of 1.14 ± 0.19 µm were observed covering the surface of the Al-Mg alloy after one hot rolling pass under low lubrication flow.

2. The structure of the blister shell was nanocrystalline and it was comprised of an outer MgO layer with an Al/Al₂O₃ lining the inside of the shell.

3. It is proposed that the formation of micro-blisters is due to a combination of two mechanisms occurring simultaneously. These mechanisms are the delamination of the oxide-rich surface layer due to adhesion to the carbide particle protrusions on the steel work roll and the evolution of hydrogen gas beneath the oxide-rich surface layer, which occurs from the reaction of water vapor (permeated through the porous oxide-rich surface layer) with the subsurface aluminum.

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CHAPTER 3

A STUDY OF THE DEVELOPMENT OF MICRO-CRACKS IN SURFACE/NEAR SURFACE OF ALUMINUM-MANGANESE ALLOYS DURING HOT ROLLING

3.1 INTRODUCTION

Enhanced shear deformation experienced during metal forming processes, such as rolling, is known to influence the surface quality of the final product [1 - 3]. Hot rolling, in particular, has been reported to induce a thin, heavily deformed tribolayer on aluminum alloys. This tribolayer has been characterized as having a microstructure comprised of ultrafine grains with oxide-decorated boundaries [2 - 10]. The surface appearance of aluminum alloys has been described as being etched with the surface morphology of the mating surface (the work roll), shingled, and having surface cracks transverse to the rolling direction [2 - 5, 7].

The shingling and surface cracks have been attributed to the interaction between the aluminum alloy and the roll surface, and result in the surface roughening of the alloy [2 - 6]. The appearance of shingles has been recognized as an indication of the presence of a near-surface layer [4, 5]. Though transverse cracks have been identified as a surface feature observed along with shingles, research into their manifestation is limited. Fishkis et al. [2] first reported that these cracks were initiated during the first few rolling passes and then covered by thin layers of aluminum on successive passes, leading to a shingled surface appearance. The lower ductility of the surface layer, shear stress reversals in the roll bite, and metal transfer/back-transfer to the roll were reported as being responsible for the crack formation [2]. Liu et al. [4] observed cracks that showed delamination of the
near surface to different degrees. Shingles have also been reported to enhance the possibility of the appearance of holes and cracks in the alloy subsurface, with holes having the potential to evolve into subsurface cracks [6, 11]. The areas around these cracks were noted to possess fine grains with diameters of about 100 nm [6]. The evolution of cracks on the alloy surface during rolling is acknowledged to affect the final surface quality of rolled products, with the depth of their propagation determining if the final product is acceptable [12, 13].

The transverse cracks in the flat rolling of steel have been extensively analyzed, and the closure and growth of cracks in relation to crack shape, contact pressure, and crack tip stress have been examined [12, 13]. It has been observed that with higher temperatures around the perimeter of cracks, compressive stresses can cause crack healing to occur [12]. The behavior of cracks during hot rolling is not well understood. Most researchers assume that crack defects exist on the strips before the rolling process and investigate their evolution during rolling using field emission microscopy (FEM), while in the rolling industry, these cracks are believed to occur only during cold rolling [12, 13]. Similar research of micro-cracks on aluminum alloy surfaces has noted that cracks initiate in the brittle oxide film at locations of maximum stress, and act as sites through which aluminum is extruded [1]. These cracks are modeled as being within the oxide layer, forming near the entry of the bite, and leaving the alloy surface with porous oxide fragments [1].

Magnesium’s diffusion to the surface of aluminum alloys is well documented [2 - 4, 14 - 17]. MgO has been observed consistently as the topmost oxide layer on magnesium containing aluminum alloys after hot rolling, due to its high diffusion rate.
and affinity for oxygen [2 - 6, 14 - 18]. The thickness of the MgO on the aluminum alloy has been reported to be affected by the magnesium concentration in the alloy [17, 18]. This oxide has also been observed to reduce aluminum adhesion to steel surfaces, especially at high temperatures [17, 18].

This paper presents results of a study on the evolution of surface damage in the form of transverse cracks occurring on aluminum alloy surfaces during hot rolling. High resolution surface and cross-sectional images are presented to show the evolution of the crack morphology during sequential passes, and a mechanism for crack formation is proposed.

3.2 EXPERIMENTAL PROCEDURE

A hot rolling tribo-simulator designed at the University of Windsor, the working principle of which has been previously described by Riahi et al. [3], was used to study the tribological aspect of the hot rolling process. It possesses a roll-on-block configuration and allows for the variation of several rolling parameters, enabling the examination of their individual effects on the rolled alloy surfaces.

The roll and workpiece can be set to run at various speeds, while a lubrication system sprays lubricant on the roll. Rolling was simulated by allowing the revolving roll to run across the face of the test specimen. A thermocouple was placed in a hole drilled into the test specimen, so as to monitor the exact surface temperature of the alloy. The lubricant used was an oil-in-water emulsion with a 4% (v/v) concentration.

An AISI 52100 steel roll with a diameter of 21 mm was used. The test specimens used were machined from an Al-Mn alloy piece (containing 0.8 – 1.4 % Mg, 0.8 – 1.3%
Mn, 0.8% Fe, 0.6% Si, 0.25% Zn, 0.05 – 0.25% Cu) cut to dimensions of 10.5 mm width, 30 mm height, and a length of 95 mm. The roll surface and the aluminum test specimens’ 10.5 mm by 95 mm faces were polished using a 1 µm diamond suspension and then cleaned ultrasonically in acetone for five minutes to remove surface contaminants. The roll was polished to eliminate the effect of roll roughness and to effectively study the effect of rolling stresses on the surface defects. It was cleaned with a 15% (wt/wt) sodium hydroxide solution to remove any aluminum transfer, and then polished to maintain the surface roughness after each pass.

The rolling conditions observed for these tests are listed in Table 3.1. A total of four passes in the forward and reverse directions were carried out, simulating industrial breakdown rolling. The roll and stage speeds were kept constant for a constant forward speed of 7%. The temperature of each subsequent pass was reduced by 10°C from a starting temperature of 550°C.

The specimen surfaces were examined with an FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. Surface roughness measurements were then made using a WYKO NT1100 optical surface profilometer in the vertical scanning interferometry (VSI) mode. The average surface roughness ($R_a$) was determined from several areas, and the mean values of these measurements were recorded for each pass. The near-surface microstructures of the specimens were examined with a ZEISS NVision 40 Cross Beam focused ion beam (FIB) microscope after each rolling pass, using gallium ions operated at low-beam currents and an operating voltage of 30kV. The area of interest was protected by depositing a thin layer of tungsten or carbon. Cross-sectional trenches were ion-milled using the FIB H-bar
method. The samples, which were prepared with the lift-out method, were examined using an FEI Titan 80-300 LB transmission electron microscope (TEM).

Table 3.1 Pass schedules in the hot rolling experiments

<table>
<thead>
<tr>
<th>Pass</th>
<th>Temperature (°C)</th>
<th>Direction of Pass</th>
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<tbody>
<tr>
<td>1</td>
<td>550</td>
<td>Forward (F)</td>
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<tr>
<td>2</td>
<td>540</td>
<td>Reverse (R)</td>
</tr>
<tr>
<td>3</td>
<td>530</td>
<td>Forward</td>
</tr>
<tr>
<td>4</td>
<td>520</td>
<td>Reverse</td>
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3.3 EXPERIMENTAL RESULTS

3.3.1 Surface Morphology Developed During Deformation: Surface Roughness

To better understand the effect of the deformation stresses on the alloy surface, the surface topography was examined after each pass and is represented in Figure 3.1. A significant increase in surface roughness ($R_a$) was observed after the first pass, from 0.020 µm (after polishing) to 0.365 µm. The surface topography taken after the second pass displayed a decrease in surface roughness, dropping to 0.167 µm. A continual reduction with each subsequent pass occurred, dropping to 0.130 µm after the third pass and 0.095 µm after the fourth pass. The surface profiles in Figure 3.1 display the surface roughness reduction after each pass, with distinct grain boundaries appearing from the
second pass in Figure 3.1 (c) through the fourth in Figure 3.1 (e). Deep depressions were observed after the first pass (shown in Fig. 3.1 (b)) and can be compared to the smooth polished surface displayed in Figure 3.1 (a).

The SEM images in Figure 3.2 display the evolution of surface features that form after each pass, and are responsible for the initial increase and subsequent decrease of surface roughness in the same area. These SEM images parallel the optical profiles displayed in Figure 3.1 only in that they are from the same surfaces. Figure 3.2 (a) hints at a misalignment of grains at their boundaries after the first rolling pass. An overlapping of grains at their boundaries, which have likely been deformed during sliding, is observed to produce a step in this figure. The significant increase in surface roughness after the first pass is due therefore to grain boundary sliding (GBS). Figure 3.2 (b) displays the surface after the second pass in the reverse direction. The surface appears slightly darker with surface features, such as transverse cracks and grain overlaps, observed after the first pass, and visually appears compressed or flattened. An examination of the grains’ step heights from the surface topography data confirmed a reduction, from an average of 0.47 ± 0.25 µm after the first pass to 0.37 ± 0.17 µm after the second pass, thus reducing the initially higher surface roughness. The surface of the sample after the third pass is shown in Figure 3.2 (c). It displays less distinct grain boundaries, while the surface profile data indicates further grain step height reduction to 0.27 ± 0.13 µm. After the fourth pass, as seen in Figure 3.2 (d), the grain steps and overlapping visually appear to have been mostly eliminated; this is supported by the reduced surface roughness evident in Figure 3.1 (e). As observed in Figure 3.2, no shingles appear on the alloy surface at any stage during the rolling process. The surface roughness increase after the first pass is
due to the sliding of the grains, and its reduction is instigated by rolling stresses, causing the sliding of the grains to yield an average grain step height reduction. Figure 3.2 also shows the initiation of transverse cracks resulting from the sliding and overlapping of grains, which will be discussed in the next section.
Figure 3.1. Surface profilometry images of the Al-Mn alloy displaying the surface roughness after (a) polishing (pre-rolling) (b) 1 pass, (c) 2 passes, (d) 3 passes, and (e) 4 passes.
Figure 3.2. Scanning electron images of the Al-Mn surface taken from the same region after (a) 1 pass, (b) 2 passes, (c) 3 passes, (d) 4 passes
3.3.1.2 Transverse Cracks on Alloy Surface

Figures 3.2 (a) and 3.3 (a) show the initiation of transverse cracks as being due to the overlapping of the grains at the boundaries. Transverse cracks have been closed by the fourth pass, as seen in Figures 3.2 (d) and 3.3 (b), due to the sliding of the grains in reverse to the original sliding direction. Figure 3.3 (a) also shows damage occurring to the surface intermetallic particles, identified from literature as Al$_6$(Fe,Mn) and α-Al$_{12}$(Fe,Mn)$_3$Si [3, 11], which appear to have been broken during rolling. Intermetallic damage was observed to increase after each pass in the form of the number of cracks and crack width.

Figure 3.4 displays a different crack tracked at higher magnification through four sequential rolling passes. The morphology of the crack after the first pass, as seen in Figure 3.4 (a), shows that the crack occurs at the grain boundary with the overlapping of the grains; by the second pass (Fig. 3.4 (b)), the grain overlap has folded back and compressed into the crack at the grain boundary, forming two parallel transverse cracks. Nano-cracks can also be observed covering the surface after this pass. These nano-cracks become more prominent and form a network by the third pass (Fig. 3.4 (c)), while the material between the transverse cracks continues to be compressed without closing either crack after four passes (Fig. 3.4 (d)).

A cross-section was made from the cracks displayed in Figure 3.4 (d), and is presented in Figure 3.5. The transverse cracks observed on the surface are seen to propagate to a subsurface depth of 0.78 µm. The cracks are seen to propagate toward each other and, as shown in Figure 3.5 (b), along grain boundaries. Some nano-cracks observed on the surface are also seen propagating to the subsurface to a depth of 0.31
\[105\, \mu m\]. These cracks, though, appear to be part of the oxide layer covering the surface of the alloy. The depth of the transverse cracks and their propagation along the grain boundaries prompted a closer examination of the subsurface evolution of these cracks and the effect that each rolling pass has on it; this is examined in the next section.

![Figure 3.3](image1.png)

**Figure 3.3.** Scanning electron images of Al-Mn surface showing evolution of the same surface crack after (a) 1 pass and (b) 4 passes.
Figure 3.4. Scanning electron images of the Al-Mn surface at high magnification showing evolution of a surface crack and appearance of nano-cracks after (a) 1 pass, (b) 2 passes, (c) 3 passes and (d) 4 passes
3.3.2 Surface Morphology of Crack Evolution during Deformation

3.3.2.1 Subsurface Crack Propagation

Subsurface examination was performed on two cracks observed at grain boundaries after deformation from the first rolling pass. Figure 3.6 displays the SEM images of both cracks, referred to as crack 1 (Fig. 3.6 (a)) and crack 2 (Fig. 3.6 (b)), and the area where the FIB ion milling was performed. These cracks both occurred at overlapping grain boundaries, forming steps. Crack 1 is a transverse crack, occurring perpendicular to the rolling direction, while crack 2 runs across the surface at a 33° angle to the rolling direction. Both cracks were monitored individually as they evolved with each pass.

Considering crack 1 with the subsurface features displayed in Figure 3.7 (a), crack propagation can be observed after the initial rolling pass, with the width of the crack opening being 0.85 µm. Crack branching, the propagation of new cracks from an existing
The crack face, is also observed, with the crack branch propagating from the subsurface face of the initial crack. The crack branch appears closed but adds to the depth of the initial crack, extending it vertically to a depth of 0.91 µm below the surface. Electron dispersive spectrometry (EDS) analysis of the cracks shows the crack branch (Fig. 3.7 (a) spectrum 1) as slightly richer in magnesium and oxygen in its surroundings than the initial crack and the bulk of the alloy (Fig. 3.7 (a) spectrum 2). The initial main crack after the second pass in the reverse direction (Fig. 3.7 (b)) appeared to have been reduced in depth, and the width of the crack opening reduced to 0.64 µm. However, the crack branch has developed, propagating to an increased depth of 0.97 µm with its crack faces separated. The EDS analysis (Fig. 3.7 (b) spectrum 1) showed significant magnesium and oxygen enrichment at the immediate surroundings of this crack. EDS analysis along the grain boundary right below the crack (Fig. 3.7 (b) spectrum 2) displayed no magnesium counts, indicating that the magnesium enrichment was peculiar to the crack.

The head and tail of the crack after the third pass is shown in Figure 3.7 (c). The initial crack, as seen in the figure, was closed up at the surface, but the crack branch had become the prominent crack, widening out (that is, the distance between the crack faces had amplified) and increasing in depth to 1.50 µm. The surface morphology of the crack face was also observed to be rougher, with a thin magnesium-rich layer surrounding the crack. This magnesium enrichment around the crack continued to be peculiar to the crack, as areas just below the crack showed low magnesium counts. At the fourth pass, the depth of the crack was 1.65 µm, with a slight opening at the surface (Fig. 3.7 (d)). The crack continued to propagate along grain boundaries, with crack branches initiating from the faces of the crack. EDS analysis along the crack continued to show a large
enrichment of magnesium close to the surface of the crack (Fig. 3.7 (d) spectrum 1), with magnesium content reducing along the depth of the crack. The region immediately surrounding the crack continued to be richer in magnesium, such that magnesium concentration was higher closer to the crack face.

The subsurface structure of crack 2, which occurred at a 33° angle relative to the rolling direction, is displayed in Figure 3.8 (a). The crack opening at the surface had a width of 0.49 µm and a crack depth of 1.21 µm. The regions immediately surrounding the crack were richer in magnesium and oxygen (Fig. 3.8 (a) spectra 1 and 2) than regions farther from the crack faces, similar to crack 1. This trend continued, with the magnesium content around the crack increasing with each pass. However, the crack depth continued to reduce with each pass, to 1.08 µm after the second pass, to 0.83 µm after the third pass, and to a final depth of 0.71 µm after the fourth pass. The crack was also observed to close up continually with each pass; at the fourth pass, it was completely closed up, with only voids and holes observed. The magnesium content within the crack continued to increase, as seen in Figure 3.8 (b) spectra 2 and 3. Similar to crack 1, the highest concentration of magnesium was observed closest to the surface (Fig. 3.8 (b) spectrum 1).
Figure 3.6. Scanning electron image of the two micro-cracks monitored (a) Crack 1 (b) Crack 2.
Figure 3.7. FIB cross sectional images and EDS analysis of propagation of Crack 1 after (a) 1 pass (b) 2 passes (c) 3 passes and (d) 4 passes.
Figure 3.8. FIB cross sectional image and EDS analysis of propagation of Crack 2 after (a) 1 pass and (b) 4 passes.

3.3.2.2 TEM Characterization of Subsurface Cracks

TEM micrographs of the cross-sections of crack 1, as monitored through the focused ion beam micrograph in Figure 3.7 (d), are illustrated in Figure 3.9 (a). It displays the crack after four passes in forward-reverse directions. The crack propagation along the grain boundaries into the subsurface is clearly indicated here. The crack opening at the crack surface was measured as 359.4 nm, with a reduced width of 65.6 nm toward the root of the crack. The tail end of the crack, which was also propagating along the grain boundary, now appears to propagate from the crack face in the form of a crack branch.
An EDS map of the crack shows the crack to be rich in magnesium and oxygen (Fig. 3.9 (b)). This correlates well with earlier EDS analysis carried out during FIB analysis. EDS line scans were carried out across the crack and the grain boundary along which the crack propagated (Figure 3.10). Figure 3.10 (a) confirms the rich magnesium and oxygen content surrounding the crack, represented by the peaks in the graphs (about 150 nm wide) for both elements, and lower contents in areas away from the cracks. The peaks in magnesium and oxygen content represented in the line scan graphs show a depletion of aluminum in the regions where the magnesium peaks occur. This correlates with the EDS maps, which showed little to no aluminum in regions where magnesium content is enriched. The same observation was made for the crack branch. The peak in magnesium and oxygen content was not observed across the grain boundary, which shows constant magnesium and oxygen content (Fig. 3.10 (b)). The lack of distinct magnesium and oxygen peaks was also observed at grain boundaries away from the crack, which indicates that the diffusion of magnesium and oxygen is to the high-energy free surface of the cracks.

A closer examination of the crack from the high resolution TEM (HRTEM) image (Fig. 3.11) exposes a layered structure of the crack. An EDS map overlay of the crack was used to define these layers, and revealed that the face of the crack was composed of aluminum and oxygen. It also hints at a possible Al₂MgO₄ spinel layer lying between the first layer and a MgO layer, which lies at the edge of the crack right before the bulk material. Comparison of this EDS map overlay with the HRTEM indicated that these layered oxides on the crack possessed different structures. Using these analyses in combination with composite HRTEM images and corresponding selected area electron
diffraction (SAED) analysis, the structures of these layers were mapped. Figure 3.12 displays the map of the crack, showing the composition and structure of the crack regions. The aluminum oxide region observed at the face of the crack is about 30 nm thick and amorphous in nature. The Al$_2$MgO$_4$ spinel region, that is, the layer lying parallel to the Al$_2$O$_3$ layer, has a nanocrystalline structure with interplanar spacing of 0.12 nm, corresponding to the {533} plane of the face centered cubic (FCC) phase. A thin amorphous layer with embedded nanocrystalline magnesium and aluminum oxides, 1–4 nm in size, lies between the Al$_2$O$_3$ and Al$_2$MgO$_4$ layers. These nanocrystalline grains have interplanar spacing of 0.24 nm and 0.26 nm, which corresponds to the {311} and {104} planes of the face centre cubic (FCC) phase for $\gamma$-Al$_2$O$_3$ and rhombohedral $\alpha$-Al$_2$O$_3$, respectively. Planes {220} and {200} were also identified within this region, providing evidence of the extent of magnesium diffusion to the crack surface. Further away from the crack’s face, the nanocrystalline structure becomes more prominent, with grain size increasing with each parallel layer up to the MgO layer surrounding the crack. This layer, which lies 80 – 130 nm from the crack’s face, is nanocrystalline, with a grain size of 10 – 30 nm, larger than those observed in the Al$_2$MgO$_4$ and Al$_2$O$_3$ layers. The EDS map overlay and line scans indicate that the oxygen content in the Al$_2$O$_3$ layer at the crack’s face was much less than that in the inner MgO layer. It is therefore probable that some unoxidized aluminum nanocrystals are present in the second amorphous Al$_2$O$_3$ layer.
Figure 3.9. (a) TEM micrograph of the cross-section of Crack 1 after 4 passes showing crack propagation along grain boundaries and (b) EDS map from the boxed region in (a).
Figure 3.10. EDS line scans of the cross-section of Crack 1 after 4 passes (a) across the crack and (b) across the grain boundary which the crack propagates.
Figure 3.11. Cross section of Crack 1 at 4 passes displaying (a) EDS map overlay of the Al (green), O (red) and Mg (blue) and (b) Bright field HRTEM image of region within the insert box in (a), layer composition is confirmed in next figure.
Figure 3.12. HRTEM images of Crack 1, mapping the individual layers observed along with 2D-FFT patterns taken from these layers confirming the amorphous to crystalline transition of the crack face. Crack region mapped is displayed in the insert above the HRTEM images.
3.4 DISCUSSION

3.4.1 Effect of Rolling Passes on Crack Evolution

As stated earlier, no shingles were observed on the alloy surface after any of the rolling passes with the smooth roll. This indicates that the surface roughness of the work roll is an important factor in the formation of shingles on aluminum alloy surfaces [2]. In earlier work with rougher rolls, shingles were observed on the alloy surface, and a mechanism for their formation was proposed [3]. In the aluminum rolling industry, rough work rolls are required to draw material into the roll bite, so while smooth rolls are not practical, the control of the work roll surface roughness would contribute to controlling shingle formation.

Micro-cracks were observed on alloy surfaces at grain boundaries, as displayed in Figure 3.5. These cracks appear to have formed by the sliding of grains due to high shear stresses experienced during hot rolling. GBS has been reported to occur during the high temperature forming of aluminum alloys [14]. The sliding of the surface/subsurface grains under the rolling forces causes overlapping at the grain boundaries, inducing the crack observed in Figure 3.4 (a) after the first rolling pass. These cracks propagate along the grain boundaries into the subsurface, as seen in the cross-sections in Figures 3.7 (a) and 3.8 (a), with depths of 0.91 µm and 1.21 µm, respectively.

The second rolling pass in the reverse direction reduces the opening of these cracks, in effect closing the cracks at the surface. This is due to the rolling forces applied by the work roll, reducing the step heights of the grains. This induces a more level surface, as observed from the distinct surface roughness reduction displayed after this
rolling pass, and the continual surface roughness drop with each subsequent pass (Fig. 3.1 (a) – (d)). The reduction of surface roughness with each pass is also observed in industrially rolled aluminum products [2]. At the subsurface region, the crack either closes or propagates deeper after the first rolling pass; this would appear to be affected by the orientation of the cracks in relation to the rolling direction (Figs. 3.7 (d) and 3.8 (b)).

Crack 1, which is oriented transverse to the rolling direction (Fig. 3.6 (a)), continues to propagate deeper into the subsurface after each pass, with crack branching observed from the first pass (Fig. 3.7 (d)). Crack 2, however, which is oriented at a 33° angle relative to the rolling direction (Fig. 3.6 (b)), begins closing by the second pass. By the fourth pass, the only evidence of this crack’s existence is the rich magnesium and oxygen content, as well as the voids and holes observed (Fig. 3.8 (b)). Therefore, cracks that occur at grain boundaries transverse to the rolling direction at the first rolling pass appear inclined to propagate, while cracks that occur preferentially in other directions experience healing on subsequent rolling passes.

3.4.2 Characterization of Transverse Cracks

The TEM micrograph of the crack in Figure 3.9 (a) confirms crack propagation along the grain boundaries. The crack faces possess features similar to those found in the near-surface layers with an amorphous Al$_2$O$_3$ layer covering a rich MgO layer and an Al$_2$MgO$_4$ spinel layer. In industrial rolling of aluminum alloys with rougher rolls, similar transverse cracks can be covered by material transferred between the work roll and ingot surface, eventually becoming part of the near-surface microstructure, as observed by
Fishkis et al. [2]. Tzedaki et al. have observed similar cracks at the surface and near-surface regions of aluminum alloys, with rich MgO layers surrounding cracks [6].

Magnesium diffusion to the crack begins from the cracks’ formation at the grain boundary with the first pass, and continues during subsequent hot rolling passes. Magnesium has a high diffusion coefficient in aluminum alloys and a high affinity for oxygen, which aids the MgO formation [15]. The magnesium-rich regions of crack 1 observed in the EDS map in Figure 3.9 (b) match the oxygen-rich regions, confirming the results from the line scan of the same crack shown in Figure 3.10 (a), where the magnesium peaks correlate with the oxygen peaks. These results indicate that these areas are rich in MgO. The EDS analyses in Figure 3.8 (b) show the same high magnesium region within the closed crack. The magnesium-rich region appears to keep growing after every rolling pass. This is due to high temperatures and deformation, which aid magnesium diffusion to free surfaces [3].

The enrichment of MgO observed at the crack faces and not at the grain boundaries confirms that the magnesium enrichment occurs after crack formation. The cracks form free surface areas, to which magnesium diffusion occurs at a high diffusion rate. At the initial formation of a crack, the aluminum surface is exposed and oxidized, allowing for the formation of an amorphous Al$_2$O$_3$ layer. The formation of nanocrystalline $\gamma$-Al$_2$O$_3$ is then influenced by temperature, oxide film thickness, and the magnesium content of the oxide film [16, 19]. Magnesium diffusion to the surface occurs because of the exposed free surface of the crack interacting with Al$_2$O$_3$, forming the Al$_2$MgO$_4$ spinel and MgO layer [16, 18].
As magnesium enrichment at grain boundaries was not observed (Fig 3.10 (b)), crack formation cannot therefore be due to incipient melting, caused by high magnesium concentrations at grain boundaries. The mechanism of crack formation is rather the movement or sliding of the surface and near-surface grains under rolling stresses. The shear stresses and frictional forces applied during rolling occur close to the surface and introduce shear stresses into the contact and near surfaces, causing grain boundaries to yield [20]. The cracks caused by this action continue to propagate or close up (depending on their orientation) on further shifting of these grains at the second rolling pass, with high deformation stresses causing branching and crack propagation in subsequent passes. Cracks at grain boundaries of superplastic aluminum alloy surfaces deformed at low strain rates and high temperatures have been reported by several researchers [14, 18].

3.4.3 Crack Healing

Crack healing in Al-Mn alloys occurs from the second rolling pass, with rolling stresses driving separated grain boundaries or crack faces together. This process allows for the welding of the crack faces once they meet during this high temperature compression process. The diffusion of magnesium to the crack faces allows for the formation of MgO in these regions; thus, the crack faces are covered in oxide. As such, metal-to-metal contact does not occur, preventing the proper healing of the crack through welding. Rather, the magnesium oxide on the crack faces fills the spaces, essentially sealing the crack (crack healing). This magnesium-enriched area lining the crack remains on subsequent passes, with void formation within it occurring during passes. The voids in this MgO-rich area, as observed in Figure 3.8 (b), are possibly due to areas of the crack
that have not completely healed or sealed with MgO. During subsequent passes, these types of cracks could end up becoming part of the MgO-enriched near surface, where voids have been typically reported.

In summary, despite the traditional belief that micro-cracks occur during cold rolling or predate rolling altogether, it has been observed that micro-cracks do occur during hot rolling of aluminum alloys. These cracks are not just part of an oxide layer, but propagate into the alloy subsurface and possess microstructures similar to those of the near-surface. It is believed that these cracks are linked with the formation of the tribolayer, and that continued research into them would offer more enlightenment about its formation.

3.5 CONCLUSIONS

The effect of hot rolling on the evolution of surface defects in an Al-Mn alloy was examined using a rolling regime of four passes at a temperature range of 550 – 520 °C. Micro-cracks that formed on the surface of the aluminum alloy were examined, and the following trends were observed:

1. Cracks were observed to occur at grain boundaries, and were initiated during the first rolling pass by the sliding and overlapping of grain boundaries.
2. Crack propagation to the subsurface occurred along the grain boundaries. The immediate regions around cracks at the subsurface were rich in magnesium, which increased after each rolling pass.
3. Cracks oriented transverse to the rolling direction were observed to continually propagate to the subsurface after each pass, while cracks with other orientations were observed to begin healing at the second pass. Therefore, the continued
propagation or healing of cracks during subsequent rolling passes was influenced by the orientation of the crack.

4. Crack healing was observed to occur by the sealing of cracks with MgO, which prevented the faces of cracks from welding together.

5. The microstructure of transverse cracks was similar to that of the near-surface, consisting of an amorphous Al$_2$O$_3$ layer at the crack face, with parallel nanocrystalline Al$_2$MgO$_4$ spinel and MgO layers lying closer to the bulk.

**BIBLIOGRAPHY**


4.1 INTRODUCTION

Hot rolling is known to alter the surface composition and properties of aluminum through the migration of alloying elements, adsorption of components from lubricants and high shear deformation [1, 2]. A surface structure, rich in oxides and covered with shingles and cracks transverse to the rolling direction, and a subsurface microstructure, with ultrafine grains and oxide rich grain boundaries, is induced by the tribological interaction of the mating surfaces [3-7]. The composition of the oxide layer of the near-surface microstructure or tribolayer varies, depending on temperature, and on processing conditions such as the number of rolling passes [1, 2]. Oxides such as \( \text{Al}_2\text{O}_3 \), \( \text{Al}_2\text{MgO}_4 \) spinel and \( \text{MgO} \) have been observed; \( \text{Al}_2\text{O}_3 \) and \( \text{MgO} \) are the dominant oxides formed during rolling of aluminum-magnesium alloys [1, 2, 4, 6]. In aluminum alloys that contain magnesium, \( \text{MgO} \) is known to form on the surface simply through heating of the alloy. The thickness of this oxide, which forms over the already existing \( \text{Al}_2\text{O}_3 \), depends on the temperature and heating period [3, 4, 8]. \( \text{MgO} \) at the surface of aluminum alloys can affect the surface quality of the product as it accumulates in piles, giving the alloy surface an “oxide related roughness” [1, 8]. An \( \text{MgO} \) layer, generated during heating of an aluminum 5052 alloy, was observed to be thick and porous in comparison to that generated on a 3003 alloy [1]. Magnesium has been noted to have a higher oxidation rate than aluminum, which is attributed to its higher diffusion rate in aluminum and its affinity for oxygen [9, 10]. Diffusion of aluminum and magnesium is hindered by the
oxide layer (Al₂O₃ and MgO respectively) formed on Al-Mg surfaces [9, 11]. The high MgO concentration at the surface of aluminum alloys is also attributed to the reduction of amorphous γ-Al₂O₃ by magnesium at temperatures exceeding 300 °C (secondary oxide formation) or to the direct interaction of magnesium with oxygen, occurring at the oxide/metal interface (primary oxide formation) [9, 12]. Internal oxidation (oxidation occurring within a bulk alloy) of Al-Mg alloys has been reported to begin at temperatures exceeding 350 °C, depending on the magnesium content. At 500 °C, Bahadur has reported that internal oxidation is only observed for aluminum alloys with magnesium content higher than 4.5 % [13].

The thickness of the MgO layer on the aluminum alloy has also been noted to depend on the magnesium concentration in the alloy [14, 15]. Thicker MgO layers were observed with higher magnesium content. This rich MgO layer has been said to have the advantage of reducing the adhesion of aluminum to steel that occurs at high temperatures [14, 15].

Most of the research into the surface oxide layers occurring on aluminum alloys has concentrated on the oxidation of aluminum alloys under controlled conditions, with focus directed mainly to temperature and heating cycle effects on these layers. Study of surface oxides developed during hot rolling have only looked at the oxide layers developed after industrial rolling that involves numerous rolling passes. This paper presents a detailed study of the oxide layers that form on Al-Mg alloys after the first and fourth hot rolling passes. The focus here is on comparing the effect of high temperature deformation induced during hot rolling on the surface oxide structure between the first and fourth rolling passes.
4.2 EXPERIMENTAL PROCEDURE

A rolling simulator with a roll-on-block configuration, designed at the University of Windsor, and previously described in [4], was used in this study. The setup simulates the tribological surface deformation of a work piece during rolling, allowing for the variation of several rolling parameters such as temperature, rolling load, work roll surface conditions and lubrication. The rotation of the roll as it runs across the surface of the aluminum block allows for the simulation of rolling and sliding.

The roll was machined from AISI 52100 steel to a diameter of 21 mm, while the Al-Mg test samples were cut from industrial transfer slabs to dimensions of 10 mm thickness, 30 mm height and a width of 95 mm. The composition of the Al-Mg alloy consisted of 4.5 % Mg, 0.2 % Mn, 0.2 % Si, 0.4 % Fe, 0.15 % Cu, 0.25 % Zn, and the balance Al (in wt%). The lubricant used was an oil-in-water emulsion at a 4 % (v/v) concentration. The surface of the roll and the aluminum test specimens were polished with a 1 µm diamond paste and then cleaned ultrasonically in acetone for 5 minutes to remove surface contaminants. The roll was polished to eliminate the effect of roll roughness and to more effectively study the effect of deformation on the alloy surface. It was cleaned with a 15 % (wt/wt) sodium hydroxide solution to remove any aluminum transfer and then polished to maintain the surface roughness after each test. The rolling simulations were carried out for 1 and 4 passes, at a typical initial industrial rolling temperature of 550 °C, with a 25 °C drop after every pass, and at a 7 % forward slip with the direction of each pass varying, simulating reverse rolling.

The specimen surfaces were examined with an FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. Surface
roughness measurements were then made using an optical surface profilometer (WYKO NT1100) in the vertical scanning interferometry (VSI) mode. The average surface roughness ($R_a$) was determined from several areas, and the mean values of these measurements were recorded for each test. The near-surface microstructure of the samples were examined using a ZEISS NVision 40 Cross Beam workstation focused ion beam (FIB), with a gallium ion beam operated at low beam currents, at an operating voltage of 30 kV. The surface was protected by depositing a thin layer of carbon. Cross-sectional trenches were ion milled using the FIB H-bar method. The samples prepared by using the lift-out method were examined using an FEI Titan 80-300 LB transmission electron microscope (TEM).

4.3 EXPERIMENTAL RESULTS

4.3.1 Surface Morphology Developed During Deformation

The surface topography of the alloy after 1 rolling pass, presented in Figure 4.1(a), hints at the fine grain structure observed on the surface of the alloy. The topography of the alloy also showed evidence of surface offsets covering the sample, which were induced during the first rolling pass. These offsets, caused by grain boundary sliding (GBS), gave the sample an increased surface roughness ($R_a$) of 0.21 µm, from the 0.03 µm recorded for the initial polished surface. The surface roughness ($R_a$) of the alloy decreased after the second pass and continued to do so with each subsequent pass. After 4 passes, it was recorded as 0.12 µm, exhibiting a surface profile with a fine grain structure but fewer surfaces offsets than displayed on the surface after 1 pass, as shown in Figure 4.1(b).
Low magnification SEM images of the alloy surface after the first pass (Fig 4.2(a)) better displayed surface offsets, in the form of steps between grains, observed with surface profilometry. An increase in magnification (Fig 4.2 (b)) exposed surface damage in the form of cracks on the sample surface. These cracks were observed to occur in two forms: micro-cracks, detected as occurring along and across grain boundaries, and nano-cracks, occurring in the surface oxide within grains. At this magnification, black or dark spots were also observed, dispersed across the alloy surface. These spots seemed to occur within the vicinity of nano-cracks spread across the sample surface. Energy dispersive spectrometry (EDS) analysis showed that these spots were rich in magnesium and oxygen, hence have been referred to as MgO-enriched dark spots. At this stage, the oxide type has been inferred from oxides recorded in literature as occurring in Al-Mg alloys [1-6]. The surface texture of the sample after 1 rolling pass, displayed at higher magnification as seen in Figure 4.2 (c), appeared to possess a further roughed appearance within the grains, due to a network of wave-like (or wrinkled) material pile-up formed across the alloy surface. Overlapping at grain boundaries was also observed, occurring at surface offsets due to GBS and inducing micro-cracks in the sample surface.

After the fourth pass, the grain structure on the sample surface (Fig 4.3 (a)), possessed a flattened morphology, with grain boundaries appearing darkened in comparison to those on the surface of the 1 pass sample. At this stage of rolling, intermetallic particles, composed of aluminum and iron (possibly Al₃Fe) [16], were also observed to be surrounded by a dark expanse. Micro-cracks running across grains were observed on the sample surface. Some of these cracks appeared to occur at deformation steps or surface offsets within grains, running across as well as along grain boundaries.
At higher magnification, micro-cracks occurring at grain boundaries, apparently initiated during the first rolling pass, appeared closed and darkened (Fig 4.3 (b)). Some of these darkened grain boundaries still showed evidence of cracks while others simply appeared to be darkened. At this pass, dark spots were also observed covering the sample surface. These spots appeared randomly dispersed, not just within the vicinity of nano-cracks, and had a higher population density than previously observed after the first rolling pass. In Figure 4.3 (c), some of these dark spots appear in the form of a wave-like network, similar to the material pile-up previously observed within grains after 1 rolling pass, across the sample surface. These dark wavy networks occurred mainly between and around nano-cracks. EDS analysis of all regions described as darkened indicated that they were all rich in magnesium and oxygen and as such are described as MgO-enriched.

![Figure 4.1. Surface profilometry images displaying the surface topographies of the Al-Mg alloy after (a) 1 and (b) 4 rolling passes.](image-url)
Figure 4.2. Scanning electron images of the surface of Al-Mg alloy after 1 rolling pass showing (a) grain structure, (b) micro-cracks and dark spots and (c) wavy surface appearance and overlap at grain boundary. R.D refers to the rolling direction.
Figure 4.3. Scanning electron images showing of surface of Al-Mg alloy after 4 rolling passes showing (a) grain structure and cracks across grains (b) MgO rich grain boundaries and closed cracks and (c) dark wavy network
4.3.2 Surface Morphology of Micro-cracks

Of particular interest in this study are the micro-cracks that occur on the alloy surface during hot rolling, thus TEM samples were prepared of cracks occurring transverse to the rolling direction from samples rolled for 1 and 4 passes. As part of the TEM sample preparation, the areas of interest were coated with carbon before being ion milled. Subsurface analysis of micro-cracks occurring on the sample with 1 rolling pass showed crack propagation into the subsurface of the alloy to depths ranging from 0.63 µm to 2.53 µm. Cracks were observed to typically propagate along the grain boundaries (Fig 4.4 (a)), but when they did not, they were observed at steps in the alloy surface (or surface offsets) that occurred due to deformation during rolling (deformation step) (Fig 4.4 (b)). Crack width, measured at the widest portion of the crack (occurring at the crack opening or at the subsurface) ranged from 0.25 µm to 1.02 µm. Figure 4.4(a) shows the thin surface oxide layer coating the alloy also covers the faces of the cracks. EDS analysis of this layer showed it to be rich in magnesium and oxygen. Narrow portions of the cracks were also observed to be filled with magnesium and oxygen, similar in appearance to the surface layer, as highlighted in Figure 4.4 (b).

Crack propagation to the subsurface was observed on analysis of the sample deformed with 4 rolling passes. Under this condition, micro-cracks were filled with a visibly porous material at the subsurface (Fig 4.5(a)), identified by EDS as being rich in magnesium and oxygen. Crack depths were observed to extend from 0.62 µm to 1.45 µm, while crack widths at the widest part of the crack subsurface were between 0.54 µm and 0.87 µm. A thin porous surface layer, composed of magnesium and oxygen, covered the alloy as displayed in Figure 4.5 (a), and had similar visible features to the porous MgO
that filled the cracks. Subsurface examination of the darkened grain boundaries, observed in Figure 4.5 (b) (an etched image attached to this figure clearly displays the grain boundaries), showed them to be filled with porous MgO similar to the micro-cracks. A cross section of the intermetallic particles (Fig 4.5 (c)) revealed that they were engulfed by this porous MgO to a depth of 1.4 µm below the surface. It also permeated into cracks in these particles, and filled them. After the fourth rolling pass, then, grain boundaries, micro-cracks and intermetallic particles were filled or surrounded by MgO.

Figure 4.4.Cross section images of the Al-Mg alloy subsurface after 1 rolling pass, showing crack propagation (a) along grain boundaries and (b) across grain boundaries.
Figure 4.5. Cross section images of the Al-Mg alloy subsurface after 4 rolling passes showing (a) crack propagating along grain boundary (b) MgO filled darken grain boundary and (c) MgO covered intermetallic particles.
4.3.3 TEM Analysis of Micro-cracks

TEM analysis of an observed crack in the sample deformed by 1 rolling pass (Fig 4.6 (a)) shows that this particular crack does not appear to propagate along the grain boundary but instead occurs within the grain. A thin layer covering the crack face is also exposed, displaying a different structure from the bulk alloy. Matching this image with the EDS map in Figure 4.6 (b), confirms that these areas are rich in magnesium and oxygen. The map also depicts the overlap, believed (from SEM imaging) to occur at the grain boundaries, to be primarily hollow and consisting of only a thin film of MgO. This overlap, from the STEM image in Figure 4.6(a), was now found to occur at the edges of the crack on the surface of the alloy rather than at the grain boundary. The deeper portion of the crack is also shown to be filled (or sealed) with magnesium and oxygen of the same structure as the thin layer covering the crack face.

The higher magnification scanning transmission electron image in Figure 4.7 (a) shows that the MgO layer lining the crack face comprises of nanocrystalline grains, which appear to have a hexagonal platelet structure that seals the deeper end of the crack and solely makes up the overlap. This nanocrystalline structure extends the full depth of the crack (133.89 nm). A HRTEM image of the MgO layer on the crack face (Figure 4.7 (b)) reveals the nanocrystalline grains as embedded in an amorphous region, although as observed from the EDS map, the entire layer comprises mainly of magnesium and oxygen. Selected area electron diffraction (SAED) analysis (Fig 4.7 (c)) of the area shown in Figure 4.7 (b) was used to identify the (200) FCC plane belonging to MgO. EDS line scans were also used to confirm the composition of these nanocrystalline layers
lying on the crack face and showed that the grain boundaries did not experience any magnesium enrichment after 1 rolling pass.

Figure 4.8 (b) displays a STEM image of a cross section (taken from the highlighted area in Figure 4.8 (a)) of a sample deformed by 4 rolling passes. The STEM image shows three labeled damaged areas, two of which are identified from the SEM image. The first damaged area (labeled as 1) represents the subsurface morphology of a micro-crack that from the surface appeared to run across the grain boundary, while the second damaged area (labeled as 2) represents a surface crack that appeared to run along the darkened grain boundary (both highlighted on the SEM image). The third damaged area (labeled as 3), which is not highlighted on the SEM, represents the subsurface of a magnesium-rich spot observed to be covering the surface of the sample. The three damaged areas possessed a similar morphology which also closely resembled the surface layer covering the bulk material. The cross-sections show that the two cracks (i.e. labeled 1 and 2) propagate along grain boundaries at the subsurface. The higher magnification image in Figure 4.8 (c), representing crack 1, clearly shows the porosity within the crack. The porous material sealing the crack, observed in the cross-sectional image, appears to be engulfed by a thin film, forming a network within the crack. An EDS map of this crack (Fig 4.9(a)) shows the porous material sealing the crack to be rich in magnesium and oxygen. Little to no aluminum was observed within the crack, leading to the conclusion that the porous material may be MgO. Carbon was also observed within the crack, an elemental mix from the crack showed the magnesium within the crack to be coated with carbon (Fig 4.9(b)).
An overview TEM image of the cross section shown in Figure 4.8(a) is given in Figure 4.10 (with damaged areas labeled). TEM images of crack 1 display the nanocrystallites (some of which are depicted by the dark spots) embedded in the amorphous carbon (Fig 4.11 (a)). Figure 4.11 (b) shows the area where the bulk aluminum matrix meets the crack, revealing the porous nature of the network of MgO nanocrystals stacked on top of each other and embedded in the amorphous carbon. The HRTEM analysis in Figure 4.12 (a) displays the MgO nanocrystals embedded in amorphous material and the SAED analysis in Figure 4.12 (b) confirms the random orientation of the nanocrystals stacked on top of each other. The diffraction data suggests that the area is polycrystalline, with random orientation of MgO planes (111), (200), (220), (222) and (400). The area covered by amorphous carbon continued to increase closer to the sample surface.

As observed in the other damaged areas (Fig 4.13 (a)), TEM analysis of the third damaged area (labeled 3 in Fig 4.10) revealed similar nanocrystals stacked on each other and embedded in an amorphous material. This damaged area did not occur at a grain boundary but instead occurred along a dark line that can be observed propagating from its edge. This area was also composed of MgO. A higher magnification image of this area (Fig 4.13 (b)) shows the high porosity within this damaged region.
Figure 4.6. (a) Scanning transmission electron micrograph showing the structure of a transverse crack at the subsurface after 1 rolling pass and (b) EDS maps displaying elemental composition of the transverse crack.
Figure 4.7. (a) Transmission electron micrographs showing the structure of a crack at the subsurface on the alloy after 1 rolling pass (b) high resolution transmission electron micrograph from the boxed region in (a) of the MgO platelets and (c) selected area electron diffraction pattern from the area shown in (b).
Figure 4.8. (a) Scanning electron micrograph of the surface of the sample deformed with 4 rolling passes depicting where Focus ion milling cross section was made (b) scanning transmission electron micrographs of subsurface showing the three damaged areas labeled, of region within the box from the SEM image and (c) crack 1 (region within the inserted box in (b)) at higher magnification.
Figure 4.9 Energy dispersive spectrometry analyses taken from crack 1 on the sample with 4 rolling passes showing (a) elemental map of individual elements contained in the crack 1, and (b) EDS color mix, from the region within the box insert, with aluminum represented by green, magnesium by red and carbon by blue.
Figure 4.10. Transmission electron micrograph of cross section of the subsurface after 4 rolling passes (previously displayed in Figure 4.8)
Figure 4.11. (a) Transmission electron micrographs from crack 1 showing its structure and (b) matrix and crack face from the same crack at the subsurface region on the alloy after 4 rolling passes.

Figure 4.12. (a) High resolution transmission electron micrograph and (b) selected area electron diffraction pattern of area shown in (a) revealing the MgO nanocrystalline structure within crack 1 after 4 rolling passes.
4.3.4 TEM Analysis of Near-Surface Oxides

Surface examination performed along with EDS line scans confirmed that the near-surface regions surrounding cracks after 1 rolling pass (Fig 4.14 (a)) and 4 rolling passes (Fig 4.14 (b)) were covered with a top layer of MgO and (possibly) lower layers of Al₂O₃ and Al₂MgO₄. After the fourth pass, some carbon was observed within the surface layer, which may come from the lubricant. This carbon content was not distinctly observed after the first pass. The thickness of the near-surface layer was about 78 nm after the first pass and 89 nm after the fourth rolling pass. Under both rolling conditions, the top layer of MgO was about 50 nm thick. A comparison of the HRTEM images of the surface layers after 1 pass (Fig 4.15 (a)) and 4 passes (Fig 4.15 (b)) shows both surfaces to be nanocrystalline in nature. The near-surface layer after the fourth rolling pass shows dark spots, which were identified as nanocrystals (Fig 4.15 (b)). High porosity was also
observed in the near-surface layer of the sample rolled with 4 passes with a structure similar to the cracks and damaged regions examined earlier, while less porosity was observed in the near-surface layer after the first pass.

![Figure 4.14. Scanning transmission electron cross-sectional micrographs with elemental linescan of near surface in crack region showing the surface oxide structure of Al-Mg alloy after (a) 1 rolling pass (green represents aluminum, red indicates magnesium and blue indicates oxygen) and (b) 4 rolling passes (green represents carbon, red indicates magnesium, cyan indicates aluminum and purple indicates oxygen).](image)
4.4 DISCUSSION

The high temperature deformation applied during hot rolling caused the increase in surface roughness of the alloy by grain boundary sliding observed after the first pass. The surface roughness was reduced during subsequent passes by the rolling forces, which compressed grains and flattened the surface. Micro-cracks on the surface of the Al-Mg alloy were observed to form at both rolling stages, in the transverse direction to rolling. Cracks observed after the first rolling pass appeared to be caused by grain boundary sliding during rolling. This mechanism caused cracks by (i) the separation at the grain boundary (Fig 4.4(a)) and (ii) induced step morphology within the grain, with cracks forming in proximity to the steps (Fig 4.4 (b)).

Cracks at deformation steps, like those along the grain boundaries, propagated to the subsurface region although not always along the grain boundaries. The sliding of
grains and the formation of deformation steps induced overlapping at grain boundaries and within grains, accompanied by crack formation. Nano-cracks could also be observed covering the alloy surface. All micro-cracks observed on the surface at this stage propagated to the subsurface having occurred due to steps formed during rolling. The flattening of the surface due to subsequent rolling passes eliminated the grain steps and overlapping at grain boundaries observed after the first pass.

After 4 rolling passes, micro-cracks were filled with MgO and appear darkened due to contrast under the SEM. The dark wavy patches observed on the surface at this stage of rolling apparently caused by the nano-cracks which had been filled with MgO. Magnesium diffusion to free surfaces formed by both nano- and micro-cracks allows for the formation of MgO, which fills the cracks. This process is extensified in aluminum alloys with high magnesium concentration [13, 14]. As such, wide cracks have their faces layered with MgO while narrow cracks are sealed with MgO after the first rolling pass. There is also magnesium diffusion to the areas around the intermetallic particles, which accumulates and causes these areas to appear darkened in subsequent rolling passes. The darkened appearance of these MgO-enriched areas is possibly due to the nanocrystalline size of the MgO grains. After the fourth rolling pass, micro-cracks also propagated to the subsurface but appear to be shallower than cracks observed after the first pass. The MgO within the cracks prevented cracks from closing during subsequent rolling passes. The formation of MgO within cracks though, enabled the healing of the cracks by filling the gaps between crack faces, thereby sealing the cracks and possibly preventing the propagation of these cracks further into the subsurface region. The filling of cracks by the formation of oxide products which prevents further crack propagation resembles an
identified mechanism of self-healing materials, referred to as oxidative healing described in literature [17 - 19].

It is clear that with the increase in rolling passes at high temperature, the diffusion of magnesium to the cracks free surfaces begins to fill the spaces formed between the crack faces and the nanocrystalline structure of the MgO inside the cracks gives them their darkened appearance. This began to occur at the second rolling pass and was assumed to be influenced by the rolling stresses. To confirm this, samples were rolled for 1 pass and held at the second pass temperature of 525 °C for the period of time it would take for a rolling pass to occur. The surfaces of these samples (Fig 4.16 (a)) were compared with samples rolled for 2 rolling passes (Fig 4.16 (b)). While the sample with 2 rolling passes displayed darkened grain boundaries, the sample with 1 pass and the heating cycle did not. This correlated to a change in MgO nanostructure after each pass due to rolling stresses. The formation of MgO within cracks aids in crack healing by the action of sealing, although it prevents the welding together of crack faces.

The difference in the MgO grain structure within cracks at each rolling pass is quite distinct. After the first pass, MgO nanocrystalline grains appear in the form of hexagonal platelet structures across the crack surface; this effect has previously been reported for a similar aluminum alloy with high magnesium concentration [2]. These platelets appear ordered and arranged along the surfaces of the cracks and appear to lie on the (200) plane. The MgO structure observed within the crack after 4 rolling passes appeared more porous than that observed after the first rolling pass. The hexagonal platelets observed after the first rolling pass were no longer observed after the fourth pass, although the MgO had a nanocrystalline structure engulfed in an amorphous matrix,
which had a high carbon concentration. The amorphous carbon film observed covering the porous MgO was probably due to lubricant residues permeating into the crack and coating the MgO nanocrystals. These were stacked on top of each other and occurred in more orientations ((111), (220), (400)) than were observed after the first rolling pass.

The structure and morphology of the surface oxide layer induced during hot rolling closely resemble the MgO rich cracks and damaged areas observed after the fourth rolling pass. This suggests that, as rolling continues; these cracks would become part of the near-surface microstructure. It also suggests that the cracks and near-surface region experience similar rolling strains and stresses during each rolling pass. These rolling shear strains influence the structure of the MgO, as is evident from experiments showing no darkening of grain boundaries from heating alone. Subsequent rolling passes are required for this effect to be observed. It can be concluded that the rolling strains influence the evolution of the MgO structure during rolling.
4.5 CONCLUSIONS

The effect of hot rolling passes on the development and propagation of surface cracks and the oxide formed within them was examined using a rolling tribo-simulator. Transverse cracks were examined after the 1st (550 °C) and 4th (475 °C) rolling pass and the following observations were made:

1. Cracks observed after the first rolling pass did not always propagate along grain boundaries, either at the surface or subsurface. However, cracks observed after the fourth rolling pass appeared to propagate along the grain boundaries.

2. From the first rolling pass, magnesium diffusion to crack faces was observed to cause the formation of MgO on crack faces, filling the spaces between narrow cracks (i.e. crack healing).

3. After the first rolling pass, MgO nanocrystals on crack faces possessed a hexagonal structure which was not evident after the fourth rolling pass.
4. MgO observed within cracks possessed a porous structure, which appears to be influenced by rolling stresses.

5. The similarities between the nanocrystalline structures observed within cracks and at the near-surface region imply that they undergo similar conditions of rolling stress and strain.

BIBLIOGRAPHY


CHAPTER 5
THE CHARACTERIZATION OF NEAR-SURFACE DEFECTS EVOLVED ON ALUMINUM-MANGANESE ALLOYS DURING HOT ROLLING

5.1 INTRODUCTION

The effect of high strains and shear deformation caused by sliding and grinding experienced during rolling has been identified to alter the near-surface microstructure of aluminum alloys with the formation of a deformed near-surface layer [1], [2]. This layer, also referred to as a tribolayer, disturbed layer, white layer and rolled-in subsurface layer, influences the tribological and corrosion properties of aluminum alloys [1]–[8]. Its subsurface features consists of ultrafine aluminum grains with oxide-decorated grain boundaries, micro-cracks, voids, aluminum and magnesium oxide particles [7]–[9], [6], [5]. Surface topography of these layers often features shingles and transverse cracks [2], [9], [5].

The conditions and mechanisms that induce the formation of the near-surface deformed layers have been a subject of interest. Kai et al. [9] have reported the observation of two types of near-surface deformed layers; type A and B, the major difference between which was the oxide decorated grain boundaries. Liu et al. [5] related the shingled topography and cracks on the aluminum alloy surface to the interaction between the workpiece and the work roll surfaces. They noted that high aspect rolling ratio combined with increased rolling speeds and rough and worn roll surfaces resulted in increased shingling and transverse crack occurrence as well as the formation of a thicker near-surface deformed layer [5]. Using automated crystallographic orientation mapping
in a transmission electron microscope (ACOM-TEM) Tzedaki et al. [8] proposed that holes formed in the roll bite could develop into subsurface cracks on subsequent passes. They noted that shingles and debris on the surface would enhance the introduction of holes and cracks into the subsurface. They also observed a grain gradient at the subsurface, with elongated grains closer to the bulk and small grains in the intermediate area, along with high amounts of oxygen and magnesium distribution around cracks [8]. Liu et al. [6] used electron energy loss spectroscopy (EELS) maps to show the presence of magnesium oxide bands and oxide particles at the grain boundaries of the near-surface layers. Watt et al. [10] reported that recrystallization coupled with magnesium content enhanced microstructure refinement during hot rolling. Riahi et al. [2] have proposed that shingle formation is a result of the local deformation of micro-wedges and that shingle formation is influenced by the forward slip. They also observed an increase in surface damage and surface concentration and depth of magnesium with the number of passes.

The present study aims to understand the mechanisms involved in the evolution of the near-surface deformed layer on an aluminum-manganese alloy surface during hot rolling. A detailed comparison of the near-surface microstructural features observed after the first and the tenth rolling passes are presented to determine their evolution during the rolling schedule. Emphasis is placed on the effect of hot rolling deformation strains and stresses induced by the work roll on the aluminum alloy to determine the mechanisms involved in near-surface layer evolution.
5.2 EXPERIMENTAL PROCEDURE

Specimens of dimensions 10 mm width, 30 mm thickness and 95 mm length were machined from an Al-Mn alloy containing about 0.8 - 1.3 wt. % Mn and 0.8 - 1.4 wt. % Mg. The 10 × 95 mm face was mechanically polished using a 1 micron diamond paste. The samples were then rolled using a tribo-simulator with a roll-on-block configuration, designed to emulate the rolling process, the working principle of which has been previously described by Riahi et al. [2].

Rolling was performed at an initial temperature of 550°C for the first rolling pass with a 10°C temperature reduction for each subsequent pass. A total of ten forward-reverse rolling passes at a forward slip of 7% were carried out on the specimens to simulate industrial reversing mill rolling. The samples were rolled using an AISI 52100 steel work roll with a diameter of 21 mm that was mechanically polished using a 1 micron diamond paste, to eliminate the effect of roll roughness, and thus, ascertain the effect of deformation forces on the work piece surface. An oil-in-water emulsion with a 4% (v/v) concentration was used as the lubricant.

The work roll and aluminum alloy surfaces were cleaned ultrasonically in acetone for 5 minutes before rolling to remove surface contaminants. The work roll was also cleaned with a 15% (wt/wt) sodium hydroxide solution after each pass to remove any aluminum transfer and then polished to maintain the surface roughness.

The specimen surfaces were examined with an FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. Surface topography was evaluated using an optical surface profilometer (WYKO NT1100) in the
vertical scanning interferometry, VSI, mode. The average surface roughness (R_a) was determined from several areas and the mean values of these measurements were compared for each pass. The near-surface microstructure of the alloy was examined using a ZEISS NVision 40 Cross Beam Focus Ion Beam (FIB) using a gallium ion beam operated at low currents with an operating voltage of 30 kV. The surface was protected by depositing a thin layer of carbon on the area of interest. Cross-sectional trenches were ion milled using the FIB H-bar method. The sample prepared by the lift-out method was examined using an FEI Titan 80-300LB Transmission Electron Microscope (TEM).

5.3 EXPERIMENTAL RESULTS

5.3.1 Surface Analysis of the Hot Rolled Al-Mn Alloy

Previous work has examined the early stage evolution of the Al-Mn surfaces from the first to the fourth hot rolling pass [11]. It was observed that after the first pass the surface roughness increased from an R_a of 0.02 µm (after polishing) to 0.37 µm and then it decreased to 0.10 µm after the fourth pass. After 10 hot rolling passes, the surface roughness of the Al-Mn alloy was 0.13 µm. The surface profile of the Al-Mn alloy after the tenth pass (Fig. 5.1(b)) had a smooth morphology covered in micro-bumps in comparison to the morphology after the first rolling pass (Fig. 5.1(a)).

SEM analysis of the alloy surfaces after the first and the tenth hot rolling passes displayed the evolution of the Al-Mn surface during the rolling schedule. After the first rolling pass (Fig. 5.2(a)), the surface revealed the misalignment of grains (in the form of surface offsets) due to grain boundary sliding (GBS). Micro-cracks were observed at
grain boundaries and fractured intermetallic particles were observed on the alloy’s surface. After 10 passes, the surface was covered with dark patches or islands that corresponded to the micro-bumps from surface topography analysis (Fig. 5.1(b)) and mostly occurred enclosing the intermetallic particles (Fig. 5.2(b)). Energy dispersive spectroscopy (EDS) analysis confirmed that these islands were rich in magnesium and oxygen, and thus, were most likely composed of MgO. The micro-cracks on the surface after the 10 pass rolling schedule appeared to dominantly occur in the transverse direction.

Higher magnification analysis of transverse micro-cracks displayed that cracks on the surface after 1 rolling pass (Fig. 5.3(a)) were wider than those observed after 10 rolling passes (Fig. 5.3(b)), indicating that the initial cracks had closed up during the rolling schedule. The SEM analysis also revealed damage to micro-crack faces, in the form of in the form of surface deformation induced crevices. Nano-cracks were also observed formed on the 10 pass hot rolled surface (Fig. 5.3(b)). While intermetallic particles were observed, fractured on the surface after the first rolling pass (Fig. 5.3(a)), by the tenth rolling pass, fractured intermetallic particles were observed embedded into the surface with material pile-up surrounding them (Fig. 5.4).

In summary, grain misalignment, because of GBS, and micro-cracks at grain boundaries, which were observed after the first pass, were no longer evident after 10 hot rolling passes. Only transverse micro-cracks could be observed on the alloy surface. Figure 5.5(a) shows a transverse crack on the Al-Mn surface after 10 passes, and Figure 5.5(b) displays an EDS map of this surface. The map revealed transverse micro-cracks, nano-cracks and grain boundaries were all enriched with magnesium. Although the grain
boundaries were no longer evident on the alloy surface, they were highlighted in the EDS map due to the magnesium enrichment. As proposed in previous work [11], micro-cracks that formed at grain boundaries not occurring in a transverse direction during the first rolling pass are preferentially healed during subsequent rolling passes. The EDS map also exposed pockets of carbon, most likely from the lubricant. The iron-rich regions identified in the map were likely fractured intermetallic particles namely Al$_6$(Fe, Mn) and $\alpha$-Al$_{12}$(Fe, Mn) according to the literature [2], [12].

![Surface profile images displaying the surface topography of the Al-Mn alloy after (a) 1 pass and (b) 10 passes.](image)

(a) (b)
Figure 5.2. Scanning electron images of the Al-Mn surface after deformation at (a) 1 pass and (b) 10 passes.

Figure 5.3. Scanning electron images of the Al-Mn surface showing evolution of surface after hot rolling for (a) 1 pass and (b) 10 passes.
Figure 5.4. Scanning electron image of the Al-Mn surface after 10 passes showing embedded intermetallic particles surrounded by oxide pile-up.

Figure 5.5. (a) Scanning electron image of the Al-Mn surface after 10 passes displaying a transverse micro-crack and (b) EDS map taken from the area in (a) displaying magnesium-rich grain boundaries and cracks.
5.3.2 Near-Surface Microstructure of the Hot Rolled Al-Mn Alloy

Examination of the near-surface microstructure of the Al-Mn alloy after 1 and 10 hot rolling passes was carried out at transverse cracks displayed in Fig. 5.6. The SEM image of the transverse crack after 1 pass (Fig. 5.6(a)) revealed that the micro-crack occurred between two overlapping grains which had formed a step. The surface after 10 passes (Fig. 5.6(b)) was much smoother with a crack that appeared to be closed up during the hot rolling schedule. The cross-sectional image of the near-surface region after 1 pass (Fig. 5.7(a)) revealed that micro-cracks propagated 0.9 - 1.4 µm deep into the subsurface region. Crack widths (the opening of the crack at the surface), had a range of 0.49 - 0.87 µm. EDS analysis exposed that the regions surrounding the cracks were rich in magnesium and oxygen.

The cross-sectional image of the near-surface region after 10 passes (Fig. 5.7(b)) displayed extensive near-surface damage in the form of nano-cracks extending 0.5 µm into the subsurface region. Crack branches were also observed propagating from the micro-cracks, which themselves reached depths of 1.0 - 2.5 µm. It should be noted that while only one micro-crack was visible on the alloy surface (Fig. 5.6(b)), two distinct micro-cracks were observed at the subsurface (Fig. 5.7(b)). The crack width of the shallower crack was 0.2 µm while that of the deeper crack was 0.6 µm. High magnification analysis of the near-surface damage after 10 passes (Fig. 5.8) showed that the shallower cracks were filled with a porous material, rich in magnesium and oxygen, while the transverse crack that propagated to a depth of 2.5 µm possessed this porous material only on the crack faces and branches. Previous work has shown that micro-cracks that did not occur transverse to the rolling direction were preferentially self-healed.
by MgO filling the crack gaps (oxidative healing) [11]. Examination of other transverse micro-cracks propagating into the subsurface region displayed crevices formed on the crack faces in addition to crack branching (Fig. 5.9(a)). Cross-sectional analysis of an embedded intermetallic particle (Fig. 5.9(b)) revealed the presence of porous magnesium-rich oxides enclosing the particle, which filled the free spaces at the sides of the particle.

Therefore, the near-surface region could be characterized as possessing micro-cracks that propagated into the subsurface and appeared to be dominantly initiated by GBS after the first hot rolling pass. By the tenth pass, the near-surface microstructure had evolved but was also characterized by micro-cracks propagating into the subsurface. Micro-cracks not transverse to the rolling direction were filled with porous magnesium-rich oxides, while transverse cracks possessed magnesium-rich faces. Damage appeared more extensive by the tenth pass and much deeper with embedded intermetallic particles being enclosed by porous magnesium-rich oxides.

![Figure 5.6. Scanning electron image of transverse micro-cracks after hot rolling at (a) 1 pass and (b) 10 passes](image-url)
Figure 5.7. FIB cross-sectional images of micro-crack propagation after (a) 1 pass and (b) 10 passes.

Figure 5.8. FIB Cross-sectional image of near-surface damage after 10 passes displaying transverse micro-crack propagation and MgO filled micro-crack.
Figure 5.9. FIB cross-sectional images of the near-surface region after 10 passes showing (a) transverse micro-crack with crevices on crack faces and (b) embedded intermetallic particle surrounded by porous MgO.

5.3.3 TEM Characterization of Near-Surface Features

TEM analysis was performed on the FIB-milled cross-sections of the transverse micro-cracks displayed in Fig. 5.7(a) (after 1 rolling pass) and Fig. 5.9(a) (after 10 rolling passes). The TEM analysis (Fig. 5.10) revealed that the cracks propagated along the grain boundary. The crack observed after 1 rolling pass (Fig. 5.10(a)) possessed a crack branch protruding from its face, while the crack observed after 10 rolling passes (Fig. 5.10 (b)) had several crack branches protruding from both faces of the crack. The crack protruding along the grain boundary of the sample deformed by the 10 pass rolling schedule was measured to run along 4 µm in length and to extend to a subsurface depth of 2 µm.

Higher magnification images shown in Fig. 5.11 depict the cracks in greater detail. These images revealed that the faces of both cracks were covered with a nanocrystalline oxide layer, which appeared less porous with less distinct grains after 1
pass (Fig. 5.11(a)) compared to that after 10 passes (Fig. 5.11(b)). It was also slightly thinner after 1 pass (82.9 ± 41nm) in comparison to that observed after 10 passes (121.6 ± 37 nm). The nanocrystalline oxide filled the crack branches, the narrow crack tail and also coated the surfaces of the alloy. EDS maps taken from regions surrounding the crack after 1 pass (Fig. 5.12(a)) and after 10 passes (Fig. 5.12(b)) were used to confirm that the nanocrystalline layer coating the cracks was rich in magnesium and oxygen. There were also slight traces of aluminum at the edges of the magnesium-rich oxide layer coating the crack faces after 10 passes (Fig. 5.12(b)). EDS line scans of the grain boundaries below the cracks confirmed that there was no magnesium enrichment at the grain boundaries after either 1 pass or 10 passes. Further examination at higher magnification of the regions surrounding the crack formed after 10 passes (Fig. 5.13) disclosed the distinct nanocrystalline oxide grains filling crevices formed on the roughened crack face due to surface deformation. At this stage of rolling, fine nanocrystalline grains could be detected lying beside the crack, within the Al-Mn bulk.

Fig. 5.14 displays a high resolution TEM analysis of the magnesium-rich oxide layer on the crack faces. Under both conditions showing the oxide layers consist of nanocrystalline grains and amorphous mixture. The HRTEM images of the crack faces in Fig. 5.14 are accompanied by corresponding selected area electron diffraction (SAED) patterns, which confirmed the structure of the oxide on the crack faces and revealed that they were composed of MgO, MgAl$_2$O$_4$ and Al$_2$O$_3$. The SAED analysis was used to identify Al$_2$O$_3$ (113) and (018) planes on the crack face after 1 pass (Fig. 5.14(a)), but only the Al$_2$O$_3$ (018) plane after 10 passes (Fig. 5.14(b)). However, the 1 pass crack face contained only the MgO (222) plane while MgO (222), (200) and (400) planes were
detected after 10 passes. Fig. 5.14(b) displays a composite image of the crack face after 10 passes along with the SAED analysis denoting the compositional change from the crack face to the bulk of the Al-Mn alloy. The face of the crack was composed of MgO, MgAl$_2$O$_4$ and Al$_2$O$_3$, while only MgO and MgAl$_2$O$_4$ were identified towards the bulk of the alloy.

The near-surface region away from the micro-cracks after the first pass (Fig. 5.15(a)) was covered in magnesium-rich oxide with a thickness of about 90 nm. The oxide layer was composed of a top MgO layer about 50 nm in thickness and a possible lower layer of Al$_2$MgO$_4$ based on EDS line scans (Fig. 5.15(b)). After 10 passes the near-surface had evolved, displaying damage due to propagating nano-cracks and extended to a depth of about 0.5 µm into the subsurface region (Fig. 5.16(a)). Comparable to the crack faces, nanocrystalline oxide grains with distinct oxide particles were also observed within the crevices of the nano-crack damaged near-surface region at this stage of rolling. In this micrograph, a top oxide layer about 60 nm in thickness was decorated by white gallium particles used in FIB milling. This oxide layer covered the oxide-filled nano-crack damaged region underneath it. EDS maps (Fig. 5.16(b)) confirmed that the top oxide layer and the oxides within the nano-crack induced damaged area at the near-surface region were rich in magnesium and oxygen. Fig. 5.17(a) displays the extensive nano-crack induced damage to the near-surface region. Damage due to propagation of nano-cracks appeared to have generated debris particles within the near-surface region. These loosen debris particles lay within the magnesium-rich oxide layer beneath the thin MgO top layer. EDS maps (Fig. 5.17(b)) identified these nano-particles embedded in the
magnesium-rich oxide layer within the nano-crack induced damaged areas to be mainly aluminum.

Figure 5.10. STEM micrographs of micro-cracks at near-surface of Al-Mn alloy after (a) 1 pass and (b) 10 pass.
Figure 5.11. TEM micrograph of micro-cracks displaying the nanocrystalline MgO layer lining crack faces after (a) 1 pass and (b) 10 passes.
Figure 5.12. Cross section of cracks displaying EDS map overlays and the accompanying area they were taken from after (a) 1 pass and (b) 10 passes.
Figure 5.13. STEM images of transverse micro-crack formed after 10 passes displaying distinct oxide grains on the roughened crack face as well as nanocrystalline grains within the near-surface of the Al-Mn alloy.
Figure 5.14. HRTEM images of the oxide-rich crack face accompanied with 2D-FTT patterns after (a) 1 pass and (b) 10 passes.
Figure 5.15. Cross sectional (a) TEM image and (b) EDS line scan of Al-Mn near-surface layer after 1 pass displaying the porous nanocrystalline magnesium-rich oxide layer.
Figure 5.16. Cross sectional (a) TEM image and (b) EDS map of Al-Mn near-surface layer after 10 passes displaying the porous nanocrystalline magnesium-rich oxide layer.

Figure 5.17. (a) TEM micrograph of the near-surface damage and (b) EDS map from the same area displaying nano-particles embedded in the oxide layer of Al-Mn alloy after 10 passes.
The increase in surface roughness observed on the Al-Mn alloy surface after the first hot rolling pass is associated with the misalignment of grains caused by GBS (Fig. 5.1 (a) and 5.2(a)). Micro-cracks observed at the grain boundaries (Fig. 5.2(a)) dominantly occur during the sliding of the near-surface grains induced by the high shear stresses experienced during the first rolling pass [11]. These micro-cracks occurred at random orientations along the grain boundaries and were observed to propagate into the subsurface region (Fig. 5.7(a)). After the tenth rolling pass, there was no evidence of grain misalignment on the alloy surface (Fig. 5.2(b)). Micro-cracks at this stage of rolling appeared to only occur in the transverse direction, but EDS maps were used to identify previous cracks on the surface that had experienced oxidative self-healing through magnesium enrichment (Fig.5.5). Earlier research has shown that magnesium diffused to the new free surfaces formed by cracks [11], thus the magnesium-rich grain boundaries could actually be previous micro-cracks that had been healed by MgO formation within the crack openings. This was confirmed by Figure 8, which displayed cracks filled with porous MgO. Also, magnesium enrichment at the grain boundaries was not observed. Crack propagation into the subsurface region along the grain boundaries was also observed after the first and the tenth passes, with cracks propagating to greater depths after the tenth pass (Fig. 5.7). Hence, the oxide decorated grain boundaries within the near-surface deformed layer, previously reported in literature [8], [9], [6], [5], could dominantly be initiated by the evolution of oxide healed cracks.

Micro-cracks propagating into the subsurface region had their faces rich in magnesium and oxygen (Fig. 5.12). The porous oxide coating the crack faces had a
nanocrystalline and amorphous mixed structure and was confirmed to be mostly composed of MgO (Fig. 5.14). This oxide was thicker after 10 passes, filling crack branches and the tail of the crack (Fig. 5.11). Cross-sectional analysis of the cracks (Fig. 5.13) revealed nanocrystalline grains in the regions close to the crack faces. Similarly, Tzedaki et al. [8] have reported nanocrystalline-amorphous mixture of MgO on crack faces. They also noted nanocrystalline grains in areas around the cracks. Nanocrystalline grains were also observed within the near-surface layer beneath the porous oxide layer (Fig. 5.16(a) and (b)). The fine grain structure of the near-surface layer has been reported to be a result of the high shear strains experienced during rolling [2], [8], [9], [4]. Damage to intermetallic particles could be observed after the first rolling pass (Fig 5.3(a)). These intermetallic particles provide free surfaces around them for magnesium diffusion (Fig 5.2 and 5.3(b)), due to the gaps and cracks that occur around the intermetallic particles during rolling (Fig. 5.9(b)). Rolling stresses and strains continue to submerge intermetallic particles into the near-surface layer, with oxide piling-up around them, thus engulfing these damaged particles into the near-surface region.

Near-surface damage after 10 passes included a porous magnesium-rich oxide layer filling crevices in the near-surface region beneath it (Fig. 5.16(a)). The crevices of the nano-crack damaged near-surface were not apparent in planar views, neither by SEM (Fig. 5.2(b)) nor by surface profilometry (Fig. 5.1(b)), probably due to the thin MgO layer coating it (Fig. 5.16(b)). This thin MgO oxide layer was about 60 nm thick and possessed a similar structure as the MgO layer present on the surface after the first pass (Fig. 5.15(b)). Near-surface damage was observed after the first pass (Fig. 5.15(a)), but it was not as extensive as that observed after 10 passes (Fig. 5.16(a)), which extended 0.5
µm in depth into the subsurface region. This indicates that the extent of near-surface damage was dependent on the number of rolling passes. Damage to the near-surface region occurred mainly due to nano-crack propagation within the areas beneath the oxide which could generate some aluminum debris (Fig. 5.17). Due to the high rolling temperatures and magnesium’s tendency to diffuse to free surfaces, these aluminum nano-particles could soon become free of magnesium, lying within the oxide layer of near-surface damaged region (Fig. 5.17(b)).

Therefore, the near-surface region continued to evolve between the first and tenth passes, with the propagation of transverse micro-cracks and healing of micro-cracks occurring in other orientations, accompanied by continued MgO enrichment to crack faces. Additionally, damage to intermetallic particles embedded into the surface continued to increase with the number of passes. There was a distinct difference in the microstructure of the near-surface region by the tenth pass in comparison to that observed after the first pass. The presence of nanocrystalline grains within the near-surface region was observed after 10 passes but not after the first pass.

Based on the cross-sectional analysis, the mechanisms for the evolution of the near-surface layer could be summarized to include the following, magnesium diffusion to the free surfaces led to the formation of a porous magnesium-rich oxide layer with a nanocrystalline structure. The formation of micro-cracks at grain boundaries provides additional free surfaces for magnesium diffusion. Nano-crack propagation within the near-surface, because of high shear stresses, form aluminum debris particles within the oxide layer, and high shear strains generate nanocrystalline grains within the near-surface region.
5.5 CONCLUSIONS

Surface and near-surface damage evolved on an Al-Mn alloy, containing a significant amount of Mg, after 1 and 10 hot rolling passes were characterized and mechanisms inducing their formation were studied. The following conclusions were derived:

1. The near-surface region after the first rolling pass was characterized by micro-cracks at grain boundaries that propagated into the subsurface region, fractured intermetallic particles, and an oxide layer covering the surface of the alloy and lining the crack faces.

2. After the tenth pass, the near-surface region was characterized by transverse micro-cracks propagating into the subsurface region, healed micro-cracks filled with MgO, embedded fractured intermetallic particles, nanocrystalline Al-Mn grains and a porous nanocrystalline MgO layer filling crack induced damaged areas.

3. Micro-cracks were initiated by grain boundary sliding in the first pass. Non-transverse micro-cracks experienced oxidative healing by magnesium diffusion to the crack faces during subsequent passes.

4. The mechanisms of the near-surface microstructure evolution included magnesium diffusion to free surfaces, nano-crack propagation within the near-surface induced by shear stresses, micro-crack formation due to grain boundary sliding and nanocrystalline grain formation due to high shear strains.
BIBLIOGRAPHY


CHAPTER 6

THE DEVELOPMENT OF NEAR-SURFACE MICROSTRUCTURES DURING HOT ROLLING OF ALUMINUM-MAGNESIUM ALLOYS IN RELATION TO WORK ROLL TOPOGRAPHY

6.1 INTRODUCTION

Surface defects induced on aluminum alloys during the rolling process are known to be influenced by the work roll material and surface conditions, lubrication conditions and composition, the tribological conditions at the roll/ work piece interface, roll speed, forward slip and other rolling parameters [1]–[2]. Surface features of the rolled aluminum products include shingles, transverse cracks, grooves and rolling ridges are induced by the surface morphology of the work roll [3]–[7]. The tribological interactions at the work roll/ work piece interface are affected by the high shear stresses experienced during rolling and the work roll topography, particularly the roll roughness, as it influences the lubricating conditions and roll separation during rolling [8]–[11]. Surface roughness is also necessary for drawing the work piece into the roll bite and for generating the required surface finish on the rolled product [10]–[12]. The surface roughness of the rolled aluminum product is a function of both the rolling reduction and the work roll roughness [1],[8]. The final morphology of the work piece surface is therefore a reflection of the work roll surface morphology [1].

The tribological interaction between the work roll and aluminum work piece also results in material transfer between the contact surfaces [13]. This material transfer or adhesion occurs almost instantaneously regardless of the roll surface morphology, and
induces a coating on the roll surface [1],[14]. However, material transfer to the work roll increases at higher roll roughness, and thus has a significant influence on the surface morphology of the roll and thereby the surface evolution of the work piece surface features during rolling [1],[14].

The high shear stresses experienced during hot rolling have been shown to induce a tribolayer with a distinct near-surface microstructure, which differs in microstructure and composition from the bulk aluminum alloy [1],[5],[7],[13],[15]–[19]. This near-surface microstructure consists of an oxide surface layer (composed of MgO, γ-Al2O3 and MgAl2O4) and a subsurface layer of metallic ultra-fine grains mixed with oxide particles [1],[5]–[7],[13],[15]–[22]. The manifestation of surface defects such as shingles and transverse cracks have been associated with the roll surface/aluminum alloy surface interactions and the development of the tribolayer on the alloy’s surface [6],[7],[13].

Studies on the formation mechanisms of near-surface layers have linked the work roll roughness to the formation of shingles either by material transfer or by the plastic deformation of micro-wedges formed by the grooves on the work roll surface [13],[18]. Investigations into the influence of roll grinding, roll wear and rolling aspect ratio on the formation of tribolayers have revealed that high rolling aspect ratio combined with rough, worn roll surfaces resulted in the rapid material removal from aluminum alloy surfaces and formation of the fine grained and oxide-rich subsurface layers [1],[19]. The thickness of the tribolayer was directly related to the height of the asperities on the work roll surface [1],[19]. Investigations into the effects of the roll surface roughness on surface quality of stainless steel strips revealed that the rate of occurrence of micro-defects reduced with lower roll roughness [23]. It was also observed that increasing work roll
roughness had a direct relation with lubricant distribution and entrapment on the surface of the steel alloy [23].

As the final rolled surface quality is a result of the rolling conditions and work roll surface morphology, modifications of work roll surface roughness can improve the rolled product surface quality as well as mill productivity [1],[4]. A detailed relationship between the development of the near-surface microstructures and the rolling conditions has yet to be fully established [1],[7]. This study focuses on the effect of work roll surface morphology on the near-surface microstructure evolution in Al-Mg alloys by using two rolls with significantly different surface morphologies, a smooth (polished) work roll and a rough (WC-coated) work roll under similar rolling conditions.

6.2 EXPERIMENTAL PROCEDURE

Hot rolling tests were carried out using a tribo-simulator with a roll-on-block configuration, whose operational principles have previously been described [18]. Work rolls were machined from AISI 52100 steel to a diameter of 21 mm. The work rolls surface conditions were smooth, (polished with 1 µm diamond paste to a roughness ($R_a$) of 0.01µm) and rough (WC-coated by an electrofusion process to roughness ($R_a$) of 5.68 µm). Optical interferometry was used to examine the surfaces using a WYKO NT1100 in the vertical scanning interferometry, VSI, mode. The surface profiles of the smooth and rough rolls are displayed in Figure 6.1. Both work rolls were cleaned after each test with a 15% (wt/wt) sodium hydroxide solution to remove any aluminum transfer. The smooth roll was polished after sodium hydroxide cleaning to maintain the surface roughness.
The Al-Mg blocks were machined to dimensions of 10 mm width, 30 mm thickness, 95 mm length and then polished with a 1 µm diamond paste before being ultrasonically cleaned in acetone to remove surface contaminants. The composition of the Al-Mg alloy contained 4.5 wt.% of Mg. Rolling tests were carried out at a 7% forward slip for 10 passes, with the rolling direction reversed after each pass (simulating reversing mill rolling). Hot rolling temperatures started at 550 °C for the first rolling pass with a 10 °C temperature reduction at each subsequent pass, such that the temperature at the final (10th) rolling pass was 460 °C. Lubrication was provided by an oil-in-water emulsion with a 4% (v/v) concentration.

The specimen contact surfaces were then examined using an FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. The near-surface microstructure of the samples was examined using a ZEISS NVision 40 Cross Beam workstation focused ion beam (FIB), with a gallium ion beam operated at low beam currents and a voltage of 30 kV. The surfaces of the samples were protected by the deposition of a thin layer of carbon. Cross-sectional trenches were ion-milled using the FIB H-bar method. The samples prepared by the lift-out method were examined using an FEI Titan 80-300 LB transmission electron microscope (TEM).
6.3 EXPERIMENTAL RESULTS

6.3.1 Surface Analysis of the Rolled Al-Mg Alloy

The surface topography of the Al-Mg alloy samples after the 10 pass schedule with the smooth and rough work rolls reflect the work roll surface topography. The surfaces of the smooth (Fig. 6.2(a)) and rough (Fig. 6.2(b)) rolled samples possessed a darkened appearance in comparison to the as-polished surface. The surface of the smooth rolled sample distinctly featured grain boundaries with a greyish appearance in comparison to the generally dark surface of the sample (Fig. 6.2(a)). Intermetallic particles could also be seen randomly dispersed on the alloy surface. The surface of the rough rolled sample clearly revealed higher roughness in the form of shingles, grooves and the imprinted surface morphology of the work roll as displayed in the SEM image in Figure 6.2(b). Intermetallic particles could not be identified on the surface, as they were likely lying beneath the shingles covering the alloy surface.

Figure 6.1. Surface topography images displaying the morphologies of the (a) smooth and (b) rough work rolls.
Micro-cracks, transverse to the rolling direction, were observed sparsely on the surface of both the smooth (Fig. 6.3(a)) and the rough (Fig. 6.3(b)) rolled samples. These transverse cracks were narrow and appeared sealed with a dark material. Both rolled surfaces were also covered with nano-cracks and cavities.

Energy dispersive spectroscopy (EDS) analysis of the surfaces of the rough and smooth rolled samples (Fig. 6.4) revealed that both surfaces were rich in magnesium and oxygen. The dark powder-like material sealing micro-cracks were also observed to be composed of magnesium and oxygen. The distribution of the magnesium and oxygen on the surface of the smooth rolled sample (Fig. 6.4(a)) was continuous in comparison to that on the rough rolled sample (Fig. 6.4(b)).

Higher magnification images of the intermetallic particles observed on the smooth rolled surface (Fig. 6.5) revealed that they were mostly embedded into the surface with oxide pile-up at the surrounding edges of these particles, while others were completely covered by the surface oxides. The surfaces of several of these intermetallic particles were worn and several others were fractured, displaying cracks running transverse to the rolling direction within them.
Figure 6.2. Scanning electron images of Al-Mg alloy surfaces after a 10 pass hot rolling schedule with (a) smooth work roll and (b) rough work roll.

Figure 6.3. Scanning electron images of Al-Mg alloy surfaces displaying transverse cracks after a 10 pass hot rolling schedule with (a) smooth work roll and (b) rough work roll.
Figure 6.4. EDS maps of Al-Mg alloy surfaces after a 10 pass hot rolling schedule with (a) smooth work roll and (b) rough work roll.

Figure 6.5. Scanning electron image of the smooth rolled Al-Mg sample displaying intermetallic particles embedded into the surface after a 10 pass hot rolling schedule.
6.3.2 Subsurface Analysis of the Rolled Al-Mg Alloy

Subsurface analysis of micro-cracks occurring transverse to the rolling direction on the surface of the Al-Mg alloy samples revealed that these cracks had become part of the near-surface microstructure. These near-surface areas are displayed in Figure 6.6 with near-surface features extending to depths of 1.5 µm and 8.6 µm for the smooth (Fig. 6.6(a)) and rough (Fig. 6.6(b)) rolled samples, respectively. There was no distinctive evidence of the surface transverse micro-crack at the subsurface of the smooth rolled Al-Mg sample (Fig 6.6(a)), while a prominent micro-crack, extending deep into the subsurface region of the rough rolled sample, with crack branches was observed (Fig 6.6(b)). Subsurface cracks in both the rough and smooth rolled samples were filled with a porous material, composed of magnesium and oxygen. The near-surface microstructure of both samples contained aluminum nano-particles, embedded in the porous magnesium-rich oxide, which filled the near-surface damaged areas. These particles could be debris from the propagation of nano-cracks or the crack branches. Extensive damage to the near-surface of the rough rolled sample included subsurface cracks parallel to the surface, which were also filled with magnesium-rich oxides, as well as cavities close to the surface (Fig 6.6(b)). Subsurface analysis away from micro-cracks revealed a continuous oxide-rich layer and uniform damage occurring at the near-surface region of the smooth rolled sample (Fig. 6.7(a)) in comparison to that of the rough rolled sample (Fig. 6.7(b)). The oxide-rich near-surface layer was 0.43 ± 0.79 µm thick on the smooth rolled sample, while on the rough rolled sample it ranged from 0.35 ± 0.09 µm at the thinnest area to 3.62 ± 0.18 µm at the thickest area. This oxide-rich near-surface layer was porous and
appeared to be similar to the porous oxide-rich material filling micro-cracks within the subsurface region. EDS analysis confirmed that the oxide-rich layers were also rich in magnesium.

Therefore, cross-sectional analysis of the near-surface region of the hot rolled samples revealed oxide filled transverse micro-cracks and aluminum debris particles embedded in a porous magnesium-rich oxide layer for both the smooth and rough rolled samples. However, transverse micro-cracks extended deeper into the subsurface, and contributed to producing a non-uniform near-surface microstructure for the rough rolled Al-Mg sample. The smooth rolled Al-Mg sample, on the other hand, possessed a continuous oxide layer and a more uniform near-surface layer. This would suggest that the thickness of the near-surface layers could be directly related to the depth of the transverse cracks and the surface topography of the work roll.

Further subsurface examination of the smooth rolled, taken from a region with a transverse crack on the surface, (Fig. 6.8(a)) and rough rolled, taken from a region with a shingle and micro-crack, (Fig. 6.8(b)) samples were used for TEM analysis which is discussed in the next section.
Figure 6.6. Cross-sectional secondary electron images displaying micro-cracks and Al-nano-particles within the Al-Mg alloy subsurface after a 10 pass hot rolling schedule with (a) smooth work roll and (b) rough work roll.

Figure 6.7. Cross-sectional secondary electron images displaying the porous Mg-rich oxide layer of the Al-Mg samples after a 10 pass hot rolling schedule with (a) smooth work roll and (b) rough work roll.
6.3.3 TEM Characterization of the Near-Surface Regions of the Rolled Al-Mg Alloy

TEM micrographs of the smooth and rough rolled samples’ cross sections in Figure 6.8 are illustrated in Figures 6.9(a) and (b) respectively. Both TEM images show the porous, nanocrystalline structure of the oxide-rich layers. Figure 6.9(a) confirms that the transverse micro-crack was part of the oxide layer of the near-surface structure, with its crack faces composed of the porous oxide. The continuous oxide layer had an average thickness of $0.69 \pm 0.20 \, \mu m$. TEM analysis revealed that the oxide layer was comprised of two regions; a thin compact nanocrystalline layer about $57 \pm 13 \, nm$ thick on top and a more porous nanocrystalline oxide layer beneath that top layer. There were breaks within the compact top layer, which could be the nano-cracks observed on the surface of the rolled Al-Mg alloy. A distinct interface was observed at the near-surface region between the oxide layer and the bulk microstructure (Fig. 6.9(a)). This interface ran along the surface of the bulk that was damaged due to nano-crack propagation within the near-surface region. Nano-crack propagation occurring lateral to the sample surface added to
the crack-induced damage within the near-surface region. These lateral nano-cracks were filled with porous magnesium-rich oxides that covered all free surfaces of the Al-Mg sample.

Figure 6.9(b) shows the cross-sectional TEM view of a shingle comprised of Al-Mg layers, and a transverse micro-crack. This material overlap was completely covered with porous oxide, even at areas where it was in full contact with the sample surface. The material overlap, which made up the shingle, concealed a round shaped debris particle that seemed to have been transferred to the subsurface of the sample from the work roll (Fig. 6.9(b)). The nanocrystalline oxide layer on the rough rolled sample varied in thickness in the range of 0.04 – 0.55 µm, with an average thickness of 0.20 ± 0.16 µm along the subsurface, areas devoid of the porous oxides were also detected. The transverse micro-crack was noted to propagate along the grain boundaries and was confirmed to be filled with porous magnesium-rich oxides. The darker color of the embedded debris particle confirmed it had a different composition (made up of a heavier element) compared to the surrounding area.

Higher magnification TEM analysis of cross sections of the smooth (Fig. 6.10(a)) and rough (Fig. 6.10(b)) rolled samples revealed that the near-surface microstructure was comprised of ultrafine grains (48 – 400 nm and 140 – 400 nm, respectively) and oxide particles. The cross-sectional micrographs also highlighted the fractured surfaces which gave the surfaces a roughened appearance underneath the porous magnesium-rich oxides. EDS maps of the near-surface layers (Fig. 6.10 (c) and (d)) confirmed that the porous oxide layer within the near-surface region were mainly composed of magnesium and oxygen. Close examination of the EDS maps showed evidence of aluminum
nanoparticles embedded in the porous magnesium-rich oxides. This is confirmed by EDS maps taken at higher magnification clearly displaying aluminum nano-particles enclosed in the magnesium-rich oxide in Figures 6.11(a) for the smooth rolled and 6.11(b) for the rough rolled Al-Mg samples. The embedded aluminum particles could be observed to have their faces enriched with magnesium.

High resolution TEM (HRTEM) analysis of the porous magnesium-rich oxide from the smooth rolled (Fig. 6.12(a)) and rough rolled (Fig. 6.12(b)) Al-Mg samples identified both to be comprised of nanocrystalline particles mixed with an amorphous material. The nanocrystalline particles were stacked on each other and possessed a random orientation, as shown in the accompanying selected area electron diffraction (SAED) patterns displayed in Figure 6.12 (c) and (d). Polycrystalline MgO with (400), (220) and (200) planes were identified for both the smooth rolled (Fig. 6.12 (c)) and rough rolled (Fig. 6.12(d)) samples.

TEM micrographs of the shingles observed on the rough rolled sample (Fig. 6.13) revealed in greater details the shingles trapped a debris particle beneath the surface. The concealed particle was most likely debris from the work roll rich in iron, tungsten and oxygen as displayed in Figure 6.13(b). The work roll debris, with a diameter of about 0.5 µm, appeared to be embedded into the subsurface and had become part of the shingle. The cross-sectional view (Fig. 6.13(a)) suggested that the debris particle was covered by two shingles, labelled shingles 1 and 2, with shingle 2 loosely lying on top of shingle 1. Shingle 1 seemed attached to the transferred debris particle and was obviously formed first, coating the debris particle. The loosely attached Shingle 2 could have been formed during subsequent rolling passes. The head of shingle 2 was separated from the bulk (in
this cross section) and was comprised of loosely attached aluminum nano-particles. Both shingles were composed of aluminum, completely coated with magnesium and oxygen (Fig. 6.13(b)) and seemed to be nanocrystalline (Fig. 6.13(a)). The magnesium-rich oxide layers appeared as interfaces between the shingles and the bulk alloy.

The subsurface of both the smooth and the rough rolled Al-Mg samples comprised of a porous nanocrystalline MgO layer, ultrafine grains less than 400 nm in size, aluminum nano-particles embedded within the oxide layers and a distinct interface between the near-surface and the bulk material. The near-surface microstructure of the smooth rolled sample had a continuous oxide layer that was coated with a thin, compact, oxide film. The rough rolled sample’s near-surface microstructure, however, included shingles and a discontinuous oxide layer.
Figure 6.9. Cross-sectional TEM images displaying the near-surface features of the Al-Mg alloy subsurface after a 10 pass hot rolling schedule with (a) smooth work roll and (b) rough work roll.
Figure 6.10. Cross-sectional scanning transmission electron images exposing the near-surface ultrafine grains and roughened surfaces within (a) smooth rolled and (b) rough rolled Al-Mg samples after a 10 pass hot rolling schedule. (c) and (d) corresponding EDS maps of areas shown in (a) and (b) respectively.

Figure 6.11. EDS analyses displaying the aluminum nano-particles embedded in porous oxide of the Al-Mg alloy near-surface regions after a 10 pass hot rolling schedule with the (a) smooth work roll and (b) rough work roll.
Figure 6.12. HRTEM micrographs revealing the nanocrystalline structure of the magnesium-rich oxide of (a) smooth rolled and (b) rough rolled Al-Mg samples after a 10 pass schedule. Corresponding SAED patterns identifying MgO planes for (c) smooth rolled and (d) rough rolled Al-Mg samples.
Figure 6.13. Cross-sectional TEM micrograph of the rough rolled Al-Mg alloy’s near-surface region after a 10 pass hot rolling schedule displaying (a) implanted work roll debris concealed by shingles and (b) EDS maps of area shown in (a).
6.4 DISCUSSION

As stated in previous sections, hot rolling was conducted using work rolls with two different surface roughness (Ra) of 0.01 µm (smooth roll) and 5.86 µm (rough roll). Surface roughness of rolls used in conventional hot rolling lie within the 1 - 3 µm range [24]. A study of the surface and subsurface features observed on the smooth and rough rolled Al-Mg alloy samples would aid in understanding the effects the work roll surface topography has on the development of the near-surface defects. The occurrence of shingles and grooves on the rough rolled sample (Fig. 6.2(b)) suggested that these features were induced by the rough topography of the work roll, as neither shingles nor grooves were observed on the smooth rolled surface (Fig. 6.2(a)). The smooth rolled Al-Mg surfaces showed no evidence of rolling marks observed on traditional hot rolled surfaces. This confirmed that the surface topography of the rolled material was a reflection of the work roll topography. Previous work [18], has suggested that shingles were formed from the shear deformation of micro-wedges induced by hot aluminum squeezing into grooves. This was most likely the mechanism of formation for shingle 1 (Fig. 6.13(a)), which formed first as evident, in part, from the magnesium-rich oxide interface between the shingle and the bulk alloy. The severe plastic deformation the micro-wedge experienced due to the deep valleys between the asperities (Fig. 6.1(b)) during the formation of this shingle resulted in its nanocrystalline structure (Fig. 6.13(a)). Shingle 2, which was observed, lying on top of shingle 1, also formed due to shear deformation of micro-wedges, with the loosely attached debris particles generated during the shear deformation.
Material transfer from the work roll to the Al-Mg alloy surfaces was observed in the form of work roll debris embedded beneath the shingles. The textured rough work roll was covered in asperities with heights between 0.5 – 20 µm as observed from the work roll surface profile (Fig. 6.1(b)). The height of the debris covered by the shingles was about 0.5 µm (Fig. 6.13(a)) and from EDS analysis (Fig. 6.13(b)) was composed of tungsten and iron. During rolling passes, the asperities are continuously embedded into the Al-Mg surface inducing grooves and shingles on the alloy surface. Shear stress experienced during rolling could easily fracture the embedded portion of an asperity. The work roll debris fractured from the work roll would be embedded in the surface of the Al-Mg alloy due to the high rolling stresses similar to the worn embedded intermetallic particles observed on the smooth rolled Al-Mg (Fig. 6.5). Shingles could then be formed over the embedded work roll debris, causing the debris to become part of the near-surface.

Transverse micro-cracks, another feature in near-surface layers, were observed on both the smooth and the rough rolled surfaces (Fig. 6.3). They propagated into the subsurface, with deeper cracks observed on the rough rolled sample (Fig. 6.6(b)) compared to the smooth rolled sample (Fig. 6.6(a)). TEM analysis of crack propagation in the smooth rolled sample showed that the crack became part of the oxide layer of the near-surface microstructure (Fig. 6.9(a)). Previous work [25] had shown that the faces of transverse cracks were preferential sites for magnesium diffusion, and the cracks in the smooth rolled sample were observed to be filled with porous MgO after the fourth rolling pass. The thickness of the oxide layer had also increased by the fourth pass, due to magnesium diffusion to free surfaces. Transverse cracks in the rough rolled Al-Mg
sample propagated along the grain boundaries of ultrafine grains and were filled with porous MgO. It was previously shown that micro-cracks within the near-surface area propagated along grain boundaries and were healed during the first rolling pass by the oxidative self-healing mechanism [25].

EDS analysis revealed a continuous near-surface oxide layer covering the smooth rolled sample (Fig. 6.4(a)), while a discontinuous oxide layer was observed on the rough rolled sample (Fig. 6.4(b)). It was also evident from the EDS maps that there were several areas on the rough rolled surface devoid of magnesium and oxygen. This correlated well with the TEM analysis of both smooth (Fig. 6.9(a)) and rough rolled (Fig. 6.9(b)) samples. The composition of the oxide layer was confirmed to be MgO by SAED analysis (Fig 6.12(c) and (d)), which showed similar crystallographic planes for both smooth and rough rolled samples and confirmed they possessed similar structures (Fig. 6.12(a) and (b)). Considering that the only difference between the rolling conditions of the samples was the surface topography of the work roll, a comparison of the oxide layer on both rolled surfaces would suggest that the rough work roll would aid the redistribution of oxides across the rolled Al-Mg surfaces more effectively. This would most likely occur due to the high asperities and deep valleys displacing ploughed oxides across the surface, which would also experience plastic deformation. Oxide redistribution was also evident on the smooth rolled surface, to a small degree, in the form of oxide pile-ups at the edges of worn, embedded intermetallic particles (Fig. 6.5). These particles, worn and embedded into the Al-Mg surface during the application of rolling strains, formed craters that would be filled by deformed oxides.
Both the smooth and the rough rolled samples had nanocrystalline aluminum nano-particles embedded into their oxide-rich layers (Fig. 6.11). The subsurface cracks propagating parallel to the surface in Figure 6.9(a) and the fractured surfaces of the near-surface grains in Figure 6.10(a) and (b) indicated that aluminum debris particles could have formed by the ablation of the surfaces and the propagation of subsurface nano-cracks, subsequently magnesium diffusion to their faces would leave aluminum nano-particles within the porous magnesium-rich oxide. Since these aluminum nano-particles and micro-cracks were observed on both the smooth and rough rolled samples, it might be suggested that crack propagation within the near-surface would be due to rolling stresses and strains rather than the roll topography.

In summary, the formation of transverse micro-cracks, damage to intermetallic particles, aluminum nano-particles, the nanocrystalline structure of the near-surface region beneath the oxide layer and the structure of the oxide layer were due to the high shear stresses and strains experienced during hot rolling. These were imposed on the Al-Mg near-surface region regardless of the work roll topography. On the other hand, the introduction of shingles, grooves and rolling marks as well as the enhanced depth and non-uniformity of the near-surface damage could all be related to the surface topography of the work roll. Therefore, the work roll morphology plays a vital role in the induction of surface damage to the Al-Mg alloys and can influence the formation mechanisms of the near-surface microstructure.
6.5 CONCLUSIONS

The surface/near-surface features formed on an Al-Mg alloy during a 10 pass hot rolling schedule with smooth and rough work rolls were examined. A comparison of both smooth and rough rolled Al-Mg surfaces was made and the following conclusions were drawn:

1. The near-surface microstructures consisting of aluminum nano-particles embedded in a magnesium-rich oxide layer formed on the surfaces of the samples hot rolled with both smooth and rough work rolls.

2. The surface morphology of the rolled samples was a reflection of the surface morphology of the work roll. Shingles and grooves were observed on the rough rolled sample, while grain boundaries and embedded intermetallic particles were prominent on the smooth rolled sample.

3. The near-surface of the smooth rolled sample possessed a continuous oxide layer and uniform near-surface damage in comparison to the rough rolled sample that had a discontinuous oxide layer and deeper, non-uniform near-surface damage.

4. The influence of the surface topography of the work roll included the formation of shingles, the redistribution of oxides and the enhancement of the near-surface damage.

5. The influence of the hot rolling stresses and strains included the propagation of transverse cracks, the nanocrystalline structure of the near-surface layers and aluminum nano-particles embedded in the magnesium-rich oxide layer.
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CHAPTER 7
THE INFLUENCE OF WORK ROLL ROUGHNESS ON THE SURFACE/NEAR-
SURFACE MICROSTRUCTURE EVOLUTION OF HOT ROLLED
ALUMINUM-MAGNESIUM ALLOYS

7.1 INTRODUCTION

The surface appearance of wrought aluminum sheets is one of the major criteria used to evaluate their commercial value. The surface quality of these sheets however, is critically influenced by hot rolling conditions. The surface and near-surface features of rolled aluminum sheets are a result of the tribological conditions at the work roll/work piece interface, which are influenced by such rolling parameters as forward slip, pressure, speed, temperature, lubrication conditions, and the surface topography of the work rolls. However, the morphology of the rolled aluminum surface is a reflection of the work roll surface morphology. Dick and Lenard [1] observed an increase in surface roughness of the rolled aluminum sheet with increased work roll roughness, while showing that the aluminum sheet roughness would also depend on the reduction chosen. Frolish et al. [2] proposed that the combination of the sticking conditions and forward/backward slip conditions, which cause ploughing and machining by the work roll asperities, would imprint the work roll surface on the rolled aluminum. Frolish et al. [3] suggested that the work roll roughness should be one of the major factors that determines the surface/near-surface features of the aluminum alloy at each stage of rolling.

Rolled aluminum sheets’ surfaces have been observed to be covered with shingles, transverse cracks, grooves and rolling ridges, all features that are a function of
the surface morphology of the work roll. However, the work roll surface morphology is actually essential for drawing the work piece into the roll bite. Dick and Lenard [1] established the work roll surface morphology’s influence on the rolling lubrication conditions as well as the roll separating force. Tan et al. [4] calculated the oil film thickness using an average flow model, and showed that the work roll surface morphology also influences the oil film formation in the deformation zone during aluminum rolling. They reported the close relation of the oil film thickness with the directionality of the work roll roughness lay. According to Sutcliffe and Le [5], while the work roll roughness generates the necessary surface finish on the rolled product, the cold rolled aluminum surface roughness is also affected by the thickness of the lubricant film. Chen et al. [6] observed that the rate of occurrence of micro-defects on stainless steel surfaces was decreased by using a lower work roll roughness. Chen et al. [6] also noted a direct relation between the work roll roughness and the lubricant distribution and entrapment on steel alloy surfaces.

Frolish et al. [2] associated the presence of shingles and transverse cracks on rolled aluminum surfaces with the development of highly deformed near-surface layers that differ in composition and microstructure from the bulk alloy. Fishkis and Lin [7] identified these deformed near-surface layers as consisting of ultra-fine grains with grain boundaries decorated with oxide particles. Frolish et al. [2] also proposed that the deformed near-surface layers were a result of the high shear stresses induced during the hot rolling process due to the tribological conditions at the work roll/ work piece interface. Scamans et al. [8] observed that these near-surface layers were not limited to rolled aluminum but were also observed on mechanically ground and machined
aluminum. While Li et al. [9] identified the two types of near-surface deformed layers present on rolled aluminum, noting the difference between them as the presence of oxide particles at the grain boundaries and the temperature at which they were formed. Frolish et al. [2] related the thickness of the near-surface layer to the height of the asperities on the work roll surface. Frolish et al. [2] also, while referring to the shingles on the aluminum surface as asperities, identified using surface contour analysis, noted that the asperities appeared to correlate with the depths of the subsurface layers. Zhou et al. [10] established that the aluminum surface roughness corresponded to its shingled appearance. Liu et al. [11,12] though, directly related the thickness of the deformed near-surface layers with the shingles and intermetallic particle distribution. Liu et al. [11] also suggested that the occurrence of shingles and other near-surface features indicated the significant interaction between the work roll and aluminum surface.

Fishkis and Lin [7] suggested that shingle formation was related to material transfer to the aluminum surface during the rolling process, little though is known of the aluminum alloy or the rolling conditions. Gjonnes [13] proposed that shingle formation was due to the deformation or smearing of large steps, peaks, or protruding features formed on the aluminum surface from previous passes and that point in a direction opposite to the rolling direction. Gjonnes [13] and later Gjonnes and Andersson [14] suggested that this deformation was governed by forward slip and that shingle size and presence were related with the surface structure of the work rolls. Noting that his research was focused on the cold rolling of cast aluminum, it should be stated that in hot rolling of aluminum, shingles have been observed on the alloy surface from the first rolling pass. Liu et al. [11] cross-sectional examination of a shingle’s head separated from the bulk
alloy led them to conclude that shingle formation was due to the metal at the surface being pushed backward and smeared across the surface. Liu et al. [11] reported that the combination of high aspect ratios, high rolling speeds and worn (but still rough) roll surfaces induced high populations of shingles and thick near-surface deformed layers, whereas low shingle populations could be attained from freshly ground roll surfaces, low aspect ratios and low rolling speeds. Riahi et al. [15] proposed that shingle formation was due to the plastic deformation of micro-wedges formed by the grooves on the work roll surface. Riahi et al. [15] work also involved exploring the effect of forward slip on shingle occurrence, noting that an increase in forward slip resulted in higher frequency of shingle occurrence. Preliminary studies by Gali et al. [16], who evaluated the effect of the work roll roughness, indicated that shingles were formed on rolled Al-Mg alloy samples when a WC-coated work roll with a surface roughness ($R_a$) exceeding 5µm was used, but none were observed with a polished work roll with a roughness of 0.01 µm. Gali et al. [16] showed that the introduction of a ground work roll in the rolling process influenced the depth of near-surface damage induced on the aluminum alloy during hot rolling.

While a relation between the occurrence of shingles and the work roll roughness has been suggested, the prospect of mitigating the manifestation of shingles with a ground work roll is still in question. This is due to the inability of previous works to examine the effect of rolling parameters individually. Consequently, the underlying mechanism of shingle formation is still in dispute. The objective of this study is to further investigate the influence of the work roll surface morphology on the development of the near-surface microstructure on Al-Mg alloys. It continues to explore the effect of work rolls with different surface roughness values on the evolution of surface damage features.
and the near-surface microstructure. This study seeks to correlate the relationship between work roll roughness, shingle manifestation and the depth of near-surface damage.

7.2 EXPERIMENTAL PROCEDURE

Hot rolling tests were carried out using a rolling tribo-simulator with a roll-on-block configuration, with operational principles previously described by Riahi et al. [15] and displayed in Figure 7.1. The block, representing the aluminum slab, was fixed on a stage which allowed motion on both the X- and Y-axes. Load cells were used to measure the normal force and cartridge heaters to heat the sample. The temperature was monitored by means of a thermocouple inserted into the aluminum block. The block was heated to the desired rolling temperature while the roll was set to revolve at a desired speed, in lubricated condition. The stage was then set to move in the desired direction, allowing the roll to run across the face of the sample, at a contact pressure of 128 MPa. The operational principles of the configuration allow for the simulation of the tribological reactions occurring during rolling and sliding.

Work rolls were machined from a steel alloy AISI 52100 to a diameter of 21 mm. The work rolls’ surfaces were ground to surface roughness (R_a) values of 1.1 µm and 0.1 µm. The surface morphologies of the work rolls, which were examined with optical interferometry using a WYKO NT1100 in the vertical scanning interferometry (VSI) mode, both consisted of discontinuous grinding grooves (Fig. 7.2). The grooves on the 1.1 µm R_a work roll (Fig. 7.2a) were deeper and wider in comparison to those observed
on the 0.1 µm Rₐ work roll (Fig. 7.2b). The work rolls were cleaned after each test with a 15% (wt/wt) sodium hydroxide solution to remove aluminum transfer.

The Al-Mg blocks, which contained about 4.5 wt.% of Mg, were machined to dimensions of 10 mm width, 30 mm thickness and 95 mm length. The blocks were polished with a 1 µm diamond paste before being ultrasonically cleaned in acetone to remove surface contaminants. The microstructure of the polished Al-Mg blocks is displayed in Figure 7.3. The rolling schedule of the Al-Mg blocks involved ten passes at a 7% forward slip, with the rolling direction reversed with each pass. Hot rolling temperatures started at 550 °C for the first rolling pass with a 10 °C temperature reduction at each subsequent pass such that the temperature at the final (tenth) rolling pass was 460 °C. Lubrication was provided by an oil-in-water emulsion with a 4% (v/v) concentration.

The specimen contact surfaces were then examined using an FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. The near-surface microstructure of the samples was examined using a ZEISS NVision 40 Cross Beam workstation focused ion beam (FIB), with a gallium ion beam operated at low beam currents and a voltage of 30 kV. The surfaces of the samples were protected by the deposition of a thin carbon layer.
Figure 7.1. Schematic illustration of the rolling tribo-simulator set-up with arrows showing the direction of motion.

Figure 7.2. Surface topographies of work rolls with an $R_a$ of (a) 1.1 µm, and (b) 0.10 µm, displaying discontinuous grooves.
7.3 EXPERIMENTAL RESULTS

7.3.1 Surface Morphology of the Rolled Al-Mg Samples

The surface morphology of the samples rolled with the 1.1 µm and 0.1 µm R\textsubscript{a} work rolls reflected the morphology of the mating work roll surface. The hot rolled sample after the first pass with the rougher work roll possessed a surface roughness (R\textsubscript{a}) of 1.00 ± 0.11µm. The surface profile observed in Figure 7.4a displayed wide and deep grooves. The surface roughness remained relatively unchanged after 10 passes (Fig. 7.4b) at 1.02 ± 0.13 µm. The surface topography of the sample rolled with the smoother work roll was covered with shallow, thin grooves with a surface roughness of 0.21 ± 0.01 µm after the first pass (Fig. 7.5a). The surface was also covered in wavy steps, which were not observed after the tenth pass (Fig. 7.5b). The surface after 10 passes possessed only ridges and grooves with a roughness of 0.16 ± 0.01 µm.

The surface of the rolled samples thus possessed a higher surface roughness with deeper, wider grooves when it was rolled with the rougher work roll with little change to
the surface roughness after ten passes. The sample rolled with the smoother work roll displayed a slight surface roughness reduction with the number of rolling passes.

Figure 7.4. Surface profilometry images of the Al-Mg samples rolled with the 1.1 µm Ra work roll displaying surface topographies after (a) 1 pass and (b) 10 passes.

Figure 7.5. Surface profilometry images of the Al-Mg samples rolled with the 0.1 µm Ra work roll displaying surface topographies after (a) 1 pass and (b) 10 passes.
7.3.2 Characterization of Surface Features on the Rolled Al-Mg samples

A comparison of the surface features of the rolled samples revealed similar features after the first rolling pass (Fig. 7.6). Both rolled surfaces were covered with rolling marks, ridges and grooves imprinted by the work rolls during the hot rolling tests. However, the surface rolled with the rougher work roll (Fig. 7.6a) prominently displayed shingles on its surface, which could not be observed on the sample rolled with the smoother work roll (Fig. 7.6b). Instead, this sample prominently displayed surface offsets in the form of micro-steps induced by the grains overlapping at their boundaries due to grain boundary sliding. The sample also possessed a smoother appearance with fewer grooves and ridges in comparison to the surface rolled with the rougher work roll. Optical interferometry analysis shown in Figures 7.4 and 7.5 confirmed that the ridges covering the sample surface rolled with the rougher work roll appeared to possess larger peaks. Higher magnification SEM images of both rolled surfaces displayed in Figure 7.7 show similar surface morphologies, overlaid with granular oxide particles, induced during hot rolling. Both surfaces were covered in transverse micro-cracks and dark spots. Energy dispersive spectrometry (EDS) analysis revealed that the dark spots were rich in magnesium and oxygen (i.e. MgO islands), while the granular oxide particles were rich in aluminum, magnesium and oxygen.

Examination of the samples after ten hot rolling passes revealed the surfaces rolled with the rougher work roll (Fig. 7.8a) to be still covered in shingles, grooves, and ridges. The shingles on this surface appeared flattened and smaller in size when compared to the same surface after one rolling pass. The sample surface rolled with smoother work roll (Fig. 7.8b) still possessed no shingles with an increase in the
frequency of occurrence of ridges and grooves. Grain boundaries, devoid of overlaps, could be observed covering the alloy surface. These grain boundaries were darker in appearance in comparison to the rest of the surface of the alloy. At higher magnification (Fig. 7.9), transverse micro-cracks could be observed on both rolled surfaces. These micro-cracks appeared closed with no overlap, as was observed for micro-cracks detected on rolled surfaces after one pass. The higher magnification images also exposed the smoother appearance of the sample rolled with the smoother work roll (Fig. 7.9b). This surface was covered with MgO islands and had less damage in comparison to the surface rolled with rougher work roll (Fig. 7.9a). Figure 7.10 displays the crack damage induced on the rolled surfaces, which exhibit nano-cracks and micro-cracks. The cracks were observed by EDS analyses to be rich in magnesium and oxygen, assumed to be MgO, consistent with observations in previous reports. Furthermore, the higher frequency of nano-cracks on the surface rolled with the rougher work roll (Fig. 7.10a) was an indication of the greater amount of surface damage on this surface.

EDS maps of the surfaces rolled with the rougher (Fig. 7.11a) and smoother (Fig. 7.11b) work rolls revealed that the surfaces were covered with magnesium and oxygen. However, the distribution of magnesium and oxygen on the surface rolled with the rougher work roll (Fig. 7.11a) was non-continuous, with areas rich in aluminum and low in oxygen concentrations clearly identified. These regions appeared mostly to occur behind shingles where wide deep grooves were observed. The sample surface rolled with the smoother work roll (Fig. 7.11b) was uniformly coated with magnesium and oxygen.
Figure 7.6. Scanning electron images of Al-Mg alloy surfaces after 1 hot rolling pass with the (a) 1.1 µm $R_a$ and (b) 0.1 µm $R_a$ work rolls displaying shingles, grooves, ridges and grain boundary overlaps.

Figure 7.7. Scanning electron images of Al-Mg alloy surfaces displaying transverse micro-cracks and MgO islands after 1 hot rolling pass with the (a) 1.1 µm $R_a$ and (b) 0.1 µm $R_a$ work rolls.
Figure 7.8. Scanning electron images of Al-Mg alloy surfaces after a 10 pass hot rolling schedule with the (a) 1.1 µm Rₐ and (b) 0.1 µm Rₐ work rolls displaying shingles, grooves, ridges and grain boundaries.

Figure 7.9. Scanning electron images of Al-Mg alloy surfaces displaying transverse micro-cracks after a 10 pass hot rolling schedule with the (a) 1.1 µm Rₐ and (b) 0.1 µm Rₐ work rolls.
Figure 7.10. Scanning electron images of Al-Mg alloy surfaces after a 10 pass hot rolling schedule with the (a) 1.1 \( \mu \)m R\(_a\) and (b) 0.1 \( \mu \)m R\(_a\) work rolls displaying MgO-rich cracks.

Figure 7.11. EDS maps of Al-Mg alloy surfaces after a 10 pass hot rolling schedule with the (a) 1.1 \( \mu \)m R\(_a\) and (b) 0.1 \( \mu \)m R\(_a\) work rolls.
7.3.3 Subsurface Analysis of the Rolled Al-Mg Samples

Figure 7.12 displays cross-sections of the ridges on the rolled surfaces occurring along the rolling direction after the ten pass hot rolling schedule. The subsurface analysis revealed that the porous oxide layer coating the sample rolled with the rougher work roll (Fig. 7.12a) was not uniformly distributed. The oxide layer, transverse to the rolling direction, on this sample was also thicker (0.59 ± 0.34 µm) when compared to the oxide formed on the sample rolled with the smoother work roll (0.34 ± 0.13 µm), as displayed in Figure 7.12b, giving the appearance that the thinner oxide layer was more uniformly distributed. The oxide layer distribution for both samples appeared to be related to the size of the ridges, with thicker oxide layers appearing to surround the higher ridges (Fig. 7.12). These images also revealed the increased damage at the surface (in the form of rougher topography) and subsurface (in the form of oxide filled subsurface cracks) for the sample rolled with the rougher work roll, as compared to the sample rolled with the smoother work roll. Higher magnification images from the areas highlighted in Figure 7.12 are shown in Figure 7.13. The magnified images revealed that the areas adjacent to the top of the ridges were covered with a thinner porous oxide layer. Thus, the occurrence of the ridges appear to contribute to the non-uniformity of the oxide layer distribution across the rolling direction. Figure 7.13 also displays the wear of the near-surface layer, by material loss from chipping and nano-crack propagation, beneath the porous oxide layer.

Subsurface analyses of micro-cracks occurring transverse to the rolling direction on the surface of the samples are shown in Figure 7.14. The transverse micro-cracks shown in these images were observed to propagate into the subsurface region down to
depths of 3.2 µm for the sample rolled with the rougher work roll (Fig. 7.14a) and 2.8 µm for the sample rolled with the smoother work roll (Fig. 7.14b). These micro-cracks depict the maximum depths of near-surface damage observed on the rolled samples with the work rolls used in this study. The micro-cracks were filled with porous oxide similar to that covering the surface of the rolled samples. EDS analysis confirms that the oxide was rich in magnesium; previous work has indicated that this oxide was likely MgO. The width of the transverse micro-cracks observed within the near-surface region of the sample rolled with the rougher work roll was measured at 1.31 ± 0.51 µm on average, compared to 0.72 ± 0.25 µm for the sample rolled with the smoother work roll. The oxide layers along the rolling direction were measured at 0.44 ± 0.16 µm for the sample rolled with the rougher work roll and 0.30 ± 0.16 µm for the sample rolled with the smoother work roll.

The cross-sectional analysis of a shingle formed on the sample rolled with the rougher work roll is displayed in Figure 7.15a. The shingle was unevenly covered with an oxide layer, with areas at the tail of the shingle having little to no oxide coverage observed, corresponding to EDS analysis performed on the surface of the alloy (Fig. 7.11a). These areas also possessed less damage due to ablation, compared to the head of the shingle where extensive damage was observed. A higher magnification image of the highlighted area is displayed in Figure 7.15b, which reveals the damage in this area occurred due to crack propagation. This image also discloses the ultra-fine grains within the shingle along with loose aluminum particles embedded in the surface oxide layer.
The near-surface of hot rolled samples could, therefore, be characterized as possessing a porous oxide layer that varied in thickness in relation to the height of the rolling ridges and the surface roughness of the work roll. Transverse cracks were also observed, with widths and depths related to the work roll surface roughness. Further, ultra-fine grains were observed within shingles on the sample rolled with the rougher work roll.

Figure 7.12. Cross-sectional secondary electron images displaying the porous Mg-rich oxide layer covering the ridges of the Al-Mg samples subsurface after a 10 pass hot rolling schedule with the (a) 1.1 µm Rₐ and (b) 0.1 µm Rₐ work rolls.
Figure 7.13. Higher magnification cross-sectional secondary electron images (from regions within the inserted boxes in Figure 10) displaying the porous Mg-rich oxide distribution around the ridges on the Al-Mg samples after a 10 pass hot rolling schedule with (a) 1.1 µm Ra and (b) 0.1 µm Ra work rolls.
Figure 7.14. Cross-sectional secondary electron images displaying transverse micro-cracks within the Al-Mg alloy subsurface after a 10 pass hot rolling schedule with (a) 1.1 µm $R_a$ and (b) 0.1 µm $R_a$ work rolls.
Figure 7.15. Cross-sectional secondary electron images of the Al-Mg alloy’s near-surface region rolled with the 1.1 µm R_a work roll after a 10 pass hot rolling schedule displaying (a) the oxide distribution around a shingle and (b) higher magnification image of the area highlighted in (a), displaying the microstructure of the head of the shingle.

7.4 DISCUSSION

Previous work of Gali et al. [16] examining the influence of work roll topography on the development of near-surface microstructure on Al-Mg alloys confirmed that a smooth work roll, with an R_a of 0.01 µm, induced no shingles, rolling marks, nor grooves on the hot rolled surface. On the other hand, a textured work roll with an R_a of 5.86 µm developed grooves and shingles, covering the sample surface. This implied that the occurrence of rolling marks and shingles were, indeed, related to the surface topography of the work roll. The present work compares the effect of work roll roughness by again comparing two work rolls with varying surface roughness values, one with an R_a of 0.1 µm (Fig. 7.2b) and the other with an R_a of 1.1 µm (Fig. 7.2a). Surface examination of the Al-Mg samples hot rolled with the rougher work roll, presented the occurrence of
shingles and rolling marks, ridges and grooves, from the first rolling pass (Fig. 7.6a). The shingles were still present after ten rolling passes (Fig. 7.8a), although they appeared flattened and compressed. The samples hot rolled with the smoother work roll did not show any evidence of shingles after the first (Fig. 7.6b) nor tenth pass (Fig. 7.8b), though rolling marks were observed across the surface with a higher frequency after ten passes. These results indicate that, although rolling marks are a persistent feature imprinted on the rolled aluminum surface regardless of the surface roughness of the ground work roll, the occurrence of shingles can indeed be mitigated by the variation of the work roll surface roughness.

As stated earlier, it has previously been suggested by Riahi et al. [15] that shingles are formed from the local shear deformation of micro-wedges induced on the alloy surface when hot aluminum squeezes into grooves on the work roll surface. The formation of shingles via this mechanism corresponds to the results observed here. They suggest an explanation for the occurrence of shingles on the surface rolled with the rougher work roll and not the smoother work roll. The surface profiles of the rolled samples (Figs. 7.4 and 7.5) were observed to be a reflection of the work roll morphology (Fig. 7.2), with the rolled samples’ surface roughness values closely matching those of the work rolls after one and ten passes. Higher and wider ridges were observed on the surface rolled with the rougher work roll, though both rolled surfaces displayed ridges with similar morphology to the grooves observed on their corresponding work roll surfaces. This implies that the ridges (i.e., micro-wedges) were formed by the hot aluminum filling out work roll grooves. Micro-wedges would be deformed during rolling due to the discontinuous morphology of the work roll grooves. Since shingle occurrence
is related to the work roll roughness, a critical work roll roughness, below which the occurrence of shingles would be mitigated, could be suggested.

As indicated in the introduction, work roll roughness is required to draw the work piece into the roll bite and for lubricant distribution. The grooves on the work roll are responsible for producing ridges, the depth and width of these grooves would therefore determine the height and width of the rolling ridges on the sample surface. The deeper and wider grooves of the rougher work roll and the existence of rolling ridges on the shingled surface rolled with the rougher work roll would imply a prerequisite critical rolling ridge height, to be plastically deformed to produce shingles. It should be noted that the critical work roll roughness would also be dependent on the amount of the shear deformation, applied load, the diameter of work roll, the work roll material, and the alloy being rolled. The size and distribution of the grooves on the work roll surface would therefore determine the size and distribution of the micro-wedges induced on the rolled aluminum surface. Hence, the frequency, size and distribution of shingles could also be related to the work roll topography. Considering that the formation of shingles is a function of the work roll roughness, and that the work roll roughness can be related to the depth of the near-surface damage, it is feasible that the occurrence of shingles on the alloy surface would not just be an indication of the existence of a near-surface deformed layer. Shingle size and frequency could also be an indication of the depth of the deformed layer. However, other parameters such as, rolling aspect ratio, applied load, rolling schedule as well as the diameter of the work roll could also influence the size, frequency, and distribution of the shingles.
Cross-sectional analyses revealed that the near-surface microstructures of the hot rolled Al-Mg samples after ten passes were composed of an oxide layer, rich in magnesium, and transverse micro-cracks (Figs. 7.12 and 7.14). A thicker oxide layer was observed at the near-surface region of the sample rolled with the rougher work roll, which was not uniformly distributed in comparison to the oxide layer on the 0.1 µm R<sub>a</sub> rolled sample (Fig. 7.12). The size of the rolling ridges also contributed to the non-uniform distribution of the oxide layer, as thicker oxide layers were noted to surround higher ridges, which were dominantly found on the sample rolled with the rougher work roll (Fig. 7.12). In addition, transverse micro-cracks propagating into the subsurface of the alloys were related to work roll roughness. These cracks were deeper and wider within the near-surface region of the sample rolled with the rougher work roll (Fig. 7.14).

This analysis thus indicated that the roughness of the work roll also has an effect on the oxide distribution and near-surface damage depth. This agrees with previous work by Gali et al. [16], who observed deeper near-surface damage induced by the formation of larger micro-cracks when using a rough roll (5.7 µm R<sub>a</sub>), as compared to a smooth work roll (0.01 µm R<sub>a</sub>). Comparing previously reported results by Gali et al. [16] with those obtained here, a trend of increasing near-surface damage (i.e., the maximum depth of micro-cracks observed) with an increase in work roll roughness is observed. The near-surface damage, in the form of micro-cracks, extended to maximum depths of 1.5 µm for a 0.01 µm R<sub>a</sub> work roll [16], 2.8 µm for the 0.1 µm R<sub>a</sub> work roll (Fig. 14b), 3.2 µm for the 1.1 µm R<sub>a</sub> work roll (Fig. 14a), and 8.6 µm for a 5.7 µm R<sub>a</sub> work roll [16]. Combining data from the current study and the previous work [16], Figure 7.16 depicts a plot of the different work
roll surface roughness in relation to the average depth of the near-surface damage due to crack propagation observed on the Al-Mg alloy samples after a ten pass hot rolling schedule. The average depth of near-surface damage was derived from measurements of micro and nano-crack propagation depths measured within several near-surface regions on a 6 cm by 1 cm area of the samples. This represents damage due to crack propagation within the near-surface region. The graph shows a linear increase in the average near-surface damage with an increase in the work roll roughness (the error bars depict the variation in crack depths measured for each work roll roughness). This relationship implies that the depth of the near-surface damage could be approximated based on the knowledge of the work roll roughness. It should be noted that this comparison would only be practical under similar rolling conditions. While extensive testing examining the depths of the near-surface damage and its relationship to the work roll roughness might not be possible on an industrial scale, computer simulations may be considered as a convenient alternative approach. The increase in the size of the error bars also displays a correlation with the increase in work roll roughness values, depicting the increase in the variation of measured crack depths with increase of the work roll surface roughness. The variance of the sizes and depths of cracks within the near-surface region which is influenced by the surface roughness of the work roll is an indication of the non-uniform near-surface damage on the rolled aluminum alloy as a result of the work roll roughness. The same non-uniformity was observed with the oxide distribution on the rolled aluminum surfaces.

EDS maps of the surface rolled with the rougher work roll (Fig. 7.11a) showed areas close to shingles were devoid of magnesium enrichment, a phenomenon that was
not observed on the surface rolled with the smoother work roll (Fig. 7.11b). Cross-
sectional analyses (Fig. 7.15a) also confirmed oxide-free areas close to the shingle. This
could be due to oxide redistribution which would be plausibly aided by the use of the
rougher work roll. Oxide redistribution would occur during shingle formation, which has
been described earlier. This is supported by the oxide free areas observed to be associated
with grooves detected at the tail of the shingles. Therefore, the work roll roughness limit
can also contribute to the redistribution of oxides across the surface.

It can, therefore, be summarized that the evolution of near-surface layers on Al-
Mg alloys is influenced by the topography (i.e., surface roughness) of the work roll, with
the depth of near-surface damage increasing as the work roll roughness increases. In
addition, the occurrence of shingles on the surface of rolled Al-Mg alloys can be related
to work roll roughness, with shingles appearing on the rolled surfaces when the surface
roughness of the work roll was greater than 1.1 µm. A critical work roll roughness was
suggested as a prerequisite for the initiation of shingle appearance on the alloy surface,
but this would also depend on the aluminum alloy, applied load, and the diameter of the
work roll. Rolling marks, transverse micro-cracks, oxide layers, and near-surface damage
induced by nano-crack propagation are features induced by shear stress and strains during
hot rolling while the extent of their propagation is influenced by the work roll
topography. Therefore, the computer modeling of the work roll topography could greatly
influence surface quality and the near-surface microstructure induced on the rolled
aluminum alloys.
7.5 CONCLUSIONS

The surface/near-surface features formed on an Al-Mg alloy during a ten pass hot rolling schedule using work rolls with surface roughness ($R_a$) values of 0.1 µm and 1.1 µm were examined. A comparison of both hot rolled Al-Mg surfaces was made, and the following conclusions were drawn:

1. It was observed that the formation of shingles on the rolled aluminum surface was directly related to the work roll roughness. Rolling marks and shingles were observed on samples rolled with a 1.1 µm $R_a$ work roll, while rolling marks and
grain boundaries were prominent surface features on the 0.1 \( \mu \text{m R}_a \) rolled sample. Although surface morphology of the rolled surfaces is a reflection of the surface morphology of the work roll, it is possible to obtain a shingle-free surface with a ground work roll.

2. A critical work roll roughness was proposed to be requisite for the initiation of shingle formation on the aluminum alloy surface. It was shown that the critical work roll roughness would be related to the depth and width of the grooves on the work roll surface.

3. The near-surface damage in the form of micro-cracks extended deeper into the subsurface region for the sample rolled with the rougher work roll compared to that rolled with the smoother work roll. A thicker surface oxide layer was also observed covering the sample rolled with the rougher work roll.

4. The oxide layer distribution in the transverse direction was noted to be related to the size (height and width) of rolling ridges. Larger ridges were observed on the sample rolled with the rougher work roll.

5. The extent of near-surface damage (i.e., depth of micro-cracks and thickness of oxide layers) was related to the surface roughness of the work roll.

**BIBLIOGRAPHY**


CHAPTER 8
THE TRIBOLOGICAL BEHAVIOR OF PVD COATED WORK ROLL SURFACES DURING ROLLING OF ALUMINUM

8.1 INTRODUCTION

The tribological aspect of the hot rolling of aluminum alloys has been the focus of research for several decades because of the complex interactions that occur between the roll and the alloy surfaces. The tribological system involves many parameters, such as; the work roll and its surface conditions, lubricant composition and conditions, work piece material and specifications, the rolling process parameters and the tribological conditions at the roll/work piece interface [1,2]. The tribological interactions that occur between the work roll and the work piece, which are both covered by their individual oxides, are influenced by the thermo-physical and mechanical properties of the roll and work piece, such as yield strength, bulk hardness, shear modulus and density [1,3,4]. These properties thus impact the transfer of heat and stresses at the roll/work piece interface, which come into play in determining the surface quality of the final product [1,2,4].

Asperity contact between the surfaces of the roll and the work piece under lubricated conditions is a result of the high shear stresses experienced during hot rolling [2,5]. As such, the frictional condition between the roll and the work piece is of particular interest; in fact friction is considered one of the main parameters influencing the rolling process [2,5-7]. Consequently, it is generally accepted that the measurement of the coefficient of friction (COF) and the effects of the tribological factors influencing the
rolling process are vital to the evaluation of the tribological behavior at the interface between the roll and the work piece [2,8]. The friction in rolling involves several material and processing parameters, which include; the lubrication condition, work roll roughness, roll speed, normal load, temperature, forward speed and reduction [2,9]. In addition, friction has been reported to be affected by the oxide layers on aluminum work piece surfaces and to increase along the length of contact and with larger reduction of the work piece [1,5,9]. However, friction is one of the main compressive forces responsible for the motion and thickness reduction of the work piece between the rolls [5].

Friction is also affected by the transfer of aluminum to the work roll surface. This transfer from the work piece induces a coating on the roll surfaces and occurs regardless of the roll topography and the applied load [2,3]. However, aluminum transfer increases with roll roughness and can impede high production rates through galling and may reduce the surface quality of the finished product [3,10]. The increase in rolling force is also known to influence the occurrence of friction pick-up [8]. The thickness of the transferred aluminum coating to the roll is determined by the size of the oxide fragments covering the work piece surfaces, which must be very small [3]. The roll coating could also reduce the COF, acting as a barrier for further aluminum transfer by weakening the adhesion tendency of aluminum to roll surfaces during subsequent rolling passes [11]. It is therefore argued that the roll coating may positively affect the work roll performance [12]. The roll coating has a large impact on the effect the surface morphology of the roll has on the surface evolution of the rolled material [2]. Lubricants have been used to reduce aluminum adhesion to work roll surfaces, friction as well as wear of the work roll [3,8,10,13].
Work roll wear is influenced by friction, load, sliding length and the presence of abrasive and corrosive particles in the lubricant [1,14]. It is important to note that although high friction can cause deterioration to the work roll topography, to the point that rolls need to be re-ground to restore the initial finish, low friction can also be detrimental, as this may cause slippage and excessive roll wear [1,12]. Wear of the rolls can occur uniformly on the contact surface or locally in deep wear bands [15]. This type of roll wear is due to the interactions between oxidation, friction and thermal fatigue, with damage mechanisms being attributed to abrasion (exerted by the oxide scale of the work piece), adhesion, thermal fatigue and oxidation [12,14,16]. Uniform wear has been attributed to abrasion and thermal fatigue causing micro-chipping and crack formation perpendicular to the roll surface [14,17]. Thermal fatigue caused cracks appear during the early stages of rolling and can trigger and develop other wear mechanisms [17]. Therefore, it can be said that the roll life is shortened by high temperature and high pressures at contact with the work piece, thus materials used in work roll manufacture require good thermal, mechanical and tribological properties [1,12,15,17].

Coatings can be used to mitigate adhesion and reduce friction, rolling force and roll wear [3,8,10]. Diamond-like-carbon (DLC) coatings have been identified as promising coatings with a high tendency for reducing aluminum adhesion [10]. The properties of DLC coatings are dependent on the coatings' bonding structure and hydrogen content. For example, the low friction of non-hydrogenated DLC (NH-DLC) coatings is attributed to the presence of water in the atmosphere [10,18]. Water dissociation to H and OH passivates the carbon bonds on the surface, allowing for friction reduction and adhesion mitigation [18]. On the other hand, interest in metal
nitride coatings is growing rapidly because of the high wear resistance attributed to them [19]. TiN coatings are recognized for their tendency of low adhesion to molten aluminum [10]. The incorporation of aluminum into TiN improves the oxidation behavior and thermal stability of the coating, enhancing the oxidation resistance of the die [19]. However, the deposition of coatings on a die surface does not guarantee improved friction and wear resistance; the coating topography or morphology has a strong influence on this. A high surface quality of the coating is required to modify the tribological interactions between the coating and the work piece [20].

Hence, because of the complex tribological conditions that occur during hot rolling, the evaluation of tribological properties of the roll materials is vital to any rolling operation. The on-line assessment of these properties is quite difficult [12]. The measurements of rolling parameters (e.g. friction) are affected by rolling conditions, which are constantly changing with each pass and for each mill [4,15]. Temperature also varies during a rolling pass due to the plastic deformation which increases the temperature and contact with the roll and emulsion, which reduces temperature [15]. Laboratory simulation of hot rolling conditions remains a hard challenge for tribologists, with test configurations such as disc on disc, pin on disc and ball on disc, being employed to reproduce specific aspects of these complex tribo-systems [1,21]. Friction measurements have included calculations using parameters such as torque and forward slip, as friction decreases with increasing roll speed [5,8]. Friction has also been estimated from the reduction or spread, the angle of contact (though inaccurately) and matching measured and calculated roll forces (Hill's formula) [5,22].
In this work, several types of PVD coatings (i.e. NH-DLC, Cr, CrN, TiN, TiCN and TiAlN) are examined to determine the possibility of the application of these coatings in extending the life time of work rolls in the hot rolling process. Adhesion and friction were used as the main criteria for the evaluation of the coatings in comparison to the uncoated steel roll. Tests were carried out first, under dry conditions at room temperature and then lubricated tests at high temperature were performed to simulate cold and hot rolling conditions using a tribo-simulator.

8.2 EXPERIMENTAL PROCEDURE

The tribological interactions in rolling were simulated using a custom designed tribo-simulator, the operational principles of which have been described in detail elsewhere [11]. It possesses a roll-on-block configuration and allows for the altering of rolling parameters independently, including temperature, rolling load, lubrication and roll surface conditions, as the roll can easily be removed and replaced. Rolling experiments were carried out by a revolving roll running across the surface of an aluminum block. The configuration allows for the measurement of the torque (G) and rolling force (P) during each rolling pass. The COF is then calculated from the torque, rolling force and the radius of the roll (R) as $\mu = G/PR$ [8].

Rolls were machined to a diameter of 25.5 mm from M2 steel, with composition 1.0 wt.% C, 4.15 wt.% Cr, 81.5 wt.% Fe, 5.0 wt.% Mo, 6.4 wt.% W, and 1.95 wt.% V. Rolling tests were conducted using coated and uncoated rolls on AA1100 samples. The PVD coatings included NH-DLC, Cr, CrN, TiN, TiCN and TiAlN. The hardness of these
coatings has been reported to be 12.8 GPa, 15.3 GPa, 21.5 GPa, 13.9GPa, 21.2 GPa, and 35.0 GPa, respectively [10, 23-26].

The AA1100 blocks were machined to dimensions of 10 mm thickness, 30 mm height and 95 mm width (the 10 mm × 95 mm face being the rolled surface). All of the rolls and the AA1100 blocks were polished with a 1 µm diamond paste and ultrasonically cleaned in acetone before every test. This was done to eliminate the effect of surface roughness during hot rolling. Lubrication was provided by an oil-in-water emulsion with a 4 % (v/v) concentration. The rolling simulations were carried out for 1 pass, at rolling temperatures of 25 °C and 450 °C individually, and with a 9 % forward slip.

The specimen contact surfaces were then examined using a FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. The subsurface features of transferred aluminum on the coatings were also examined, using a ZEISS NVision 40 Cross Beam workstation focused ion beam (FIB), with a gallium ion beam operated at low beam currents at an operating voltage of 30 kV. The surface was protected by the deposition of a thin layer of carbon. Cross-sectional trenches were ion milled using the FIB H-bar method. The samples prepared by using the lift-out method were examined using a JEOL 2010F transmission electron microscope/scanning transmission electron microscope (TEM/STEM).
8.3 EXPERIMENTAL RESULTS

8.3.1 Coefficient of friction (COF) of Coatings

Initial dry rolling tests were conducted at room temperature in order to examine the performance of the coatings under this condition. TiAlN displayed the highest COF (0.87 ± 0.01), while DLC had the lowest COF (0.47 ± 0.01). The uncoated roll performed better than most of the coatings with the second lowest COF (0.54 ± 0.07). A graph displaying the COF recorded for all the coated and the uncoated rolls under this condition is presented in Figure 8.1a.

Figure 8.1b displays the COF recorded under lubricated hot rolling conditions. As stated earlier, the COF values were calculated from the mean torque and rolling force values. Tests were repeated thrice and the mean COF values were calculated and plotted. The TiAlN displayed the lowest COF (0.35 ± 0.01), while the DLC coating had the highest COF (0.55 ± 0.06). The uncoated roll was recorded as having the fifth highest COF (0.45 ± 0.06), with only the DLC and Cr (0.55 ± 0.06) coatings displaying higher COF values. All the nitride coatings had a lower COF in comparison with the uncoated M2 steel roll.

As indicated earlier, higher reduction and spread are related to higher friction. Thus these parameters can be used as a measure of friction. The spread on the aluminum samples were measured and are compared in Figure 8.2. The performance of the rolls based on spread and that based on the COF were mostly coincident, with minor variations. The Cr and DLC coatings had the highest spread of 29.88 ± 0.11 % and 28.52 ± 0.11 % respectively and the TiAlN coating had the lowest spread of 21.22 ± 0.11 %.
the cases of CrN and TiN, the COF and spread values did not occur in the same order, but these small discrepancies were found to be negligible.
Figure 8.1. Average COF values for coatings during (a) dry cold rolling and (b) lubricated hot rolling of AA1100 at 450°C
Wear of Coated and Uncoated Rolls

After dry cold rolling tests, aluminum transfer to the work rolls could be observed with the naked eye. The most severe aluminum transfer was observed for the Cr-based coatings and uncoated rolls. The Ti-based coatings displayed better adhesion mitigation with TiN presenting the least aluminum transfer of these coatings. The DLC coating showed no aluminum transfer on its surface, thus proving to have the highest adhesion mitigation performance among the tested coatings under this condition.

Aluminum transfer to the rolls under the lubricated hot rolling condition was not as distinctive. Examination by SEM revealed no wear damage observed in the form of
micro-chipping or cracking on any of the work roll surfaces. Adhesion was still difficult to observe on the work roll surfaces even under the SEM, but transfer material was randomly dispersed across the rolls' surfaces. The SEM images of all the work roll surfaces are presented in Figure 8.3. Of all these surfaces aluminum transfer was distinctly observed on only the uncoated and Cr-based coated rolls (Fig 8.3a, e, f). Energy dispersive spectroscopy (EDS) mapping was carried out next, to determine the extent of aluminum transfer to the roll surfaces. The maps for aluminum, oxygen and the main element of the coating presented in Figure 8.4 are taken from the same areas displayed in the corresponding SEM images in Figure 8.3. The maps display aluminum transfer to all rolls' surfaces after one hot rolling pass. All roll surfaces except the Cr-based coated rolls were free from a significant amount of oxygen. While little oxygen was detected on the CrN coated surface (Fig. 8.4e), the Cr coated surface (Fig. 8.4f) possessed higher amount of oxygen. The amount of aluminum transfer was quantified using an image analyzer. The surface area fraction covered with aluminum for each coating was plotted in Figure 8.5. Aluminum transfer to the TiAlN coating was difficult to evaluate due to the aluminum concentration in the coating itself. Analysis of aluminum transfer to this coating was performed with a combination of backscattered electron (BSE) images, EDS spot analysis along with the EDS maps (Fig 8.4b). Areas where aluminum transfer was observed on the TiAlN coated roll are displayed in Figure 8.4b.

TiAlN coating displayed the highest surface area fraction of aluminum transfer (0.98 ± 0.008 %) within the margin of error allowed during its calculation. The Cr coating had the next largest surface area fraction of aluminum transfer (0.74 ± 0.04 %), while both the uncoated (0.19 ± 0.001 %) and the DLC coated (0.12 ± 0.001 %) rolls had
the smallest surface area fraction covered in aluminum transfer. The CrN (0.2 ± 0.006 %) and TiN (0.28 ± 0.01 %) coatings also displayed a small surface area fraction of transferred aluminum. Thus, the DLC and TiN coatings exhibited adhesion mitigation behavior under dry cold rolling and lubricated hot rolling testing conditions, while the Cr coating was not as effective under both conditions.

The surfaces of the rolled AA1100 alloys were also examined. They all displayed similar features, such as evidence of grain boundary sliding, with no significant difference in their morphology, regardless of the work roll coating that was used to roll them.
Figure 8.3. SEM images displaying wear damage and aluminum transfer to (a) uncoated, (b) TiAlN, (c) TiN, (d) TiCN, (e) CrN, (f) Cr, and (g) DLC coated rolls after hot rolling AA1100 blocks at 450°C. The rolling direction for all images is from left to right.
Figure 8.4. EDS maps displaying aluminum transfer to (a) uncoated, (b) TiAlN, (c) TiN, (d) TiCN, (e) CrN, (f) Cr, and (g) DLC rolls after hot rolling AA1100 blocks at 450°C. Blue represents aluminum, cyan represents oxygen and magenta represents the main element.
8.3.3 Microstructural Analysis of Coatings

Subsurface damage features were studied using FIB-milled cross sections made along the rolling direction from locations where aluminum transfer to the coated roll surfaces’ was observed. The cross-sectional view of transferred aluminum adhered to the TiN coating is displayed in Figure 8.6a. It shows partial contact with narrow interfacial gaps between the aluminum transfer and the coating indicating adhesion of the aluminum to the coating (at least in two areas). The aluminum surface in contact with the coating appeared smooth and no damage to the coating was observed. The aluminum transfer to
the TiCN coating was loosely adhered to the coating surface evident from the gap at the interface (Fig. 8.6b). The aluminum transfer was connected to the coating at an edge. The interfacial surface of the aluminum was slightly roughened and the coating showed no evidence of damage. The cross-sectional view of the transferred aluminum to the TiAlN coating was of particular interest. The coating was oxidized with the aluminum transfer adhered to the oxide surface. There was also delamination observed, occurring by the cracking of the oxide layer in the vicinity of the aluminum–oxide interface (Fig. 8.6c). The oxidation of the TiAIN coating was evident in the form of an oxide-rich surface layer. This layer was observed to be delaminating at several regions including below the aluminum transfer. Oxidation of this layer was confirmed by EDS analysis which identified the elements Al and O.

The DLC coating was also free of damage beneath the aluminum transfer (Fig. 8.6d). The aluminum transfer appeared loosely adhered to the coating with a distinct spacing at the interface. The transferred aluminum at the interface had a smooth appearance. Thin fibers were observed at the interface bridging the aluminum to the DLC coating.

Aluminum adhesion to the Cr coating occurred at several points at the Cr-aluminum interface (Fig. 8.6e). Gaps observed between the coating and the adhered aluminum could still be observed, giving a rough appearance to the aluminum surface at the interface. There was no damage to this coating observed. Figure 8.6f displays the cross-sectional image of the aluminum transfer to the CrN coating, with no damage to the coating at this particular region. The aluminum transfer was not fully adhered to the coating surface with interfacial gaps between the aluminum transfer and the CrN coating.
This was confirmed with TEM analysis, displayed in Figure 8.7a, where the STEM image of the aluminum transfer to the CrN coating is featured. The image clearly depicts the gap at the interface between the aluminum transfer and the CrN coating. A small portion of the aluminum transfer can be seen fully adhered to the CrN coating at the edge of the aluminum transfer. The elemental composition of this area is confirmed with the EDS map displayed in Figure 8.7b. The rich iron layer observed at the interface, adhered to the aluminum transfer, most likely occurred during the milling of the sample. High magnification images from this area of the transfer highlights the full adhesion of the aluminum to the CrN coating (Fig 8.8). These images revealed a thin interface about 50nm thick between the aluminum transfer and the CrN coating (Fig 8.8b). EDS analysis from line scans run across the aluminum transfer to the CrN coating confirms that this interface is situated at an overlap between the chromium and aluminum peaks. Figure 8.9 displays the EDS analysis from the interface, with aluminum and chromium peaks.
Figure 8.6. Cross-sectional SEM images displaying aluminum transfer to (a) TiN (b) TiCN (c) TiAlN (d) DLC (e) Cr (f) CrN coated rolls after hot rolling AA1100 block at 450°C.
Figure 8.7. (a) Scanning transmission electron micrograph and (b) EDS map of aluminum transfer to the CrN coated roll after hot rolling of the AA1100 block at 450°C

(a)  
(b)

Figure 8.8. (a) Scanning transmission electron micrograph and (b) transmission electron micrograph of aluminum adhered to the CrN coated rolls after hot rolling AA1100 block at 450°C

(a)  
(b)
8.4 DISCUSSION

The coatings’ performances can be compared in the graph presented in Figure 8.10, which plots the COF of coatings during hot rolling against the surface area fraction of aluminum transfer to the coatings. The graph shows the CrN, DLC and uncoated rolls as having the smallest surface area fraction of transferred aluminum. TiN also possessed a small surface area fraction with transferred aluminum, slightly larger than that on the CrN, DLC and uncoated rolls but much smaller than recorded for Cr and TiCN. The
TiAlN, CrN, TiN and TiCN coatings were also seen to all display COF values less than that obtained for the uncoated roll.

Based on these observations, the CrN coating possessed one of the highest performances under aluminum hot rolling conditions, as evident from its low COF and small surface area fraction of aluminum transfer. This was supported by the FIB cross-section observations, which showed the aluminum transfer to be loosely adhered to the coatings’ surface (Fig 8.6f). TEM analysis indicated that the only point of aluminum adhesion at the edge of the transfer possessed an interface (Fig 8.8b) composed of chromium and aluminum (Fig 8.9). Adhesion of aluminum to the CrN coating at this region was due to this interface. A comparison of the COF values under dry cold rolling and lubricated hot rolling also identified CrN as having one of the lowest COF values under both conditions, although under dry cold rolling it displayed a high adhesion tendency. This would indicate that the CrN coating has an improved performance during lubricated hot rolling.

The uncoated roll also appears to perform well with a slightly smaller surface area fraction of aluminum transfer than the CrN coating, though it exhibits a higher COF during hot rolling. Under dry cold rolling tests, it displayed a low COF but high tendency for adhesion, highlighting how the change in rolling conditions affects the performance of the roll. TiN also appears to perform well during hot rolling, with a low COF, but with higher aluminum transfer than both the uncoated and the CrN coated rolls. It displays a low adhesion tendency during dry cold rolling but a high COF, making it better suited for lubricated hot rolling conditions. The Cr coating appeared to have a low performance based on its high COF and large surface area fraction of aluminum transfer. This could be
linked to the high oxygen content observed on its surface (Fig. 8.4f), though no oxide was observed on examination of its cross-sectional view (Fig. 8.6e). Strong adhesion was observed between the transferred aluminum and the Cr coating with several junction points of the rough aluminum interface in full contact with the coating (Fig. 8.6e). It also displayed the largest adhesion tendency accompanied by high COF during the dry cold rolling tests. It appears to display the one of the lowest performances among the compared coatings during rolling either in the dry (cold rolling) or lubricated condition (hot rolling). In the aluminum industry, steel rolls are sometimes coated with Cr after grinding.

While the TiAlN coating showed the lowest COF, it had evidence of damage in the form of cracks and delamination of the oxide layer at its surface (Fig. 8.6c). It was the only coating that showed such damage. The layer was confirmed to be oxidized from EDS analysis, which identified Al and O peaks within it. The formation of an Al$_2$O$_3$ layer on this coating has been confirmed in literature [27,28]. It is reported that TiAlN is resistant to oxidation due to this Al$_2$O$_3$ layer that forms on its surface, but this oxide layer must be stable and adherent to be protective [27,28]. Figure 8.6c shows that the oxide layer formed during the hot rolling test is mechanically unstable and delaminating. Therefore, the mechanism of the performance must be delamination of the adhered oxide layer with the adhered aluminum. During the cold rolling tests this coating displayed one of the highest COF values but appeared to have one of the lowest tendencies for adhesion. It appeared to constantly perform worse than the TiN coating except in the aspect of COF during hot rolling tests.
The DLC coating displayed the highest COF, but possessed one of the lowest tendencies for aluminum adhesion. This was also supported by the cross sectional view which showed the transferred aluminum loosely adhered to the DLC coating surface (Fig. 8.6d). During dry cold rolling tests it performed better with the lowest COF and tendencies for aluminum adhesion. Adhesion mitigation by NH-DLC coatings has been attributed to its absorption of water vapor in humid environments and the formation of easily sheared carbonaceous transfer layers on the counter-face running against these coatings [18,29,30]. However, DLC coatings in sliding contact with aluminum surfaces experience a ‘running-in period’ of high COF due to the wear and the plastic deformation of the aluminum surface during the formation of the carbonaceous layer. Once this layer has been transferred, the COF drops and a steady state condition is reached. During rolling, the generation of fresh aluminum surfaces is continuous. This is expected to keep the contact conditions between the aluminum work piece and the DLC coated rolls experiencing a high COF, during the continuous transfer of the carbonaceous layer.
8.5 CONCLUSIONS

The application of PVD coatings in the mitigation of roll wear and adhesion during hot rolling of aluminum was examined. Rolls, uncoated or coated with TiN, TiCN, TiAlN, CrN, Cr and DLC were used to cold and hot roll AA1100 under dry and lubricated conditions respectively. A rolling schedule of 1 pass at aluminum temperature of 450 °C was used. Roll surfaces were then examined and the following trends observed:
1. The CrN coating displayed one of the lowest coefficients of friction (COF) and low aluminum adhesion. Additionally, the coating showed no evidence of damage, either on the surface or at the subsurface region during hot rolling.

2. The Cr coating possessed one of the highest COF values and aluminum adhesion tendencies, under both dry cold rolling and lubricated hot rolling conditions, among the coatings but showed no evidence of damage.

3. The TiAlN coating had the lowest COF during hot rolling but an examination of the subsurface region showed a delaminating oxide layer at the contact interface.

4. While the DLC coating displayed the highest COF during hot rolling, it showed the lowest aluminum transfer under both cold and hot rolling conditions. The high friction of this coating is attributed to the plastic deformation of the aluminum alloy and the transfer of a carbonaceous layer to the aluminum surface.

**BIBLIOGRAPHY**


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9.1 INTRODUCTION

The tribological interactions that occur during rolling between the steel work roll surface, the lubricant and the hot aluminum surface are characterized by the formation of an aluminum roll coating on the work roll surface. This roll coating is formed by the buildup of material transfer from the aluminum surface. Material transfer from the workpiece surface to the work roll surface is referred to as pickup, and for aluminum alloys occurs regardless of the roll topography and the applied load [1,2]. The buildup of material transfer, pickup, from the aluminum surface to the work roll increases with work roll roughness and highly influences the morphology of the work roll [1–5]. The buildup of the roll coating is therefore influenced by the surface morphology of the work roll, rolling force and the rate of cooling [1,6]. The thickness of the roll coating however, is thought to be dependent on the size of the oxide fragments covering the work piece surface, the rolling stage and emulsion [1,2,5,7–9]. While little relation has been found between roll coating development, rolling load and coefficient of friction, Budd et al. [10] have related the thickness and distribution of the roll coating on the work roll surface to the emulsion viscosity and additive type, concentration, pairing and the hydrocarbon chain length [7]. Yoshida et al. [7,8,11,12] observed a relation between the roll coating thickness and surface appearance with the oil concentration, particle size, state, composition and preparation method of the emulsion, and the molar ratio of oleic acid to
triethanol amine. Yoshida et al. [11] also observed the buildup of a uniform roll coating with the oleic acid additive. They proposed that the roll coating was caused by the accumulation of aluminum debris sticking to a polymerized lubricant layer formed on the work roll surface due to the oxidation of the hydrocarbon chain at elevated temperatures [11,12]. The thickness of the roll coating would therefore be dependent on the quantity of the lubricant oil adhered to the work roll surface, inferring the important role that emulsions play in roll coating formation and composition [2,3,6,8,11].

The appearance of the roll coating during lubricated rolling has been described as patchy, discontinuous and streaky, irrespective of the work roll surface structure, especially during the early stages of buildup with more continuous coverage observed with an increasing number of passes [8,9,13,14]. Smith et al. [9] have described the initial aluminum pickup to the work roll as appearing as isolated lumps streaked out on the work roll surface. Tripathi's [1] observations of roll coatings noted a difference in the color of the roll coatings at different stages of the rolling process. It was reported in the reversing mill as shiny grey, the tandem mill as dark black, and the cold rolling mills as bluish black. It has been suggested that the color of roll coatings is an indication of the thickness of the coating and its lubricant induced polymeric film. Based on his observations, Tripathi [1] proposed two mechanisms for the formation of roll coatings depending on the speed of rolling. The first mechanism was proposed to occur at low speeds by micromechanical entrapment, i.e. the micromechanical interlocking of plastically deformed metal on the rough profile of the work rolls. The second mechanism was proposed for higher rolling speeds, as the tribo-chemical generation of a polymeric film from the lubricant oxidation adhered to the work rolls, which entrapped wear debris
particles from the rolled aluminum slab and work roll, similar to that proffered by Yoshida et al.’s [11]. Hui et al. [15] have reported that the chemical reaction between the lubricant and the surface of aluminum produces a soap, polymer and absorption film, depending on the lubricant composition. The formation of these films was related to aluminum dissolving in the lubricant during rolling and to the transfer of the rolled aluminum to the work roll surface [15]. Treverton et al. [16] reported the chemisorption of lubricant additives by aluminum surfaces during the hot rolling process. Smith et al. [9] reported the presence of carbon observed within the roll coating. Treverton et al. [4], however, suggested that areas of roll coating were possibly separated from the work roll surface by a relatively featureless film of aluminum metal, which would thus influence the interaction of the roll coating with the work piece during contact. Roll coating composition has been observed, using X-ray photoelectron spectroscopy (XPS), to include aluminum oxide, ($\text{Al}_2\text{O}_3$) and metallic aluminum, the ratio of which appears to depend on lubrication and temperature [7,9,14].

Roll coatings are believed to be linked to the wear of the work rolls during the back transfer of the roll coating to the aluminum workpiece, which form pickup defects or grooves on the aluminum surface [1,9]. Back transfer of aluminum buildup from the work roll is believed to occur when the roll coating is unstable, which can manifest due to thermal and mechanical stress cycling [1,9,13]. Defects in the roll coating are also imprinted on the rolled aluminum alloys and were believed to force oxide particles into the aluminum alloy [4]. Thus, the properties of the roll coating influences the surface quality of the rolled aluminum sheets [8]. Yoshida et al. [7] reported a smooth and fine
rolled aluminum sheet surface when the roll coating thickness and aluminum metal to oxide ratio of the roll coating were small.

In the aluminum rolling industry, however, roll coatings are thought to be beneficial, and the development of uniform, fine roll coatings on fresh work rolls is promoted as an industrial practice, as the refusals of slabs is thought to occur in their absence [1,17,18]. The roll coating is also understood to mitigate against further aluminum adhesion to the work roll surface by weakening the adhesion affinity of aluminum to the steel work roll surface during subsequent rolling passes [19]. However, the performance of the roll coating is dependent on its thickness and uniformity as back transfer from the coating to the rolled aluminum sheet (pickup defects) would occur when the coating is too thick, while a thin coating has been associated with unfavorable and unstable friction conditions [1,18].

Previous works have been based on the metallographic examination of the roll coating and pickup defects developed during the rolling of commercially pure aluminum alloys. Analysis used to determine the structure of the roll coating has been limited to XPS, optical microscopy and scanning electron microscopy (SEM). While preliminary transmission electron microscopy (TEM) of the aluminum pickup on a CrN-coated work roll revealed a nanocrystalline structure, the analysis was performed after only the first pass of hot rolling a commercially pure aluminum alloy and limited discussion was provided [20]. The present study intensively examines the initial buildup of a roll coating developed on a steel work roll during a 20 pass hot rolling schedule of an Al-Mg alloy. The microstructure of the roll coating has been investigated using focus ion beam (FIB) and transmission electron microscopy (TEM).
9.2 EXPERIMENTAL PROCEDURE

Hot rolling experiments were performed using a tribo-simulator with a roll-on-block configuration, the operational principles of which have been described in detail previously [19]. The tribo-simulator was designed to emulate the rolling processing conditions. The work roll was machined from an AISI 440C steel alloy to a diameter of 21 mm. The surface of the work roll was then polished to an average roughness (Ra) of 0.02 µm. Rolling tests were conducted with an Al-Mg alloy with a 4.5 wt% Mg content. The Al-Mg blocks were machined to dimensions of 10 mm width, 30 mm thickness and 95 mm length, and then polished with a 1 µm diamond paste. The work roll and the Al-Mg blocks were then ultrasonically cleaned in acetone before rolling to remove surface contaminants. A rolling schedule of 20 passes with a 7 % forward slip and the rolling direction reversed after each pass was carried out. Rolling began at a temperature of 550 °C for the first two rolling passes, with a 10 °C temperature reduction after every two subsequent passes, so that the temperature at the final rolling pass was 460 °C. Lubrication was provided by an oil-in-water emulsion with a 4% (v/v) concentration.

The specimen surfaces were then examined using a FEI Quanta 200 FEG environmental scanning electron microscope (SEM) under high vacuum. The roll coating microstructure was also examined, using a ZEISS NVision 40 Cross Beam Workstation focused ion beam (FIB), with a gallium ion beam operated at low beam currents and an operating voltage of 30 kV. The surface was protected by the deposition of a thin layer of carbon. Cross-sectional trenches were ion milled using the FIB H-bar method. The samples prepared by using the lift-out method were examined using an FEI Titan 80-300 LB transmission electron microscope (TEM).
9.3 EXPERIMENTAL RESULTS

9.3.1 Surface Analysis of Roll Coating

The roll coating buildup on the work roll surfaces was examined with a SEM after 20 rolling passes. The roll coating observed initiated on the work roll surface was patchy, discontinuous and randomly dispersed (Fig. 9.1a). The non-uniform patches, which represent the initial stages of the buildup of the roll coating, were streaked in the rolling direction, spread over the carbides and surface of the work roll. Examinations at higher magnification revealed isolated, smaller patches of material transfer on the work roll surface that appeared at lower magnification as blotches on the work roll surface. (Fig. 9.1b). In other areas, the roll coatings possessed a wavy surface appearance, while darker material at the edges of the roll coating could be observed lying on the surface of patches of roll coating, appearing in some areas as a network of dark blotches. There were dark expanses detected at the edges of these patches and the streaks of material transfer forming the roll coating. A closer examination of these dark areas at the side of the roll coatings revealed wear debris particles embedded within this dark region, which was suspected to be polymerized lubricant (Fig. 9.1c), as well as cracks within the thicker regions of the roll coating (Fig. 9.1d). The presence of these cracks suggested that the roll coating was unstable at these regions.

Another feature observed imprinted on the work roll surface at lower magnification, was a network of lines in the form of grain boundaries (Fig. 9.2a). The patches of material transfer that made up the roll coating could be seen to be located within these grain boundaries. A comparison of the rolled aluminum surface (Fig. 9.2b)
with the steel work roll surface (Fig. 9.2a) revealed that these imprinted grain boundaries were corresponded with the elevated grain boundaries on the rolled aluminum surface. The grain boundaries distinctly observed on the rolled aluminum surface were rich in magnesium. These elevated grain boundaries on the rolled aluminum surface were possibly imprinted onto the steel work roll surface during the hot rolling schedule.

Energy dispersive spectrometry (EDS) analysis, in the form of mapping, of the work roll surfaces revealed that the roll coating buildup was primarily composed of aluminum, magnesium and oxygen (Fig. 9.3). The carbide particles were observed to be rich in chromium (Fig. 9.3e). The carbon map exposed the dark regions surrounding the roll coating as carbon, confirming the likelihood that these regions were carbon from the polymerized lubricant (Fig. 9.3c). The carbon map also revealed the rich carbon content of the carbides, which were observed to coincide with oxygen. The EDS maps suggest that the carbon concentration of the roll coating was possibly from the lubricant used during the hot rolling tests. The maps confirmed a rich carbon layer overlapping with the aluminum and magnesium maps (Fig 9.3). The aluminum map displayed rich aluminum islands, which were richer in aluminum than within the coating (Fig 9.3a). An overlay of the chromium map with the magnesium map confirms the roll coating covered several carbide particles.
Figure 9.1. SEM images of the roll coating initiated on the surface of the AISI 440C steel work roll after 20 hot rolling passes against the Al-Mg sample displaying (a) roll coating distribution (b) dark expanse surrounding the roll coating (c) the debris caught within the dark expanse (polymerized lubricant) and (d) cracks within the thicker regions of the roll coating.
Figure 9.2. SEM images displaying (a) the imprinted grain boundaries on the surface of the AISI 440C steel work roll and (b) the grain boundaries on the Al-Mg alloy after 20 hot rolling passes.
Figure 9.3. EDS maps displaying (a) aluminum (b) magnesium, (c) carbon, (d) oxygen, (e) chromium and (f) iron observed on the AISI 440C steel work roll after the 20 hot rolling pass schedule against an Al-Mg Alloy.

9.3.2 Subsurface Morphology of Roll Coating

Focus ion beam (FIB) milled cross-sections of the material transfer that made up the roll coating initiated on the steel work roll were prepared and used to examine the coating’s subsurface features. The roll coating was loosely adhered to the work roll
surface in several areas (Fig. 9.4a), while at other locations it was fully adhered to the steel work roll surface (Fig. 9.4b). At locations where the coating was fully adhered, there were rich carbon deposits observed at the interface between the roll coating and the work roll surface (Fig. 9.4b). The regions with loosely adhered roll coating were observed to be porous and several iron-rich nanoparticles were identified within the coating. The roll coating possessed an average thickness of 0.5 ± 0.12 µm and was identified to be comprised of a complex mixture of aluminum, magnesium, iron, chromium, carbon and oxygen. The top layer close to the protective carbon deposit was observed to be richer in aluminum and magnesium than other regions, with almost none of the other roll coating elements detected.

Further analysis revealed thicker regions of the roll coating with cracks propagating from the surface of the coating through to the work roll surface (Fig. 9.4c). The material transfer making up the roll coating was observed to fill cavities of the work roll surface. A close examination of these cavities revealed the roll coating was loosely adhered to the material filling the cavity (Fig. 9.4c). EDS analysis revealed that the darker material within the cavity closer to the delaminating roll coating were rich in aluminum, magnesium, carbon, iron, chromium and oxygen, indicating that these regions were still a part of the roll coating. The bright particle within the darker portion of the roll coating within the cavity was rich in chromium, iron, carbon and oxygen, highlighting that it was most probably a debris particle from the carbides. A fractured carbide particle was also observed at the surface of the work roll. The region below the carbide debris particle was observed to be rich in magnesium, carbon, iron, chromium and oxygen. These regions appeared to be composed of fractured carbide particles mixed with
magnesium from the roll coating. The cavity observed here can be inferred to have been induced by the fracture of a chromium, iron-rich carbide particle, which could have occurred during the rolling schedule.

Figure 9.4. Cross-sectional secondary electron images displaying the roll coating (a) loosely adhered to the work roll surface (b) fully adhered to the work roll surface and (c) within the cavity created by a fractured carbide particle after 20 hot rolling passes against an Al-Mg sample.
9.3.3 Microstructural Characterization of the Roll Coating

Transmission electron microscopy (TEM) of the cross-sections of the material transfer buildup on the work roll surface was used to study the microstructure of the roll coating. The complex microstructure of the roll coating is displayed in the scanning TEM (STEM) image featured in Figure 9.5. The roll coating was comprised of a thin magnesium-rich oxide (MgO) layer lying on top of an aluminum/magnesium transfer layer (Fig. 9.5a). Randomly dispersed iron and chromium-rich nanoparticles were observed embedded within the aluminum/magnesium transfer layer. There were also regions within the roll coating that were rich in aluminum and carbon. The roll coating was observed to be loosely adhered to the iron-rich oxide on the steel work roll surface. However, the steel work roll was not uniformly covered with this iron-rich oxide. There were regions of the work roll observed beneath the roll coating which did not possess this oxide, Figure 9.5b displays one such region. There was extensive damage to the work roll surface in the form of fractured iron particles observed delaminating and protruding from the work roll surface. The fracture of the steel work roll surface induced crevices on its surface, which could trap aluminum/magnesium transfer material. The fractured work roll debris particles were detected beneath the aluminum/magnesium transfer layer, becoming part of the roll coating.

EDS line scans of the roll coating (Fig. 9.6) showed fluctuating peaks for all of the elements, including the magnesium concentration at the top of the roll coating, chromium and iron peaks within the roll coating and the work roll substrate, as well as the aluminum, oxygen and carbon peaks. EDS maps (Fig. 9.7) exposed the distribution of elements within the roll coating, with the EDS map overlay displaying the rich
magnesium layer at the top of the roll coating as well as a small concentration of magnesium within the roll coating. The oxygen map showed the distribution of this element within the roll coating, implying it was extensively oxidized. The roll coating also appeared to possess an even distribution of carbon, while bright areas were primarily rich in aluminum and oxygen. There were also chromium and iron-rich particles observed within the roll coating, some of which also contained aluminum. The EDS overlay comprised of the aluminum, magnesium and iron distribution clearly depicts the iron oxide covering the work roll surface.

High resolution TEM images (HRTEM) of the roll coating (Fig. 9.8) revealed the amorphous nature of the roll coating. Figure 9.8a reveals that the roll coating was composed of an amorphous layer of MgO lying on top of a complex amorphous mixture of aluminum, magnesium, iron, carbon and oxygen adhered to the nanocrystalline iron-rich oxide on the steel work roll surface. Striations were revealed within the porous, amorphous aluminum, magnesium, iron, carbon and oxygen mix. A crater covered with the nanocrystalline iron-rich oxide, which might have previously held a carbide particle that could have been fractured and become part of the iron and carbon-rich roll coating, was observed. High magnification HRTEM images of the loosely adhered porous roll coating (Fig. 9.8b) revealed the striations towards the top of the roll coating. The roll coating here possessed amorphous nanoparticles rich in iron, chromium and oxygen. The microstructure of the roll coating appeared to transition from fully amorphous at the surface to an amorphous/nanocrystalline mixture closer to the nanocrystalline oxide on the work roll surface. The nanocrystalline iron-rich oxide layer on the work roll surface
was approximately 20 nm thick. The roll coating could therefore be described as possessing a very complex composition and microstructure.

Figure 9.5. STEM micrographs displaying (a) the complex structure of the roll coating and (b) fractured debris particles from the AISI 440C steel work roll embedded in the roll coating after 20 hot rolling passes against an Al-Mg alloy.
Figure 9.6. EDS line scans displaying the elemental composition of the roll coating formed on the AISI 440C steel work roll after 20 hot rolling passes against an Al-Mg alloy.
Figure 9.7. EDS maps displaying elemental composition distribution of the roll coating developed on the AISI 440C steel work roll after 20 passes against Al-Mg alloy.
Figure 9.8. HRTEM micrographs displaying (a) the amorphous structure of the roll coating and (b) the amorphous nanoparticles embedded in the roll coating on the AISI 440C steel work roll after 20 hot rolling passes against an Al-Mg alloy.

9.4 DISCUSSION

In the industrial aluminum rolling operations, the uniform, stable roll coating desired is only obtained after several hundred rolling passes. Initial roll coating formation has been described as isolated lumps that are streaked out in the rolling direction after the first pass, and as patchy and discontinuous after several passes [7–9,13], similar to roll coating distribution observed in this work (Fig. 9.1). The roll coating developed in this study, developed after 20 hot rolling passes, could be considered as the initiation of the roll coating buildup. The cracks observed on the surface of the roll coating (Fig. 9.1d) and propagating through the roll coating (Fig. 9.4c) are an indication of the instability of the roll coating at this initial stage. The breakdown of this unstable roll coating can easily result in the back transfer of the roll coating to the workpiece surface, reducing the
surface quality of the rolled product. Roll coating breakdown has also been attributed to the lower rolling temperatures that were experienced during the last few rolling passes during the investigated rolling schedule [21]. The grain boundaries imprinted on the work roll surface (Fig. 9.2a) by the rolled Al-Mg sample (Fig. 9.2b) are an indication of the complex tribological interactions between the work roll and the rolled aluminum workpiece that take place during the severe rolling conditions. It is therefore important to understand the roll coating developed at this rolling stage for a better understanding of its evolution through the rolling process.

As earlier stated, previous works have examined roll coatings using XPS, estimating the thickness of the roll coating, from this analysis, as the point where the iron concentration is identical to or is more intense than the aluminum concentration. Similarly, surface examination of the roll coating developed in this study (Fig. 9.3) revealed it was rich in aluminum, magnesium and carbon. However, cross-sectional TEM analysis (Fig. 9.8) revealed iron nanoparticles embedded within the roll coating, closer to the work roll surface. While it is expected that on subsequent passes the transfer of aluminum/magnesium from the workpiece will continue to buildup, increasing the thickness of the roll coating, these elements would still comprise the base of the roll coating. Thus, the constant transformation of the roll coating microstructure due to the dynamic process of its buildup during hot rolling as previously proposed [22] is confirmed. The iron and chromium-rich oxides observed within the roll coating actually indicates that the work roll material would play an essential role in the formation and composition of the roll coating. The severe tribological interactions that occur between the work roll and the aluminum sample during constantly alternating temperatures can
induce wear as well as thermal and mechanical fatigue on the work roll, damaging the brittle carbide particles which are rich in iron and chromium. The extent of wear and damage is influenced by the size, distribution, amount and type of carbides. Carbides have been reported to act as routes for crack initiation, to allow oxide growth to penetrate into the work roll and to cause spalling, all due to the repetitive stress concentration they experience during hot rolling [23,24]. The spaces created by the fracture of the carbides would become filled with aluminum and magnesium transfer during the rolling process, causing more damage to the carbide particles. Eventually, the fractured debris from the work roll surface becomes embedded in the mixture of aluminum and magnesium of the roll coating (Fig. 9.4c). The fractured work roll surface observed in Figure 5b displays pieces of the work roll breaking off the work roll surface. These work roll debris are also observed within the portion of the roll coating closest to the work roll surface. Aluminum and magnesium transfer could also be observed caught within some of the crevices induced by the fracture of the work roll surface, indicating that some of this fracture could have occurred before adhesion of the rolled Al-Mg to the work roll.

The roll coating possessed a complex layered structure composed of amorphous aluminum and magnesium at the surface with its nanocrystallinity increasing closer to the work roll surface (Fig. 9.8). The striations observed towards the top of the roll coating hints that either its formation is due to layers of adhered material piling up on each other or of the mechanical mixing of the elements occurring during rolling contact. It is interesting to note that while there are areas rich in all the major elements of the work roll and rolled aluminum, there are also isolated areas rich in work roll elements (Fe and Cr) as well as the areas rich in aluminum and magnesium while the top layer is rich in
magnesium (Fig. 9.6). This highlights again the complex layered structure of the roll coating. Mechanical mixing of the roll coating elements would explain the mixture of elements observed in several regions of the roll coating closer to the work roll surface including the work roll debris particles observed mixed with the roll coating as observed in Figure 4c and 8. However, roll coating generation due to the pilling up of adhered material leading to buildup also clarifies the layered microstructure and variation in composition at different regions of the coating. The rich oxygen concentration of the entire roll coating (Fig. 9.6 and 9.7) imply that all of the elements within the roll coating are oxidized. No regions were detected without oxygen concentrations, therefore metallic aluminum was not observed in the roll coating under these rolling conditions, at this stage of rolling, as had been observed by previous researchers [7–10,12,13,25]. This could be related to the rolling conditions employed, including the work roll material and surface roughness as well as the number of rolling passes. The carbon concentration observed through the roll coating (Fig. 9.6 and 9.7), has previously been reported by several individual researchers [8]. The carbon is known to be from the lubricant used during rolling and to form chemisorbed species on the work rolls [16]. Aluminum has been observed to react with the additives of the emulsions (carboxylic acids) used in hot rolling to produce complex aluminum soaps [22,26–28]. These aluminum soaps, in addition to aluminum and aluminum oxide have been reported as being part of the transfer films that comprise the roll coating [16,21,29]. The occurrence of these soaps have been credited with the mitigating of further aluminum adhesion, especially when they have been formed with low molecular compounds by reducing the contact between the rolling surfaces [22,28,30]. It has been proposed that the generation of fresh naked
aluminum surfaces and the contact pressures experienced during a bulk deformation process like metal rolling is required for the formation of these soaps [22,30].

Aluminum debris from the work piece caught within the polymerized chemisorbed layers has been proposed as one of the mechanisms for roll coating formation. While this mechanism is meant to be dominant at higher rolling speeds, debris was observed on the work roll surface trapped in the polymerized carbon surrounding the roll coating (Fig. 9.1c) as well as a portion of the roll coating observed lying on rich carbon deposits at the roll coating/ work roll interface (Fig. 9.4b) [1,12]. Cross-sectional analysis carried out on the polymerized lubricant observed at the edges of the roll coating exposed aluminum-rich debris particles embedded in the carbon layer (Fig. 9.9a), while similar analysis or other areas of the roll coating displayed a carbon-rich layer which was underneath an aluminum and magnesium-rich layer, and had aluminum, iron and chromium-rich debris from the Al-Mg sample and the work roll embedded within it (Fig. 9.9b). Mechanical interlocking of the plastically deformed metal was the more dominant mechanism observed here with debris observed on the fractured work roll surface (Fig. 9.5b and 9.8a) as well as on the iron-rich oxide of the work roll surface (Fig. 9.5a and 9.8b) [1].

It is apparent from studies of the roll coating that it possesses a more complex compositional make-up and microstructure than that expected from basic sliding tests. This is obviously due to the complex and severe tribological interactions that occur at the work roll/ workpiece interface. These interactions during severe shear stresses occur in the presence of lubricant films, contaminants, metallic and non-metallic materials [1]. The roll coating composition induced during hot rolling is a combination of all these
materials, from the material transferred from the workpiece, lubricant and also wear debris from the work roll. Therefore, the work roll material as well as the alloy being rolled are relevant in determining the composition and the structure of the initial roll coating.

Figure 9.9. Cross-sectional secondary electron images displaying (a) aluminium debris embedded in polymerized carbon lying at edge of the roll coating (b) carbon-rich layer of the roll coating sandwiched between wear debris adhered to the AISI 440C steel work roll surface after 20 hot rolling passes against an Al-Mg alloy.

9.5 CONCLUSIONS

The initiation of roll coatings adhered to an AISI 440C steel work roll, with a surface roughness (R_a) of 0.02 µm, during a 20 pass hot rolling schedule of an Al-Mg alloy was investigated. Analysis of the roll coating, under these rolling conditions examined after the 20th pass led to the following observations:

1. The roll coating initiated on the work roll surface possessed a complex layered structure comprised of an amorphous magnesium-rich oxide layer lying on an
amorphous mixture of aluminum, magnesium, carbon and oxygen with amorphous iron and chromium-rich particles from the work roll surface embedded within the coating.

2. The roll coating microstructure and composition observed suggest that the work roll material used during hot rolling and the workpiece alloy being rolled are relevant in determining the composition and microstructure of the roll coating.

3. Tribo-chemical and micromechanical entrapment were the two mechanisms identified to be responsible for the initiation of the roll coating.

**BIBLIOGRAPHY**


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CHAPTER 10

THE EFFECT OF SURFACE CONDITIONS ON THE ELEVATED TEMPERATURE SLIDING CONTACT DEFORMATION OF AA5083 ALLOY

10.1 INTRODUCTION

During rolling of certain aluminum alloys, tribolayers with microstructures and compositions different from the bulk material are generated on their surfaces. The tribolayers influence the materials' performance during subsequent manufacturing steps and hence they have been a topic of interest [1-4]. The microstructures of tribolayers typically consist of a top layer of mixed oxides (MgO, Al₂O₃ and MgAl₂O₄) and an inner layer characterized by metallic ultra-fine grains mixed with nano-crystalline oxide particles [1-5]. A proper understanding of the properties of the tribolayers during superplastic deformation of Al-Mg alloys is important in the production of lightweight components for vehicles. Our previous work studied the deformation behaviour of the tribolayers formed on the surfaces of rolled Al-Mg alloys [5-7]. Superplastic deformation of Al-Mg alloys occurs at temperatures above 150 °C and is controlled by solute drag and grain boundary sliding (GBS) within fine grain microstructures [8-12]. Grain boundary sliding is known to provide a mechanism for strain relief but also results in an increase in surface roughness of the alloy [6-8]. Recent work on AA5083 aluminum alloys whose surfaces were covered with tribolayers reported that the surfaces consisted of nanocrystalline grains of MgO and MgAl₂O₄ when examined after being tested at 450 °C and at a strain rate of $4 \times 10^{-2}$ s⁻¹ [5-7]. At 450 °C and warm forming temperatures of 250 °C to 350 °C, examination of surfaces of AA5083 alloys deformed at a constant strain rate of $4 \times 10^{-2}$ s⁻¹ revealed formation of fibrous structures within the cracks generated in
the tribolayer [7]. The location of the cracks where fibres were generated in the tribolayers coincided with the grains of the underlying bulk material that exhibited GBS [7]. Fibres were stretched to lengths exceeding ten times their diameters [13, 14]. It was proposed that fibre formation was induced during the GBS of near surface grains and promoted by oxidation and the outward diffusion of magnesium during elevated temperature deformation [5-7].

In addition to the studies reviewed above, where the generation of fibres was found to originate from the existing tribolayers, there exist many other reports where similar fibrous structures were observed to occur during tensile tests on the fracture surfaces [11, 15]. These fibrous structures also referred to as ligaments [10], filaments [10, 13] and whiskers [8, 15] occurred parallel to the tensile deformation direction on freshly fractured surfaces of aluminum alloys [8, 11-16]. The lengths of fibres were reported to reach 100 - 200 µm and their widths ranged between 0.05 and 3 µm [8, 13, 17]. The lengths of fibres were observed to generally increase with the test temperature and the amount of deformation while their thickness decreased [5, 7, 10, 13, 14]. Fibres with droplet-like nodes have been reported by several researchers [13, 18, 19]. Coarsening of fibres with an increase in temperature was observed by Chen et al. [13, 16]. Fibre composition has been noted to vary with the concentrations of the solute elements [17]. Cao et al. [10] suggested that a high magnesium concentration was required in aluminum for fibre formation.

Shaw et al. [20, 21] proposed that exposing the samples to high temperatures, the application of low strain rates that promotes superplastic deformation and the presence of oxygen in the environment are among the conditions that would facilitate fibre formation.
Some studies have attributed the formation of fibres to dynamic oxide growth [5, 13, 17, 20, 22]. Blandin et al. [15], based on their TEM observations of fibres with a crystalline structure with different orientations along the fibre, proposed that fibre formation occurred by the re-alignment of ultrafine grains during superplastic deformation. Yin et al. [23] suggested that fibres in magnesium alloys result from flow localization of cavities occurring at grain boundaries due to stress concentration. Proposed mechanisms for fibre formation that are still under consideration by researchers include single crystalline plasticity, viscous flow, superplastic flow in micro-volumes and mechanisms linked with a pre-existing oxide film [12, 17, 19, 24]. Arguments have been made for fibre formation by incipient melting due to the presence of a liquid phase at the grain boundaries and observation of droplet-like nodes on individual filaments [18, 19]. Arguments against this view, point out the fact that fibres can occur at temperatures below the incipient melting temperature [8, 13]. Fibres were observed to hold together the opposing edges of cavities and cracks (crack bridging) [20]. It has therefore been speculated that they prevent cavity growth by providing resistance to their propagation [20, 21].

The role that fibre formation plays during the deformation of Al-Mg alloys still needs to be clarified and the role of fibres in aluminum adhesion to steel dies during forming processes needs to be further studied. Accordingly, this study investigates the fibres generated on the surfaces of AA5083 alloy during elevated temperature deformation at 350 °C – 545 °C (a temperature range that is of interest for warm/hot forming). The emphasis of the work is on the determination of the microstructural and compositional evolution of fibres generated within the existing tribolayers on the AA5083 alloy surface during elevated temperature deformation. The results of high
resolution SEM and TEM observations on fibres were used to discuss the role of the surface conditions of AA5083 on its coefficient of friction (COF) when this alloy is placed in sliding contact with a tool steel counterface.

10.2 EXPERIMENTAL PROCEDURE

Samples of 1.5 m length and 1.8 mm in width were tested under a constant tensile strain rate using a high temperature tribometer at different temperatures. The strips were wire cut from highly cold worked 1.2 mm thick AA5083 (H18 temper) sheets. The operation principles of the tribometer were previously described in [5-7]. The tribometer was designed to simulate the deformation observed during the sheet forming process and consisted of a friction measurement assembly and a loading system capable of applying a constant strain rate to the sample. The loading system consisted of two synchronized linear actuators (a slave and a master) to which the strip was attached and a stainless steel roller which was used to guide the strips. The speed of the slave actuator was synchronized with respect to the master actuator so that when the strip was pulled it was deformed at a constant strain rate and moved in the forward direction at a constant speed. The strip passed over the roller which rotated freely and was heated to the desired temperature using a cartridge heater. To determine the COF, a cylindrical pin (counterface) was brought into contact with the top surface of the strip to simulate die contact during high temperature forming. Each point on the top surface of the strip being deformed at a constant rate made contact with the counterface as the strip moved forward so that a linear wear tract was generated. The COF between the strip and the P20 pin in contact was measured at a normal load of 0.22N.
Samples were deformed at test temperatures ranging from 350 °C to 545 °C at a constant strain rate of $4 \times 10^{-2} \text{s}^{-1}$. Major and minor strains were determined by measuring the changes in the initial diameter of circular grids inscribed on the surface of the strip. The calculated major and minor strains for the strip tested at 450 °C and 545 °C were 0.4 and 0.1 respectively. The AA5083 strips were tested in (i) as-received condition with surfaces covered with a tribolayer formed during previous hot rolling and (ii) with surfaces mechanically polished to a 1 µm diamond finish to remove the tribolayer (5 cm length of the surface, corresponding to the strip length where friction measurements were taken, was polished). The AA5083 alloy had a composition consisting of 4.5% Mg, 0.7% Mn, 0.1% Si, 0.25% Fe, 0.09% Cr, 0.02% Cu and the balance Al (in wt %). The cylindrical pin counterface used in the friction experiments was machined from P20 steel (a typical die material) and had a composition of 0.35% C, 1.90 Cr, 0.83% Mn, 0.04% Ni, 0.70% Si, 0.49% Mo, ≤ 0.01 S and the balance Fe (in wt %). The pin surface was polished to a 1 µm diamond finish and ultrasonically cleaned with acetone.

The cross-sectional FIB/SEM image of the AA5083 alloy deformed at 545 °C shown in Figure 10.1 illustrates the microstructure of the tribolayer. A MgO rich top surface and a mixture of $\gamma^{-}\text{Al}_2\text{O}_3$ and MgAl$_2$O$_3$ as previously determined [1] were the main constituents of the tribolayer. Surface analyses on the AA5083 strips were first performed using an environmental scanning electron microscope, ESEM, (FEI Quanta 200 FEG). Then a high resolution SEM, HR-SEM, (FEI Magellan 400) was used to characterize the critical features of fibres at 50,000 – 350,000 times magnification. Cross-sectional high resolution transmission electron and scanning electron microscopy HR-TEM/STEM analyses were performed using FEI Titan 80-300 LB. The system was fitted
with a mono-chromator and energy loss spectrometer (Gatan 865) for electron dispersive spectrometric quantitative linescans. Cross sectional TEM samples were prepared using the H-bar technique [25] with a ZEISSL NVision 40 Cross Beam workstation, focussed ion beam (FIB) system operated at 30kV for the FIB in-situ lift-out of ion beam milled samples. Details of FIB preparation methods can be found in [5, 25]. The surface of the samples was protected by depositing a thin layer of carbon. Cross sectional TEM samples were perpendicular to the loading direction of the strips.

![Figure 10.1. Cross-sectional FIB/SEM micrograph of tribolayer on AA5083 strip after deformation at 545 °C at a strain rate of $4 \times 10^{-2}$ s$^{-1}$ and a strain of 0.4.](image)

10.3 EXPERIMENTAL RESULTS

10.3.1 Surface Roughness of AA5083 Alloy Induced by Tensile Deformation

Surface roughness ($R_a$) measurements were made after the tests conducted at different temperatures using an optical surface profilometer (WYKO NT1100) and the average $R_a$ values are plotted in Figure 10.2. $R_a$ values for the polished surfaces ranged between $0.33 \pm 0.02 \mu m$ (350 °C) and $0.41 \pm 0.03 \mu m$ (545 °C), slightly increasing with
the temperature. Surface roughness measurements for the as-received AA5083, whose surface was covered with a tribolayer ranged between 0.45 ± 0.04 µm (450 °C) to 0.62 ± 0.05 µm (545 °C). The surface roughness (Rₐ) values (Fig. 10.2) measured for the as-received strips were higher in comparison to the polished strips at all temperatures.

**Figure 10.2.** Surface roughness (Rₐ) vs. temperature plot for the AA5083 alloy with as-received and polished surface conditions after deformation at 4 × 10⁻² s⁻¹ at different temperatures.

### 10.3.2 Coefficient of Friction (COF) of the As-Received and Polished AA5083 Strips

The COF values of the as-received and polished AA5083 strips in contact with P20 pins were measured for the sliding contact experiments run between 350 °C and 545
The average COF obtained from the three tests for each surface condition was calculated and the mean values along with their standard deviations are reported in Figure 10.3.

From the plot it was observed that the COF increased with temperature in a similar way regardless of surface conditions of the AA5083 alloy. The highest COF values were obtained at 545 °C as 2.09 ± 0.24 for the as-received and 2.12 ± 0.17 for the polished surfaces, while the lowest were observed at 350 °C, 0.80 ± 0.51 (as-received) and 0.90 ± 0.10 (polished). The COF of the polished AA5083 surfaces were noted to be generally higher than those recorded for the as-received AA5083 alloys. This difference was more pronounced at 450 °C, while at 350 °C and 545 °C, it was less pronounced. The average COF of the as-received strip at 450 °C was 1.45 ± 0.18 while a COF of 1.67 ± 0.18 was calculated for the polished strip.
10.3.3 Surface Damage Induced by Sliding Contact

10.3.3.1 Material Transfer to P20 Steel Pin

Material transfer that occurred from the worn surface of the AA5083 to the contact surface of the P20 steel pin counterface during sliding was examined with SEM. The morphologies of the material transferred and adhered to the P20 pins run against the as-received AA5083 alloy strips are shown in Figures 10.4. The material transferred to the surface of the pins run against the as-received AA5083 was composed of aluminum, magnesium and oxygen according to EDS analyses. The transferred material was...
distributed discontinuously over an area of the pin extending to lengths of about 400 µm and widths of 88 ± 9 µm at 350 °C (Fig. 10.4 (a)) and 147 ± 12 µm at 450 °C (Fig. 10.4 (b)). At 545 °C (Fig. 10.4 (c)), material transfer was not uniformly distributed with build-up observed at the sides of the sliding track and minute quantities of material transfer spread across wide areas of the pin surface.

Similar morphologies were observed for material transferred to the steel pin run against the polished AA5083 surfaces (Fig. 10.5). Material transfer was adhered however in a localized area, comprising of a circular head and a tail having a semi elliptical appearance. The circular shape of the area (A_o) at the tip of the material transferred to the counterface suggests that this is the location where the initial contact between the AA5083 strip and the P20 pin occurred. The area of material transferred adhered to the pin extended to lengths of 229 ± 18 µm, 364 ± 12 µm and 392 ± 42 µm at 350 °C, 450 °C and 545 °C respectively. The initial area of contact (A_o) is highlighted with a red circle, the size of which was observed to increase with the temperature (Fig. 10.5). The diameter of the initial contact area (A_o) at 350 °C (Fig. 10.5 (a)) was 62 ± 9 µm, while at 450 °C (Fig. 10.5 (b)), it was 88 ± 11 µm and at 545 °C (Fig. 10.5 (c)), it was 116 ± 27 µm. This area is indicative of the adhesive junction formed between the steel and aluminum [26]. Its size is related to the junction strength of the aluminum alloy which was observed to grow larger with increasing temperature.
Figure 10.4. Secondary electron images of material transferred to P20 steel pin from the as-received (with tribolayer) AA5083 aluminum alloy at (a) 350 °C (b) 450 °C (c) 545 °C

Figure 10.5. Secondary electron images of material transferred to P20 steel pin from the polished AA5083 aluminum alloy at (a) 350 °C (b) 450 °C (c) 545 °C illustrating the initial contact area ($A_o$).

**10.3.3.2 Sliding Damage on Wear Track of AA5083**

Surface damage to the AA5083 alloy induced on contact with the steel pin was estimated from measurements of the sliding track width on the as-received and polished samples for each test run at temperatures 350 °C, 450 °C and 545 °C. Figure 10.6 displays the variation of sliding track width with temperature and AA5083 surface
condition. Sliding track width was observed to increase with temperature for both polished and as-received surface conditions. The widest sliding track occurred at 545 °C for both polished (42.8 ± 4.9 µm) and as-received (43.0 ± 2.6 µm) and the narrowest at 350 °C, 20.9 ± 1.8 µm and 15.9 ± 2.1 µm for the as-received and polished surfaces respectively. Sliding track of the as-received surfaces at 350 °C was wider, while at 450 °C the polished surface had the wider sliding track. At 545 °C sliding track widths were similar for both surface conditions. Sliding track damage appeared to correlate with the temperature increase as both surface conditions for AA5083 displayed increasing damage in the form of overlaps, grooves and compacted oxide debris due to plastic deformation.

In summary, while the surface roughness ($R_a$) measurements for the polished AA5083 alloy surfaces were lower than those recorded for the as-received (with tribolayer) AA5083 alloy surfaces, the reverse was the case for the recorded COF values. Material transfer to the P20 pin from the as-received AA5083 alloy was discontinuous and possessed a morphology different from that observed on the P20 pin in contact with the polished AA5083 surface, which had a continuous transfer with a morphology that depicted an initial area of contact ($A_o$).
10.3.4 Damage Morphologies of AA5083 due to Tensile Deformation

10.3.4.1 Damage Features of As-Received AA5083 Surfaces

The surface roughness (Ra) results for AA5083 strips reported in Section 10.3.1 prompted an examination of the surface features developed during tensile deformation (without sliding contact). This was done to determine the high temperature deformation induced surface morphologies and microstructures that influence the tribological behaviour. The SEM images in Figures 10.7 (a – c) show typical surface morphologies that were developed during deformation when the AA5083 alloy strips, with surfaces...
covered with tribolayers were tested at 350 °C, 450 °C and 545 °C. The surface offsets observed were due to the steps formed on the surface as a result of the sliding of Al-Mg grains beneath the tribolayer [5, 7]. The height of the surface offsets increased as a result of the temperature increase. Formation of cracks in the tribolayer was observed and the long axes of these cracks extended in a direction normal to the direction of the applied tensile stress. These cracks were the prominent features of the tribolayer damage at temperatures ranging from 350 °C to 450 °C (Fig. 10.7 (a) – (b)) but were less prominent on the tribolayer surface at 545 °C (Fig. 10.7 (c)). Cracks that were generated on the surface of the strip tested at 545 °C appeared to occur preferentially at the grain boundaries. An important observation was the formation of fibrous structures that appeared within the cracks. Fibres were also observed at lower temperatures of 450 °C (Fig 10.7 (b)) and at 350 °C (Fig 10.7 (a)).

Most fibres were observed to extend across the width of the cracks and bridge the edges of these cracks so that the average fibre length was equal to the width of the cracks where they occurred. The average fibre length at 350 °C was 1.15 ± 0.22 µm. At 450 °C fibres extending to average lengths of 2.58 ± 0.67 µm and 545 °C to 3.04 ± 0.59 µm were observed. Fibres had diameters of 85 ± 39 nm at 545 °C. At 350 °C, several fibres that were still attached to each other were observed in the form of a uniform membrane immediately underneath the top surface of the tribolayer (Fig. 10.8 (a)). This could be the first phase of the formation of individual fibres before their separation. Many individual fibres were also distinguished. Individual fibres did not always extend to the full width of the crack; some fibres were ruptured (after necking). Others showed evidence for buckling at 350 °C and 450 °C (Fig. 10.8 (b) and (c)). At 450 °C fibres were also
observed to occur in bundles as well as separated from each other and standing individually (Fig. 10.8 (b)). Fibre bundles at 545 °C occurred at tribolayers stretched over at grain boundary steps, and some were entwined (Fig. 10.8 (c)) as a result of high ductility. In fact the high length-to-diameter ratios of fibres indicate that superplastic deformation occurred during the formation of these fibres.

Higher magnification images in Figure 10.9 (a) revealed that at 350 °C the individual fibres appeared to have been formed by separating from a membrane, consistent with the previous observations noted in Figure 10.8 (a). Fibres at 350 °C also appeared to have a smooth surface with a diameter of 104 ± 39 nm and thus had a high aspect ratio of 11.1. At 450 °C some fibres showed a rougher surface morphology (Fig. 10.9 (b)). Fibres were still observed to form by separating from a membrane but occurred closer to the surface. The fibres had an aspect ratio of 26.8 and appeared to be just underneath a thin surface layer. A larger aspect ratio of 35.8 was calculated for the fibres observed at 545 °C. Fibres had distinctly rougher surface morphology with protrusions giving a nodular surface appearance (Fig. 10.9 (c)).

![Figure 10.7](image_url)

Figure 10.7. Secondary electron images of the as-received AA5083 aluminum alloy strip surface after deformation at (a) 350 °C (b) 450 °C (c) 545 °C. The direction of applied tensile stress is horizontal to these images.
Figure 10.8. Secondary electron images of AA5083 aluminum alloy fibres observed within cracks in strip tribolayer after deformation at (a) 350 °C (b) 450 °C and (c) 545 °C displaying fibre bundles and individual fibres at high temperatures.

Figure 10.9. Secondary electron images of AA5083 aluminum alloy fibres observed within cracks in the tribolayer after deformation at (a) 350 °C (b) 450 °C (c) 545 °C showing changes in fibre surface morphologies from a smooth appearance to a nodular surface morphology as the temperature increased.

10.3.4.2 Damage Features of Polished AA5083 Surfaces

The SEM images of the polished strip surfaces deformed at 350 °C, 450 °C and 545 °C are shown in Figure 10. The surfaces of strips deformed at 350 °C exhibited offsets of Al-Mg grains (Fig. 10.10 (a)). The fine grained structure of the alloy surface could be observed from Figure 10.10 (a) with individual grains being clearly defined. It
should be noted that the motion of the grains did not always occur by individual grain boundary sliding (sliding between two adjacent grains). Surface offsets were observed at regions where groups of grains (4 – 10 grains) were displaced collectively indicating that a cooperative grain boundary sliding (CGBS) mechanism likely took place to accommodate the imposed strain at near surface grains of the alloy. Cavities were observed at the boundaries of grains that slid past each other. AA5083 strips deformed at 450 °C (Fig. 10.10 (b)) also displayed evidence of both individual GBS and CGBS on its surface yet the surface steps and cavities at grain boundaries were more prominent. Oxide fibres that appeared at sliding grain boundaries can also be seen (Fig 10.10 (b)). Cavities generated between the grains were bridged by the oxide fibres. At 545 °C (Fig. 10.10 (c)), surface offsets arising from the grain boundary steps were higher providing evidence for more individual GBS at this temperature.

Examination of oxide fibres by SEM at high magnification revealed that on the surface of the polished strip deformed at 350 °C (Fig. 10.11 (a)) the typical features of the fibres were similar to those generated from the tribolayers. Oxide fibres were harder to locate on the polished AA5083 surfaces in comparison to those on the as-received (with tribolayer) AA5083 surfaces. There were a larger number of fibres that did not extend to the entire width of the crack, as they were either ruptured or buckled (Fig. 10.11 (a)). At 450 °C and 545 °C, fibres were located at the grain boundaries that had experienced sliding (Figs. 10.11 (b) and (c)). Fibres bridging a grain boundary cavity at 450 °C are shown in Figure 10.11 (b). Fibres at these temperatures were extended to lengths of 1.35 ± 0.31 µm at 450 °C and 0.97 ± 0.70 µm at 545 °C. The diameters of fibres were 54 ± 16
nm at 450 °C and 74 ± 19 nm at 545 °C. These fibres were thus superplastic with aspect ratios of 25 at 450 °C and 13 at 545 °C.

Fibres observed on the polished AA5083 surfaces appeared to occur at or very near the surface of the alloy. The surface features of the fibres at higher magnification are shown in Figure 10.12. The smooth surface morphology of fibres formed at 350 °C can be observed in Figure 10.12 (a). Oxide fibres formed at 450 °C (Fig. 10.12 (b)) possessed a rougher surface morphology in comparison to those observed at 350 °C. While at 545 °C (Fig. 10.12 (c)) coarser oxide fibres (evidence for oxidation will be given in section 10.3.6) were commonly observed similar to the nodular surface morphology of fibres detected within the cracks of the tribolayer deformed at the same temperature.

![Figure 10.10. Secondary electron image of polished AA5083 aluminum alloy strip surface after deformation at (a) 350 °C (b) 450 °C. (c) 545 °C](image-url)
Figure 10.11. Secondary electron image of fibres observed on polished AA5083 aluminum alloy after deformation at (a) 350 °C (b) 450 °C (c) 545 °C

Figure 10.12. Secondary electron images of polished AA5083 aluminum alloy deformed at (a) 350 °C (b) 450 °C (c) 545 °C showing structure of oxide fibres at grain boundaries at higher magnification.

10.3.5 HRSEM Analyses of Fibres Formed within Cracks on As-Received AA5083 Surfaces

High resolution SEM (HR-SEM) images were taken in order to elucidate details of the surface morphologies of fibres observed at 350 °C, and at 545 °C. The analyses were performed on the as-received samples. HR-SEM images confirmed the smooth surface texture of fibres formed at 350 °C (Fig. 10.13(a)). The HR-SEM images revealed that these fibres occurred immediately below the top surface of the tribolayer and appeared to originate from a thin continuous membrane shown at the lower end of Figure
10.13 (a). The presence of ruptured fibres, with needle-like appearance at the point of fracture was indicative of localized necking as shown in Figure 10.13 (a). Many other fibres were extended along the whole width of the crack and displayed uniform elongation (Fig. 10.13 (b)). These fibres had diameters of about 115 - 130 nm and reached lengths of 1.30 µm at the widest area of the crack giving a high fibre aspect ratio of about 11.

A HR-SEM image of fibres formed at 545 °C (Fig. 10.14 (a)) revealed the rough surface texture of these fibres. Almost all fibres were found to run the across the width of a crack. This image also shows that these fibres occurred immediately beneath a thin oxide film (of the tribolayer). Examination of individual fibres formed at 545 °C with the HR-SEM (Fig. 10.14 (b)) revealed that the surface texture of fibres was characterized by protrusions on the fibre surface possibly due to oxidation during or after the formation of the fibres. These fibres had lengths of 4.4 µm at the widest area of the crack. Figure 10.14 (b) shows a section of the AA5083 surface where fibres were fractured in a brittle manner (fracture surfaces were perpendicular to fibre axis, without indication of necking). This type of fracture could likely have occurred subsequent to the high temperature deformation, while the alloy was cooling as the oxide would become brittle at low temperatures. Otherwise most fibres formed at this temperature had uniform thicknesses with no evidence of fracture or localized necking.

To understand the origin of the fibres the fibre/tribolayer interface was examined. Figure 10.15 shows a high magnification image of the fibres formed at 350 °C from the tribolayer. The fibres were observed to arise from a region within the tribolayer that possesses a porous granular appearance.
Figure 10.13. High resolution secondary electron image of AA5083 aluminum alloy fibres observed within cracks on strip covered with tribolayer after deformation at 350 °C showing (a) fibre morphology with uniform elongation and some ruptured fibres, (b) localized necking occurring in fibres along with unbroken and uniformly deformed fibres with aspect ratios exceeding 11, some ruptured fibres can also be seen.

Figure 10.14. High resolution secondary electron image of AA5083 aluminum alloy fibres observed within cracks on a strip covered with tribolayer after deformation at 545 °C showing (a) fibres with ‘nodular’ morphology of surface protrusions, (b) nodes on fibres that extended to large aspect ratios. The ‘brittle’ fracture (fracture at the centre) may indicate that the fibres that were deformed superplastically at this temperature were fractured during or after cooling to room temperature.
10.3.6 TEM Characterization of Fibres

The microstructures of the fibres were studied using a cross-sectional TEM and STEM. Figure 10.16 (a) shows the TEM image of the cross-section of a single oxide fibre formed at 545 °C on a nano aluminum grain lying on the tribolayer surface with a distinct grain boundary separating it from the tribolayer. According to the EDS map of this fibre shown in Figure 10.16 (b), the fibre consisted of an aluminum core with low oxygen counts at the centre. EDS linescans (Fig. 10.16(c)) showed the oxygen count increased away from the core and an oxide shell of 10 nm in thickness was formed on the outer surface of the fibre. The ESD map and linescan showed no indication of magnesium within the core. However, the shell of the fibre consisted of high concentrations of
magnesium, depicting an Al-Mg-O layer rich in aluminum oxide towards the core and magnesium oxide on the outside.

A high resolution TEM image taken from the boxed area indicated at the edge of the fibre in Figure 10.16 (a) was examined at greater detail as shown in Figure 10.16 (d). Selected area diffraction patterns identified that the outer shell structure consisted of MgO. The HR-TEM of the fibre also indicated that the MgO shell was comprised of nano-grains which were embedded in an amorphous matrix (Fig. 10.16 (d)). The Selected area diffraction patterns (Fig. 10.16 (e)) taken from the boxed area in Figure 10.16 (d) determined the (222), (220) planes of Al₂O₃ and (311), (440) planes of MgAl₂O₄ spinel. Both Al₂O₃ and MgAl₂O₄ were found to consist of a nanocrystalline structure with grain sizes between 3 and 5 nm (Fig. 10.16 (d)). Similar structures consisting of an Al core and an oxide shell were also observed for oxide fibres generated at 450 °C.

In summary, the fibres formed on the AA5083 alloy consisted of a MgAl₂O₄ interface layer between a MgO rich outer layer and an Al₂O₃ and they had a metallic aluminum core. Previous work suggested that visco-plastic deformation namely diffusional creep and or/Cobble creep [5-7] would occur in fibres with nano-crystalline oxide grains.
Figure 10.16. TEM analysis of fibres formed at 545 °C illustrating (a) TEM image of fibre lying on an aluminum nano-grain (b) EDS map displaying the shell structure of the fibre with blue indicating oxygen, red indicating magnesium and green indicating aluminum (c) EDS linescan displaying element distribution across the fibre with blue indicating oxygen, red indicating magnesium and green indicating aluminum (d) HRTEM image showing MgO, MgAl$_2$O$_4$ and Al$_2$O$_3$ and (e) SAED of area within the box in (d).

10.4 DISCUSSION

10.4.1 Adhesion and Friction Mechanisms during High Temperature Sliding

The AA5083 showed high friction when tested in the temperature range between 350 °C and 545 °C and the COF increased with the temperature. Also the wear of AA5083 and the amount of material transferred to the counterface both increased with the temperature. One of the main factors that should be considered when examining the high COF values is the increase in surface roughness with the temperature. The surface roughness was found to be induced by grain boundary sliding (GBS) as evidenced by the cross-sectional microscopy [6]. As the GBS becomes more prominent with an increase in temperature, the steps that are formed on the surface become more prominent [6]
increasing the COF. A lower COF (Fig. 10.3) was observed for the as-received surfaces in comparison to the polished surface despite having a higher surface roughness (Fig. 10.2). Although the disparity in COF values for the polished and the as-received AA5083 alloys was low at 350 °C and 545 °C, it was higher at 450 °C which is of interest for the hot forming process of this alloy. An additional factor that should be taken in account when interpreting the friction results is the differences in the adhesion behaviour of the two surfaces.

The morphologies of the material adhesion to the P20 pin displayed in Figure 10.4 (as-received strip) and Figure 10.5 (polished strip) are critical in rationalizing the difference in COF values of the polished and tribolayer covered samples. The morphology of the material transferred to the P20 pin from the as-received strip was discontinuously distributed over a wide area, while material adhesion to the pin from the polished strip was localized and continuous. This would suggest that the surface condition would influence material adhesion behaviour and this would in turn affect the COF.

The examination of the adhesion behaviour of the polished surface (with the tribolayer removed) is interesting because the morphology of the adhered area in Figure 10.5 suggests that the contact caused a strong localized adhesion and higher junction strength compared with the surface covered with a tribolayer. The increase in initial contact area ($A_o$) with the temperature (Fig. 10.5) would increase the COF of the polished surface compared with the tribolayer covered surface. This observation implies that the tribolayer will serve to reduce the adhesion and hence the COF. However caution should be exercised in generalizing this argument. The junction strength was shown to decrease
with the percentage of magnesium content of the alloy, as the alloy magnesium content would increase the amount of MgO formed on the alloy surface [26] and MgO can act as a solid lubricant [1-6, 26]. The Mg concentration of the surface is expected to increase with temperature hence the role of diffusion of Mg [26, 27] should be further investigated. It has been previously shown that surface roughening of the tribolayer increases damage to the counterface when placed in sliding contact with AA5083 alloy subjected to plastic deformation [7]. Thus there are complex relationships between the deformation mechanisms that modify the surface morphology (and mechanical properties) and the dynamic changes in surface compositions, which influence the wear and adhesion behaviour of this alloy. Some basic trends can be discussed by considering the following points.

The increase in the COF of the tribolayer covered surface with the temperature is consistent with the increase in material transfer (adhesion) to the pin with more material build-up occurring at high temperatures and also to the surface roughness increase due to GBS.

The morphology of the continuous material transfer associated with the polished strip is indicative of the strong adhesion that occurs within that particular region. This continuous material transfer morphology could be described as a stick-slip type sliding behaviour and as indicated above, when a polished surface comes into sliding contact with the counterface the adhesion forces are higher (Fig. 10.5). It is possible that the tribolayer was richer in MgO and this would cause the lower friction observed for the as-received samples. However the role of the oxide fibres should be taken into account. The
Oxide fibres have been observed to exhibit a visco-plastic behaviour [5, 6], which will be discussed in the next section.

### 10.4.2 Role of Oxide Fibres on the Tribological Behaviour

Previous work has observed that fibre formation occurred in AA5083 within the cracks in the tribolayers that corresponded to locations above the boundaries of the grains of the near-surface subjected to relative sliding (GBS) at temperatures above 300 °C [5-7]. All fibres formed within the tribolayer were observed to be generated from a thin oxide film immediately below the porous top surface (Fig. 10.15). Fibres observed on the polished surfaces appeared to be formed on the surface of the alloy between intergranular cavities that were possibly formed during relative sliding of the grain boundaries (Fig. 10.11 and 10.12). As the grain boundaries provide channels to facilitate Mg diffusion [26] it is possible that these fibres were formed from MgO rich locations where the grain boundaries reach the surface. The fibres were stretched to large elongations as a result of relative shear of adjacent Al-Mg grains [6]. The fibre formation would have been aided by dynamic oxidation repairing broken bonds during applied strain and leading to viscous creep type growth of the oxide fibres [28]. Hence distinction should be made between the superplastic deformation of the bulk alloy that occurs by GBS of Al-Mg grains and local ‘microsuperplasticity’ of the fibres by visco-plastic deformation mechanism.

TEM, SAED analyses and EDS mapping in Figure 10.16 confirmed that the structure of the fibres consisted of an Al$_2$MgO$_4$ shell with an MgO rich outer layer with an Al$_2$O$_3$ rich inner layer and aluminum core. The important point was that the Al core
consisted of nano-crystalline grains while the oxide phases were also nano-crystalline with the MgO outer layer comprising of both nano-crystalline grains and an amorphous structure. Accordingly the observed complex microstructure of the fibres supports the superplastic deformation mechanisms responsible for the large aspect ratios reached by fibres without rupture. It can therefore be proposed that fibres' superplastic behaviour was due to two factors, nano-crystalline grain plasticity and viscous flow of the amorphous regions in agreement with previous microscopic and computational models [5-7, 28]. The plastic deformation behaviour of aluminum nanowires with a core (pure Al) – shell (aluminum oxide) structure was modelled using first principle calculations [28]. It was shown that while the pure aluminum would be subjected to fracture immediately after yielding, the oxide shell would increase its ductility by decreasing the image forces and increasing the density of mobile dislocations. Therefore the oxidation would enhance the plasticity of the pure aluminum. It was also shown that oxygen diffusion to the oxide shell would lead to the healing broken Al-O and Mg-O bonds and thus would facilitate a viscous flow mechanism that leads to fibre superplasticity [28].

The exact role of the fibres during sliding contact is subject to further discussion. The sliding tracks of the as-received strips placed in contact with the counterface while the strips were deformed at 545 °C are shown in Figure 10.17 (a). Clearly, oxide fibres could be observed within the sliding track. The fibres that were generated during deformation at the same strain and strain rate but not subjected to contact are seen on the upper portion of this figure. The upper surfaces of the fibres subjected to sliding contact appeared flattened and exhibited some wear damage as seen in the higher magnification SEM image in Figure 10.17 (b). The flattened appearance of fibres suggests that they
may have taken part in controlling the friction of AA5083 alloy. As the fibres are subjected to visco-deformation it can be argued that they would provide low shear during sliding. The viscous behaviour of the fibre could therefore be a factor in lowering the friction, a point that requires further investigation.

In summary, the surface condition of an AA5083 alloy during elevated temperature sliding contact deformation influences the friction between the alloy and the steel die. The formation of tribolayers on the surface of the alloy during prior deformation processes appears to be an advantage in reducing friction and adhesion. The removal of tribolayers generated during hot rolling from the surface of the AA5083 alloy by polishing the surfaces causes an increase in the friction. This could be attributed to the formation of stronger adhesive forces (higher junction strength) between the polished surface and the counterface. The change in the surface roughness induced by tensile deformation during sliding is another factor that should be taken into consideration. As the surface roughness increases with the temperature because of grain boundary sliding, the COF of the alloy increases with the temperature. The role of MgO in the tribolayers and on the polished surfaces in controlling friction is a topic that requires further attention. The high resolution SEM and TEM studies revealed that the micro-superplasticity of the fibres generated at the contact surface could be one of the key factors in rationalizing the microscopic details of adhesion mechanisms. The oxides with nano-crystalline or amorphous structures likely play a role in friction control. Oxide fibres could contribute to friction reduction as they are low shear constituents due to their viscoplastic flow during deformation. Therefore factors that would promote oxide formation on the surface could be beneficial to achieve low adhesion and friction during
forming of Al-Mg alloys, including conducting warm and hot forming operations in oxygen rich environments.

Figure 10.17. Secondary electron images of (a) oxide fibres located within the sliding track generated on the as-received AA5083 aluminum alloy at 545 °C and also shows the fibres formed on the tribolayer surface outside the sliding track (b) higher magnification image of boxed area in (a) which shows sliding debris lying on the oxide fibres

10.5 CONCLUSIONS

The tribological behaviour of AA5083 Al-Mg alloy with as received surfaces, covered with tribolayers generated during sheet rolling process were examined during tensile deformation at temperatures between 350 °C and 545 °C in comparison with surfaces without tribolayers (removed by polishing). The following conclusions were drawn:
1. The COF of AA5083 increased with the test temperature. Also wear of AA5083 surfaces and the amount of material transferred to the counterface both increased with temperature.

2. Fibres formed during plastic deformation on both polished and as-received surfaces exhibited superplastic deformation reaching lengths at least 11 times longer than their diameters at 450 °C and above. Fibres had a core-shell structure consisting of an outer shell of nanocrystalline or amorphous MgO, Al\textsubscript{2}MgO\textsubscript{4} and Al\textsubscript{2}O\textsubscript{3} and a nanocrystalline aluminum core.

3. Fibres formed on the as-received surfaces propagated from cracks within the tribolayer while those on the polished surfaces were observed to generate at the surface cavities between the grains of the AA5083 alloy.

4. Oxide fibres were observed within sliding tracks of both as-received and polished AA5083 surfaces and their visco-plastic deformation was suggested to contribute to reduce the friction.

The formation of tribolayers on the surface of the alloy during prior hot rolling processes appear beneficial for elevated temperature tribological properties of AA5083 as the polished surfaces show generally higher COF and wear, with a tendency to form localized adhesive joints with the counterface. An oxidizing environment that promotes the formation of the nano-crystalline oxides on the alloy surface could further reduce the friction. Further studies are needed to establish the role oxide fibres on tribological properties of Al-Mg alloys under different atmospheres.
BIBLIOGRAPHY


CHAPTER 11

GENERAL SUMMARY AND CONCLUSIONS

The surface appearance and quality of aluminum sheets is dependent on the occurrence of such surface defects like the generic near-surface deformed layer, which is developed during thermomechanical processes. The major objective of this study is to use laboratory scale tribo-simulators to gather an increased understanding of the tribological conditions responsible for the development and evolution of the near-surface microstructure (NSM) features on Al-Mg alloys during hot rolling and subsequent elevated temperature forming processes. Consequently, an examination of the surface and NSM developed on magnesium containing aluminum alloys hot rolled with the tribo-simulator (Chapters 2 - 7) displayed several features: magnesium enriched grain boundaries, micro-cracks, micro-blisters, shingles, fractured intermetallic particles, magnesium-rich surface oxide layers, aluminum nanoparticles, ultrafine grains, and rolled-in oxides. These are all typically associated and found on the rolled aluminum surface and NSM. These features were determined to be influenced either by the stresses and strains experienced during hot rolling, or the surface topography of the work roll. However, the influence of individual rolling parameters on the evolution of these features and the individual mechanisms associated with their development were identified. An investigation into blisters, which have been observed on heat treated and rolled aluminum alloys, showed the influence of lubrication flow rates, the reduction of which led to an increased tendency for lubrication breakdown and aluminum adhesion to the counter surface,
and work roll topography on the aluminum surface during the initial stages of rolling (examined in Chapter 2). Nanocrystalline structures were observed on the aluminum alloy surface from the first rolling pass. A systematic study of the evolution of the near-surface deformed layer (Chapters 3 – 5) revealed that, for aluminum alloys containing magnesium, the diffusion of magnesium to free surfaces and the formation of micro-cracks developed within the near-surface have a vital role in the NSM development. This held true at both 0.8 -1.4 wt.% Mg and at 4.5 wt.% Mg concentrations. In fact, a comparison of both the Al-Mn (Fig. 5.2 (b)) and the Al-Mg (Fig. 6.2(a)) surfaces after a 10 pass hot rolling scheduled using a smooth roll with the tribo-simulator revealed that the major differences were that at this stage of rolling, the surface of the Al-Mg was completely covered with MgO, giving it a darkened surface appearance. Alternatively, the Al-Mn surface possessed MgO islands (referred to as magnesium piles and dark spots in some Chapters of this dissertation). An examination of both alloy surfaces after a 4 pass rolling schedule under a similar temperature regime of 550 °C – 520 °C, revealed that the Al-Mn alloy surface (Fig. 11.1a) possessed few MgO islands, while the Al-Mg alloy surface was covered with MgO islands and possessed darkened magnesium enriched grain boundaries. The magnesium diffusion rates in both alloys would differ because of the difference in the bulk magnesium concentration, although it is also influenced by temperature and the rolling process [1–6]. The surface, near-surface features, and crack morphology of aluminum alloys would therefore be critically influenced by the rate of magnesium diffusion to the free surfaces.
In order to study the effect of the work roll surface topography on the aluminum surfaces, Al-Mg surfaces were examined after a 10 pass rolling schedule against work rolls of various surface roughness while their surface and near-surface features were analysed and revealed to be related to the formation of shingles on the alloy surface and the depth of the near-surface layer (Chapters 6 and 7). Another objective of this dissertation was to characterize the aluminum adhesion to the work roll, which revealed a nanocrystalline structure for metallic aluminum adhered to a Cr-coated work roll after one rolling pass and a complex layered amorphous mixture on an AISI 440C steel work roll after a rolling schedule of 20 passes (Chapters 8 and 9). An examination of the near-surface deformed layer, referred to as a tribolayer in Chapter 10, being plastically deformed during a hot forming operation with another tribo-simulator showed that these layers influenced the COF and the material transfer behaviour during forming, indicating that the NSM might actually have a useful effect during subsequent elevated temperature forming processes. Through this dissertation, the evolution of NSMs were evaluated through tribological experiments performed using two thermomechanical tribo-simulators. Surface and subsurface characterizations were then carried out on the formed samples to examine the microstructural and composition changes that occurred on the aluminum alloys and work roll surfaces. The NSMs development and evolution, as well as the material transfer buildup on forming tools, were monitored using SEM/EDS, cross-sectional SEM/EDS, FIB, and TEM in relation to varying individual forming parameters, such as the number of rolling passes, temperature, work roll surface topography, and strain rate. Therefore during the course of this study the purposed research objectives
stated at the beginning of this dissertation were achieved. The itemized objectives and the achieved results are displayed below:

- To determine the micromechanisms involved in the formation of the surface and subsurface features of the NSM.

Several micro-mechanisms were identified as being responsible for the various features of the NSM which included grain boundary sliding, which was responsible for introducing cracks to the aluminum near-surface; magnesium diffusion to free surfaces which was responsible for the oxide decorated grain boundaries; the plastic deformation of rolling ridges which induced shingles on the alloy surface as well as rolled-in oxides and crack propagation at the alloy subsurface which introduced aluminum particles within the near-surface region.

- To understand the influence of individual rolling parameters on NSM formation.

Individual parameters such as the number of rolling passes were identified to initiate cracks due to grain boundary sliding on the first rolling pass and to induce crack propagation during subsequent rolling passes. The surface roughness of the work roll was also shown to be responsible for shingle formation and to enhance the depth of the near-surface deformed layer.
• The characterization of the microstructure of the roll coating.

The microstructure of the roll coating was determined to depend on the deformation conditions experienced during the rolling process. The roll coating was revealed to possess a complex microstructure with composition depending on the work roll, rolled aluminum alloy and lubricant compositions.

• An examination of the influence of PVD coatings on the work roll on reducing material transfer.

This examination exposed that while PVD coatings were effective at reducing aluminum transfer to the work roll surface, they were unable to totally mitigate against aluminum to the work roll surface. They however showed promise with extending the life of the work rolls.

• Studying the effect of the near-surface microstructure on subsequent forming processes.

Hot forming experiments comparing aluminum surfaces with and without NSMs indicated that the near-surface microstructure contributed to reducing the high friction typically experienced during aluminum hot forming operations. The NSM influenced the morphology of the material transfer to
the die surface, wear of the aluminum surface and the frequency of oxide fibres occurrence within cracks and at grain boundaries on the plastically deformed aluminum alloy surface.

The impact of this research is surmised in the subsequent sections:

11.1 The Influence of Individual Rolling Parameters on the Surface and Near-Surface Microstructure Features

The rolling tribo-simulator, locally designed at the University of Windsor, was used to carry out the research for this dissertation. It is unique in that it permits the variation of individual rolling parameters, which is not easily achieved with industrial or pilot rolling mills. This is of importance to assess the effect of individual rolling parameters on the development of the NSM. The work roll, which can be easily replaced and as such allows for the variation of the surface topography, was polished to a surface roughness ($R_a$) of 0.01 $\mu$m during initial experiments. This was done to eliminate the effect of surface roughness and evaluate the effect of rolling stress and the number of rolling passes on the NSM evolution. The near-surface region of both Al-Mg and Al-Mn alloys was characterized by micro-cracks induced at the grain boundaries that propagated into the subsurface region, fractured intermetallic particles, and an oxide layer that covered the surface of the alloy and lined crack faces. The micro-cracks, grain boundaries, and fractured intermetallic particles could be observed on the surface of the rolled aluminum alloys. In the case of the Al-Mg alloy, some of the micro-cracks were filled with the oxides at the subsurface. The oxides observed at this stage of rolling corresponded to $\text{Al}_2\text{O}_3$, $\text{MgAl}_2\text{O}_4$ and MgO, similar to those constantly identified on the
aluminum alloy surface at this rolling stage \([2,7–11]\). Observations of the near-surface microstructure after the fourth rolling pass revealed increased damage to the near surface with continued crack propagation, and intermetallic particle damage, as well as an increase in the porosity and evolution of the near-surface oxides. Crack propagation had been shown with the Al-Mn alloy to progress with each rolling pass depending on the crack orientation along the grain boundary in relation to the rolling direction. After a 10 pass rolling schedule, the aluminum alloy NSM was comprised of transverse micro-cracks that propagated into the subsurface region, micro-cracks filled with MgO, embedded fractured intermetallic particles, a nanocrystalline grain structure, aluminum nano-particles, and a porous dominant nanocrystalline MgO layer that filled the crack induced damage areas. These features have been identified as typical of the developed NSM \([2,8,12–16]\). The NSM has been reported to continually reduce in thickness during the hot rolling process, in part due to the high level of plastic deformation induced from the reductions of about 50 mm – 75 mm per pass of the as-cast ingot (i.e., about 13% - 18% reduction per pass, depending on the rolling plant and thickness of the cast ingot) to produce sheets of 2 mm and 6 mm thickness, in addition to the distribution or spread of the NSM features from the extension of the aluminum sheet constantly being formed during the rolling process \([2,8,9,14–17]\). However, while the increase in the number of rolling passes might induce a reduction of the thickness of the NSM, as a result of rolling stresses and strains induced at each rolling pass, it distinctly contributes to the increase of near-surface damage induced on the aluminum alloy. A schematic of the evolution of the near-surface region with the increase in the number of rolling passes is displayed in Figure 11.2.
The influence of rolling stresses and strains has been shown to induce the formation of cracks, both micro and nano, within the near-surface region. These stresses and strains also influence the propagation of cracks and therefore the near-surface damage induced on the alloy and the nanocrystalline structure of the NSM due to severe plastic deformation. These features of the NSM were observed on both aluminum alloys regardless of the work roll material or topography, number of passes, or lubrication conditions. Therefore, they can be attributed to the stresses and strains experienced during rolling. The structure of the oxides developed on Al-Mg were also observed to be influenced by this parameter, as dark grain boundaries, which were shown to be a result of MgO enrichment, were only observed on subsequent rolling passes, likely appearing dark due to their nanocrystalline microstructure. An analysis of the effect of surface roughness was performed initially by comparing work rolls of surface roughness (R$_a$) of 0.01 µm and 5.68 µm, which only displayed shingles on the surface of the aluminum alloy rolled with the rougher work roll after a 20 pass rolling schedule. Further experiments under similar conditions, with work rolls of surface roughness (R$_a$) of 0.1 µm and 1.1 µm, again only exposed shingles on the aluminum alloy surface rolled with the rougher work roll after the 1$^{st}$ and 20$^{th}$ passes. These experiments led to the conclusion that the work roll roughness had a distinct influence on the formation of shingles on the surface of aluminium alloys. They also resulted in the inference of a critical work roll roughness for the initiation of shingles that would vary depending on the roll diameter, applied load, aluminum alloy, and rolling temperature. The work roll roughness was also observed to induce increased near-surface damage, with an increase in the depth of near-surface damage in the form of crack depth after 20 hot rolling passes.
observed with an increase in the roll roughness. In addition, the work roll was responsible for inducing the rolling marks and the redistribution of the surface oxides. The effect of the roll microstructure was compared using work rolls with carbides emerging or protruding from their polished surfaces and rolls without this microstructural feature. Carbides emerging from the roll surface have previously been reported to result in faster transfer layer formation and influence the surface quality of rolled sheets [18,19]. Increased aluminum adhesion to carbide surfaces was observed when they were protruded from the roll surface, resulting in the formation of blisters on the rolled aluminum surface. However, these blisters were not present on aluminum surfaces formed with rolls that did not possess these protruding carbides. Lubrication was also observed to contribute to the formation of these blisters and increase aluminum adhesion to the work roll surface as the combination of protruding carbides and low lubrication flow were credited as the parameters responsible for the blister formation. The reduction of lubrication on its own was not observed to induce blister formation, and the occurrence of protruding carbides appeared to be required for the introduction of this surface defect. The rolling parameters and their influence on the NSM features are summarized in Table 11.1.

The near-surface features observed on the aluminum alloys rolled with the rolling tribo-simulator in this work, like the transverse cracks, shingles and blisters are similar to those observed on the industrial rolled aluminum alloys. Although aluminum surfaces rolled with the polished work roll, which was performed to observe the effect of rolling stresses and strains, possessed features like darkened grain boundaries on the alloy surface, not typically observed with industrial rolled products, their subsurface features
represented those observed within the near-surface of rolled aluminum alloys. However, aluminum alloys rolled with ground work rolls using the rolling tribo-simulator mirrored both surface and near-surface features observed on industrially rolled aluminum alloys. In fact, a comparison of the surface features and appearance of the aluminum alloy rolled with the ground work rolls displayed in Figures 7.6 and 7.8 with industrially rolled aluminum alloys examined by Wang et al. [13] shown in Figure 1.7, Liu et al. [14] and Frolish et al. [2] expose that they possess similar surface features and appearance. Similarly, the surface evolution of the rolled aluminum alloy due to rolling passes which induces smoother surfaces as displayed with Al-Mg alloys rolled with the tribo-simulator in Figures 7.6a and 7.8a of this dissertation has been observed with industrially rolled aluminum as examined by Zhou et al. [16]. Zhou et al. examination of the industrial rolled aluminum alloy also displayed the porous nanocrystalline oxide layer observed using FIB and TEM examination in Figures 5.16 6.6, 6.9 of this dissertation. The propagation of cracks within the near-surface region of the rolled aluminum alloys shown through this dissertation have similarly been observed on rolled aluminum alloys by researchers like Fishkis et al. [8] and Tzedaki et al. [12], with Tzedaki et al. [12] similarly observing the nanocrystalline MgO on the surface of cracks within the near-surface layers of industrially rolled aluminum sheets. Finally the nanocrystalline structure of the near-surface shingles and the rolled-in oxides which appear in Figures 6.13 and 7.15 of this dissertation have been noted in Liu et al. [14] examination of the industrially rolled aluminum alloy. Therefore it can be said that the near-surface features and the influence of the rolling parameters observed here display the trends observed during industrial aluminum rolling.
Consequently, this work shows that the variation of rolling parameters such as the work roll roughness, the rolling strains, passes and the lubrication conditions can lead to an improvement of the surface quality of the rolled aluminum alloy and a reduction in the thickness of the NSM. However, the variation of these parameters would also have a distinct impact on productivity. It is clear that a better understanding of other rolling parameters, such as rolling load, forward slip and the ingot surface would also be beneficial to give a full picture of which parameters can be altered to improve the rolled aluminum surface with the least impact on the productivity of the rolling plant. It should also be noted that while several of the results of this work have been applied in the rolling industry they are not discussed due to confidentiality and a conflict of interest.

Figure 11.1 Scanning electron images showing MgO islands and magnesium distribution at grain boundaries on surface or (a) Al-Mn, and (b) Al-Mg alloys after a 4 pass rolling schedule
Figure 11.2 Schematic of the evolution of the near-surface deformed layer of an Al-Mn alloy during hot rolling schedule after (a) 1 pass, (b) 4 passes, and (c) 10 passes.
Table 11.1 Summary Table of the individual rolling parameters and the related near-surface microstructure features

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<thead>
<tr>
<th>Rolling Parameter</th>
<th>Influence on NSM Features</th>
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<td>Rolling Passes</td>
<td>Increased near-surface damage</td>
</tr>
<tr>
<td>Rolling Stresses and Strains</td>
<td>• Crack formation and propagation within the near-surface</td>
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<td></td>
<td>• Nanocrystalline structure of NSM</td>
</tr>
<tr>
<td></td>
<td>• MgO structure</td>
</tr>
<tr>
<td>Lubrication</td>
<td>Increased adhesion</td>
</tr>
<tr>
<td>Work Roll Topography</td>
<td>• Increased adhesion</td>
</tr>
<tr>
<td></td>
<td>• Blister formation</td>
</tr>
<tr>
<td>Work Roll Surface Roughness</td>
<td>• Shingle formation</td>
</tr>
<tr>
<td></td>
<td>• Increase near-surface damage</td>
</tr>
</tbody>
</table>

11.2 The Development and Influence of Roll Coatings

The roll coating structure was examined under two varying conditions, initially on a CrN-coated roll with a diameter of 25.5mm, after one rolling pass against a commercially pure aluminum alloy (AA1100) at 450 °C and a forward slip of 9%. The aluminum transfer to the CrN-coated roll was not fully adhered to the coating surface and was characterized as a predominantly nanocrystalline structure composed of mostly
metallic aluminum with oxides observed at face of the aluminum transfer close to the 
CrN coating. Subsequently, the roll coating developed on an AISI 440C steel work roll 
with a diameter of 21 mm after a 20 pass rolling schedule against an Al-Mg alloy (4.5 
wt.% Mg) at a temperature regime of 550 °C to 460 °C and a forward slip of 7% was 
examined. The AISI 440C steel work roll was used because its polished surface was 
covered with protruding carbides, which increase the formation of a transfer layer [19]. 
The roll coating under this condition differed from that observed on the CrN coating in 
that it possessed a more complex and layered amorphous structure that was comprised of magnesium-rich oxides, an amorphous mixture of aluminum, magnesium, and carbon 
besides iron, and chromium-rich particles. The complex structure of the roll coating 
developed on the AISI 440C steel roll highlighted the relevance of the roll material to the 
microstructure of the roll coating. Its complex structure also underline that mechanical 
mixing of the roll coating elements might occur while noting that the layered structure 
and the striations within the roll coating might be due to a buildup of adhered material. 
The composition of the rolled alloy is also of great influence in the roll coating 
composition. Aluminum and oxygen were observed with the material adhered from the 
commercial purity aluminum while aluminum, magnesium, and oxygen when the coating 
was built from rolling an Al-Mg alloy. 

The difference in the structures of the roll coating could be related to severe 
plastic deformation (SPD), which is one of the mechanisms reported by Zhou et al. to be 
responsible for the nanocrystalline structure of the NSM [20]. Severe plastic deformation 
has also been observed to induce amorphous phases, dissolution of precipitates, and 
thermally activated phases during accumulative roll bonding, cold rolling, cold drawing
of wire, extrusion, high pressure torsion, and equal channel angular pressing [21–24]. The structures induced by severe plastic deformation are influenced by the thermal conductivity, number of shear deformations, strain, and temperature at the interface [25–30]. The mechanisms behind the formation of amorphous structures during severe plastic deformation, described as deformation-induced amorphization, has been related to a critical grain size at which the nanocrystalline phase becomes unstable; deformation-induced plastic flow; intermixing; the interdiffusion of elements, which is promoted by the increase in density of lattice defects; localization of severe deformation within shear bands; and the fragmentation of coarse particles [21,24–27,30,31]. The deformation conditions and parameters, such as the number of enhanced shear deformation passes, temperature regimes, thermal conductivity of the work roll, work roll material, and the temperature at the tribological interface, influence the roll coating structure developed on the work roll at any stage of the rolling process [25,28–31]. The compositional distribution of the roll coating after 20 passes due to mechanical mixing could also be aided by the intermixing and interdiffusion of elements observed during SPD.

These works highlight the need to understand the influence that the work roll material, and rolling parameters have on the microstructural evolution of the roll coating. The structure and composition of the roll coating is of importance during the course of rolling. For example, back transfer of the roll coating to the aluminum alloy would introduce this microstructure into the near-surface. Subsequent research has shown that the delamination of the adhered material on an uncoated M2 work roll, induced under similar rolling conditions observed for the AISI 440C steel roll, would be introduced back unto the rolled Al-Mg surface on successive rolling passes [32]. The pickup defects
on the rolled Al-Mg surface identified in that work occurred as islands of metallic aluminum on the rolled surface covered with MgO [32]. As these roll coating structures are reintroduced into the near-surface deformed layer, they would influence its microstructure, composition, and the distribution of elements in the near-surface. Thus NSMs developed on the aluminum alloy would be transferred to the roll coating, endure deformation until they are unstable, and then would be reintroduced back to the aluminum alloy surface. This could be another reason for the carbon and carbides observed within the NSM as the roll coating observed after 20 passes, showed areas of polymerized lubricant and carbon within the amorphous aluminum and magnesium mixture [9,10,33]. Aluminum debris caught within the polymerized lubricant on the work roll surface and the mechanical interlocking of deformed material indicated that both the tibo-chemical and mechanical entrapment mechanisms were responsible for the formation of the roll coating [34].

This work reveals the complex microstructure and composition of the roll coating and that the composition and structure of the roll coating depend on the composition of the rolled alloy, work roll and lubricant. Industrially little is known of the roll coating outside of work done with commercial purity aluminum alloys. This research project has highlighted that cracks within the roll coating could be an indication of the instability of those regions of the roll coating which could be applied industrially as there is typically no indication as to thickness the roll coating becomes unstable.
11.3 Micro-Mechanisms Responsible for the NSM Features

One of the main objectives of this dissertation is to determine the micro-mechanisms responsible for the formation of the surface and subsurface features of the near-surface deformed layer developed during hot rolling. Several NSM features have been observed and the mechanisms behind these features have been identified. The occurrence of transverse cracks for instance has been related to grain boundary sliding, which occurs during the first rolling pass, during which the sliding of grains within the near-surface grains induces cracks at the grain boundaries. These cracks can be clearly seen when the surface of the work roll and aluminum slab has been polished to a mirror finish. Otherwise, these cracks are covered by the oxide scales or shingles induced on the alloy surface. These cracks are either healed or continue to propagate depending on the orientation of the crack with respect to the rolling direction. It has been observed that after several rolling passes, only transverse cracks are observed on the alloy surface. The healing of cracks has been shown to be one of the mechanisms responsible for introducing oxides into the near-surface region. The introduction of oxide particles within the near-surface region occurs through a two mode mechanism:

1. Crack formation occurs due to grain boundary sliding during the first rolling pass, and magnesium diffusion to the free surfaces created by these cracks results in the faces of cracks to be covered with MgO.

2. The oxide covered cracks are closed due to the compression of the grains during the second pass in the opposite direction.

The formation of MgO on crack faces was also observed to result in the healing of cracks from the first rolling pass on Al-Mg alloys, where magnesium diffusion occurs.
faster, but the closing of the cracks has been observed to occur primarily during the second rolling pass. The filling of these cracks with MgO has been referred to as crack healing through this dissertation. It should be noted that the term ‘crack healing’ does not imply that the effect of the crack has been eliminated or that the crack is fully repaired at this stage; it simply refers essentially to the sealing of the cracks with oxide material (oxidative healing) thereby bridging the contact between the crack faces [35]. Magnesium diffusion to damage induced areas has also resulted in these areas being filled with MgO.

MgO islands observed on the alloy surface after the first rolling pass (Al-Mg alloys) and forth rolling pass (Al-Mn) alloys are related to the diffusion of magnesium to nano-cracks at the alloy surface and around fractured intermetallic particles. The formation of shingles is due to the shear deformation of micro-wedges or rolling ridges formed by the aluminum squeezing into the grooves of the work roll. The deformation or folding over of these rolling ridges is the mechanism responsible for the rolled-in oxides, as the micro-wedges would be covered with oxides during their high temperature formation. Their deformation would introduce an oxide layer between the shingle and the aluminum surface below it, as shown in Fig 6.13 (b).

The mechanism behind the occurrence of aluminum nano-particles has been proposed to be generated by the propagation of cracks within the near-surface, causing the formation of aluminum debris particles. The formation of these debris particles has been observed from near-surface examinations of both the Al-Mg and Al-Mn alloys after 4 hot rolling passes (Fig. 11.3). These particles, which are initially magnesium-rich aluminum nano-particles, experience continued magnesium diffusion to their free surfaces until these particles are metallic and covered with MgO as shown in Figures 5.17
and 6.11. These nano-particles referred to as aluminum nanocrystals had been proposed to occur due to the reduction of Al$_2$O$_3$ by magnesium, resulting in aluminum particles within the magnesium-rich oxides [36,37]. Therefore the occurrence of aluminum nanocrystals during thermomechanical processing would be a result of (i) the continued outward diffusion of magnesium from aluminum debris particles formed from crack propagation and (ii) the magnesium reduction of Al$_2$O$_3$. Blisters observed on the rolled aluminum surface were suggested to be induced by two mechanisms: (i) the delamination of the surface oxide layer after its adhesion to carbides protruding from the roll surface after and (ii) the evolution of hydrogen gas beneath the delaminated porous oxide layer, from the interaction of the subsurface aluminum with water vapor in addition to the magnesium evaporation. Table 11.2 summarizes the NSM features and their micro-mechanisms. In summary the mechanisms responsible for the near-surface evolution include but are not limited to: magnesium diffusion to free surfaces; crack formation due to grain boundary sliding; crack propagation within the near-surface induced by shear stresses; nanocrystalline grain formation due to high shear strains; and the deformation of micro-wedges induced on the aluminum surface by grooves on the roll surface.
Figure 11.3 Cross-sectional secondary electron images of the (a) Al-Mn and (b) Al-Mg alloys displaying the formation of aluminum debris from crack propagation at the near-surface.
Table 11.2 Summary Table of the near-surface microstructure features and their related micro-mechanisms

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<th>Micro-mechanisms</th>
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<td>Grain boundary sliding</td>
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<td>Shingles</td>
<td>Shear deformation of rolling ridges (micro-wedges)</td>
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<tr>
<td>Oxide decorated grain boundaries</td>
<td>Magnesium diffusion to free surfaces created by cracks</td>
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<td>Rolled-in oxides</td>
<td>Deformation of oxide covered rolling ridges during the formation of shingles</td>
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<tr>
<td>Free Al particles</td>
<td>Al debris from ablation from crack propagation within near-surface</td>
</tr>
<tr>
<td>Blisters</td>
<td>Hydrogen evolution and delamination of oxide surface layer due to adhesion to carbides</td>
</tr>
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</table>

11.4 General Conclusions

To acquire an increased understanding of the evolution of the near-surface deformed layer during the tribological interactions occurring during hot rolling and
The results derived indicate that:

1. The near-surface deformed layer is influenced by various individual rolling parameters which induce the various surface and near-surface features that comprise the near-surface deformed layer.

2. The features of the near-surface layer induced during elevated temperature tribological contact are influenced by the diffusion rate of the alloying elements of the aluminum alloy.

3. The microstructure and composition of the material transferred to the mating die surface during elevated temperature forming process is influenced by several parameters ranging from the deformation conditions to the composition of the surfaces at the tribological interface.

4. The near-surface deformed layer induced during hot rolling could prove beneficial for subsequent elevated temperature forming processes where high friction and material transfer is detrimental. This is could be linked to the MgO concentration at the surface of the alloy and the high frequency of oxide fibres formed between cracks and grain boundaries during plastic deformation.

Through this dissertation, research work has been presented from experiments that have been performed under specific rolling conditions to systematically identify the individual rolling parameters and the mechanisms behind the initiation and evolution of NSM features. The formation of the near-surface deformed layer has generally been simply attributed to be a result of the high shear stress experienced by the aluminum
surface during thermomechanical processing. This appears to be an oversimplification of a complex phenomenon. Zhou et al. [16] have observed that the generation of the near-surface deformed layer during rolling is a result of the combined effect of a couple of mechanisms they have identified. This dissertation as well as several other authors have identified several mechanisms involved in the formation of the NSM features [2,5,8,9,12–16,33,36,38,39]. Based on all these works, it is proposed, from a tribological perspective, that the near-surface deformed layers are a result of several micro-mechanisms occurring simultaneously during the tribological interactions that prevail through the rolling process as a result of the various rolling parameters at play. Therefore the varying of individual or a combination of rolling parameters would influence the formation, structure and depth of the near-surface deformed layer.

11.5 Suggestions for Future Work

The evolution of the near-surface microstructure needs to be further studies in relation to the influence of other rolling parameters such as forwards slip, aspect ratio, rolling load, roll diameter and lubrications on the NSM development. An understanding of the influence of further rolling parameters would yield a more in-depth understanding pertaining to which combination of individual rolling parameters to vary to control the formation, depth and features of the near-surface deformed layer and maybe eliminate it. As the rolling parameters would also influence productivity these parameters could be controlled to give a desirable balance between both productivity and near-surface features that would be transferable and useful for subsequent forming processes.
The influence of rolling parameters on the evolution of near-surface microstructures was studied at elevated temperatures. An extension of this work to lower temperature forming processes like cold rolling would be ideal. The evolution of the NSM during cold rolling and as well as the mechanisms at play during the NSM formation during cold rolling would be the ideal next step in the evolution of this project. It is expected that the diffusion of magnesium would not be a huge contributing factor at lower temperatures. The absence of this mechanism during crack formation and propagation would be of great interest in understanding the NSM evolution.

The identification of further mechanism responsible for other features of the NSM and its evolution would greatly contribute to the understanding of the near-surface deformed layer, especially the role it plays during subsequent forming processes. This work could be extended to other aluminum series to understand the influence of the various aluminum alloying elements have on the formation of the NSM. The influence of these alloying elements on the already identified mechanisms would also prove useful in further understanding the NSM evolution.

It is evident from this initial study of the roll coating that further work is required. The extent to which the work roll composition influences the roll coating structure and composition still needs to be investigated further. The influence of the rolled alloy and lubricant composition on the roll coating composition, especially pertaining to the influence of alloying elements for the aluminum alloy and additives for the lubricant. While the influence of additives has been touched in previous research, this did not extend to their influence on the microstructure and carbon distribution within the roll coating. The influence of the deformation conditions is also a research area that should be
paid particular attention and these would determine the microstructure of the roll coating. Subsequent work would also include the influence of the roll coating microstructure on its stability and its performance during rolling.

Future studies should also include an examination of the microstructure and composition of the roll coatings developed during cold rolling. As the diffusion of alloying elements of the aluminum alloy occur at elevated temperatures, this would prove idea in understanding their influence on the formation and microstructure of the roll coating. A comparison of the roll coating with material transfer buildup on other dies and tools applied to elevated and low temperature forming would prove useful to applying the benefits of this coating observed in the rolling industry to other aluminum forming operations.

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APPENDIX A

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Chapter 2 The Formation of Micro-blisters on Al-Mg Alloy Surfaces during Hot Rolling

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Chapter 4 The Influence of Hot Rolling on Oxide Development within Micro-cracks of Aluminum-Magnesium Alloys
INTRODUCTION

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