



University of Leicester

Rapidly Solidified Aluminium-High Silicon Alloys

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To

My brother Turke

My wife Nouf

My sons Nawaf and Mohamed

My daughters Hend and Hnouf

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Faraj D. Alshmri

ABSTRACT

Aluminium silicon alloys are the most used raw material for automotive applications. One of the main limitations on using aluminium *high* silicon alloys is the formation of coarse brittle phases under conventional solidification conditions. However, rapid solidification processing (RS) is very effective in limiting the coarsening of primary silicon due to high cooling rate.

Aluminium high silicon alloys can be processed by different RS methods. However, melt spinning is the most familiar of all RS commercial methods.

Rapid solidification is a material production route. It does not give the final shape. Rapidly solidified materials require consolidation after solidification. Moreover, consolidation requires mechanical and/or thermal treatment. These treatments may degrade the microstructure. Also hot extrusion may be used as a final step, which may cause deterioration of the initially useful properties due to hot working. In general, although rapid solidification may suppress the coarsening of Si during solidification, the Si particulates may still coarsen during the subsequent thermo-mechanical treatment due to heat effect during consolidation and extrusion.

In the present work characterisation of the material at various stages of production has been carried out and understanding of the metallurgical phenomena involved. Several alloy variants were examined. Coarsening behaviour of the Silicon particles and hardness tests were carried out. Wear properties of the alloys have been studied under dry sliding conditions against a steel ball bearing. Analyses and observation of the wear surfaces and wear mechanisms of these alloys are discussed. Wear of RS materials was compared with piston material A390 made by casting.

Degassing and HIPping at 400 °C for 12 hrs prior to HIPping seems to be the optimum the degassing is effective whilst avoiding excessive coarsening of the silicon particles.

Alloy number RSP461 (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) shows excellent wear resistance due to its fine microstructure.

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CHAPTER 1

INTRODUCTION

Introduction

Increase in the demand for quality materials in the automotive industry has been a focal point for researchers due to constant development. Specific qualities of materials such as resistance to corrosion, resistance to wear, specific strength, ability to withstand high temperatures are of a great advantage in the automotive industry. There is therefore a need for continuous research to understand new processes which may be able to provide superior properties.

Aluminium silicon alloy has proven to be exceptional due to corrosion resistance, low wear rates and low thermal expansion. Aluminium is widely used in the automotive industry because of its low density (2.7g/cm^3), an advantage to weight reduction and gas mileage. These properties have become ideal in the production of a variety of automotive parts such as pistons, connecting rods and many more. Silicon is one of the elements which when added to aluminium has minimum weight increment (Si density 2.33g/cm^3). Increase in silicon in aluminium silicon alloys results in increase in the wear resistance and mechanical properties as long as it is below eutectic range [1, 2]. However, increasing silicon content beyond the eutectic composition causes the formation of large silicon particles which affect the toughness of the alloy. Rapid solidification processing is a technique used for refining the primary silicon and seems to be the most promising technique for the production of high Si Al-Si alloys (i.e. Si content greater than 17 wt.%).

There are number of routes which can be used to produce rapid solidification, including spray methods, weld methods, and chill methods. Of these, melt spinning is

the most widely used industrially due to its high cooling rate and the ability to process large volumes of materials. In the work described here, the material supplied is melt spun ribbon which has been chopped to produce flakes. It then requires degassing, consolidation, and hot extrusion after solidification. These thermo-mechanical treatments may degrade the initial useful properties obtained by melt spinning.

Here the consolidation step involves Hot Isostatic Processing (HIPping) which densifies the flakes. However, at this stage oxide films are still present around the flakes. Hot extrusion is carried out to break up the oxide film layers which would otherwise be a source of weakness.

The aim of this project is to characterise the material at various stages of production and to deepen understanding of the metallurgical phenomena involved. Several alloy variants will be examined. Hardness tests were carried out to evaluate the hardness of each alloy. Wear testing has been carried out to characterize the dry sliding wear of Al- high Si alloys and relate the wear resistance to the microstructure.

CHAPTER 2

LITERATURE SURVEY

2.1 Introduction

Al-high Si alloys are well known for their use as lightweight components in engineering applications, particularly within the automotive industries, due to their high wear resistance and low thermal expansion. It is desirable to increase the hot strength of these alloys by increasing the Si content. However, increasing the Si content beyond about 17 wt. % Si causes the primary Si crystals to become coarse, resulting in deteriorating toughness. The problems associated with conventional hypereutectic Al-high Si casting can be effectively overcome by the use of rapid solidification processing such as spray, weld, and chill methods (e.g. melt spinning). The final product of rapid solidification processing by melt spinning is flakes or ribbons. Hence, a consolidation step is required after degassing. In the work in this project, the steps involved in consolidation are Hot Isostatic Pressing (HIPping) and hot extrusion. In this chapter aluminium and aluminium silicon alloys, high silicon aluminium silicon alloys, rapid solidification, rapid solidification of high Si Al-Si alloys, production methods of rapid solidification, degassing, Hot Isostatic Processing (HIPping), and hot extrusion will be discussed. In addition, as wear resistance is an important property studied in the project, there is a section on wear at the end of the chapter.

2.2 Aluminium and Aluminium Silicon Alloys

The most important properties of aluminium are its low specific gravity, which is only 2.7 as compared with 7.8 for steel and 8.8 for copper, high corrosion resistance

in various media due to the formation of natural oxide film on its surface, and high ductility [3]. Commercial aluminium and aluminium alloys are divided into two major groups [4-6]:

1. Wrought alloys, which are shaped by hot and /or cold plastic deformation such as forging and rolling.
2. Casting alloys which are made with foundry practice such as sand and pressure die casting.

Silicon is advantageous in metallic alloys used for aluminium casting. This is because it increases the fluidity of the melt, reduces the melting temperature, decreases the contraction associated with solidification and is very cheap as a raw material. Al-Si alloys represent the base for most aluminium casting alloys. Figure 2.1 [1] shows the aluminium-silicon phase diagram and the cast microstructure of pure Al and alloys of various compositions. Additions of silicon to aluminium fall into three major categories [1, 7]: eutectic, hypoeutectic, and hypereutectic. ‘Eutectic’ is the point of saturation of Si in aluminium and occurs when the silicon level reaches 11.7 wt. % (weight percent). Hypoeutectic means the silicon content is less than 11.7 wt. %. ‘Hypereutectic’ occurs when the silicon content is higher than 11.7 wt. % (weight percent). Hence, aluminium with 16 wt. % silicon has 12 wt. % dissolved silicon and 4 wt. % shows up as primary silicon [1, 3].

2.2.1 The Effect of Alloying Elements

The alloys under consideration here contain Al, Si, Zr, Cu, Mg, Fe and Ni. Silicon is the main alloying element; it imparts high fluidity and low shrinkage in casting.

Alloying elements in Al-base alloys has been summarised in Ref [8]. Addition of Si to aluminium alloys reduces the thermal expansion coefficient and increases wear

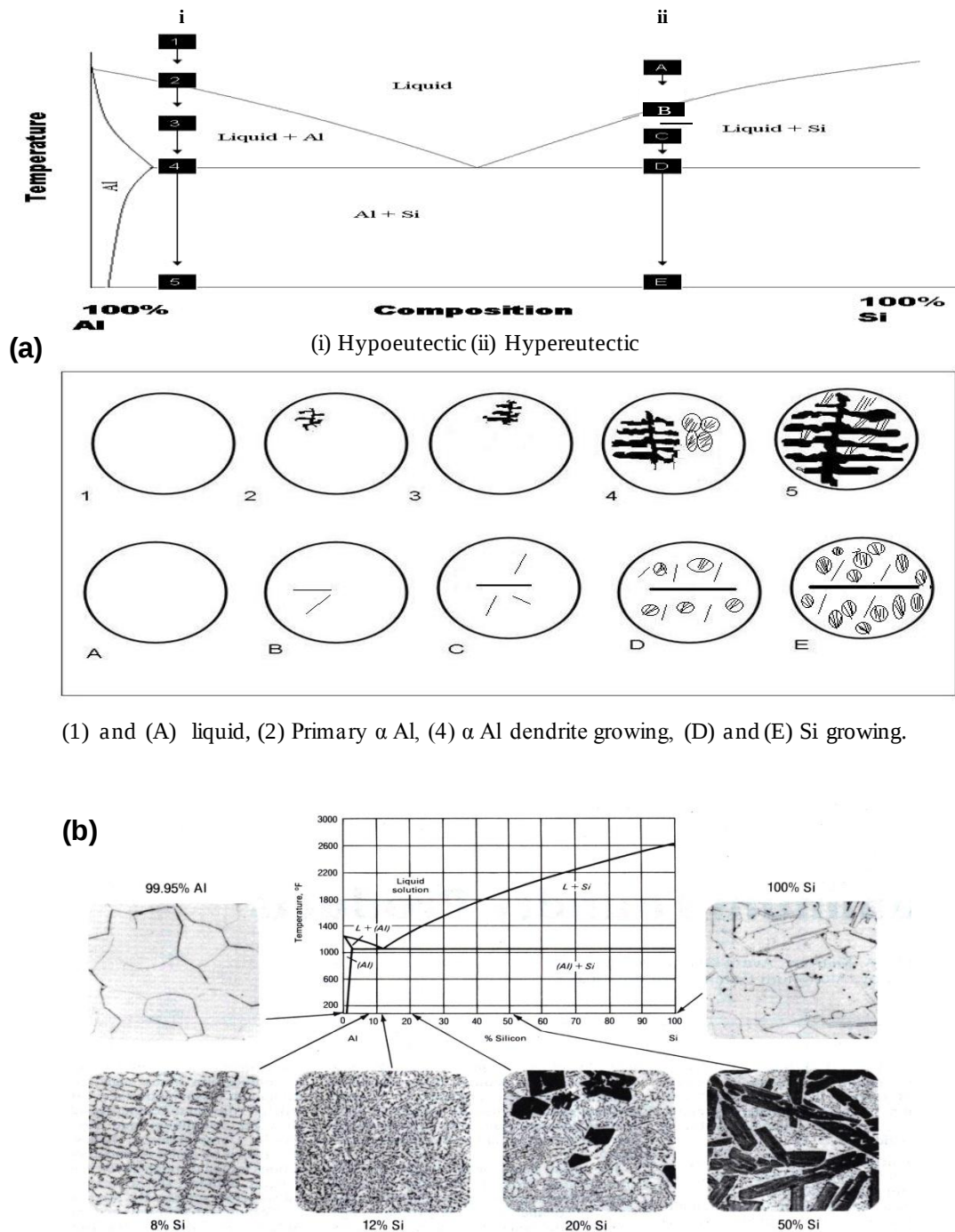


Figure 2.1 a) Schematic diagram of aluminium-silicon phase diagram to illustrate hypoeutectic and hypereutectic b) Aluminium-silicon phase diagram and associated microstructures [6].

resistance because the Si not in solid solution is present as hard particles embedded in a soft matrix. Addition of nickel to aluminium-base alloys forms dispersoids of Al_3Ni resulting in a lowering of the thermal expansion coefficient and improved high temperature mechanical strength. The Fe and Ni additions to Al-Si alloys promote the formation of many intermetallic compounds which will improve the proof stress by up to 45MPa at elevated temperatures. These intermetallic compounds reduce the coefficient of thermal expansion. Moreover, Nickel can improve the alloy strength and hardness at elevated temperature when combined with copper. Zirconium has been known to be effective in modifying the primary silicon particles and refining the grains. The addition of Zr increases the tensile strength by 12% and has no effect on the wear resistance. Iron has low solubility in the solid state. Hence most of the iron present in aluminium appears as an intermetallic second phase with aluminium and mostly with other elements. Iron increases the strength slightly, but decreases the ductility. Copper strengthens the alloy by precipitation of CuAl_2 and improves the corrosion resistance. Addition of small amounts of Cu to Al-Si alloys increases the fatigue strength under cyclic tensile loading as well as the tensile strength. The addition of magnesium markedly increases the strength of Al without unduly decreasing the ductility. Magnesium strengthens the alloy through precipitation of fine Mg_2Si . In general, Cu and Mg increase the strength due to precipitation hardening. Fe and Ni increase elevated mechanical properties due to the present of intermetallic particles [9-19].

2.3 High Silicon Aluminium-Silicon Alloys

Recently, high Si Al-Si alloys have attracted much attention as light-weight materials because of their excellent wear resistance, and low thermal expansion coefficient [20-

23]. These properties are generally attributed to the presence of a high volume fraction of hard Si particles embedded in an alloy matrix [21, 22]. The properties of these alloys can be correlated with the morphology, size, and distribution of the primary silicon in the microstructure [24].

There has been a need to increase further the wear resistance and the mechanical properties of these alloys. Increasing the silicon will increase the wear resistance of Al-Si alloys. However, increasing the Si content will make the alloy less ductile and harder [25]. Moreover, the addition of iron (Fe) will improve the tensile strength at elevated temperatures and improve wear resistance as well due to the formation of intermetallic compounds which are fine and hard. The improvement of hot strength is due to the formation of fine dispersoids in the aluminium matrix. The increase of wear resistance is due to the presence of hard Al-Si-Fe intermetallic compounds. Hence, increasing the hot strength and the wear resistance requires increased Si content. However, as the Si content increases beyond the eutectic composition, particularly beyond about 17 wt %, this leads to the formation of coarse brittle Si phase due to the relatively low solidification rate in casting processes [20, 22, 23, 26, 27]. The materials then have low toughness. Moreover, the addition of Fe will form coarse intermetallic particles due to the solid solubility limit of Fe in Al at room temperature under equilibrium conditions [20, 22, 26, 27]. These coarse particles will degrade the mechanical behaviour of these alloys [20, 22, 23, 27, 28].

In summary, casting of high silicon Al-Si alloys will generate large degrees of segregation and coarse microstructures due to the low rates of solidification. The problems associated with ingot casting of hypereutectic Al-Si alloys (i.e. segregation, coarse microstructures and porosity) may be overcome by:

1. Rheocasting which involves stirring the Al-Si alloy during solidification to produce a semi-solid slurry, then injecting the slurry directly into the die [29, 30].
2. Rapid solidification processing where there is a high cooling rate such as melt spinning, atomization and spray deposition [26, 31].

Rheocasting and rapid solidification do not permit the formation of coarse Si.

2.4 Rapid Solidification

Rapid solidification (RS) was initially developed approximately 35 years ago by Dewez and his colleagues at California Institute of Technology. They chilled a thin layer of liquid metal on to a very conductive substrate. RS is defined as the transformation of liquid to solid by removing superheat and latent heat at a cooling rate of 10^2 - 10^6 Ks⁻¹ [4, 5, 32]. In order to achieve RS the following criteria must be satisfied [4]:

- a) High undercooling before the occurrence of solidification.
- b) High velocity of the solidification front during continuous solidification.
- c) High cooling rate during solidification.

RS affects the microstructure (see figure 2.2) and phase equilibrium and offers the following advantages [2, 4, 33-40]:

1. Refinement of microstructure.
2. Extension of solid solubility.
3. Increased chemical homogeneity.
4. Precipitation of non-equilibrium crystalline phases.
5. Formation of amorphous phases.

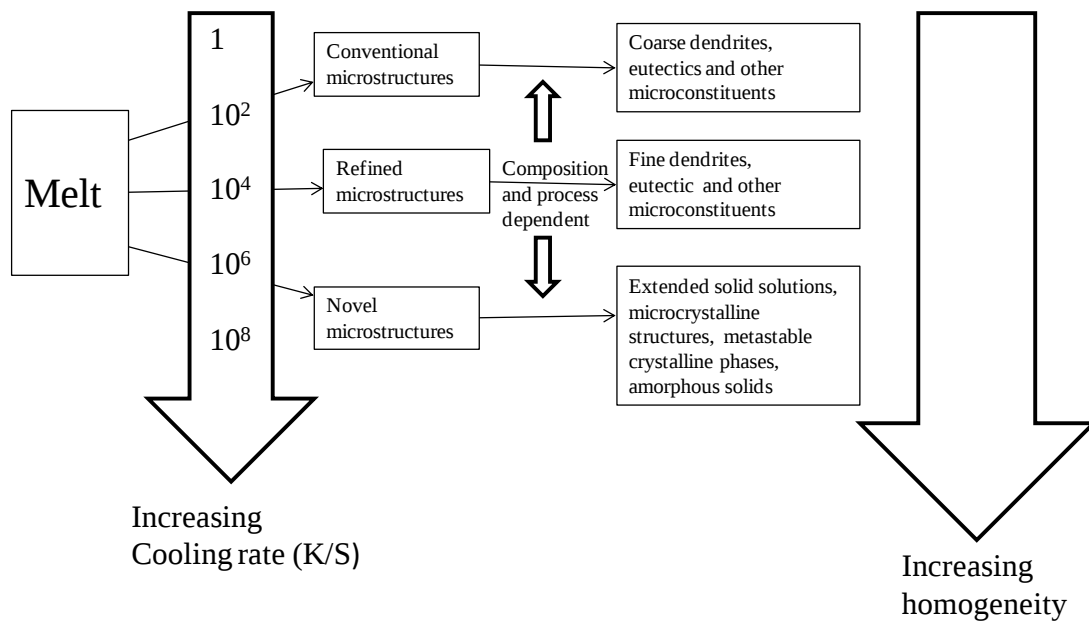


Figure 2.2 Effects of RS on microstructure [41].

Rapid cooling has a great effect on the microstructure. The cooling rate of conventional casting is approximately 1K/S. The expected microstructure at this stage is very coarse accompanied with high segregation. As cooling rate increase gradually i.e. greater than 10^2 K/S, the microstructure is finer compared with the conventional casting. The microstructure is composed of dendrites, eutectic and other microconstituents. The refinement of the microstructure is mainly due to the high cooling rate which provides shorter time for coarsening to take place. However, chemical segregation for the cooling rates in the range of 1K/s to 100K/s is almost the same because of local equilibrium is achieved at the solid/liquid interface because temperatures and concentrations are provided by equilibrium phase diagram.

Undercooling of the molten metal occurs as the cooling rate increases. Therefore novel microstructure exists at the solidification rate of about 10^6 K/s. At this solidification rate the novel microstructures are extended solid solutions,

microcrystalline structure, metastable crystalline phases and amorphous solids; there is a huge deviation from equilibrium at the solid/liquid interface accompanied with the solid phase entrapping supersaturated solute and impurity atoms.

Massive solidification rate can be achieved at high cooling rate (10^8K/s and above) when homogenous structure can be obtained because the composition of the solid is almost the same as the molten metal. Moreover, a microcrystalline structure is formed due to the effect of high nucleation rates accompanied with very low growth rates at very low temperature [33, 41].

2.4.1 Refinement of Microstructure

In RS, there is a large chemical driving force available to transform the liquid phase to the product crystalline phase. A grain size of the order of 10 nm can be achieved. Strength is an important factor that can be improved by the reduction of the grain size of material produced by RS [42, 43]. This is related to the Hall-Petch effect, which can be expressed as follows [33, 44]:

$$\sigma = \sigma_0 + k d^{-1/2}$$

where:

σ = yield strength.

σ_0 and k = constant.

d = grain diameter.

Moreover, reduction of grain size by RS improves both strength and toughness simultaneously. In addition, RS reduces the second phases that precipitate during solidification such as the carbide phases in tool steel. Another advantage of microstructural refinement in RS is in reducing the effect of inclusions, which exist in commercial alloys, by reducing their size. This will mean that manufacturers can

produce some aircraft and automotive parts from scrap which contains impurities, hence reducing alloy costs [32, 33, 45].

Furthermore, grain size reduction can lead to superplasticity due to the production of fine, stable microstructures. In order for superplasticity to occur equiaxed grains less than 10 μm must be formed, where grain boundary sliding can take place. In addition, a second phase must exist to stabilize the small grains. RS is an excellent process to form fine stable microstructures and to enhance superplasticity [33, 46-48].

2.4.2 Extension of Solid Solubility

In RS a phenomenon known as solute trapping exists. This means the formation of highly supersaturated solid due to the deviation from local equilibrium. The chemical potential of the solute actually increases during crystallization. When the solid/ liquid interface is below the equilibrium liquidus temperature, a wide range of solid compositions is permissible at the interface. In RS where high interface velocities take place, the interfacial solute partition coefficient (k), which can be expressed by the ratio of the solute concentration in the solid to that of the liquid, deviates from its equilibrium value. Hence, the existing solid phase will contain more solute than allowed by equilibrium, if the interface is undercooled. The most important advantage of the extended solid solubility is the reduction of particle size, which increases the strength [33, 49, 50].

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2.4.3 Increase in Chemical Homogeneity

In RS, segregation of alloy elements is minimized due to the shortness of time for diffusion processes to take place. This explains the reduction in compositional

variations and in phase separation within a phase. An advantage of chemical homogeneity is the ability of RS alloys to be treated thermally for shorter times compared with other conventional alloys [33, 51].

2.4.4 Precipitation of Non-Equilibrium Crystalline Phases

This phenomenon exists due to the fast cooling rate, which allows the precipitation of crystalline phases to take place in non-equilibrium conditions. For example, it means displacement of equilibrium graphite by nonequilibrium cementite on chilling of cast irons, which becomes widespread at cooling rates of 10^6 Ks^{-1} [52]. It occurs in alloy systems not forming intermediate phases under equilibrium conditions. These phases are intermediate rather than terminal alloy phases. They tend to show disordered rather than ordered arrangements [5, 52].

2.4.5 Formation of Amorphous Phases

In rapid solidification, there may be insufficient time for nuclei to form and grow due to high and rapid cooling rates. When this occurs, the liquid structure is locked into place and amorphous material forms. Noncrystalline phases are characterized by a complete lack of long range crystallographic order i.e. no regularity in relative atomic position (no structural periodicity) beyond a distance of a few atomic diameters. This disordered structure is an extreme nonequilibrium condition obtained in metallic materials at high cooling rates. It should be mentioned that glasses occur when continuous cooling (1 Ks^{-1}) of silica or silicate materials take places. Pure metals tend to require 10^{10} Ks^{-1} to obtain the required suppression of crystallization on solidification. However, rapid solidification processing (cooling rate 10^6 - 10^{10} Ks^{-1})

reduces this critical cooling rate mainly by water quenching. These materials have high hardness and corrosion resistance [5].

In summary, the effect of RS on the material properties can be summarized as in table 2.1.

Table 2.1 Effects of RS on material properties [33].

Structural features	Expected property improvement
Size-refinement	Hall-Petch strengthening Improved fracture and impact toughness Enhanced superplasticity
Extended solid solubility	Increased solid solution strengthening Increased precipitation and dispersion strengthening Minimized brittle equilibrium phase precipitation
Chemical homogeneity	Improved corrosion/oxidation resistance Better response to working Better response to heat treatments
Precipitation of non-equilibrium crystalline phases	Improved physical properties Improved mechanical properties
Formation of amorphous phases	Improved physical properties Improved mechanical properties (strength, wear) Improved corrosion resistance

2.5 Rapid Solidification of High Si Al-Si Alloys

RS affects the microstructure of Al-Si alloys by:

- a) Constituent changes.
- b) Size refinement.
- c) Formation of metastable phases.

Rapidly solidified hypereutectic Al-Si alloys can be fabricated by [53-55]:

- a) Atomization and powder metallurgy.
- b) Spray deposition.
- c) Melt spinning.

Al-20%Si alloys produced by RS show a fine distribution of primary Si of about $3\mu\text{m}$ diameter embedded in an aluminium matrix [56]. Rooyen et al. [56] reported that the microstructure of flakes and ribbons contains two different phases. Also, it was reported that the size of Si precipitates can be less than 150 nm [57]. The micrographs in figure 2.3 show the microstructure of RS Al-17.48 % Si compared with the same composition of alloy produced by conventional casting. Figure 2.3(a) reveals a uniform, homogeneous distribution, and reduced dendrite size compared with figure 2.3 (b) [58].

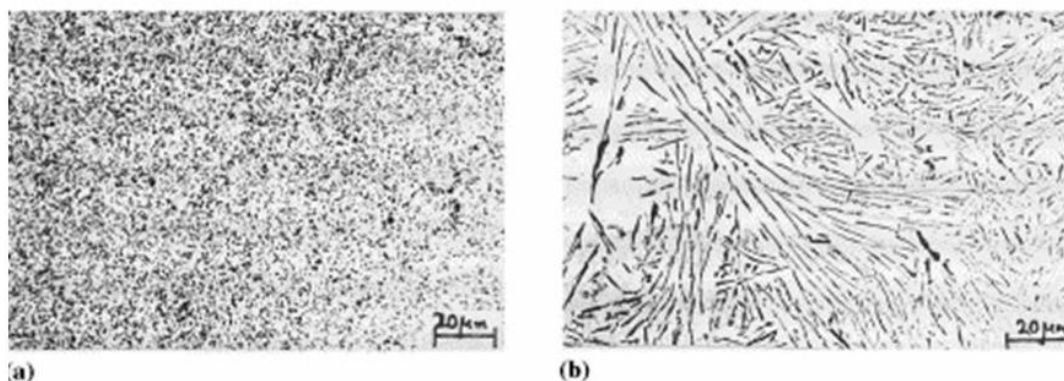


Figure 2.3 Optical micrograph of Al-Si alloys: a) Rapidly solidified and extruded (Al 17.26 Si 0.23Fe) b) Conventionally cast and extruded (Al 17.36Si 0.22Fe) [58].

It was reported that Al_5FeSi phase which gives strength is formed in RS Al-high Si alloys from Al_9FeSi_3 [59].

RS improves the mechanical properties of Al-Si alloys. Table 2.2 compares the mechanical properties of Al-17.48%Si produced by RS and conventional casting. A 50% increase in both the Yield and UTS of rapidly solidified extruded bar has been reported. Further increase of 30% to these properties has been reported due to the addition of 1.48% Cu [58, 60-63]. Figure 2.4 shows optical micrographs of Al-Si-Cu alloys (a) rapidly solidified and extruded and (b) conventionally cast and extruded.

As solidification rate increases, grain size decreases because there are more dispersed particles formed and they are finer. Hence, the strength increases due to finer dispersoids strengthening mechanism (particle size is less than $1\mu\text{m}$) which will impede grain boundary movement. The hardness of RS hypereutectic Al-Si alloys increases as a result of dispersion strengthening. In addition, the hardness increases when the material is heat treated at around 473K (i.e. due to formation of precipitate from Cu alloying when heat treated). However, hardness decreases when the material is heat treated at above 573K due to coarsening of the structure [56, 64-67]. Also, hardness decreases as silicon content decreases [68].

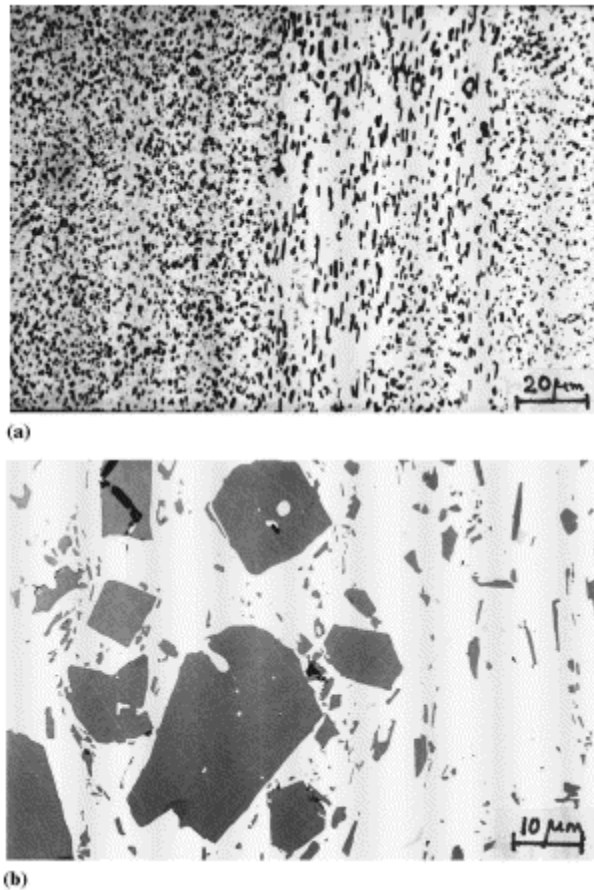


Figure 2.4 Optical micrograph of Al-Si-Cu alloys a) Rapidly solidified and extruded (Al 17.39Si 1.48Cu 0.16Fe) b) Conventionally cast and extruded (Al 17.37Si 1.46Cu 0.19Fe) [58].

Table 2.2 Tensile properties of Al-Si alloys [58].

Alloy	Processing route	Yield strength (MPa)	UTS (MPa)	Elongation (%)	Composition (wt.%)		
					Si	Cu	Fe
Al-Si	CCE	108	110	2.4	17.63	0.04	0.22
	RSE	168	173	3.2	17.26	0.06	0.23
Al-Si-Cu	CCE	224	227	2.5	17.37	1.46	0.19
	RSE	290	295	4.4	17.39	1.48	0.16

CCE means conventionally cast and extruded; RSE rapidly solidified and extruded.

2.6 Production Methods of Rapid Solidification

There are number of methods which can be used to produce rapid solidification, including spray methods, weld methods, and chill methods. Final products include:- spherical particulates, continuous or discontinuous ribbon, flat ribbon or sheet, surface layers or multilayered deposits. Figure 2.5 illustrates different types of products and their production methods.

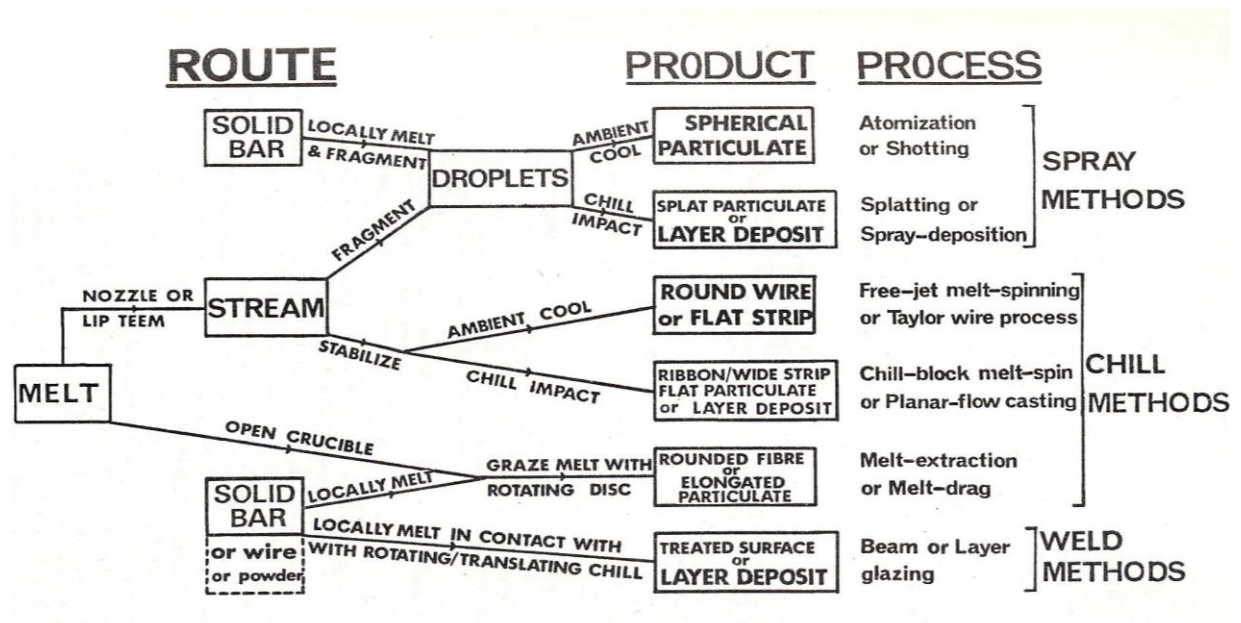


Figure 2.5 Production routes for RS and their products [5].

2.6.1 Spray Methods

Spraying involves fragmentation of melt into droplets prior to quenching. It involves breaking up molten charge by direct impingement by a jet of fluid such as water, steam or gas or by a high speed rotation to form spray droplets.

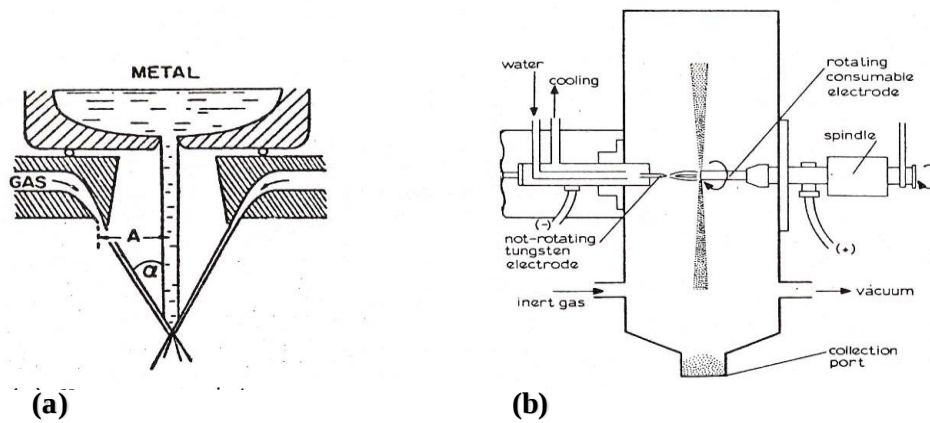


Figure 2.6 Spray methods a) V or cone jet atomization b) Rotating electrode process [5].

Jet fragmentation involves breaking up the molten metal at a distance from the discharge nozzle to avoid blocking the tip of the nozzle, with the aid of pressurised cooling gas (figure 2.6a) [33, 69]. Another technique is breaking up a rotating melt into droplets using a spinner which could be an electrode or a circular disc. Figure 2.6 (b) illustrates the rotating electrode process where an arc provides melting and droplet formation at the tip of a rotating round bar. This process is suitable for most reactive metals due to the absence of a crucible.

2.6.2 Weld Methods

The weld methods involve rapid melting and solidification of a limited depth at the surface of a workpiece. A traversing heat source in the form of a single pulse or a continuous beam is used to rapidly melt the surface of the block of material, which acts as heat sink during solidification (see figure 2.7).

Another approach is to inject alloy or dispersoid at the surface to form a different composition from the workpiece [5].

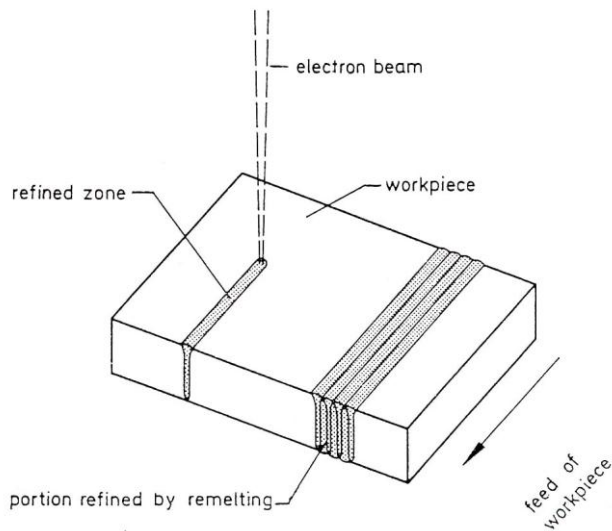


Figure 2.7 Principle of rapid solidification at the surface showing rapid local melting with a traversing heat source [33].

2.6.3 Chill Methods

Chill methods involve continuous melting up to and during quenching. These methods require stabilization of the melt source before breaking it up. The spinning method (figure 2.8) is probably the most industrially used. This is due to its ability to process large volumes of material, and high cooling rates of up to 10^6 Ks^{-1} . It involves ejecting the molten metal through an orifice by pressurized gas. Then, the jet is directed onto a cooled rotating copper drum. The drum is maintained at a low temperature (5°C) by constantly circulating pressurized cold water. The molten metal is dispersed and instantaneously solidified in the form of continuous ribbon, when it comes into contact with the copper drum [70-72].

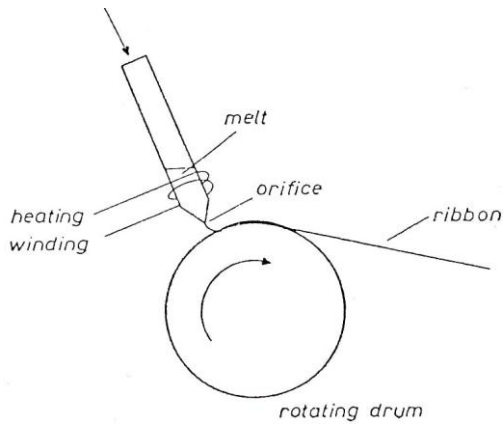


Figure 2.8 Schematic of melt spinning [33].

2.7 Degassing

Aluminium has high reactivity, which prevents the production of oxide free ribbons or flakes. Moreover aluminium oxide will form as hydrate on ribbon and flake surfaces in a humid environment. Therefore, it is necessary to remove water and hydrogen from surface oxide before complete consolidation because during consolidation the water of hydration can react with the metal surface to form hydrogen. This hydrogen will form porosity in the final product. Failing to properly degas will lead to the development of high internal pressure during subsequent hot working (i.e. hot extrusion) or service at high temperatures. This problem can be overcome by degassing the ribbons prior to consolidation. Researchers have used a variety of different heating sequences [71, 73-75].

The maximum degassing temperature must be equal to or higher than the consolidation temperature but not lower than 300 °C [74].

2.8 Hot Isostatic Processing (HIPping)

Rapidly Solidified flakes and ribbons are not of any commercial interest unless they are consolidated into fully dense bodies. The consolidation sequence consists of degassing, and HIPping, followed by hot extrusion [4].

HIPping is mainly used to upgrade castings, to consolidate powders, or for interfacial bonding. The process involves subjecting components to high temperatures and high gas pressures, within a specially constructed pressure vessel. This removes internal porosity and diffusion bonds interfaces in contact.

HIPping temperatures are greater than $0.7 T_m$ [76]. The process involves diffusion of atoms and the plastic deformation of flakes and ribbons at the consolidation temperature [4]. The isostatic pressure comes from atoms of gas colliding with the surface of the object, where each gas atom is behaving as a tiny atomic forge. The process is capable of removing both macro-and micro-porosity from a component without affecting the microstructure. The pressurizing medium is applied equally in all directions (isostatically). Hence, (in theory) there is no distortion of the microstructure or the overall geometry. However, there are dimensional changes due to porosity removal.

The main advantages of HIPping cast components are:

1. Improved static (i.e. density) and dynamic properties (i.e. fatigue).
2. Decreased scatter of properties.
3. Improved machined surface finish (i.e. by closing sub-surface porosity).

Flake consolidation by HIPping is usually used in order to obtain a microstructure and properties not readily available by casting. Moreover the process is used to

produce dense powder billets for further shaping, for semi-finished shapes for closed die forging, and for net-shapes [76, 77].

To achieve good bonding, proper vacuum degassing of the unconsolidated material must be done prior to consolidation. Failure to degas will cause decohesion during further heat treatment or service. The degassing temperature should be higher than any subsequent process temperature such as extrusion, HIPping, and heat treatment temperature [33].

Flakes or ribbons are the first step in the production process. Rapidly solidified materials require a consolidation process as a second step after solidification. Moreover, the consolidation process requires mechanical and/or thermal treatment. These treatments may deteriorate the properties in comparison with the initial product. Also hot extrusion is used as a final step, which may cause deterioration of the initially useful properties due to hot working.

2.9 Hot Extrusion

Hot extrusion is carried out to break up the oxide film layers which would otherwise be a source of weakness and to form useful shapes by plastic deformation [33, 78, 79]. The extrusion process is controlled by many parameters. The most important of these parameters are extrusion ratio, working temperature, ram speed, and friction conditions [56].

2.10 Production Route for the Material Used in this Study

Melt spinning, discussed previously in section 2.6.3, is the route used by RSP Company in The Netherlands (who have sponsored the current project). The overall

process used by RSP is shown in figure 2.9. Flakes are produced by chopping melt spun ribbon.

The flakes are either ‘free encapsulated’ (i.e. placed in a canister without compaction prior to degassing) or consolidated (i.e. cold precompacted to 60% density) by cold work to a density of about 60% theoretical before degassing. The flakes in the canister are then degassed before the canister is sealed and hot isostatically pressed to full (or almost full) density. The capsule is then machined off to avoid the mixing of aluminium with Al-Si material and the billet extruded. Figure 2.10 illustrates RSP steps for processing bar from flakes and table 2.3 shows the mechanical properties of RSP alloys.

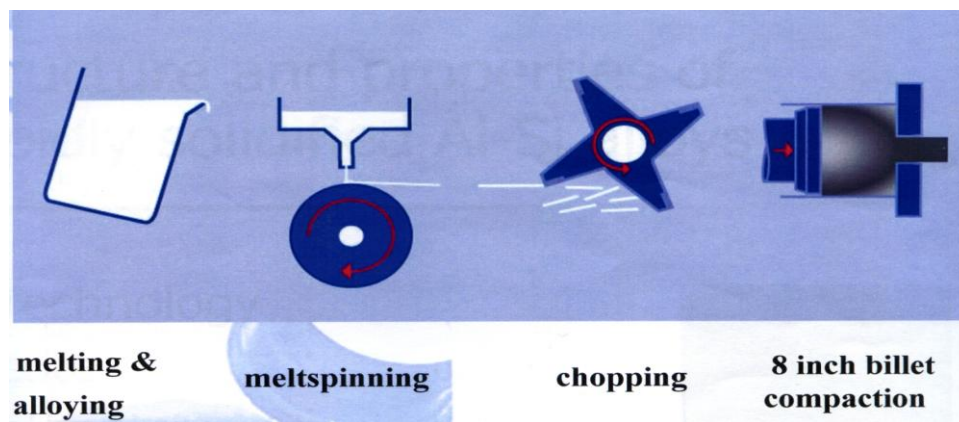


Figure 2.9 Melt spinning technique used by RSP Company

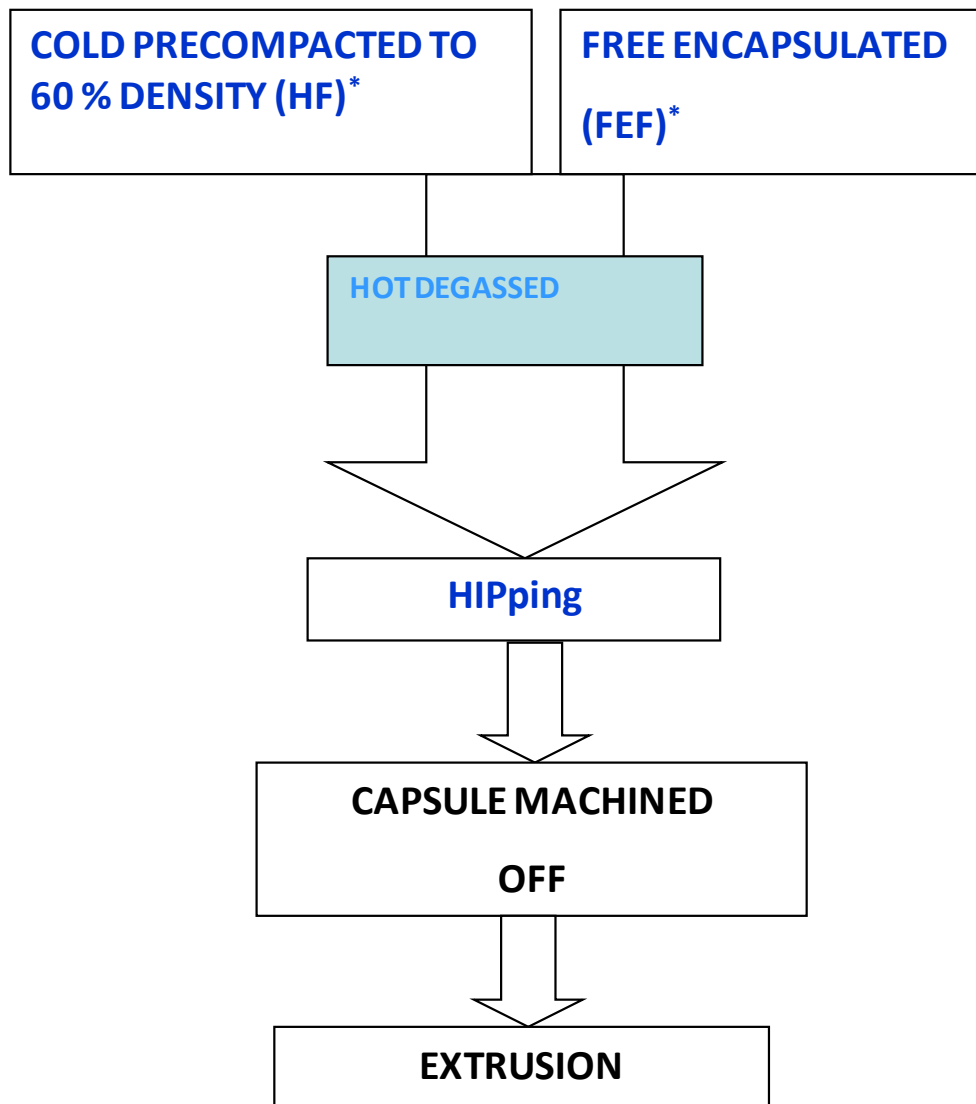


Figure 2.10 RSP steps for processing bar from flakes.

*RSP use the designation HF for cold precompacted and FEF for free encapsulated.

Table 2.3 Mechanical properties of RSP alloys [80].

RSP alloy	Hardness (HB)	Elongation (%)	Thermal expansion $\mu\text{m}/\text{m}^\circ\text{C}$	UTS (Mpa) Rm = 20 °C	YS (Mpa) Rm = 20 °C
444	170	1	15	470	390
446	170	1.8	17	530	425

2.11 Wear of Aluminium High Silicon Alloys

2.11.1 Introduction

Wear is defined as the progressive loss of material from a surface or surfaces and is measured as volume of material lost per unit sliding distance. It is also known as undesired transfer of materials from one surface to another [81, 82]. It may be defined as surface damaged due to the relative movement of two components are in contact with each other [83].

Wear is considered to be one of the most severe engineering problems which necessitates the replacement of numerous mechanical components. However, there are also a number of applications which make positive use of wear, such as sand blasting and cutting processes [81]. Wear can occur due to shearing of weld surface and sub-surface crack propagation and corrosive action. There are many methods to reduce the wear of engineering components e.g. lubrication and materials modification. Analysis of wear debris can sometime indicate the operating wear mechanism [83].

Amount of wear, which is the removal of material, is expressed as volume loss or mass. The volume of the material loss is equal to the weight loss of a sample divided by the density of the sample [84]. The wear rate is defined as the volume of material lost per unit sliding distance. The wear coefficient K is a further measure of severity of wear and is the ratio of wear rate to true contact area [83].

Many efforts have been made to study wear characterization and mechanisms. One method of studying material removal from the sample in contact with a hard surface is the reciprocating sliding test.

2.11.2 Types of Wear

There are four primary wear mechanisms: Adhesive wear, abrasive wear, corrosive wear and surface fatigue wear.

Adhesive wear

This is the most common type of wear. It occurs when two smooth surfaces are slid over each other and the asperities of the soft materials have been sheared and welded on the hard surface.

Abrasive wear

There are two types mild and severe. The severe abrasive wear is very dangerous because it occurs suddenly i.e. catastrophic failure due to the high wear rate. It occurs when two surfaces are sliding over each other and one of them is rougher and harder. Loose and non adhering particles are sliding under pressure against another surface and creating grooves due to microcutting. This type of wear can be two-body when a single surface is roughened by particles and three-body when particle (i.e. the abrasive) is blocked between two surfaces [81, 83, 85]. Three-body wear is more severe than two-body wear.

Corrosive wear

This type of wear occurs in a corrosive medium. It is mainly a combination of wear and harsh chemical substance such as acid.

Surface fatigue wear

Such wear occurs when sliding is repeated. This type of repeated action will create subsurface cracks resulting in the breakup of the surface by introducing large pits [81, 82, 85].

2.11.3 Wear of Al-Si Alloys

The wear behaviour of Al-Si alloys has attracted growing interest. There are different opinions regarding the optimum silicon content which will lead to higher wear resistance. However, controversy exists in the literature regarding the effect of silicon content and pressure on dry sliding wear [86]. Clark and Sarkar [87] found that the increase in silicon content of the binary Al-Si alloy leads to increased wear resistance for pin on bush up to the near eutectic composition and the hypereutectic (about 21% Si) alloys wear more than the hypoeutectic alloys. Also, they found that a high silicon alloy (about 21% Si) causes wear of the counterface, even when this is very hard steel, because the silicon particles act as abrasives and they are the main cause of wear on the counterface. They concluded that the beneficial effect of silicon is to minimize the tendency for seizure.

Sharma et al. [88] found that increasing silicon content from 4 to 20% increased the wear resistance. Also, as the silicon content of hypereutectic Al-Si alloys increased the wear resistance increased [89].

It has been reported that at intermediate contact pressures around 117 kPa, the wear resistance increases with increasing the silicon content [89].

Bai and Biswas [90, 91] reported that wear of alloy without silicon is higher than that of binary alloys containing 4-24 wt. %Si and there is no regular trend in wear with respect to silicon content. Additionally, they found that the wear resistance increases with the addition of alloying elements such as Cu, Fe, Mg, Mn, Ti, Zn and Ni and there is significant improvement (i.e. to Al without Si) when the alloy contains 17% Si for dry sliding pin on disc test [92]. However, as the pressure increases the wear rate increases [93].

Wang et al. [94] found that the wear rate decreases with increasing the Si content at low load (8.9 N). They also reported that the wear mechanism was oxidative. They found that Al-20Si showed superior wear resistance compared with Al-12Si and Al-25Si at high load. They also observed that at a high load of 35.6 N as the silicon content increased the wear debris become more powdered (i.e. there was an increase in fineness) and as silicon decreases the wear debris is flake type for dry sliding pin on disc test.

Some studies have shown that heat treatment (i.e. T6) improves the wear properties of Al-Si alloys due to the increase of hardness [95, 96]. However, the existence of silicon particles unaccompanied with other elements is not sufficient for resistance to wear load [97].

Some studies have shown that the effect of alloying elements on the wear resistance of Al-(4-20)%Si-0.3%Mg alloy is due to solid solution strengthening and precipitation of Mg_2Si [88, 95].

When subjecting Al-Si alloys to dry sliding tests, two main regimes are observed to take place. The first is a mild wear region which is mainly mechanical mixing and surface oxidation. The second is a severe wear regime which is metallic wear because of the shiny appearance of deformed surfaces [87]. However, there is no sharp transition separating mild and severe regimes [91].

Bai and Biswas [91] found that the debris of Al-Si alloys is flake shaped and the debris quantity collected at 10 N showed a larger number of flakes compared with 2N. They also found that as load increases (i.e. from 10-65 N) debris size increases.

Hitoshi and Fusao [98] reported that the wear track of Al-(18-26%) Si alloys showed a black spotty surface due to high sliding speed (i.e. 8m/sec) and high silicon contents.

Methods and material	Material	Wear rate (m ³ .m ⁻¹)x10 ⁻¹²				Wear rate (m ³ .m ⁻¹)x10 ⁻¹²			Wear rate (m ³ .m ⁻¹)x10 ⁻¹²				Wear rate (m ³ .m ⁻¹)x10 ⁻¹²		
[89] Pin on a disc. D=15mm. Dry sliding on steel (50HRC). Pins are cast Al-Si alloys Time 60 min. Area= 1.8E-04 m ²		Contact pressure (kPa)				Contact pressure (kPa)			Contact pressure (kPa)				Contact pressure (kPa)		
		40	80	120	158	304	457	609	492	984	1476	1968	509	2038	3057
	Al-7%Si	0.9	1	3.5	5										
	Al-13%Si	0.5	2.2	1.5	2										
	Al-22%Si	0.9	1.9	3	4										
[87] Pin on a disc. Dry sliding on steel (50HRC). Pins are cast Al-Si alloys. Speed= 196 cms ⁻¹ Area =3.2E-05 m ²	Al-15%Si					0.0053	0.0076	0.0087							
	Al-16%Si					0.0076	0.0133	0.0133							
	Al-21%Si					0.0134	0.017	0.0229							
[94] Pin on a disc. D = 4.64mm. Dry sliding on steel (50HRC). Pins are spray deposited Al-Si alloys. Speed = 0.48 m/s Distance = 1.7 km. Area= 1.8E-05 m ²	Al-12%Si								0.022	0.023	0.038	0.069			
	Al-20%Si								0.015	0.023	0.026	0.03			
	Al-25%Si								0.011	0.023	0.034	0.042			
[91] Pin on a disc. D = 5mm. Dry sliding on steel (50RC). Pins are cast Al-Si alloys. Area = 2E-05 m ²	Al-12%Si												4	10	10
	Al-15%Si												4	9	13

Table 2.4 Wear rate of Al-Si and High Si alloys.

Table 2.4 shows the wear rate of Al- Si and high Si alloys. A full detail of the table calculations are shown in appendix (1). The data shows that there is no correlation between the Si content and wear rate or the contact pressure and the wear rate and

that the wear performance of these materials is a complex function of the structure and mechanical properties.

2.11.4 Wear Measurement Examining the sliding material before and after wear is the most commonly used method. The weighing method is a simple and suitable way of measuring wear. It consists of weighing the sample before and after the test. The weight loss is equal to the difference between the weight before and the weight after. This method can be inaccurate due to the difficulty of cleaning and drying the specimen. The mechanical gauging method is also called the micrometer method. It consists of taking the dimensions of the component before and after the test. It is used for large components such as automobile cylinders [81, 99, 100]. Another method is using a profilometer to take a profile of the track from which the depth or volume of the wear track is determined [99, 101]. The aim of all these techniques is to determine the volume of material lost from the surface. The different techniques are all subject to different experimental difficulties but are the most commonly used methods for determining the wear volume.

2.12 Summary

Low density, high thermal conductivity, and superior mechanical properties make the high silicon Al-Si alloys attractive for automotive applications such as pistons and connecting rods, where weight saving is vital. These alloys cannot successfully be produced by traditional manufacturing process such as ingot casting due to excessive embrittlement from coarse Si precipitates. Melt spinning based rapid solidification processing has the potential to eliminate this problem by strong refinement of the Si microstructure. Rapidly solidified products such as flakes require consolidation by

degassing, hot isostatic processing and hot extrusion. The material used in this project has been supplied by RSP Ltd. and includes: ribbons, flakes, as-HIPped material and HIPped plus extruded material. The potential for improvement in wear resistance is an important feature of Al-high Si materials. Wear mechanisms and methods of measurement have been briefly described alongside the literature on wear resistant for Al-high Si alloys.

CHAPTER 3

ALUMINUM SILICON ALLOYS IN THE AUTOMOTIVE INDUSTRY

3.1 Introduction

There have been tremendous improvements in automotive design and materials selection in general and in racing cars especially where there is demand for continuous improvement for all parts and the engine in particular. The improvement of mechanical properties such as strength, toughness and modulus of elasticity of rapidly solidified Al-Si alloys makes them excellent materials to be used in aircraft and aerospace applications. In this work, we have concentrated on the use of Al-high Si alloys for land vehicles. The automobile, engine components, the piston engine, four-stroke cycle and Al-high Si alloys in automotive applications will be discussed in this chapter.

3.2 The Automobile

An automotive system is a group of related parts which perform a specific job, for example, the clutch pedal is one part in the gearbox system.

An automotive system can be grouped as follows:

1. Engine system, which provides power for the vehicle.
2. Power train systems which use engine power for propulsion e.g. the drive shaft.
3. Electrical systems, which operate the electrical devices such as starting.

3.2.1 Engine Components

Basic engine components are shown in figure 3.1.

1. The piston.

- transfers the force power produced by the high pressure to the connecting rod. The piston head experiences high heat, hence, it must be made out of materials resistant to high temperatures.

2. Connecting Rod.

- transfers the force motion produce by the piston to the crankshaft. The I-beam of the connecting rod has a high strength to weight ratio, which prevents it from breaking and bending.

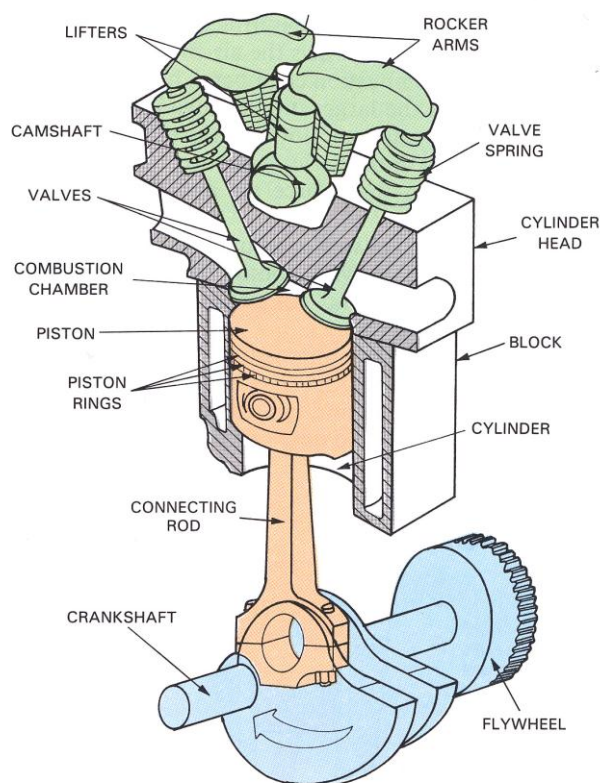


Figure 3.1 Engine components [102].

3. Engine Block (cylinder block).

- represents the body of the engine.

4. Piston Rings.

- fit on the gap (clearance) around the side of the piston, hence, filling the clearance between the piston and the cylinder wall. Their function is to maintain combustion pressure and prevent oil from leaking.

5. Crankshaft.

- transfers the perpendicular motion up and down movements (reciprocating) of the piston to horizontal (rotating) motion.

6. Cylinder head.

- protects and seals the top of the cylinder.

7. Valves.

- control the flow rate of the fuel mixture and exhaust out of the cylinder. There are two valves in each cylinder, one valve called the intake and the second is the exhaust valve.

8. Camshafts.

- main function is controlling the opening of the valves. They have lobes, which open each valve.

9. Rocker Arms.

- change the camshaft action to the valves which allows them to open and close when needed [102, 103].

3.2.2 The Piston Engine

The engine is the source of power for the vehicle. Fuel is burned inside the engine to produce heat, the heat causes enlargement of the fuel vapours inside the engine. High pressure is produced in combustion chambers due to burning and expansion. The pressure is converted into motion for moving the vehicle by the engine pistons, connecting rods, and crankshaft.

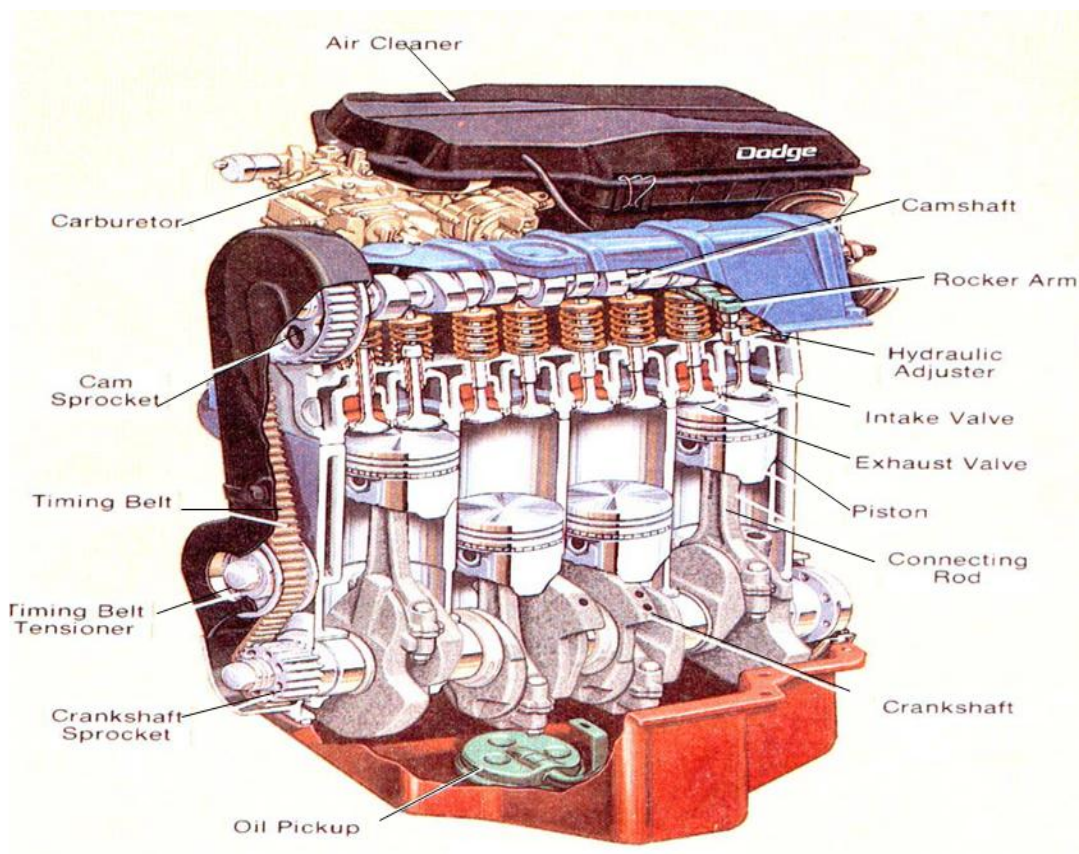


Figure 3.2 Automotive engine [102].

Vehicle engines have more than one piston and cylinder and hence are called multiple cylinder engines. Vehicles usually have 4, 6, or 8 cylinder engines. As the number of the cylinders increases, the smoothness of the engine operation increases due to the decrease of time between each power stroke. The power output of the engine is also increased. Figure 3.2 shows a four stroke engine [104].

3.2.3 Four-Stroke Cycle

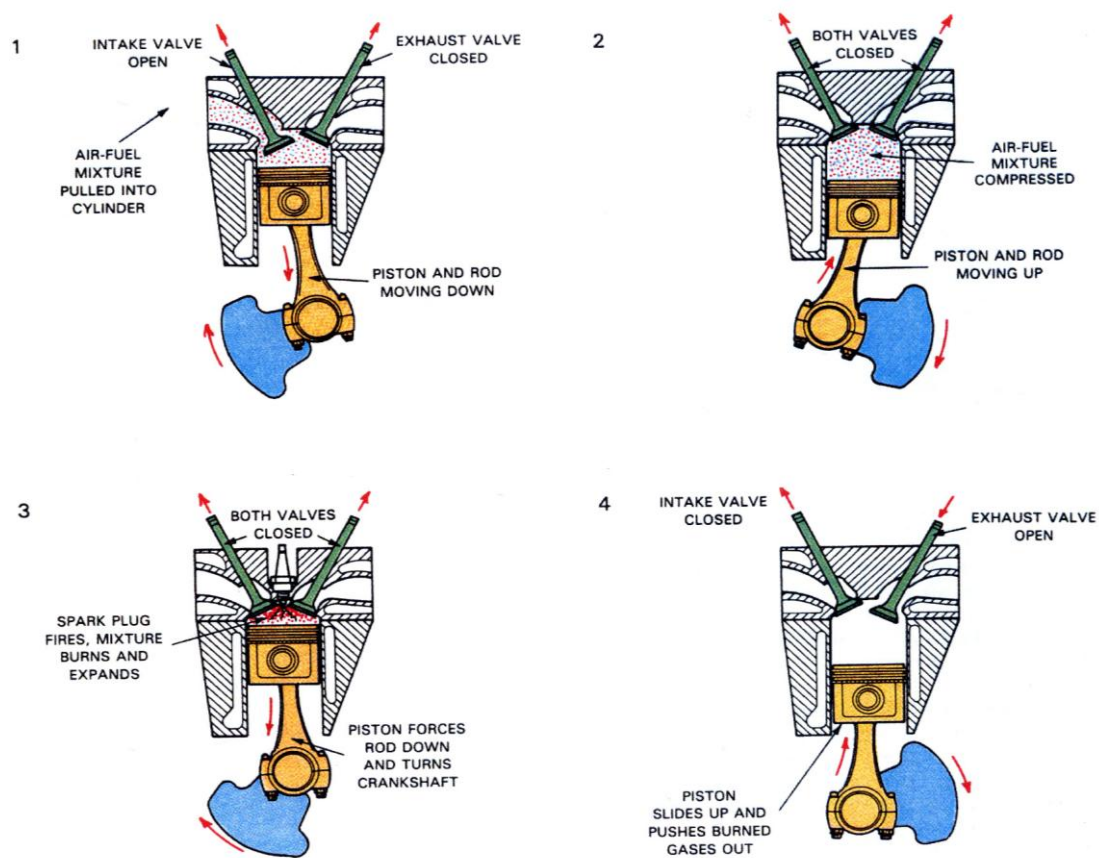


Figure 3.3 Basics of four-stroke cycle [102].

Four-cycle stroke engines are commonly used. One cycle means the up and down movement in a piston stroke. Figure 3.3 describes the function of four strokes and can be summarized as follows:

1. The intake stroke where the piston slides down, allowing the air fuel mixture into the engine combustion chamber.
2. The compression stroke where the piston slides up and squeezes the fuel mixture.
3. The power stroke where the spark plug arcs and ignites the fuel. High pressure develops in the combustion chamber, which pushes the piston and the connecting rod. Hence, the crankshaft will rotate until the next power stroke is due.
4. The exhaust stroke where the piston slides up pushing the burned gases out of the combustion chamber.

This action is repeated to obtain smooth operation of the crankshaft [102, 104].

3.3 Al High Silicon Alloys in Automotive Applications

Lately automotive industries have been scrutinising the performance of engine parts such as connecting rods, rocker arms, cylinders, pistons, valves, and cylinder blocks. (See figure 3.2). They are looking for materials which will fulfil the following requirements:

1. Engine performance: uniform heat distribution around the cylinder and improved fuel economy [105].
2. Light weight: reduction of weight by up to 20-30% [79, 106].
3. Manufacturability: preferably using the same production tools and capable of machining.

4. Abrasion resistance: to allow adequate abrasion resistance without further treatment [105-107].

Performance is the most important criterion for materials selection for a material which is to be used in the automotive industry. However, a compromise between materials selection and cost must be arrived at. Increased performance will lead to increased payload, higher speed, and reduced fuel consumption [4, 108, 109].

The importance of Al-Si-X alloys for automotive application with some engine parts, such as the piston, and the valve retainer connecting rod, has been well established [7, 110-112].

Al-high Si alloys have been used in the automotive industry due to their lightweight and hence reduced fuel consumption. It is possible to tailor the properties (density, strength) of Al-hypereutectic Si alloys by adjusting the volume fraction of Si [4, 106]. Also, Al-high Si alloy components are recyclable at low energy cost [113].

Vehicle pistons undergoing high acceleration and deceleration were the first application of aluminium-high-silicon alloys in the engine block. Lately, Al-high Si alloys have been used as pistons, piston rings and cylinder liners [106, 114, 115]. These materials are used for armoured personnel carriers due to their light weight which allows the vehicle to be easily transported by aircraft. In addition Al-high Si alloys have excellent mechanical properties such as high strength, ductility and fatigue resistance such that it is used in hull construction where it functions as armour [116].

CHAPTER 4

EXPERIMENTAL MATERIALS AND METHODS

4.1 Introduction

This chapter introduces the range of techniques used in the analysis of RSP alloys (ribbons, flakes, as-HIPped and HIPped plus extruded) in this research. The chapter will explain the experimental materials, microstructure analysis, hardness testing, wear testing and experimental setup for degassing. The aim was to build an understanding of the effect of alloying additions and varying degassing conditions on the materials.

4.2 Experimental Materials

The exact compositions of all the materials supplied by RSP are given in table 4.1. Each RSP alloy has different Si, Zr, Cu, Mg, Fe and Ni. The aim is to characterise the material at different alloying element contents. The materials was supplied as either ribbon, flake, as-HIPped material or HIPped plus extruded bar. The material supplier provided the compositions which were obtained by using a specific analysis technique. The technique consists of chemical dissolution, Si determination via gravimetry and determination of metallic elements by XRF using borate fusion, standard DIN ISO 4503. The production route carried out by RSP and Bodycote HIP is summarised in figure 2.10. Flakes produced by melt spinning and chopping (figure 2.9) are placed in a canister and either cold compacted to 60% density (denoted “HF”) or free encapsulated (i.e. without compaction-denoted “FEF”). The material is then hot degassed at either 400°C or 500°C for 3 or 12 hours. The temperature must be high enough to remove water and hydrogen (i.e. above 300°C see section 2.7) but

not so high that excessive coarsening occurs. These degassing conditions were established by “custom and practice” at Bodycote HIP. After degassing the material is HIPped in the same capsule (aluminium) under HIPping conditions which are proprietary to HIP and then the capsule machined off and the HIPped material extruded at RSP.

For some flakes, the cold compaction and degassing were carried out at Leicester to examine these steps in the process in more detail. Further details of the Leicester treatment are given in section 4.6.1.

The flake average thickness is 40 μm , length is 1.4 mm, and width is 1.2 mm. Ribbon average thickness is 40 μm , and width is 2.5 mm. This suggests the ribbons have been split lengthways during chopping.

Piston A390 (Al 17Si 4.5Cu 0.6Mg 0.5Zn wt. %) supplied by KB performance pistons USA, is used for comparison with RSP materials.

The extruded bar has been extruded at RSP following degassing and HIPping at Bodycote HIP. The degassing and HIPping conditions are proprietary to Bodycote HIP.

RSP alloy	BATCH NUMBER	TYPE	COMPOSITION (wt.%)						CONDITION
			Si	Zr	Cu	Mg	Fe	Ni	
446-2	RSA-446-4051-FEF2	Bar	20.3	0.2	2.0	1.5	1.8	1.9	Wear test
446	RSA-446-15058	Flake	20.6	0.2	2.0	1.6	1.9	1.9	
446-3	RSA-446-4047-FEF3	Bar	20.6	0.2	2.0	1.6	1.9	1.9	Wear test
461	RSA-461-16030	Flake	21	0.4	3.9	1.2	2.4	1.4	*Degassed for 3 hours either at 300°C or at 500°C in Leicester. *Degassed at 400°C, 500°C for 3 and 12 hour then HIPped. *Wear test
461	RSA-461-L2	Ribbon	21	0.4	3.9	1.2	2.4	1.4	Degassed for 3 hours either at 300°C or at 500°C in Leicester.
461	RSA-461-5017-HF	Bar	21	0.4	3.9	1.2	2.4	1.4	Wear test
431	RSA-431-14053	Flake	29.8	0.3	1.3	1.4	0.3	0.3	*Degassed at 400°C, 500°C for 3 and 12 hour then HIPped. *Wear
431-6	RSA-431-4046-HF-6	Bar	29.8	0.3	1.3	1.4	0.3	0.3	Wear test
444	RSA-444-4042-FEF	Bar	30.3	0.2	1.5	1.2	1.1	1.4	Wear test
444	RSA-444-15041	Flake	30.3	0.2	1.5	1.2	1.1	1.4	

Table 4.1 Composition of materials supplied by RSP Technology and type of tests carried out.

4.3 Microstructure Analysis

4.3.1 Sample Preparation for Optical and FEGSEM Microscopes

Ribbons and Flakes

Two to three flakes or ribbons from each flake or ribbon batch supplied by RSP were placed in a plastic clip to hold them. Samples were hot mounted (see figure A.2.1, appendix 2 for the ribbon orientation). Mounting was conducted in a Struers LaboPress-3 with a cure temperature of 180°C and a mounting pressure of 15kN. Samples were then ground and polished using a Buehler Metaserv polishing machine. Grinding operations used SiC paper of 240, 400, 600, 800, 1000 and 2500 grit, followed by polishing with 6µm and 3 µm, and a final polish with a colloidal silica Dp-Com. Grinding and polishing times were one minute for each stage. The grinding liquid was water. Polishing liquids were propanol plus water and 6-micron diamond were used for 6 µm polishing, Struers oil based lubricating liquid for diamond polishing (red) plus 3-micron diamond were used for 3 µm polishing and aqueous solution of 0.1 µm colloidal SiO₂ was used for colloidal silica polishing . After each grinding stage, specimens were then washed with water and soap. Then, after each polishing stage, specimens were cleaned using a Struers Ultrasonic bath for 2 minutes to remove any dirt or particles remaining on the polished surface. Specimens were washed with water and soap. Finally, they were washed with alcohol to remove any final dirt and water residue. Specimens were then hot dried.

As-HIPped Billets and Extruded Bars

For microstructural analysis of as-HIPped billets and extruded bar a 3 mm thickness slice was cut from each sample. Samples were hot mounted. Mounting was conducted using the same regime as for ribbons and flakes. Samples were ground and polished using the Struers Tegra Pol-21 process sequence as shown in table 4.2. After

each grinding stage, specimens were then washed with water and soap. Then, after each polishing stage, specimens were cleaned using a Struers Ultrasonic bath for 2 minutes to remove any dirt or particles remaining on the polished surface. Specimens were then washed with water and soap. Finally, they were washed with alcohol to remove any final dirt and water residue. Specimens were then hot dried.

Grinding and polishing times of ribbons and flakes are less than for as-HIPped billets and extruded bars because ribbons and flakes are thin and fragile.

Etching: Ribbon, flake, as-HIPped and HIPped plus extruded bar specimens were electrolytically etched for 15-20 seconds in 50 % vol. HBF_4 in water at 10 volts.

Step	Surface	Disc rotation speed (rpm)	Force (N)	Suspension	Lubricant	Time (min.)
1	SiC-Paper #220	300	20		Water	10
2	SiC-Paper #500	300	20		Water	5
3	SiC-Paper #800	300	20		Water	5
4	SiC-Paper#1000	300	20		Water	2
5	DP-largo 3 μm	150	15	DiaPro Allegro Largo (9 μm)		10
6	DP-Mol 3 μm	150	15			10
7	DP-Nap 1 μm	150	15	DP, A1 μm	Dp Blue	10

Table 4.2 Sample preparation method for as-HIPped billets and extruded bars.

4.3.2 Sample Preparation for TEM

Small specimens (13x18mm) were sliced from each as-HIPped billet and extruded bar. Samples were then hot mounted. Sample thickness was reduced from 500 μm to 100 μm using a mechanical grinding machine and a final thickness of 25 μm was achieved using the Gatan 623 Disc Grinder (see figure A 2.2, appendix 2). However, flake and ribbon samples were exempted from the above procedure since their original thickness was 40 μm and further reduction was impossible to achieve due to the brittleness of the material. Thin foil samples were prepared in the Precision Ion Polishing System (PIPS) using argon ions at an energy of 3-5 keV. The bombardment angles were first set at 4° for 1hr, and reduced to a smaller angle of 3° to finish until penetration was visible. The time required was 2 hrs.

4.3.3 Analysis Techniques

4.3.3.1 Optical Microscopy and Particle Size Measurement

Optical microscopy was conducted using an Olympus Vanox. The highest magnification 100x objective lens as used in this project. Some Si particle size measurement was carried out on optical micrographs (see section 4.3.3.4). Using image analysis in the FEGSEM was explored but the clustering of the Si particles made it difficult to distinguish separate particles. Image analysis was carried out on optical micrographs as described in section 4.3.3.4

4.3.3.2 Field Emission Gun Scanning Electron Microscopy

Field emission gun scanning electron microscopy (FEGSEM) was conducted using an FEI Sirion 200 FEGSEM equipped with electron backscattered diffraction

(EBSD). The operating voltage used was 20 kV and the working distance was 5 mm. Both backscattered and secondary electron techniques were used. Quantitative analysis was carried out to determine the chemical composition of each sample with Energy Dispersive X-Ray Analysis Priston Gamma Technology Avalon (EDX).

4.3.3.3 Transmission Electron Microscopy

Transmission electron microscopy (TEM) was carried out using a JEM-2100 LaB₆ TEM at 200keV and fitted with a PGT Energy Dispersive X-ray analysis system (EDX).

4.3.3.4 Image Analysis

Image analysis is conducted using Image-Pro plus software which is used to measure length, roundness, area and holes. The software has a wide range of measurements and is capable of filtering unwanted objects. It is also capable of using colours to spread and measure objects in colour images. The software will export measurements for statistical analysis and spreadsheets. It works as follow:

- i. Load the micrograph into the software.
- ii. Calibrate.
- iii. Set the area of interest.
- iv. Adjust the histogram.
- v. Finally, count the size of the desired particles.

To find the silicon particle size the maximum and minimum ferret of each particle identified as Si was measured. The mean for each particle was then found by the software. The mean for all particles was then calculated by the software. The use of ferrets does mean that “long” silicon particles (i.e. where several particles have

joined up to form a network) tend to skew the mean to a higher value than it would had if an equivalent spherical diameter had been taken.

4.3.3.5 X-Ray Diffraction

X-Ray diffractions (XRD) were carried out in the Geology Department. The (XRD) data were obtained by using a Philips PW 1716 diffractometer with a PW 1729 generator and a PW 1050/25 detector.

Samples were run from 4 to 90 degrees 2 theta with a step size of 0.0204.

4.4 Hardness Testing

Hardness tests were conducted to study the hardness of ribbons, as-HIPped billets and HIPped plus extruded bars. Measurements were carried out on a macro Vickers Hardness Tester MV-1S with a load range from 0.3 to 20 kgf. The load applied was 1 kgf for ribbons due to their small thickness (i.e. 40 μm) and 10 kgf for billets and bars. The load time was 10 sec. and the load speed was 50 $\mu\text{m}/\text{sec}$. Calibration of the hardness machine was carried out according to BS427. Hardness number is taken as an average of 10 readings.

4.5 Wear Testing

Wear properties of Al-Si alloys have been studied under dry sliding reciprocating multi-pass scratch testing conditions against a steel ball bearing ($r = 2.5\text{mm}$). This work is aimed at understanding the effect of the main alloying elements (i.e. Si, Fe and Cu) on dry sliding wear of rapidly solidified Al-high Si alloys. Analyses and observation of worn surfaces and the wear mechanism of these alloys will be

discussed. The tests were conducted using a reciprocating sliding wear tester ST-200 manufactured by Teer Coatings Ltd.

The ball bearing was cleaned with alcohol to remove any dirt and oil residue. After each test a new ball bearing was used.

4.5.1 Test Parameters

Test Fixed Parameters

Table displacement = 5 mm

Table speed = 300 mm/min

Number of cycles = 1000 cycle

Test Variables

Load = 10 and 100N

These parameters were chosen due to the fact that the test is carried out on reciprocating sliding wear tester which required small a sample approximately 10 x 10 mm. The table displacement is 5 mm which will give 10 mm per cycle. To achieve a reasonable number of results in the time available with a variety of samples, 1000 cycles was chosen as the duration. This gave 10 m sliding distance in approximately 35 minutes. Two loads were used one high (100N) and one low (10N). A load of 10 N generated a measurable wear volume.

4.5.2 Materials

The full table of materials for wear testing is given earlier in table 4.1. Hence, the samples are summarised into two categories:

- 1) RSP extruded bars table 4.3.

2) Degassed and HIPped samples table 4.4.

RSP alloy	Sample No Extruded bars	Composition
446-2	RSA-446-4051-FEF2	Al 20.3Si 2Cu 1.5Mg 1.8Fe 1.9Ni 0.2Zr
446-3	RSA-446-4047-FEF3	Al 20.6Si 2Cu 1.6Mg 1.9Fe 1.9Ni 0.2Zr
461	RSA- 461-5017-HF	Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr
431-6	RSA-431-4046-HF-6	Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr
444	RSA-444-4042-FEF	Al 30.3Si 1.5Cu 1.2Mg 1.1Fe 1.4Ni 0.2Zr

Table 4.3 RSP extruded bars.

Billet sample number (degassed and HIPped)	Degassing temperature (°C)	Degassing time (hr)
No 461	400	3 and 12
No 461	500	12
No 431	400	3 and 12
No 431	500	12

Table 4.4 Degassed and HIPped samples.

4.5.3 Sample Preparation for Wear Testing

Wear test samples in the form of 4 mm x 10 mm x 10 mm blocks were cut from each bar and billet. Samples were then hot mounted and ground. Grinding operations used

SiC paper of 240, 400, 600, 800, 1000 grit. Grinding time was two minutes. The grinding liquid was water. After each grinding stage, specimens were washed with water and soap. Finally, they were washed with alcohol to remove any final dirt and water residue. Specimens were then hot dried. In general the specimens were thoroughly cleaned prior to conducting the wear tests. Debris was collected using sticky holder after the wear tests and finally the specimens were cleaned.

Wear volume measurements

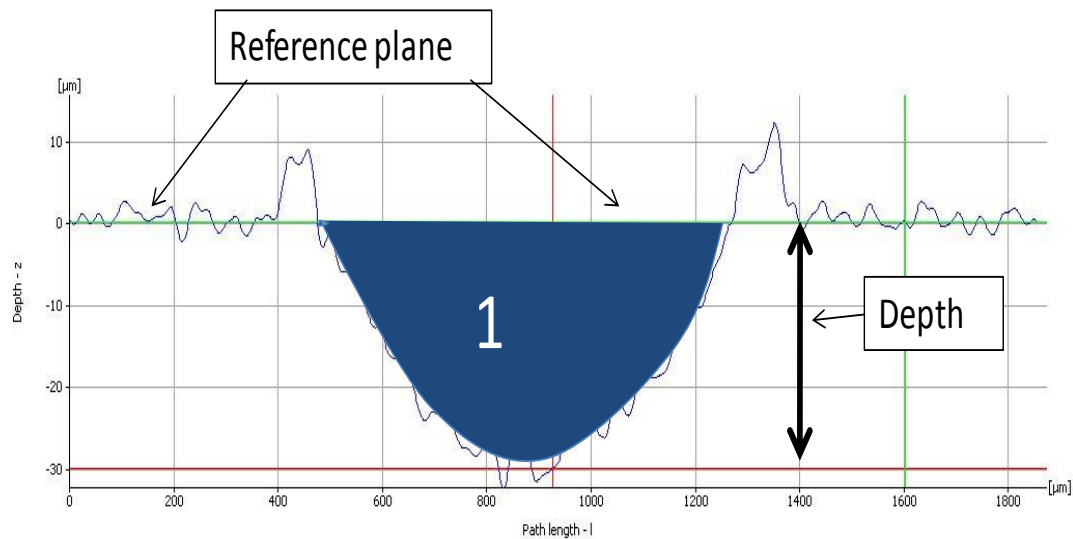


Figure 4.5.3.1 illustration of wear volume measurements.

The wear tracks were characterised in Geology Department. The wear volume was obtained by using an Alicona IFM infinite focus microscope. The software works as follows (see figure 4.5.3.1):

- I. Assigning a reference plane by selecting arbitrary points around the wear track which is on the top of the sample.

- II. Calculating the area of shape number 1 which is the wear track.
- III. Measuring the depth.
- IV. Finally, calculating the volume by adding up all of these areas along the length of the track.

4.6 Experimental Set Up for Degassing

Alloy 461 (see table 4.1) ribbons and flakes were degassed at the University of Leicester and they were coded as UOL (i.e. UOL 461). The degassing temperature was either 300°C or 500°C and the degassing time 3 hours. Table 4.4 shows the materials that were degassed at Bodycote HIP and they were coded as BDC (i.e. BDC 461). At the Bodycote HIP the degassing was for either 3 hours or 12 hours and the temperature was either 400°C or 500°C. The Bodycote HIP degassed samples were then HIPped at Bodycote HIP so as to characterise material at this stage in the process and clarify the effect of degassing conditions.

4.6.1 Degassing and Cold Compaction at the University of Leicester

Ten canisters of approximately 38Ø x 61 mm were made out of aluminium tube with wall thickness of 2.9 mm (see figure A.2.3, appendix 2). An aluminium tube of approximately 5 mm inner diameter and 50 cm length was inserted in the middle of the top cover of the canisters. Welding was performed around the tube and the canister cover to prevent any leakage. Then the aluminium tube was connected to a rotary vacuum pump. Ribbons or flakes were placed inside the canisters, the aluminium cover put in place, and welding performed around the cover and the canister wall to prevent any leakage.

An electrical furnace was then used. Temperature was controlled with a Chromel/alumel (K type) thermocouple. Two thermocouples were used, one was built in the furnace and the second thermocouple was installed outside the canister (see figure A.2.3, appendix 2). Initial process variables were as followed:

a) Degassing temperatures = 300 or 500°C. These values were chosen to represent extremes because 300°C is the minimum temperature expected from experience to give adequate degassing (see section 2.7) and 500°C is the temperature which is close to the maximum temperature that can be applied without melting phases.

b) Degassing time = 3 hrs

The colour of as-received ribbons is shiny. It was observed that the colour of ribbons was no longer shiny after degassing. Also, it was noticed that the ductility of ribbons decreases as the degassing temperatures increases. The ribbons became friable after degassing at 500°C.

At RSP some flakes are cold compacted prior to degassing (the “HF” route). To mimic the effect of this at Leicester, prior to degassing, RSP 461 flakes were poured into a cylindrical steel mould. The inner diameter of the mould is 12.56mm and the length is 75.12mm. The outer diameter is 75.12mm and the outside length is 100.12mm. The mould has two pistons: one is fixed (stationary L= 25mm) and the second is moving (see figure A.3.1, appendix 3). A force of approximately 93kN was used to force the moving piston to compact the flakes. Cylindrical billets (green body) of about 9mm diameter and 75mm length were obtained. Compaction at the University of Leicester was carried out using a hydraulic press.

4.6.2 Degassing and HIPping at Bodycote

Specimens were degassed in a process similar to that used by the University of Leicester previously described. Bodycote canisters are shown in figure A.2.4, appendix 2. Degassing conditions are given in table 4.5.

Temp. (°C)	Time (hrs)	Alloy No.431	Alloy No.461
400	3, 12	Degassed Degassed+ HIPped	Degassed Degassed+ HIPped
500	12	Degassed, Degassed+ HIPped	Degassed Degassed+ HIPped

Table 4.5 Materials treated at Bodycote and their conditions. 3, 12 means one sample is treated for 3 hours and another for 12 hours. Degassed, degassed + HIPped means one sample is degassed and one sample is degassed then HIPped.

CHAPTER 5

Experimental Results

5.1 Introduction

This chapter represents the experimental results. Section 5.2 is flake and ribbon microstructure as-received. Section 5.3 is cold compaction and degassing at the University of Leicester. Section 5.4 is mapping and line scans of as-received and degassed ribbon. Section 5.5 is degassing and HIPping at Bodycote. Section 5.6 is bar microstructure as-received. Section 5.7 is Si particle measurements. Section 5.8 is X-Ray diffraction (XRD). Section 5.9 is hardness testing. Section 5.10 is wear and finally section 5.11 is comparison of wear rates with other sources.

5.2 Flake and Ribbon Microstructure As-Received

Figure 5.2.1 shows optical micrographs of sample 431 flake etched and 461 ribbon. Figure 5.2.1a shows a fine grained aluminium matrix with clusters of silicon particles dispersed within the microstructure. Figure 5.2.1b shows a ribbon at low magnification in end down (transverse) cross section. It can be clearly seen that there are two distinct zones in the microstructure which are zone 1 wheel side and zone 2 air side. Based on evidence from literature it has been assumed that the straight line (i.e. 3) in the cross section is the wheel side. Figure 5.2.2 shows optical and FEGSEM micrographs of sample 461 ribbon. Figure 5.2.3 shows TEM micrographs of sample 461 ribbon and points 1, 2, 3, and 4 were analysed by EDX (figure 5.2.4). Figure 5.2.5 shows FEGSEM backscattered micrograph of sample 461 ribbon. Figure 5.2.5 is taken at point 3 of figure 5.2.2 which is very close to the wheel side. Points 1 and 2 on the micrograph were analysed by EDX (figures 5.2.6 and 5.2.7). Figure 5.2.8 shows FEGSEM backscattered micrographs of sample 461. Based on evidence from

literature it has been assumed that the fine zone is wheel side and coarse side is air side in cross section. Figure 5.2.9 shows an EDX spectrum for zone 1 in figure 5.2.8. Similarly, figure 5.2.10 shows an EDX spectrum for zone 2 in figure 5.2.8.

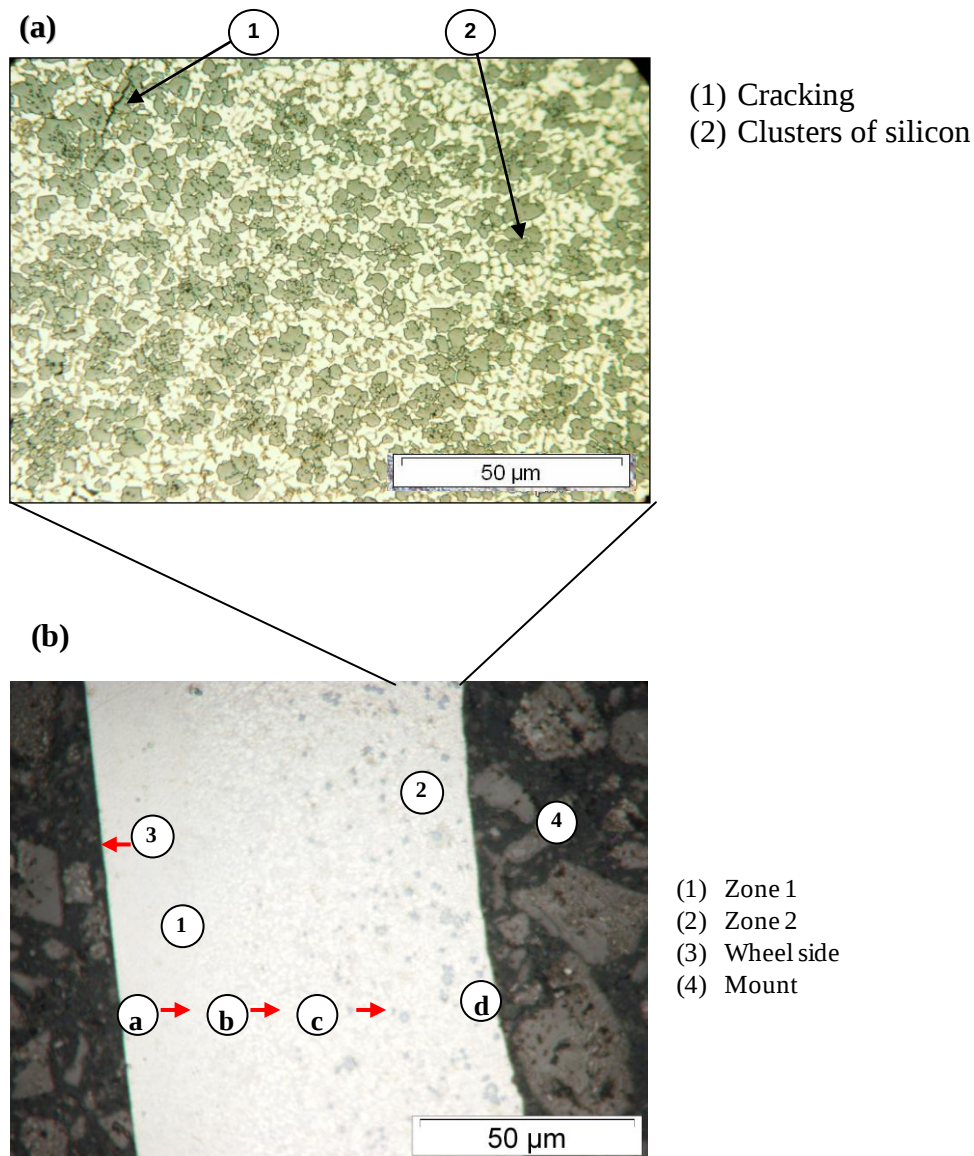


Figure 5.2.1 (a) Sample 431-14053 flake optical micrograph etched (according to table 4.1 Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr), air side longitudinal cross section, (b) Sample 461-L2 ribbon optical micrograph (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) ribbon, drum side. As indicated in the figure (b) solidification starts at (a) and ends at (d) .

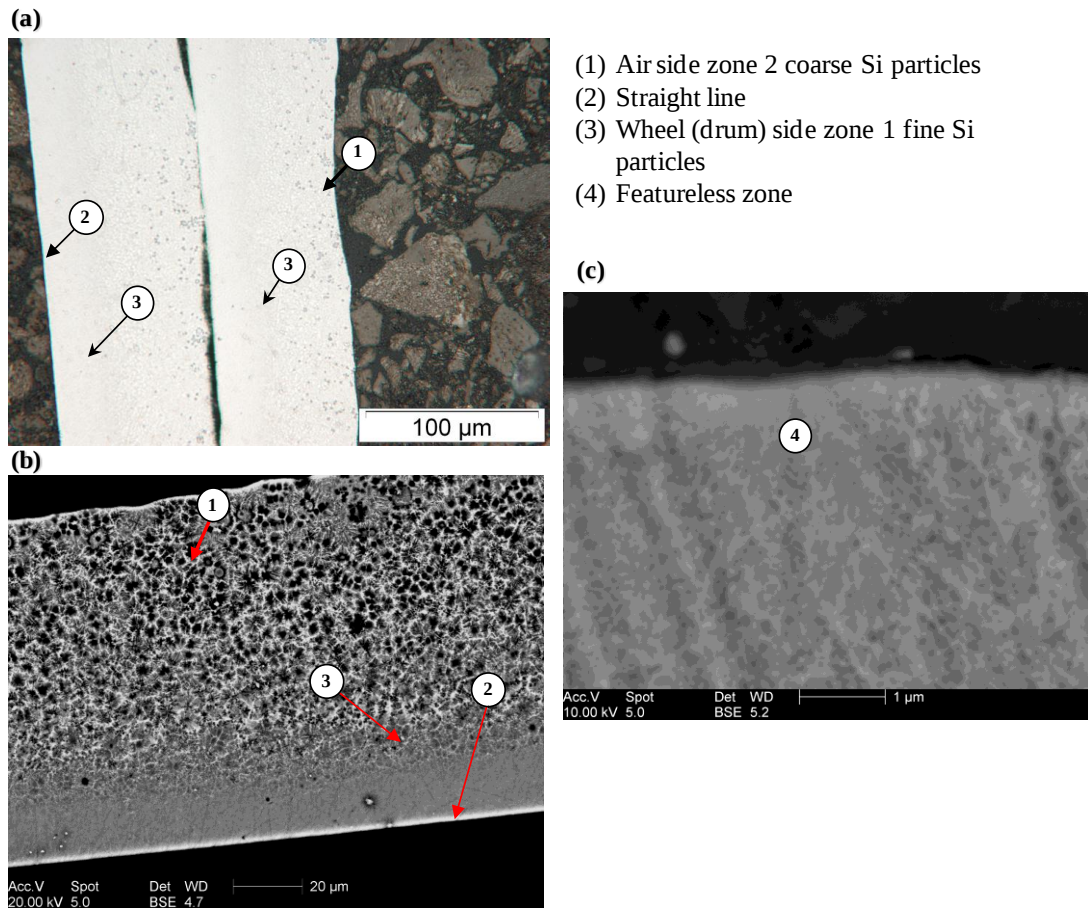
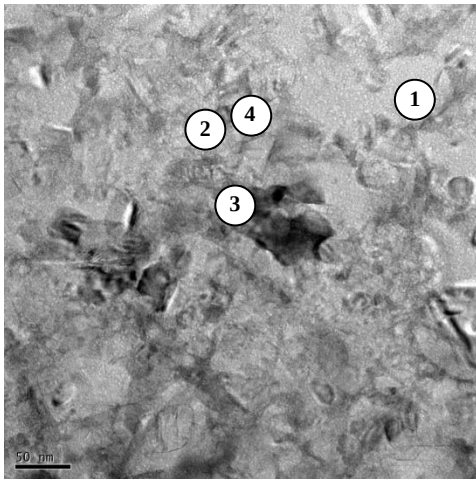
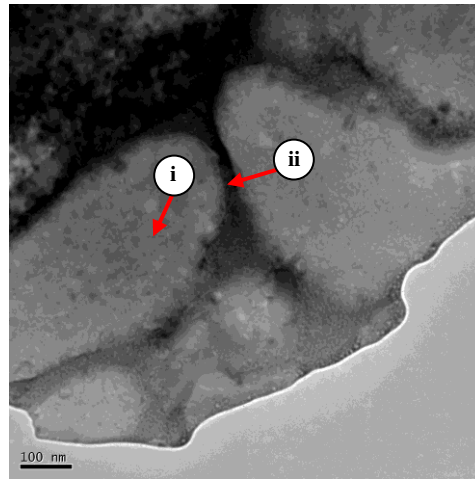


Figure 5.2.2 Sample 461 ribbon unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) (a) Optical micrograph (b) FEGSEM backscattered micrograph (c) FEGSEM backscattered micrograph of featureless zone.

(a)



(b)



(i) Si (ii) Aluminium

Figure 5.2.3 TEM micrograph sample 461 ribbon unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr). Points 1,2,3,4 and 2 were analysed by EDX (see figure 5.2.4)

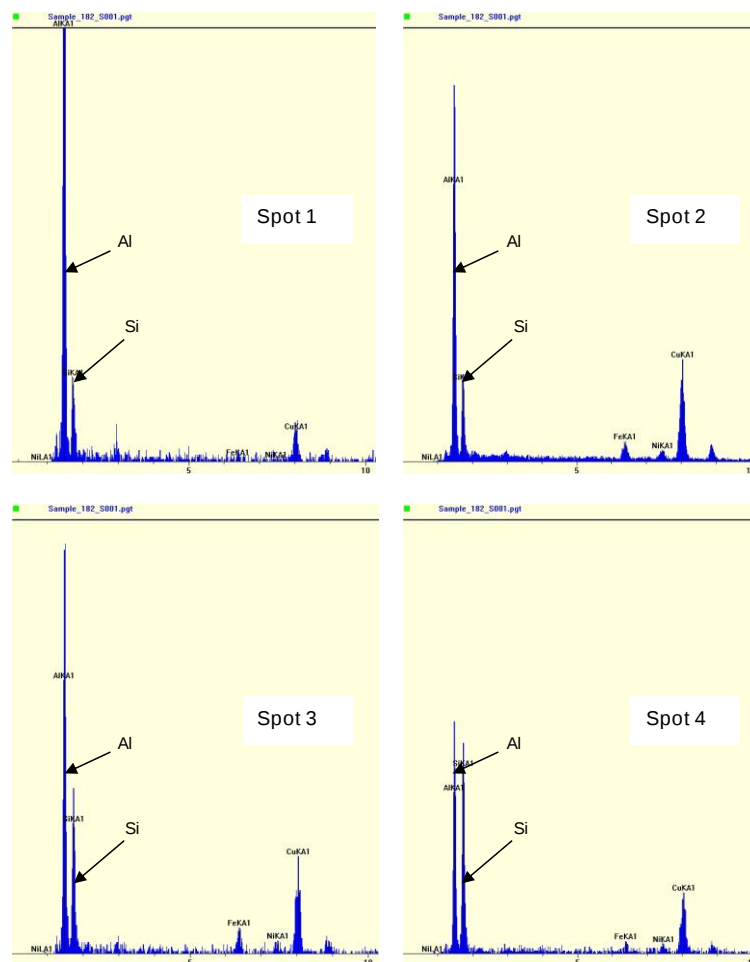


Figure 5.2.4 EDX spectrum for points 1. 2. 3 and 4 in figure 5.2.3 showing relatively, high Al in points 1 and 2. High Al and Si for points 3 and 4.

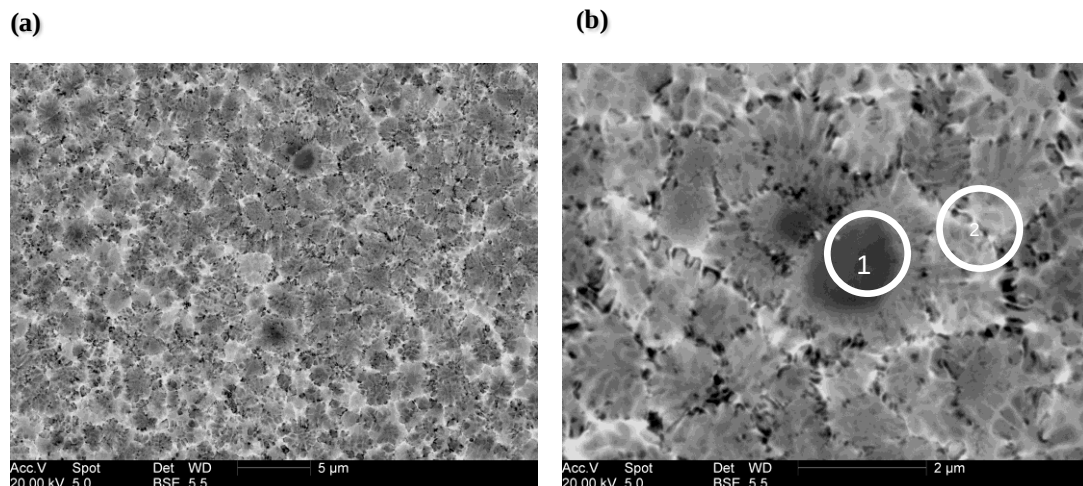


Figure 5.2.5 Sample 461 ribbon FEGSEM backscattered micrograph (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr). Points 1 and 2 were analysed by EDX (see figures 5.2.6 and 5.2.7). This micrograph is taken at point 3 in figure 5.2.3.

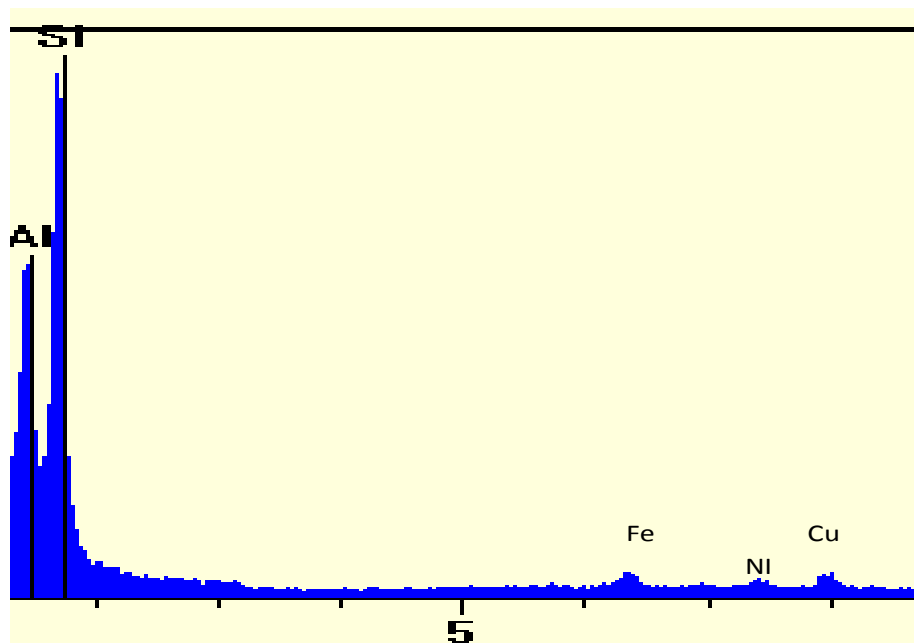


Figure 5.2.6 EDX spectrum for point 1 in fig. 5.2.5 showing relatively high Si.

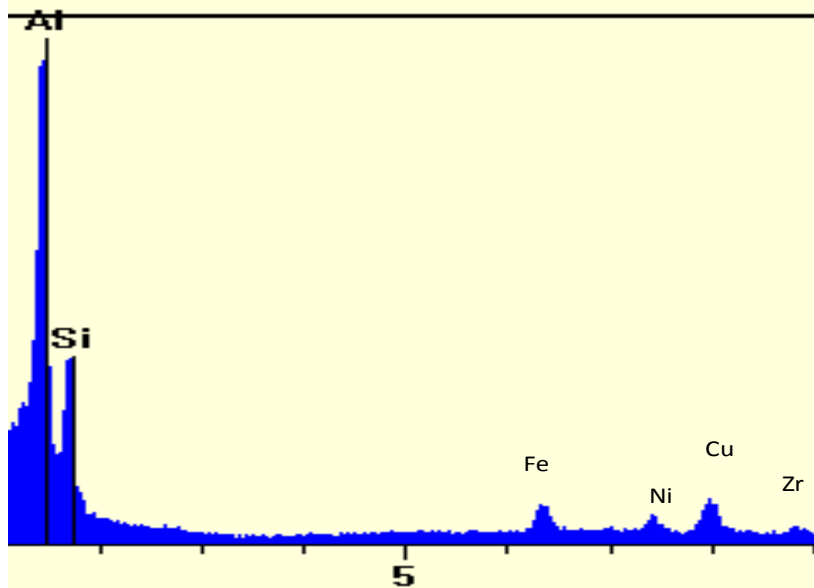
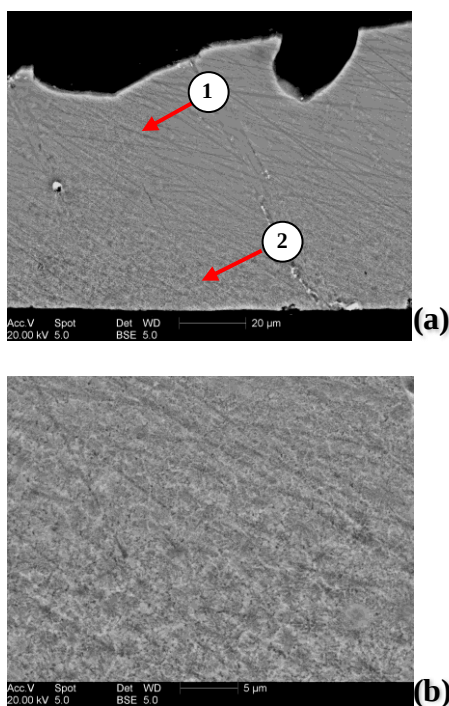


Figure 5.2.7 EDX spectrum for point 2 in fig. 5.5 showing relatively high Al.



- (1) Zone 1 wheel side. Si = 23.81wt. %
(see quantitative analysis for zone 1 in appendix 4.
- (2) Zone 2 air side. Si = 22.60 wt. %
(see quantitative analysis for zone 1 in appendix 4.
- (b) Air side

Figure 5.2.8 FEGSEM backscattered micrographs sample 461-L2 ribbon unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) (a) Overall view of ribbon (b) Micrograph of zone 1 in fig.5.2.8 a (c) Micrograph of zone 2 in fig.5.2.8.

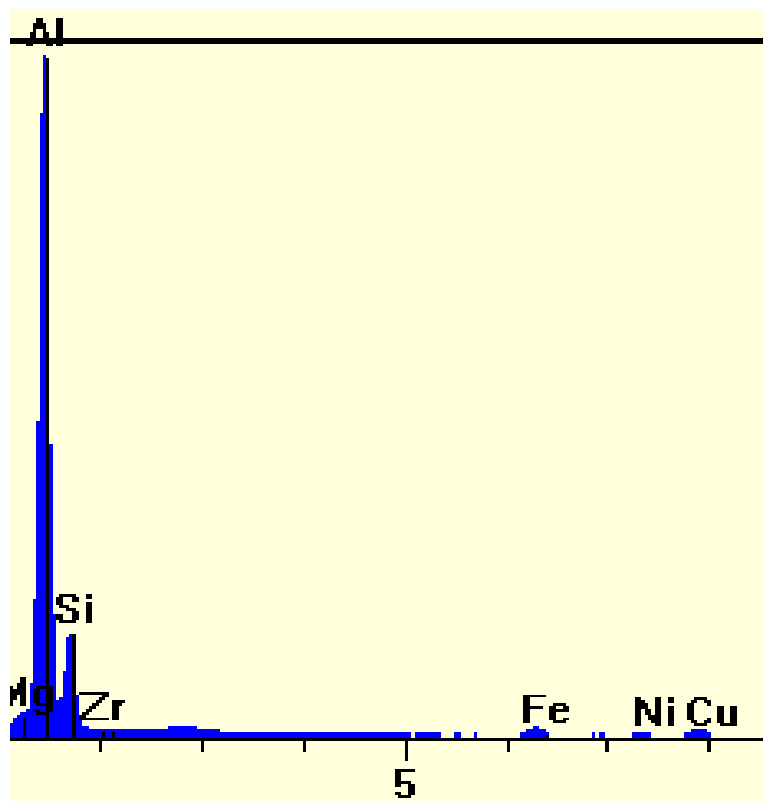


Figure 5.2.9 EDX spectrum for zone 1 in fig. 5.2.8

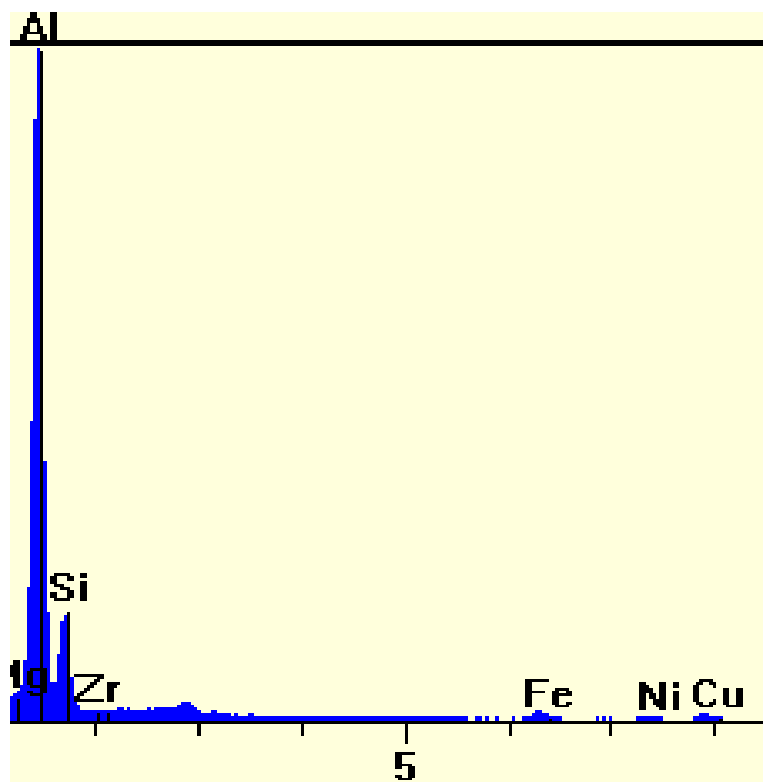


Figure 5.2.10 EDX spectrum for zone 2 in fig. 5.2.8

5.3 Degassing and Cold Compaction at the University of Leicester

Figure 5.3.1 shows optical micrographs of sample 461 ribbon as-received. Relatively large (coarse) particles sizes were measured to give an indication of particle size. The results are given in table 5.3.1. Two measurements were taken on each particle for accuracy (hence two measurements are shown against each particle on the micrograph). Figure 5.3.2 shows optical micrographs of sample 461 ribbon degassed at 500°C for 3 hrs. Figure 5.3.3 shows optical micrographs of sample 461 ribbon degassed at 300°C for 3 hrs. Table 5.3.1 shows large Si particle diameter measurements at different degassing conditions. Figure 5.3.4 shows optical micrographs of sample UOL 461 flake cold compacted. In figure 5.3.4(b) the transition between coarse and fine particles is quite abrupt. Figure 5.3.5 shows optical micrographs of the same sample after cold compaction and degassing at 500°C for 3 hrs.

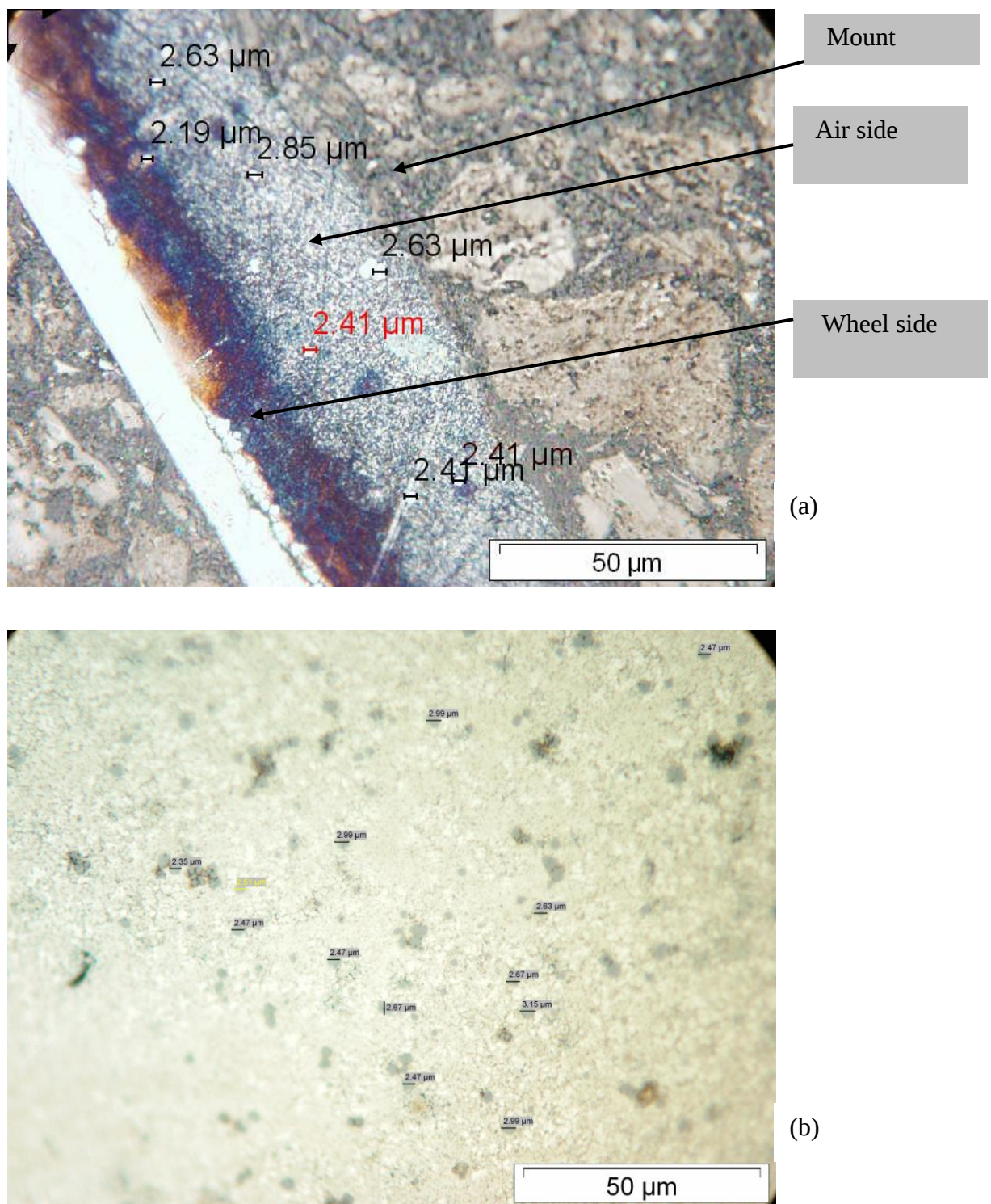
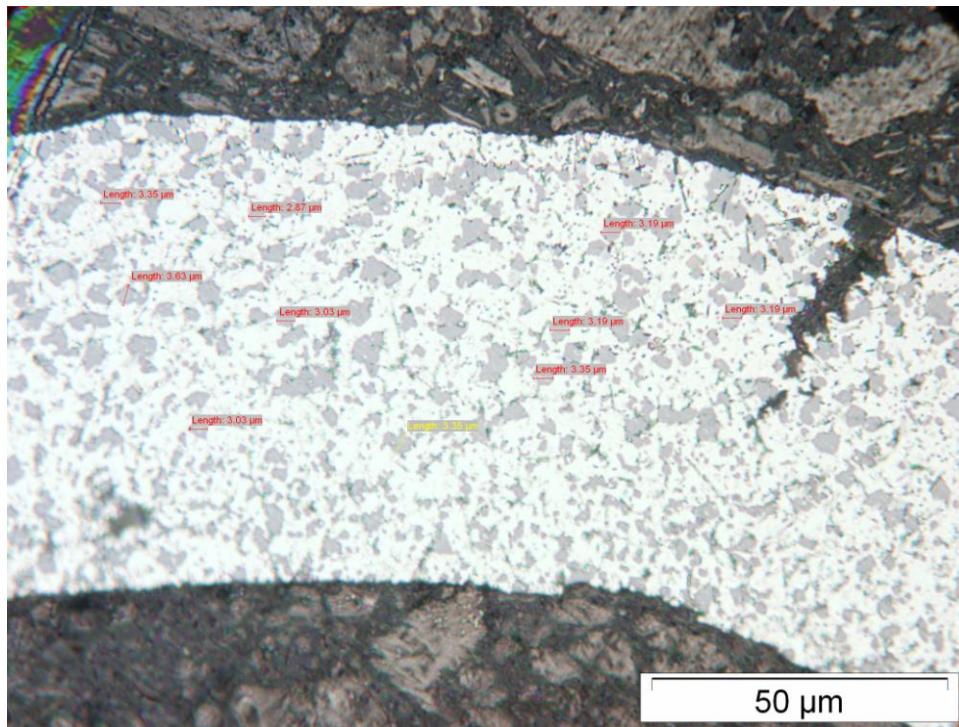
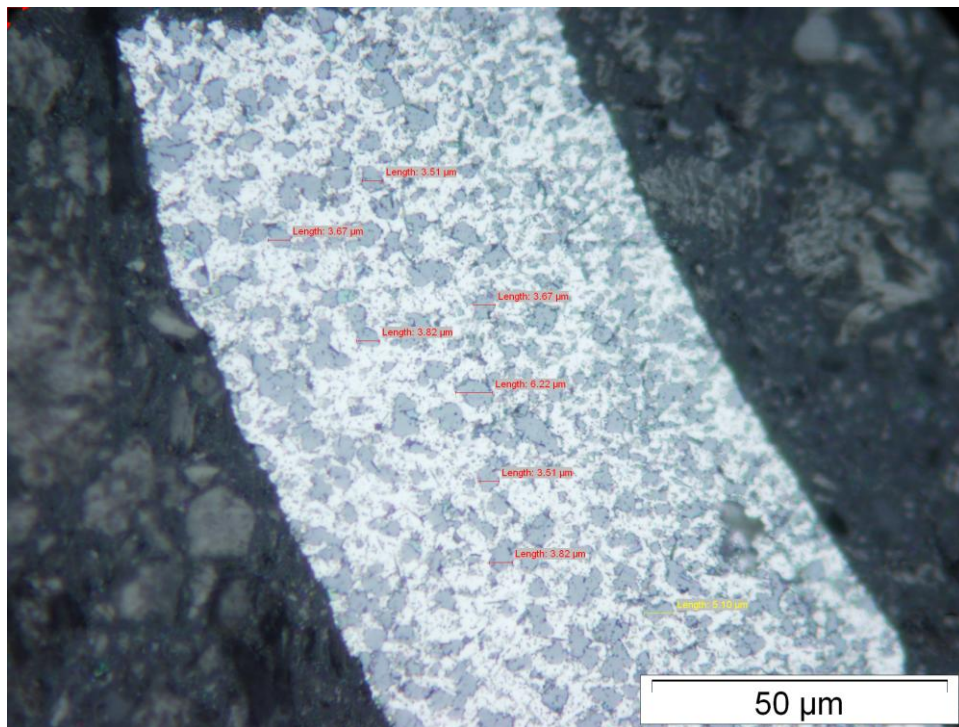


Figure 5.3.1 Optical micrographs of sample 461 ribbon (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) as-received (a) End down (transverse see fig.A.2.1 appendix 2) cross section unetched (b) Longitudinal cross section etched.

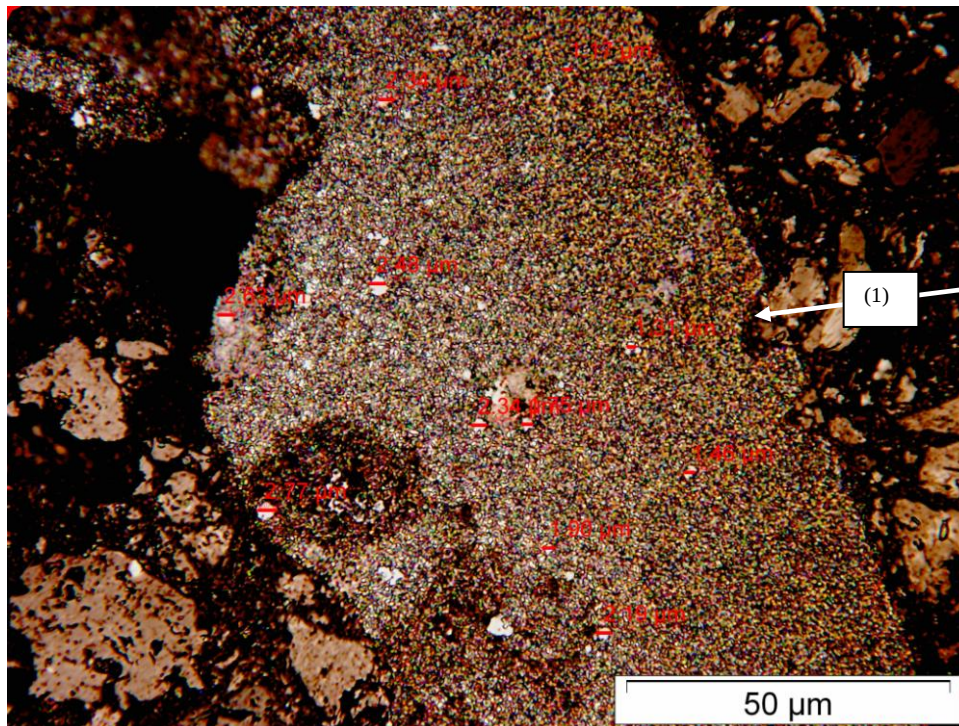


(b)



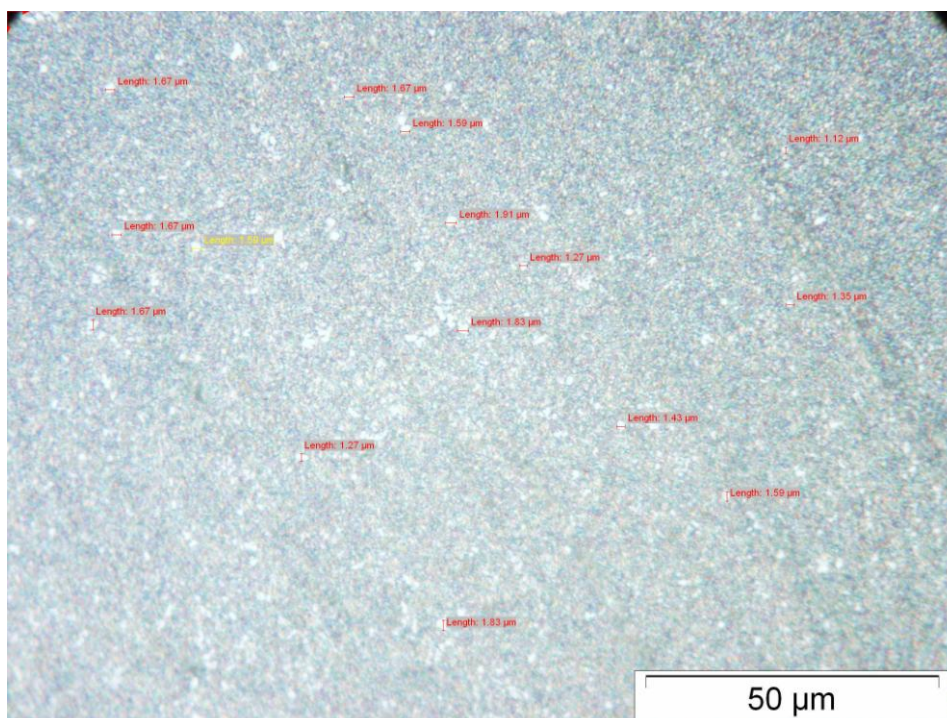
(b)

Figure 5.3.2 Optical micrographs of sample 461 ribbon (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 500°C for 3 hrs. End down (transverse see fig.A.2.1 appendix 2) cross section. (a) Unetched (b) Etched



(1)
Edges of ribbon often break up during preparation because of the fragility of the

(a)



(b)

Figure 5.3.3 Optical micrographs of sample 461 ribbon (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 300°C for 3 hrs. (a) End down (transverse see fig. A.2.1 appendix 2) cross section unetched (b) Longitudinal cross section etched.

Sample No.	Measurement number	Condition	Typical large Si particle (μm)	Mean (μm)	S.D. (μm)
461 Ribbon	1	As-received end down cross section (air side)	2.6, 2.2, 2.6, 2.4, 2.4, 2.4, 2.9	2.5	± 0.2
	2	As-received longitudinal cross section (thought to be air side)	2.9, 2.5, 2.7, 2.5, 2.6, 2.4, 3.0	2.6	± 0.2
	3	Degassed @ 300 °C for 3 hrs longitudinal cross section(thought to be wheel side)	1.7, 1.6, 1.9, 1.8, 1.3, 1.8, 1.4	1.6 *	± 0.2
	4	Degassed @ 300 °C for 3 hrs end down cross section (air side)	2.8, 2.3, 2.5, 2.2, 2.5, 2.6, 2.4	2.5	± 0.2
	5	Degassed @ 500 °C for 3 hrs end down cross section (air side)	3.4, 2.9, 3.6, 3.0, 3.0, 3.4, 3.4	3.2	± 0.3

***Zone (1) wheel side hence smaller than in the as-received.**

Table 5.3.1 Silicon particle diameter measurement. Typical large particles in the field of view.

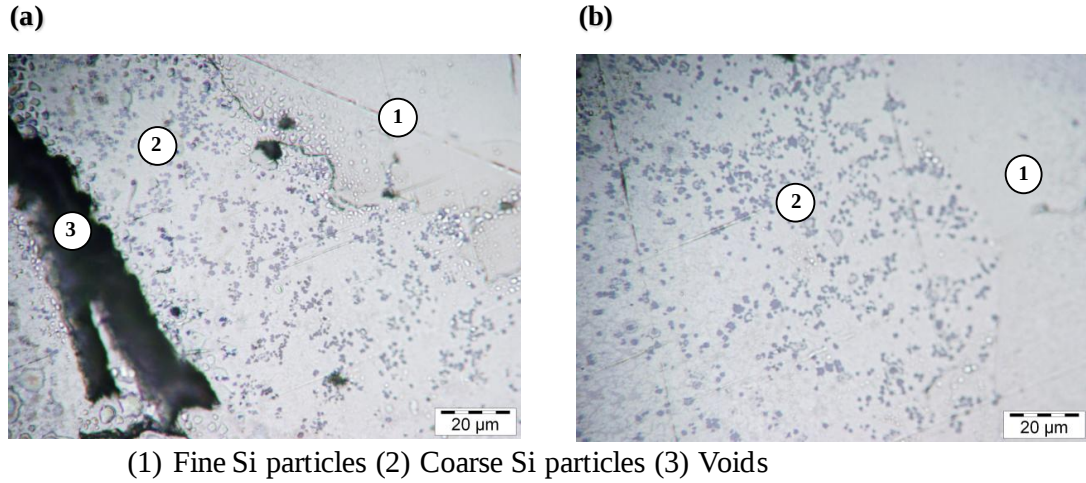


Figure 5.3.4 Optical micrographs of sample UOL 461flake (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) unetched cold compacted (i.e. after cold compaction but before degassing) at UOL. (a) Porosity. (b) Coarse and fine particles.

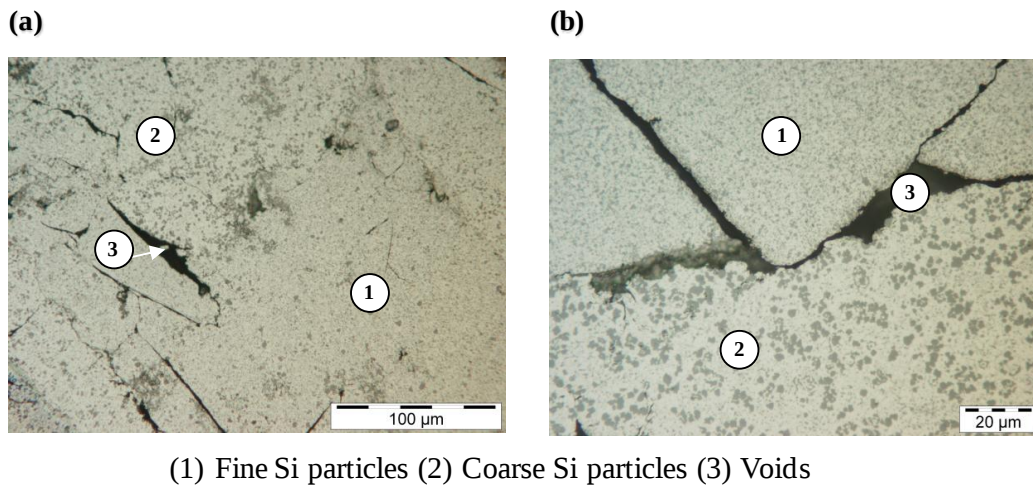


Figure 5.3.5 Optical micrographs of sample UOL 461 (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) unetched cold compacted and degassed at 500°C for 3 hrs at UOL. (a) Porosity. (b) Porosity, coarse and very fine particles.

5.4 Mapping and Line Scans of As-Received and Degassed Ribbon

Figure 5.4.1 show FEGSEM elemental mapping of sample 461 ribbon degassed at 500°C for 3hrs. Figure 5.4.2 shows line scan micrograph of sample 461 ribbon. Figure 5.4.2a is for an as-received sample and figure 5.4.2b degassed at UOL at 500°C for 3hrs. Figures 5.4.2a and 5.4.2b were put on the same page for comparison.

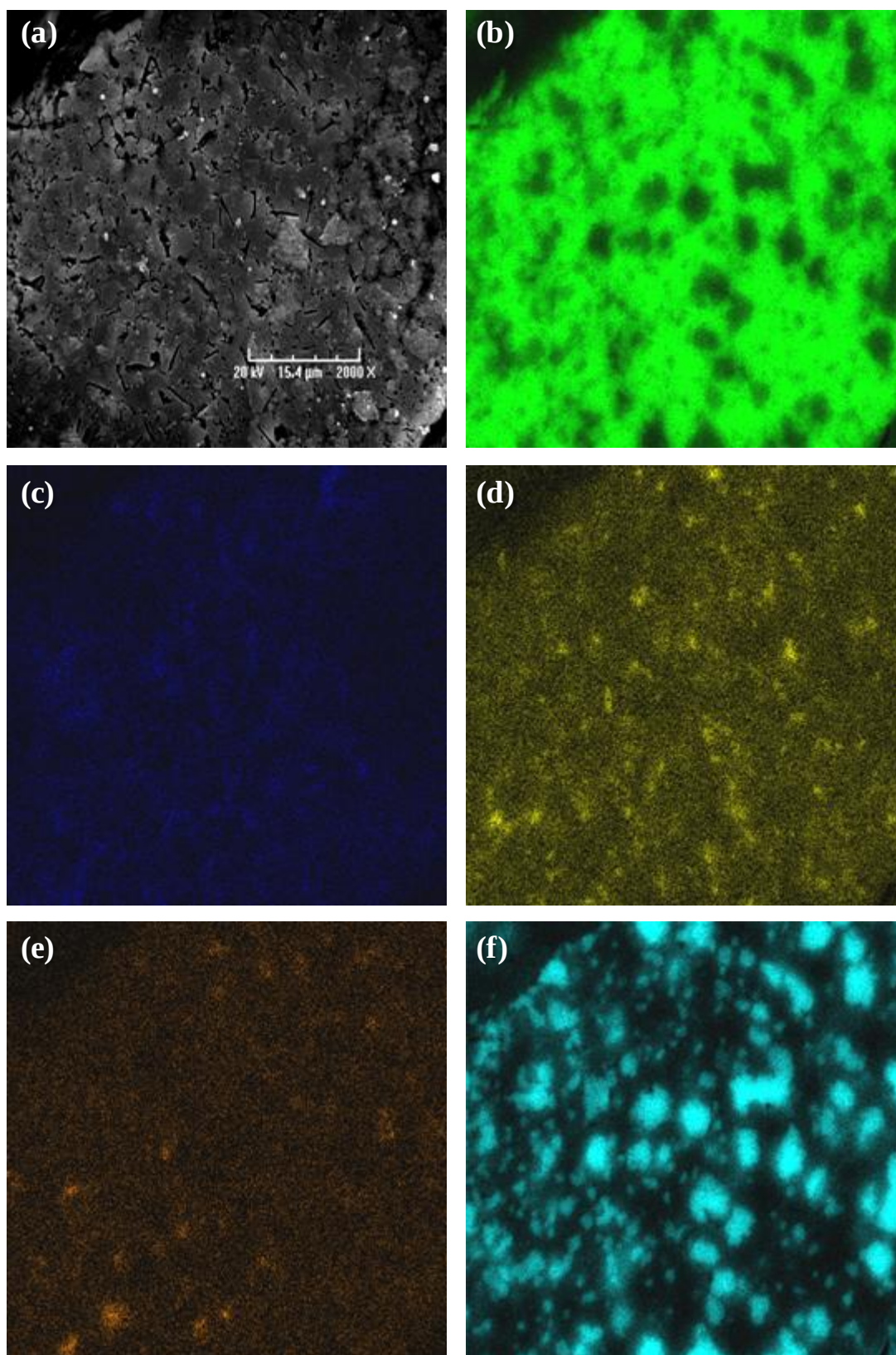


Figure 5.4.1 FEGSEM micrograph elemental mapping of sample 461-L2 ribbon (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 500°C for 3 hrs. WD = 5 mm, V = 20 kV, t = 45 min. (a) Secondary electron overall view of ribbon (b) Al (c) Cu (d) Fe (e)Mg (f) Si element.

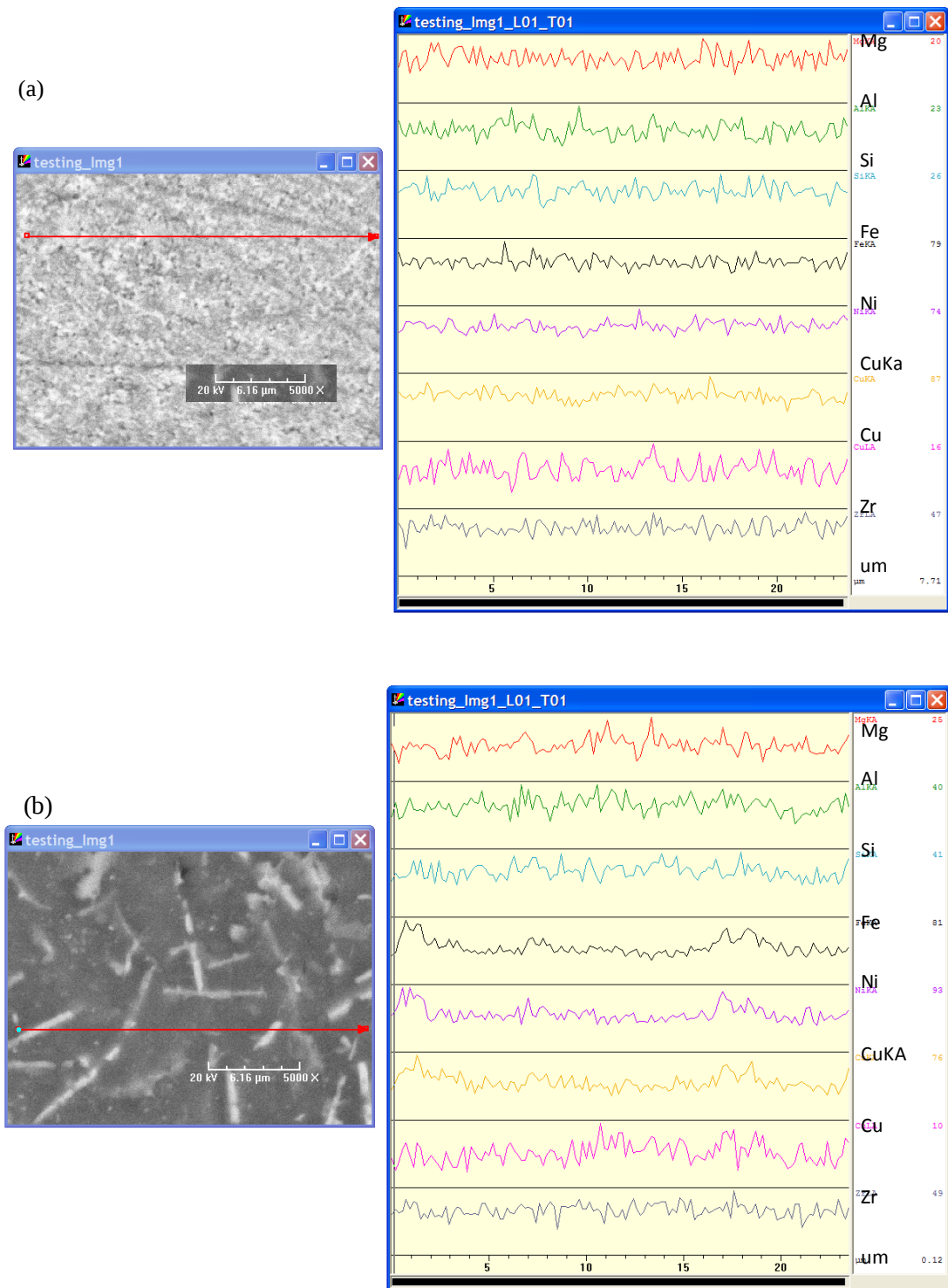


Figure 5.4.2 Line scan micrographs of sample 461 ribbon scan duration 200 sec. (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr). (a) As-received (b) degassed at UOL at 500 °C for 3hrs.

5.5 Degassing and HIPping at Bodycote

Optical micrographs of sample BDC 461 unetched degassed at 400°C for 3hrs and HIPped is shown in figure 5.5.1 and FEGSEM backscattered micrographs of the same sample under the same conditions is shown in figure 5.5.2. Figure 5.5.3 shows EDX analysis for a two points on FEGSEM backscattered micrograph to show that some regions are relatively free of Si.

Figures 5.5.4 and 5.5.5 show optical micrographs of sample 461 unetched, degassed at 400°C for 12 hrs and HIPped. Both micrographs show inhomogeneity. Figure 5.5.6 shows FEGSEM backscattered micrographs BDC of sample 461 unetched, degassed at 400°C for 12 hrs and HIPped. Figure 5.5.7 shows EDX spectra of two points which were taken on figure 5.5.6a to illustrate the inhomogeneity in the microstructure.

Figure 5.5.8 shows an optical micrograph of sample BDC 461 unetched, degassed at 500°C for 12 hrs and HIPped. The sample was taken from the top edge (corner) of the canister. Figure 5.5.9 shows an optical micrographs of sample BDC 461 unetched degassed at 500°C for 12 hrs and HIPped. The sample was taken from the middle of the canister to compare it with the edge sample previously described. Figure 5.5.10 shows FEGSEM micrographs of sample BDC 461 unetched, degassed at 500°C for 12 hrs and HIPped. The needle shaped of particles are assumed to be intermetallic particles based on literature evidence [9, 22]. They are too small to analyse by EDX as the width is less than 1µm. Figure 5.5.11 shows FEGSEM backscattered micrograph of sample BDC 461 unetched, degassed at 500°C for 12 hrs and HIPped. Figure 5.5.11b was taken on a fine particle area of figure 5.5.10b.

Figure 5.5.12 is an optical FEGSEM backscattered micrograph of Sample BDC 431 unetched, degassed at 500°C for 12 hrs and HIPped. Figure 5.5.13 shows FEGSEM backscattered micrograph of sample BDC 431 unetched, degassed at 500°C for 12 hrs and HIPped. Seven different points were taken on the micrograph for spot analyses. Figures 5.5.14-5.5.20 show the EDX spectrum for each point. Table 5.5.2 shows the EDX quantitative analysis for each spot (i.e. elements wt.%). The reason for doing spot analysis is to illustrate the variation of Si and Al on the microstructure. Figure 5.5.21 shows FEGSEM micrograph elemental mapping of sample BDC 431 degassed at 500°C for 3 hrs.

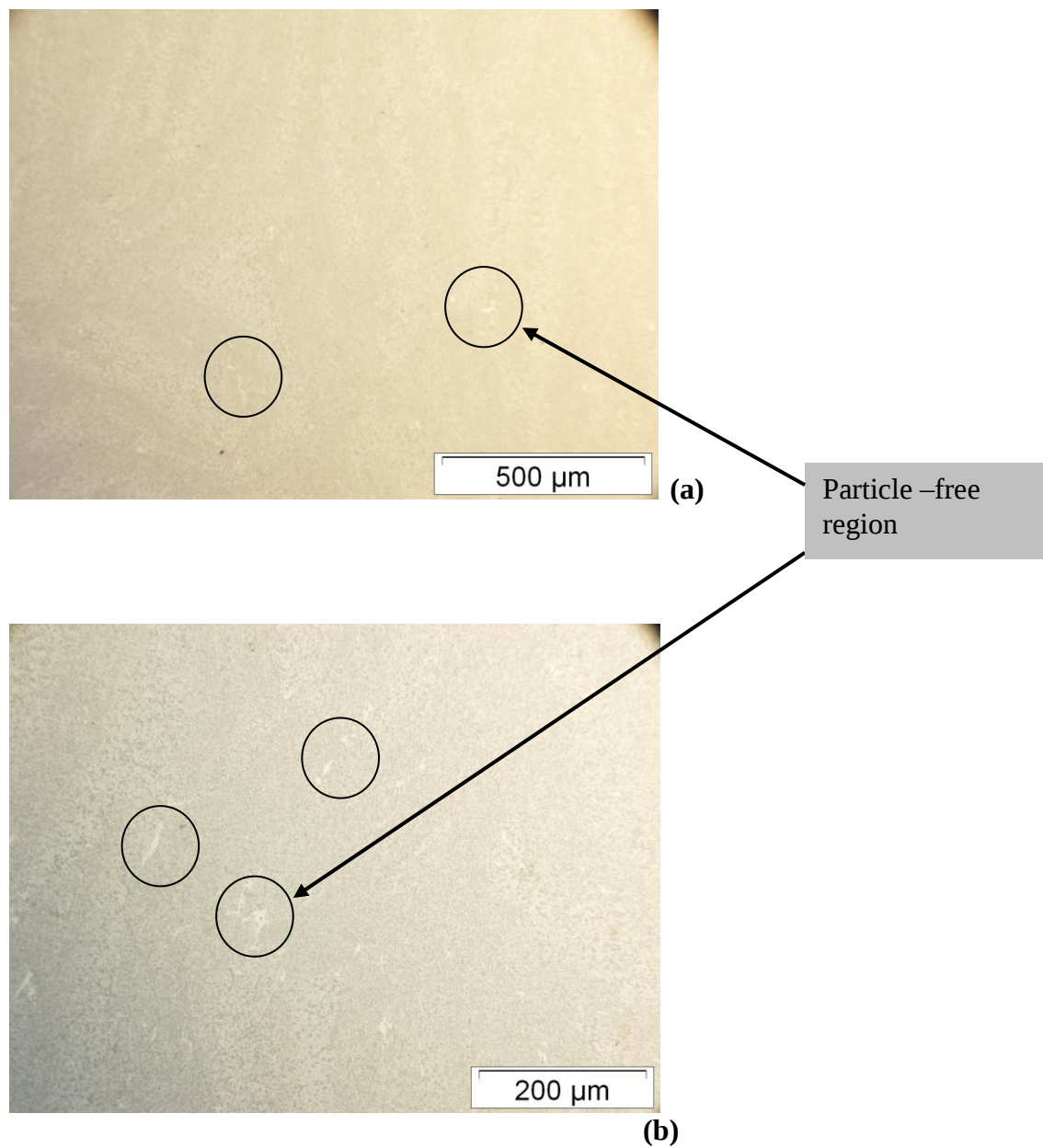
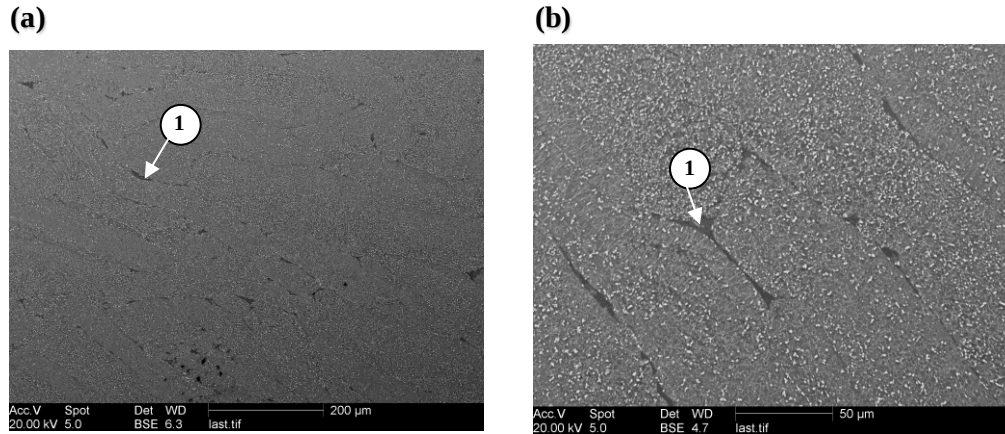


Figure 5.5.1 Optical micrograph sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 400°C for 3hrs and HIPped. (a) All over view (b) Higher magnification



(1) Particle free region

Figure 5.5.2 FEGSEM backscattered micrographs sample BDC 461 unetched (acc to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 400°C for 3h HIPped. (a) Overall view (b) Higher magnification.

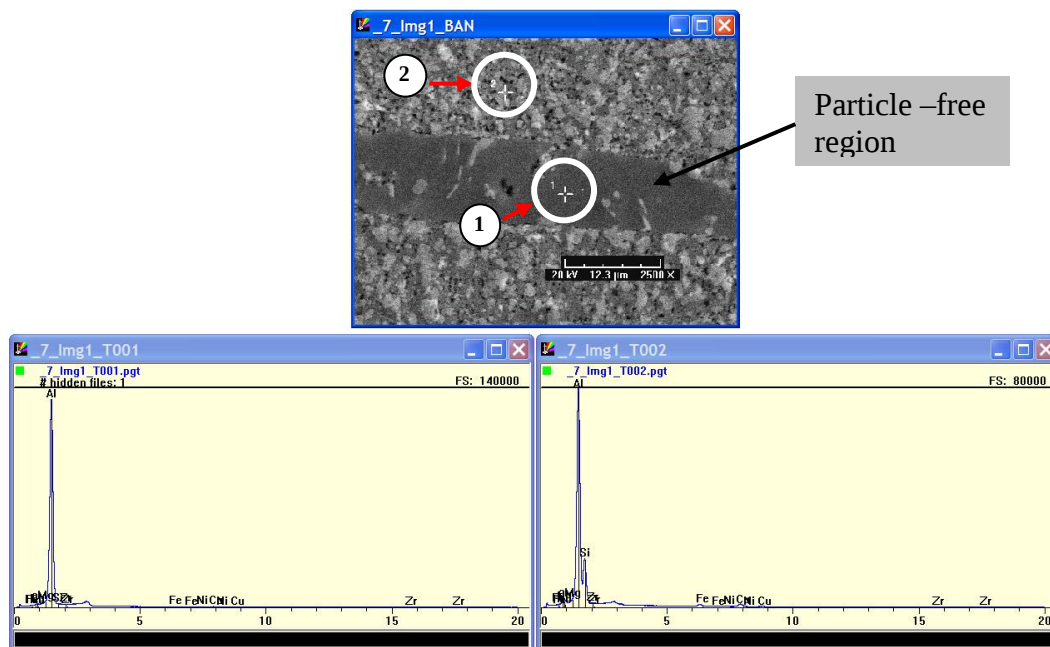
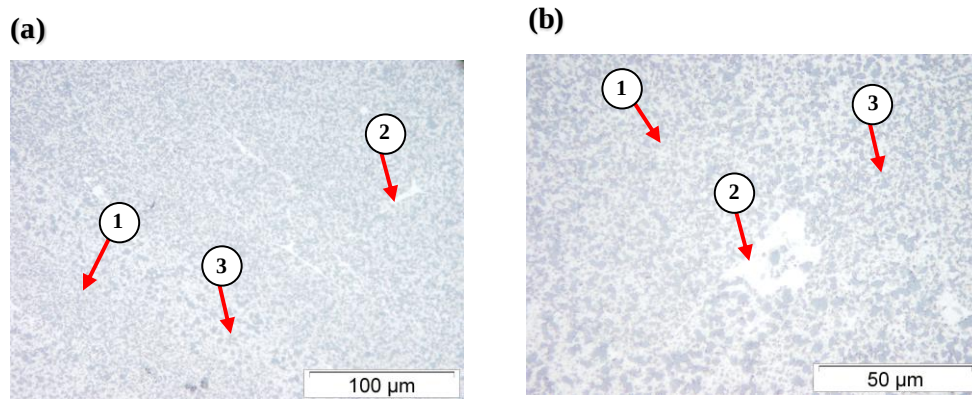
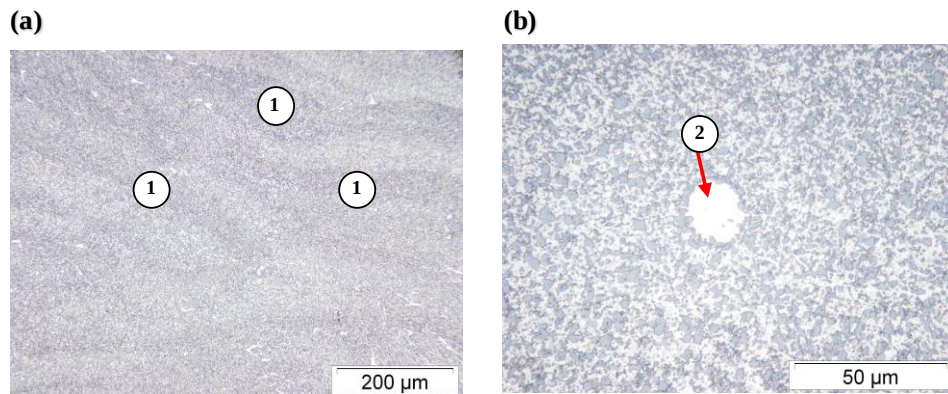


Figure 5.5.3 Sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 400°C for 3 hrs and HIPped FEGSEM micrograph taken at 2 micron and 20.00 KV. EDX spectra for points 1 and 2.



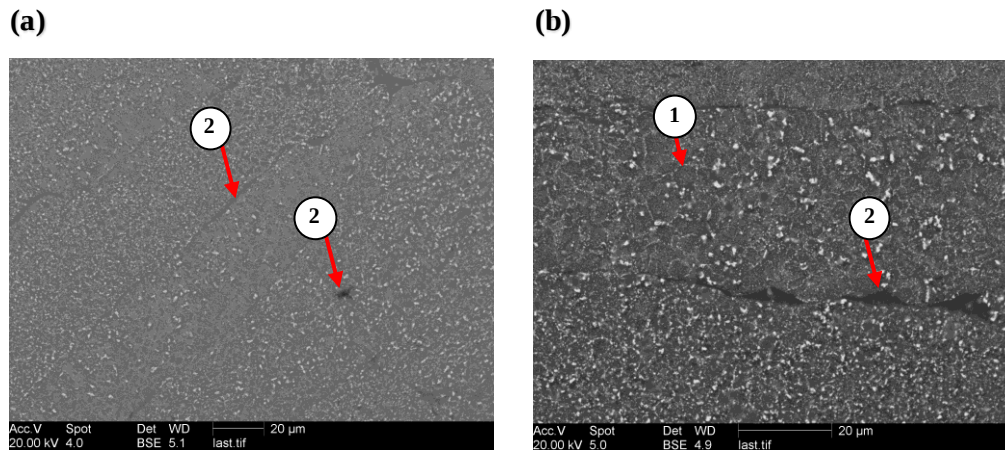
(1) Fine silicon particles (2) Aluminium matrix (3) Coarse silicon particles

Figure 5.5.4 Optical micrograph sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 400°C for 12 hrs and HIPped. (a) All over view (b) Higher magnification.



(1) This region illustrates inhomogeneity in the microstructure (2) Aluminium matrix

Figure 5.5.5 Optical micrographs sample BDC 461 etched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) etched degassed at 400°C for 12 hrs and HIPped. (a) All over view (b) Higher magnification showing isolated large round area apparently free of Si particles.



(1) Network of silicon particles (2) Particle-free region

Figure 5.5.6 FEGSEM backscattered micrographs sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 400°C for 3hrs and HIPped. (a) Overall view (b) Higher magnification.

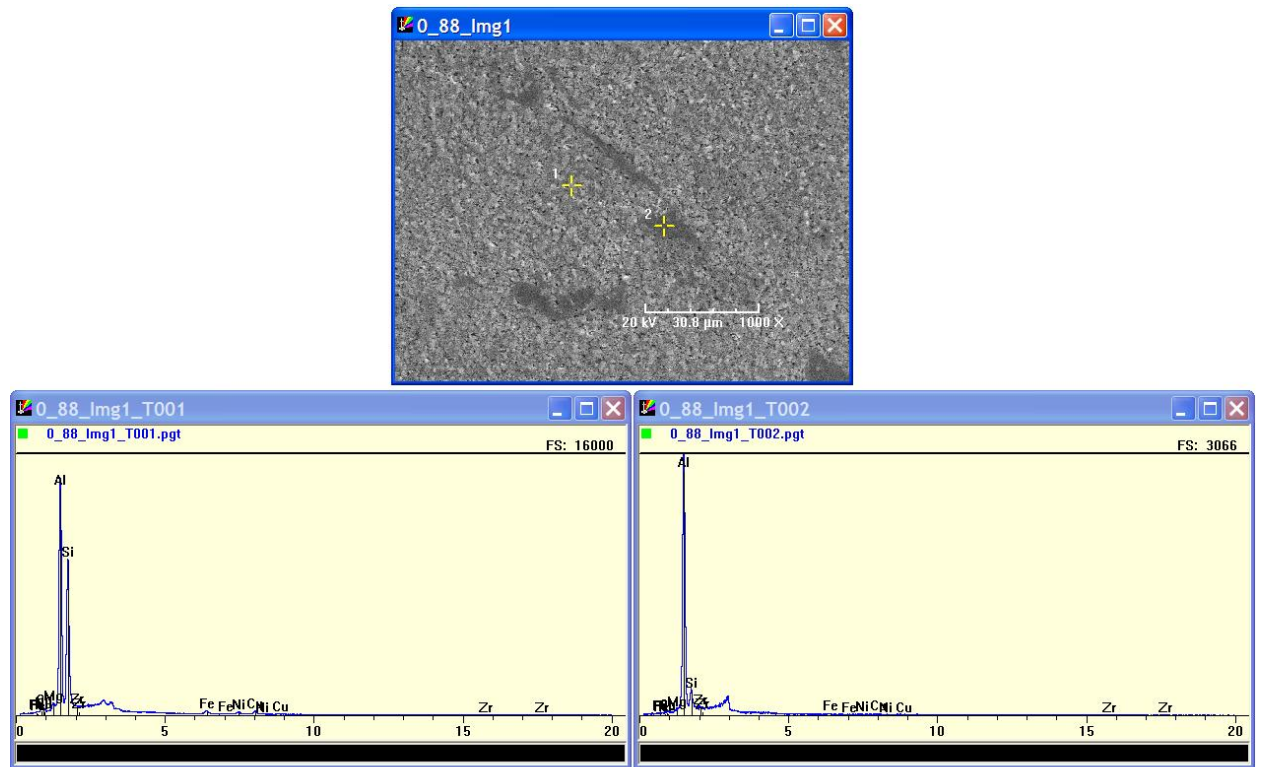
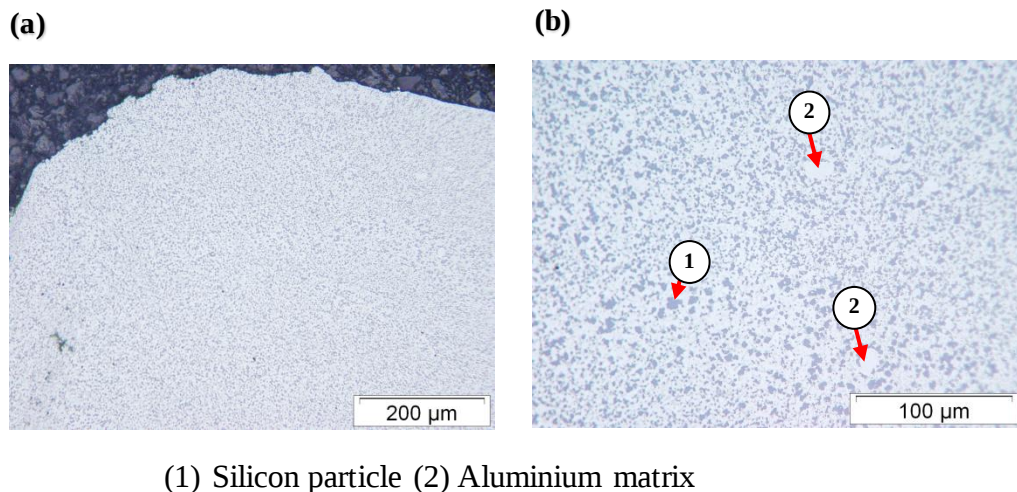


Figure 5.5.7 Sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 400°C for 12 hrs and HIPped FEGSEM micrograph taken at 2 micron and 20.00 KV. EDX spectra for points 1 and 2.



(1) Silicon particle (2) Aluminium matrix

Figure 5.5.8 Optical micrograph sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 500°C for 12 hrs and HIPped taken from the top edge of the billet. (a) All over view (b) Higher magnification.

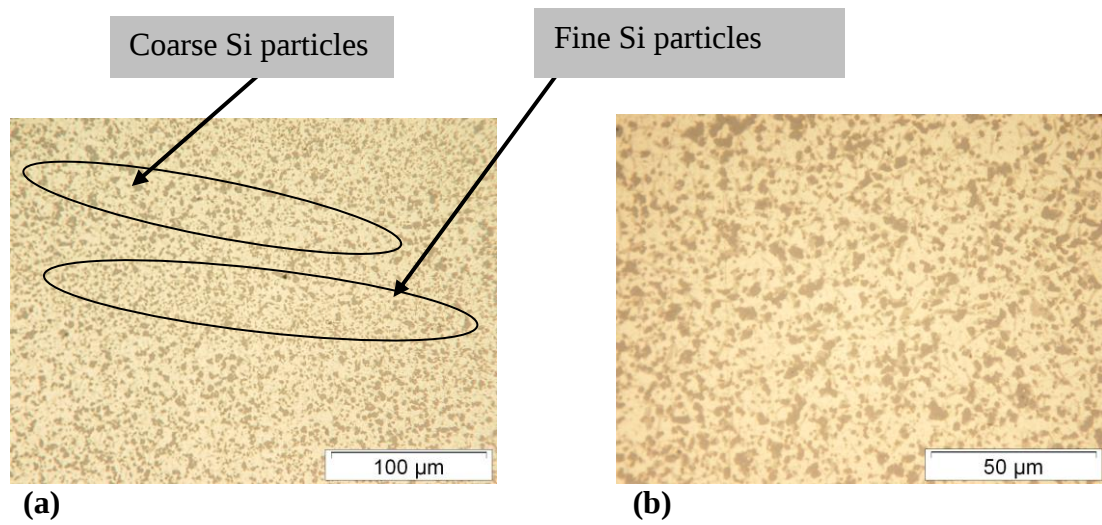


Figure 5.5.9 Optical micrograph sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 500°C for 12 hrs and HIPped. The sample was taken from the middle of the billet. (a) All over view (b) Higher magnification.

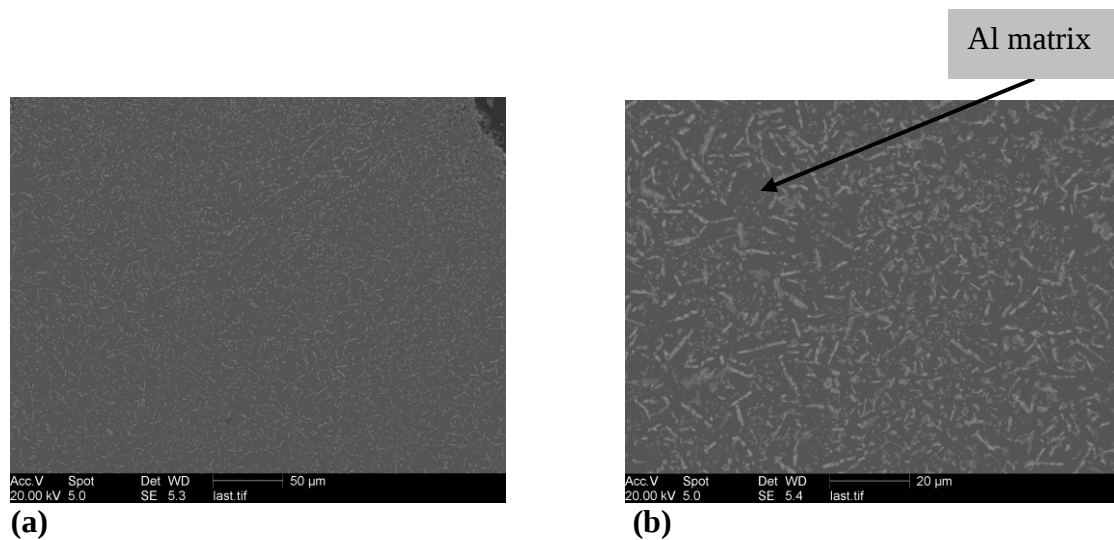
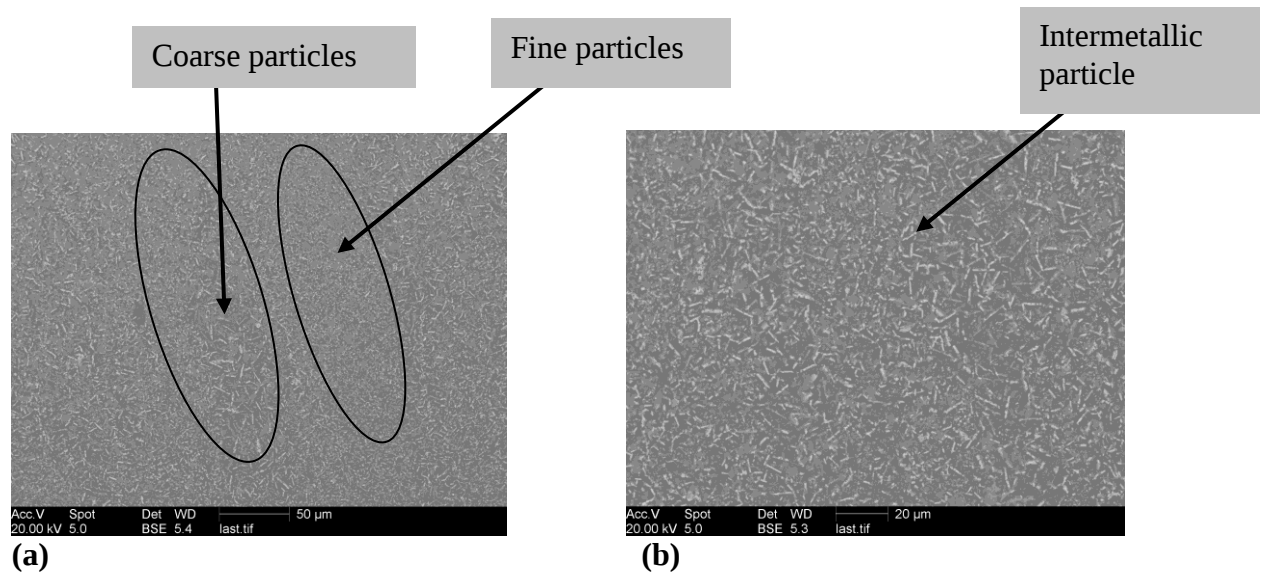


Figure 5.5.10 FEGSEM micrograph sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 500°C for 12 hrs and HIPped. (a) Overall view (b) Higher magnification.



(a)
(b)
Figure 5.5.11 FEGSEM backscattered micrograph sample BDC 461 unetched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) degassed at 500°C for 12 hrs and HIPped. (a) Overall view (b) Higher magnification.

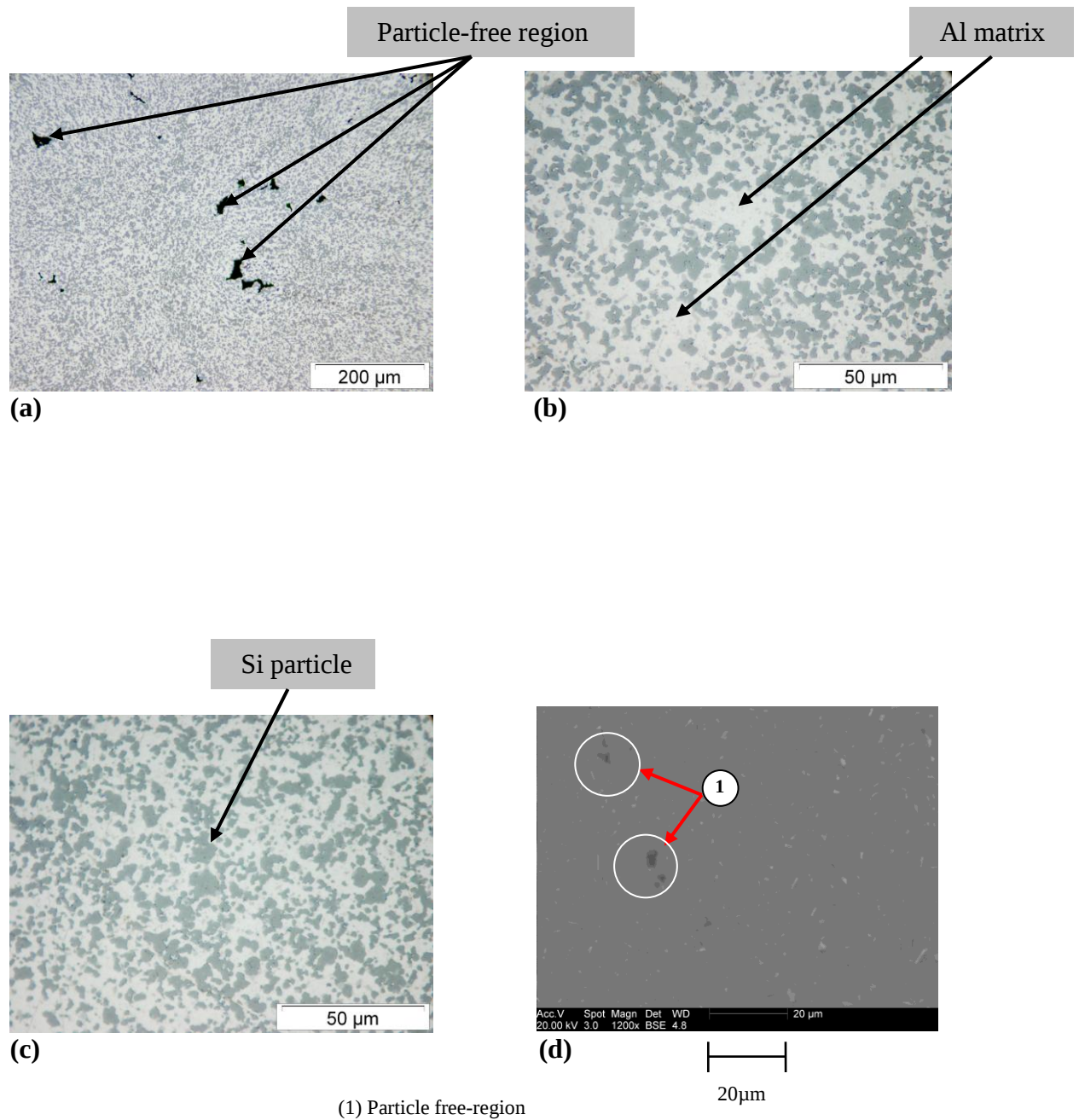


Figure 5.5.12 Sample BDC 431 unetched (according to table 4.1 Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr) degassed at 500°C for 12 hrs and HIPped. (a,b,c) optical micrographs (d) FEGSEM backscattered micrograph.

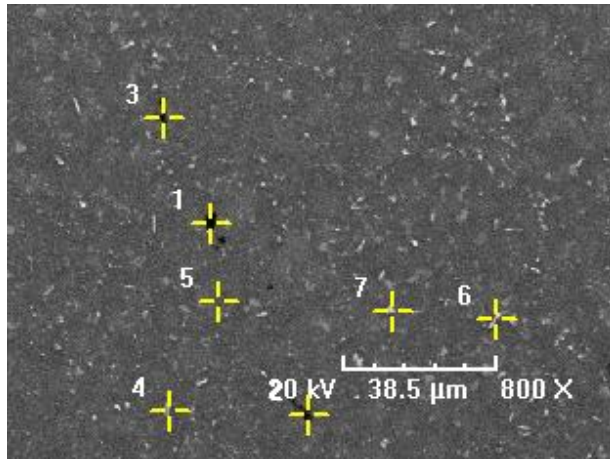


Figure 5.5.13 FEGSEM backscattered micrograph sample BDC 431 unetched (according to table 4.1 Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr) degassed at 500°C for 12 hrs and HIPped.

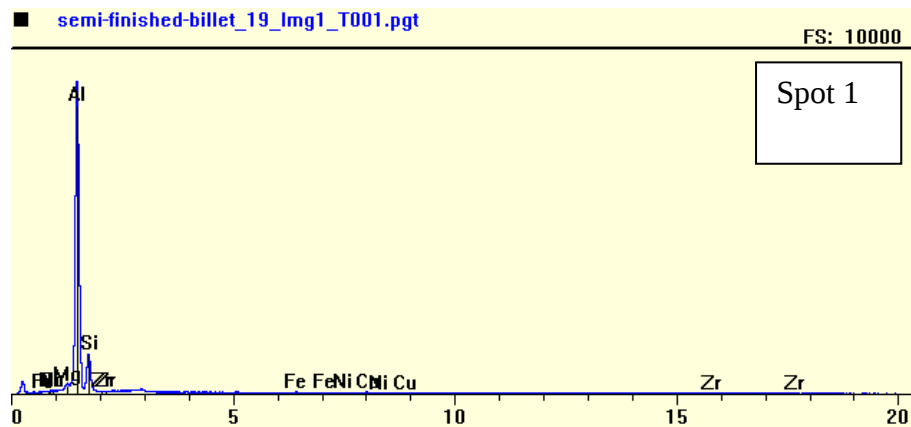


Figure 5.5.14 EDX spectrum for spot 1 in figure 5.5.13.

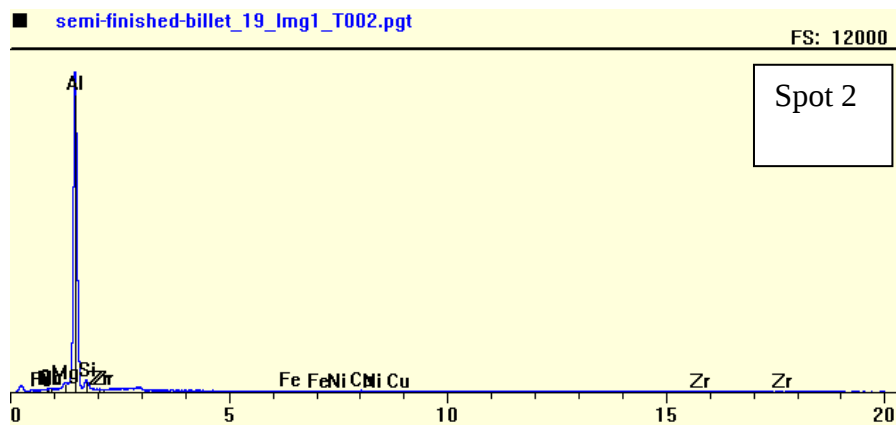


Figure 5.5.15 EDX spectrum for spot 2 in figure 5.5.13.

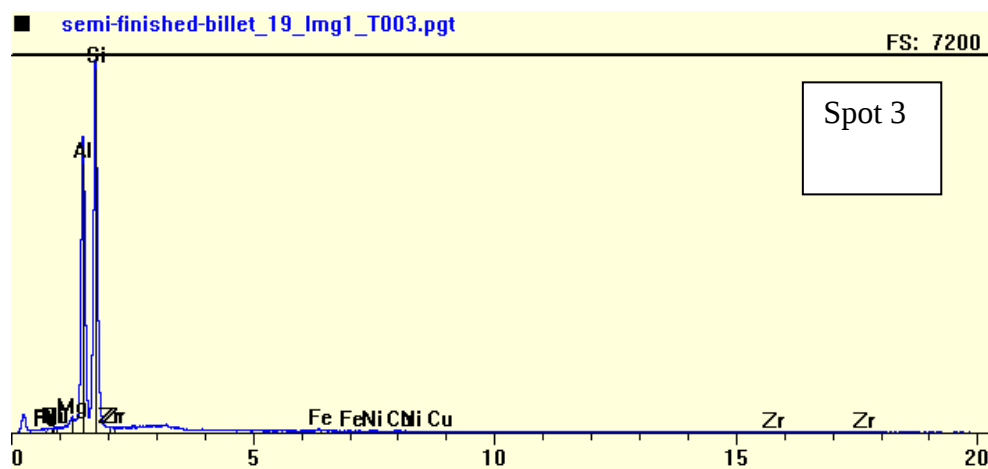


Figure 5.5.16 EDX spectrum for spot 3 in figure 5.5.13.

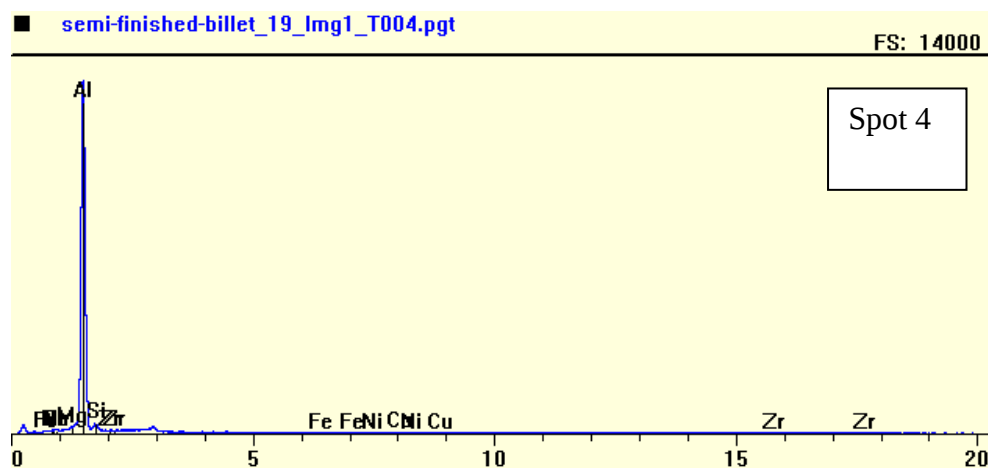


Figure 5.5.17 EDX spectrum for spot 4 in figure 5.5.13.

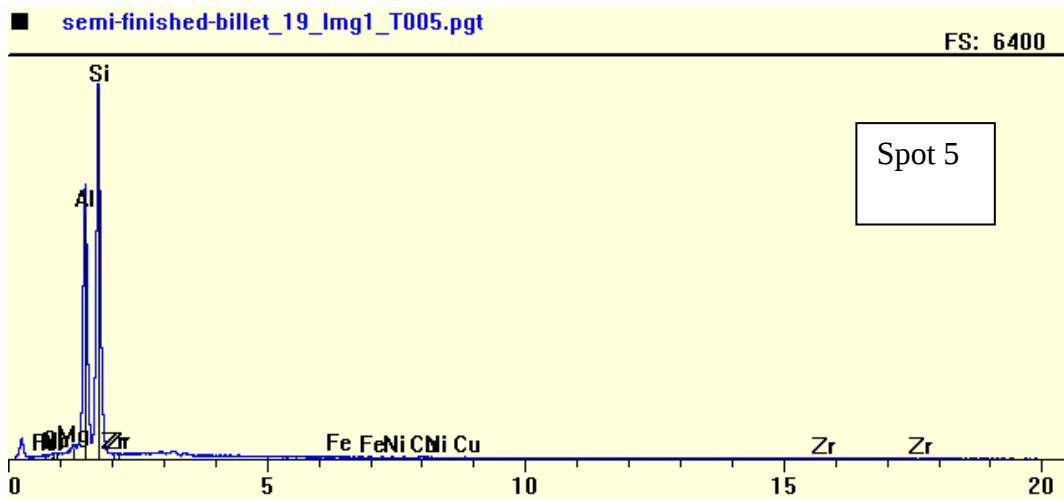


Figure 5.5.18 EDX spectrum for spot 5 in figure 5.5.13.

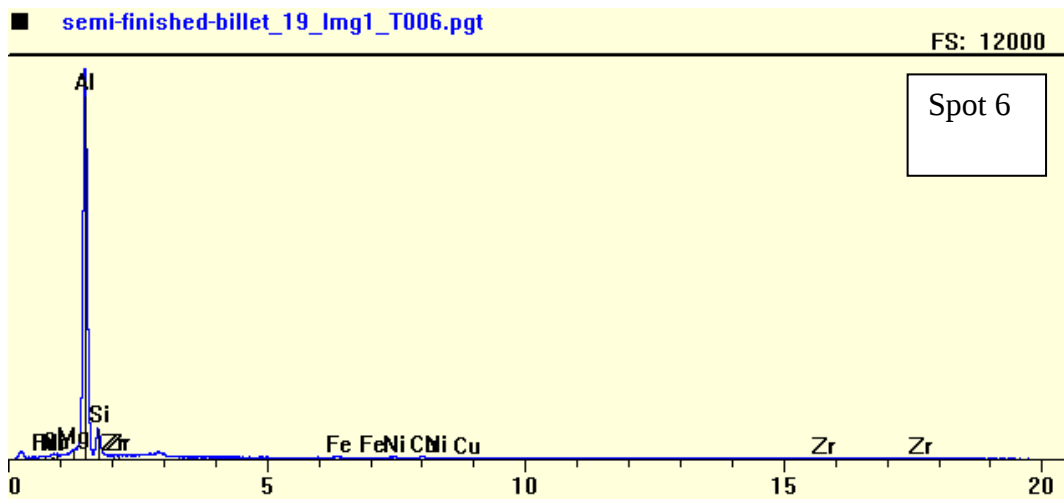


Figure 5.5.19 EDX spectrum for spot 6 in figure 5.5.13.

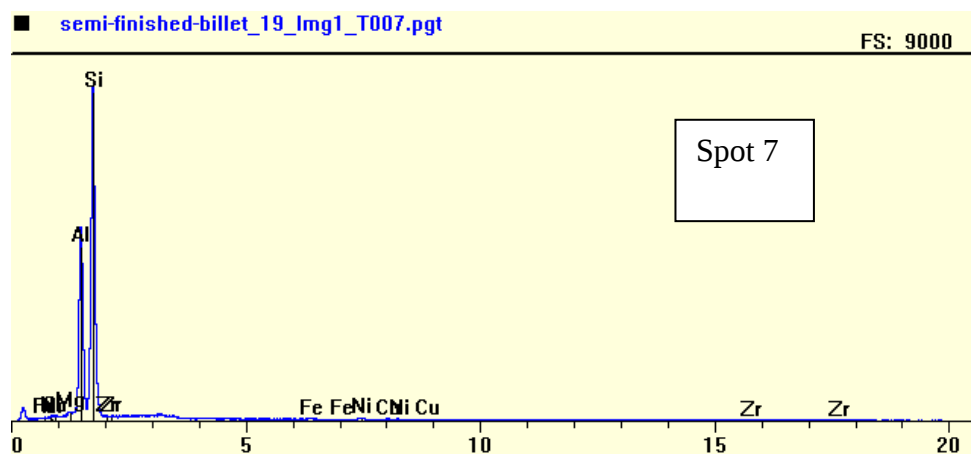


Figure 5.5.20 EDX spectrum for spot 7 in figure 5.5.13.

Spot No	Elements (wt. %)						
	Al	Si	Mg	Fe	Cu	Zr	Ni
1	75.0	21.8	1.3	0.4	1.0	0	0.0
2	90.4	6.0	1.6	0.3	1.6	0	0.0
3	32.0	65.9	0.9	0.6	0.4	0	0.2
4	93.5	3.9	0.9	0.2	1.2	0	0.2
5	30.3	67.9	0.8	0.3	0.7	0	0.2
6	82.2	12.6	1.1	1.0	1.9	0	1.3
7	26.8	69.9	0.5	0.7	0.8	0.5	0.8

Table 5.5.1 EDX quantitative analysis (wt. %) for each spot on figure 5.5.13.

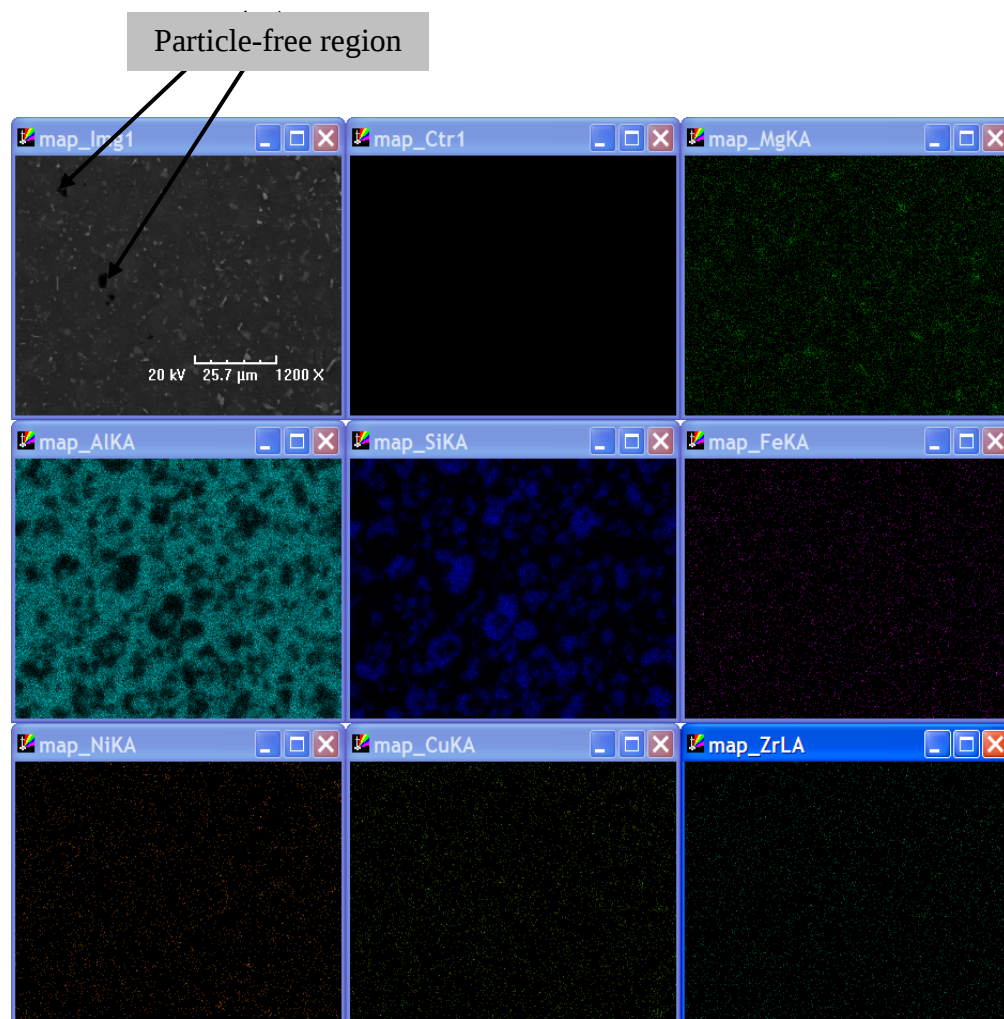


Figure 5.5.21 FEGSEM micrograph elemental mapping of sample BDC 431 (according to table 4.1 Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr) degassed at 500°C for 12 hrs. WD = 5 mm, V = 20 kV, t = 45 min. Overall view of ribbon, Mg, Al, Si, Fe, Ni, Cu, Zr elements.

5.6 Bar Microstructure As-Received

Firstly, the result of the two bars 446-4051-FEF-2 and 446-4047-FEF-3 will be presented. The two bars have similar composition and in both cases the flakes were free encapsulated. Afterwards, a different alloy composition 431-4046-HF-60 with higher silicon content where the flakes are cold compacted before degassing “HF” will be presented. Then, 461-5017-HF which has a similar Si content to the first but with flaks that are cold compacted prior to degassing. Finally, 444-4042 FEF which has very high silicon content (i.e. 30.3 wt%) where the flakes were free encapsulated. An optical and FEGSEM backscattered micrographs of sample 446-4051-FEF-2 etched is shown in figure 5.6.1. Figure 5.6.2 shows a FEGSEM backscattered micrograph and EDX spectra for points 1 and 2 in the FEGSEM micrograph of Sample 446-4051-FEF-2. Figure 5.6.3 shows spot analysis of sample 446-4051-FEF-2 bar to illustrate the variation of Si in the microstructure. Figure 5.6.4 shows an optical and a FEGSEM backscattered micrographs of sample 446-4047-FEF-3. Figure 5.6.5 shows an optical micrograph of sample 461-5017-HF etched. Figure 5.6.6 shows an optical and a FEGSEM backscattered micrographs of sample 431-4046-HF-6 etched. Figure 5.6.7 shows an optical and a FEGSEM backscattered micrographs of sample 444-4042 FEF etched. Figure 5.6.8 shows an optical micrograph of sample 461-5017-HF.

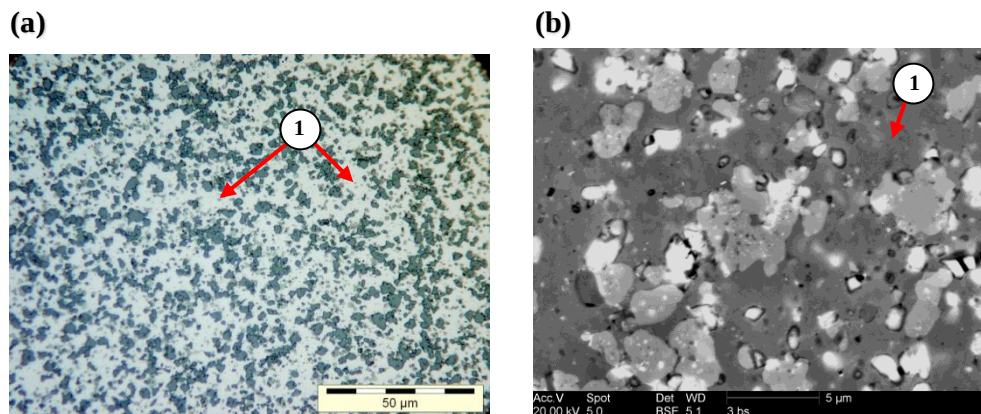


Figure 5.6.1 Sample 446-4051-FEF-2 etched (according to table 4.1 Al 20.3Si 2Cu 1.5Mg 1.8Fe 1.9Ni 0.2Zr) (a) Optical micrograph (b) FEGSEM backscattered micrograph.

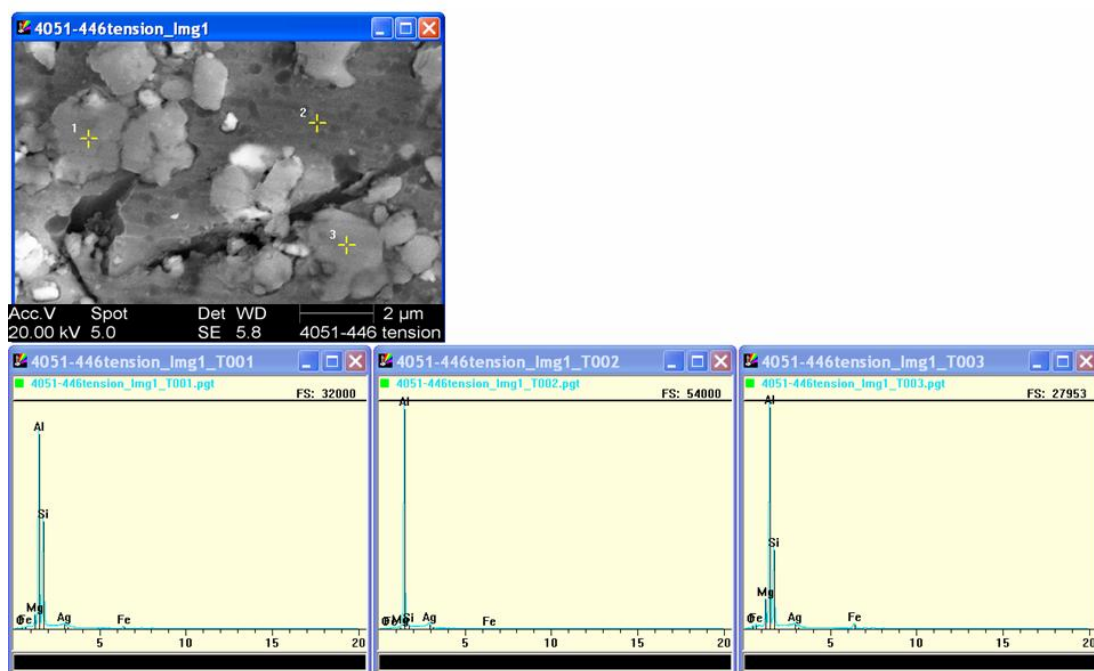


Figure 5.6.2 Sample 446-4051-FEF-2 (according to table 4.1 Al 20.3Si 2Cu 1.5Mg1.8Fe 1.9Ni 0.2Zr) FEGSEM micrograph taken at 20 kV. EDX spectra for points 1, 2 and 3.

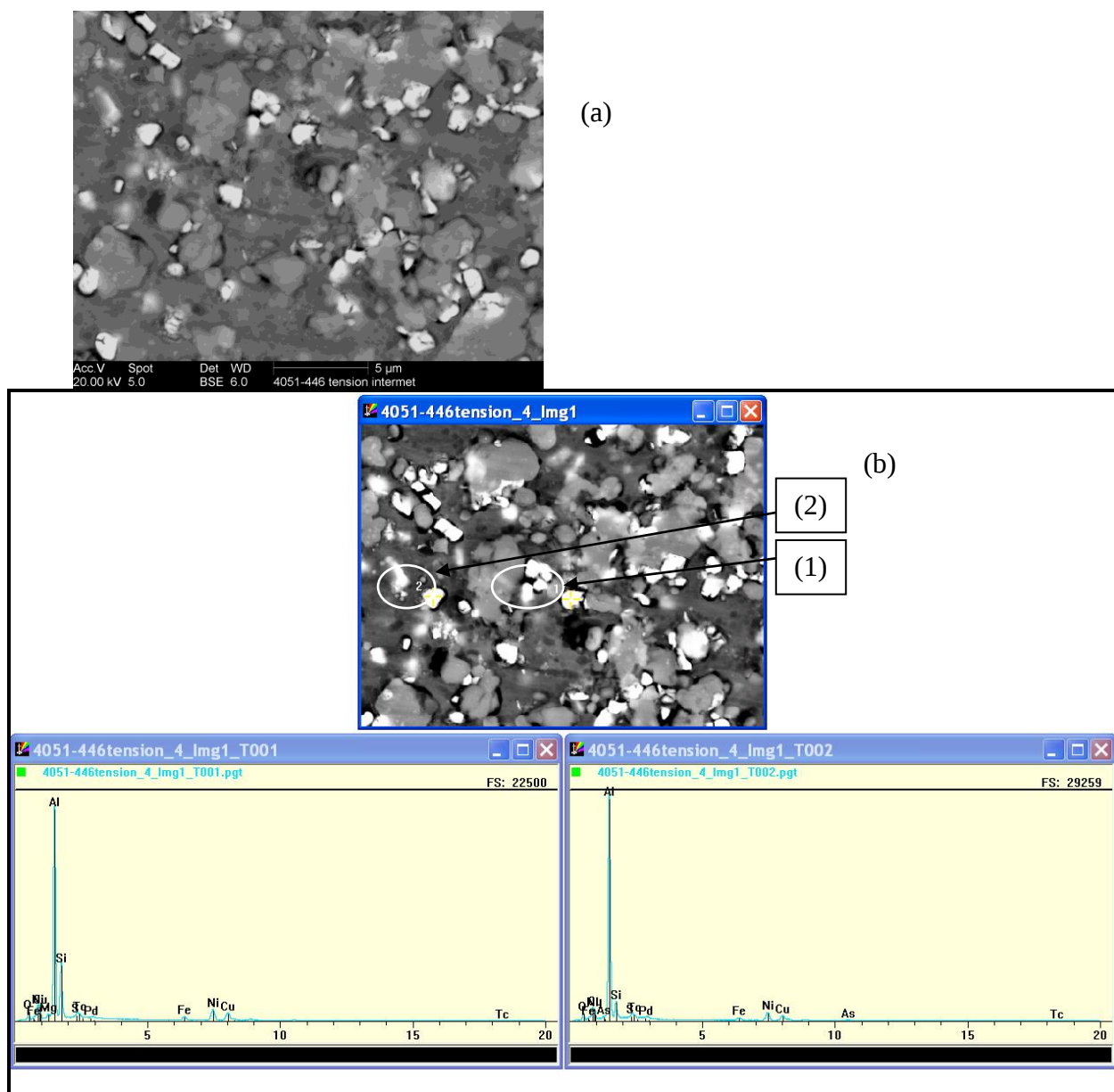


Figure 5.6.3 Sample 446-4051-FEF-2 (according to table 4.1 Al 20.3Si 2Cu 1.5Mg 1.8Fe 1.9Ni 0.2Zr) (a) FEGSEM backscattered micrograph taken at 20.00 kV. (b) FEGSEM backscattered micrograph and EDX spectra for points 1 and 2 in the FEGSEM micrograph.

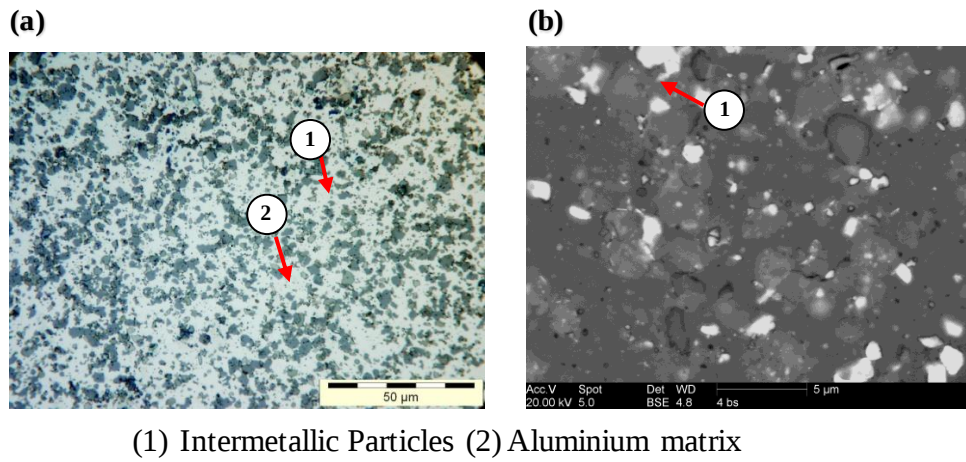


Figure 5.6.4 Sample 446-4047-FEF-3 etched (according to table 4.1 Al 20.6Si 2Cu 1.6Mg 1.9Fe 1.9Ni 0.2Zr) (a) Optical micrograph (b) FEGSEM backscattered micrograph

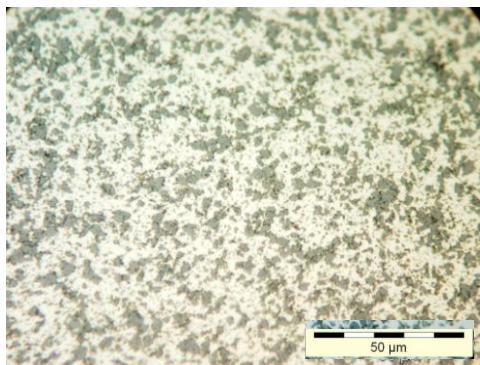


Figure 5.6.5 Optical micrograph of Sample 461-5017-HF etched (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr)

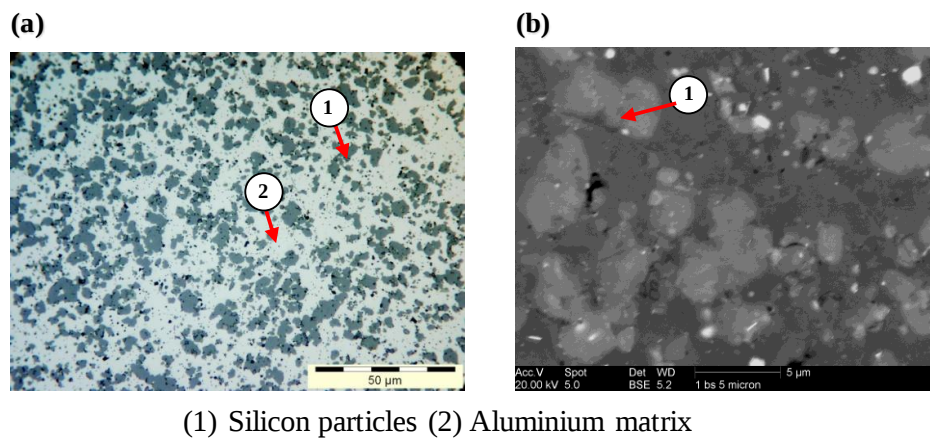


Figure 5.6.6 Sample 431-4046-HF-6 etched (according to table 4.1 Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr) (a) optical micrograph (b) FEGSEM backscattered micrograph

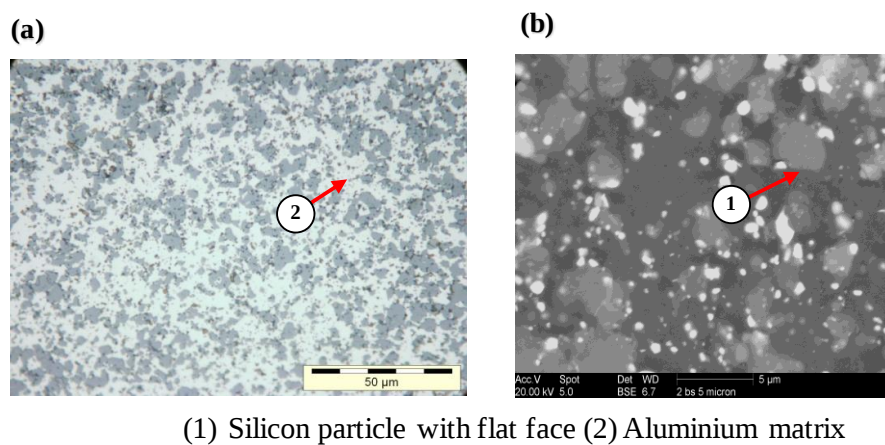


Figure 5.6.7 Sample 444-4042 FEF etched (according to table 4.1 Al 30.3Si 1.5Cu 1.2Mg 1.1Fe 1.4Ni 0.2Zr) (a) Optical micrograph (b) FEGSEM backscattered micrograph.

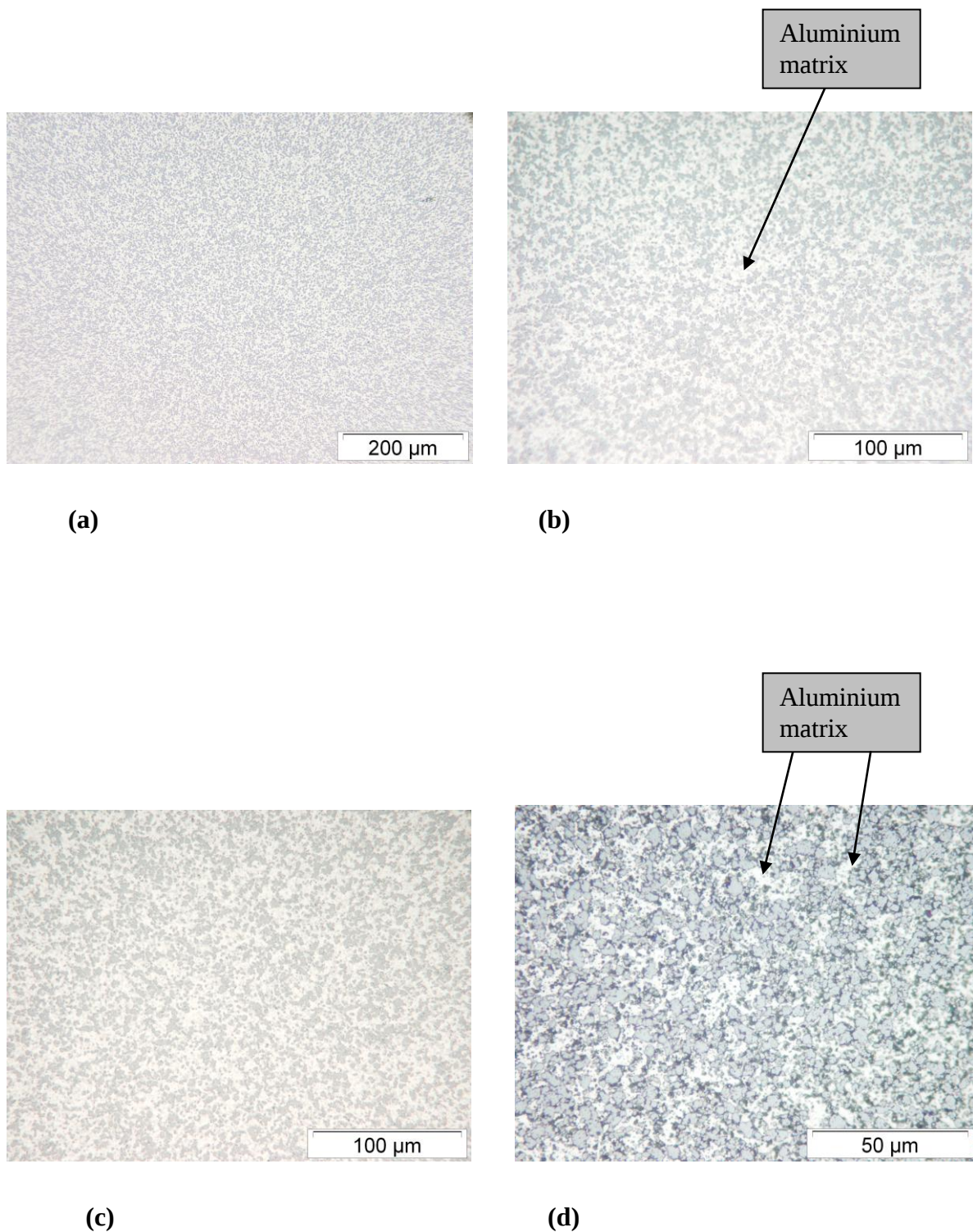


Figure 5.6.8 Optical micrograph of sample 461-5017-HF (according to table 4.1 Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr). (a) All over view unetched. (b) Higher magnification unetched. (c) All over view etched. (d) Higher magnification etched.

5.7 Si Particle Measurements

Area fractions obtained from image analysis (section 4.3.3.4) are showing on table 5.7.1. The calculated area fraction is donated by” calculated Si area fraction (wt %)” and used for comparison with the area fraction obtained from image analysis. Calculate area fraction obtained using the rule of mixture.

$$f = [f_w] / [f_w + (1 - f_w)(\rho_f / \rho_m)]$$

where:

f = calculated area fraction.

f_w = weight fraction.

ρ_f = density of particle (for Si 2.3 g/cm³).

ρ_m = density of matrix (for Al 2.7 g/cm³).

In table 5.7.2 the particle sizes obtained from image analysis are showing. Its clear that Si particle readings represent means for fields of view and for each sample several fields of view are obtained. There are at least three different fields of view.

RSP No	Condition	Si wt. %	Area fraction readings (%)	Area fraction Si%	Calculated Si area fraction (%)
446-2	bar	20.3	21.1,21.08,20.0	20.7 $\pm$ 0.6	23
446-3	bar	20.6	21.8,22.17,22,22	22.0 $\pm$ 0.2	23.3
461	bar	21	17.24,23.67,17.46,16.55	18.7 $\pm$ 3.3	23.8
431	bar	29.8	32.6,31.9,33.73,34.24	33.1 $\pm$ 1	33.3
444	bar	30.3	33.2,33.31,33.81	33.4 $\pm$ 0.3	33.8
461	400°C/3hr	21	27.43,31.6,27.6,25.3,28.44	28.1 $\pm$ 2.3	23.8
461	400°C /12hr	21	23.2,23.76,23.13,24.46	23.6 $\pm$ 0.6	23.8
461	500°C /12hr	21	26.22,28.19,24.78,25.44,24.72	25.8 $\pm$ 1.4	23.8
431	400°C /3hr	29.8	27.37,27.37,30.3,30.52,30.04	29.1 $\pm$ 1.6	33.3
431	400°C /12hr	29.8	27.15,28.35,31.4,31.57	29.6 $\pm$ 2	33.3
431	500°C /12hr	29.8	31.85,32.09,29.57,30.63	31.1 $\pm$ 1	33.3

Table 5.7.1 Silicon area fraction (%).

RSP No.	Condition	Si %	Si particle readings (µm)	Si Particle Mean (µm)	S.D (µm)
461	bar	21	2,2,2.1,1.8	2.0	±0.1
431	bar	29.8	2.75,26,2.64	2.7	±0.1
444	bar	30.3	2,2.03,1.6	1.9	±0.2
461	400°C/3hr	21	1.3,1.3,1.4,1.3,1.4	1.3	±0.1
461	400°C /12hr	21	1.22,1.4,1.5,1.66	1.5	±0.2
461	500°C /12hr	21	2.21,2.1,2.44,2.21	2.3	±0.2
431	400°C /3hr	29.8	1.7,1.2,1.3,1.4,1.3	1.4	±0.2
431	400°C /12hr	29.8	1.6,1.97,1.67,1.85	1.8	±0.2
431	500°C /12hr	29.8	2.82,2.83,2.5,2.85	2.8	±0.2

Table 5.7.2 Silicon particles (%).

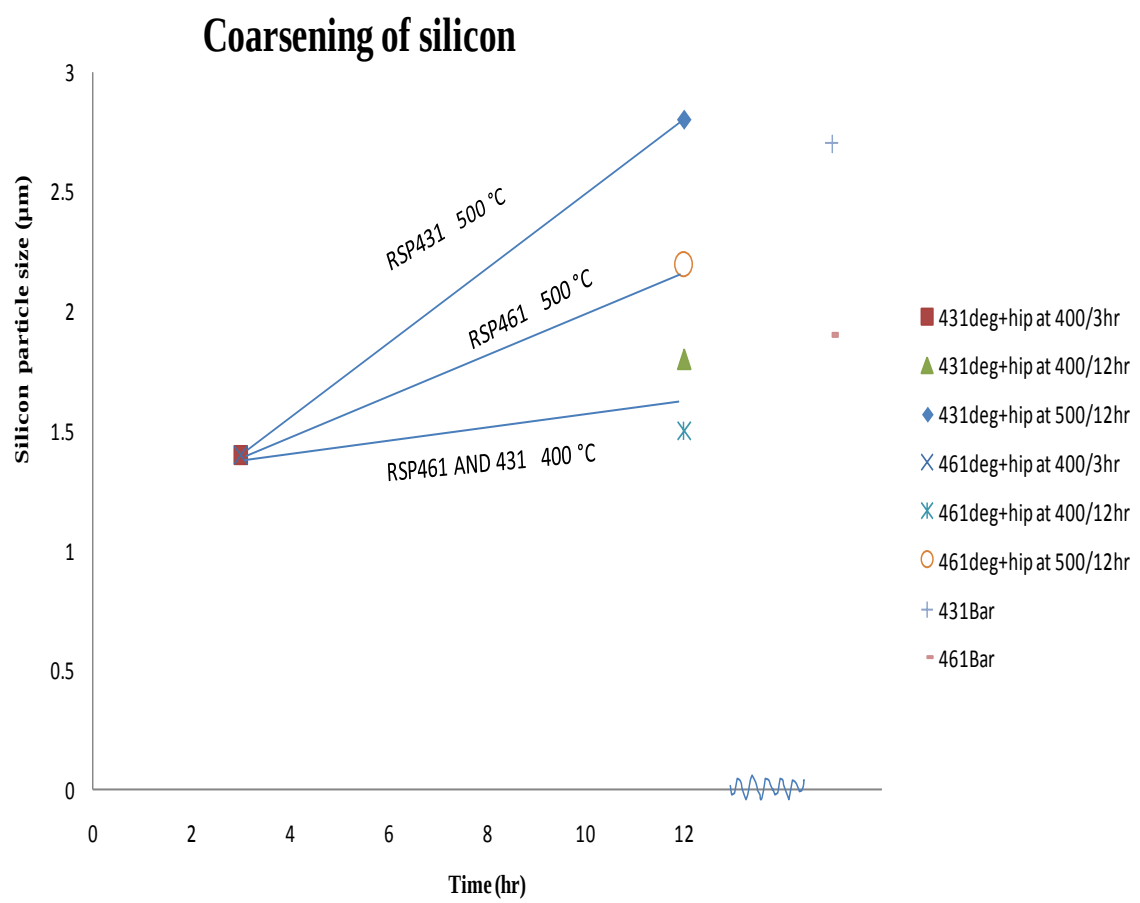


Figure 5.7.1 Coarsening of silicon particles.

5.8 X-Ray Diffraction

The XRD spectra are shown in figure 5.8.1. The figure shows the intensity versus 2θ of extruded bars 446-3, 446-2, 444, 431 and 461.

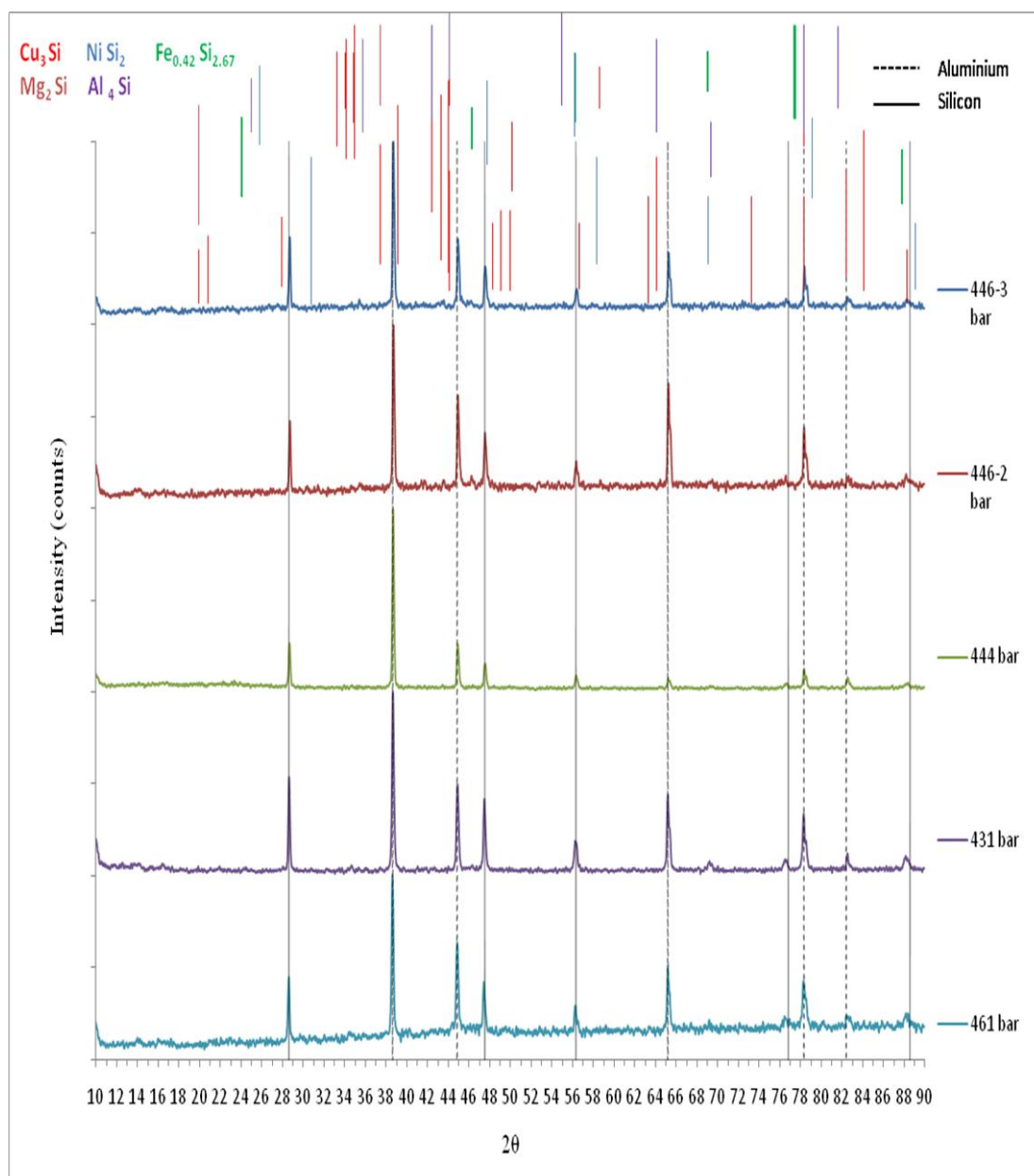


Figure 5.8.1 XRD spectra of extruded bars 446-3,446-2,444,431 and 461.

5.9 Hardness Testing

The results for the extruded bar are shown in the first columns. The next six columns show results for degassed and HIPped material from Bodycote HIP. The right hand column is A390 piston alloy for compression.

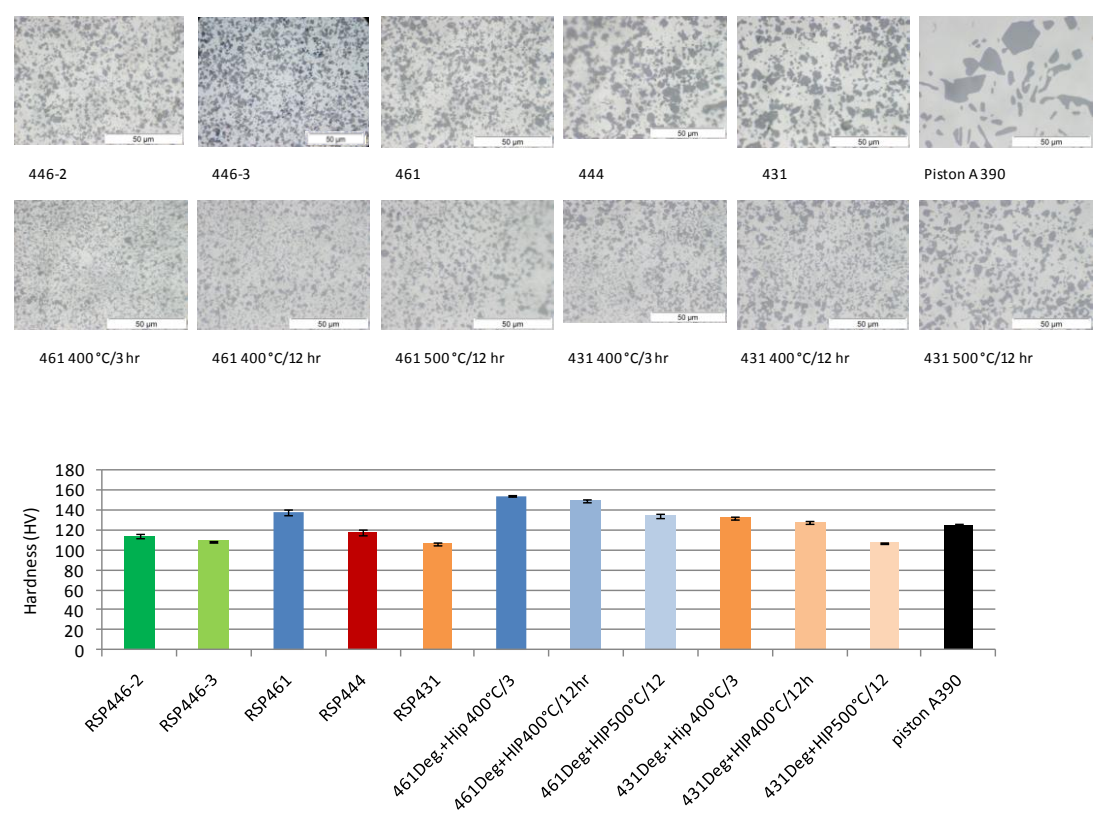


Figure 5.9.1 Hardness test. According to table 4.1: 446-2 (Al 20.3Si 2Cu 1.5Mg 1.8Fe 1.9Ni 0.2Zr), 446-3 (Al 20.6Si 2Cu 1.6Mg 1.9Fe 1.9Ni 0.2Zr), 461(Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr), 444(Al 30.3Si 1.5Cu 1.2Mg 1.1Fe 1.4Ni 0.2Zr), 431(Al 29.8Si 1.3Cu 1.4Mg 0.3Fe 0.3Ni 0.3Zr). Piston A390 (Al 17Si 0.5Zn 4.5Cu 0.6Mg).

RSP alloys	Mean (Vickers Hardness Number)	S.D
RSP446-2	113.2	±2.2
RSP446-3	108.1	±0.5
RSP461	137.3	±2.7
RSP444	117.9	±2.9
RSP431	106	±2
461Deg.+HIP 400°C/3hr	153.6	±0.7
461Deg.+HIP 400°C/12hr	148.9	±1.4
461Deg.+HIP 500°C/12hr	133.9	±1.6
431Deg.+HIP 400°C/3hr	131.6	±1.2
431Deg.+HIP 400°C/12hr	127.9	±1.5
431Deg.+HIP 500°C/12hr	106.8	±1.0
Piston A390	124.5	±0.8

Table 5.9.1 Hardness test.

5.10 Wear

A FEGSEM micrograph of sample 461 bar at load of 10 N is shown in figure 5.10.1. Figure 5.10.1a shows the beginning of the worn surface and figure 5.10.1 b shows the end of the worn surface. Aluminium matrix is shown on the micrograph dark grey because backscattered electron imaging is sensitive to atomic weight and therefore shown darker (i.e. in BS light atomic weight shows darker and heavy atomic weight shows lighter).

Figure 5.10.2 shows a FEGSEM backscattered micrograph of sample 461 bar at load of 10 N. Figure 5.10.2a shows the beginning of the worn surface and figure 5.10.2b represent the end of the worn surface at higher magnification. A FEGSEM micrograph of sample 461 bar at load of 100N is shown in figure 5.10.3. Figure 5.10.4 represents a FEGSEM micrograph of sample 461 bar where a micro cracks is shown. Figure 5.10.5 shows a FEGSEM backscattered micrograph of sample 461 bar at load of 100 N. Figure 5.10.6 shows the wear track profiles. Figure 5.10.6a shows the wear track of sample number 461extruded bar at 10N. Figure 5.10.6b shows the wear track of sample number 431extruded bar at 10N. Figure 5.10.6c represents the wear track of sample number 441extruded bar at 10N. Figure 5.10.6d shows the wear track of sample number 461extruded bar at 100N. All wear track profiles are shown in appendix 7. All wear debris are examined by FEGSEM illustrative examples of debris types are shown in figures 5.10.7 and 5.10.8. Figure 5.10.7 shows a FEGSEM micrograph of samples, 444-4042 FEF and 446-4051-FEF-2 at 10 N. However, figure 5.10.8 shows a FEGSEM micrograph of the same sample but at 100 N. A FEGSEM micrograph of the ball bearing is shown in figure 5.10.9. Figure 5.10.10 shows an optical and EDX spectra for unused ball bearing. Figure 5.10.11 shows a

FEGSEM micrograph of the ball bearing at 10 N and points 1, 2 and 3 were analysed by EDX. Figure 5.10.12 shows a FEGSEM micrograph of the ball bearing at 100 N and points 1, 2 and 3 were analysed by EDX. Figures 5.10.13-5.10.17 show wear volume at 10N. Figures 5.10.18-5.10.21 shows wear volume at 100N. Figure 5.10.22 shows a comparison of wear volume at 10 and 100N. Figures 5.10.23-5.10.25 show wear volume versus Si area fraction at 10 and 100N. The figures are divided up under headings:

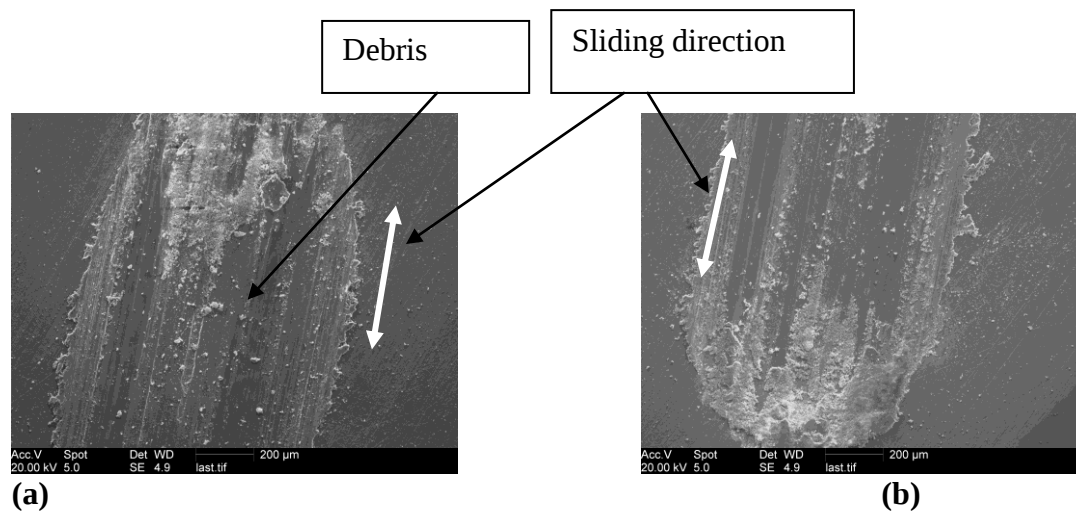
5.10.1 Wear Mechanism.

5.10.2 Wear Debris.

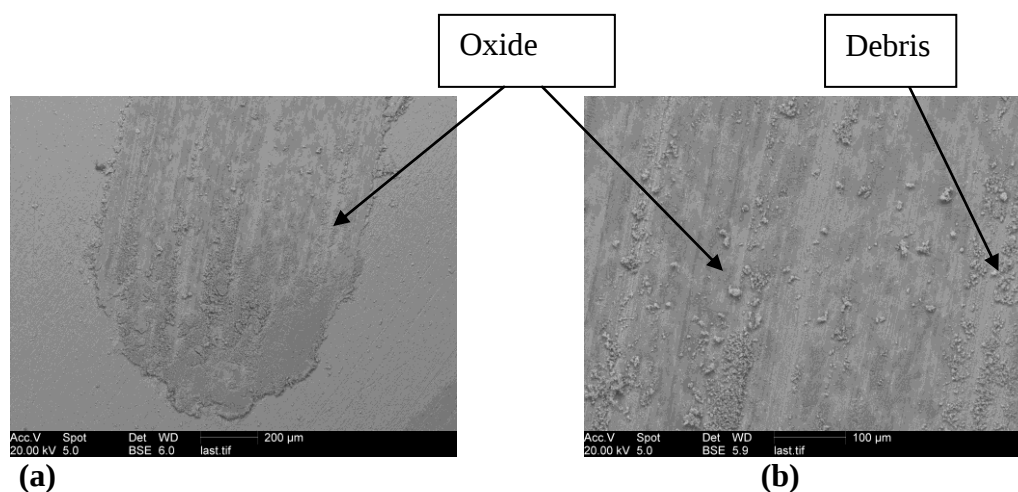
5.10.3 The Counterface.

5.10.4 Effect of Alloying Elements.

5.10.1 Wear Mechanism



(a) **(b)**
Figure 5.10.1 FEGSEM micrograph sample 461 bar (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) at load of 10 N. (a) beginning of the worn surface (b) end of the worn surface.



(a) **(b)**
Figure 5.10.2 FEGSEM backscattered micrograph sample 461 bar (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) at load of 10 N. (a) beginning of the worn surface (b) end of the worn surface at higher magnification.

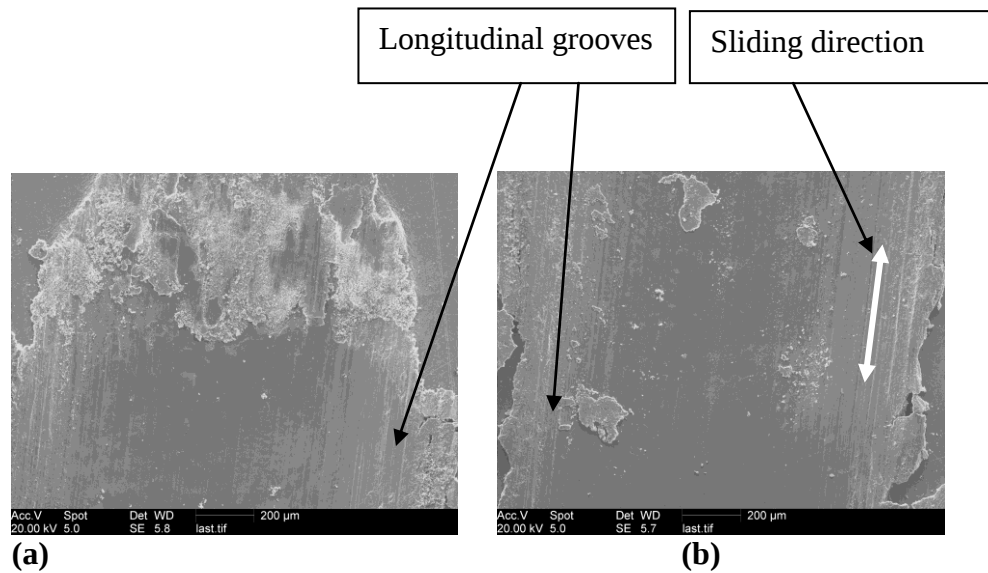


Figure 5.10.3 FEGSEM micrograph sample 461 bar (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) at load of 100N.

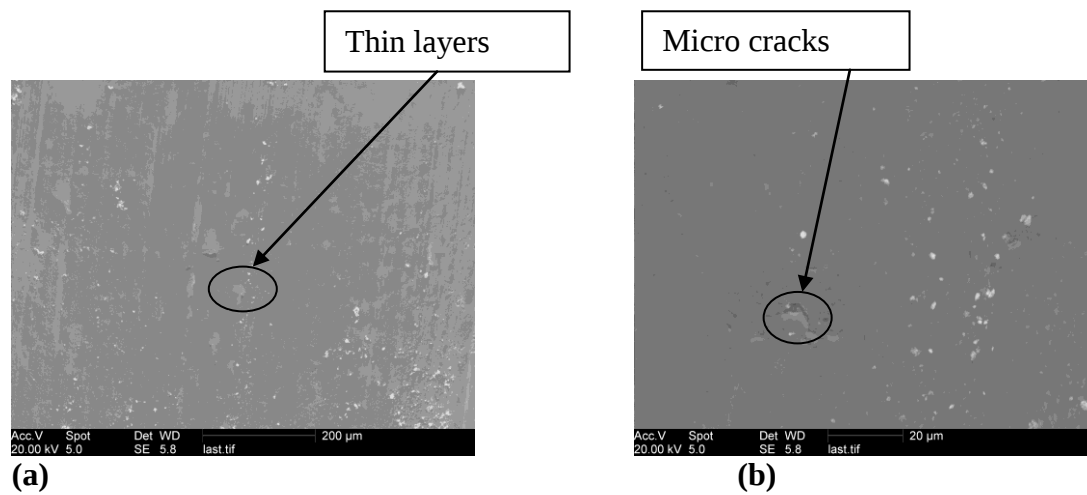


Figure 5.10.4 FEGSEM micrograph sample 461 bar (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) at load of 100N.

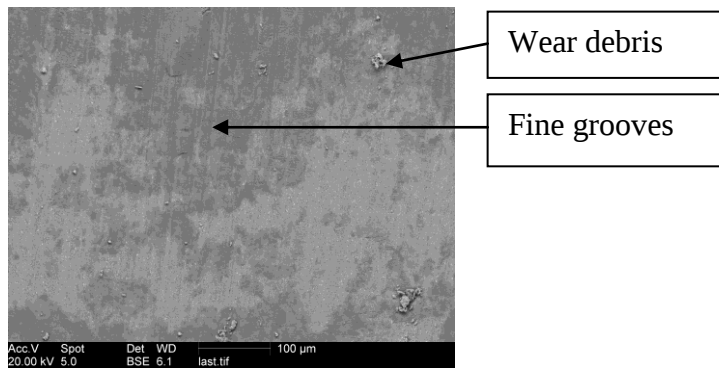
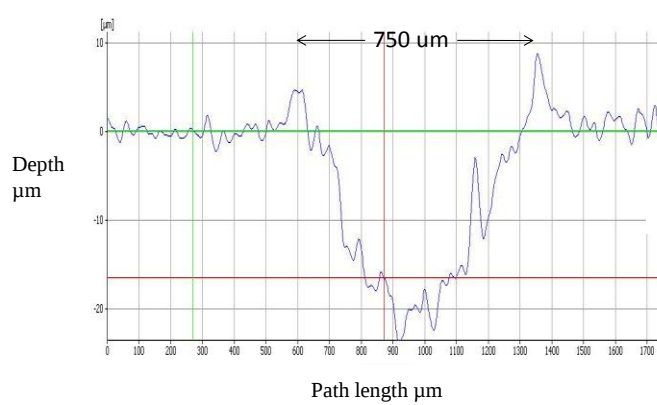
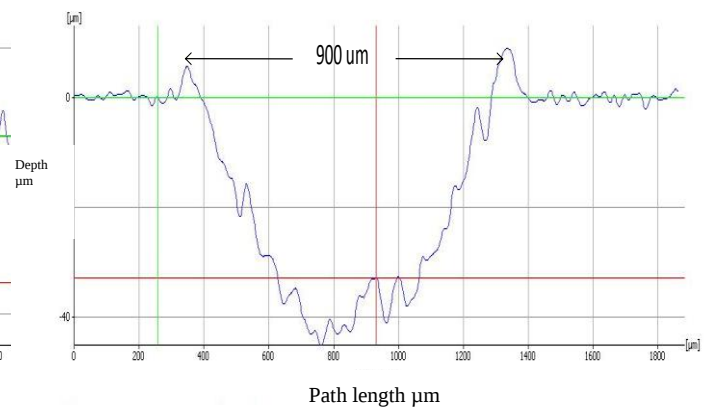


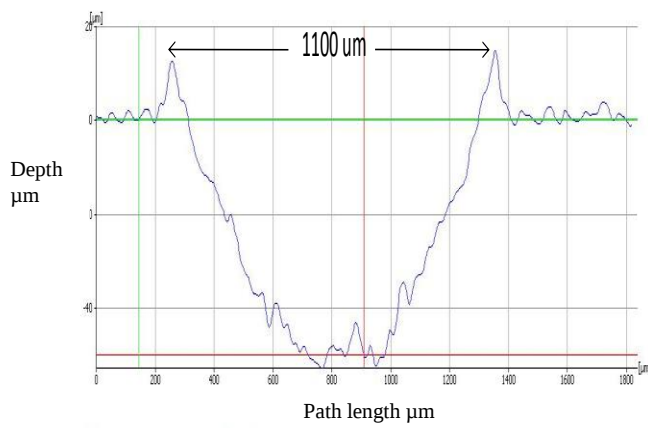
Figure 5.10.5 FEGSEM backscattered micrograph sample 461 bar (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) at load of 100 N.



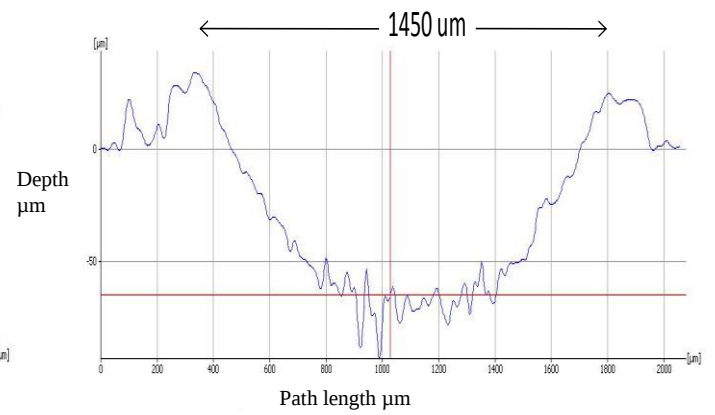
(a)



(b)



(c)



(d)

Figure 5.10.6 Wear volume profiles. (a) Sample number 461 extruded bar at 10N. (b) Sample number 444 extruded bar at 10N. (c) Sample number 431 extruded bar at 10N. (d) Sample number 461 extruded bar at 100N.

5.10.2 Wear Debris

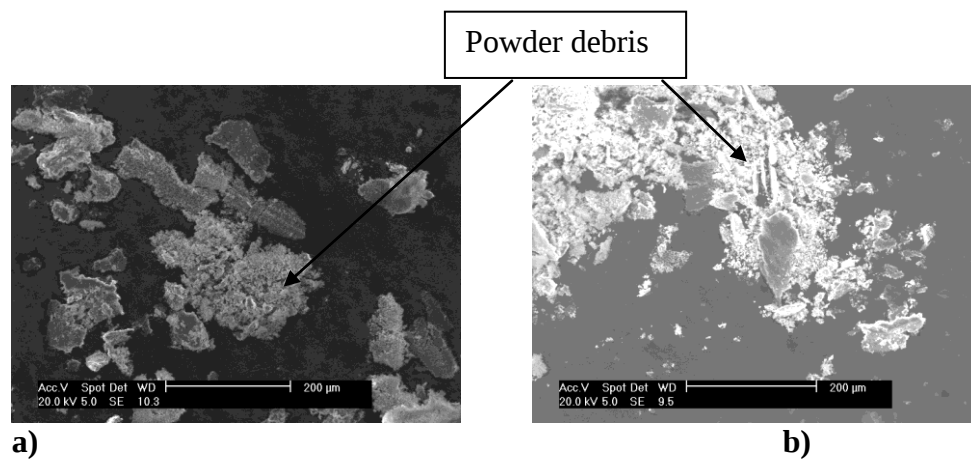


Figure 5.10.7 Wear debris at 10 N

a) Sample 444-4042 FEF (according to table 4.1 Al 30.3Si 1.5Cu 1.2Mg 1.1Fe 1.4Ni 0.2Zr) FEGSEM micrograph taken at 20 keV.

b) Sample 446-4051-FEF-2 (according to table 4.1 Al 20.3Si 2Cu 1.5Mg1.8Fe 1.9Ni 0.2Zr) FEGSEM micrograph taken at 20 keV.

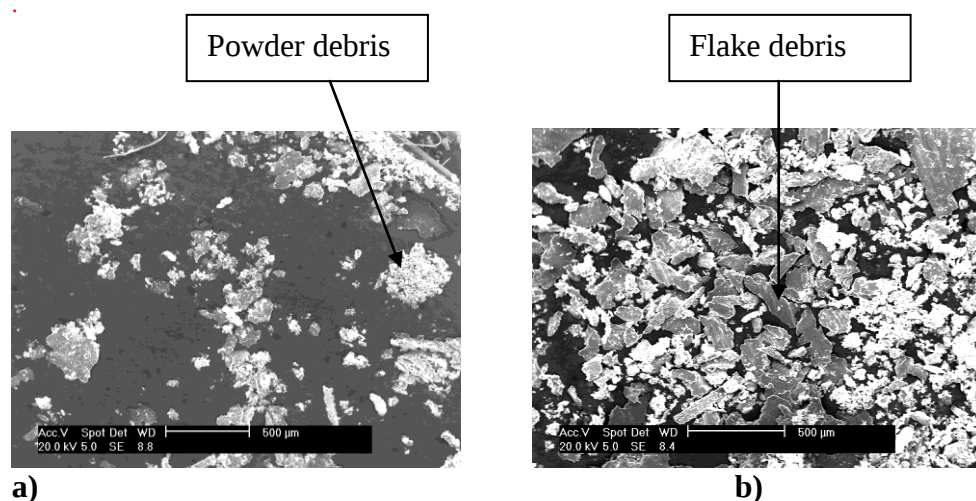


Figure 5.10.8 Wear debris at 100 N

a) Sample 444-4042 FEF (according to table 4.1 Al 30.3Si 1.5Cu 1.2Mg 1.1Fe 1.4Ni 0.2Zr) FEGSEM micrograph

b) Sample 446-4051-FEF-2 (according to table 4.1 Al 20.3Si 2Cu 1.5Mg1.8Fe 1.9Ni 0.2Zr) FEGSEM micrograph taken at 20 keV.

5.10.3 The Counterface

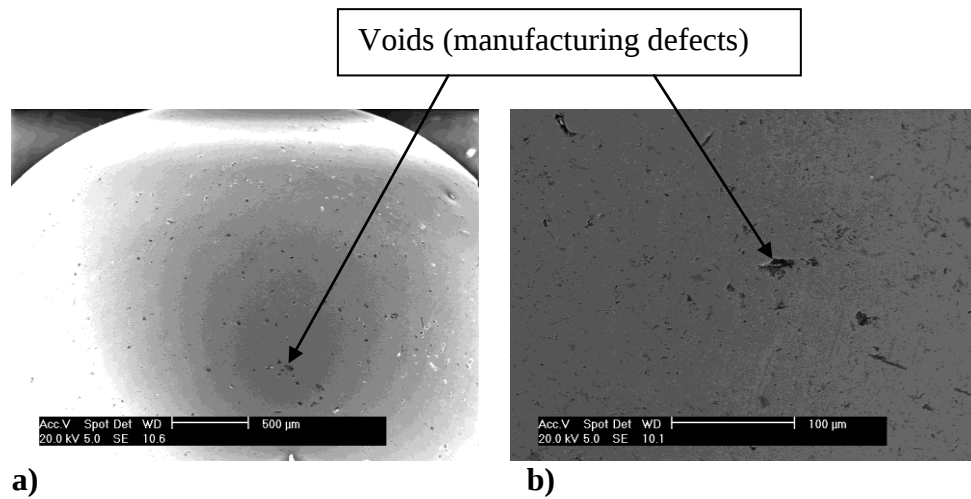


Figure 5.10.9 FEGSEM micrograph taken at 20 keV of the ball bearing.

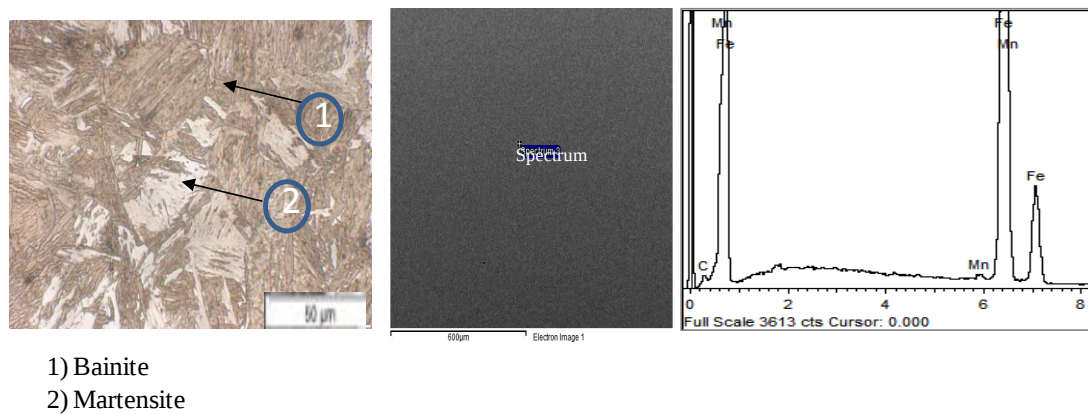
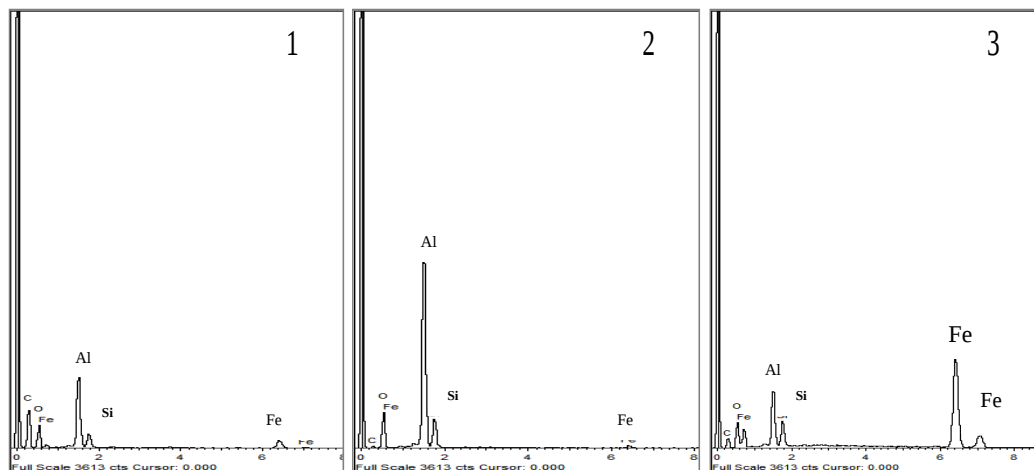
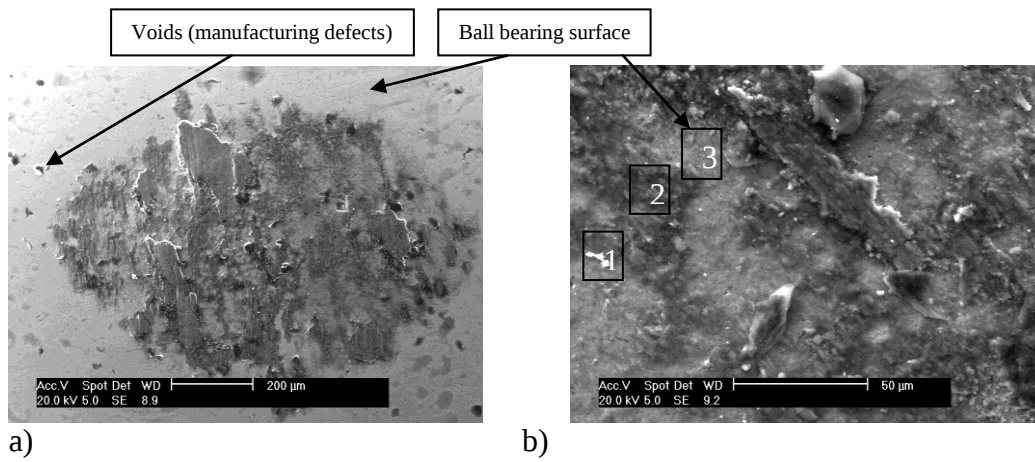
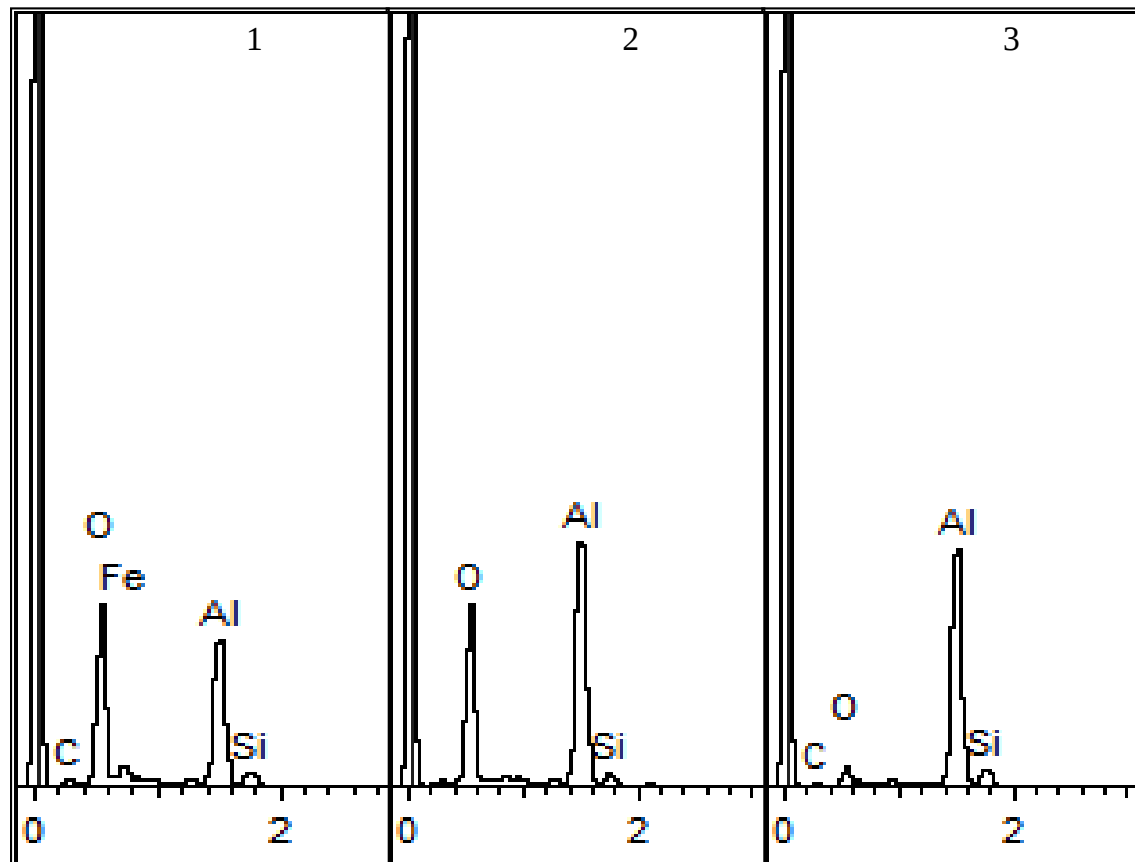
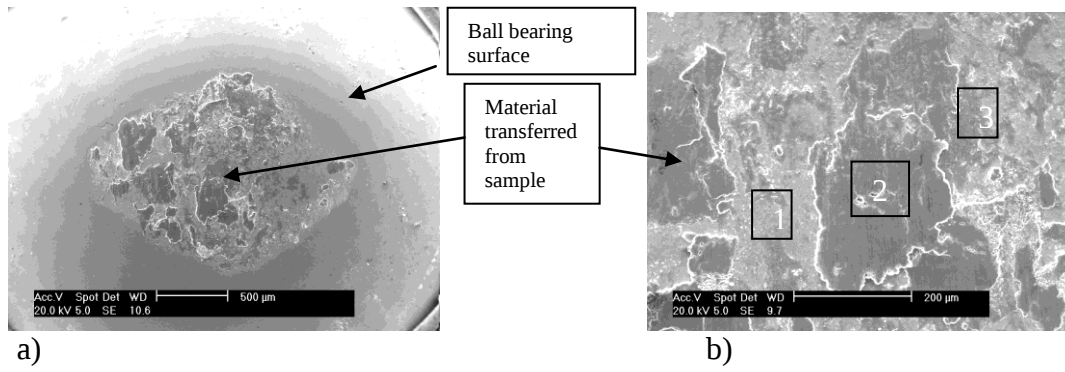


Figure 5.10.10 Optical and EDX spectra for unused ball bearing.



c)

5.10.11 FEGSEM micrograph taken at 20 keV of the ball bearing at 10 N and EDX spectra for points 1, 2 and 3 in the FEGSEM micrograph. (Wear sample No. 461).



c)

5.10.12 FEGSEM micrograph taken at 20 keV of the ball bearing at 100 N and EDX spectra for points 1, 2 and 3 in the FEGSEM micrograph for sample 461 extruded bar.

5.10.4 Effect of Alloying Elements

Wear resistance and hardness are known to be closely interrelated hence wear volume versus the hardness is plotted. The wear volume has then been plotted against every individual alloying element. Only Si, Cu and Fe show any consistent trend. Therefore, only these graphs are shown here.

The effect of alloying elements is analysed at two different loads, low load i.e. 10N and high load i.e. 100N. It should be noted that some alloying elements (e.g. Zr) are only present at a relatively low level (i.e. 0.2wt%) and some did not vary much between the materials provided by RSP (e.g. only between 1.2 and-1.6wt% and Ni between 1.4 and-1.9wt.% - see Table 4.1).

5.10.4.1 Wear at 10 N

Figure 5.10.13 illustrates the wear volume versus hardness at 10N. The effect of individual element on wear resistance was attempted in this study. The difficulty is that the alloys contain more than 6 alloying elements, but only limited number of samples was provided by the industry and each sample has different level of alloy concentration. Therefore, elements with strong effect on the wear resistance of Al-high Si alloys from the literature were analyzed based on the samples I received. These elements are Si, Cu and Fe. The measured wear volume results versus Si, Cu and Fe contents are plotted in Figures 5.10.14-5.10.16 respectively at a load of 10N. In the Figure 5.10.14, samples with two different levels of Si contents are highlighted in two circles. In this plot alloy 446-2, 446-3 461 contain a about 20wt% Si, while alloy 461 and 444 have about 30wt%Si. Although Cu and Fe concentration varies in these alloys, there is probably a clear trend illustrating the effect of Si on wear resistance. In the Figure 5.10.15, the wear resistance versus Cu concentration are

analyzed. It can be seen that increasing Cu content reduces the wear resistance. It shall be noted that variation in other alloying elements also occurs in the alloys analysed here. In the Figure 5.10.16, the wear resistance versus Fe concentration are presented. A similar trend as the Cu effect can be observed for Fe. Finally, Wear volume versus hardness of billet 431 and 461 degassed plus HIPped at 10N is plotted in figure 5.10.17.

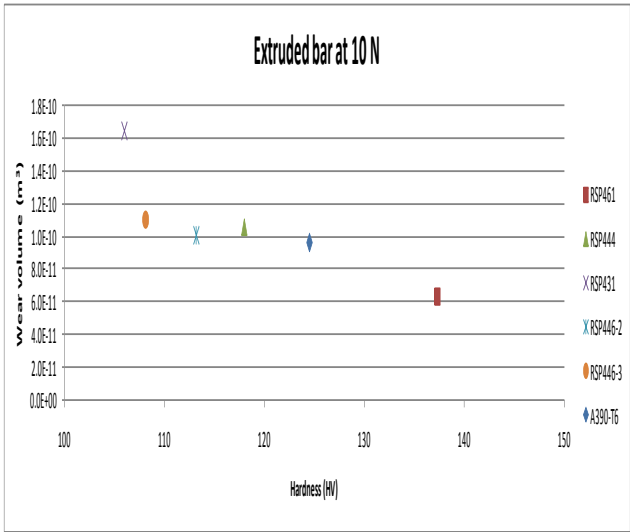


Figure 5.10.13 Wear volume versus hardness at 10N.

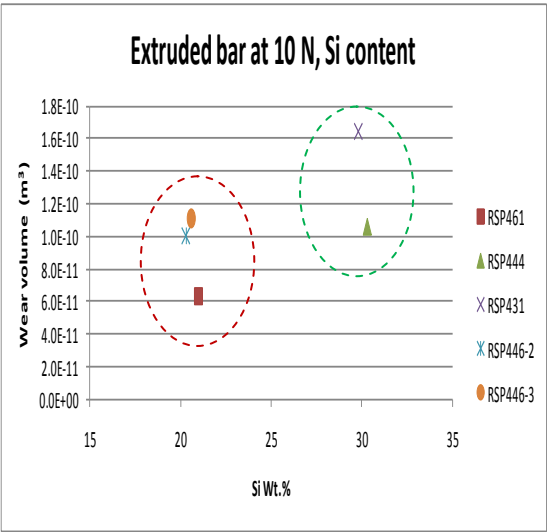


Figure 5.10.14 Wear volume versus Si wt.% at 10N.

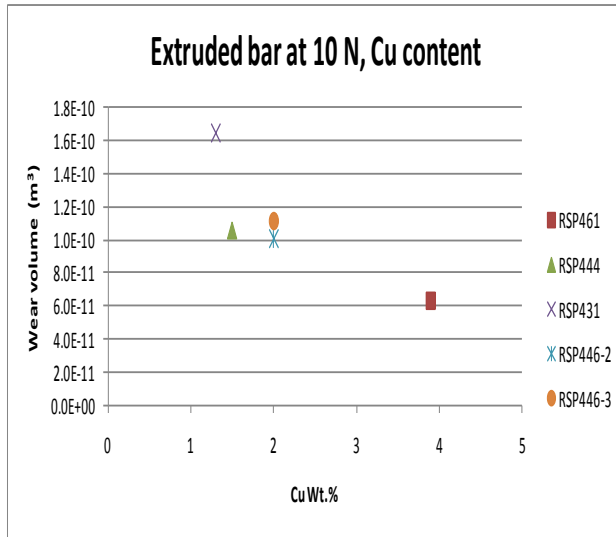


Figure 5.10.15 Wear volume versus Cu wt.% at 10N.

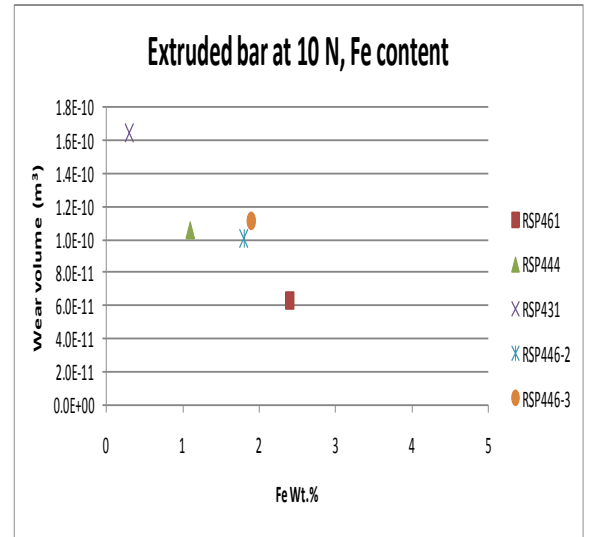


Figure 5.10.16 Wear volume versus Fe wt.% at 10N.

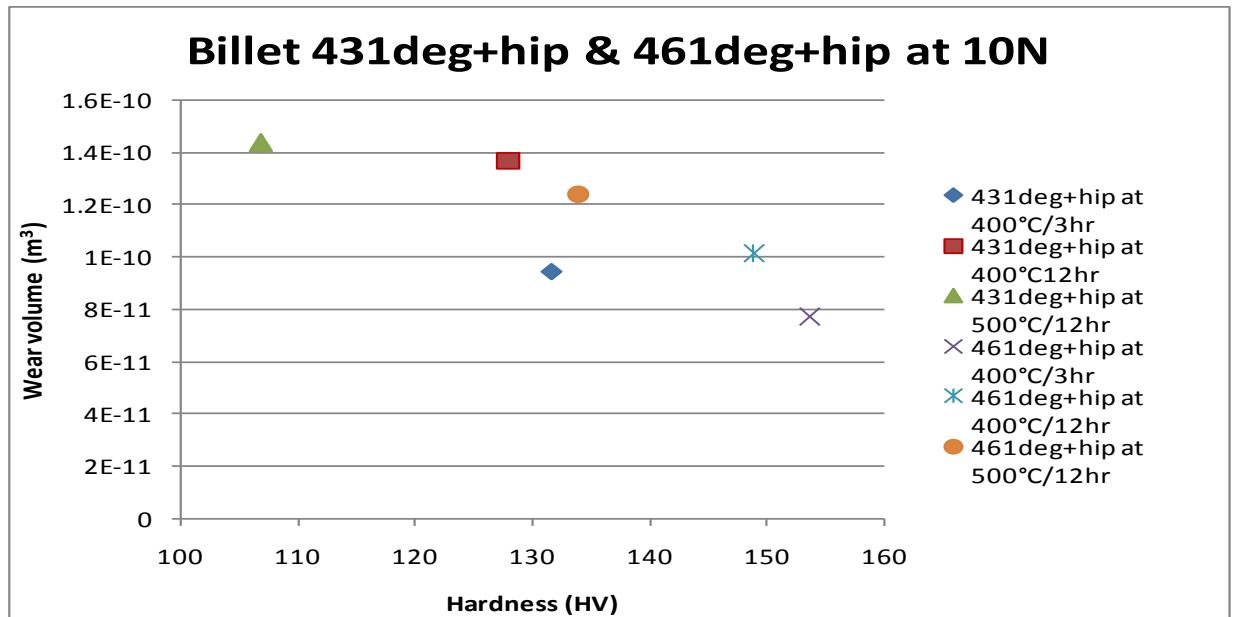


Figure 5.10.17 Wear volume versus hardness of billet 431and 461 degassed plus HIPped at 10N.

5.10.4.2 Wear at 100 N

Wear volume versus hardness at 100N is shown in figure 5.10.18. The alloys used at 100N are the same as those used under 10N. Analysis on the effect of Cu, Fe on wear resistance was attempted and results are shown in Figures 5.10.19 - 5.10.20 respectively. Again due to the limited number of samples, concentration of other elements, such as Si, Ni, also varies in the plot. Figure 5.10.21 shows wear volume versus hardness of billet 431 and 461 degassed plus HIPped at 100N.

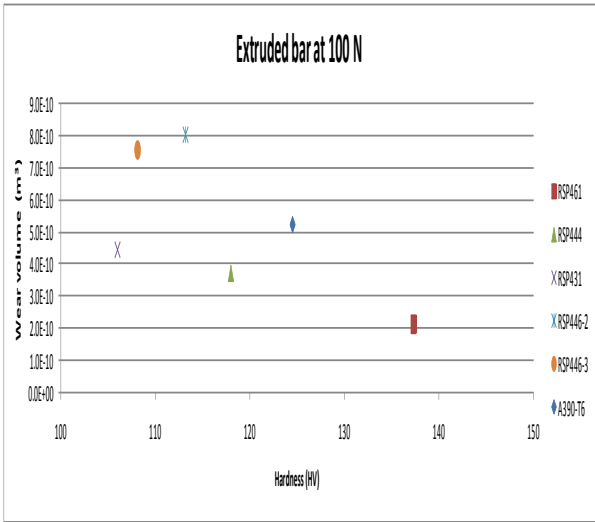


Figure 5.10.18 Wear volume versus hardness at 100N.

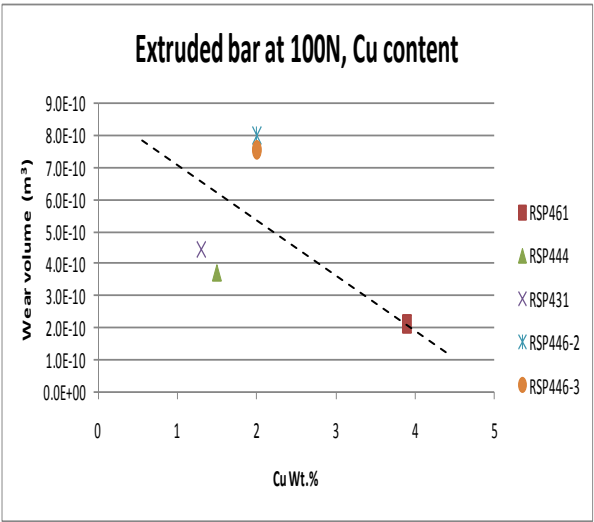


Figure 5.10.19 Wear volume versus Cu wt.% at 100N.

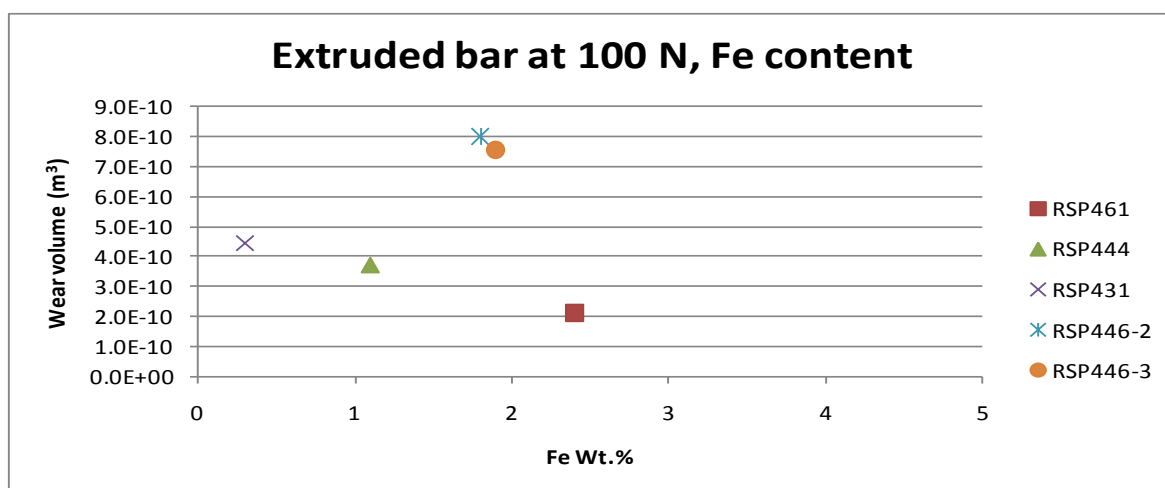


Figure 5.10.20 Wear volume versus Fe wt.% at 100N.

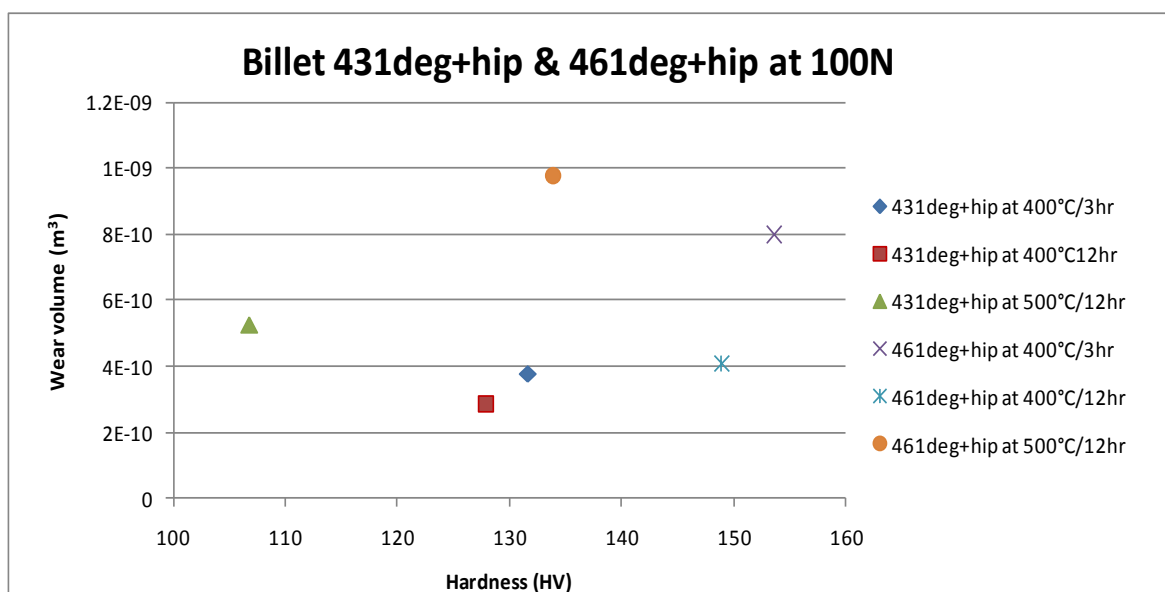


Figure 5.10.21 Wear volume versus hardness of billet 431and 461 degassed plus HIPped at 100N.

5.10.4.3. Comparison of Wear Volume at Load of 10N and 100N

Wear volume versus the hardness of both the extruded bars and A390 is plotted in figure 5.10.22. This is mainly to characterise the wear behaviour of these materials at low and high loads. Data for 100N test are enclosed by a dotted red line and the data for 10N are enclosed by a dotted blue line for comparison.

To analyse the effect of area fraction of Si (i.e. as an alloying element) on the wear resistance, the wear volumes versus the measured area fraction of Si for extruded bars and degassed plus HIPed billets are illustrated in figures 5.10.23-5.10.25.

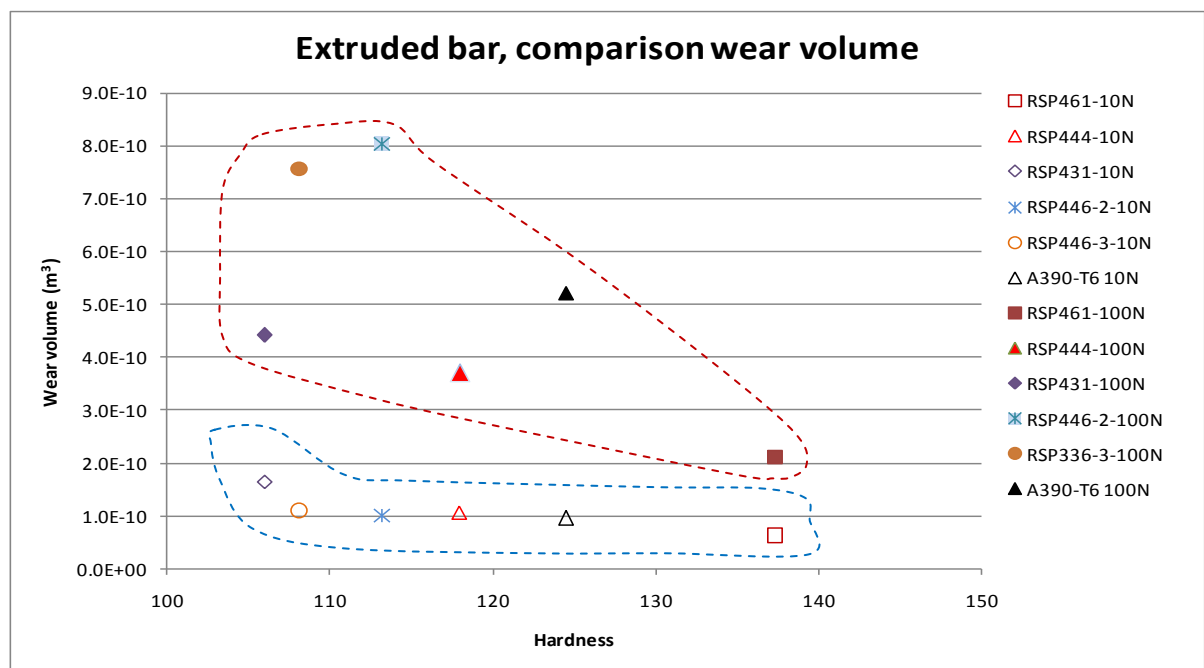


Figure 5.10.22 Wear volume versus hardness at 10 and 100N.

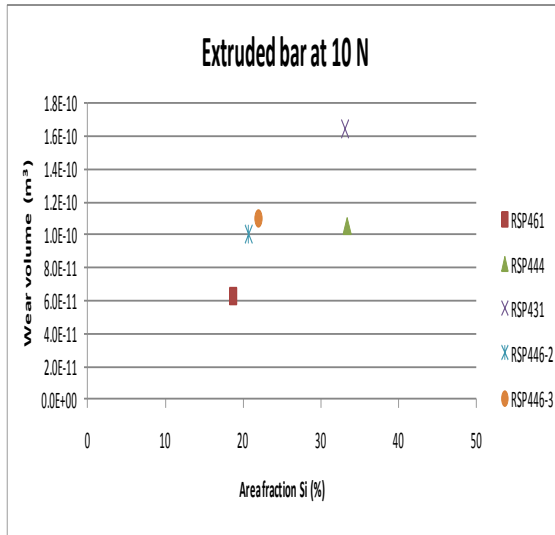


Figure 5.10.23 Wear volume versus calculated area fraction of Si of extruded bars at 10N.

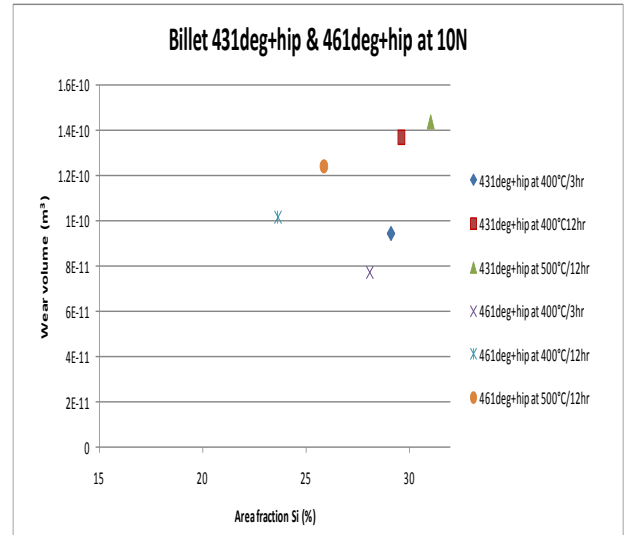


Figure 5.19.24 Wear volume versus calculated Si area fraction of degassed plus HIPped billets 431 and 461 at 10N.

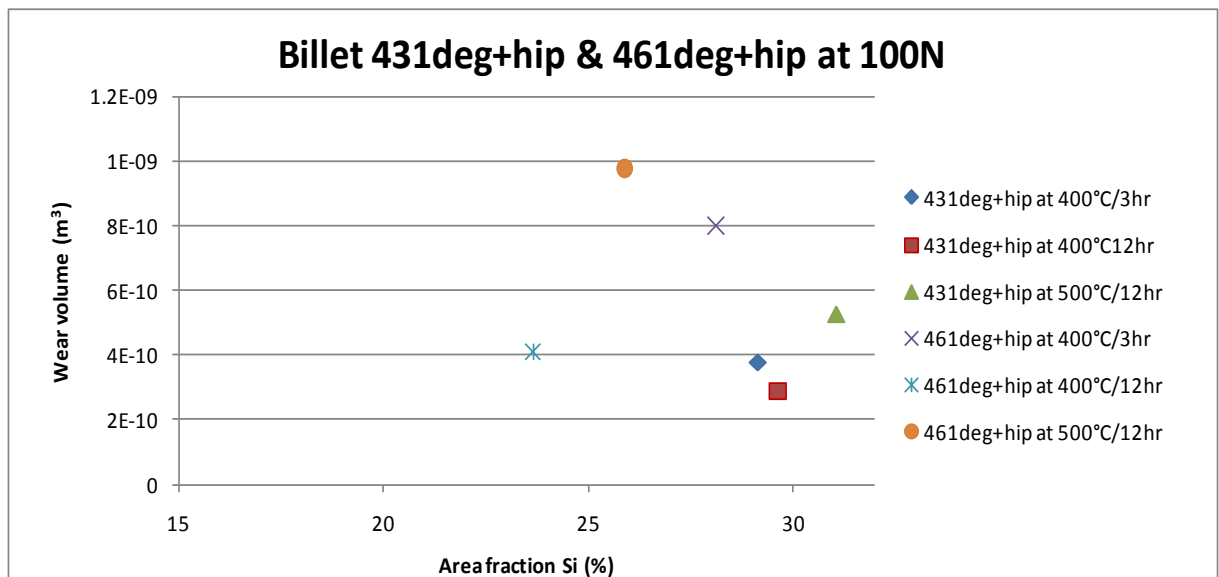


Figure 5.10.25 Wear volume versus calculated Si area fraction of degassed plus HIPped billets 431 and 461 at 100N.

5.11 Comparison of Wear Rate with Other Sources

Comparison of wear rates cannot be accurate unless the contact pressure is known for all the wear rates. Contact pressure for all RSP materials (.i.e. carried out in this research) will be calculated here. Also, wear rates and contact pressure from the literature (see table 2.4 section 2.11.3) will be compared with RSP wear rates in the discussion in chapter 6.

Ball on flat contact area can be calculated as follow [117]:

$$a^3 = \frac{3 PR}{4 E^*}$$

$$a = \left(\frac{3 PR}{4 E^*} \right)^{1/3}$$

where:

a=the radius of point contact circle

P= Load

R=ball radius

Pressure= Force/Area

$$A = \pi R^2$$

$$\frac{1}{E^*} = \frac{(1 - \nu_s^2)}{E_s} + \frac{(1 - \nu_b^2)}{E_b}$$

where: E^* = reduced modulus

For the ball [118]:

Elastic modulus of the ball (E_b) = 193 GPa

Poisson's ratio of the ball (ν_b) = 0.285

For RSP alloy [118]:

Elastic modulus of the alloy (E_s) = 75 GPa

Poisson's ratio of the alloy (ν_s) = 0.33

Contact pressure (MPa)	Contact pressure (kPa)	Contact pressure (Pa)	Area of contact (m ²)	Radius, a (m)	Load, P (N)	Ball radius, R (m)	E* (reduced modulus)	1/E*	ν_s (alloy)	E_s (alloy) (Pa)	ν_b (ball)	E_b (ball) (Pa)
6.9 X10 ²	6.9 X10 ⁵	6.9 X10 ⁸	1.4 X10 ⁻⁸	6.7 X10 ⁻⁵	10	2.5 X10 ⁻³	6.01 X10 ¹⁰	1.664 X10 ⁻¹¹	0.33	7.50 X10 ¹⁰	0.285	1.93 X10 ¹¹
1.5 X10 ³	1.5 X10 ⁶	1.5 X10 ⁹	6.7 X10 ⁻⁸	0.00014	100							

Table 5.11.1 Calculation of contact pressure for RSP materials at 10 and 100 N.

Wear rates must be calculated in equivalent units (i.e. m³.m⁻¹) to compare for all materials. Therefore, wear rate in table 2.4 section 2.11.3 was converted to (m³.m⁻¹) for ease of comparison.

RSP material	Load 10 N	Load 100 N
	Contact pressure (kPa)	
	6.9×10^5	1.5×10^6
	Wear rate ($\text{m}^3 \cdot \text{m}^{-1}$) $\times 10^{-12}$	Wear rate ($\text{m}^3 \cdot \text{m}^{-1}$) $\times 10^{-12}$
RSP461	6.3	21.2
RSP444	10.6	37.2
RSP431	16.5	44.2
RSP446-3	11	75.6
RSP446-2	10	80.4
461deg at 400°C/3hr+hip	7.7	79.9
461deg at 400°C/12hr+hip	10.2	40.8
461deg at 500°C /12hr+hip	12.4	97.6
431deg at 400°C /3hr+hip	9.5	37.8
431deg at 400°C /12hr+hip	13.7	28.8
431deg at 500°C /12hr+hip	14.4	52.4

Table 5.11.2 Wear rates of RSP materials.

Summary

This chapter is the result for flake and ribbon microstructure as-received, cold compaction and degassing at the University of Leicester, degassing and HIPping at Bodycote, bar microstructure as-received, Si particle measurements, hardness testing and wear.

Key results are the microstructure on the wheel side is very fine indeed and coarse on the air side. On the wheel side Al and Si regions cannot be clearly distinguish on the microstructure. Based on the microstructure, rapid solidification process reduced the amount of silicon segregation tremendously.

There is clear evidence from large Si size particle for coarsening during degassing particularly at 500°C but far less at 400°C. Air side and wheel side microstructures have great influence on the microstructure during cold compaction or Hipping stage. Extruded bar 461 shows very fine microstructure. Based on the Si particle measurements, there is a clear coarsening behaviour of the Si particles at 500°C. There is clear evidence that hardness has a great influence on the wear resistance of aluminium silicon alloys i.e. as hardness increase the wear resistance increases. The microstructures approved a mixture of adhesive and abrasive wear mechanism. The counterface showed a clear evidence of adhesion mechanise where a transferred of ductile matrix i.e. Al to the ball bearing take a place. Moreover, the grooves which were on the sliding direction approved abrasion resistance due to the entrapment of Si. Wear debris microstructure approved flake debris at high load and low Si content. Wear resistance is effected by the Si, Cu and Fe content.

CHAPTER 6

DISCUSSION

Flake and Ribbon Microstructure As-Received

Figure 5.2.1 shows an optical micrograph of the microstructure of sample numbers 431-14053 and 461-L2. Figure 5.2.1a shows air side microstructure which consists of fine silicon particles uniformly distributed throughout the aluminium matrix. Similar results have been reported elsewhere [58, 119, 120].

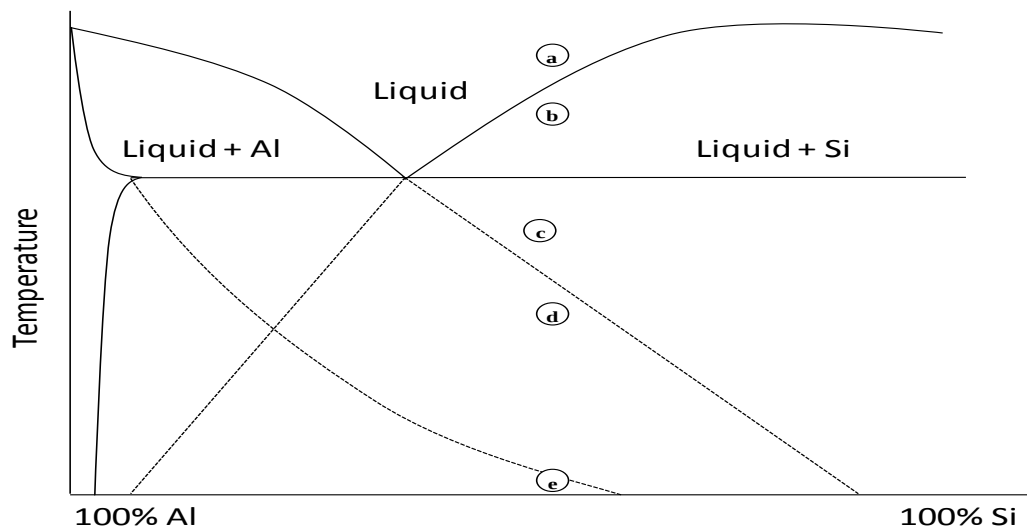


Figure 6.1 Schematic diagram of Al-high Si extended phase diagram to illustrate figures 5.2.1 and 5.2.2.

Figure 5.2.1b shows two zones: featureless zone and coarse Si particle zone. The highest cooling and solidification rates occur at the wheel side which is known as the chill zone. This leads to very small crystallites in this region. Therefore, zone (1) is believed to be the wheel side and zone (2) is the air side. An initial development of columnar grain boundaries in the direction of the wheel (drum) normal is due to high

cooling rate. Similar results have been reported [56, 121-123]. The wheel side with the relatively featureless zone can also be seen in figure 5.2.2a-c. It can be seen in these micrographs that the microstructures for the air sides are coarser than those of the wheel side. Similar results have been reported elsewhere [70, 124-126].

The featureless zone which is point (a) in figure 5.2.1b is amorphous and this can be clearly seen in point (1) figure 5.2.2c. The phase expected at this stage is liquid based on point (a) in figure 6.1. Undercooling of the molten metal occurs due to the high cooling rate. Some structure is visible in figure 5.2.1points (c,b) which correspond to point (4) in figure 5.2.2c however, it is not clear whether it is amorphous or a crystalline and this only can be clarified with TEM which is not possible due to the difficulty with specimen preparation on this project. The solidification of this sample can be explained using figure 6.1. With slow cooling of an Al-20%Si alloy, at 'a' the alloy is liquid. As the alloy cools, solid Si starts to form at 'b'. With further decrease of temperature, at 'c' the remaining liquid becomes eutectic.

With fast cooling (but not so fast) the liquid is quenched to an amorphous structure of the same composition, little diffusion can take place. It is as though the liquidus and solidus from the left hand side are extended. At 'b', solid Si does form, and at 'c' eutectic, but at 'd' some alpha aluminium will also be present and this can be seen in figure 5.2.2.1a. This is not an equilibrium phase in an Al-20%Si alloy so will only be present under rapid solidification conditions.

Figure 5.2.3 shows TEM micrographs of the ribbon sample 461. Figure 5.2.3a shows four different points. Points 1 and 2 which are relatively pale on the micrograph have relatively high content of aluminium and points 3 and 4 which are appearing dark on the micrograph and have a high content of silicon. These points were analysed in

figure 5.2.3b. Figure 5.2.4 shows this finding. Figure 5.2.5(a,b) shows FEGSEM micrographs of the flake sample 461-16030. The microstructure shows rosette like morphology. Similar results have been reported elsewhere [119, 127, 128]. Figure 5.2.5b shows two different points. Point number one which is appearing dark grey on the micrograph has a high content of Si and point number two which is appearing white on the micrograph has a high content of Al (see figures 5.2.6 and 5.2.7). The difference in contrast must be due to the difference in the composition in terms of Fe Ni and Cu since these elements heavier than Al and Si which have very similar atomic numbers.

Figure 5.2.8(a-b) shows FEGSEM micrographs of sample 461-L2. Figure 5.2.8a shows two different zones for the same material. Zone (1) appears to have smooth (fine) structure on the micrograph and is believed to be the wheel side, and zone (2) which appears to have rough (coarse) structure on the micrograph is believed to be the air side. Figure 5.2.8(b) show air side at higher magnification. Figure 5.2.9 gives the EDX spectrum for zone (1) in micrograph 5.2.8a and figure 5.2.10 shows EDX spectrum for zone 2 in micrograph 5.2.8a. Both zones showed similar amounts of Si and Al i.e. there is little evidence for segregation from the wheel side towards the air side during solidification. The amount of Si for zone 1 is 23.81 wt.% and the amount of Si for zone 2 is 22.60 wt.%. EDX quantitative analysis can be seen in appendix 4 tables A 4.1 and A 4.2.

Cold Compaction and Degassing and at the University of Leicester

Figure 5.3.1 shows as-melt spun microstructure of 461 ribbon (Al 21Si 3.9Cu 1.2Mg 2.4Fe 1.4Ni 0.4Zr) alloy. The microstructure of figure 5.3.1b is comprised of a high Si phase embedded in an Al matrix. It is clear from this figure that the Si phase exhibits equiaxed particulate like morphology. The average diameter of the large Si particles (i.e. those which can be measured optically) in the as-melt spun condition in the zone near the air side is $2.5\mu\text{m} \pm 0.2$. Figure 5.3.1a shows an optical micrograph of sample 461 ribbon end down cross section (see figure A.2.1, appendix 2). It shows coarse Si particles in what is believed to be the air side zone. However, a featureless microstructure is evident on the other side which is believed to be the wheel side. It should be mentioned that air and wheel sides can be easily distinguished in end down (transverse) cross section because it is only the air side which shows coarse Si particles. The diameters of Si particles in figure 5.3.1a increase on moving towards the air side. Silicon particle diameter measurements in figure 5.3.1(a,b) are shown in table 5.3.1. Since the average diameter of the larger Si particles in figure 5.3.1b ($2.6\mu\text{m}$) is almost the same as in figure 5.3.1a ($2.5\mu\text{m}$), therefore, figure 5.3.1b is deduced to be air side. If the average diameter of the Si particle in figure 5.3.1b is less than the average diameter of Si particle in 5.3.1a, then figure 5.3.1b would be a wheel side. Longitudinal micrograph cross sections can only be identified as to whether they are air side or wheel side by comparing Si particle diameter measurements with end down (transverse) cross section measurement. Following degassing at 500°C for 3hrs as shown in figure 5.3.2(a,b), the average diameter of the large Si particles is $3.2\mu\text{m} \pm 0.3$ (see table 5.3.1). It can be observed that the Si particles have very clear dimensions, especially in the unetched samples, compared with as-received samples. Sastry et al. [129] reported that 500°C caused coarsening

of silicon particles in solidified melt spun flakes. Figure 5.3.3a shows the optical micrograph of 461 ribbon degassed at 300°C for 3 hours end down cross section. The average diameter of the large silicon particles is $2.5 \mu\text{m} \pm 0.2$. Figure 5.3.3b shows a micrograph of the same sample in longitudinal cross section. The average diameter of the silicon particle is $1.6 \mu\text{m} \pm 0.2$ (see table 5.3.1). Similarly, since the average diameter of the silicon particles in figure 5.3.3b is less than that in figure 5.3.3a; therefore, figure 5.3.3b is a wheel side sample.

Figures 5.3.4 and 5.3.5 show optical micrographs of UOL 461 flakes cold compacted. The cold compaction pressure calculation can be seen in appendix 5. Figure 5.3.4a and 5.3.5 (a,b) show particle-free (i.e. Al matrix) region due to lack of compaction. All the micrographs in figures 5.3.4 and 5.3.5 show a region of very fine Si particles and just next to it a region of coarse Si particles. This makes it difficult to measure the Si particle size within the sample. A potential explanation for the observation of regions of fine Si particles next to regions of coarse Si particles is that fine Si particles are coming from drum side and coarse Si particles are coming from the air side of flakes. Also flakes are splitting into two parts along the lengthway of the ribbon (one wheel and the second air) during the production stage.

Mapping and Line Scan of As-Received and Degassed Ribbon

Elemental mapping of sample 461 ribbon is shown in figure 5.4.1. Figure 5.4.1b shows high concentration of Al element and figure 5.4.1f shows high concentration of Si element. Other elements have low concentration; this is probably due to their low content (less than 3.9 wt. %). These are Cu, Fe and Mg (3.9, 2.4, 1.2 wt. %). However, there are some regions where Cu, Fe and Mg are concentrated. Ni element and Zr element maps show that these elements are uniformly distributed. Particles

based on Cu, Fe and Mg are expected to present in RSP samples. It was very difficult to prepare TEM samples to confirm the identity of the particles. TEM samples tend to break whenever taking them out of the holder due to brittleness.

FEGSEM line scan micrographs of sample 461ribbon are shown in figure 5.4.2. Figure 5.4.2a shows high peak of Al and Fe at 5 μ m from the left hand of the scan while figure 5.4.2b shows high Fe and Ni peaks at 1-2 μ m on the horizontal axis. The high peak for Fe in both cases is believed to be associated with an intermetallic particle. Cu shows its maximum at 14 μ m in figure 5.4.2a while it is at 11-12 μ m in figure 5.4.2b. It should be mentioned that the microstructure is very fine therefore it was difficult to get a line scan which shows the particles up in the as-received.

Degassing and HIPping at Bodycote

Optical micrograph of sample BDC 461 unetched is shown in figure 5.5.1. The sample was degassed at 400°C for 3 hrs and HIPped. Figure 5.5.1a shows fine Si particles; the distribution is inhomogeneous with some regions relatively sparsely populated and others much denser. Figure 5.5.2 shows FEGSEM backscattered of sample BDC 461. Figure 5.5.2b is the same micrograph as in figure 5.5.2a but at high magnification. There is an inhomogeneity in the microstructure. Both micrographs show particle-free regions (i.e. Al matrix). Figure 5.5.3 shows two different points for the same material as in figure 5.5.2b. EDX spectra for these two points are shown. Point number one which is appearing dark on the micrograph has very high content of Al and little Si. Point two which is appearing light grey on the micrograph has high content of Al and some Si. This shows that there is an inhomogeneity in the microstructure at the HIPping stage.

Figure 5.5.4 and figure 5.5.5 are optical micrographs of sample BDC 461 unetched and etched. Figure 5.5.4b and figure 5.5.5b show big “spots” of aluminium matrix. In these two micrographs there are two regions, one region has high content of fine Si particles and another one has high content of coarse Si particles. Figure 5.5.5a shows the “wave type” of microstructure. The explanation for this is that during the HIPping step air side flakes are high in one region, hence formation of the coarse silicon region. Similarly, wheel side flakes tend to have very fine Si particles. The flakes are thought to be splitting lengthways along the interface between the wheel and the air side forming very fine particles in one region and coarse particles on another. The flake average width is 1.2mm and length is 1.4mm. However, ribbon average width is 2.5mm. This suggests the ribbons have been split lengthways during chopping.

Figure 5.5.6 shows a FEGSEM backscattered micrograph of sample BDC 461. The Si particles (which are grey rather than the intermetallics which are white) are forming a necklace pattern (i.e. network of Si particles) in figure 5.5.6b. This mesh type of Si is believed to be the reason of giving the matrix the strength but brittle. To analyse the particle-free region on figure 5.5.6b two different points are taken on the micrograph of figure 5.5.7. Point number one which is appearing light grey on the micrograph is high in both Al and Si and point number two which is appearing dark on the micrograph has a high content of Al but very little Si. EDX spectra are shown for these findings.

Figure 5.5.8 shows optical micrograph of sample BDC 461 unetched. The sample is taken from the top edge of the canister. It shows white spots in the Al-matrix. This could be that the Al is being extruded between the Si particles by the HIPping to form these Al pockets. Figure 5.5.9 shows optical micrograph of sample BDC 461 unetched

degassed at 500°C for 12 hrs and HIPped. The sample was taken from the middle of the canister. Figure 5.5.9a shows two regions, one with coarse Si and the other with fine Si particles. Figure 5.5.9b is the same area as figure 5.5.9a but taken at higher magnification. Figure 5.5.10 shows a FEGSEM micrograph of sample BDC 461 degassed at 500°C for 12 hrs and HIPed. Figure 5.5.10a shows an overall view while figure 5.5.10b shows the same micrograph but at higher magnification. It is the same sample as in figure 5.5.8 i.e. billet top edge. Micrograph 5.5.10b shows coarse white particles which are needle shaped believed to be the intermetallic particles. The coarsening characteristic of figure 5.5.10b is due to the high heat effect of degassing at 500°C for a relatively long period compared with at low temperature.

Figure 5.5.11 shows FEGSEM backscattered micrograph of sample BDC 461 degassed at 500°C for 12 hrs and HIPed. The sample is taken from another top edge (left hand side) of the same billet on figure 5.5.8. Figure 5.5.11a shows fine and coarse Si particles regions. Figure 5.5.11b is the same micrograph as on figure 5.5.11a but at higher magnification. Figure 5.5.11b show needle coarse particles believed to be intermetallic particles.

Figure 5.5.12 shows an optical micrograph and a FEGSEM backscattered micrograph of sample BDC 431 degassed at 500°C for 12hrs and HIPped. Figure 5.5.12a shows a lot of black spots on the micrograph which are believed to be particle-free regions (possibly porosity). Figure 5.5.12b shows the same micrograph (i.e. the top right corner) but at higher magnification. This micrograph (5.5.12b) shows high concentration of Si in one region which lead to leaving the matrix without support. This type of material will yield poor mechanical properties. Micrograph 5.5.12c

shows coarse Si particles. Figure 5.5.12d shows dark spots which is believed to be particle-free region (possibly porosity). Needle shaped intermetallics are visible.

Figure 5.5.13 shows FEGSEM backscattered of sample BDC 431. Seven different points are taken on the micrograph. Point numbers 1, 2, 4 and 6 which appear dark on the micrograph have a high content of aluminium but very low in Si. EDX spectra for these points are shown on figures 5.5.14, 5.5.15, 5.5.17 and 5.5.19 which prove this finding. It should be mentioned that there is a possibility of oxides (BS is atomic sensitive, heavy elements appear darker than light elements). Similarly, point numbers 3, 5 and 7 which appear light grey on the micrographs have a high content of Si. EDX spectra for these points are shown on figures 5.5.16, 5.5.18 and 5.5.20. Table 5.5.1 shows the amount of Al and Si (i.e. wt. %) in the above mentioned points. EDX quantitative analysis can be seen in appendix 4, table A.4.3-A.4.9.

Figure 5.5.21 shows elemental mapping for sample 431 degassed at 500°C for 3 hrs and HIPped. It shows a high concentration of Al and Si, this is probably due to their high content (29.8Si 85Al wt. %). It also shows coarsening of Si particles due to high heat effect at long time as well. However, other elements have low concentration because of their low content (less than 4 wt. %); these elements are evenly distributed. It is expected that the needle shape particles are intermetallic Fe, Cu and Ni from literature evidence. However, the particles are too small to be analysed by EDX and cannot be identified here.

Bar Microstructure As-Received

Figure 5.6.1(a,b) shows optical and FEGSEM micrographs of extruded bar sample number 446-4051. The micrographs show very fine Si particles homogeneously distributed (with some clustering). The sample is free of porosity and defects. To

analyse Al and Si particles, three different spots were taken on figure 5.6.2. Spots numbers one and three are high Si particles, they had almost the same amount of Al and Si. Spot number two is the Al matrix. Figure 5.6.3 (b is the same region as a because there is no micron bar on b) shows two different points for the same material as in figure 5.6.1, both points showed intermetallic particles appearing white on the micrograph having the same amount of aluminium but slightly different amounts of silicon.

Figures 5.6.4-5.6.8 show optical microscopy and FEGSEM micrographs of hot extruded bars. The microstructures are composed of fine grey silicon particles and smaller intermetallic particles (black). It has been observed that silicon particles (appearing grey on optical micrographs) and intermetallic particles (appearing black on optical micrographs and white on FEGSEM) are present in the aluminium matrix. Figure 5.6.8b shows more intermetallic particles than figure 5.6.7b. This could be due to the fact that the composition of the material for figure 5.6.8 contains more Fe and Ni than the composition of figure 5.6.7. All samples showed a reasonably homogeneous overall distribution of the silicon particles. The micrograph of some samples (e.g. figure 5.6.8) showed very fine silicon particles. This is due to the addition of alloying elements such as Fe, Ni.

A comparison between the silicon particles of the optical micrographs in figures 5.6.7a and 5.6.8a reveal that the silicon particle size decreases as the silicon content increases (see table 5.7.2). This could be also due to the increase of Cu, Fe and Ni. Similar results have been reported by Rooyen et al. and Kaneko et al. [56-59, 64]. They reported that as Si content increases, the particle sizes decreases.

Figure 5.6.9 shows optical microscopy micrographs of hot extruded bar of sample 461. Figure 5.6.9a shows an overall view micrograph while figure 5.6.9b shows the same micrograph but at higher magnification. There are high content of Si in one part of the micrograph and leaving the other part free of particles which is called particle – free region. This can be seen clearly on the etched micrographs on figure 5.6.9(c, d). A potential explanation for this kind of behaviour is that the Si particles are forming an interconnected network during extrusion which can be overcome by heat treatment. This kind of inhomogeneous distribution of Si will lead to poor mechanical properties.

Si particle Measurements

Table 5.7.1 shows the area fraction from image analysis and that expected based on wt. %. Most of the actual area fraction of silicon is very close to the calculate area fraction. However, the actual area fraction of silicon for bar 461 (18.7%) is lower than the calculated (23.8%). Also, the actual fraction silicon for 461 degassed and hipped at 400°C for 3 and 12 hours and 500°C for 12 hours is higher than the calculated area fraction. However, this kind of deviation is expected in experimental work.

Table 5.7.2 shows the average silicon particle sizes of extruded bars and degassed and HIPped samples (appendix 6 shows images of area fraction and a sample of excel calculation). Extruded bar 444 and 461 have smaller particle size than extruded bar 431. Sample number 461 degassed and HIPped at 400°C for 3hrs shows the smallest particle size (1.3 μ m) of all the degassed and HIPed samples. The table also shows that as the degassing temperature or degassing time increases the particle sizes increase because of the heat effect.

Figure 5.7.1 represents the silicon particle size versus the degassing time. The graphs shows that there is a slight an increase in the particle size as the degassing time increases from 3 to 12 hrs for the 400°C degassing temperature for sample 461 and 431. However there is a sharp increase in particle sizes as the degassing temperature and degassing time increases. For instance the particle size of silicon for sample 431 degassed at 400°C for 3hrs is 1.4µm but it increases to 2.8µm when degassed for 500°C for 12 hr.

It is noticeable that sample 431 and 461 degassed at 400°C for 3hrs have almost the same silicon particle size. However, as the degassing time and HIPping temperature increase to 500°C for 12hrs both samples have different silicon particle size (i.e. 2.8 µm for sample 431 and 2.3 µm for sample 461 both degassed and HIPped at 500°C for 12 hrs). It should be mentioned that sample number 431 has 29.8 Si (wt.%) but lower other elements and 461 has 21 Si (wt.%) but higher other elements (see table 4.1 section 4.2). If we assume here that the coarsening rate is diffusion controlled rather than interface controlled the diffusion is being inhibited in the 461 in comparison with the 431. This could be due to the presence of intermetallics in the mixture between the Si particles or due to elements in solid solution in the matrix. Therefore, the diffusion rate of silicon on sample 431 is higher than sample 461. This is mainly due to the fact that intermetallic between the Si particles on sample 461 acts as a barrier which hinders the diffusion.

Extruded bar sample numbers 431 and 461 shows lower silicon particles (i.e. 2.7 and 2µm respectively) compared with 431 and 461 degassed and HIPped at 500°C for 12 hour (i.e. 2.8 and 2.3µm respectively). This is mainly due to the fact that extrusion breaks the particles and consequently smaller particles are obtained. The degassing

conditions for the extruded bar are likely to be close to the 400°C than 500°C. The actual degassing temperature for the extruded bar is not known because it is proprietary to Bodycote HIP.

X-Ray Diffraction

Figure 5.8.1 shows the intensity versus 2θ of extruded bar samples 446-3, 446-2, 444, 431 and 461 respectively. The intermetallic particles that are showing on sample 446-3 are Cu_3Si and $\text{Fe}_{0.42}\text{Si}_{2.67}$. However, Mg_2Si , Al_4Si and NiSi_2 are found in samples 461, 431 and 446-2 bars respectively. Other intermetallics in sample 444 were below the limit of detection. Also, if other intermetallics such as Al_3Ni and CuAl_2 were present, they were also below the limit of detection.

There are high amounts of Cu_3Si and NiSi_2 compared with other intermetallics. However, some of the intermetallics lines are overlapping making differentiation difficult. The intermetallics in each alloy can be seen in more detail in appendix 8.

It has been observed that intermetallics are present in samples which contain around 21% Si such as samples i.e. 446-3, 446-2 and 461. However, 444 which has a high content of Si (i.e. 30%Si in 444) did not show intermetallics. There is a small amount of Al_4Si in 431 which is a high Si alloy.

The microstructures in figures 5.6.6, 5.6.4b and 5.6.6b for samples 446-2, 446-3 and 431 respectively reveal the intermetallics having rounded morphology. These intermetallic particles are responsible for the increase of hardness and high wear resistance of the RSP samples. Also, they have a great affect on the coarsening behaviour of silicon particles during degassing because they act as a barrier which hinders the diffusion.

Hardness Testing

Figure 5.9.1 shows the hardness reading of each RSP sample. It is clear that alloy RSP 461 extruded bar has the highest hardness of all extruded bars. This is mainly due to the fact that RSP 461 has an optimum combination of Si and other alloying elements (low Si and high Fe+Cu) uniformly distributed in the Al matrix (see extruded bar micro structure in section 5.9). The high hardness of sample RSP 461 is due to the high amount of Cu (3.9 wt. %) and Fe (2.4 wt. %). This is probably due to solid solution strengthening mechanisms during extrusion. The second highest is RSP 444 and that is due to the combination of high Si (30.3) and lower Cu (1.5) and Fe (1.1) weight percent. The third highest is RSP446-2 and the reason for that is due to low weight percent Si (20.3) and lower Cu (2) and Fe (1.8) but higher Ni (1.9) and Mg (1.5) relative to 461.

Sample numbers RSP 431 and 446-3 have the lowest hardness. This could be due to the combination of high Si (29.8), low Cu (1.3) and very low Fe (0.3) for sample 431. However, for sample 446-3, it is the reverse combination of sample 446-3 which is low Si (20.6 wt. %) and high Cu (2 wt. %) and Fe (1.8 wt. %).

Therefore, high hardness is associated with the weight percent of Si, Cu and Fe. Hardness number increases, as the weight percent of Si increase and Cu and Fe decreases. Also, it increases as the as the weight percent of Si decreases and Cu and Fe increase.

When, comparing the hardness of degassed and HIPped samples, it is clear that BDC461 degassed at 400°C for 3hrs and HIPped has the highest hardness and alloy BDC461 degassed at 400°C for 12 hrs and HIPped is the second highest. However, alloy BDC431 degassed at 500°C for 12 hrs and HIPped and extruded bar

RSP431 show the lowest hardness among the degassed and HIPped and extruded bar respectively. This could be explained as a result of the relatively large silicon particle size (see table 5.7.2).

When comparing the hardness of RSP alloys with piston A390, it is clear that RSP 461 extruded bar, RSP 461 degassed and HIPped at 400°C for 3 and 12 hours, RSP 461 degassed and HIPped at 500°C for 12 hours and RSP 431 degassed and HIPped at 400°C for 3 hours have higher hardness than A390. This is due to the fact that all RSP alloys mentioned above have finer Si particles than A390 (see microstructure in section 5.9).

Wear Mechanism

In order to analyse the wear mechanism of the alloy, the surfaces of the worn sample were examined under FEGSEM. Figure 5.10.1 shows FEGSEM micrograph of worn surface of sample 461 extruded bar at load 10 N. Figure 5.10.1a shows the beginning of the worn surface and figure 5.10.1b shows the end of the worn surface.

Figure 5.10.2 shows backscattered micrograph of sample 461 extruded bar. Figure 5.10.2a shows the beginning of the worn surface and figure 5.10.2b shows the same micrograph but at higher magnification.

Debris was clearly seen on figure 5.10.1a and figure 5.10.2b. The debris shows that the steel counterface has worn the Al-Si alloy. Oxide is shown on the micrograph dark grey because the backscattered is sensitive to atomic weight and therefore shown darker.

Figure 5.10.3 shows FEGSEM micrograph of worn surface of sample 461 extruded bar at load 100 N. Figure 5.10.3a shows the beginning of the worn surface and figure

5.10.3b shows the middle of it. Longitudinal grooves have been observed on both micrographs, their directions are the same as the sliding direction.

Figure 5.10.4 shows FEGSEM backscattered micrograph of sample 461 extruded bar at load of 100N. Figure 5.10.4a shows thin layer coming out of the surface. Figures 5.10.4 (a,b) shows micro-crack growth. Figure 5.10.4b shows micro-crack growth and crack propagation.

Figure 5.10.5 shows FEGSEM backscattered micrograph of sample 461 extruded bar at load of 100N. Oxides are appearing dark grey on the micrograph. The micrograph shows fine grooves. The number of fine grooves in figure 5.10.5 is higher than figure 5.10.2b. This could be due to the fact that figure 5.10.5 experienced higher load (i.e. 100 N) than figure 5.10.2b (i.e.10N). It has been observed that as the load increases, the number of grooves and the width of track increases.

In general, the worn surface of the 100N shows smoother and finer grooves compare with the 10N. Grooves are due to abrasion either by entrapped particles or by asperities of the hard counterface.

At low load (i.e. 10N)

In a ball on flat contact, the maximum shear stress is below the surface. In sliding contact, the effect of friction is to move the maximum contact stress to the surface. The higher the load, the higher the contact stress and the greater the wear.

Since the maximum stress takes place at the surface or into it in sliding wear and the local stresses generated beneath the slider are lower than the fracture strength of the Si particles. Therefore, the unfractured Si particles act as load supporting elements

and scratch the counterpart surface. Consequently, this will prevent the Al matrix from getting involved directly in the wear. However, some particles which are chipped either from the silicon or other particle will wear the aluminium matrix because Al is softer component.

At high load (i.e. 100N)

As load increases (i.e. 100 N), the Si particles will fracture. Therefore, Si particles will be unable to support the load. As a result of this Al- matrix will be directly involved in the wear process (i.e. Al matrix will be in direct contact with the counterface). Therefore, large strain will be generated with the Al matrix adjacent to the contact surface. This will lead to the initiation of micro cracks and the propagation of the cracks (see figure 5.10.4). Moreover, the broken Si particles will act as third body abrasers, which are responsible for the initiation of the groove profile (laminar) on the worn surface. These longitudinal grooves indicate abrasive wear. Therefore, it is an abrasive mechanism.

Figure 5.10.6 shows the wear track profiles of samples 461,444 and 431 extruded bars. The wear track profiles width of each sample is respectively at 10 N is 750, 900 and 1100 μm and the depth is 17, 33 and 43 μm respectively. The wear track profiles show that sample number 461 has the least width and depth of all samples. The wear track profile of sample 461 shows shallower wear depth compared with 444 and 431. Examining the bottom of the wear profile shows that 431 has wider and smoother flat type which indicate less wear resistance compared with 444 and 461. In general the wear depths are similar but grooves wider for 431. All wear track profiles can be seen in appendix 7.

Examining the wear track profiles of extruded bar sample number 461 at 10 and 100N figure 5.10.6(a,d) shows that the width at the 100 N is greater on both top and bottom due to load effect.

Wear Debris

Figure 5.10.7(a, b) shows FEGSEM micrograph of samples 444-4042 FEF and 446-4051-FEF-2 respectively. The micrographs show the wear debris at 10 N. Figure 5.10.8(a, b) show FEGSEM micrograph of samples 444-4042 FEF and 446-4051-FEF-2. The micrograph shows the wear debris at 100 N.

The debris at 10 N is powder type for both samples. However, debris at 100 N is powder type for sample 444-4042 FEF and flake type for sample 446-4051-FEF-2. Therefore, wear debris is powder type as Si content increases and flake type as Si content decreases (at high load).

Comparing the wear debris of figure 5.10.7b and figure 5.10.8b it can be seen on these micrographs that the wear debris of figure 5.10.7b is powder type and on figure 5.10.8b is flake type. Therefore, as load increase, i.e 100 N, the wear debris is flake type and as the load decrease the wear debris is powder type (for the same material). Similar results have been reported by Bai and Biswas [91].

These findings can be explained as follows, Si acts as an abrasive material. At high Si contents some of the Si particles chip off and act as another abrasive material i.e. abrasive mechanism. Consequently, the wear debris is fine powder. On other hand, as load increases to 100 N plastic deformation take place which leads to flake debris (for the same material).

Counterface

Figure 5.10.9 shows FEGSEM micrograph of the counterface i.e. steel ball bearing. Figure 5.10.9a is taken at lower magnification and figure 5.10.9b is taken at higher magnification of the same spot. Both micrographs show small voids or pits on the surface of the ball bearing. These voids are manufacturing defects. During the wear process these voids are filled with the softer elements of the work piece. Figure 5.10.10 shows optical micrograph and EDX spectra of unused ball bearing. The EDX shows high amount of iron.

Figure 5.10.11 is a FEGSEM micrograph of the used ball bearing and EDX spectra of figure 5.10.11b at load of 10 N for sample 461. Figure 5.10.11a is taken at lower magnification while figure 5.10.11b at higher magnification. Figure 5.10.11a shows transferred material from sample 461 extruded bar to the ball bearing surface. To analyse particles transferred on to the ball bearing i.e. Al and Si particles, three different spots were taken on figure 5.10.11b. Spots numbers one and two are Al, Si and Fe particles, spot number two has a higher amount of Al, Si and a lower amount of Fe than spot number one. Therefore, these two spots confirm the transfer of particles from the work piece to the counterface. Spot number three shows high amount of Fe and very small amount of Al and Si. Spot number three is the ball bearing surface with small amount of particles transferred to the counterface. The ball bearing surface appears light on the micrograph while particles transferred on to the ball bearing appear as dark black spots. Similar observations have been reported by Elmadagli and Alpas [130].

Figure 5.10.12 shows FEGSEM micrographs of the ball bearing at 100 N for sample 461 and the EDX spectra of figure 5.10.12b. Figure 5.10.12a shows the ball bearing

surface at lower magnification while figure 5.10.12b shows the same spot but at higher magnification. Figure 5.10.12b shows three different points on the micrograph. Point 1 which is pale on the micrograph has high content of iron with little amount of aluminium and silicon. Points 2 and 3 which are relatively dark on the micrograph have relatively high content of aluminium and silicon. However, point 3 has relatively higher amount of Si than point 2. These points were analysed in figure 5.10.11c. Therefore, point 1 represents the ball bearing surface with very little amount of material transferred from the sample while points 2 and 3 represent the material transferred to the ball bearing from wear process.

Comparing the amount of material transferred at 10N (figure 5.10.11a) with the 100N (figure 5.10.12a), figure 5.10.12a shows thicker and more dark spots than figure 5.10.11a which means the amount of material transferred at 100N is more than 10N due to high load effect. Also, it is probably due to plastic deformation involvement at higher load. Increasing the load will increase area of contact which will increase the frictional forces between the two surfaces. Therefore, increasing the frictional force and surface area in contact, will increase the wear.

Correlation of Wear, Alloying Element Content and Hardness

Figure 5.10.13 shows the wear volume versus the hardness of as-received extruded bar samples at 10 N. The figure indicates that sample numbers 431 and 446-3 have the highest wear volume and sample number 461 has the lowest wear volume. Moreover, it shows that sample 431 and 446-3 have the lowest hardness number and sample 461 has the highest hardness. Therefore, the line trend shows that as hardness increases the wear resistance increases or wear volume decreases. It should be

mentioned that as hardness increases (i.e. higher σ_y) the abrasion resistance increases. However, in adhesion resistance, as hardness increases this does not give an increase of adhesion resistance due to the fact that adhesion mostly is related to tendency to stick. In fact, in adhesion wear, hardness affects junction growth. As hardness increases, junction growth decreases which means less adhesion. Figure 5.10.13 therefore adds to the evidence that abrasion is the wear mechanism.

The figure also shows that RSP sample number 461 has higher hardness and higher wear resistance than piston A390. Moreover, RSP samples 446-2 and 444 have lower hardness than piston A390. The wear resistance of A390, is similar to that for 446-2, 446-3 and 444.

Figure 5.10.14 represents the wear volume versus Si contents of as-received extruded bars at 10 N. There is little correlation between wear volume and Si content except that sample 431 with a relatively high Si content also shows the lowest wear resistance. Clark and Saker [87] and Wang et al. [94] have reported that as silicon content increases up to 20-21 weight percent the wear resistance decreases. This can be explained as follows. Si particles at certain amount of weight percent, i.e. 20-21, will be able to withstand the load, consequently providing high wear resistance. However, at higher amounts of Si, some Si particles will chip out of the matrix and act as another abrasive material which will reduce the wear resistance.

Figure 5.10.15 shows the wear volume versus the Cu content. The general trend of the graph shows that as the Cu content increases the wear volume decreases. This is likely to be due to formation of hard precipitate (i.e.intermetallic) during hot processing i.e. extrusion. The precipitates are present at too small a volume fraction to show up in XRD and are too fine to be visible in SEM.

Figure 5.10.16 shows the wear volume versus the Fe content. The trends of the curve indicate that as the Fe contents increases, the wear volume decreases probably due to the formation of intermetallic particles.

Figure 5.10.17 shows the wear volume versus the hardness of degassed and HIPped billets of 431 and 461 at a load of 10 N. It indicates that as hardness increases, the wear volume decreases. Wear resistance is associated with hardness; harder materials will have higher wear resistance in abrasion wear. The figure also shows that billet 461 has higher wear resistance than billet 431 at the same condition (i.e. degassing temperature and time); this could be due to the fact that billet 461 has finer microstructure than billet 431 (see figures 5.5.9b and 5.5.12b).

At 100 N

Figure 5.10.18 shows the wear volume against the hardness of as received extruded bar samples at 100 N. The trend of the curve shows that wear resistance increases as hardness increases. RSP 461, 444 and 431 show higher wear resistance than piston A390.

Figure 5.10.19 shows the wear volume versus Cu content. If the 464-2 and 446-3 results are disregarded, the trend of the curve shows that as Cu content increases, the wear resistance increases.

Figure 5.10.20 shows the wear volume versus Fe. If the 464-2 and 446-3 results are disregarded, the trend of the curve shows that as the alloying element contents increase, the wear resistance increases.

Figure 5.10.21 represents the wear volume of billet numbers 431 and 461 degassed and HIPped at different degassing time and HIPped versus the hardness. The graph shows that material number 461 degassed at 400°C for 12 hrs has the highest wear resistance of the 461 group. Also, sample number 431 degassed at 400°C for 12 hrs has the highest wear resistance of the 431 group. The graph also shows the effect of degassing temperature on wear volume for the two groups i.e. 461 and 431. For instance, degassing at 500°C for 12 hr shows the least wear resistance of the two groups due to heat effect which will coarsen the particles. However, the degassing at 400°C for 12hr shows the least wear volume of the two groups. This could mean that the optimum degassing temperature is 400°C for 12hr.

Figure 5.10.22 shows the wear volume versus the hardness of all the extruded bar samples. The graph shows a comparison of the wear volume of extruded bars at different wear loads i.e. 10 and 100 N. The graph indicates that as hardness increases, the wear resistance increases. The graph also shows that samples at 100N have higher wear volume than samples at 10N this is mainly due to plastic deformation involvement at higher load. The graph also shows that RSP 461 has higher wear resistance than A390 at 10 and 100 N. It is clear that RSP 446-3, 446-2 and 444 have lower hardness than piston A390 and have almost the same wear resistance as A390. The high wear resistance of RSP sample is mainly due to the fact that RSP sample has finer silicon particles and very well distributed in the aluminium matrix. This also shows good evidence of the advantages of rapid solidification process over casting process (i.e. piston A390).

Image analysis

Figure 5.10.23 shows wear volume versus area fraction (measured) for extruded bar samples at 10 N. The figure indicates that as area fraction of Si increases the wear volume increases. This is due to the fact that silicon will behave as another abrasive material beyond 21w.t %. However, analysing samples having 20-21 w.t. % silicon (461, 446-2 and 446-3) it is clear that silicon plays an important role in increasing the wear resistance on at 10 N load. This means that 461, 446-2 and 446-3 show higher wear resistance as area fraction of Si increases.

Figures 5.10.24 and 5.10.25 show the wear volume versus the area fraction of silicon of degassed and HIPped billet at two different loads. Figure 5.10.24 shows that sample number 461 as silicon area fraction increases the wear resistance increases because it has the optimum silicon content 21w.t %. Moreover, as degassing temperature increases the wear resistance decreases due to coarsening behaviour of Si particles. However, sample 431 shows that as area of fraction increases wear resistance decreases because silicon beyond 29 will act as another abrasive material. Also, as degassing temperatures increases wear resistance decreases due to heat effect. It was shown previously on figure 5.7.1 section 5.7 that sample 431 (degassed and HIPped) has bigger silicon particle size than 461 (degassed and HIPped).

The trend line of figure 5.10.25 shows that as fraction area of silicon increases, wear resistance decreases. This could be due to plastic deformation involvement at high load i.e. 100 N. Also, both materials show decreased wear resistance especially at high degassing temperature i.e. 500°C due to heat influence which coarsens the particles.

Comparing the Image-pro plus images (see appendix 6) for particle shape, maximum and minimum radius of extruded bar sample number 461 (which has the highest wear resistance), and sample numbers 431 and 446-2 (which have almost the least wear resistance) general conclusions can be drawn as follow:

- i. Silicon particles of sample 461 tend to be more rounded than 431 and 446-2.
- ii. Silicon is more clustered (i.e. in group) in 431 and 446-2 than 461.

Comparisons of RSP wear rates with other sources

When comparing the wear rate of other references (table 2.4 section 2.11.3) with RSP samples (table 5.11.2), RSP samples showed lower wear rate than references [89, 91] at 10 and 100 N. However, references [87, 94] show slightly better wear resistance than RSP. However, it should be mentioned that RSP samples experienced very high contact pressure compared with other references due to the fact that using small ball radius (2.5mm) will generate higher contact pressure than pin on disc. Contact pressure increases as the contact area decreases. It should be mentioned that most of the references contact area is almost 12000 times higher than here. Also, RSP materials shows fine, rounded and will distributed silicon particles on the aluminium matrix. In general, wear rate of RSP materials is lower than other reference.

Summary

This chapter is the discussion of the results for flake and ribbon microstructure, cold compaction and degassing at the University of Leicester, mapping and line scans of as-received and degassed ribbons, degassing and HIPing at Bodycote, bar microstructure as-received, Si particle measurements, X-Ray diffraction, hardness testing, wear and comparison of wear rate with other sources.

There is a clear evidence that the microstructure of RSP samples is composed of three zones; air side, wheel side and featureless. The microstructure of the air side is coarser than the wheel side based on large Si particles measurement.

Flakes tend to split lengthways during the production stage and hence forming two regions (i.e. fine and coarse) during the HIPping stage. Degassed at 400°C for 12hrs is shown to be the optimum degassing temperature since it causes lower coarsening behaviour to the Si particles compared with 500°C. The intermetallic particles are present in sample which contains around 21% Si. Based on hardness and wear tests 461 has the highest hardness and wear resistance of all the samples mainly due to the fact that it has the optimum alloying elements (i.e. 21Si 3.9Cu 2.4Fe). RSP 461,444 and 431 show higher wear resistance than piston A390.

CHAPTER 7

SUMMARY AND CONCLUSIONS

Summary and Conclusion

Flake, ribbon and bar microstructures of as-received samples have been obtained using optical microscopy and FEGSEM. The chill zone has the highest cooling and solidification rate; it is also known as the drum side or the wheel side. The other side is called the air side. The microstructures of the air sides are coarser than those of the wheel side. The wheel sides microstructures are very fine. The edge of the sample at the wheel side formed a straight line but it is irregular at the air side.

It has been observed that silicon particles (appearing grey on optical micrographs) and intermetallic particles (appearing black on optical micrographs and white on FEGSEM) are present in the aluminium matrix. All RSP extruded bar samples showed a reasonably homogeneous overall distribution of the silicon particles.

Elemental mapping of ribbons and as-HIPped billet showed high concentration of Al and Si. Other elements have low concentration due to their low content (less than 3.9 wt.%).

Degassing has been done at maximum 500°C and minimum temperatures 300°C for 3hrs and 12hrs to study the effect on samples microstructures. The average diameter of the large “typical” Si particles is 3.2 μm for degassing at 500°C for 3hrs and is 2.5 μm for degassing at 300°C for 3hrs. The microstructure after degassing at 500°C for 3 hrs showed the Si particles having very clear dimensions compared with as-received sample (see figures 5.3.1a, 5.3.2a).

Most of the Bodycote degassed and HIPped billet samples show an inhomogeneous distribution of silicon particles with some regions having very fine silicon particles while others are coarse. These regions look like “waves” at low magnification. This is mainly due to the fact that during the HIPping stage air side flakes are high in one region and wheel side flakes in another region. Porosity was seen in some of the samples which could be due to gas entrapped inside the canister i.e. hydrogen or argon.

It should be mentioned that RSP could consider the following steps to overcome the above mentioned problems:

1. If possible reduce ribbon thickness to minimize the coarser silicon particles which originate from the air side.
2. If possible use two chill drums on top of each other (twin rolls) with the ribbons are produced in between them. This will help reducing the coarser particles originating from the air side.

Wear

Wear debris type is affected by two factors, the alloying composition and the load. It is powder type as silicon content increases and flake type as silicon content decreases (at 100N). Also, it is powder type as load decreases i.e. 10N and flake type as load increases i.e. 100N (for the same material).

It is found that material is transferred from the work piece i.e. RSP samples to the counterface i.e. the ball bearing. The amount of material transferred to the ball bearing is higher at high load than at low load 10 N.

As received sample number RSP 461 has the highest hardness of all extruded bars. It is also higher than the piston A390. Sample number 461 degassed and HIPped at 400°C for 3hrs has the highest hardness of all degassed and HIPped samples and indeed of all the test samples (including those subjected to extrusion or heat treatment).

Harder materials resist wear more than softer materials. Therefore, wear resistance increases as hardness increases. Hardness and wear is affected by alloying elements content.

Silicon plays an important role in increasing the wear resistance mainly because silicon is a very hard material. Silicon increases the wear resistance up to a certain amount i.e. 20-21(wt. %). However, at high content of silicon i.e. 30 (wt.%), it thought to have a negative effect on the wear resistance process because some of the silicon particles may chipped out and act as another abrasive material.

Cu and Fe have a great influence in increasing the wear resistance of RSP samples. It has been mentioned previously that as Cu and Fe content increases the wear resistance increases. The reason for this is that Cu will enhance the formation of hard precipitates when exposed to heat (these precipitates are below the resolution of the FEGSEM) and Fe will react with other elements to form intermetallic particles which are visible in the FEGSEM but are too small to analyse.

Extruded bar 461 has the highest hardness and hence the highest wear resistance of all samples due to the fact that it combines the hardness of an intermediate level of Si (21%) with relatively high content of intermetallic i.e. Mg_2Si . Degassing condition has great influence on material wear resistance. For instance, a high degassing

temperature (i.e. 500 °C) for a long period of time (i.e. 12 hrs) leads to a reduced wear resistance of the material due to heat effect which will coarsen the particles.

The wear results show that the production of Al-high Si alloys by RSP has the potential to develop good wear resistant alloys since the fine microstructure that is developed has high hardness. The wear mechanism is a mixture of adhesive wear leading to transfer to the ball bearing of ductile matrix and abrasive wear from Si particle entrapment leading to three-body wear. Therefore, there is not additional benefit from increasing the Si content due to the fact that Si tends to chip out of the matrix and give third body wear (i.e. more severe wear).

To optimize the wear resistance in these materials the following should be considered:

- I. Optimize the Si content at around 21%.
- II. Increasing the hardness by having intermetallics in the matrix which act to minimize junction growth as they decrease the flow of the ductile matrix and decrease the tendency of the material to adhere to the counterface.

CHAPTER 8

FUTURE WORK

This work could be extended by examining various degassing temperatures such as 200°C, 300°C, 400°C, and 500°C for the ribbon and compared the same temperature for the degassed and HIPped billet using sample containing only aluminium and different silicon contents.

It could also be extended to conduct mathematical modelling of the coarsening behaviour of the silicon during degassing and HIPping stages.

Fatigue strength at high temperatures relationship with microstructure. Some considerable effort was put into attempting to prepare TEM samples. This was not successful. However, TEM is clearly important for identifying the intermetallic phases and therefore a next step would be developing a method for preparing TEM specimens so that the phases could be identified.

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Appendix 1: Literature wear rates

References	Material	Wear rate ($\text{cm}^3 \cdot \text{cm}^{-1} \cdot 10^8$) at each pressure (KPa)				Wear rate ($\text{g cm}^{-1} \cdot 10^{-9}$) at each load (kg)			Wear rate (10^{-6} g/m) at each load (N)				Wear rate ($10^{-2} \text{ cm}^3/\text{cm}$) at each load (N)		
		40 KPa	80 KPa	120 KPa	158 KPa	1 kg	1.5 kg	2 kg	8.9 N	17.8 N	26.7 N	35.6 N	10 N	40 N	60 N
K.Mohamed Wear, 119 (1987)119- 130.	Al-7%Si	0.9	1	3.5	5										
	Al-13%Si	0.5	2.2	1.5	2										
	Al-22%Si	0.9	1.9	3	4										
J.Clark and A.Saker. Wear, 54(1979) 7- 6	15%Si					14	20	23							
	16%Si					20	35	35							
	21%Si					35	45	60							
F.Weng Material and design 25 (2004)163- 166.	Al-12%Si								5.8	6	10	18			
	Al-20%Si								4	6	7	8			
	Al-25%Si								3	6	9	11			
B.Bai and S. Biswas ASLE Tra. 29(1986)11 6-120	12%Si												0.4	1	1
	15%Si												0.4	0.9	1.3

Table A.1.1 The original wear data taken from different sources.

The original wear data is showing in table A.1.1. Units of data collected will be unified for ease of comparison.

For reference (k. Mohamed):

Pressure

Pressures given are 40, 80, 120 and 158KPa.

Wear rate

Wear rate is given in ($\text{cm}^3 \cdot \text{cm}^{-1} \cdot 10^{-8}$) and should be converted to ($\text{m}^3 \cdot \text{m}^{-1}$).

therefore,

$$1\text{cm}^3 = 0.000001\text{m}^3$$

$$0.9 \times 10^{-8} \text{ cm}^3 \cdot \text{cm}^{-1} \times 10^{-6} = 0.9 \times 10^{-14} \text{ m}^3 \text{ cm}^{-1} = 0.9 \times 10^{-14} \times 10^2 \text{ m}^3 \text{ m}^{-1} = 0.9 \times 10^{-12} \text{ m}^3 \text{ m}^{-1}$$

All wear rates are calculated in the same way for ease of comparison.

Appendix 1: Literature wear rates

For reference (J.clark and A.saker):

Pressure

The given load is in (Kg).

Contact pressure can be calculated as follow:

$$\text{Pin diameter} = 6.2\text{mm} \quad r = 3.1\text{mm} = 3.1 \times 10^{-3} \text{ m}$$

$$\text{Contact area} = A = \pi r^2 = \pi (3.1 \times 10^{-3})^2 = 3.217 \times 10^{-5} \text{ m}^2$$

$$\text{Pressure} = F/A = \quad \text{given pressure as } 1\text{kg}_f \quad 1\text{kg}_f = 9.80665 \text{ N}$$

$$\text{For load 1 kg, Pressure} = 1 \times 9.80665 / 3.217 \times 10^{-5} = 304 \text{ Kpa}$$

$$\text{For load 2 kg, Pressure} = 2 \times 9.80665 / 3.217 \times 10^{-5} = 609 \text{ Kpa}$$

Wear rate

The wear rate is given in ($\text{g cm}^{-1} 10^{-9}$)

For alloy composition 15%Si and 85%Al

$$\text{Al density} = 2.7\text{g/cm}^3 \quad \text{Si density} = 2.33 \text{ g/cm}^3$$

$$85\% \text{ Al} = 85\% \text{ of aluminium density } 2.7\text{g/cm}^3 = 0.85 \times 2.7 = 2.295 \text{ g/cm}^3$$

$$\text{and } 15\% \text{ Si of Si density } 2.33 = 0.15 \times 2.33 = 0.3495\text{g/cm}^3$$

$$\text{The total density of the alloy} = 2.295 \text{ g/cm}^3 + 0.3495\text{g/cm}^3 = 2.64 \text{ g/cm}^3$$

$$\text{volume} = \text{mass} / \text{density} = 14 \times 10^{-9} / 2.64 = 5.3 \times 10^{-9} \text{ cm}^3$$

$$\text{wear rate at (1kg) or at 304 KPa} = 5.3 \times 10^{-11} \text{ cm}^3 \text{ cm}^{-1} = 5.3 \times 10^{-17} \text{ m}^3 \text{ cm}^{-1} =$$

$$= 5.3 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$$

$$\text{volume} = \text{mass} / \text{density} = 20 \times 10^{-9} / 2.64 = 7.575 \times 10^{-9} \text{ cm}^3$$

$$\text{wear rate at (1.5kg) or at 457 KPa} = 7.6 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$$

$$\text{volume} = 23 \times 10^{-9} / 2.64 = 8.7 \times 10^{-9} \text{ cm}^3$$

Appendix 1: Literature wear rates

wear rate at (2kg) or at 609 KPa = $8.7 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$

For alloy 16%Si and 84%Al

84% Al=84% of aluminium density $2.7 \text{ g/cm}^3 = 0.84 \times 2.7 = 2.268 \text{ g/cm}^3$

and 16% Si of Si density $2.33 \text{ g/cm}^3 = 0.16 \times 2.33 = 0.368 \text{ g/cm}^3$

the total density of the alloy = $2.268 \text{ g/cm}^3 + 0.368 \text{ g/cm}^3 = 2.64 \text{ g/cm}^3$

volume = $20 \times 10^{-9} / 2.64 = 7.57 \times 10^{-9} \text{ cm}^3$

wear rate at (1kg) or at 304 KPa = $7.57 \times 10^{-11} \text{ cm}^3 \text{ cm}^{-1} = 7.57 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$

volume = $35 \times 10^{-9} / 2.64 = 13.25 \times 10^{-9} \text{ cm}^3$

wear rate at (1.5kg) or at 457 KPa = $13.25 \times 10^{-11} \text{ cm}^3 \text{ cm}^{-1} = 13.25 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$

For alloy 21%Si and 79%Al

79% Al=79% of aluminium density $2.7 \text{ g/cm}^3 = 0.79 \times 2.7 = 2.133 \text{ g/cm}^3$

and 21% Si of Si density $2.33 \text{ g/cm}^3 = 0.21 \times 2.33 = 0.483 \text{ g/cm}^3$

the total density of the alloy = $2.133 + 0.483 = 2.616 \text{ g/cm}^3$

volume = $35 \times 10^{-9} / 2.61 = 13.4 \times 10^{-9} \text{ cm}^3$

wear rate at (1kg) or at 304 KPa = $13.4 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$

volume = $45 \times 10^{-9} / 2.61 = 17.24 \times 10^{-9} \text{ cm}^3$

wear rate at (1.5kg) or at 457 KPa = $17.24 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$

volume = $60 \times 10^{-9} / 2.61 = 22.9 \times 10^{-9} \text{ cm}^3$

wear rate at (2kg) or at 609 KPa = $22.9 \times 10^{-15} \text{ m}^3 \text{ m}^{-1}$

Appendix 1: Literature wear rates

For reference (F. Wang):

Pressure

The given loads are 8.9, 17.8, 26.7 and 35.6N

Pin diameter = 4.8mm $r = 2.4\text{mm} = 2.4 \times 10^{-3} \text{ m}$

Contact area = $A = \pi r^2 = \pi(2.4 \times 10^{-3})^2 = 18.0864 \times 10^{-6} \text{ m}^2$

pressure = $F/A = 8.9/18.0864 \times 10^{-6} = 492 \text{ KPa}$

pressure = $17.8/18.0864 \times 10^{-6} = 984 \text{ KPa}$

pressure = $26.7/18.0864 \times 10^{-6} = 1476 \text{ KPa}$

pressure = $35.6/18.0864 \times 10^{-6} = 1968 \text{ KPa}$

Wear rate

For alloy 12%Si and 88%Al

88% Al=88% of aluminium density $2.7\text{g/cm}^3 = 0.88 \times 2.7 = 2.376 \text{ g/cm}^3$

and 12% Si of Si density $2.33 \text{ g/cm}^3 = 0.12 \times 2.33 = 0.276 \text{ g/cm}^3$

the total density of the alloy = $2.376 + 0.276 = 2.652 \text{ g/cm}^3$

wear rate = $5.8 \times 10^{-6} / 2.652 = 2.2 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

Wear rate = $6 \times 10^{-6} / 2.652 = 2.3 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $10 \times 10^{-6} / 2.652 = 3.8 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $18 \times 10^{-6} / 2.652 = 6.88 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

For alloy 20%Si and 80%Al

80% Al=80% of aluminium density $2.7\text{g/cm}^3 = 0.8 \times 2.7 = 2.16 \text{ g/cm}^3$

and 20% Si of Si density $2.33 \text{ g/cm}^3 = 0.2 \times 2.33 = 0.46 \text{ g/cm}^3$

Appendix 1: Literature wear rates

the total density of the alloy = $2.16 + 0.46 = 2.62 \text{ g/cm}^3$

wear rate = $4 \times 10^{-6} / 2.62 = 1.5 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $6 \times 10^{-6} / 2.62 = 2.3 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $7 \times 10^{-6} / 2.62 = 2.6 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $8 \times 10^{-6} / 2.62 = 3 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

For alloy 25%Si and 75%Al

75% Al = 80% of aluminium density $2.7 \text{ g/cm}^3 = 0.75 \times 2.7 = 2.025 \text{ g/cm}^3$

and 25% Si of Si density $2.33 \text{ g/cm}^3 = 0.25 \times 2.33 = 0.575 \text{ g/cm}^3$

the total density of the alloy = $2.025 + 0.575 = 2.6 \text{ g/cm}^3$

Wear rate = $3 \times 10^{-6} / 2.62 = 1.1 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $6 \times 10^{-6} / 2.62 = 2.3 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $9 \times 10^{-6} / 2.62 = 3.4 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

wear rate = $11 \times 10^{-6} / 2.62 = 4.2 \times 10^{-14} \text{ m}^3 \text{ m}^{-1}$

For reference (B.Bai):

Pressure

The given loads are 10, 40, and 60N

Pin diameter = 5mm $r = 2.5 \text{ mm} = 2.5 \times 10^{-3} \text{ m}$

Contact area = $A = \pi r^2 = \pi (2.5 \times 10^{-3})^2 = 19.625 \times 10^{-6} \text{ m}^2$

Appendix 1: Literature wear rates

$$\text{Pressure} = F/A = 10/19.625 \times 10^{-6} = 509 \text{ KPa}$$

$$\text{Pressure} = 40/19.625 \times 10^{-6} = 2038 \text{ KPa}$$

$$\text{Pressure} = 60/19.625 \times 10^{-6} = 3057 \text{ KPa}$$

Wear rate

Wear rate is given in ($\text{cm}^3 \cdot \text{cm}^{-1} \times 10^{-7}$) and should be converted to ($\text{m}^3 \cdot \text{m}^{-1}$).

therefore,

$$\begin{aligned} 0.4 \times 10^{-7} \text{ cm}^3 \cdot \text{cm}^{-1} \times 10^{-6} &= 0.4 \times 10^{-13} \text{ m}^3 \cdot \text{cm}^{-1} = 0.4 \times 10^{-13} \times 10^2 \text{ m}^3 \text{m}^{-1} \\ &= 0.4 \times 10^{-11} \text{ m}^3 \text{m}^{-1} \end{aligned}$$

in the same way all wear rates calculated as follow:

$$0.9 \times 10^{-7} \text{ cm}^3 \cdot \text{cm}^{-1} \times 10^{-6} = 0.9 \times 10^{-11} \text{ m}^3 \cdot \text{m}^{-1}$$

$$1.3 \times 10^{-7} \text{ cm}^3 \cdot \text{cm}^{-1} \times 10^{-6} = 1.3 \times 10^{-11} \text{ m}^3 \cdot \text{m}^{-1}$$

Appendix 1: Literature wear rates

Appendix 2

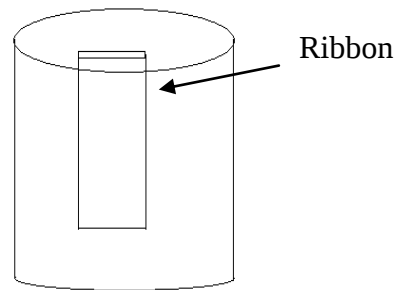


Figure A.2.1 Ribbon mounting



Figure A.2.2 Gatan 623 disc grinder.

Appendix 2

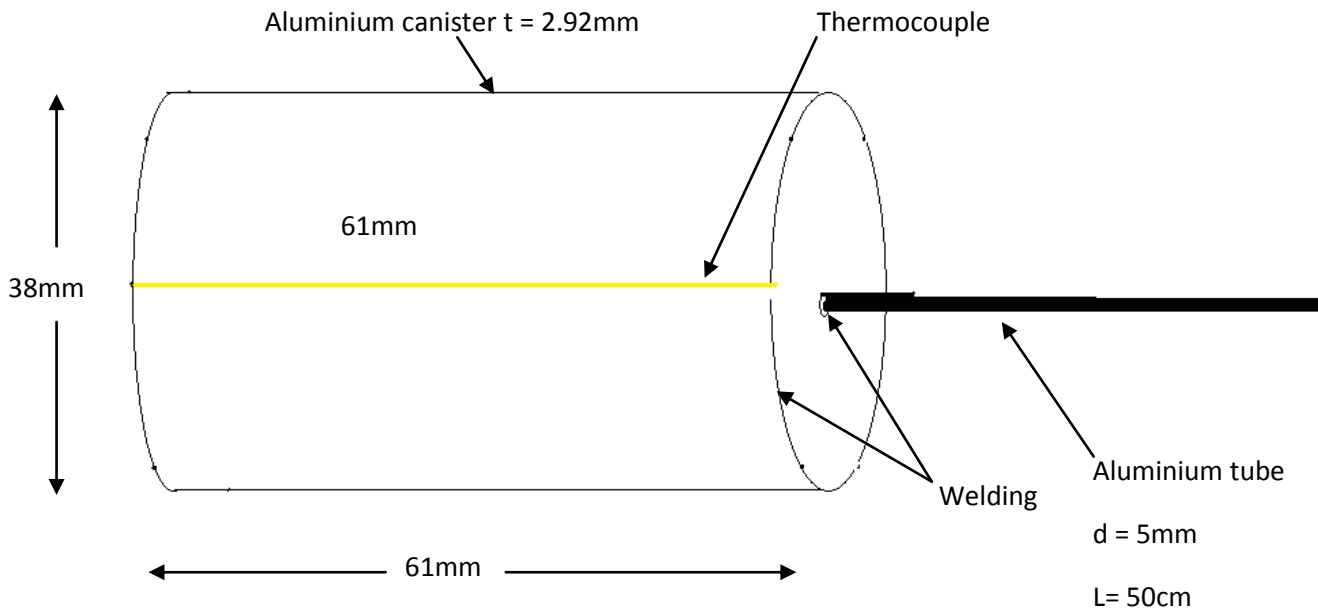


Figure A.2.3 Degassing set up

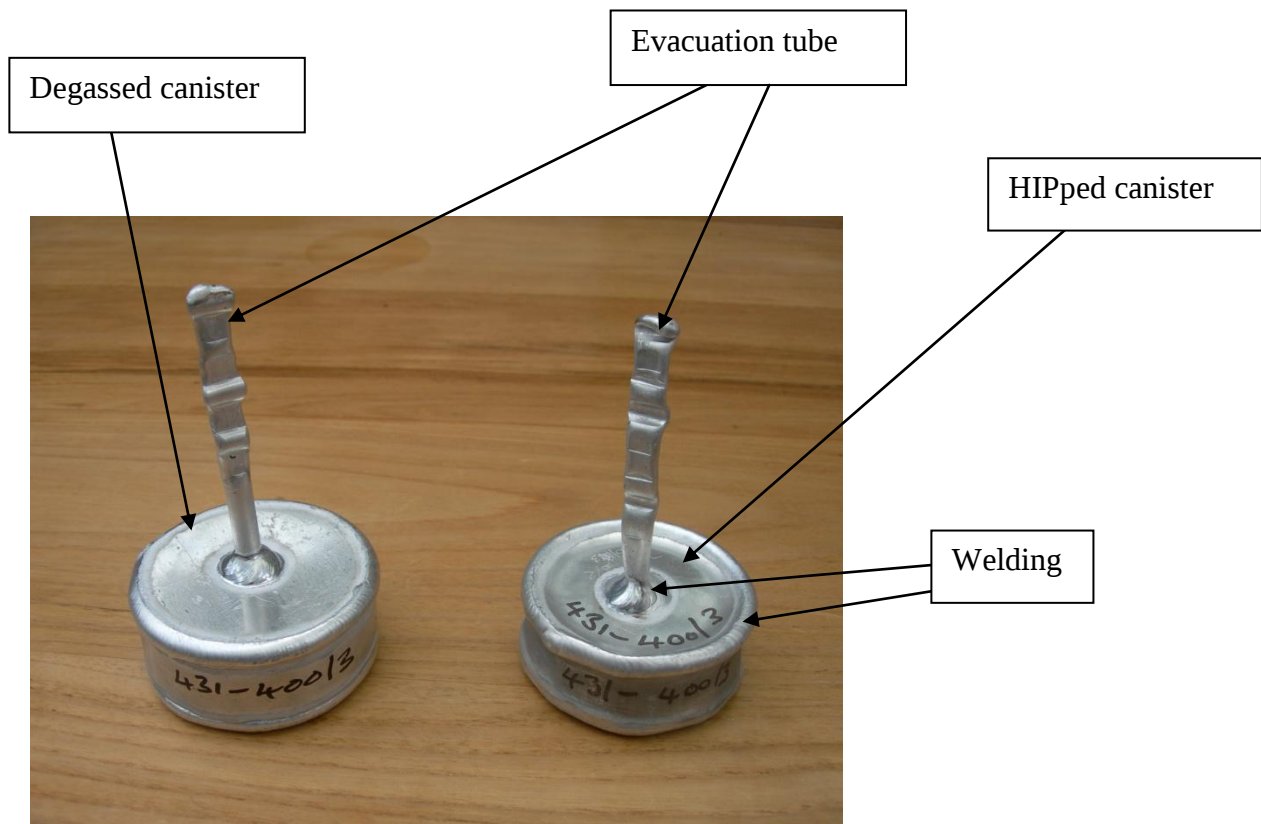


Figure A.2.4 Bodycote canisters.

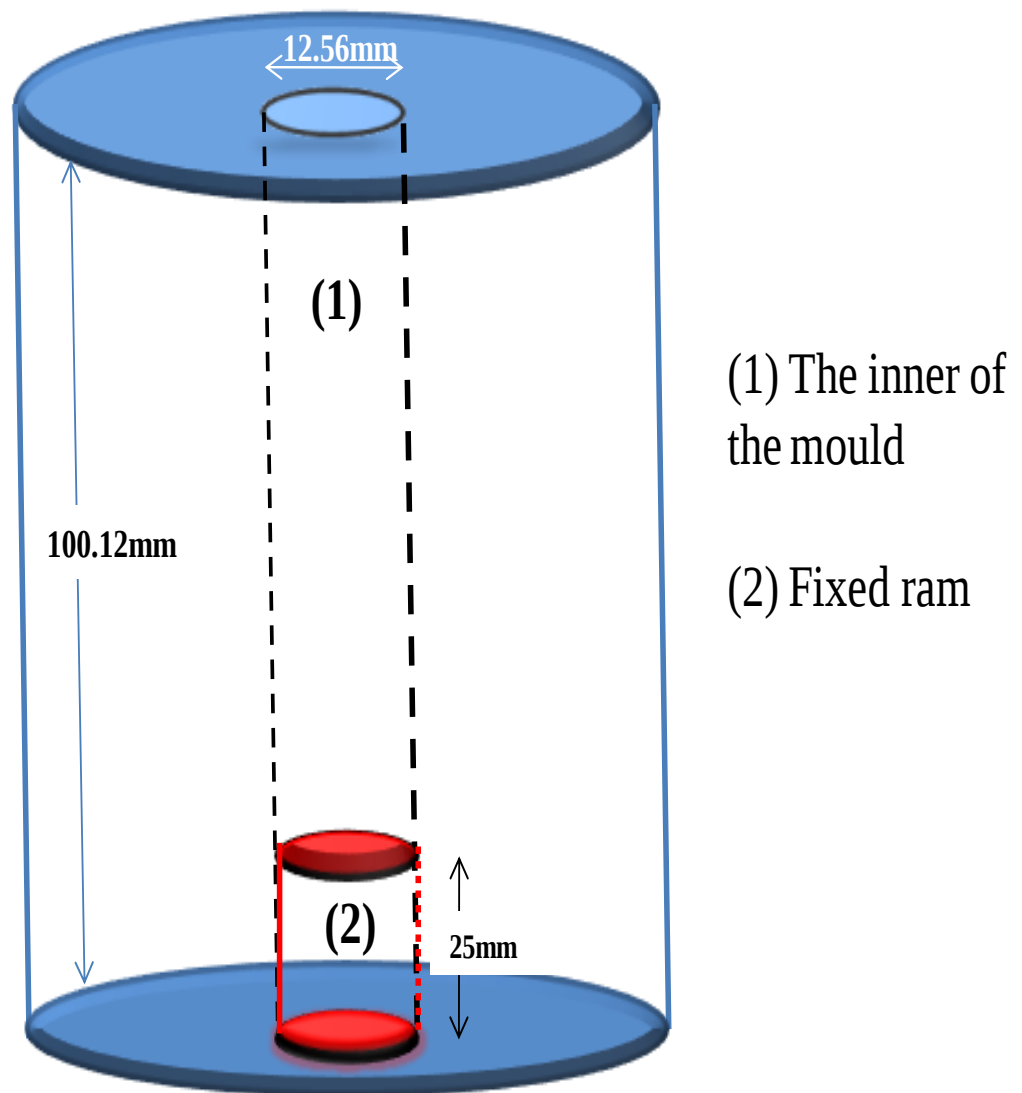


Figure A.3.1 Cold compaction mould.

Appendix 4

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Mg	KA1	1.254	0.0144	1.62	1.87	0.0	3826.60
Al	KA1	1.487	0.6068	69.79	72.25	0.0	3826.60
Si	KA1	1.740	0.0935	23.81	23.67	0.0	3826.60
Fe	KA1	6.403	0.0158	1.81	0.90	0.0	121.13
Cu	KA1	8.046	0.0184	2.19	0.96	0.0	121.13
Zr	LA1	2.042	0.0005	0.12	0.04	0.0	3826.60
Ni	KA1	7.477	0.0059	0.66	0.32	0.0	121.13
Total			0.7554	100.00	100.00	0.0	2903.02

Table A.4.1 EDX quantitative analysis for zone 1 in figure 5.2.8.

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Mg	KA1	1.254	0.0174	2.00	2.31	0.0	1413.41
Al	KA1	1.487	0.6022	69.94	72.61	0.0	1413.41
Si	KA1	1.740	0.0966	22.60	22.53	0.0	1413.41
Fe	KA1	6.403	0.0176	2.02	1.01	0.0	38.23
Cu	KA1	8.046	0.0218	2.60	1.15	0.0	38.23
Zr	LA1	2.042	0.0000	0.00	0.00	0.0	
Ni	KA1	7.477	0.0074	0.84	0.40	0.0	38.23
Total			0.7630	100.00	100.00	0.0	1043.81

Table A.4.2 EDX quantitative analysis for zone 2 in figure 5.2.8.

Appendix 4

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.7058	75.46	76.64	35514.1	43.18
Si	KA1	1.740	0.0852	21.75	21.21	9830.3	43.18
Mg	KA1	1.254	0.0133	1.33	1.49	692.0	43.18
Fe	KA1	6.403	0.0035	0.41	0.20	92.7	0.93
Cu	KA1	8.046	0.0087	1.03	0.45	206.7	0.93
Zr	LA1	2.042	0.0000	0.00	0.00	0.0	
Ni	KA1	7.477	0.0001	0.01	0.01	3.0	0.93
Total			0.8166	100.00	100.00	46338.8	38.08

Table A.4.3 EDX quantitative analysis for spot 1 in figure 5.5.13.

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.8412	90.44	91.51	7388.3	60.05
Si	KA1	1.740	0.0229	6.04	5.86	473.5	60.05
Mg	KA1	1.254	0.0157	1.58	1.77	142.8	60.05
Fe	KA1	6.403	0.0022	0.25	0.12	10.0	0.96
Cu	KA1	8.046	0.0135	1.61	0.69	55.8	0.96
Zr	LA1	2.042	0.0000	0.00	0.00	0.0	
Ni	KA1	7.477	0.0007	0.08	0.04	3.0	0.96
Total			0.8961	100.00	100.00	8073.4	49.34

Table A.4.4 EDX quantitative analysis for spot 2 in figure 5.5.13.

Appendix 4

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.2979	32.03	33.07	1436.6	36.17
Si	KA1	1.740	0.3829	65.88	65.32	2837.9	36.17
Mg	KA1	1.254	0.0086	0.93	1.06	46.2	36.17
Fe	KA1	6.403	0.0053	0.63	0.31	13.5	1.02
Cu	KA1	8.046	0.0032	0.39	0.17	7.4	1.02
Zr	LA1	2.042	0.0000	0.00	0.00	0.0	
Ni	KA1	7.477	0.0013	0.15	0.07	3.0	1.02
Total			0.6991	100.00	100.00	4344.6	32.27

Table A.4.5 EDX quantitative analysis for spot 3 in figure 5.5.13.

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.8861	93.49	94.48	2951.9	65.85
Si	KA1	1.740	0.0145	3.91	3.79	118.4	65.85
Mg	KA1	1.254	0.0087	0.86	0.96	30.1	65.85
Fe	KA1	6.403	0.0015	0.18	0.09	2.7	0.98
Cu	KA1	8.046	0.0115	1.37	0.59	18.3	0.98
Zr	LA1	2.042	0.0000	0.00	0.00	0.0	
Ni	KA1	7.477	0.0018	0.21	0.10	3.0	0.98
Total			0.9242	100.00	100.00	3124.4	55.90

Table A.4.6 EDX quantitative analysis for spot 4 in figure 5.5.13.

Appendix 4

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.2827	30.28	31.29	1347.7	30.66
Si	KA1	1.740	0.4052	67.87	67.35	2901.2	30.66
Mg	KA1	1.254	0.0069	0.75	0.86	37.1	30.66
Fe	KA1	6.403	0.0022	0.26	0.13	5.6	0.95
Cu	KA1	8.046	0.0057	0.69	0.30	13.0	0.95
Zr	LA1	2.042	0.0000	0.01	0.00	0.1	30.66
Ni	KA1	7.477	0.0013	0.15	0.07	3.0	0.95
Total			0.7041	100.00	100.00	4307.6	27.04

Table A.4.7 EDX quantitative analysis for spot 5 in figure 5.5.13.

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.7409	82.19	84.46	409.4	55.13
Si	KA1	1.740	0.0504	12.55	12.39	60.1	55.13
Mg	KA1	1.254	0.0099	1.07	1.22	5.9	55.13
Fe	KA1	6.403	0.0085	0.97	0.48	2.3	0.94
Cu	KA1	8.046	0.0161	1.91	0.83	4.0	0.94
Zr	LA1	2.042	0.0000	0.00	0.00	0.0	
Ni	KA1	7.477	0.0116	1.31	0.62	3.0	0.94
Total			0.8373	100.00	100.00	484.8	44.55

Table A.4.8 EDX quantitative analysis for spot 6 in figure 5.5.13.

Appendix 4

Element	Line	keV	KRatio	Wt%	At%	At Prop	ChiSquared
Al	KA1	1.487	0.2460	26.78	28.00	221.6	39.39
Si	KA1	1.740	0.4398	69.92	70.19	555.6	39.39
Mg	KA1	1.254	0.0043	0.49	0.57	4.5	39.39
Fe	KA1	6.403	0.0057	0.67	0.34	2.7	1.00
Cu	KA1	8.046	0.0067	0.81	0.36	2.9	1.00
Zr	LA1	2.042	0.0022	0.53	0.17	1.3	39.39
Ni	KA1	7.477	0.0069	0.79	0.38	3.0	1.00
Total			0.7116	100.00	100.00	791.6	33.43

Table A.4.9 EDX quantitative analysis for spot 7 in figure 5.5.13.

Appendix 5: Pressure calculation

1) RSP cold compaction 30 MPa

2) University of Leicester cold compaction

a) Using aluminium canister

$$D = 32.33 \text{ mm} \quad r = D/2 = 32.33/2 = 16.156 \text{ mm}$$

$$F = 95.9 \text{ kN}$$

$$A = \pi (r)^2 = \pi (16.156 \text{ mm})^2 = 819.591 * 10^{-6} \text{ m}^2$$

$$P = F/A = 95.9 \text{ kN}/819.591 * 10^{-6} = 0.117 * 10^6 \text{ kN/m}^2 \\ = 117 \text{ MPa}$$

b) Using steel mould

$$D = 12.56 \text{ mm} \quad r = D/2 = 12.5/2 = 6.28 \text{ mm}$$

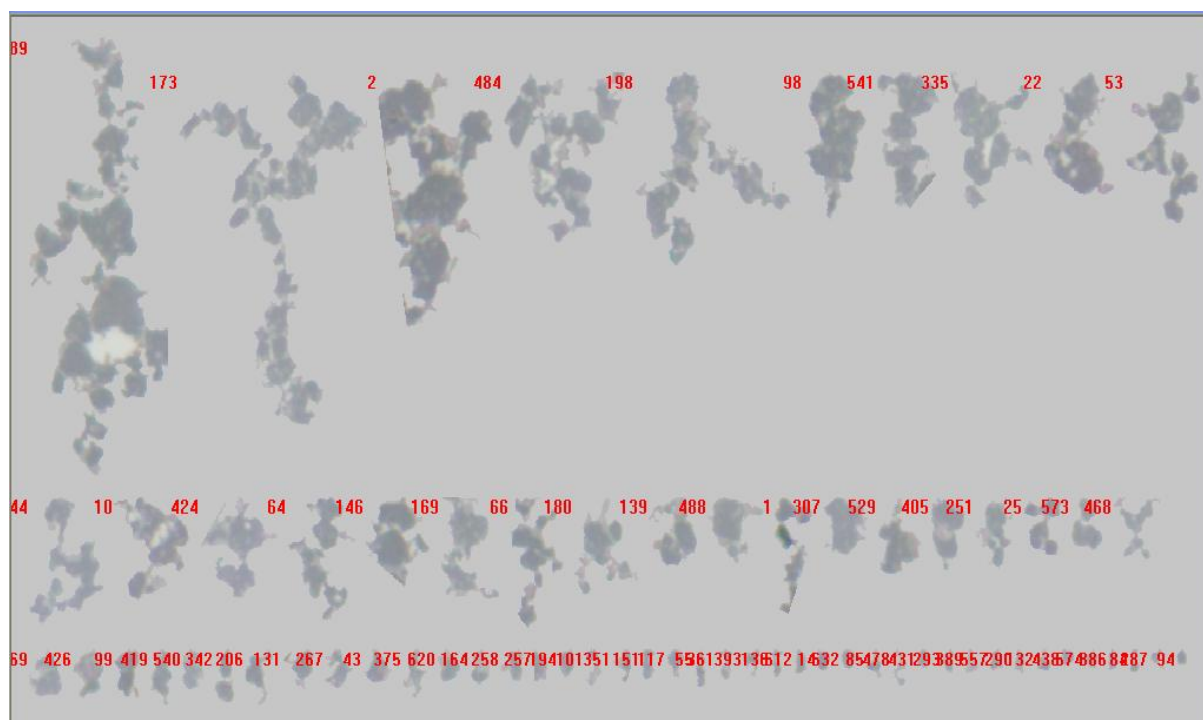
$$F = 93 \text{ kN}$$

$$A = \pi (r)^2 = \pi (6.28 \text{ mm})^2 = 123.836 * 10^{-6} \text{ m}^2$$

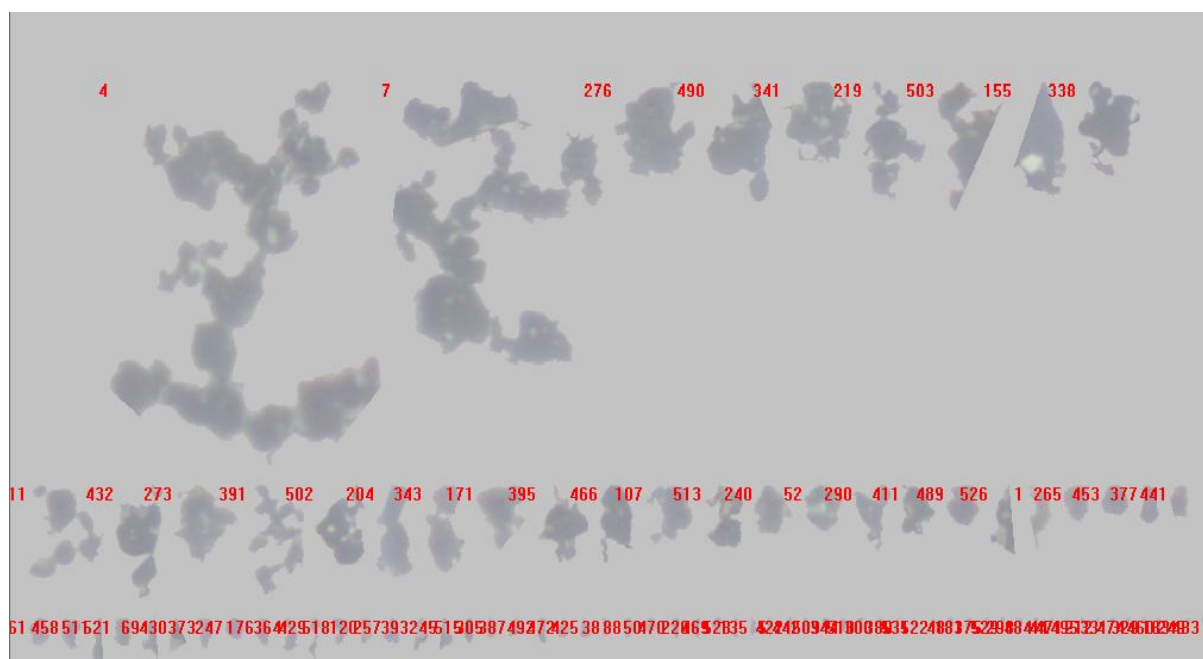
$$P = F/A = 93 \text{ kN}/123.836 * 10^{-6} = 0.750 * 10^6 \text{ kN/m}^2 \\ = 750 \text{ MPa}$$

[illegible]

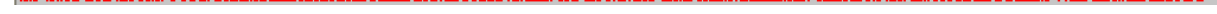
Appendix 6: Image analysis



461 Bar



444 Bar

[illegible]

Appendix 6: Image analysis



461 degassed and HIPed 400°C° for 12hrs.

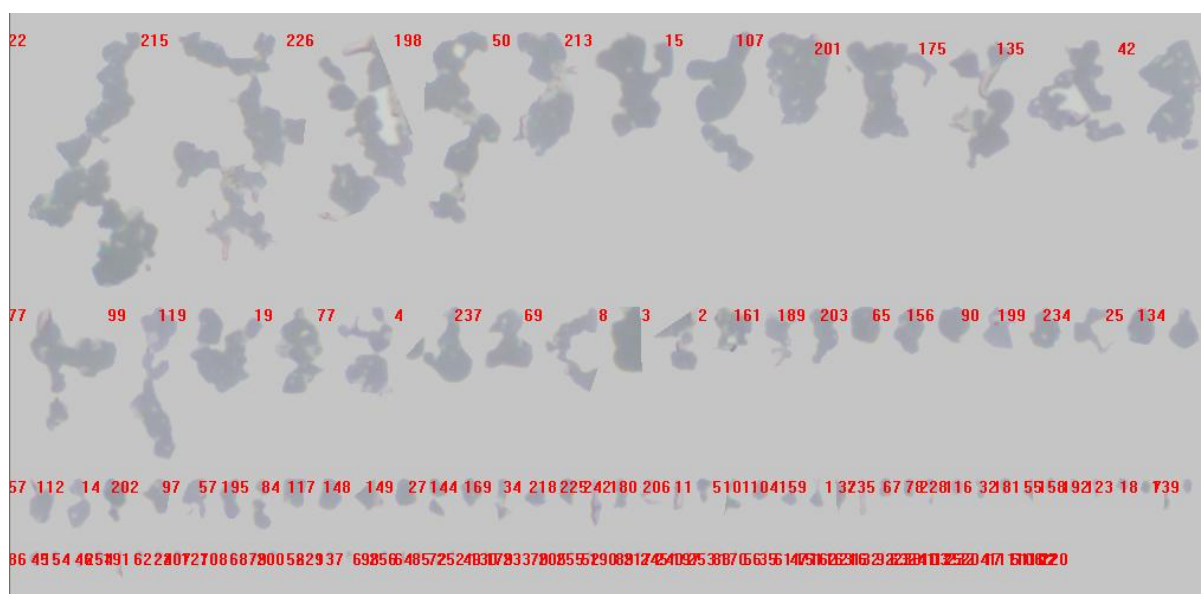


461 degassed and HIPed 500°C° for 12hrs.

Appendix 6: Image analysis



431 degassed and HIPed 400°C° for 3hrs.

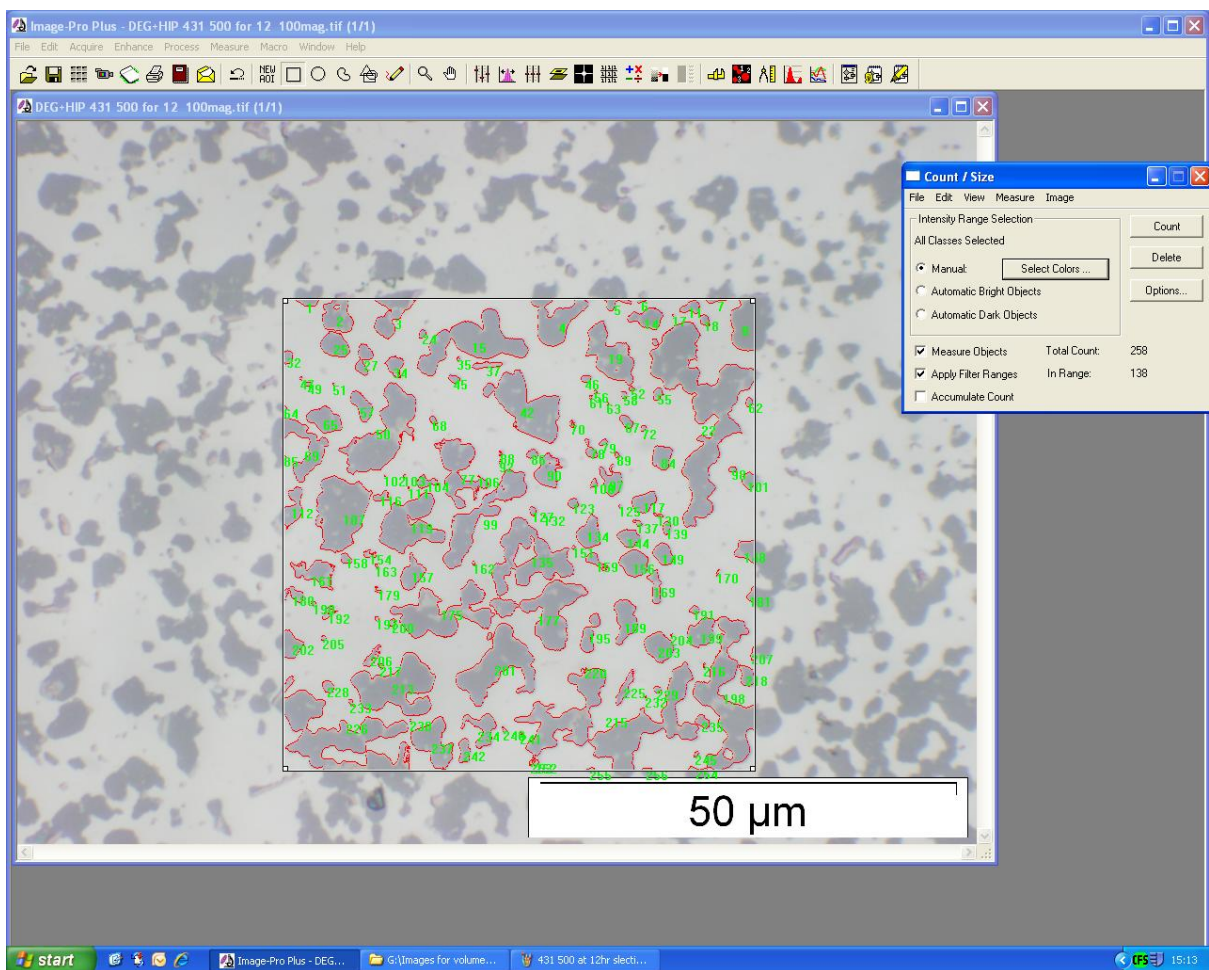


431 degassed and HIPed 400°C° for 12hrs.

Appendix 6: Image analysis



431 degassed and HIPed 500°C° for 12hrs.



Sample for Image Pro count for 431 degassed and HIPed 500°C° for 12hrs.

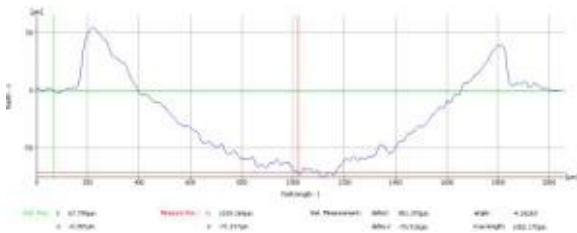
Appendix 6: Image analysis

Sample of Excel calculation for 431 degassed and HIPed 500°C° for 12hrs.

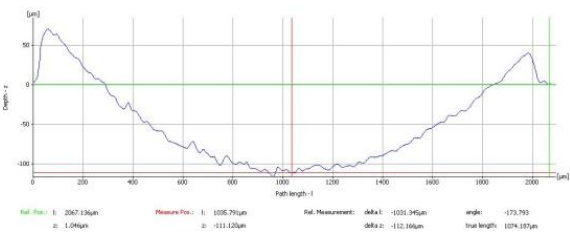
Total Count:	128					AOI, X (µm) =>	AOI, Y 56 (µm) =>
Obj#	Area	Radius (max)	Radius (min)	Radius Ratio	Feret (mean)	Total silicon area (µm ²)	Area fraction silicon
1	1.872621	1.582347	0.34396	4.600384	2.086249	960.6641	30.63%
2	10.04782	2.536093	1.127206	2.249893	4.082936		
3	10.09878	3.071583	0.015446	198.8571	4.858203		
4	18.43003	4.299112	0.65436	6.569948	6.471087		
5	1.815296	1.860995	0.06672	27.89248	2.538321		
6	0.248409	0.403259	0.143284	2.814408	0.587738		
7	0.82803	1.642477	0.126116	13.02351	1.805329		
8	15.00644	3.427097	1.215749	2.818917	5.247699		
9	1.875806	1.294231	0.336115	3.850564	1.858101		
11	5.799392	2.343828	0.761467	3.078044	3.24727		
12	39.14351	7.132931	1.424965	5.005688	10.3142		
14	0.76752	0.618326	0.329563	1.8762	1.012932		
17	20.35361	4.179233	1.053815	3.965812	6.323727		
18	92.38263	14.34122	0.79159	18.11699	18.29645		
21	0.420384	0.458722	0.212049	2.16328	0.758371		
23	6.853538	1.816264	1.155131	1.572344	3.073802		
24	0.200638	0.359881	0.128582	2.798855	0.534065		
28	2.162431	1.053798	0.538731	1.956075	1.721231		
32	1.070069	0.709565	0.407289	1.742166	1.213841		

Appendix 7: Wear profiles

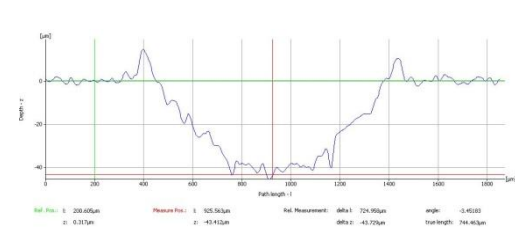
Extruded Bars



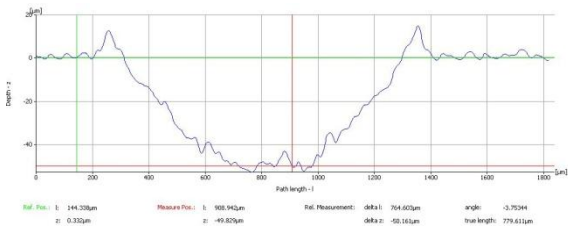
431, 100 N (1)



431, 100 N (2)

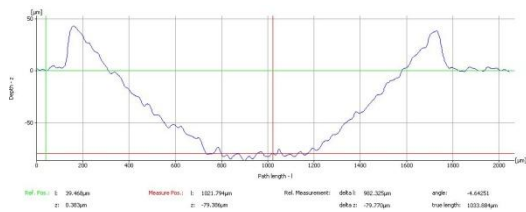


431, 10 N (1)

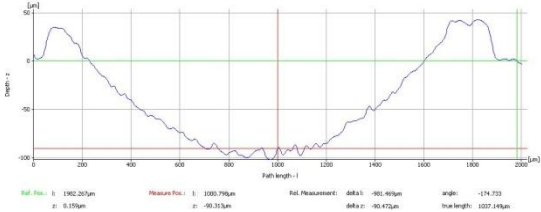


431, 10 N (2)

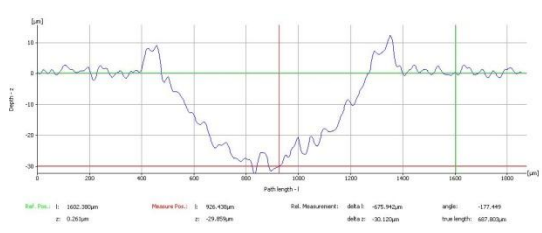
Appendix 7: Wear profiles



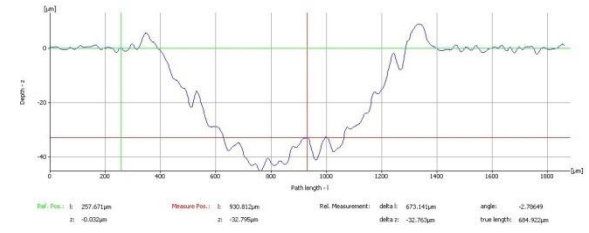
444 100N (1)



444 100N (2)

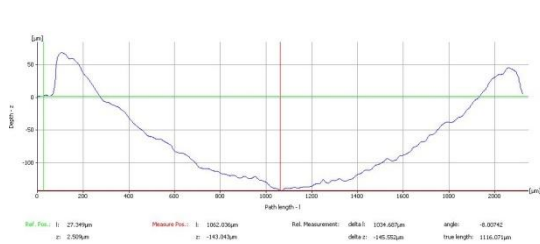


444 10N (1)

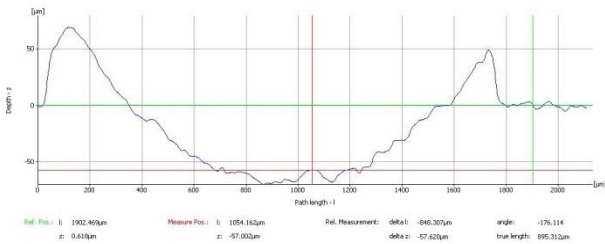


444 10N (2)

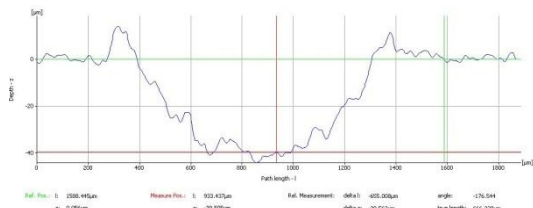
Appendix 7: Wear profiles



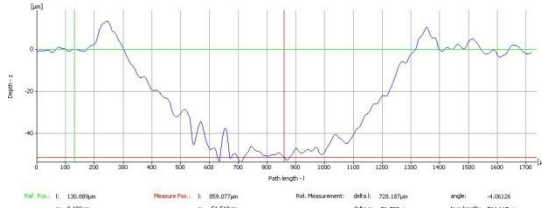
446-2 100N (1)



446-2 100N (2)

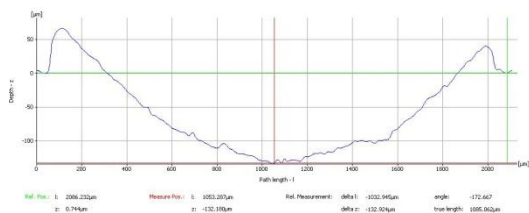


446-2 10N (1)

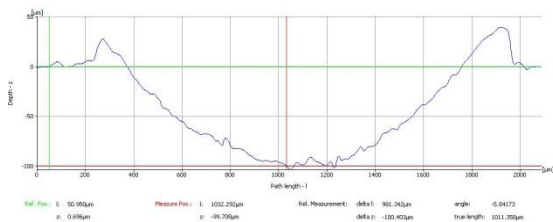


446-2 10N (2)

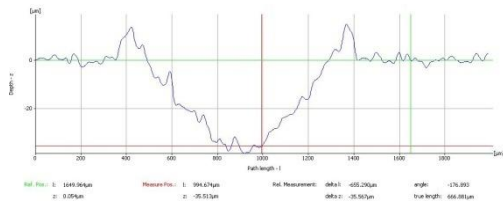
Appendix 7: Wear profiles



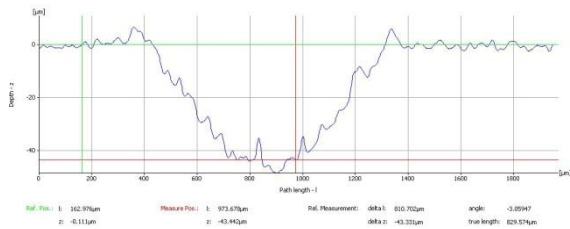
446-3 100N (1)



446-3 100N (2)

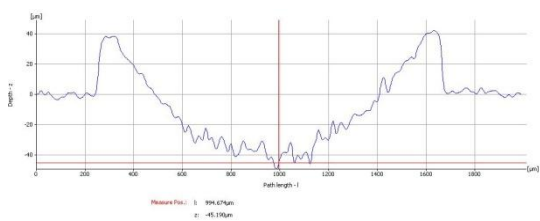


446-3 10N (1)

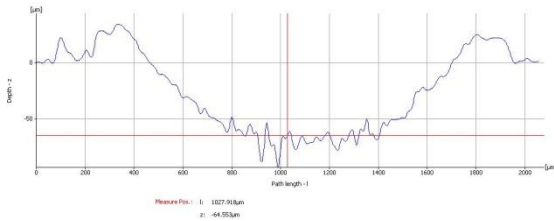


446-3 10N (2)

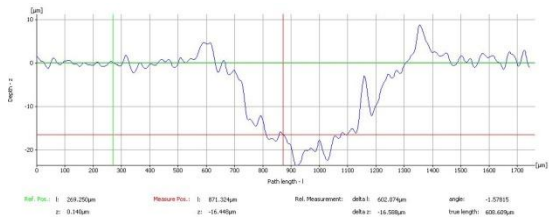
Appendix 7: Wear profiles



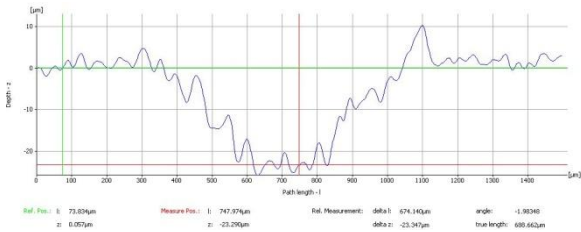
461 100N (1)



461 100N (2)



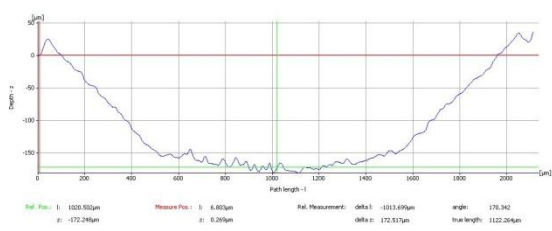
461 10N (1)



461 10N (2)

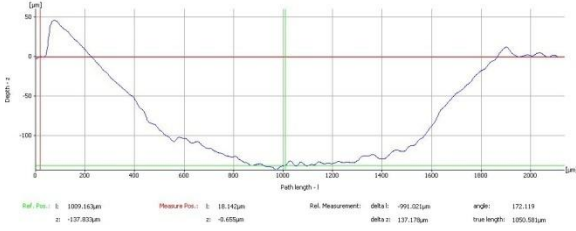
Appendix 7: Wear profiles

461 deg.+HIPped



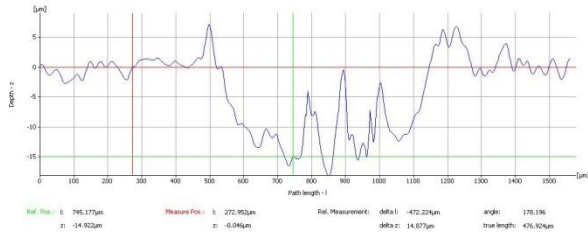
461 400°C/3hr 100N (1)

b



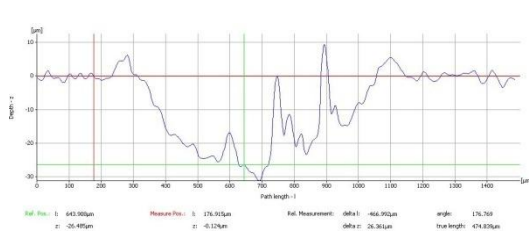
461 400°C/3hr 100N (2)

b



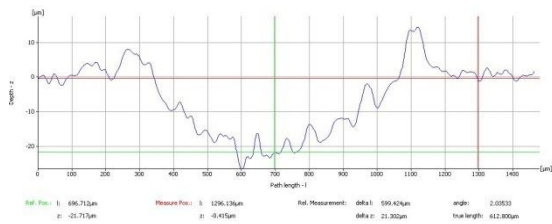
461 400°C/3hr 10N (1)

a

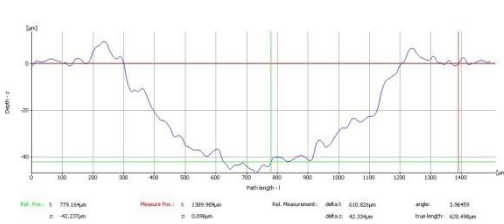


461 400°C/3hr 10N (2)

a

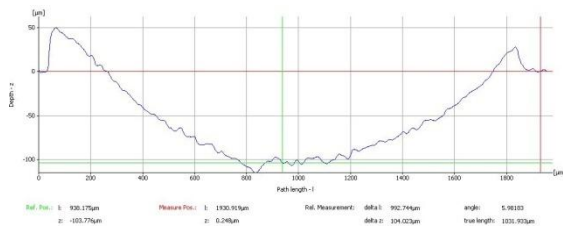


461 400°C/12hr 10N c1

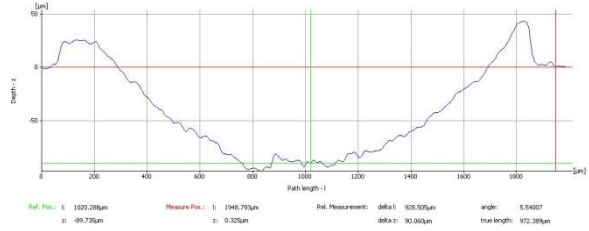


461 400°C/12hr 10N c2

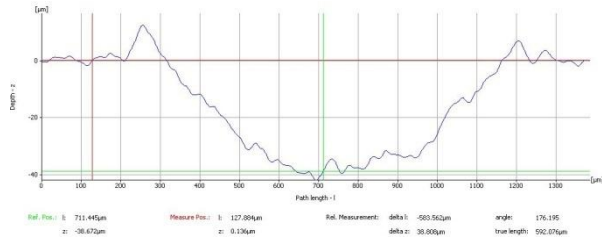
Appendix 7: Wear profiles



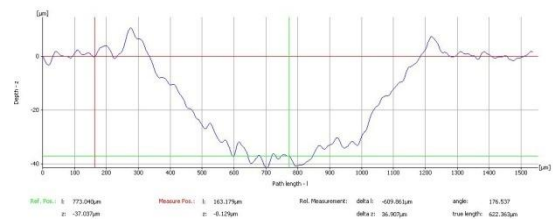
461 400°C/12hr 100N d1



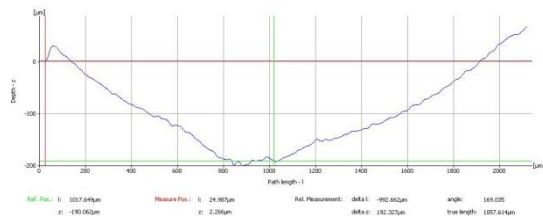
461 400°C/12hr 100N d2



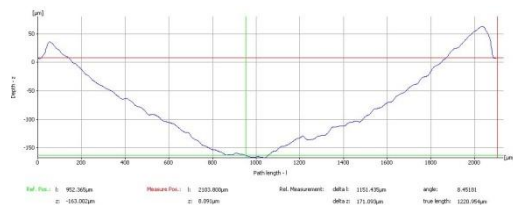
461 500°C/12hr 10N e1



461 500°C/12hr 10N e2



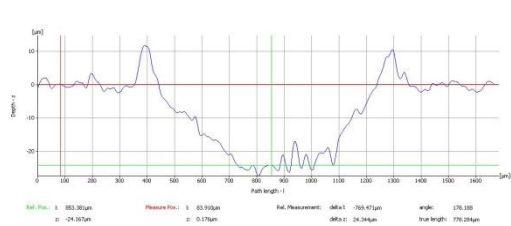
461 500°C/12hr 100N f1



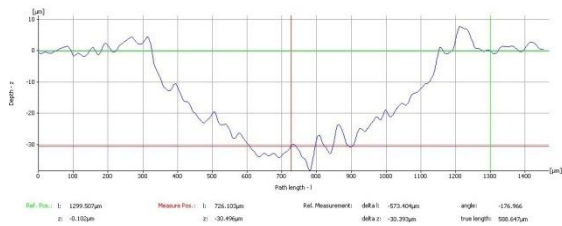
461 500°C/12hr 100N f2

Appendix 7: Wear profiles

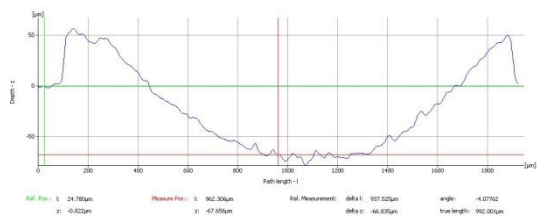
431 deg.+HIPped



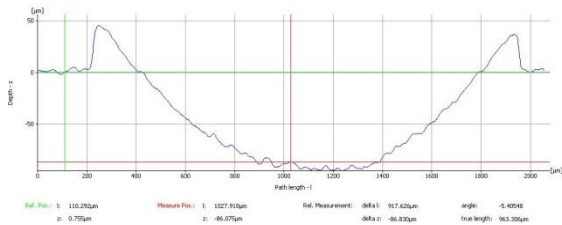
431 400°C/3hr 10N (1) g



431 400°C/3hr 10N (2) g

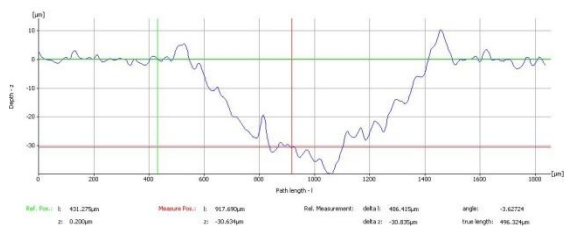


431 400°C/3hr 100N (1) h

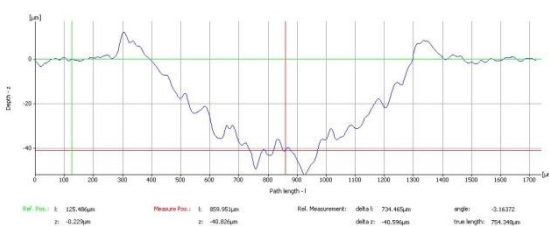


431 400°C/3hr 100N (2) h

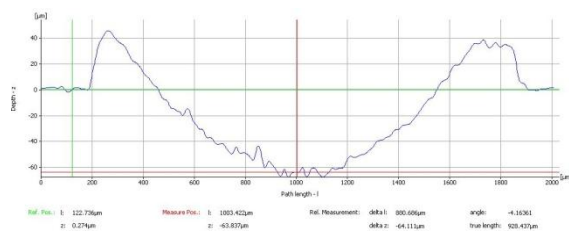
Appendix 7: Wear profiles



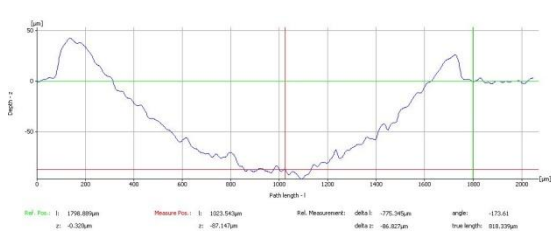
431 400°C/12hr 10N (1) i



431 400°C/12hr 10N (2) i

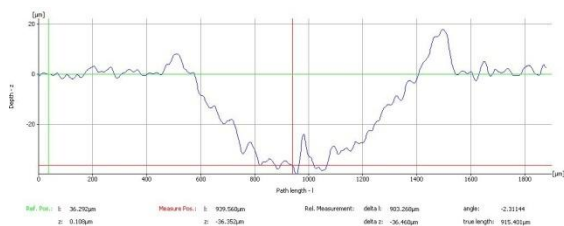


431 400°C/12hr 100N (1) j

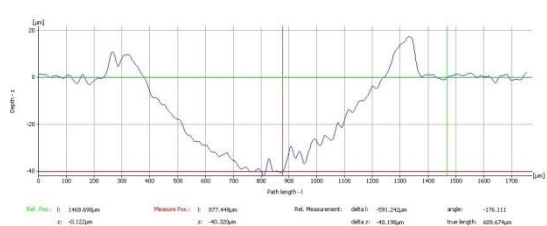


431 400°C/12hr 100N (2) j

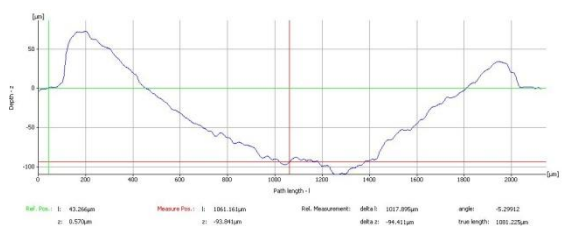
Appendix 7: Wear profiles



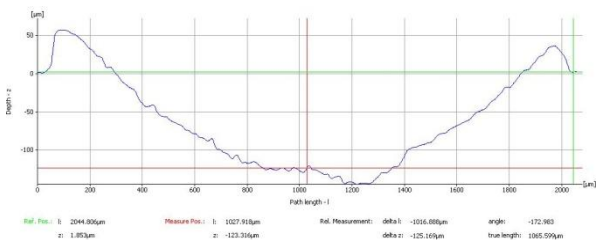
431 500°C/12hr 10N (1) k



431 500°C/12hr 10N (2) k



431 500°C/12hr 100N (1) L



431 500°C/12hr 100N (2) k

Appendix 7: Wear profiles

Appendix 8: XRD spectra

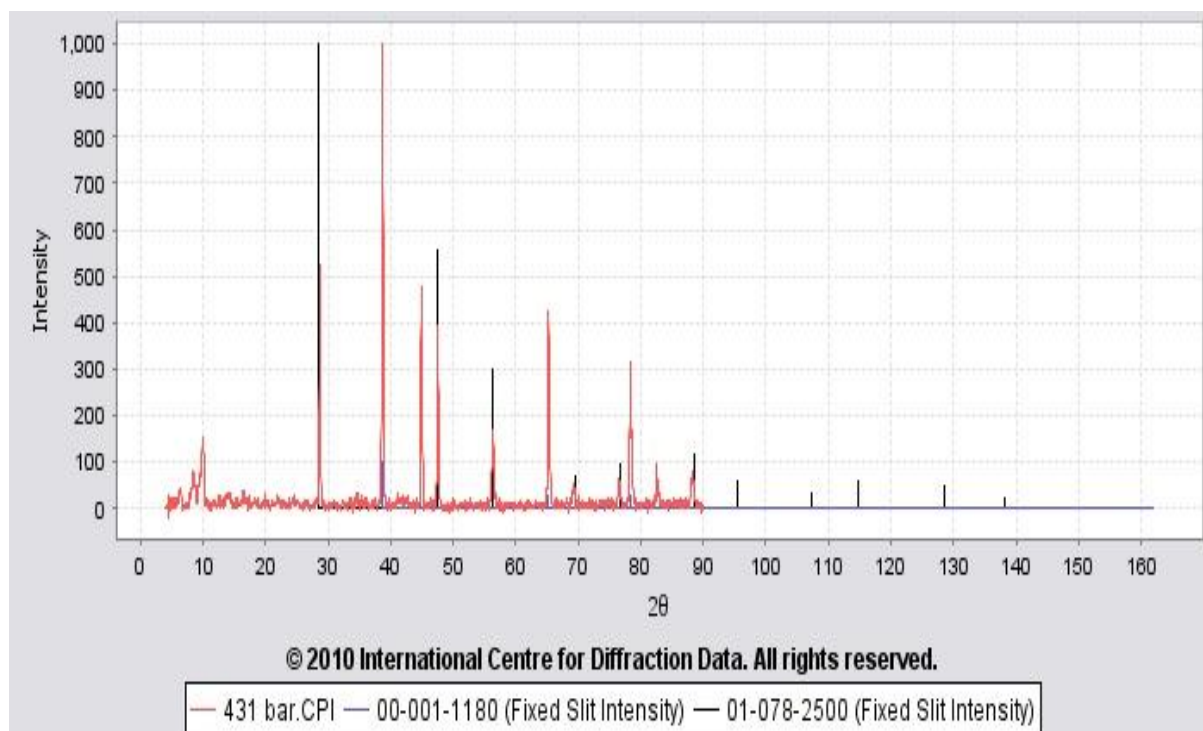


Figure 1 XRD spectra of extruded bar 431, the code 00-001-1180 = Al and 01-078-2500 = Si.

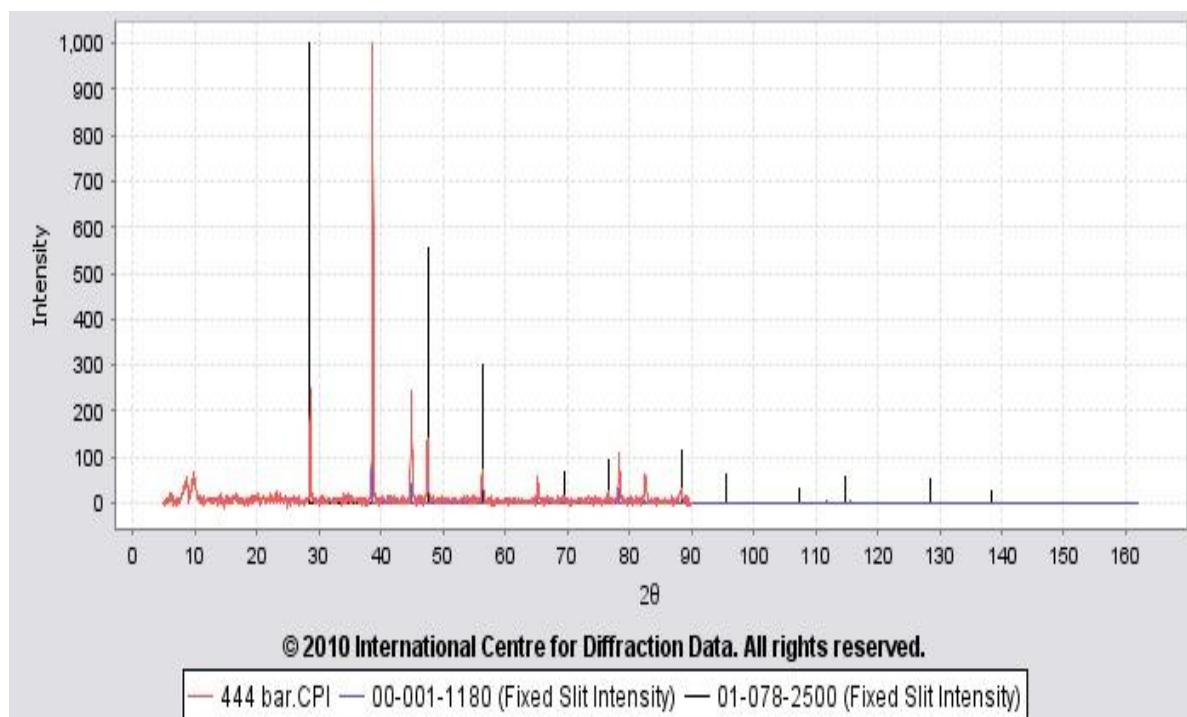


Figure 2 XRD spectra of extruded bar 444, the code 00-001-1180 = Al and 01-078-2500 = Si.

Appendix 8: XRD spectra

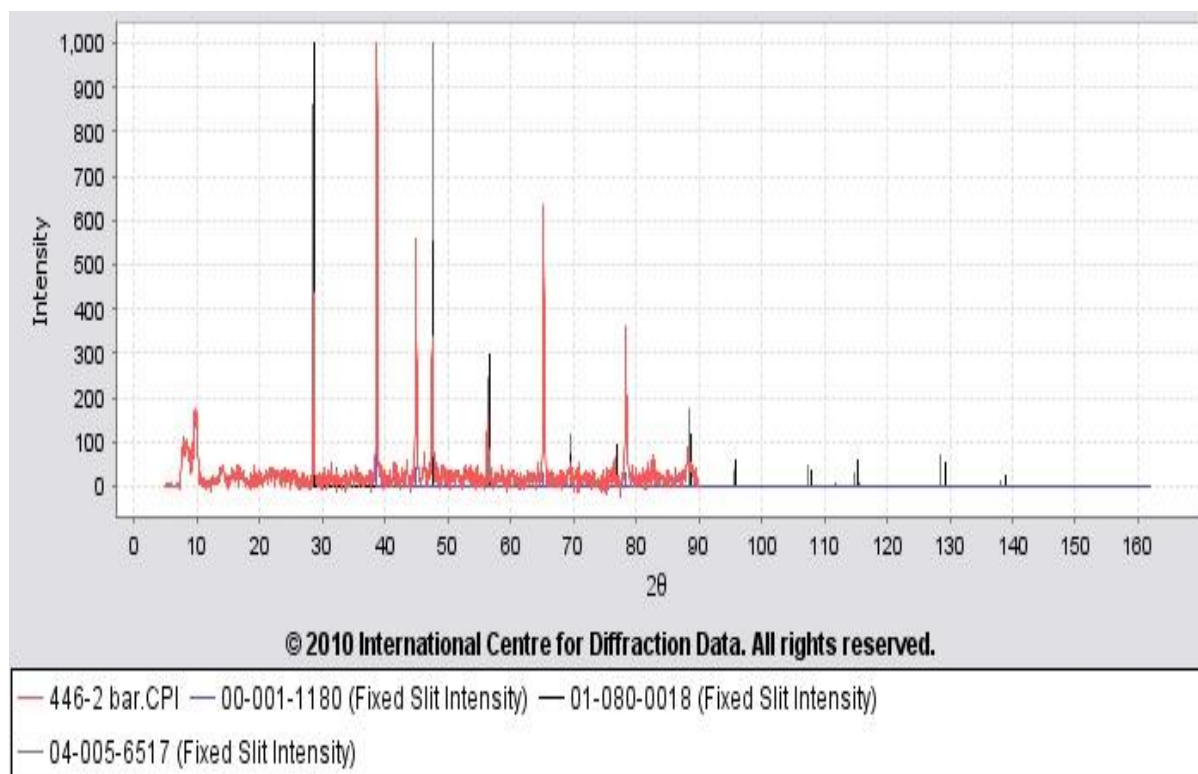


Figure 3 XRD spectra of extruded bar 446-2, the code 00-001-1180 = Al, 01-080-0018 = Si and 04-005-6517 = Ni Si₂.

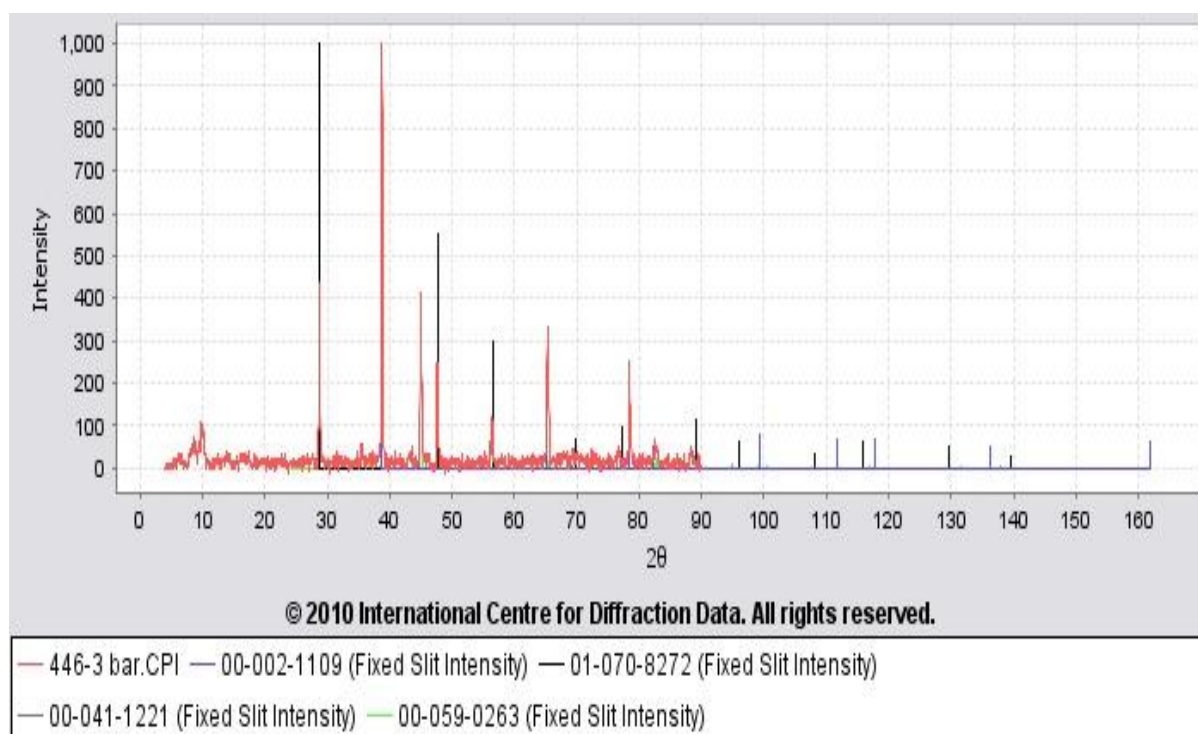


Figure 4 XRD spectra of extruded bar 446-3, the code 00-002-1109 = Al, 01-070-8272 = Si, 00-041-1221 = Fe_{0.42} Si_{2.67} and 00-059-0263 = Cu₃ Si.

Appendix 8: XRD spectra

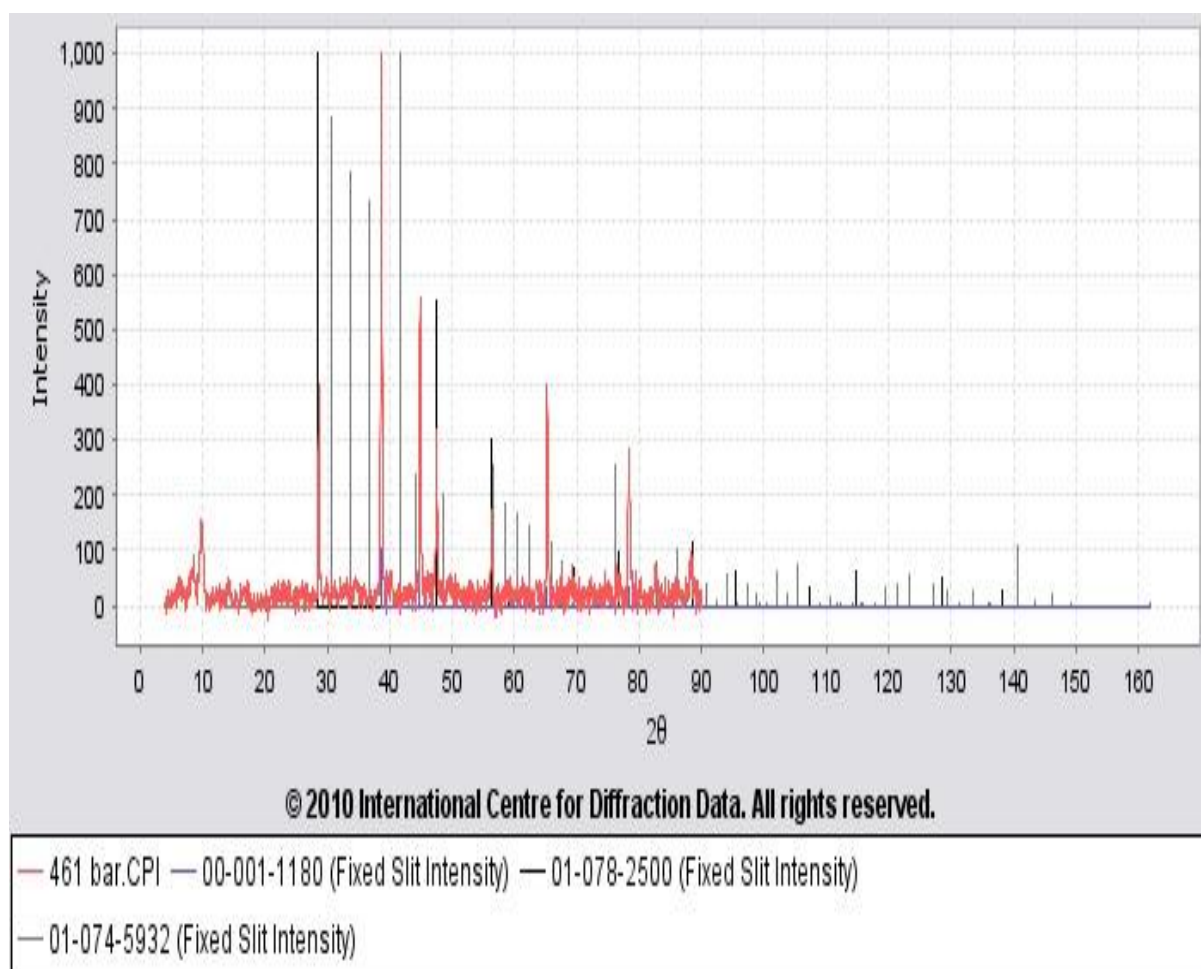


Figure 5 XRD spectra of extruded bar 461, the code 00-001-1180 = Al, 01-078-2500 = Si and 01-074-5932 = Mg₂ Si.