INFLUENCE OF PROCESSING CONDITIONS ON THE MICRO-MECHANICAL PROPERTIES OF PARTICULATE-REINFORCED ALUMINIUM MATRIX COMPOSITES

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ABSTRACT

During processing, metal matrix attempts to deform and this deformation plays the key role in the micro-structural events of segregation and precipitation at the matrix-reinforcement interface. The important aspect of this behaviour is to identify the strengthening micro-characteristics which enhance the material's interphasial reactions in order to improve the bonding properties of the matrix-reinforcement interface. This work focuses on the non-equilibrium segregation which arises due to imbalances in point defect concentrations set up around interfaces during non-equilibrium heat treatment processing of SiC particle reinforced aluminium matrix composites. The important factors affecting the heat treatment process are the temperature, the cooling rate, the concentration of solute atoms and the binding energy between solute atoms and vacancies. Aluminium – silicon – magnesium alloy matrix reinforced with varying amounts of silicon carbide particles were used in this study. Samples in the as-received and heat treated condition were examined by microstructural and micro-hardness analyses. Based on the analysis, it has been observed that the macroscopic mechanical behaviour of the composite is influenced by several factors including the manufacturing process, the processing conditions, the inter-particle distance, as well as the mean size and the percentage of reinforcement.

Keywords: Metal matrix composites (MMCs); Heat Treatment; Interfacial strength; Particulates; Precipitation.

1. INTRODUCTION

Silicon carbide particulate-reinforced aluminium matrix composites are attractive engineering materials for a variety of structural applications, due to their superior strength, stiffness, low cycle fatigue and corrosion fatigue behaviour, creep and wear resistance, compared to the aluminium monolithic alloys.

An important feature of the microstructure in the Al/SiC composite system is the increased amount of thermal residual stresses, compared to unreinforced alloys, which are developed due to mismatch in thermal expansion coefficients of matrix and reinforcement phases. The introduction of the reinforcement plays a key role in both the mechanical and thermal ageing behaviour of the composite material. Micro-compositional changes which occur during the thermo-mechanical forming process of these materials can cause substantial changes in mechanical properties, such as ductility, fracture toughness and stress corrosion resistance.

Particulate-reinforced composites are not homogeneous materials; hence bulk material properties not only are sensitive to the constituent properties, but strongly depend on the properties of interface. The strength of particulate-reinforced composites depends on the size of the particles, interparticle spacing, and the volume fraction of the reinforcement [1].

In the case of particulate-reinforced aluminium composites, the microstructure and mechanical properties can be altered by thermo-mechanical treatment as well as by varying the reinforcement volume fraction. The strengthening of monolithic metallic material is carried out by alloying and supersaturating, to an extent, that on suitable heat treatment the excess alloying additions precipitates out (ageing).

2. MATERIALS

The metal matrix composites studied were aluminium – silicon – magnesium alloy matrix A359 reinforced with varying amounts of silicon carbide particles. Aluminium alloys A359 are important materials in many industrial applications, including

aerospace and automotive applications.

For the investigation, four types of material were used: 1) Hot Rolled A359/20%SiC, with an average particle size of 17±1 micron, 2) Hot rolled A359/31%SiC with an average particle size of 17±1 micron and 3) Cast alloy A359/30%SiC with particles of F400grit, with an average particle sizes of 17±1 micron. Table 1, contains the details of the chemical composition of the matrix alloy as well as the amount of silicon carbide particles in the metal matrix composites according to the supplier specifications [2].

Table 1: Chemical Composition (wt %) [MC-21]

TYPES	Si	Mg	Mn	Cu	Fe	Zn	SiC
ROLLED A359	9.5	0.5	0.1	0.2	0.2	0.1	20
ROLLED A359	9.5	0.5	0.1	0.2	0.2	0.1	31
CAST A359	9.5	0.5	0.1	0.2	0.2	0.1	30

The alloys from the Al-Si-Mg system are the most widely used in the foundry industry thanks to their good castability and high strength to weight ratio. Si improves the fluidity of Al in the molten state and, also, Si particulates improve the wear resistance of reinforced aluminium alloy. By adding Mg, Al – Si alloy become age hardenable through the precipitation of Mg2Si particulates. An additional advantage of Al – Si alloys for casting applications is that Si expands on solidification and Si is needed to form Mg2Si. The precipitation sequence is supersaturated solid solution → GP zones → β' → β (Mg2Si). The GP zones are needled-shaped along the aluminum matrix and the β'phase is rod-shaped along the matrix. The equilibrium phase β is face centred cubic and forms platelets on the matrix [3].

The materials used were produced by MC-21, Inc.[2] using a patented mixing process that allows SiC particles to be mixed into molten aluminium more rapidly with the benefit of a wider range of volume fractions and sizes of reinforcement.

3. HEAT TREATMENT

3.1 Precipitation Hardening

Properties in particulate-reinforced aluminium matrix composites are primarily dictated by the uniformity of the second-phase dispersion in the matrix. The distribution is controlled by solidification and can be later modified during secondary processing. In particular, due to the addition of magnesium in the A359 alloy, the mechanical properties of this material can be greatly improved by heat treatment process. There are many different heat treatment sequences and each one can modify the microstructural behaviour as desired [4]. Precipitation heat treatments generally are low temperature, long-term processes. Temperatures range from 110°C to 195°C for 5 to 48 hours. The selection of the time temperature cycles for precipitation heat treatment should receive careful consideration. Larger precipitate particulates result from longer times and higher temperatures. On the other hand, the desired number of larger particles formed in the material in relation to their interparticle spacing is a crucial factor for optimising the strengthening behaviour of the composite. The objective is to select the heat treatment cycle that produces the most favourable precipitate size and distribution pattern. However, the cycle used for optimising one property, e.g. tensile strength, is usually different from the one required to optimise a different property, e.g. yield strength, corrosion resistance.

Heat treatment of composites though has an additional aspect to consider, the particles introduced in the matrix. These particles may alter the alloy's surface characteristics and increase the surface energies [5].

3.2 Heat Treatment

The heat treatments were performed in Carbolite RHF 1200 furnaces with thermocouples attached, ensuring constant temperature inside the furnace. There were two different heat treatments used in the experiments, T6 and modified-T6 (HT-1) [6].

The T6 heat treatment consists of the following steps: solution heat treatment, quench and age hardening (Fig.1). In the solution heat treatment, the alloys have been heated to a temperature just below the initial melting point of the alloy for 2 hours at 530±5 ºC where all the solute atoms are

Fig.1: T6 Heat treatment diagram showing the stages of the solution treatment for 2 hours and artificial ageing for 5 hours.

allowed to dissolve to form a single phase solid solution. Magnesium is highly reactive with Silicon at this temperature and precipitation of Mg_2Si is expected to be formed. The alloys were then quenched to room temperature. In age hardening, the alloys were heated to an intermediate temperature of 155ºC for 5 hours where nucleation and growth of the β' phase. The desired β phase Mg_2Si precipitated at that temperature and then cooled at room temperature conditions. The precipitate phase nucleates within the grains at grain boundaries and in areas close to the matrix-reinforcement interface, as uniformly dispersed particles. The holding time plays a key role in promoting precipitation and growth which results in higher mechanical deformation response of the composite.

The second heat treatment process was the modified-T6 (HT-1) heat treatment, where in the solution treatment the alloys have been heated to a temperature lower than the T6 heat treatment, at 450±5ºC for 1 hour, and then quenched in water. Subsequently the alloys were heated to an intermediate temperature of 170±ºC for 24 hours in the age hardened stage and then cooled in air (Fig.2).

In both heat treatments undesired formation of phases, like the Al_4C_3, is a possibility and selection of the solution treatment as well as the age hardening processes should be carefully considered. Tempera-

Fig.2: Modified T6 (HT-1) showing stages of solution treatment for 1 hour and artificial ageing for 24 hours.

ture and time control, therefore, is extremely important during heat treatment. If the melt temperature of SiC/Al composite materials rises above a critical value, Al_4C_3 is formed increasing the viscosity of the molten material, which can result in severe loss of corrosion resistance and degradation of mechanical properties in the cast composite; excessive formation of Al_4C_3 makes the melt unsuitable for casting. In the A359/SiC composite high silicon percentage added in excess aids to the formation of some oxides (SiO_2) around the SiC reinforcement something that retards the formation of Al_4C_3 [5].

4. MICROSTRUCTURAL ANALYSIS

In order to analyse the microstructure, both as received and heat treatment conditions, a series of sample preparation exercises were carried out, consisted of the cutting, mounting, grinding and polishing of the samples. The microstructures were investigated by using a Philips XL40 Scanning Electron Microscope with a link 860 EDAX, a Philips FEI Nova Nano – Scanning Electron Microscope and X-ray diffraction (XRD) technique with a link to Philips X'Pert High Scores software 2000. The microhardness was determined by a Mitutoyo Muk-H1 Hardness tester.

4.1 SEM-EDAX-Mapping Results

The microstructures of the examined MMCs in the as received condition have four distinct micro phases as clearly marked on the image micrograph, which are as follows: the aluminium matrix, the SiC

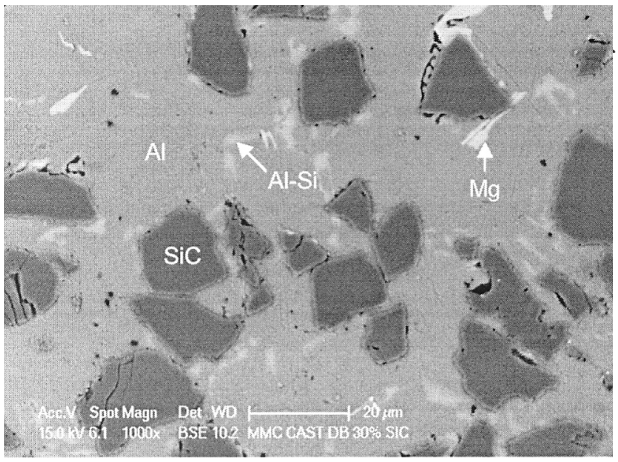

Fig.3: Microstructure of cast 30% SiC in the as received condition showing four distinct phases: Aluminium matrix, SiC particles, eutectic region of aluminium and silicon and *M*g phase.

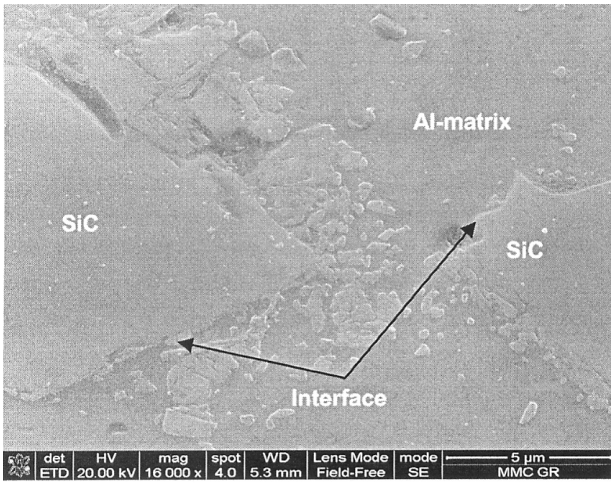

Fig.4: Microstructure of rolled 31% SiC in the as received condition showing matrix-reinforcement interfaces.

ticle clustered zones were observed.

Matrix-reinforcement interfaces were identified by using high magnification Nano-SEM microscope. In the as received hot rolled images the Al matrix/ SiC reinforcement interface is clearly identified (Fig.4). These interfaces attain properties coming from both individual phases of constituents and facilitate the strengthening behaviour of the composite material.

In the modified T6 (HT-1) condition the microstructure of the cast 30% SiC has the same phases as in the as received state, plus one rod-shape phase (Fig.5a, 5b) along the matrix and at the matrix-reinforcement interface has been identified to be Mg_2Si precipitates in an early stage which are not fully grown. This evidence shows that β' phase has been formed with magnesium and silicon reacting together but β phases forming platelets of precipitates have not been formed in this HT-1 heat treatment, and this is probably due to the solution treatment temperature that did not allow enough reactivity time between the main alloying elements.

In the rolled 20% SiC the microstructure of HT-1 heat treatment shows an increase of the Silicon phase as shown in the image (Fig 6a). Silicon has been expanded during solidification and subsequent ageing. This formed round areas around the whole area of the composite. Comparing with the cast 30% SiC sample, in the rolled material the silicon phase

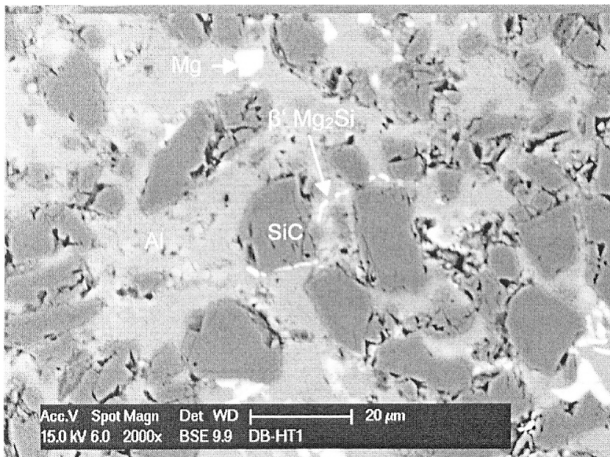

Fig.5a: Microstructure of cast 30% SiC in the HT-1 condition showing rod shape β' phases of Mg2Si around the matrix and the interface of the reinforcement.

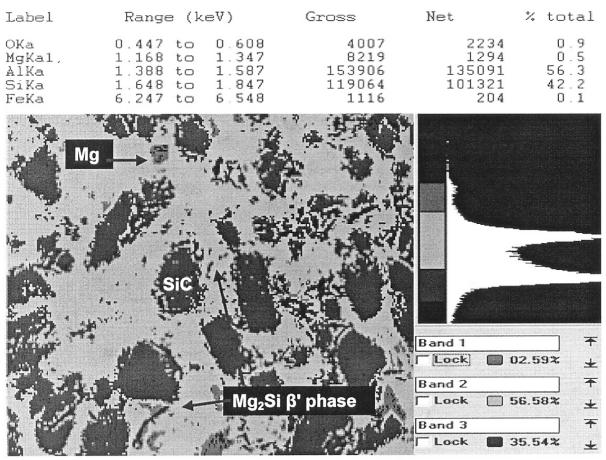

Fig.5b: Cast 30% SiC - HT-1 sample showing phases and percentages. Aluminium (green), SiC (blue), Mg_2Si phase (red and dark as pointed in the image). Oxygen and Fe is also present in small percentages.

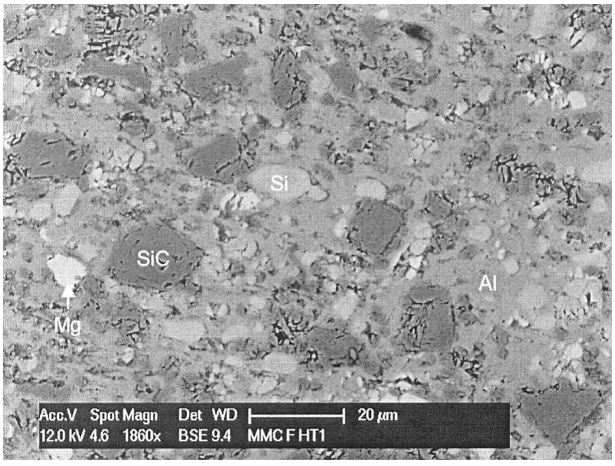

Fig.6a: Hot rolled HT-1 sample showing phases of Aluminium, SiC, Silicon, Mg.

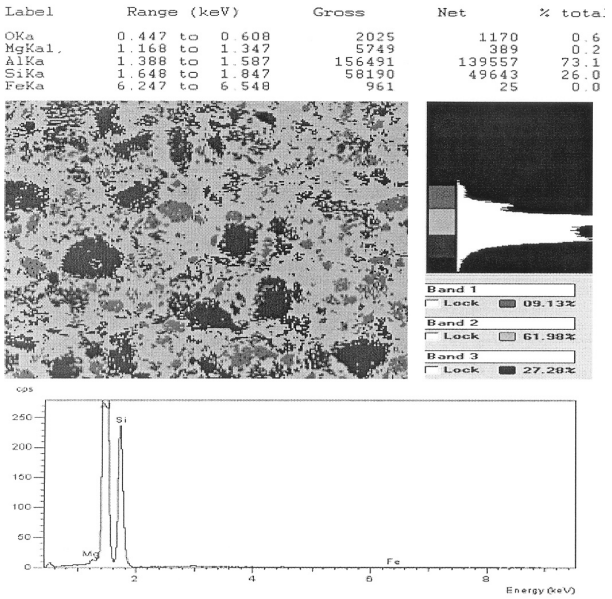

Fig.6b: Hot rolled 20% SiC - HT-1 microstructural analysis showing phases and elemental percentages. Silicon phase (red) has been expanded.

is increased by 6%, as shown in (Fig 6b). This increase under the same heat treatment conditions is explained by the difference in the percentage of reinforcement in the material. Therefore, it becomes evident that the introduction of SiC reinforcement promotes zone kinetics and phase formation reactions during heat treatment process. The reinforcement, depending on its percentage in the matrix material, accelerates or restrains events such as precipitation and segregation. This is further supported by the fact that precipitation has not been observed in the HT-1 heat treated 20% SiC rolled material, where lower percentage of SiC reinforcement slowed-down the precipitation kinetics and β' phases could not be created in a similar manner as the 30% SiC cast sample.

In the T6 condition the microstructural results showed that in the rolled 31% SiC sample precipitates of Mg2Si have been formed in a platelet shape in the matrix as well as in areas close to the interface (Fig.7a, 7b). The higher solution temperature and lower age hardening holding time that exist in the T6 heat treatment process, promoted the forming of this type of precipitates, which act as support to strengthening mechanisms of the reinforcement-matrix interface. In the case of presence of a crack in the matrix, these precipitates act as strengthening aids promoting crack deflection at the interface

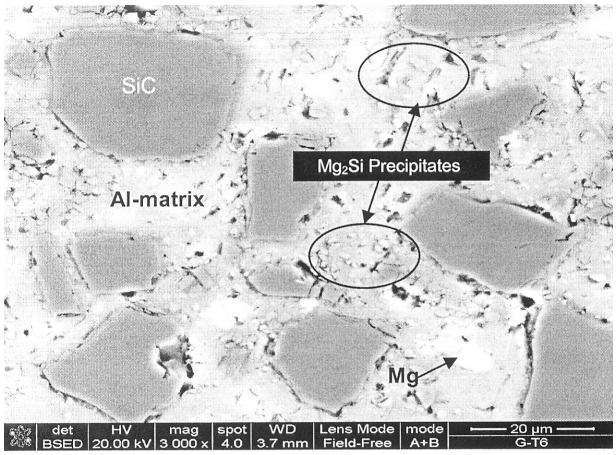

Fig.7a: Hot rolled 31% SiC –T6 showing precipitates formed around the reinforcement.

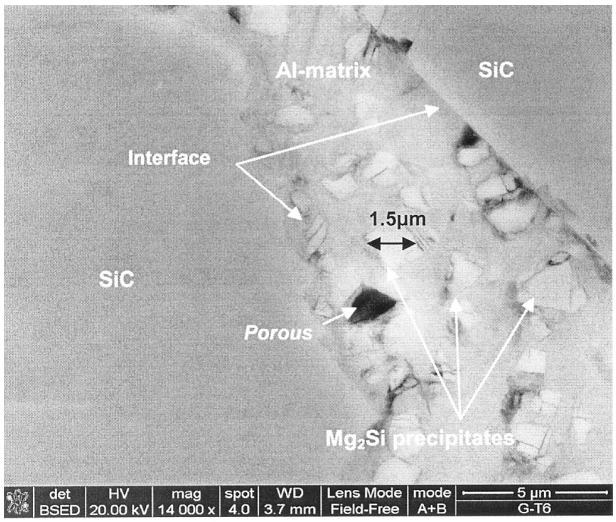

Fig.7b: Hot rolled 31% SiC – T6 showing Mg_2Si precipitates formed between the SiC reinforcement interface in a platelet shape of around 1-3 µm. A porous close to the interface has been identified in a similar size.

resulting in an increase of the composite's fracture toughness [7-8].

Furthermore, in the T6 condition, Fe elements have been identified by Edax-mapping technique, therefore, demonstrating the existence of a new phase in the composite due to the reaction of Fe with other major alloying elements (Fig.10c).

4.2 XRD Result

The X-ray diffraction was carried out on the MMCs in the as received, as well as, in the heat treatment conditions, in samples with 20%, 30% and 31% of SiC particulates. Even though some peaks were superimposed, the results clearly showed the phases present in the microstructures. In particular, in the as received condition and in the heat treatment conditions the results showed existence of aluminium matrix material, eutectic silicon, SiC, Mg_2Si, SiO_2 phases as the distinct ones, and also $MgAl_2O_4$ and Al_2O_3 phases (Fig.8). $MgAl_2O_4$ and Al_2O_3 oxides give good cohesion between matrix and reinforcement when forming a continuous film at the interface. The presence of $MgAl_2O_4$ (spinel) shows that low percentage of magnesium reacted with SiO_2 at the surface of SiC and formed this layer in the interphacial region between the matrix and the reinforcement (Eq.1). This layer has been identified by SEM-EDAX technique (Fig.9).

$$2SiO_2 + 2Al + Mg \rightarrow MgAl_2O_4 + 2Si \qquad (1)$$

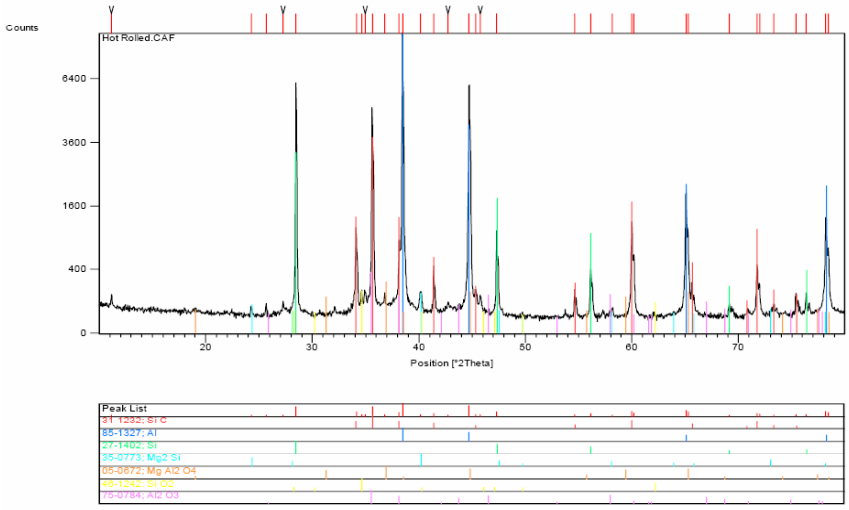

Fig.8: XRD of hot rolled 31% SiC as received sample showing phases present and some superimposed oxides ($MgAl_2O_4$ and Al_2O_3).

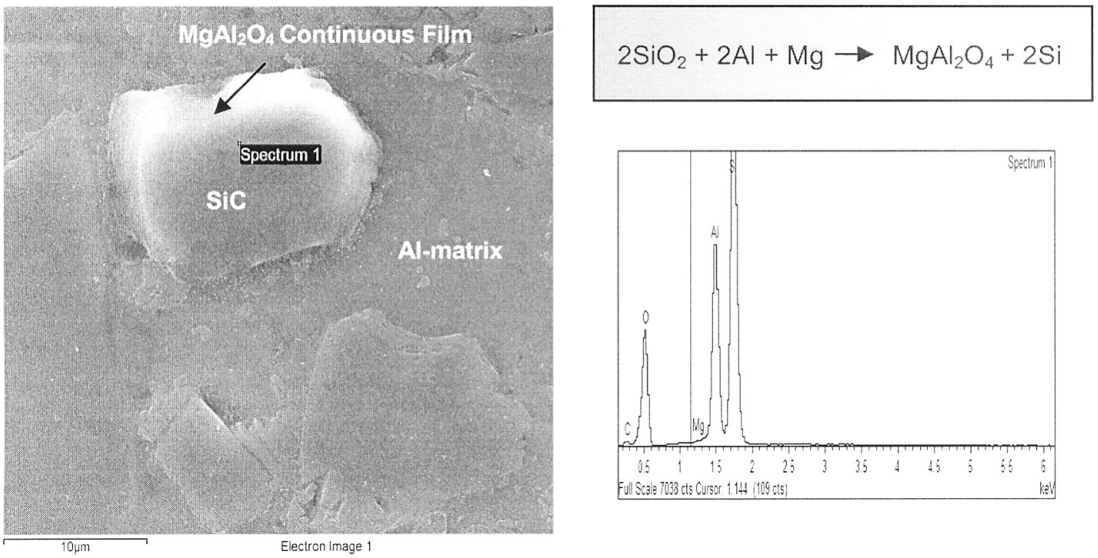

Fig.9: $MgAl_2O_4$ phase observed to be a continuous film around the SiC particle. (white area)

The layers of $MgAl_2O_4$ protect the SiC particles from the liquid aluminium during production or remelting of the composites. This layer provides more than twice bonding strength compared to Al_4C_3. Furthermore, the layer of Al_2O_3 oxide is formed as a coating when SiO2 is reacting with liquid Aluminium (Eq.2).

$$3SiO_2 + 4Al \rightarrow 2Al_2O_3 + 3Si \quad (2)$$

The presence of Al_4C_3 could not be identified by XRD in all samples in the as received or heat treated states, something that verifies that high percentage of Si added in the composite during manufacturing, leading to forming of Al_2O_3, retards Al_4C_3 formation in the composite [9].

The same phases have been identified in the HT-1 modified condition. In the T6 condition XRD results showed one more phase present which is the spinel-type mixed oxide $MgFeAl0_4$ showing that Fe trace reacted with Mg and in the presence of aluminium and oxygen formed this oxide (Fig.10a). The presence of Fe has also been identified in this study by microscopic analysis (Fig.10b, 10c).

Porosities were observed in some of the samples. A total avoidance of porosity is difficult to achieve, because the lower thermal conductivity of ceramic reinforcements requires them to be pushed to the solidifying front of a freezing melt in such way that shrinkage porosities appear around the particulate as the matrix shrinks during solidification. Also, as magnesium is surface active, it effectively reduces interfacial energies, resulting in the development of gas (due to air) and shrinkage porosity when an optimum amount of reinforcements is present [10].

Microscopic porosity was observed in specific areas of the reinforced and unreinforced regions of the composites in the as received as well in the heat treatment conditions. (Fig.7b). Porosities of 1-3 μm in size and ≈1 wt% were present in the materials examined. In the heat treated samples porosity was increased and found to be 1.5 wt% in the material. This is due to the treatment condition and these porosities may have been formed by solidification shrinkage, thus cannot be considered as major defects.

5. MICROHARDNESS TESTING

The three samples have been compared in relation to their microhardness performance based on the reinforcement percentage, the heat treatment conditions and the different manufacturing forming processes. Microhardness of the three composites has been measured in order to get the resistance of the material to indentation, under localized loading conditions. The microhardness test method, according

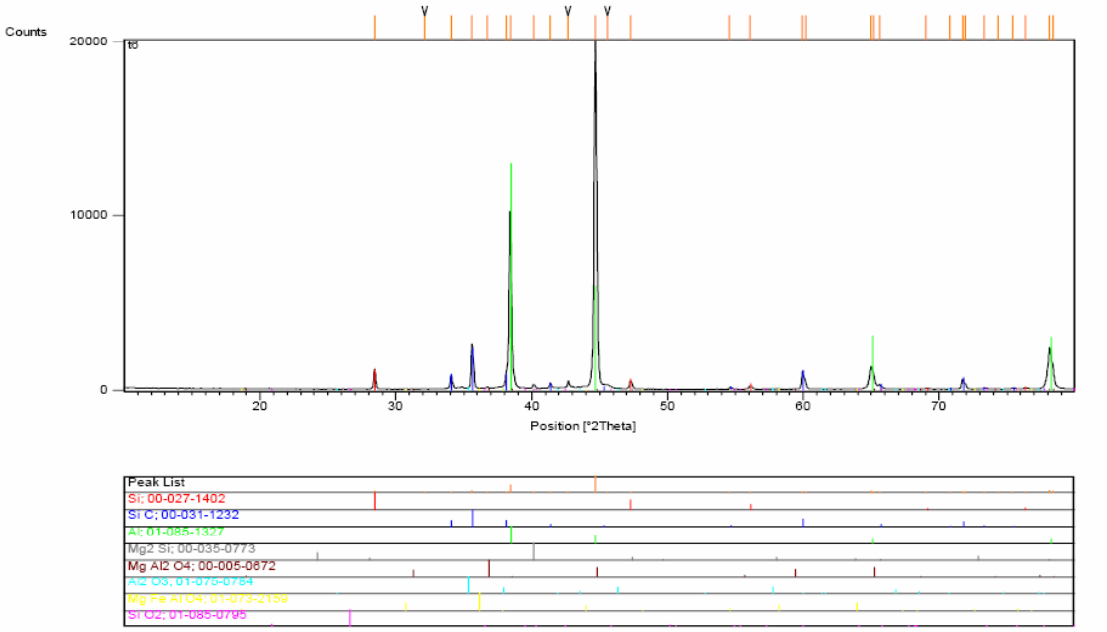

Fig.10a: XRD of hot rolled 31% SiC - T6 sample showing phases present and $MgFeAl0_4$ phase.

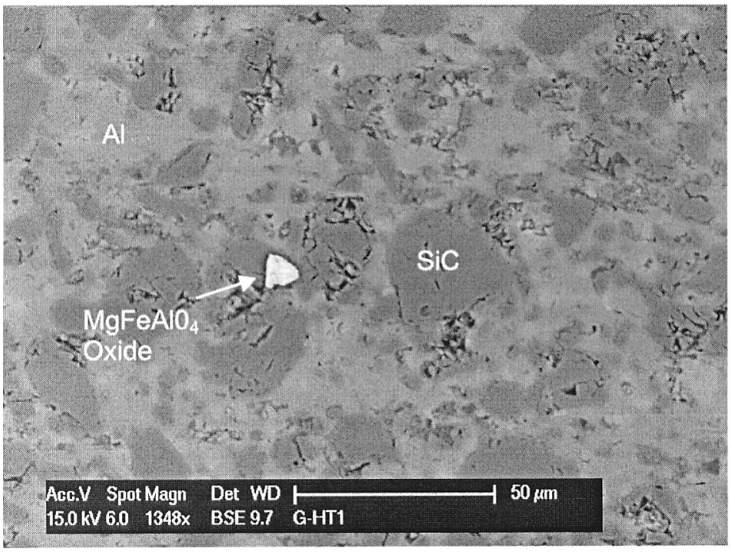

Fig.10b: SEM image of rolled 31% SiC showing phase of $MgFeAl0_4$ formed in the composite.

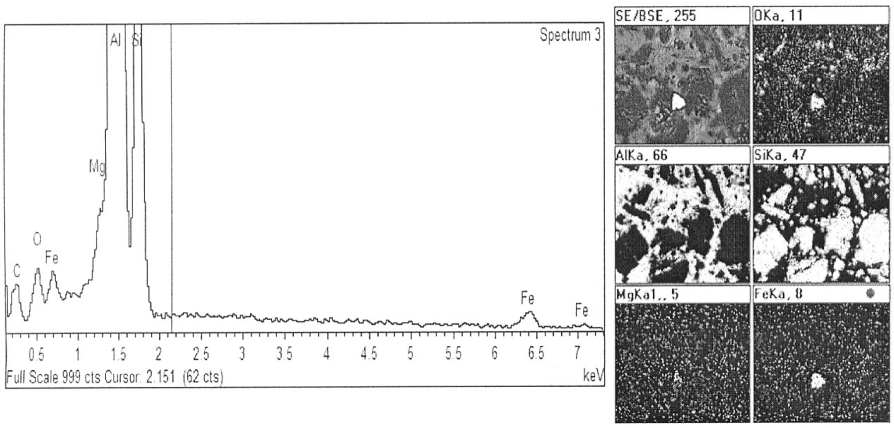

Fig.10c: EDAX-Mapping Techniques used showing Fe, O, Al, Mg, Si elements present.

to ASTM E-384, specifies a range of loads using a diamond indenter to make an indentation, which is measured and converted to a hardness value [11].

Measuring the different phases in the micro-level it is quite challenging, as the SiC reinforcement of ≈17μm in size was not easy to measure, due to small indentation mark left when a small load on the carbide is applied. When introducing higher values of load, the indentation was not localized in the carbide but covered some of the matrix area too. The load was finally set to 50 grams in order to obtain valid measurements coming from different areas of the samples: SiC, aluminium matrix, and the overall composite – MMC i.e. areas superimposing matrix and reinforcement.

There are many factors influencing the microhardness of a composite material, including the reinforcement percentage, interparticle spacing and also particle size. Moreover, manufacturing forming processes influence material's microhardness behaviour in relation to the reinforcement percentages in the composites.

The cast sample in the as received condition has the highest MMC microhardness, where the rolled 20% SiC with lower percentage of reinforcement has the lowest values. By altering the microstructure with modified T6 (HT-1) heat treatment all values of the three samples show an increase between 20-45% from the initial state (Fig.11). This shows the effect of the heat treatment in the micro-deformation of the

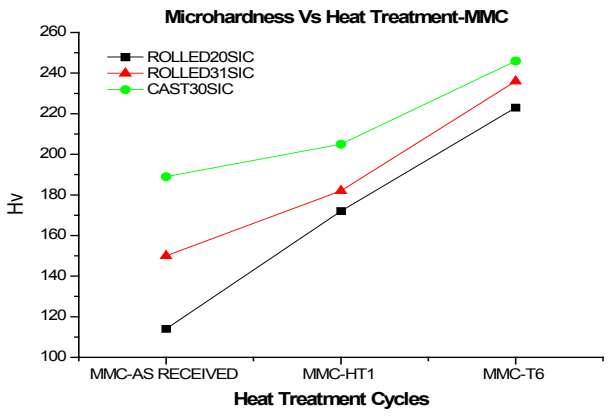

Fig.11: Microhardness values Vs. Heat treatment cycles for the MMC areas.

matrix- reinforcement interface due to the presence of precipitates and other phases and oxide layers.

In the T6 condition it was observed the larger increase in microhardness values from the as received state, ranging from 20% to 90% depending on the reinforcement percentage and manufacturing process. In particular, in the rolled 20% SiC material the increase in microhardness values is in the order of 90%.

Furthermore, variability in microhardness values was observed when comparing cast and rolled materials with different percentage of SiC. However, this variability varied when samples processed at different heat treatment conditions were compared. Highest variability showed samples in the as received condition, whereas lowest variability showed samples in the T6 condition, with samples in the HT-1 condition in between. This can be explained by the fact that precipitates act as strengthening mechanisms and affect the micromechanical behaviour of the composite material.

In the absence of precipitates (in the as received condition), the volume percentage of SiC and the manufacturing processing play a significant role in micromechanical behaviour of the composite. As precipitates are formed due to heat treatment process they assume the main role in the micromechanical behaviour of the material. In the HT-1 heat treatment condition there is presence of β' precipitates which affect the micromechanical behaviour in a lesser degree than in the case of T6 heat treatment condition where fully grown β precipitates are formed. It becomes clear that after a critical stage, which if related to the formation of β precipitates in the composite the dominant strengthening mechanism is precipitation hardening.

While Fig. 11 shows results in areas that include the interface region (where precipitates are concentrated) Fig.12, shows results on microhardness values in the aluminium matrix (where precipitates are dispersed). In Fig. 12 there is similar variability for all three materials processing states, as received, HT-1, and T6. This implies that in the matrix material the

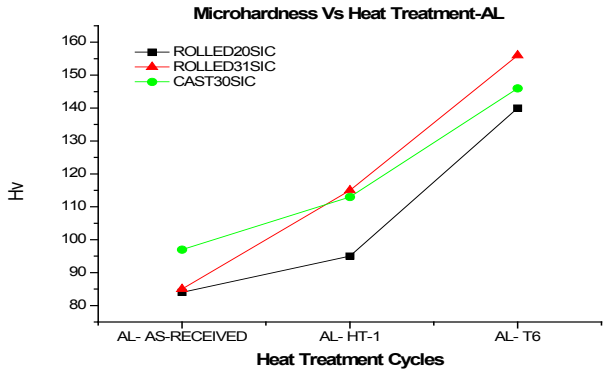

Fig.12: Microhardness Vs. Heat treatment cycles for Aluminium areas.

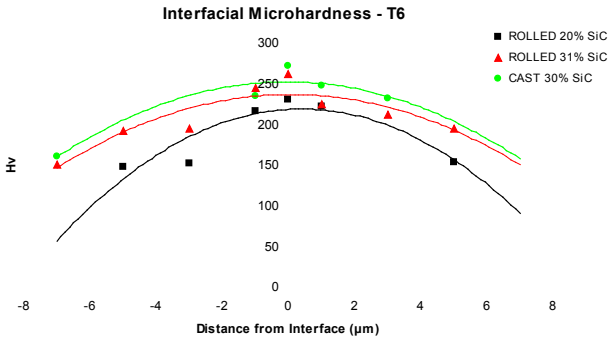

Fig.13: Interfacial microhardness showing measurements obtained from areas close to the matrix- reinforcement interface in the T6 condition.

percentage of the reinforcements, the manufacturing process, as well as the precipitation hardening, are strengthening mechanisms of equal importance.

Fig. 13 shows microhardness measurements obtained from areas around the matrix-reinforcement interface in a composite heat treated in the T6 condition. The microhardness values are higher in the close proximity of the interface. It is observed that cast material has higher values than the rolled material. In the case of rolled material, the microhardness raises as the percentage of reinforcement increases.

6. CONCLUSIONS

The influence of processing conditions in the micromechanical behaviour of Al/SiC composites has been investigated. Two different manufacturing processes (cast and rolled), three reinforcement percentages (20%, 30%, 31%) and three processing states (as received, HT-1, T6 heat treated) have been compared.

The importance of processing conditions in the micro-structural events of segregation and precipitation has been investigated, at the micro/nano level using microhardness measurements and nano-scale phase identification of the matrix-reinforcement interface, and the developments of strengthening mechanisms in the composite have been identified.

HT-1 heat treatment clearly showed an increase in the microhardness, due to β' precipitates as well as other phases and oxides formed in the composite. T6 heat treatment showed the highest microhardness values due to formation of β precipitates, which contribute to strengthening of the interface.

Microhardness testing results showed that the composite's micro-mechanical behaviour is influenced by certain factors. In the absence of precipitates (as received state) or in the case of dispersed precipitates (aluminium matrix) the dominant parameters influencing the micromechanical behaviour of the composite are the reinforcement percentage, the interparticle distance, the mean size of particulates, and the manufacturing processing conditions. However, when precipitates are concentrated in the areas close to the interface (T6 condition), these precipitates contribute to the strengthening of the composite material.

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