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Heat Treatment and Interface Effects on the Mechanical Behavior of SiC-Particle Reinforced Aluminium Matrix Composites

ABSTRACT: The interface plays a vital role in composites. Strengthening behavior of SiC-particle reinforced aluminium matrix composites relies on load transfer behavior across the interface, whereas toughness is influenced by crack deflection at the boundary between matrix and reinforcement and ductility is affected by relaxation of peak stresses near the interface. In general, metal matrix composites often behave asymmetrically in tension and in compression and have higher ultimate tensile strength, yet lower proportional limits, than monolithic alloys. Such behavior of composites lies with the factors governing matrix plasticity, which can be divided into two areas: those affecting the stress rate of the matrix, and those which alter the flow properties of the matrix through changes in microstructure induced by inclusion of the reinforcement. This work focuses on the characterization of the mechanical response of the interface to stresses arising from an applied load in SiC-particle reinforced aluminium matrix composites. The composites have been studied in the as-received (T1) and in the T6 and modified T6 (HT1) conditions. In the nonequilibrium heat treatment processing of the composites, nonequilibrium segregation arises due to imbalances in point defect concentrations set up around interfaces. Mechanical properties, including microhardness and stress-strain behavior, of aluminum matrix composites containing various percentages of SiC particulate reinforcement have been investigated. The elastic modulus, the yield/tensile strengths, and ductility of the composites were controlled primarily by the volume percentage of SiC reinforcement, the temper condition, and the precipitation hardening.

KEYWORDS: metal matrix composites, heat treatment, interfacial strength, precipitation hardening

Introduction

Silicon carbide particulate reinforced aluminium matrix composites are attractive engineering materials designed for a variety of structural applications due to their superior strength, stiffness, low cycle fatigue, and corrosion fatigue behavior, 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 aging behavior of the composite material. Microcompositional 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 [1,2].

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 [3–6].

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 a monolithic metallic material is carried out by alloying and supersaturating to the point that, on suitable heat treatment, the excess alloying additions precipitates out (aging).

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TABLE 1—*The chemical composition of the materials used in this work.*

	Elements (wt %)					
Materials	Si	Mg	Mn	Cu	Fe	Zn
Rolled Al A359-SiC-20p	9.5	0.5	0.1	0.2	0.2	0.1
Rolled Al A359-SiC-31p	9.5	0.5	0.1	0.2	0.2	0.1

Materials

The metal matrix composites studied in this work 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 this investigation, two types of materials were used: (1) Hot Rolled A359/20 % SiC, with an average particle size of 17 ± 1 μm and (2) Hot rolled A359/31 % SiC with an average particle size of 17 ± 1 μm. 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.

The alloys from the Al–Si–Mg system are the most widely used in the foundry industry due to their good castability and high strength-to-weight ratio. Silicon improves the fluidity of aluminium in the molten state and, also, Si-particulates improve the wear resistance of reinforced aluminium alloy [7]. By adding magnesium, an Al–Si alloy becomes age hardenable through the precipitation of Mg_2Si particulates. An additional advantage of Al–Si alloys for casting applications is that silicon expands on solidification and Si is needed to form Mg_2Si. The precipitation sequence is supersaturated solid solution $\rightarrow$ GP zones $\rightarrow \beta'$ $\rightarrow \beta$ (Mg_2Si).

Heat Treatment

The objective here is to select the heat treatment cycle that produces the most favorable precipitate size and distribution pattern. Heat treatment of composites has an additional aspect to consider; the particles introduced into the matrix may alter the alloy's surface characteristics and increase the surface energies [8,9].

There were two different heat treatments used in this study; T6 and modified-T6 (HT-1) [10]. The T6 heat treatment process 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 h at 530 ± 5°C, where all the solute atoms are allowed to dissolve to form a single-phase solid solution then quenched in water. Next, the composites were heated to a temperature of 155°C for 5 h then cooled in air. The second heat treatment process was the modified-T6 (HT-1) heat treatment, where the alloys in the solution treatment were heated to a temperature lower than the T6 heat treatment, at 450 ± 5°C for 1 h, and then quenched in water. Subsequently, the alloys were heated to an intermediate temperature of 170°C for 24 h in the age hardened stage and then cooled in air (Fig. 2).

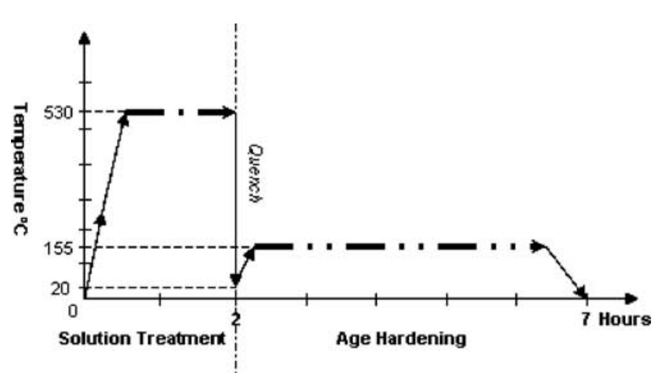


FIG. 1—*T6 heat treatment diagram showing the stages of the solution treatment for 2 h and artificial aging for 5 h.*

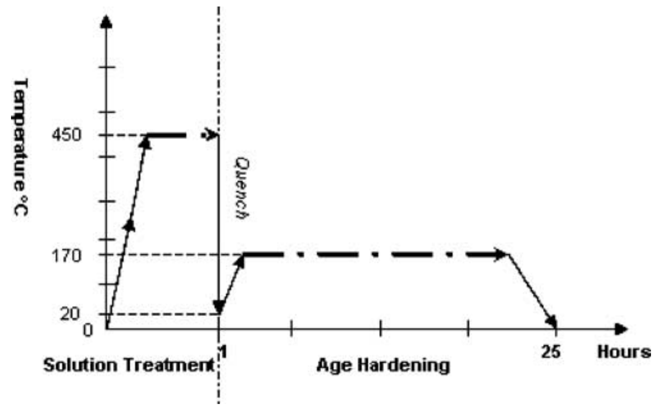


FIG. 2—*Modified T6 (HT-1) showing stages of solution treatment for 1 h and artificial aging for 24 h.*

Microstructural Analysis

The microstructure of the composites was investigated in the as-received and heat-treated conditions, using a Philips XL40 scanning electron microscope with a link 860 EDAX and a Philips FEI Nova Nano scanning electron microscope.

The microstructures of the examined MMCs in the as-received condition (T1) have four distinct microphases clearly marked on the image micrograph, which are as follows: the aluminium matrix, the SiC particles, the eutectic region of aluminium and silicon, and the Mg phase (Fig. 3). Small voids and cracks are also observed in Fig. 3. Total avoidance of voids is difficult because the lower thermal conductivity of ceramic reinforcements requires them to be pushed to the solidifying front of a freezing melt in a way that shrinkage porosities appear around the particulates as the matrix shrinks in solidification. Also, as magnesium is surface active, it effectively reduces interfacial energies only with an optimum amount of reinforcements; otherwise, both gas (due to air layer) and shrinkage porosities will result [11].

Matrix-reinforcement interfaces were identified by using high magnification Nano-SEM microscope. These interfaces attain properties coming from both individual phases of constituents and facilitate the strengthening behavior of the composite material.

In the modified T6 (HT-1) condition of the rolled 31 % SiC, one rod-shape phase (Fig. 4) 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 for the kinetics dissolution of the main alloying elements.

In the rolled 20 % SiC the microstructure of HT-1 heat treatment shows an increase in the silicon phase, as shown in the image (Fig. 5). The HT-1 heat treatment resulted in enlargement and rounding of

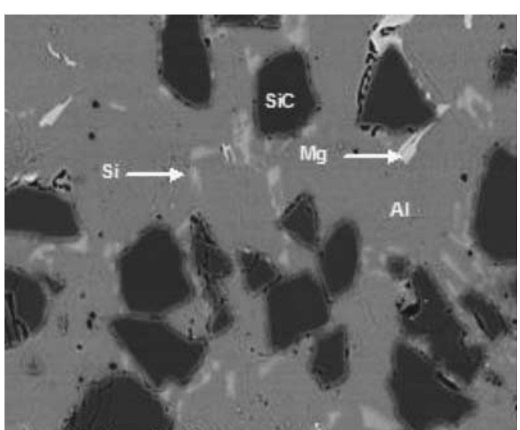


FIG. 3—*Microstructure of rolled 31 % SiC in the as-received condition (T1) showing four distinct phases: aluminium matrix, SiC particles, eutectic region of aluminium, and silicon and Mg phase.*

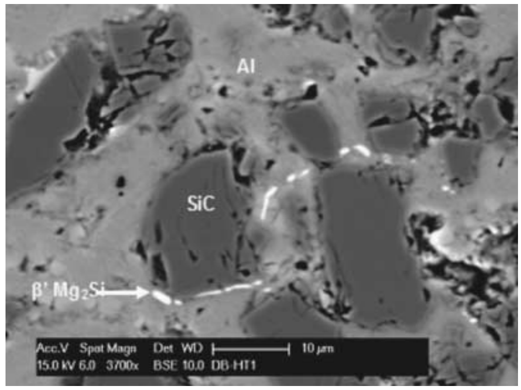


FIG. 4—*Microstructure of rolled 31 % SiC in the HT-1 condition showing rod shape β' phases of Mg_2Si around the matrix and the interface of the reinforcement.*

the Si-eutectic particles. This formed round regions around the whole area of the composite.

Therefore, it becomes evident that the introduction of SiC reinforcement promotes zone kinetics and phase formation reactions during the heat treatment process. The reinforcement, depending on its percentage in the matrix material, accelerates or restrains events such as precipitation and segregation [11–13]. 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 31 % SiC rolled sample.

In the T6 condition the microstructural results showed that in the rolled 20 % and 31 % SiC sample precipitates of Mg_2Si have been formed in a platelet shape in the matrix as well as in areas close to the interface (Figs. 6 and 7). 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 the presence of a crack in the matrix, these precipitates act as strengthening aids promoting crack deflection at the interface resulting in an increase of the composite's fracture toughness [14–19].

Microhardness Testing

The microhardness of the composites has been measured in order to get the resistance of the material to indentation under localized loading conditions and compare them in relation to their microhardness performance, based on the reinforcement percentage and the heat treatment conditions. The microhardness test method, according 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 [20]. The load was set to 50 g in order to obtain valid measurements coming from different areas of the metal matrix composite (MMC), i.e., areas superimposing matrix and reinforcement (interface areas).

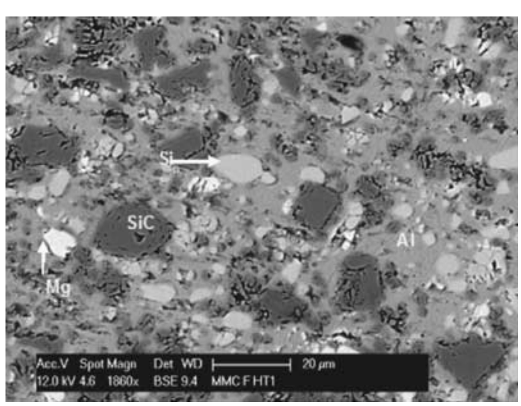
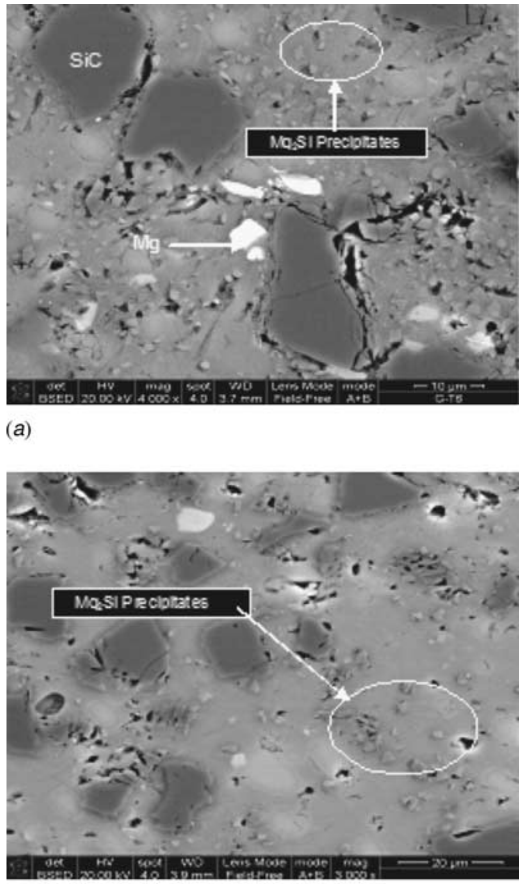


FIG. 5—*Hot rolled 20 % SiC HT-1 sample showing phases of aluminium, SiC, silicon, magnesium.*



(a)

(b)

FIG. 6—*Hot rolled T6 heat treatment showing precipitates formed around the reinforcement. (a) 31 % SiC. (b) 20 % SiC.*

Measuring the different phases in the micro-level was quite challenging as the interface areas close to SiC reinforcement (~17 μm in size) was not easy to measure when a small load was applied. By introducing higher values of load, the indentation was not localized in the interface region but covered some of the matrix area too [21]. The size of the microhardness indentation mark was much smaller than the average spacing between SiC particles. The indentation mark was varying in size depending on the distance from the interface, as well as from the reinforcement percentage. By getting closer to the SiC particulates, the marks became smaller and the microhardness values higher. Furthermore, the 31 % SiC

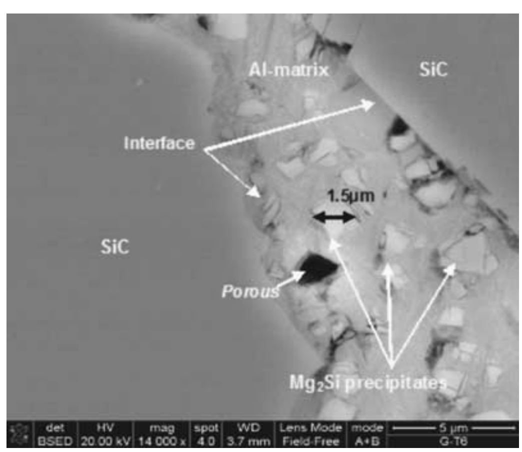


FIG. 7—*Hot rolled 31 % SiC—T6 showing Mg_2Si precipitates formed between the SiC reinforcement interface in a platelet shape of around 1–3 μm.*

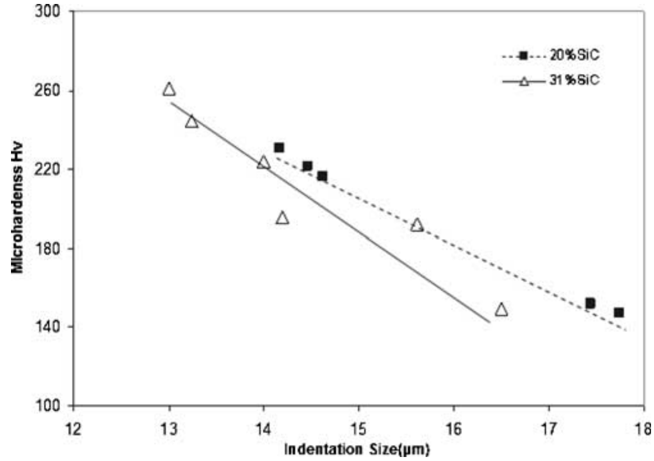


FIG. 8—*Interfacial microhardness values versus microhardness indentation mark size in the T6 condition.*

composite left the smallest indentation marks verifying that, with a higher percentage of SiC, the inter-particle distance becomes smaller and, therefore, the microhardness increases in areas close to the interface (Fig. 8).

The results showed that the rolled 31 % SiC 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 samples show an increase between 20–45 % from the initial state (Fig. 9). As can be observed in Fig. 9, the reinforcement percentage (20 % versus 31 %) has little impact on the microhardness, while the heat treatment process is more important. In the T6 condition a larger increase in microhardness values from the as-received state was observed, ranging from 20 % to 90 %, depending on the reinforcement percentage. In particular, in the rolled 20 % SiC material, the increase in microhardness values is in the order of 90 %. This shows the effect of the heat treatment in the micro-deformation of the matrix-reinforcement interface due to the presence of precipitates and other phases and oxide layers.

In the absence of precipitates in the as-received condition, the volume percentage of SiC plays a significant role in micromechanical behavior of the composite. As precipitates are formed due to heat treatment process, they assume the main role in the micromechanical behavior of the material. In the HT-1 heat treatment condition there is presence of β' precipitates which affect the micromechanical behavior in a lesser degree than in the case of T6 heat treatment condition, where fully-grown β precipitates are

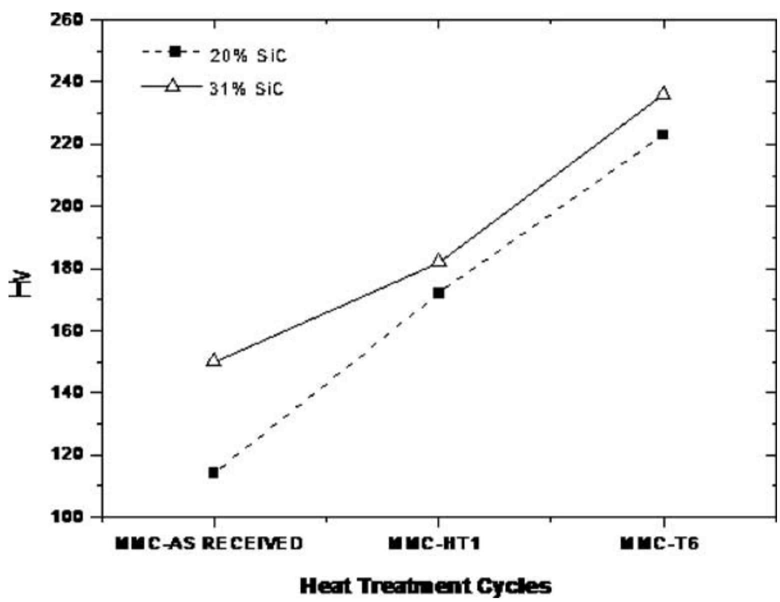


FIG. 9—*Microhardness values versus heat treatment cycles.*

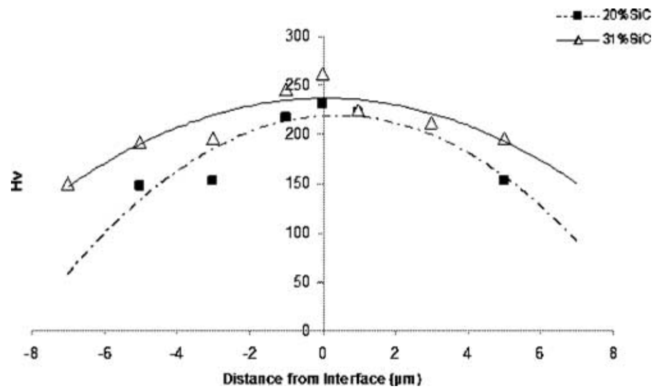


FIG. 10—*Interfacial microhardness showing measurements obtained from areas close to the matrix-reinforcement interface in the T6 condition.*

formed. It becomes clear that after a critical stage, which is related to the formation of β precipitates in the composite, the dominant strengthening mechanism is precipitation hardening.

Figure 10 shows microhardness measurements obtained from areas around the matrix-reinforcement interface in a composite heat treated in the T6 condition. It was found that the microhardness values are higher in the close proximity of the interface. It is further observed that the microhardness rises as the percentage of reinforcement increases. This is expected due to the fact that interparticle spacing decreases in the composite as the volume fraction of particles increases, thereby stresses arising through changes in microstructure induced by inclusion of the reinforcement significantly affects the overall behavior of the composite.

Tensile Testing

Aluminium-SiC particulate composite samples were tested in tension for two different volume fractions, 20 % and 31 %, in reinforcement. The dog-bone coupons were tested according to ASTM E8-04 [22] in the as-received and, following two different heat treatments, modified T6 (HT-1) and T6 heat treatment conditions. Tensile tests were conducted using a 100 KN Instron hydraulic universal testing machine and the strain was monitored using a clip gauge.

The mechanical properties of the composites are presented in Table 2. The engineering stress/strain curves of the composite are shown in Fig. 11. As can be clearly seen in Fig. 11, the HT-1 heat treatment improved both the strength and strain to failure than the untreated composites for both volume fractions. Furthermore, the failure strain for this temper is considerably higher than for the T6 heat treatment; this may be attributed either to the nucleation of the β′ precipitate phases which, although not yet visible, may lead to the increase of the plastic deformation through crack deflection mechanisms and/or to annealing, which acts competitively to the precipitation leading to the toughening of the composite. However, the T6 heat treatment exhibits the highest strength followed by the HT-1 and the as-received state. Finally, as was expected, the "as-received" composites behavior in tension deteriorates with increasing filler concentration.

The experiments showed that, for the same range of conditions tested, the yield and the ultimate tensile strengths of the SiC/Al composites were mainly controlled by the percentage of reinforcement as well as by the intrinsic yield/tensile strengths of the matrix alloys. The addition of the SiC reinforcement created

TABLE 2—*The mechanical properties of Al/SiC composites.*

Material	Condition	$\sigma_{0.2}$ (MPa)	σ_{uts} (MPa)	ε (%)	E	$HV_{0.5}$
Rolled Al A359-	T1	146	157	1.5	100	114
SiC-20p	HT-1	147	190	4	102	172
	T6	326	360	2.1	112	223
Rolled Al A359-	T1	158	168	1	108	150
SiC-31p	HT-1	155	187	2	110	182
	T6	321	336	1.3	116	236

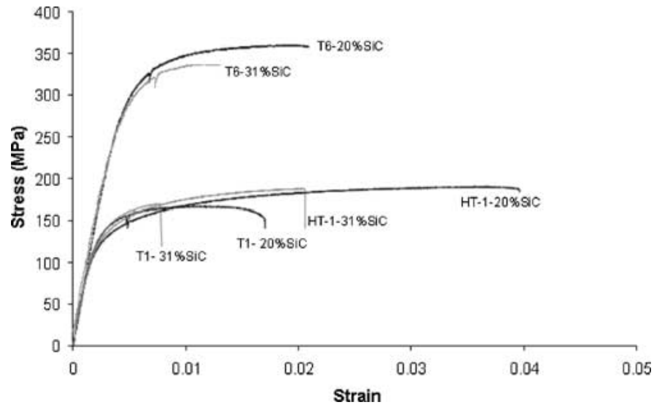


FIG. 11—*Stress/strain curves of Al/SiC composites.*

stress concentrations in the composite and, thus, the aluminium alloy could not achieve its potential strength and ductility due to the induced embrittlement. Composites in the as-received condition failed in a brittle manner with increasing percentage of reinforcement. As a result, with increasing reinforcement content, the failure strain of the composites was reduced, as shown in Fig. 11. From the above postulations it is obvious that the phase that dominates the mechanical behavior of the composite is the precipitation phase created by age hardening while the reinforcement phase plays a secondary role.

The heat treatment affected the modulus of elasticity of the composites by altering the transition into plastic flow (see Table 2 and Fig. 12). Composites in the T6 condition strained elastically and then passed into a normal decreasing-slope plastic flow. Composites tested in the HT-1 condition exhibit a greater amount of strain than the as-received and those heat-treated in the T6 condition. The failure strain increasing from about 1.5 % strain to about 4 %, but the greater influence was a sharper slope of the stress-strain curve at the inception of plastic flow.

This increase in elastic proportional strain limit and the steepening of the stress-strain curve were reflected by the higher yield and ultimate tensile strengths observed in the heat-treated composites. The increase in flow stress of composites with each heat-treatable matrix probably indicated the additive effects of dislocation interaction with both the alloy precipitates and the SiC reinforcement. The combination increased the strain in the matrix by increasing the number of dislocations and requiring higher flow stresses for deformation, resulting in the higher strengths observed. Ductility of SiC/Al composites, as measured by strain to failure, is again a complex interaction of parameters. However, the prime factors affecting these properties are the reinforcement content, heat treatment, and precipitation hardening.

Conclusions

The influence of processing conditions in the micromechanical behavior of Al/SiC composites has been investigated. Two reinforcement percentages (20 % and 31 %) and three processing states (T1, HT-1, and T6) have been compared.

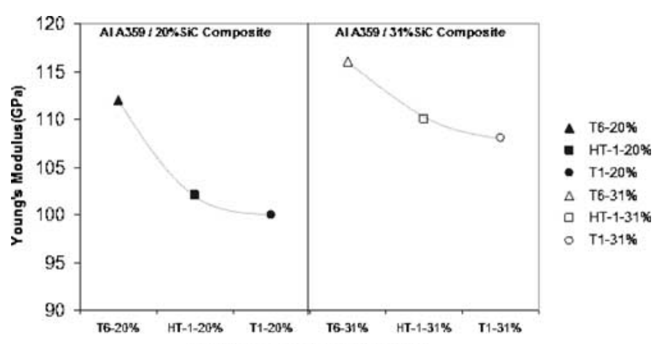


FIG. 12—*Young's modulus versus processing conditions curves showing T6 treated composites having the highest modulus.*

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

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

Microhardness and tensile testing results showed that the composite micro-mechanical behavior is influenced by certain factors. In the absence of precipitates (as received state T1) or in the case of dispersed precipitates (aluminium matrix) the dominant parameters influencing the micromechanical behavior of the composite are the reinforcement percentage, the interparticle distance, and the mean size of particulates. However, when precipitates are concentrated in the areas close to the interface (T6 condition) these precipitates contribute to the strengthening of the composite material.

The results of this work can be summarized as follows:

- Microhardness and tensile testing results showed that the composite micro-mechanical behavior is influenced by certain factors such as precipitates, the reinforcement percentage, and interparticle distance.
- It was observed that the T6 heat-treated composite with 20 % volume fraction of SiC particles had higher strength compared to the 31 % SiC composite. This is expected since the strength of the composite in the T6 condition comes from the formation of the Mg2Si precipitates [Fig. 6(*a*) and 6(*b*) and Table 2]. Less precipitation was formed around the reinforcement phase when the higher volume fraction of SiC particles attract more Si phases close to the interface region, thereby Mg_2Si precipitation formation decreases. Furthermore, Si phase tends to be absorbed by the SiC particles and this causes the matrix embrittlement due to the lack of Si phase remaining in the matrix, thus, eutectic regions are not any more formed and failure to strain decreases in comparison with the 20 % SiC composite, where precipitation is more homogenously distributed around the reinforcement. It should also be pointed out that micro-void formation in the material is promoted as the volume faction of SiC reinforcement increases, which would further contribute to decreasing the overall strength of the composite.
- In the HT-1 heat-treated samples the Si phase showed an enlargement and rounding homogenously around the matrix. This explains the higher strain to failure value than the other two states, T1 and T6.
- As precipitates are formed due to the heat treatment process, they assume the main role of strengthening during micromechanical deformation of reinforced metal matrix composites.
- Finally, the cycle used for optimizing one property, e.g., tensile strength, is usually different from the one required to optimize a different property, e.g., yield strength, so the processing of materials requires careful consideration regarding the particular application.

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"Heat Treatment and Interface Effects on the Mechanical Behaviour of SiC-Particle Reinforced Aluminium Matrix Composites"

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In accordance with criteria set by the ASTM Committee on Publications, the acceptance of papers must be determined by the following:

A = Acceptable as is
O = Optional Revision
M = Mandatory Revision

R = Rejection

Manuscript #JAI101624-07R:

Editor's Comments:

Reviewer Comments:
Editorial Board Member Evaluations:
Editorial Board Member Recommendation: Publish as is Publication Priority: 3

Editorial Board Member (Editoral Board Member Comments to Author):

Thank you for making and explaining the revisions. The paper has been reviewed and is deemed to be acceptable for publication.

Reviewer #1 Evaluations:
Recommendation: Acceptable
1. Is the title descriptive and concise?: N/A 2. Does the paper contribute to the permanent literature or to the immediate interests of the technical community?: N/A 3. Are all descriptions of commercial products, or their use, objective and not presented in an advertising manner?: N/A 4. Is the paper acceptable in literary form and structure, continuity of thought, and logical development?: N/A 5. The Substance of the paper shall not have been published unless the paper is clearly defined as a review paper. Does the paper meet this requirement?: N/A 6. Is the paper concise, yet thorough? If condensation is required, please specify exactly which material is extraneous.: N/A 7. Are the results properly correlated with pertinent work in the field, and has that work been referenced thoroughly?: N/A 8. Are the illustrations readable, relevant, and adequately explained? (An indication of scale is required in all photographs): N/A 9. Are the trend curves properly drawn to conform with the data?: N/A 10. Are the materials tested, the testing conditions, and the test results sufficiently described to permit other investigators to check the procedures and the conclusions?: N/A 11. Are the conclusions reasonable, succinct, and consistent with the data?: N/A 12. Do all references provide sufficient information to enable the reader to obtain copies?: N/A
Rating: N/A

Reviewer #1 (Elaboration on Question 1.):

Electronic form not filled out. See Remarks to Editor below.

Reviewer #2 Evaluations:
Recommendation: Acceptable
1. Is the title descriptive and concise?: A 2. Does the paper contribute to the permanent literature or to the immediate interests of the technical community?: A 3. Are all descriptions of commercial products, or their use, objective and not presented in an advertising manner?: A 4. Is the paper acceptable in literary form and structure, continuity of thought, and logical development?: A 5. The Substance of the paper shall not have been published unless the paper is clearly defined as a review paper. Does the paper meet this requirement?: A 6. Is the paper concise, yet thorough? If condensation is required, please specify exactly which material is extraneous.: A 7. Are the results properly correlated with pertinent work in the field, and has that work been referenced thoroughly?: A 8. Are the illustrations readable, relevant, and adequately explained? (An indication of scale is required in all photographs): A 9. Are the trend curves properly drawn to conform with the data?: A 10. Are the materials tested, the testing conditions, and the test results sufficiently described to permit other investigators to check the procedures and the

conclusions?: A 11. Are the conclusions reasonable, succinct, and consistent with the data?: A 12. Do all references provide sufficient information to enable the reader to obtain copies?: A
Rating: Average

Reviewer #2 (Comments to the Author):

The authors have revised the text/figures in accordance with requests by reviewers. The paper is now clearer and will enable the readers to gauge the contribution in the light of other work in the field.
