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Microstructure and Grain Growth Behavior of an Aluminum Alloy Metal Matrix Composite Processed by Disintegrated Melt Deposition

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In this study, a silicon-carbide particulate (SiC_p), reinforced aluminum alloy-based, metal-matrix composite was synthesized using disintegrated melt deposition. Microstructural characterization of the disintegrated melt deposition processed composite samples revealed the presence of columnar-equiaxed shaped grain structure, noninterconnected porosity associated with the reinforcing carbide particulates, improved interfacial integrity between the reinforcement and the aluminum alloy matrix coupled, and a near uniform distribution of the reinforcing SiC particulates in the alloy matrix. An examination of grain growth with the objective of delineating the effects of the silicon carbide particulates revealed a diminishing to minimal role of the reinforcing phase with an increase in temperature from 450 to 590 °C.

Keywords Aluminum alloy, composites, grain growth, metal matrix composite, microstructure

1. Introduction

The impetus for making available structural materials that are cost-effective, while concurrently offering high performance resulted in continuous attempts in the areas spanning alloy design and use of innovative or novel processing techniques to develop composites or hybrid materials as serious competitors to the traditional engineering alloys. Much emphasis was given to the development of reinforced metallic materials that offered significant improvements in density; stiffness; strength-to-weight ratio, σ/ρ; fatigue strength; wear resistance; structural efficiency; reliability; and overall mechanical performance over the monolithic counterpart. The metal matrix was reinforced with high strength, high modulus, and often brittle second phases either in the form of continuous fibers or discontinuous in the form of whiskers, chopped fibers, and particulates.

The use of aligned continuous fiber-reinforced metallic matrices offers advantages in applications where highly directional properties such as high specific stiffness, E/ρ, in the reinforcement direction are of prime importance (Ref 1-3). Conversely, the discontinuous metal-matrix composites (MMCs) based on discontinuous fibers, whiskers, nodules, or particulate reinforcements are preferred because they offer a number of advantages such as substantially improved modulus and strength properties compared to the unreinforced matrix material. In particular, the particulate reinforced MMCs are attractive because they exhibit more near isotropic properties than the continuously reinforced counterpart (Ref 2-5). Other reported advantages these materials have to offer, compared to the unreinforced matrix material, include:

- Potential for high abrasion resistance (Ref 6)
- Improved fatigue crack propagation resistance (Ref 7-11)
- Increased elevated temperature strength (Ref 2, 5)
- Improved creep rupture properties (Ref 12)
- Excellent microcreep performance (Ref 13)

Overall, the ability to achieve the desired properties is largely dependent on the appropriate selection of the metallic phase and the reinforcing ceramic phase as dictated by the end application (Ref 2, 3,14-16). For performance-critical applications requiring frequent exposure of the composite components to ambient, cryogenic, and even elevated temperatures it is imperative that the microstructure of the composite exhibit a near uniform distribution of the reinforcing ceramic phase in the matrix and improved integrity at the metal-ceramic interfaces coupled with the presence of minimal porosity (Ref 14,18). Enhanced mechanical properties can be realized as a direct result of the distribution of the reinforcing ceramic phase in the metal matrix to ensure a uniform distribution of the applied load across the composite microstructure coupled with a concurrent minimization of stress concentration sites (Ref 19). However, at the higher test temperatures the strength properties can be enhanced depending on the ability of the reinforcing ceramic phase to resist microstructural coarsening, while concurrently serving as a load carrier (Ref 14,17). Recent research studies have provided convincing evidence for an enhancement of ambient temperature strength properties (Ref 2, 21), high temperature strength, and creep resistance of ceramic phase-reinforced metal matrices (Ref 20, 21). However, there exists a paucity of information on understanding the effects of a reinforcing ceramic phase on microstructural coarsening, such as grain growth behavior.

With the objective of rationalizing the appropriate mechanism, discontinuous silicon-carbide particulate (SiC_p) reinforced aluminum alloy MMCs were synthesized using the disintegrated melt deposition (DMD) technique to obtain a composite with the desirable microstructural features required for realizing superior mechanical properties at both ambient and elevated temperatures. The as-processed Al-Cu/SiC_p

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MMC samples were microstructurally characterized to examine (a) morphology of grains, (b) presence and/or absence of porosity, (c) distribution of the reinforcing SiC_p in the aluminum alloy metal matrix, and (d) integrity at the reinforcement-metal interfaces. Particular emphasis was placed on examining the role of the reinforcing SiC_p in inhibiting grain growth in the absence of matrix strengthening precipitates.

2. Material and Experimental Techniques

2.1 *Material and Processing*

The nominal composition of the matrix alloy used in this study (in weight percent) was 2.0% Cu and balance (98.0%) aluminum. Silicon carbide particulates (α-SiC_p) with an average size of 23 μm were selected as the reinforcing phase.

The discontinuously reinforced MMCs were synthesized using DMD. The technique involved the following procedure:

- The elemental materials were thoroughly cleaned prior to melting to eliminate surface impurities.
- The cleaned elemental materials were melted and superheated to 940 °C in a graphite crucible.
- Particulates of SiC (equivalent to 15 wt%), preheated to 900 °C, were then added to the molten metal and continuously stirred using an impeller. The stirring time of the SiC_p in the molten metal was maintained between 10 and 15 minutes. Stirring was carried out to facilitate the incorporation and uniform distribution of the SiC_p in the molten metal. The composite melt was stirred for no longer than 10 minutes, after which the composite slurry was ready for disintegration.
- The composite melt was then disintegrated using argon gas oriented normal to the melt and located 180 mm from the melt pouring point. The argon gas flow rate was maintained at 4.17×10^{-4} m^3/s throughout the disintegration process. The disintegrated composite melt slurry was subsequently deposited on a circular-shaped metallic substrate located at a distance of 250 mm from the gas disintegration point.

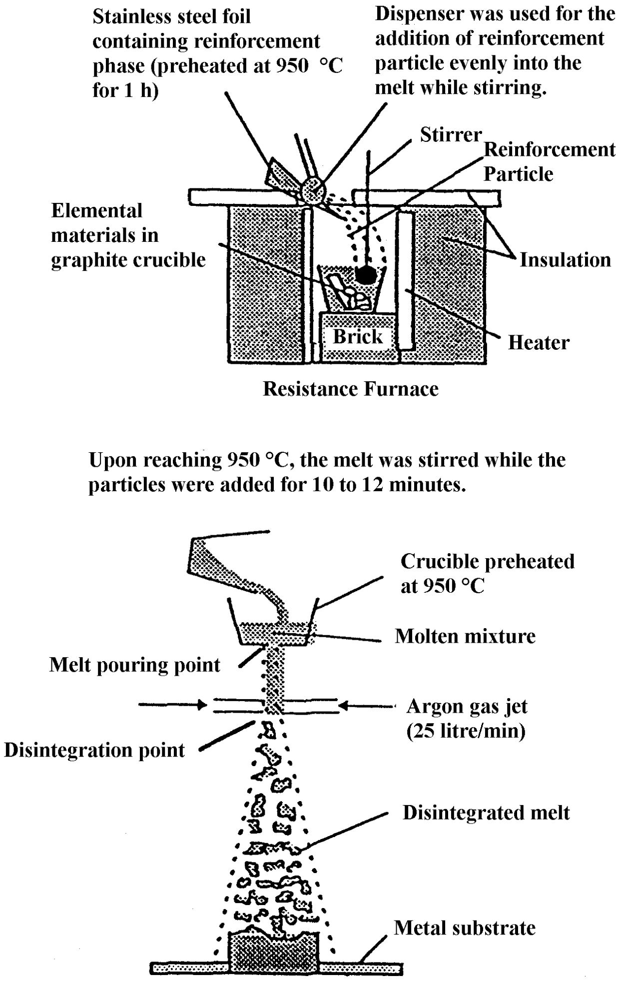


Fig. 1 Schematic representation of the disintegrated melt deposition (DMD) processing technique used for synthesizing the aluminum alloy-based metal-matrix composite

The experiment was conducted under ambient atmospheric conditions (temperature, 27 °C; relative humidity, 55%). It should be noted that unlike the spray atomization and deposition (SAD) technique, the DMD process employs higher superheat temperatures, lower impinging gas jet velocity, and the resulting end product is bulk composite material (Ref 22-24). Figure 1 shows a schematic of the DMD processing technique.

2.2 *Quantitative Assessment of the SiC particulates*

Quantitative assessment of the reinforcing SiC particulates in the as-processed Al-Cu/SiC_p MMC samples was achieved using the chemical dissolution method. This method involved (a) measuring the mass of the composite sample, (b) dissolving the sample in hydrochloric acid, and (c) filtering to separate the reinforcing ceramic particulates. The particulates were then dried and the weight fraction determined. Details of this method can be found in Ref 25.

2.3 *Microstructural Characterization*

Microstructural characterization studies were conducted on the as-processed Al-Cu/SiC_p MMC samples to investigate grain size, porosity, presence and distribution of secondary phases, distribution of the reinforcing SiC_p in the aluminum alloy metal matrix, and interfacial integrity between the reinforcing SiC_p and the soft metal matrix.

Optical microscopy was carried out using an Olympus BH2-UMA (Olympus America, Melville, NY) microscope on the polished and etched Al-Cu/SiC_p composite samples in both the as-processed and heat-treated conditions. Grain boundaries and other coarse microstructural features were revealed using Keller's reagent (a solution mixture of hydrofluoric acid + hydrochloric acid + nitric acid + distilled water). The average grain size was determined from the photomicrographs using the method described in ASTM Standard E 112-84.

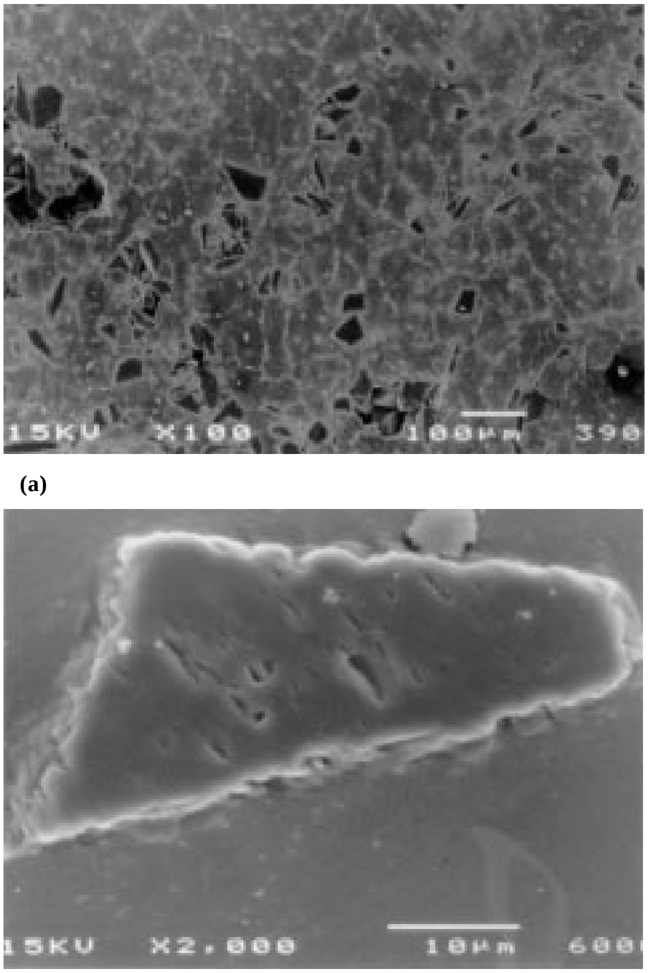
Scanning electron microscopy (SEM) observations were made using a JEOL JSM-T330A (Japan Electro-Optics Laboratory; Peabody, MA) scanning electron microscope equipped with energy dispersive spectroscopy (EDS) on both the etched and unetched samples to investigate the presence and/or absence of porosity and other secondary phases in the microstructure. Porosity was determined using Quantimet image analysis software on digitized images of the scanning electron micrographs.

Grain growth studies in the Al-Cu/SiC$_p$ composite samples (measuring 10 by 10 by 5 mm) were carried out to study the influence of temperature, in the single-phase regime of the phase diagram, and annealing time on grain growth. This was accomplished by heat treating the Al-Cu/SiC$_p$ composite samples at temperatures of 450, 490, 540, and 590 °C for various intervals of time.

3. Results

3.1 Macrostructure

The overall dimensions of the as-processed composite preforms were approximately 35 mm in height and 55 mm in diameter. The composite preforms in the as-processed, machined, and polished condition did not reveal the presence of either macroscopic pores or macrosegregation of the reinforcing SiC$_p$. In addition, results of the mass balance studies provided a processing yield of approximately 85%. Results of the acid dissolution experiments conducted on the DMD processed Al-Cu/SiC$_p$ MMC specimen revealed the weight percentages of the SiC$_p$ in the aluminum alloy metal matrix to be approximately 10.6%.



(a)

(b)

Fig. 2 Representative scanning electron micrographs taken from disintegrated melt deposition (DMD) processed composite samples showing (a) matrix microstructure and (b) interfacial integrity between a SiC particulate and an aluminum-copper matrix

3.2 As-Received Microstructure

Optical microscopy studies of the as-processed Al-Cu/SiC$_p$ samples revealed the presence of both dendritic- and equiaxed-shaped grains in the microstructure of the composite. Grain size measurements provided an average of 52 ± 2.1 µm for the Al-Cu/SiC$_p$ samples in the as-processed condition.

Scanning electron microscopy conducted on polished and etched samples of the composite revealed:

- A combination of dendritic- and equiaxed-shaped grains in the composite microstructure
- A finite amount of micrometer-sized pores
- A near uniform distribution of the reinforcing SiC$_p$ through the composite microstructure
- An interdendritically located copper-rich intermetallic phase coupled with good interfacial integrity (Fig. 2)

The volume fraction of microporosity was found to be 4.6% using the Quantimet image analysis technique. In addition, results of energy dispersive x-ray analysis (EDXA) elemental mapping revealed a preferential segregation of copper in the immediate vicinity of interfaces formed between the aluminum alloy matrix and the reinforcing SiC particulates and at the grain boundaries (Fig. 3).

3.3 Grain Growth Behavior

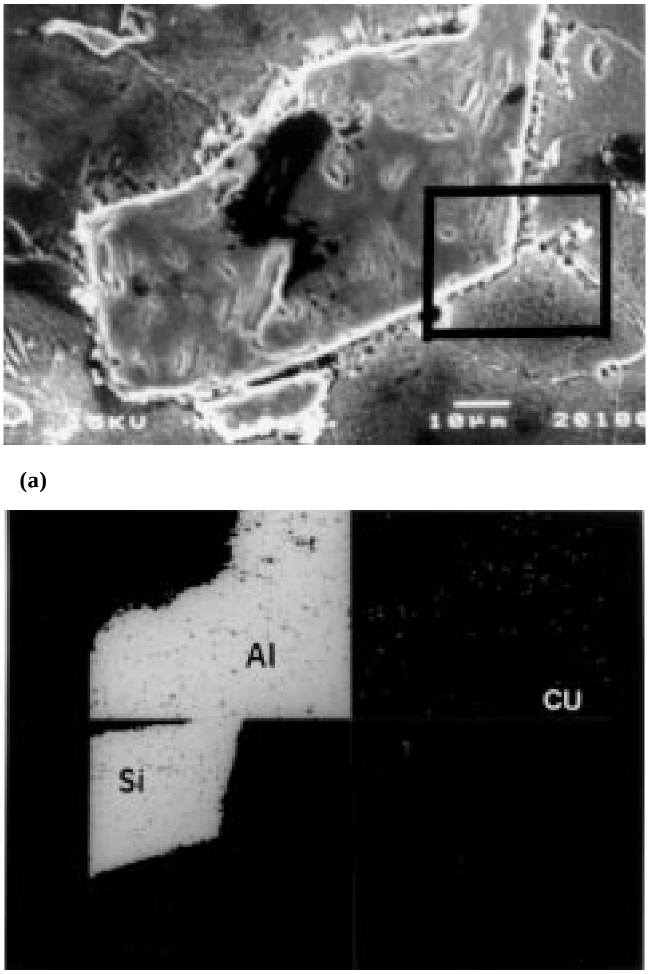
Figure 4 shows the results of grain growth studies carried out on the DMD processed Al-Cu/SiC$_p$ MMC samples. The results reveal a logarithmic progression of grain growth with time at all temperatures investigated and an increase in the rate of grain growth with an increase in test temperature from 450 to 590 °C.

4. Discussion of Results

4.1 As-Received Microstructure

The microstructure of the DMD processed composite samples revealed three salient features: presence of mixed columnar/equiaxed microstructure, presence of porosity, and presence of interdendritic copper-rich phase. The columnar-equiaxed matrix microstructure, commonly referred to as an ingot-type structure (Ref 25), indicates that the remaining liquid temperature subsequent to the onset of solidification from the mold wall remained above the nucleation temperature. The underlying principles responsible for the development of an ingot-type structure are well established and documented elsewhere (Ref 25).

Another important microstructural feature observed in reinforced samples was the presence of porosity. Three types of



(a)

(b)

Fig. 3 Results of scanning electron microscopy/electron dispersion spectroscopy showing (a) Al-Cu/SiC interfacial area and (b) elemental mapping of the area enclosed in the square showing the enrichment of copper in the immediate vicinity of SiC particulates

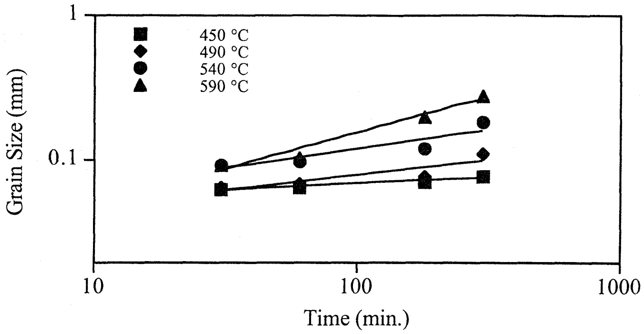


Fig. 4 Graphical representation of the logarithmic grain growth relationship observed in the case of disintegrated melt deposition (DMD) processed Al-Cu/SiC$_p$ samples

porosity were observed in the microstructure: microporosity in the metallic matrix, porosity associated with individual SiC particles, and porosity associated with the SiC$_p$ clusters. The formation and presence of microporosity in the discontinuously reinforced Al-Cu/SiC$_p$ samples, under the experimental conditions used in the present study, was inevitable primarily as a result of the columnar-equiaxed type of solidification structure observed. In analogous studies (Ref 25), it has been convincingly shown that the microporosity associated with materials exhibiting a columnar-equiaxed solidification structure can be attributed to an innate inability of the high viscosity liquid, in the interdendritic regions, to be “sucked” into the groove sufficiently fast to keep pace with the shrinkage that accompanies solidification. The formation and presence of porosity associated with the reinforcing SiC$_p$ can be attributed to an inability of the high viscosity particulate-molten alloy slurry to negotiate sharp corners. The presence of porosity associated with the clusters of SiC$_p$ can be ascribed to an inability of the disintegrated molten Al-Cu/SiC$_p$ composite melt to infiltrate the micrometer-sized crevices in the inefficiently packed SiC particulate clusters formed during solidification. Furthermore, the presence of a finite amount (4.6 vol%) of porosity in the as-processed Al-Cu/SiC$_p$ composite samples is the same order of magnitude as exhibited by the near-net shape synthesizing processes of conventional casting and spray atomization and deposition processing (Ref 23, 24, 26). In related studies, Gupta et al. have reported the presence of 2 to 6 vol% of porosity in discontinuously reinforced aluminum alloy-based metal matrix composites processed by spray deposition (Ref 23, 24)

The presence of an interdendritic/intercellular copper-rich phase observed in the DMD processed Al-Cu/SiC$_p$ composite samples (Fig. 2) is attributed to the conjoint and mutually interactive influences of (Ref 22, 26) (a) a sluggish solidification front velocity achieved during primary processing of the material and (b) rejection of copper ahead of the moving liquid-solid interfaces and subsequent solidification when temperature of the remaining liquid reaches the eutectic temperature (Ref 23, 24).

4.2 Amount and Distribution of the Reinforcing SiC Particulates

In this study, 10.6 wt% SiC$_p$ was successfully incorporated in an Al-2wt%Cu alloy using the DMD processing technique. The successful incorporation of SiC$_p$ in the aluminum alloy metal matrix is attributed to an enhanced wettability of the reinforcing SiC particulates as a direct result of preheating them to 900 °C prior to their addition to the superheated liquid metallic melt (Ref 26-28).

The relatively uniform distribution of the reinforcing SiC$_p$ observed in the as-processed Al-Cu/SiC$_p$ composite samples can be attributed to the conjoint and competitive influences of nonequilibrium conditions during melt disintegration and the dynamic events that occur during deposition as a result of an impingement of the disintegrated melt stream on the deposition surface (Ref 29).

The dynamic events during deposition bear a significant importance in achieving near uniform distribution of the reinforcing SiC particulates because the solidification front velocity, which is governed by the rate of heat extraction, is sluggish enough to engulf the reinforcing SiC$_p$ during solidification even at a gas flow rate of 0.013 kg/s (Ref 30, 31). It can be noted that a lower gas flow rate (7×10^{-4} kg/s) was used to synthesize the Al-Cu/SiC$_p$ MMCs.

4.3 Interfacial Characteristics

Results of microstructural characterization studies conducted on the particulate/matrix interfacial regions exhibited minimal debonding coupled with the presence of fine microscopic voids associated with the reinforcing SiC particulates in the aluminum-copper metal matrix in the as-processed condition (Fig. 2). This microstructural observation is indicative of the excellent fluid flow characteristics of the composite slurry achieved during deposition on a metal substrate. In addition, the results of microstructural characterization studies conducted on the interfacial regions of the SiC particulate/metal-matrix revealed a fairly high concentration of copper (Fig. 3). This phenomenon is ascribed to the presence of an enhanced dislocation density in the interfacial regions. The enhanced dislocation density results from the difference in coefficient of thermal expansion between the constituents of the metal-matrix composite—the reinforcing SiC particulates, and the soft aluminum alloy metal matrix (Ref 32), and facilitates dislocation-assisted diffusion of the alloying elements from the dislocation depleted areas in the matrix. In related studies conducted on conventionally cast and spray deposited aluminum alloy-based metal-matrix composites, Gupta et al. (Ref 23) and Gupta and Surappa (Ref 28) reported a similar enrichment of the primary/key alloying elements at the SiC particulate/matrix interfaces. The present experimental findings provide convincing evidence that the interfacial segregation of the alloying elements is largely governed by the physical properties of the composite constituents—the soft metal matrix, and the hard reinforcing ceramic particulate, and is independent of the processing technique used to synthesize the composite.

4.4 Grain Growth Behavior

The grain growth studies were carried out to specifically delineate the intrinsic influence of the reinforcing SiC particulates on coarsening behavior of the composite microstructure at temperatures selected in the single-phase regime of the phase diagram. The selection of temperatures, in the single phase regime of the phase diagram, was made in order to eliminate the effects of strengthening aluminum-copper precipitates (θ-type) in influencing the rate of grain growth. The kinetic analysis of test data, shown in Fig. 3, was carried out to calculate the grain growth exponent of the DMD processed samples at the various temperatures. The grain growth exponent, *n*, represents the slope of the line when the grain size (in mm) is plotted as a function of time (minutes) on a bilogarithmic plot (Ref 33-35). The empirical relationship, providing a correlation between the grain size, *D*, annealing time, *t*, and the grain growth exponent, *n*, can be expressed as:

$$D = Ct^n \quad \text{(Eq 1)}$$

where *C* and *n* are positive empirical constants. The values of both *C* and *n* depend on both alloy composition and annealing temperature (Table 1). The values of *n* have been reported to range from 0.05 to 0.50 (Ref 36). The results summarized in Table 1 reveal the value of the grain growth exponent *n* to increase with an increase in temperature. This is consistent with the results obtained by other investigators (Ref 36, 37). A limiting value of *n* equal to 0.496 indicates an almost negligible role of the reinforcing SiC particulates in inhibiting grain growth at 590 °C. In related studies (Ref 38), conducted on aluminum-lithium alloys containing a low volume fraction of SiC particulate reinforcements, the value of *n* was found to be in the range of 0.065 to 0.070 for soaking temperatures in the range of 400 to 600 °C. These values were considerably lower in comparison to the values obtained in this study, indicating the diminishing to minimal role of SiC particulate reinforcements in inhibiting grain growth in the Al-2wt%Cu alloy with increasing temperature in the single phase regime of the phase diagram. Furthermore, an increase in the value of *n* with an increase in temperature can be attributed to the occurrence of thermally activated diffusion mechanisms responsible for the grain growth in a material containing deformation free grains (Ref 39).

Table 1 Results of grain growth exponents

Exponent	Temperature, °C 450	490	540	590
C	0.046	0.030	0.035	0.016
n	0.089	0.210	0.266	0.496

An increase in grain size was observed with an increase in annealing time and temperature and is directly attributed to the mutually interactive influences of (a) reduction in overall free energy of the microstructure as a result of reduction in a grain boundary area, (b) reduction in chemical potential that exists between two adjacent grains, and (c) reduction in curvature and the concomitant pressure differences between any two adjacent grains (Ref 39).

5. Conclusions

The key observations that can be inferred from this study are:

- Aluminum-copper, alloy-based metal-matrix composites containing up to 10.6 wt% of reinforcing SiC_p can be successfully synthesized by DMD.
- A low volume percent (4.6%) of porosity observed in the as-processed DMD Al-Cu/SiC_p samples is indicative of the potential of the technique to make near-net shape products.
- Kinetic analyses of the grain growth data revealed the value of grain growth exponent to be between 0.089 and 0.496 for the Al-Cu/SiC_p composites. An increase in value of the grain growth exponent, *m*, with an increase in temperature, in the single phase regime of the phase diagram, provided evidence for the thermally activated diffusion mechanism being responsible for grain growth.
- Grain growth studies conducted on the as-processed Al-Cu/SiC_p composite samples indicate a diminishing effect of the reinforcing SiC particulates in inhibiting grain growth with an increase in temperature in the single phase regime of the phase diagram and a negligible influence of the particulates at a soaking temperature of 590 °C.

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