Microstructural Development in a Metal Matrix Composite During Thermomechanical Processing

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Although a considerable amount of research on particulate reinforced metal matrix composites (MMCs) has concentrated either on their primary processing, or on their failure while in service, little attention has been given to their mechanical and/or physical behavior during secondary processing. Hence, there is only limited information available to facilitate the design of suitable thermomechanical processing schedules for these alloys. However, in order to design an appropriate processing route, it is necessary to develop an understanding of the microstructural development of both the matrix alloy and reinforcing particles during hot deformation. This research investigated the effect of high temperature deformation on the microstructural development of the aluminum alloy 2618 reinforced with 17% SiC particles by volume. Particular attention was directed towards the fracture behavior of the reinforcing particles deformed under conditions of plane strain. Results from extensive quantitative metallography show that SiC particles can be significantly refined during thermomechanical processing. Additionally, at these elevated deformation temperatures, particles redistribute themselves within the matrix, resulting in a more homogeneous distribution. This behavior has been characterized using Dirichlet tessellations. Finally, it is shown how the refinement and redistribution of the SiC particles can lead to the enhancement of the fracture-related properties.

Keywords: Dirichlet tessellations, image analysis, metal matrix composites, microstructural refinement, particulate fracture, plane strain deformation, quantitative metallography, thermomechanical processing

I. Introduction

Particulate reinforced metal matrix composites (MMCs) remain an interesting class of materials for technological development, fulfilling the needs of numerous applications where the properties of current monolithic alloys are not adequate. Two major attractions of MMCs for use in structural applications are their exceptionally good strength-to-weight and stiffness-to-weight ratios. These properties suggest many possible uses for weight sensitive components for aerospace, automotive and naval applications. However, although it is now possible to produce MMCs exhibiting very high tensile strengths⁽¹⁻³⁾, these materials often display low values of fracture toughness and ductility⁽⁴⁻⁶⁾. Hence, it is often these fracture-related properties which have hindered the further development and exploitation of MMCs.

In spite of the poor fracture-related properties, considerable interest in particulate reinforced MMCs remains, from both an industrial and academic perspective. This interest arises because MMC billets can be mechanically processed using the existing technology developed for monolithic alloys. The microstructure which evolves during the thermomechanical processing of MMCs will be dependent on both the matrix alloy and the reinforcing media. Therefore, in order to improve or optimize the mechanical properties (e.g., strength, ductility

and fracture toughness), it is important that an understanding of the microstructural development in MMCs during thermomechanical processing is understood. Any serious perusal of the literature dealing with the development of these materials shows that there are large gaps in our fundamental understanding of the evolution of the matrix microstructure and reinforcement during thermomechanical processing, and of those particular microstructural parameters which can be systematically tailored to enhance the ductility and fracture-related properties.

Low levels of ductility and fracture toughness in discontinuously reinforced composites can often be traced back to those microstructural features such as reinforcement size, shape and their distribution within the matrix. There have been investigations⁽⁶⁻¹³⁾ reporting that the particulate size is instrumental in decreasing the fracture-related properties of composites, either through particle-matrix decohesion or through fracture of the particles. When either of these two events occur, voids will be nucleated at or near the matrix/particle interface, provided that two separate conditions are satisfied. First, the elastic energy released upon nucleation must be greater than the increase in surface energy on forming the void. The second condition is that the local stress at the particle must be greater than the interfacial bond strength⁽¹⁴⁾. Once nucleated, the voids will grow under the applied stress and strain fields until either the plastic constraint between the voids is surmounted, or

when secondary nucleation mechanisms are activated. Finally, void coalescence will occur from the joining of voids along regions of intense shear^(6,15).

Typically, it has been observed that particles greater than 10 μm in diameter are more prone to particle fracture. Other studies^(6,13,16-17) have shown a correlation between composite fracture and particulate aspect ratio (e.g., ratio of particulate length to particulate width). Particles exhibiting high aspect ratios (>4), and aligned in the direction of loading, were observed to encourage void formation leading to premature composite failure. Finally, in addition to particulate size and shape, the distribution of particles in the matrix material has been shown by a number of investigators⁽¹⁸⁻²⁴⁾ to be the major factor responsible for the poor fracture-related properties in composites. In general, it has been shown that the composite fracture is often associated in those microstructural regions where a high concentration of reinforcing particles exist. Within these regions of high particulate density (i.e., particle clustering), initiation and propagation of cracks can readily occur leading to low values of fracture toughness. Moreover, in composites which exhibit a non-uniform distribution of particles, deformation often occurs inhomogeneously. Specifically, the onset of plastic deformation is associated with those microstructural regions which contain a low particle density.

In spite of the existing knowledge that using particles less than 10 μm would be advantageous to achieving more attractive properties in these materials, it is unlikely that primary fabricators will do so because of the health hazards associated with the handling of fine powders. Furthermore, the cost associated with the fabrication of powders finer than 10 μm is high. For example, the cost associated with the powder processing of SiC to an average diameter of 50 μm is \$4.50/kg, compared with the cost of \$10.00/kg for processing SiC to an average diameter of 6 μm ⁽²⁵⁾. Likewise, although most of the primary fabrication processes are subject to stringent quality control inspection, these materials will continue to exhibit regions in the as-cast or consolidated microstructure where: (1) porosity and (2) non-uniform particle distributions will be present. In light of this, it appears that the most cost-effective and substantial improvements made on the final mechanical properties of composites will be realized through secondary processing.

The primary objective of this research was to use a systematic approach directed towards understanding the microstructure-property-processing relationships in particulate reinforced metal matrix composites during thermomechanical processing. Specific attention was given to the reinforcing particles and to the extent to which high temperature deformation could promote their refinement and redistribution within the matrix.

II. Experimental Details

A spray formed monolithic 2618 Al alloy (2.5 wt%Cu-1.1%Fe-1.5%Mg-1.1%Ni-0.2%Si) and a 2618+17% SiC_p MMC were supplied by Cospray (Alcan International Ltd., Banbury, UK). This material was in the form of extruded

rectangular bars having dimensions 63x25 mm. Following extrusion, these bars were solution treated at 530°C for one hour and were water quenched. Both materials were then aged at 190°C to obtain maximum strength. To achieve the peak aged condition, the monolithic alloy was held at temperature for 24 hours. Due to faster precipitation kinetics, the composite reached the peak aged condition after 20 hours. Specimens were subsequently machined from this as-extruded material for hot deformation testing utilizing plane strain indentation. The length and width of the indentation specimens were equivalent at 50 mm and the thickness was 10 mm. At the mid-thickness of each specimen, a hole was drilled for the insertion of a thermocouple, which enabled the temperature to be recorded during deformation. Prior to deformation, the top and bottom surface of each specimen was coated with graphite to minimize any adverse frictional effects at the specimen/indenter interface. The width of the indenter was 10 mm. Specimens were heated to one of five possible temperatures (250, 350, 400, 450 and 500°C) and held for 30 minutes to obtain equilibrium conditions. Following equilibration, specimens were isothermally deformed in a single-stroke to a true strain of 1.5, and at a constant true strain rate of 1 sec⁻¹. Immediately after deformation, specimens were quenched to room temperature.

As-deformed specimens were sectioned, cold mounted, and metallographically prepared for quantitative microscopy. In all cases, the plane of observation was perpendicular to the compression axis, and was along the 50 mm length of each specimen. Quantitative metallography was performed using a Leco 2001 image analyzer.

III. Results and Discussion

The high temperature flow behavior of the composite is shown in Figure 1. At any deformation temperature, the

Figure 1. High temperature flow behavior of MMC, deformed under conditions of plane strain

flow stress rises to a maximum and begins to soften, characteristic of dynamic recrystallization. The onset of dynamic recrystallization, indicated by the strain associated with the peak stress, occurs at a higher level of strain with decreasing deformation temperature. Since deformation was conducted at a constant true strain rate of 1 sec^{-1} , it could equivalently be stated that the onset of dynamic recrystallization is suppressed with decreasing deformation temperature. For example, at 500°C , the peak stress is obtained after 0.17 seconds, whereas at 250°C , the peak stress is obtained after 0.40 seconds. The large difference in flow behavior between 250°C and 350°C is due to deformation being carried out below and above the S' CuMgAl_2 metastable solvus. Below 350°C , lath-shaped $0.2 \text{ }\mu\text{m}$ long S' precipitates are present throughout the matrix, resulting in a higher flow stress.

Prior to deformation, extensive quantitative metallography was performed to characterize the SiC particle diameter, in addition to the particle morphology and distribution

Figure 2. Distribution of SiC_p diameter (a) prior to deformation and (b) after deformation at 250°C

throughout the matrix. Measurements were performed over an approximate area of 1 mm^2 , which resulted in the analysis of at least 2700 particles per sample. Figure 2a shows the distribution of SiC particle diameter for the MMC prior to deformation. The average diameter was $7.8 \text{ }\mu\text{m}$. However, the distribution in particle diameter was large, ranging from $1.7 \text{ }\mu\text{m}$ to $46.6 \text{ }\mu\text{m}$. During deformation, these large particles fractured, which resulted in a refinement of the average SiC particle diameter. This is shown for one particular deformation temperature (250°C) in Figure 2b. In this case, the average diameter was $4.6 \text{ }\mu\text{m}$, and the range in particle size varied between $0.7 \text{ }\mu\text{m}$ and $23 \text{ }\mu\text{m}$. As the initially large SiC particles fractured, the alloy matrix was able to flow between the newly created surfaces, filling any voids that were created during the fracture process. This is shown in Figure 3, depicting the etched microstructure of the MMC deformed at 250°C . This figure clearly shows the flow behavior of the alloy matrix, both between and around fractured SiC particles. Hence, one result of this simple deformation schedule was that SiC particles were refined during high temperature deformation. Moreover, as these particles fractured, the structural integrity of the composite was maintained as a result of the alloy matrix flowing between the fractured particles.

Figure 3. Micrograph of the MMC deformed at 250°C , depicting the flow of the alloy matrix between fractured SiC particles

Deformation at the other four temperatures resulted in similar particle refinement, with no clear trend in the extent of refinement as a function of deformation temperature. This is shown in Table I. It is clear from Table I that for deformations at temperatures $\geq 350^\circ\text{C}$, there is little difference in the average particle size. This can be rationalized by the flow behavior shown in Figure 1. Since the flow curves are similar (and low) at temperatures between 350°C and 500°C , the alloy matrix can easily flow with respect to the particles, resulting in low localized stress fields around the particles. Consequently, at any given level of strain, particles are less susceptible to fracture. However,

as the deformation temperature is lowered to 250°C, the alloy matrix flow behavior with respect to the particles is more difficult, resulting in higher localized stress fields around particles. Hence, at this temperature, particles are more susceptible to fracture, and the average particle diameter is smaller.

Table I. Quantitative metallographic results for SiC particle diameter prior to deformation and after deformation at elevated temperatures

Deformation Condition	Average SiC _p Diam. (μm)	Minimum SiC _p Diameter (μm)	Maximum SiC _p Diameter (μm)
Undeformed	7.8	1.7	46.6
250°C	4.6	0.7	23.0
350°C	5.1	0.8	23.7
400°C	5.1	0.7	25.9
450°C	5.0	0.8	21.3
500°C	5.2	0.5	23.7

Perhaps of greater significance than the particle refinement during deformation is the fact that the particles, once fractured, were able to flow with the alloy matrix, thereby leading to their redistribution within the alloy matrix. Presented in Figure 4 is a frequency distribution of the SiC_p area fraction of the MMC prior to deformation, measured from 400 fields at a magnification of 200X. The distribution appears to be normal, exhibiting a mean area fraction of 16.8%. Note, however, that the range in area fractions is rather large, extending from a minimum value of 10.3% to a maximum value of 29.5%. Hence, the histogram in Figure 4 shows that the distribution of SiC particles in the

Figure 4. Histogram of measured SiC_p area fractions in the MMC prior to deformation

MMC prior to deformation were non-uniform. Those regions which exhibit low area fractions represent particles which are widely separated, whereas those regions which exhibit high area fractions represent particles which are clustered together.

Similar measurements were performed on the specimens after deformation and the results are shown in Table II. As would be expected from performing analysis over a large sample area, the average % area fraction is similar for the undeformed specimen and those specimens deformed at any of the respective deformation temperatures. Although particles fracture, and are subsequently refined during deformation, conservation of mass is maintained. However, noticeable differences can be observed in both the minimum and maximum % area fraction measurements. After deformation at any given temperature, the minimum % area fraction is greater than that measured for the undeformed MMC. Conversely, after deformation at any given temperature, the maximum % area fraction is less than that measured for the undeformed MMC. These data show that as a result of particulate fracture, and their subsequent flow with the alloy matrix, a more homogeneous spatial distribution develops as both the minimum and maximum values converge towards the mean value.

Table II. Quantitative metallographic results for SiC_p area fraction prior to deformation and after deformation at elevated temperatures

Deformation Condition	Average % Area Fraction	Minimum % Area Fraction	Maximum % Area Fraction
Undeformed	16.8	10.3	29.5
250°C	17.9	13.1	21.4
350°C	17.9	14.8	22.1
400°C	19.1	14.1	26.3
450°C	17.6	13.7	22.2
500°C	17.4	12.5	23.1

The influence of thermomechanical processing on the SiC particles is shown in Figure 5. Prior to deformation, the SiC particles are inhomogeneously distributed within the alloy matrix. Visible bands are present, exhibiting regions where particles are clustered together and regions where particles are absent, Figure 5a. These banded regions were aligned with the original extrusion axis. Additionally, it is apparent from Figure 5a that there is a large variation in particle size. In comparison, the particle size after deformation at 250°C is more uniform, Figure 5b. Also, it is clearly obvious that the spatial distribution of particles at this deformation temperature is more homogeneous. Deformation at the other temperatures yielded similar microstructures.

In order to further quantify the spatial distribution of particles before and after deformation, a Dirichlet tessellation technique was used^(19,26,27). This technique involves the division of the microstructure into polygonal cells, where each cell contains a single particle and an area

Figure 5. Microstructure of 2618+17% SiC_p composite: (a) before deformation and (b) after deformation at 250°C

of the adjacent matrix that surrounds the particle. Each tessellated cell can be characterized by an average diameter, in a manner similar to that used to characterize a grain diameter. Particles clustered together would tend to have small cell diameters, whereas particles spaced widely apart would have larger cell diameters. Therefore, in a system containing a random, but uniform dispersion of particles, a unimodal distribution of tessellated cell diameters would result. In contrast, a system containing a non-uniform dispersion of particles could potentially result in a multi-modal distribution of tessellated cell diameters. By comparison, a regular square lattice array of particles having a constant diameter would result in a single and uniform tessellated cell diameter.

The results from Dirichlet tessellations are shown in Table III. This table presents the average of the tessellated cell diameters for the MMC prior to, and after deformation. For comparison, the tessellated cell diameter for a regular square lattice array of particles is included. This regular array of particles was constructed assuming a volume

fraction (17%) of spherical particles having a constant diameter (7 μm). These values were chosen to reflect the average SiC_p diameter and area fraction in the undeformed MMC. At any given deformation temperature, the tessellated cell diameter ranges between 8.4–10.8 μm . This indicates that among the respective deformation temperatures used in this investigation, there was very little difference regarding particulate dispersion. However, deformation at any temperature did result in a substantial change in cell diameter from that cell diameter of the undeformed MMC.

Table III. Characterization of particulate spatial distribution before and after deformation using Dirichlet tessellation analysis

Deformation Condition	Average Tessellated Cell Diameter (μm)
Undeformed	18.2
250°C	8.4
350°C	8.4
400°C	10.8
450°C	8.8
500°C	10.2
Regular Square Lattice Array	11.7

The tessellated cell diameters measured from the as-deformed specimens were significantly smaller than the cell diameter measured from the undeformed specimen. The large average cell diameter from the undeformed specimen is largely due to the banded microstructure shown in Figure 5a, where regions containing relatively few particles were present. As particles were refined and redistributed during deformation, these regions containing few particles vanished, as did those regions where particles were clustered together. This resulted in a more homogeneous distribution of the SiC particles, similar to that exhibited by the square lattice array of particles.

One objective in using thermomechanical processing to achieve particulate refinement and a homogeneous spatial distribution of particles, is to improve the fracture-related properties of MMCs. Room temperature tension tests performed on as-deformed composites revealed that a 2.5-fold increase in ductility was obtained from this simple deformation schedule. The strain to fracture (ϵ_f) for the composite prior to thermomechanical processing was 1.9% and after deformation at 250°C, a ϵ_f of 4.8% was obtained. This increase in ductility was realized while maintaining a similar level of strength. Prior to thermomechanical processing, the MMC exhibited a respective yield and tensile strength of 460 and 532 MPa. After deformation at 250°C, the yield and tensile strength were 425 and 526 MPa, respectively. Deformation at temperatures greater than

250°C exhibited similar, and in some cases greater improvements in ductility. However, these improvements in ductility were at the expense of significantly lower strength properties. Clearly, this demonstrates that thermomechanical processing schedules should be tailored to those conditions conducive for both microstructural refinement of the alloy matrix and the reinforcement.

IV. Conclusions

The following conclusions can be made regarding the microstructural development of MMCs during thermomechanical processing.

1. Significant particulate refinement can be achieved during high temperature deformation. The average SiC_p diameter was refined from 7.8 μm to a low of 4.6 μm . This refinement was due to the fracture of large particles during deformation, and subsequent flow of the alloy matrix between particles.
2. As particles flowed during deformation, they were able to redistribute within the alloy matrix. As a result of this redistribution of particles, a more homogeneous spatial distribution of particles within the alloy matrix was obtained. Quantitative characterization revealed that the spatial distribution of particles after deformation was much more homogeneous than the distribution of particles prior to deformation. Furthermore, the spatial distribution of particles after deformation was similar to that expected based on a regular and uniform dispersion of particles.
3. The combined effect of particle refinement and redistribution led to improvements in the fracture-related properties, as measured from the tensile ductility performed on as-deformed specimens. A 2.5-fold increase in ductility was achieved, at similar levels of strength, as a result of thermomechanical processing.

Acknowledgement

The authors would like to thank Alcan International Ltd. for supplying the materials used in this investigation and the University of Pittsburgh for financial support through a Central Research Development Grant.

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