Effect of Hot Working on Structure and Tribological Properties of Aluminium Reinforced with Aluminium Oxide Particulates

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Al-Al₂O₃ (18%) composite was prepared by stir-cast melt technique. The microstructures showed uniform distribution of particulates, dispersed in the matrix. There exists discontinuity (\sim 0.25 µm) in the interface between particulates and matrix. The composite was hot forged. Hot working resulted in fine recrystallized microstructure with particulates dispersed along grain boundaries. Formation of pancake microstructure with some inhomogeneity in the microstructure along three faces of the forged composite was observed. The discontinuity across the interface between Al-Al₂O₃ was reduced to 0.125 µm after forging. The as-cast and forged Al-Al₂O₃ composites showed higher wear resistance than pure Al. In lubricant media, there was no significant wear observed for either the as-cast or forged composite, whereas Al had shown higher wear at 50 N load.

Keywords composite-metal matrix, forming, interface, wear

1. Introduction

Aluminium metal matrix composite (AMC) is gaining importance in automotive and aerospace industries because of its attractive properties, such as low densities, high specific stiffness, and good abrasion resistance (Ref 1, 2). Several reports have been published addressing the problems associated to their developments, mechanical behavior, microstructure and distribution of particulates (Ref 3-9). Presently, particulatereinforced composites are being produced by three different methods; stir casting, powder metallurgy, and spray deposition techniques. Among these three methods, conventional stir casting technique is currently considered to be easily adaptable and economically viable owing to its high production rate and low processing cost (Ref 9). However, some challenges need to be addressed toward the development of AMCs to intensify their uses in different engineering fields. Some of the questions associated to the development of AMCs are recognized to be (i) microstructural integrity due to different factors including agglomerates in AMCs which include interfaces between particulates of different sizes/shapes and the matrix, (ii) mechanical workability of the composites, (iii) solidification and age-hardening behavior of the composite in the aluminum

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alloy matrix containing different sizes and the amount of particulates, (iv) fracture behavior of the composite, (v) Poor wettability of the particulates in the matrix, and (vi) economics of the process (Ref 3).

Among these factors, we have undertaken the processing of AMC and the influence of hot working on the interface and properties of the composite. In the present study, composite is prepared using Al₂O₃ particulates. Several studies (Ref 10-12) have been made on the plasticity of composites. Stress-strain curves, determined from tension and compression loading, indicate that yielding and flow of the composite take place at a lower applied load in tension than in compression (Ref 13, 14). Matrix plasticity of the composite is controlled by the presence of hard and brittle ceramic particles. The amount of distribution together with varied sizes and shapes of the particulates largely control the deformation process. There also exists a large difference in the thermal coefficient between the matrix and particulates, which becomes the prime factor for creating discontinuities between the matrix and particulates. Some researchers have studied the hot-working process in aluminum alloy (2000/6000 series)-Al₂O₃ composites (Ref 15, 16). Those studies have revealed several metallurgical processes operating during hot deformations. Dynamic recrystallization during hotworking was observed for these systems. Presently, it is thought to study the effect of hot-working on the microstructure of Al-Al₂O₃ (18% volume) composite materials, especially when composites are prepared with particulates of different sizes, shapes, and non-uniform distributions. Effect of hot forging on Al-Al₂O₃ composites is thought to be an important proposition. This article is an effort in this direction. Studies on the tribological behavior of the AMC are an important aspect since these products find wider applications in automobile industries. Properties such as friction and wear resistance in unlubricated or lubricated conditions are of particular importance for the manufacturing of brakes, engine pistons, etc. (Ref 17). The present study will, therefore, report the tribological behavior of Al-Al₂O₃ composites both in cast- and in hot-forged conditions.

2. Experimental

2.1 Composite Preparation

Aluminum of 99.6% purity was used for the preparation of the composite. Al₂O₃ (purity 98.8%) was used as reinforcing particulates. The size range of nearly spherical Al2O3 particulates is 1-8 μ m with an average size of ~5 μ m. The metal ingots, before melting, were properly cleaned to eliminate surface impurities. The melting furnace used was pit-type resistance-heating system with a bottom pouring arrangement (shown in Fig. 1). During the melting, the temperature was controlled at 800 °C. 0.5% Mg was added to the melt to enhance wettability of the Al₂O₃ particulates in liquid Al. The molten metal was stirred with a BN coated stainless steel rotor at a speed of 700 rpm. A vortex was created in the melt because of stirring where preheated (~350 °C) Al₂O₃ was poured centrally into the vortex. The rotor was pushed down slowly by keeping its clearance of 12 mm from the bottom. The rotor was then pushed back slowly to its initial position. The pouring temperature of the liquid was maintained at a lower temperature of \sim 700 °C. The composite melt thus obtained was poured into a steel mould $(100 \times 20 \times 40 \text{ mm}^3)$.

2.2 Mechanical Working

In order to hot forge, the cast samples of $Al-Al_2O_3$ composite were slowly heated in a muffle furnace to a temperature of ~450 °C for 4 h. Prolonged soaking was followed by hot forging with a pneumatic hammer. Deformations were carried out slowly in different stages. In between two stages, intermittent annealing was done for 30 min at 350 °C. The total deformation carried out was 80%.

2.3 Metallography and Hardness

Metallographic samples were cut from the central part of the ingot. Both unetched and etched samples were examined under optical microscope. Quantitative phase analysis was made using the image processor (XJL-17). Hardness tests were performed using a micro-hardness tester (DHV-1000) at 0.01 N load.

The cast as well as forged materials of Al-Al₂O₃ composite was tested for abrasion resistance using a pin-on-disk wear test machine (Ducom, TR-201LE) both in air and in lubricant. The test samples of dimension $40 \times 8 \times 8 \text{ mm}^3$ (shown in Fig. 2) were fabricated from the as-cast as well as from the forged composite samples and made to slide against a low-alloy steel disk (material: 103 Cri-Eng-31HRS60W61, equivalent to AISI 4340) of diameter 215 mm and hardness 62 Rc. Three loads, namely, 10, 30, and 50 N were applied for each test material. Tangential force and hence, the coefficient of friction were measured continuously with an electronic sensor attached to the machine and recorded. Frictional force in N and cumulative wear loss in micrometers were measured from the sensor output as a function of time. The wear test was conducted with a track diameter of 55 mm and a rotational speed of 600 rpm for a period of 30 min. The sliding distance and the speed were maintained at 3100 m and 1.7 m/s, respectively. The contact surfaces of the pins and disks were polished to a roughness of $R_{\rm a} = 0.1 \ \mu {\rm m}$ before wear testing.

3. Results and Discussion

3.1 Microstructure

Figure 3(a) shows the optical micrograph of Al-Al₂O₃ composite in an unetched condition. The microstructure showed fairly uniform distribution of Al₂O₃ particulates in the matrix. The volume fraction of the particles, as measured by an image analyzer, is ~18%. The composite sample reveals comparatively finer grain size than the pure Al sample (microstructure not shown) cooled at identical condition. The finer grain size of the matrix is due to Al₂O₃ particle-stimulated heterogeneous nucleation of the primary phase. The particle's density also determines the size of the primary phase. It is observed (Fig. 3a) that most of the particles are at the grain. During the solidification, the growth of primary phase pushes the particles toward the grain boundary. The clustering of the small particles (Fig. 3a) was also observed. The high surface



Fig. 1 Schematic diagram of pit furnace showing the formation procedure of composite



Fig. 2 Geometry and photograph of $\rm Al/Al_2O_3$ as-cast/forged, and pure Al test samples



Fig. 3 Micrographs of $Al-Al_2O_3$ as-cast composite: (a) optical and (b) SEM

tension due to the large surface area-to-volume ratio at the interface and the small mass of the particles contribute to the agglomeration of the particles and their clustering at the grain boundaries. In order to examine the interface between the particles and the matrix, some areas were magnified and examined under SEM (Fig. 3b). The width of the interface between the particles and the matrix is $\sim 250-280$ nm.

3.2 Hardness

The micro hardness values are found to be 27 ± 2 VPN for aluminum matrix and 35 ± 2 VPN at the region near Al₂O₃particles under 0.01 N load. The hardness value reported is the average of 20 indentations ignoring the high value contributed by the direct encounter of hard Al₂O₃-particles. The composite sample shows higher hardness values than those of their unreinforced counterparts. This is due to the presence of hard particles, which aids to the load-bearing capacity of the material and also restricts the matrix deformation by constraining dislocation movement. During the indentation, Al is plastically deformed, and dislocations accumulate at the interface. This pileup together with discontinuity in the interface region will cause a barrier to the deformation process. The microhardness near the particle/matrix interface is higher than that of interior region of the matrix in all the cases. The higher hardness near the interface is due to the presence of hard Al₂O₃-particles, which restricts the matrix deformation by hindering the dislocation movement. During indentations, a large amount of dislocations are generated around finer particles (Ref 18). Reference 11 reports the modification in the in-situ behaviour of the matrix owing to the presence of the particulates.



Fig. 4 Micrographs of Al-Al₂O₃ composite after 80% hot deformation: (a, b) optical; F1 is perpendicular to the forging direction, F2 is along the forging direction, and F3 is lateral transverse to the forging direction; and (c) SEM

3.3 Hot Working

The as-cast Al-Al₂O₃ composite material has shown good response to forging at a temperature between 400 and 450 °C. Figure 4(a) shows the three-dimensional view of optical micrograph of the hot-forged Al-Al₂O₃ composite sample (80% deformation). The particles are aligned transversely across the sample, perpendicular to the forging direction. The top surface marked F1 is the surface perpendicular to the forging direction. It is evident that recrystallization has occurred because of repeated hot forging. Typical recrystallization-textured grains can be observed in Fig 4(b). Appearance of pancake structure, within which fine aluminum grains nucleated, can easily be observed in the microstructure. It is a typical microstructure usually observed in thermo-mechanically processed metals and alloys. Alumina particles are observed at the boundaries of the grain, thereby restricting the grain growth. The presence of different sizes and shaped ceramic particles, non-homogeneously dispersed in the matrix, creates possibilities for different diffusion-related processes during hot deformation. High-energy interfaces between metal and ceramic particulates are created owing to difference in the thermal coefficients between the two (Ref 19). The high-energy interface accumulates dislocations and point defects that simultaneously diffuse during hot working. During the process, climbing of dislocations become prevalent, creating possibilities for dynamic recrystallization (Ref 16). All the above reasons help us to explain the occurrence of pancake structure as shown in Fig. 4(b). Diffusion mechanism differs appreciably if the sizes of the particulates differ in sizes and shape. For small spherical particles, volume diffusion is more prevalent compared to the pipe diffusion. Thus, the prediction of microstructure in particulate composites becomes a very difficult proposition, especially where the particulates of different sizes exist. This result also suggests that there is inhomogeneity in the microstructure, possibly, due to the presence of different sizes and shapes of the particulates.

The interfacial bonding of Al₂O₃ particles with the matrix is found to be better after forging. Figure 4(c) showed that the particle-matrix interface distance is 100-125 nm after deformation as compared to that in the as-cast composite of \sim 250-280 nm (Fig. 3b). After deformation, the mechanical bonding between particulates and the matrix improved in the forged composite because of transfer of load from the matrix to the particulates. Mechanical keying between the two surfaces can also lead to bonding (Ref 16, 17).

3.4 Wear Properties

In view of wide the application of AMCs in automobile industry where higher abrasion resistance is mandatory, especially in brake system (Ref 20, 21), the abrasion rate was measured for the composite prepared in the present investigation. Figure 5(a)-(c) represents the cumulative wear loss as a function of time for Al and Al-Al₂O₃ composites in both as-cast and forged conditions at three different loads, namely, 10, 30, and 50 N, tested in air without any lubrication. After a transient period, the wear loss was found to increase linearly with increasing time, i.e., with increasing sliding distance. The wear loss increases rapidly with increasing load for pure Al, whereas for composite samples, the increase of wear loss is mild with increasing wear load. The wear loss in case of pure Al sample was distinctly higher than that of the as-cast and forged composites at all loads. The extent of wear with time is quantified in the form of regression equations using best-linearfit technique. The coefficient attached to X denotes wear rate, which is a constant for a particular material under the test condition. Under all the conditions of loading, the wear rate was found to be the highest for aluminum in comparison with the as-cast and the forged Al-Al₂O₃ composites. The magnitude of abrasion rate for the cast composite in comparison to forged composite increased at a faster rate for the higher applied load. The average value of coefficient of friction (μ) of the test samples at a load of 10 N is 0.48, 0.34, and 0.28 for pure Al, as-cast and forged composites, respectively. The data for coefficient of friction confirm the wearing of aluminum, whereas the composite material in either the forged or the as-cast condition shows minimum abrasion. However, it is worthy to mention that a quantitative comparison between the

literature data and the results of the present study is not possible owing to the various conditions, such as test materials, test configurations, nominal contact stress, sliding velocity, etc. Therefore, a qualitative comparison is given here. In the present study, the addition of reinforcing particulates to Al has significantly reduced the μ value. This is in agreement with those of Roy et al. (Ref 22) and Hosking et al. (Ref 23). However, Sato and Mehrabian (Ref 24) had reported an increase in μ value with the addition of reinforcing particulates in Al alloys. The reasons for such observations are not clear. Figure 6 depicts the extent of abrasion rate with different applied loads. Compared with pure Al, the wear rates of the composites are about a factor of 3-4 lower at higher loads (Fig. 6). The reported magnitude of reduction in the wear rate lies in the range of 4-160 at different load ranges (Ref 22-24). The present experiment suggests that there is enhancement in the abrasion rate for the cast composite in comparison with the forged composite because of the modification in the interface region between particulates and the matrix as discussed earlier. The modification of the interface by hot working of the composite helps in improving the bonding between the two, thereby improving the abrasion resistance of the material.



Fig. 5 The variation of cumulative wear loss versus time of sliding of pure Al, as-cast, and forged Al-Al₂O₃ composites under the loads of (a) 10 N, (b) 30 N, and (c) 50 N



Fig. 6 Extent of abrasion rate of pure Al, as-cast, and forged composites with different applied loads



Fig. 7 Wear at 50 N load for different materials in lubricant

The abrasion test was also carried out for the above three materials at 30 and 50 N loads under lubricant condition. For 30 N load, no abrasion was observed till the time of experiment for all the materials. At 50 N load, aluminum had shown significant amount of wear (Fig. 7), whereas the as-cast composite did not show any significant abrasion. However, forged material did not show any abrasion within the stipulated time of experiment.

4. Conclusions

From the present study, the following points can be inferred:

- (i) Al-Al₂O₃ was prepared by stir casting technique. The composite could be hot worked up to 80% by forging in steps. Hot working resulted in better mechanical bonding between particulates and the matrix as compared with that in the as-cast sample. Owing to high-temperature working, the diffusion across the interface owing to the presence of defects, further aided the bonding between the two.
- (ii) The microstructure of hot-worked composite material showed a recrystallized texture with particulates dispersed

in the grain boundaries. Pancake structure was caused by hot working.

(iii) The forged $Al-Al_2O_3$ composite material had shown the highest resistance among the materials tested. This is due to the increase in bond strength between the matrix and the particulate caused by hot working of the forged material. Under lubricating condition, there was no abrasion observed in the case of either the as-cast or forged composite, whereas Al had shown the highest abrasion at 50 N load.

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