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Dynamic mechanical analysis of prestrained Al_2O_3/Al metal-matrix composite

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The effect of prestraining on the elastic modulus, *E*, and damping capacity, tanϕ, of 10 and 20 vol% Al_2O_3 particle-reinforced composites has been investigated as function of temperature using dynamic mechanical analysis. Both elastic modulus and damping capacity were found to increase with volume fraction. At 10 vol % the modulus and damping were relatively insensitive to prestrain. However, at 20 vol % it was observed that the modulus decreased with increasing prestrain while damping increased significantly. These results are discussed in terms of fraction of broken particles, particle size, and differential in thermal expansion between the matrix and Al_2O_3 particulate.

1. Introduction

Ceramic particulate-reinforced aluminium alloy-based metal-matrix composites (MMCs) display substantial increases in strength, stiffness and wear resistance over conventional unreinforced aluminium alloys [1–3]. In addition, they exhibit a reduced coefficient of thermal expansion [4, 5] and increased thermal conductivity [5]. These excellent properties combined with practical pricing, availability and the economics of liquid processing have enabled the production of such diverse components as bicycle frames and tyre stud jackets as well as stimulating interest in large volume automotive applications such as brake rotors, drive shafts, differential housings, etc.

One of the major advantages of particle-reinforced wrought MMCs is that they can be deformed in a manner similar to conventional aluminium alloys at both elevated and ambient temperatures. However, under elevated stresses during cold deformation processing, the mechanical properties of the composite, particularly the modulus, can deteriorate. Recently, Jin and Lloyd [6] and Mochida *et al.* [7], reported that the Young's modulus of SiC/6061 and Al_2O_3/6061 composites are reduced by plastic straining and that the reduction is related to particle damage during the deformation process. The authors also reported that the larger Al_2O_3 particles are the first to fracture during straining.

The main objective of this study was to determine the effect of prestraining on the elastic modulus, *E*, and damping capacity, tan ϕ, of Al_2O_3/Al composites as function of temperature using dynamic mechanical analysis (DMA). In this investigation the testing was performed on composites containing 10 and 20 vol% aluminium oxide particles. The variation in *E* and tan ϕ between 25 and 450 °C for specimens having undergone prestrain ranging from 0 %–14 % are presented. Scanning electron microscope analysis of metallographic sections of strained composite specimens are used to explain the physical and mechanical results obtained.

2. Damping mechanisms

Damping capacity is a measure of a material's ability to dissipate elastic strain energy during mechanical vibration or wave propagation. Elasticity describes a material's behaviour under an applied load in the stress–strain region where the resultant deformation obeys Hooke's law (which states that the resultant strain is proportional to the applied stress). Hooke's law neglects the time effect, that is, the applied load and the resultant deformation are assumed to be perfectly in phase. This is only valid when the loading rate is sufficiently low so that the deformation process may be considered instantaneous and therefore static. In fact, materials respond to an applied load not only by an instantaneous elastic strain, which is independent of time, but also by a strain lagging behind the applied load, which is dependent on time [8].

As a result of the lag induced by the relaxation, the stress–strain curve forms a hysteresis loop when the material undergoes cyclic loading. If the maximum amount of elastic energy that is stored in the sample during the cycle is *W* and the energy dissipated *DW*, the ratio, *DW*/*W*, is called the "specific damping capacity" of the material. This parameter may be regarded as the ratio of the area of the hysteresis loop in the

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$\sigma - \varepsilon$ curve to the maximum energy stored in the sample during the cycle. In fact, for a periodic stress imposed on the anelastic material, the expression for stress, σ, and strain, ε, can be given [8] as

$$\sigma = \sigma_0 \exp(iwt) \tag{1}$$

$$\varepsilon = \varepsilon_0 [i(wt - \phi] \tag{2}$$

where σ_0 and ε_0 are the stress and strain amplitude, respectively, $w = 2\pi f$ is the circular frequency and f is the vibrational frequency, ϕ is the loss angle by which the strain lags behind the stress. In an ideally elastic material, $\phi = 0$ and $\sigma/\varepsilon = E$, the elastic modulus from Hooke's law. However, most materials are anelastic, so ϕ is not zero and the ratio is a complex modulus defined as

$$E^* = \frac{\sigma}{\varepsilon} = \frac{\sigma_0}{\varepsilon_0}(\cos\phi + i\sin\phi) = E' + iE'' \tag{3}$$

where

$$E' = \frac{\sigma_0}{\varepsilon_0}\cos\phi \tag{4}$$

is the storage modulus, and

$$E'' = \frac{\sigma_0}{\varepsilon_0}\sin\phi \tag{5}$$

is the loss modulus; the ratio of the two moduli is

$$\frac{E''}{E'} = \tan\phi \tag{6}$$

where tan ϕ is the damping capacity.

3. Experimental procedures
3.1. Composite materials
The 6061 Al composites reinforced with 10 and 20 vol % aluminium oxide particulate used in this study were produced by the Duralcan™ (Trademark, Alcan Aluminium Ltd, San Diego, USA) molten metal mixing process [9–11] in the form of the cast extrusion billets. The composite billets were hot extruded at 550 °C to 9.5 mm diameter rod with an extrusion ratio of about 40:1. The microstructure of the composites was examined using both optical and scanning electron microscopy. Fig. 1 is a low-magnification micrograph of the longitudinal section of extruded 20 vol % Al_2O_3/6061 composite showing a uniform distribution of particles throughout the aluminium matrix. This uniformity of microstructure is believed to be responsible for the isotropic nature of mechanical properties reported in this study and elsewhere [5].

Tensile samples were machined from the extruded composite rods, then heat treated to the T4 condition (solution treated at 550 °C for 1 h then quenched in water followed by room-temperature ageing for a minimum of 2 days). These specimens were then prestrained to a specific plastic strain, ε_p, at room temperature at a crosshead speed of 2.5 mm min^{-1}. To establish the level of particle cracking with tensile prestrain, optical microscopy was used: 200 fields at × 500 were examined, the broken particles counted and classified by size range using IBAS2 image analyser.

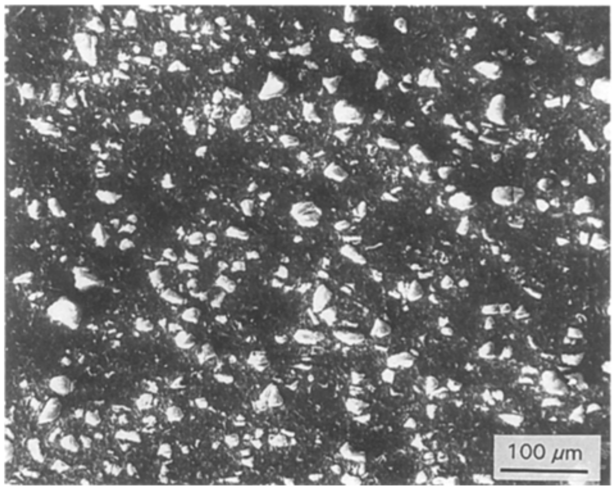

Figure 1 Typical microstructure of 20 vol % Al_2O_3/6061.

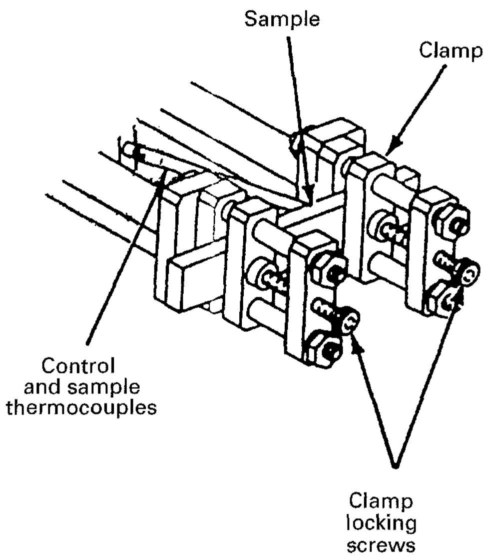

Figure 2 DMA clamping system.

3.2. DMA test
Specimens for DMA testing 50 mm × 8 mm × 1 mm in size, were cut from the prestrained composites using a diamond saw. Specimen surfaces were then polished using 1 μm diamond paste. Four samples of each composite were tested under each condition to verify reproducibility of the data.

The DMA, model 983 from Dupont was used to measure the Young's modulus and damping capacity of the specimens as a function of temperature between 25 and 450 °C. The rate of heating was about 5 °C min^{-1}. The specimens were clamped between two parallel arms (Fig. 2) and then subjected to a uniform sinusoidal displacement of a constant maximum strain $\varepsilon = 2 \times 10^{-4}$. The oscillation frequency was fixed at 1 Hz. The specimen displacement was monitored by an LVDT and the measured lag between the drive signal and the LVDT was the phase angle. The phase angle and drive signal were used to calculate the elastic modulus and damping capacity of the specimen.

4. Results

4.1. Particle distribution

The microscopic observations combined with the results of the image analysis show that the particulates used in the composite containing 20 vol % Al_2O_3 are larger than those used in the 10 vol %. A comparison of cumulative volume of the particles as a function of mean particle size of each of the particulates used is shown in Fig. 3. At 10 vol % the composite contains an aluminium oxide particulate having a mean particle size of 12 μm and 54% of the particles are below 12 μm in size (Fig. 3a); at 20 vol % the mean particle size is 20 μm and 56% of the particles are below 20 μm (Fig. 3b). Figs 4 and 5 show the particle-size distribution of fractured particles at different strains for each of the composites tested. The vertical axis is the ratio of the number of broken particles to the total number of particles having the same mean size. The results

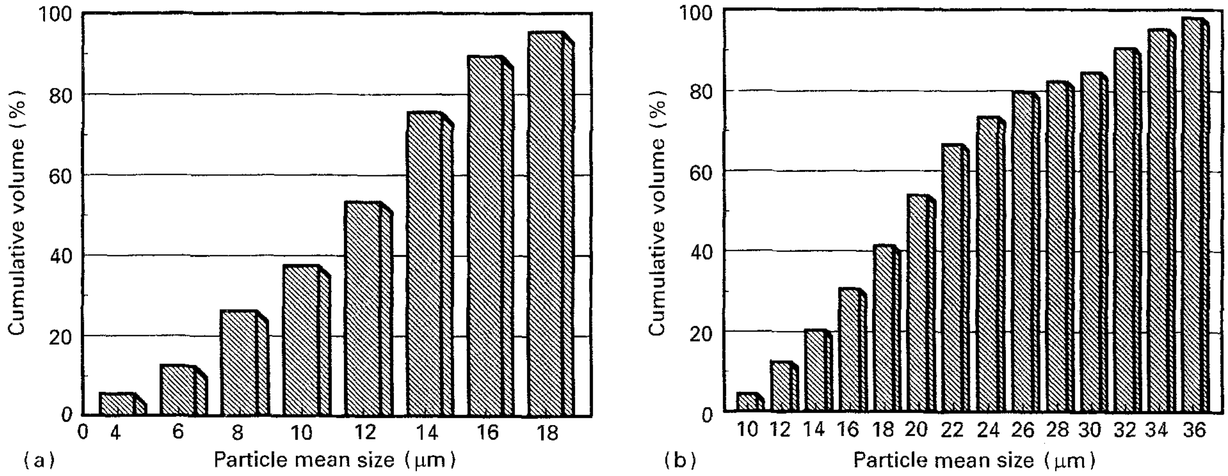

Figure 3 Cummulative volume fraction of particle prior to plastic straining: (a) 10 vol % Al_2O_3, (b) 20 vol % Al_2O_3.

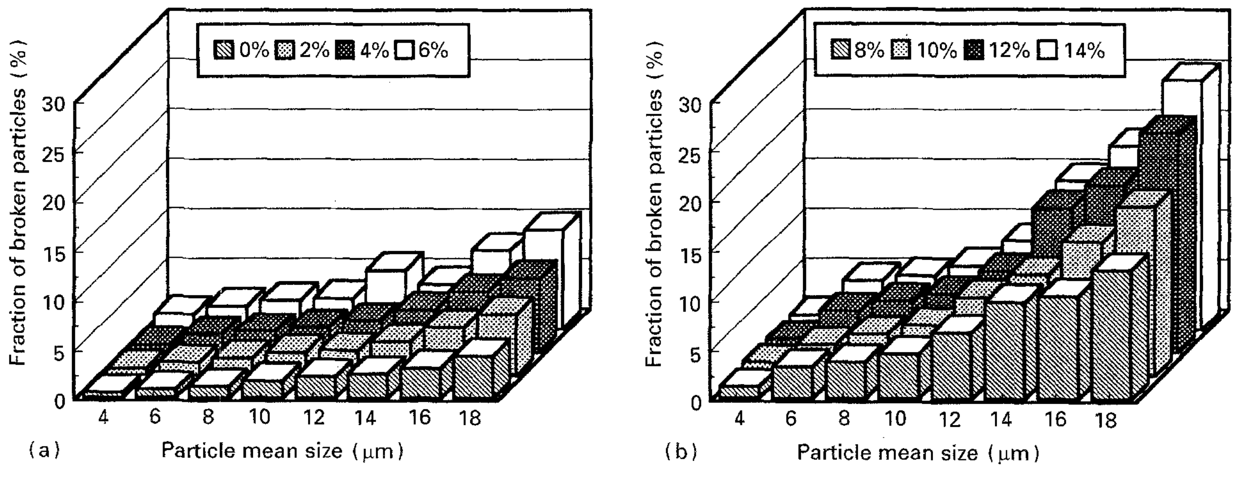

Figure 4 Fraction of broken particles as a function of particle size for various plastic strain of the 10 vol % Al_2O_3/6061 composite: (a) $\varepsilon_p = 0\%$–6%, and (b) $\varepsilon_p = 8\%$–14%.

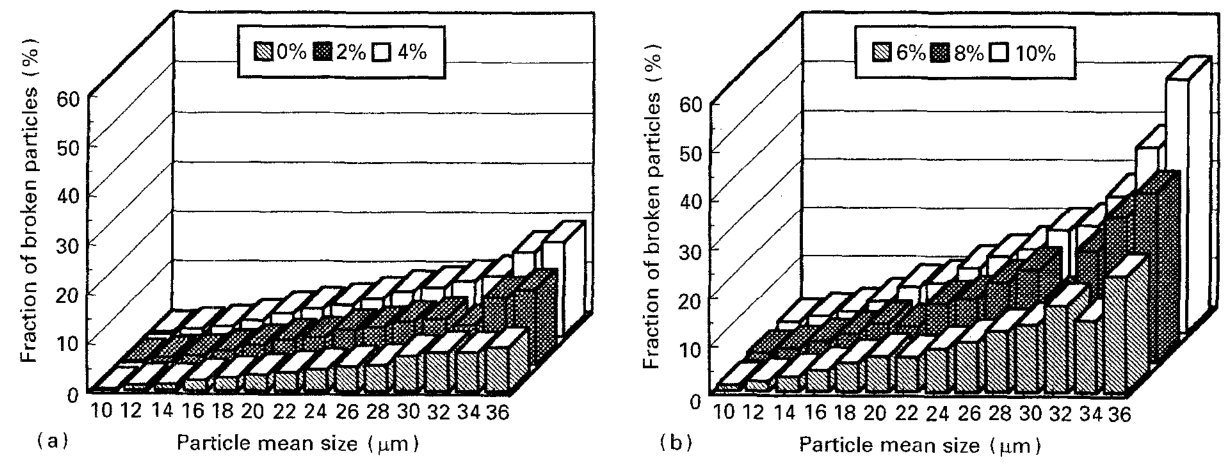

Figure 5 Fraction of broken particles as a function of particle size for various plastic strain of the 20 vol % Al_2O_3/6061 composite: (a) $\varepsilon_p = 0\%$–4%, and (b) $\varepsilon_p = 6\%$–10%.

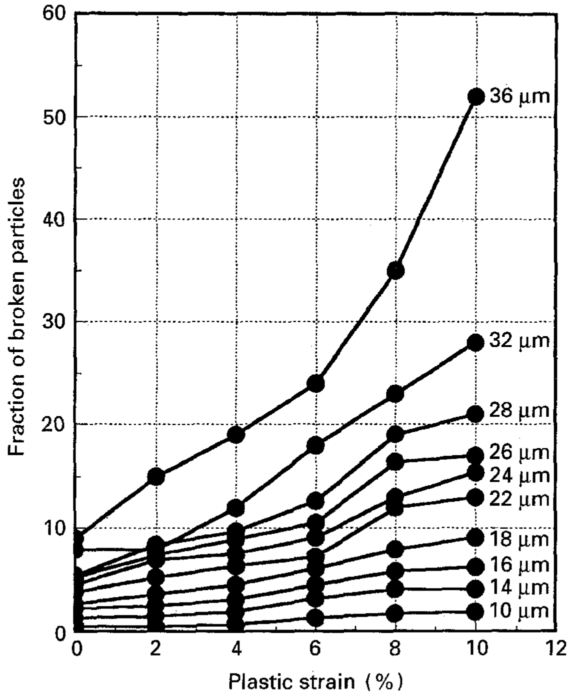

Figure 6 Fraction of broken particles as a function of plastic strain for various particle sizes of the 20 vol % Al_2O_3/6061 composite.

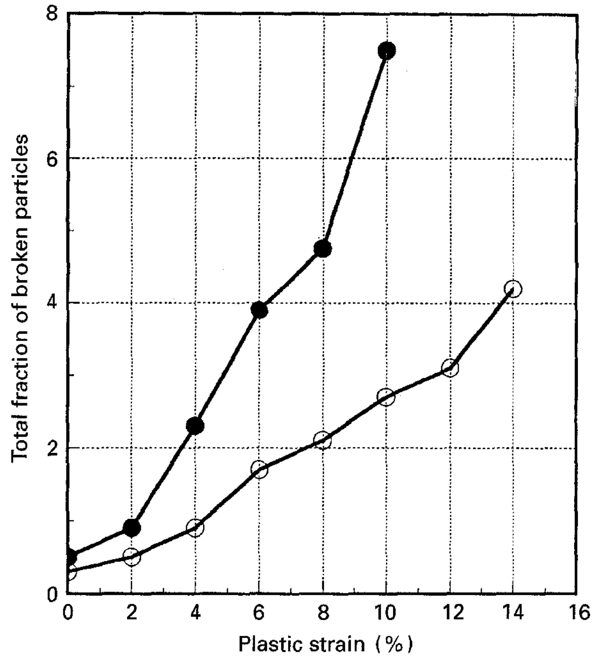

Figure 7 The plastic strain dependence of particle fracture for (○) 10 vol % Al_2O_3 and (●) 20 vol % Al_2O_3.

indicate that with plastic strain, the fraction of broken particles increases with mean particle size. For example, in 20 vol % Al_2O_3/6061 samples prestrained to 10 %, 52 % of the particles having a mean size of 36 μm (the largest sampled) were broken. These data suggest that at a given strain a large particle would have a greater tendency to fracture than a smaller one. This is well supported by Fig. 6. For example, in 20 vol % Al_2O_3/6061 at 6% prestrain the fraction of broken particles is 8% and 25%, respectively, for the 20 and 36 μm particle sizes. The variation in the total fraction of broken particles with strain is shown in Fig. 7. It is apparent that the total fraction of broken particles increases with ε_p for both composites. The particle cracking increases more rapidly with strain at 20 vol % reinforcement, which also has the larger particle size. Micrographs taken of 20 vol % composite prestrained at 10% are shown in Fig. 8. Broken particles are clearly evident in Fig. 8. The fracture of the particle occurs perpendicular to the loading axis, indicating that the particles are broken by tensile stresses induced in the particle. The fractures themselves are devoid of matrix material and could be considered as voids.

4.2. Young's modulus and damping capacity of the composites

A typical DMA run, showing changes in the elastic modulus, E (storage modulus) and damping capacity, tan ϕ, as a function of temperature, is presented in Fig. 9. The room-temperature measurements of elastic modulus at 10 and 20 vol % were 81 and 94 GPa, respectively. These values agreed with moduli measured on similar materials using standard tensile specimens and strain gauges reported in the literature [5]. The agreement tends to support the validity of measurements of E generated through the use of DMA.

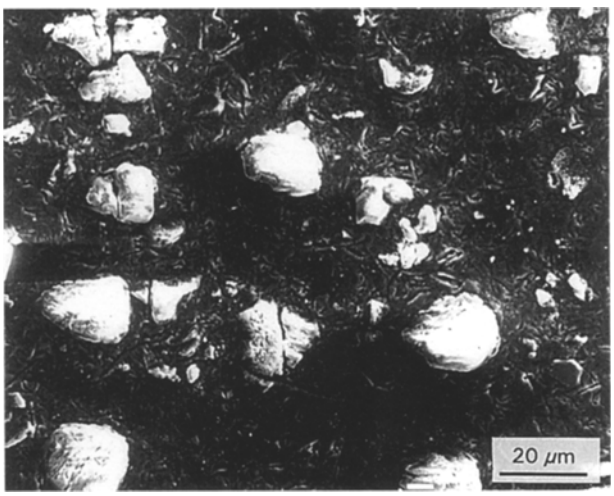

Figure 8 Scanning electron micrograph of prestrained 20 vol % Al_2O_3/6061 composite ($\varepsilon_p = 10$ %) showing broken particles.

The temperature dependence of elastic modulus and damping, over the temperature range 25–450 °C as measured by DMA leads to three interesting observations.

1. The composites show a monotonic decrease in modulus between 25 and 200 °C (Fig. 9a). This is consistent with the decreases in modulus as well as yield strength of the 6061 matrix with temperature which should result in a reduction in the load-transfer effectiveness of the particulate. For example, 20 vol % Al_2O_3 reinforced 6061 without prestrain, $\varepsilon_p = 0$, exhibits a modulus of 94 and 83 GPa at ambient temperature and 200 °C, respectively. Above 200 °C, Young's modulus of this composite decreases more rapidly and reaches 26 GPa at 450 °C.

2. The damping capacity generally exhibits an increase with increasing temperature above 200 °C (Fig. 9b). In

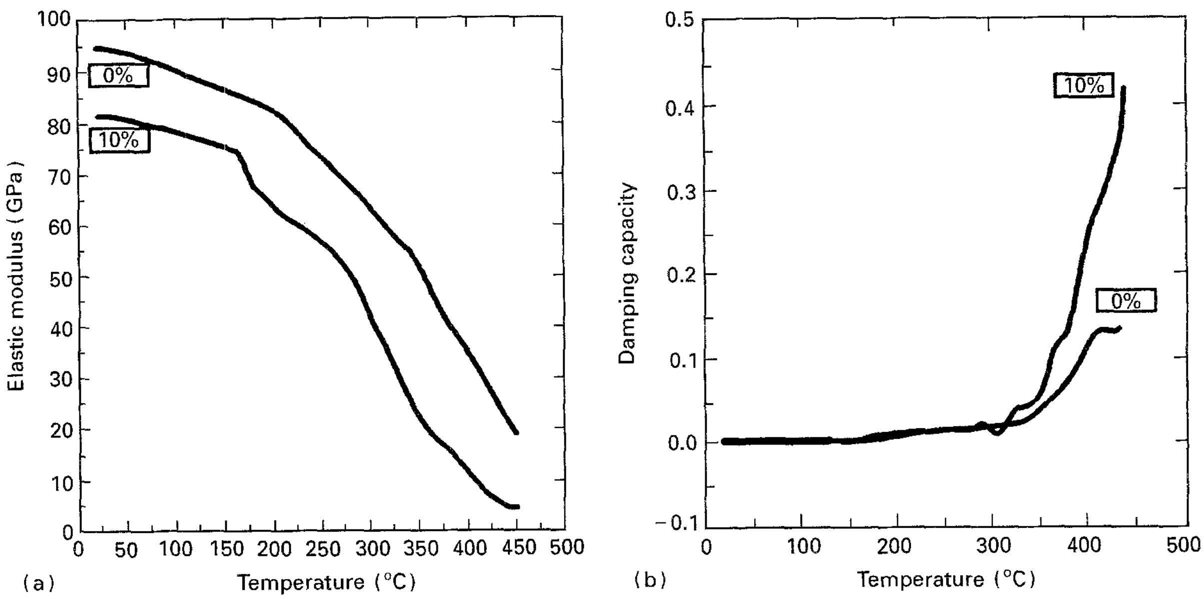

Figure 9 Typical DMA plots for 20 vol % Al_2O_3/6061 composite in the prestrained condition ($\varepsilon_p = 0$ % and 10 %) at 1 Hz and 5 °C min^{-1}: (a) elastic modulus, and (b) damping capacity.

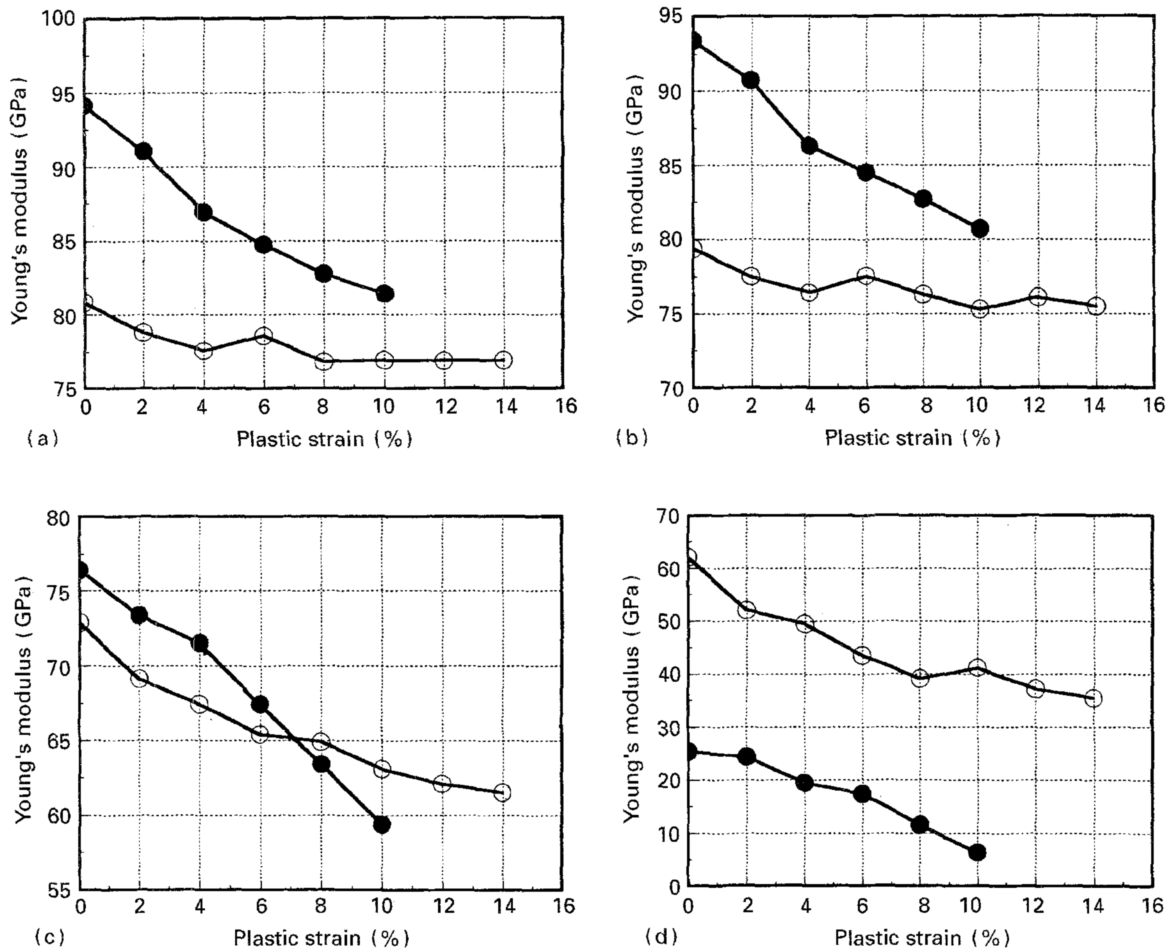

Figure 10 Young's modulus of the (○) 10 and (●) 20 vol % Al_2O_3/6061 composites as a function of plastic strain, at various temperatures: (a) 25 °C, (b) 50 °C, (c) 250 °C, and (d) 450 °C.

addition, the temperature at which a maximum (peak) in damping capacity is observed is approximately 430 °C. This result and prior work [12] lead to the conclusion that the mechanisms responsible for the damping peak are probably related to the transformation processes (growth of precipitates) in the material.

3. Prestrain has a negative influence on the elastic modulus of the 20 vol % Al_2O_3/6061 composite where approximately 8 % of the reinforcing phase is fractured at 10% prestrain. In the same specimen, damping capacity shows a significant increase.

The Young's modulus and damping capacity of the composites prestrained to different plastic strains (0%–14%) are plotted at various temperatures in Figs 10 and 11, respectively. The results indicate that the room-temperature elastic modulus of the 20 vol %

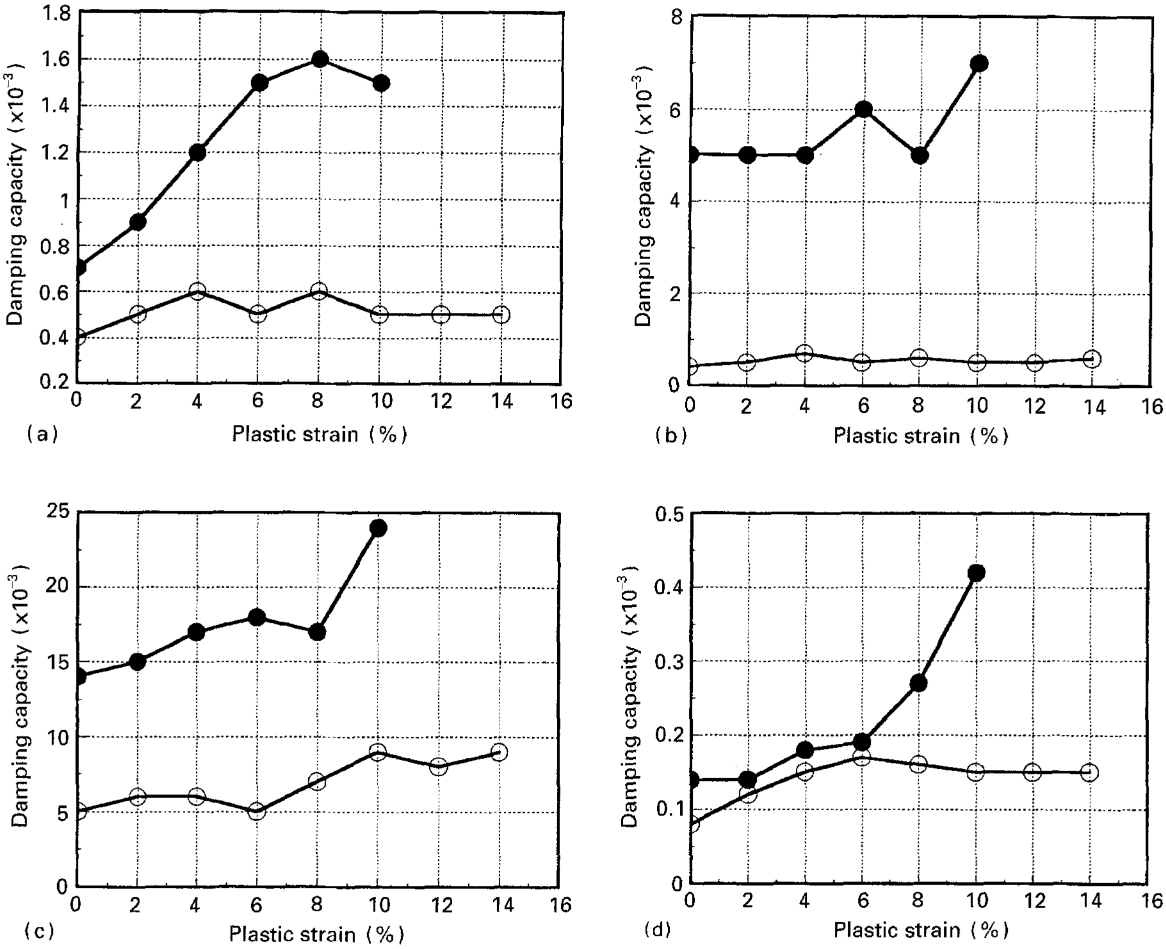

Figure 11 Damping capacity (tan ϕ) of the (○) 10 and (●) 20 vol % Al_2O_3/6061 composites as a function of plastic strain, at various temperatures: (a) 25 °C, (b) 50 °C, (c) 250 °C, and (d) at peak.

Al_2O_3 composite decreases as plastic strain increases ranging from approximately 94 GPa at 0% prestrain to 82 GPa at 10% deformation (Fig. 10a). In contrast, the Young's modulus at low temperatures (25–50 °C) of the composite containing 10 vol % alumina showed a much more gradual decrease with increasing plastic strain (Fig. 10a and b), within the bounds of experimental error (3%). Unexpectedly, above 300 °C, the modulus of specimens containing 10 vol % Al_2O_3 appeared actually to be higher than the modulus measured at 20 vol %. It should be noted that each experimental point on Figs 10 and 11 is the average of four DMA tests. Consequently, it is believed that the observed response is a material characteristic and should not be dismissed as experimental scatter.

As shown in Fig. 11, it is evident that the damping capacity increases with increasing particulate content. With increasing plastic strain, the tan Φ of the 10 vol % Al_2O_3/6061 composite was found to change insignificantly with increasing plastic prestrain. On the other hand, the damping capacity of the composite containing 20 vol % reinforcement increases with plastic strain, ranging for example, at the peak, from 0.15 at 0 % prestrain to 0.42 at 10 % prestrain (Fig. 11d).

5. Discussion

Image analysis results show that the average size of the broken particles is larger than the overall average particle size in the prestrained composite samples. This suggests that larger particles fracture more readily than smaller ones under tensile deformation. The increasing mean size of the broken particles with increasing plastic strain shows that the larger particles tend to fracture in the earlier stages of prestraining. Mochida *et al.* [7] observed the same phenomena in a 15 vol % Al_2O_3/6061 composite under *in situ* tensile straining in a scanning electron microscope. They reported that the larger sized particles are more likely to be fractured for a given applied plastic strain. In addition, Vanstone *et al.* [13] found that fracture of particles and void nucleation in aluminium matrix composites occurred first at the largest particles and progressed to smaller particles as loading increased. It has been observed that the plastic strain necessary to fracture particles increases as particle size decreases, thus the smaller the particle size the better the damage tolerance at a given strength level [14].

By combining the results of image analysis and DMA we can make three important points.

1. In the case of 20 vol % Al_2O_3/6061 composite, which contains larger particles (median size of 20 μm), a significant number of broken particles is noted. As a consequence, this composite displays a substantial decrease in elastic modulus with increasing prestrain. It is likely that the presence of voids at particle fracture sites not only reduces the capacity of the larger aluminium oxide particulate to reinforce the matrix, but also creates a structure of pores randomly distributed

throughout the matrix. This defect structure has a deleterious effect on elastic modulus. Some previous studies of metal-matrix composites [7, 15–18] have also indicated that the generation of thermal residual stresses during quenching from the processing temperature significantly decreases the initial stiffness during subsequent mechanical loading.

2. The 20 vol % composite also showed the highest levels of damping capacity (tan $\phi = 0.42$ at 10 % prestrain). It appears that the presence of broken particles and the associated void-like defects leads to an increase in damping capacity. The effect of these defects on damping is likely to be augmented by the high density of dislocations generated during MMC processing as a result of the thermal mismatch between matrix and ceramic particulate. This is believed [7, 19] to have an important effect on the dissipation of elastic energy.

3. The plastic strain did not have a strong influence on the damping capacity of the 10 vol % Al_2O_3/6061 composite. The composite was reinforced with smaller particles (12 μm mean size) and showed fewer broken particles with prestrain. Therefore, it appears likely that the particle-size range and the amount of broken particles are critical values affecting the damping behaviour of these composites.

The difference in temperature sensitivity of the moduli at 10 and 20 vol % Al_2O_3 and the apparent cross-over at elevated temperatures is difficult to rationalize. However, one may speculate on a mechanism which involves the differences in the number of particles and the resulting interparticle spacing between the two composites. The particles used at 20 vol % are nearly twice the size as those used at 10 vol % (20 μm versus 12 μm) and, if perfectly spherical, would have nearly six times the volume of the smaller particles. Although the 10 vol % composite contains one-half the volume of particles, it will have three times more particles than the composite reinforced at 20 vol % and must, therefore, have a smaller interparticle spacing. At elevated temperatures where the shear strength of the matrix is extremely low, the greater number of particles in closer proximity may be a more effective reinforcement than a higher volume fraction of larger particles. In other words, as the interparticle spacing decreases, the matrix becomes more constrained and the local stress state may be largely triaxial. This effect vanishes at low temperatures where the shear strength of the matrix is many times higher and true particle reinforcement is occurring.

The present study described a strong influence of both particle mean size and plastic strain on elastic modulus and damping capacity of the aluminium oxide-reinforced 6061 composite. This influence becomes considerable when a critical amount of broken particles exists in the composite. There is a synergetic effect of particle size and plastic strain in that the larger particles are more susceptible to fracture during straining. This effect could also be considered as a result of difference in thermal and plastic properties of the matrix and aluminium oxide particulate.

6. Conclusions

1. The results of image analysis of prestrained 10 and 20 vol % Al_2O_3 reinforced 6061 T4 composites showed that the fraction of broken particles increased with prestrain. In the higher volume fraction composite, which was reinforced with larger alumina particulate (20 μm), particle cracking increased more rapidly with prestrain and there was a significantly larger total number of broken particles.

2. As a result of the larger propensity for particle fracture with prestraining, the composite reinforced with 20 vol % Al_2O_3 showed the greatest decrease in modulus with increasing plastic strain. The modulus measured at ambient temperature dropped from 94 GPa at 0% plastic strain to 82 GPa at 10 %.

3. The 20 vol % composite which showed the greatest particle damage with prestrain also had the highest damping capacity, tan $\phi = 0.42$ at 10 % prestrain. Therefore, the damping capacity measured with the DMA can be used as a sensitive indicator of microstructural damage in MMCs.

4. The plastic strain did not have a significant influence on the damping capacity of the 10 vol % Al_2O_3/6061 composite, which was reinforced with a smaller particulate (12 μm). It appears that particle-size range and the number of broken particles have a strong effect on the damping behaviour of particle-reinforced composites.

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References

1. J. W. LUSTER, M. THUMANN and R. BAUMANN, *Mater. Sci. Technol.* **9** (1993) 853.
2. T. J. REINHART and H. F. HALBERT, "Engineered materials handbook", Vol. 1 (ASM International, Metals Park, OH, 1987).
3. A. MORTENSEN, in "Proceedings of the Conference on Fabrication of Particulates Reinforced Metal Composites", Montreal, Canada, September 1990, edited by J. Masounave and F. G. Hamel (ASM International, Metals Park, OH, 1990). p. 235.
4. M. S. ZEDALIS, P. S. GILMAN and S. K. DAS, in "High performance composites for the 1990'S", edited by S. K. Das, C. P. Ballard and F. Marikar (TMS, Warrendale, PA, 1990) p. 61.
5. "Duralcan composites-Mechanical and physical property, foundry composites, SI Units" (Duralcan^{USA}, San Diego, CA, 1992).
6. I. JIN and D. J. LLOYD, in "Proceedings of the Conference on Fabrication of Particulates Reinforced Metal Composites", Montreal, Canada, September 1990, edited by J. Masounave and F. G. Hamel (ASM International, Metals Park, OH, 1990) p. 47.
7. T. MOCHIDA, M. TAYA, and D. J. LLOYD, *Mater. Trans.* **32** (1991) 931.
8. J. ZHANG, R. J. PEREZ and E. J. LAVERNIA, *J. Mater. Sci.* **28** (1993) 835.
9. M. D. SKIBO and D. M. SCHUSTER, US. Pat. 4759 995, 26 July, 1988.
10. *Idem* US Pat. 4786 467, 22 November, 1988.
11. D. M. SCHUSTER, M. D. SKIBO and W. R. HOOVER,

Light Metal Age February (1989) 15.
12. S. P. RAWAL, J. H. ARMSTRONG and M. S. MISRA, in "M^3D Mechanisms of Material Damping", edited by V. Kinra and A. Wolfenden (American Society for Testing and Materials, Philadelphia, PA, 1992) p. 158.
13. R. H. VANSTONE, R. H. MERCHANT and J. R. LOW, ASTM STP 556 (American Society for Testing and Materials, Philadelphia, PA, 1974) p. 93.
14. M. J. HADIANFARD, J. HEALY and W. MAI, *J. Mater. Sci.* **29** (1994) 2321.
15. Y. L. SHEN, A. NEEDLEMAN, and S. SURESH, *Metall. Trans.* **25A** (1994) 839.
16. Y. L. SHEN, M. FINOT, A. NEEDLEMAN and S. SURESH, *Acta Metall. Mater.* **24** (1994) 77.
17. T. NAKAMURA and S. SURESH, *ibid.* **41** (1993) 1665.
18. D. J. LLOYD, *Int. Mater. Rev.* **39** (1994) 1.
19. J. ZHANG, R. J. PEREZ, M. GUPTA and E. J. LAVERNIA, *Scripta Metall. Mater.* **28** (1993) 91.

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