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Yield stress of a copper single crystal containing B_2O_3 particles

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The yield stress of a copper single crystal containing vitreous B_2O_3 particles was measured as a function of temperature (77–1073 K) and strain rate (5.6×10^{-6}–5.6×10^{-4} sec^{-1}). Although the B_2O_3 particles in the copper matrix are a plastically non-deformable solid at low temperatures, they became liquid-like at high temperatures, above approximately 550 K. The yield stress of the Cu–B_2O_3 alloy at low temperatures was explained by the Orowan mechanism and the modulus-corrected yield stress of the Cu–B_2O_3 alloy at 1073 K was about four-fifths of the values at low temperatures. It was found that the liquid B_2O_3 particles could be the effective hardening centres even at high temperatures. At 873 and 1073 K, the yield stresses of the Cu–B_2O_3 alloy varied with the logarithm of the strain rates.

1. Introduction

Low-temperature yielding behaviour of dispersion-hardened materials containing hard (plastically non-deformable) inclusions is well explained by the Orowan mechanism. On the other hand, at high temperatures where various diffusion processes become operative, several different hardening mechanisms have been proposed [1–6]. To clarify the hardening mechanisms, basic studies are necessary as to investigate the yield stresses in materials containing inclusions at various temperatures and strain rates and their relation to the character of inclusions. In the experiments, single crystalline materials should be used to avoid possible grain-boundary sliding and other grain-boundary related phenomena. However, as far as the authors know, previous experimental studies were performed only for alloys with hard dispersed particles [7–9].

In the present paper, we will report experimental results on the temperature and strain rate dependence of the yield stress of a copper single crystal containing vitreous B_2O_3 particles. The Cu–B_2O_3 alloy system has been selected since the B_2O_3 particles have a rather unique character: although these particles in the copper matrix are a hard solid at low temperatures, they become soft and liquid like at high temperatures over approximately 550 K.

2. Experimental procedure

2.1. Specimen preparation

A plate-like single crystal (1.2 mm × 20 mm × 150 mm) of a Cu–0.031 wt % B solid solution alloy was grown by the Bridgman method in vacuum. In order to avoid the chemical reaction of boron with a mould, a split mould made of boron nitride was used. To obtain dispersion of B_2O_3 particles, the Cu–B single crystal was internally oxidized with a Cu–Cu_2O–Al_2O_3 (weight ratio 1 : 1 : 2) mixed powder at 1173 K for 36 h. For comparison, a Cu–0.080 wt % Si single crystal was also grown by the Bridgman method and was internally oxidized at 1223 K for 24 h to obtain dispersed SiO_2 particles.

After the internal oxidation, the thickness of these single crystals was reduced to 0.7 mm by chemical and mechanical polishing. Tensile specimens with a gauge length of 30 mm and a cross-sectional area of 3.0 mm × 0.7 mm were spark-cut from these single crystals. The tensile axis was chosen close to the [2 1 6] direction to encourage deformation by single slip. After electrolytic polishing, these tensile specimens were annealed at 1173 K for 24 h before the tensile tests.

By preparing thin foils and carbon-extraction replicas from as-annealed specimens, oxide particles were observed on a transmission electron microscope and a scanning electron microscope. The B_2O_3 and SiO_2 particles were found to be spherical in shape and the absence of discrete reflection spots in diffraction patterns indicated that they were amorphous.

The volume fraction of the particles, f, and the arithmetic mean of the particle diameter, d, for each alloy are tabulated in Table I. The value of f was calculated on the assumption that all solute atoms had been oxidized. As shown in Table I, since the Cu–B_2O_3 alloy has the lower volume fraction and larger diameter particles than the Cu–SiO_2 alloy, the mean spacing between the particles in the Cu–B_2O_3 alloy is longer than that in Cu–SiO_2 alloy.

2.2. Tensile tests

Specimens were tensile tested at various temperatures

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TABLE I The volume fraction of the particles, f, and the arithmetic mean of particle diameter, d, for Cu–B_2O_3 and Cu–SiO_2 alloys used in the present study

Alloy	f	d (nm)
Cu–B_2O_3	5.5×10^{-3}	104
Cu–SiO_2	6.6×10^{-3}	75

from 77 to 1073 K using an Instron-type testing machine. Test temperatures were controlled within ± 1.5 K and an argon atmosphere was employed for the tests above room temperature. The temperature dependence of the yield stress with the shear strain rate of 5.6×10^{-5} sec^{-1} was examined by repeatedly deforming one specimen until the tensile strain just exceeded the elastic limit. At first, the specimens were tensile tested at room temperature. Then they were heated to 1073 K and were tensile tested. Thereafter, the specimens were cooled stepwise to the next temperature and were tensile tested. This procedure of cooling and tensile testing was repeated until 77 K was reached, with a 50 or 100 K interval. The yield stress was defined as the stress at which plastic tensile strain amounts to 1×10^{-4}. Plastic tensile strain applied to the specimens at each temperature was less than 5×10^{-4}.

The yield stress as a function of strain rate was also measured by repeatedly deforming one specimen with various strain rates from 5.6×10^{-6} to 5.6×10^{-4} sec^{-1} at 873 or 1073 K. For each test, the applied plastic tensile strain did not exceed 5×10^{-4}. To ensure the reliability of the data, the yield stresses at the strain rates of 5.6×10^{-5} and 5.6×10^{-4} sec^{-1} were also obtained independently by deforming other specimens with the same orientation.

3. Results

3.1. The temperature dependence of the critical resolved shear stress

Figure 1 shows the critical resolved shear stress (CRSS) as a function of temperature for the Cu–B_2O_3 and Cu–SiO_2 alloys with the shear strain rate of 5.6×10^{-5} sec^{-1}. CRSS of both alloys decreased with increase in temperature. Because the mean particle spacing in the Cu–SiO_2 alloy is shorter than that in Cu–B_2O_3, the Cu–SiO_2 alloy has higher CRSS. It should be noted that CRSS obtained at room temperature during the cooling run is in agreement with the virgin value obtained by the first test for both alloys. This means that the introduced amount of work hardening during each step-deformation test is negligible.

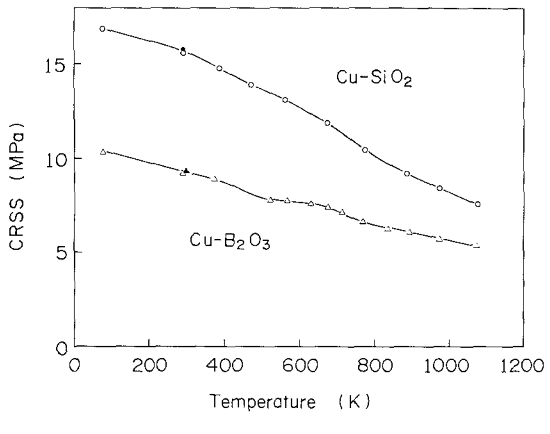

Figure 1 The temperature dependence of CRSS obtained under a shear strain rate of 5.6×10^{-5} sec^{-1} for Cu–B_2O_3 and Cu–SiO_2 alloys used in the present study. Close symbols indicate the values obtained by the first tests. Open symbols indicate the values during the cooling runs.

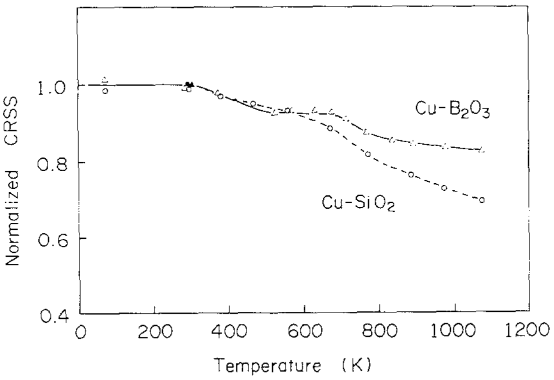

Figure 2 The temperature dependence of the normalized CRSS for Cu–B_2O_3 and Cu–SiO_2 alloys used in the present study.

The value of CRSS at room temperature, about 9.4 MPa, in the Cu–B_2O_3 alloy is in reasonable agreement with that (about 10.1 MPa) in a Cu–SiO_2 alloy with almost the same volume fraction (5.4×10^{-3}) and the slightly smaller diameter of particles (87 nm) [8]. Therefore, the low-temperature strength in the Cu–B_2O_3 alloy is explained by the Orowan mechanism, as in the case for the Cu–SiO_2 alloy [10, 11].

To compare the temperature dependence of CRSS of the two alloys, and to compensate CRSS for the change in the shear modulus of a copper matrix with temperature, the normalized CRSS $[(\tau_T/\mu_T)/(\tau_{RT}/\mu_{RT})]$, is plotted as a function of temperature, as shown in Fig. 2. Here, τ_T and μ_T are, respectively, CRSS and the shear modulus at a temperature, T, τ_{RT} the CRSS obtained by the first tests at room temperature (300 K) and μ_{RT} the shear modulus at room temperature. The values of μ_T have been evaluated using the following equation [12, 13]:

$$\mu_T = 4.21 \times 10^{10}[1 - (T - 300) \times 3.97 \times 10^{-4}] \quad (1)$$

It is found from Fig. 2 that although the normalized CRSS remains constant below room temperature, it gradually decreases with increase in temperature above room temperature for both alloys. It should be noted that the Cu–B_2O_3 crystal retains a large fraction of the low-temperature strength at high temperatures.

3.2. The strain-rate dependence of CRSS

Figure 3 shows the modulus-corrected CRSS $[\tau_T(\mu_{RT}/\mu_T)]$, as a function of the shear strain rate at 873 and 1073 K. Data obtained from different specimens are in good agreement, ensuring the reproducibility of the tests. Linear relationships are found from Fig. 3 between the modulus corrected CRSSs of the two alloys at 873 and 1073 K and the logarithms of the strain rates in the range between 5.6×10^{-6} and 5.6×10^{-4} sec^{-1}. The linear relationships were also found for several copper-base dispersion-hardened alloys such as Cu–Al_2O_3 and Cu–BeO [7]. Shewfelt

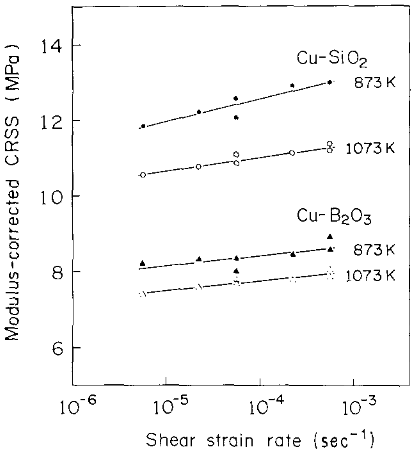

Figure 3 The modulus-corrected CRSS as a function of strain rate at 873 K and 1073 K for Cu–B_2O_3 and Cu–SiO_2 alloys used in the present study.

and Brown [8] also reported the linear relationships in Cu–SiO_2 at temperatures between 873 and 1073 K. Slopes of the lines for the Cu–SiO_2 alloy in Fig. 3 are in agreement with their results.

4. Discussion

Here, it is worth while to compare the deformability between the vitreous B_2O_3 particles and the copper matrix. The shear stress, τ, to cause the Newtonian viscous flow of B_2O_3 can be obtained from the equation, $\eta = \tau/\dot{\gamma}$, where η is the viscosity of vitreous B_2O_3 and $\dot{\gamma}$ the shear strain rate. Provided that the CRSS of the copper matrix after the internal oxidation and annealing is $\tau = 2$ MPa [10], η becomes 4×10^{10} N sec m^{-2} for $\dot{\gamma} = 5.6 \times 10^{-5}$ sec^{-1}. The values of η and their temperature dependence were reported by Napolitano *et al.* [14] and Shelby [15]. According to their results, η rapidly decreases with increase in temperature. At 550 K the value of η is about 4×10^{10} N sec m^{-2} and it becomes two orders of magnitude smaller at 600 K. Therefore, when deformation temperature is above about 550 K, the shear stress to cause the viscous flow of B_2O_3 becomes smaller than the CRSS of the copper matrix and the B_2O_3 particles in the copper matrix behave like liquid particles. On the other hand, since the viscosity of SiO_2 is much larger than that of B_2O_3 [16], the SiO_2 particles in the copper matrix are hard and non-deformable even during the high-temperature tests employed in the present study [8].

As shown in Fig. 2, although the B_2O_3 particles become liquid-like at high temperatures, the Cu–B_2O_3 alloy retains most of the low temperature strength even at 1073 K. Moreover, no distinct change in the normalized CRSS of the Cu–B_2O_3 alloy was observed at around 550 K. This shows that the liquid B_2O_3 particles can be the effective hardening centres even at high temperatures. It is also found from Fig. 2 that the normalized high-temperatures CRSS of the Cu–B_2O_3 alloy is even larger than that of the Cu–SiO_2 alloy. Although the reason for this fact remains to be clarified, it can be concluded that the high-temperature strength of alloys containing liquid inclusions is no smaller than that of alloys containing hard and solid inclusions.

Weeks *et al.* [17] have considered the interaction between a straight dislocation and a void or a bubble and have found that the interaction is attractive. Moreover, Srolovitz *et al.* [3, 4] have concluded that when diffusional relaxation occurs along the matrix–inclusion interface, the interaction between a dislocation and an inclusion becomes attractive regardless of the character (hard or soft) of the inclusions. In these cases, a criterion for the dislocation to overcome the barrier may be determined by the detachment process of the dislocation from the inclusions and the threshold stress can be comparable to a stress given by the Orowan mechanism [5, 6, 17]. The present experimental results indicate that the origin of the strength of the Cu–B_2O_3 alloy at high temperatures most likely comes from the attractive interaction between a dislocation and a liquid particle.

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References

1. L. M. BROWN and R. K. HAM, in "Strengthening Methods in Crystals", edited by A. Kelly and R. B. Nicholson (Elsevier, Amsterdam, 1971) p. 9.
2. R. S. W. SHEWFELT and L. M. BROWN, *Phil. Mag.* **35** (1977) 945.
3. D. J. SROLOVITZ, M. J. LUTON, R. PETKOVIC-LUTON, D. M. BARNETT and W. D. NIX, *Acta Metall.* **32** (1984) 1079.
4. D. J. SROLOVITZ, R. PETKOVIC-LUTON and M. J. LUTON, *Scripta Metall.* **18** (1984) 1063.
5. E. ARTZ and D. S. WILLKINSON, *Acta Metall.* **34** (1986) 1893.
6. E. ARTZ and J. RÖSLER, *ibid.* **36** (1988) 1053.
7. F. J. HUMPHREYS, P. B. HIRSCH and D. GOULD, in Proceedings of the 2nd International Conference on the Strength of Metals and Alloys, Asilomar, California, 1970 (Amer. Soc. Metals) p. 550.
8. R. S. W. SHEWFELT and L. M. BROWN, *Phil. Mag.* **30** (1974) 1135.
9. G. DOREY, Royal Aircraft Establishment, Technical Report No. 65126, No. TR68230, 1968 (cited in [8]).
10. R. EBELING and M. F. ASHBY, *Phil. Mag.* **13** (1966) 805.
11. M. F. ASHBY, in "Physics of Strength and Plasticity", edited by A. S. Argon (MIT Press, Cambridge, Massachusetts, 1969) p. 113.
12. W. C. OVERTON Jr and J. GAFFNEY, *Phys. Rev.* **98** (1955) 969.
13. Y. A. CHANG and L. HIMMEL, *J. Appl. Phys.* **37** (1966) 3567.
14. A. NAPOLITANO, P. B. MACEDO and E. G. HAWKINS, *J. Amer. Ceram. Soc.* **48** (1965) 613.
15. J. E. SHELBY, *J. Appl. Phys.* **45** (1974) 5272.
16. G. HETHERINGTON, K. H. JACK and J. C. KENNEDY, *Phys. Chem. Glasses* **5** (1964) 130.
17. R. W. WEEKS, S. R. PATI, M. F. ASHBY and P. BARRAND, *Acta Metall.* **17** (1969) 1403.

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