Letters

Grain size strengthening and weakening in a copper base alloy

At low temperatures metals show an increase in strength as the grain size is decreased. This phenomenon has been recognized for some time [1] and plays an important role in the fabrication of alloys for low temperature applications. Under conditions of creep at elevated temperatures a reversal of the grain size influence on strength is often observed [2, 3]. The ultimate effect of this grain size weakening is the phenomenon of micrograin superplasticity where a very low flow stress and high ductility is obtained in metals with an ultrafine grain size [4]. Superplastic forming is now receiving attention as a possible industrial process [5]. Apart from the actual financial benefits of superplastic forming over conventional forming an added attraction is that components for low temperature applications will have increased strength as a result of their fine grain size. It is the purpose of this note to present results on grain size strengthening at low temperatures and grain size weakening at elevated temperatures in an industrial copper base alloy (Coronze CDA 638) which exhibits superplasticity [6].

Round tensile specimens (16 mm gauge length, 4.54 mm gauge diameter) were machined from 9.3 mm thick hot-rolled plate of an industrial copper dispersion alloy known as Coronze (95.0% Cu, 2.8% Al, 1.8% Si, 0.4% Co) which is manufactured by Olin Corporation. Grain size variations were obtained by isochronal anneals (150 min) in the temperature range 700 to 950° C (Table I). An ultrafine grain size (1 μm) was obtained by a separate thermo-mechanical treatment of the hot-rolled plate and flat tensile specimens (12.7 mm gauge length, 9.53 mm gauge width and 1.52 mm thick) were machined from this material. Grain sizes were measured by the linear intercept method. Creep and hot tensile testing was carried out using three zone furnaces controlled to ± 3° C over 150 mm length.

TABLE I Influence of annealing temperature on grain size.

Annealing temperature (° C)	Grain size (μm)
700	16
850	126
900	263
950	720

The influence of grain size on the room temperature hardness (V.P.N.) is recorded in Fig. 1 where an increase in hardness with a decrease in grain size, following the usual Hall-Petch relationship [7], is observed. The results of dead load creep tests at 400° C and 127.2 MN m^{-2} are presented in Figs. 2, 3 and 4. The influence of grain size on the minimum creep rate ($\dot{\epsilon}_{\mathrm{mcr}}$) is recorded in Fig. 2 where $\dot{\epsilon}_{\mathrm{mcr}}$ is observed to decrease with an increase in grain size. In Fig. 3 the grain-size dependence of ductility (% elongation) is presented and a ductility minimum is observed at an intermediate grain size. The variation of time to rupture (t_r) with grain size is recorded in Fig. 4 where t_r is observed to increase with an increase in grain size. Figs. 5 and 6 record the results of dead load creep tests at 600° C and 26.8 MN m^{-2}. The influence of grain size on $\dot{\epsilon}_{\mathrm{mcr}}$ is presented in Fig. 5 where a decrease in $\dot{\epsilon}_{\mathrm{mcr}}$ with an increase in grain size is observed. Fig. 6 shows that for these test conditions t_r increases with an increase in grain size. Results of constant strain-rate tests (7.0 × 10^{-2} h^{-1}) at 400 and 600° C are recorded in Table II. Fig. 7 shows the influence of test temperature on the per cent elongation of 1 μm grain sized material tested at a nominal constant strain-rate of 3.94 × 10^{-2} min^{-1}. At the temperature of maximum elongation, 550° C, the 1% flow stress was 24 MN m^{-2}.

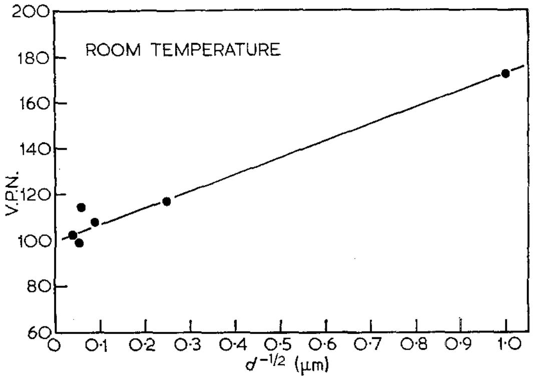

Figure 1 Variation of V.P.N. with grain size at room temperature.

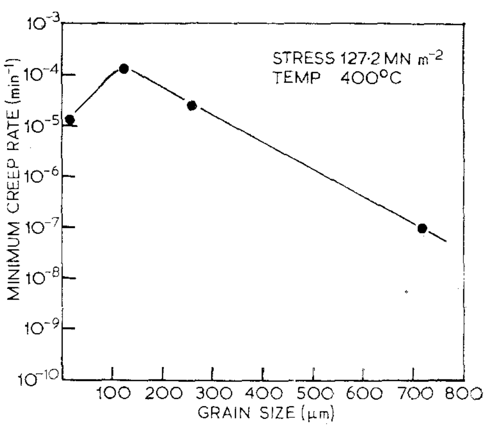

Figure 2 The influence of grain size on the minimum creep rate at 400°C and 127.2 MN m^{-2}.

The results from Fig. 1 indicate that the room temperature strength is increased by refining the grain size from 720 to 1 μm, as is predicted by the Hall-Petch [7] relation:

$$\sigma_y = \sigma_0 + k_y d^{-\frac{1}{2}} . \quad (1)$$

Grain size weakening at 400 and 600° C is shown by Figs. 2 and 5 and Table II. Where it is shown that $\dot{\epsilon}_{mcr}$ decreases with an increase in grain size and that the 1% flow stress decreases with a decrease in grain size. Barrett *et al* [3] have attributed this to the increased importance of grain boundary sliding at finer grain sizes. This explanation fits in with the present set of results as it has been shown, for this alloy [8], that the contribution of grain boundary sliding to total strain increases as the grain size is decreased. However, an increased contribution by diffusional flow and via grain-boundary dislocation sources is also possible with decreasing grain size. The results of the grain-size dependence of t_r, Figs. 4 and 6, show reverse trends for the different

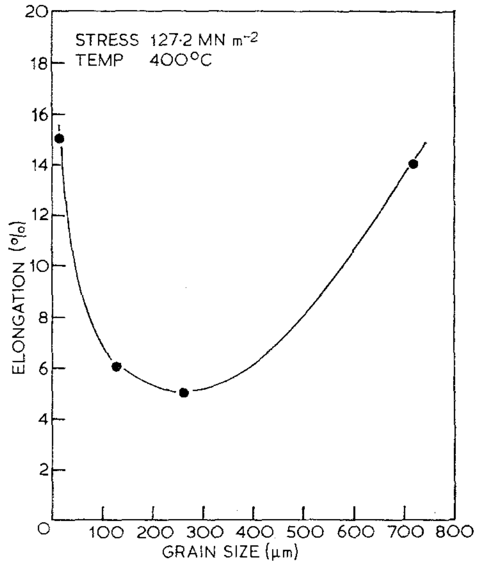

Figure 3 The influence of grain size on the per cent elongation at 400° C and 127.2 MN m^{-2}.

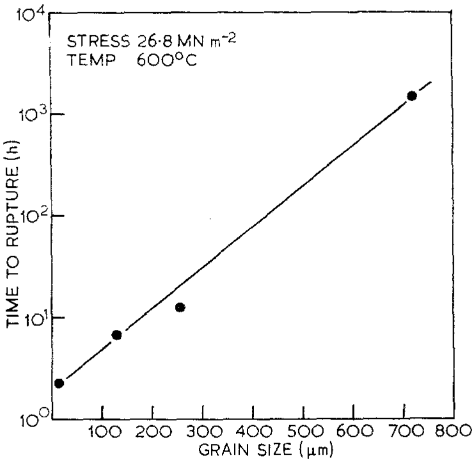

Figure 4 The influence of grain size on the time-to-rupture at 400°C and 127.2 MN m^{-2}.

TABLE II Tensile data at 400 and 600° C as a function of grain size for a strain-rate of 7.0 × 10^{-2} h^{-1}.

Test temperature (°C)	Grain size (μm)	Elongation (%)	Strain-rate sensitivity	Flow stress (1%) MN m^{-2}
400	1	200	0.27	36
	16	41	0.16	55
	720	8	—	146
600	1	282	0.4	20
	16	52	0.13	34
	720	9	—	55

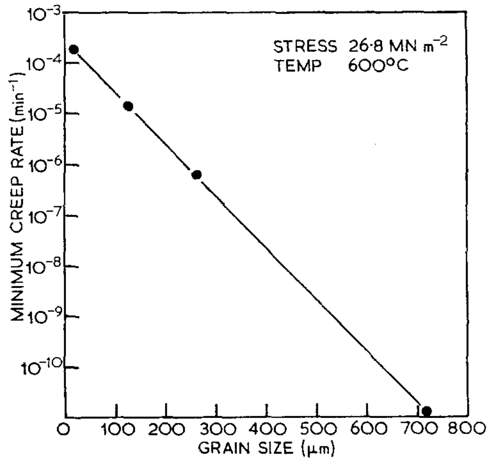

Figure 5 The influence of grain size on the minimum creep rate at 600° C and 26.8 MN m^{-2}.

conditions investigated. The time to rupture can be considered as the time required to develop a favourable crack length or distribution for spontaneous growth and interlinkage to give fracture [2]. The results from the tests at 600° C, Fig. 6, suggest that this critical length or distribution becomes more difficult to obtain as the grain size is increased. Garofalo [9] suggests that this is a result of the crack distribution being controlled by the creep rate and that it is the expected trend under conditions where $\dot{\epsilon}_{mcr}$ is decreased by increasing the grain size. This relationship can also be explained in terms of strain controlling the crack length or distribution as at fine grain sizes higher strains will be obtained for a given time than at coarse grain sizes. The results in Fig. 4 from tests at 400°C and 127.2 MN m^{-2} show a decrease in t_r with an increase in grain size. Similar results have been observed by Lagneborg [10] who explains them in terms of a decrease in the incubation time for crack nucleation with an increase in grain size [11]. If it is assumed that nucleation is controlled by grain-boundary sliding then this explanation fits in with the present set of results as it has been shown [8] that the mean sliding step height ($\bar{V}$) increases with an increase in grain size. Langdon [12] has suggested that the trend of a decrease in t_r with an increase in grain size is a result of excessive straining of the specimen on applying the load. This is unlikely to be the case in the present study as the loads were applied slowly to the specimen.

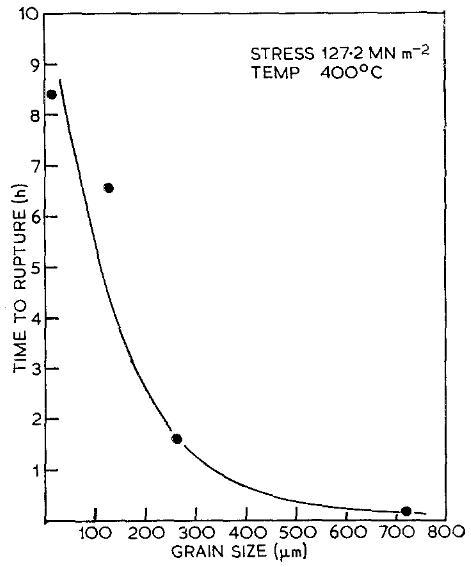

Figure 6 The influence of grain size on the time-to-rupture at 600° C and 26.8 MN m^{-2}.

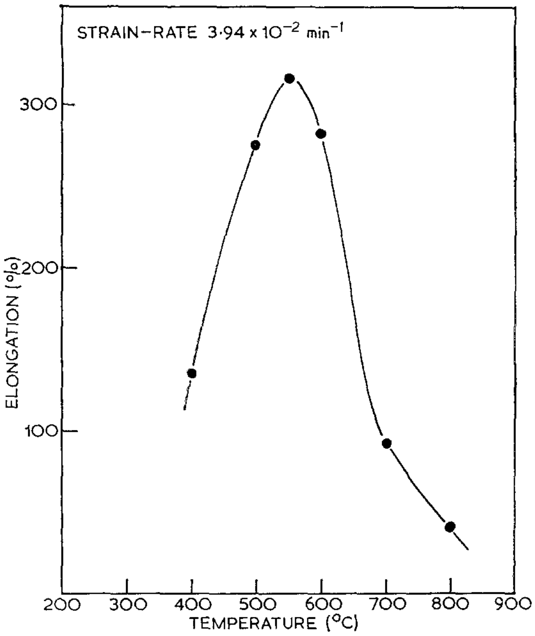

Figure 7 The variation of per cent elongation with temperature at a strain-rate of 3.94 × 10^{-2} min^{-1}.

Fig. 3 shows a ductility minimum at an intermediate grain size for tests at 400°C and 127.2 MN m^{-2}. This effect has also been noted in α-iron [13] and explained in terms of a marked increase in the importance of grain-boundary sliding at an intermediate grain size. However, this point requires further study before a more definite explanation can be put forward.

Armstrong [14] has suggested that at intermediate stress (σ) levels and at high temperatures, superplastic behaviour of fine grain sized materials is a result of the pronounced temperature dependence of the self diffusion coefficient (D) in the following equation

$$\dot{\epsilon}_{\mathrm{mcr}} = \left[\frac{10\Omega D\sigma}{RT}\right] d^{-2} \qquad (2)$$

where Ω is the atomic volume and d the grain size. The present set of results indicate that superplastic behaviour occurs at intermediate temperatures 0.5 T_{m} (T_{m} is the melting temperature in degrees absolute) and at fast strain-rates 3.94×10^{-2} min^{-1}, which suggests that the grain-size weakening effect is not explicitly dependent on the self diffusion coefficient. A more likely source for this effect is the marked increase in grain boundary sliding with a decrease in grain size.

In conclusion it has been shown for this alloy that:

(i) the room temperature strength can be increased by a decrease in grain size,

(ii) the minimum creep rate can be decreased by an increase in grain size and

(iii) that superplastic deformation occurs at 0.5 T_{m} at a strain-rate of 3.94×10^{-2} min^{-1} and a 1% flow stress of 24 MN m^{-2} in 1 μm grain sized material which exhibits high room temperature strength.

Acknowledgements

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Flux growth of the rare earth borates, RBO_3

Single crystals of the aragonite and calcite type RBO_3 compounds have been prepared. With R = Sm to Yb, it appears that the high temperature vaterite structure transforms to a room-temperature structure of lower symmetry. Hexagonal pseudomorphs with this structure were flux-grown by the slow cooling method.

The rare earth borates, RBO_3, fall into three groups according to their crystal structure, each group being structurally related to one of the three forms of calcium carbonate. At room temperature, those with R = La to Nd have the orthorhombic structure of aragonite [1-3]. Those with R = Sm to Yb have a structure which resembles that of vaterite, but Levin *et al* remark that these are really pseudo-hexagonal, as it was not possible to index all the lines on the X-ray powder pattern [1]. The divergence from the vaterite structure has been confirmed by studies of vibrational spectra [4-6]. Below 1310°C,