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The stress dependence of the subgrain size in aluminium

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Etch-pit (EP) technique and transmission electron microscopy (TEM) have been used to investigate the subgrain size, δ, as a function of applied shear stress, τ, during high-temperature creep of aluminium. Examination of thin foils, prepared from deformed specimens, in the electron microscope shows the presence of very large equiaxed subgrains that approximate those observed in etch-pit photographs. By measuring the average subgrain size from transmission micrographs of representative areas of the foils, two observations are made. First, the average subgrain size is smaller than that determined from etch-pit procedure, but exhibits the same stress dependence:

$$\delta/\mathbf{b} \propto (\tau/G)^{-1},$$

where b is the Burgers vector and G is the shear modulus. Second, the TEM subgrain size data of aluminium and those of other metals and alloys, when plotted in the normalized form of $\delta\tau/G\mathbf{b}$ against $\gamma/G\mathbf{b}$ on a logarithmic scale, where γ is the stacking-fault energy of the material, fall within a narrow, horizontal band, confirming earlier reports that the subgrain size is insensitive to stacking-fault energy.

1. Introduction

Subgrains are formed during the creep of metals and solid-solution alloys whenever the climb mechanism is rate controlling [1]. Several different techniques including X-ray diffraction analysis, polarized-light, etch-pit, and transmission electron microscopy have been used to examine and measure the subgrain size. The average subgrain size, δ, has been experimentally reported [1, 2] as being related to the normalized shear stress, τ/G, through a relationship of the form

$$\frac{\delta}{\mathbf{b}} = K(\tau/G)^{-r}, \qquad (1)$$

where $\mathbf{b}$ is the Burgers vector, K is a constant, τ is the applied shear stress, G is the shear modulus and r is a constant. While analyses of subgrain size data of many metals and alloys deformed under creep conditions, as documented elsewhere [1–3], show that $r = 1$ and $K = 10$, investigations on aluminium [1, 2, 4, 5] seem to suggest that the values of r and K are influenced by the type of technique used to measure δ. This apparent dependence of r and K on the type of technique, not only questions the suitability of a particular technique for examining the subgrain size in aluminium, but also imposes a limitation on developing substructural correlations between aluminium and other metals. In the present paper, experimental results on the subgrain size obtained using the etch-pit and transmission electron microscopy techniques, along with earlier data, are examined in an attempt to clarify this technique dependency.

2. Experimental technique

Pure aluminium (of purity 99.99%), obtained from the Kaiser Aluminum and Chemical Corporation, was tested at 473, 573, 673 and 923 K in both tension and double shear [6, 7]. The choice of these temperatures was dictated by the need to obtain measurable creep rates over a wide range of normalized stresses. Prior to testing, all specimens were annealed *in situ* for at least 10 h at 923 K to remove effects of machining and to produce a stable, uniform grain size. This anneal-

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ing treatment resulted in a grain size of approximately 5 mm. Tensile specimens were tested in a three-zone furnace and the temperature was kept to within ± 1 K of the reported value over the gauge length (9.5 cm). Creep tests conducted under double-shear conditions have been discussed elsewhere [6, 7] and it is only important to mention that sufficient strain was attained in each test to unequivocally establish the steady-state creep. After straining, specimens were cooled rapidly in air to preserve the substructure developed during creep.

Etch-pit (EP) samples were individually mounted, mechanically polished, electro-polished in a mixture of 15% perchloric acid in methanol and, finally, etched at room temperature using a mixture of 50% HCl, 47% HNO_3, and 3% HF. TEM samples were mechanically polished to a thickness of 0.1 mm. Thin foils were then prepared using the window technique and a polishing solution of 15% perchloric acid in methanol which was surrounded by a dry-ice–acetone bath to keep the electrolyte temperature at 193 K. Thin foils were examined in a JEOL-100C electron microscope operating at 100 kV.

For TEM measurements, efforts were made (a) to prepare a large number of thin foils from specimens deformed at each stress, and (b) to obtain numerous transmission micrographs (about 30) of representative areas of these foils; the number of subgrains ranged from 5 to 20 in each micrograph, depending on the subgrain size. The subgrain size was measured using the linear intercept method. Ninety-five per cent confidence limits, which were equivalent to an error of approximately 10% in the subgrain size, were calculated.

3. Experimental results and discussion

Two types of subgrains were observed in each foil examined in TEM: (a) banded subgrains and (b) both small and large equiaxed subgrains.

Fig. 1a is a micrograph of banded subgrains and, as can be seen, subgrains are elongated and arranged into bands of a common axis of elongation. Banded subgrains were observed at all stresses, although the frequency of their presence decreases with decreasing stress level. Also, banded subgrains were observable in thin foils prepared from double-shear specimens that experienced shear strains as high as 70%, suggesting that they are a persistent feature of the substructure.

Fig. 1b is a micrograph of small equiaxed subgrains. For any stress level, it was found that no difficulty was encountered, when operating the transmission microscope at either high or intermediate magnifications, to observe these small subgrains.

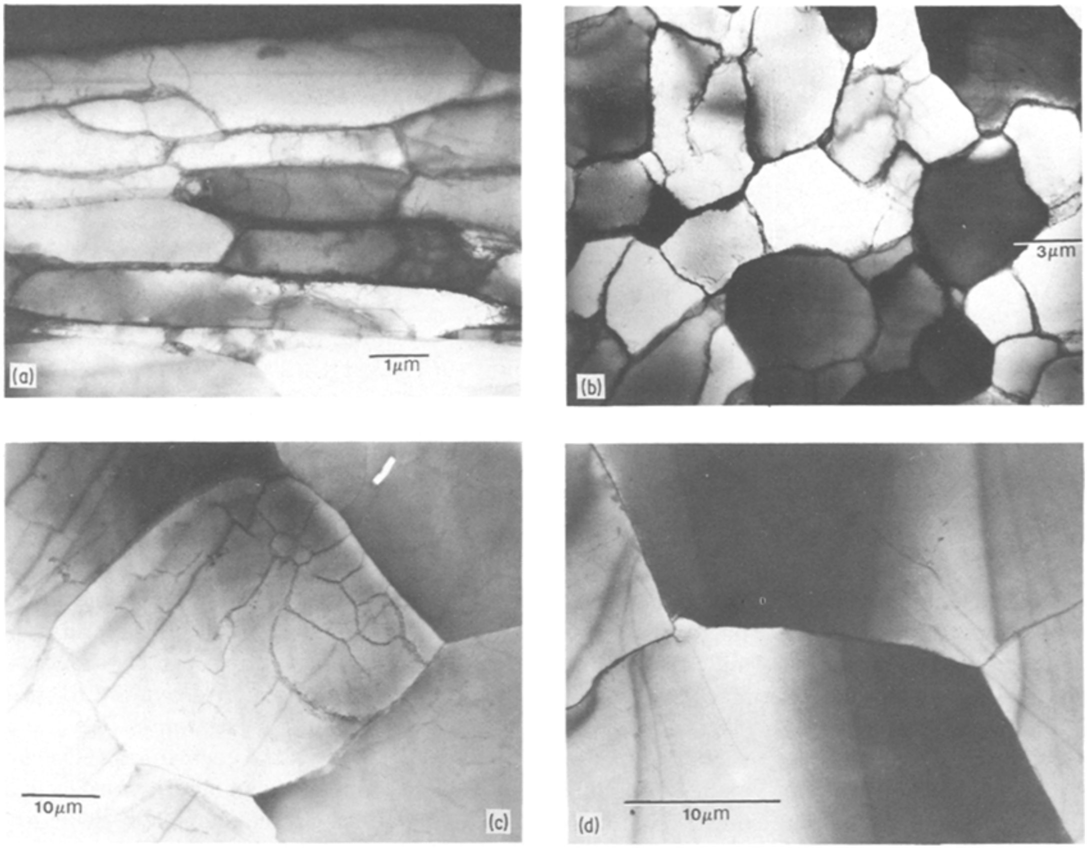
In contrast, extensive efforts that included preparation of numerous foils having large thin areas and the use of very low magnifications (x 660 to x 1000) were necessary to examine the large equiaxed subgrains shown in Fig. 1c. These efforts were successful at intermediate stresses ($5 \times 10^{-5} < \tau/G < 2 \times 10^{-4}$), but not at low stresses when $\delta/\mathbf{b}$ exceeded 4×10^5. Nevertheless, the presence of the large subgrains at low stresses was inferred from the observations of a single, well-developed sub-boundary, extending across the whole thin area, and/or triple-junctions of sub-boundaries (Fig. 1d). Observation of large subgrains at high stresses did not call for special efforts and, in many cases, both small and large subgrains were seen in the same micrographs (Fig. 1e).

Etch-pit photographs revealed generally equiaxed subgrains and also showed inhomogeneous subgrain sizes (very fine and large subgrains could be found) within each grain. Banded subgrains were occasionally observed, but they were localized in very small areas.

The present results are shown in Fig. 2, where $\delta/\mathbf{b}$ against τ/G is plotted on a logrithmic scale; the relation $\tau = \sigma/2$ was used for tensile samples, where σ is the applied tensile stress.

With the exception of the datum point at the lowest normalized stress, the EP data of Fig. 2 fit a straight line that can be described by Equation 1 with $r = 1$ and $K = 20$. The present value of K is a factor of 2 higher than that estimated by Bird *et al.* [1] from data of metals and alloys, excluding aluminium. The data of TEM show two observations. First, the size of the large equiaxed subgrains agrees reasonably well with that predicted from the extrapolation of the EP line. Second, the data points representing an average of measurements taken from representative micrographs (solid circles) fall on a segment of a straight line that is parallel to the EP line and separated from it by almost a factor of 2.

In Fig. 3, $\delta/\mathbf{b}$ values obtained during several different investigations [4, 5, 8–14] on aluminium are plotted against τ/G on a logarithmic scale. All points represent data obtained by etch-pit (EP) [8–12], polarized-light technique (OPT) [4], or



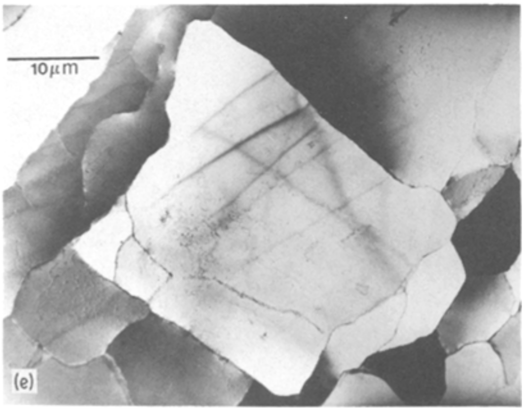


Figure 1 (a) Banded subgrains observed at $\tau/G = 6 \times 10^{-5}$; (b) small equiaxed subgrains observed at $\tau/G = 3 \times 10^{-5}$; (c) large equiaxed subgrains observed at $\tau/G = 1.5 \times 10^{-4}$; (d) two triple junctions of sub-boundaries observed at $\tau/G = 9 \times 10^{-6}$; and (e) equiaxed subgrains observed at $\tau/G = 2.7 \times 10^{-4}$.

transmission electron microscopy (TEM) [4, 5, 13, 14], but do not include data obtained by X-ray diffraction methods. Also, care has been exercised to include only data obtained from constant-stress creep tests that were conducted at or above 473 K (0.5 T_m, where T_m is the melting point of aluminium). Fig. 3 shows that the EP data obtained from five independent investigations are bounded by two straight lines having $K = 15$ and $K = 40$ due to the presence of a small, but distinct, separation* between the data points of [8] and those of [9].

Also, as indicated by Fig. 3, the present EP data agree with the position of the narrow band of $\delta/\mathbf{b}$ against τ/G. Despite this agreement, the EP technique, as found in the present investigation, seems to suffer from a serious limitation at high stresses; in this range it is extremely difficult to determine the sizes of small subgrains, basically because etch-pits that result from the presence of high dislocation densities in subgrain interiors may interfere with the identification of sub-boundaries. Examination of TEM micrographs shows that the EP procedure also tends to exclude the contribution of small subgrains to measurements even over the intermediate stress range. This is clearly

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*Also, most data points of [10] are higher than those both of [9] and of the present investigation.

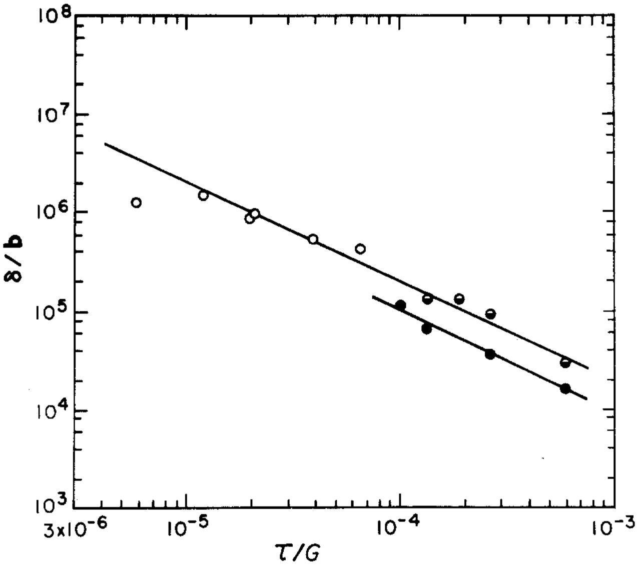

Figure 2 $\text{Log}_{10}\delta/b$ plotted against $\log_{10}\tau/G$ for the present results. Open circles represent measurements taken from photographs of etch pitted specimens, circles filled in their lower half represent the large equiaxed subgrains observed in TEM and solid circles represent averages taken from TEM micrographs of representative areas of thin foils.

demonstrated by the observation that small subgrains observed in transmission micrographs, over this range of stresses, appear significantly smaller than the smallest subgrain determined from several EP photographs at any particular value of the applied stress. However, it is possible that at very low stresses small subgrains become resolvable, partly due to low dislocation densities and partly because of the increase of the subgrain size with decreasing stress; as a result, small subgrains may significantly contribute to the EP measurements. This possibility may explain the tendency of experimental data to scatter downward to smaller sizes when $\tau/G < 10^{-5}$, as shown in Figs 2 and 3.

Also, Fig. 3 provides a comparison between the EP data and those of TEM observations, including earlier investigations on aluminium [4, 5, 13], tested under creep conditions. An examination

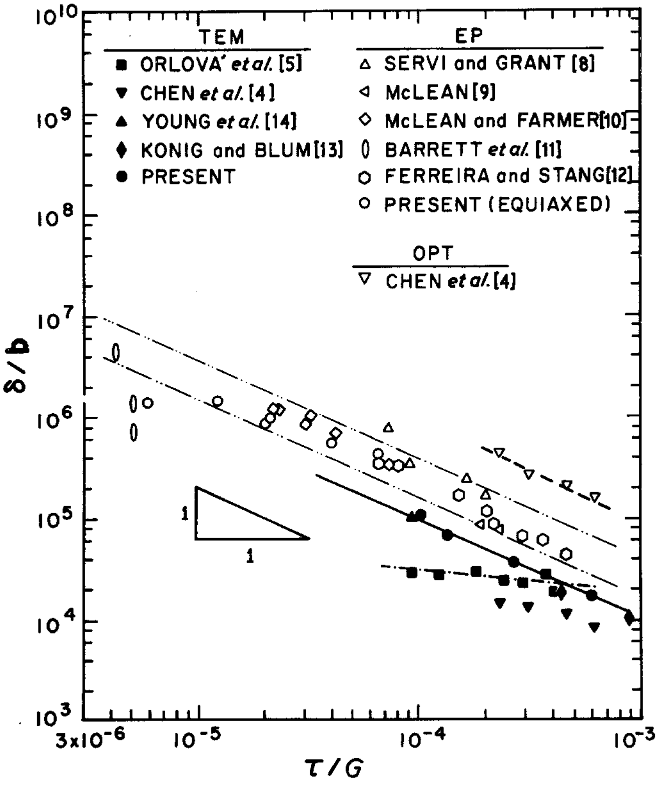

Figure 3 $\text{Log}_{10}\delta/b$ plotted against $\log_{10}\tau/G$ for aluminium tested under constant stress creep conditions at or above 473 K, where δ is determined by etch-pit [8–12], optical technique [4], or transmission electron microscopy [4, 5, 13, 14].

of Fig. 3 shows that the data of Orlová *et al.* [5] exhibit a much weaker stress dependence as well as smaller subgrain sizes when compared with the present TEM and EP data. Also, the TEM data of Chen *et al.* [4], with the exception of the datum point at the highest τ/G, show a similar trend. While experimental conditions under which measurements of the subgrain size were performed in those two investigations [4, 5] are not clearly known, it seems most likely that the discrepancy between the present and earlier TEM data [4, 5] arises from a lack of representative TEM measurements in the earlier investigations [4, 5]. This suggestion is supported by several observations. First, the observation of the large equiaxed subgrains at intermediate stresses in the present investigation (Fig. 2) required extensive efforts that included preparation of many thin foils for each stress and the use of the lowest practical magnification. The sizes of these large subgrains agree with those measured by the EP procedure and, therefore, the failure to observe and include them in measurements would undoubtedly result in an underestimation of the average subgrain size; this underestimation, if it occurred, is expected to be more significant as the applied stress is decreased. Comparison between the positions of the data of Orlová *et al.* [5] and those of the present EP and TEM data tends to support this view since the divergence between the two sets of data becomes more distinct at lower stresses. Second, data points representing averages taken from transmission micrographs of representative areas of thin foils fall on a straight line that is parallel to the EP line, showing that the subgrain size, as expected, varies inversely with the applied stress. Third, the two TEM data points taken from a recent investigation by Konig and Blum [13] are consistent with the position of the present TEM line (solid line). These two points were obtained at high stresses, a range in which the electron microscopy technique is expected to be efficient and practical in providing representative measurements of the subgrain size due to the fine scale of the substructure. It might be mentioned that Young *et al.* [14] reported a single measurement of the subgrain size using TEM. Their measurements, which were obtained under the constant strain rate condition, correspond to $\tau/G \simeq 10^{-4}$ and agree very well with the present TEM data (constant stress condition) as shown in Fig. 3.

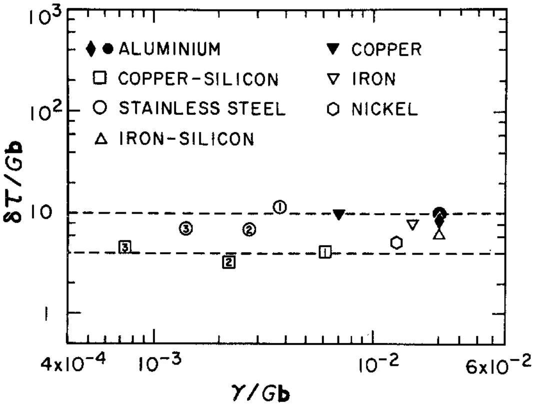

Figure 4 $\text{Log}_{10}\,\delta\tau/G\mathbf{b}$ plotted against $\log_{10}\gamma/G\mathbf{b}$ for several metallic systems [1–3, 14–18].

Based on the above discussion, it is suggested that the solid line of Fig. 3 represents the stress dependence of the subgrain size in aluminium. The position of this line is given by

$$\frac{\delta}{\mathbf{b}} \simeq 10\,(\tau/G)^{-1}. \qquad (2)$$

As mentioned earlier, the difference in the values of K obtained from the EP procedure ($K = 20$) and from TEM ($K = 10$) is most probably due to the difficulty of resolving small subgrains in EP photographs. This interpretation is also consistent with the position of the OPT data relative to those of EP and TEM; the OPT technique is known to be very insensitive to small subgrains [1] and, as shown by Fig. 3, gives the highest value of K ($K = 90$). The value of K given by Equation 2 is identical to that determined from TEM data on copper [15], showing that the subgrain size is not influenced by stacking-fault energy, γ; $\gamma_{\text{Cu}} = 50\,\text{erg cm}^{-2}$, while $\gamma_{\text{Al}} = 200\,\text{erg cm}^{-2}$.

The insensitivity of the average subgrain size, δ, to stacking-fault energy, γ, has previously been inferred from an examination of the subgrain size in Cu–Si alloys [16], in which γ varies by a factor of 8 but subgrains developed are of identical size, or from the overlap of subgrain size data of several metallic systems over the same range of stresses [1, 2, 14]. Over the past decade, a compilation of data on stacking-fault energy has become available, and it is therefore possible to introduce a different type of plot which examines the correlation between $\delta/\mathbf{b}$ and γ. This plot is shown in Fig, 4, where $\delta\tau/G\mathbf{b}$ is plotted against the normalized stacking-fault energy parameter, $\gamma/G\mathbf{b}$, on a logarithmic scale. The data on the subgrain size and stacking-fault energies were taken from several recent works [1–3, 14–18]. As shown by Fig. 4, the parameter $\delta\tau/G\mathbf{b}$ exhibits no systematic dependence on $\gamma/G\mathbf{b}$, which varies by almost one order of magnitude*, and is bounded by the two limits of $K = 10$ and $K = 4$, presumably due to experimental scatter. Also, Fig. 4 demonstrates that aluminium is no exception, as previously thought, to the concept that the subgrain size is not influenced by stacking-fault energy.

4. Conclusions

TEM measurements taken from numerous micrographs of representative areas of thin foils show that the variation of the subgrain size of aluminium with the applied stress agrees with the semi-empirical equation, Equation 2, $\delta/\mathbf{b} \simeq 10(\tau/G)^{-1}$, which was developed from consideration of data on several materials. This agreement not only indicates that optical techniques tend to overestimate the subgrain size but it also confirms the concept that the subgrain size is independent of stacking-fault energy.

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*In contrast, the same range of $\gamma/G\mathbf{b}$ results in creep rates that span four orders of magnitude.