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Superplastic testing conditions and grain growth

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The strain-rate sensitivity index m of the Cu-9.5 wt % Al-4 wt % Fe alloy has been determined by two different procedures: (1) from a rate-change test and (2) from the slopes of the maximum engineering stress/initial strain-rate curve. The discrepancies between the results obtained according to the two different procedures are outlined and discussed.

Besides the necking behaviour during deformation, factors such as (1) changes in the primitive defect structure, (2) large variations of m over the strain-rate interval covered and (3) grain growth during deformation, are to be considered in explaining the origin of the discrepancies. Dunlop and Taplin's method is considered to be the better of the two procedures used for determining m, since it introduces fewer errors. In this alloy, grain-coarsening is caused by thermal activation as well as by the deformation process itself.

1. Introduction

The phenomenon of anomalously high elongations in a tension test was observed by Pearson, as early as 1934 [1]. However, it was not until the work of Backofen *et al* [2] that this domain received considerable attention. It was pointed out that for a micrograined Zn-22 wt % Al alloy, there was a strong relationship between the flow stress, σ, and the strain-rate, $\dot{\epsilon}$, which was expressed by the equation

$$\sigma = K\dot{\epsilon}^m$$

where K is a constant of proportionality and is a function of grain size and temperature. It was found that an increase in the exponent

$$m = \frac{\mathrm{d}\log\sigma}{\mathrm{d}\log\dot{\epsilon}}$$

resulted in an increase in overall elongation. The exponent, m, is termed the *strain-rate sensitivity index* and is a measure of the degree of superplasticity in a given specimen. It has also been shown by several workers that the value of m attains a maximum for a given strain-rate (usually less than 0.1 sec^{-1}) at a temperature usually greater than 0.5 T_M (T_M is the homologous temperature expressed in Kelvin) [3-8]. The superplastic materials have been known to possess a very fine grain size, and in the case of duplex structures, equal fractions of the distinct phases.

As grain size is an important parameter in the description of superplastic behaviour, it is interesting to investigate the phenomenon of grain growth during deformation. The present paper gives experimental evidence for grain growth and discusses the influence of this effect on the results of the rate-change method, which is the commonly used superplastic test method.

2. Experimental procedure

The material used in this study was a Cu-9.5 wt % Al-4 % wt Fe alloy.(*) This material is superplastic at about 800°C [9] and contains equal proportions of α and β.

Two types of tensile specimens have been cut out of plate material, namely, series A, 0.5 mm thick (Fig. 1a) and series B, 2 mm thick (Fig. 1b). The specimens were mounted in an ADAMEL air furnace on an Instron TT-DM-L tensile machine. Without the tensile clamps in position the furnace displays a zone of constant temperature (± 1°C), 18 cm long. These tensile clamps introduce varying temperature gradients depending on their relative position as shown in Figs. 2a and b. The temperature variation was about ± 3°C for the A-specimens, whereas for

*The Olin CDA-619 alloy, which has kindly been supplied by the Olin Corporation.

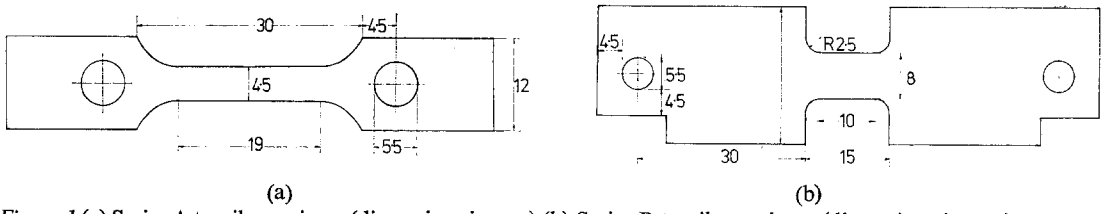

Figure 1 (a) Series A tensile specimen (dimensions in mm).(b) Series B tensile specimen (dimensions in mm).

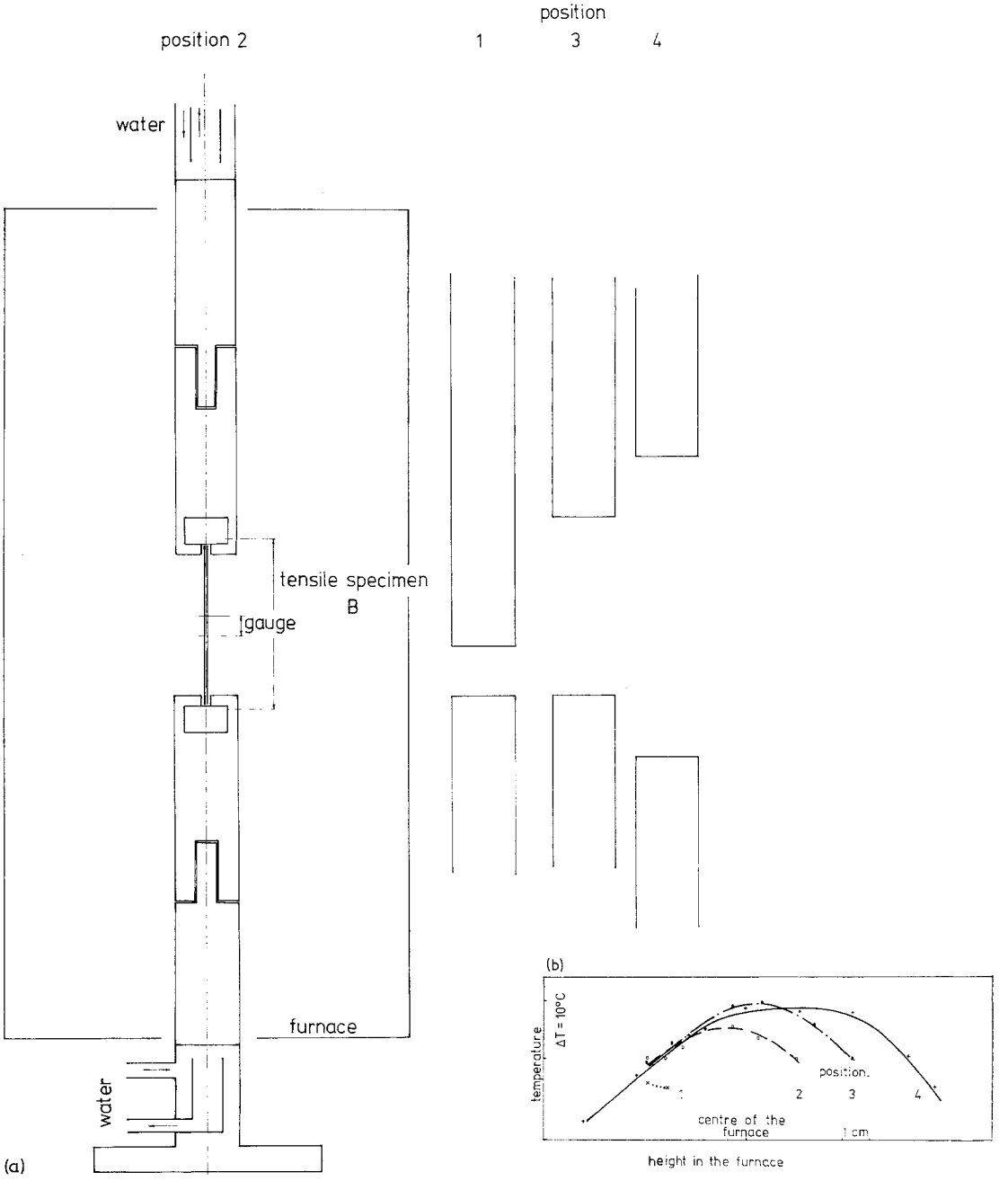

Figure 2 (a) Schematic drawing of the tensile devices and the furnace for four different positions of the tensile clamps. (b) Temperature gradient for the different positions of the tensile devices of (a).

the B-specimens, with a more remote clamp attachment, it stayed at about ± 1°C. The time prior to testing, which is necessary for heating up and stabilizing the temperature of the specimen, was 50 min. The specimens of series B were ice-quenched at the end of the test.

3. Results

3.1. Mechanical

From tests on series A-specimens σ-$\dot{\epsilon}$ and m-$\dot{\epsilon}$ diagrams have been obtained, following the methods proposed by Backofen *et al* [2] and that developed by Dunlop and Taplin [9].

1. In the first method (Fig. 3), the value of m is calculated from a rate-change test, according to the equation

$$m = \frac{\log (P_A/P_B)}{\log (v_2/v_1)} \qquad (1)$$

where v_1 is the smaller cross-head speed; v_2, the the larger cross-head speed; P_A the maximum load after cross-head speed changing; P_B the load which would have been reached at the original cross-head speed, for similar deformation as for P_A.

Evidently, m must change slowly over the strain-rate range covered by the rate-change test, for the deviation between this calculated value and the theoretical value,

$$m = \left(\frac{\partial \log \sigma}{\partial \log \dot{\epsilon}}\right)_{\epsilon, T, L},$$

to be small ($\bar{L}$ = mean intercept length).

2. In the second method, a σ-$\dot{\epsilon}$ curve is obtained by plotting the value of the engineering stress at the beginning of each test versus the initial strain-rate. The values of m are obtained by calculating the slope at various values of strain-rate. The results of these experiments with series A-specimens are shown in Figs. 4 and 5.

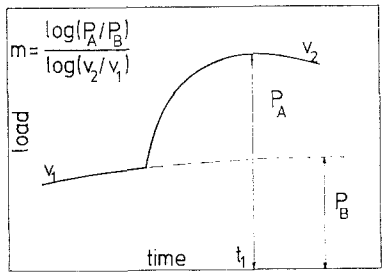

Figure 3 Principle of the rate-change test in order to determine the strain-rate sensitivity index, m.

Series B-specimens were used to evaluate grain growth during deformation. Results of this set of experiments are given in Table I. Fig. 6 represents the load-time curve for specimen B_1, while Fig. 7 shows specimen B_1 before and after deformation.

3.2. Microstructural

The structure of the deformed specimens is shown in Figs. 8 and 9. In Figs. 8a and b the structure of specimen B_1 in the thinnest zone, as well as near the end of the gauge length, is represented. Figs. 9a and b are corresponding photographs of the deformed B_2 specimen. The high temperature β-phase transforms to martensite on subsequent quenching. This phase etches dark, whilst the α-phase is lightly etched (alcoholic $FeCl_3$ solution was used for etching).

In order to draw comparisons, photomicrographs of annealed and quenched specimens are shown in Figs. 10 to 13. Fig. 10 represents the microstructure of a specimen heated from 650 to 800°C in 40 min, and held at this temperature for 10 min. Thus, this specimen was heated up in an identical manner to the tensile specimens. Figs. 11 to 13 represent the microstructures of speci-

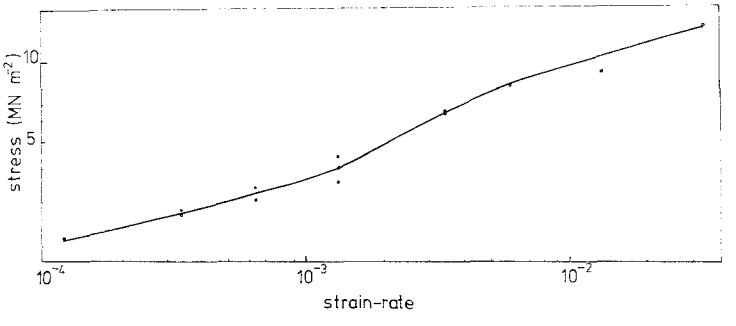

Figure 4 σ-$\dot{\epsilon}$ curve obtained following the Dunlop and Taplin method. Strain-rate in sec^{-1}.

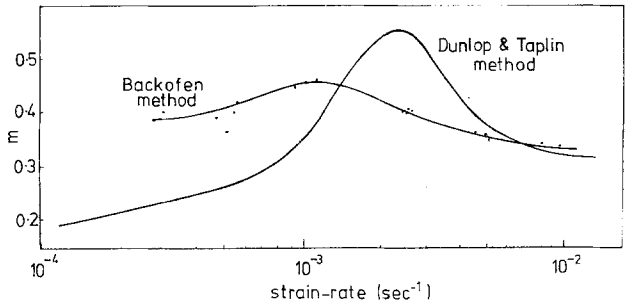

Figure 5 m-$\dot{\epsilon}$ curve obtained following the two described methods.

TABLE I

Specimen no.	Cross-head speed (mm min^{-1})	$\dot{\epsilon}$ initial (sec^{-1})	Deformation time (h)	Measured % elongation of the 10 mm gauge length
B_1	2	3.3×10^{-3}	1	1020
B_2	0.2	3.3×10^{-4}	4	350

Note: the measured % elongation of specimen B_2 is about half the maximal attainable extension.

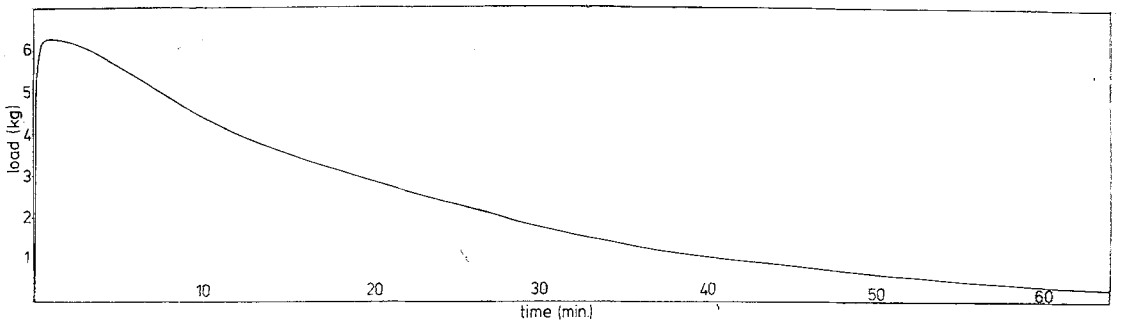

Figure 6 Representation of the recorded load-time curve of the tensile test on specimen B_1.

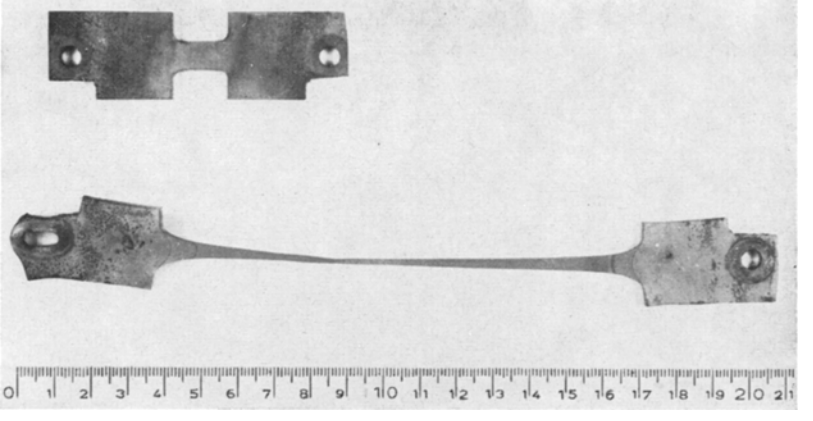

Figure 7 Tensile specimen before and after 1020% deformation at constant cross-head speed. The initial strain rate was 3.3×10^{-3} sec^{-1}.

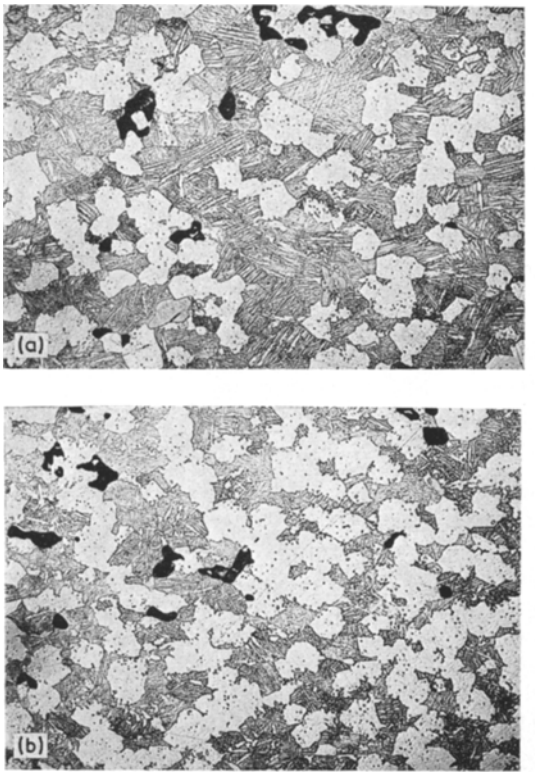

Figure 8 (a) Structure of the alloy deformed at an initial strain-rate of 3.3×10^{-3} sec^{-1}. Deformation time, 64 min; % elongation, 1020; photograph of the central part of the gauge (taken at cm 11 on Fig. 7) ($\times$175). (b) Microphotograph of the same deformed specimen as in (a), taken near the extensity of the gauge length (taken at cm 5 on Fig. 7) ($\times$ 175).

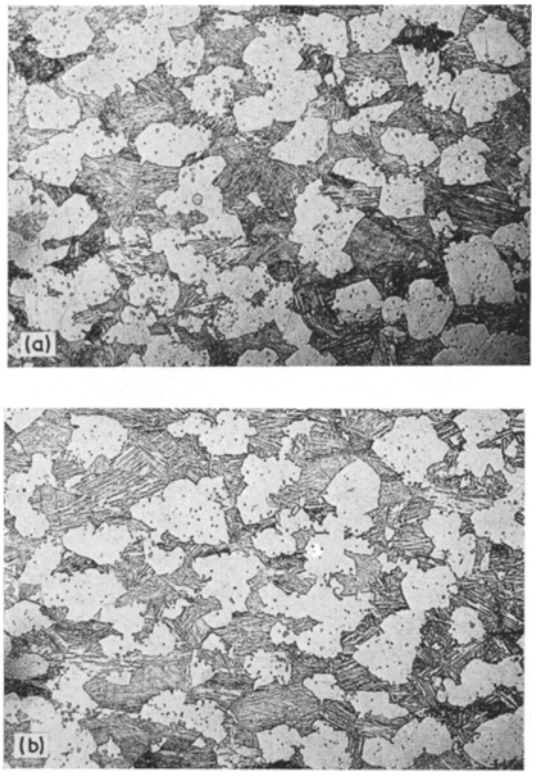

Figure 9 (a) Structure of specimen B_2 deformed at an initial strain-rate of 3.3×10^{-4} sec^{-1}. The time of deformation amounts to 4 h. View of the area with smallest cross-section ($\times$ 175). (b) Microphotograph near the extensity of the gauge length of the same specimen of (a), namely B_2 ($\times$ 175).

mens annealed at 800°C for 50, 110 and 290 min. As such, the time of annealing was equal to the heating up and deformation time of the various tensile specimens.

All photographs are taken at the same magnification, namely $\times$ 175, and with the tensile axis, if any, parallel to the longer edge of the picture.

It follows from Figs. 8 to 13 and Table II that grain coarsening has occurred. These also show the enhanced grain growth owing to the deformation process.

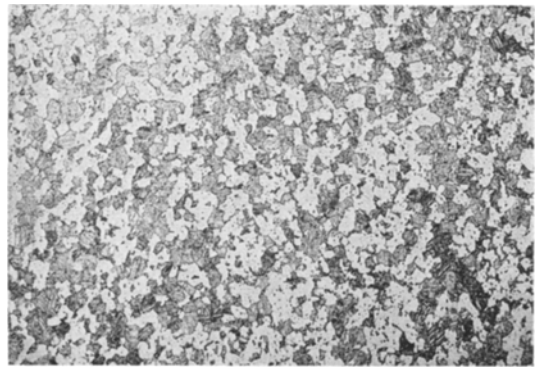

Figure 10 Microstructure of the CDA-619 alloy after heating to 800°C for a period of 50 min in an identical way as the tensile specimen ($\times$ 175).

4. Discussion

Most work on superplasticity has been performed using the rate-change method. By comparing the m-$\dot{\epsilon}$ curve determined by this method with that determined from the slopes of the σ-$\dot{\epsilon}$ curves, it follows that the curve determined by the second or Dunlop-and-Taplin method, has a higher maximum, although at extreme strain-rates, the values so determined are

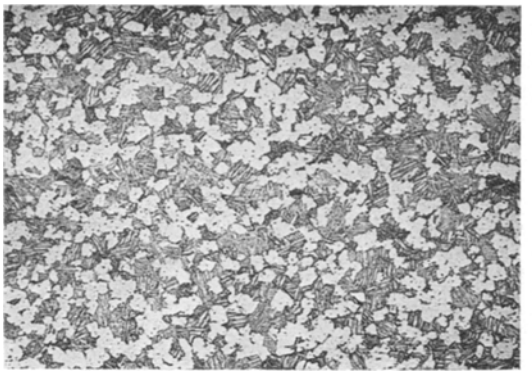

Figure 11 Microstructure of the alloy after 50 min annealing at 800°C and water-quenching (× 175).

Figure 12 Microstructure of the alloy after 110 min annealing at 800°C and water-quenching (× 175).

lower than those obtained by the former or Backofen method. These facts have also been reported by Dunlop and Taplin [9] although the second finding has not been stressed. They supposed that necking, as suggested by Morrison [10], affected this behaviour.

Although necking exists and is an essential phenomenon of superplastic tensile testing [11, 12], it seems also that the following considerations play an important role in explaining the above mentioned behaviour.

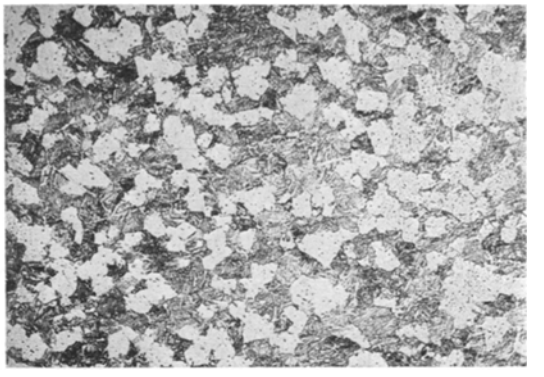

Figure 13 Microstructure of the alloy after 290 min annealing at 800°C and water-quenching (× 175).

1. In analogy to creep experiments in which the deformation conditions are changed during testing, primitive defect structures (such as matrix and grain-boundary dislocation densities) are not constant during superplastic deformation, as suggested by Hedworth and Stowell [13].

2. The variation of the strain-rate sensitivity index must be small over each strain-rate interval covered by the rate-change test, as has been deduced by Backofen *et al* [2]. This is not the fact, as can be seen from Fig. 5, where m determined with the Dunlop and Taplin method, is shown to vary significantly with strain-rate; we consider this method to be the better one, as will be stressed later.

3. The initial microstructure is also continuously changing owing to grain growth. As the flow stress is also dependent on the grain size, it follows that, if grain growth is not taken into account, errors will arise. Indeed, if we put $\sigma = f(\epsilon, \dot{\epsilon}, T, \bar{L})$ or, in the differential form, at constant temperature, $d(\log \sigma) = \gamma d(\log \epsilon) + m\, d(\log \dot{\epsilon}) + \alpha d\bar{L}$, according to Baudelet and

TABLE II Mean intercept length L of the β-grains after various conditions of deformation and/or annealing $\left(L = \frac{\text{Sum of the length of the intercepts of } \beta\text{-grains parallel to the tensile axis}}{\text{number of } \beta\text{-grains}}\right)$

	Tensile specimens (central part of the gauge, parallel to the tensile axis) (heated from 650 to 800°C in 40 min, then at 800°C)	Annealed specimens (at 800°C)
50 min	8.5 μm (Fig. 10)	11 μm (Fig. 11)
1 h + 50 min	30 μm (1020% deformation; Fig. 8a)	15.5 μm (Fig. 12)
4 h + 50 min	34 μm (350% deformation; Fig. 9a)	19 μm (Fig. 13)

Suery [14], it becomes clear that a contribution to the increase of the flow stress is caused by grain growth. If grain growth is neglected, a greater value of m would be computed on the basis of the latter equation.

Yet, by determining m from Equation 1, a somewhat smaller value of m is obtained if the maximum load value, P_A, is reached after roughly 5% additional straining, subsequent to the change of cross-head speed. This follows from the fact that both P_A and P_B are increased by the same amount, relative to the load which would have been recorded without grain coarsening. A rough calculation indicates that the decrease in m is of the order of 0.020 for this alloy.

As has been shown, grain growth is a phenomenon induced by the superplastic deformation of this aluminium-bronze containing iron. White streaks of the α-phase are visible in the microstructures of both the deformed specimens (Figs. 8 and 9) and the annealed ones (Figs. 10 to 13); these are owing to a transformation of β to α in the temperature range between 800 and 600°C. However, the proportion of α-phase to β-phase is not altered, or only slightly so, by quenching, therefore we can assume that no growth or shrinkage of the grains has occurred during quenching. This evidence of fairly large grain growth is not in close agreement with the work on the same alloy reported by Dunlop and Taplin [9, 15].

Watts and Stowell [16] also established grain growth in the superplastic Al-33 wt % Cu alloy. They stressed that the important factor governing the grain size of the homogenized material was the time interval at the high temperature and not the strain. Cutler and Edington [17] reported on the phenomenon of grain growth as well. They found that for the Sn-38 wt % Pb alloy, the grain size had doubled. It was only caused by superplastic deformation with the following testing conditions: temperature 160°C, $m = 0.7$ and elongation 500%, obtained after 15 min annealing and 20 min straining. Baudelet and Suery [14], however, claimed that in this alloy deformation at room temperature did not cause grain growth. Finally, grain coarsening has also been seen for the industrial Cu-40 wt % Zn alloy [18] with a grain size of about 3 μm. Grain growth was much more pronounced at lower strain-rates, just as in the present investigation. However, Sagat *et al* did not mention if this coarsening was owing to thermal activation and/or the superplastic deformation process.

An approach to the phenomenon of grain growth by the deformation process itself has been given by Bäro [19]. Supposing that grain-boundary sliding is accompanied by dislocation gliding, he concludes that the dislocations must not interact in the grains themselves, but must be caught at the grain boundaries, where they cause stress concentrations. This produces a stress-induced diffusion, which largely enhances the mobility of the grain boundaries, by which grains will grow.

In the recent model of Ashby and Verrall [20] grain growth is not excluded. Superplastic material deforms by grain-boundary sliding, accommodation at edges being accomplished by diffusive processes. The work done by straining the material drives several irreversible processes; grain growth could be one of these for the present alloy.

5. Conclusions

1. In the case of Cu-9.5 wt % Al-4wt % Fe alloy, grain growth is induced by the superplastic deformation process itself. Work is currently in progress to reveal details of the correlation between grain growth and the cavity formation on the one hand and the strain-rate and the deformation mechanisms on the other hand.

2. The method proposed by Dunlop and Taplin to evaluate the superplastic properties seems to be the better one, because the same structure – the initial structure – is tested.

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