JOURNAL OF MATERIALS SCIENCE 8 (1973) 363–369

Flow stress and microstructure in superplastic 60/40 brass

MICHEL SUERY, BERNARD BAUDELET
Laboratoire de Physique du Solide, Laboratoire associé au C.N.R.S. no. 155 Ecole des Mines, Nancy, France. Faculté des Sciences, Metz, France

Stress-strain curves have been obtained for a superplastically deformed industrial 60/40 brass tested in tension under constant true strain-rate conditions at 600° C. This alloy produced by extrusion shows a fibrous structure with elongated grains. It is shown that the flow stress is influenced by two microstructural factors, i.e. the change of phase shape which becomes approximately equiaxed and the variation of the phase size during superplastic deformation. These factors are of different relative importance according to the strain-rate.

1. Introduction

The term superplasticity is currently employed to describe the behaviour of many metals and alloys with very fine grains which exhibit abnormal neck-free elongations when tested under low stresses in tension at temperatures greater than about half the melting point [1]. As long as the deformation remains uniform, the flow stress depends on the true strain, the true strain-rate, the test temperature and the microstructure of the alloy. In the superplastic range, it is shown that the strain-hardening coefficient can be considered as negligible [2]. Most of the currently published research has been concerned with the mechanical properties of these materials in relation with the temperature, the strain-rate and the grain or phase size without taking their shape into account. The materials are generally produced by rolling or extrusion and these processes give them a fibrous structure with elongated grains. After deformation in the superplastic range, this structure becomes equiaxed [3]. A recent theoretical study on the deformation mechanisms of superplasticity has shown that the dependence of stress and grain size on strain-rate should be different in materials having fine elongated grain structures and in materials having fine equiaxed grain structures [4]. The aim of the present work is to study the influence of grain shape and size on the flow stress during superplastic deformation of an industrial 60/40 brass [5]. This alloy contains two phases α and β up to about 760°C [6]. The only researches on this alloy have shown that it is characterized by high values of the strain-rate sensitivity coefficient m [7] ($m = (\delta \ln \sigma)/\delta \ln \dot{\epsilon}$, σ is the true stress, $\dot{\epsilon}$ the true strain-rate) and it was found to exhibit elongations of about 500% [8].

2. Experimental

The 60/40 brass was prepared industrially by the "Etablissements Reboud-Roche". It was extruded at 600°C to rods of 6 or 10 mm in diameter. Tensile specimens of 25 mm gauge-length and 3 or 6 mm in diameter, were machined from these rods. Tensile tests were performed with an Instron testing machine under an argon atmosphere. A furnace with a constant temperature zone ($\pm$ 2°C) was used. For each test, the temperature was controlled in the furnace and was reproducible with an error of less than 2°C. After inserting the specimen, the furnace was held at the test temperature for 45 min to make the temperature distribution uniform.

In order to characterize the superplastic range of this alloy, the strain-rate sensitivity parameter m was determined at 490, 550, 600, 700 and 800°C using the velocity change tests [9]. The variation of flow stress with strain, and the influence of grain shape and size were studied at 600°C using nominally constant true strain-rates of 4×10^{-1}, 10^{-1}, 4×10^{-2}, 10^{-2} min^{-1} with an accuracy of $\pm 0.5\%$. The test was stopped after an engineering deformation of 100%, after which the diameter all along the specimen remains constant within $\pm 3\%$. The apparatus has been described elsewhere [2].

After deformation, samples for metallographic examination were prepared by conventional techniques. In order to reveal the microstructure of this alloy, a solution of 5 cc $FeCl_3$, 5 cc HCl and 10 cc H_2O was used. To permit the identification and the determination of the mean phase diameter, a solution of 50 g NH_4Cl in 250 cc H_2O, 33 g CuCl and 85 cc NH_4OH was used. Etching times ranged from 3 to 5 sec. This etching reveals the β-phase in dark contrast.

The average transverse and longitudinal phase diameter were obtained during the deformation using the lineal intercept method. On longitudinal cuts, lines were drawn in both transverse and longitudinal directions and the mean phase diameter is obtained by averaging not less than 100 interceptions on each specimen.

3. Results

3.1. Mechanical

Fig. 1 shows the variation of the strain-rate sensitivity parameter m, with true strain-rate at different temperatures. We note that a temperature increment leads to an increase of the m-value. These curves confirm the existence of a phase transformation between 700 and 800°C [6]. Indeed at 800°C, the 60/40 brass is single phase and the m-values less than 0.2 can be applied to a coarse-grain material. These curves justify the study at 600°C in the strain-rate range of 10^{-2} to 4×10^{-1} min^{-1}; in this range, the strain-rate sensitivity parameter is greater than 0.3. This study is however very qualitative because the m-determination remains open to discussion [10]: it may be supposed that the elongation is uniform and the structure remains constant (not only grain shape and size but also all defect distribution in the material).

Fig. 2 shows the variation of flow stress with true strain at 600°C for constant true strain-rate tensile tests. The curves are different according to the strain-rate. For high values of the strain-rate, the flow stress decreases with strain. On the contrary, for low values of the strain-rate, the flow stress increases and this increase becomes larger as the strain-rate decreases.

3.2. Microstructural

The as-extruded structure of the material homogenized and held for 45 min at 600°C is shown in Fig. 3a. There is a marked directionality of structure with phases elongated along the extrusion direction. It is difficult to study the microstructural change by considering both the phases: this structure may be considered as α-grains in a β-matrix and it may be meaningless to characterize this structure change by a study of the β-phase.

Fig. 4 shows the variation of the average longitudinal and transverse α-phase diameter with strain for two different strain-rates. We note that the phases lengthen at first parallel to the tensile axis. This lengthening is characterized

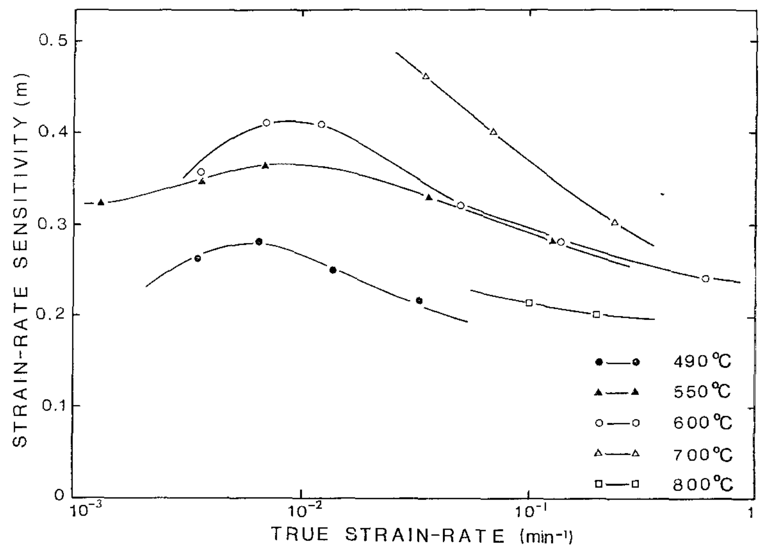

Figure 1 Strain-rate sensitivity coefficient against true strain-rate at different temperatures.

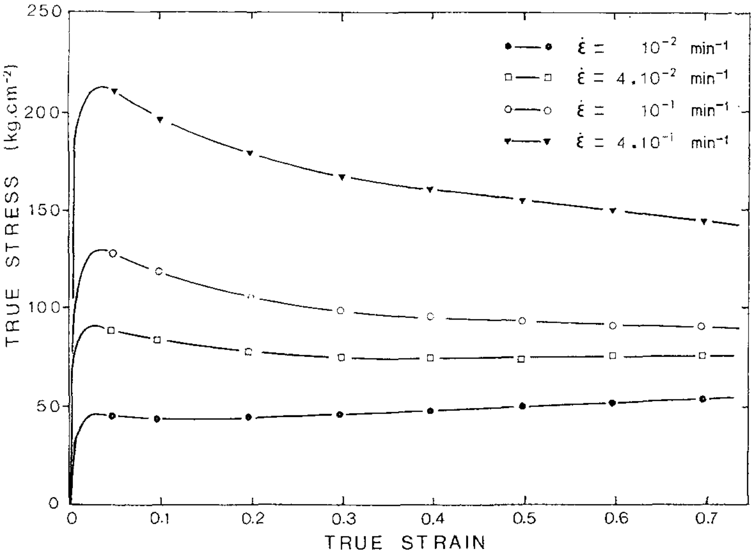

Figure 2 True stress against true strain at constant true strain-rates and at 600°C.

by a $D_{\parallel}$ increase and a $D_{\perp}$ decrease; the ratio $D_{\parallel}/D_{\perp}$ shows a maximum which moves to higher strains at higher strain-rates. Then the structure becomes approximately equiaxed. It is to be noted that $D_{\parallel}/D_{\perp}$ does not lead to 1, which is a characteristic of true equiaxed structures, but to a higher value approximately equal to 1.2. That agrees with the results obtained in the same conditions with the AlCu eutectic alloy [11]. For the lowest true strain-rates, the average α-phase diameter increases and the ratio $D_{\parallel}/D_{\perp}$ remains approximately constant.

This phase diameter increase is not only due to time with which the sample is kept at the test temperature, it is also aided by deformation. Indeed tensile specimens were at first deformed at low strain-rate ($\dot{\epsilon} = 4.2 \times 10^{-3}$ min^{-1}) to 25% true strain. This is necessary to develop an approximately equiaxed structure (D = 21.2 μm). They were then either deformed to 70% true strain at the same true strain-rate, or kept at the same temperature during the same time. The final α-phase diameters were 34 μm and 27 μm respectively.

In Fig. 5 which is a plot $D_{\parallel f}$ and $D_{\perp f}$ (the values of $D_{\parallel}$ and $D_{\perp}$ after 70% true strain) versus true strain-rate, we note than $D_{\parallel f}$ and $D_{\perp f}$ are decreasing functions of strain-rate; we particularly note that the final phase diameter are lower than the initial phase diameter $D_{\parallel i}$ and $D_{\perp i}$ for high strain-rates. All these microstructural results are shown in Fig. 3.

This microstructural study clearly indicates the existence of two factors; on one hand, the evolution of the α-phase shape which becomes approximately equiaxed and on the other hand the phase increase which is an increasing function of the deformation in the superplastic range.

4. Discussion

The α-phases lengthen at first parallel to the tensile axis and lead then to an approximately equiaxed structure; thus it may be supposed that this shape change is performed by a division of the phases and it is proposed a mechanism entailing dislocation cell structure formation to explain this. Indeed, when the grain length exceeds the thickness, the expected dislocation cell size which is approximately equiaxed [12] may be smaller than the length. With continued straining, the subgrains which correspond to the cell boundaries become gradually grain boundaries [4], and along these boundaries the separation may start. The ratio $D_{\parallel}/D_{\perp}$ does not lead to 1, and this may be explained by the fact that the calculation on a evolutive structure results in an average. Studies by transmission electron microscopy are undertaken to confirm this cell structure mechanism.

A microstructural coarsening occurs during deformation. The deformation acting with subgrain rotation [13], facilitates the microstructural coarsening by assembly of the α-phases and by mass-diffusion along the phase-interfaces. This

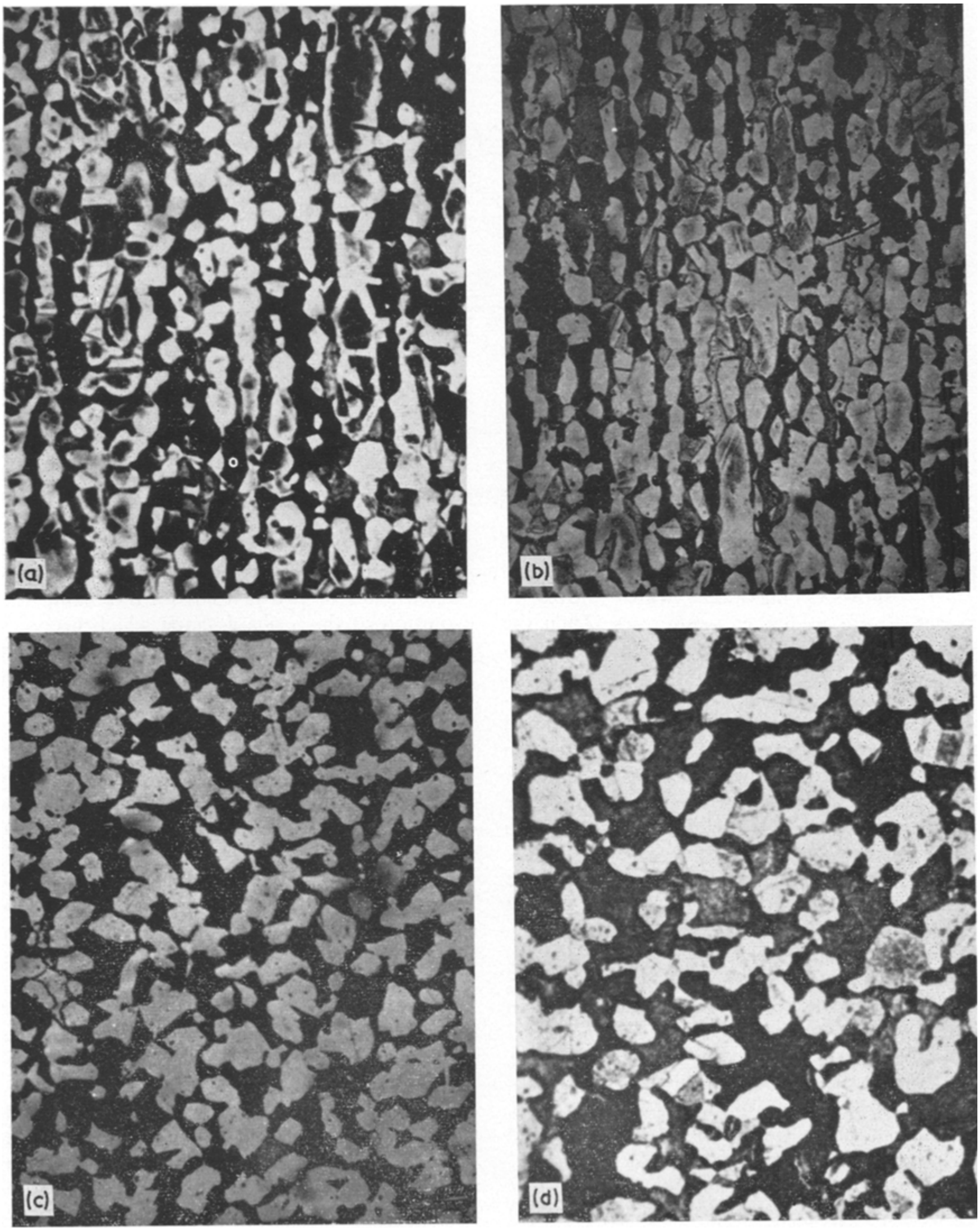

Figure 3 Microstructure of homogenized material, superplastically deformed at constant true strain-rate. Temperature 600°C. Tensile axis vertical (× 275). (a) $\epsilon = 0$; (b) $\epsilon = 5\%$, $\dot{\epsilon} = 10^{-2}\ \text{min}^{-1}$ (the ratio $D_{\parallel}/D_{\perp}$ is maximum); (c) $\epsilon = 70\%$, $\dot{\epsilon} = 10^{-1}\ \text{min}^{-1}$; (d) $\epsilon = 70\%$, $\dot{\epsilon} = 10^{-2}\ \text{min}^{-1}$.

microstructural change during superplastic deformation influences the mechanical properties of the material. Then, the flow stress during constant true strain-rate tensile tests is influenced

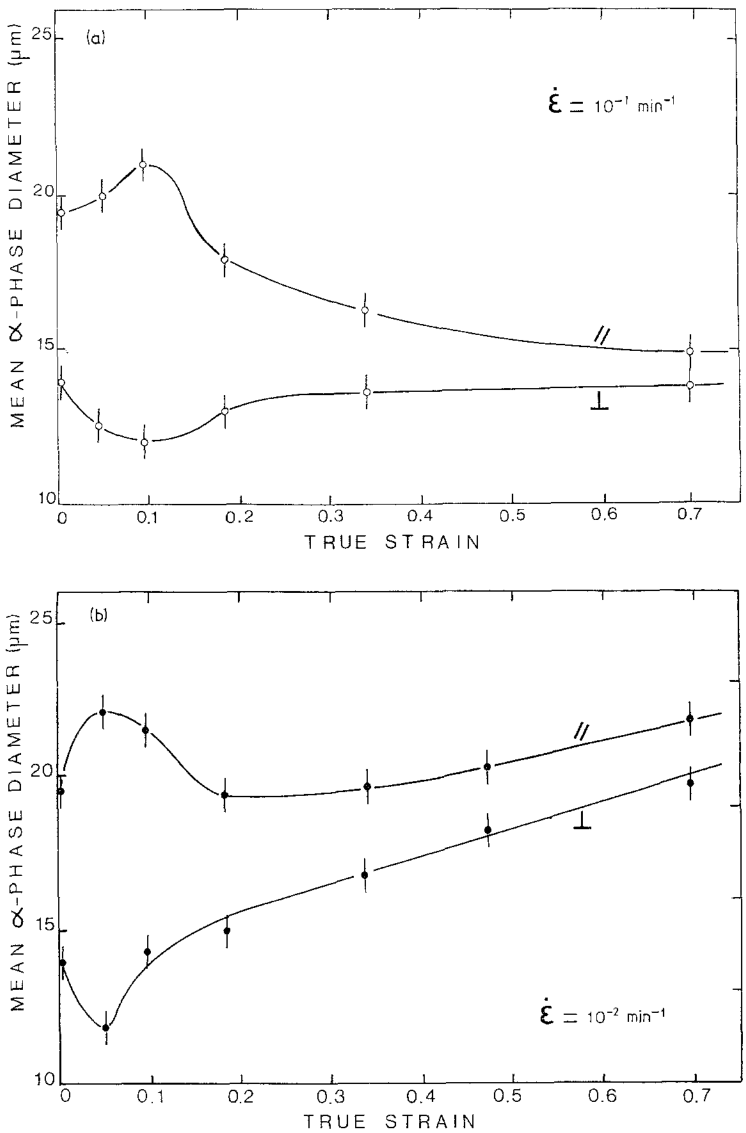

Figure 4 Average longitudinal and transverse α-phase diameter against strain at constant true strain-rate. Temperature 600° C. (a) $\dot{\epsilon} = 10^{-1}$ min^{-1}; (b) $\dot{\epsilon} = 10^{-2}$ min^{-1}.

by three factors. 1. The strain-rate through the medium of the strain-rate sensitivity parameter m. 2. The α-phase shape change which becomes approximately equiaxed and which allows an easier grain-boundary sliding. This process leads to a decreasing stress [4]. 3. The influence of the phase size according to a function of the type $\sigma = AD^b$ where A is a constant and $b = ma$ [14]. Then, as the strain-rate sensitivity coefficient becomes higher, the influence of the grain size on the flow stress increases. These last two factors are of relatively different importance according to the strain-rate.

For high values of the strain-rate, the phase diameter decrease (Fig. 5) associated with their shape change leads to a strong decrease of the flow stress (Fig. 2). It is to be noted that the flow stress shows a maximum which corresponds approximately to the $D_{\parallel}/D_{\perp}$ maximum (Fig. 4). At low strain-rate, the phase increase is the most important factor and the true stress thus increases with strain, whereas at intermediate

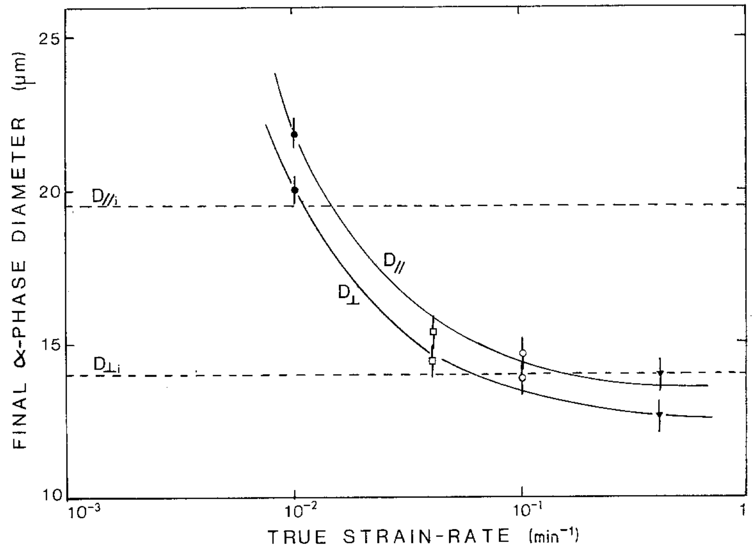

Figure 5 Variation of the average longitudinal and transverse α-phase diameter with strain-rate after 70% true strain. Temperature 600° C.

strain-rate, both the factors can cancel each other and the stress remains approximately constant. The maximum in the stress-strain curves is all the more marked as the phase increase is lower. We observe that this maximum does not reappear if we redeform a specimen which was previously deformed to a strain corresponding to an approximately equiaxed structure.

The stress-strain curves are comparable with those obtained on the superplastic aluminium bronze tested in tension at constant cross-head velocity [15]. These authors ascribe the stress decrease with strain to a non-uniform elongation, the maximum corresponding to the onset of plastic instability. For our experiments the diameter all along the specimen remains constant within ±3% after 70% true strain. It is likely that the result obtained with the aluminium bronze arises from both non-uniform elongation and structure change of the material which was obtained by rolling, and showed a fibrous structure with elongated grains. All the results agree with those obtained by the authors on the eutectic alloy Pb–Sn [2] and the industrial solder Cu7 wt% P alloy [16] which were both extruded.

On the PbSn eutectic tested in creep under constant stress and in tension under constant true strain-rate conditions, a transition has been observed which can be attributed to structure change. Then, if grain size is constant, the strain-rate remains constant with strain whatever the stress, or the flow stress remains constant with strain whatever the strain-rate.

A similar result has been observed on the industrial solder Cu7 wt% P alloy tested in tension under constant true strain-rate. The stress increase due to microstructural coarsening and to a high value of the strain-rate sensitivity coefficient is more important than the stress decrease due to structure change. Then no maximum is observed in the stress-strain curves.

5. Conclusions

(a) The superplastic behaviour range of an industrial 60/40 brass has been determined.

(b) The stress-strain curves are influenced by three factors. 1. The strain-rate. 2. The phase shape change which becomes approximately equiaxed. This change has been attributed to a dislocation cell structure formation at the beginning of the elongation. 3. The variation of the phase size and, in particular, the microstructural coarsening aided by the deformation. This coarsening may be attributed to the assembly of phases of the same nature, caused by their rotation during the deformation and by mass-diffusion along the phase interfaces.

Acknowledgements

We gratefully acknowledge the help of the "Etablissements Reboud-Roche" who supplied this alloy and should also like to thank the

"Centre National de la Recherche Scientifique" – Action Thématique Programmée: "Propriétés Mécaniques des Solides" for their financial support.

References

1. R. H. JOHNSON, *Metall. Rev.* **146** (1970) 115.
2. B. BAUDELET and M. SUERY, *J. Mater. Sci.* **7** (1972) 512.
3. C. M. PARKER, Ph.D. Thesis, Standford University, California (1967).
4. H. W. HAYDEN, S. FLOREEN, and P. D. GOODELL, *Met. Trans.* **3** (1972) 833.
5. A. A. PRESNYAKOV and G. V. STARIKOVA, *Fiz. Metal. Metaloved* **12** (1961) 873.
6. M. HANSEN, "Constitution of Binary Alloys" (McGraw-Hill Book Company, New York, Toronto, London, 1958), p. 650.
7. D. M. R. TAPLIN, G. L. DUNLOP, S. SAGAT, and R. H. JOHNSON, "An Interamerican Approach for the Seventies Materials Technology", **I** (1970) 253.
8. M. S. STAMFORD, *Copper* **3** (1969) 10.
9. W. A. BACKOFEN, I. R. TURNER, and D. H. AVERY, *Trans. ASM Quart.* **57** (1964) 980.
10. J. HEDWORTH and M. J. STOWELL, *J. Mater. Sci.* **6** (1971) 1061.
11. B. M. WATTS and M. J. STOWELL, *ibid* **6** (1971) 228.
12. J. P. HIRTH and J. LOTHE, "Theory of Dislocations", (McGraw-Hill Book Company, New York, London, Sydney, 1968), p. 640.
13. D. J. DINGLEY, Proceedings of the third Annual Scanning Electron Microscope Symposium, Chicago (1970), p. 329.
14. T. H. ALDEN, *J. Aust. Inst. Metals* **14** (1969) 207.
15. G. L. DUNLOP and D. M. R. TAPLIN, *J. Mater. Sci.* **7** (1972) 84.
16. G. HERRIOT, M. SUERY, and B. BAUDELET, *Scripta Met.* **6** (1972) 657.

Received 24 July and accepted 3 August 1972.