3. C. Y. CHIN, L. G. VAN UITERT, M. L. GREEN and G. ZYDZIK, *Scripta Met.* **6** (1972) 503.
4. C. A. BROOKES, R. P. BURNAND and J. E. MORGAN, *J. Mater. Sci.* **10** (1975) 2171.
5. E. AERTS, S. AMELINCKX and W. DEKEYSER, *Acta Met.* **7** (1959) 29.
6. W. H. VAUGHAN and J. W. DAVISSON, *ibid.* **6** (1958) 554.
7. M. PERINOVÁ and A. A. URUSOVSKAIA, *Czechoslov. J. Phys.* **B16** (1966) 791.
8. D. N. FRENCH and D. A. THOMAS, *Trans Met. Sco. AIME* **233** (1965) 950.
9. M. V. SWAIN and B. R. LAWN, *Phys. Stat. Sol.* **35** (1969) 909.
10. E. S. BERKOVICH, *Zavodskaya Laboratoriya* **10** (1963) 1250.

Received 28 February
and accepted 7 June 1978

Yu. S. BOYARSKAYA
D. Z. GRABKO
E. I. PURICH
Institute of Applied Physics,
Moldavian Academy of Sciences,
Kishinev-28, USSR

On creep ductility of two dilute zinc alloys

The effect of small additions of Al and Cu on slow strain-rate tensile ductility of Zn at elevated temperatures are reported. Two dilute alloys, high purity Zn (99.99% pure) with addition of 0.16 at.% Al (Alloy A) or 0.14 at.% Cu (Alloy C) have been examined. The study assumes significance in view of the fact that Al is a solute in Zn with a distribution coefficient K less than 1 (about 0.25) while for the Cu solute, K is greater than 1. The work of Aust *et al.* [1] has shown that Al atoms experience attractive interactions with grain boundaries in polycrystalline Zn, tend to get adsorbed at the grain boundaries and cause grain-boundary hardening. On the other hand, Cu atoms experience repulsive interactions, stay away from grain boundaries in Zn and give rise to grain-boundary softening. Our interest was to investigate the effect of this distinct difference in solute distribution on the creep ductility of Zn.

Standard Hounsfield tensile specimens of gauge diameter 4.7 mm and gauge length 16 mm were machined from extruded rods of 10 mm diameter in each case. The specimens were chemically polished in a solution containing 32 g chromic acid and 4 g sodium sulphate in 100 ml water to remove the deformed layer. The polished specimens were annealed at 250° C for 1 h to yield average intercept grain sizes of 80 and 120 μm in Alloys A and C respectively. The specimens were tested in tension at 23° C (0.41 T_m, where T_m is the melting temperature in degree Kelvin), 67° C (0.49 T_m), 100° C (0.54 T_m), 150° C (0.61 T_m) and 200° C (0.68 T_m) on a modified Hounsfield tensometer [2] at a nominal strain-rate of 3×10^6 sec^{-1}. The fractured specimens were rapidly cooled by a jet of cold air in order to retain the microstructure developed at the test temperature. The tested specimens were longitudinally sectioned and polished for metallography. The extent of creep cavitation in the fractured samples was assessed by estimating the percent volume fraction (f) of cavities, using the formula [3]:

$$f = \frac{8}{3\pi} \cdot \frac{N_L^2}{N_A} \times 100 \qquad (1)$$

where N_L and N_A are respectively the number of cavities per unit length and unit area. Measurements of N_L and N_A were made at × 200 magnification and the entire area covering the gauge length of the samples was scanned for the purpose.

The variation with test temperature of percent elongation at fracture of the two alloys is shown in Fig. 1. The ductility of alloy C remains more or less the same over the entire temperature range. On the other hand, the ductility of alloy A increases sharply at temperatures above 67° C, attains a maximum value at 150° C and then again decreases to a lower value at 200° C. At any test temperature the ductility of Alloy A is higher than that of Alloy C and at 150° C Alloy A exhibits three times as large a percentage elongation as observed in Alloy C. There is some difference in the grain sizes of the above two alloys as mentioned above (see also Fig. 2) but the vast difference in ductility cannot be attributed to difference in grain size alone. Furthermore, similar ductility differences in these two alloys have been observed by Roth *et al.* [4] who tested specimens of equal grain sizes of these alloys. We shall

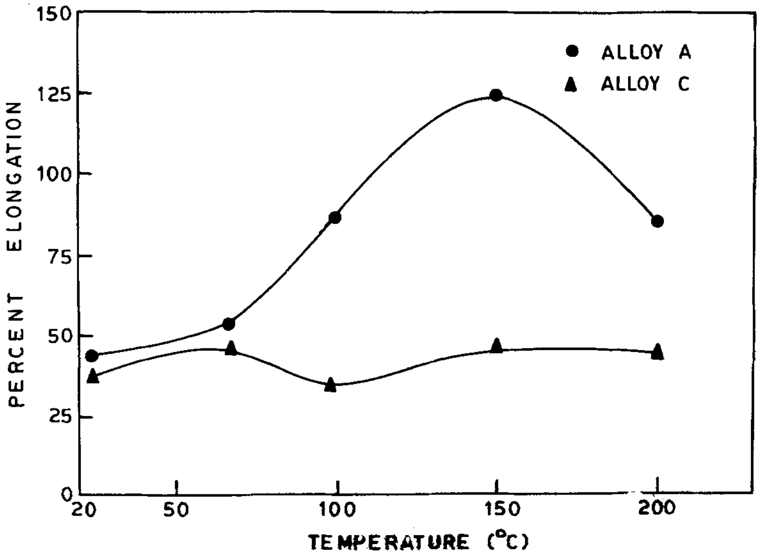

Figure 1 Variation of percent tensile elongation with temperature (nominal strain-rate: 3×10^{-6} sec^{-1}).

attempt an explanation for the observed differences in the ductility behaviour on the basis of metallographic observations.

Fig. 3 shows optical micrographs of longitudinal sections of fractured specimens tested at 67° C. There is a striking difference between Alloy A and Alloy C. Recrystallized grains in the vicinity of grain boundaries are clearly seen in Alloy A (Fig. 3a). In contrast, while no recrystallized grains are discernible, marked boundary migration is obvious in the micrograph of Alloy C (Fig. 3b).

The micrographs of Fig. 4 pertain to specimens fractured at 150° C. Contrasting features between tested specimens of Alloy C and Alloy A pointed out above, are further accentuated in the micrographs of Fig. 4. Recrystallized grains appear extensively in Alloy A, particularly in the vicinity of the grain boundaries (Fig. 4a) while no evidence of recrystallization but essentially of grain-boundary migration (Fig. 4b and c) is seen in Alloy C.

Micrographs of specimens of Alloy A and Alloy C tested at 200° C which indicate great similarity are shown in Fig. 5. Appearance of recrystallized grains is not a noticeable feature in *either* case.

The effect of test temperature on volume fraction of cavities in fractured samples is given in Table I. It is noteworthy that up to 100° C, volume fractions of cavities in the two cases are comparable. At 150° C, Alloy C develops a cavity volume fraction twice as large as alloy A. At 200° C there is a remarkable rise in development of creep cavities in Alloy A with volume fraction exceeding that measured for Alloy C.

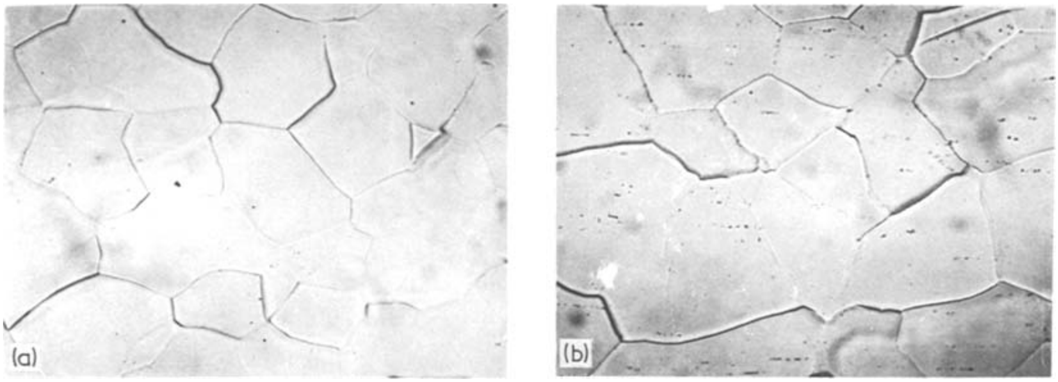

Figure 2 (a) Annealed Zn–Al alloy (× 180), (b) annealed Zn–Cu alloy (× 180).

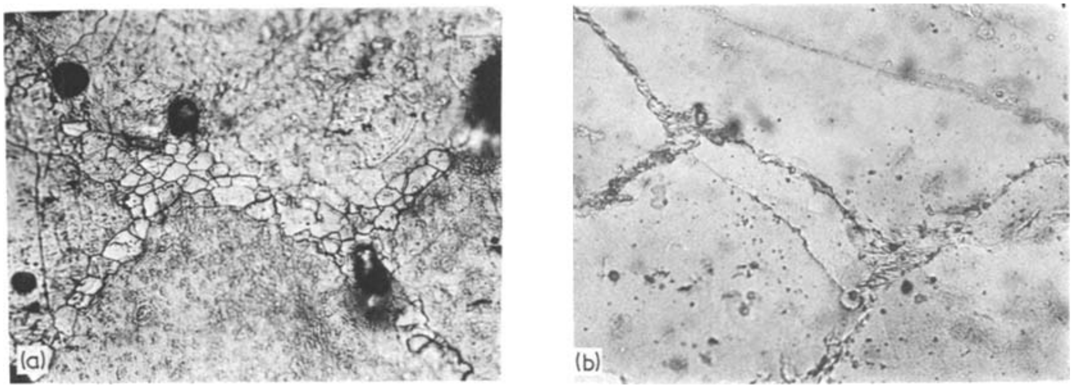

Figure 3 (a) Zn–Al alloy tested at 67° C (X 460), (b) Zn–Cu alloy tested at 67° C (X 460).

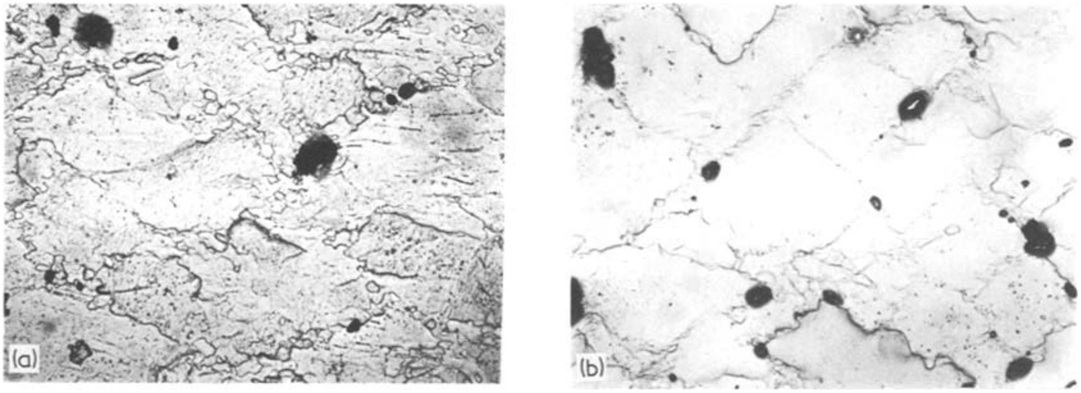

Figure 4 (a) Zn–Al alloy tested at 150° C (X 180), (b) Zn–Cu alloy tested at 150° C (X 180), (c) Zn–Cu alloy tested at 150° C (X 925).

It has been established earlier [4] through grain growth studies, under purely thermal conditions, that the extent of grain-boundary migration is very small in Alloy A in comparison to Alloy C up to 390° C. This has been attributed to Al atom adsorption at grain boundaries. Stresses developed at grain-boundary irregularities consequent to grain-boundary sliding are normally relieved by one or more of the three processes, namely intragranular deformation, grain-boundary migration and intergranular cavitation. In Alloy C the effect of added plasticity resulting from the higher test temperature is counterbalanced by increasing propensity for intergranular cracking (Table I) and consequently ductility remains nearly the same over the temperature range investigated. The sudden rise in the level of cavitation at 150° C is due to development of a diamond grain morphology, aided by grain-boundary migration, in which the majority of boundaries is oriented at 45° from the stress axis (Fig. 4b and Fig. 6). This aspect has been discussed in detail earlier by Singh *et al.* [5]. The striking observation in Alloy A is in regard to recrystallized grains up to a test temperature of 150° C. There are two possibilities for the origin

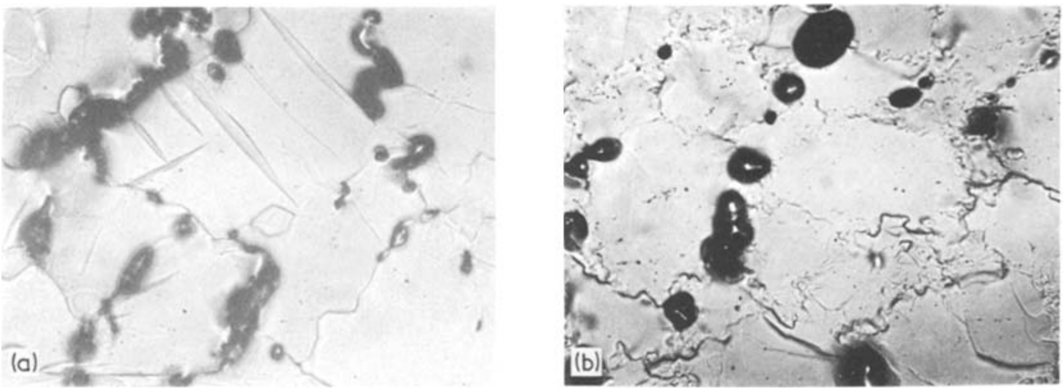

Figure 5 (a) Zn–Al alloy tested at 200° C (X 180), (b) Zn–Cu alloy tested at 200° C (X 180).

of the new grains. One of these is that dynamic recrystallization has taken place *during* the high temperature deformation as an accommodation process. The other possibility is that, due to grossly reduced grain-boundary mobility most likely in Alloy A, intense intragranular deformation occurred which gave rise to the recrystallized grains during subsequent cooling following the test. When large-scale grain-boundary migration can occur, as for instance at test temperature of 200° C, no evidence of recrystallized grains is to be found (Fig. 5) and the micrographs of Alloy A and Alloy C acquire much similarity.

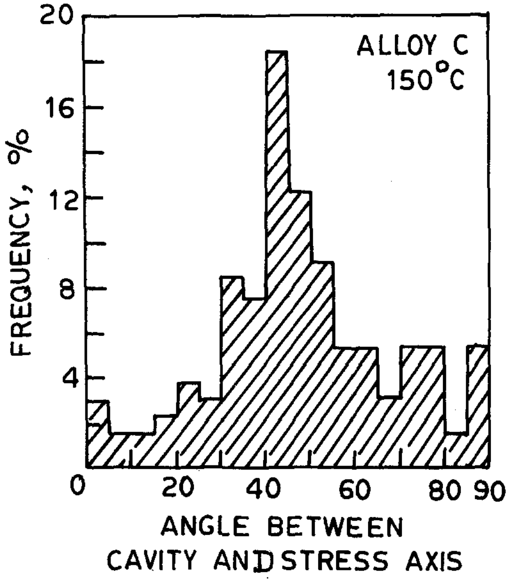

Figure 6 A histogram showing angular distribution of cavities with respect to the stress axis for Alloy C tested in slow tension at 150° C.

TABLE I The effect of test temperature on volume fraction of cavities in fractured samples of Alloys C and A

Test temperature (° C)	Volume fraction of cavities (%)	
	Alloy C	Alloy A
23	0.9	1.3
67	2.3	3.0
100	4.8	4.0
150	11.7	5.0
200	13.3	19.1

The important result of the present work is contained in the observation that under restricted grain-boundary migration in Alloy A, conditions for recrystallization seem to develop, cavitation is inhibited (Table I) and ductility is considerably large when tested at 150° C.

Acknowledgements

The authors are grateful to Professor. T. R. Anantharaman and Professor S. L. Malhotra for encouragement and provision of facilities, and to Professor D. M. R. Taplin and Dr V. V. P. Kutumba Rao for a helpful discussion. The alloys were kindly supplied by the COMINCO Research Centre, Sheridan Park, Canada.

References

1. K. T. AUST, R. E. HANNEMAN, P. NIESSEN and J. H. WESTBROOK, *Acta Metallurgica* 16 (1968) 291.
2. V. V. P. KUTUMBA RAO, D. M. R. TAPLIN and P. RAMA RAO, *Trans. Ind. Inst. Metals* 23 (1970) 61.
3. R. L. FULLMAN, *Trans. Met. Soc. AIME* 197 (1953) 447.
4. K. H. ROTH, R. G. FLECK and D. M. R. TAPLIN, *Mat. Sci. Eng.* 16 (1974) 251.

5. VAKIL SINGH, P. RAMA RAO, G. J. COCKS and D. M. R. TAPLIN, *J. Mater. Sci.* 12 (1977) 373.

Received 17 May
and accepted 17 July 1978.

VAKIL SINGH
P. RAMA RAO
Department of Metallurgical Engineering,
Banaras Hindu University,
Varanasi,
India

Transmission electron microscopy and X-ray analysis of coloured anodized films prepared by ultramicrotome without embedding

Direct investigation of thin anodized and subsequently electrolytically coloured oxide films with a high resolution is only possible if a method is used which is not selective during the decomposition of different materials. During ion-bombardment, this requirement is not fulfilled [1], therefore ion-beam etching, proposed earlier [2], should be avoided in this case. Furthermore, all treatments having an effect on the sample (including embedding [3]) should be avoided to be sure not to influence the colouring agent. A very simple method to provide thin sections is described below, and the interpretation of structures observed in this way is given.

In order to prepare thin sections for direct examination, anodic layers were prepared on 5 mm thick sheets of aluminium. From these sheets 5 mm wide and 15 mm long strips were cut, the strips were trimmed and then cut with the ultramicrotome. The same procedure was carried out in the case of layers built up on bulk material. Blocks with dimensions 5 mm × 5 mm × 15 mm were self-

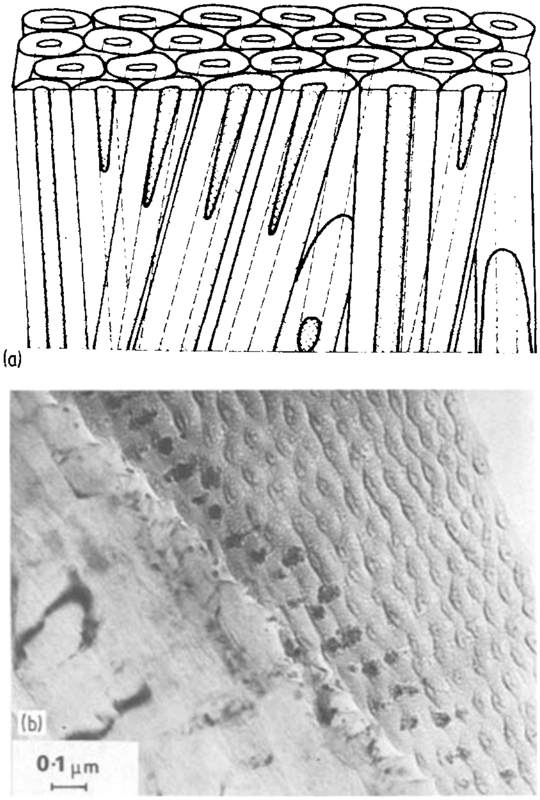

Figure 1 (a) Schematic model of a section. (b) Micrograph of a section inclined to the surface. Anodic layer coloured for 20 sec.

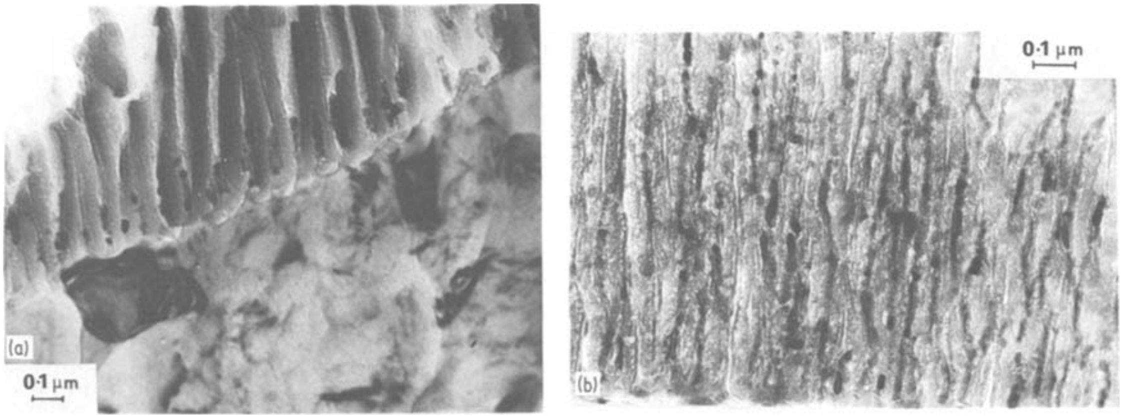

Figure 2 (a) Vertical section of an anodic layer coloured for 20 sec. (b) Vertical section of an anodic layer coloured for 5 min.