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HIGH-TEMPERATURE CREEP OF EQUIAXED Cd-26.5 at % Zn EUTECTIC IN THE SUPERPLASTIC REGIME

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HIGH - TEMPERATURE CREEP OF THE SUPERPLASTIC Cd + 26.5 at.% Zn EUTECTOID

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The temperature and stress dependence of the secondary creep rate of the Cd + 26.5 Zn eutectoid in the superplastic domain were studied in constant-stress compression creep. Experiments were performed in the following ranges of temperature, stress and grain size : $170°C < T < 227°C$, $11 < \sigma < 122$ g/mm^2, $1 < d < 10$ µm.

In all cases secondary creep was established after a strain $\varepsilon \stackrel{\sim}{\sim} 4\%$. The strain rate sensitivity m was measured by stress increments or decrements and by using independent measurements on different samples.

For temperatures higher than 200°C all these techniques yielded the same value for m ($m = 0.49 \pm 0.03$) in the whole investigated range of stresses. For $T = 170°C$ a lower value of m was found ($m = 0.33$).

The activation energy for the creep rate was determined by the conventional Arrhenius plot and by the Dorn temperature-compensated time method, and found equal to $Q \stackrel{\sim}{\sim} 25$ Kcal/mol.

Micrographic examinations were performed on sectioned samples at several stages of deformation. The grain size was found to be identical for various conditions of temperature and stress and very stable with respect to deformation. The stereology of the phases was investigated and their respective role in the deformation ascertained. The experimental results of the creep tests are discussed in relation with the microstructural aspects.

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INTRODUCTION : Superplasticity has been extensively studied in microduplex two-phased alloys and several models have been proposed for the elementary mechanisms leading to the high value of the strain rate sensitivity of the stress, m and to the activation energy, Q, characteristic of the superplastic regime (1). However, it is still not clear whether the two phases act only by preventing grain growth or whether the interphase boundaries play a particular role, different from the one played by grain-boundaries within one phase. Also, practically no attention has been given to the possible role of the topology of the phases and their connectivity.In this respect, two obviously quite different physical situations can arise, according to the volume ratio of the two phases :

i) Phase B is dispersed in unconnected globules within phase A,

ii) Phase A and phase B form two highly connected imbricated networks. Percolation can be expected for a volume ratio of one phase as low as 15% (2), and Cahn (3) has shown that a volume ratio of 20% should lead to a high value of the connectivity.

The two situations (and the intermediate ones) cannot be distinguished merely by looking at micrographs of sections of the sample, even if taken in different planes, and 3-dimensional serial sectioning must be resorted to.

The purpose of the present study is to investigate in this light a superplastic system with a volume ratio of phases such that the highly connected situation ii) may be expected. We chose as a material the eutectic Cd 26.5 at % Zn (17.4 Wt %) where both phases

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are h.c.p. terminal solid solutions. The volume fraction of the Zn-rich phase is 26 % and microhardnesses of the phases are in the ratio 2:1, the Zn-rich phase being harder. The superplasticity of this alloy has been investigated by Merriman (4).

EXPERIMENTAL : The alloy was cast and extruded at 120°C with a 225:1 area reduction. Cylindrical compression specimens (12 mm long and 5 mm dia.) were machined from the extruded rod and annealed at 230°C for 2, 8 or 130 hours.
Constant-stress creep experiments in compression between 201 and 227°C were preferred to more traditional constant cross-head speed experiments in traction for the following reasons :
i) The values of Q and $n = \frac{1}{m} = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma}$ can be obtained more easily and in ~~different well-controlled conditions (stress increment~~ or decrement) which can provide more reliable information.
ii) The samples are easier to prepare and metallographic observations are better performed after deformation on the stockier compression samples.
Some experiments were made in compression at constant cross-head speed on an Instron machine and a few experiments in traction were performed (samples 2 cm long, 2 mm dia.) in the higher m range at T = 210°C to check the superplastic behavior.

Metallography : The observations were performed on longitudinal and transversal sections of the samples. The Cd-rich phase grain boundaries and the phase boundaries were revealed by etching for 15 s at R.T. with the solution : 8 g CrO_3, 2.5 g Na_2SO_4, 25 ml H_2O. The Zn-rich phase appeared on both sections as concentrated at the Cd-rich phase grain boundaries in equiaxed domains, becoming more elongate for longer annealing times. The Cd-rich phase grains were about 4 times larger and equiaxed on both sections, their size was measured by a mean intercept technique (5), it was found to be respectively 3.3, 7.1 and 10.1 µm for annealing times of 2, 18 and 130 hours.
Serial sectionning was carried out on specimens with 7.1 and 10.1 µm grain size, undeformed and deformed (ε = 40%), by chemically polishing away repeatedly the surface of a section and observing it under the microscope. Rapidity is required in order not to oxidize too much the surface which would result in having to remove too thick layers. We succeeded in obtaining successive sections distant of about 2 µm.

RESULTS : a) For all specimens a secondary creep regime at constant $\dot{\varepsilon}$ was obtained after a primary regime. The secondary creep rate depended only on grain-size, temperature and applied stress and did not depend on the previous stress and strain history of the sample (Fig. 1).
The results are given Fig. 2 and 3 on the usual $\ln\sigma - \ln\dot{\varepsilon}$ plot. The curves are sigmoïdal and the strain rate sensitivity reaches

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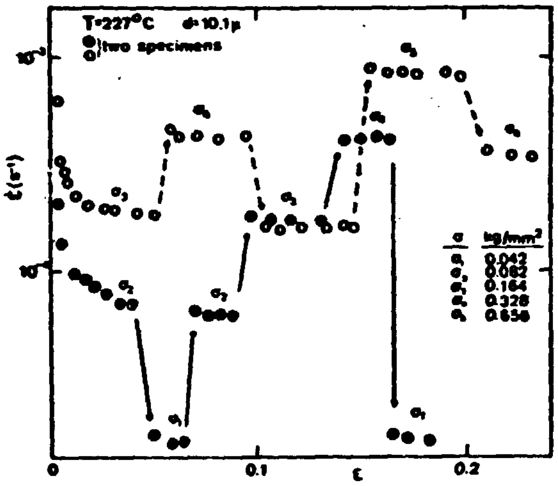

Fig. 1

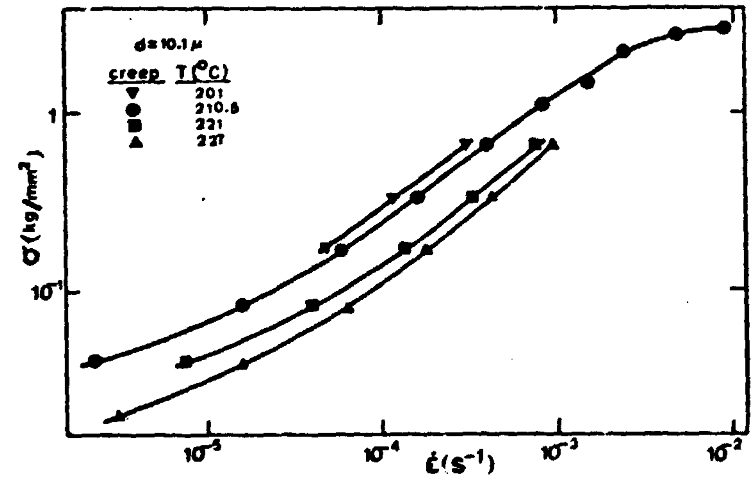

Fig. 2

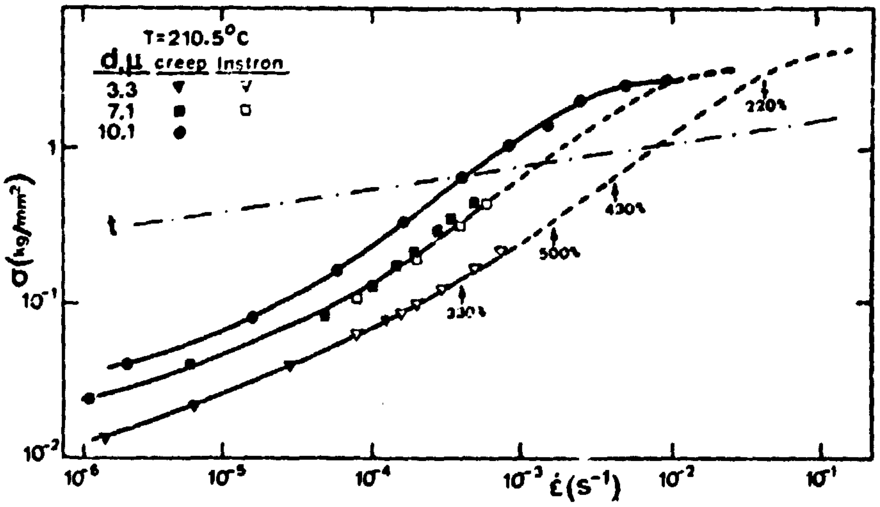

Fig. 3

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values as high as $m = 0.75$. The experiments performed in compression at constant speed are reported on fig. 3 and agree very well with the creep experiments. In the superplastic region, elongations to failure up to 500% were observed in the few runs performed in tension (Fig. 3). Identical values were found for m in the superplastic range by measuring the slope of the $\ln\sigma - \ln\dot\varepsilon$ curves or by stress changes (stress increments or decrements) performed on the same specimen after the secondary creep rate was established.

Curves for various grain sizes d can be deduced from one another by a translation as previously found for other superplastic materials (6).

The results can be gathered in an empirical constitutive relation:

$$\dot\varepsilon = \dot\varepsilon_0 \sigma^n d^p \exp-\left(\frac{Q}{RT}\right)$$

with $n = \frac{1}{m}$ in the range $1.3 < n < 2.9$.

The exponent p expressing the dependence of $\dot\varepsilon$ on the grain size d is constant in all the range of $\dot\varepsilon$ investigated and equal to : $p = -(2.2 \pm 0.2)$.

The activation energy Q measured at constant grain size and applied stress, using experiments with comparable values of m, does not depend on the grain size and is equal to : $Q = (23 \pm 3)$ kcal/mole.

b) The Cd-rich phase grain size was found to be very stable, it did not vary with strain up to $\varepsilon = 40\%$, and the grains remained equiaxed.

Serial sectioning showed that the Zn-rich phase appeared at the Cd-rich phase grain boundaries and disappeared in 3 consecutive sections at most (∿ 6 µm) (Fig. 4), its connectivity is very low. The Zn-rich phase, therefore, is dispersed as spheroïdal globules in the grain boundaries of the Cd-rich phase.

DISCUSSION AND CONCLUSION : a) The experimental results agree on the whole with Merriman's results (5). This author, however found $p = -1.4$ (compared to our $p = -2.2$); he also found two ranges for activation energies : below 210°C, $Q = 21.3$ kcal/mole (which agrees with our value $Q = 23$ kcal/mole) and $Q = 32.2$ kcal/mole above 210°C; however he found the latter value by using values of $\dot\varepsilon$ corresponding to curve portions where m was widely different. Using his published results, Q was recalculated with $\dot\varepsilon$ values corresponding to the same value of m and was found to agree with our value.

b) The fact that identical values for m were found by stress increments and stress decrements and the fact that the strain rate did not depend on the previous stress history of the sample suggest that the $\ln\sigma - \ln\dot\varepsilon$ plot corresponds both to minimum creep rate and to constant structure (7). Hence the superplastic regime in this case is probably a true stationary state (8).

c) Despite the theoretical predictions (2)(3) the Zn-rich phase, whose volume fraction is as high as 26%, is still present as unconnected, dispersed spheroids. This may be due to a high interfacial energy not taken into account in the topological predictions. Anyway this topology is incompatible with any significant role being attributed to the interphase boundaries, the harder Zn-rich particles probably being passively rotated and the creep rate being controlled by the deformation accommodated by diffusion of the connected softer Cd-rich phase. This is consistent with the fact that the activation energy is quite comparable to the activation energy for self diffusion of cadmium (Q_{Cd} = 19.7 kcal/mole).

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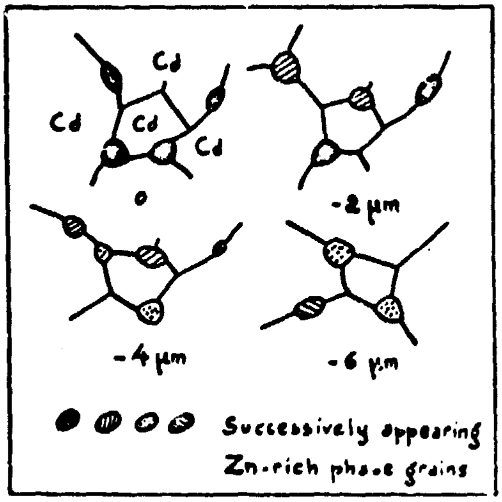

Fig. 4