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Creep of dilute zinc–copper alloys

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A series of zinc–copper alloys containing up to 2.0 wt % Cu have been prepared and tested in creep under constant load at temperatures up to 200° C. A metallographic study has also been made of the crept specimens. The creep resistance of Zn is shown to increase as the Cu content is raised, although the creep strength increment is small above 1 wt % Cu. Ageing the alloys also improves the creep strength, but precipitation during creep can generate voids which may lead to premature failure. The effect of increasing the Cu content is to make slip and grain-boundary sliding progressively more difficult, and to raise ΔH_c, the apparent activation energy for creep. A higher copper content also enables a low value of the stress exponent to persist to higher creep stresses.

1. Introduction

In spite of the importance of the wrought Zn–Cu alloys, little systematic work appears to have been undertaken to establish the relationship between their microstructure and mechanical properties. The room temperature tensile properties of these alloys have been studied [1], and no dramatic age-hardening response was observed, although discontinuous precipitation of the ϵ-phase appears to contribute a small aggregate hardening effect in alloys of higher (~ 2.5 wt %) copper content.

The aim of the present work was to investigate the creep behaviour of these alloys at temperatures between room temperature and 200° C, and to attempt to correlate these properties with the microstructures observed.

2. Experimental methods

2.1. Alloys and specimens

8.5 kg casts of four alloys of compositions listed in Table I were prepared. Each ingot was hot-rolled at 250° C to a thickness of 9.5 mm, and finish rolling was carried out at 100° C by a number of passes to a final thickness of 0.75 mm. The width of the final sheets was 125 mm, and from these the test-pieces were produced by blanking with a die and punch with their axis parallel to the rolling direction: the gauge dimensions were 21.19 mm in length and 4.25 mm in width.

2.2. Heat-treatment

Heat-treatments above 200° C were performed in a salt bath, and those below 200° C were carried out in an oil bath. Details of the solution heat-treatment and the resulting grain sizes are given in Table I. A subscript notation is used in describing the ageing heat-treatment: namely $1.6_{200,100}$; the base (1.6) represents the weight % Cu, the first subscript (200) the ageing temperature in °C and the second subscript (100)

TABLE I Alloy compositions (wt%) and solution treatment

% Cu		Impurities wt% max			1 h solution annealing temperature (°C)	Grain size (μm)
Nominal	Actual	Cd	Fe	Pb		
0.3	0.3	0.0002	0.0005	0.001	275	100
1.0	0.995	0.0002	0.001	0.001	300	70
1.6	1.6	0.0002	0.001	0.001	330	50
2.0	2.05	0.001	0.0005	0.002	385	90

Al, Mg, Sn, Si and Ti were not detected.

the ageing time in hours. An alloy without subscripts refers to the solution-annealed condition.

2.3. Creep testing

The specimens were tested in air in a dead-loading creep rig, employing an LVDT for strain measurement which gave a strain sensitivity of $\pm 10^{-4}$. Tests were conducted at room temperature, 100, 150 and 200° C, which represent values of 0.42, 0.54, 0.61 and 0.68 T_m for pure zinc. A number of crept specimens were examined by optical microscopy: details of the metallographic technique have been given elsewhere [1].

3. Results

3.1. Stress sensitivity

The effect of stress on the secondary creep rate can be seen from Fig. 1 a to d, in which log σ versus log $\dot{\epsilon}_s$ is plotted for the four alloys. The individual curves correspond to the different test temperatures. In Fig. 1c creep data for the aged 1.6% Cu alloy are also given: when these aged alloys were tested at 150 or 200° C, the specimens exhibited tertiary creep from the beginning of the test, so the curves corresponding to these results are shown dashed. The 2% Cu alloy tested at 150 and 200° C also showed wholly tertiary creep, and thus are shown as dashed lines in Fig. 1d since a true $\dot{\epsilon}_s$ could not be assessed. In Figs. 1c and d, the creep rate plotted for the dashed curves is that observed at the commencement of the test.

3.2. The effect of temperature

The influence of temperature on the secondary creep rate can be examined by plotting $\dot{\epsilon}_s$ versus $1/T$ for a chosen stress. From the slopes of the individual curves, the apparent activation energies may be calculated for each alloy. The data obtained by this approach are shown in Fig. 2.

3.3. Metallography of crept specimens

Since deformation modes are strain-rate dependent, in order to examine the effect of composition in this context, a comparison has been made of the different alloys after creep at approximately 10^{-3} h^{-1} to a total strain of $\sim 4\%$. This was effected by examining the surface of crept specimens which had been metallographically prepared before a creep test.

The solution-treated alloys, crept at room temperature, show a change in deformation behaviour with increasing copper content. Thus

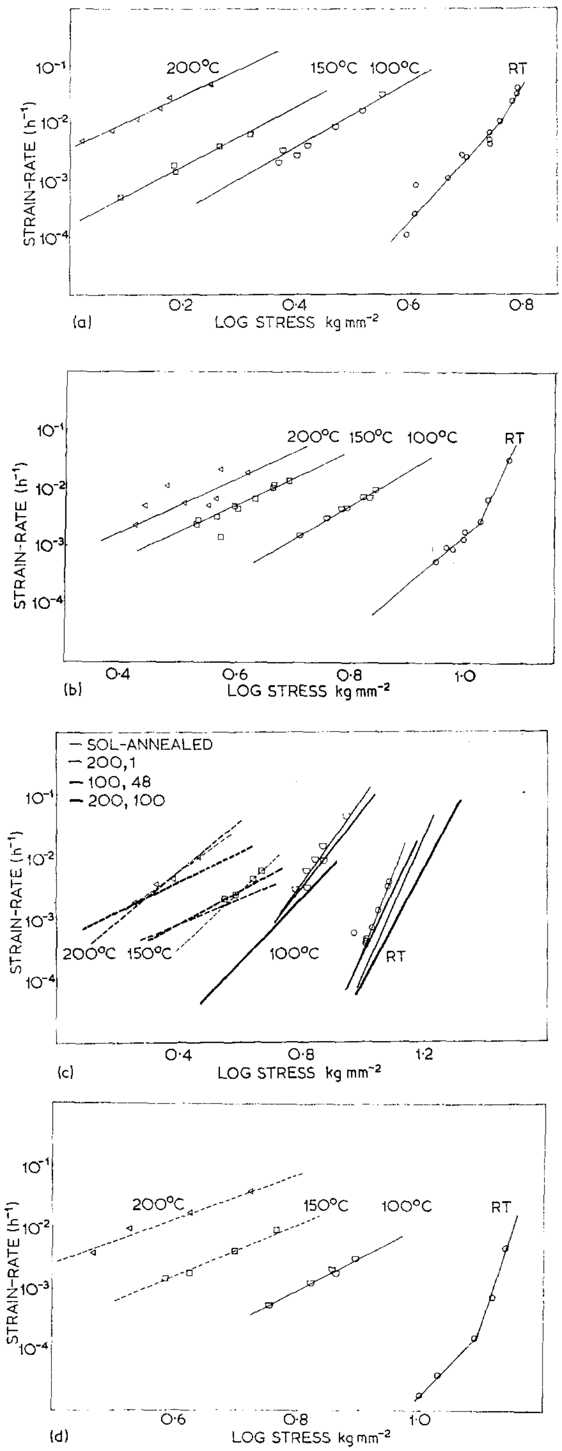

Figure 1 Variation of strain-rate with stress for (a) 0.3% Cu alloy, (b) 1.0% Cu alloy, (c) 1.6% Cu alloy and (d) 2.0% Cu alloy.

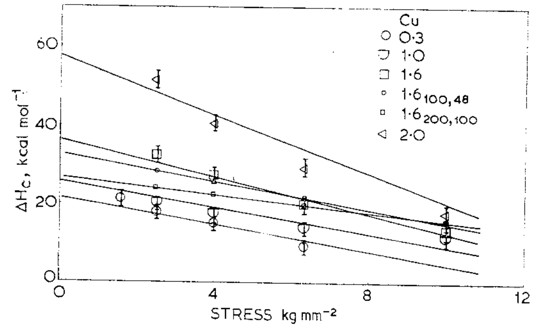

Figure 2 The influence of stress upon the apparent activation energy for creep.

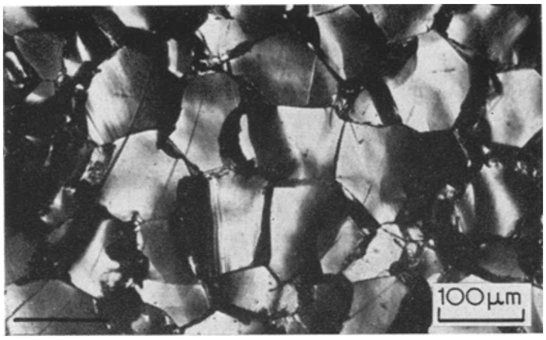

Figure 5 Surface of Zn-1.0% Cu after creep at 200°C. The stress axis is marked.

the 0.3% Cu alloy (Fig. 3) shows predominantly straight, parallel slip bands in most of the grains. There is also clear evidence of relative grain displacement due to grain-boundary sliding. The 2% Cu alloy (Fig. 4) shows prolific deformation twins, with the majority of the slip bands being wavy in appearance. The alloys of intermediate Cu content show a transition between these types of behaviour, with decreasing evidence of grain boundary sliding as the Cu content is raised.

Figure 3 Surface of Zn-0.3% Cu alloy after creep at room temperature. The stress axis is marked.

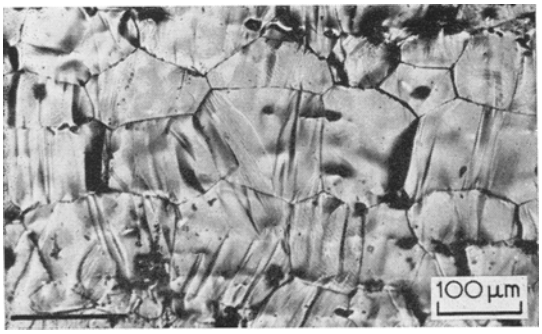

Figure 4 Surface of Zn-2.0% Cu alloy after creep at room temperature. The stress axis is marked.

An increase in the creep temperature of the alloys of higher Cu content has the effect of reducing the number of deformation twins present, and of increasing the incidence of wavy slip bands (Fig. 5). The amount of grain-boundary sliding also appears to increase as the temperature is raised.

The effect of ageing the alloys of higher Cu content prior to creep testing has the effect of decreasing the incidence of twinning, deformation by coarse slip, or grain-boundary movements. Thus a $2.0_{200,72}$ alloy crept at room temperature appeared to have deformed wholly by a process of fine slip.

It has already been reported [1] that none of these alloys, when aged at room temperature, reveal any precipitation within at least 3 months. Under conditions of creep at room temperature, however, grain-boundary precipitation is readily detected after relatively short times. Thus Fig. 6 shows grain-boundary precipitation of the ϵ-phase in the 1.6% Cu alloy crept under 10.5 kg mm^{-2} at room temperature for 54 h. There is also evidence of the presence of grain-boundary cavities on certain of the boundaries lying perpendicular to the direction of application of the stress.

A metallographic examination has also been made of those specimens which exhibited tertiary creep throughout the test, such as the 1.6 and 2.0% Cu alloys (in any initial heat-treated condition) when crept at 150 or 200°C. There appear to be two structural changes which can give rise to this continuous tertiary creep:

(a) If the testing temperature is such that extensive precipitation can take place during the

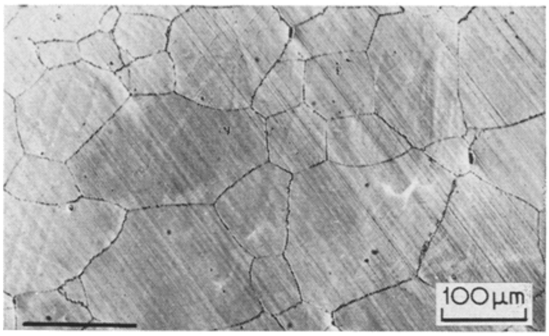

Figure 6 Optical micrograph of a 1.6% Cu alloy crept at room temperature. The direction of the stress axis is marked.

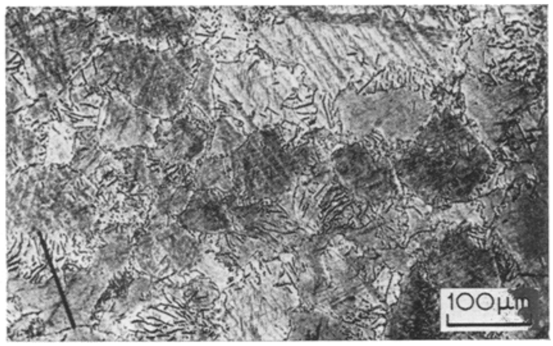

Figure 8 Optical micrograph of a $2.0_{200,100}$ alloy crept at 200° C. The stress axis is marked.

test, cavities form which are associated with the discontinuous grain-boundary precipitate. Fig. 7 illustrates such cavities (whose growth will be associated with an accelerating creep deformation) in the case of a 1.6% Cu alloy tested at 150° C. They have formed predominantly at the advancing interface of the cellular precipitate, and appear to be associated particularly with grain boundaries lying transversely with respect to the applied stress.

(b) Continuous microstructural change without cavity formation is observed in the case of the pre-aged specimens undergoing creep. Thus Fig. 8 illustrates a $2.0_{200,100}$ alloy crept at 200° C in which no cavities are observed, although marked grain growth has taken place, and so the latter process of continual structural change accompanies the tertiary creep.

4. Discussion

4.1. Stress sensitivity

The stress dependence of the secondary creep rate of pure metals has been reviewed by Sherby and Burke [2]. At low stresses the creep rate varies linearly with the stress; in a range of intermediate stress, $\dot{\epsilon}_S$ obeys an exponential stress dependence:

$$\dot{\epsilon}_S = k^n \tag{1}$$

where k and n are stress-independent coefficients within the region considered. For pure metals the approximate value of n is 5. At still higher stresses the stress-dependence of $\dot{\epsilon}_S$ is greater than that predicted by Equation 1, and empirically it is found that

$$\dot{\epsilon}_S = k' \exp \beta\sigma \tag{2}$$

From Fig. 1a to d the stress exponent, n, of Equation 1 can be determined, although some uncertainty must be attached to those values obtained from specimens showing tertiary creep characteristics from the start of the test. Lagneborg [3] has pointed out that values of n should be examined as a function of both stress and temperature, and following this approach Fig. 9 shows the relationship between n and the stress/temperature ratio for all the alloys in the present study.

Owing to the absence of steady-state creep in certain specimens, as already discussed, there is uncertainty in some of the n values, nevertheless the alloys appear to show an n value of about 4.8 at low σ/T values. There is a threshold value of σ/T above which a rapid increase in the value of n occurs, and this threshold appears to increase with increasing copper content of the alloys.

An n value of about 5 corresponds to a creep process controlled by dislocation climb involving an equilibrium vacancy concentration. Sherby

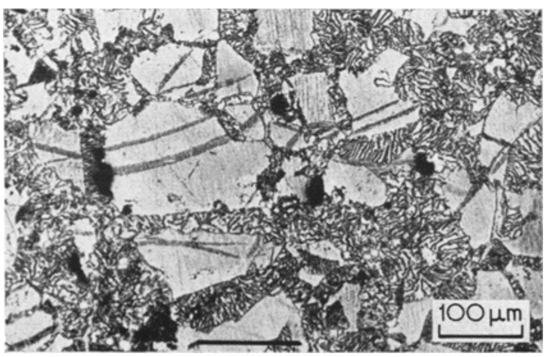

Figure 7 Optical micrograph of a 1.6% Cu alloy crept at 150° C. Direction of stress axis is marked.

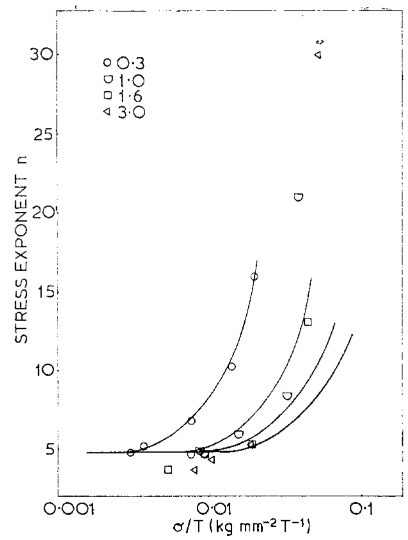

Figure 9 Relation between the stress exponent, n, and the ratio of the creep stress and absolute temperature.

and Burke suggest that the large increases in n observed at high creep stresses correspond to dislocation climb creep processes under conditions where the vacancy concentration is greater than that in thermal equilibrium.

4.2. Apparent activation energy for creep

It is apparent from Fig. 2 that the value of ΔH_c is stress-dependent, and if the values are extrapolated to zero stress one obtains values for ΔH_c of 22, 26, 36 and 57 kcal mol^{-1} for the 0.3, 1.0, 1.6 and 2.0% Cu alloys respectively. They thus tend to the value of ΔH_c observed for pure Zn (21 kcal mol^{-1}) there being a progressive increase in ΔH_c as the copper content of the matrix increases.

The curves for the pre-precipitated 1.6% Cu alloys are shallower than that when the alloy is in the solution-treated condition. It is suggested that this arises because the matrix solute content decreases as precipitation occurs: thus the $1.6_{100,48}$ is only partially precipitated and thus has a higher matrix Cu content than the $1.6_{200,100}$ alloy. It may thus be concluded that the apparent activation for creep depends primarily upon the solute content of the matrix and not upon the presence of a second phase.

4.3. Modes of creep deformation

It is seen (e.g. Figs. 3 and 4) that, with increasing copper content, the deformation modes change from a planar slip process to non-planar slip and twinning, although there is less twinning as the temperature of deformation rises. The extent of grain-boundary sliding appears to decrease with decreasing temperature, increasing copper content, and with the presence of precipitation in the boundaries.

The acceleration of grain-boundary precipitation by creep deformation has been demonstrated, and, in particular, in those specimens showing wholly tertiary creep behaviour, there were either grain-boundary voids forming in association with the precipitate phase, or a process of structural softening by grain growth occurred. In the latter case the pre-existing cellular precipitate must dissolve and re-precipitate as the grain boundaries migrate.

4.4. The relation between creep strength and composition

If one disregards the data obtained at 150 and 200°C because of the predominantly tertiary creep behaviour observed, there is clear indication that the creep resistance of Zn increases as the copper content is raised. The stress required for a strain rate of 10^{-3} h^{-1} is plotted against the copper content in Fig. 10; and it is seen that the

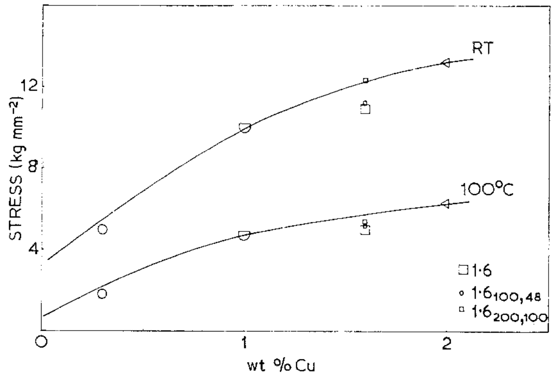

Figure 10 The effect of copper content on the creep stress to yield a strain rate of 10^{-3} h^{-1}

creep strength increment is small above 1% wt Cu. It can also be seen from this diagram that ageing improves the creep properties, although this is not a major effect. If an alloy is not fully aged, however, precipitation during creep can generate voids which may lead to premature failure.

The effect of increasing the copper content is to make slip and grain-boundary sliding progressively more difficult, and to raise ΔH_c the apparent activation energy for creep. With regard to the stress exponent (Fig. 8), the results suggest that for a constant temperature of test, a higher copper content enables a low n value to persist to higher creep stresses.

Acknowledgement

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