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Structure Changes in a Single-Phase Superplastic Zn-Cd Alloy

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The paper deals with the investigation of structure changes in the superplastic single-phase Zn-0.25wt.% Cd alloy by means of electrical resistivity measurements, light microscopy and mechanical testing. The results obtained make possible to explain processes taking place in the alloy during its heating to the deformation temperature as well as in the course of its superplastic deformation. The experimental findings were used in the explanation of the observed favourable influence of superplastic prestrain on the stability of fine-grained structure of the alloy. This effect is beneficial because it leads to the shift of the temperature interval of superplastic behaviour to higher temperatures and also to better superplastic characteristics.

1. Introduction

A very fine-grained structure which is stable at high temperatures ($T > 0.4\ T_m$, where T_m is the melting temperature) is considered as a necessary structural precondition for the occurrence of superplasticity. Such a structure is usually attained by rather complicated thermomechanical treatment and consequently superplastic materials have a complicated initial microstructure. Various processes can take place during annealing of such specimens due to the release of deformation energy stored in specimens in the course of their preparation. The fine-grained structure in superplastic materials is usually stabilized by second phase particles. However, in alloys with a low thermal stability of the structure the grain coarsening can occur in the course of their heating to the deformation temperature resulting in a deterioration or loss of superplastic behaviour. Such a situation is typical especially for the small group of alloys which are superplastic in the simple single-phase state.

Recently we have reported the results of the investigation of the thermal stability of the Zn-0.25 wt. % Cd alloy [1]. This alloy belongs to the family of alloys which are superplastic in the state of a solid solution. Fig. 1 shows the isochronal resistivity annealing curves of the alloy. The curve 1 belongs to the specimen prepared to the

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microcrystalline state by a multi-step rolling at high temperature ($T > 0.42\ T_m$). Two stages may be clearly seen on the annealing curve. As confirmed by light microscopy, recrystallization is responsible for the first stage (320K – 360K), while in the

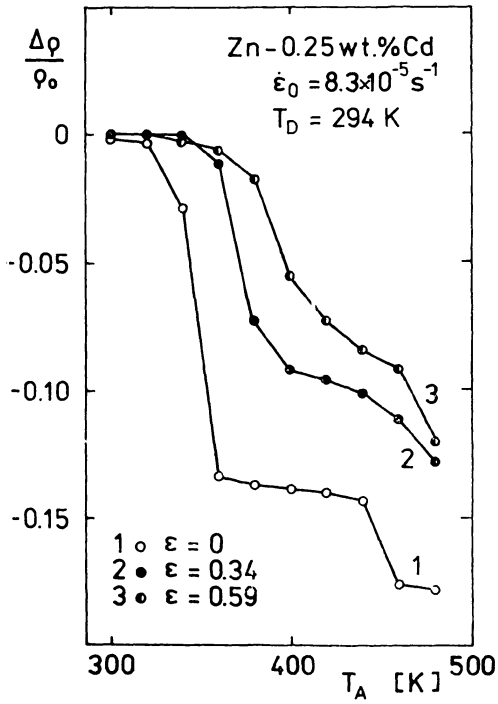

Fig. 1. Strain dependence of resistivity annealing curves after deformation at 294 K and at strain rate $8.3 \times 10^{-5}\ s^{-1}$, ε is the true strain, ϱ_0 stands for the initial value of resistivity.

second one the recrystallized grains are growing. The grain size was 1 μm in the initial state, 8 μm at the end of recrystallization stage and 30 μm at the end of the second stage. The recrystallization stage was analysed by measuring the isothermal annealing curves of resistivity. These curves may be well fitted by the Avrami's equation and the values of the kinetic exponent may be explained by the classic theory of the static recrystallization.

Recently, the superplastic behaviour of the Zn-0.25 wt. % Cd alloy has been investigated in a broad temperature range and the deterioration of the superplastic properties has been observed around 350K [2]. It is the aim of the present paper to explain this effect using the results of resistivity measurements [1] and to propose a way to the stabilization of the fine-grained structure in this alloy.

2. Experimental details

The casting was homogenized in vacuum at 573K for 100 hours and rolled at the same temperature with a total reduction of 40 %. The final rolling was performed at 300K with further reduction of 80 %. The prepared sheets of a thickness of 1 mm were stored in dry ice in order to prevent their structure changes. The mean grain size of 1 μm was determined using the method of TEM of carbon replicas. The phase composition of the alloy was tested by means of electrical resistivity measurements. No redistribution of Cd atoms was revealed both in quenching experiments and during linear heating of specimens up to the melting point. On the ground of these results and of the equilibrium phase diagram data [3] it may be concluded that the Zn-0.25 wt. % Cd alloy is an oversaturated solid solution of Cd in Zn which does not decompose in the whole temperature range studied. The specimens with the axis parallel to the rolling direction were deformed in tension at temperatures between 275K and 514K (i. e. 0.39 and 0.73 of T_m) in air atmosphere.

3. Results and discussion

Fig. 2 demonstrates the influence of temperature on the deformation curves measured at the initial strain rate $\dot{\varepsilon}_0 = 8.3 \times 10^{-4}\ s^{-1}$. The strain softening region beyond the flow stress maximum at the beginning of straining may be observed at all curves except the curves number 4 and 5 which exhibit a strain hardening. A nearly steady

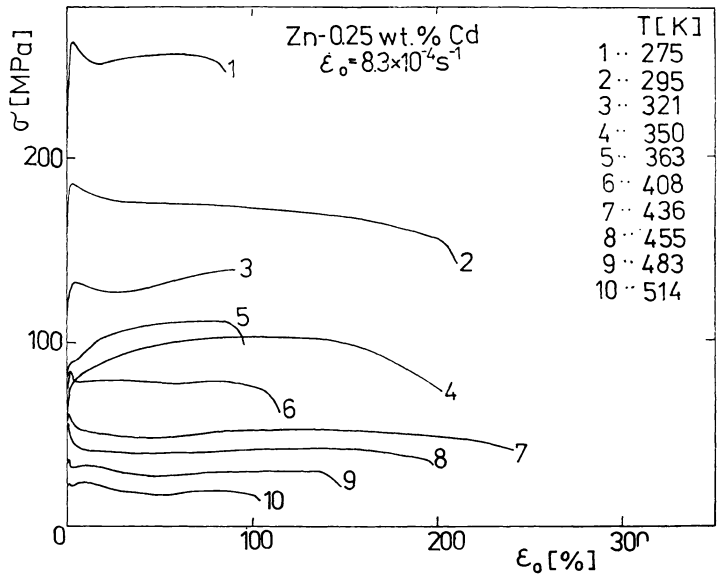

Fig. 2. The effect of temperature on the deformation curves. σ is the true stress and ε_0 stands for the relative elongation.

state character of deformation curves is established at about 400K. Fig. 3 shows the temperature dependence of the flow stress at the relative elongation $\varepsilon_0 = 30\,\%$ (curve 1). This dependence is not monotonous and exhibits a local maximum at

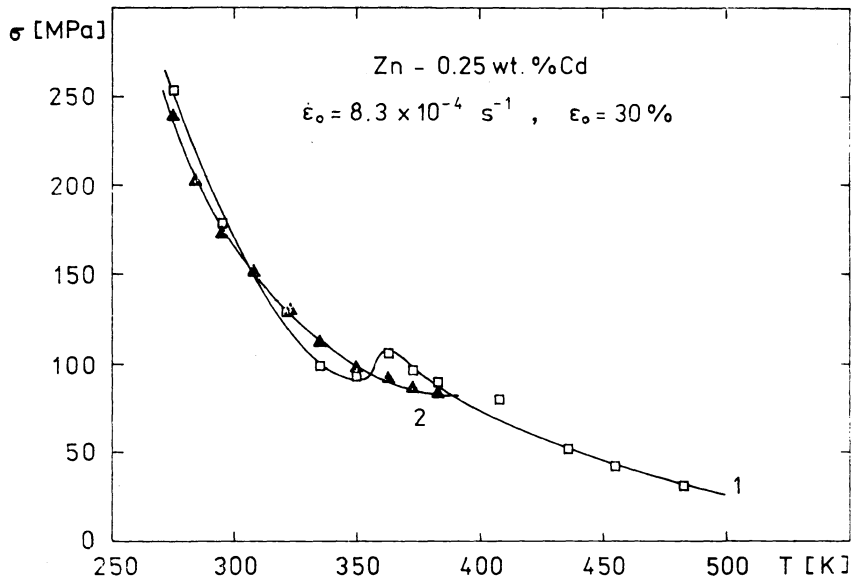

Fig. 3. Temperature dependence of the flow stress at $\varepsilon_0 = 30\,\%$. The superplastic prestraining at 303 K with $\dot{\varepsilon}_0 = 10^{-4}$ s^{-1} to $\varepsilon_0 = 40\,\%$ preceded the measurements represented by the curve 2.

360K. The influence of the temperature on the strain rate sensitivity parameter m is shown in Fig. 4. The experimental points of the curve 1 were obtained from strain rate changes at $\varepsilon_0 = 30\,\%$ whereas the curve 2 resulted from stress relaxation tests at $\varepsilon_0 = 50\,\%$. Both dependences exhibit a decrease of the strain rate sensitivity parameter m near the temperatures where the local maximum on the temperature dependence of the flow stress was found. This deterioration of the superplastic behaviour is due to the recrystallization taking place between 320K and 360K in the course of heating of specimens to the deformation temperature. This recrystallization results in an increase in the grain size from 1 to 8 μm. Similarly to other superplastic materials which are characterized by an increase in the flow stress with increasing grain size [4] the flow stress around 350K is enhanced in comparison with finer grained specimens deformed at lower temperatures and the strain rate sensitivity parameter decreases.

We have found that this deleterious effect may be suppressed using a superplastic predeformation at the room temperature prior to tensile testing. The procedure follows from our older measurements [1]. We observed the influence of a small superplastic deformation at the room temperature ($\dot{\varepsilon}_0 = 8.3 \times 10^{-5}$ s^{-1}) on resist-

ivity annealing curves (Fig. 1). The superplastic character of this deformation was demonstrated by the value of $m = 0.4$. As it is shown in Fig. 1 the magnitude of the stage of recrystallization decreases with increasing strain and the stage shifts to

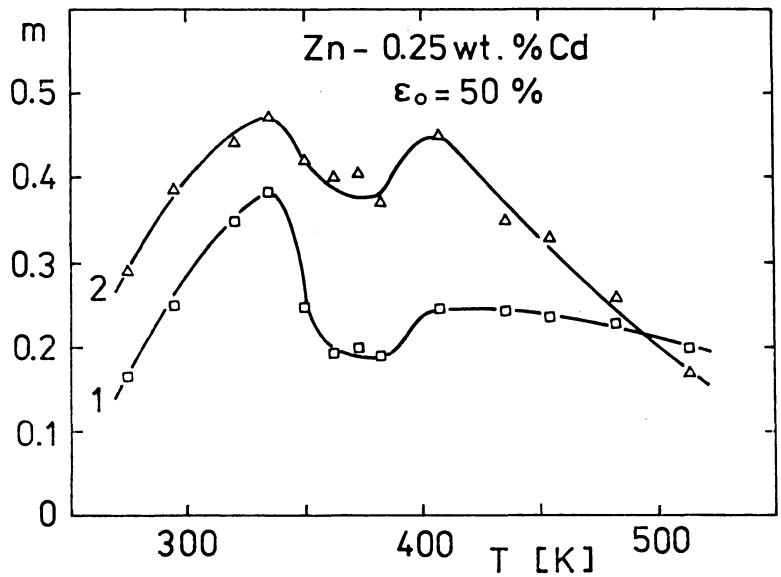

Fig. 4. Temperature dependence of the parameter m evaluated from strain rate chagnes at $\varepsilon_0 =$ $= 30$ % (1) and from stress relaxation tests at $\varepsilon_0 = 50$ % (2).

higher temperatures at the same time. Such a behaviour is due to dynamic softening (dynamic recovery and/or dynamic recrystallization) during superplastic deformation when a part of deformation energy stored in specimens in the course of their preparation is released. We verified this shift of the recrystallization stage also by means of light microscopy. The most important findings of the light microscopy performed on specimens exposed to the additional superplastic deformation at 303K ($\dot{\varepsilon}_0 \simeq$ $\simeq 10^{-4}\ s^{-1}$) are given in Table 1, where ε_0 is the relative elongation, T_a is the upper temperature limit of the stable fine-grained structure and d stands for the mean grain size at the end of the superplastic prestraining.

TABLE 1
The results of the light microscopy performed on specimens exposed to the superplastic straining at 303 K with $\dot{\varepsilon}_0 \simeq 10^{-4}\ s^{-1}$

ε_0 [%]	0	40	80
T_a [K]	320	350	370
d [μm]	1	1.7	2.7

It is obvious that the superplastic prestraining near the room temperature enhances the thermal stability of the fine-grained structure. This effect has a favourable influence on the deformation behaviour of the alloy investigated. Both local maximum

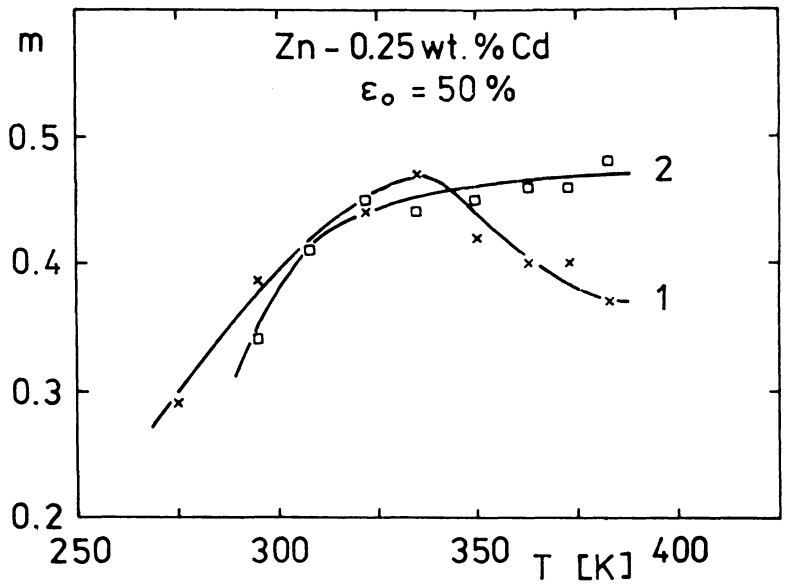

Fig. 5. Temperature dependence of the parameter m evaluated from stress relaxation tests at $\varepsilon_0 = 50\,\%$. The superplastic prestraining at 303 K with $\dot{\varepsilon}_0 \simeq 10^{-4}\ s^{-1}$ to $\varepsilon_0 = 40\,\%$ preceded the measurements represented by the curve 2.

of the flow stress at 360K (Fig. 3, curve 2) and the local minimum of the parameter m situated in the same temperature range (Fig. 5, curve 2 – the strain rate sensitivity parameter was evaluated from stress relaxation tests at $\varepsilon_0 = 50\,\%$) disappear. Due to the better thermal stability of the fine-grained structure the temperature interval of superplastic behaviour is extended to higher temperatures.

4. Conclusion

Using a suitable superplastic prestrain, the fine-grained structure of some superplastic materials may be stabilized. The grain coarsening occurring during the heating of these materials to the deformation temperature may be suppressed and the range of superplastic behaviour extended to higher temperatures. This positive influence may be explained on the assumption that a part of deformation energy stored in these materials during their preparation is relased by dynamic processes occurring during their superplastic prestrain. This results in a decrease of driving force for recrystallization during the following annealing.

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