Deformation of Single Crystals of Cd₃Mg

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The deformation behaviour of single crystals of Cd₃Mg and Cd-28.5 at. % Mg has been studied over a range of temperature from -196 to 200° C, and the critical resolved shear stresses associated with both basal and $\{1\overline{1}00\} \langle 11\overline{2}0 \rangle$ prismatic slip have been measured. The CRSS for prismatic slip has been found to show a marked increase with increasing temperature prior to the onset of appreciable disordering while that for basal slip remains athermal over the same range. Both slip systems, however, showed a CRSS peak within the two-phase, ordered plus disordered, region. The athermal CRSS for basal slip in the stoichiometric alloy is shown to be too low to be consistent with a previous explanation advanced to account for the CRSS for basal slip in Mg₃Cd.

1. Introduction

The formation of a DO_{19} superlattice at both Mg₃Cd and Cd₃Mg makes the magnesiumcadmium system particularly suitable for the study of the mechanical properties of this structure. Previous deformation studies have mainly been concerned with the former alloy in both polycrystalline [1-3] and single crystal [4-6] forms, and in both the ordered and the disordered condition. It has been established that in the ordered state basal slip and $\{1\overline{1}00\}$ $\langle 11\overline{2}0\rangle$ prismatic slip can occur with equal facility and that the critical resolved shear stress (CRSS) for each system is athermal between about $-120^{\circ}C$ and room temperature. A flow stress peak occurs at elevated temperatures, associated with disordering through a narrow two-phase region [2], though the exact location of this peak in single crystal studies was uncertain due to deviations from stoichiometry of the specimens tested. Elevated temperature tests on disordered material showed a significant decrease in CRSS for both basal and prismatic slip relative to the low temperature values for the ordered state.

The purpose of the present work is to extend the single crystal studies of basal and prismatic slip carried out on Mg₃Cd to the alloy Cd₃Mg. Like Mg₃Cd, superlattice formation at this composition involves a two-phase region in the equilibrium diagram, but for Cd₃Mg the temperature and composition limits of this region exist over a larger range and have been accur-



Figure 1 The two-phase region associated with Cd₃Mg.

ately determined by Kamath, Craig, and Wallace [7]. The equilibrium diagram obtained by these workers is shown in fig. 1. Because of the onset of the two-phase region at 25° C for the stoichiometric composition it was decided to supplement the study of the stoichiometric alloy by also investigating the behaviour of a Cd-28.5% Mg alloy, which would enable the single phase ordered material to be retained at higher temperatures.

2. Experimental Procedure

2.1. Materials and Specimen Preparation

High purity cadmium (99.999%) and magnesium (99.98%) were melted in the appropriate proportions to obtain ingots of the required

compositions; one nominally stoichiometric the other containing 28.5% Mg. The materials were induction melted, cast under argon, and the resulting ingots sliced to provide starting material for single crystal growth. Blanks for this purpose were cut from the sliced ingots and fitted into split graphite moulds which were lowered through the hot zone of a furnace under a highpurity argon atmosphere. The hot zone had a maximum temperature of 390°C and each run lasted about 24 h. The resulting single crystal slab was chemically polished, its orientation determined by back-reflection Laue and single crystal specimens for tensile testing, having orientations required for either basal or prismatic slip, were cut out using a jewellers saw. The crystals were lightly ground to shape and a reduced gauge section was electropolished on the specimens to give a gauge length of 1 cm. The cross-section was approximately 1 mm². The finished specimens were annealed for 1 h at 200°C, slow cooled in an oil bath, and their exact orientations were determined by further back-reflection Laue photographs.

2.2. Analysis and Dilatometry

Composition determinations on several single crystal specimens indicated that the average compositions of the "as-grown" crystals were within $\frac{1}{2}$ % of the intended compositions. As an additional check, the disordering of selected crystals was followed dilatometrically using a variable inductance dilatometer as the temperature of the specimen was incrementally raised through the two-phase region. The specimen was held for 1 h at each temperature but in fact all

changes in length occurred within the first 5 min. It was not possible to determine the lower limit of the two-phase region by this technique, but the upper limit was characterised by a very sharp discontinuity in the slope of a length versus temperature plot enabling its temperature, and composition (from fig. 1) to be accurately fixed.

2.3. Mechanical Testing

All mechanical tests were performed in uniaxial tension at a strain-rate of 3×10^{-4} sec⁻¹. Low temperature tests were performed in the appropriate constant temperature bath, high temperature tests in a thermostatically controlled oil bath. In the latter tests temperature control was better than $\pm \frac{1}{2}^{\circ}$ and specimens were held at temperature for 1 h prior to testing. Slip system identification was by two-surface analysis.

3. Experimental Results

3.1. Dilatometry

Fig. 2 shows an example of a dilatometric run performed using a crystal of the 28.5% Mg alloy which was oriented such that its *c*-axis lay at 13° to the measuring direction. Both the temperature indicated for the two-phase region upper limit and the measured change in length agree with the results expected for this composition according to a previous dilatometric study which utilised single crystals [8].

3.2. Mechanical Tests

The temperature dependence of the CRSS for Cd_3Mg is shown in fig. 3 for both basal slip and $\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$ prismatic slip. The CRSS has been defined as 0.01% proof stress and examples



Figure 2 The change in length of a single crystal of Cd-28.5 at. % Mg during disordering.



Figure 3 Temperature dependence of the CRSS for basal slip and $\{1\overline{1}00\} \langle 11\overline{2}0 \rangle$ prismatic slip in Cd₃Mg.

of the stress-strain curves from which these data are derived are given in figs. 4 and 5. The curves for basal slip, like those obtained for Mg_3Cd , exhibited jerky flow over a range of temperature, from -120 to 75°C, and were relatively smooth outside this range. In contrast to the behaviour of Mg_3Cd , however, prismatic slip in Cd_3Mg was not characterised by jerky flow at any temperature.

The temperature dependence of the CRSS for basal and prismatic slip in the Cd-28.5 at. % Mg alloy is shown in fig. 6. The results reflect basically similar behaviour to that shown by Cd_3Mg , the main difference lying in the higher stress levels compared with the "stoichiometric" alloy. The stress-strain curves were also basically similar in form.



SHEAR STRAIN

Figure 4 Typical stress-strain curves for single crystal Cd_3Mg deforming by basal slip.



Figure 5 Typical stress-strain curves for single crystal Cd_3Mg deforming by prismatic slip.



Figure 6 Temperature dependence of CRSS for basal slip and prismatic slip in Cd-28.5 at. % Mg.

The deformation of both alloys by prismatic slip above 25° C was accompanied by a decrease in work hardening capacity typified by the rounded stress-strain curver shown in fig. 5. In this region there was also a marked increase in strain-rate sensitivity as determined by stress relaxation tests on specimens deforming by prismatic slip. The results of such tests are shown in fig. 7 in which the strain-rate sensitivity exponent, *m*, is plotted as a function of temperature, assuming the relationship

$$\dot{\gamma} \propto \tau^m$$

where $\dot{\gamma} =$ strain rate, $\tau =$ applied stress.

4. Discussion

4.1. The CRSS for Basal Slip

The athermal nature of the CRSS for basal slip, $\tau_{\rm b}$, and the transition from jerky to smooth



Figure 7 Variation of strain-rate sensitivity exponent "m" with temperature for prismatic slip in specimens of Cd₃Mg and Cd-28.5 at. % Mg. At temperatures below about 10° C there was no detectable strain-rate sensitivity.

deformation are features common to both Mg₃Cd and Cd₃Mg but the actual magnitudes of τ_b are very different. For Mg₃Cd, $\tau_b \sim 5 \text{ kg mm}^{-2}$ compared with the value of 0.5 kg.mm⁻² found here for Cd_3Mg . The value of 5 kg mm⁻² for Mg₃Cd was interpreted [4] in terms of the stress required to create anti-phase boundary (APB) on the basal plane by the movement of individual components of dissociated superlattice dislocations, on the basis that facile cross-slip onto low APB energy prism planes would result in the proposed dissociation of basal dislocations. Quantitative agreement with this model was obtained, though this necessitated assuming a rather low value of ordering energy. However, even allowing for a still lower ordering energy for Cd₃Mg (judging by the lower disordering temperature) it cannot be envisaged that the ten times lower value of $\tau_{\rm b}$ found for this alloy can be ascribed to the same process. If it is assumed that the explanation was correct for Mg₃Cd then its non-applicability to the present case might indicate a lower basal plane stacking fault energy which would inhibit cross-slip and stabilise the superlattice dislocations on this plane, removing the necessity to initiate slip by the movement of unpaired dislocations.

The peak in the τ_b versus temperature curve for the Cd-28.5% Mg alloy is not well defined by the experimental results and is largely inferred from the behaviour of prismatic slip in the same alloy. For Cd₃Mg, however, the peak in τ_b is quite well defined and is located within the twophase region at about 76°C. At this temperature, according to Kamath *et al* the alloy consists of 60% disordered phase at equilibrium and, as these workers found equilibrium to be complete with $\frac{1}{2}$ h, this proportion should be applicable to the tensile specimens at peak strength. There is some uncertainty over this figure, however, since dilatometric work on the stoichiometric alloy seemed to indicate an unexpectedly low degree of transformation up to about 76°C followed by a rapid increase in extent of transformation with increase in temperature up to the upper limit of the two phase region.

4.2. The CRSS for Prismatic Slip

The increase in CRSS for prismatic slip, τ_p , with increase in temperature, which characterises the deformation of both Cd₃Mg and Cd-28.5% Mg at temperatures below the temperatures of the respective maxima in τ_p , appears to occur continuously from about - 196°C upwards. Below the two-phase region for the "stoichiometric" alloy this increase is accompanied by virtually no change in degree of order [7], there is no parallel increase in $\tau_{\rm b}$, and no appreciable strain-rate sensitivity, inverse or otherwise. The rather rapid increase in τ_p above room temperature occurs within the two-phase region for Cd_3Mg , but the results for the 28.5% Mg alloy indicate a corresponding increase over the same temperature range for the single phase material, as one would expect from extrapolation of the Cd₃Mg data from the single phase region into the two-phase region.

Approaching the two-phase region from the high temperature side, the increase in τ_p for disordered material with decrease in temperature occurs predominantly within the single-phase region. The formation of a CRSS peak within the two-phase region is, therefore, inevitable given the opposite temperature dependences of the single phase material on either side and, apparently, irrespective of any two-phase hardening mechanism within the two phase region. This last statement must, however, be qualified since it is possible that the "single-phase" disordered material just above the two phase region should be regarded as a coherent, two-phase mixture itself [9] the second phase consisting of very small ordered domains in the disordered matrix, incapable of producing the distinct superlattice lines detectable within the two-phase region. In that case at least part of the rapid temperature variation of τ_p in both alloys above the two-phase region could be ascribed to "particle" hardening as the proportion of ordered phase increased with decrease in temperature, resulting in peak strength at a critical fraction of ordered phase within the two-phase region. This interpretation would be consistent with the similar values of τ_p at the upper limit of the twophase region in both Cd₃Mg and Cd-28 % Mg (even though the actual temperatures are different) and with the fact that the fall off in τ_p with temperature above this point is very similar in both alloys – the curves being superimposable if shifted horizontally so that the upper limits of the two-phase regions coincide.

4.3. Jerky Flow

As far as we are aware there have been virtually no investigations or previous reports of dynamic strain ageing effects in long-range ordered alloys and we have not been able to establish categorically whether the jerky flow found in this work (and in Mg₃Cd) can be attributed to this cause. More work is required to fully investigate these phenomena, but the disappearance of jerky flow at low temperatures in all the crystals which exhibit it certainly suggests a diffusion controlled mechanism. The temperatures down to which serrated stress-strain curves are observed in Mg₃Cd and Cd₃Mg are low (~ 0.2 Tm), but they are not inconsistent with observations of jerky flow associated with dynamic strain ageing in Al-Mg [10] alloys at similar temperatures. In addition the absence of serrated flow in polycrystals [11] in regimes where single crystals do exhibit this behaviour is consistent with work on the latter alloy.

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