Letters

On the orientation of grain boundaries in cadmium recrystallized during hot deformation

A striking feature of grain-boundary migration during high temperature fatigue is the metallographic observation on plane surfaces of the so-called diamond or orthogonal configuration of grain boundaries occurring as a result of their migration to stable positions at ~ 45° to the stress axis [1-6]. A similar microstructural development has recently been shown to occur in zinc during creep deformation [7]. Important consequences of diamond grain configuration to life in fatigue [6, 8, 9], creep strength [9, 10] and creep cavitation [7] have been noticed as a result of 45° grain boundaries experiencing maximum shear stresses. However, the cause of reorientations of grain boundaries to stable diamond configurations is still a matter for research.

In the present work, creep and hot fatigue were performed on cadmium. Measurements of angular distribution, relative to the stress axis, of grain boundaries were made to study grain-boundary migration and to investigate the propensity to formation of diamond pattern of grain boundaries. It has been suggested [5-7, 10] that the diamond morphology arises as a result of the combined action of grain-boundary sliding, intragranular deformation and grain-boundary migration. In the present work, an indirect indication of the importance of grain-boundary sliding in causing the observed changes in grain-boundary alignments has been obtained by experiments in which recrystallization in cadmium was induced under (a) purely thermal conditions (b) during hot-working (c) during creep and (d) during hot fatigue, followed by measurements of angular distribution of grain boundaries associated with the newly formed grains.

Cadmium of 99.9% purity was used. Constant strain-rate creep tests were performed in a modified Hounsfield tensometer using a nominal strain rate of 3.6% per hour at room temperature (0.50 T_m, where T_m is the melting point in degree Kelvin), 55°C (0.55 T_m), 100°C (0.63 T_m) and 150°C (0.71 T_m). Fatigue testing (3000 Hz) was carried out at room temperature and 100°C on an Avery Midget Pulsator (Type No. 7304). Typical stress range was 100 kg cm^{-2} (σ_{max}) to -50 kg cm^{-2} with a positive mean stress of 25 kg cm^{-2} to avoid buckling. Constant stress creep experiments were carried out in a creep machine described elsewhere [11]. For optical metallography, samples were polished chemically in a solution containing 32 parts chromic acid, 2 parts sodium sulphate and 100 parts water and etched in a solution of 70 ml glacial acetic acid and 30 ml fuming nitric acid. Angular distribution of grain boundaries relative to the stress axis was determined following a method used by Kutumba Rao and Rama Rao [12] and for plotting each histogram a minimum of 200 measurements was made.

As-cast cadmium was given a reduction of 75% in thickness (final thickness was 1.8 mm) by rolling at room temperature followed by annealing at 200°C for 2 h to yield an average grain size of 0.2 mm. The angular distribution of grain boundaries in the annealed sample relative to the longitudinal axis of the sheet sample is shown in Fig. 1a. Creep as well as fatigue at all the test temperatures resulted in grain-boundary reorientations with a marked tendency for grain boundaries to align at about 45° relative to the stress axis (typically Fig. 1b and c). This appears to be the first instance in which a tendency for the diamond grain configuration has been observed in the same metal during creep as well as fatigue at elevated temperatures.

Wigmore and Smith [5], as well as Westwood and Taplin [6], suggest models for the occurrence of diamond pattern of grain boundaries in which the role of grain-boundary sliding is emphasized. It has been suggested [5] that grain-boundary sliding generates a defect imbalance by causing higher local strains ahead of boundaries already at 45° positions as compared to adjoining boundaries which are not initially at 45° orientations. These shear zones provide the driving force for the migration of grain boundaries, not initially at 45°, towards such orientations. The experiments of Walter and Cline [10] illustrate excellently the inter-relationship between grain-boundary sliding, the resulting shear zones and migration.

In order to elucidate the role of grain-boundary sliding in a process governed by grain-boundary migration, which is relevant to the above observations of grain-boundary reorientations during hot fatigue and creep of cadmium,

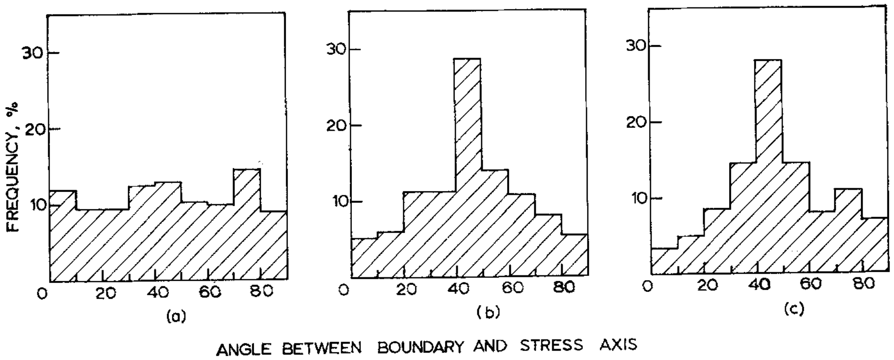

Figure 1 Angular distribution of grain boundaries relative to stress axis in cadmium (a) annealed (b) after 5% tensile strain at 150°C (0.7T_m) at 3.6 % per hour (c) after 10^4 cycles of fatigue at 25°C (0.50 T_m).

experiments were performed in which recrystallization was caused in cadmium under three different conditions of hot deformation namely, hot-working, hot fatigue and creep. Hot-working was carried out by rolling annealed (as before) cadmium at 150°C. Fatigue was performed at 1000°C on annealed cadmium. In order to induce recrystallization during creep, the annealed sample was deformed to 30% strain at liquid nitrogen temperature in the constant stress creep apparatus and then crept to 3.5% strain at 100°C using a load of 31 kg cm^{-2}. As before, the angular distribution of grain boundaries associated with the *newly formed* grains was determined in all the cases and the histograms are shown in Fig. 2. The number of grain boundaries of the recrystallized grains lying in 30 to 60° range is recorded in Table I.

TABLE I % of boundaries of recrystallized grains in cadmium in the 30 to 60° angular range relative to the longitudinal or stress axis

Treatment	% of boundaries in range 30 to 60° from stress axis
Annealed at 200°C for 2 h	35.6
Rolled at 150°C	35.2
Creep-deformed at 100°C (following straining to 30% at −196°C)	50.4
Fatigue at 100°C (3000 Hz and a mean stress of 25 kg cm^{-2})	61.0

During annealing, in the absence of an applied stress, we obtain equiaxed grains with a random distribution of grain boundaries (Fig. 1a). During hot-working, grain-boundary sliding is

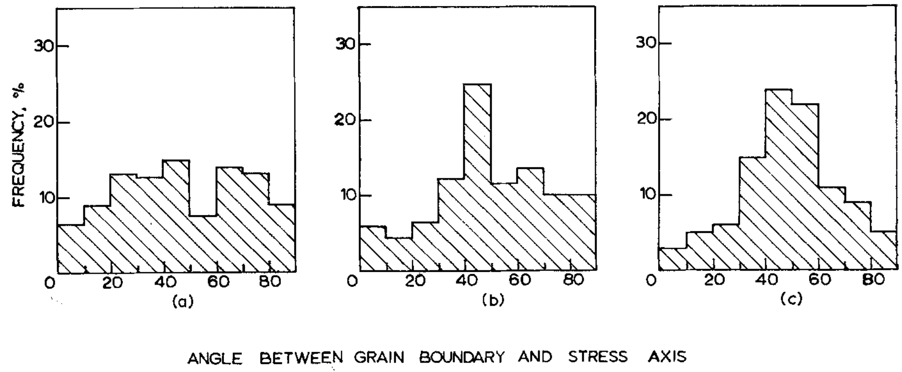

Figure 2 Angular distribution of boundaries of recrystallized grains in cadmium (a) rolled at 150°C (0.7 T_m) (b) after creep at 100°C (0.63 T_m) following 30% strain at liquid nitrogen temperature (c) after fatigue at 100°C (0.63 T_m).

suppressed as a consequence of high deformation rate, and recrystallization in this situation produces once again equiaxed grains with a random distribution of grain boundaries (Fig. 2a). The shape of the solid grain in these instances is close to Kelvin tetrakaidecahedron with the value of dihedral angle between grain boundaries being 120° when equilibrium of the forces of surface tension is established. Topological as well as thermodynamic considerations have been invoked in the study of the shapes of solid grains in annealed materials [13-15]. It has been well documented that during creep and fatigue at temperatures greater than 0.5 T_m grain-boundary sliding is an important process. When recrystallization is caused in cadmium under conditions of creep and fatigue such that grain-boundary sliding occurs, it is observed that a strong deviation from a random angular distribution of grain boundaries takes place in favour of 45° positions (Fig. 2b and c and Table I). There is clearly a determining influence of grain-boundary sliding on the process of grain-boundary migration involved in recrystallization during creep and fatigue of cadmium. This result has certainly important bearing on the observation of a tendency for the grain boundaries to align preferentially at 45° relative to the stress axis during creep and fatigue (Fig. 1b and c). A further understanding of the phenomenon seems possible only on the basis of similar topological and thermodynamic considerations as have been used in the understanding of grain shapes in annealed materials.

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Mixed conductivity of β-alumina electrolyte in aqueous concentration cells

Sodium β-alumina (Na_2O; 5.3 to 8.5 Al_2O_3 [1]) is well known for its high sodium ion conductivity, a feature which has led to its use as the electrolyte in the sodium-sulphur battery. The conductivity is highly anisotropic, being high within the basal plane of the hexagonal crystal structure, and essentially zero along the *c*-axis. This aspect was emphasized in the recent study [2] of β-Al_2O_3 single crystals as solid electrolyte in a concentration cell, where the presence or absence of an emf in the cell

$$\mathrm{Pt} \mid \mathrm{Na_2SO_4\,(aq)}_{a_1} \mid \mathrm{NaAl_{11}O_{17}\,(s)} \mid \mathrm{Na_2SO_4\,(aq)}_{a_2} \mid \mathrm{Pt}$$

was found to depend on the orientation of the crystal.

The present work was undertaken to survey the possible use of β-Al_2O_3 as a monitor for Na^+ in aqueous solutions under more general conditions.

The β-alumina was prepared in the form of a