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Subgrain boundary formation in single and polycrystalline CoO

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Subgrain boundaries were observed in creep-tested samples of single and polycrystalline CoO. The structure of the subgrain boundary was characterized by transmission electron microscopy (TEM). In single crystal CoO, subgrain boundaries are formed typically by two sets of $a_0/2\ \langle 1\,1\,0\rangle$ dislocations on nonorthogonal $\{1\,1\,0\}$ slip planes. In polycrystalline CoO, either single tilt boundaries or hexagonal networks formed by three sets of $a_0/2\ \langle 1\,1\,0\rangle$ dislocations were observed. These observations suggest that dislocation climb is the predominant creep mechanism in single and polycrystalline CoO.

1. Introduction

Direct observation of microstructure of a creep-tested material provides useful clues to the underlying creep mechanisms. Among materials with NaCl type structure, CoO has been widely studied for its creep behaviour [1–4]. Conventional etch-pit techniques have been used to reveal the dislocation distribution and subgrain-boundary formation. A few direct transmission electron microscopic observations of subgrain boundaries have also been reported by Clauer and co-workers [1, 5]. However, no detailed analysis of the dislocation structure has been carried out. Among other materials with NaCl-type structure, direct observations and analyses of dislocation networks have been reported by Amelinckx [6] for NaCl and Washburn *et al.* [7] and Narayan [8] for MgO. However, these analyses are made for materials in as deformed or as-grown conditions. The purpose of the present investigation was to report some direct TEM observations and analyses of a few typical examples of subgrain-boundary formation in creep-tested samples of single and polycrystalline CoO.

2.1. Sample preparation

Creep-tested samples of single and polycrystalline CoO were sliced approximately into thin sections of 0.05 cm × 0.25 cm × 0.25 cm. Single crystal samples were sliced parallel to the $\{1\,1\,0\}$ planes. The sliced samples were lapped on 600 grit SiC paper to a thickness of 0.09 mm (0.0035 in.) and solution etched (90% lactic, 7.5% nitric and 2.5% HF) for about 5 min. Thin films for TEM were prepared by ion micromilling. The milling operation was terminated at the first observation of a pinhole. This occurred approximately after 65 h milling at 13° tilt for an ion beam of 100 μA at 8 kV.

2.2. Electron microscopy

TEM observations were made on a Philips EM-300 K to 100 kV microscope using ± 45° single/double tilt specimen holders. Kikuchi maps were extensively used in identigying and setting up of proper diffraction conditions. Two beam bright-field conditions were used in imaging the structure. The pre-determined image-diffraction rotation calibration for the instrument was used to correlate the images with the selected-area diffraction patterns. From this information as well as the established knowledge of the slip ssytem in CoO, the Burgers vector and the nature of dislocations were easily identified.

3. Results and discussion

Electron microscopic observations for a CoO single crystal were made on a foil prepared by cutting a thin section along what appeared to be slip lines on a (1 0 0) plane of a parallelepiped specimen whose stress axis was along the [0 0 1] direction. However, on scanning the thin region, no glide dis-

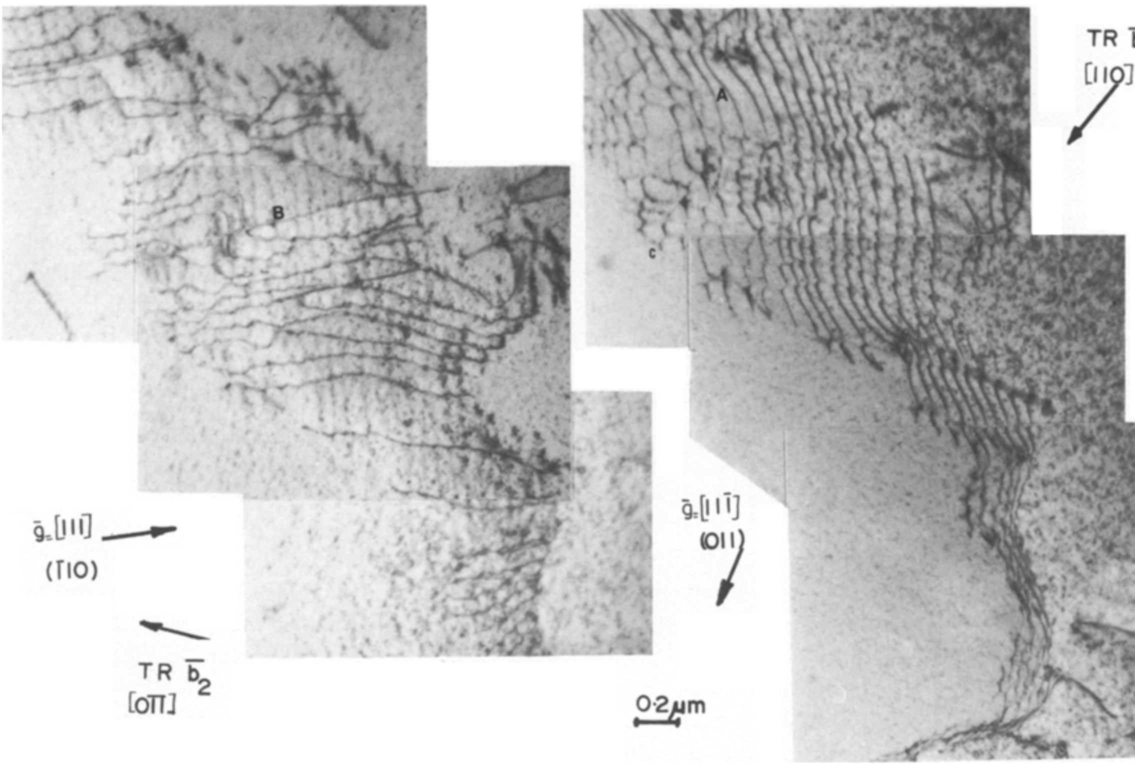

Figure 1 Subgrain boundaries in single crystal CoO creep tested at 1273 K, 13.78 MPa (2000 psi) and strain $\epsilon \approx 6\%$. Sets A and B consisting of parallel edge dislocations are in contrast for $\mathbf{g} = [1\,1\,\bar{1}]$ and $\mathbf{g} = [1\,1\,1]$ reflections

locations were observed; instead, subgrain boundaries consiting of two sets of predominantly edge dislocations were observed as shown in Fig. 1. In Fig. 1, each set of dislocations A, or B, is formed by alignment of edge dislocations on parallel slip planes. The arrangement within each set appears to be similar to a simple tilt-boundary. However, with the presence of two sets of dislocations the boundary has a twist component. Note that in each set (A or B) of Fig. 1 only one set of dislocations is in contrast. The micrograph corresponding to a $(1\,\bar{1}\,1)$ reflection is not shown here but both sets of dislocations were out of contrast in this condition. Thus from the out-of-contrast condition corresponding to $\mathbf{g} \cdot \boldsymbol{b} = 0$, the Burgers vector for each set of dislocations were identified as belonging to two non-orthogonal (120°) $\{1\,1\,0\}$ slip systems. The configuration of these dislocations with respect to stress direction is represented schematically in Fig. 2. As the details of Fig. 2 bear out, a combination of glide and climb of edge dislocations on two non-orthogonal slip systems, such as $\frac{1}{2}\,[1\,1\,0] + \frac{1}{2}\,[0\,\bar{1}\,\bar{1}] = \frac{1}{2}\,[1\,0\,\bar{1}]$ is possible. In Fig. 1, a similar reaction can be seen to have occurred at point C.

In contrast to single crystal CoO, the examination of a polycrystalline CoO sample revealed typically simple tilt boundaries or a network consisting of three sets of dislocations. Of course, this relatively minor difference in the structure of subgrain boundaries in single and polycrystalline CoO may not be significant and perhaps can be attributed to the constraints imposed by compatible

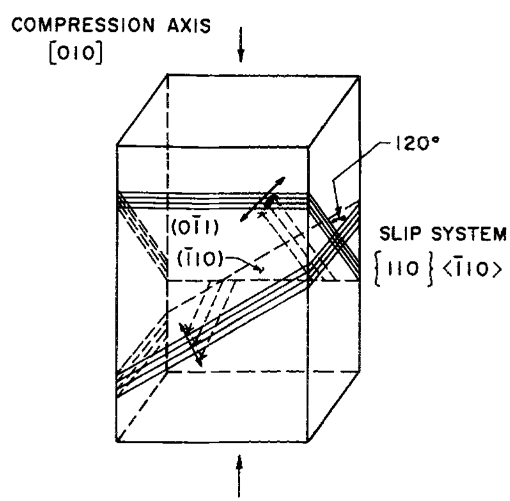

Figure 2 Non-orthogonal slip systems in CoO.

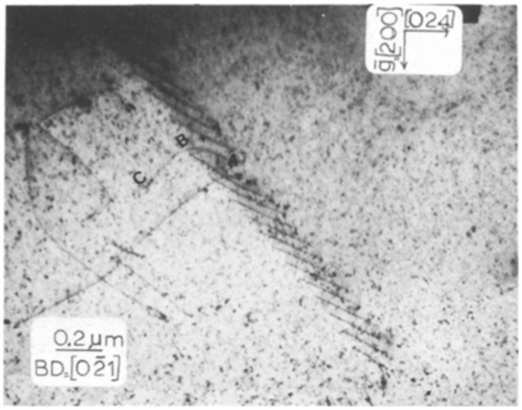

Figure 3 Subgrain boundary in polycrystalline CoO, creep tested at 1273 K, 27.57 MPa and strain $\epsilon \approx 8\%$. It consists of parallel edge dislocations forming a tilt boundary and ABC is a mixed dislocation.

deformation of different grains in polycrystalline materials and the crystallographic orientation in the case of single crystal.

Fig. 3 is in an electron micrograph of creep tested (at 1273 K and 27.57 MPa) polycrystalline CoO. It shows a typical structure of a simple tilt boundary formed by parallel edge dislocations of Burgers vector $a_0/2\langle 1\,0\,1\rangle$. The plane of the tilt boundary was determined to be $\{0\,1\,2\}$ and the tilt angle was estimated to be 1.4°. A mixed dislocation such as ABC is a reminiscence of the glide process that must have led to the formation of tilt boundary.

Fig. 4a shows a typical three-dimensional network consisting of dislocations A, B and C. As apparent in Fig. 4a, all three sets of dislocations A, B and C are in good contrast for reflection $\mathbf{g} = [0\,2\,2]$. However, as shown in Fig. 4b, the A dislocations are out of contrast for $\mathbf{g} = [\bar{2}\,2\,2]$. From the $\mathbf{g} \cdot \boldsymbol{b} = 0$ condition, and trace analysis, the Burgers vectors, of A, B, and C were identified to be $a_0/2\ [1\,1\,0]$, $a_0/2[\bar{1}\,0\,1]$ and $a_0/2[0\,1\,1]$. At nodes, the following dislocation reaction was identified to occur: $a_0/2[1\,1\,0] + a_0/2[\bar{1}\,0\,1] \rightarrow a_0/2[0\,1\,1]$. This reaction occurs on two intersecting slip planes, $(\bar{1}\,1\,0)$ and $(1\,0\,1)$, which are at an angle of 120° to each other. The resultant dislocation is a pure edge lying parallel to $[1\,\bar{1}\,1]$ as illustrated in Fig. 5. The slip plane of the resultant dislocation is $(2\,\bar{1}\,1)$, which is not a favourable slip plane for the NaCl-type structure and, therefore, the resultant dislocation remains sessile, forming a barrier to other dislocations and contributing to strain hardening.

According to Weertman's [9] creep mechanism, an edge dislocation climbs out of its slip plane to clear its obstacles and sweep out a new area and produce creep strain. During steady state creep, the strain hardening rate due to formation of entanglements or obstacles such as sessile dislocations is balanced by the rate of recovery due to climb of edge dislocations over the obstacles. Recently, Krishnamachari [3] has suggested that dislocation climb is the recovery mechanism during high temperature creep of CoO single crystals. Based on the creep data of polycrystalline CoO, Krishnamachair and Notis [4] have suggested that dislocation climb is also operative in polycrystalline CoO. The present TEM observation of subgrain boundaries in single and polycrystalline CoO supports the dislocation climb mechanism.

If dislocation climb is the predominant mechanism for creep of CoO at high temperature then the formation of subgrain boundaries indicates that

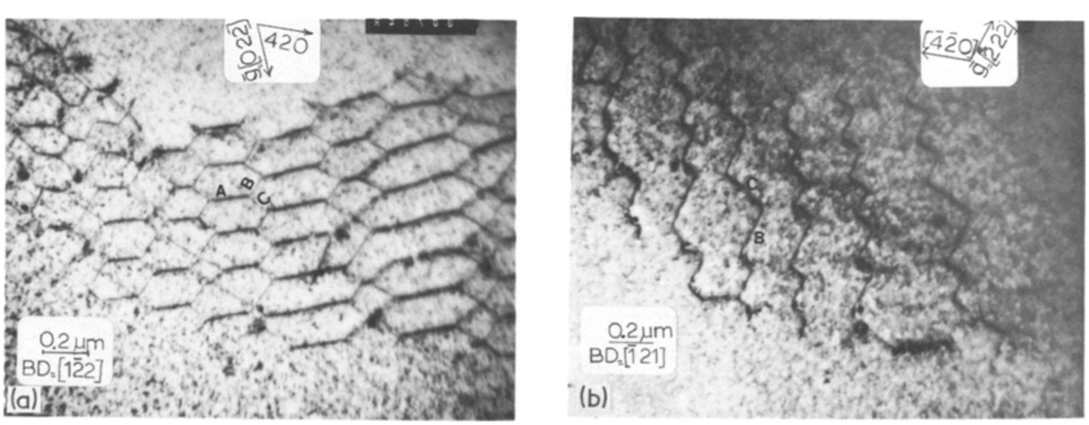

Figure 4 Three-dimensional dislocation network in polycrystalline CoO, creep tested at 1273 K 27.57 MPa and strain $\epsilon \approx 8\%$.

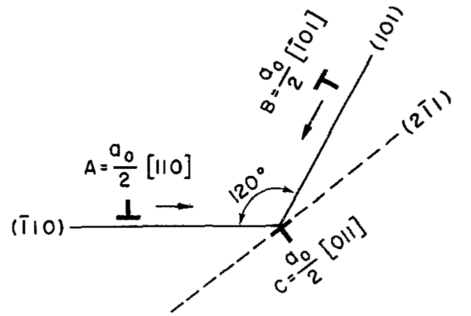

Figure 5 Formation of sessile dislocation.

the total creep strain must be related to the misorientation across the subgrain boundary and the dislocation density in the subgrain boundary. This quantitative aspect as well as the effect of impurities on dislocation structure and hence on subgrain boundary formation are being further investigated.

4. Conclusions

(1) Subgrain boundaries were observed in creep-tested samples of single and polycrystalline CoO. Subgrain boundaries were observed to be formed by two sets of dislocations on non-orthogonal {1 1 0} planes. In polycrystalline CoO either simple tilt boundaries or a three-dimensional network consisting of three sets of dislocations were observed.

(2) The formation of tilt boundaries and network formation confirm that dislocation climb is operative during high temperature creep of single and polycrystalline CoO.

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