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Creep recovery of CoO single crystals

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The recovery of creep resistant substructure in CoO single crystals was studied at temperatures of 1000, 1050, and 1100° C in air. Compressive creep specimens were crept under a stress of 13.8 MN m^{-2}* to a strain early in the secondary stage of creep, then allowed to recover for varying periods of time under a reduced stress of 0.69 MN m^{-2}. Recovery was detected by increased amounts of creep strain which occurred upon reapplication of the 13.8 MN m^{-2} stress. An apparent activation energy of 71.7 ± 10 kcal mol^{-1} was obtained for the recovery process. Experimental evidence suggests that the primary recovery mechanism involves the climb of dislocations within subgrain boundaries.

1. Introduction

Recovery of high temperature creep resistant substructure is an important design factor in many applications. As a result, Dorn and co-workers [1, 2] studied the effect of reduced stress on creep deformation in pure Al. They observed that a reduced stress treatment resulted in the recovery of creep resistant substructure by subgrain-boundary migration. Reduced stress treatments with Pb [3, 4] have also demonstrated creep recovery. Bell [5] was observed no such recovery in a dilute Al–Mg solid solution, which he attributed to pinning of subgrain boundaries by the solute atoms. Bell *et al.* [6] have observed creep recovery in rutile single crystals in air. They suggested that the primary recovery mechanism involves the sweeping out of dislocation barriers within the material by the migration of dislocation walls. Krishnamachari *et al.* [7] have also studied the creep recovery of rutile single crystals in vacuum and under various reduced stresses. They suggested that the basic recovery mechanism in near stoichiometric [6] and vacuum-reduced rutile [7] under reduced stress is the same. They also suggested that the creep recovery in air is stress assisted.

This investigation was initiated to study the recovery of creep resistant substructure in cobaltous oxide single crystals under reduced stress treatment. Cobaltous oxide was chosen as test material because of its previously reported creep data [8, 9]. Recovery is an important creep parameter. An intimate understanding of the formation, nature, and recovery of the creep resistant substructure is essential to solve the problem of steady state creep. Therefore, a systematic investigation is needed on the recovery of creep resistant substructure.

2. Experimental procedure and results

Single crystals of CoO were grown by d.c. arc technique [10, 11] from Co rods 5 mm x 15 cm of 99.999% purity. The as-grown CoO crystal boules were oriented using back-reflection X-ray technique. Specimens having ⟨1 0 0⟩ axial orientation (Fig. 1) were cut for {1 1 0}⟨1 1 0⟩ deformation. The specimens were polished on dry 600-grit SiC grinding paper to 0.406 cm x 0.203 cm x 0.203 cm.

Creep recovery tests were performed in air at temperatures of 1000, 1050 and 1100° C for various periods of time, t_r. The creep recovery apparatus consists of a split furnace which was proportionately controlled to +2° C. The sample was loaded by polycrystalline alumina rams with the deformation continuously monitored, using LVDT system and recorded autographically. A stress of about 0.69 MN m^{-2} was imposed on the sample throughout the heating stage in order to maintain tautness in the loading assembly. In each

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* 13.8 MN m^{-2} = 2000 psi.

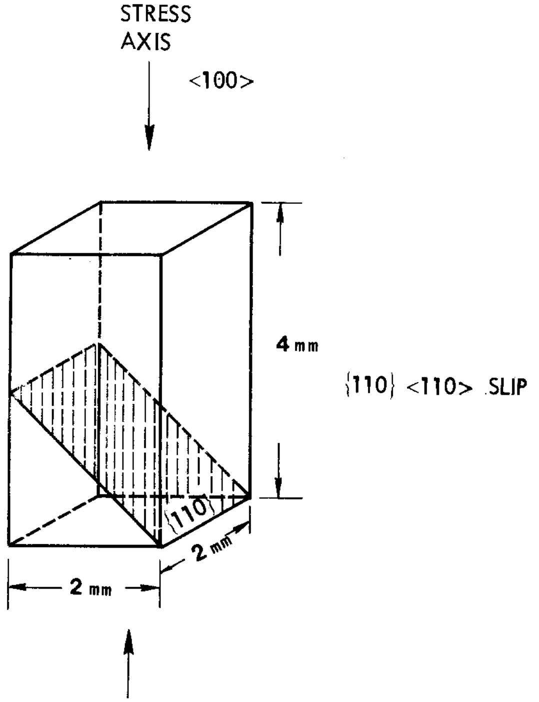

Figure 1 Orientation and size of CoO single crystal specimen.

test the load was applied by pouring lead shots in a container on one end of a lever system to minimize the chance of impact loading and difference in the rate of load application.

Specimens were crept under a stress of 13.8 MN m^{-2} (200 psi) to the same creep strain in the early secondary stage of creep, $\epsilon_c = 0.08$ (and to the same temperature compensated time $\theta_c = 4.7 \times 10^{-10}$ min), then allowed to recover for varying periods of time t_r, under a reduced stress of 0.69 MN m^{-2} (100 psi). Recovery was detected by the increased amount of creep strain which occurred upon re-application of the 13.8 MN m^{-2} stress. A typical example of a recovery test under a reduced stress of 0.69 MN m^{-2} is shown in Fig. 2. After a recovery period of 180 min at 0.69 MN m^{-2} and 1000° C, the initial stress of 13.8 MN m^{-2} was applied; the plastic creep strain DG resulted. The fractional recovery index n, which is defined [6] as:

$$n = \frac{\epsilon_r - \epsilon_u}{\epsilon_c - \epsilon_u}$$

was determined at each test temperature for various recovery times t_r, and plotted on a log–log graph as shown in Fig. 3. Identical substructural states can be obtained by allowing samples to recover for appropriate times t_{r1} and t_{r2} at temperatures T_1 and T_2 to yield the same degree of recovery (value of n). For a given amount of recovery

$$t_{r1} \exp(-H_r/kT_1) = t_{r2} \exp(-H_r/kT_2) \quad (2)$$

which upon rearrangement yields

$$H_r = \frac{k \ln(t_{r1}/t_{r2})}{(1/T_1 - 1/T_2)}, \quad (3)$$

where H_r = apparent activation energy for the recovery process, k = Boltzmann's constant, t_{r1}, t_{r2} = time under reduced stress.

It can be observed from Fig. 3 that the data for three test temperatures fall on three parallel straight lines on log–log coordinates. The activation energy for recovery was determined to be 71.7 ± 10 kcal mol^{-1}. The validity of this value is demonstrated by the data in Fig. 4, in which n is plotted on log–log coordinates against θ_r, the tem-

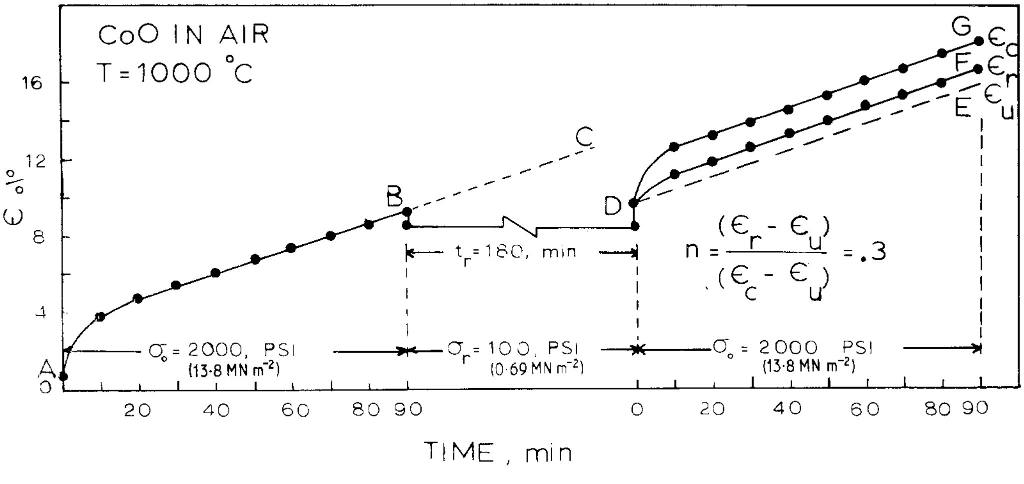

Figure 2 Illustration of creep recovery experiment.

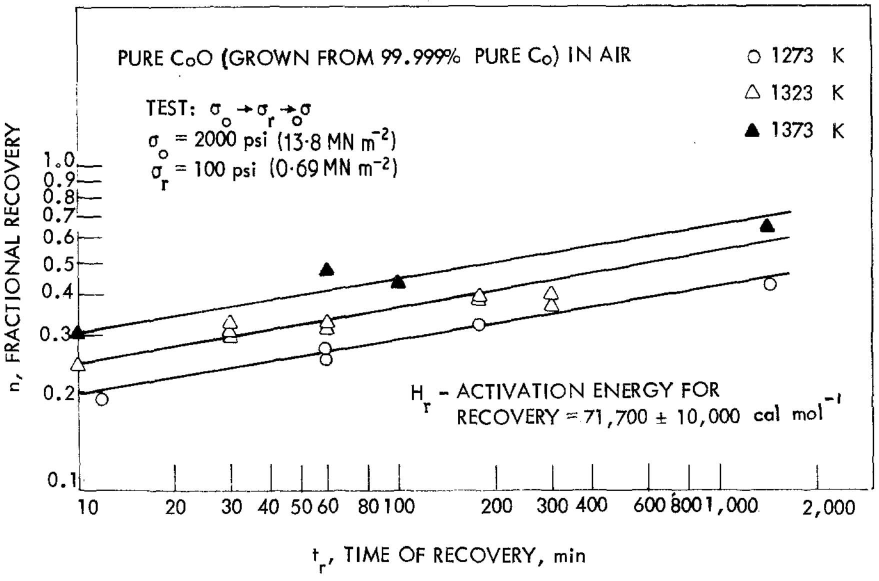

Figure 3 Fractional recovery index as a function of time at a reduced stress of 0.69 MN m^{-2} (100 psi).

perature compensated time for recovery ($\theta_r = t_r \exp(-H_r/kT)$). Since all datum points fall on the same line, this indicates that H_r is insensitive to the temperature and substructural details attending creep recovery over the range of conditions investigated.

Creep tested (at 1000° C, 13.8 MN m^{-2}) and recovered (24 h at 0.69 MN m^{-2} reduced stress) samples, were sliced parallel to the (1 1 0) slip plane into small samples of 0.5 mm x 2.54 mm x 2.54 mm. The sliced samples were lapped on 600-grit SiC paper to a thickness of 0.1 mm. These samples were etched (etchant: 90% lactic acid, 7.5% nitric acid, and 2.5% HF) for 5 min and further thinned in ion micro milling machine. A hole was formed in the sample after 60 h of argon ion-bombardment at 8 kV and 100 MA beam current. Figs. 5 and 6 are TEM micrographs of creep tested (13.8 MN m^{-2}, 1000° C) and recovered (24 h) samples.

3. Discussion

Krishnamachari and Jones [8] have observed steady state creep in CoO single crystals in the same range of temperature and stress. Steady state creep has been regarded as arising from a balance

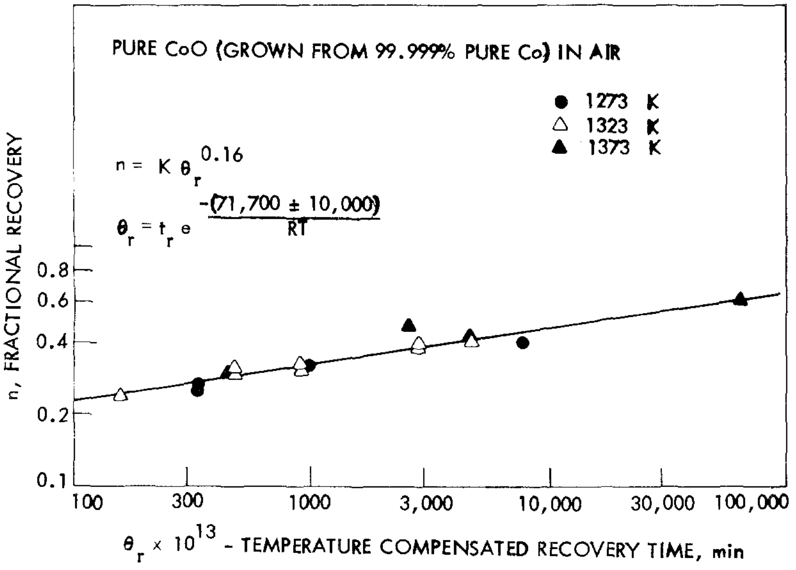

Figure 4 Thermal recovery of creep.

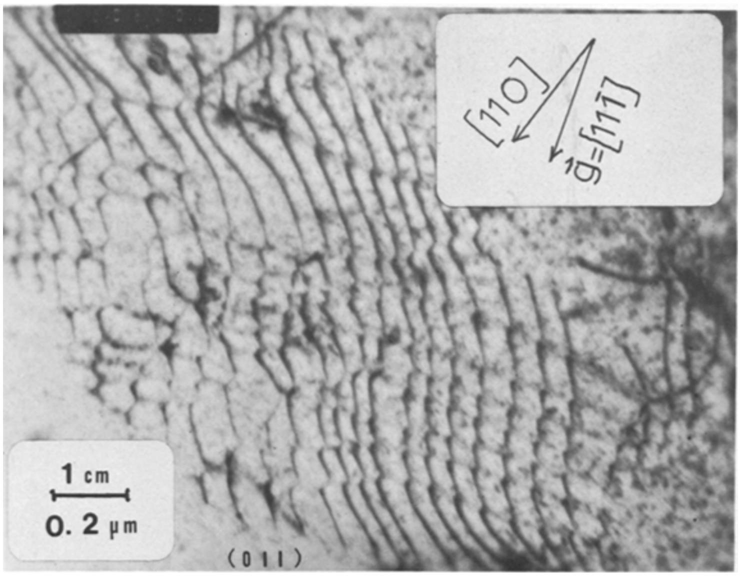

Figure 5 Dislocation configuration of CoO creep specimen crept for 60 min at 13.8 MN m^{-2} (2000 psi), 1000° C.

of two competing processes: work hardening and recovery [12–14]. Clauer *et al.* [9] and Krishnamachari and Jones [8] have observed the formation of subgrain walls during high temperature creep. At high temperatures dislocations can climb out of the slip plane by diffusional mass transport of atoms and form subgrain boundaries. Dislocation multiplication and glide are regarded as hardening mechanisms and dislocation annihilation and climb as recovery mechanism. Krishnamachari and Jones [8] have suggested that dislocation climb mechanism is operative during high temperature creep of CoO single crystals. They obtained a fifth power stress exponent for the steady state creep rate, and the activation energy for creep was 66.1 ± 5.4 kcal mol^{-1}, which they attributed to the oxygen controlled pipe diffusion. The observed activation energy (71 ± 10 kcal mol^{-1}) for the recovery of creep resistant substructure is in close agreement with the activation energy for high temperature creep.

Fig. 5 is a TEM micrograph consisting of parallel edge dislocations of Burgers vector $a_0/2$ [1 1 0] that are climbed out of the slip plane ($\bar{1}$ 1 0). The sample was creep tested at 13.8 MN m^{-2} (2000 psi) and 1000° C, and the formation of tilt boundaries indicate the dislocation climb is operative during high temperarure creep of CoO single crystals. Fig. 6 is a TEM micrograph of a creep recovered sample for 24 h. It consists of

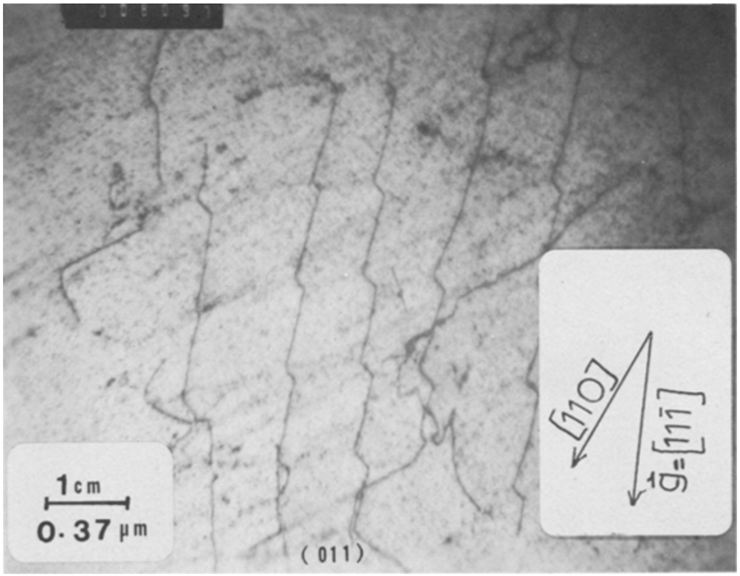

Figure 6 Dislocation configuration of CoO creep specimen crept for 60 min at 13.8 MN m^{-2}, 1000° C and recovered at t_r = 24 h at 0.69 MN m^{-2}, 1000° C.

parallel edge dislocations of Burgers vector $a_0/2$ [1 1 0]. It may be noticed in Fig. 6 that dislocations might have climbed out of the slip plane ($\bar{1}$ 1 0) by creation or annihilation of vacancies at jogs. It may also be noticed an increase in dislocation spacing and decrease in dislocation density, indicating that during recovery under reduced stress the dislocations might have climbed out of the slip plane and annihilated with each other.

These observations appear to be consistent with a model which attributes that the recovery of creep resistant substructure to dislocation climb. Under constant stress conditions subgrains are formed and the creep recovery at constant stress is controlled by climb of edge dislocations within subgrains. Under reduced stress conditions the edge dislocations still climb out of the slip plane and annihilate with each other. The observed increase in dislocation spacing and decrease in dislocation density suggests that the recovery mechanism under reduced stress is also dislocation climb.

4. Conclusions

The creep resistance of cobaltous oxide single crystals in the temperature range 1000 to 1100° C is reduced by reduced stress treatment. This is due to the recovery of creep resistant substructure. The apparent activation energy for recovery is in close agreement with the apparent activation energy for creep. TEM micrographs revealed the formation of subgrain boundaries, and the dislocation density within subgrains decreased, whereas the dislocation spacing increased, during the recovery process. The recovery of creep resistant substructure under reduced stress conditions also involves the climb of edge dislocations within the subgrain boundaries.

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