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Negative creep and recovery during high-temperature creep of MgO single crystals at low stresses

K. S. RAMESH, EIICHI YASUDA, SHIUSHICHI KIMURA
Research Laboratory of Engineering Materials, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-Ku, Yokohama 227, Japan

High-temperature creep equipment with very high precision has been used to measure the creep of MgO single crystals above 1948 K and stresses lower than 4 MPa. A transition in exponent, *n*, from 3 at stresses higher than 2 MPa to almost unity at lower stress region was observed. Since in a single crystal deformation can only occur by the generation and movement of dislocations, the transition in stress exponent from high to low stress region cannot be interpreted in terms of a change from dislocation to diffusional creep processes. Decreasing the stress by a small amount during steady-state creep resulted in an incubation period of zero creep rate before creep commenced at lower stress. However, large stress reduction led to a period of negative creep during which the dislocation substructure coarsens and the subgrain cell boundaries straighten. On the basis of dislocation substructure studies, it is proposed that the kinetics of backflow are thought to be based on the local network refinement caused by the reverse movement of dislocations and that recovery is necessary before further movement of dislocation can occur. It is shown that the network theory proposed by Davis and Wilshire can satisfactorily account for all stress reduction observed during forward creep.

1. Introduction

As early as 1946, Orowan and West [1] proposed that strain hardening is exactly balanced by recovery to maintain a constant mechanical state, in which the flow stress, σ, does not vary with time. Subsequently, stress reduction experiments were proposed by Mitra and Mclean [2], and later by Davies and Wilshire [3], as a method for determining the rate of recovery during creep. Several authors [4–6] applied this technique extensively to a number of pure metals and ceramics for studying the rate-controlling mechanism during creep. They reported that stress reduction during steady state creep is always followed by an incubation period of zero creep.

However, the above results and interpretations are in conflict with the results of Ahlquist and Nix [7] and Blum *et al.* [8] who reported the occurrence of an incubation period of zero creep rate to be very much dependent on the extent of stress reduction performed. Based on the incubation period of zero creep, they found [7, 8] that a critical degree of stress reduction exists, after which the creep rate is zero for some time. Depending on whether the stress change is similar or larger than the critical stress, the creep rate immediately after the stress change is negative or positive. The occurrence of a period of negative creep immediately after large stress reduction has often been explained on the basis that creep occurs under an effective stress [7] as:

$$\sigma_{\text{eff}} = (\sigma_A - \sigma_i) \quad (1)$$

where σ_A is the applied stress and σ_i the internal stress developed during creep.

In spite of the wide use of stress change tests for investigating creep deformation behaviour, no attempt has been made to evaluate the results on the basis of dislocation substructure which develops and changes after stress reductions.

In the present investigation, stress change experiments were carried out at temperatures above 1948° C and stresses lower than 4 MPa, particular attention being given to any incubation period of zero creep rate that may follow a stress change, with special reference to changes in substructural features.

2. Experimental procedures

The single crystals of MgO used in the present study were of the same purity as that used in our earlier studies [9]. Parallelepipedic samples with dimensions 4 mm × 4 mm × 8 mm were used. All the samples were deformed in compression under constant load along the ⟨1 0 0⟩ direction in a creep machine specially fabricated for this study (Fig. 1). Creep measurements were done with a pair of differential linear variable transducers (Hottinger Baldwin Messtechnik GMBH type W 0.5T) having a resolution better than 10^{-5} cm. The output from these transducers was amplified (MBM-MGT 33.0 type) and then fed directly to a recorder. Temperature measurement was made by using a Pt · 6% Rh–Pt · 30% Rh thermocouple, and the temperature was maintained constant within

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± 2 K for any test. Stresses employed ranged from 1.5 to 4 MPa.

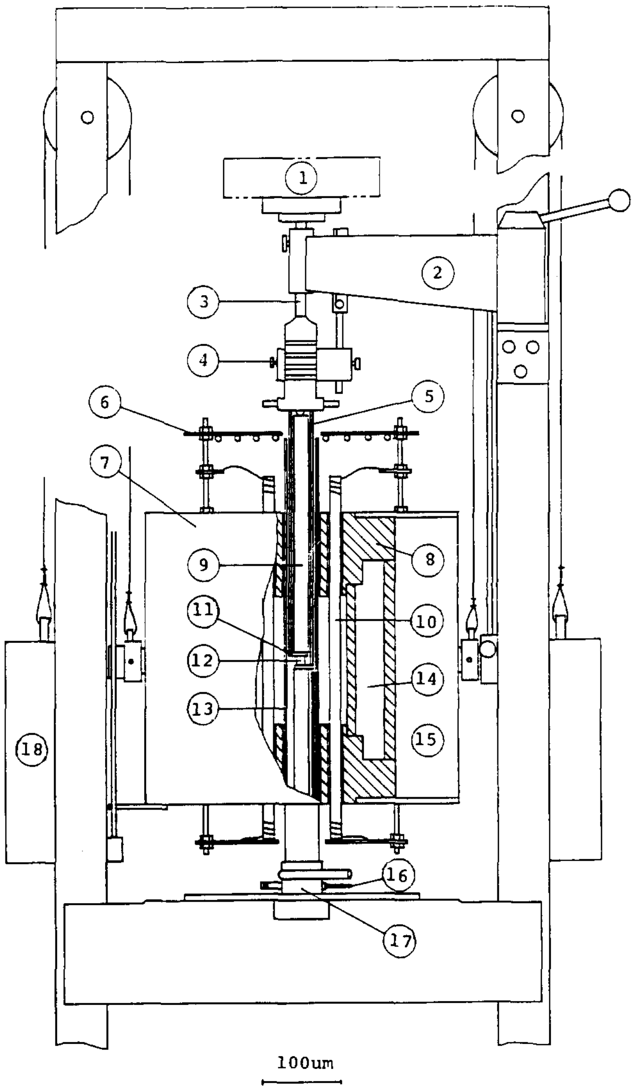

Figure 1 Sectional view of the high-temperature low-stress compression creep equipment. 1, Loading pan; 2, movable arm; 3, connecting steel rod; 4, LVDT housing; 5, saphire detecting rod; 6, water cooling plate; 7, furnace – max. 1750° C; 8, refractory muffle; 9, fused cast alumina push rod; 10, $LaCrO_3$ heating element; 11, single crystal alumina plate; 12, specimen; 13, recrystallized alumina tube; 14, bubble alumina insulation; 15, Kaowool fibre insulation; 16, thermocouple; 17, gas port; 18, balancing weight.

3. Experimental results

3.1. Stress reductions during creep

Small stress reductions of the order 0.05 to $0.170\,\sigma_A$ were made during steady stage creep. In every case the stress change was followed by a period of zero creep. The length of this incubation period (Δt) increased with increasing stress reduction, $\Delta\sigma$ (Fig. 2).

Furthermore, in order to establish if the incubation period was a true aspect of creep and not due to measuring technique, the creep stress was reduced from 3 to 2.5 MPa for various times at constant temperature; then the stress was again increased to the original stress. In all instances, incubation period was observed (Fig. 3). The incubation period was followed by a new creep rate always lower than the creep rate obtained in an uninterrupted creep test, carried out at the reduced stress level. This lower creep rate continued for a while, but eventually returned to the value for the reduced stress level.

However, with larger stress reductions, $\Delta\sigma$, the incubation periods, Δt, tended to become excessively long. With sufficiently large stress change, negative deformation immediately followed.

3.2. Stress dependence of creep and recovery

The stress dependence of the steady state creep rate ($\dot{\varepsilon}$) for single crystals at temperatures above 1948 K and lower than 4 MPa, is shown in Fig. 4. A noteworthy observation from the figure is the transition of stress exponent from 1 to 3 for low and high stress.

When steady state creep conditions had been established, the stress was instantaneously reduced by a small amount. These stress reductions were followed by an incubation period of duration Δt, when the creep rate is zero. Following previous authors [10–12] the recovery parameter $\Delta\sigma/\Delta t$ was calculated and found to be linearly dependent on the creep rate prior to stress change, Fig. 5. The results show that the

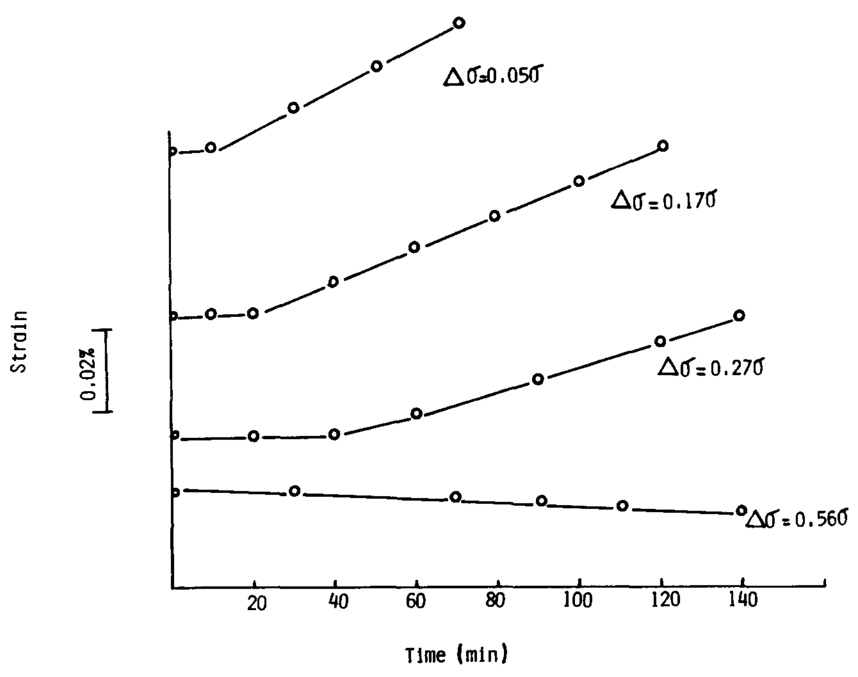

Figure 2 Incubation periods following stress reductions during steady state creep at 4 MPa and 1948 K.

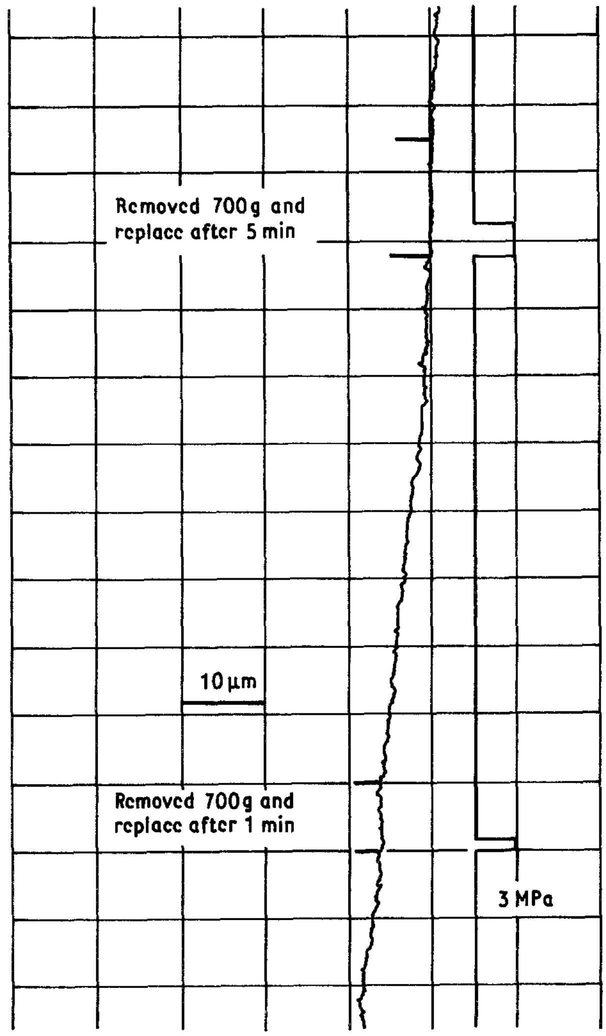

Figure 3 Strip chart recordings for MgO single crystals showing the effect of stress decrement/increment on incubation periods.

steady state creep rate is directly proportional to the rate of recovery. Birch and Wilshire [13] have also observed a similar relationship for MgO polycrystal in the region of $n = 3$, but at stresses higher than 50 MPa.

4. Discussion

High-temperature low-stress creep of MgO single crystals shows a transition in the stress exponent from 1 to 3 as the stress is increased. The former value of $n = 1$ is not anticipated for single crystals as it is usually associated with boundary mechanisms for polycrystalline materials. However, such a transition has been observed for creep of CaO [14] and aluminium [15] single crystals as well as polycrystalline materials. Barrett *et al.* [15] observed for aluminium a dislocation subgrain structure during creep, and the transition is attributed to a change in the stress dependence of the network density. Microstructural studies of the crept sample revealed a subgrain network (Figs 6a and b). In order that the model proposed by Barrett *et al.* [15] for aluminium to be valid for MgO at low stresses, i.e. in the region where $n = 1$, it is necessary to ascertain if dislocation density within subgrains becomes independent of stress. Definitive experiments at low stresses and subsequent analysis are planned for the future.

4.1. Backflow phenomenon in creep

During steady state creep of MgO single crystals a small stress decrease results in an instantaneous specimen contraction which is followed by an incubation period of zero creep rate, before entering a new positive creep rate. However, with large stress reductions negative creep was observed which implies that the stress

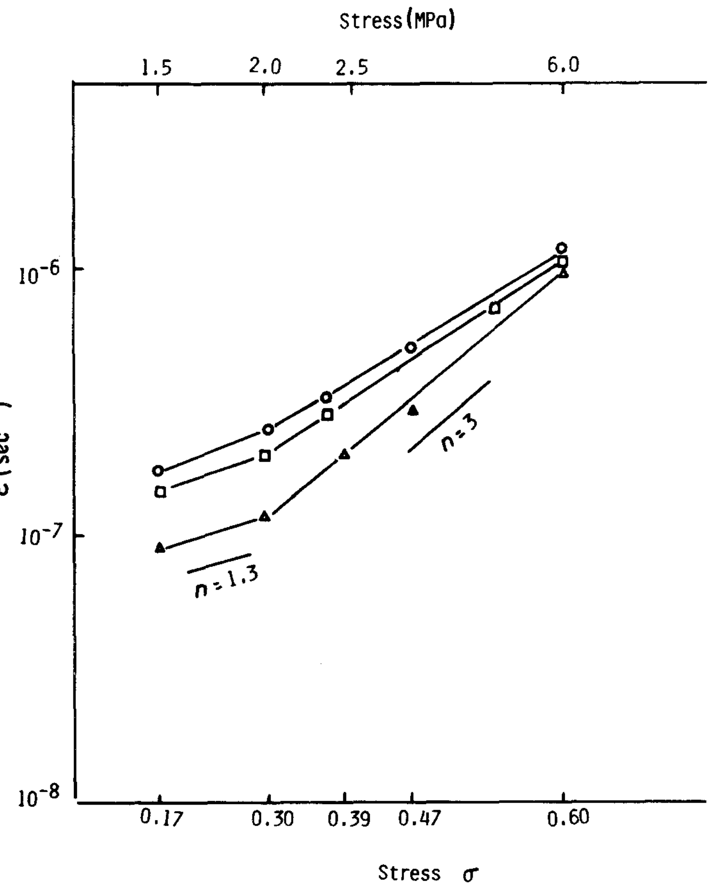

Figure 4 Steady state creep rates as a function of applied stress. ○, 2008 K; □, 1973 K; △ 1948 K.

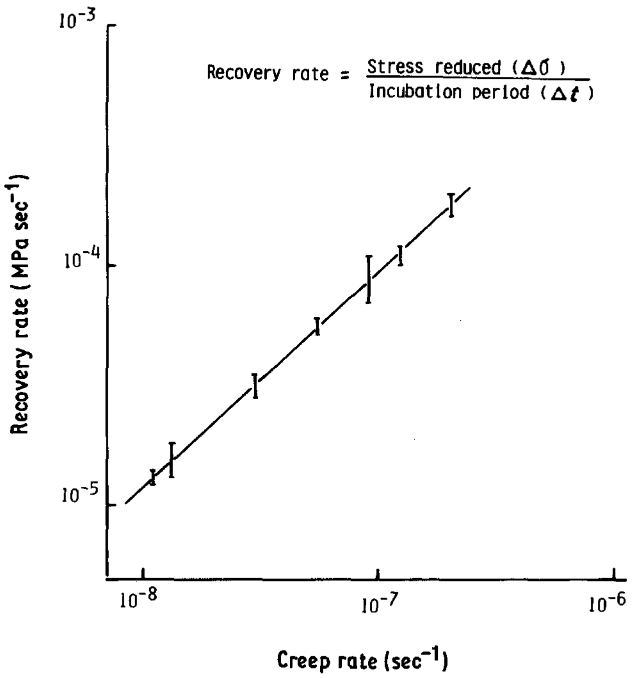

Figure 5 Relationship between recovery parameter and steady state creep rate before stress change.

has been reduced to below the level of the internal stress and that the creep behaviour is governed by the effective stress ($\sigma_A - \sigma_i$).

It is well known that the compression creep test does not induce a simple stress state in the sample and the structure is heterogeneous [16]. Fig. 7 shows the structure in the middle portion of the specimen after creep to a strain of 0.038 and recovered fully for 30 min at 198 MPa and 1973 K. Different types of structures can be observed in different localities of the sample, with certain areas consisting of small subgrain cells in the direction of ⟨0 1 1⟩. Equiaxed substructure was observed mosly in the bottom part of the samples. During the primary creep piling of dislocations, and deformation bands were observed in the bottom part of the specimen, which indicate that they play an important role in the formation of the subgrain. Such a heterogeneity of substructure has also been observed on copper [17] and molybdenum [18]. No convincing explanation can be provided for such a non-uniformity of structure except that it can be attributed to the operation of two glide planes out of four in parallelepipedic specimens [19–21]. Hasegawa *et al.* [17] and Gibeling and Nix [22] have reported that the heterogeneous structure associated with subgrain formation provides some driving force for backflow and concluded that the source of anelasticity is creep substructure.

In our earlier work it was shown that the subgrain diameter is inversely proportional to applied stress [9]. Therefore, any change in applied stress should affect the mean diameter. Obviously, this change will partly involve sub-boundary migration [23]. Fig. 8a shows the microstructure of a specimen crept to a strain of 0.038 at 1.98 MPa and 1973 K. The subgrain cell of size 50 μm can be observed inside the sub-boundaries of size 300 μm. However, when another specimen (Fig. 8b) was crept to nearly the same strain 0.04 at a stress of 2.5 MPa, and subsequently recovered to 1.98 MPa for 30 min, evidence of subgrain coarsening and straightening of curved subgrain cells is seen. The microstructure shows dislocations blocked against subgrain boundaries and some of them have been arrested when moving backward, due to their mutual interactions. It is complex to quantify the contributions of different mechanisms to backflow, but it is evident that more than one mechanism contributes to the backflow.

From the above discussion it is clear that subgrain boundaries do move during anelastic flow and that backflow most likely involves motion of dislocations from the interior to the boundaries. This can be corroborated with the microstructure and is in agreement with the work of Gibeling and Nix [22].

Davies and Wilshire [3] have recently suggested that the dislocation mechanism for forward creep is the growth of the three-dimensional dislocation network to generate links in the network long enough to act as dislocation sources. Once a link of sufficient length is formed, a burst of slip takes place increasing the local dislocation density, so that the next slip event is likely to occur in another place. Thus, the rate-controlling process is considered to be the rate of recovery and growth of network.

Following stress reduction, there can be no sources

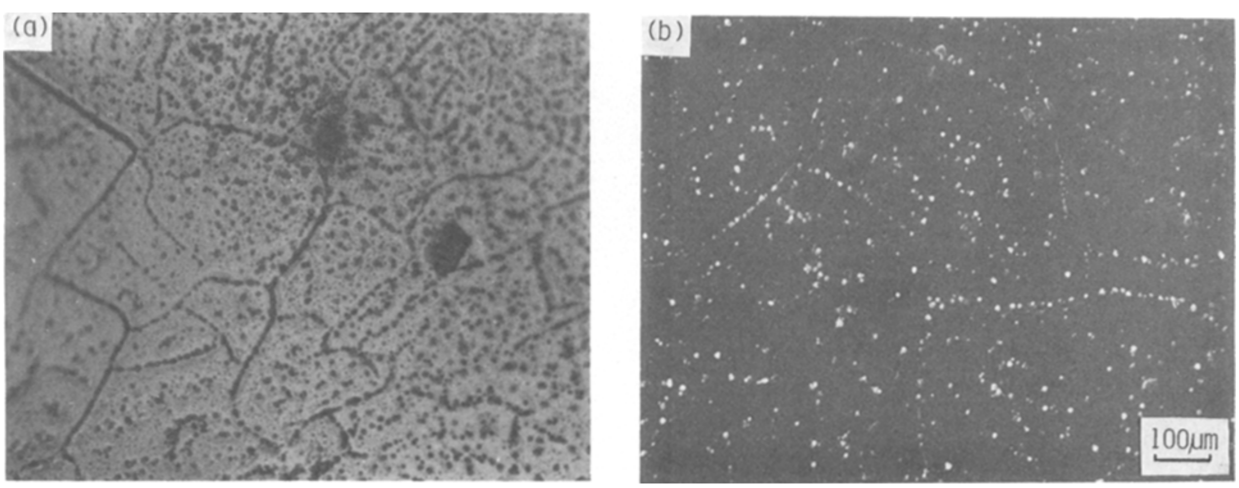

Figure 6 Evolution of creep substructure during steady state creep: (a) optical microscopy; (b) scanning electron microscopy.

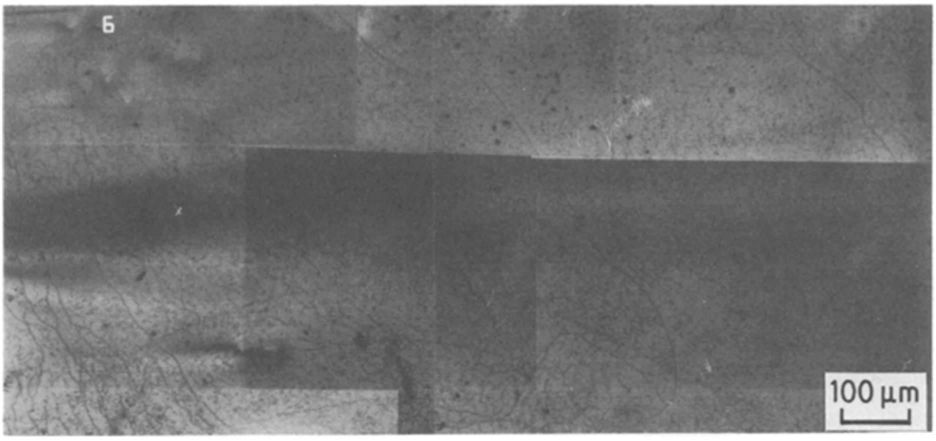

Figure 7 Substructure in the middle portion of the specimen after crept to a strain of 0.038 and recovered from 2.5 to 1.98 MPa for 30 min at 1973 K.

capable of producing forward creep, and an incubation period of zero creep rate will result. Under these conditions, the direct relationship between creep rate and $\Delta\sigma/\Delta t$ presented in Fig. 5 is usually obtained. Depending upon the extent of stress reduction, this will cause some relaxation of bowing in bowed dislocations, and run-back of any dislocation pile-ups [3]. In the case of small stress reduction, they will be small, very rapid, and not distinguishable in time from instantaneous contraction. Thus the only time-dependent process will be the recovery of the network structure to produce dislocation links sufficiently long to be unstable under the reduced stress; these will then act as slip sources and initiate forward creep.

When a large stress reduction is made the enormous driving force provided by the internal stress will cause dislocations blocked against sub-boundaries by the applied stress to move backward after unloading.

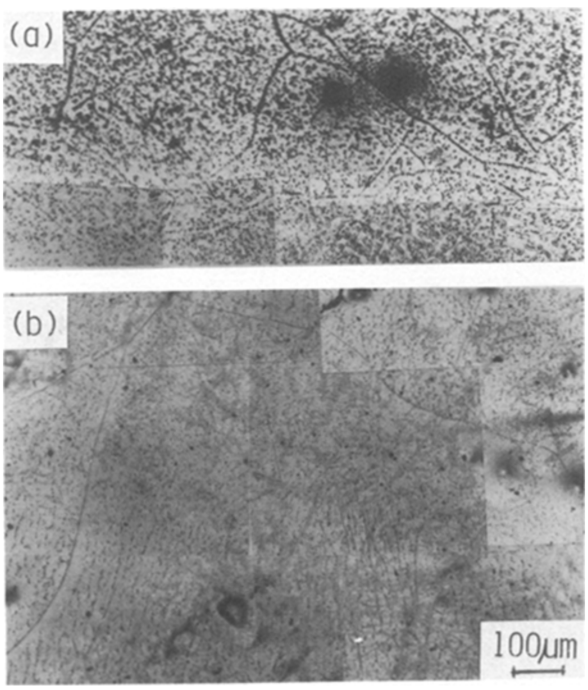

Figure 8 Dislocation structure in the subgrain interior of MgO after a creep at 1973 K: (a) crept at 1.98 MPa and a strain of 0.04; (b) crept at 2.5 MPa and a strain of 0.038 followed by 30 min recovery after unloading to 1.98 MPa.

Recovery processes are required to coarsen the network before run-back can occur, which leads to the observation of negative creep.

The presence of internal stress, σ_i, only indicates the reduction of stress before the dislocations moving in the backward direction from the undispersed pile-ups cause sufficient interactions to refine the network, thus requiring the recovery process to take place before the relaxation process is completed.

5. Conclusions

High temperature creep of MgO single crystals in the stress range 1.5 to 4 MPa and temperatures between 1948 and 2023 K has been investigated. The results can be summarised as follows:

1. A transition in the stress exponent from 1 to 3 was observed as the stress was increased.
2. Subgrains were formed during creep.
3. Subsequent stress decreases by a small amount, result in incubation periods of zero creep rate. The rate-controlling step is the growth of a dislocation network to produce links of sufficient length capable of operating as slip sources, as proposed for forward creep by Davies and Wilshire [3].
4. However, for large stress reductions, the driving force provided by internal stress causes dislocations, blocked against sub-boundaries by applied stress, to move backwards after unloading. Recovery processes are required to coarsen the network.
5. Microstructural evidence before and after stress reduction reveals that sub-boundaries move, and demonstrates evidence of coarsening of sub-boundaries.

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References

1. E. OROWAN and J. WEST, *Scotland Iron Steel Inst.* **45** (1946) 54.
2. S. K. MITRA and D. McLEAN, *Proc. Roy. Soc. A* **295** (1966) 288.
3. P. W. DAVIES and B. WILSHIRE, *Scripta Metall.* **5** (1971) 475.
4. P. L. THREADGILL and B. WILSHIRE, *Met. Sci.* **8**

(1974) 117.
5. J. M. BRICH and B. WILSHIRE, *Proc. Brit. Ceram. Soc.* **25** (May 1975) 227.
6. P. W. DAVIES, G. NELMES, K. R. WILLIAMS and B. WILSHIRE, *Met. Sci. J.* **7** (1973) 37.
7. C. N. AHLQUIST and W. D. NIX, *Scripta Metall.* **3** (1969) 679.
8. W. BLUM, J. HAUSSELT and G. KONIG, *Acta Metall.* **24** (1976) 293.
9. E. YASUDA, K. S. RAMESH and S. KIMURA, 1st International Conference on Refractories preprint November (The Technical Association of Refractories, Japan, 1983) p. 171.
10. J. D. BAIRD, *Jernkontorests Ann.* **155** (1971) 311.
11. K. R. WILLIAMS and B. WILSHIRE, *Met. Sci.* **7** (1973) 176.
12. B. SIDEY and B. WILSHIRE, *Met. Sci. J.* **3** (1969) 56.
13. J. M. BRICH and B. WILSHIRE, *J. Mater. Sci.* **9** (1974) 794.
14. P. J. DIXON-STUBBS and B. WILSHIRE, *Phil. Mag.* **A45** (1982) 519.
15. C. R. BARRETT, E. C. MUEHLERSEN and W. D. NIX, *Mater. Sci. Eng.* **10** (1972) 33.
16. T. BRETHEAU and C. DOLIN, *J. Mater. Sci.* **13** (1978) 587.
17. T. HASEGAWA, R. HASEGAWA and S. KRASHIMA, *Trans. Jpn. Inst. Metals* **11** (1970) 101.
18. A. H. CLAUER, M. S. SELTZER and B. A. WILCOX, *J. Mater. Sci.* **6** (1971) 1379.
19. W. BLUM and B. ILSCHNER, *Phys. Status Solidi* **20** (1967) 33.
20. J. P. POIRIER, *Phil. Mag.* **26** (1972) 701.
21. V. PONTIKIS and J. P. POIRIER, *Scripta Metall.* **8** (1974) 1427.
22. J. C. GIBELING and W. D. NIX, *Acta Metall.* **29** (1981) 1769.
23. S. F. EXCELL and D. H. WARRINGTON, *Phil. Mag.* **26** (1972) 1121.

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