Recovery Processes during Creep of Fe-0.75%Mn

By Yoichi Ishida*

A Fe-0.75%Mn alloy was creep tested at 500°C to examine the following three points: (1) whether or not the steady state creep of the alloy was in a thermodynamical steady state, (2) which configuration of the dislocation network was contributing to the creep strength, and (3) which change in the dislocation network was the cause of the creep recovery. Specimens were furnace-cooled under stress and then observed by transmission electron microscopy. The creep stress was reduced during the test and the recovery of the creep rate was found to consist of two stages. The decrease in dislocation density was not connected with the rapid and extensive first stage recovery, but with the second stage recovery. The subgrain size changed during steady state creep, indicating that the subgrain boundary was not controlling the creep strength. Though not by a single stage, the recovery was completed; i.e. the history effect disappeared in the true steady state. The structure factor, therefore, did not exist as an independent variable during the true steady state creep of the present simple and stable alloy. This suggests that the thermodynamical steady state was realized at least for the factors controlling the creep strength.

I. Introduction

When a small portion of applied stress is subtracted during the creep test, a large reduction in creep rate occurs after an instantaneous contraction of the specimen. The creep rate, however, recovers gradually to a level which can be expected for the new stress as shown in Fig. 1. Opposite is the case when stress is increased.

Fig. 1 Changes in creep rate when a small portion of applied stress is subtracted. The creep rate may recover only partly (1) or fully (2).

The time needed to regain the new steady state creep rate is short when stress is increased, while a long time is required to recover when stress is reduced. For some metals such as nickel the recovery is reported to be incomplete and the steady state creep was realized when the creep rate was still below the proper steady state creep rate expected for the new reduced stress(3), as indicated by curve 1 in Fig. 1. Nickel appears to be the metal of such nature. The prestrain effect, for example, was very persistent during creep(3). If such a deformation history effect lingers on through the steady state creep, another variable (structure factor) must exist in addition to usual variables such as stress, temperature and chemical composition during the steady state creep, which implies that even the strength contributing factors (not to mention of non-strength contributing factors) are not at a thermodynamical steady state during the steady state creep. A simple thermodynamical explanation of the steady state creep then has to be abandoned. Whether such a structure factor does exist as a variable in steady state creep, or there is still another slower recovery process overlooked in experiment (like curve 2 of Fig. 1) should be reexamined to understand the nature of the steady state in creep. Dislocation density is known to be constant during the steady state creep. A square law in stress is satisfied between the dislocation density and the creep stress(4) as in the case of cold worked structure. The dislocation density, however, did not decrease immediately on reduction of applied stress(5). The stress-dislocation density relationship, therefore, appears to be of an indirect nature and a closer examination of the dislocation arrangement during each stage of the recovery process is needed. Subgrain size has often been thought as an important factor to control the creep strength. There are, however, no theoretically firm grounds to postulate that the subgrain boundary acts as an effective barrier against the dislocation motion(6). Even for nickel with a large prestrain effect, the effect of subgrain size was of secondary nature(3). For the effect of subgrain size, therefore, the existence of the effect itself has to be examined first. Fe-0.75%Mn was chosen in the present test because the alloy gives a well-defined steady state creep at 500°C.

II. Material and Experimental Procedures

The specimen composition was Fe-0.75%Mn, 0.003% N, 0.0017% C(7). Sheet specimens 5 cm long, 1 cm wide in gauge length and 0.5 mm thick were prepared.

* Institute of Industrial Science, University of Tokyo, Tokyo, Japan.

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from a cold rolled sheet. For the final heat treatment specimens were annealed in vacuum at 850°C for 30 min and then furnace-cooled to room temperature. The resulting grain size was 0.054 mm. Constant load creep tests were performed at 500 ± 1°C under stresses of 4, 6 and 8 kg/mm² and strains of 6~17% in an atmosphere of 90% N₂ and 10% H₂ to avoid excessive oxidation. The specimens were cooled in air under loading, and the temperature of the specimens was dropped from 500°C to below 150°C in 15 min. The stress was reduced from 8 kg/mm² to 6 and 4 kg/mm², respectively; the stress reduction was repeatedly made stepwise by the decrement of 0.18 kg/mm² each time after the steady state creep was established. The above stress reduction was performed at the time when the additional creep strain of 0.01% was reached or the stressing time exceeded 30 min. The additional strain of 0.01% was experimentally found to be enough to regain the steady state creep, while the longest recovery time was estimated from the Bailey-Orown equation to be 30 min for 0.18 kg/mm² stress reduction under a stress of 4 kg/mm². After the creep test, pieces approximately 1 cm square were cut out from the specimen, lapped to about 0.12 mm in thickness, thinned electrolytically in a bath of 5% perchloric acid and 95% glacial acetic acid by volume and then observed under an electron microscope. Dislocation densities were determined by the "exit method", i.e., by counting their intersection with the surface (and dividing by 2). This method does not require a knowledge of the foil thickness. Densities of repulsive junctions, attractive nodes and super jogs were counted from the same photographs with the criterion described elsewhere. Subgrain size was measured from 50~100 micrographs obtained from the same foils, half of them along the grain boundaries and the rest well within the grains. The line intercepts method was used to measure the subgrain gize. For the subgrain size along the grain boundary, two lines were drawn along both sides of grain boundaries with 2 μ distance. Areas near triple points were excluded from statistics since the deformation was extremely large in this area.

III. Results

Fig. 2 shows the creep rate-strain curves of the Fe-Mn alloy under stresses of 4, 6 and 8 kg/mm² at 500°C. The steady state creep range of the alloy was estimated from this figure. The increase in creep rate around the point C is probably not a tertiary stage because the test was carried out under a constant load. The dislocation density and the subgrain size were measured for the points ABCD and G. Fig. 3 is the result where the creep stress of 8 kg/mm² was reduced to 6 kg/mm² stepwise by the decrement of 0.18 kg/mm². The resulting steady state creep rate was below the one when the specimen was creep tested at 6 kg/mm² from the beginning. The recovery curve of Fig. 3 on reduction of stress is exaggerated for clarity. The recovery in fact occurred more rapidly. A still larger discrepancy was observed when the stress was reduced from 8 kg/mm² to 4 kg/mm². As shown in Fig. 4, the resulting creep rate was 0.002%/hr, about one-fifth of the steady state creep rate that should have occurred when the same stress of 4 kg/mm² was applied from the beginning. Similar results were found by Mitra for nickel and aluminium and led him to a conclusion that the structure factor remains as a variable in the steady state creep as already described in the introduction. However, in the present tests, the continuation of creep deformation after the stress reduction process revealed a slow and small recovery of the creep rate as shown in the

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Fig. 2 Creep rate vs. strain of Fe-0.75% Mn at 500°C. ABCD and G represent points where the dislocation density and subgrain size were measured.

Fig. 3 Creep rates as stress is reduced successively from 8 to 6 kg/mm². Dotted horizontal line is the steady state creep rate when 6 kg/mm² was applied from the beginning.
Fig. 4. After 20 days of the additional creep test, the resulting creep rate coincided with the steady state creep rate of 0.01%/hr within the experimental accuracy. It was not the tertiary stage creep since the creep rate became constant at the value instead of it being in-
creased. The time to attain the final steady state creep was about 400 hours when the stress was reduced from 8 kg/mm$^2$ to 4 kg/mm$^2$, while it took about 40 hours when the stress was reduced to 6 kg/mm$^2$. The steady state creep rates, however, varied by about 10 times so that the additional strains to regain the real steady state were both 1~2% as shown in Fig. 5. The abscissa refers to the strain after the stress was reduced. The early part of the recovery curve of Fig. 4 is plotted in Fig. 5 because of the low creep rate. The second stage recovery process appears more evidently in the latter figure. E and F in Fig. 5 indicate points where the dislocation density and the subgrain size were measured. The difference in the dislocation arrangements between the two points may reveal the nature of the second stage recovery. The strain at F is obviously higher than that at E. The effect of strain on these parameters during the steady state creep needs first to be examined in order to single out the recovery effect. Dislocation densities and subgrain sizes for points A, B, and C in Fig. 2 were measured and are shown in Figs. 6 and 7. Dislocation density, junction density and super-

![Fig. 4 Extended creep after the stress was reduced successively from 8 to 4 kg/mm². About 400 hours later the creep rate recovered to the true steady state creep rate $\dot{\varepsilon}_s$.](image)

![Fig. 5 Secondary recovery in creep rate with additional strains. E and F represent points where dislocation density and subgrain size were measured. Additional strains of 1~2% were needed for the second stage recovery.](image)

![Fig. 6 Dislocation density, junction densities and super jog density vs. creep strain tested at 500°C and 6 kg/mm². Points AB and C are shown in Fig. 2.](image)

![Fig. 7 Subgrain size vs. strain in steady state creep. Fe-0.75%Mn, 500°C, 6 kg/mm². Points AB and C are shown in Fig. 2.](image)

jog density did not change appreciably during the steady state creep. However, the subgrain size changed appreciably. The average subgrain size gradually reduced with strain. The subgrain size along the grain boundary was decreasing faster than ones inside the grain. At point A, the subgrain boundary did not develop inside some of the grains, but at point C, the subgrain boundary developed well inside the grain. The subgrain boundary developed so much along the grain boundary, and it was shown that the grain boundary made so prominent cusps at the intersection with the subgrain boundary, it was sometimes difficult to distinguish the grain boundary and the subgrain boundary. The subgrain size can reach a steady size only after more than 20% strain. The comparison of subgrain size for the points E and F should, therefore, take the strain effect into account. The fact that the creep rate was constant during the
change in the subgrain size, seems to imply that subgrain size does not affect the creep rate at least during its early stage. On the other hand, it appears that properties such as dislocation density and dislocation junction density contribute to the creep strength since they were constant during the steady state creep. Fig. 8 shows the change in dislocation density and subgrain size during the stress reduction test. For reference, point D in Fig. 2 corresponding to the state before the stress reduction and point G crept-tested at 4 kg/mm² from the beginning are included in the Fig. 8. The change between points E and F involved in the change in dislocation density but not in subgrain size. Since some part of the second stage recovery must have occurred during the stress reduction process, the change in dislocation density between points D and E appears to have resulted from that portion of the second stage recovery. The primary quick recovery then is the one not involving the dislocation density change, while the second stage recovery is strongly related to the dislocation density change.

**IV. Discussion**

In general, the creep equation is written in the following form, if the rate controlling process is single:\(^{(10)}\):

\[ \dot{\varepsilon} = f(\sigma, T, St)e^{-U(\sigma, T, St)/kT} \]  

(1)

where St is the structure factor which is affected by the deformation history of the specimen. According to the present creep curve analysis the structure factor seems to drop out from the steady state creep equation of simple and stable metals:

\[ \dot{\varepsilon} = f(\sigma, T)e^{-U(\sigma, T)/kT} \]  

(2)

where \( \dot{\varepsilon} \) is the steady state creep rate. Since the second stage recovery process brought about the dislocation density change, the change seems to have occurred mainly on \( f \), while the first stage recovery process showing a far more extensive and rapid change in creep rate without changing the dislocation density, appears to represent changes in the arrangement in the three-dimensional dislocation network in the matrix and hence to involve in a change in \( U \).

The first stage recovery rate \( r_I \) may be estimated from the Bailey-Orowan equation:

\[ \dot{\varepsilon} = r_I / h \]  

(3)

where \( h \) is the work hardening coefficient and is 4000 kg/mm²/unit strain for the present specimen. The steady state creep rate \( \dot{\varepsilon} \), of the alloy at 500°C and the applied stress of 6 kg/mm² was 0.065%/hr. \( r_I \) is then 2.6 kg/mm²/hr. The second stage recovery rate \( r_2 \) may be estimated from Fig. 4. \( r_2 \approx \dot{\varepsilon}/\dot{t} = 0.06 \text{ kg/mm}^2/\text{hr} \), so that \( r_I / r_2 \approx 40 \). Similarly, for the test at 4 kg/mm², \( r_t \approx 0.4 \text{ kg/mm}^2/\text{hr} \), \( r_I \approx 0.01 \text{ kg/mm}^2/\text{hr} \), so that \( r_I / r_2 \approx 40 \). Since some part of the second stage recovery had already occurred during the stress reduction process, the real \( r_I / r_2 \) ratio is higher than 40: i.e. the second stage recovery rate is more than 40 times slower than that of the first stage recovery. The ratio has the accuracy only in the order of magnitude since a sort of incubation time was detectable in the secondary creep rate of Fig. 4.

It appears natural for the second stage recovery to take 1~2% additional strain when the recovery involves in the dislocation density change. For the dislocation density \( \rho \) of 10⁹ cm⁻² and the grain size \( D \) of 0.05 mm, the order of strain for the process fits to the experimental result if a half of the dislocations move one-fifth of the grain diameter:

\[ \varepsilon \approx \rho / 2 \cdot b \cdot D / 5 \approx 10^9 / 2 \cdot 2 \cdot 10^{-8} \cdot 5 \cdot 10^{-3} / 5 \approx 1\% \]

The magnitude of the second stage recovery in terms of creep rate is very small compared with that of the first stage recovery. The coefficient of decrease in creep rate on the reduction of stress for pure iron at 5.2 kg/mm², 500°C\(^{(7)}\) was 90 mm²/kg (\( \approx \lim_{\sigma \to \sigma_0} \frac{d \log \dot{\varepsilon}}{d \sigma} \)), while the second stage recovery of the present test at 500°C and 6 kg/mm² was 0.1 mm²/kg as estimated from Fig. 5. (The steady state creep rate of pure iron at 500°C and 6 kg/mm² was about 60 times higher than that of Fe-0.75%Mn at the same temperature and stress. But the result on pure iron may still be used for comparison, since the stress dependence was the same in Fe-0.75%Mn\(^{(7)}\) alloy.) The magnitude of the second stage recovery, then, is about one-thousandth of the first stage recovery process in logarithmic scale. The fact that the second stage recovery is so small in magnitude compared with that of the first stage recovery appears to indicate that the strength of the creeping structure depends mainly on the mode of dislocation arrangement such as attractive nodes, repulsive junctions and super jogs but not on properties directly depending on the dislocation density such as the long-
range stress field of dislocations.

V. Conclusion

Creep analysis of Fe-0.75%Mn at 500°C has indicated its steady state creep rate is a function of applied stress and temperature only. It appears that the deformation history effect of the specimen, i.e. the structure factor, does not exist as a variable in the true steady state creep, and instead, a second stage recovery process takes place after the normal first stage recovery. The magnitude of the second stage recovery was far smaller and its rate was much slower than that of the first stage recovery. The second stage recovery involved in the dislocation density change, while the first stage recovery did not. The subgrain size did not affect the steady state creep rate.

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