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Journal of Nuclear Materials 307–311 (2002) 946–950

journal of nuclear materials

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In situ TEM observation of dislocation movement through the ultrafine obstacles in an Fe alloy

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Abstract

Dislocation movement through ultrafine obstacles in a thermally aged Fe–Cu alloy has been studied by in situ transmission electron microscope observation. A very effective technique for quantitative estimates of radiation embrittlement is proposed. The obstacle strength has been estimated from the critical bow-out angle, ϕ, of dislocations. The increase in shear stress, which is estimated from the averaged strength of ultrafine obstacles and the averaged distance between the two neighboring obstacles on a dislocation in the in situ observation, is in good agreement with the hardening obtained from macroscopic mechanical tests. This technique is very useful to predict the mechanical properties of irradiated fusion and fission reactor materials from the microstructure obtained from experimental observations and/or the computer simulations.

1. Introduction

It is widely accepted that the radiation-induced obstacles to dislocation movement are the primary reason for the radiation hardening of fusion and fission reactor materials by neutron irradiation. Such obstacles are precipitates, dislocation lines and loops, and defect clusters, etc. Understanding dislocation movement through such radiation-induced obstacles is important for the quantitative estimate of radiation hardening. Extensive studies on the relation between defect structures (which are obstacle size and the density of obstacles) and mechanical properties were performed [1]. The value of α has been determined by correlating microstructural parameters with mechanical properties. This value signifies the average obstacle strength against dislocation movement. However, ultrafine obstacles, which are not observed by transmission electron microscope (TEM), were not considered for these estimates. It is therefore important to determine the value of α for such ultrafine obstacles in order to estimate $\Delta\tau$ from the results of computer simulation on defect structures or from the observations by such useful techniques as SANS, positron annihilation, atom probe, etc.

In this study, we performed in situ TEM observations of dislocation movement through obstacles in thermally aged Fe–Cu alloys under stress in order to estimate α from the bow-out angle ϕ of dislocations. The increase of the critical shear stress due to the obstacles has been estimated using relationship by Foreman and Makin [2,3]. Finally, a comparison is made between our hardening estimates based on the critical bowing angles and those obtained from mechanical property measurements.

2. Experiment

The Fe–1.0wt%Cu alloy was made by arc melting and cold rolling. The samples were machined to coupons of 1.0 mm × 4.0 mm × 0.2 mm for in situ tensile tests,

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PII: S0022-3115(02)01163-7

plate tensile specimen of 5.0 mm in length and 1.2 mm × 0.3 mm in cross section in the gauge section (type SSJ [4]), and discs of 5.0 mm diameter for coincidence Doppler broadening (CDB) measurement of positron annihilation [5]. They were annealed at 825 °C for 4 h and subsequently quenched into ice water. Some of the samples were thermally aged at 525 °C for 1/3 h (20 min) or for 10 h. The coupons (for in situ tensile tests) were electrolytically polished to 30–40 μm in thickness. The dimensions of the microtensile specimens were 4.0 mm × 1.2 mm. The thin foil specimens were prepared by a standard twinjet electrolytic polishing method.

In situ tensile deformation experiments were performed in a JOEL 4000FX TEM operated at 400 kV with a single tilt tensile holder at room temperature. The tensile load capacity is about 0.5 kg. The motion of dislocations has been recorded by a digital video camera with a time resolution of 1/30 s.

Tensile tests were conducted at a strain rate of 6.67×10^{-4} s^{-1} at room temperature in air. The CDB of positron annihilation was measured (the details of this experimental method is described in Ref. [5]).

3. Results and discussion

Table 1 shows the yield stress, for the Fe–1.0wt%Cu specimens as quenched, aged at 525 °C for 20 min and for 10 h, respectively. Fig. 1 shows the ratio curve of the CDB spectra for the samples with the different heat treatments where the spectra are normalized to pure iron. For the as-quenched sample, the ratio curve of the CDB spectra is constant at unity. This means that positrons annihilate only with the iron electrons. It has been shown by a theoretical computation that the copper precipitates, if any, are smaller than about 0.6 nm in such cases [5]. For the 10 h-aged sample, the ratio curve is almost the same as that of pure copper indicating all the positrons annihilate with copper electrons. Othen et al. [6] studied the growth process of Cu precipitates in Fe–1.3wt%Cu alloy during thermal aging at 550 °C. They showed that the hardness peaked at about 2 h of aging, when the ultrafine Cu precipitates were smaller than 4 nm. Nagai et al. [5] showed, the hardness peaked at about 10 h of aging in Fe–1.0wt%Cu alloy during thermal aging at 550 °C. Therefore under the present experimental conditions, the Cu precipitate size is most likely much less than 4 nm.

Table 1
Value of yield stress obtained by tensile tests

	Yield stress σ (MPa)	Increase of yields stress $\Delta\sigma$ (MPa)
As quenched	74	–
20 min aged	144	70
10 h aged	278	204

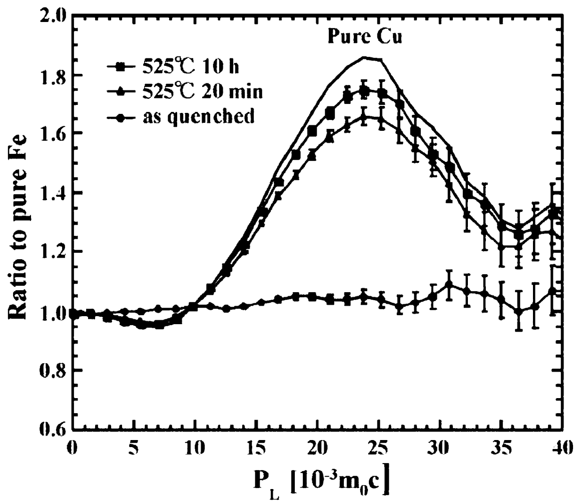

Fig. 1. Ratio curve of the CDB spectra of pure Cu, Fe–1.0wt%Cu alloys as quenched, after 20 min and 10 h aging with respect to pure Fe.

Fig. 2(a)–(c) shows snap shots of the in situ TEM observation of Fe–1.0wt%Cu samples as quenched, after 20 min aging and 10 h aging at 525 °C. In as-quenched condition (Fig. 2(a)), dislocation movement was smooth, i.e. no pinning was observed on moving dislocations while they gradually passed across the view field. After 20 min aging (Fig. 2(b)), pinning-depinning behavior of moving dislocations was clearly observed in the entire view field. After 10 h aging (Fig. 2(c)), dislocation movement was more complex than after 20 min aging. It suggests that the obstacles are Cu precipitates detected by positron annihilation only in the aged samples, and that the Cu precipitates that have grown larger after prolonged aging pinned dislocations more strongly.

Before measuring bow-out angles of dislocations, the slip systems had to be determined. Slip systems being activated were assumed to be $\langle 1\,1\,1 \rangle$ $\{1\,0\,\bar{1}\}$ or $\{1\,1\,\bar{2}\}$ systems under the temperature condition in this experiment. Fig. 3(a)(1)–(4) shows snapshots of the primary slip system activity. The moving dislocations were in the shape of an arc as is schematically illustrated in Fig. 4(a)(1)–(3). It was easily noticed that the glide velocity depended on the direction of the dislocation line. The glide velocity along $\langle 1\,1\,1 \rangle$ directions was significantly higher than that in its parallel direction. This observation suggests that the well-known fact that the glide velocity of the edge component is much faster than that of the screw component is still effective under the present experimental conditions. Assenting that the choice of the slip system follows Schmid's law and considering the slip lines, the slip system was determined as $[1\,1\,1]$ $(0\,\bar{1}\,1)$.

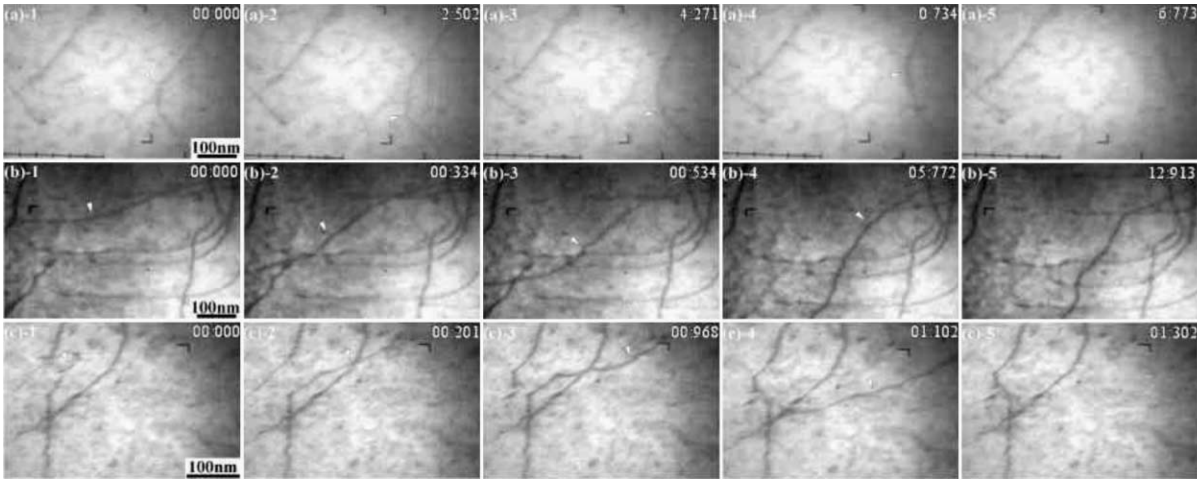

Fig. 2. In situ TEM observation in Fe–1.0wt%Cu alloy as quenched (a), after 20 min (b) and 10 h aging (c).

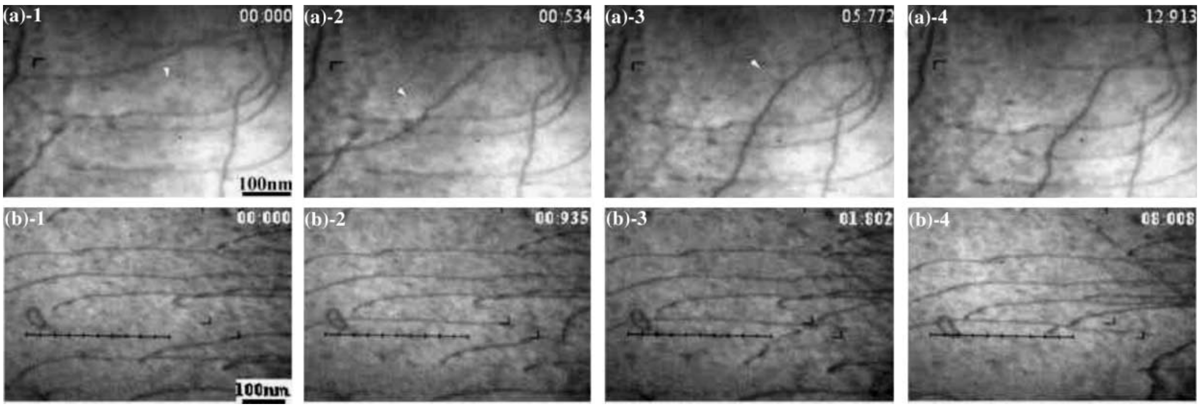

Fig. 3. Dislocation glide in the primary slip system (a), and the secondary slip system (b) in Fe–1.0wt%Cu alloy after 20 min aging.

Fig. 3(b)(1)–(4) shows the activity of a secondary slip system in the later stage of observation. Almost all dislocations are straight. The glide velocity dependence on the direction of the dislocation line was more significant than was observed for the primary slip system. The movement of non-screw dislocation segment is in the form of kink propagation along screw dislocation segments as is schematically illustrated in Fig. 4(b). From these features, the Burgers vector was determined to be pararel to the straight-lines, hence $[\bar{1}\,\bar{1}\,1]$. Apparently the fraction of screw component was much larger than that of the edge component. It is quite reasonable that the slower component occupies the larger fraction. According to Schmid's law and considering slip line directions, the slip system was determined as $[\bar{1}\,\bar{1}\,1]$ $(\bar{1}\,2\,1)$.

Fig. 5 shows the histogram of the obstacle strength parameter $\alpha = \cos(\phi_c/2)$, which has been estimated from the critical bow-out angle, ϕ_c, of screw dislocations through the obstacles measured just before the dislocations broke away from the obstacles for the 20 min aged samples. The relation between obstacle strength parameters and macroscopic mechanical property is given by the empirical relation by Foreman and Makin [2,3]:

$$\Delta\sigma_c = T\Delta\tau_c = T(\mu b/L)\cos(\phi_c/2)^{3/2}(1 - \phi'/5\pi), \qquad (1)$$

where $\Delta\tau_c$ is the hardening, $\Delta\sigma_c$ is the tensile stress increase and ϕ' is the complementary breaking angle. The value of the Taylor factor T is 3.06 [7]. The average of $\cos(\phi_c/2)$ values for screw dislocations was 0.22, and the average distance between the two neighboring obstacles on dislocations, L, was 111 nm by in situ TEM observation. The value of tensile stress increase, $\Delta\sigma_c$, calculated from the experimentally obtained values using Eq. (1) is 76 MPa. On other hand, the increase of the yield stress $\Delta\sigma$ after 20 min aging obtained by tensile test was

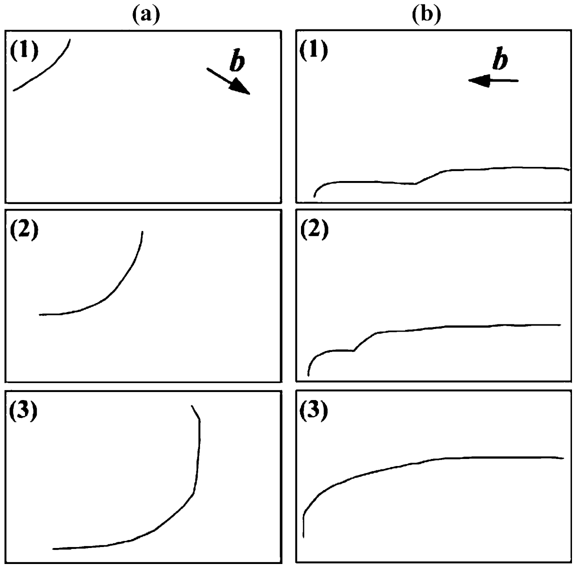

Fig. 4. Schematic illustration of the dislocation movement though obstacles in Fe–1.0wt%Cu alloy after 20 min aging shown in Fig. 2. (a) The primary system, (b) the secondary system.

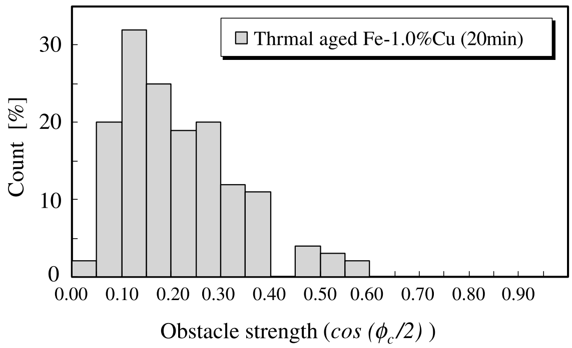

Fig. 5. The obstacle strength parameter, $\alpha = \cos(\phi_c/2)$ estimated from the critical bow-out angle, ϕ_c, of screw dislocation through obstacles by in situ TEM observation.

70 MPa, which was in good agreement with that estimated from the microstructural information.

Fig. 6 shows an in situ TEM observation for a 10 h aged specimen. In the specimen, the dislocation movement was not very active, so that a sufficient number of pinning points to estimate the value of $\cos(\phi_c/2)$ were not observed. Instead the local stress field, τ_l, given by the relation [2,3]

$$\tau_l = \mu b/(2\rho), \tag{2}$$

where ρ is the radius of curvature, was evaluated. The radius of the curvature was about 190 nm and the local stress field τ_l was calculated to be 76 MPa. The tensile stress σ_λ estimated using the Taylor factor is 233 MPa. Since the radius of curvature cannot become smaller than the value corresponding to the critical stress of the obstacle holding the dislocation segment, it is considered that this value gives the minimum of the precipitate strength. On the other hand, the tensile yield stress was 144 and 278 MPa after 20 min and 10 h aging, respectively. It is reasonable that the σ_l took a value between yield stress after 20 min aging and that after 10 h aging.

4. Conclusions

Direct observation of dislocation movement through obstacles was performed in thermally aged Fe–1wt%Cu alloy samples. The behavior of the dislocations clearly varied with the thermal aging time. The obstacle strength was estimated as $\cos(\phi_c/2)$ using the bow-out angle. Experimentally obtained macroscopic mechanical properties were in good agreement with the values estimated by dislocation bow-out model.

The understanding of the dislocation movement through radiation-induced obstacles is important for the quantitative estimate of the radiation hardening based on the microstructural evolution. The in situ TEM observation technique performed in this study has proven to be quite useful for quantitative estimation of the dislocation-obstacle interactions especially when the obstacles are too small to be observed by TEM.

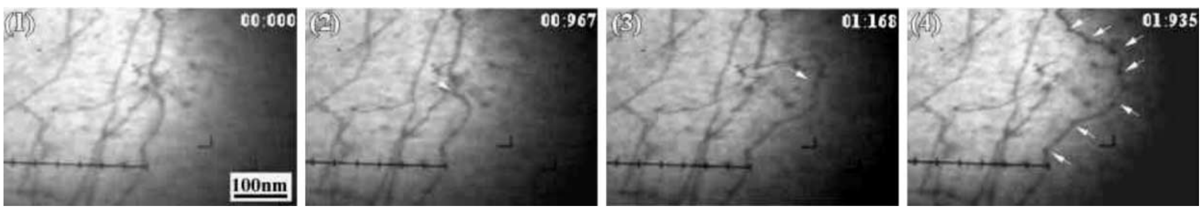

Fig. 6. In situ observation in Fe–1.0wt%Cu alloys after 10 h aging.

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