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Quantitative analysis of the dependence of hardening on copper precipitate diameter and density in Fe–Cu alloys

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Abstract

In order to investigate the dependence of hardening on copper precipitate diameter and density, in situ TEM observations during tensile tests of dislocation gliding through copper precipitates in thermally aged Fe–Cu alloys were performed. The obstacle strength has been estimated from the critical bow-out angle, ϕ_c, of dislocations. The obstacle distance on the dislocation line measured from in situ TEM observations were compared with number density and diameter measured by 3DAP (three dimensional atom probe) and TEM observation. A comparison is made between hardening estimation based on the critical bowing angles and those obtained from conventional tensile tests.
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1. Introduction

It is well known that the radiation-induced defect clusters in metals are responsible for significant changes in mechanical properties. Examples of extended crystal defect clusters include precipitates, voids, dislocation lines and loops. Understanding the quantitative effect of such radiation-induced defect clusters on dislocation motion is important for construction of predictive models to estimate the lifetime of power plant components. Based on Orowan's simple model, the most commonly used expression for the change in shear stress, $\Delta\tau_c$, by a regular array of obstacles is given in the following equation: $\Delta\tau_c = \alpha\mu b(Nd)^{1/2}$, where μ is the shear modulus, b the magnitude of the dislocation Burgers vector of the dislocations, N the number density of defect cluster, d the defect cluster diameter and the square-root factor is the reciprocal of the average distance between obstacles. The α factor is known as the obstacle strength and is determined as $\alpha = \cos(\phi_c/2)$. The critical bow-out angle, ϕ_c, was taken as the angle between two tangent lines drawn from the cusp in the line tension approximation. However, relatively few works have been performed to determine the α, and, in practice, microstructure and mechanical property are merely compared to infer the α for different types of defect clusters [1].

In recent molecular dynamics (MD) simulation studies [2–4], the dependence of hardening on obstacles diameter and the distance between obstacles was investigated. However, it is difficult to compare MD results with experimental observations because obstacles responsible for hardening are often too small to observe by transmission electron microscopy (TEM). In our previous study [5], the in situ

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TEM observation technique has proven to be quite useful for quantitative estimation of the dislocation–obstacle interactions.

In this study we investigate the dependence of hardening on copper precipitate diameter and number density by performing in situ TEM observations during tensile tests in thermally aged Fe–Cu alloys. The obstacle strength α was determined at the point of dislocation break away from the obstacle. Only a single type of precipitate is assumed as the obstacle, hence the angle, ϕ_c, depends on the diameter. The number density of obstacles was determined from the dislocation segment length L between two adjacent pinning points. The spatial randomness of the pinning point separation has been taken into account by using a relationship of Foreman and Makin [6,7]. Comparison is made between number densities estimated from in situ observations and those obtained by three dimensional atom probe (3DAP) and conventional TEM observations.

2. Experimental procedure

Fe–1.0 wt%Cu alloy was prepared by arc melting followed by cold rolling. The samples for in situ tensile tests were machined to coupons of $1.0\,\text{mm} \times 4.0\,\text{mm} \times 0.2\,\text{mm}$ and samples for 3DAP were cut into small square rods of $0.25 \times 0.25 \times 7$ mm which were subsequently electro-polished to sharp needles. Type SSJ plate tensile specimens ($16 \times 4 \times 0.25$ mm with a $1.2\,\text{mm} \times 5\,\text{mm}$ gauge section) were used for macroscopic property tests. All samples 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 20 min, 1 h, 10 h and 100 h. The coupons (for in situ tensile tests) were electro-polished to 30–40 μm thicknesses. The thin foil specimens were prepared by electropolishing with a twinjet Struers Tenupol-3 using a polishing solution of 5% perchloric acid in ethanol cooled to −20 °C.

In situ tensile deformation experiments were performed at room temperature in a JEOL 4000FX TEM operating at 400 kV with a single tilt straining holder. The motion of dislocations was recorded by a CCD camera with a time resolution of 1/30 s. The slip planes had to be determined and the bow-out angles of dislocation should be measured in slip plane. Slip systems being activated were determined to be $\langle 111\rangle$ $\{10\bar{1}\}$ or $\{11\bar{2}\}$. The method for determination of the actual slip system was described in detail in Ref. [5]. The α was determined by a frame-by-frame analysis of the interaction between a moving dislocation and the copper precipitates, in which the critical angle was assessed at the configuration just before the dislocation broke away. Tensile tests were carried out at a strain rate of $6.67 \times 10^{-4}\,\text{s}^{-1}$ at room temperature in air. The 3DAP measurements were performed with the energy compensated position-sensitive atom probe facilities which consist of a reflectron energy compensator, a position-sensitive detector and a high-resolution flight time detector [8]. Measurements were carried out at a sample temperature of 60 K with the pulse fraction of 0.15 under ultrahigh vacuum conditions.

3. Results and discussion

Fig. 1 shows the typical morphology of dislocations during an in situ TEM observation of Fe–1.0 wt%Cu after aging 20 min, 1 h, 10 h and 100 h at 525 °C. Dislocations are along $\langle 111\rangle$ direction and anchored at pinning points. After 20 min and 1 h aging, copper precipitates were not observed by TEM in Fig. 1(a) and (b). On the other hand, copper precipitates were clearly observed at pinning points and in the matrix after 10 h and 100 h aging. Table 1 shows the average diameters and number densities of copper precipitates measured by 3DAP and TEM observations. The diameter was about 1 nm after 20 min and 1 h aging. In our previous study of coincidence Doppler broadening measurements [5], it is confirmed that copper precipitates are larger than about 0.6 nm after 20 min aging at 525 °C. The copper precipitates diameter increased with the aging time. The homogeneous distributions of copper precipitates were observed by TEM. It has been reported that precipitates of b.c.c. clusters are formed at first, then their transformation to 9R structure occurs when they grow beyond a critical diameter of 4 nm [9]. Therefore under the present experimental conditions, copper precipitates are deduced to be a coherent b.c.c. structure after 20 min and 1 h aging. On the other hand, it is considered that the precipitates were in the stage of transformation from b.c.c. structure to 9R structure after 10 h aging. However, it is difficult to observe ultrafine precipitates of b.c.c. structure by TEM. Therefore most of the copper precipitates observed by TEM were expected to have 9R structure and small invisible b.c.c. copper precipitates might also exist after 10 h aging. There is a peak in number density of copper precipitates at 1 h aging.

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