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High-resolution electron microscopy observation and dislocation reaction mechanism of fivefold twinning in a Cu-rich precipitate in a cold rolled ferritic steel containing copper

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ABSTRACT

Ferritic steels containing copper have been studied as model systems for clusters/precipitate formation in reactor pressure vessel steels. The samples were aged at 400 °C for 4000 h and subsequently cold rolled to 30% reduction at room temperature. The microstructural characteristics of the samples were analyzed using high-resolution transmission electron microscopy. Direct evidence was found that the fivefold twinning occurs via simultaneous emission of two Shockley partial dislocations from two particular α-Fe/Cu interfaces, and then the pileup tips of the twofold twin.

1. Introduction

Irradiation-induced hardening that leads to embrittlement of reactor pressure vessel (RPV) steels is long accepted to be in most cases caused by the precipitation of copper, which is highly insoluble in iron [1,2]. Many studies have been carried out to investigate the precipitation of Cu-rich clusters during neutron irradiation [3,4], proton irradiation [5], thermal aging [6–8] and cold deformation before thermal aging [9–11]. It has been observed that both point defects and dislocations can accelerate the copper precipitation kinetics [12]. Pre-deformation before thermal aging also affects the copper precipitation in a similar way and causes precipitation at dislocations [13]. Actual RPV steels like A508-III may contain many kinds of lattice defects, such as point defect clusters and line dislocations, and what's more, the interactions of these defects with copper-rich precipitates will influence the evolution of Cu-rich precipitates [14,15].

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In this study, we report experimental evidence that fivefold twinning could form in cold rolled ferritic steels containing copper. A formation mechanism of fivefold twinning is proposed and discussed based on the observed morphologies of the fivefold twinning formed during cold rolling.

2. Materials and Methods

The samples for this study were taken from a model steel having higher Cu content than that in the real RPV steel with a composition of 0.6% Cu, 0.85% Ni, 1.58% Mn, 0.39% Si, 0.016% P, 0.22% C, 0.006% S, 0.54% Mo and balance Fe (in wt.%). The experimental materials were prepared by vacuum inducting melting. The resulting ingot (~40 kg) was hot forged and air-cooled. The slabs were hot-rolled to sheets of 4 mm in thickness, and then cut into samples with the dimensions of $30 \times 30 \times 4$ mm^3. After an initial heat treatment of 0.5 h at 880 °C and water quenching, the samples were tempered at 660 °C for 10 h followed by air-cooling to room temperature. The samples were then isothermally aged at 400 °C for various duration conditions up to 4000 h. Subsequently, the samples were cold rolled to 30% reduction at room temperature. For the accurate analyses of chemical composition and microstructure of the precipitates without including noise from the matrix, carbon replicas were used for the electron microscopy because of their greater stability under the exposure of an electron beam. Procedures of extracting Cu-rich precipitates were described in reference [16]. HRTEM observation was performed on a JEM-2010F with EDS system operating at 200 kV.

3. Results and Discussion

Cu-rich precipitates in aged RPV model steel (4000 h at 400 °C) were nearly spherical in shape with a diameter about 5–30 nm before deformation. The precipitates have been determined to be Cu–Fe solid solutions with 9R, 2H or fcc [16–18]. No fivefold twinning was detected after careful examination of the whole thinning area of the TEM foil or specimen for extraction precipitates. However, fivefold twinning appeared as shown in Fig. 1(a) after the aged sample was cold rolled to 30% reduction at room temperature.

The EDS spectrum of the precipitate is shown in Fig. 1(b). The analysis result indicates that the precipitate has a chemical composition of ~63.3 Cu–36.7 Fe (in at.%). No other composition in the spectrum was considered besides copper and iron, since the peaks of C and Mo yield mainly from the carbon film and the molybdenum grid.

The corresponding fast Fourier transformation (FFT) of the region and the inverse FFT (IFFT) image of fivefold twinning are shown in the inset in Fig. 1(a) and in Fig. 1(c), respectively. The twin boundaries (TBs) are indicated by white lines and each twin boundary in fivefold twinning is marked as TB1–TB5, respectively, in Fig. 1(c). A schematic illustration of the FFT pattern is shown in Fig. 1(d).

The first finding of a copper fivefold twinning was reported in 1882 by von Lasaulx [19]. Fivefold twinning is a wide-spread habit of nanoparticles [20], found not only in synthetic materials as reported for the first time in 1957 [21], but also in nanocrystalline and ultrafine-grained fcc alloys and metals synthesized by severe plastic deformation techniques such as ball milling and high pressure torsion [22–25]. Cao and Wei [26] observed a fivefold twinning in nanocrystalline Cu under uniaxial tensile conditions. Bringa et al. and Huang et al. [27,28] found that fivefold twinning can form during annealing of nanocrystalline Cu with no external stress. Although extensive studies on fivefold twinning have been carried out as aforementioned, so far there have been no reports on fivefold twinning in the Cu-rich precipitates of cold rolled ferritic steels containing copper. It should be noted that the angles between TBs shown in Fig. 1(c) are more proportional than those reported previously. Generally, the ideal twin angle between {111} planes is 70.5° in fcc materials and a fivefold twinning leaves a gap of ~7.3° when closing the 360° circle. As shown in Fig. 1(c), the deviation between the maximum and minimum angles between the twin boundaries is only 2°, whereas, the deviation in fivefold twinning formed via a proposed sequential twinning mechanism is as large as 12.5° [25] and 12° [27], respectively. This comparison indicates that the 7.3° gap is shared by several twins in the fivefold twinning shown in Fig. 1(c), implying that some of the TBs may form simultaneously instead of sequentially during cold rolling.

In nanocrystalline Cu, deformation twinning is generally believed to occur via partial dislocations emitted from grain boundaries and requires higher resolved shear stresses that can only be reached under high strain rate and/or low temperature conditions [25]. In contrast, in a Cu-rich precipitate of a cold rolled ferritic steel containing copper, the twin may be induced via partial dislocation emission from matrix/precipitate interfaces instead of grain boundaries.

Kohler et al. [29] investigated the interaction between dislocations and spherical Cu precipitates in α-Fe during plastic deformation using classical molecular dynamics simulations. It was found that the dislocations are mainly located at the α-Fe/precipitates interface, as shown in Fig. 2. It can be seen that Burgers vector distributions (BVD) are inhomogeneous and the pinned dislocation lines are predominantly located in the interface regions in which are marked as I_1 and I_2, respectively. This can be attributed to a consequence of the tendency of the Cu-rich precipitate to undergo a structure modification into the fcc structure inside the precipitate while the boundary region can still be considered as a bcc structure during cold rolling, which results in a considerable change in stress distribution on the dislocation slip plane, as well as the dislocation core atomic structure. The compressive stress and the distortion of the atomic structure in front of the dislocation restrain the dislocation from slipping forward. For mismatching interface, the interfacial shear resistance is weak and the core of the interfacial segment can spread. In contrast, for crystals of matching interface, the interfacial shear resistance is relatively high and the core of the interfacial segment remains compact. The self-energy will be lower for the interfacial segment with the wider core [30]. Hence, partial dislocation nucleation is easier in the vacant regions than in the other parts.

To make the discussion easier, Thompson tetrahedron is unfolded onto a two-dimensional representation, as shown in Fig. 3(a). The (1-1-1) and (1-11) planes are shaded here because