MATERIALS CHARACTERIZATION 62 (2011) 438–441

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High-resolution electron microscopy characterization of 2H and 9R variant in the ferritic steels containing copper

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ARTICLE DATA

Article history:
Received 10 February 2011
Received in revised form
18 February 2011
Accepted 22 February 2011

Keywords:
Crystal Structure
High-resolution electron microscopy
Cu precipitate
9R structure
2H structure

ABSTRACT

Ferritic steels containing copper have been studied as model systems for cluster/precipitate formation in reactor pressure vessel steels. The samples were aged at 400 °C for 2000 h and subsequently analyzed using high-resolution electron microscopy. Direct evidence was found that, besides the 9R structure occurring, there exist also 2H variant and stacking faults in local regions of the same Cu precipitate. The 2H variant has a hexagonal unit cell with lattice parameters a = b = 0.254 nm and c = 0.417 nm and axial ratio c/a = 1.642.
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1. Introduction

Much work has been carried out to study the precipitation of Cu-rich clusters during thermal aging of ferritic steels containing copper [1–3]. It is well known now that neutron irradiation hardening and related embrittlement of reactor pressure vessel (RPV) steel are at least partially due to the precipitation of copper [4], and thus understanding the phase transformation and subsequent precipitate structure is a necessary step in understanding the embrittlement process. Several investigations on Fe—Cu model alloys using high-resolution transmission electron microscopy (HRTEM) have shown that the growing precipitates undergo a transformation from BCC structure to a twinned 9R. Upon further aging, Cu precipitates grow and transform to a 3R (FCT) structure, and finally reach the expected FCC structure [5,6]. Although extensive studies on the precipitation of Cu in aged model Fe—Cu alloys have been carried out as aforementioned, the detailed microscopic features of this series of structural transformations were not well understood.

This study aimed to carry out further structural details of copper precipitates in aged RPV model steel using the carbon extraction replica technique, with HRTEM and energy dispersive spectrometer (EDS). Besides precisely analyzing the chemical composition and microstructure without the noise from the matrix, carbon replicas were used for the electron micrographs because of their greater stability under the exposure of electron beam.

2. Materials and Methods

The samples for this study were taken from RPV model steel having higher Cu content 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 induction melting. The resulting ingot (~40 kg) was hot forge and air-cooling. The slabs were hot-rolled to sheets of 4 mm in thickness,

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doi:10.1016/j.matchar.2011.02.006

and then cut into samples with the dimension of $40 \times 30 \times 4\ mm^3$. After an initial heat treatment of 0.5 h at 880 °C inside the tubular furnace and quenched into water, the samples were tempered at 660 °C for 10 h followed by air cooling. The samples were then isothermally aged at 400 °C for different times up to 2000 h.

To prepare the replica specimen for extraction precipitates (extraction replicas [7]), the samples were first mechanically polished up to a mirror finish and then electrochemically etched using a non-solution of 10% acetylacetone-1% tetra-methylammonium chloride-methyl alcohol. After etching and carbon coating, the carbon film on the specimen surface was lightly scored into small squares, stripped by etching again with the solution mentioned above, and gently removed from the surface in deionized water. Finally, the sections of film were of the correct size for capture onto molybdenum TEM grids. HRTEM observation was performed on a JEM-2010F with EDS system operating at 200 kV.

3. Results and Discussion

Fig. 1(a) shows bright-field TEM micrographs of some precipitates in an extraction replica in a sample aged for 2000 h at 400 °C. It can be seen that there are a number of precipitates on an extraction replica. Precipitates by the white arrows are cementite particles. Fig. 1(b) shows the HRTEM micrograph of a precipitate as shown by black arrow in Fig. 1(a). It can be seen that the precipitate is nearly spherical in shape with a diameter about 20 nm.

The EDS spectrum taken from a precipitate as shown by black arrow in Fig. 1(a) is shown in Fig. 2. The analysis result indicates that the precipitate has a chemical composition of 65.8 Cu–34.2 Fe (in at.%). Here, there is no consideration of the other compositions' influence besides copper and iron, since the peaks of C and Mo elements yield from the carbon film and the molybdenum grid.

Othen et al. [5] had studied the copper precipitates in Fe—Cu and Fe—Cu—Ni alloys by HRTEM. These authors found that the orientation relationship between the BCC matrix and the 9R precipitates is $(011)_b \parallel (\bar{1}\bar{1}4)_{9R}$ and $[\bar{1}\bar{1}1]_b \parallel [\bar{1}10]_{9R}$. This relationship leads to the occurrence in HRTEM images of characteristic herring-bone fringes. Moreover, only about a quarter of the 9R precipitates show the typical "herring-bone" fringes when observed along [111] direction of the ferrite matrix. The Fast Fourier transform (FFT) pattern of the copper precipitate as shown in Fig. 1 is given in Fig. 3(a). Some of the spots on the FFT patterns were identified as 9R structure in according to the references [8]. Indeed, these patterns match well with the lattice parameters of a = 0.442 nm, b = 0.255 nm, c = 1.885 nm and $\beta = 86°$. It can be seen that one spot appears in the middle between (000) and $(00\bar{9})$ spots. This spot is the (001) position of 2H variant that due to the effect of double diffraction additional spots appear on forbidden positions between the central (000) and the $(00\bar{9})$ spots in [010] orientation. This result is coherent with the Inverse Fast Fourier transform (IFFT) pattern of the high resolution image observation.

Fig. 3(b) shows an IFFT pattern of the high resolution image in Fig. 3(a). It can be seen that there are a number of stacking faults on (001) planes in the pattern, examples of them are shown by the arrows labeled S. A high-magnification IFFT pattern from selected area 1 in Fig. 3(b) is shown in Fig. 3(c). From the IFFT pattern, one can see that there is a nine-layer (sequenced as ABCBCACAB) unit-cell structure. Therefore, the above stacking sequences in the copper precipitate can be classified as a 9R structure. The IFFT pattern shows that the (001) plane of the 9R structure copper phase is not perpendicular to the (100) plane but rather has a relative orientation of about 86.9°. This distortion can also be clearly observed in the corresponding FFT pattern shown in Fig. 3(a). A high-magnification IFFT pattern from selected area 2 in Fig. 3(b) is shown in Fig. 3(d). From the IFFT pattern, one can see that there is a two-layer (sequenced as AB) unit-cell structure. Therefore, the above stacking sequences in the copper precipitate can be classified as a 2H structure. The IFFT pattern shows that one atomic layer has much brighter contrast than the others in one unit cell, and the (001) plane of the 2H structure copper phase is perpendicular to the (010) plane. The contrast difference may be caused by the segregation of Cu atoms as reported by Abe et al. [9]. The spacings of the $(002)_{2H}$ and $(010)_{2H}$ planes for the 2H variant are

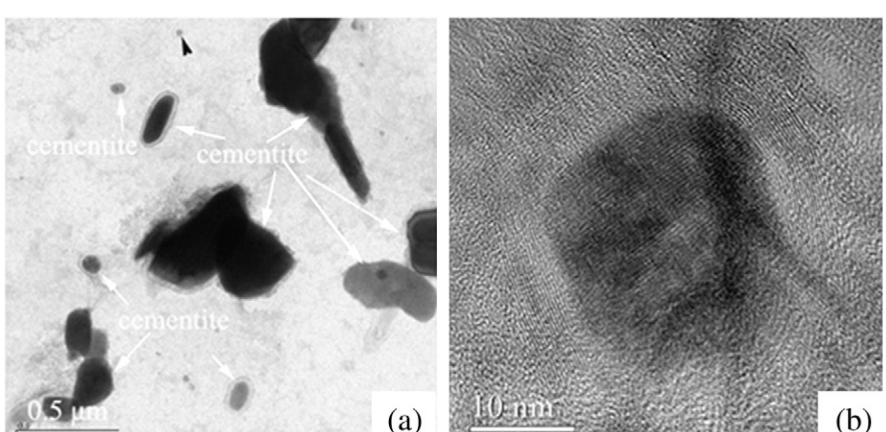

Fig. 1 – (a) Bright-field TEM micrographs of some precipitates in an extraction replica in a sample aged for 2000 h at 400 °C; (b) HRTEM micrograph of a precipitate as shown by white arrow in Fig. 1(a).

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