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Microstructure evolution to reach the single variant in an ordered Fe–55at.%Pd alloy

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ABSTRACT

Recently, we reported single variant formation in an Fe–55at.%Pd is certainly realized from a disordered fcc-phase to an ordered $L1_0$-phase by heat-treatment under magnetic field. In the present study, we have investigated microstructure evolution during the process of the single variant formation by an X-ray diffraction and an electron microscopy observation. As a result, followings are obtained: size of the ordered particles at the early stage of ordering is about 2 nm and the nucleation ratio of preferable variant, whose easy axis lies in the field direction, is higher than that of other variants. Each of the ordered preferable variant grows by consuming the order variants and finally come together to become a single variant. Based on the observation, a model is proposed for the single variant formation of the ordered $L1_0$-phase under magnetic field.

1. Introduction

Microstructure control is quite important in materials science and technology because microstructure is closely related to the physical properties of materials, such as strength, toughness, electric and magnetic ones. Microstructure can be formed by solid–solid phase transformations such as martensitic transformations [1–4], reconstructive transformations [5–8] and precipitations [9–12]. Recently, magnetic field is found to be effective for controlling the microstructure formed by above transformations. Rearrangement of martensite variants in ferromagnetic shape memory alloys [13–16] and alignment of α- and γ-phases in steels under magnetic field [17–20] are typical examples of such microstructure control.

Disorder–order transformation of near-equiatomic FePd, CoPt and FePt is another example through which we can control the microstructure using a magnetic field. The details are as follows. Three alloys transform from a disordered A1-type (cubic) structure to an ordered $L1_0$-type (tetragonal) structure and the tetragonal phase has three variants [21–23]. Compared with a disordered phase, the ordered phases have relatively high magnetocrystalline anisotropy, where the easy axis is the *c*-axis and the hard axis is the *a*-axis. Therefore, there arises an energy difference among variants under a magnetic field, and the variant with the lowest magnetic energy will be selected to form preferentially during the disorder–order transformation under magnetic field. In fact, we found in Co–50at.%Pt and Fe–55at.%Pd alloys that formation of a single variant is certainly realized by a two-step ordering heat-treatment under magnetic field. The first step corresponds to the nucleation process, and is made under a magnetic field at a temperature below the Curie temperature of the disordered phase, 740 K. The second step corresponds to the growth process, and is made at an elevated temperature below the order–disorder transformation temperature of 1025 K without magnetic field to accelerate the ordering [24,25]. However, we have no information on microstructure formed during the two-step ordering process.

In this study, therefore, we will show crystal structure change and the microstructure formed during the two-step ordering process in Fe–55at.%Pd alloy by using an X-ray diffraction (XRD) measurement and a transmission electron microscope (TEM) observation. Furthermore, we will discuss the reason why preferential formation of an ordered variant occurs under magnetic field.

2. Experimental procedure

The Fe–55at.%Pd alloy used in the present study was prepared from high purity elemental starting materials (Fe 99.99% and Pd 99.98%) using arc melting. A single-crystalline rod of Fe–55at.%Pd was grown by a floating zone method at a growth rate of 3.5 mm/h under a purified argon gas flow of 1 L/min. Homogenization of the single-crystalline rod was carried out at 1273 K for 168 h followed by ice-water quenching. Crystallographic orientation was determined using a Laue back reflection X-ray technique. Cubic specimens with three edges parallel to ⟨0 0 1⟩ directions

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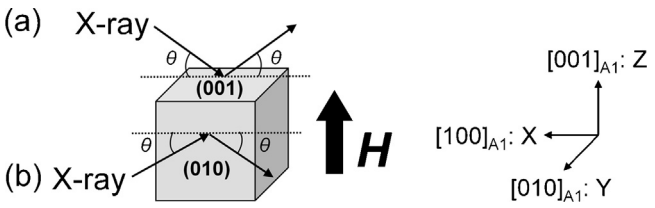

Fig. 1. Schematic illustration showing orientation of the specimen and X-ray beam irradiation to the (0 0 1) surface (a) and (0 1 0) surface (b).

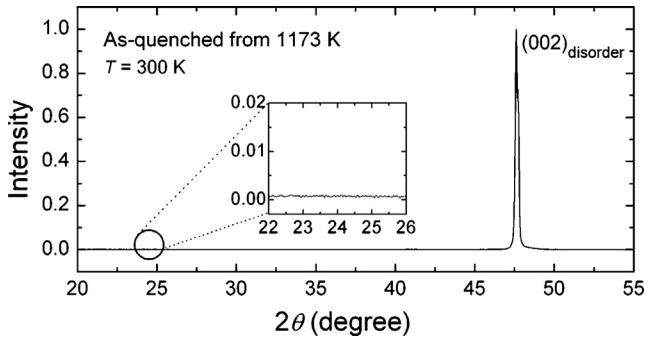

Fig. 2. XRD profile of as-quenched specimen. The cubic specimen was heat-treated at 1173 K for 1 h followed by ice-water quenching. The inset shows the area where 0 0 1 superlattice reflection appears.

(hereafter called *X*-, *Y*-, *Z*-directions) were prepared. After quenching from the disordered state (1173 K), the specimens were subjected to a two-step heat-treatment. The first step was made at 673 K for 1 h under magnetic field of 10 T applied in the *Z*-direction, and the second step at 773 K for 5 min or 24 h in the absence of the magnetic field.

Structural change during the ordering process was examined by XRD measurements and transmission electron microscope (TEM) observations. XRD measurements were made with a Rigaku RINT 2000 X-ray diffractometer using Cu K_α radiation. The specimens were set to the stage so that one of the {1 0 0} surfaces were parallel to the scattering vector as shown in Fig. 1. For TEM observations, cubic specimens were cut out so as the beam direction becomes parallel to the *X*-direction. According to our previous studies [24,25], the volume fraction of the *X*- and *Y*-variants are essentially the same. Consequently, the present results were discussed based on superlattice reflection of the *Z*- and *Y*-variants. Thin foils for the electron microscopy were prepared using a jet-polishing method with an electrolyte composed of 85 vol.% acetic acid and 15 vol.% perchloric acid. TEM observation was made by using Hitachi H-800 operated at 200 kV.

3. Results

First, we examine the as-quenched state (from 1173 K). Figs. 2 and 3 show XRD profile and electron diffraction pattern of the as-quenched specimen at 300 K, respectively. In the X-ray

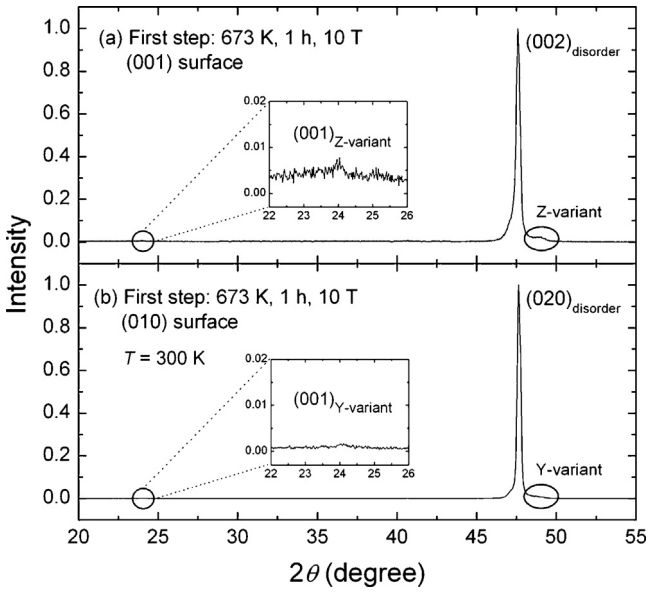

Fig. 4. XRD profile after the first step of ordering at 673 K for 1 h under magnetic field of 10 T.

profile shown in Fig. 2, the 0 0 2 fundamental reflection can be detected, and the 0 0 1 superlattice reflection is missing. The lattice parameter at 300 K is obtained to be 0.382 nm. The electron diffraction pattern shown in Fig. 3a is essentially indexed by the fcc structure and no specific microstructure can be observed in the bright field micrograph, Fig. 3b at this stage. In this way we confirmed that nearly complete disordered state is obtained by quenching the specimen from 1173 K.

Second, we examine the state after the first step of ordering at 673 K for 1 h under the magnetic field of 10 T. Fig. 4 shows the corresponding XRD profile, where besides the fundamental reflections, a very weak 0 0 1 superlattice reflection of the *Z*- and *Y*-variants can be observed as shown in the inset of Fig. 4a and b, respectively. We notice that the intensity of the superlattice reflection of the *Z*-variant is a little bit higher than that of the *Y*-variant. We also notice that the (0 0 2) fundamental reflection of the *Z*-variant is stronger than that of the *Y*-variant. Electron diffraction pattern after the first step of ordering at 673 K for 1 h under the magnetic field of 10 T is shown in Fig. 5a. The direction of the magnetic field applied in the first step is shown by a horizontal arrow in the figure. In the diffraction pattern, the superlattice reflections of the *Z*-variant and *Y*-variant are indicated by circles. We notice that the intensity

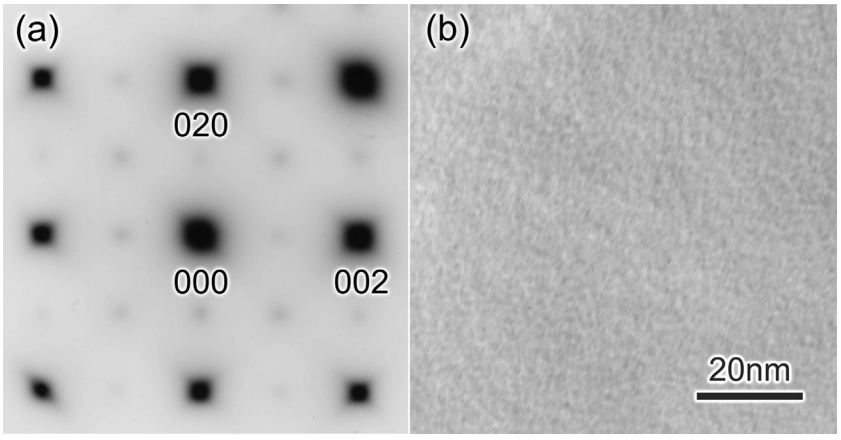

Fig. 3. Electron diffraction pattern (a) and bright field image (b) of as-quenched (from 1173 K) specimen.