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Dynamic mechanical analysis of weak first-order martensitic transformation in an iron–palladium alloy

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ABSTRACT

A disordered Fe-31.2Pd (at.%) alloy exhibits a weak first-order martensitic transformation (MT) from a face-centered cubic structure to a face-centered tetragonal structure. We employed a Dynamic Mechanical Analysis (DMA) to understand the transformation behavior. As the temperature decreases, the storage modulus E' decreases in the parent phase and increases in the martensite phase. The loss tangent (tanδ) shows a slight increase when approaching M_s (=225 K), suggesting the formation of movable interfaces even above M_s. The value of tanδ is nearly proportional to $(1 - c/a)^2$ below M_s, meaning that highly mobile twinned interfaces are essential for internal friction below M_s. The so-called transitory term of tanδ is negligibly small for the MT of this alloy.

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1. Introduction

Iron–palladium alloys with a Pd content of approximately 30 at.% exhibit a martensitic transformation (MT) from a face-centered cubic (FCC) structure to a tetragonal structure. The martensite phase is slightly contracted along one of the <001> directions and is traditionally called face-centered tetragonal (FCT) martensite [1–3]. This MT is considered to be a weak first-order transformation with two significant characteristics. First, the change in lattice parameters at the transformation temperature is small, and the lattice parameters of the martensite phase change gradually below the transformation temperature. Second, this transformation is associated with a significant softening of the elastic constant C' $(=(C_{11}-C_{12})/2)$ as the transformation temperature is approached [4].

Recently, several interesting phenomena have been found in Fe–Pd alloys exhibiting the FCC–FCT MT. One is a large magnetic-field-induced strain in the martensite phase, which is caused by the rearrangement of martensite variants by the magnetic field [5,6]. The high magnetocrystalline anisotropy of the FCT martensite phase and the low detwinning stress of the martensite phase are responsible for this behavior [7]. The second interesting phenomenon is the appearance of a large elastic-like strain of more than 6% under compression loading in the [001] direction [8,9]. Another is the large temperature change with the application or removal of stress (elastocaloric effect) within a wide temperature range with a very small hysteresis loss [10,11]. These fascinating properties of Fe–Pd alloys are probably closely related to the softening of the elastic constant C' and the movement of interfaces between martensite variants and between martensite and parent phases.

Dynamic Mechanical Analysis (DMA) is an effective method to obtain information on the elastic properties and the movement of interfaces simultaneously. Therefore, it has been used to understand the MT behavior in representative shape memory alloys such as Ti–Ni alloys [12,13], Cu–Al–Ni alloys [14], and Mn–Cu alloys [15]. However, DMA has not been extended to the weak first-order FCC–FCT MT of Fe–Pd alloys yet. In this study, we employed DMA in order to derive new aspects of the weak first-order FCC–FCT MT in Fe–Pd alloys, including the movement of interfaces.

2. Experiments

An ingot of Fe-31.2Pd (at.%) alloy was prepared by arc melting from a high-purity Fe rod (99.998%) and a Pd sheet (99.9%). From the ingot, two specimens with dimensions of 3.1 mm × 1.2 mm × 2.5 mm (Specimen-A) and 3.3 mm × 1.2 mm × 17.5 mm (Specimen-B) were cut out. They were

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sealed in an evacuated quartz tube and then homogenized at 1375 K for 24 h, followed by quenching in ice water to retain the disordered parent phase in the FCC structure.

Magnetic susceptibility was measured using Specimen-A under a magnetic field of 500 Oe with a constant cooling rate of 2 K/min in a superconducting quantum interference device magnetometer. DMA was performed in a TA-Q800 using Specimen-B. A single cantilever mode was used for all measurements. The vibrational amplitude of the specimen was between 15 μm and 150 μm; the corresponding maximum strain was between 2×10^{-4} and 2×10^{-3}. The frequency of the vibration was between 0.2 and 10 Hz. Measurements were made in a step cooling process; at every temperature, we waited 3.5 min to reduce the temperature difference between the furnace and the specimen. In addition, measurements were made during a continuous cooling process with rates of 1 K/min and 2 K/min to evaluate the influence of the so-called transitory term, which will be described later. In the holding test, the specimen was cooled from room temperature to the target temperature at a rate of 1 K/min, and then the specimen was maintained at the temperature for 1 h. In DMA, we evaluate complex Young's modulus $E^* = E' + iE''$. The real part E' is termed the storage modulus, and the ratio E''/E' is termed the loss tangent (tanδ).

3. Results

3.1. Change in storage modulus and loss tangent associated with MT

Fig. 1 (a) shows the temperature dependence of the magnetic susceptibility χ measured during the cooling process. We notice a bend point near 225 K. This temperature corresponds to the FCC–FCT MT start temperature, M_s. The decrease in χ below M_s is due to the increase in the magnetic anisotropy of the martensite phase. The transformation behavior of this specimen agrees with that in a previous report [16]. Fig. 1b and c, respectively, show the temperature dependence of the storage modulus (E') and loss tangent (tanδ) of Specimen-B measured under different frequencies in the step cooling process with a strain-amplitude of 15 μm.

According to Fig. 1b, the storage modulus E' is insensitive to the frequency over the whole temperature range examined. We also notice that the temperature at which E' is minimum agrees with the M_s determined by the magnetic susceptibility measurement. Above M_s, the storage modulus decreases monotonically with decreasing temperature, which is consistent with the softening of C' reported previously [4]. Note that dE'/dT remains almost constant above 263 K, and gradually increases with decreasing temperature in the temperature range between 263 K and M_s. The accelerated softening near M_s is possibly related to the formation of tweed, which was observed previously by transmission electron microscopy in an Fe-30.0Pd (at.%) alloy [17]. The tweed structure consists of fine striated contrasts nearly parallel to the traces of {110} planes. The contrast is explained by local tetragonal distortions associated with small coherent FCT embryos embedded in the FCC matrix [17]. Below M_s, the storage modulus increases with decreasing temperature, which implies that the elastic constants of the martensite phase increase as temperature decreases.

In Fig. 1c, we notice that tanδ is almost independent of frequency when the temperature is above M_s. The value of tanδ increases gradually in the parent phase with decreasing temperature. The change in tanδ is not linear; the increase in tanδ is 0.002 between 323 K and 263 K ($\Delta T = 60$ K), while the increase is 0.007 between 263 K and 228 K ($\Delta T = 35$ K). The cause for such behavior in tanδ implies the formation of interfaces. Presumably, the tweed microstructure in the parent phase [17] is responsible for the behavior. Below M_s, tanδ shows a frequency dependence; it increases with decreasing temperature at all frequencies examined. It shows a maximum and then decreases with a further decrease in temperature. The temperature at which tanδ shows a maximum increases with increasing frequency, which is frequently observed for relaxation processes [18,19].

3.2. Influence of strain amplitudes

Fig. 2 shows the storage modulus E' (a) and tanδ (b) of the Fe-31.2Pd (at.%) alloy measured under different strain amplitudes at a fixed frequency of 0.2 Hz. Above M_s, the storage modulus is independent of the strain amplitude. Below M_s, however, it decreases with increasing strain amplitude over the entire temperature range examined. The strain amplitude dependence of E' is probably not related to the decrease in elastic modulus but is related to the detwinning process of the FCT martensite; the applied stress induces strain by the motion of twinning planes as well as pure elastic strain. We also notice that tanδ depends strongly on the

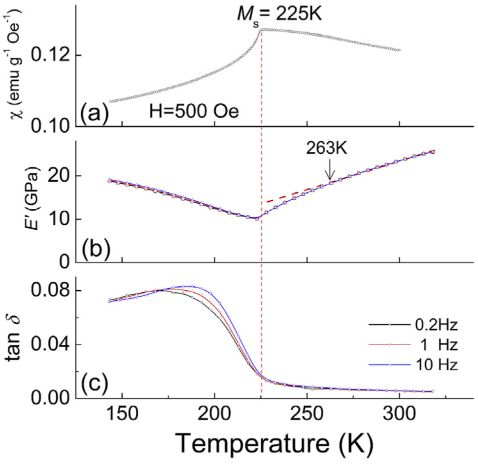

Fig. 1. Temperature dependence of (a) the magnetic susceptibility, χ, (b) the storage modulus, E', and (c) the loss tangent, tanδ, of the Fe-31.2Pd (at.%) alloy. χ was measured in a continuous cooling process with a rate of 2 K/min under a magnetic field of 500 Oe. E' and tanδ were measured in a step cooling process under a strain amplitude of 15 μm with frequencies of 0.2, 1 and 10 Hz.

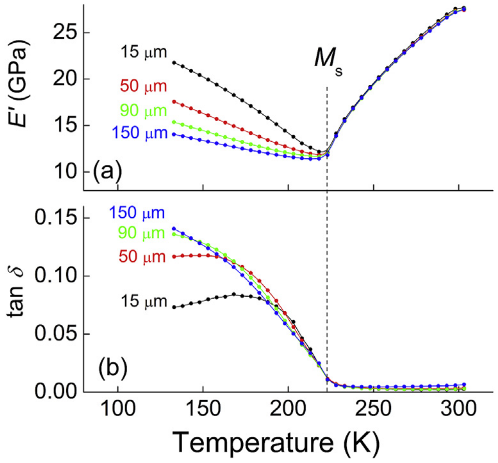

Fig. 2. Temperature dependence of (a) E' and (b) tanδ of the Fe-31.2Pd (at.%) alloy measured under different strain amplitudes as indicated in the figure. Measurements were made at 0.2 Hz in a step cooling process.

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