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Ferromagnetic Shape Memory of Nanostructure Fe-Pd Alloy: The Texture Observation Study by Laser and Electronic Microscopes

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ABSTRACT

Ferromagnetic shape memory Fe-29.6at%Pd alloy ribbon (about 60 µm thickness) prepared by the rapid solidification melt-spinning method, has a large magnetostriction of 1.0×10^{-3} when a magnetic field of 10 kOe is applied normal to the ribbon surface at room temperature. The strain induced by magnetic field is caused by the conversion of variants in the martensite phase, and increases with temperature and has a maximum at phase transformation temperature of 380-400 K. However, the mechanically shape recovery effect of the ribbon has two-step phase transformation temperature of 300-330 K and 380-420 K. To investigate the origin, we observed the texture by using laser microscope and high resolution electronic microscope. The cross section of ribbon shows columnar structure of about 10µm in width. The ribbon consists of three parts: both upper and bottom surfaces have small grains of 2-3 µm with strong [100] texture and the inner part has fine layer-structures of 30-40 nm thickness in grains. It can be concluded that this nano-scale composite structure makes phase transformation temperature increase from 300 K in surface to 380-400 K in inner part.

INTRODUCTION

Ferromagnetic shape memory alloy (FSMA) Fe-Pd is expected to be useful as a magnetic-field-drive sensor/actuator material for a micro-machine and intelligent/smart material system. The FMSA exhibits a large strain caused by martensitic twin's initiations and its movements, i.e., a new type of mangetostriction [1]. Although Fe-31.2at%Pd single crystal [2] and polycrystalline bulk [3] samples exhibit large magnetostriction, phase transformation temperatures are low than room temperature. In previous study [4, 5], we showed that Fe-29.6at%Pd alloy ribbon prepared by the rapidly solidification melt-spinning method has stronger crystal anisotropy and giant magnetostriction of 1.0×10^{-3} at room temperature with good shape memory effect. The magnetostriction of this ribbon is 10 times as large as polycrystalline bulk value. Its value increases with temperature and has a maximum at the phase transformation temperature of 380-400 K, though the austenite finishing temperature A_f of Fe-29.6at%Pd single crystal is 320-330 K [6].

In the present study, to investigate the origin of the temperature discrepancy, we observed the texture of ribbon sample by using laser microscope and high resolution electronic microscopes. We found that the martensite twin in the inner part of ribbon consists of fine layer-structures of 30-40 nm thickness.

EXPERIMENTAL DETAILS

The rapidly solidified Fe–29.6at%Pd ribbon sample with 60 µm in thickness, was prepared by originally designed electro-magnetic melt-spinning single-roll method from bulk alloy [4]. Some of ribbon was annealed at 1173 K for 1 h in vacuum atmosphere to study the effect of heat treatment on magnetostriction. The structure of ribbon was researched by XRD method with Cu Kα radiation. The microstructure of texture of ribbon was observed by

using laser and high resolution electronic microscopes. The magnetization M vs. applied magnetic field H loop was measured by VSM.

The magnetostriction ε was measured by a strain-gauge attached on the sample (see Fig.4(a)). The magnetic field was applied perpendicular to the rolling direction (RD) and strain changes were measured along RD. The shape memory effect was evaluated from the changes of shape recovery of the curled ribbon with temperature.

RESULTS AND DISCUSSION

Microstructure of ribbon

Figure 1 shows laser microscope photographs of (a) the contact surface and (b) the free surface of as-spun ribbon. Microstructure of roll contacting surface, which is covered by roller traces, is observed in (a). As seen in (b), the free surface consists of microstructure of grains with about 3 μm size.

Figure 2 shows (a) the schematic diagram of the rapidly solidification apparatus and processed ribbon sample, (b) the micrograph of as-spun Fe-29.6at%Pd ribbon. The cross section of ribbon shows unevenness of columnar texture of about 20 μm. Moreover, there is a fine chilled region of 10-15 μm on roll contacting side.
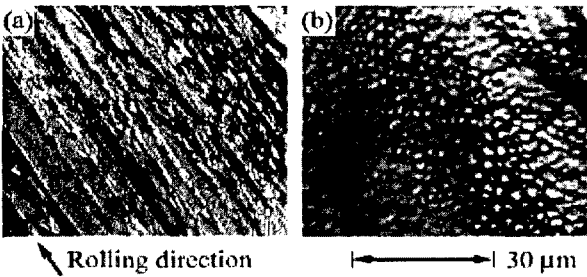

Fig. 1 Laser microscope photographs of (a) the contact surface and (b) the free surface of as-spun Fe-29.6at%Pd ribbon.
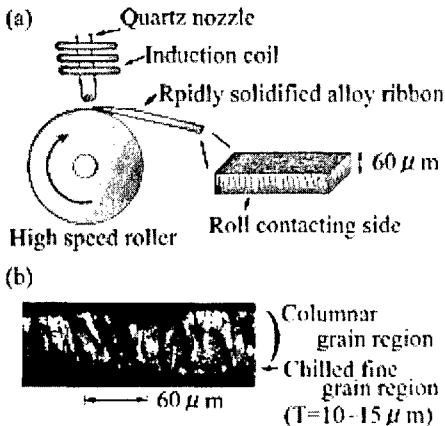

Fig.2 (a) Schematic diagram of the rapidly solidification apparatus and processed ribbon sample, (b) a micrograph of as-spun Fe-29.6at%Pd ribbon.

Figure 3 shows the XRD profiles of (a) roll contact surface and (b) free surface for as-spun and annealed ribbons. The fct martensite and fcc austenite phases coexist in the ribbon because fcc and fct {111} peak at near 2θ=42 K fct(200), (020), fcc{200} and fct(002) peaks at 47 K<2θ<52 K arise in Fig.3. The second and third peaks are larger than the first one for the free surface, indicating that the ribbon has strong [100]-oriented texture. Moreover, the fct(200), (020) and fct(002) peaks characteristic of martensite phase increases after annealing at 1173 K for 1 h.
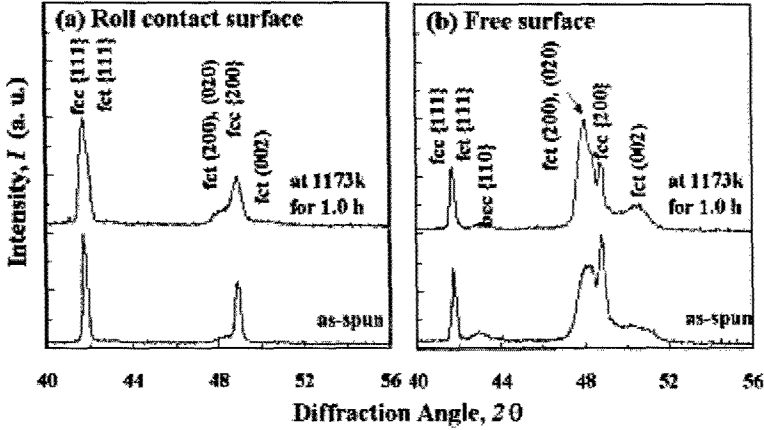

Fig. 3   XRD profiles on (a) roll contact surface and (b) free surface of
as-spun and annealed Fe-29.6at%Pd ribbons.

Magnetic and magnetostriction properties

Figure 4(a) is the M–H loops of as-spun ribbon, where θ is the rotation angle between the transverse direction of ribbon and H, and θ=0 K and 90 K denote H parallel and perpendicular to plate of ribbon.
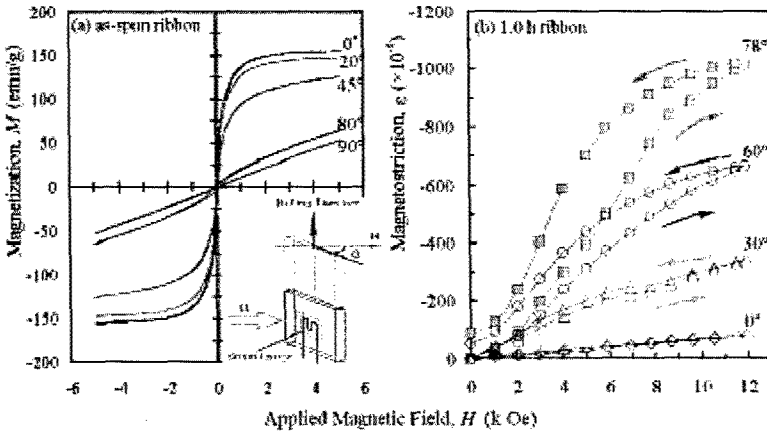

Fig. 4 (a) M–H loops of as-spun Fe-29.6at%Pd ribbon and (b)
magnetostriction vs. H curves after annealing for 1 h at 1173K.

When H=2.5 kOe parallel to plate, the saturation magnetization Ms of ribbon is 153 emu/g, indicating that it is ferromagnetic. When H of 5 kOe applies to the θ=90 Kcase, the magnetization of ribbon can not be saturated because of a large demagnetizing field and the coercive force (Hc) is 65 Oe, which is larger than one for θ=0 K The above results indicate that the ribbon has magnetic anisotoropy. Figure 4(b) shows the magnetostriction ε vs. H curves of the ribbon annealed for 1 h at 1173 K, which measures change in strain in RD. The value depends remarkably on θ, and has a maximum of - 0.1 % strain at θ=78 Kdirection. The strain decreases with H and goes almost back to the H=0 value. The reason that the maximum arises at thickness direction is considered to be that [100] directions of the texture of ribbon are distributed around the center of pole figure [7].
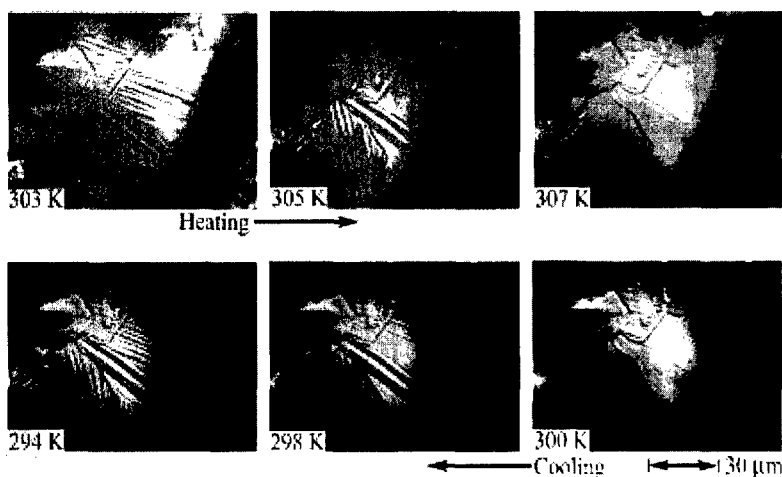
Phase transition temperature

Fig. 5 Laser microscope photographs on surface of coarse grain (about 30μm).

Figure 5 shows the laser microscope photographs of surface of coarse grain (about 30 μm). On heating process, the martensite twin observed at 303 K disappears at 307 K. On cooling process, the same martensite twin is recovered at 298 K. From these results, A_f and the martensite starting temperature M_s of the grain are determined to be 307 K and 298 K, respectively, in almost agreement with the values obtained from XRD measurement for the same ribbon [4] and single crystals [6].

Dependence of the magnetostriction and shape recovery ratio of as-spun ribbon on temperature is shown in Fig. 6(a). The magnetostriction increases from $3.2×10^{-4}$ at room temperature to $7.0×10^{-4}$ at 376 K with temperature. In the range of T>376 K, the strain decreases and suddenly falls to $1.6×10^{-4}$. We consider that this phenomenon is closely related with the mobility of martensite twin, that is, the mobility of variants is activated by heating, and a maximum strain arises near the phase transformation temperature, A_s. A_s and A_f of the ribbon are found to be 376 K and 450 K, respectively. Moreover, the shape recovery ratio Φ_{T2}/Φ_{T1} increases with temperature, where Φ_{T1} and Φ_{T2} are diameters of curled ribbon at 300 K and T K, respectively (see Fig. 6(b)). The variation is rapid in temperature ranges of 300-330 K and 380-420 K. These results suggest that the ribbon has two-step phase transformations from martensite phase to austenite one. Low A_s-A_f, 300-330 K is consistent
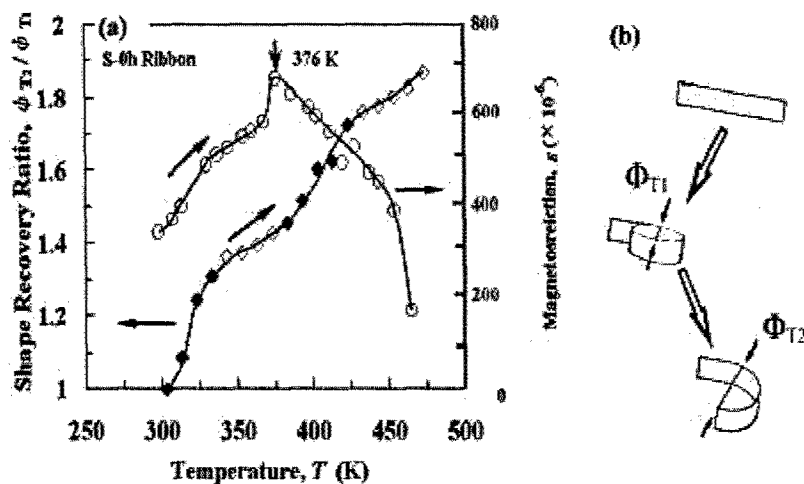
Fig. 6 Temperature dependence of the magnetostriction and
shape recovery ratio of as-spun Fe-29.6at%Pd ribbon.

with the value obtained from laser microscope observation of the surface in Fig.5 and XRD
[4]. Therefore, the low A_s-A_f is net phase transformation temperature of Fe-29.6at%Pd alloy.
Note that high A_s-A_f, 380-420 K is peculiar to rapidly solidification ribbon.

Nano-scale composite structure

↦ 50 nm

Fig. 7 High-resolution electron-microscope photograph
of the inner part of ribbon.

To investigate the origin of high A_s-A_f, we observed inner part of ribbon by using high resolution electronic microscope. The observed photograph is shown in Fig.7. There, it is seen that grains of about 1µm size consist of fine layer-structures of 30–40 nm thickness. That is , they are martensite twins because the widths of dark and light layers are in the ratio of about 2 to 1. These nano-scale layers are parallel to the columnar structure which is in the same direction of thermal diffusion on roll. This nano-scale composite structure is stress-induced martensite twin. Therefore, it can be considered that nano-scale martensite twins make phase-transformation temperature increase from 300-330 K in surface to 380-420 K in inner part.

CONCLUSION

Rapidly solidification FSMA Fe-29.6at%Pd ribbon has two-step phase transformation temperatures. To investigate the origin, we observed the texture by using laser and high resolution electron microscopes. The cross-section of ribbon has columnar structure of about 10 µm in width. The ribbon consists of three parts: both upper and bottom surfaces have small grains of 2-3 µm with strong [100] texture and the inner part has fine layer-structures of 30–40 nm thickness in grains. It can be concluded that this nano-scale structure makes phase-transformation temperature increase.

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