



Constricted hysteresis loops in Fe and Ni single crystals

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ABSTRACT

Magnetic hysteresis loops reflect the variety of magnetic domain structures and have been considered to have normal rectangular or leaf-like shapes in standard ferromagnets such as Fe and Ni metals. We report on observations of constricted hysteresis loops in Fe and Ni single crystals with very low defect densities. The constricted loops were observed below $T=150$ K and in a medium temperature range from 150 to 430 K in Fe and Ni single crystals, respectively. These constricted loops disappear by weak plastic deformation for both single crystals. The origin of constricted hysteresis loops was explained by eddy current effects under less domain wall pinning due to dislocations.

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1. Introduction

Since the discovery of ferromagnetism, fundamental hysteresis loops for Fe and Ni metals have been considered to have normal rectangular or leaf-like shapes and the magnetization models have been constructed according to their properties from the viewpoint of irreversible movement of domain walls and the rotation of magnetic moments [1–3]. The hysteresis loops reflect intrinsic features of ferromagnets including magneto-crystalline anisotropy, magnetostriction, etc. and are affected by lattice defects such as dislocations, impurities, grain boundaries, voids, etc. In addition to lattice defects, eddy current effects would exert an influence on the domain wall movement. Of our interest is the role of two effects in the magnetization process.

The constriction of hysteresis loops, associated with very small remanence and low permeability at coercive field was first discovered in heat-treated Fe–Co–Ni alloys called “Perminvars” [4]. The constriction depends both on condition of heat treatment and compositions, and is lost at elevated temperatures. The appearance has been considered to be related with both uniaxial anisotropy and atomic short-range order, and was explained as due to two neighboring uniaxial domains whose axes intersect perpendicularly with each other, yielding strong resistance to domain wall displacement [5]. So far, constricted hysteresis loops have been observed in other materials and various mechanisms have been proposed. For instance, stabilization of 90° wall by compressive stress [6–8] or both 180° and 90° walls by interstitial atoms [9,10] through magnetoelastic coupling causes loop

constriction in Fe-based alloys, while that of stripe domains and a circular domain does in oblique-incidence Permalloy films [11] and single-domain nanomagnets with a vortex state [12,13], respectively. On the other hand, constricted loops were observed in hard-rolled Co–Fe alloys [14], polished amorphous alloys [15], heat-treated polycrystalline ferrites [16] and Cu–Mn–Al alloys [17,18], and exchange-spring nanocomposite magnets [19], where magnetostatic and/or exchange coupling between magnetic domains with different magnetic properties play an important role. Generally, these systems are associated with artificial metallurgical treatments or size effects or sample inhomogeneity. The constricted hysteresis loops have been therefore considered to occur in ferromagnetic materials under special conditions.

In this paper, we report on observations of constricted hysteresis loops in bulk Fe and Ni single crystals with low defect densities. We find that the constriction occurs below $T=150$ and 150–430 K for Fe and Ni single crystals, respectively, and is lost after weak plastic deformation. A different mechanism for the constriction based on eddy current effects is introduced to explain the observations.

2. Experimental

Sheets of Fe single crystals with 1 mm in thickness and with low impurity contents ($< \sim 0.002$ wt%) were prepared with strain-annealing method [20]. The sheet samples have (100) plane on the sheet surface. Cylindrical Ni single crystals with 12 mm in diameter and with [113] growth direction were prepared with electron-beam floating zone melting in vacuum. To see effects of dislocations on magnetic properties, some sheet Fe samples were plastically deformed in tension at room

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temperature up to a true stress of 142 MPa (25% true strain) with an Instron-type testing machine along the crystal direction of $\langle 100 \rangle$, whereas cylindrical Ni samples were compressively deformed along the $\langle 113 \rangle$ direction up to a true stress of 25 MPa (5% true strain). The strain rate for Fe and Ni samples was 1.5%/min and 1.0%/min, respectively.

For magnetic measurements, the Fe and Ni samples were cut into picture frames with $[100]$ and $[010]$ frame axes and rings, respectively [21–23]. To obtain a sample with low defect densities, a ring sample of as-grown Ni single crystal used in previous measurements [21,22] was annealed at $T=1373$ K for a day. Both exciting and pickup coils were wound around the samples. The samples were placed in a He-gas closed-cycle refrigerator with a high-temperature stage, by which the temperature was varied from 10 to 600 K.

A triangular voltage with a frequency of 0.02 and 0.05 Hz for the Fe and Ni samples, respectively, obtained from a function generator was applied to a bipolar power supply. The bipolar power supply converts the voltage to current and amplifies the

current. The amplified current was applied to the exciting coil to generate a cyclic magnetic field and magnetize the sample. The magnetic field within the sample was obtained from the voltage across a $1\text{-}\Omega$ resistance connected to the exciting coil in series. The induced voltage of the pickup coil was amplified and then purified by a low-pass filter with a cutoff frequency of 40 Hz. After this signal processing, the induced voltage was integrated in order to obtain a magnetic flux within the sample. To examine dependence of field amplitude of a cyclic field, H_a , on hysteresis properties, a set of magnetic minor hysteresis loops with various H_a up to 4 kA/m, which are symmetrical about the origin, was measured. Before measuring each minor loop, the sample was demagnetized with decaying alternating magnetic field.

3. Results and discussion

Fig. 1(a) shows a set of minor hysteresis loops at $T=10$ K for as-grown Fe single crystal. The constricted hysteresis loops

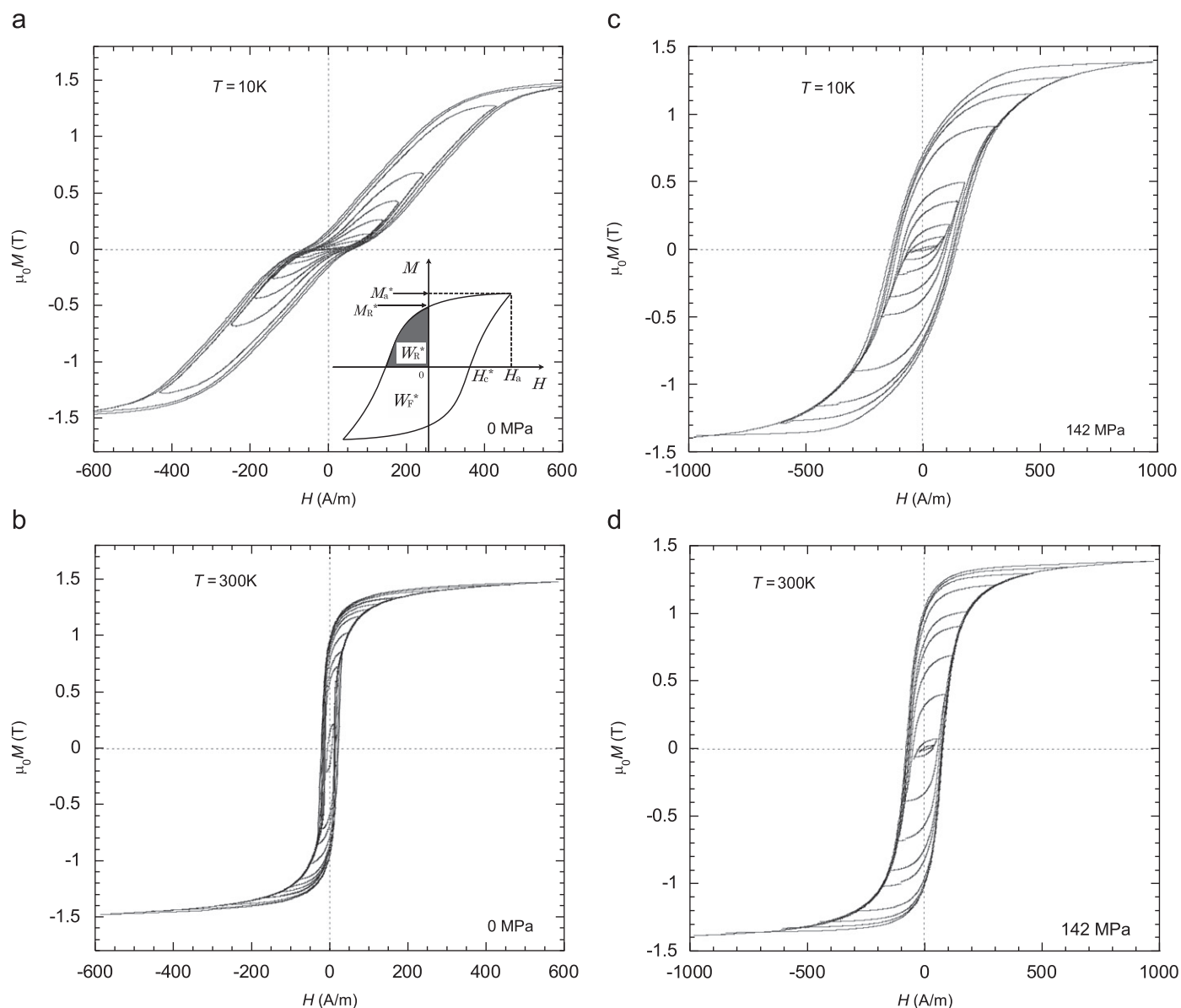


Fig. 1. Set of magnetic minor hysteresis loops for Fe single crystals before plastic deformation, taken at (a) 10 K and (b) 300 K and after plastic deformation by 142 MPa (25% true strain), taken at (c) 10 K and (d) 300 K. The inset in (a) shows parameters of a minor hysteresis loop.

were observed both in minor and major loops and have characteristics of small remanence and low permeability at coercive field. The constriction of the loops at $T=10\text{K}$ is reflected in the field dependence of differential permeability μ shown in Fig. 2(a), where μ exhibits a maximum both above and below the zero applied field on the descending branch of hysteresis loop. Such constricted behavior was only observed for minor loops with $\mu_0 M_a^* > 0.05\text{ T}$ where irreversible movement of domain walls takes place. With increasing temperature, the maxima of μ rapidly develop associated with a shift of the position toward $H=0$. At $T \sim 150\text{ K}$, the constricted behavior disappears and μ shows only a single peak at $H < 0$ on the descending branch of hysteresis loop as shown in Figs. 1(b) and 2(a). No constricted loops were observed at higher temperatures up to 500 K . The constriction of hysteresis loops disappears after plastic deformation as shown in Fig. 1(c) and (d) and only a single peak of μ is present in the field dependence as shown in Fig. 2(b).

On the other hand, for the annealed Ni sample, hysteresis loops at $T=10\text{ K}$ are normal in shape but are constricted at $T=300\text{ K}$ as shown in Fig. 3(a) and (b). The constricted hysteresis loops appear above $T \sim 150\text{ K}$ and their characteristics are the same as those for as-grown Fe single crystal below $T \sim 150\text{ K}$. The changes with temperature can be seen in the field dependence of μ on descending branch of hysteresis loop shown in Fig. 2(c) and (d); μ shows a single peak at $T=10\text{ K}$ at $H < 0$, but it exhibits two peaks above $T \sim 150\text{ K}$. As the temperature increases, the peak at $H < 0$ gradually shifts toward zero field, whereas that at $H > 0$ is weakly temperature dependent. At $T=430\text{ K}$, μ again exhibits a single peak at $H < 0$ on the descending branch of hysteresis loop, though it is rather broad. These observations show that hysteresis loops become constricted in the medium temperature range from 150 to 430 K for the annealed Ni single crystal unlike below $T=150\text{ K}$ for as-grown Fe single crystal. This constriction behavior is absent for as-grown Ni single crystal [21,22] and also for compressively deformed sample as shown in Figs. 3(c), (d) and 2(d).

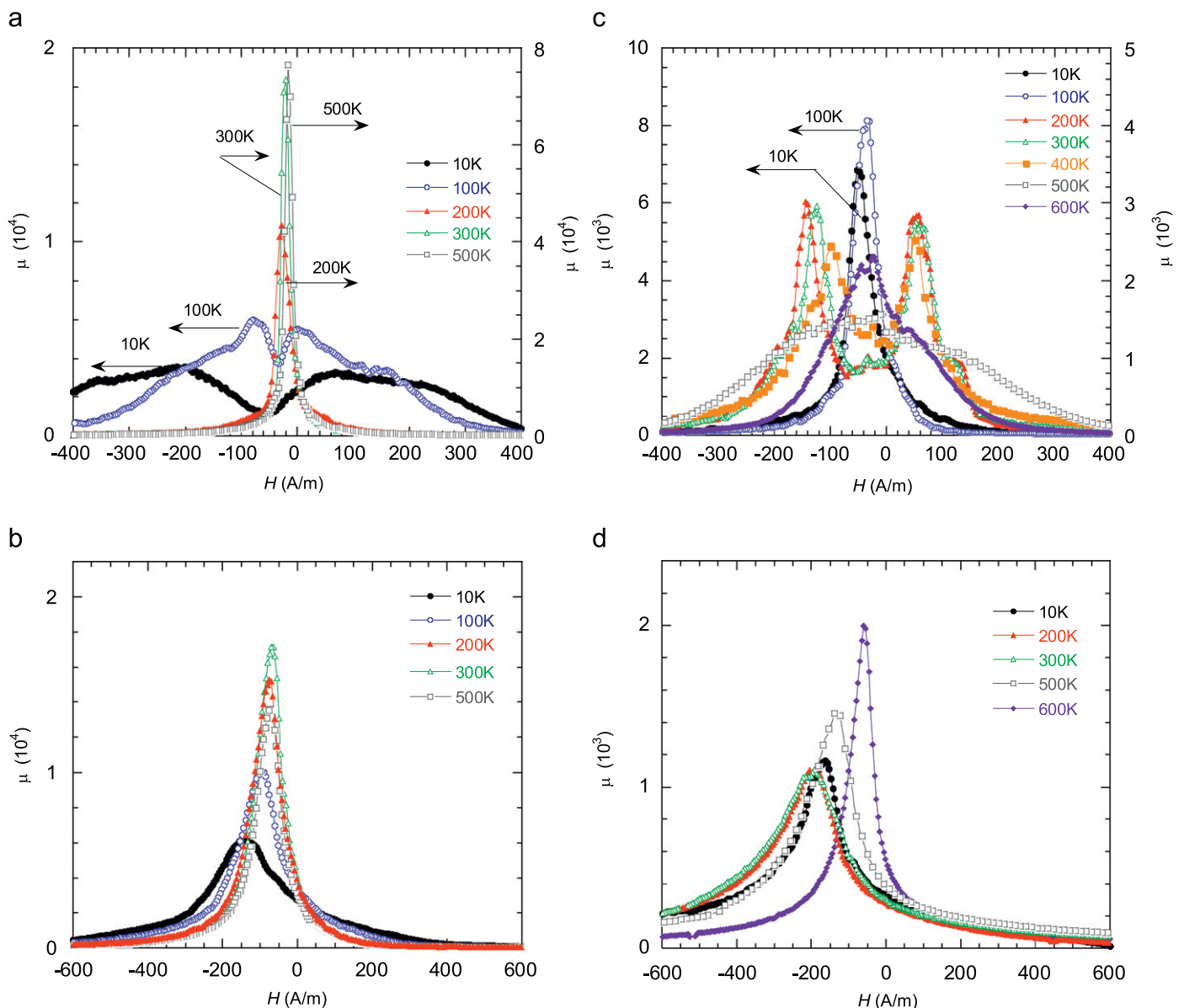


Fig. 2. Differential permeability as a function of magnetic field for Fe single crystals (a) before and (b) after plastic deformation by 142 MPa (25% true strain), and for Ni single crystal (c) before and (d) after plastic deformation by 25 MPa (5% true strain), taken at various temperatures. The data were obtained from descending branch of minor hysteresis loop with $H_a = 400\text{ A/m}$ for 0 MPa and 600 A/m for 142 or 25 MPa.

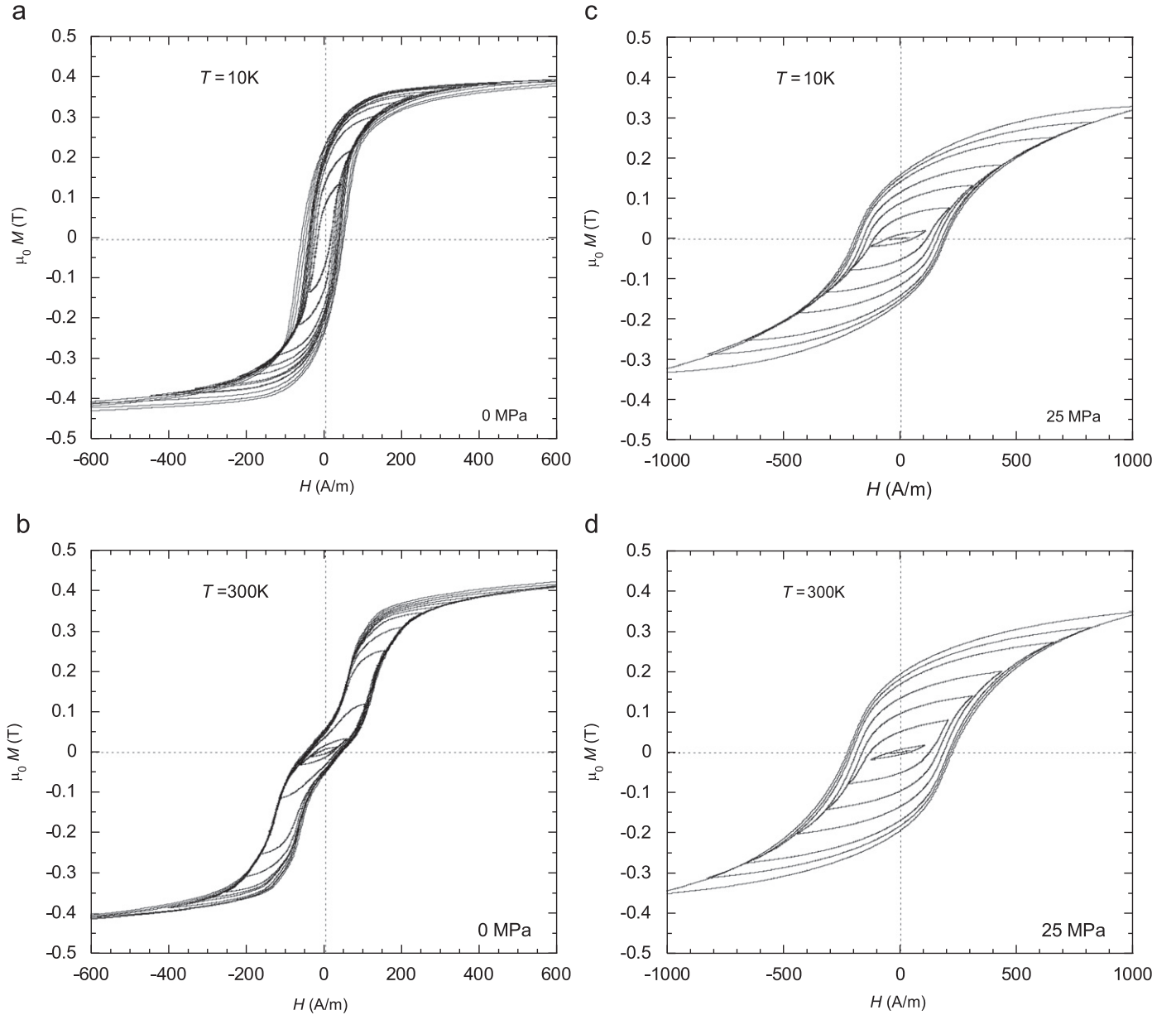


Fig. 3. Set of magnetic minor hysteresis loops for Ni single crystals before plastic deformation, taken at (a) 10 K and (b) 300 K and after plastic deformation by 25 MPa (5% true strain), taken at (c) 10 K and (d) 300 K.

To investigate effects of the constriction on magnetic properties in detail, a set of minor hysteresis loops was analyzed using scaling power-law relations between parameters of minor loops. In the second stage of magnetization process where the irreversible movement of domain walls plays a dominant role, there exist several scaling power-law relations [21–23], given by such as

$$W_F^* = W_F^0 \left(\frac{M_R^*}{M_S} \right)^{n_F}, \quad (1)$$

$$W_R^* = W_R^0 \left(\frac{M_R^*}{M_R} \right)^{n_R}, \quad (2)$$

and

$$H_c^* = H_c^0 \left(\frac{M_R^*}{M_R} \right)^{n_c}, \quad (3)$$

where W_F^* is minor-loop hysteresis loss, W_R^* is minor-loop remanence work, M_R^* is minor-loop remanence, and H_c^* is minor-loop coercive force as denoted in the inset in Fig. 1(a). M_S and M_R are saturation magnetization and remanence, respectively. Exponents n_F , n_R , and n_c are about 1.5, 1.5 and 0.45, respectively, being independent of ferromagnets, stress and temperature. Here, Eq. (1) corresponds to the well-known Steinmetz law [24]. From the least-squares fits to the equations, minor-loop coefficients W_F^0 , W_R^0 , and H_c^0 which are sensitive indicators of internal stress were obtained. Since both coefficients show a similar behavior to each other, only results of W_F^0 will be given.

Fig. 4(a) shows the temperature dependence of W_F^0 for Fe single crystal before and after plastic deformation by 142 MPa. W_F^0 does not depend remarkably on temperature above 200 K, but increases rapidly with decreasing temperature below 200 K for both samples. On the other hand, for the annealed Ni single crystal, W_F^0 shows two local maxima at $T \sim 220$ and 540 K as

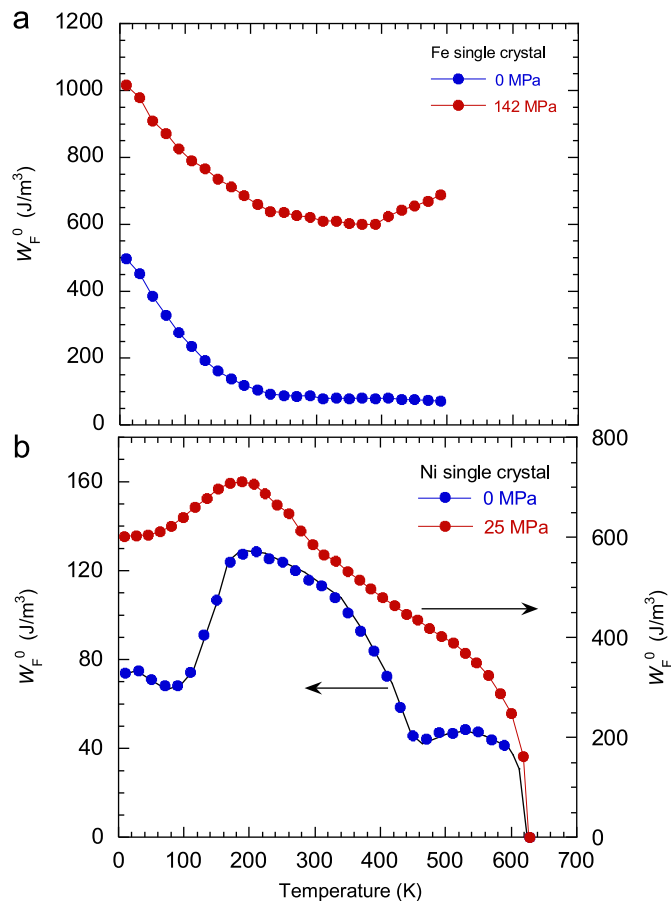


Fig. 4. Temperature dependence of W_F^0 before and after plastic deformation, for (a) Fe and (b) Ni single crystals.

shown in Fig. 4(b), whereas for plastically deformed Ni single crystal the absolute value largely increases and W_F^0 shows only a small local maximum at $T \sim 200$ K. The comparison of the behavior of W_F^0 with the temperature variation of permeability data shown in Fig. 2 clearly indicates that the constriction of hysteresis loops is associated with anomalous increase in minor-loop coefficients for both Fe and Ni single crystals.

Now, we discuss the origin of constricted hysteresis loops for Fe and Ni single crystals with low defect densities. For Fe single crystal, the characteristics of the constriction are most pronounced at the lowest measuring temperature of 10 K. Considering the enhancement of eddy current effect at low temperatures due to the increase of electron conductivity, these behaviors would be explained as follows. As the domain walls displace, the local magnetization around the domain wall changes and eddy current is induced within the domain wall. Since the force due to eddy current acts on domain walls so as to reverse the direction of their movement, the eddy current has an effect of the viscosity on the domain wall displacement, resulting in low permeability around coercive field below $T = 150$ K. When the applied field is reduced from a high field, the magnetic moments turn to the easy direction, 90° domain walls appear and start to displace, followed by the nucleation of 180° domain walls and their displacement. When the applied field is removed completely, most 180° domain walls are pinned by dislocations, yielding high remanence at $T = 300$ K. On the other hand, at $T = 10$ K, 180° domain walls can jump the highest pinning point at a higher field due to the reaction force against the applied field. Therefore, domain walls can go down to remanence without pinning under the dissipation

force due to eddy current. As a result, 180° domain walls settle close to a nearly demagnetized state at zero field, yielding very small remanence at $T = 10$ K. The dissipation force due to eddy current also makes the prompt change of magnetization toward the opposite easy direction difficult, yielding a very low permeability around coercive field at $T = 10$ K.

For the annealed Ni sample, the same explanation would be applied to the appearance of constricted loops. The negligible pinning effect and pronounced eddy current effect may yield the similar constricted loops at $T = 300$ K for the Ni sample to those at $T = 10$ K for the Fe sample. This eddy current effect may also decide the shape of hysteresis loops at low temperatures, but the shape below $T = 150$ K is normal. The decrease of domain wall thickness below $T \sim 200$ K, associated with a rapid increase of magneto-crystalline anisotropy [25] might cause three effects on the domain wall movement: (I) a decrease of the resistance to the movement because of the decrease in the number density of obstacles in a wall, (II) a weakening of eddy current effect, and (III) strengthening of interaction between 180° domain wall and obstacles due to an increasing angle between neighboring magnetic moments in a wall. Moreover, the increase of magnetostriction constants below 200 K [26] would enhance the interaction. The combination of these effects may make the pinning effect predominant at low temperatures, yielding a large remanence and high permeability around coercive field as shown in Fig. 3(a). This pinning effect is further enhanced in the sample deformed plastically and the hysteresis loops are unconstricted even at $T = 300$ K as shown in Fig. 3(d).

Generally, temperature variations of hysteresis properties including minor-loop coefficients and coercive field reflect the temperature dependence of magnetostriction constant, elastic constant, domain wall thickness and saturation magnetization, etc. [2]. For Fe single crystal, variations of these constants at low temperatures are very small [27,28] and the anomalous increase below $T \sim 200$ K may be primarily due to the eddy current effect. On the other hand, for Ni single crystal, both magnetostriction constant and domain wall thickness show a remarkable temperature dependence and will exert a significant influence on minor-loop coefficients; a steep decrease of W_F^0 below $T \sim 200$ K can be due to a decrease of the domain wall thickness, while a local minimum around $T \sim 450$ K may reflect a shrinking of domain walls around this temperature where the magneto-crystalline constant K_1 takes a local maximum [25]. Theoretical study on the constriction in connection with hysteresis properties varying temperature and field amplitude would shed light on the origin of hysteresis loops in ferromagnets with very low defect densities.

4. Conclusion

We observed constricted hysteresis loops in single crystals of Fe and Ni with low defect densities at temperatures below 150 K and from 150 to 430 K, respectively. The appearance was explained from the viewpoint of competition between pinning effects of domain walls due to dislocations and eddy current effects. So far, constricted loops have been observed in other systems and there seems to be three major mechanisms which dominate the constriction; (i) stabilization of 90° (and 180°) walls by artificial metallurgical treatments such as uniaxial stress, charging interstitial atoms, (ii) stabilization of special magnetic domain(s) by size effects, (iii) magnetostatic and/or exchange coupling between magnetic domains with different magnetic properties. The first mechanism accompanies an increase in internal stress, while both second and third ones assume ferromagnets with reduced dimensions and sample inhomogeneity, respectively. These mechanisms are not expected in bulk single

crystals with low defect densities. The present observations clearly demonstrate that constricted hysteresis loops can occur even in simple single-crystal ferromagnets if defect density is low.

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