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A scaling power-law relation in magnetic minor hysteresis loops in Fe and Ni metals

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Abstract

A simple scaling relationship has been found in the limited amplitude range of quasistatic minor hysteresis loops: $W_a^* = W_a^0(M_a^*/M_s)^n$, where W_a^* is the area enclosed by the minor hysteresis curve and minor-loop magnetization M_a^* in the first quadrant. W_a^* is related to the energy dissipated during the restoration process of domain walls toward the zero applied field state. M_s is the saturation magnetization and n has a constant value of about 1.0, being independent of ferromagnets, temperature, and lattice defects such as dislocations. The minor-loop effluence coefficient W_a^0 has information on microstructure and shows a simple linear relation to the applied stress. The dependence of W_a^0 on applied stress and temperature can be explained by use of magneto-striction constants, the magneto-crystalline anisotropy, and electrical conductivity.

1. Introduction

Magnetic hysteresis loops of ferromagnets are characterized by domain wall motion and rotation of magnetic moments. Because of the magneto-elastic coupling, the hysteresis loop is strongly affected by the strain field produced by lattice defects such as dislocations, grain boundaries, and impurity atoms. Both experimental and theoretical works revealed the simple relationships between magnetic properties and the dislocation density that both coercive force and the inverse of initial susceptibility increase in proportion to the square root of dislocation density [1].

Minor hysteresis loops, which are obtained at magnetic fields less than the saturation field, also contain much information about lattice defects [2–9], although minor-loop properties strongly depend on magnetic field amplitude H_a. Recently, we found an analysis method of a set of minor loops in single crystals of Fe [6] and Ni [7], Fe polycrystals [8], and A533B steels [9], by the introduction of several minor-loop properties as shown in figure 1: minor-loop magnetization M_a^*, minor-loop coercive force H_c^*, minor-loop remanence M_R^*, minor-loop hysteresis loss W_F^*, minor-loop remanence work W_R^*, and three minor-loop susceptibilities χ_H^*, χ_R^* and χ_a^*. These parameters depend on microstructures as well as the magnetic field amplitude H_a. We found six scaling rules between a pair of minor-loop properties in the first and second stages of the magnetization process [10], given by

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

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

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

$$R_C^* = \frac{1}{\chi_H^*} = R_C^0 \exp\left(-b\frac{H_c^*}{H_c}\right), \quad (4)$$

$$\chi_R^* = \chi_T^0 \left(\frac{M_R^*}{M_R}\right)^{n_T}, \quad (5)$$

and

$$\chi_a^* = \chi_S^0 \left(\frac{M_a^*}{M_s}\right)^{n_S}, \quad (6)$$

where W_F^0, W_R^0, H_c^0, R_c^0, χ_T^0, and χ_S^0 are minor-loop coefficients sensitive to lattice defects. M_S and M_R are saturation magnetization and remanence, respectively. The power-law exponents n_F, n_R, and n_C are about 1.5, 1.5, and 0.45, respectively and nearly constant, being independent of the kind of ferromagnet, whereas b, n_T, and n_s, which are related to minor-loop susceptibilities, have values of 2–6, 0.1–0.5, and 0.5–1.0, respectively, depending on the kinds of ferromagnets. Note that the relation between W_F^* and M_a^* with $n_F = 1.6$

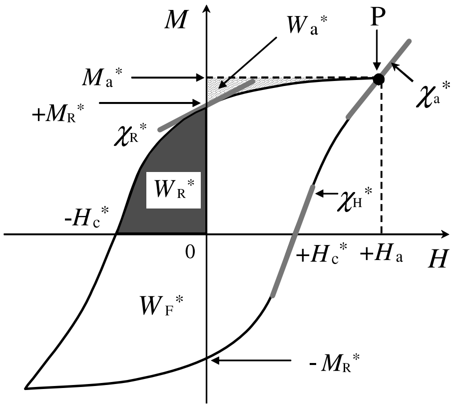

Figure 1. Parameters of a minor hysteresis loop.

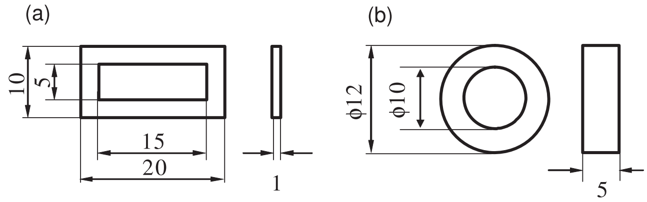

Figure 2. Size of (a) a picture frame sample and (b) a ring sample. The dimensions are in mm.

was discovered by Steinmetz about one century ago and is well known as the Steinmetz law [11].

Our detailed studies [6–9] showed that the exponents n_F, n_R, and n_C are almost independent of crystal structure, crystal orientation, and applied stress. All minor-loop coefficients W_F^0, W_R^0, H_c^0, R_c^0, $1/\chi_T^0$, and $1/\chi_S^0$ increase with dislocation density and are more sensitive to lattice defects compared with traditional structure-sensitive properties of major hysteresis loop such as coercive force. Further, the coefficients can be obtained with low magnetic fields less than about 2 kA m^{-1}, unlike the coercive force which requires high magnetic fields of several tens of kA m^{-1}. These advantages over the major loop will offer a new magnetic inspection method of evaluating material degradation in ferromagnetic materials.

In this study, another scaling rule between minor-loop properties has been examined experimentally in single crystals of Fe and Ni, and Ni polycrystals, where dislocations were introduced by plastic deformation in tension or compression. Besides the parameters introduced in previous works [6–9], we focus on the minor-loop stored energy W_a^*, which is the area enclosed by M_R^*, P, and M_a^* in figure 1 (the shaded area). Generally, the magnetic free energies which contribute to W_a^* are primarily due to magneto-static energy and magneto-crystalline energy. When the applied magnetic field is removed to zero from $H = H_a$, the value of magnetization in the system is changed from M_a^* to M_R^* by the restoring force on magnetic domain walls. W_a^* is related to the energy dissipated during the restoration process of domain walls toward a state in zero applied field and has a different meaning from other energies W_F^* and W_R^*; W_F^* is the energy dissipated in one cycle of the minor hysteresis loops and W_R^* is the externally added work for domain walls to displace in order to make the magnetization zero. In the present study, a relationship between W_a^* and M_a^* was investigated in detail, varying applied stress and temperature in a wide temperature range from 10 to 600 K. An explanation for the coefficient was given from the viewpoint of the interaction between magnetic domain walls and dislocations.

2. Experimental procedure

Sheets of Fe single crystals and cylindrical Ni single crystals, and sheets of Ni polycrystals with purity of 99.9% were prepared. The Ni polycrystals were annealed at 1273 K in vacuum for 6 h, followed by slow cooling. The average grain size was about 180 μm. The sheets of Fe single crystals and Ni polycrystals were deformed in tension at room temperature with an Instron-type testing machine, while cylindrical Ni single crystals were compressively deformed. The yield stress τ_0 was about 50, 17, and 9 MPa for Fe and Ni single crystals, and Ni polycrystals, respectively.

For magnetic measurements, Fe single crystals were cut into picture frames with [100] and [010] frame axes, while Ni samples were cut into rings, as shown in figure 2. Both exciting and pickup coils were wound around the samples. The samples were mounted in an He-gas closed cycle refrigerator with high temperature stage by which temperature was varied from 10 to 600 K. A set of magnetic minor hysteresis loops with various amplitudes of a cyclic field up to 4 kA m^{-1} was measured. The frequency of a cyclic field was 0.02, 0.1, and 0.05 for Fe and Ni single crystals, and Ni polycrystals, respectively. Before measuring each minor loop, the sample was demagnetized with decaying alternating magnetic field.

3. Experimental results

From a set of minor hysteresis loops, the parameters shown in figure 1 were determined for each minor loop and a relation between W_a^* and M_a^* was examined. Figure 3 shows the dependence of minor-loop stored energy W_a^* on minor-loop magnetization M_a^* in Fe single crystal samples at $T = 10, 100, 200, 300,$ and 500 K before and after plastic deformation. W_a^* and M_a^* are plotted on a double logarithmic scale. As can be seen, the relation shows a straight line in a limited range of M_a^* both before and after the deformation, though the slope becomes steeper at low temperatures below 200 K for undeformed and lightly deformed samples. This field range of M_a^* corresponds to the second stage of the magnetization process where magnetization against applied field shows a steep slope and the scaling relations of (1)–(3) hold true.

The linear relation in the double logarithmic plot indicates that there exists a power-law scaling relation between W_a^* and M_a^*. To elucidate the dependence of the relation on temperature and applied stress, we assume an equation given by

$$W_a^* = W_a^0 \left(\frac{M_a^*}{M_s}\right)^{n_a}, \qquad (7)$$

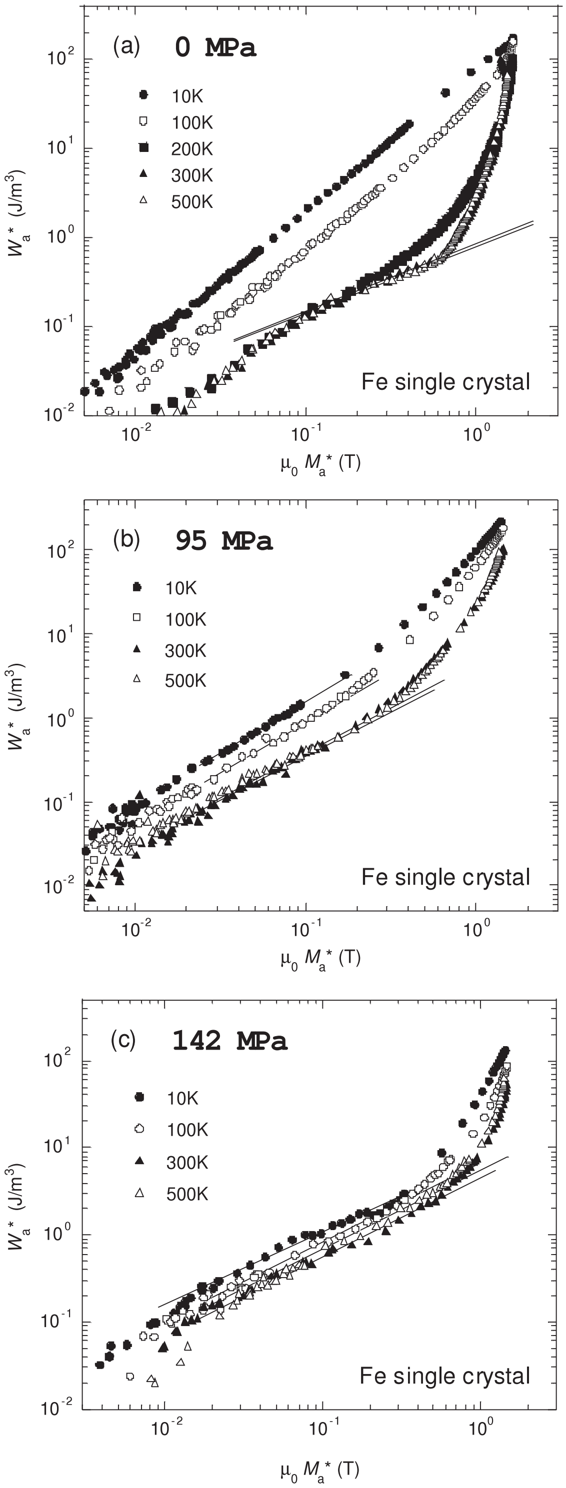

Figure 3. Dependence of the minor-loop stored energy W_{a}^{*} on minor-loop magnetization M_{a}^{*} in Fe single crystal samples at various temperatures, taken before and after plastic deformation by the true stress of 95 and 142 MPa.

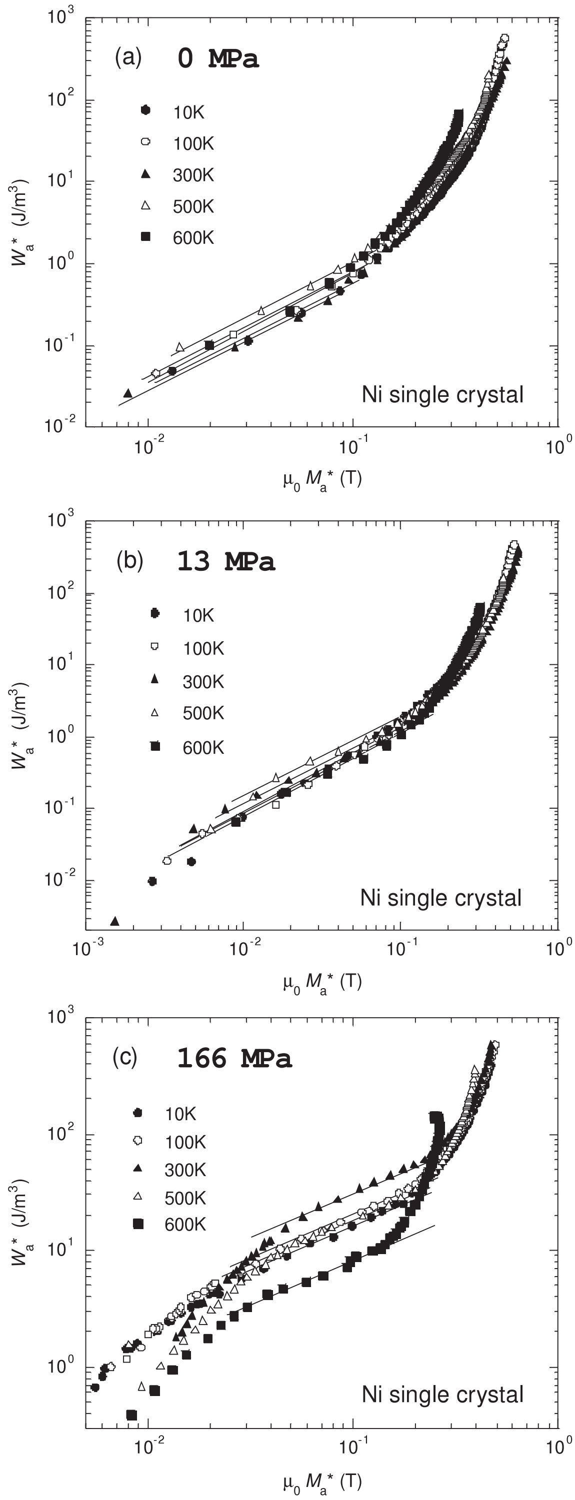

Figure 4. Dependence of the minor-loop stored energy W_{a}^{*} on minor-loop magnetization M_{a}^{*} in Ni single crystal samples at various temperatures, taken before and after plastic deformation by the true stress of 13 and 166 MPa.

where W_{a}^{0} is a coefficient and n_{a} is an exponent of the power law; hereafter we call W_{a}^{0} a minor-loop effluence coefficient. Least-squares fits of the data to (7) using minor loops with $\mu_0 M_{\mathrm{a}}^{*} = \sim 0.08$–$0.4$ T yielded an almost constant value of $n_{\mathrm{a}} = 0.92 \pm 0.08$. Here, the data at low temperatures for undeformed and lightly deformed samples which give a steep slope in the W_{a}^{*}–M_{a}^{*} relation were not used. The origin of the steep slope will be explained later.

Figure 4 shows the relations between W_{a}^{*} and M_{a}^{*} in Ni single crystals before and after plastic deformation in

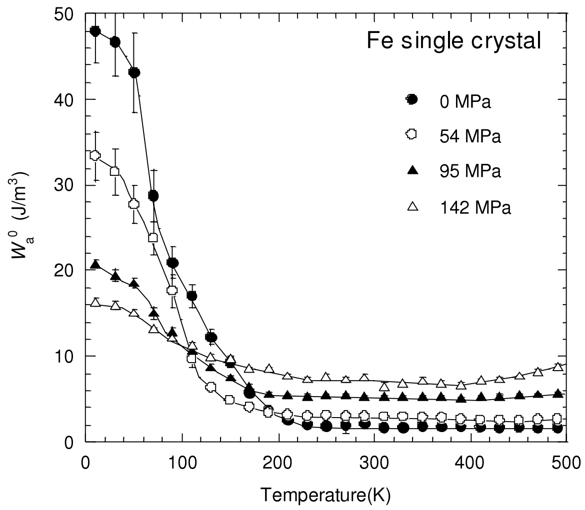

Figure 5. (a) Temperature dependence of W_a^0 in Fe single crystal samples before and after plastic deformation by the true stress of 54, 95 and 142 MPa.

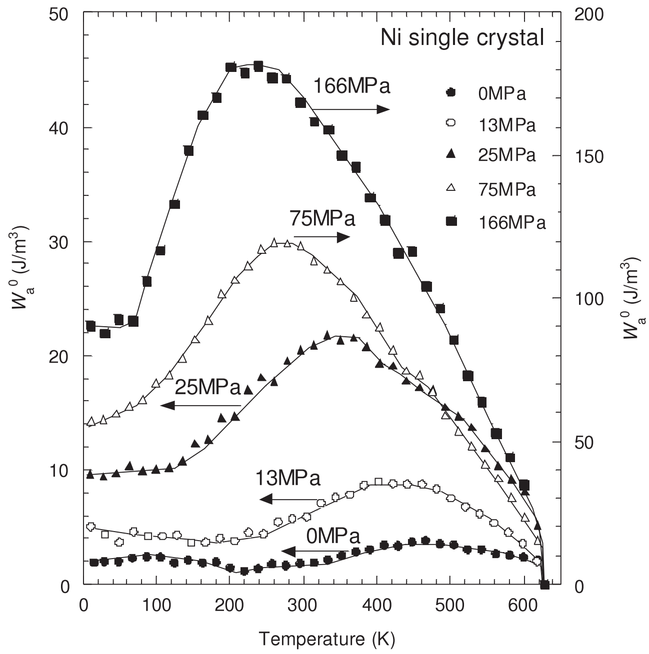

Figure 6. Temperature dependence of W_a^0 in Ni single crystal samples before and after plastic deformation by the true stresses of 13 , 25, 75, and 166 MPa.

compression, taken at various temperatures. As is seen in a linear relation in the double logarithmic plot, we confirmed that the relation of (7) holds true also in Ni single crystals in a limited range of $\mu_0 M_a^* = 0.01$–0.1 T. For samples after plastic deformation, we obtained $n_a = 0.88 \pm 0.08$ independently of the level of compressive deformation, though a slightly higher value of 1.28 ± 0.09 was obtained for the undeformed samples as in the case of Fe single crystals. The relation of (7) was also confirmed in Ni polycrystals with tensile deformation, where $n_a = 0.95 \pm 0.08$ was obtained.

Now, we pay attention to the minor-loop effluence coefficient W_a^0 obtained from the least-squares fits. Figure 5 shows the temperature dependence of W_a^0 in Fe single crystal samples before and after plastic deformation by the true stress of 54, 95, and 142 MPa. Here, we used the value of $n_a = 0.92$ for the fits in all temperature–stress conditions. W_a^0 is almost independent of temperature and increases with the increase of true stress above $T = 200$ K. However, below $T = 200$ K, W_a^0 increases remarkably and its order does not necessarily follow the sequence of true stress. The rate of increase of W_a^0 at low temperatures is the largest in the sample without plastic deformation.

Figure 6 shows the temperature dependence of W_a^0 in Ni single crystal samples after plastic deformation, taken at various true stresses up to 166 MPa. With decreasing temperature below its Curie temperature of 628 K, W_a^0 increases, takes a maximum, and then decreases. The maximum point moves toward a lower temperature with the increase of true stress. The dependence of W_a^0 on temperature and stress is similar to that of coercive forces in Ni single crystals studied previously [10, 12] and may reflect the temperature dependence of magneto-striction and magneto-crystalline anisotropy of Ni.

The minor-loop effluence coefficient W_a^0 is sensitive to plastic deformation, as shown in figures 5 and 6 and reflects the interaction between domain walls and dislocations. Generally, the applied stress τ is a function of the dislocation density ρ,

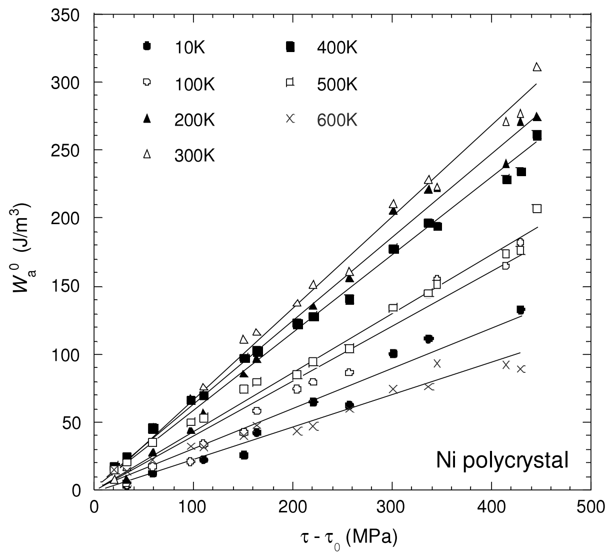

Figure 7. Stress dependence of W_a^0 in Ni polycrystals, taken at various temperatures. τ_0 is yield stress.

i.e. in the first stage of the stress–strain curve, [10]

$$\rho \propto (\tau - \tau_0), \tag{8}$$

where τ_0 is the critical shear stress. In the second stage, the total dislocation density increases with plastic deformation as

$$\rho \propto \tau^2. \tag{9}$$

Figure 7 shows the stress τ dependence of W_a^0 in Ni polycrystals, taken at $T = 10$, 100, 200, 300, 400, 500, and 600 K. W_a^0 increases in proportion to $(\tau - \tau_0)$ at all measuring

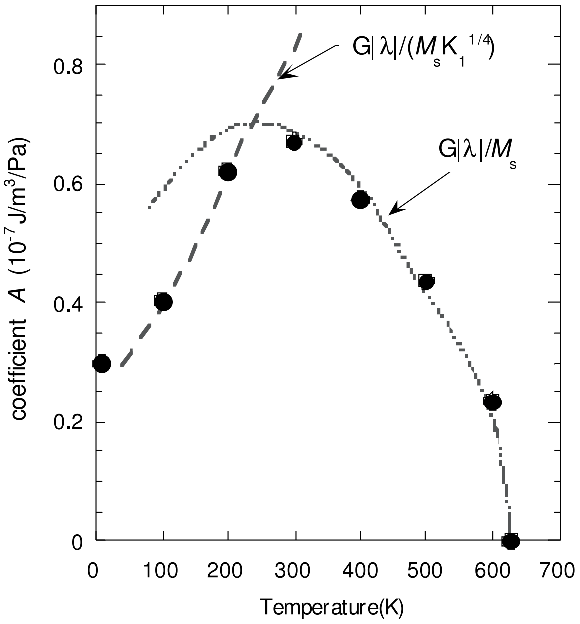

Figure 8. Temperature dependence of coefficient A for Ni polycrystals. The dashed and dotted lines denote calculated curves obtained from (13)–(15), respectively.

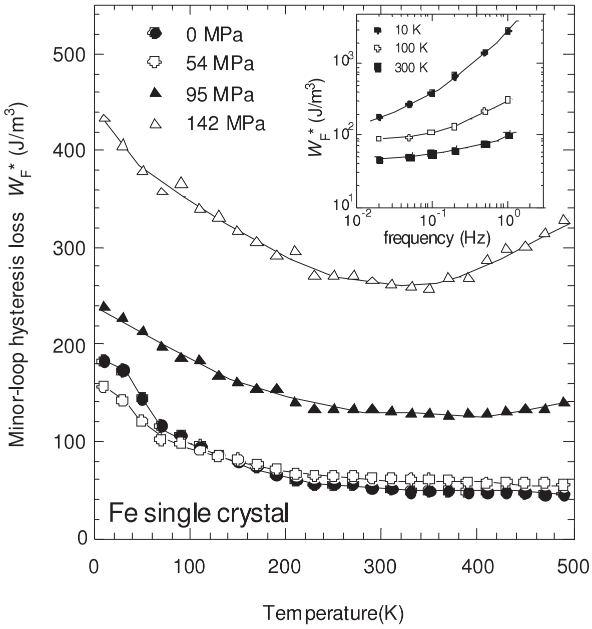

Figure 9. Temperature dependence of minor-loop hysteresis loss W_F^* at $\mu_0 M_a^* = 1.0$ T in Fe single crystals before and after plastic deformation. The inset shows the frequency dependence of W_F^* at $\mu_0 M_a^* = 1.0$ T, taken at 10, 100, and 300 K in the undeformed Fe single crystal sample.

temperatures and can be fitted to a simple linear function, i.e.

$$W_a^0 = A(\tau - \tau_0), \tag{10}$$

where A is a coefficient. Figure 8 shows the temperature dependence of coefficient A in Ni polycrystals. The coefficient A has a maximum near room temperature and becomes zero at the Curie temperature of Ni.

Minor-loop effluence coefficient W_a^0 increases remarkably at low temperatures in the Fe sample without plastic deformation. We have investigated the cause of increasing W_a^0 below $T = 200$ K. The minor-loop coefficient W_a^0 is a magnetic property found in the present study and is unfamiliar to us. We have investigated the temperature dependence of minor-loop hysteresis loss W_F^* instead of W_a^0. Figure 9 shows the temperature dependence of W_F^* at $\mu_0 M_a^* = 1$ T in Fe single crystal deformed plastically. We do not admit any large change above $T = 200$ K in W_F^* but it increases remarkably below $T = 200$ K in all samples with and without plastic deformation. Since the electrical conductivity increases at lower temperatures, eddy current effects may occur in the magnetization process below $T = 200$ K in particular. In fact, the frequency dependence of W_F^* at $\mu_0 M_a^* = 1$ T revealed a remarkable increase of W_F^* with frequency at low temperatures, as shown in the inset in figure 9, indicating the importance of eddy current effects on W_a^0 at low temperatures.

4. Discussion

We found the new scaling rule of (7) in the analysis of the family set of minor hysteresis loops. Minor-loop stored energy W_a^* is a different parameter from minor-loop hysteresis loss W_F^* and minor-loop remanence work W_R^* in the notion of energy. W_F^* is the dispersion energy in one cycle of minor hysteresis loops and W_R^* is the externally added work for domain walls to displace in order to make the magnetization zero. W_a^* is the energy dissipated from the free energy stored by the applied field of H_a. The relation (7) is independent of the relations (1) and (2). We confirmed this scaling rule in single crystals both of Fe and Ni, and Ni polycrystals with and without plastic deformations in the wide temperature range from 10 to 600 K. The exponent n_a is about 1.0 and almost independent of the kind of ferromagnet, temperature, and applied stress, except for low temperature data in undeformed and lightly deformed samples; $n_a = 0.92 \pm 0.08, 0.88 \pm 0.08$, and 0.95 ± 0.08 in Fe and Ni single crystals, and Ni polycrystals, respectively. The generality of (7) was experimentally proved.

W_a^0 is proportional to the true stress τ or to the dislocation density represented by (10). If the behavior of W_a^0 predominantly results from irreversible movement of the 180° domain wall due to dislocations, the coefficient A would be given, as in the case of H_c [10], by

$$A \propto (G|\lambda|\sqrt{\delta d})/M_s. \tag{11}$$

Here δ, G, and λ are the domain wall thickness, the shear modulus, and the magneto-striction constant, respectively. d is the mean length of dislocations by which dislocations run parallel to the domain walls. The 180° domain wall thickness is written as

$$\delta = \pi\sqrt{\frac{JM_s^2}{K_1 a}}. \tag{12}$$

Here J is the exchange integral and a is a lattice constant. K_1 is a constant of magneto-crystalline anisotropy. If the interaction length d is constant and independent of temperature, A is given by

$$A \propto \frac{G|\lambda|}{M_s K_1^{1/4}}, \quad (13)$$

whereas

$$A \propto \frac{G|\lambda|}{M_s K_1^{1/2}}, \quad (14)$$

when d is proportional to $\delta^{1/2}$.

On the other hand, when stress anisotropy is large and the magnetization change is mainly due to the domain rotation against stress anisotropy, A would be expressed [10] by

$$A \propto \frac{G|\lambda|}{M_s}. \quad (15)$$

From the calculation of the coefficient A using published data of constants of magneto-striction [13] and magneto-crystalline anisotropy [14, 15], we found that at temperatures below a maximum of A, (13) is well fitted to the observed data, while (15) explains well the high temperature data, as shown in figure 8. This fitting result is consistent with that of H_c [16, 17] and indicates that the coefficient A and therefore W_a^0 reflect the strength of magneto-elastic interaction between magnetic moments and lattice defects.

Next, we remark on the anomalous increase of W_a^0 at low temperatures observed in Fe single crystals. As shown in figure 5, W_a^0 for undeformed and lightly deformed samples increases remarkably below $T = 200$ K and W_a^0 does not show a systematic change against stress below $T = 200$ K. Taking account of an increase of the electrical conductivity with decreasing temperature, the increase below 200 K can be attributed to the appearance of the eddy current effect. This effect is further enhanced in the samples with low density of dislocations where the mobility of domain walls is high [18]. The detailed investigation of the eddy current effect in samples with extremely low dislocation density is now in progress and will appear elsewhere.

The present study showed that the minor-loop effluence coefficient W_a^0 is sensitive to dislocations and contains information about the microstructure of ferromagnetic materials. W_a^0 is independent of amplitude H_a as well as magnetic field H in the second stage of the magnetization process. W_a^0 is the same physical property as H_c and W in the major loop. We have an advantage in the measurement of W_a^0 compared with H_c and W, because W_a^0 can be obtained with a low measurement field. Further, we need only half of the minor loops with $H \geqslant 0$ for W_a^0, yielding shorter measurement time than for other coefficients which require full minor loops. These advantages are important in the application of non-destructive evaluation and material characterization.

5. Conclusion

The 180° domain wall displacement has been experimentally examined by the minor hysteresis loops in single crystals of Fe and Ni, and Ni polycrystals, deformed plastically. We can say conclusively the following experimental facts:

(i) A simple scaling power-law relation is obtained experimentally between minor-loop stored energy W_a^* and minor-loop magnetization M_a^*. For deformed samples, the exponent n_a is about 1.0, being independent of the kind of ferromagnet, applied stress, and temperature.

(ii) The minor-loop effluence coefficient W_a^0 is sensitive to dislocations and there exists a simple linear relation between W_a^0 and applied stress.

(iii) For undeformed and lightly deformed Fe single crystals, a remarkable increase of W_a^0 below $T = 200$ K was observed. This is due to the appearance of the eddy current effect, associated with the high conductivity at low temperatures and the high mobility of domain walls.

Acknowledgments

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