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Magnetic hysteresis minor loops in Fe single crystal

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Abstract

Magnetic hysteresis minor loops were measured with step by step increase of the magnetic field amplitude, H_a, in plastically deformed Fe single crystal. In order to analyse minor loops in connection with the lattice defects, we defined some magnetic parameters, such as the pseudo-coercive force, H_c^*, and the differential susceptibility under the pseudo-coercive force χ_H^*. H_c^*, for instance, is the magnetic field where the magnetization becomes zero in minor loops. In this work, we found the relationship $2H_c^* = H_a$ to hold over a fairly wide H_a range. These parameters are important for representing Bloch wall displacement and the potential energy. The parameters are remarkably sensitive to lattice defects in the field range below the coercive force H_c. The relation of $1/\chi_H^*$ and H_c^* shows better sensitivity—90 times higher than that for H_c—for revealing information on dislocations. In the minor loop measurement, for getting full information on dislocations it is sufficient to have $H_a = 400$ A m^{-1}.

Magnetism of ferromagnetic materials has been studied experimentally by the use of hysteresis loops for a long time and much information about domain structures, domain wall movements and the rotation of magnetic moments has been obtained. The hysteresis loops are very sensitive to the kinds of material and lattice defects such as dislocations, grain boundaries, impurity atoms, precipitations, vacancies and interstitial atoms. The relationship between magnetism and dislocations was studied by the Stuttgart group at the Max Planck Institute three decades ago [1–5]. The structure sensitive magnetic properties are represented by the dislocation density, ρ, and its distribution; the coercive force, H_c, increases in proportion to the square root of ρ and the initial susceptibility decreases with the inverse of the square root of ρ. The relationship is experimentally confirmed in Ni, Co and Fe single crystals [4].

The hysteresis minor loops have not been studied as intensively as the major loop. The hysteresis major loop is a universal relation, but the minor loops depend on the magnetic field amplitude, H_a. In minor loops, the magnetic properties depend on H_a and are difficult to give physical meaning; therefore they failed to attract the interest of scientists. Few data on

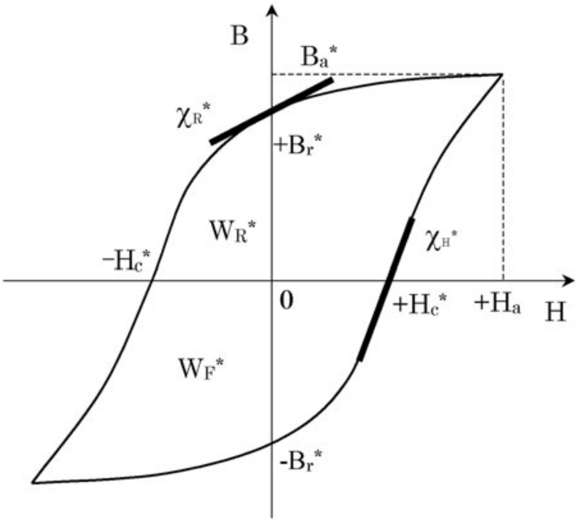

Figure 1. The pseudo-coercive force H_c^*, the susceptibility at the pseudo-coercive force χ_H^*, the magnetic field amplitude H_a, the magnetization B_a^* at H_a, the pseudo-hysteresis loss W_F^*, the pseudo-remanence B_R^*, the pseudo-remanence work W_R^* and the susceptibility at B_R^*, χ_R^*, in a minor loop.

Table 1. Symbols and names for the pseudo-magnetic parameters defined in minor loops.

Symbol	Name
H_a	Magnetic field amplitude
H_c^*	Pseudo-coercive force
χ_H^*	Differential susceptibility at the pseudo-coercive force
B_a^*	Magnetization at H_a
W_F^*	Pseudo-hysteresis loss
W_R^*	Pseudo-remanence work
B_R^*	Pseudo-remanence
χ_R^*	Differential susceptibility at the pseudo-remanence
R^*	Resistance of the Bloch wall at H_c^*, $R^* = 1/\chi_H^*$

minor loops of ferromagnetic materials can be found in the literature [6–10]. We found a new method for analysing the minor loops. This method can be used to scrutinize the Bloch wall potential, and detailed information on the Bloch wall potential that consists of lattice defects can be obtained. It has advantages over the traditional analyses of major hysteresis loops: the Barkhausen noise method, the magneto-acoustic emission method and analysis of the Preisach function. The purpose of the present study is to clarify the magnetic properties due to minor loops on the supposition that all domain walls receive a force from the same potential; the potential is the average over the individual ones.

The minor loops are characterized by H_a, though the major loop is independent of H_a. In order to obtain magnetic information related to defects in materials from minor loops, some new magnetic parameters have been defined and these are shown in figure 1; their symbols and names are listed in table 1. These parameters depend on H_a.

The new parameters agree with the corresponding parameters in the major loop when the applied field is large enough to saturate the magnetization. For instance, H_c^* has a similar character to the coercive force. In this paper two parameters, H_c^* and χ_H^*, are studied in the Fe single crystal with tensile deformation and the physical meaning is discussed according to the experimental data. Both H_c^* and χ_H^* contain important information about the potential energy of domain wall movement.

We prepared a sheet of Fe single crystal 1 mm thick with purity 99.99% and a surface plane (100), provided by courtesy of Dr Tomoyuki Takeuchi. During the manufacturing process it was annealed between 860 and 890 °C; this was followed by water quenching and tempering between 650 and 665 °C for 139 min. Tensile test samples along the crystal direction of $\langle 100\rangle$ were prepared by wire cutting from the sheet. Each sample was tensile deformed at room temperature with an Instron testing machine. The strain rate was 1.5% $\min^{-1}$. The true stresses applied to each sample were 54, 95 and 135 MPa, respectively. The true stress of 54 MPa is just above the yielding point, and the strain is only 0.7%; therefore, most magnetic properties of this sample are very close to those of the undeformed sample. Picture frame shape samples with surfaces and directions of $\{100\}$ and $\langle 100\rangle$ were cut by wire cutting for the magnetic measurements. Minor loops were measured by using a fluxmeter at room temperature. The magnetizing and detecting coils on these samples had 80 and 100 turns, respectively. It was ascertained that the results are independent of the number of turns in both coils if they are between 50 and 150 turns.

The hysteresis minor loops were measured from 0 to 800 A m^{-1} with incrementation of H_a by 8 A m^{-1}. The new pseudo-magnetic properties were calculated from each minor loop by a computer program for minor loop analysis produced by the authors. Figures 2(a) and (b) show the relation between H_c^* and H_a for the Fe single crystal. The relation between H_c^* and H_a depends on the applied stress remarkably above $H_a = 40$ A m^{-1}. Below $H_a = 24$ A m^{-1} the values of H_c^* without plastic deformation become larger than those of deformed samples, as shown in figure 2(b). H_c^* is saturated above 320 A m^{-1} before plastic deformation. H_c^* increases linearly within a limited range of H_a as shown in figure 2(a). The slope of the linear relation is about $1/2$; this means that H_c^* is half of H_a. We have proved experimentally that this relationship is also realized in other materials, such as Fe polycrystalline samples and low carbon steels. Figure 3 shows the H_a dependence of χ_H^* before and after tensile deformation. The reciprocal of χ_H^* decreases rapidly with increase of H_a in minor loops.

The reciprocal susceptibility indicates the resistance of the domain wall motion. The domain wall displaces easily with application of H_a. Initially the domain wall is located at the minimum state of potential energy and is difficult to displace near the minimum state with an applied magnetic field. For large H_a the domain wall moves over a wide range. The slope of the potential becomes steep with increase of ρ and the mobility of the domain walls decreases.

It is known that in the magnetism of ferromagnetic materials, the Bloch wall moves reversibly in the first stage and the irreversible displacement of the Bloch wall contributes in the second stage. The Bloch wall receives a force from dislocations during its displacement within a limited range by the applied field below H_a. The force is caused by the potential. The Bloch wall stays at a stable position of the potential after the applied field is removed and it climbs over a peak of potential and is displaced over a large distance by the inverse field, H_c^*. The displacement of the domain wall governs χ_H^*.

The domain wall is located at the minimum potential position in the demagnetized state and displaces along the harmonic potential direction, E_w, under the small field amplitude, H_a:

$$E_w = \tfrac{1}{2}\alpha x^2, \tag{1}$$

where α is a coefficient and x is the displacement of the domain wall. The harmonic part of the potential changes by plastic deformation; an example is $\alpha \sim \rho^{1/2}$. The length of the Bloch wall displacement gives the magnetization and the initial susceptibility, χ_i, can be obtained in

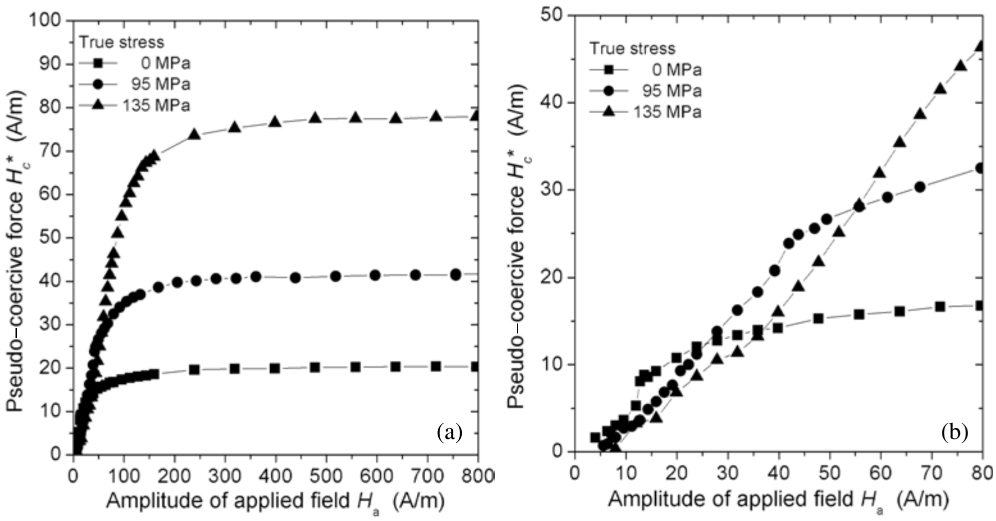

Figure 2. (a), (b) The magnetic field amplitude, H_a, dependence of the pseudo-coercive force, H_c^*, in Fe single crystal with tensile deformation.

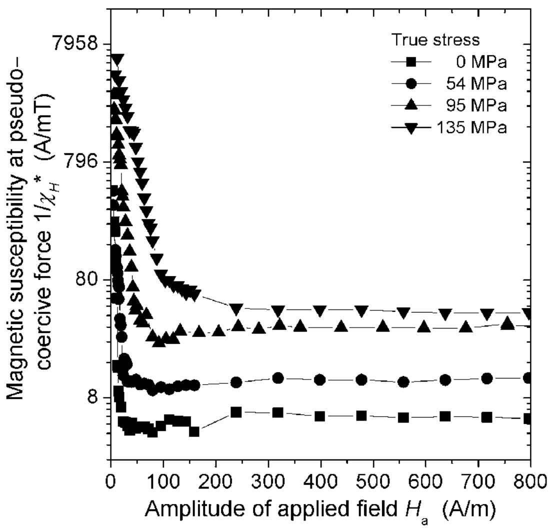

Figure 3. The magnetic field amplitude, H_a, dependence of the magnetic susceptibility, χ_H^*, at the pseudo-coercive force in Fe single crystal with tensile deformation.

the reversible region from

$$\chi_i = \frac{4M_s^2 F \cos\phi}{\alpha L}, \qquad (2)$$

where M_s is the spontaneous magnetization, F is the area of the 180° wall, L is the average distance of the walls and ϕ is the angle between the external magnetic field and the magnetic moments. The initial susceptibility decreases with increase of the dislocation density: $\chi_i \sim \rho^{-1/2}$ [5]. The initial susceptibility can be obtained experimentally from the relation B_a^* and H_a from the minor loop, if H_a is small enough.

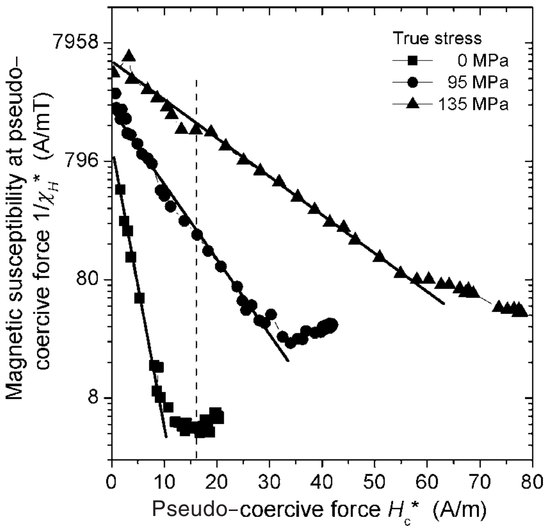

Figure 4. The relation between H_c^* and $1/\chi_H^*$ in Fe single crystal with tensile deformation.

The ⟨100⟩ is the direction of easy magnetization in Fe metal. The highest mobility of Bloch walls is attained with ⟨100⟩ magnetization in the single crystal. There is a potential energy of the domain wall even before plastic deformation. But the potential is much smaller, so the domain wall attains the highest potential peak for $H_a = 40$ A m^{-1} and sweeps out of the crystal for $H_a = 80$ A m^{-1}; this has been examined on the basis of the relationship of W_F^* and H_a.

In the irreversible stage, H_c^* and χ_H^* increase remarkably with increasing H_a; χ_H^* represents the mobility of the Bloch wall. The potential energy depends on the dislocations; the H_a dependences of H_c^* and χ_H^* change remarkably upon plastic deformation. H_c^* increases in proportion to H_a and its rate of increase is about 1/2 in the plastically deformed samples. H_c^* in the irreversible stage corresponds to the height of the potential peak over which the domain wall cannot climb when the applied field is removed. The height of the potential which the domain wall attains for a given value of H_a is nearly the same in the plastically deformed samples and is independent of the strength of the stress. The displacement and the number of domain walls become largest and their movement becomes the most pronounced at H_c^*, which is half H_a. The relationship of $2H_c^* = H_a$ may be caused by the frictional energy of the domain walls. The detailed mechanism is currently under consideration.

The amplitude of the magnetic field is an external parameter and is arbitrary. It is convenient to remove H_a from the representation of domain wall movement. Figure 4 shows the H_c^* dependence of $1/\chi_H^*$. We found a simple relation between H_c^* and $1/\chi_H^*$, given as equation (3):

$$R^* = \frac{1}{\chi_H^*} = \exp(a - bH_c^*) \tag{3}$$

where R^* is the resistance of the Bloch wall movement. a and b are constants; b is very sensitive to dislocations—its value is changed from 40 to 6 by applying a true tensile stress of 135 MPa.

The coercive force is changed from 19.1 to 79.6 A m^{-1} by tensile deformation under 135 MPa and its increment is by a factor of four. The resistance R^* at $H_c^* = 16$ A m^{-1} changes from 4.5 to 1615 A mT^{-1} as shown in figure 4. The increment of R^* is by a factor of

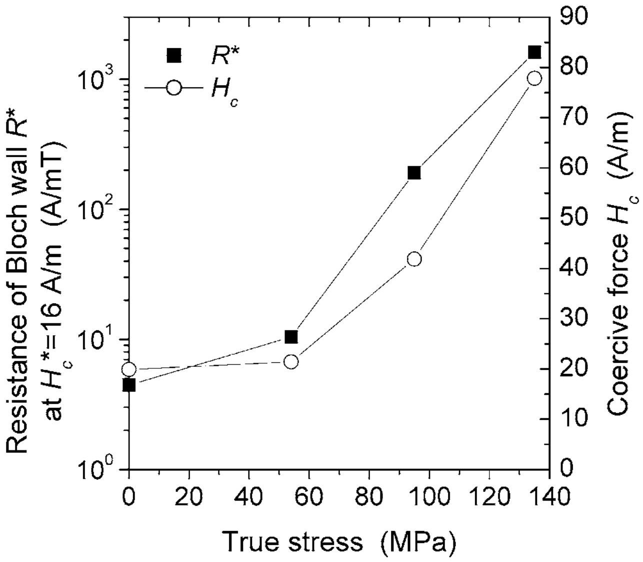

Figure 5. The dependences of R^* and H_c on the true stress.

360. The sensitivity due to the H_c^*–$1/\chi_H^*$ relation is 90 times that for H_c. The dependences of R^* and H_c on the true stress show similar forms, as shown in figure 5.

A detailed analysis of minor loops is in progress, using the other pseudo-magnetic parameters [11].

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