Vol. A52 (1977) *ACTA PHYSICA POLONICA* No 5

THE ANISOTROPY OF CHANGES OF THE SUSCEPTIBILITY OF DEFORMED Ni MONOCRYSTALS*

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(Received December 6, 1976)

The paper discusses the results of investigations concerning anisotropic changes of the reversible susceptibility in approach to saturation of Ni-monocrystals deformed in the direction that favours a multisystem slip. An analysis was carried out in order to determine the influence of stresses caused by the dislocations of both the primary and secondary slip systems as well as the short-range stresses on the anisotropy of susceptibility changes which is to be found experimentally.

1. Introduction

In studies of the dislocational structure of monocrystals in various stages of deformation, the method of electron microscopy has found wide applications. From among the intermediate methods which are used in the case of ferromagnetic materials, magnetic methods have become most important. Their development in large magnetic fields had been originated by Brown's investigations [1, 2] of the interaction between the internal stresses and spontaneous magnetization, due to magnetostriction. Seeger and Kronmüller continuing Brown's investigations, devoted their attention to theoretical studies of the influence of stresses arising from dislocations on the reversible susceptibility in approach to saturation [3, 4]. The influence of the distribution of dislocations on the susceptibility was discussed in paper [5]. Experimental investigations [6, 7] dealt with the influence of primary slip system dislocations on the anisotropy of susceptibility changes in the case of Ni crystals oriented for easy glide.

In this paper the results of studies concerning the anisotropic changes of susceptibility in the case of deformed crystals oriented for hard glide [8] have been presented. The influence of the edge dislocations of the secondary slip systems have been taken into account.

* Supported by the Institute of Physics of the Polish Academy of Sciences.

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2. The anisotropy of susceptibility changes in approach to saturation. Theory and experimental

In large magnetic fields, the magnetization of undefected monocrystals is homogeneous. Changes of the magnetization component J_H, parallel to the direction of field H, result from the spontaneous magnetization rotation occurring uniformly within the whole volume of the sample. In perfect crystals the changes of the component J_H are controlled by the energy of crystalline anisotropy E_k and the magnetostatic interaction energy E_H. When dislocations occur however, the directions of magnetization are arranged non-uniformly, as they are influenced by magnetoelastic coupling energy E_m. The final distribution of the directions of the spontaneous magnetization is the result of a compromise of different energies in order to minimize the free energy of crystals.

The quantitative conception of the effect of dislocations on the magnetization distribution was solved for the first time by Seeger and Kronmüller [3, 4]. As the starting point a set up of micromagnetic equations was used

$$\Delta\gamma_i - \frac{\partial u}{\partial x_i} - \frac{HJ}{2A}\gamma_i - \frac{1}{2A}\frac{\partial E_k}{\partial \gamma_i} - \frac{1}{2A}\frac{\partial E_m}{\partial \gamma_i} = 0, \quad i = 1,3, \tag{1}$$

which holds true when the direction of the magnetizating field coincides with the direction of the x_2 axis. The succesive terms describe in turn the influence of the exchange energy, stray-field energy, magnetostatic interaction energy, magnetocrystalline energy and magnetoelastic coupling energy, on the value of the direction cosines $\gamma_i(\vec{r})$ of the spontaneous magnetization, measured relatively to the x_i axis. J is the saturation magnetization, A — exchange energy constant. The magnetization distribution around the dislocations is non-uniform. The magnetization component J_2, parallel to the field direction in point $\vec{r}$ may be written as follows:

$$J_2 = J\gamma_2(\vec{r}).$$

Its value is dependent on both on the type of dislocation and on the reciprocal arrangement of the direction of field $\vec{H}$ relatively to the dislocation line. The susceptibility χ measured along the direction x_2 is defined by the expression

$$\chi = \frac{dJ_2}{dH}; \quad J_2 = J\langle\gamma_2\rangle,$$

where $\langle\gamma_2\rangle$ denotes the average value of γ_2 throughout the whole volume of the sample. This way of finding the value of $\langle\gamma_2\rangle$ from Eq. (1), as proposed in [4], does not take into account the energy of the stray field. As has been shown in [9] however, in the case of the influence of that energy at dislocation densities of about 10^8 cm^{-2} is negligible for small fields, of about 3 kOe.

As far as the picture of the real dislocation structure is concerned, it is most expressively reflected in the directional changes of the magnetic susceptibility. That problem has been dealt with in Gessinger's theoretical [7] and experimental [6] studies. They were a continuation of Seeger's and Kronmüller's investigations [3, 4] and made it possible to determine the influence of a variety of types of dislocations on the anisotropic changes of susceptibility within the {111} plane, which are connected with dislocations lying in this plane. For dislocations of the i-type Gessinger obtained the following expressions:

$$\chi_i(\varphi, H) = \chi_0^i + \chi_2^i \sin 2(\varphi + \varphi_2) + \chi_4^i \sin 4(\varphi + \varphi_4), \tag{2}$$

where φ is the angle lying in the {111} plane, between the direction of the field and the reference direction both lying in this plane.

The anisotropic changes of the susceptibility are described then with Fourier's second and fourth components. For a wide field range, $\chi_{2,4}^i \sim H^{-3}$, and a contribution to the anisotropy of susceptibility $\chi(\varphi, H)$ resulting from i-type dislocations and N_i densities this can be expressed as follows:

$$\chi(\varphi, H) = \sum_i BC_0^i(\varphi) N_i H^{-3}. \tag{3}$$

The anisotropy is described by $C_0^i(\varphi)$ factors, which on the $(11\bar{1})$ plane have got the following form:
for edge dislocations; $\vec{l} \,||\, [\bar{1}21]$, $\vec{b} \,||\, [101]$;

$$C_0 = 10^{-8}[26.681 + 17.332 \sin 2(\varphi + 45°) + 3.811 \sin 4(\varphi - 22.5°)], \tag{4a}$$

for screw dislocations; $\vec{l} \,||\, [101]$, $\vec{b} \,||\, [101]$;

$$C_0 = 10^{-8}[31.368 + 19.160 \sin 2(\varphi + 45°) + 12.233 \sin 4(\varphi + 22.5°)], \tag{4b}$$

for 60° dislocations; $\vec{l} \,||\, [011]$, $\vec{b} \,||\, [101]$;

$$C_0 = 10^{-8}[27.866 + 15.298 \sin 2(\varphi + 100.5°) + 7.340 \sin 4(\varphi - 10°)], \tag{4c}$$

for 120° dislocations; $\vec{l} \,||\, [110]$, $\vec{b} \,||\, [101]$;

$$C_0 = 10^{-8}[27.866 + 15.298 \sin 2(\varphi - 10.5°) + 7.340 \sin 4(\varphi - 35°)], \tag{4d}$$

for L. C. dislocations; $\vec{l} \,||\, [011]$, $b \,||\, [01\bar{1}]$;

$$C_0 = 10^{-8}[19.702 + 16.688 \sin 2(\varphi + 75°) + 3.065 \sin 4(\varphi + 7.5°)]. \tag{4e}$$

B is a function of material constants.

In this paper there has been found an analytical form of $C_0^i(\varphi)$ for edge dislocations across the $(11\bar{1})$ plane. As the components $C_0^i(\varphi)$ were found analogously to the dislocations lying in $(11\bar{1})$ plane, the final results of calculations are as follows:
for edge dislocations; $\vec{l} \,||\, [\bar{1}12]$, $\vec{b} \,||\, [110]$;

$$C_0 = 10^{-8}[4.673 + 1.254 \sin 2(\varphi + 85°) + 1.178 \sin 4(\varphi - 13.5°)], \tag{4f}$$

for edge dislocations; $\vec{l} \,||\, [1\bar{2}1]$, $\vec{b} \,||\, [10\bar{1}]$;

$$C_0 = 10^{-8}[4.673 + 1.254 \sin 2(\varphi + 25°) + 1.178 \sin 4(\varphi + 16.5°)]. \tag{4g}$$

Experimental investigations of anisotropic susceptibility changes realised in the course of this work were carried out by means of a magnetometer with rotating sample. The samples used for studies were disc-shaped with a diameter of 9.3 mm and a hight of 2.5 mm. They were cut off parallel to the $(11\bar{1})$ plane of primary system slip [8]. Anisotropic changes of susceptibility have been found on the basis of Fourier's analysis of the voltage induced upon the measuring coil of the magnetometer. If the direction of the lines of the magnetic field remains parallel to the sample surface during its rotation, the voltage induced upon the measuring coil can be expressed [6] as below

$$U(\varphi, H) = -c\,\frac{dJ_2(\varphi, H)}{dH} = U_2(H)\cos 2(\varphi+\varphi_2)+U_4(H)\cos 4(\varphi+\varphi_4). \tag{5}$$

The constant c describing the coil-sample system was in these studies equal to 79830 $cm^2\,s^{-1}$. For samples of the $(11\bar{1})$ orientation the second and fourth voltage components result only from the dislocations, as the contribution resulting from the magnetocrystalline anisotropy is just equal to zero [7]. Fourier's analysis was carried out with a homodyne nanovoltmeter. Later on there are presented the only result obtained for the fourth voltage component. The results of the second component, however, were affected by the noncentricity of the sample position relative to the rotation axis of the magnetometer, which led to false results concerning the contribution of dislocations. Measurements of both $U_4(H)$ and φ_4 make it possible to calculate the anisotropy of susceptibility changes according to [6]:

$$\chi_4(H) = \frac{1}{4c}\left|\frac{dU_4(H)}{dH}\right|, \tag{6}$$

$$\chi_4(\varphi, H) = \chi_4(H)\sin 4(\varphi+\varphi_4). \tag{7}$$

3. *Results and discussion*

Experimental studies of the anisotropic changes of the component of susceptibility were carried out for four samples plastically deformed by the tension in the direction favouring the multisystem slip. The work-hardening characteristics of deformed crystals have been presented in Fig. 1. The deformation of less deformed samples $\tau = 1.8$ kG/mm² was carried out at temperatures of 200, 300 and 400 K; and for more deformed ones $\tau = 5.3$ kG/mm² at the temperature 200 K. The results of investigations concerning the dislocational structure have been presented in paper [8].

Fourier's analysis of voltages which are induced in the measuring coil of the magnetometer was carried out in a magnetic field, which changed over the range from 2.5 to 5.5 kOe. It had been confirmed for all the samples that the phases φ_4 do not change their values within this range of field. In the case of fields smaller than 2.5 kOe, however, the phase values are alternately changed. This might be connected with the domain structure, occurring at the sample edges of weak magnetic fields. The established values of the phase φ_4 have been listed in Table I and the dependence of U_4 on the field in Fig. 2. According to (3) and (6) a power dependence should be expected for $U_4(H)$. The dependence $U_4(H)$ is illustrated in Fig. 3 in a double-logarythmic representation. The measuring points are distributed along straight lines, which means that the dependency $U_4(H)$ could be

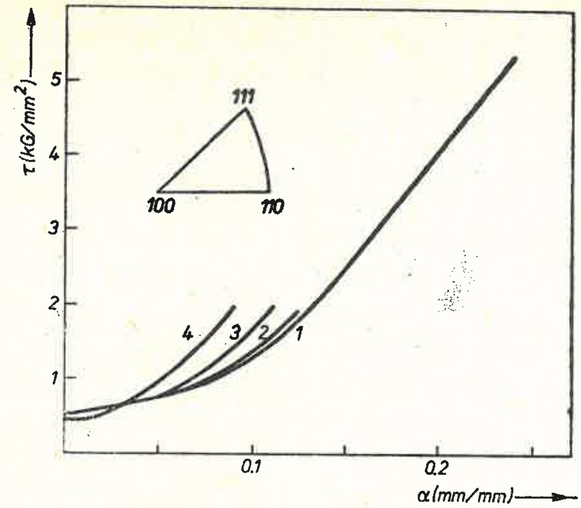

Fig. 1. Work-hardening curves of Ni-monocrystals. *1* — for a sample much deformed at 200 K, *2*, *3*, *4* — for samples less deformed at 200, 300 and 400 K respectively. The stereographic projection displays the direction of the strain activity at the begining of the deformation process

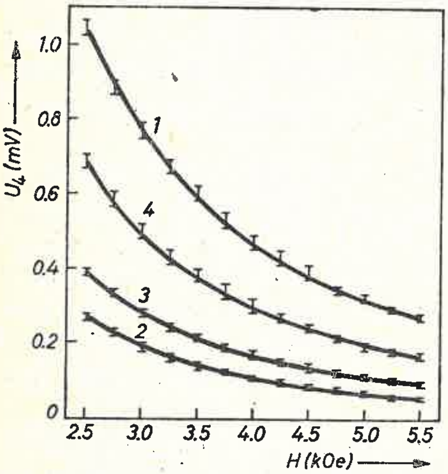

Fig. 2

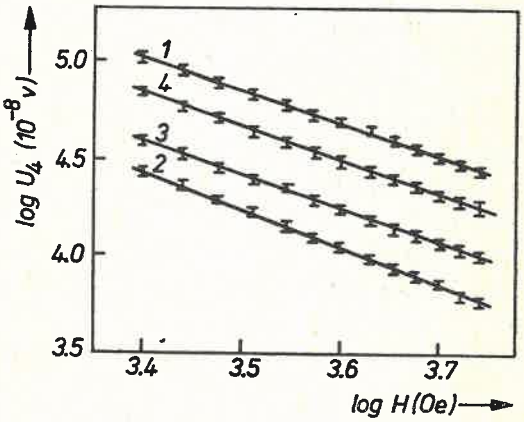

Fig. 3

Fig. 2. Dependence the amplitude of the voltage of U_4 induced in the measuring coil on the field H (the samples have been designated like in Fig. 1)

Fig. 3. Dependence of the amplitude of the voltage U_4 on the field H plotted in the double-logarythmic representation. The values of the parameters U_4^0 and n have been listed in Table I

expressed as $U_4 = U_4^0 H^{-n+1}$. The parameters values U_4^0 and n determined by means of the least squares method have been listed in the Table I. The value of the power index of n is smaller than 3 for all the samples and decreases as both the degree of deformation and the deformation temperature increase.

The developed theory of susceptibility for straight-line dislocations, whose tension σ changes with the distance r from the dislocation lines in accordance as r^{-1}, predicts (3) that $\chi(H) \sim H^{-3}$. If we assume that in the deformed crystals there occur also sources short-range tension, it is to be expected that the influence of the field on the change of the magnetization distribution and thus also on the susceptibility will become smaller.

TABLE I

Values of the phases φ_4 of the fourth components of voltage measured in relation to the [101] reference direction and values of the parameters U_4° and n in the expression $U_4(H) = U_4^{\circ}H^{-n+1}$. This expression describes strictly the dependences of $U_4(H)$, determined experimentally, if H denotes a number of Oe

Sampie	1	2	3	4
φ_4	$-15°$	$-22°$	$-21.5°$	$-21°$
U_4°[V]	623	887	491	749
n	2.70	2.92	2.79	2.78

In the deformed crystals the short-range tensions may result both from the dislocation dipoles and loops of small geometrical sizes, for which $\sigma \sim r^{-2}$. Around them the perturbations in the homogeneity of the magnetization distribution occur in small regions and the distribution of the directions of the spontaneous magnetization is controlled mainly by the exchange interaction. The influence of the magnetostatic interaction on the magnetization distribution is in this case smaller than in an environment of straight-line dislocations, around which the perturbation radius is great and the inhomogeneity of the magnetization distribution is relatively small.

Structural investigations of the deformed samples showed [8] an increase in the relative density of both the loops and dislocation dipoles accompanied by an increase of both temperature and deformation degree. Thus the differences in the value of the powers exponent n should be connected with changes in the contribution of the both the loops and dislocation dipoles upon the total dislocation structure. Because, however, the power value n was only a little smaller than 3, the experimentally determined anisotropy of susceptibility is conditioned mainly by the influence of the long-range dislocational stresses.

The estimated influence of the crystalline anisotropy on the amplitude of susceptibility $\chi_4(H)$, resulting from the disorientation of the mosaic blocks of the magnitude of 30′ [8], is negligible. The presence of mosaic blocks tends towards the susceptibility component $\Delta\chi_k$, whose dependence on the field is also of the type; $\Delta\chi_k \sim H^{-3}$. The small value of $\Delta\chi_k$ does not affect the phase value χ_4, determining the susceptibility anisotropy of dislocational origin. Thus taking into account that the effect of short-range tensions and of the disorientation of the mosaic blocks on $\chi_4(\varphi, H)$ is rather small, the determined course of $\chi_4(\varphi, H)$ may be explained mainly by the influence of dislocational stresses. As a criterion for the choice of elements of the dislocational structure creating a real crystal structure there has been assumed a pair of equalities $\varphi_4^{exp} = \varphi_4^{th}$ and $\chi_4^{exp}(H) = \chi_4^{th}(H)$. Because the power exponent $n < 3$, the dislocational structure model was adjusted to the experimental results only for $H = 4$ kOe but not for the whole measuring range. It has been assumed, similary as in paper [7], that the effects of various dislocation types are added linearly, according to (3). For $C_0^i(\varphi)$ the anisotropy has been adopted which is described by the formulas (4a)–(4e) in the case of dislocations within the primary slip-system and by formulas (4f)–(4g) for edge dislocations of secondary slip-systems.

TABLE II

Dislocational structure models motivating the experimentally determined anisotropy $X_4(\varphi, H)$ of the deformed samples

Sample number j	1	2	3	4
dislocation density N_j [cm^{-2}]	5×10^9	6×10^8	7×10^8	8×10^8
dislocation type	relative density of various types dislocations			
⊥ primary $\vec{l} \parallel [\bar{1}21]$	0.31	0.80	0.66	0.60
⊥ secondary $\vec{l} \parallel [\bar{1}12]$	0.20	0.05	0.12	0.15
⊥ secondary $\vec{l} \parallel [1\bar{2}1]$	0.07	0.02	0.02	0.05
⊙ primary $\vec{l} \parallel [101]$	0.04	0.01	0.05	0.01
60°-primary $\vec{l} \parallel [011]$	0.09	0.04	0.04	0.05
120°-primary $\vec{l} \parallel [\bar{1}10]$	0.10	0.05	0.06	0.07
barrier L.C. $\vec{l} \parallel [011]$	0.19	0.03	0.05	0.07
φ_4^{exp}	−15°	−22°	−21.5°	−21°
φ_4^{th}	−15°	−22°	−21.5°	−21°
χ_4^{exp} (4000 Oe) [G × Oe^{-1}]	6.26×10^{-5}	1.64×10^{-5}	2.40×10^{-5}	4.18×10^{-5}
χ_4^{th} (4000 Oe) [G × Oe^{-1}]	6.32×10^{-5}	1.51×10^{-5}	2.42×10^{-5}	4.16×10^{-5}

A good agreement with the experimental data was obtained for models in which the ratio of dislocation densities of various types is similar to that presented in Table II. Such models reflect adequately the dislocational structure [8] investigated by means of electron microscope.

I wish to thank Professor dr W. Żdanowicz for his help.

REFERENCES

[1] W. F. Brown, *Phys. Rev.* **60**, 132 (1941).
[2] W. F. Brown, *Phys. Rev.* **58**, 736 (1940).
[3] A. Seeger, H. Kronmüller, *J. Phys. Chem. Solids* **12**, 298 (1960).
[4] H. Kronmüller, A. Seeger, *J. Phys. Chem. Solids* **18**, 93 (1961).
[5] H. Kronmüller, *Int. J. Nondestr. Test.* **3**, 315 (1972).
[6] H. Gessinger, *Phys. Status Solidi* **38**, 277 (1970).
[7] H. Gessinger, *Phys. Status Solidi* **39**, 581 (1970).
[8] T. Uliasz, *Acta Phys. Pol.* **A51**, 393 (1977).
[9] A. Holz, H. Kronmüller, *Phys. Status Solidi* **28**, 755 (1968).