Measurement of the thickness of 180° domain boundaries in silicon iron from the refraction of cold and thermal neutrons

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Experiments have been carried out on the refraction of cold and thermal neutrons at a system of 180° domain boundaries in a silicon iron crystal. A qualitative difference between the refraction pattern of cold and thermal neutrons has been detected, consisting of the absence of the refraction of cold neutrons in a certain range of grazing angles of the beam at the boundaries. The observed difference of the refraction intensity of neutrons with different wavelengths is direct evidence that the neutron spin rotates as it passes through a Bloch-type domain boundary. The domain-boundary thickness is measured from the magnitude of the effect, and is found to be 179±10 nm.
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1. INTRODUCTION

The domain-boundary thickness in a ferromagnet is one of the fundamental quantities determined by the relationship of the exchange energy and the anisotropy and plays an important role in the domain-structure formation and the magnetization processes of ferromagnets. The comparison of the theoretically predicted domain-boundary thickness and the experimental value in a bulk ferromagnet can be one of the criteria for the adequacy of theoretical representations of the domain structure. Therefore, much attention has been devoted to experimentally determining the domain-boundary thickness. Methods have been developed, based on Lorentz electron microscopy[1,2] and magnetooptic methods[3–5] that make it possible to determine the domain-boundary thickness in thin films and on the surface of ferromagnets. To measure the domain-boundary thickness in a bulk material, a method has been proposed that uses the transmission of a neutron beam through a domain boundary, in which case adiabatic neutron-spin rotation is possible.[6] In principle, this effect manifests itself by changing the polarization vector of the neutron beam or by refracting it. It is virtually impossible to distinguish the beam-polarization variation at the domain boundary, because the contribution of this process is small by comparison with the effect of the volume of the domains. Neutron refraction occurs only at a domain boundary, and therefore it is possible to use neutron-refraction experiments to distinguish the adiabatic spin-rotation effect and to measure the domain-boundary thickness from its value.[7]

Experiments to determine the thickness of a 90° domain boundary in iron were described by Schaerpf and Strothmann.[8] However, the domain-boundary thickness was determined in their paper by comparing the observed intensity of the refracted beams with a value calculated from the domain-structure parameters. They used a simplified model of the volume domain structure, based on a study of the domain boundaries where they emerge from the surface of the sample; however, it is known[9] that the volume domain structure of bulk samples can substantially differ from the surface structure. This prevents the results of Ref. 8 from being conclusive. Reference 10 proposed a method for observing the adiabatic spin-rotation effect from the refraction of neutrons with different wavelengths, in which the differential effect is determined by the domain-boundary thickness and is virtually independent of the domain-structure parameters. The small neutron-wavelength range used in Ref. 10 (0.15–0.23 nm) made it possible to measure the thickness of a 180° domain boundary in iron with an accuracy of about 25%. Cold neutrons can be used to improve the accuracy of the measurement. The purpose of our work was to measure the 180° domain-boundary thickness in iron from the refraction of cold and thermal neutrons. The idea of the experiment, as in Ref. 10, is to measure how the intensity of the unrefracted neutron beam varies for two wavelengths in the same volume of a test crystal with grazing incidence of the beam on a domain boundary.

We should point out that the observation of the adiabatic-flip effect directly confirms the presence of rotation of the magnetic induction vector inside a domain boundary; i.e., it confirms that Bloch walls really exist in a ferromagnet in which the induction vector rotates in the plane of the domain boundary.

2. THEORY

Let us consider the behavior of the spin of a neutron when it passes through a domain boundary, following the results of Ref. 6. We use a very simple model of the domain boundary, in which the magnetic induction vector **B** uniformly rotates by 180° in a layer of thickness δ, with the direction of vector **B** being always parallel to the domain boundary (Fig. 1):

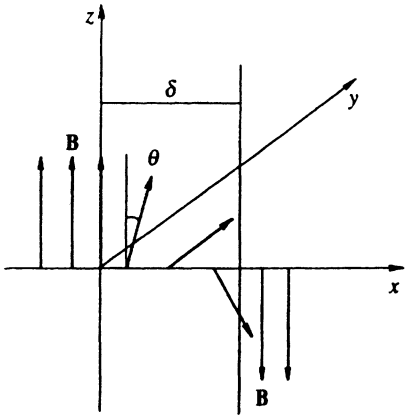

FIG. 1. Diagram of a domain boundary: rotation of the magnetic induction vector **B** by 180° in a plane of thickness δ (θ is the angle between the induction vector and the z axis).

$$B_x=0, \quad B_y=B \sin \theta, \quad B_z=B \cos \theta, \quad \theta=\pi x/\delta. \quad (1)$$

The x axis, with its origin at the entrance surface of the domain boundary, is perpendicular to its plane ($0 \leqslant x \leqslant \delta$), while the z axis is directed along the **B** vector in the first domain. If the neutron moves along the x axis with velocity v, then, in the coordinate system connected with it, it lies in a field that rotates with angular velocity $\omega=\pi v/\delta$:

$$B_x=0, \quad B_y=B \sin \omega t, \quad B_z=B \cos \omega t. \quad (2)$$

At $t=0$, let the components of the averaged angular momentum vector of the neutron equal

$$M_x=M_y=0, \quad M_z=-M_0. \quad (3)$$

The value of **M** as a function of time is a solution of the classical equation

$$\frac{d\mathbf{M}}{dt}=\frac{\mu}{h}\mathbf{M}\times\mathbf{B}, \quad (4)$$

where μ is the magnetic moment of the neutron. Solving the equation for the component of interest to us, M_z, at time $t=\pi/\omega$ gives

$$\frac{M_z}{M_0}=\frac{1}{1+y^2}[y^2+\cos(\pi\sqrt{1+y^2})], \quad (5)$$

where $y=\omega_L/\omega$, and $\omega_L=2\mu B/\hbar$ is the Larmor precession frequency of the neutron spin in field B. Transforming from the magnetic-moment component to the probability P of adiabatic neutron-spin flip, we recall the relation $M_z/M_0=1-2P$, and then

$$P=\frac{1}{1+y^2}\left[y^2+\cos^2\left(\frac{\pi}{2}\sqrt{1+y^2}\right)\right]. \quad (6)$$

For grazing incidence of the neutron on the domain boundary, we can consider only the normal component of its velocity and use $\omega_{\text{eff}}=\omega \sin \alpha$, where α is the grazing angle. If there is scatter σ of the grazing angles because the domain boundary is nonplanar and misoriented and the beam is divergent, the probability is averaged over angle:

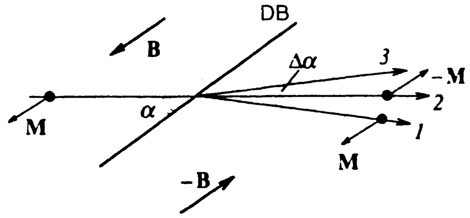

FIG. 2. Refraction of a beam of unpolarized neutrons at a 180° domain boundary (DB): α is the grazing angle of the beam, $\Delta\alpha$ is the refraction angle, **B** and **M** are the magnetic induction and the magnetic moment vectors of a neutron in the two domains, *1* is the refracted beam for the **B**‖**M** polarization component in the first domain, *2* is the beam not refracted as a result of spin flip, *3* is the refracted beam for the opposite neutron-polarization component (the unrefracted beam for this polarization also coincides with beam *2*.

$$P_\sigma(\alpha)=\frac{1}{\sqrt{2\pi}\sigma}\int P(\alpha-x)\exp\left(-\frac{x^2}{2\sigma^2}\right)dx. \quad (7)$$

When a neutron passes through a domain boundary with no spin flip, its potential energy changes by $2\mu B$, and it is refracted by the angle

$$\Delta\alpha=\pm\frac{\mu B}{E}\cot \alpha, \quad (8)$$

where E is the neutron energy, and the $\pm$ signs correspond to the different spin states of the neutron in the first domain. However, if adiabatic neutron-spin flip occurs, the neutron's potential energy does not change, and no refraction occurs. Thus, a beam of unpolarized neutrons passing through a domain boundary separates into two refracted beams and one unrefracted beam (Fig. 2), with the fraction of unrefracted neutrons being proportional to P_σ.

The dependence of the intensity of the unrefracted beam on the grazing angle for a system of quasi-parallel domain boundaries is characterized by a transmission maximum when the beam passes parallel to the domain boundary,[11] so that, for infinitely thin 180° domain boundaries, the presence of an unrefracted beam is associated with the passage of part of the beam past the boundaries. The specific form of this maximum is determined by such average parameters of the system of domain boundaries as their extension along the beam, the distance between them, and the misorientation. Let the shape of this maximum in the absence of the spin-flip effect be described by the function $F(\alpha)$; then the effect of neutron spin flip on the unrefracted beam intensity can be taken into account in the form

$$I_0(\alpha,\lambda)=F(\alpha)+[1-F(\alpha)]P_\sigma(\alpha,\lambda). \quad (9)$$

If one experimentally measures the functions $I_0(\alpha,\lambda_{1,2})$ and independently measures the parameter σ, one can use Eqs. (7) and (9) to determine the domain-boundary thickness for which the function $F(\alpha)$ obtained from the experimental $I_0(\alpha,\lambda_1)$ curve, taking into account $P_\sigma(\alpha,\lambda_1)$, and corrected to the flip probability $P_\sigma(\alpha,\lambda_2)$, gives the best description of the experimental $I_0(\alpha,\lambda_2)$ curve.

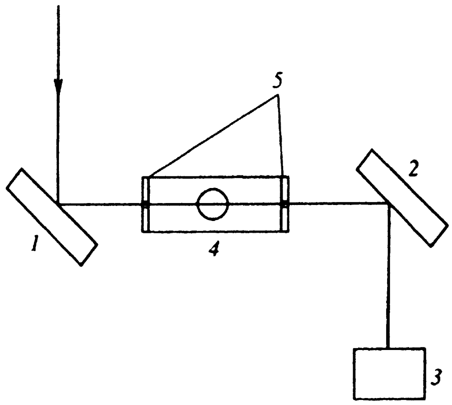

FIG. 3. Experimental layout: *1* and *2*—crystals: monochromator and analyzer; *3*—detector; *4*—sample; *5*—rotatable slit system.

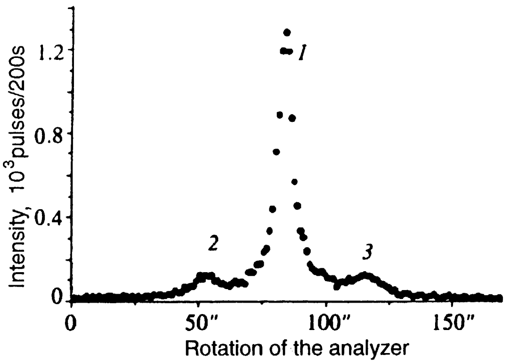

FIG. 4. Scattering curve of thermal neutrons for a grazing angle of $\alpha=0.5°$: *1*—unrefracted beam; *2* and *3*—refracted beams. Measurement time of one point is 200 s.

3. EXPERIMENTAL TECHNIQUE

We used as a sample a single crystal of silicon iron (3 wt% silicon) obtained by floating-zone melting.[12] The crystal was cylindrical, with a diameter of 13 mm and a length of 60 mm, and the cylinder axis was parallel to the [001] crystallographic axis. The domain structure of such crystals is described in Ref. 9. Reference 13 showed that the application of a compressive stress of about 30 MPa along the cylinder axis produces a nearly perfect system of 180° domain boundaries misoriented by no more than 1°.

The experimental layout is shown in Fig. 3. The measurements were made on two-crystal spectrometers with perfect crystals, which were mounted on RNTs reactors of the Kurchatov Institute in Moscow[14] and the Gana–Meitner Institute in Berlin.[15] The neutron wavelength equalled 0.15 nm on the first device and 0.48 nm on the second. The angular resolution of both devices exceeded by more than an order of magnitude the angular deviations of the neutrons when they were refracted at grazing angles of $|\alpha|<10°$.

To ensure that the same section of the domain structure was used on the two different devices, a sample holder was made in combination with a system of $1.5\times 15\text{-mm}^2$ input and output slits relative to which the sample could freely rotate about its axis. The holder was mounted on a rotating table on the device, and the system of slits was adjusted to be parallel to the neutron beam and maintained its position during the measurements, while the sample was rotated relative to the slits. The stability of the position of the slits as the system rotated was controlled with a micrometer. The holder allowed a compressive stress of about 30–40 MPa to be applied to the sample; this stress was monitored in accordance with Ref. 13 from the shape of the transmission curve and did not change during the measurements.

4. RESULTS AND DISCUSSION

When neutrons were refracted at the system of domain boundaries, we observed a beam passing through the sample without refraction and beams refracted at angles determined by Eq. (8) (Fig. 4). Figure 5 shows how the intensity of a refracted beam of thermal and cold neutrons transmitted by a single crystal of silicon iron through a system of 180° domain boundaries depends on the grazing angle. For the thermal neutrons, the curve has a maximum, but, for the cold neutrons, it has a "table" shape, whose width is close to 2°. The intensity of the unrefracted neutron beam with grazing incidence on the domain boundary is thus greater for cold neutrons than for thermal neutrons. According to Eq. (9), this is associated with the adiabatic neutron-spin flip effect when the neutrons pass through a domain boundary, the probability of which is greater for cold neutrons than for thermal neutrons. According to Eq. (6), the width of the "table" is proportional to the thickness δ of the domain boundary, which can be roughly determined from this width. Since the adiabatic spin-rotation effect is largest close to $\alpha=0$, and in order to avoid the distorting influence of multiple refraction, which is significant for $|\alpha|>3°$,[10] we used only the $|\alpha|>2°$ values when processing the data. The scatter of the grazing angles α was determined in accordance with Eq. (8) from the broadening of the refracted beams and equalled 0.4°.

The domain-boundary thickness was determined by curve fitting, which was done as follows: The transmission curve for thermal neutrons was approximated by an analytical function to 0.1%, and this function was used to calculate the shape of the transmission curve without the influence of adiabatic spin flip for some value of δ. Using this shape and

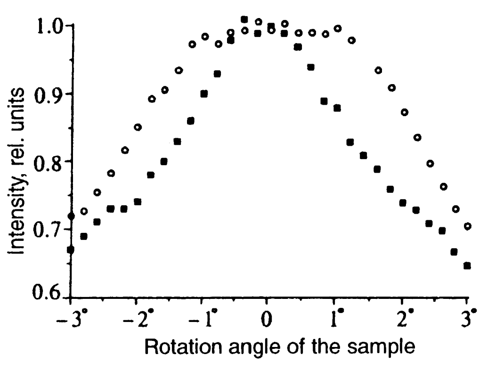

FIG. 5. Experimental dependence of the intensity of the unrefracted neutron beam on the rotation angle of the sample for thermal (■) and cold (○) neutrons.

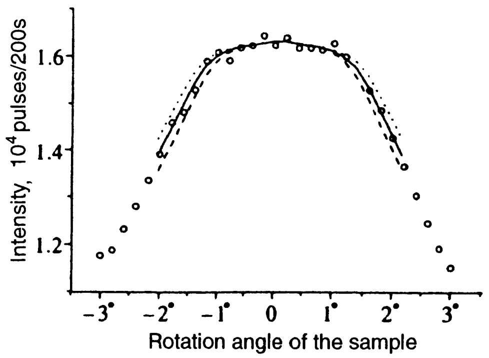

FIG. 6. Experimental dependence of the intensity of the unrefracted beam of cold neutrons vs the rotation angle of the sample (points) and calculated curves obtained for the following thicknesses of the domain boundary: 159 nm (dashed curve), 179 nm (solid curve), and 199 nm (dotted curve).

the same δ value, we calculated the shape of the transmission curve of the cold neutrons which also was fitted to the experimental data. The domain-boundary thickness, the intensity ratio of the incident beams of thermal and cold neutrons, and the value of the zero angle were used as adjustable parameters. The best result of the fitting was achieved when $\delta = 179$ nm (Fig. 6). The accuracy with which δ was determined was estimated from the width of the interval of δ values in which there is no substantial change in the χ^2 fitting criterion; it is estimated as $\Delta\delta = 10$ nm. The thickness of a 180° domain boundary in silicon iron is thus $\delta = 179 \pm 10$ nm. The resulting value agrees with the theoretical thickness of 180° domain boundaries in iron, which equals 180–200 nm.[16,17]

This paper has reported a direct observation of the adiabatic spin-flip effect as neutrons pass through domain boundaries in iron and has demonstrated that the domain boundary has a Bloch structure. The results of this paper show that domain-boundary thickness measurements in neutron-refraction experiments are sufficiently accurate that the real structure of the domain boundary can be studied, and that its thickness can be studied quantitatively in bulk ferromagnetic crystals under various conditions.

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