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X-RAY TOPOGRAPHIC INVESTIGATION OF FERROMAGNETIC DOMAIN STRUCTURES WITH CLOSURE DOMAIN CONFIGURATIONS IN IRON-SILICON SINGLE CRYSTALS

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Résumé. — On a étudié la structure en domaines de plaques monocristallines de fer-silicium (2,4 % en masse), de surface (110), soumises à une traction parallèle à [110] à l'aide de l'effet Kerr et de la méthode de topographie en rayons X par transmission de Lang. L'usage de sections et de toographies par translation renseigne sur les distorsions du réseau, et donc sur la structure des domaines à l'intérieur d'échantillons d'épaisseur voisine de 0,1 mm. En particulier, certaines sections semblent fournir des images de parois à 90° internes, et les franges qui apparaissent sur les toographies peuvent s'interpréter comme des lignes de niveau sur des parois internes.

Abstract. — The domain structure of single-crystal (110) plates of 2.4 wt % silicon-iron was investigated under tensile stress parallel to [110] by means of the Kerr technique and of Lang's method of transmission X-ray topography. It was possible, by using both section and traverse topographs, to gain information about the lattice distortions, and therefore the domain structure, within specimens about 0.1 mm thick. In particular, some section topographs seem to provide images of internal 90° walls, and the fringe system which appears on traverse topographs can be interpreted as a set of equal-depth contours for internal walls.

I. Introduction. — X-ray topographic techniques, and in particular Lang's method [1], have now gained, primarily through Polcarova's work [2-6], a place of their own as methods for observing ferromagnetic domain structures.

It is well known that the contrast of crystal defects such as dislocations can be interpreted within the framework of the dynamical theory of X-ray diffraction in nearly perfect crystals (reviewed in [7] and [8]); it arises from the effect of slight local departures from perfect crystal periodicity on the propagation of wave-fields [9]. It was generally assumed from the beginning, and also proved experimentally [10], that the contrast of ferromagnetic domain walls has the same origin. Because of magnetostriiction, the crystal suffers a distortion related to the direction of the magnetization, $\mathbf{M}$ and $-\mathbf{M}$ being equivalent in this respect. Thus 180° walls are not visible. In particularly simple domain structures, the black or white contrast of 90° walls in iron-silicon could thus be associated with the sign of the difference in distortion of the neighboring domains [11] and interpreted in terms of the dynamical theory of X-ray diffraction by invoking effects of total reflection and refraction of wavefields at the boundary [12].

One of the great assets of this method is that it should make it possible to probe the domain structure within comparatively thick samples, when only surface domain configurations can be studied by conventional methods. In an attempt to take advantage of this asset, we investigated situations where the domain structure involves simple closure configurations, induced either by a magnetic field or by elastic stress.

II. Experimental results. — 1. EXPERIMENTAL METHOD. — We shall discuss the results concerning domain structures induced by elastic stress in (110) plates of single-crystal Fe-Si (2.4 wt %) grown by the Bridgman method. The plates were cut into squares $12 \times 12 \, \text{mm}^2$, 100 to 150 $\mu$ thick, with sides parallel to [001] and [110]. A tensile force could be applied along [110] by means of a jig designed to enable observation of the stressed specimen either on the Lang camera or by means of a Kerr magneto-optical device for surface-domain examination.

The application of stress led to the appearance of a pattern of surface domains elongated along [110] but magnetized along $\pm$ [001], the well-known « pattern I » investigated by Dijkstra and Martius [13].

2. Metastability of the stress-induced domain structure when the stress is removed. — A striking feature of this pattern is that, when the stress is relieved, the domain structure remains unaltered; it is then in a metastable state, as a slight disturbance such as vibration or a magnetic field causes it to vanish into the familiar structure of bands parallel to [001]. The traverse and section topographs obtained from this domain structure with and without external stress were found identical (see, e.g., Fig. b and c).

3. General features of the topographs. — a) A comparison of figure a and b shows that the period of the X-ray images corresponds to half the period of the surface domain structure.

b) Topographs made with the same MoKα1 radiation, but with either 110 (Fig. b or c) or 220 (Fig. e) reflections, look very different; we showed that the dark line in the 220 topographs is shifted by one-half of a surface-domain width with respect to the sharp black fringe of the 110 topographs.
a) Surface domains, Kerr effect, state I; b) Mo Kα 110 traverse topograph (T. T.) of same region, same state as in a; c) Mo Kα 110 T. T., state II; d) Co Kα 110 T. T., state I; e) Mo Kα 220 T. T., state I; f) Mo Kα 220 section topograph (S. T.), state II; g) Co Kα 110 S. T., state I; h) Mo Kα 110 S. T., state I; i) cross-section along (110) according to model of [13].

State I = metastable condition, no external stress applied. State II = with tensile stress ~ 8 x 10^5 dyne cm^-2 applied along [110]. The mark next to each photograph corresponds to 100 μ.

c) A 110 topograph made with CoKα radiation (Fig. d) reveals a system of much closer-spaced fringes, the over-all period being of course the same as for Mo 110 or 220 topographs.

These features can better be understood if we remember that, in this case, the traverse topographs can be considered as the result of traversing along [110] a section topograph and integrating the intensities. A better insight may therefore be gained by referring to the corresponding section topographs (Fig. f, g, h); then the low contrast of Mo 220 topographs and the visibility of a few fringes only in figure d become readily understandable.

III. Discussion. — 1. The observed period of the X-ray topographic images is consistent with Dijkstra and Martius’ model (Fig. i), in which the main domains have antiparallel magnetizations; for reversing all the magnetization directions should not change the lattice distortions.

2. A close examination of the X-ray topographic and Kerr-effect images of the tips of the elongated domains (not shown here for lack of space) shows that the sharp fringe in the Mo 110 topographs correspond to the intersection of closure-domain boundaries with the surface (see also Fig. a and b), whereas the dark stripe in the Mo 220 topographs corresponds to the position of the wall separating the main domains.

3. The variety in aspect of the various topographs shown, which are all made using the same family of (110) planes as reflecting planes, is certainly connected to the different values of two essential parameters of the dynamical theory of X-ray diffraction: the Pendellosung period Λ and the angular width δ of the intrinsic rocking-curve, which is a measure of the sensitivity of the reflection used to crystal distortion. The values are respectively:

\[ Λ = 9.7 \mu \delta = 4.2 \times 10^{-5} \text{ rad for MoKα 110} \]
\[ Λ = 14.8 \mu \delta = 1.4 \times 10^{-5} \text{ rad for MoKα 220} \]
\[ Λ = 3.5 \mu \delta = 1.1 \times 10^{-5} \text{ rad for CoKα 110} \]

4. The difference in distortion on crossing a (111) wall between a closure domain magnetized along [001] and a main domain magnetized along [100] or [010] causes the characteristic parameter of a wave-field, the departure from Bragg’s angle Δθ, to change by ±0.75 δ_110 ≈ 2 x 10^{-5} rad. This value was calculated using the results of [11], i.e. assuming the wall to be infinite and isolated; it is smaller than δ for Co 110 and Mo 110 reflections, but larger than δ for the Mo 220 reflection. This means that, in the Mo 220 case, the wave-fields will suffer large deviations on crossing the (111)-90° boundaries.

5. The fringe periods observed on 110 traverse topographs taken with AgKα, MoKα, and CoKα radiation are proportional to the corresponding Pendellosung periods. It is well known that one fault-plane [14] or twin-lamella [15] inclined with respect to the surface induces on traverse topographs equal-depth contours with a depth-interval equal to Λ. The spacing observed here is consistent with the angle of about 35° between a {111} — 90° wall and the surface which is assumed in Dijkstra and Martius’ model.

6. The striking feature of the Mo 220 section topograph (Fig. h) is that it looks like the expected cross-sectional view (Fig. i). However, we must emphasize: a) that, under the conditions of high absorption used (μd ~ 4), the contribution of «direct images» — the only ones for which a kinship to the cross-section can be claimed — is very slight. Therefore, although it is likely that the images which look like the closure-domain walls actually do originate from them, care should be taken in their analysis;

b) that the horizontal V-shaped streak can not be thought of as the image of the wall between main domains, which must be parallel to (001). It is probably related to the complex distribution of distortions which prevails at the intersection of the three walls.

The situation there bears resemblance to a twin tip; the corresponding stress could be partly relaxed by a complex magnetization configuration, which has not yet been calculated. A thorough understanding of the contrast may yield some information about the nature of the wall intersections.
References

[10] SCHLENKER (M.) and BRISONNEAU (P.), to be published.