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OF (100) Fe<sub>3</sub>wt%Si CRYSTALS COMPRESSED ALONG AN EASY DIRECTION OF MAGNETIZATION

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Magnetic domain wall contrast in the synchrotron x-ray topographs  
of (100) Fe<sub>3</sub>wt%Si crystals compressed along an easy direction of  
magnetization

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Abstract

Transmission and back reflection x-ray topographs of a large near (100) Fe-3wt%Si subgrain compressed along an easy surface direction of magnetization are taken with white synchrotron radiation. Disappearance of the [001] stripe domain contrast is observed only in the 001 transmission topograph. High resolution back reflection topographs reveal a zig-zag fine structure of the stripes. A domain model to explain the results is presented.

### §1. Introduction

Compression along [010] in grain oriented (100) [001] Fe-3wt%Si commercial crystals was found to create stripe domains, aligned parallel to the stress direction. Transmission synchrotron x-ray topographs taken at successively increased values of compressive stress from 0.5 to 10.8 Nmm<sup>-2</sup> also showed that the stripe domain width decreased with increasing stress (Tuomi, Stephenson, Tilli and Kelh  1979). The results were in agreement with energy considerations and Bitter pattern observations (Corner and Mason 1963).

Iron has a positive magnetostriction coefficient in the [100] direction as measured by Webster (1925) and calculated by Heisenberg (1931) applying statistical considerations. When the sample is compressed magnetization tends to align perpendicularly to the direction of stress, because this has the same effect as compression: a decrease of the length of the sample in the direction of compressive stress. The resulting domain configuration has been described by a model with zig-zag-boundaries (Chikazumi and Suzuki 1955, Corner and Mason 1963). This model is the starting point for the x-ray topographic contrast analysis of this work.

Polcarova and Kacz r (1967) have shown that a 90° domain wall in an iron-silicon crystal is not visible if

$$\Delta\vec{M} \cdot \vec{g} = 0. \quad (1)$$

In eq. (1)  $\Delta\vec{M} = \vec{M}_1 - \vec{M}_2$ , where  $\vec{M}_1$  and  $\vec{M}_2$  are the magnetization vectors of the two adjacent domains, and  $\vec{g}$  is the diffraction vector. The contrast is caused by the magnetostrictive deformation which is different on the two sides of the wall. A 180° domain wall is expected to show no contrast, because the magnetostrictive deformations on each side of the wall are equal.

From the theoretical domain configurations and from eq. (1) one can find

out in which x-ray topographs the 90° domain walls of the model should disappear and in which cases they are visible. In this work such x-ray topographs are taken and the observed contrast in them is compared with the theoretical predictions. The x-ray topographic method that utilizes white synchrotron radiation (Tuomi, Naukkarinen, Laurila, and Rabe 1973, Tuomi, Naukkarinen, and Rabe 1974, Hart 1975) is particularly well suited for the contrast analysis, because several reflections can be obtained at a time and exposure times for taking high resolution topographs are short (a few seconds).

### § 2. Experimental

An about 0.15 mm thick grain oriented (100) Fe-3wt%Si platelet was glued on an aluminium frame of a compression-tension jig (Tuomi et al. 1979). A constant compressive stress causing a strain of  $1.6 \cdot 10^{-5}$  was applied to the specimen in the horizontal [010] direction. The strain was measured with a strain gauge glued on the sample. Transmission and back reflection topographs were taken on Kodak Industrex type R single coated films with the white synchrotron radiation from the DORIS storage ring, whose positron energy was 3.0 GeV and whose current was 15 to 30 mA. In the back reflection geometry fluorescence radiation was effectively removed by means of a 3 mm plastic sheet in front of the film cassette. In the transmission mode a cassette in which the film was behind a 2 mm thick aluminium cover was used for the same purpose. The distance between the film of the size 100 x 100 mm<sup>2</sup> placed perpendicularly to the incident beam and the sample was 50 mm in each case. A circular aperture, 10 mm in diameter, bored in a 80 mm thick lead shield limited the size of the incident beam. In the back reflection geometry the centre hole of the film cassette, the diameter of which was 5 mm, further decreased the size of the beam.

### § 3. Synchrotron x-ray topographs

Fig. 1 a) shows a 001 transmission topograph taken from the compressed crystal. For this measurement the sample was tilted  $18^\circ$  about the horizontal  $[010]$  -axis from the position in which the surface normal ( $2^\circ$  off the  $[100]$  direction) was parallel to the synchrotron radiation beam.

According to a calculation performed in the same manner as in previous works (Tuomi, Naukkarinen, Rabe 1974, Hart 1975, Petroff and Sauvage 1978) the principal wavelength of the diffracted beam on the film turned out to be 39  $\mu\text{m}$  (reflection 004). Horizontal stripe domain areas are clearly visible in the topograph. Between them there are regions where no domain structure is visible.

Fig. 1 b) shows a 411 reflection topograph (principal wavelength 127  $\mu\text{m}$ ) taken from the same part of the crystal as in Fig. 1a. For taking this topograph the sample was turned back to the position in which  $[100]$  was almost parallel to the synchrotron radiation beam. The 411 reflection topograph shows exactly the same horizontal stripe domains as seen in the 001 reflection and in addition also stripes in the vertical direction. Moreover, the fine zig-zag structure of the domain walls can be distinguished, mainly in the  $[010]$  horizontal stripes partly due to the better geometrical resolution in the vertical direction than in the horizontal one. Because the positron beam height at 3.0 GeV in DORIS is about 1 mm (Materlik 1979) the vertical resolution in the topograph of Fig. 1 is about 1.5  $\mu\text{m}$ . The horizontal resolution calculated from the formula given in a previous work (Tuomi et al. 1974) is about 3  $\mu\text{m}$ . Several other transmission and reflection topographs were examined. There was a very weak horizontal  $[010]$  stripe domain contrast in the  $1\bar{3}0$  and in the  $130$  reflection. The vertical  $[001]$  stripes almost disappeared in the  $10\bar{3}$  and in the  $103$  reflection (see the Laue patterns in the previous work (Tuomi et al. 1979)).

### § 4. Stripe domain model

Fig. 2 a) shows a magnetic domain model for the  $(100)$  Fe-3wt%Si crystal compressed along the  $[010]$  direction. It is essentially the same as that presented previously by Chikazumi and Suzuki (1955) in which  $90^\circ$  domain walls, e.g. CDEF and EFGH intercept the surface and join to each other along zig-zag lines, e.g. along EF. The zig-zag junction line of the two walls  $90^\circ$  on the surface is justified by the reflection topograph shown in Fig. 1b. Zig-zag lines have also been observed by Roessler (1967), who took x-ray topographs under anomalous transmission conditions, and in the Bitter patterns of compressed iron-silicon crystals (Chikazumi and Suzuki 1955, Bates and Carey 1960, Corner and Mason (1963)).

The direction of magnetization is also shown in the domain model. The difference between the magnetization vectors on both sides of a horizontal  $[010]$   $90^\circ$  domain wall such as ABCD is

$$\Delta\vec{M}_1 = \vec{M}_1 - \vec{M}_2 = [101] \quad (2)$$

and for the wall CDEF

$$\Delta\vec{M}_2 = \vec{M}_2 - \vec{M}_3 = [10\bar{1}]. \quad (3)$$

For the vertical  $[001]$  stripe domain walls this difference vector is

$$\Delta\vec{M}_3 = [1\bar{1}0] \quad (4)$$

or

$$\Delta\vec{M}_4 = [\bar{1}10]. \quad (5)$$

If the diffraction vector is  $\vec{g} = [001]$ , then  $\vec{g} \cdot \Delta\vec{M}_3 = 0$  and  $\vec{g} \cdot \Delta\vec{M}_4 = 0$ , which means that the vertical stripe domains should disappear in the 001 reflection. This was also observed in the synchrotron x-ray topographs (Fig. 1a). A similar disappearance of vertical stripe domains (called fringes) was observed by Kuriyama and McManus (1968) in the 002 reflection using anomalous transmission of  $\text{CrK}_\alpha$  radiation.

It is evident from the model that it is not possible to observe the disappearance of either vertical or horizontal stripe domains in any other

reflection than 001 or 010, respectively. The horizontal stripe domain wall ABCD for which  $\vec{AM}_1 = [101]$  should disappear e.g. in the  $14\bar{1}$  topograph. However, for the adjacent domain wall CDEF  $\vec{AM}_2 = [\bar{1}01]$  and therefore the horizontal stripes are visible in the  $14\bar{1}$  reflection as was also found experimentally.

The 411 reflection topograph, in which  $2\vec{M}_i \cdot \vec{g} \neq 0$  ( $i = 1,2,3,4$ ), shows dark (weak x-ray intensity) zig-zag lines which are approximately 5  $\mu\text{m}$  wide. In the model of Fig. 2a) the zig-zag lines are junctions of  $90^\circ$  domain walls penetrating the surface. In a first approximation the lines may be regarded as boundaries between two surface domains with opposite magnetization,  $\vec{M}_2$  and  $\vec{M}_4$ , and therefore they may be regarded as  $180^\circ$  domain walls. Magnetostriction would not result in any noticeable distortion of the lattice and consequently no contrast will be observed in the x-ray topographs.

It is, however, possible that the zig-zag lines of the  $90^\circ$  domain wall junctions widen out into bands, when the compressive stress is increased, as shown schematically in Fig. 2b). This kind of an "opening-up" of the closed flux structure on the surface was suggested by Dijkstra and Martius (1953) who observed Bitter patterns in  $(110)$  iron-silicon crystals compressed along  $[001]$ . Simultaneously with the widening of the zig-zag lines the width of the surface domains, the magnetization of which is  $\vec{M}_2$  or  $\vec{M}_4$ , decreases with increasing compressive stress. The reflection topograph of Fig. 1b) lends some support to this model of Fig. 2b). In the regions of small stripe width the black lines are wider than the white ones, whereas the broad white stripes are accompanied by narrow black zig-zag stripes. Williams, Bozorth and Shockley (1949) made calculations which show that the "opening-up" of the flux reduces the total energy.

As a conclusion, the contrast analysis of the x-ray synchrotron topographs renders support to the stripe domain model of Chikazumi and Suzuki (1955) and to the suggestions made by Dijkstra and Martius (1953).

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Figure captions

Fig. 1. (a) 001 transmission and (b) 411 reflection topograph of a near (100) Fe-3wt%Si crystal taken with synchrotron radiation from DORIS in Hamburg. The crystal is compressed along horizontal  $[010]$  direction. Measured (average) compressive strain is  $1.6 \cdot 10^{-5}$ .

Fig. 2 a) Model of a magnetic domain configuration in a uniaxially compressed near (100) Fe-3wt%Si crystal. b) Widening of the stripes at a large compressive stress.

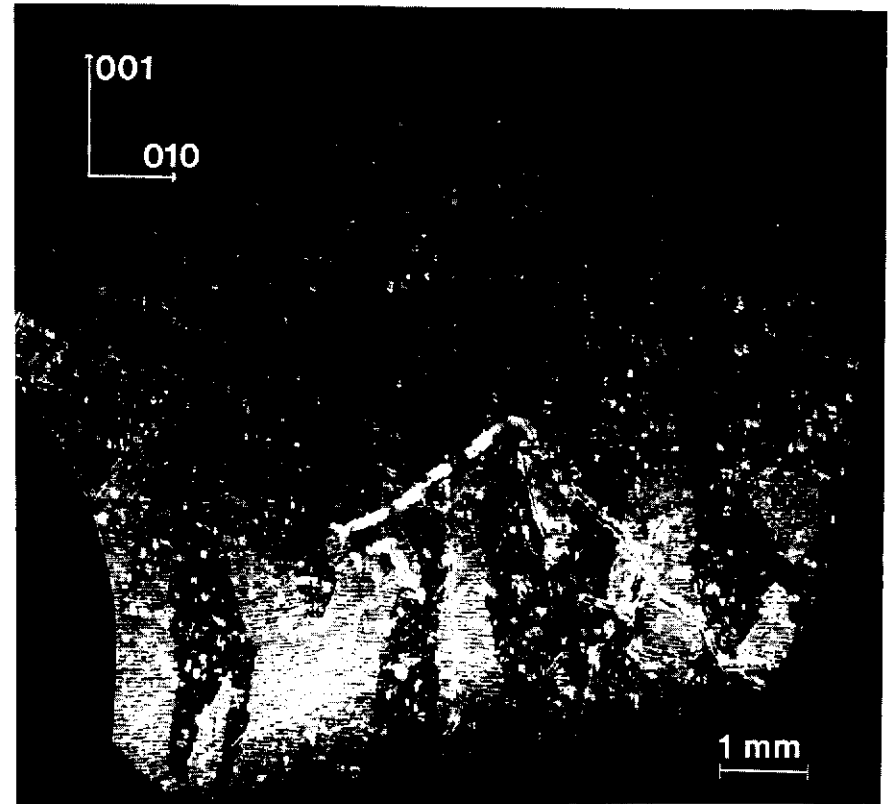


Fig. 1 a

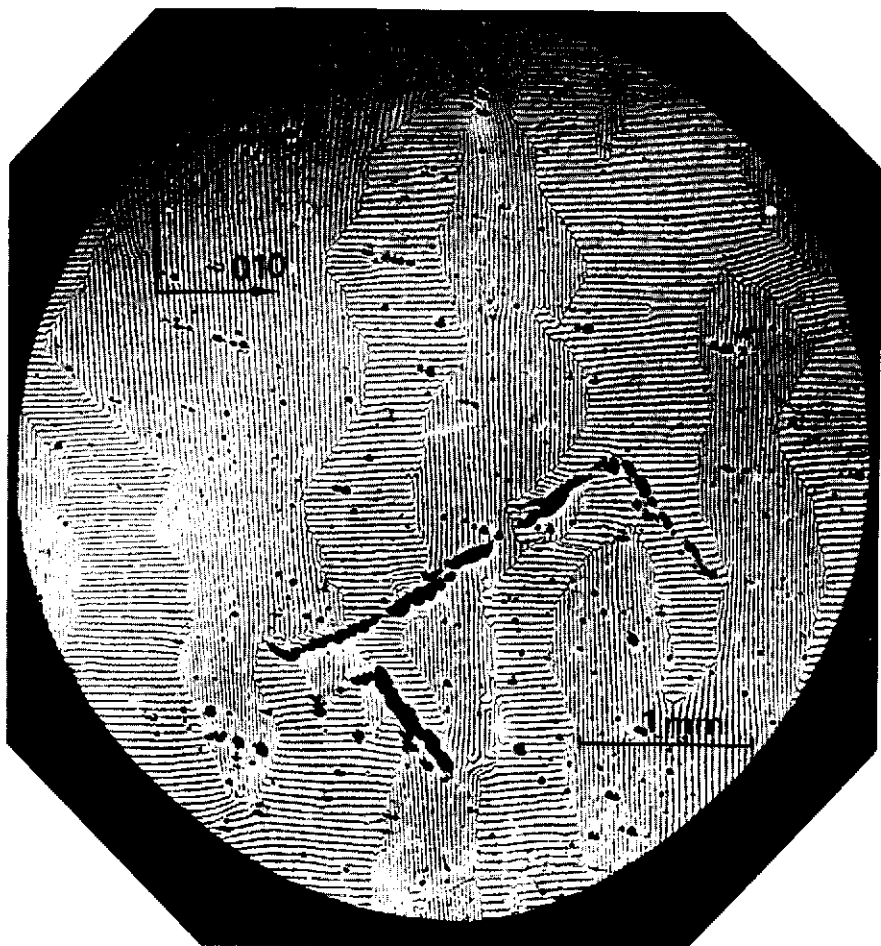
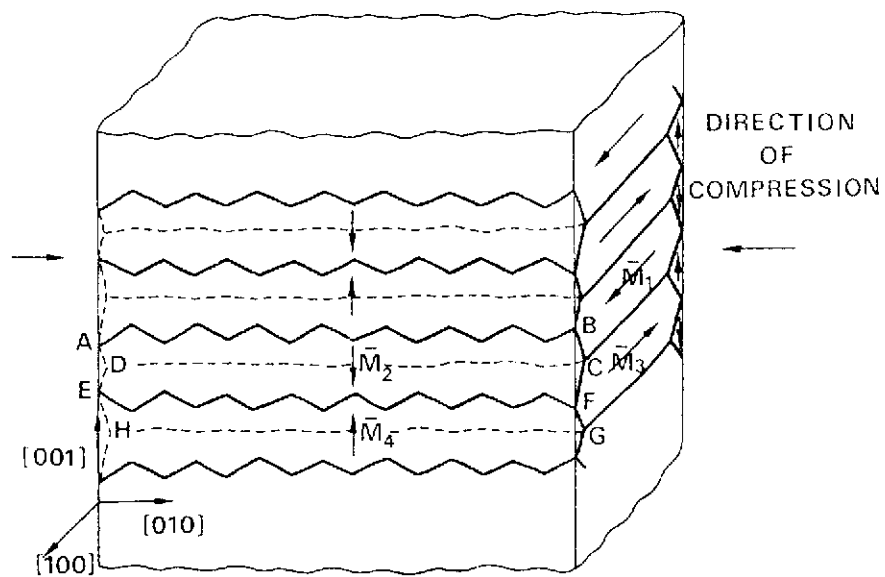
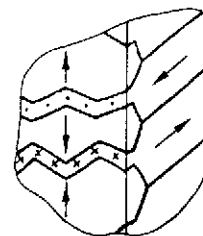


Fig. 1 b



a)



b)

Fig. 2