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OBSERVATION OF DOMAIN STRUCTURE IN A (111) ORIENTED GRAIN OF FE(SI) BY X-RAY WHITE BEAM TOPOGRAPHY

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ABSTRACT

Using white synchrotron radiation from the storage ring DORIS the structural changes with time in the domain pattern of a Fe-3wt% Si polycrystal are observed without any external magnetic fields and stresses applied. In a (111) oriented grain a magnetic domain configuration with <211> directed 90° domain walls perpendicular to the sample surface is detected. The residual stress state is estimated with the help of the stripe domain pattern observed in the neighbouring (100) oriented grain. Observations performed during five months show that the domain wall distance in the (111) grain remains constant and only the area covered by this structure changes.

Mit Hilfe der weißen Synchrotronstrahlung vom Speicherring DORIS werden zeitliche strukturelle Änderungen der Domänenkonfiguration einer grobkristallinen Fe-3Gew.% Si - Probe beobachtet, ohne daß ein externes Magnetfeld oder mechanische Spannungen angelegt sind. In einem (111)-orientierten Korn wird eine magnetische Domänenstruktur nachgewiesen, deren 90°- Wände in <211> Richtung und senkrecht zur Oberfläche verlaufen. Der Restspannungszustand wird mit Hilfe des Streifendomänenmusters im angrenzenden (100) orientierten Korn abgeschätzt. Beobachtungen, die sich über fünf Monate hinzogen, zeigen, daß der Domänenwandabstand im (111)- Korn konstant bleibt und sich nur die Flächenbedeckung dieser Struktur ändert.

1. INTRODUCTION

The use of X-ray topography is a very convenient and nondestructive tool to observe magnetic domain walls because of the sensitivity of this method to the lattice distortion caused by magnetostrictive deformations [1-5]. During the last ten years white synchrotron radiation has been employed [6-9] to study magnetic structure and also magnetic domain movement in Fe(3% Si) polycrystalline specimens both with or without the influence of external stresses or magnetic fields. The advantage of using synchrotron radiation is not only in much shorter exposure times (two or three orders of magnitude in comparison to a rotary anode or conventional X-ray tube) but mainly in the fact that at the same time one obtains a set of Laue images which give information under different diffraction conditions about the same crystal state. In addition, pictures of the neighbouring grains of quite different orientation can be simultaneously taken which is of great importance to follow changes in the magnetic domain structure over the grain boundary.

It is well known that only when directions of easy magnetization are parallel to the sample surface the domain pattern has a simple structure. Usually such a structure is representative of the interior and can be solved by using Bitter pattern or Kerr effect. It was shown that the images obtained by these techniques closely correspond to those obtained by X-ray topography [3,10]. Using transmission and reflection geometry it is in principle possible to distinguish between the surface closure configuration and the main bulk domain set. When the direction of easy magnetization points out of the sample surface like it is the case with a (111) surface, an increase of the surface closure domain density is revealed [11] and the closure domains form the complicated maze pattern. Nevertheless Williams et al. [12] observed by Bitter pattern sets of stronger lines forming an angle of 120° with each other corresponding to the threefold symmetry of the crystal plane. They did not give an explanation of this image suggesting only that the observed pattern might be restricted to the surface. Their lines were running closely to the <112> directions. A model of this structure was given by Hubert [13] consisting of main domains with the direction of easy magnetization [100] and [$\bar{1}$00] separated by 180° walls perpendicular to the surface and some closure domains with all three directions of easy magnetization.

In this work synchrotron radiation transmission and reflection white beam topographic experiments are reported to show the domain structure of a (111) oriented grain which was found in a sample consisting mainly of (100) oriented mutually twisted grains and to follow its behaviour in time without any external magnetic field (except earth's magnetic field) and especially applied stresses.

2. EXPERIMENTS.

A grain oriented Fe(3% Si) commercial crystal was used in the measurements. The initial preparation of the sample was similar to that reported in [8][1]. Nevertheless a grain with a (111) surface orientation was found. Observations were performed several times during a period of six months. When taking topographs the storage ring was running in either of two modes: 5.3 GeV, 30 mA or 3.7 GeV, 80 mA. Typical exposure times were 5 s in transmission and 15 s in reflection geometry. In the latter case a 2 mm thick lucite absorber was placed in front of the film to reduce the background caused by fluorescent radiation from the sample. All the topographs were recorded on Kodak Industrex type R single emulsion film. The film distance was usually 55 mm. All presented topographs are the direct copies of the originals i.e. lower intensity of the diffracted beam means darker area of the image.

3. RESULTS AND DISCUSSION.

3.1 Area under Observation.

Two neighbouring grains, topographs of which were reassembled in a "jig-saw puzzle" fashion from enlarged Laue spots, are of primary interest here. As it is schematically shown in Figure 1 the surface orientation of the grains is (111) and (001), respectively. The [$\bar{1}\bar{1}$2] direction of the (111) oriented grain and the [100] direction of the (001) oriented grain are twisted by 7°. Practically all the surface of the (001) grain was covered by stripe domains during the observations. The (111) grain usually showed complicated tiny domain configuration with the exception of two areas where a well defined pattern of parallel lines and bands were seen. These areas are indicated in Figure 1.

[1] The sample was obtained by courtesy of T. Tuomi, Helsinki Univ..

3.2 Time dependent changes of the magnetic domain pattern.

3.2.1 (001) oriented grain.

The main purpose to follow this time changes was to obtain information about the residual stress situation [8,14] in the vicinity of pattern 2 of the (111) oriented grain as discussed below. Generally the following types of domain image changes were observed : a) appearing and disappearing stripe domains, b) decreasing and increasing domain width (0.03 to 0.15 mm), c) substitution of [010] domains by [100] directed domains and vice versa and climbing of areas of perpendicular domain orientation, d) creation of alternating broad and narrow domain widths [15]. All these changes are shown in Figure 2 with indication which amount of time has passed between one state and the other. The influence of the strong direct beam on the domain pattern was also observed. It was done using a short (14 s) and subsequently a long (50 s) exposure time (Fig. 3). Generally the domain width decreases with longer exposure time what can be caused by increasing internal stress due to the heating of the sample by the beam. In addition the pattern becomes more uniform thus less sensitive to local stresses induced by crystal imperfections. This sequence of short and long exposures was repeated several times, each time with full reconstruction of the observed structure. As a conclusion it is worth to underline that when following time changes of the domain pattern with white beam topography the influence of beam heating should be taken into account. The beam heating problem is in practice more or less always present in white beam topography experiments.

3.2.2 (111) oriented grain

The pattern observed in the (111) grain is discussed in detail in the next paragraph. Here its time behaviour is considered. The only change observed is a variation of the domain length without changing the width. Pattern 1 first extended its area then vanished. Pattern 2 was more stable and only reduced its extension. These changes with time are shown in Figure 4.

3.3 Magnetic domain structure of the (111) oriented grain

Except the complicated and rather tiny domain structure which can be seen in Figure 4(b) well defined black and white lines parallel to the $[\bar{2}11]$ direction (pattern 1) and to $[\bar{1}\bar{1}2]$ (pattern 2) are present. Depending on the diffraction vector also black and white bands with the same orientation are observed in transmission but never in reflection. Analysis of the images taken from both sides of the sample indicates that the lines are traces of planar defects crossing the sample perpendicular to the surface. A direct confirmation was achieved by taking section topographs. The set of $[\bar{1}\bar{1}2]$ directed planar defects lying in $(\bar{1}10)$ crystallographic planes with strong X-ray contrast suggests a set of 90° domain walls with $\Delta\boldsymbol{M} = \boldsymbol{M}_1 - \boldsymbol{M}_2$ ($\boldsymbol{M}_1$ and $\boldsymbol{M}_2$ are the magnetization vectors of two adjacent domains) parallel to either [110] or $[\bar{1}\bar{1}0]$ direction. Hence the magnetization vectors parallel to the directions of easy magnetization, which in Fe(Si) are <100>, in neighbouring domains are [100] and $[0\bar{1}0]$ (or [010] and $[\bar{1}00]$) for $\Delta\boldsymbol{M}$ = [110] and $[\bar{1}00]$ and [010] (or $[0\bar{1}0]$ and [100]) for $\Delta\boldsymbol{M} = [\bar{1}\bar{1}0]$.

As it is known [1] the visibility condition for a 90° wall in Fe(Si) requires $\Delta\boldsymbol{M}\cdot\boldsymbol{g} \neq 0$ ($\boldsymbol{g}$ is the diffraction vector). The contrast is either black or white depending on the sign of the scalar product and is due to the magnetostriction deformation which is different on both sides of the domain wall in contrary to the 180° wall where the deformation is equal in both domains, thus no contrast is expected in principle.

A set of topographs needed to follow the contrast changes with different diffraction vectors $\boldsymbol{g}$ was taken. Because of the poor quality of the grain under study it was impossible to utilize all Laue spots obtained. Blurring of the wall image when projected under certain angle and overlapping with the band contrast forced us to choose only reflections belonging to the $[1\bar{1}0]$ zone. Having a horizontal diffraction plane the investigated domain walls were placed horizontally and the crystal was rotated about the vertical $[1\bar{1}0]$ axis starting from the position where the surface normal [111] was parallel to the incident beam. Planes satisfying no contrast condition belong to the zone with axis $\Delta\boldsymbol{M}$ thus all the topographs taken "to the right" of the (001) reflection should show an inversed contrast in comparison to topographs taken "to the left". The obtained topographs, some examples of which are shown in Fig.5, indicate that the planar defects are indeed 90° walls with alternating [110] and $[\bar{1}\bar{1}0]$ directions of the vector $\Delta\boldsymbol{M}$. The section topographs show the same contrast as the Laue spots (Fig. 5(a)).

The information that we could obtain from this grain was not sufficient to analyze the band contrast in detail. It can be caused eventually by flat closure domains or complicated phenomena connected with changes of the magnetization direction close to the sample surface and corresponding magnetostrictive deformations. Some discussion is included in the next paragraph. Here we only state the presence of a set of 90° walls crossing the sample perpendicular to the surface and visible on both sides of the sample.

3.4 Energy considerations

The energy per unit area E of the sample surface can be written as the sum of magnetostatic E_m, magnetoelastic E_σ and domain wall energy E_w:

$$E = E_m + E_\sigma + E_w$$

According to [16]

$$E_m = 2/(1 + \mu^*)\ 1.7\ I_s^{*2}\ d\ \sin^2\Theta$$

where I_s^* is the normal component of magnetization, d the domain width and $\Theta = 35.27°$, the angle between the easy magnetization axis and the surface. The μ^* correction term is needed because spins turn from the direction of easy magnetization under the influence of the surface field. In our case with d ~ 0.005 - 0.01 cm E_m becomes ~ 350 -700 erg/cm^2

The magnetoelastic energy density [17] with a stress σ_{hkl} applied along [hkl] is given by

$$E_\sigma = -(3/2)\ \lambda_{100}\ \sigma_{hkl} \sum (\alpha_i\gamma_i)^2 - 3\ \lambda_{111}\ \sigma_{hkl} \sum \alpha_i\gamma_i\alpha_j\gamma_j$$

where α_i are the direction cosines of magnetization in the domain, γ_i the direction cosines of the applied stress and λ_{100} and λ_{111} the saturation magnetoelastic constants along [100] and [111], respectively. Assuming compressive stress $\sigma_{\bar{1}\bar{1}2} < 0$ we obtain a magnetoelastic energy density equal to 0.975 $\lambda_{100}\ \sigma_{\bar{1}\bar{1}2}$ for M_{001} (or $M_{00\bar{1}}$) and 0.25 $\lambda_{100}\ \sigma_{\bar{1}\bar{1}2}$ for M_{100} and M_{010} (or $M_{\bar{1}00}$ and $M_{0\bar{1}0}$). In both cases the magnetostatic energy will be the same but the magnetoelastic energy is lower in the second case which makes this configuration energetically more favorable.

The internal stress was estimated by observation of the stripe domain pattern [14,8] in the neighbouring (001) grain. As it is shown in Fig. 1 the angle between [100] of the (001) grain and [$\bar{1}\bar{1}2$] of the (111) grain is 7°. The presence of the stripe domains without any external magnetic field is caused by compressive stress with a significant component in the [100] direction. As it was shown above it is exactly this direction of stress which prefers the creation of the pattern observed in the neighbouring (111) grain. Its absolute value is not so important because the magnetoelastic energy term does not influence the calculation of the domain wall separation and the stress will only influence the wall energy. The separation of the observed stripe domain walls was 0.03 - 0.15 mm which gives a compressive stress from 0.02 to 50.8 N/m^2.

Calculations of the wall energy under stress were performed using the Kaczér and Zelený theory [18]. The estimated range of stress leads to a value of the reduced wall energy from 1.65 to 1.73.

By minimizing E with respect to d, the equilibrium domain wall spacing may be found. This distance was calculated to be ~ 0.005 mm which is about ten times smaller than the observed spacing. A few remarks are necessary. Both patterns 1 and 2 observed in the (111) grain are not typical and representative for the whole grain. Especially pattern 2 which was very stable in time seems to be created by local stress conditions which concern only this part of the grain. It is seen that the walls are pinned between the grain boundary and a long lattice defect (Fig. 4). In addition, the presence of magnetic charges at both the grain boundary and the lattice defect may be an important feature favoring the formation of the observed pattern. But these effects are not easily incorporated into the calculation. Our result is that the domain spacing in the (111) grain is too large in comparison to the one expected from the stresses which in turn are estimated from the stripe domain pattern in the neighbouring grain.

A way to decrease the total energy is the formation of a surface closure domain system. The observed band contrast suggests the presence of such a set. Some support to this suggestion is shown in Fig. 6, where below the upper surface of this section pattern a diffuse zig-zag contrast is seen. On the other hand it is also seen how strongly strained and deformed the sample is. The lower surface in this image is far from being a straight line and only some walls are partly seen. A sample of higher quality and a full control of the stress is needed to propose the final unambiguous form of the reported domain structure.

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FIGURE CAPTIONS

Figure 1. : Sketch of domain image locations and grain shape. Mutual misorientation between [100] direction of the (001) oriented grain and $[\bar{1}\bar{1}2]$ direction of the (111) oriented grain is about 7°. Stripe domains are schematically shown in the (001) grain. Dotted lines indicate the location of <112> directed domain walls in the (111) oriented grain.

Figure 2. : Time dependent changes of stripe domains in the (001) grain. In a) both [100] and [010] domains are seen whereas in b), which was taken two days later, the same area is fully covered by [100] domains. Time between c) and d) was three days. This pair of topographs mainly shows a strong increase of the domain width with a tendency to form alternating broad and narrow domains.

Figure 3. : [100] stripe domain patterns after different exposure time being 14 s for topograph (a) and 50 s for (b). The most distinct changes are seen on the right side of the long white strained area.

Figure 4. : Time changes of the domain images in the (111) grain. Pattern 1 which is seen in a) disappeared after two months and was never seen during the next three months. Instead, a tiny complicated domain structure was observed as it is shown in b). Pattern 2 was visible during all the observations but reduced its area without any change of domain width as it is seen in c) and d).

Figure 5. : Transmission topographs of pattern 2 taken with different diffraction vectors from both sides of the sample. a) is the section image of the $(22\bar{2})$ reflection, b) and c) are $(22\bar{2})$ and $(11\bar{2})$ reflections. Topographs d) and e) are taken with the second side of the sample as an entrance surface and are $(\bar{1}\bar{1}2)$ and $(22\bar{2})$ reflections. Note the inversed wall contrast in d).

Figure 6. : Section topograph showing a weak diffused zig-zag contrast below the upper surface of the sample (see text).

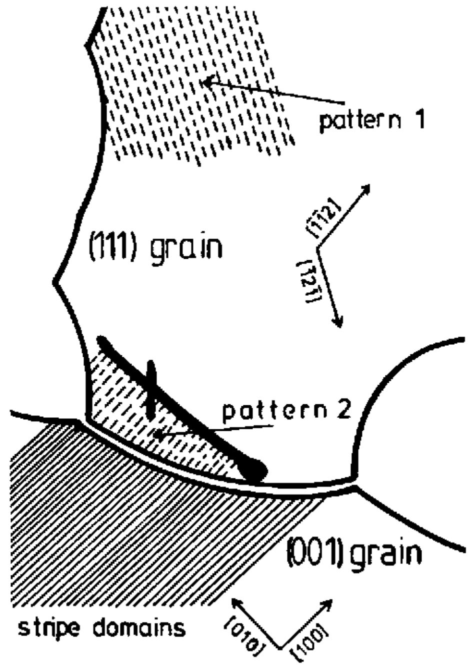

Figure 1

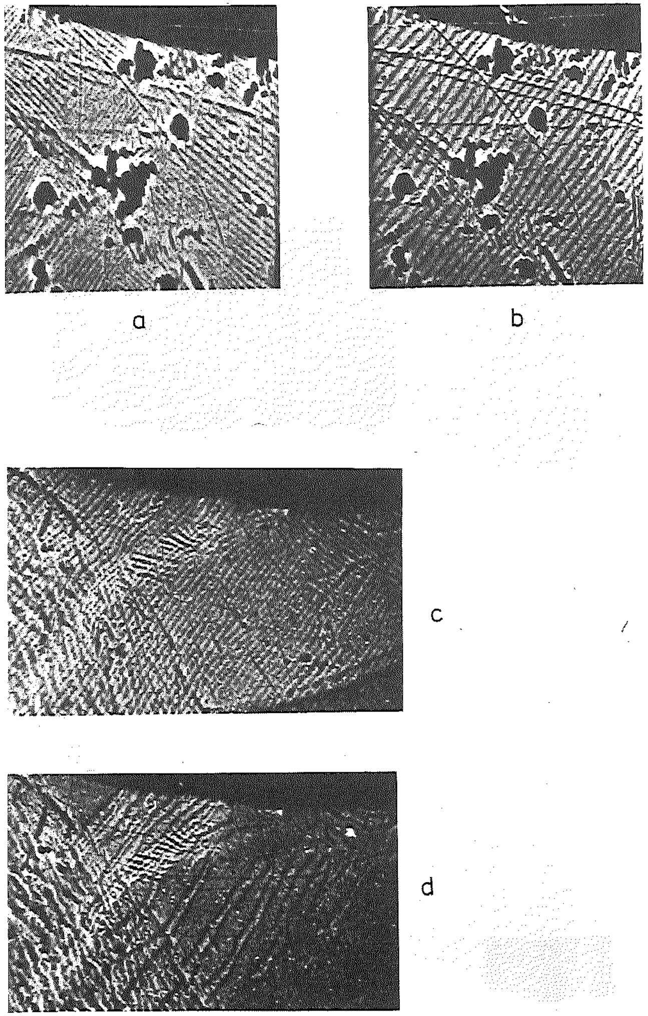

Figure 2.

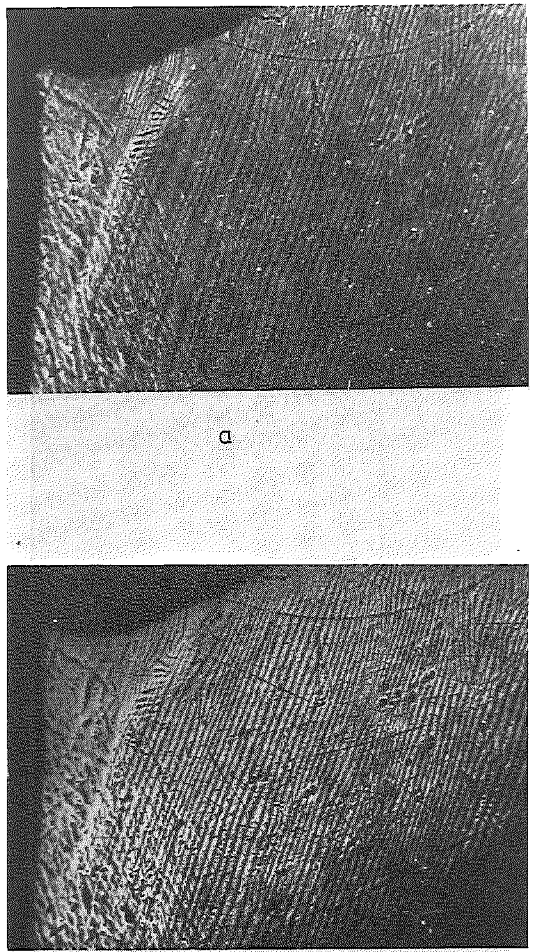

Figure 3.

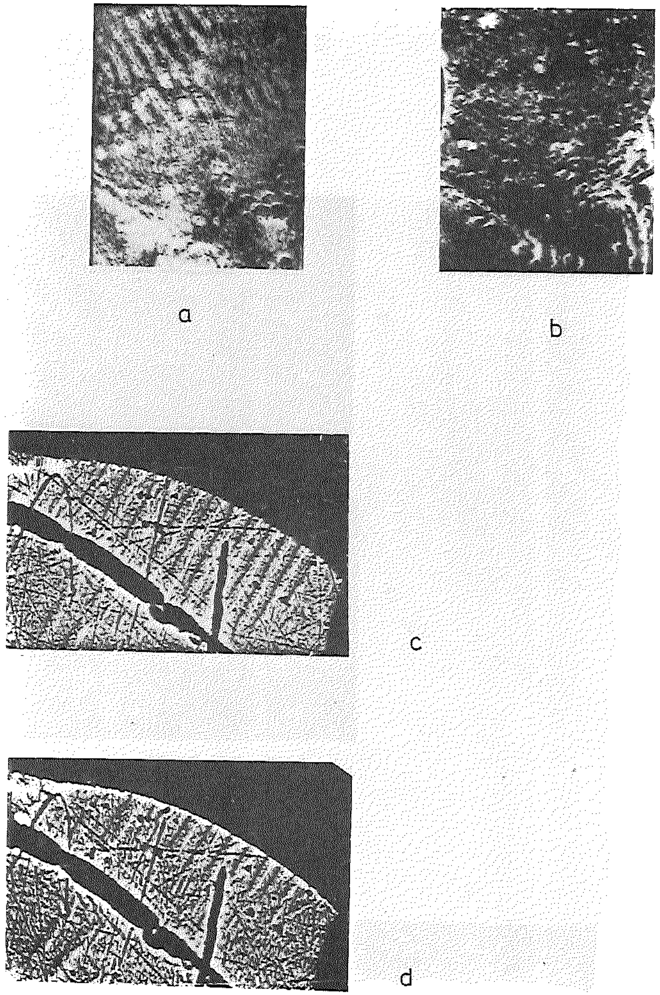

Figure 4.

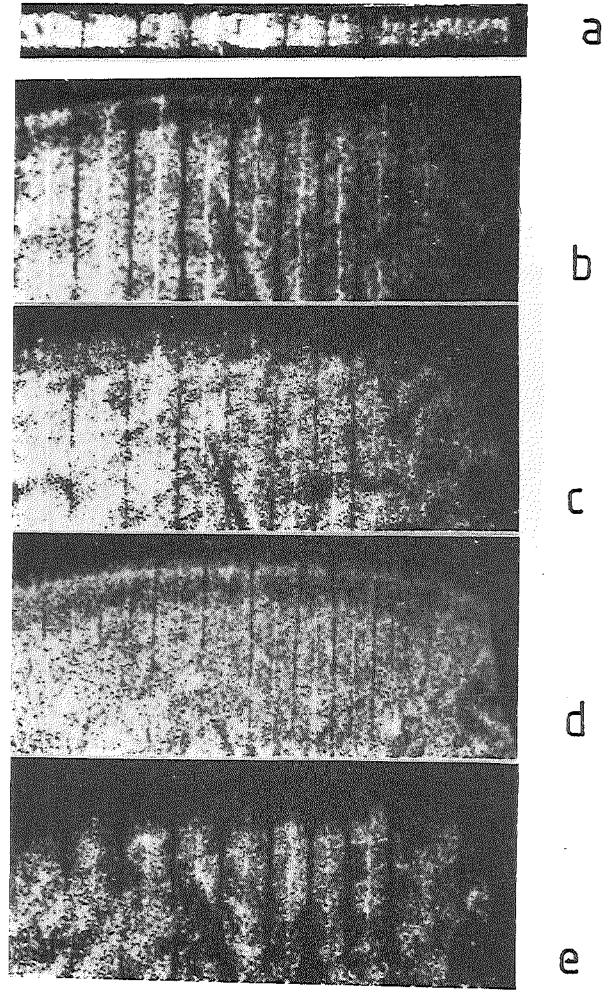

Figure 5.

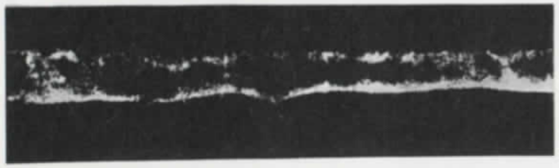

Figure 6.