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DIRECT OBSERVATION OF FERROELECTRIC DOMAINS IN BARIUM TITANATE BY MEANS OF THE ELECTRON MICROSCOPE

by

S. AMELINCKX (C.E.N.)

H. BLANK (Euratom)

1963

Euratom - United States Agreement for Cooperation
Work performed under the Euratom/C.E.N. contract No. 063-61-10 RDB
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DIRECT OBSERVATION OF FERROELECTRIC DOMAINS IN BARIUM TITANATE BY MEANS OF THE ELECTRON MICROSCOPE

H. Blank[1] and S. Amelinckx
Solid State Physics Department
C. E. N. – S. C. K., MOL (Belgium)
(Received 14 March 1963)

The well established methods for studying directly the geometry of ferroelectric domains are: (1) optical observation in polarized light;[2] (2) etching.[3] It is the purpose of this Letter to draw attention to the use of electron transmission microscopy as a new tool to study ferroelectric domains.

Recent attempts to prepare barium titanate single-crystal foils suitable for electron microscopic observation have been successful[4,5] and it has been shown that ferroelectric domains can be revealed.[5] The electron microscope offers the attractive possibility of increased resolution as compared with the optical microscope, and of simultaneous diffraction experiments.

Figure 1 is an example of the type of image which can be obtained in tetragonal barium titanate. The domains which have a differently oriented *c*-axis exhibit varying shades because of differences in diffraction conditions. This can clearly be seen in Fig. 1 and 2 as a shift of the extinction contours within the narrow domains, with respect to the contours outside. The boundaries themselves, when inclined with respect to the foil plane, produce interference fringes similar to those seen at stacking faults or twin boundaries, Fig. 2 and 3. The contrast is clearly diffraction contrast, as can be concluded from tilting experiments. Boundaries which are perpendicular to the foil plane, and hence are roughly parallel to the beam, have a small apparent width (see Fig. 1). Sometimes the opposite boundaries of a domain show different contrast as in Fig. 3 along AA′ and BB′. This may be related to the polar character of the *c*-axis. This thickness of the walls seen end on is in any case much smaller than 4000 Å, as has been assumed in the past from optical observations.[6]

A very striking feature of these walls is the instability of their configuration. Their position changes with the intensity of the electron beam current, with focusing conditions, or when the specimen is displaced in the field of view. This of course makes it difficult to perform selected area diffraction experiments. If the beam intensity is

Fig. 1. Domain boundaries in $BaTiO_3$ seen end on. From the picture one may estimate the wall thickness as being below 100 Å.

INDEXING CATEGORIES

A. barium titanate	
B. ferroelectric domains	
C. electron microscopy	
C. diffraction	
E	

Fig. 2. A domain in a crystal wedge showing thickness fringes. The displacement of the thickness fringes in the domain is indicated by a local stronger contrast in the fringes of the domain boundaries.

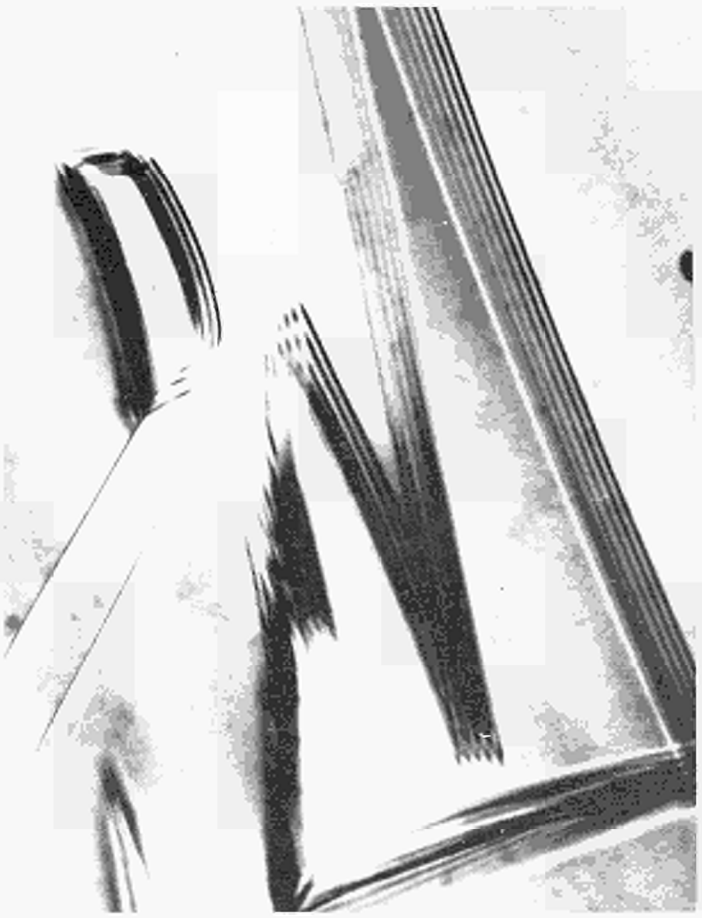

Fig. 3. Domain boundaries showing deviations from the easy <110> directions. Notice the bent boundary in the closed loop. The other indications are explained in the text.

turned up the specimen can easily be heated above the transition point (120°), with disappearance of the domain patterns.

From Fig. 3 it is clear that the domain boundaries are not strictly bounded to {110} planes, as is usually observed optically. The deviations can be judged from the crystallographic orientation of the foil which has been indicated on the figure. The *z*-shaped boundary in *Q* has presumably this peculiar shape as a result of its tendency to remain more or less parallel to a {110} plane. All boundaries visible in Fig. 3 are apparently of the 90°-type. Interactions are visible in *P* between the boundaries perpendicular to the foil and boundaries inclined 45° with respect to the foil. Notice the small closed loop of boundary.

Another frequently occuring type of boundary usually shows only weak contrast and moves rather steadily at the same speed at which one moves the specimen stage, *i. e.*, the image of the boundary remains more or less stationary on the screen while it actually sweeps across the moving crystal [see Fig. 4(*a*)]. From several indications one may conclude that it is a 180° boundary. If this boundary remains fixed for several minutes in the crystal it slowly changes its appearance and obviously serves as a site of nucleation for a new domain as can be seen from Fig. 4(*b*) which shows the same area as Fig. 4(*a*) but after several minutes.

A more detailed account of these observations will be published elsewhere.

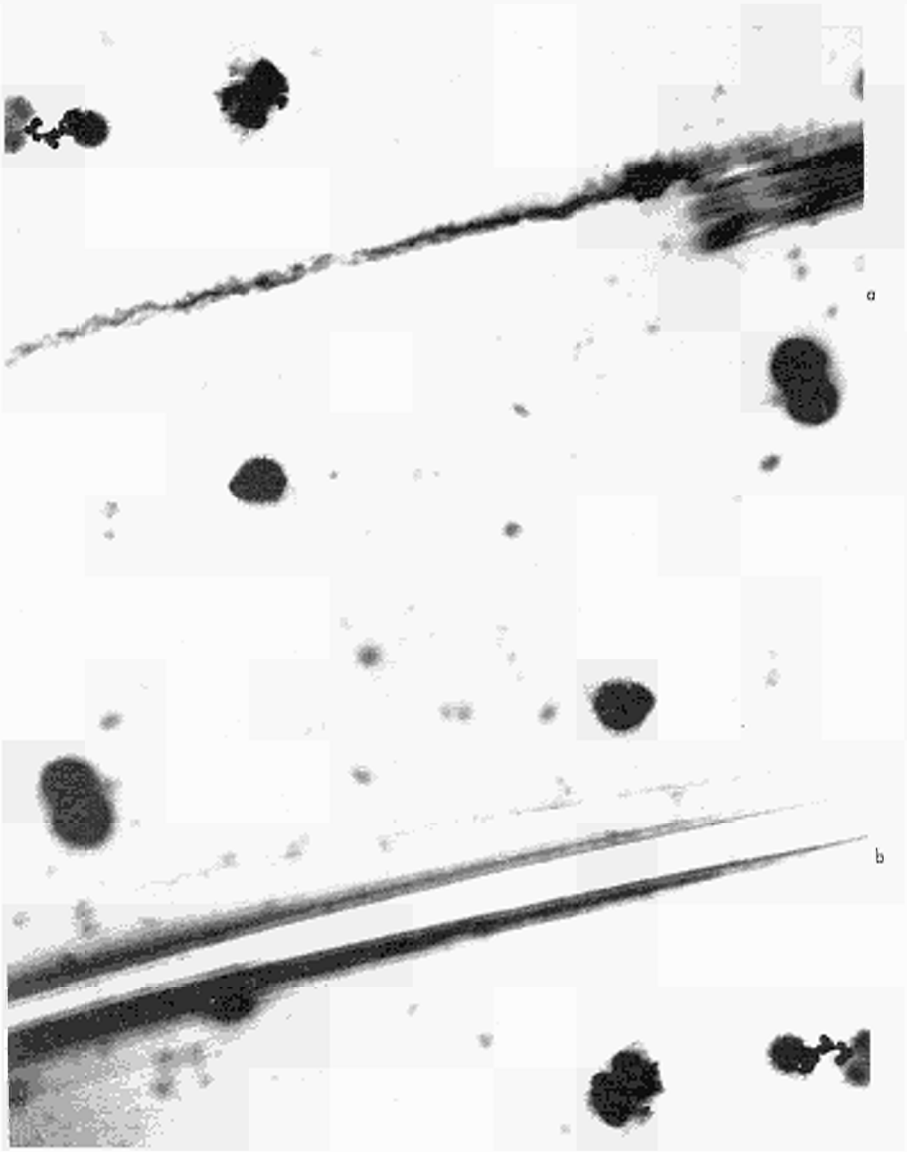

Fig. 4. Change in domain configuration.

(*a*) A 180° boundary has come to rest and slowly changes its appearance. At the right one can see the tip of a new domain nucleated in the 180° boundary.

(*b*) The new domain has suddenly advanced to the left along the old 180° boundary.

[1]Permanent address: Euratom, Brussels.

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