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SOME RECENT APPLICATIONS OF X-RAY TOPOGRAPHY

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ABSTRACT

Dislocations, Inclusions, and Precipitates. Impurity precipitated after growth and foreign particles accidentally included during growth both produce intense local strain fields which give rise to diffraction contrast effects resembling those seen in electron microscope images of precipitates. The relationship between the dislocation configuration and these localized strain centers can show whether the latter arise from inclusions or precipitates. Precipitates will generally be found strung along the grown-in dislocations, decorating them. On the other hand, inclusions often generate dislocations by lattice closure errors; such dislocations then fan out from the inclusion in the general direction of advance of the growth interface.

Twin Boundaries and Fault Surfaces. The cases when the twins have parallel lattices, such as in Brazilian and Dauphiné twinning in quartz, are interesting. When the crystals on either side of the twin boundary are both Bragg reflecting, the twin boundary may exhibit 'stacking fault' type fringes. From an analysis of the variation of visibility of these fringes in different reflections, the fault vector at the twin boundary and its variation with boundary orientation may be found. In quartz, other types of fault surfaces producing fringe contrast may lie parallel to growth horizons or they may mark growth sector boundaries. In synthetic quartz, they can also mark cell boundaries under conditions of cellular growth.

Internal Magnetic Domain Structures. In plates of Fe + 3% Si roughly parallel to (110), a variety of previously undetected domain structures has been discovered and analyzed. Diffraction contrast is produced by 90° domain walls but not by 180° walls. The 90° walls produce strong diffraction contrast even though the magnetostriction of silicon-iron is only about 10^{-5} . In plates parallel to (112), the main lamination pattern below the complex pattern of surface closures can be revealed and, in favorable cases, interpreted.

X-ray Moiré Patterns. The most direct method of observing X-ray moiré patterns—by topography of one crystal closely superimposed upon another—involves considerable theoretical complexities and produces a variety of curious diffraction patterns. However, it shows promise of providing a means for the comparison of lattice spacings to about one part in 10^7 and for mapping strain fields very sensitively.

INTRODUCTION

The aim of this paper is to illustrate some current activities in the practical use of X-ray topography for detecting and displaying lattice defects, and at the same time to demonstrate the interplay of this work with investigations of the diffraction behavior of nearly perfect crystals. X-ray topographic study of single crystals in which individual dislocations can be resolved remains a leading activity, and it is especially directed toward investigations of the growth history of crystals. The ability to sample a large crystal volume and present on a single topographic record the variation in degree and

type of imperfection over a distance in the crystal corresponding to a substantial fraction, if not all, of its period of growth is an asset of the X-ray topographic method which in some measure counterbalances its inferior resolution compared with the electron microscope. Grown-in defects in diamonds have been extensively studied¹⁻⁶, and natural and synthetic quartz, and the amethyst variety of quartz⁷ have been investigated to a considerable degree. In the cases of both quartz and diamond, added interest arises from the availability of both natural and synthetic specimens; it is always instructive to compare the products of the laboratory with those of nature. The nearly perfect crystals upon which the bulk of X-ray topographic work has been done have all had quite simple structures, such as the diamond structure, the face-centered cube (e.g., aluminium⁸) and the body-centered cube (e.g., iron⁹). In the more complex structure of quartz we might expect to find diffraction evidence for a wider variety of lattice imperfections than those present in the simplest structures. Such variety has indeed been found. In the earliest X-ray topographic studies of quartz various sheet defects or 'fault surfaces' were revealed.^{10,11} Such fault surfaces may be of general occurrence in complex structures; the recent observations by Yoshimatsu¹² on fault surfaces in *ADP* crystals are suggestive.

Observations on fault surfaces in alpha quartz will be reported below. The type of fault surface most amenable to exact study is the boundary between twins whose lattices are parallel. The two common types of twinning in quartz—those following the Brazil and Dauphiné laws—fall in this category. The experiments described here illustrate the timeliness of the new theoretical work of Kato, Usami, and Katagawa¹³ reported elsewhere in this volume. They also show the need for development of the theory to take into account X-ray absorption.

Two aspects of X-ray topographic study that do not directly involve dislocations will also be discussed. These are the study of internal magnetic domain structures and of X-ray moiré fringes, both of which have implications outside the field of lattice imperfections.

The question may be asked, how far is it possible to use X-ray topography merely as a tool for detecting and identifying lattice imperfections without reference to complicated diffraction theory? The answer is that one can proceed satisfactorily quite a useful distance in many directions. Examples are the study of the sizes and shapes and deformations of subgrains in single crystals, the locating of surface damage and the verification of its satisfactory removal, and the counting of dislocations and even the determination of their Burgers vectors. However, in the last-mentioned work one soon runs into diffraction phenomena which require some understanding of the processes of production of diffraction contrast if a proper relation of the image to the nature of lattice defect is to be made, as some examples in this paper will show. Indeed, it is worth emphasizing that due regard should be taken of the theoretical work on diffraction by perfect and nearly perfect crystals. It is certainly necessary if X-ray topographic experiments are to be designed and executed to give the maximum information yield. The most significant theoretical papers for transmission X-ray topography are those by Kato and his colleagues, and a summary of work done up to 1962 will be found in a useful review by Kato.¹⁴

DISLOCATIONS, INCLUSIONS, AND PRECIPITATES

A common dislocation configuration found in crystals is one in which dislocations radiate from a central point within the crystal and run outward to the crystal faces.

It is frequently seen in natural diamonds.¹ The interpretation of the pattern—that the dislocations were generated at the crystal nucleus and were subsequently grown into the crystal—is doubtless correct, but it leaves unsettled the question whether the nucleation was homogeneous or heterogeneous. Rapid initial growth under the conditions of supersaturation that attend homogeneous nucleation could lead to the introduction of dislocations, especially if the initial growth were dendritic. However, if the topographs show a concentration of strain at the nucleus greater than that attributable to the dislocations, then it is likely that a foreign body is present there, and that nucleation was heterogeneous, the dislocations being generated by initial imperfect growth on this body or by lattice closure errors arising in the course of its envelopment. Evidence on the origin of dislocations drawn from X-ray topographic studies of a wide variety of crystal species, but of necessity restricted to those specimens which are of sufficient purity and perfection of crystal matrix to allow individual dislocation images to be recognized, indicates that the dislocations in these crystals are generated by lattice closure errors at foreign bodies incorporated during growth rather than by some mechanism involving growth accidents in the course of crystallization of pure material.

The configurations adopted by grown-in dislocations, their relationship with inclusions, and the effects of impurity segregation at dislocations after growth, are well illustrated by natural and synthetic quartz; some recent observations on these materials will now be described. The record of the growth history of quartz crystals that appears on X-ray topographs includes not only the configuration of grown-in dislocations but also the disposition of fault surfaces, the shapes of twins, and the presence of growth stratifications which delineate the surfaces upon which material was crystallizing during the stages of growth of the crystal.

The structure and twinning of quartz are described in a standard work.¹⁵ Note that there has been some confusion in the indexing of planes of quartz in Miller-Bravais axes. In certain X-ray crystallographic work, the plane which is structurally and morpho-

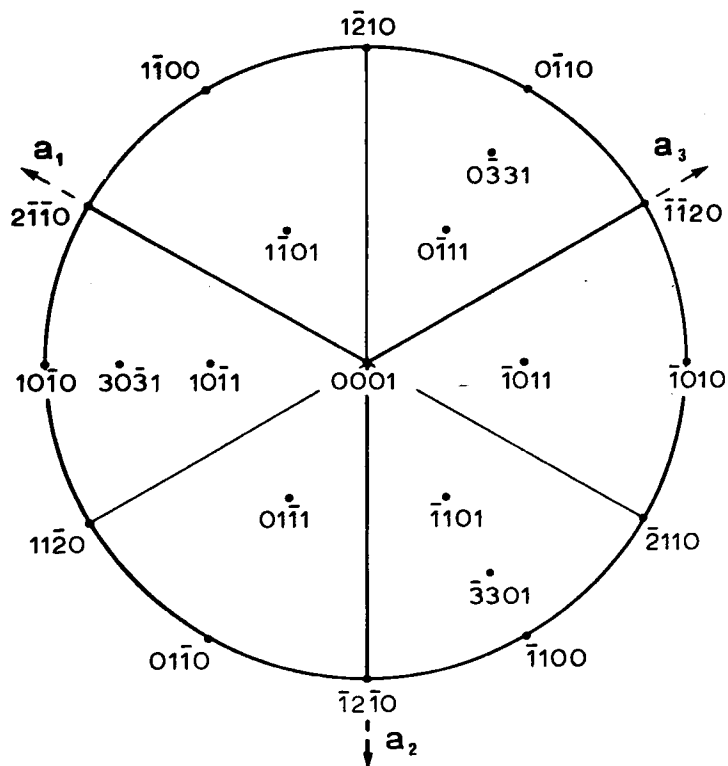


Figure 1. Stereographic projection on to the basal plane of quartz showing poles of reflecting planes most used in X-ray topographic surveys.

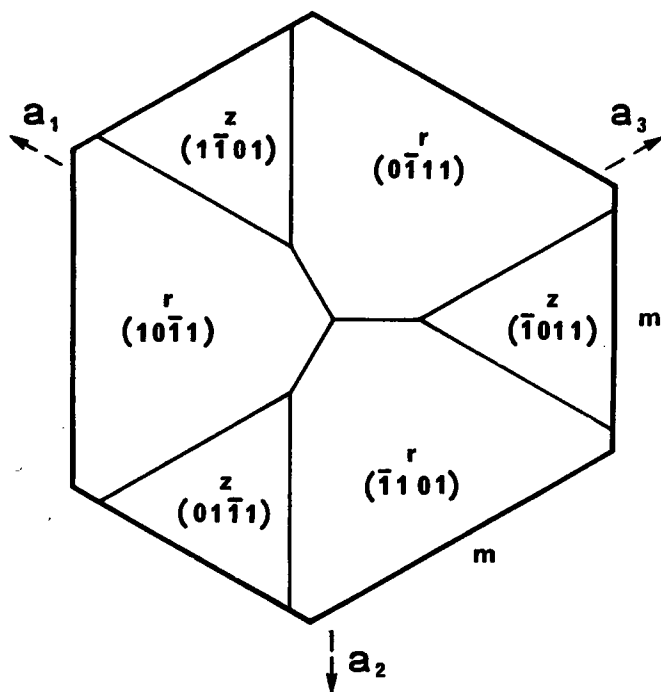


Figure 2. Idealized division into major and minor rhombohedral growth sectors in a quartz slice cut parallel to the basal plane.

logically recognized as the major rhombohedron is given the index of the minor rhombohedron. The history of this confusion, and a mnemonic for the correct orientation of the Miller-Bravais axes with respect to the structure have been stated;¹⁶ the correct orientation is adopted here.

Figure 1 is a stereographic projection on the basal plane of quartz which indicates the planes chiefly used in X-ray topographic studies, in some cases in several orders of reflection. Most of the work on natural quartz has been done on plates about 15 mm square and 1 mm thick cut in the *BT* orientation. This lies between $(10\bar{1}1)$ and (0001) , being 11° off $(10\bar{1}1)$. Suppose a slice were cut from a quartz prism parallel to the basal plane. Then, assuming growth had occurred only on the major and minor rhombohedral faces, the slice would be divided into areas which had grown on these faces in the way shown ideally in Figure 2. These divisions are called *growth sectors*, and it will be seen that the *r*-*z* growth sector boundaries are parallel to $\{10\bar{1}0\}$ and the *r*-*r* sector boundaries are parallel to $\{11\bar{2}0\}$. Growth stratifications parallel to either *r* or *z* faces will cut the slice in lines parallel to the sides of the hexagon. When topographs are taken using some of the various planes indicated on Figure 1, the specimen may perforce be viewed from directions making large angles with the *c*-axis, and the geometry of Figure 2 will be appreciably distorted in consequence. Nevertheless, it is usually a straightforward matter to determine the orientations of growth layers and growth sector boundaries. Possibly section topographs¹⁷ may be needed to supplement the projection topographs.¹⁸

Figures 3 and 4 show projection topographs of parts of plates cut in the *BT* orientation. On Figure 3 there can be seen the diffraction images of strings of centers of intense localized strain. These strings lie on surfaces parallel to the *r* and *z* planes. Traces of these planes are more directly indicated by the pattern of growth stratifications revealed in Figure 4. In both Figures 3 and 4 it is easy to identify the trace of $(\bar{1}011)$ which is vertical and that of $(\bar{1}101)$ which slopes upward to the right. (On these figures there also appear images of twin boundaries and growth sector boundaries; these will be described below.)

The X-ray diffraction contrast arises from strain gradients in the specimen

Figure 3. X-ray topograph of natural quartz plate about 1 mm thick. Field edge length 8 mm; reflection 2420, diffraction vector vertical; Mo K_{α} radiation; fault fringes at Dauphiné twin boundaries.

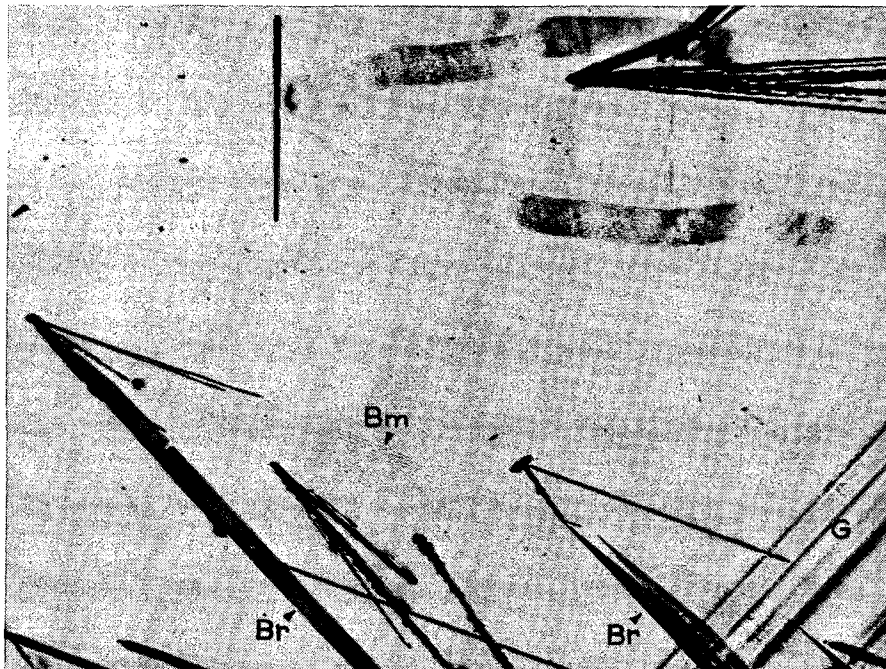


Figure 4. Topograph of natural quartz plate, thickness about 1 mm. Field area 0.9 mm by 1.1 mm; reflection $10\bar{1}\bar{1}$, Ag K_{α} radiation. Projection of diffraction vector is horizontal, direction $[\bar{1}2\bar{1}0]$ is vertical on topograph (orientation similar to Figure 3). Growth layers parallel to $(\bar{1}101)$ marked (G); Brazil twin boundaries (*Br*) are parallel to $(0\bar{1}11)$ and (*Bm*) parallel to $(0\bar{1}10)$.

variability from point to point of the identity, mode of incorporation in the structure, or quantity of impurity will cause changes in cell dimensions; but lattice curvature is more effective than a gradient of dilation in producing diffraction contrast. Experiments show that the diffraction contrast arises chiefly from lattice tilts where the growth layers