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The application of scanning electron beam anomalous transmission patterns in mineralogy

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SUMMARY. It is possible with a standard Geoscan microanalyser to produce back-scattered electron pictures that resemble Kikuchi patterns but which are produced by a different mechanism. These effects have been called scanning electron beam anomalous transmission (SEBAT) patterns and they are produced by the interaction of a parallel scanning beam of high-energy electrons with a crystalline sample. The patterns are excellent indicators of the crystallographic perfection and orientation of samples and can provide information about the lattice dimensions of solid, bulk specimens. The same specimens can, if necessary, be chemically analysed in the same instrument.

In order to produce the patterns the electron beam must be deflected through a minimum angle of approximately 20° and, on existing instruments, the beam covers an area of about 2×2 mm during such a deflection. Minor modification of the authors' microanalyser has reduced the scanned area to $0{\cdot}5\times 0{\cdot}5$ mm and attempts are being made to reduce this even further.

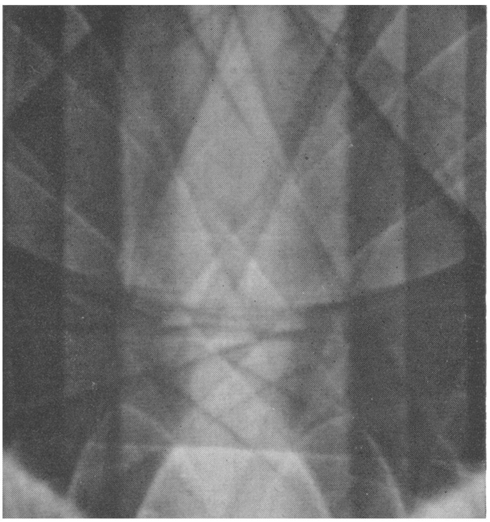
In favourable circumstances the technique enables the mineralogist to determine, rapidly and easily, the crystallographic structure and orientation of large, bulk specimens. Examples are given of patterns that have been produced from cleaved, as grown, and polished mineral faces. The degree and nature of the crystallographic damage produced during mechanical polishing of a galena crystal are illustrated.

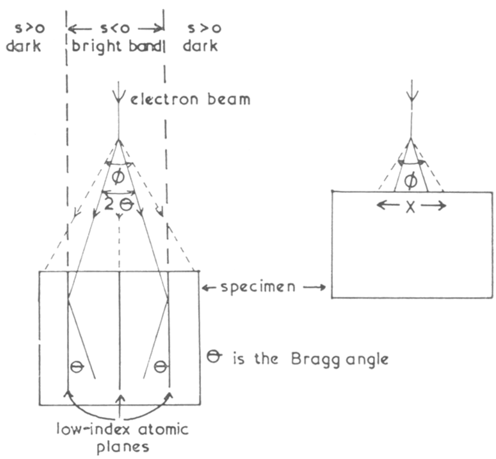
COATES (1967) showed that orientation-dependent electron diffraction patterns could be formed in a scanning electron microscope. It is also possible, under certain circumstances, to obtain similar electron diffraction effects from bulk specimens in the Geoscan electron probe X-ray microanalyser. These diffraction effects (see fig. 1), have been conveniently called by the acronym, SEBAT (Holt *et al.*, 1968, Scanning Electron Beam Anomalous Transmission patterns).

The geometry of the patterns obtained is identical with the Kikuchi line patterns, which have been known for over forty years but which are produced by a different mechanism. The Kikuchi patterns are caused by double diffraction of electrons (first by incoherent scattering and then by coherent scattering) from atomic planes that lie roughly parallel to the specimen surfaces. The SEBAT patterns are formed by electrons that are back-scattered from solid, bulk specimens. The lines and bands that make up the patterns are due to the anomalous transmission effects that occur when an incident beam of electrons is nearly parallel to crystallographic planes in the specimen that are roughly normal to the specimen surface (Booker *et al.*, 1967). On one side of the exact Bragg angle there is anomalously high transmission of electrons and, consequently, little back-scattering. On the opposite side of the Bragg angle condition there is an enhancement of the back-scattering effect (see fig. 2).

Pattern production. The back-scattered electrons are detected and displayed as a video-signal on a cathode ray tube that is scanned in synchronism with the electron beam that scans the specimen. The SEBAT patterns are readily produced from appropriate specimens in the Geoscan microanalyser but the electron path geometry of some other microanalysers may need modification before the patterns can be obtained.

The optimum conditions for producing SEBAT patterns on a Geoscan are: high accelerating voltage, 35 to 45 kV; parallel, defocused electron beam, produced by turning off the electron lenses and collimating the beam by apertures only; the beam must be deflected through the maximum scanning angle, i.e. the condition normally



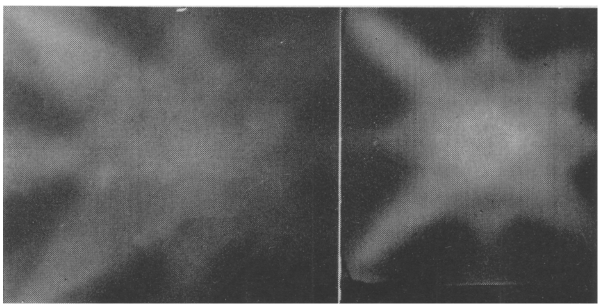


FIGS. 1 and 2: Fig. 1 (left). Typical pattern produced from a high-purity single crystal of silicon. The pattern shows that the 311 plane of the silicon is normal to the central position of the electron beam. The considerable line detail indicates that the sample was a crystal of a high degree of perfection. Fig. 2 (right). Diagrammatic indication of the geometry of the formation of SEBAT patterns (after Booker *et al.* and Holt *et al.*). The atomic planes for which the Bragg condition holds are indicated. Inside these positions (where $S < 0$) low transmission of electrons occurs and a bright band appears in the pattern. Outside these planes (where $S > 0$) there is high electron transmission giving a dark band on the pattern. The effect of moving the specimen nearer the point of deflection of the incident beam is also shown.

employed for minimum magnification; and low gain on the electron detector photomultiplier. Thus, the patterns are best formed when a suitable specimen is scanned by a parallel beam of high-energy electrons that is deflected through relatively large angles. Under these conditions the pattern is immediately visible on the cathode ray tube and visual inspection is facilitated by using a white, short-persistence tube and a fast scanning speed. Very faint patterns on the tube may be difficult to examine but the contrast can be enhanced by suitable photographic techniques.

In order to produce the sharpest and most detailed patterns the samples should be monocrystalline over a minimum area of about 2×2 mm (this area can be reduced to $0{\cdot}5 \times 0{\cdot}5$ mm by minor modification to the Geoscan—fig. 2), and the surface should be reasonably flat but need not necessarily be optically flat. It is possible to produce good SEBAT patterns from *as-grown* surfaces, from cleaved surfaces, or from chemically polished specimens, but no patterns have been obtained from mechanically polished specimens. Coates (1967) claims to have obtained similar patterns from a mechanically polished GaAs crystal.

Advantages. The SEBAT technique offers a number of advantages in the examination of mineralogical specimens. For example, it is possible to determine the crystallographic structure, orientation, and degree of surface structural damage of a solid bulk specimen and also to determine the chemical composition of the same specimen. The SEBAT pattern of a suitable specimen can be obtained on a standard Geoscan microanalyser in a matter of seconds and can be recorded on a Polaroid film. Although a

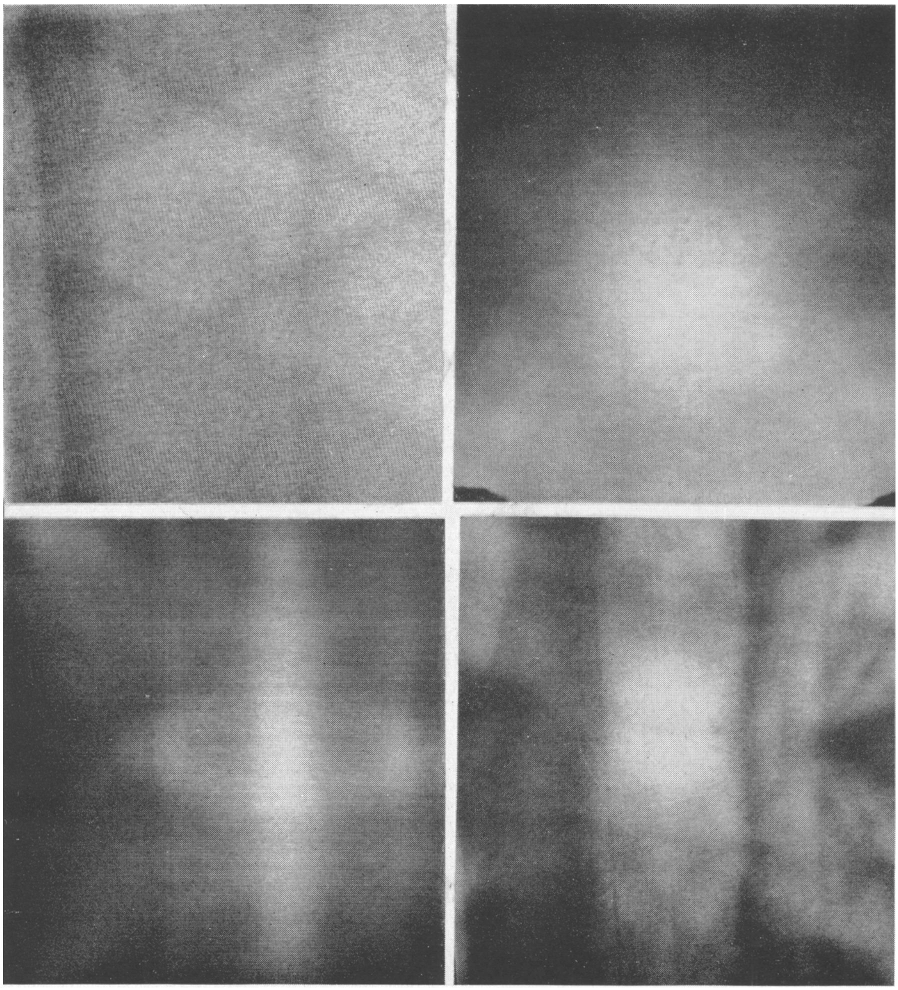


FIGS. 3 and 4: Fig. 3 (left). Pattern obtained from a slightly misorientated (100) plane of a crystal of common salt, consistent with the cubic symmetry. Fig. 4 (right). Cubic symmetry of the SEBAT pattern produced from a (100) cleavage face of galena. This pattern was destroyed by gentle polishing on 0·25 μ diamond for 30 sec, and was recovered when the polished face was etched in hydrochloric acid.

complete interpretation of the patterns can be time-consuming it is often possible to obtain useful information by a simple comparison with published Kikuchi maps (Levine *et al.*, 1966). In addition, it is a simple matter to compare the SEBAT patterns obtained from a mineral before and after a treatment process such as polishing, etching, etc.

The high accelerating voltage that is required to produce the conventional electron-diffraction effects can seriously damage the structure of a number of minerals. However, when similar accelerating voltages are used in a *scanning* defocused beam the effects are much less damaging and minerals like common salt have been successfully examined (see fig. 3). However, the salt crystal was damaged by prolonged scanning and the SEBAT pattern slowly faded. Similarly, the structure of a galena crystal was slowly altered by the electron beam.

It has been shown by Holt *et al.* (1968) that the sharpness of detail in a SEBAT pattern is an excellent indication of the perfection of the crystalline structure of a sample. This effect has been used to show the nature and degree of structural damage that occurs in minerals as a result of the usual mechanical polishing procedures. Fig. 4 shows the pattern produced from a cleavage face of galena, destroyed by gentle polishing on diamond dust and the original pattern recovered when the polished face



FIGS. 5–8: Fig. 5 (top left). Pattern obtained from the basal plane of muscovite mica (001). Fig. 6 (top right). The pattern obtained after etching a polished single crystal of quartz in hydrofluoric acid. A layer of highly disturbed material approximately 1 μ thick was removed by the etchant. The picture shows that the quartz has a low degree of crystallographic perfection, cf. synthetic material in fig. 1. Fig. 7 (bottom left). The pattern obtained from a natural prism face of a large crystal of zircon. This shows that the sample does not possess a high degree of crystallographic perfection. Fig. 8 (bottom right). The pattern obtained from a cleavage face (010) of stibnite. Although this picture does not provide the same detail as fig. 1 it is still possible to measure many of the band widths and line distances in order to derive some crystallographic parameters.

is etched with acid. A similar loss of pattern was observed when the muscovite mica shown on fig. 5 was mechanically polished.

This technique provides a method for determining the degree and depth of structural damage sustained by minerals during the usual polishing procedures. For example, fig. 6 shows how a poor pattern is slowly produced by etching what was considered to be a single crystal of quartz. The depth of the structural damage produced by polishing in this specimen was approximately 1 μ. Figs. 7 and 8 show the SEBAT patterns obtained from a crystal face of natural zircon and from a cleavage fragment of stibnite.

The SEBAT patterns are also extremely sensitive to changes in crystal orientation and it is readily possible to determine the orientation of bulk specimens. This has proved useful in the study of mineral properties, such as chemical reactivity, where it is important to determine the orientation of the crystal face that is being attacked. The SEBAT patterns can also be used to facilitate the preparation of orientated sections of minerals for optical or other study.

REFERENCES

COATES (D. G.), 1967. *Phil. Mag.* **16**, 1179–84.
BOOKER (G. R.), SHAW (A. M. B.), WHELAN (M. J.), and HIRSCH (P. B.), 1967. Ibid. **16**, 1185–91.
HOLT (D. B.), GAVRILOVIC (J.), and JONES (M. P.), 1968. *Journ. Mat. Sci.* **3**, 553–8.
LEVINE (E.), BELL (W. L.), and THOMAS (G.), 1966. *Journ. Appl. Phys.* **37**, (5) 2141–8.

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