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Scanning-Electron-Beam Anomalous Transmission Patterns

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The operating conditions required to obtain scanning-electron-beam anomalous transmission (SEBAT) patterns were investigated and the factors likely to limit the range of materials to which the technique may be applied were studied. It was found that the SEBAT technique is less restricted than is transmission electron microscopy. SEBAT patterns were obtained on a Geoscan X-ray microanalyser from samples of Si, Ge, Ag, CdS, NaCl, epitaxial Si on sapphire, mineralogical mica, and galena. The geometry of SEBAT patterns is discussed and it is emphasised that they are not Kikuchi patterns despite the very close resemblance.

1. Introduction

Coates recently discovered that scanning electron micrographs could be obtained at low magnifications, from monocrystalline silicon and gallium arsenide, which closely resembled Kikuchi line patterns [1]. The lines in these micrographs were explained by Booker *et al* [3] as due to the anomalous transmission that occurs when the incident electron beam is approximately parallel to a low-index crystallographic plane. When transmission of the electron beam into the crystal is anomalously high, losses from the beam are anomalously low. The losses include the high energy electrons that are back-scattered, detected, amplified and displayed as a video signal on the CRT scanned in synchronism with the beam scanning the specimen. Dynamical electron diffraction theory shows that anomalously high transmission will occur on one side of the exact Bragg condition and anomalously high absorption on the other [2], giving rise to the pattern as shown in fig. 2a [3]. It was shown that the geometry of the patterns produced in this way is the same as that of the Kikuchi lines produced in transmission electron diffraction for the same crystallographic orientation of the specimen [3]. The nature and geometry of these patterns will be further discussed below.

For terminological convenience, it is proposed to refer to scanning-electron-beam anomalous transmission patterns by the acronym SEBAT, formed from the initial letters of the descriptive phrase. This agrees with the use of the acronym SEM (Scanning Electron Microscope), SEBERR [4] (Scanning-Electron-Beam-Excited Recombination Radiation) and SEBECC [5] (Scanning-Electron-Beam-Excited Charge Collection) for related techniques. The results of the examination of a number of materials in a Geoscan microanalyser by this new technique are reported in this paper.

2. Experimental Methods

On the Geoscan it was found that the best results were obtained with a high-beam current and with both the lenses turned off and the beam collimated only by apertures. Similar observations were made independently by D. G. Coates* (private communication). The sharpness of the patterns was improved when the beam accelerating voltage was increased (figs. 1a, b). Specimen preparation was not critical. Chemically polished, cleaved, and as-grown surfaces have been employed successfully for different materials. The surfaces have merely to be flat and free of gross contamination and mechanical damage.

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It was found that greater detail appeared in the photographs of the recording CRT than could be seen on the viewing CRT. SEBAT patterns may thus be present even though they are not visible to the eye. The immediate availability of the prints when a Polaroid Land Camera is used for recording is therefore of great advantage in such investigations. The total time taken to place the specimen in the Geoscan and obtain the SEBAT pattern is then less than 2 min.

3. Results and Discussion

The patterns moved when the specimen was tilted or rotated as though rigidly attached to the crystal lattice, as shown in figs. 1a, c.

In order to observe SEBAT patterns it is necessary that the electron beam be deflected through comparatively large angles. This results in large areas of the specimen being scanned. In many cases this can be a disadvantage, for example when it is desired to examine polycrystalline materials in which the sizes of individual grains may be smaller than the area scanned. This limitation can be overcome by moving the specimen nearer to the deflection coils than is normal, as shown in fig. 2. It was found that in the Geoscan microanalyser, specimens could be moved in to less than half their normal distance from the deflection coils without losing the pattern. A specimen area of about 0.5 × 0.5 mm was then scanned. In addition, pattern sharpness was improved as fig. 1c shows.

In order to determine the practical value of the SEBAT technique, the factors that limit the types of materials from which the patterns can be obtained were investigated.

Orientation presented no problem. Patterns were obtained from a number of surfaces, for example the (113) surface of silicon and the (112) surface of germanium as shown in fig. 3. In all cases the patterns could be identified with the Kikuchi patterns for the same orientation, e.g. by comparison with published Kikuchi maps [6] and these identifications were confirmed by other methods of orientation determination.

The technique is not confined to semiconductors of high purity and perfection, for it was found possible to obtain SEBAT patterns from, for example, silver and from a cleavage flake of natural mica (fig. 4). The lines of the mica pattern were found to be broader than for

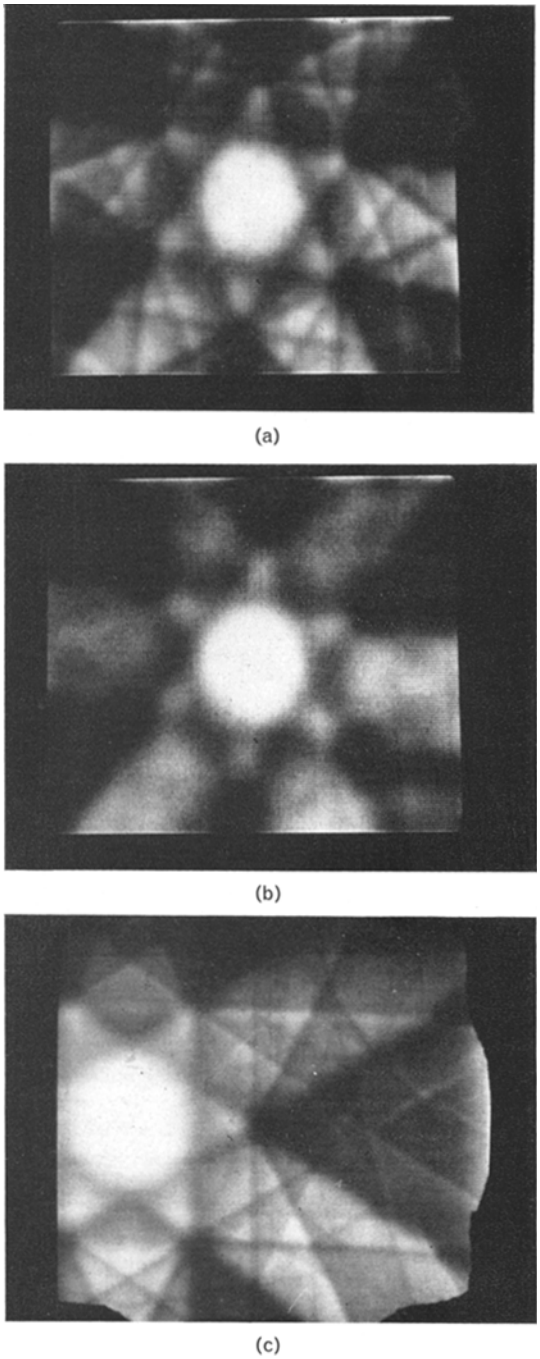

Figure 1 Scanning electron micrographs from a (111) surface of silicon. These are SEBAT patterns obtained using a large beam current with the beam defocused. (a) Accelerating voltage 45 kV (instrumental magnification setting "90 ×"). (b) 20 kV ("50 ×"). (c) Specimen tilted and rotated slightly relative to (a) and moved in to half the normal distance from the beam deflecting coils; 35 kV ("70 ×").

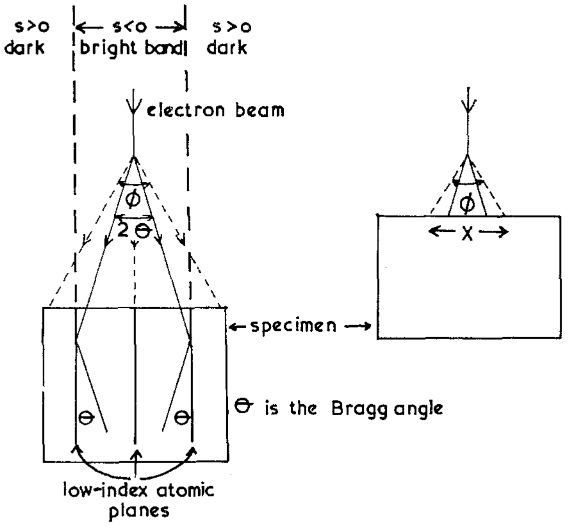

Figure 2 Geometry of anomalous transmission into electron-beam-scanned crystals (after Booker *et al* [3]). The atomic planes for which the Bragg condition is satisfied are shown. Inside these positions ($S < 0$), anomalously low transmission and correspondingly high "reflection" occurs, and a bright band arises in the SEBAT patterns. Outside these planes ($S > 0$) anomalously high transmission occurs and a dark region arises. The effect of moving the specimen nearer to the deflection coils is to retain the same scanning angle ϕ while reducing the area of specimen surface scanned (X^2).

the semiconductors. It was also found possible to obtain SEBAT patterns from silicon films vacuum-evaporated on to sapphire [7]. These gave even less sharp patterns than did the mica. Specimens of germanium were examined which had been masked so that vacuum-evaporated ZnSe had been deposited only over a part of the surface. Sharp SEBAT patterns were obtained from the germanium, but nothing was detectable in the ZnSe covered areas. This was not an effect characteristic of the film/substrate combination, for SEBAT patterns have been obtained from ZnSe films deposited on germanium by a chemical vapour transport technique (T. Rallins†, private communication). It is thought that these observations are indications that SEBAT patterns are sensitive to crystalline perfection in much the same way that Kikuchi patterns are. Point defects, point defect aggregates, or other defects that produce deviations from planarity or periodicity in the atomic planes concerned, would alter the conditions for anomalous transmission and broaden the features of the SEBAT patterns. This was the case for mica which contains a large concentration of impurity atoms (by semiconductor standards), and for epitaxial silicon and ZnSe which contain a large concentration of vacancies by comparison with melt-grown crystals.

A factor which must limit the range of materials to which the SEBAT technique is applicable is electron bombardment damage, since the operating conditions required involve relatively large beam currents and accelerating voltages. However, the electron bombardment is by no means as severe as in transmission electron microscopy where higher beam voltages, larger current densities, and specimens of much smaller thermal and electrical conductances are used. Thus, whilst transmission specimens of ZnS and CdS sublimate unless the beams are carefully controlled [8], no damage was observed in making SEBAT patterns from the same materials. To test the extent of radiation damage more critically a crystal of NaCl was examined and it was found that SEBAT patterns could be obtained from fresh areas, and previously scanned areas no longer gave the patterns. Brown discolouration was found in the irradiated areas. This is the characteristic colouration due to large numbers of charged F centres. That this form of damage prevented subsequent observation of SEBAT patterns supports the view expressed above regarding the

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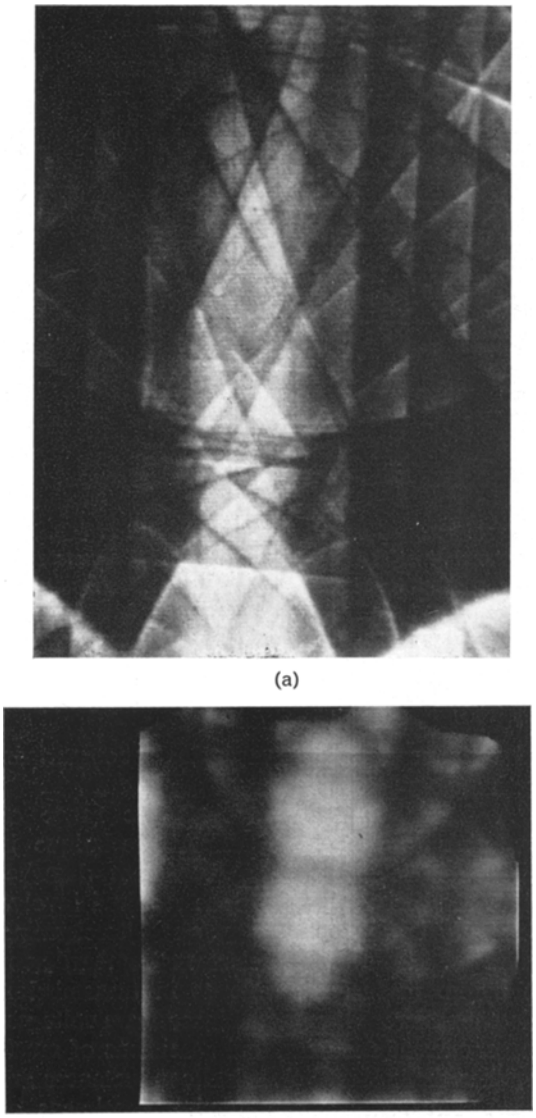

(a)

(b)

Figure 3 SEBAT patterns, 45 kV ("90 ×"). (a) (311) silicon surface; (b) (112) germanium surface.

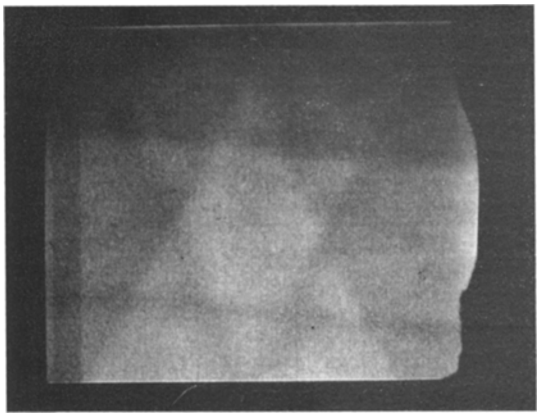

Figure 4 SEBAT pattern from a cleavage face (0001), of biotite mica; 35 kV ("70 ×").

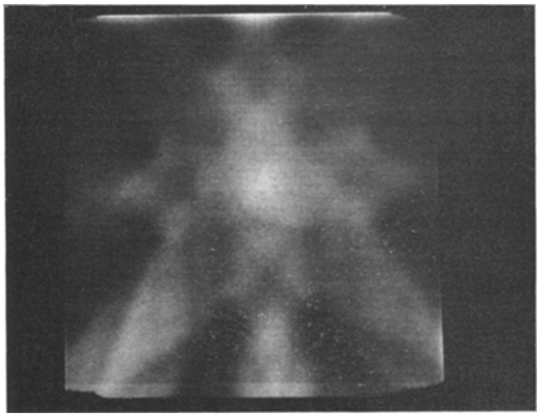

Figure 5 SEBAT pattern from a (100) cleavage face of NaCl; 35 kV ("70 ×").

sensitivity of the patterns to large point defect concentrations.

Surface preparation also influences the patterns obtained. In the case of CdS it was found to be impossible to obtain SEBAT patterns from mechanically polished surfaces whereas chemically polished surfaces gave clear patterns. Patterns were also obtained from cleavage faces (e.g. mica and PbS) and from as-grown faces (epitaxial silicon). Thus any preparation technique that results in a flat surface without gross mechanical damage or contamination was found to be acceptable. The sensitivity to damage is consistent with the discussion, given above, regarding the influence of defects. In the case of mechanical damage the defects involved are dislocations and microcracks. Some surface damage is tolerable however for Coates [1] succeeded in obtaining SEBAT patterns from mechanically polished (111) surfaces of GaAs. Specimen preparation and handling is thus much less exacting than in the case of transmission electron microscopy. Suitable chemical etches and polishes are available for most metals, alloys, and semiconductors, and for many ceramics. In the case of minerals, however, specimens have generally been mechanically polished, and little has been done to develop polishing reagents.

Cleavage faces of galena (mineralogical PbS) and of synthetic, semiconductor quality, PbS were examined. It was found that the patterns from the synthetic PbS were rather poorer than those from galena, and that in both cases the material rapidly altered during examination, so that patterns could no longer be obtained. It is thought that this is due to the chemical reactivity of PbS which, under the influence of electron bombardment, combines with one or more of the constituents of the residual gases in the microanalyser. This constitutes an additional type of limitation on the range of materials to which the technique is applicable. This type of limitation will be less restrictive when ultra-high-vacuum stages for scanning electron microscopes become available.

As a result of the work just described it is clear that the SEBAT technique is applicable to a considerably wider range of materials than is transmission electron microscopy. This is the result of (i) the reduction of the electron bombardment damage, so that, for example, the alkali halides can be examined, and (ii) the removal of the requirement of a thinning technique so that difficult-to-thin multicomponent materials, and high temperature materials, such as refractory ceramics, can be examined.

The discovery of SEBAT patterns by Coates [1] is important because, for the first time, it makes available from scanning-electron-beam instruments crystallographic electron diffraction information. Experience of transmission electron microscopy has demonstrated the importance of the combination of micrographic and diffraction information for unambiguous interpretation. The number of types of scanning-electron-beam micrography to which SEBAT patterns may be related is large. This is because incident electrons may excite a large number of physical effects, each of which may be separately detected, amplified and displayed as video signal on the synchronously scanned CRT. In this way micrographs may be produced that are based on (i) fluorescent X-rays in the case of microanalysis, (ii) scanning-electron-beam-excited recombination radiation (infrared or visible) in the case of the SEBERR technique [4], (iii) scanning-electron-beam-excited charge collection by contacts to the n- and p- sides of a semiconducting specimen containing a p-n junction in the case of the SEBECC technique [5], (iv) specimen current to earth, or (v) secondary and/or high-energy backscattered (primary) electrons, in the case of the standard scanning electron microscope (SEM) micrographs, and of SEBAT patterns. The main use of the information provided by SEBAT patterns is likely to be for the interpretation of contrast in the above-mentioned types of SEM micrography.

4. Interpretation

The geometry of SEBAT patterns from massive crystals and of transmission Kikuchi patterns from thin crystals in parallel orientation is the same [3]. Therefore SEBAT patterns may be analysed by the same geometrical methods already developed for Kikuchi patterns [2], or simply identified by comparison with Kikuchi maps [6].

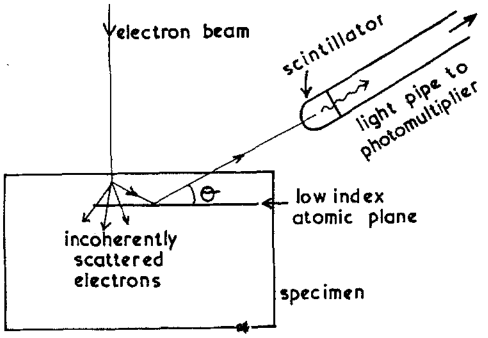

Figure 6 Geometry of the Kikuchi mechanism of double incoherent-coherent scattering of electrons into the detector. This mechanism does *not* contribute to SEBAT patterns.

It is necessary to emphasise that SEBAT patterns are however *not* Kikuchi patterns. Kikuchi patterns are produced by double diffraction: first incoherent and then coherent (Bragg) scattering. This double diffraction would necessarily have to take place in the geometrical arrangement in the Geoscan microanalyser as shown in fig. 6. By comparing figs. 2a and 6, the difference in geometry between the two mechanisms can be seen to be that anomalous transmission involves atomic planes normal to the specimen surface, or more generally taking into account the possibility of tilting the specimen, parallel to the undeflected electron beam, whereas Kikuchi double diffraction involves planes approximately parallel to the specimen surface, or generally, normal to the undeflected beam. Thus, on the anomalous transmission interpretation, the pattern observed ought to correspond to the symmetry of the axis parallel to the undeflected beam, and when the specimen is rotated, it ought to simply rotate about its centre. This is observed to happen [1] e.g. from figs. 1a-c. On rotating the specimen about the electron beam, the Kikuchi patterns would change as one crystal axis after another was brought into the azimuth from the point of incidence of the beam toward the electron detector. No such effect occurs. Therefore the patterns observed are not Kikuchi patterns. It is also important to emphasise that SEM micrographs are not real images. There is no position at which a fluorescent screen could be placed so as to produce the observed patterns. The patterns are point-by-point displays of the strength of signal produced as the electron beam falls in particular directions on to the specimen. The positioning of the detector is insignificant. There is no foreshortening of the patterns due to the "viewpoint" of the scintillator being near the specimen surface, and no change in the pattern due to moving the specimen up toward the scintillator. The micrographs are oriented as if "seen" in the direction of the undeflected beam.

5. Summary and Conclusions

SEBAT patterns are obtainable from a wide range of materials: semiconductors, metals, minerals and alkali halides. No difficult surface preparation is required. Electron bombardment damage is a less serious problem in scanning-electron-beam observations than in transmission electron micrography.

Sharp SEBAT patterns are indicative of crystalline perfection.

References

1. D. G. COATES, *Phil. Mag.* **16** (1967) 1179.
2. P. B. HIRSCH, A. HOWIE, R. B. NICHOLSON, D. W. PASHLEY, and M. J. WHELAN, "Electron Microscopy of Thin Crystals" (Butterworths, London, 1965) pp. 204-447.
3. G. R. BOOKER, A. M. B. SHAW, M. J. WHELAN, and P. B. HIRSCH, *Phil. Mag.* **16** (1967) 1185.
4. H. C. CASEY, *J. Electrochem. Soc.* **114** (1967) 153.
5. D. B. HOLT, and B. D. CHASE, *J. Materials Sci.* **3** (1968) 178.
6. E. LEVINE, W. L. BELL, and G. THOMAS, *J. Appl. Phys.* **37** (1966) 2141.
7. B. A. UNVALA, D. B. HOLT and M. O'HARE (1968) to be published.
8. D. B. HOLT, R. PORTER, and B. A. UNVALA, *J. Sci. Inst.* **43** (1966) 371.