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## The rotation between selected area channelling patterns and micrographs in the SEM

Selected-area channelling patterns (SACPs) are now in wide use as a means of obtaining detailed crystallographic information from solid specimens in the scanning electron microscope (SEM) [1]. The best technique for generating these patterns is that described by van Essen et al. [2, 3]. In their method, the incident beam is made to rock about the selected area by deflecting the beam off-axis with a single set of scan coils, and then using the probe forming lens to bring the deflected rays back on to the axis. With this technique the minimum area from which a pattern can be obtained is set by the spherical aberration of the lens and the angle through which the beam is rocked. Areas as small as 1 µm diameter, for a total rock angle of 10° are attainable [4, 5].

When such patterns, generated by the deflection focusing technique, are used for crystallographic orientation determinations, it has been usually assumed that there is no relative rotation between the scan axes of the SACP and those of the normal micrograph. Such a rotation might be expected by analogy with that observed between diffraction patterns and micrographs in the transmission electron microscope. The rotation of the diffraction pattern is caused by the change in the intermediate lens excitation needed to go from diffraction to micrograph operation. In the SEM, however, the change from micrograph to SACP is made by simply switching off one set of scan coils. The lens excitation remains unchanged, and no apparent rotation would, therefore, be expected.

However, recent experimental work has established that, when this system is used on the Cambridge "Stereoscan", there is a rotation. Received 30 September and accepted 16 October 1974

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This is made up of two components. The first is an inversion of  $180^{\circ}$ , and is a consequence of the change in scan direction (but not in scan angle) which occurs at the second scan pivot point. Both normal and selected-area channelling patterns are, therefore, inverted with respect to the micrograph. (The existence of this effect can readily be observed by noting that surface detail and channelling lines move in opposite senses during a rotation about the optic axis.) The second component is a variable rotation dependent on the working distance. Although this has been noted previously [6], no figures have been given for its magnitude. This can be measured in the following way. The SEM is set up in the electron-optical conditions used for SACPs. That is, a probe diameter of about 0.5 µm, a beam collimation of about  $3 \times 10^{-3}$  rad and an incident current of at least 10<sup>-9</sup> A. A large single crystal (such as silicon) is then observed at  $\times 20$ magnification. A conventional topographic image will be produced, together with an electron channelling pattern. With the specimen set normal to the optic axis of the microscope, the specimen is rotated so as to line up a band on the pattern with a line on the display screen (e.g. a "line-set" scan). The SEM is then switched to the SACP mode. The angle through which the specimen must now be rotated to bring the chosen band parallel once again to the line on the display screen is the required angle of rotation between the SCAP and the micrograph. Fig. 1 shows the result of this measurement as a function of the excitation of the final lens. The rotation is in a clockwise sense between the SACP and micrograph, and must be added to the 180° inversion.

This effect arises from the presence of stray fields in the back bore of the lens. The ray paths traversed by the beam through this region are different in the SACP and micrograph conditions.



*Figure 1* Experimental measurement of rotation between SACP and micrograph for S2 and S4-10 Stereoscans at 30 kV.

The net rotation through the lens will, therefore, be slightly changed, by an amount depending on

the form of the field distribution. This might be expected to differ from one instrument to another, but measurements made on three "Stereoscans" (two mark 2 and one S4-10) gave similar results at 30 kV.

When two orientations are being compared (as for example across a grain boundary) these rotations are of no significance. But when accurate absolute determinations of orientations are being made, or when crystallographic directions in the specimen surface are being found, the rotations must be taken into account.

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## Hertzian fracture of glass ceramics

Whereas many studies have been made of the formation and structure of a vast range of glass ceramics in recent years, comparatively little attention has been devoted to the fracture properties. Nevertheless, it has become clear [1-3] that microstructural parameters can assume a controlling role in the fracture of this type of solid. Basically, the microstructure can affect the material strength in two ways: first, it can determine the size of incipient surface flaws from which catastrophic cracks may initiate; second, it can provide "obstacles" to the passage of a "well developed" crack, thereby influencing the fracture surface energy. Of these two factors, it is the second which characterizes more the underlying fracture processes of a given material.

Studies of the mechanics of Hertzian fracture [4, 5] show that the "critical load" (the load at which a cone-shaped crack suddenly forms just outside the circle of elastic contact between spherical indenter and flat specimen) provides a measure of the fracture surface energy under conditions of crack equilibrium. Moreover, the test is reasonably insensitive to any variations in the elastic properties of the material (as long as the stiffness of the specimen remains small in comparison to that of the indenter) or in the state of microstructural flaws. The need to