Letters

X-ray diffraction from micro-crystals in scanning electron beam instruments

Kossel X-ray diffraction, using a focused electron beam to generate a divergent source, is a very useful method of studying the crystallography of micro-crystals in polycrystalline specimens. A single pattern contains complete information about orientation, lattice constants, plane spacings, crystal type and symmetry. There have been several applications of this method to solve problems in physical metallurgy [1-3]. In the opinion of the authors the use of the technique has not been as widespread as it could have been because of the shortcomings of the camera designs which have interfered with the main use of the electron beam instrument used to generate the pattern. For this reason Vieth and Yakowitz [4] have produced a Kossel pattern generator to be used just for this purpose. A design of camera is also required which can be incorporated into electron optical instruments, e.g., scanning microscopes and microanalysers so that Kossel patterns could always be recorded without affecting the main use of the instrument. It is desirable that the design should provide for rapid interpretation of the Kossel patterns and it should enable patterns to be obtained either in reflection or transmission. The reflection mode is consistent with the use of scanning instruments to study bulk specimens.

The constraints on the design of Kossel cameras in the past have been due to the use of methods of interpretation which have needed a prior knowledge of the distance between the specimen and the film and of the pattern centre. The pattern on a flat film is a gnomonic projection of circles which lie on the surface of a sphere. The source is the centre of the sphere and the pattern centre is therefore the projection of this point onto the film and is the centre of the projection. The pattern centre has usually been defined by centring the film on the electron beam. Developments in interpretation techniques [5-7] allow these requirements to be relaxed and enable orientation and plane spacings to be obtained without knowing the specimen-to-film distance and the pattern centre. It has also been realized recently that the Rowlands and Bevis chart method [8], which is the most rapid method of interpreting Kossel patterns, while originally designed for beam-centred cameras, can be used when the pattern centre is not known. The only pre-requisite for the use of the charts is that the source-to-film distance should be known and that it should remain constant.

The design requirements outlined above can be met by the camera illustrated diagrammatically in Fig. 1, which is the subject of a Patent Application [9]. The flat film is mounted away from the electron beam axis and parallel to it so that if the specimen is tilted, or moved while it is tilted, the perpendicular distance from the film to the X-ray source will be unchanged. The specimen holder can be rotated about an axis parallel to the film but perpendicular to the electron beam so that either back reflection or transmission patterns can be recorded with the same film position. The tilted specimen is in such a position that the solid angle of the pattern includes the region near the specimen horizon, which can be important when evaporated layer targets are used in back reflection. With the provision of a suitable X-ray window the film can be outside the main instrument vacuum. If this camera were fitted to a scanning electron

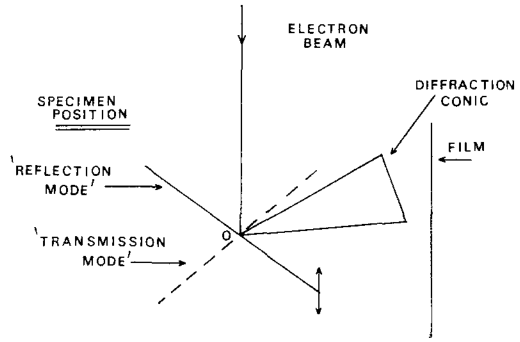

Figure 1 Schematic diagram of the proposed design of a Kossel X-ray diffraction camera. The positions of the specimen for reflection and transmission modes of operation are represented by continuous and broken lines respectively. The film position is the same for both modes of operation and is parallel to the optical axis of the instrument. The pattern may be interpreted without a knowledge of the pattern centre thus allowing movement of the specimen parallel to the electron beam, as indicated by the vertical arrow. The specimen may be moved in all directions normal to the optical axis allowing an area of interest to be located and the diffraction pattern from that area to be recorded.

microscope, then the film would occupy the position normally filled by the electron detector viewing the surface at 45°. This apparent conflict need not cause any serious difficulties and there are two possible solutions. The size of features which would be examined by Kossel microdiffraction (> 2 μm) means that conducted electron imageing would be a satisfactory mode of operation for most purposes and the detector could be removed. A better solution would be to re-arrange the relative positions of the specimen and detector so that while the surface was tilted towards the film in the manner shown in Fig. 1, the detector viewed the surface along a line which did not intrude into the space occupied by the solid angle subtended by the film. This would retain normal viewing. It may be necessary to increase the working distance of the final lens in order to provide a large enough solid angle for the pattern and to accommodate the changes indicated above. Since the electron image requirements are not severe this should not be a problem. The beam will usually be stationary for generating the pattern and, hence, it should in principle always be in the same position in the scanning frame. This restriction applies only to the use of charts to interpret the pattern. In practice the chart line curvatures are not sensitive to such small changes as would be caused by a movement of the beam within the scanning frame.

The main practical difference between this design and electron beam-centred cameras for reflection patterns is that, for a given film size, the solid angle at the film is dependent both on the working distance of the final lens and on its shape. In beam-centred cameras the solid angle depends only on the working distance, unless the film is positioned at the back of the lens.

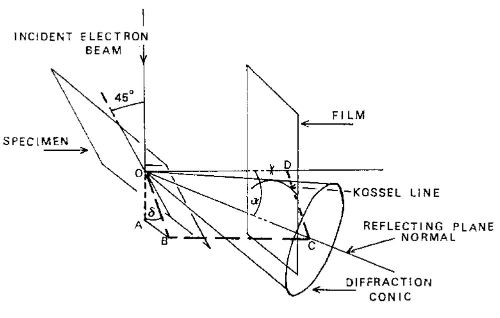

Figure 2 Schematic diagram showing the relationship between a Kossel line formed on a film and the orientation of the associated diffraction conic.

The patterns obtained with this camera can be used to determine crystal orientations and relative orientations, using the method of Ryder, *et al* [5] or to determine plane spacings and orientation by the method of Harris and Kirkham [7]. Indexing, orientations and orientation relationships can also be determined with the use of Rowlands-Bevis charts and since this procedure is simple, rapid and accurate [10] its adaptation to this camera geometry is worth describing in detail.

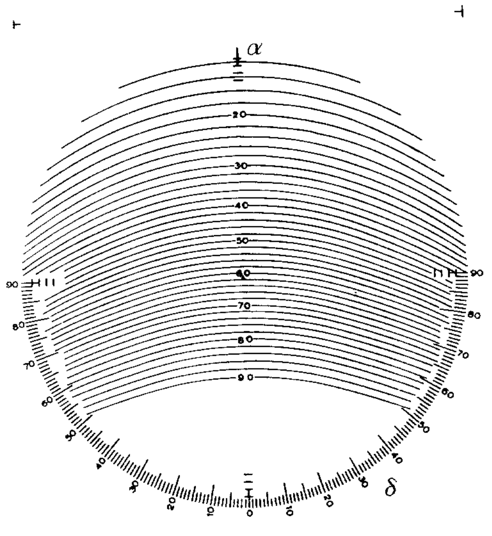

Figure 3 A Rowlands-Bevis chart [12] for indexing Kossel lines associated with a diffraction conic of semi-apex angle of 62°. The ratio of the chart diameter to the specimen-to-film distance is 2.5.

Fig. 2 shows the relationship between the Kossel lines formed on a film and the diffraction conics of semi-apex angle $\phi = (90 - \theta)$ where θ is the Bragg angle for the reflecting plane. The cone axes are normal to the diffracting plane in the crystal and are defined with respect to a reference basis in the film by the angular co-ordinates α and δ. If the angle α were varied while keeping δ fixed, a series of line positions would be obtained. The appearance of the line would change and in the series of positions, would be as represented on the chart shown in Fig. 3. This chart is for a cone with a semi-apex angle of 62° and a source-to-film distance of 4 cm. The source-to-film distance is the length of the normal from the film to the source. In use the chart is rotated about the pattern centre θ until a line on the chart coincides with a line on the pattern. The co-ordinates α and δ are then read off directly. If the centre of the pattern needs to be established then, from the geometry of the projection, it must lie at the intersection of the major axes of the ellipses which the Kossel cones form on the film. When the chart is oriented correctly on a line, therefore, the central axis of the chart will lie along the major axis of that ellipse. The cone angle of the line can also be used for indexing the corresponding plane if the crystal is known and with low symmetry crystals the extra information from the angular co-ordinates of the plane normal is invaluable for indexing purposes. The accuracy of orientation determination is not very sensitive to the cone angle and $\pm 1^\circ$ can be tolerated in the fit. The fitting procedure is easiest between the range of Bragg angles from 25° to 60° and hence with charts constructed at 2° intervals the number of charts required for the orientation determination of all crystals types would be less than 20. In particular, since the source-to-film distance is fixed in this proposed design only one set of charts would be needed to index and orientate accurately all crystals. In practice the orientation of a known crystal can be established from the original pattern in approximately 5 min.

This proposed design of camera takes full advantage of the fact that the generation of a Kossel pattern is independent of the electron beam direction and utilizes the capability of modern interpretation procedures to deal with patterns where the centre and the source-to-film distance are not known in advance. The position of the film, off the electron beam axis, should not interfere with other uses of the system and the camera could therefore be a permanent attachment to an electron probe microanalyser or scanning electron microscope. The design could serve equally well as the basis of a relatively low cost instrument which would be used primarily for the generation of back-reflection and transmission Kossel patterns.

The Kossel technique provides a comprehensive diffraction system and in the opinion of the authors, the widespread use of such a system is justified by the value of the results obtained by its application. A full description of the practical procedures to be followed in order to obtain good Kossel patterns is being prepared for publication [11].

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M. BEVIS
N. SWINDELLS
Department of Metallurgy and
Materials Science,
University of Liverpool,
Liverpool, UK

Friction changes in ion-implanted steel

We wish to report a change in the frictional properties of an En steel implanted with various metallic and non-metallic ions using a 500 kV accelerator at AERE, Harwell. To our knowledge, this is the first reported case of a significant mechanical effect brought about by the ion implantation of various elements into an industrial steel.

Ion implantation may be described as the projection and deposition of atoms by the bombardment of a solid substrate. In conventional implantation equipment a beam of ions is accelerated across a high potential to strike a solid target at the end of a vacuum tube. Pene-