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Selected Area Channelling Patterns in the Scanning Electron Microscope

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A technique is presented and illustrated for generating orientation dependent electron channelling patterns in the scanning electron microscope from small selected areas on the surface of bulk specimens. Methods of reducing the pattern area to 1 to 2 μm in diameter are discussed. Numerous applications not previously possible are listed.

1. Introduction

The selected area electron diffraction technique in transmission electron microscopy has had no equivalent in scanning electron microscopy. This has prevented using the scanning electron microscope (SEM) for crystallographic analysis. The recent observation [1, 2] of orientation dependent electron channelling patterns on bulk single crystal specimens in the SEM has only partially removed this limitation, because the area over which the patterns are generated is at least 1 mm across. A method for generating patterns from small selected areas was previously mentioned by the authors [3] and has now been developed into a practical system. The purpose of this paper is to describe the technique and to suggest possible applications.

2. Pattern Generation

Channelling occurs when an electron beam is incident upon a given set of crystal planes in directions close to the Bragg angle [2]. Patterns are detected by collecting the reflected primary and/or secondary emitted electrons, or by using the specimen current, and arise from the changing direction of incidence of the beam at the surface of the crystal. In the standard scanning operation of the Cambridge Scientific Instrument Co Ltd "Stereoscan" SEM, the beam is deflected in two mutually perpendicular directions (x and y) by a double-deflection system such that the scanning crossover lies just above the final lens. The beam passes through a small final aperture (typically 100 μm diameter) at the centre of the lens and scans an area on the specimen surface some 10 to 20 mm below (fig. 1e). The variation in direction of incidence occurs over the scanned surface, which is typically 5 mm square for the largest scanning angle ($\sim 15°$, obtained at $\times$ 20 magnification setting) where identifiable patterns are most readily obtained. By attenuating the current in the lower coils of the double-deflection system, the scanning crossover point may be brought down to the specimen surface. In this case the variation in the direction of incidence is slightly reduced, and occurs about a "point" the size of which is determined by the size of the probe. Contrast arises from the variation in direction of incidence only. This, therefore, allows channelling patterns to be generated from small selected areas.

3. Practical Details

With the modified scanning system the diameter of the final aperture must be increased: for instance, if the crossover is lowered $\sim$ 10 mm, the diameter should be $\sim$ 2.5 mm. This prevents the beam from being wholly or partially cut off at the extremes of its $\pm$ 7° deflection, and keeps the illuminated spot stationary.

In the selected area channelling technique the probe size must be as small as, or smaller than, the area of interest, and the beam divergence must be sufficiently low to permit good detection and angular resolution of patterns. At the same time the beam must be of sufficient current at the specimen surface so that the signal-to-noise ratio is high enough to observe the low-contrast channelling effect. The current acceptable depends on the amplifying system used. A compro-

mise must be established, and some possible values of the quantities are given in table 1 with the corresponding lens settings. These settings give rise to a narrow beam (i.e. 100 to 200 μm diameter) entering the third lens. The divergence has been calculated by estimating the diameter of the beam at the final lens (i.e. close to the final aperture) and dividing by the specimen-aperture distance. It is apparent from the table that a probe of diameter 1 or 2 μm has a low current and high divergence. Successful generation of patterns from such small areas will thus probably require the use of a pointed tungsten filament or lanthanum hexaboride cathode [4] and/or a pulsed-beam/phase lock detector system. Signal processing using a differentiating amplifier [5] or a deflection modulated display [6] should also improve both pattern detection and angular resolution.

4. Limitations

The spot sizes given in the table ignore the effects of aberrations such as astigmatism, or spherical aberration, which may arise from the greatly enlarged final aperture. Experiment shows that the settings for the smaller spot sizes do not give the resolution expected, and this is attributed to astigmatism. It is thought that the spherical aberration is not significant for spot sizes above ~ 2 μm.

Smaller areas might be selected by positioning an aperture just above the specimen, analogous to the selected area procedure in transmission electron microscopy. In this case it would probably be advantageous to use the specimen current for pattern detection.

5. Operation

In operation a lens-current attenuator is inserted into the lens 2 circuit since the current required is less than the normal minimum. Another attenuator which selects "standard scan" (as in fig. 1e) or "selected area" (as in fig. 1f) is inserted into the scanning circuit. When "selected area" is chosen, a series/shunt pair of resistors is switched into each lower scanning coil circuit, x and y. Pure resistances can be used as the reactive impedance of the coils in the "Stereoscan" at scan frequencies is negligible. The specimen is adjusted so that its surface is in the same plane as the lowered scanning crossover point, and is then observed under "standard scan" with one of the lens conditions listed in the table. The area to be examined is brought to a predetermined position on the viewing screen. On switching to "selected area" a channelling pattern from the selected area appears on the screen and may be photographed. The exact point on the screen corresponding to the selected area must be determined: it need not be the centre.

To illustrate the technique, patterns were obtained from areas 50 to 100 μm across on various grains of an annealed copper polycrystalline bulk specimen (fig. 1a). Three of the patterns are shown in fig. 1b, c and d. The pattern obtained from each grain is different and may be used to determine the crystallographic orientation of the individual grains. The indexing of channelling patterns has been discussed in a separate paper [7]. The reflected primary and secondary emitted electrons were collected, and the signal was amplified by a DC-coupled amplifier. That patterns did, in fact, come from the areas indicated was established by translating the specimen under the beam, noting points when the pattern changed, and comparing these with the positions of grain-boundaries seen on "standard scan".

The lens settings used were as shown in table 1, number 1. Other conditions for smaller spot sizes, using lens 3, were also tried, but the crossover area was found to be larger than the probe size. This discrepancy is being investigated.

6. Specimen Preparation

To date, patterns have been generated from

TABLE I Spot size, divergence and beam current at the specimen surface in the "Stereoscan" for various lens settings

	Approx. lens currents (A)			Spot size	Divergence	Rel. beam
	1	2	3	(μm)	(rad.)	current
1	0.2	0.025	0	50-100	3×10^{-3}	1
2	0.2	0.02	0.4	20-40	5×10^{-3}	1
3	0.5	0.08	0.7	10-20	5×10^{-3}	0.1
4	1.0	0.06	0.7	5-10	5×10^{-3}	0.01
5	1.0	0.07	0.6	1-2	10^{-2}	0.01

For final aperture diameter 2.5 mm, aperture-specimen distance 10 mm and standard tungsten hair-pin filament. Lens 1 without pole-piece, lens 2 with pole-piece.

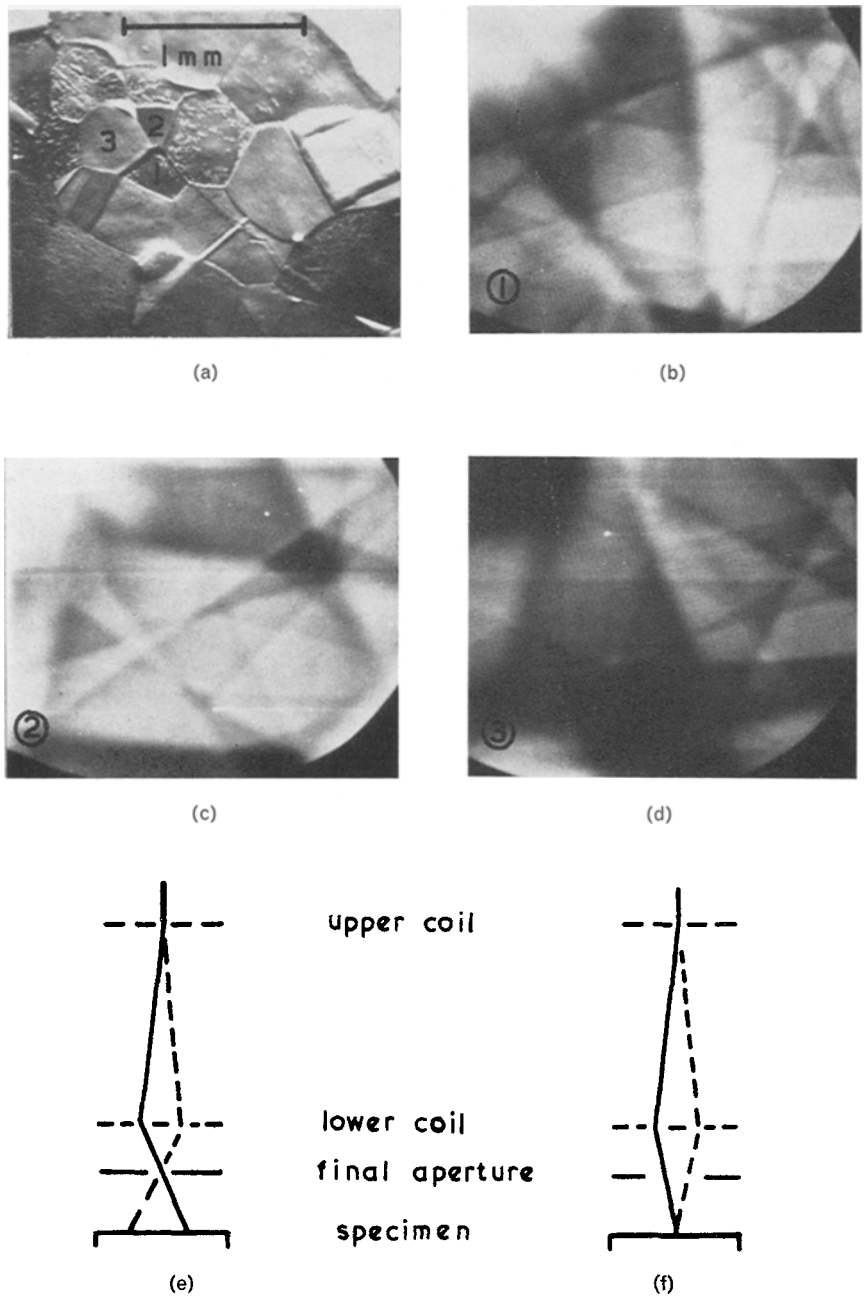

Figure 1 a Microstructure of annealed copper polycrystalline specimen, seen under "standard scan"; b, c, d selected area channelling patterns (SACP) from areas 1, 2 and 3 respectively of the specimen, seen under "selected area"; e ray deflection diagram for the "Stereoscan" under "standard Scan"; f ray deflection diagram for the "Stereoscan" under "selected area".

various metals, semiconductors, and ionic and covalently bonded solids. If the surface is mechanically damaged or coated, for example with an oxide film, pattern contrast is usually reduced. However, patterns can often still be obtained and identified.

7. Applications

The scanning electron microscope is rapidly becoming a valuable instrument for the examina-

tion of materials. The main reasons for this are that bulk specimens can be examined with little or no preparation, that resolution is relatively good (for commercially available instruments $\sim$ 200 Å), and that depth of focus is extremely large. The ability to obtain selected area electron channelling patterns, i.e. to obtain specific crystallographic information from any particular small area of the specimen, enables the range of application of the SEM to be appreciably extended. Some applications not previously possible are as follows:

(i) Crystallographic orientation of very small crystals, of individual grains in a polycrystal, and of eutectic plates and rods.

(ii) Determining the crystallographic misorientation across grain-boundaries, sub-grain-boundaries, deformation bands, kinks, microcracks, etc.

(iii) Identifying precipitates and inclusions, and determining their crystallographic orientation with respect to the matrix.

(iv) Locating the positions of grain-boundaries, sub-grain-boundaries, twins, transformed regions, etc on surfaces which are unetched.

(v) Recovery, recrystallisation and grain growth studies using a hot stage.

(vi) Phase change and martensitic transformation studies using a hot or cold stage.

(vii) Deformation studies, e.g. slip, twinning fatigue and fracture, using a straining stage. Surface strains might be measured by the channelling line broadening.

It is further suggested that if the angular resolution of channelling patterns is high enough, changes in lattice parameter may be measured. It may also be possible to detect changes in the degree of long range order in ordering systems.

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