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SHORT NOTE

DIRECT OBSERVATION OF DIFFRACTIVE PROBE SPREADING

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Preliminary results for the diffractive probe spreading in a STEM are described. Clear evidence is found for two beam Bormann fan effects due to high order beams.

1. Introduction

One of the growing areas of electron microscopy is microanalysis, using the elementary characteristic loss processes of a swift (~ 100 keV) electron probe propagating through a specimen to determine local chemical compositions. In order to quantify the experimental results, it is essential to know what is the volume of the specimen which the electron probe sees, in particular the transverse spreading perpendicular to the incident probe direction. Over the last few years a number of authors have investigated the probe spreading in a STEM theoretically (e.g. ref. [1]). All these theories have employed a ballistic model for the electrons, i.e. classical hard particles which have a certain probability of being scattered as a function of distance. However, the failure of a ballistic model of electrons is well known, the superior model being Quantum Mechanics. For instance, ballistic models fail to represent diffraction correctly which is frequently the dominant scattering process for swift electrons.

A more appropriate analysis for the STEM probe spreading has been available for many years in the X-ray literature, the so-called spherical wave diffraction theory (e.g. refs. [2,3]) which grew out of the topography experiments of Kato and Lang [4] and the theoretical analysis of Kato [5]. The application of these ideas to electrons will be reviewed elsewhere [6] and is quite straightforward. In general, the n-beam dynamical dispersion surface is treated by standard optical dispersion techniques (e.g. ref. [7]), ray diagrams being constructed from the vectors normal to the dispersion surface. Analytical solutions are also available for some simple cases.

This note is a preliminary report of direct observation of diffractive probe spreading obtained by directly imaging the probe in a Philips 400ST FEG electron microscope. A more detailed description will be given elsewhere [8].

2. Experimental procedure

Experiments to look for the diffractive probe spreading were performed in a Philips 400ST FEG electron microscope. In nanoprobe mode, a probe can be produced of order 20 Å in diameter and imaged straightforwardly in TEM mode at high magnifications. The specimens used were $\langle 111 \rangle$ oriented sillicon single crystals, chemically thinned in a HF/HNO_3 solution and cleaned by standard semiconductor solvents immediately before use. The latter both reduced contamination problems and reduced the amorphous SiO_2 coverage to a thickness of 10–20 Å (determined by high resolution electron microscopy). To further reduce contamination, the specimens were cooled to liquid nitrogen temperatures.

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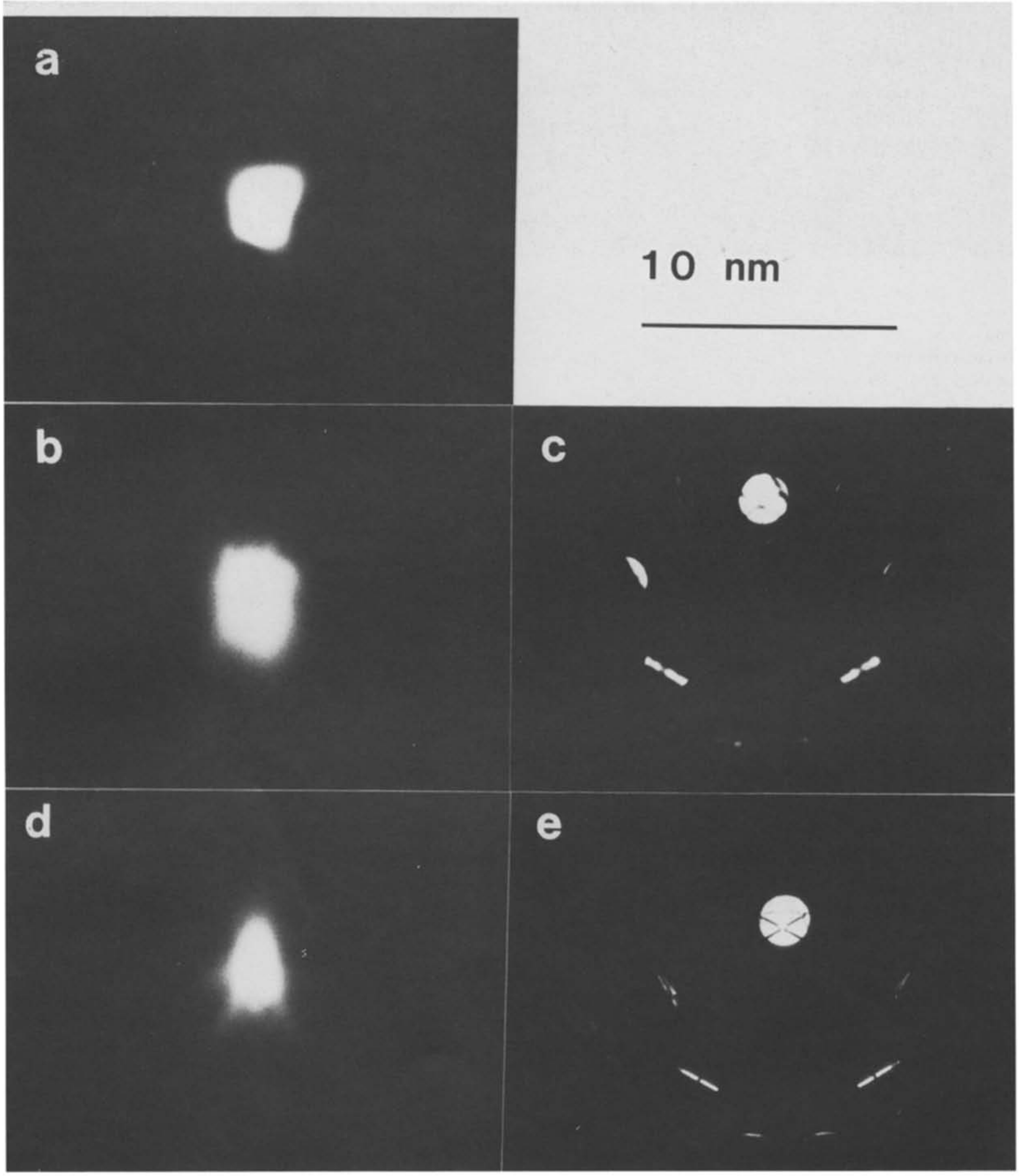
3. Experimental results

One problem with detecting the diffractive probe spreading is that the effects from low order diffracted beams is small, of order λgt, t being the thickness. Furthermore, λgt is an upper limit for



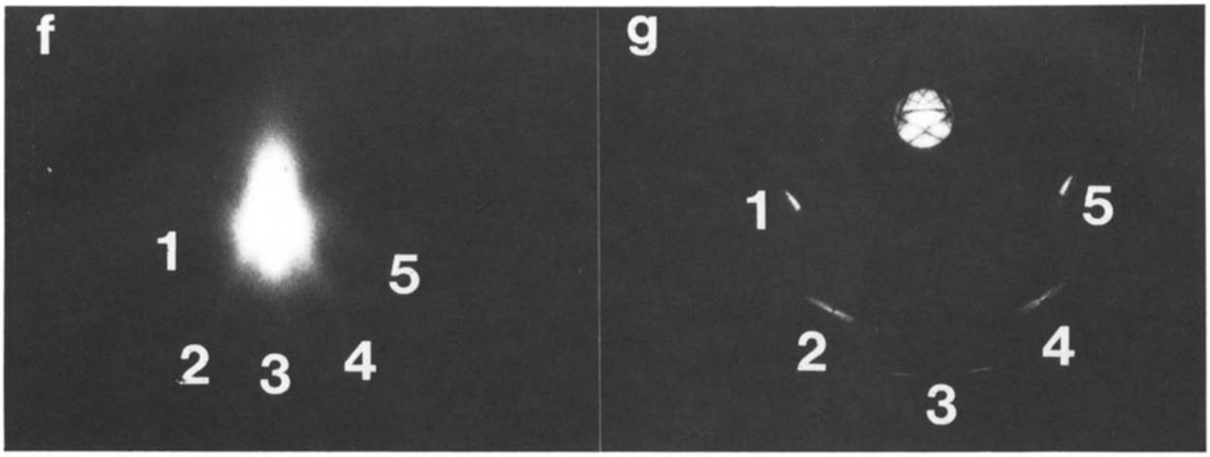


Fig. 1. In (a), real space image of the probe without a specimen. Figures (b)–(g) are image, diffraction pairs across the page, for increasing thickness down the page. All the image exposures are the same, so (a) and (b) are slightly overexposed and show a larger probe than was in fact present. The specimen is Si, near a (111) zone axis, at a temperature of −180°C. Note that the streaks (arrowed) are along the ***g*** diffraction directions as indicated in (f) and (g), and increase in length as the thickness increases. The diffracted beams have been excluded from the images by an objective aperture.

nearly free electron Bloch waves whilst most materials at 100kV are quite tightly bound (e.g. ref. [6]). (The spreading for tightly bound levels is substantially smaller since the dispersion surfaces are relatively flat.)

What could in practice be readily observed were effects from higher order spots – with a large converence angle it is essentially impossible to avoid exciting "stray" spots. A series of images showing the diffractive streaks from these high order spots for different thicknesses are shown in fig. 1.

4. Discussion

In the two-beam case, the diffraction is contained within a Bormann fan of length λgt directed along the ***g*** direction, *both* the transmitted and diffracted being so contained [6]. Dealing with the diffraction conditions as a set of simultaneous two-beam cases, theory predicts a set of streaks superimposed on the transmitted probe, the diffracted waves themselves being intensity streaks in the image plane. (In simplified terms the image can be considered as a Fourier transform of the diffraction pattern, lines as evident producing the streaks.)

The experimental results show precisely this type of streaking. This confirms the inappropriate nature of the accepted theories of STEM probe spreading, the preferred theory being based upon X-ray spherical wave theory [2–6].

Conclusions

Clear experimental evidence for Bormann fan streaking of a STEM probe has been obtained.

Acknowledgments

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References

[1] P. Rez, Ultramicroscopy 12 (1983–84) 29, and references therein

[2] L.V. Azaroff, R. Kaplow, N. Kato, R.L. Weiss, A.J.C. Wilson and R.A. Young, X-Ray Diffraction (McGraw-Hill, New York, 1974).

[3] Z.G. Pinsker, Dynamical Scattering of X-Rays in Crystals (Springer, New York, 1978).
[4] N. Kato and A.R. Lang, Acta Cryst. 12 (1959) 787.
[5] N. Kato, Acta Cryst. 14 (1961) 526, 627.
[6] L.D. Marks, in preparation.
[7] M. Born and E. Wolf, Principles of Optics (Pergamon, London, 1959).
[8] L.D. Marks, in preparation.