The Suppression of Contamination in the Convergent-Beam Electron Diffraction Camera

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It has been demonstrated that specimen contamination in convergent-beam diffraction operation can be prevented by maintaining the specimen temperature between −110 °C and −165 °C, without the use of especially high or clean vacuum conditions. At these temperatures, surface migration of molecules causing contamination is evidently inhibited. Precautions to prevent deposition from the vapour phase both before and after cooling are also required.

The technique of convergent beam electron diffraction, for which small areas of crystals (< 10 nm diameter) are irradiated, is generally limited by very rapid contamination build-up which can exceed 100 nm/sec in extreme cases. Even in systems which are fitted with anti-contamination devices, specimens maintained at room temperature are contaminated rapidly unless special procedures are adopted. Following the advice of Ennos [1] and Heide [2], Goodman and Lehmpfuhl [3] avoided contamination by passing a current through the supporting grid to bring the specimen temperature above 250 °C. This approach can introduce complications due to local fields and the elevated temperature limits the range of specimens which can be examined; however, it has proved very effective for lengthy convergent-beam investigation of substances which do not decompose or suffer phase changes when moderately heated under vacuum. The method is successful even in the absence of cold traps, since the residence time of hydrocarbons and other organic molecules on the hot substrate is very short.

While it has been demonstrated that contamination on substrates at or near room temperature can be suppressed under totally-clean vacuum conditions, it is essential that a pre-cleaned specimen is used. In this laboratory one of the convergent-beam electron diffraction cameras is a converted Elmiskop 1. This is fitted with a modified Mills-Moodie type stage [4]. These authors designed a specimen holder for contamination-free operation in high resolution electron microscopy over a wide range of temperatures, rather than for ultra-low specimen temperatures. Our need for a low-temperature convergent beam stage lead us to modify the original thermal design. With our instrument we have kept to the original Elmiskop 1 concept of working in a standard vacuum (5×10^{-7} Torr), with specimens which undergo no special pre-cleaning treatment. The convenience which this permits in specimen preparation and handling must, of course, not be achieved at the expense of contamination control. The approach adopted has been to cool the specimen substrate and traps with a view to suppressing both surface migration and deposition from the vapour. To this end the thermal insulation of the specimen and anti-contamination thimble and their cooling links was modified to permit cooling to between −100 °C and −175 °C, depending upon the type of specimen holder [5]. Before the specimen is chilled, however, the vapour pressure of contaminants in the microscope column is reduced by means of an auxiliary liquid nitrogen cooled trap.

Cooling of the specimen also has the advantage that cold crystals give better data than hot ones. Figure 1 is a boron nitride zone-axis pattern which, at low temperatures, shows in the central beam very fine upper-layer deficiency lines of the type previously reported for Si [6]. This observation for boron nitride is an exacting test since the individual perfect domains are extremely small. Precise focusing of the beam is required and the appearance of the lines is temperature dependent.

The method described is quite different from that of Raith [7], who also cooled both specimen and surround but relied on a delicate balance, at a particularly critical temperature of about −80 °C, between etching and contamination to keep his specimens clean.

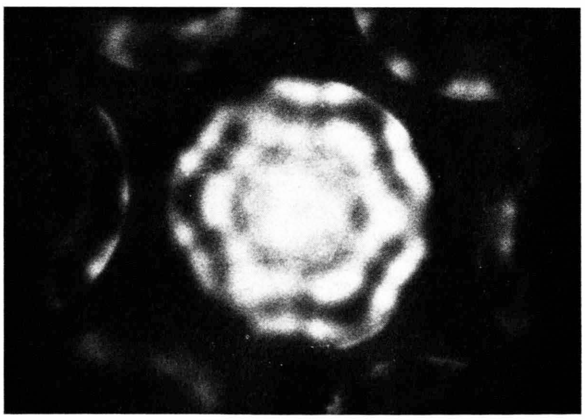

Fig. 1. Zone-axis convergent-beam electron diffraction pattern from hexagonal boron nitride at −110 °C.

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