

Early Use of Reflection Electron Diffraction in the TEM

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My TEM experience started nearly fifty years ago. I began in 1961 as a total novice when, as a fledgling graduate student in geology at Rice University, I inherited responsibility for a Shimadzu transmission electron microscope, perhaps the only Shimadzu TEM to come into this country. A previous student showed me the on-off switch and a few other basics and then dropped it and its

Microscopy (1953) was my only crutch. It took me three years of spare-time fiddling, but I finally got fairly adept at using this rather crude instrument. As an illustration of that crudeness, filaments were handmade from a 1000-foot coil of fine tungsten wire. A length of wire was bent into a V-shape by pressing it down over a single-edged razor blade. You can imagine that centering the beam from this filament was a tedious job. But, amazingly, it worked.

Eventually, I graduated to a more advanced instrument, a Hitachi HU 11A, at Pennsylvania State's Mineral Constitution Lab. Sample preparation was via carbon replicas or powder dispersions. Replicas were made in a vacuum evaporator by vapor-casting a few hundred Angstroms of carbon onto the sample surface, then shadowing the surface with platinum from a point source. Platinum-shadowed carbon replicas looked remarkably "life-like" in the TEM (for that time), and they could be faithful in resolution to about 25,000x.

Frequently some of the components of the sample would remain with the replica. For example, grain boundary precipitates could remain in place on replicas of metal samples. But if they didn't, it was also possible to make intentional "extraction replicas." It was these surface constituents that often provided suitable specimen material for electron diffraction, powder, or single crystal. These techniques allowed identification of very thin layers or small amounts of deposits without energy-dispersive x-ray spectroscopy, which had not yet been developed. Powder patterns were much the simplest, of course, being sets of concentric circles, the diameters of which were inversely

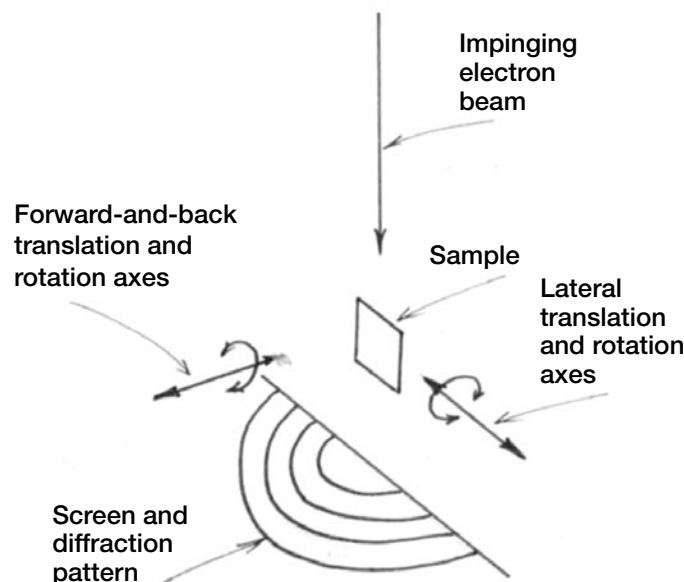
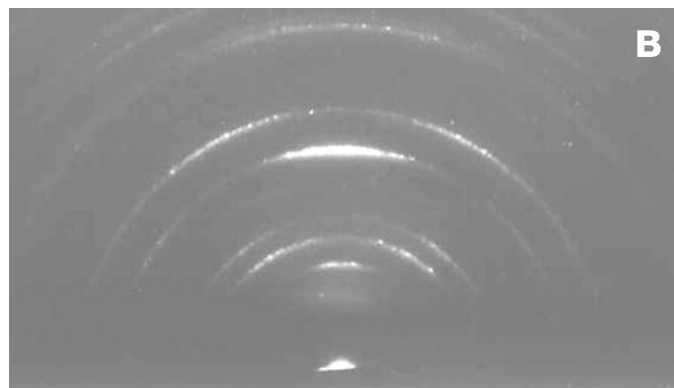
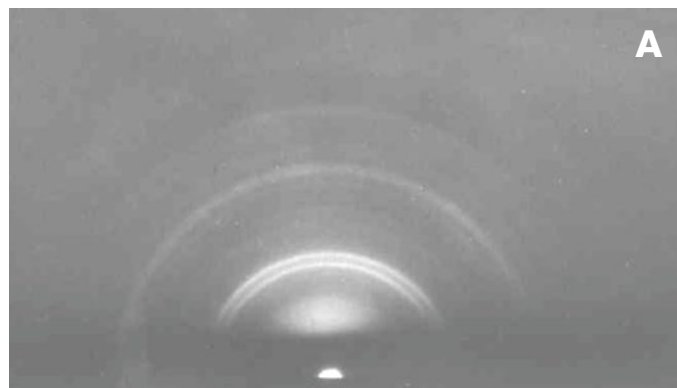
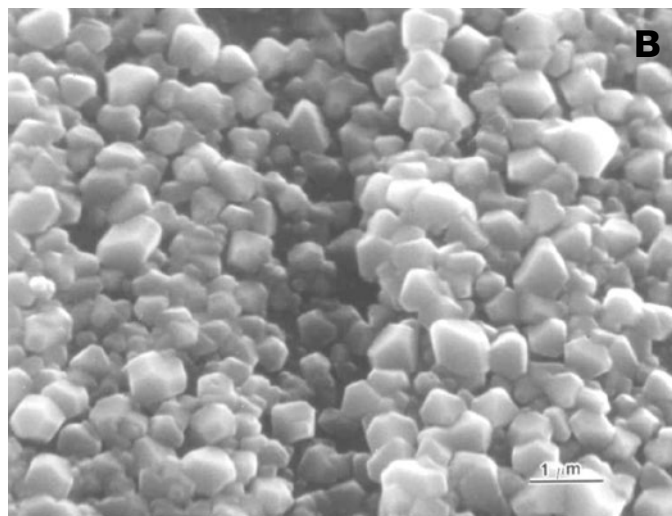
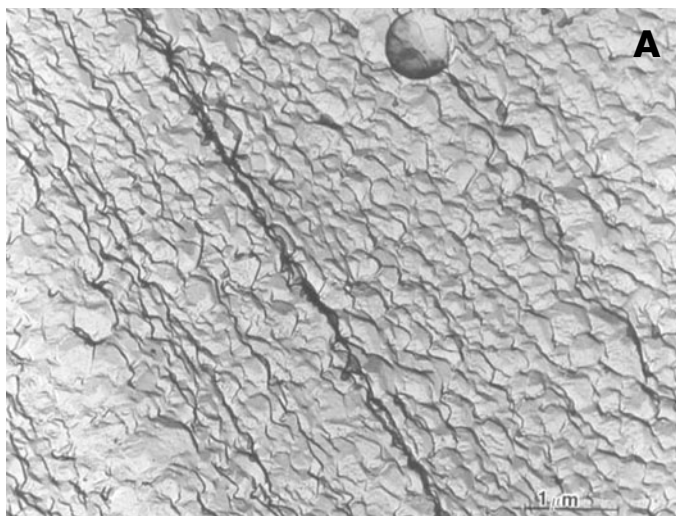


Figure 1: General diagram of reflection electron diffraction set-up.

pseudo-English instruction manual into my lap to figure out on my own. From that inauspicious beginning, microscopy became my life's work. Cecil Hall's *Introduction to Electron*



Figures 2: Reflection electron diffraction patterns from Cu_2O (A) and CuO (B), cubic and triclinic crystalline corrosion products on copper coupons.



Figures 3: (A) Platinum-carbon replica TEM micrograph and (B) SEM micrograph of cuprite, Cu_2O .

proportional to the d -values of the compounds.

An alternative to this was to use reflected, instead of transmitted, electrons. I learned this interesting technique on a Phillips 300 TEM. Delivered with that microscope was one of the few reflection diffraction stages in this country. It mounted low in the column above the final lens. Figure 1 is a crude diagram showing how it relates to the beam and the screen. The idea was to simply present the beam to the sample in grazing incidence and use the final lens to suitably enlarge the diffraction pattern. I could never understand why this technique did not catch on because it's far easier than struggling to get x-ray diffraction patterns from tiny samples.

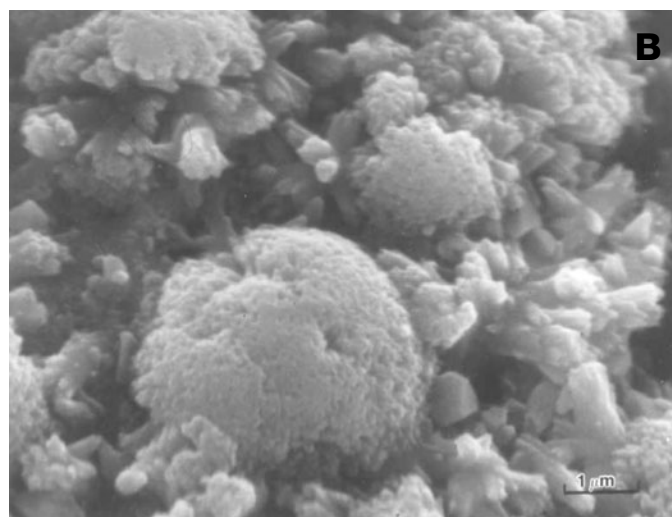
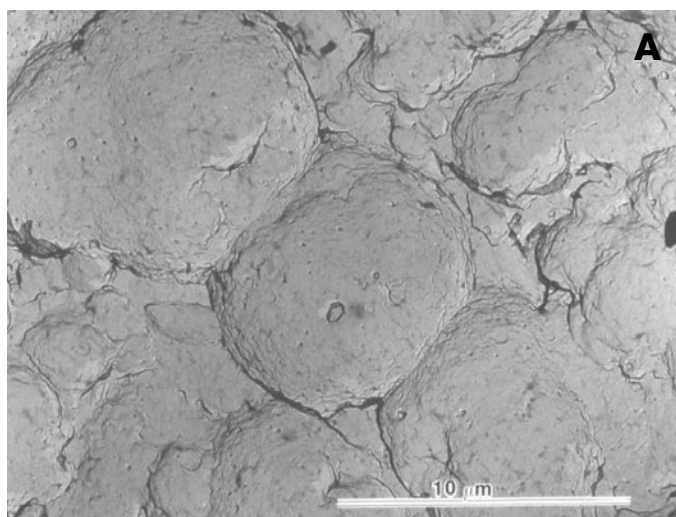
Figure 2 compares reflection electron diffraction patterns from cuprite (Cu_2O) and tenorite (CuO), the red and black copper oxides. For purists, the electrons aren't truly reflected. They pass through atomic-level asperities that exist on the surface of almost everything. The beam streams down the surface of the sample, partly passing through these little protrusions, and is diffracted when Bragg's law is satisfied. The d -values are quite low-precision because the pattern originates at different points down the

face of the specimen. But all this can be sharpened up if need be.

It is interesting to compare the carbon replica and SEM images of these copper oxides. Figure 3 shows replica and SEM micrographs of Cu_2O , and Figure 4 shows similar micrographs of CuO .

Electron diffraction did help my professional work. The only *Science* publication of my career came from transmission electron diffraction carried out on the high-resolution diffraction stage of that old HU11A. The venerable George Brindley, of clay-mineralogy fame, had me determine the lattice constants of a special form of kaolinite to three decimal places, far better precision than is usually possible by "selected area diffraction" in the normal sample position.

Editor: Reflection high-energy electron diffraction (RHEED) was revived in the 1980s and became the basis for the technique of reflection electron microscopy (REM) that can reveal fine surface steps and reconstructions. For more details see the special issue on REM in T. Hsu (ed.) J. Elect. Microsc. Tech. 20 (1992) part 4. MT



Figures 4: (A) Platinum-carbon replica TEM micrograph and (B) SEM micrograph of tenorite, CuO , corrosion deposit on copper coupon.