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DETERMINATION OF STRUCTURE FACTORS OF COPPER BY CONVERGENT BEAM ELECTRON DIFFRACTION.

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Cu Paper

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There has been considerable recent interest in the determination of structure factors from convergent beam electron diffraction (CBED) patterns and the ultimate goal is the ability to retrieve the crystal structure of an unknown crystal by inversion of a CBED pattern. There are a number of different methods that have been used to extract structure factor information¹⁻². The zone-axis pattern fitting technique of Bird and Saunders¹ has recently been used to obtain structure factors for silicon that compare well with those obtained by X-ray methods³. This work extends the techniques to f.c.c. metals, specifically copper.

CBED patterns were recorded from [110] zone axes of electropolished foils of pure copper (99.999% purity) in the Philips EM420T at Argonne National Laboratory. The patterns were energy-filtered by scanning the whole pattern across the entrance aperture of a Gatan #607 serial energy loss spectrometer and collecting the zero loss intensity only (energy window ~5eV). An EDAX PV990 computer was used to control the scans and digitize the patterns into 512 by 512 pixel data arrays with an intensity range of 12bits (4096 counts). The patterns were recorded such that the direct disc and the six lowest order reflections filled the field of view. An example diffraction pattern is shown in figure 1.

The data was processed at the University of Cambridge, using SEMPER running on a Silicon Graphics workstation, to extract 25x25 pixels regions from the centers of the seven low order discs. The intensities from these regions were then matched to calculated patterns at the University of Michigan on Hewlett Packard HP9000/710 workstations. The fitting process is a quasi-Newton method that is described in reference 1 and it proceeds by varying a set of parameters to minimize of the difference between a theoretical pattern and the experimental pattern. The fitting parameters include: a set of low order structure factors (both elastic and absorptive), the specimen thickness, a normalization coefficient and a number of background intensity values. For copper [110] the fit solves for 77 beams exactly and another 74 beams are added by perturbation theory. The initial starting estimate for the structure factors included real part elastic values of Doyle and Turner⁴ and the imaginary absorptive values from Bird and King⁵. At the start of the fitting process only the 002 and 111 structure factors were varied, to limit the possibility of the fit being trapped in a local minimum, and then, as the fit began to converge, the higher order structure factors were varied. Fine tuning of the fit was accomplished by variation of the Debye-Waller factor. Structure factors as fitted from patterns from two different sample thicknesses are shown in table 1. The {111} and {002} structure factors differ appreciably from the neutral atom values, while the differences for {220} and {113} are of the order of the estimated error. Figure 2, however, compares the experimental data from a 111 reflection with the computer generated fit and shows the high quality of the fit.

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Figure 1. Energy Filtered (zero loss only, $\sim 5\text{eV}$ window) Cu [110] Zone Axis CBED pattern. Recorded at 120kV, sample held at liquid nitrogen temperature.

Parameter	Data Set 1 U_g values	Data Set 2 U_g values	Data Set 1 f_x values	Data Set 2 f_x values	Neutral Atom f_x values
{111}	3.898(3)	3.888(6)	21.50(1)	21.52(1)	21.78
{002}	3.435(4)	3.419(7)	20.13(1)	20.17(2)	20.35
{220}	2.436(11)	2.393(18)	16.14(5)	16.35(5)	16.21
{113}	2.044(10)	2.025(19)	13.96(5)	14.09(12)	14.09
t (in Å)	1052(1)	652(1)	-	-	-

Table 1. The U_g values are in units of $\text{\AA}^{-2}$ and are fitted at liquid nitrogen temperature. The Cu lattice parameter is $3.61\text{\AA}$ and the accelerating voltage was 118kV . The estimated 2σ values are shown in parentheses. The authors believe that the results from the thicker pattern are more reliable as the sum of squares is smaller than for the thinner pattern and the residual map shows very little structure. The f_x values are the X-ray structure factors at liquid nitrogen temperature derived from the fitted U_g s using the Mott formula. The neutral atom structure factors are derived from Doyle and Turner⁴. It can be seen that the {111} and {002} structure factors differ appreciably from the neutral atom values, while the differences for {220} and {113} are of the order of the estimated error.

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