

A Summary of Low-angle X-ray Atomic Scattering Factors Measured by the Critical Voltage Effect in High Energy Electron Diffraction*

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Abstract

A summary of all the accurate ($\sim 0.1\%$) low-angle X-ray atomic scattering (form) factors for cubic and hexagonal close-packed elements which have been determined by the critical voltage technique in high energy electron diffraction (HEED) is presented. For low atomic number elements ($Z \leq 40$) the low-angle form factors can be significantly different to best free atom values, and so the best band structure calculated and/or X-ray measured form factors consistent with the critical voltage measurements are also indicated. At intermediate atomic numbers ($Z \approx 40-50$) only the very low-angle form factors appear to be different to the best free atom values, and even then only by small amounts. For heavy elements ($Z \geq 70$) the best free atom form factors appear to agree very closely with the critical voltage measured values and so, in this case, critical voltage measurements allow accurate determinations of Debye-Waller factors.

1. Introduction

When a crystal, which is thick enough for sharp Kikuchi lines to be exhibited (usually 1000 Å or greater), is set at the Bragg reflecting position in HEED, the intensity of the diffracted beam is usually strong owing to the constructive interference of waves scattered in the diffracted beam direction. However, for reflections higher than first order in a systematic row, at a particular electron accelerating voltage known as the critical voltage V_c , the diffracted beam intensity can be very small owing to destructive rather than constructive interference (see e.g. Lally *et al.* 1972). The critical voltage is very sensitive to the low-order Fourier coefficients of the crystal potential for the systematic row concerned, and hence can be used to determine these and related quantities with high accuracy. In particular, low-angle X-ray structure factors can be measured with an accuracy which far exceeds conventional X-ray measurements, and is similar to those achieved by X-ray Pendellösung methods (see e.g. Aldred and Hart 1973). This high accuracy has allowed Smart and Humphreys (1980) to produce accurate sets of atomic scattering (form) factors and thus deformation electron density distributions for several cubic elements. The object of this work is to review all the critical voltage measurements that have been made on both cubic and hexagonal close-packed elements, and to present the best values of the low-angle form factors deduced from them.

* Paper presented at the International Symposium on Accuracy in Structure Factor Measurement, held at Warburton, Australia, 23-26 August 1987.

2. Measurement of Systematic Critical Voltages

Sample preparation for critical voltage measurements is very straightforward; all that is required is a conventional electropolished transmission electron microscope specimen. To make the greatest range of V_c measurements, it is necessary to have a high (as possible) voltage electron microscope (HVEM). Most HVEMs operate at 1 MeV, but 1.5 and 3 MeV machines are also available, and these can improve the amount of information available by this technique.

As mentioned in Section 1, a critical voltage is associated with the minimisation of a Bragg excited reflection of second or higher order in a systematic row. These minimisations can be observed by three methods:

- (1) the disappearance of the Kikuchi lines associated with the Bragg reflection (see e.g. Thomas *et al.* 1974);
- (2) the disappearance of the central maximum of the dark field bend contour associated with the minimising reflection (Lally *et al.* 1972); and
- (3) the disappearance of the central maximum of the rocking curve profile in the Bragg excited convergent beam disc (Sellar *et al.* 1980).

Also associated with these minimisations are changes in the asymmetry of Kikuchi lines in the diffraction patterns [for methods (1) and (3)] which allow accuracies of $\sim 0.2\%$ to be obtained in V_c , although about 1% is the routine error for measurements on second-order reflections. Higher order minimisations are often less distinct (see e.g. Jones 1978; Fox 1985), but can still give accurate information about low-angle X-ray structure factors.

3. Analysis of V_c Measurements

As discussed by Lally *et al.* (1972), a critical voltage occurs when electron waves (Bloch waves) interfere destructively in their contribution to a Bragg excited reflection, and consequently a V_c value can be analysed using a suitable computer program for the dynamical matrix for electron diffraction in a systematic row (see e.g. Fisher 1968). It is clear from this that V_c depends on all the Fourier coefficients of the systematic row concerned and Lally *et al.* (1972) and Sellar *et al.* (1980) have pointed out that the major contribution to V_c comes from the first-order low-angle form factor. The second-order low-angle form factor and the Debye–Waller factor also make important contributions, with higher angle form factors being an order of magnitude less in their effect on V_c . This means that where higher angle form factors are very different to relativistic Hartree–Fock (RHF) free atom values (which are usually used in V_c analyses), it is important to use the best higher angle data available in the analysis of critical voltage measurements. As shown by Lally *et al.* (1972), with accurate high-angle data, the first-order form factor for electrons f_e can be determined to within $\sim 0.2\%$ if V_c is measured to within 1%. The factor f_e is related to the form factor for X-rays, f , by the usual Mott equation

$$f_e = \frac{me^2}{2h^2} \left(\frac{\lambda}{\sin \theta} \right)^2 (Z - f), \quad (1)$$

where m , e , h , λ and θ have their usual meanings and Z is the atomic number for the atom concerned. For low-angle form factors $Z - f$ is small, and hence if f_e is known to a certain percentage accuracy, equation (1) gives f to a much greater accuracy.

Typically, f can be measured to within 0.1% or better using the V_c methods (see e.g. Hewat and Humphreys 1974) and this technique forms the basis for the results presented in the next section.

4. Results

In this section the room temperature critical voltage results V_c (with appropriate superscript showing the reflection minimising and with all values in units of kV) for numerous hexagonal and cubic elements are presented together with the best room temperature Debye–Waller factors B (in \AA^2) and higher angle form factors available (consistent with the V_c measurements). The low-angle X-ray form factors f (with appropriate superscript) determined from the V_c results are also shown together with the equivalent RHF form factors in parentheses.

(a) Hexagonal Elements

Apart from beryllium and zinc, very few accurate form factor measurements have been made on these materials, and in general free atom RHF (Doyle and Turner 1968) form factors are used for the higher angle reflections, together with the best available room temperature, anisotropic Debye–Waller factors B_c and B_a . This seems to be quite reasonable for h.c.p. elements, as only the low-angle form factors appear to be significantly different from free atom values.

Beryllium. $B_c = 0.395$, $B_a = 0.435$ (Larsen *et al.* 1980). The best higher angle form factors are the γ -ray values of Hansen *et al.* (1984) or X-ray values of Larsen and Hansen (1984):

$$\begin{aligned} V_c^{0004} &= 1230(30) \text{ (Fox and Fisher 1987)} & f^{0002} &= 1.679 \text{ (1.741)} \\ V_c^{20\bar{2}2} &= 718(10) \text{ (Thomas *et al.* 1974)} & f^{10\bar{1}1} &= 1.704 \text{ (1.718)}. \end{aligned}$$

There is excellent agreement between these results and the values of Hansen *et al.* (1984) and Larsen and Hansen (1984).

Magnesium. $B_c = 1.23$, $B_a = 1.34$ (Brindley and Ridley 1938). For higher angle form factors the RHF free atom values of Doyle and Turner (1968) were used:

$$\begin{aligned} V_c^{0004} &= 678(6) \text{ (Thomas *et al.* 1974)} & f^{0002} &= 8.774 \text{ (8.84)} \\ V_c^{20\bar{2}2} &= 310(5) \text{ (Thomas *et al.* 1974)} & f^{10\bar{1}1} &= 8.624 \text{ (8.677)}. \end{aligned}$$

Titanium. $B_c = 0.55$, $B_a = 0.57$ (Schoening and Witt 1965). Higher angle form factors from Doyle and Turner (1968) were used:

$$V_c^{0004} = 236(10) \text{ (Arii *et al.* 1973)} \quad f^{0002} = 15.47 \text{ (15.638)}.$$

α -*Cobalt.* $B_c = B_a = 0.39$ (Barron *et al.* 1957). Higher angle form factors from Doyle and Turner (1968) were used:

$$\begin{aligned} V_c^{0004} &= 278(2) \text{ (Thomas *et al.* 1974)} & f^{0002} &= 19.461 \text{ (19.518)} \\ V_c^{22\bar{4}0} &= 1850(50) \text{ (Present work)} & f^{11\bar{2}0} &= 14.903 \text{ (14.718)} \\ V_c^{20\bar{2}6} &= 1210(40) \text{ (Present work)} & f^{10\bar{1}3} &= 13.879 \text{ (13.762)}. \end{aligned}$$

Zinc. $B_c = 2.045$, $B_a = 0.884$ (Skelton and Katz 1968). Higher angle form factors from Doyle and Turner (1968) were used:

$$\begin{aligned} V_c^{0004} &= 112.5(12.5) \text{ (Jones 1978)} & f^{0002} &= 24.19 \text{ (24.193)} \\ V_c^{20\bar{2}2} &\approx 40 \text{ (Present work)} & f^{10\bar{1}1} &\approx 22.714 \text{ (22.869)} \\ V_c^{20\bar{2}6} &= 730(30) \text{ (Jones 1978)} & f^{10\bar{1}3} &= 17.75 \text{ (18.288)}. \end{aligned}$$

These results are in reasonably good agreement with the low-angle X-ray Pendellösung results of Takama *et al.* (1984).

Cadmium. $B_c = 2.61$, $B_a = 1.11$ (Brindley and Ridley 1939). Higher angle form factors from Doyle and Turner (1968) were used:

$$\begin{aligned} V_c^{30\bar{3}0} &= 900(30) \text{ (Jones 1978)} & f^{10\bar{1}0} &= 39.30 \text{ (39.278)} \\ V_c^{0006} &= 530(30) \text{ (Jones 1978)} & f^{0002} &= 40.126 \text{ (40.224)} \\ V_c^{22\bar{4}0} &= 1030(30) \text{ (Jones 1978)} & f^{11\bar{2}0} &= 31.516 \text{ (31.387)} \\ V_c^{20\bar{2}6} &= 215(15) \text{ (Present work)} & f^{10\bar{1}3} &= 31.617 \text{ (31.671)}. \end{aligned}$$

(b) Diamond Cubic Elements

These elements (in particular silicon) have been the subject of considerable study by many workers, and the current results agree very well with the best X-ray data.

Silicon. $B = 0.4613$ (Aldred and Hart 1973). Higher angle form factors were taken from the X-ray Pendellösung work of Aldred and Hart (1973) with Cromer (1965) anomalous dispersion values:

$$\begin{aligned} V_c^{333} &= 1104(6) \text{ (Hewat and Humphreys 1974)} & f^{111} &= 10.730 \text{ (10.536)} \\ V_c^{333} &= 1101(7) \text{ (Shishido and Tanaka 1975)} & f^{222} &= -0.185. \end{aligned}$$

The V_c^{333} measurement allows a simple relationship between f^{111} and f^{222} to be developed, and by a best choice of f^{111} (considering all the values available), f^{222} was calculated.

$$V_c^{440} = 1101(6) \text{ at } 515^\circ\text{C} \text{ (Thomas } et al. \text{ 1974)} \quad f^{220} = 8.688 \text{ (8.718)}.$$

The analysis of V_c^{440} used an appropriate high temperature Debye–Waller factor.

Germanium. $B = 0.543$ (Mair and Barnea 1975). Higher angle form factors were taken from the X-ray work of Matsushita and Kohra (1974) with Cromer (1965) anomalous dispersion values:

$$\begin{aligned} V_c^{333} &= 922(5) \text{ (Hewat and Humphreys 1974)} & f^{111} &= 27.53 \text{ (27.366)} \\ V_c^{333} &= 908.5(6.4) \text{ (Shishido and Tanaka 1976)} & f^{222} &= -0.16. \end{aligned}$$

The factor f^{222} was estimated from a combination of V_c and convergent beam diffraction measurements by Shishido and Tanaka (1976).

$$V_c^{440} = 994(5) \text{ (Hewat and Humphreys 1974)} \quad f^{220} = 23.75 \text{ (23.797)}.$$

(c) Body-centred Cubic Elements

Surveys of the best Debye-Waller factors for many cubic elements have been carried out by Thomas *et al.* (1974), Smart and Humphreys (1980) and Fox and Fisher (1986), and the best values of B from these workers are adopted in this subsection and the next.

Vanadium. $B = 0.66$. Higher angle form factors were taken from the band structure calculations of Laurent *et al.* (1978) which have recently been re-evaluated by Fox (1987, unpublished):

$$\begin{aligned} V_c^{220} &= 230.0(2.5) \text{ (Thomas } et al. \text{ 1974)} & f^{110} &= 15.90 \text{ (15.964)} \\ V_c^{400} &= 1141(37) \text{ (Terasaki } et al. \text{ 1975)} & f^{200} &= 13.22 \text{ (13.208)}. \end{aligned}$$

Chromium. $B = 0.24$. Higher angle form factors were taken from the band structure calculations of Wakoh and Yamashita (1971):

$$\begin{aligned} V_c^{220} &= 265(3) \text{ (Thomas } et al. \text{ 1974)} & f^{110} &= 16.259 \text{ (16.715)} \\ V_c^{400} &= 1285(31) \text{ (Terasaki } et al. \text{ 1975)} & f^{200} &= 13.47 \text{ (13.582)}. \end{aligned}$$

α -Iron. $B = 0.35$. Higher angle form factors were taken from the band structure calculations of Wakoh and Yamashita (1971) or De Cicco and Kitz (1967), which are in good agreement with each other:

$$\begin{aligned} V_c^{220} &= 305(3) \text{ (Thomas } et al. \text{ 1974)} & f^{110} &= 18.278 \text{ (18.449)} \\ V_c^{400} &= 1278(4) \text{ (Terasaki } et al. \text{ 1975)} & f^{200} &= 15.143 \text{ (15.231)}. \end{aligned}$$

Niobium. $B = 0.47$. Higher angle form factors were taken from the RHF values of Cromer and Waber (1974):

$$\begin{aligned} V_c^{220} &= 35(3) \text{ (Thomas } et al. \text{ 1974)} & f^{110} &= 31.350 \text{ (31.584)} \\ V_c^{400} &= 749(3) \text{ (Thomas } et al. \text{ 1974)} & f^{200} &= 27.470 \text{ (27.602)}. \end{aligned}$$

Molybdenum. $B = 0.254$. Higher angle form factors were taken from Cromer and Waber (1974):

$$\begin{aligned} V_c^{220} &= 35(3) \text{ (Thomas } et al. \text{ 1974)} & f^{110} &= 31.637 \text{ (31.906)} \\ V_c^{400} &= 789(2) \text{ (Thomas } et al. \text{ 1974)} & f^{200} &= 27.579 \text{ (27.667)}. \end{aligned}$$

Tantalum. $B = 0.358$. Higher angle form factors were taken from Cromer and Waber (1974):

$$V_c^{400} = 651(2) \text{ (Thomas } et al. \text{ 1974)} \quad f^{400} = 54.595 \text{ (54.624)}.$$

Tungsten. $B = 0.192$. Higher angle form factors were taken from Cromer and Waber (1974):

$$V_c^{400} = 660(3) \text{ (Thomas } et al. \text{ 1974)} \quad f^{400} = 54.479 \text{ (54.517)}.$$

(d) Face-centred Cubic Elements

Aluminium. $B = 0.85$. There are many band structure calculated and experimental form factor values of this element [see e.g. Walter *et al.* (1973), Tawil (1975) (band structure calculations), Takama *et al.* (1982) (X-ray Pendellösung), Rantavuori and Tanninen (1977) (X-ray powder) and Inkinen *et al.* (1970) (X-ray reflection)]. Of these, those by Inkinen *et al.* appear to be best and were therefore adopted in the calculations:

$$\begin{aligned} V_c^{222} &= 425(10) \text{ (Lally } et al. 1972) & f^{111} &= 8.843 (8.956) \\ V_c^{400} &= 918(5) \text{ (Thomas } et al. 1974) & f^{200} &= 8.386 (8.509). \end{aligned}$$

ε Cobalt. $B = 0.39$. The higher angle form factors were taken to be the free atom RHF values of Doyle and Turner (1968), which seem reasonable for cobalt as its electron charge distribution does not appear to be much different from the free atom case:

$$\begin{aligned} V_c^{222} &= 276(2) \text{ (Thomas } et al. 1974) & f^{111} &= 19.526 (19.564) \\ V_c^{400} &= 555(3) \text{ (Thomas } et al. 1974) & f^{200} &= 18.182 (18.300) \\ V_c^{400} &= 1850(50) \text{ (Present work)} & f^{220} &= 14.872 (14.713). \end{aligned}$$

Nickel. $B = 0.4$. Higher angle form factors were taken from the band structure calculations of Wang and Callaway (1977) (ν Bh calculation):

$$\begin{aligned} V_c^{222} &= 298(2) \text{ (Thomas } et al. 1974) & f^{111} &= 20.482 (20.544) \\ V_c^{400} &= 588(3) \text{ (Thomas } et al. 1974) & f^{200} &= 19.181 (19.249). \end{aligned}$$

Copper. $B = 0.54$. Similar to aluminium, there are many band structure calculated and experimental form factor values for this element [see e.g. Wakoh and Yamashita (1971), Bagayoko *et al.* (1980), McDonald *et al.* (1982) (band structure calculations), Takama and Sato (1982) (X-ray Pendellösung experimental) and Schneider *et al.* (1981) (γ -ray experimental)]. Of these, those by Wakoh and Yamashita appear to be the most consistent with the critical voltage measurements:

$$\begin{aligned} V_c^{222} &= 310(3) \text{ (Thomas } et al. 1974) & f^{111} &= 21.702 (22.076) \\ V_c^{400} &= 605(3) \text{ (Thomas } et al. 1974) & f^{200} &= 20.421 (20.725) \\ V_c^{440} &= 1750(50) \text{ (Rocher and Jouffrey 1972)} & f^{220} &= 16.675 (16.782). \end{aligned}$$

Of the experimental results, the X-ray Pendellösung values by Takama and Sato (1982) show the best agreement with the critical voltage measurements; the γ -ray values of Schneider *et al.* (1981) seem somewhat low.

Silver. $B = 0.655$. Higher angle form factors were taken from Doyle and Turner (1968):

$$\begin{aligned} V_c^{222} &= 55(3) \text{ (Fukuhara and Yanagisawa 1969)} & f^{111} &= 37.331 (37.438) \\ V_c^{400} &= 225(10) \text{ (Lally } et al. 1972) & f^{200} &= 35.418 (35.496) \\ V_c^{400} &= 919(5) \text{ (Thomas } et al. 1974) & f^{220} &= 29.873 (30.096). \end{aligned}$$

Platinum. $B = 0.323(10)$ (calculated from the V_c^{622} measurement—see Section 5). Higher angle form factors were taken from Cromer and Waber (1974):

$$\begin{aligned} V_c^{440} &= 800(10) \text{ (Present work)} & f^{220} &= 54.262 \text{ (54.439)} \\ V_c^{622} &= 1420(20) \text{ (Present work)} & f^{311} &= 50.553 \text{ (50.553)}. \end{aligned}$$

Gold. $B = 0.655$. Higher angle form factors were taken from Doyle and Turner (1968):

$$\begin{aligned} V_c^{400} &= 108(2) \text{ (Thomas } et al. \text{ 1974)} & f^{200} &= 63.580 \text{ (63.594)} \\ V_c^{440} &= 726(5) \text{ (Thomas } et al. \text{ 1974)} & f^{220} &= 55.845 \text{ (56.090)}. \end{aligned}$$

5. Discussion

It is clear that for heavy elements ($Z \approx 70$) the experimental (crystal) form factors are very close to the best RHF free atom values, and so critical voltage measurements on these can be used to obtain accurate Debye–Waller factors. The V_c^{622} measurement for platinum was used in this way to produce $B = 0.323(10) \text{ \AA}^2$ for Pt.

For elements with intermediate atomic number ($Z \approx 40\text{--}50$), only the very low-angle form factors are different from the best free atom values, and so for niobium and molybdenum the results presented here should be sufficient to plot the charge density [in fact, Smart and Humphreys (1980) have produced a deformation electron density map for Nb]. For cadmium, accurate values of $f^{10\bar{1}1}$ and $f^{10\bar{1}2}$ are needed to complete a charge density analysis.

For light elements ($Z \leq 35$), the crystal form factors may deviate considerably from best free atom values, although these differences seem more severe for cubic elements than they are for h.c.p. elements. This means that free atom form factor values at higher angles are usable in V_c analyses on low Z h.c.p. elements, which is important because, apart from beryllium, very few good theoretical or experimental crystal form factors are available for these elements. For cubic elements there are plenty of theoretical and experimental crystal form factors for higher angles to choose from, and those which appear to be best are given in Section 4. It should be noted that nearly all the crystal form factors shown in the present work are less than their equivalent free atom values and, as discussed by Smart and Humphreys (1980), this means that electron charge is being depleted from the outside of atomic sites (in the free atom case) to positions between the atoms.

Acknowledgments

The authors would like to thank the following persons: Professor R. E. Smallman, Head of Department of Metallurgy and Materials, University of Birmingham, for the use of an AEI 1 MeV HVEM; Dr B. Jouffrey at the Laboratoire D'Optique Electronique du CNRS, Toulouse, for the use of a 3 MeV HVEM; Dr K. H. Westmacott, National Centre for Electron Microscopy, Lawrence Berkeley Laboratory, for the use of the Kratos 1.5 MeV HVEM; and The Director of the Polytechnic, Wolverhampton, for the provision of computing facilities. The work was supported in part by the Director, Office of Energy Research, Office of Basic Sciences, Materials Science Division of the US Department of Energy, under Contract DEAC03-76SFOO098.

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