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X-Ray Measurement of the Atomic Scattering Factor of Iron

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The x-ray integrated intensities of Bragg reflections of iron, copper, and NaCl powders were measured with monochromatic Fe K_α radiation. By investigating the effect of cold work on intensities of the metal powders and comparing the measured intensities of different orders with theoretical values, it was shown that extinction, surface roughness, and preferred orientation effects were negligible. The four measured iron reflections gave atomic scattering factors relative to either copper or NaCl which agreed, within experimental error, with values calculated from wave functions of the isolated iron atom. It is estimated that the experimental form factor for the lowest order reflection of copper and iron agrees with free-atom values to an error corresponding to the contribution of ± one free-atom-3*d* electron. This is to be contrasted with results of Weiss and DeMarco, who concluded from single-crystal measurements that metallic iron has (2.3±0.3) 3*d* electrons, compared with 6 for the free atom.

INTRODUCTION

In a recent paper Weiss and DeMarco[1] have reported an x-ray measurement of the number of 3*d* electrons in Cu, Ni, Co, Fe, and Cr single crystals. Their method was to measure the absolute intensities of Bragg reflections from these crystals, apply a correction for extinction, and using the equation for the integrated intensity of an imperfect crystal, compute the atomic scattering factor for each reflection. The difference between the measured and theoretical scattering factors was attributed to a change in the 3*d* electron distribution from a free atom configuration. They report that metallic iron has (2.3±0.3) 3*d* electrons compared with 6 for the isolated atom. Since the Weiss-DeMarco results were unexpected in light of present theoretical considerations, it was felt that an independent measurement of the atomic scattering factor of iron should be made. The results of the present measurements indicate that the atomic scattering factor for metallic iron agrees with that calculated for the isolated atom. These results have already been reported.[2] To facilitate a critical evaluation of the validity of the reported atomic scattering factors, the experimental procedures, the measured data, and the calculations will be presented in detail.

THEORY

The integrated intensities of Bragg reflections from ideally imperfect crystals are proportional to the square of the atomic scattering factors. The integrated intensity of a Bragg reflection of an ideal powder specimen measured with monochromatic radiation is[3]

$$P=k\frac{mF^2\exp(-2M)(1+\cos^2 2\theta_M\cos^2 2\theta)}{\mu_\rho(\text{M.W.})v_a\sin\theta\sin 2\theta},$$

where k is a constant for all measurements with a given wavelength; m is the multiplicity; F is the structure factor of the cell of molecular weight (M.W.) and volume v_a; $\exp[-2M]=\exp[-2B\sin^2\theta/\lambda^2]$ is the Debye temperature factor; θ_M is the Bragg angle for the monochromator; and μ_ρ is the *mass* absorption coefficient.

If a crystal is not ideally imperfect, one must apply a correction for extinction based on an assumed model for the crystal imperfection. To eliminate the necessity of employing such a correction, it was decided to perform the experiment in such a manner as to render extinction effects negligible.[4] It has been shown[5,6] that cold-worked metal powders can be made free of extinction. Measurements on powders, however, introduce problems of preferred orientation and surface roughness absorption. The latter reduces the intensities of low-angle reflections due to unevenness of the specimen surface. This has been observed by McKeehan and Warren[5] and qualitatively measured by deWolff.[7] If the particle size and absorption coefficient are low enough, this effect can be negligible. Both extinction and surface roughness tend to reduce the intensity of lower order reflections more than higher orders. By investigating the effect of cold work and surface preparation on these lower order reflections, one can establish whether or not extinction or surface roughness are significant. A further check can be made by comparing the observed and theoretical intensities of several orders if the theoretical atomic scattering factors are not in question.

In this work, two experimental determinations of the atomic scattering factor of iron have been made. In one, the iron intensities were standardized against the copper

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[1] R. J. Weiss and J. J. DeMarco, Revs. Modern Phys. 30, 59 (1958).

[2] B. W. Batterman, Phys. Rev. Letters 2, 47 (1959). A reply to this preliminary report has been made by Weiss and DeMarco, Phys. Rev. Letters 2, 148 (1959).

[3] R. W. James, *The Optical Principles of the Diffraction of X-Rays* (G. Bell and Sons, London, 1950).

[4] In the iron single crystal measurements of Weiss and DeMarco, the extrapolated value of $f^2(110)$ (presumably with no extinction) ranged from 1.2 to 2.0 times the observed values (Fig. 3 of their paper).

[5] M. McKeehan and B. E. Warren, J. Appl. Phys. 24, 52 (1953).

[6] B. L. Averbach and B. E. Warren, J. Appl. Phys. 20, 1066 (1949).

[7] P. M. deWolff, Acta Cryst. 9, 682 (1956).

TABLE I. Effect of sample preparation on integrated intensities of powder compresses of iron and copper.

			P_{hkl}		
Iron		110	200	211	220
Pressed 40 000 psi		924	166	417	269
Cold-worked in ball mill pressed 30 000 psi		916	160	399	266
Only Duco binder		873	165	403	261
Copper	111	200	220	311	222
Less than 100 psi	640	333	261	447	197
2000 psi	639				
60 000 psi	655	340	261	458	200
120 000 psi	625	338	267	459	181

(111) reflection using Fe K_α and Mo K_α radiation. In the second experiment, the iron intensities were standardized against NaCl using Fe K_α radiation.

EXPERIMENTAL

(1) General Considerations

The x-ray intensities were measured with a General Electric x-ray diffractometer which was adapted to receive monochromatic Fe K_α radiation from a singly bent lithium fluoride monochromator. Iron radiation was chosen because it would be expected to give the smallest surface roughness absorption of the more conventional radiations. The tube voltage for all measurements with iron radiation was 20 kv. From a measurement with an NaCl single crystal, the primary beam was found to contain 0.3% $\lambda/2$ component. This can be neglected in these measurements.

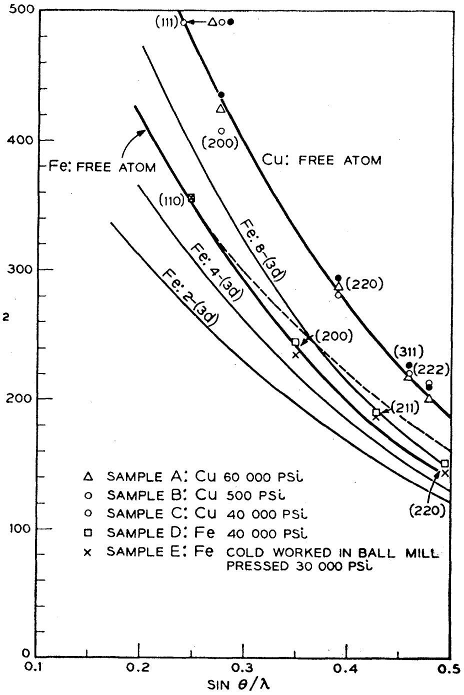

FIG. 1. Experimental values of f^2 for iron and copper *vs* $\sin\theta/\lambda$. Theory and experiment are matched at a single point, the (111) reflection of copper. The three copper specimens are matched at the (111) and the two iron specimens at (110). The curves for iron with 2, 4, and 8 (3*d*) electrons are from *f*-curves of electrons of the isolated atom. The dotted curve is the Fe:2 (3*d*) curve times a constant to match the free-atom curve at the (110) reflection.

The iron specimens were made from powders of carbonyl iron containing particles about 3 μ in diameter and purity 99.5%. The copper specimens were made from an electrolytic powder of purity 99.2% with a particle size of about 5 μ estimated microscopically.[8] The powders were pressed in a highly polished cylindrical mold. Preliminary measurements were made with both powders to check the effect of molding pressure, and consequently the density of sample and state of the surface on the integrated intensities of the various reflections. In addition to the cold work introduced in the molding process, some of the powders were initially cold worked in a small oscillating ball mill before being pressed. The data from these measurements are presented in raw form in Table I. The intensities are the total number of counts ($\times\frac{1}{10}$) entering the counter as the diffractometer rotates counter and sample a fixed 2θ distance bracketing the reflection, and consequently includes the background under the peaks. (The background was shown to be independent of sample preparation.) The relative intensities of different *hkl* reflections are not significant; only the intensities of a given reflection from the different samples are to be compared. The purpose of presenting these data is to show the sensitivity of integrated intensities to sample preparation. The copper briquets made at the higher pressures showed considerable line broadening and reduction of peak heights by as much as 30% over those made at low pressures. At the high pressures the plastic flow was great enough to produce a mirror-like finish, which, upon microscopic observations showed local variations in flatness of about 3 μ. The <100 psi specimen had a surface variation about 3–5 times those at the higher pressures, and showed no appreciable difference in intensities. Only at the very highest pressure of 120 000 psi was there possibly a slight texture developed as evidenced by the slightly lower (111) and (222) intensities. Data not presented in the table showed that copper powder cold worked in a ball mill and lightly pressed into a briquet gave (111) intensities within the range presented in the tables. The fraction of the final metallic density ranged from approximately 0.2 in the loosely packed copper powder to 0.8 with the high molding pressures.

The results for the iron powders are similar to those for copper. The powder held together merely with a Duco cement binder had a density of approximately 0.50 of solid iron, while at the highest molding pressure a value of 0.72 was reached. The Duco-bonded specimen

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[8] Manufactured by Whitaker Metals Corporation; supplied by Charles Hardy, Inc., New York, New York.

TABLE II. Summary of experimental and calculated parameters used in Eq. (1) to evaluate atomic scattering factors.

Sample	a_0 (A)	μ_ρ(cm²/g) (Fe K_α)	μ_ρ(cm²/g) (Mo K_α)	$\Delta f'$ Mo K_α[a]	$\Delta f'$ Fe K_α[b]	$\Delta f''$ Mo K_α[a]	$\Delta f''$ Fe K_α[b]	B(293°K)
Copper	3.615	96.3	51.0[c]	0.4	-1.51	1.4	0.93	0.555[d]
Iron	2.866	70.5	38.9	0.4	-2.45	1.0	0.61	0.368[d]
NaCl	5.639	148			$+0.42$[e]		1.2[e]	1.36[f]

[a] See reference (11).
[b] See reference (10).
[c] See reference (23).
[d] K. Londsdale, Acta Cryst. 1, 142 (1948). Based on Debye θ for copper and iron of 315°K and 420°K, respectively.
[e] These values are the sum of Na^+ and Cl^- dispersion corrections [calculated from reference (10)] and are valid for those reflections for which Na^+ and Cl^- scatter in phase.
[f] I. Waller and R. W. James, Proc. Roy. Soc. (London) **A117**, 214 (1927).

showed about a 5% lower (110) reflection, while the higher orders were within experimental error of intensities from higher pressure samples. The (110) intensities from samples with intermediate molding pressures were consistent with those from the higher pressure samples reported in the table. These results for the copper and iron powders indicate that no preferred orientation is introduced in the compression and that extinction and surface roughness effects are not important.[9]

The parameters used to calculate the atomic scattering factors from the intensity data are given in Table II. The Hönl corrections are calculated by the method of Parratt and Hempstead,[10] or were taken from the tabulation of Dauben and Templeton[11] based on this method. A description of the measurements of the mass absorption coefficients is given in the Appendix.

(2) Measurement of Iron Relative to Copper

The complete Fe K_α spectra of copper and iron were measured from three copper and two iron compressed powders. The total number of counts entering the proportional counter was recorded as the diffractometer was driven through a Bragg reflection between two points beyond the tails of the peak. The background was determined from the average of the fixed counts at these points. The measured intensities corrected for background are given in Table III. The intensities for the three copper specimens were matched at the (111) reflection, and the two iron specimens at the (110). These low order reflections were picked because their high signal-to-noise ratio gave them the highest precision. The weakest reflections, having poor signal-to-noise ratios have the largest experimental errors. The two iron (110) reflections and three copper (111) reflections were measured with a carefully monitored primary beam. The spread in intensity of each set was less than 4%. Using the average values of these results, the two sets of spectra were put on a common intensity scale. Equation (1) was matched to experimental intensities at the copper (111) reflection. This evaluates the constant k, and from this single value, all the theoretical intensities for iron and copper in Table III were computed. In the last two columns of Table III are the theoretical[12,13] and average experimental values of the atomic scattering factors (without dispersion) using the same one-parameter fit.

In Fig. 1 the experimental values of f^2 for the individual specimens are plotted against $\sin\theta/\lambda$. The relative intensities of the copper specimens A and B (Fig. 1) can be matched separately with the theoretical free atom values to ±3% for all reflections, while for sample C, the match can be made to ±6%. If surface roughness and extinction were important, the experimental points would be considerably higher than theory at high values of $\sin\theta/\lambda$. It is felt that the good fit between theory and experiment for all the copper reflections shows that the copper specimens are free of extinction and surface roughness effects.

TABLE III. Experimental intensities and average atomic scattering factors of iron and copper obtained from a single-parameter match between the experimental copper (111) scattering factor and that calculated from free-atom wave functions. The arrows show where data have been matched.

	P measured[a]				f	
				Theoretical intensities (free atom)	Exp. av	Free atom
	A	B	C			
Cu 111	578 ↔	578 ↔	578 ↔	578	22.2 ↔	22.16
200	273	263	283	280	20.6	20.78
220	206	200	211	200	17.0	16.78
311	323	325	337	320	14.9	14.76
222	113	121	119	113	14.5	14.20
	D	E				
Fe 110	816 ↔	816		813	18.8+	18.77
200	139	134		138	15.5	15.57
211	360	352		338	13.7	13.45
220	213	201		197	12.1	11.91

[a] Sample A: pressed 60 000 psi; B: pressed 500 psi; C: pressed 40 000 psi; D: pressed 40 000 psi; E: powder cold-worked in ball mill, then pressed 30 000 psi.

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[9] These points will be further checked for copper by comparing the relative intensities of the different hkl reflections with theoretical predictions. For iron, the relative intensities will be compared to theoretical values for the iron atom with different numbers of d electrons.

[10] L. G. Parratt and C. F. Hempstead, Phys. Rev. **94**, 214 (1954).

[11] C. H. Dauben and D. H. Templeton, Acta Cryst. **8**, 841 (1955).

[12] The theoretical f(Cu) values from J. Berghius *et al.* [Acta Cryst. **8**, 478 (1955)] are for Cu^+. The difference between Cu^+ and Cu is negligible in the range of $\sin\theta/\lambda$ involved.

[13] The f values for iron are those of A. J. Freeman and J. H. Wood, Acta Cryst. **12**, 271 (1959).

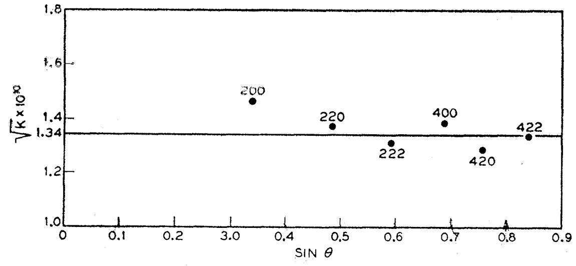

FIG. 2. Plot *vs* sinθ of

$$\sqrt{k}=[P\mu_\rho(\text{M.W.})v_a/m(\text{LP})F^2\exp(-2M)]^{\frac{1}{2}}$$

for h, k, l even reflections of NaCl with Fe K_α radiation. (LP) is the Lorentz and polarization factors.

Since this experiment is concerned primarily with the electron distribution in iron, one must compare measured iron f values with those expected for the atom with different numbers of $3d$ electrons. In Fig. 1 are the f^2 curves for iron with 2, 4, 6 (isolated atom), and 8 free-atom ($3d$) electrons.[14] The iron intensities can be matched to the free-atom curve to ±4% for sample D and ±3% for E. To facilitate discussion of the existence of extinction and surface roughness in iron, drawn in Fig. 1 is a dotted curve of f^2 for iron with two ($3d$) electrons matched at the (110) value for the free-atom curve. Considering only the relative intensities of the different reflections, it can be seen that the f^2 curve for two ($3d$) electrons is higher at high sinθ/λ. If the iron specimens had extinction or surface roughness, correcting for these would depress the data points at large sinθ/λ relative to low values, and the departure from the functional variation predicted for two ($3d$) electrons would be greater.[15]

In light of the measured values of f^2 for iron, and its variation with sinθ/λ, it is concluded that the experimental data are not consistent with metallic iron containing significantly less than six ($3d$) electrons. From the average value of the ratio of the intensity of copper (111) to iron (110) for several pairs of specimens, Eq. (1) gives for $f(110)$ of iron 18.9 ± 0.3 relative to the theoretical value $f(111)=22.16$[12] for copper. The free-atom value $f(110)$ of iron is 18.77.[13]

The experimental atomic scattering factors for iron relative to copper depend upon the theoretically determined dispersion corrections. A cross check on these theoretical values can be made by measuring the iron and copper reflections with another wavelength having a different dispersion correction. Another check would be provided by using the Fe K_α radiation to measure another material with a different dispersion correction and thus standardizing the iron and copper intensities.

TABLE IV. Intensities (P) of NaCl used to standardize copper and iron data.

	hkl	P	$k^{\frac{1}{2}}\times10^{10}$	Theoretical intensity
NaCl	200	769	1.46	647
	220	447	1.37	429
	222	137	1.31	143
	400	71	1.38	66.6
	420	178	1.28	193
	422	158	1.33	162
Fe	110	3300		3220
Cu	111	2324		2290

The first check was made by measuring the iron (110) and copper (111) with unfiltered molybdenum radiation with a Philips diffractometer. Two pairs of specimens gave intensity ratios $P_{\text{Fe}}(110)/P_{\text{Cu}}(111)$ of 1.52 and 1.57, as determined from the peak areas on the strip charts. Using Eq. (1) with $\theta_M=0$ (unpolarized incident beam) and the constants in Table II, these area ratios reduce to $f(110)=18.6$ and 18.9 relative to the free atom scattering factor for the (111) reflection of copper. These results are in good agreement with those obtained using iron radiation.[16]

(3) Standardization of Iron and Copper Against NaCl

Rocksalt is considered a good intensity standard because experimental results[17] have shown that the high-order reflections from single crystals are free of extinction. Since in powdering rocksalt, plastic deformation occurs, it is reasonable to expect that extinction should be even smaller than in the single crystals. Reagent NaCl was ground in a ball mill, passed through a 400-mesh screen and pressed into a briquet. In the initial attempt using a molding pressure of about 14 000 lb/in.², a strong (100) texture was developed. This was overcome by dusting a layer of NaCl powder (thick enough to completely absorb Fe K_α radiation) over a highly compressed NaCl substrate and pressing the surface merely with the weight of the mold. This corresponded to a pressure of about 2 lb/in.². In this way the weakly pressed powder adhered to the sub-

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[14] The curves are taken from reference (13) and do not include any change in the wave functions of other electrons in the atom due to a change in the $3d$ electron screening.

[15] If a number of the $3d$ electrons have a wider spatial distribution than in the isolated atom, the corresponding reduction in screening would contract the distribution of the remaining electrons (Walter Marshall, private communication). This would flatten the f^2 curve for an atom with less than six ($3d$) electrons and result in even greater departure from experiment than is indicated by the dotted curve of Fig. (1).

[16] In using polychromatic radiation, it becomes necessary to use a filter to suppress the characteristic β line. This filter produces a discontinuity in the background near a Bragg reflection and consequently adds uncertainty to the determination of the background under the peak. With molybdenum radiation, the copper (111) and iron (110) could be measured without a filter, and yet not have any serious interference from a higher order β line. Since only a single reflection of iron and copper has been measured, it has not been explicitly proven that extinction and surface roughness are absent with molybdenum radiation. The fact that there is good agreement with the Fe K_α results can mean that these effects are negligible, or that a combination of errors are present. That the intensity ratios were the same between specimens having different compressions and degrees of cold work lends support to the former contention.

[17] Bragg, James, and Bosanquet, Phil. Mag. 41, 309 (1921); 42, 1 (1921). Also, reference 3 of this paper, pp. 287–292.

TABLE V. Measured values of the atomic scattering factors for iron and copper standardized against sodium chloride. The arrows show where data have been adjusted to the same intensity scales. All values of atomic scattering factors for the three substances are calculated from the value of $\sqrt{k}$ determined in Fig. (2).

	NaCl				Copper					Iron	
hkl	Fitted	Theory[a]	*hkl*	*A*	*B*	*C*	Theory[b]	*hkl*	*D*	*E*	Theory[c]
200	23.4	21.47	111	22.3 ↔	22.3 ↔	22.3	22.16	110	19.0 ↔	19.0	18.77
220	18.6	18.21	200	20.6	20.3	21.0	20.78	200	15.9	15.5	15.57
222	15.8	16.17	220	17.1	16.9	17.3	16.78	211	14.0	13.8	13.45
400	15.2	14.76	311	14.9	15.0	15.2	14.76	220	12.5	12.1	11.91
420	13.1	13.70	222	14.3	14.8	14.7	14.20				
422	12.7	12.85									

[a] See reference (18).
[b] See reference (12).
[c] See reference (13).

strate, and presented a flat surface to the x-ray beam. It was found that the high-multiplicity (422) reflection changed only about 4% between the highly and lightly compressed specimens, while the texture lines (200) and (400) changed by about 50%. Since surface roughness effects would be small for high-angle reflections, it is reasonable to assume that the high-multiplicity, high-angle reflections are fairly insensitive to texture and that for these, the small differences in flatness of surface between the two compressed specimens should be negligible.

The intensity data from this specimen and from the (110) of iron and the (111) of copper are given in Table IV. Using the experimental values of P_{NaCl} and $(\mu_\rho)_{\mathrm{NaCl}}$, and the tabulated values[18] of f_{NaCl} corrected for dispersion, the values $\sqrt{k}$ in Eq. (1) were calculated for each NaCl reflection. The values are plotted against $\sin\theta$ in Fig. 2. The horizontal line gives the average value of $\sqrt{k}$ so drawn to favor the higher order reflections. Using this single parameter, the theoretical intensities listed in Table IV were calculated. Upon comparing the theoretical and experimental intensities of the NaCl reflections, it can be seen that there is a small (100) texture.[19]

The experimental values of the atomic scattering factors for all the copper and iron specimens calculated from the NaCl standardization, together with the f values for NaCl are given in Table V.[20] There is good agreement between theoretical free-atom f values for all copper and iron reflections.

SUMMARY AND CONCLUSIONS

The intensities of Bragg reflections of iron and copper pressed powders have been measured with monochromatic Fe K_α radiation. By investigating the effect of different methods of sample preparation upon integrated intensities and comparing these intensities with theoretical predictions, it was concluded that the specimens were free of extinction, preferred orientation or surface roughness effects. A comparison of the measured iron intensities relative to those from copper shows that the form factor for metallic iron agrees with that of the free atom, assuming the free atom values for the form factor of copper. The most precise iron reflection, the (110), has a measured atomic scattering factor relative to copper estimated at 18.9±0.3. This variation corresponds to a relative intensity measurement of uncertainty ±3%. The theoretical value based on the calculation of Freeman and Wood[13] is 18.77. The ratio of the copper (111) to iron (110) measured with molybdenum radiation gave $f(110)$ of iron in good agreement with the Fe K_α results.

From the measured intensities of the Fe K_α Bragg reflections of lightly pressed NaCl powder, an average parameter was obtained which allowed the copper and iron intensities to be reduced to atomic scattering factors relative to sodium chloride. Relative to NaCl, the Fe(110) scattering factor is estimated as 19.0±0.6 and the Cu(111) as 22.3±0.6, compared to theoretical values of 18.77 and 22.16, respectively. The limits correspond to an estimated precision of ±6% in the measured intensities. The uncertainty in the reported values involved, in addition to the actual errors in the observed intensities, the uncertainty in the mass absorption coefficients, the Debye temperature factors and the theoretical scattering factors for the standard.

We wish to report in this paper only the measured atomic scattering factors for iron and copper and conclude that these are consistent with calculations

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[18] Na^+ values are taken from A. J. Freeman, Acta Cryst. **12**, 261 (1959). Cl^- values are taken from reference 12.

[19] The experimental ratio $P(200)/P(400)$ is about 11% higher than predicted by theory. This is attributed in part to the inaccuracy in the (400) intensity measurement. The (400) is the weakest of all the measured reflections and consequently has the most unfavorable signal-to-background ratio. In addition, the amount of texture is not necessarily uniform across the specimen surface and consequently the (400) and (200) reflections could involve different degrees of preferred orientation.

[20] The f values for NaCl and samples B and E differ slightly from those presented in reference 2. The experimental value of $\mu_\rho(\mathrm{NaCl})=148$ was used in the present calculations instead of the tabulated value. In addition, after reconsideration of the experimental methods involved, it was decided to use the Waller and James value of 0.227 A for the rms amplitude for NaCl rather than the Wasastjerna value of 0.239 A [J. A. Wasastjerna, Soc. Sci. Fennica, Commentationes Phys.-Math. **13**, No. 5 (1945)]. The experimental μ_ρ value would give closer agreement with the free-atom f values for iron and copper by about 2%, while the different rms amplitude would be of opposite sense. The net change in the iron and copper f values over those in reference 2 is the order of -1%.

from self-consistent wave functions of the respective isolated atoms. These results are not in agreement with those reported by Weiss and DeMarco from their measurements on a single crystal of iron. In order to facilitate a comparison of the two results, we present our measurements in terms of an *effective* number of *3d* electrons. To do this, we assume that the contribution to the atomic scattering factor of a *3d* electron is independent of the number of such electrons in the atom and is the value calculated for the free atom. The contribution of a free atom-like *3d* electron at the (110) reflection of iron is 0.60 unit,[13] and at the (111) reflection of copper approximately 0.62 unit.[1] In terms of these contributions, the number of *effective* *3d* electrons in iron is estimated at 6.3 ± 1.0 and in copper 10.2 ± 1.0,[20] while the reported Weiss and DeMarco value for iron is (2.3 ± 0.3) *3d* electrons.

The reduction of the experimental iron scattering factors to a number of *3d* electrons involves, of course, a comparison with theoretical calculations for the isolated atom. The present results were compared to the free-atom values for iron calculated by Freeman and Wood since their values are probably the most reliable. However, it has been pointed out to the writer that their open-configuration method might tend to overemphasize exchange. A recent publication of iron scattering factors[21] calculated from the nonexchange wave functions of Manning and Goldberg are lower than the Freeman and Wood values in the vicinity of the (110) reflection. Comparing the present data to these nonexchange form factors would give about 7.5 as the number of *effective* *3d* electrons in iron, and a comparison with the Thomas-Fermi-Dirac values calculated by Thomas and Umeda[22] would give about 7 *3d* electrons.

We bring in these latter comparisons to stress the point, that considerable caution must be exercised when deducing the number of electrons in an atom from an experimental value of the atomic form factor.

ACKNOWLEDGMENTS

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APPENDIX

The absorption coefficients for Fe K_α were experimental values obtained in the following manner: The area densities of uniform foils of copper and iron were measured directly. The intensity of monochromatic Fe K_α transmitted through the foils was measured. From the ratio of these intensities and the measured area densities, a linear relation between the two mass absorption coefficients can be obtained. Using a multiple-foil technique with the copper, the absolute value $\mu_\rho=96.3$ cm^2/g was obtained. From this value and the experimental linear relation, the value $\mu_\rho=70.1$ cm^2/g for iron was obtained. For the purposes of this work, knowledge only of the ratios of the mass absorption coefficients is necessary. For the foil sizes chosen, the error in this ratio can be shown to be about 0.14 times the sum of the errors in the measured intensity ratio, and the single μ_ρ value for copper. Thus, this difference technique provides a simple and accurate means of obtaining the ratios of absorption coefficients and is especially useful in obtaining these ratios when it is inconvenient to obtain thin, uniform foils of the absorber. It is with this difference technique that the mass absorption coefficient of NaCl was measured. Two single crystal slabs were ground to thickness of approximately 0.025 cm and a powder compress to about 0.040 cm (a powder compressed with 20 000 psi could be readily ground and polished). The two single-crystal values of μ_ρ(NaCl) relative to copper were 146 and 150, while the powder gave 148 cm^2/g. The average value of 148 cm^2/g was used. The coefficients for Fe K_α radiation in Table II are all relative to the measured value of 96.3 for Cu. The values for iron and copper for Mo K_α radiation were obtained with a fluorescence spectrometer using the difference technique. The value μ_ρCu$=51.0$ cm^2/g is taken from Compton and Allison[23] and μ_ρFe$=38.9$ cm^2/g is experimental, relative to the copper value, the ratio being insensitive to the tabulated copper value.

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[21] Veenendaal, MacGillavry, Stam, Potters, and Romgens, Acta Cryst. **12**, 242 (1959).

[22] L. Thomas and K. Umeda, J. Chem. Phys. **26**, 293 (1957).

[23] A. H. Compton and S. K. Allison, *X-Rays in Theory and Experiment* (D. Van Nostrand, Inc., New York, 1935).