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X-ray Attenuation Coefficients for Magnesium and Aluminium in the Range 0·3 to 1·3 Å

By J. L. Lawrence

School of Physical Sciences, University of St Andrews, St Andrews, Fife, Scotland

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Abstract

X-ray mass attenuation coefficients for polycrystalline samples of magnesium and aluminium have been measured over the wavelength range 0·3 to 1·3 Å. Estimates of the contributions from Bragg, thermal diffuse and Compton scattering have been made and the photoelectric coefficients deduced and compared with theoretical values. The photoelectric coefficient is found to be proportional to $\lambda^{3\cdot09}$ for both materials.

Introduction

The interaction between an X-ray beam and a crystalline material involves many physical processes. For the large wavelengths usually employed in the investigation of charge densities, the predominant effect is usually the photoelectric process but significant contributions to measured X-ray absorption coefficients come from Bragg, thermal and Compton scattering. Further, the amount of Bragg scattering depends on whether the sample is of a single crystal or a polycrystalline type and, in some cases, on preferred orientation and impurity content. This paper describes the measurement of the X-ray absorption coefficients for polycrystalline samples of magnesium and aluminium over the wavelength range from 0·3 to 1·3 Å and the deduction from these results of the photoelectric coefficients.

Experimental

The X-ray mass attenuation coefficients were measured in a similar manner to those of graphite (Berry & Lawrence, 1979), based on the method of Lawrence & Mathieson (1976). The radiations used were the white radiation and some characteristic L lines from a tungsten target, each wavelength being measured by determining the 2θ angle through which a large crystal of lithium fluoride was rotated in moving from the (002) to the $(00\bar{2})$ diffracting position, assuming a(LiF) = 4·0262 (1) Å (Thewlis, 1955).

The method involved measuring the attenuation of an X-ray beam through specimens of uniform thickness as the angle between the incident beam and the normal to the surface, φ, was varied, nine values of φ being used at each wavelength. For the short wavelengths, the φ values were chosen such that $1/\cos\varphi = n$, where n took all integer and half integer values between one and five. For longer wavelengths, the attenuation became too great to allow transmission of the beam for $n = 5$ and the range of n was progressively reduced from 1 to 5 to 1 to 1·5 at the largest wavelengths, nine intensities being measured in each range.

The apparatus, method of monochromatizing the main beam, considerations of the spectral purity of the main beam, methods of detection and of measurement of intensities, the tolerances of the wavelength measurements and the final determination of the linear absorption coefficient μ were as for the graphite study (Berry & Lawrence, 1979).

Both samples were supplied by Goodfellow Metals of Cambridge. The main impurities in the aluminium sample (99·999%) were iron, magnesium and silicon, all having a concentration of less than three parts per million by weight and thus a negligible effect on the absorption coefficients. The magnesium sample was less pure (99·9%), the main impurity being silicon which can be neglected since its atomic number is similar to magnesium. However, the sample also contained 0·01% of both iron and chromium, resulting in the absorption coefficients being overestimated by 0·2% over the wavelength range and for this a small correction was applied. Diffraction photographs of the samples showed no evidence of preferred orientation.

The average thicknesses of the samples over the surfaces traversed by the X-ray beam were measured using a micrometer and verified by mass and area measurements.

$$t(\mathrm{Mg}) = 0\cdot995\ (3)\ \mathrm{mm},$$

$$t(\mathrm{Al}) = 0\cdot970\ (5)\ \mathrm{mm}.$$

The densities were taken to be

$$\rho(\mathrm{Mg}) = 1\cdot738 \times 10^{-3}\ \mathrm{g\ mm^{-3}},$$

$$\rho(\mathrm{Al}) = 2\cdot699 \times 10^{-3}\ \mathrm{g\ mm^{-3}}.$$

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Results

Tables 1 and 2 show the values of the measured mass absorption coefficients $(\mu/\rho)_o$ at the various wavelengths, the magnesium coefficients being corrected for impurities. A study of a graph of $\ln\lambda$ against $\ln(\mu/\rho)_o$ for magnesium showed a linear relation between $\ln\lambda$ and $\ln(\mu/\rho)_o$ at wavelengths greater than 0·65 Å, the equation of the best straight line being

$$\ln(\mu/\rho)_o = 2{\cdot}945\ (7)\ln\lambda + 2{\cdot}405\ (2).$$

For aluminium, the graph was also linear above 0·65 Å, the equation of the best straight line being

$$\ln(\mu/\rho)_o = 2{\cdot}940\ (7)\ln\lambda + 2{\cdot}612\ (2).$$

Thus the wavelength dependences of the mass absorption coefficients of polycrystalline samples of magnesium and aluminium are the same. Weiss (1966) gives possible values of 2·75 (5), 2·91 (5) and 2·89 (5) for aluminium and 2·85 (5) for magnesium.

There have been few recent measurements of the mass absorption coefficient of magnesium reported. Table 3 shows the results of Millar & Greening (1974) (MG) over a range of wavelengths, a result of DeMarco & Suortti (1971) (DS) for Mo $K\alpha$ radiation and the values quoted in *International Tables for X-ray Crystallography* (1974) (IT) along with the results from this study, interpolated if necessary either from a graph or using the above equation. There is excellent agreement between values from this study and the MG values, while the IT values are on average 2% lower.

The wavelength dependence of the mass absorption coefficient of aluminium has been investigated by Hopkins (1959) and by Millar & Greening (1974) (MG). The results of Hopkins are not significantly different from the results quoted here but the random errors quoted are very large, up to 10% at the lower

Table 1. *The experimental mass absorption coefficient of polycrystalline magnesium* $(\mu/\rho)_o$, *the single crystal value* $(\mu/\rho)_1$, *the contribution from Bragg and thermal diffuse scattering,* $(\mu/\rho)_s$, *from Compton scattering,* $(\mu/\rho)_c$, *and the resulting photoelectric coefficient,* $(\mu/\rho)_p$, *all in units of* 10^2 mm² g⁻¹

λ (Å)	$(\mu/\rho)_o$	$(\mu/\rho)_1$	$(\mu/\rho)_s$	$(\mu/\rho)_c$	$(\mu/\rho)_p$
0·306	0·461 (5)	0·443	0·043	0·159	0·259
0·353	0·657 (5)	0·633	0·055	0·156	0·446
0·398	0·814 (4)	0·784	0·067	0·153	0·594
0·448	1·130 (5)	1·092	0·080	0·151	0·899
0·481	1·364 (8)	1·320	0·089	0·150	1·125
0·508	1·60 (1)	1·55	0·10	0·15	1·35
0·560	2·09 (1)	2·03	0·11	0·15	1·83
0·601	2·49 (1)	2·42	0·12	0·14	2·23
0·655	3·20 (1)	3·12	0·14	0·14	2·92
0·710	4·03 (2)	3·94	0·16	0·14	3·73
0·757	4·86 (2)	4·76	0·17	0·14	4·55
0·802	5·77 (2)	5·66	0·19	0·13	5·45
0·842	6·71 (2)	6·59	0·20	0·13	6·38
0·884	7·60 (2)	7·47	0·22	0·13	7·25
0·921	8·71 (4)	8·57	0·23	0·13	8·35
0·970	10·05 (4)	9·90	0·25	0·12	9·68
1·010	11·34 (6)	11·18	0·26	0·12	10·96
1·065	13·22 (8)	13·05	0·28	0·12	12·82
1·099	14·63 (8)	14·45	0·29	0·12	14·22
1·165	17·23 (8)	17·03	0·31	0·11	16·81
1·183	18·1 (1)	17·9	0·32	0·11	17·7
1·244	21·2 (1)	21·0	0·34	0·11	20·8
1·281	23·2 (2)	23·0	0·35	0·11	22·7

Table 2. *The experimental mass absorption coefficient of polycrystalline aluminium* $(\mu/\rho)_o$, *the single crystal value* $(\mu/\rho)_1$, *the contribution from Bragg and thermal diffuse scattering,* $(\mu/\rho)_s$, *from Compton scattering,* $(\mu/\rho)_c$, *and the resulting photoelectric coefficient,* $(\mu/\rho)_p$, *all in units of* 10^2 mm² g⁻¹

λ (Å)	$(\mu/\rho)_o$	$(\mu/\rho)_1$	$(\mu/\rho)_s$	$(\mu/\rho)_c$	$(\mu/\rho)_p$
0·315	0·587 (8)	0·556	0·063	0·147	0·377
0·371	0·819 (6)	0·776	0·082	0·145	0·592
0·423	1·179 (7)	1·125	0·103	0·142	0·934
0·459	1·491 (9)	1·428	0·113	0·140	1·238
0·508	1·91 (1)	1·83	0·13	0·14	1·64
0·567	2·58 (2)	2·49	0·16	0·14	2·28
0·609	3·18 (2)	3·08	0·17	0·13	2·88
0·669	4·19 (2)	4·07	0·20	0·13	3·86
0·710	4·99 (2)	4·85	0·22	0·13	4·64
0·766	6·16 (3)	6·01	0·24	0·13	5·79
0·806	7·29 (4)	7·12	0·26	0·12	6·91
0·862	8·76 (5)	8·57	0·28	0·12	8·36
0·921	10·75 (7)	10·54	0·31	0·12	10·32
0·971	12·5 (1)	12·3	0·33	0·12	12·1
1·056	16·1 (1)	15·8	0·38	0·11	15·6
1·100	18·1 (1)	17·8	0·39	0·11	17·6
1·170	21·7 (1)	21·4	0·42	0·11	21·2
1·244	25·8 (2)	25·5	0·46	0·11	25·2
1·281	28·2 (2)	27·9	0·48	0·10	27·6

Table 3. *Comparison of experimental mass absorption coefficients of magnesium,* $(\mu/\rho)_o$ (10^2 mm² g⁻¹), *with other studies* $(\mu/\rho)_x$

λ (Å)	$(\mu/\rho)_o$	$(\mu/\rho)_x$	Reference*
0·492	1·45 (1)	1·42 (1)	MG
0·497	1·51 (1)	1·44 (3)	IT
0·521	1·71 (1)	1·63 (3)	IT
0·536	1·84 (1)	1·79 (1)	MG
0·546	1·93 (1)	1·86 (4)	IT
0·561	2·10 (1)	2·00 (1)	IT
0·587	2·33 (1)	2·28 (5)	IT
		2·29 (1)	MG
0·615	2·67 (1)	2·60 (5)	IT
0·632	2·88 (1)	2·83 (6)	IT
0·644	3·03 (1)	3·09 (2)	MG
0·710	4·03 (2)	3·98 (8)	IT
		4·06 (2)	MG
		3·98 (1)	DS
0·788	5·48 (2)	5·49 (2)	MG
0·927	8·82 (4)	8·88 (2)	MG
1·106	14·88 (8)	14·96 (6)	MG
1·256	21·7 (1)	22·0 (1)	MG
1·295	23·7 (1)	24·2 (4)	IT

* MG, Millar & Greening (1974); DS, DeMarco & Suortti (1971); IT, *International Tables for X-ray Crystallography* (1974).

wavelengths. The MG measurements were made using certain characteristic wavelengths and have an average random error of 0·4%. Other measurements have been made by Cooper (1965) (C) with Mo $K\alpha$ and Ag $K\alpha$ radiations, by DeMarco & Suortti (1971) (DS) with Mo $K\alpha$ radiation and by Alonzo & Grodzins (1965) (AG) using 14·4 keV γ-rays. Table 4 shows these authors' results with the corresponding values from this study, interpolated if necessary to the required wavelength. Also shown are the values quoted in *International Tables for X-ray Crystallography* (1974) which have been given uncertainties of 2%.

In determining the true polycrystalline X-ray mass absorption coefficient, the avoidance of systematic error is of the utmost importance. The main sources of error in absorption measurements arise from non-monochromaticity of the main beam, preferred orientation in the samples, errors in the thickness measurements and the presence of impurities. The experimental technique used ensured that significant contributions from other wavelengths would have been detected and the diffraction patterns ruled out preferred orientation. The errors in the thickness measurements are the main sources of error for both samples. The MG aluminium results seem to be systematically 1% higher than the results quoted here and such a systematic difference could be partly accounted for by the uncertainties in the thickness measurements, provided they are true measures of the uncertainties in these measurements and not a measure of variations in the thicknesses of the samples since these should not have systematic effects on (μ/ρ). Corrections had been applied to the MG results for very small amounts of impurities.

Table 4. *Comparison of experimental mass absorption coefficients of aluminium,* $(\mu/\rho)_o$ (10^2 mm^2 g^{-1}), *with other studies* $(\mu/\rho)_x$

λ (Å)	$(\mu/\rho)_o$	$(\mu/\rho)_x$	
0·492	1·76 (1)	1·77 (1)	MG
0·497	1·80 (1)	1·81 (4)	IT
0·521	2·05 (2)	2·06 (4)	IT
0·536	2·24 (2)	2·25 (1)	MG
0·546	2·33 (2)	2·35 (5)	IT
0·561	2·51 (2)	2·54 (5)	IT
		2·65 (1)	C
0·587	2·86 (2)	2·89 (6)	IT
		2·90 (6)	MG
0·615	3·28 (2)	3·30 (7)	IT
0·632	3·54 (2)	3·59 (7)	IT
0·644	3·74 (2)	3·78 (2)	MG
0·710	4·99 (2)	5·04 (10)	IT
		5·04 (1)	MG
		5·10 (2)	C
		5·01 (2)	DS
0·788	6·76 (2)	6·83 (2)	MG
0·863	8·84 (5)	8·73 (2)	AG
0·927	10·90 (7)	11·02 (3)	MG
1·106	18·3 (1)	18·7 (1)	MG
1·256	26·6 (2)	27·2 (1)	MG
1·295	29·1 (2)	30·0 (6)	IT

MG, Millar & Greening (1974); C, Cooper (1965); DS, DeMarco & Suortti (1971); AG, Alonzo & Grodzins (1965); IT, *International Tables for X-ray Crystallography* (1974).

For both materials, the values quoted here for Mo $K\alpha$ radiation agree with those of DeMarco & Suortti (1971) which were corrected for impurity content but the values of Cooper (1965) for aluminium at Ag $K\alpha$ and Mo $K\alpha$ wavelengths are significantly greater. However, there is no information regarding impurities for the samples used in this study.

Absorption measurements from polycrystalline samples include contributions from Bragg scattering, thermal diffuse scattering and Compton scattering as well as photoelectric absorption. The mass attenuation coefficients applicable to single crystals can be considered to be those for polycrystalline samples with corrections for Bragg scattering. DeMarco & Suortti (1971) have given the appropriate equations for determining the contribution of Bragg scattering to the mass absorption coefficient in the case of a cubic crystal structure and the equation has been adapted for the hexagonal case for magnesium. Using the notation of DeMarco & Suortti (1971), the contribution to Bragg scattering in the hexagonal case is

$$\sigma_{\text{Bragg}} = \frac{\lambda^2 r_o^2}{2V_c^2} \sum_{hkl} \frac{K(\text{pol})}{\chi_{hkl}} j_{hkl} F_{hkl}^2 \exp(-2M),$$

where $\chi_{hkl} = [4(h^2 + k^2 + hk)/3a^2 + l^2/c^2]^{1/2}$.

In Tables 1 and 2 the 'single crystal' mass absorption coefficients, $(\mu/\rho)_1$, are listed and it can be seen that the effect of Bragg scattering ranges from about 5% at the low wavelengths to 1% at the high wavelengths. The Debye–Waller factors used in the calculation were $\bar{B}$(Mg) = 1·43 Å^2 (Capkova & Sedivy, 1977) and B(Al) = 0·849 Å^2 (Dingle & Medlin, 1972).

Approximate corrections for thermal diffuse scattering can be made if it is assumed (DeMarco & Suortti, 1971) that the total thermal diffuse scattering is equal to the scattering lost from the Bragg reflection due to thermal vibrations. The correction for Bragg scattering and for thermal diffuse scattering can then be calculated assuming only Bragg scattering is present but with zero Debye–Waller factors. These combined contributions, $(\mu/\rho)_s$, are also shown in Tables 1 and 2 and can be seen to vary over the wavelength range from 10 to 1·5%.

Azaroff, Kaplow, Kato, Weiss, Wilson & Young (1974) have calculated the Compton cross section for Mo $K\alpha$ radiation and the contribution of Compton scattering to the mass absorption coefficient, $(\mu/\rho)_c$, has been found to be

$$\text{magnesium } (\mu/\rho)_c = 0{\cdot}14 \times 10^2 \text{ mm}^2 \text{ g}^{-1},$$

$$\text{aluminium } (\mu/\rho)_c = 0{\cdot}13 \times 10^2 \text{ mm}^2 \text{ g}^{-1}.$$

Hubbell, Veigele, Briggs, Brown, Cromer & Howerton (1975) have calculated incoherent cross sections at photon energies of 10, 15 and 20 keV. These show that the contribution to the absorption coefficient from Compton scattering decreases linearly with increasing wavelength over this range and give a result for Mo $K\alpha$ radiation which is in agreement with that of Azaroff *et al.* (1974). Assuming this linearity, the contributions from Compton scattering were calculated and are shown in Tables 1 and 2.

Finally, the observed mass absorption coefficients for the polycrystalline samples were corrected for these effects and the photoelectric absorption coefficient, $(\mu/\rho)_p$, calculated.

Stephenson (1976) has calculated photoelectric mass absorption coefficients for magnesium and aluminium from screened hydrogen-like eigenfunctions, determined from ionization potential screening constants for incomplete shells, over a range of characteristic wavelengths. Tables 5 and 6 show the interpolated results from the present study, $(\mu/\rho)_o$, the correction factors $(\mu/\rho)_s$ and $(\mu/\rho)_c$, the resulting photoelectric absorption coefficient $(\mu/\rho)_p$, along with the results of Stephenson (1976), $(\mu/\rho)_{t_1}$. $(\mu/\rho)_{t_2}$ are results from Hildebrandt, Stephenson & Wagenfeld (1975) which were calculated at the same wavelengths by a similar method but using screening constants which were taken from averages of full shell values.

Table 5. *The experimental mass absorption coefficient of magnesium* $(\mu/\rho)_o$, *the contribution from Bragg and thermal diffuse scattering,* $(\mu/\rho)_s$, *from Compton scattering,* $(\mu/\rho)_c$ *and the resulting photoelectric coefficient,* $(\mu/\rho)_p$, *compared with theoretical results of Stephenson* (1976), $(\mu/\rho)_{t_1}$, *and Hildebrandt, Stephenson & Wagenfeld* (1975), $(\mu/\rho)_{t_2}$, *in units of* 10^2 mm^2 g^{-1}

λ (Å)	$(\mu/\rho)_o$	$(\mu/\rho)_s$	$(\mu/\rho)_c$	$(\mu/\rho)_p$	$(\mu/\rho)_{t1}$	$(\mu/\rho)_{t2}$
0·498	1·51 (1)	0·09	0·15	1·27	1·24	1·21
0·561	2·10 (1)	0·11	0·15	1·84	1·79	1·74
0·632	2·88 (1)	0·13	0·14	2·61	2·60	2·53
0·710	4·03 (2)	0·16	0·14	3·73	3·70	3·62
1·129	15·78 (8)	0·30	0·12	15·36	15·19	14·83
1·256	21·7 (1)	0·34	0·11	21·3	20·84	20·36
1·295	23·7 (1)	0·36	0·11	23·2	22·96	22·44

Table 6. *The experimental mass absorption coefficient of aluminium* $(\mu/\rho)_o$, *the contribution from Bragg and thermal diffuse scattering,* $(\mu/\rho)_s$, *from Compton scattering,* $(\mu/\rho)_c$ *and the resulting photoelectric coefficient,* $(\mu/\rho)_p$, *compared with theoretical results of Stephenson* (1976) $(\mu/\rho)_{t_1}$, *and Hildebrandt, Stephenson & Wagenfeld* (1975), $(\mu/\rho)_{t_2}$, *in units of* 10^2 mm^2 g^{-1}

λ (Å)	$(\mu/\rho)_o$	$(\mu/\rho)_s$	$(\mu/\rho)_c$	$(\mu/\rho)_p$	$(\mu/\rho)_{t1}$	$(\mu/\rho)_{t2}$
0·498	1·80 (1)	0·13	0·14	1·53	1·60	1·56
0·561	2·51 (2)	0·15	0·14	2·22	2·29	2·23
0·632	3·54 (2)	0·18	0·14	3·22	3·33	3·26
0·710	4·99 (2)	0·22	0·13	4·64	4·73	4·62
1·129	19·5 (1)	0·40	0·11	19·0	19·18	18·77
1·256	26·6 (2)	0·46	0·11	26·0	26·24	25·67
1·295	29·1 (2)	0·48	0·10	28·5	28·88	28·35

For magnesium, agreement between $(\mu/\rho)_p$ and $(\mu/\rho)_{t_1}$ is excellent, better than that between $(\mu/\rho)_p$ and $(\mu/\rho)_{t_2}$, the latter results being rather low. For aluminium the better agreement at short wavelengths is between $(\mu/\rho)_p$ and $(\mu/\rho)_{t_2}$ but at higher wavelengths, the $(\mu/\rho)_{t_2}$ values again appear too low. However, Stephenson (1975), using averaged screening constants, gives values of $2{\cdot}24 \times 10^2$ and $4{\cdot}62 \times 10^2$ mm^2 g^{-1} for the photoelectric mass absorption coefficients at 0·561 and 0·710 Å respectively, in excellent agreement with $(\mu/\rho)_p$.

Graphs of $\ln\lambda$ against $\ln(\mu/\rho)_p$ are linear over the whole range of wavelengths, unlike the graphs of $\ln\lambda$ against $\ln(\mu/\rho)_o$. The equations of the best straight lines are

$$\text{magnesium } \ln(\mu/\rho)_p = 3{\cdot}087\ (10) \ln\lambda + 2{\cdot}367\ (5),$$

$$\text{aluminium } \ln(\mu/\rho)_p = 3{\cdot}086\ (12) \ln\lambda + 2{\cdot}567\ (6).$$

The gradients are again identical, the applied corrections having increased them from 2·94 to 3·09. This similarity between these elements is not surprising considering their adjacent position in the periodic table.

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