HARADA, J. & SAKATA, M. (1974). *Acta Cryst.* A**30**, 77–82.
KITAIGORODSKY, A. I. (1973). *Molecular Crystals and Molecules*. New York: Academic Press.
KROON, P. A. (1977). Thesis, State Univ. of Groningen, The Netherlands.
NYE, J. F. (1969). *Physical Properties of Crystals*. Oxford: Clarendon Press.
REYNOLDS, P. A. (1973). *J. Chem. Phys.* **59**, 2777–2786.
WITH, G. DE (1976). *J. Appl. Cryst.* **9**, 502.
WITH, G. DE, HARKEMA, S. & FEIL, D. (1976). *Acta Cryst.* B**32**, 3178–3184.

Acta Cryst. (1979). A**35**, 316–318

X-ray Attenuation Coefficients of Graphite in the Range 0·40 to 1·54 Å

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Abstract

The mass attenuation coefficients of graphite have been measured at 23 different wavelengths in the range 0·40 to 1·54 Å. Above 1 Å, the coefficient is proportional to $\lambda^{2\cdot88}$.

Introduction

A method of measuring accurate X-ray attenuation coefficients has been described by Lawrence & Mathieson (1976) and has been used to measure the attenuation coefficient of silicon for Mo $K\alpha$ and Cu $K\alpha$ radiations (Lawrence, 1977). The method consists of measuring the intensity, I, of a monochromatic beam of X-rays transmitted through a regular specimen of crystal of thickness t as the angle between the incident beam and the normal to the crystal face, φ, is varied. Thus, if I_0 is the incident intensity,

$$I = I_0 \exp(-\mu t/\cos\varphi)$$

and a plot of $\ln I$ *versus* $1/\cos\varphi$ will yield values of μt from which μ can be deduced. μ is of course dependent on the wavelength of the radiation and the presence of any spectral components in the main beam will be detected by departures from linearity of the plot.

In this study, the method was applied to an investigation of the wavelength dependence of the absorption coefficients of graphite in the wavelength range 0·40 to 1·54 Å.

Experimental

The crystal used was a single crystal of pyrolytic graphite of uniform thickness and rectangular cross-section 20 × 10 mm. Its average thickness over the surface traversed by the X-ray beam was measured using a linear differential transducer, carefully calibrated using slip gauges, and found to be $t = 0\cdot922$ (3) mm.

The apparatus used was similar to that employed in the measurement of the attenuation coefficients of silicon (Lawrence, 1977), the main beam being first diffracted by the (111) planes of a near-perfect silicon crystal and collimated by two circular apertures, of radius 0·5 mm, separated by 150 mm, one aperture being 60 mm away from the X-ray source. The crystal was centred on a spectrometer which was capable of being rotated through a minimum of 3″ of arc.

The wavelengths used were narrow ranges of the white radiation from a tungsten target, certain tungsten characteristic L lines, the $K\alpha$ and $K\beta$ lines of copper and silver targets and the $K\alpha$ line of a molybdenum target. In each case the wavelength was measured by determining the 2θ angle through which the graphite crystal was rotated in moving from the $000\bar{2}$ to the 0002 reflecting position, assuming c(graphite) = 6·7079 Å (Nelson & Riley, 1945). The diffraction profiles of these reflections were fairly wide, about 10′ of arc, and were found to be independent of wavelength. On the instrument used, the centres of each peak could be determined to ± 12″, giving an uncertainty in each characteristic wavelength of 0·001 Å. [The wavelengths quoted for the characteristic lines will not necessarily correspond exactly to the accepted values since, for the copper, molybdenum and silver targets, different proportions of the α_1 and α_2 lines have been gathered by the collimator and, for the tungsten characteristic lines, more than one line could be present.] For the wavelengths obtained from the white radiation of the tungsten tube, the divergence through the collimator was 0·0014 rad, giving a wavelength spread of less than 0·01 Å.

The φ angles were chosen such that $1/\cos\varphi = n$, where n took all integer and half-integer values between

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1 and 5. The (000*l*) planes were parallel to the surface of the crystal and the crystal was set to $n = 1$ from the known position attained during the wavelength measurement. Any uncertainty in the determination of the wavelength will result in an error in φ and the error quoted leads to a maximum error of 0·6% in the path length for $n = 5$. The averaging equivalent transmitted intensities at $\pm \varphi$ and $180 \pm \varphi$ should make this error negligible. If the $n = 1$ position were fixed accurately for a characteristic wavelength and used thereafter, this error would be eliminated.

Of fundamental importance in absorption measurements is the spectral purity of the incident beam. For all readings at all wavelengths the excitation voltage of the X-ray tube was kept as far below the excitation voltage of the $\lambda/2$ wavelength as possible to ensure no component of the anharmonic 222 reflection from the silicon crystal was present in the main beam.

The intensities were measured by a scintillation counter attached to a Siemens counting chain, all intensities being corrected for dead-time. For each wavelength, the number of counts per second at the $n = 1$ position was less than 4000 and each intensity was measured for sufficient time to accumulate 40 000 counts (*i.e.* 0·5% counting statistics error). For the largest wavelength, the counting times for the larger n values were up to 6 min. Four equivalent intensities for each value of n were measured and averaged and the μt value was determined from the least-squares fit of $\ln I$ against n.

Results

Table 1 shows the values of μ/ρ [$\rho = 2{\cdot}266 \times 10^{-3}$ g mm^{-3} (Moore, 1973)] and the corresponding wavelengths. The errors in the μ/ρ values combine the standard deviations obtained from the least-squares fit with the error in the thickness measurement.

Consideration has to be given to the Bragg scattering processes which occurred during the absorption measurements and Calvert, Killean & Mathieson (1976) have studied the transmitted-beam absorption pattern for a similar sample of pyrolytic graphite using Cu $K\alpha_1$ radiation. In their experiment, intensities of the incident beam and the transmitted beam at various values of φ were measured and values of the effective absorption coefficient μ' deduced for each position. In the range $0° < \varphi < 3°$, no Bragg scattering was taking place and measurements here were assumed to yield the normal attenuation coefficient of carbon, μ.

The experiment described here included a reading at $\varphi = 0$ (*i.e.* $n = 1$) and at all other points there will be scattering. The greatest scattering will occur when the 000*l* reflections are in the scattering position but these reflections were easily predicted and the corresponding intensities eliminated from the calculation of the absorption coefficient. Also, any transmitted intensity which lay a significant distance below the straight line through the other intensity values was assumed to be affected by scattering and eliminated.

The effective absorption coefficient μ' measured by Calvert, Killean & Mathieson (1976) for Cu $K\alpha_1$ radiation outside the regions of large scattering was fairly constant and about 1% higher than μ. In these regions, therefore, the scattering can be considered to be a small additional absorption factor. DeMarco & Suortti (1971) have investigated the contribution of Bragg scattering to the total attenuation coefficient and have shown that for Ag, Mo and Cu $K\alpha$ radiations, the contribution of Bragg scattering to attenuation for carbon is 9, 8 and 2·5% respectively. However, the contribution from Bragg scattering to the results presented here will be much less since the 000*l* reflection positions were avoided.

The most commonly used source of attenuation coefficients is *International Tables for X-ray Crystallography* (1974) but since these values include full contributions from Bragg scattering they cannot be

Table 1. *Mass absorption coefficients of graphite*

λ (Å)	$\frac{\mu}{\rho}$ (10² mm² g⁻¹)	λ (Å)	$\frac{\mu}{\rho}$ (10² mm² g⁻¹)
0·399	0·215 (10)	0·856	0·804 (10)
0·464	0·267 (3)	0·917	0·946 (7)
*0·497	0·277 (5)	0·978	1·113 (8)
0·553	0·327 (8)	*1·031	1·282 (6)
*0·559	0·337 (4)	*1·096	1·501 (8)
0·583	0·356 (4)	*1·134	1·710 (10)
0·623	0·391 (5)	*1·218	2·039 (14)
0·664	0·458 (5)	*1·280	2·403 (11)
*0·711	0·513 (4)	*1·392	3·060 (18)
0·728	0·525 (6)	*1·421	3·204 (17)
0·786	0·632 (7)	*1·477	3·584 (16)
		*1·541	4·121 (16)

* Denotes characteristic radiation.

Table 2. *Mass absorption coefficients of carbon* (10² mm² g⁻¹) *for some characteristic radiations*

(I) *International Tables* (1974). (II) *International Tables* (1974) corrected for Bragg scattering. (III) Results from this study.

Radiation	(I)	(II)	(III)
Ag $K\beta$	0·301	0·274	0·277
Pd $K\beta$	0·319	0·290	0·298
Rh $K\beta$	0·340	0·308	0·320
Ag $K\alpha$	0·354	0·320	0·337
Pd $K\alpha$	0·379	0·343	0·364
Rh $K\alpha$	0·408	0·370	0·389
Mo $K\beta$	0·429	0·389	0·411
Mo $K\alpha$	0·535	0·488	0·513
Zn $K\beta$	2·493	2·402	2·46
Cu $K\beta$	3·093	2·996	3·06
Zn $K\alpha$	3·399	3·294	3·32
Ni $K\beta$	3·878	3·760	3·76
Cu $K\alpha$	4·219	4·120	4·12

directly compared with the graphite results. The values for carbon given in the *International Tables* have been corrected for Bragg scattering by the method suggested by DeMarco & Suortti (1971). Table 2 shows the published and the corrected values of μ/ρ for various characteristic radiations along with the value obtained in this study, either directly using the same characteristic radiation or interpolated from the results in Table 1.

If there is assumed to be a 2% error in the values in the *International Tables*, these values corrected for Bragg scattering are not significantly different from the measured values, although in all cases the measured values are higher, perhaps owing to the small amount of Bragg scattering.

The value of μ/ρ for Cu $K\alpha_1$ radiation, 4·121 (16) × 10^2 mm^2 g^{-1}, can be compared with the value due to Chipman (1955) of 4·15 × 10^2 mm^2 g^{-1} and the value due to Calvert, Killean & Mathieson (1975) of 4·080 (12) × 10^2 mm^2 g^{-1}. (The standard deviation of the latter result is higher than that quoted by the authors since the standard deviation they quote did not include the standard deviation in the thickness measurement.)

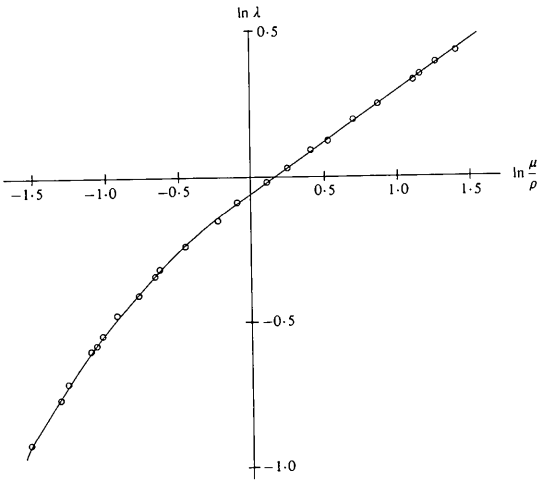

Fig. 1. Graph of ln (μ/ρ) against ln λ for graphite.

A graph of ln (μ/ρ) against ln λ is shown in Fig 1. Above 1 Å the graph is fairly linear having a gradient of 2·88 (2), showing that in this range

$$\frac{\mu}{\rho} = C\lambda^{2\cdot88}$$

where C is a constant. Stiglich, Weiss & Hansen (1974) have deduced a value of 2·73 from experimental measurements.

References

CALVERT, L. D., KILLEAN, R. C. G. & MATHIESON, A. MCL. (1975). *Acta Cryst.* A**31**, 855–856.

CALVERT, L. D., KILLEAN, R. C. G. & MATHIESON, A. MCL. (1976). *Acta Cryst.* A**32**, 648–652.

CHIPMAN, D. R. (1955). *J. Appl. Phys.* **26**, 1387.

DEMARCO, J. J. & SUORTTI, P. (1971). *Phys. Rev.* B**4**, 1028–1033.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

LAWRENCE, J. L. (1977). *Acta Cryst.* A**33**, 343.

LAWRENCE, J. L. & MATHIESON, A. MCL. (1976). *Acta Cryst.* A**32**, 1002–1004.

MOORE, A. W. (1973). *Chemistry and Physics of Carbon.* Vol. XI, pp. 87, 106, 107. New York: Marcel Dekker.

NELSON, J. B. & RILEY, D. P. (1945). *Philos. Mag.* **36**, 711–714.

STIGLICH, J., WEISS, R. J. & HANSEN, A. M. (1974). *Report of the IUCr ad interim Commission on Charge Spin and Momentum Densities*, obtainable from Dr R. J. Weiss, Materials Science Division, Army Materials and Mechanics Research Center, Washington, Massachusetts 02172, USA.