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Line Shape Analysis of Cold-worked Magnesium

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A single-reflexion method due to Pines for line-shape analysis has been used to determine the particle size in cold-worked magnesium. A new expression has also been derived to determine strain from a single line. The results have been compared with those obtained by the Averbach–Warren method.

Introduction

X-ray line-broadening investigation of deformed metals has mostly been concentrated on the cubic class. Among metals having h.c.p. structure cobalt has received the greatest attention (*e.g.* Van Arkel, 1939; Edwards & Lipson, 1942; Wilson, 1942; Anantharaman & Christian, 1956; Houska, Averbach & Cohen, 1960; Mitra & Halder, 1964). Among others, zirconium (Mogard & Averbach, 1958) and magnesium (Lele & Anantharaman, 1964) have been studied to some extent. Lele & Anantharaman (1964) have studied the integral widths of intensity distributions around several reciprocal-lattice points for magnesium filings. They attributed the entire broadening successively to particle size and strain respectively and found that the mean deviation from the mean for all the reflexions for strain was much lower than the same value for particle size; hence they concluded that the line broadening was due to strain only, and that the strain was of the order of $1{\cdot}0\times10^{-3}$. This work is clearly not satisfactory since the individual contributions of particle size and strain to the line broadening, assuming it to be due to the composite effect of the two factors, have not been separated. Besides this work by Lele & Anantharaman (1964) no other study of cold-worked magnesium has been reported. The aim of this paper is to apply the technique of line-shape analysis in determining particle size and strain in cold-worked magnesium.

Experimental

Spectroscopically pure rods of magnesium supplied by Johnson Matthey and Co., London, were used for the study. The rods were rotated on a lathe head and lightly touched by a fresh alcohol-washed file and the filings were collected on a clean glass tray. Minute grains of iron from the file which might have become mixed with the magnesium filings were separated magnetically. The filings were passed through a sieve having 325 meshes per square inch. X-ray diffraction line profiles for $10\bar{1}0$, 0002, $11\bar{2}0$, $10\bar{1}3$ reflexions only could be obtained by using the photographic technique due to Mitra (1963). Line profiles for other reflexions were not satisfactory and were therefore not used for the present purpose. The background of each line profile was carefully determined by using a technique due to Mitra & Misra (1966). For obtaining the geometrical broadening for each reflexion similar studies were carried out on magnesium filings annealed in vacuum at 500 °C for three hours. Fourier coefficients for the pure diffraction line profile for each reflexion were determined from the two sets of line profiles by using the deconvolution method due to Stokes (1948). For calculating the Fourier coefficients 3° Beevers–Lipson strips and an electrical desk calculating machine were used.

Theoretical considerations

Since no pair other than $10\bar{1}0$, $11\bar{2}0$ has a simple relation, the usual Averbach–Warren (1949) technique is inapplicable. Hence the single-reflexion method due to Pines (1953) has been used for determining the particle size. According to this method,

$$\left(\frac{dA_L}{dL}\right)_{L=0}=\left(\frac{dA_L^P}{dL}\right)_{L=0}=-\frac{1}{p} \qquad (1)$$

where

A_L = nth order Fourier coefficient of the pure diffraction profile.
A_L^P = nth order Fourier coefficient of the particle size line profile.
L = nd, a real distance in the crystallite.

d is given by

$$\frac{2d(\sin\theta_1-\sin\theta_0)}{\lambda}=\frac{2d(\sin\theta_0-\sin\theta_2)}{\lambda}=\frac{1}{2} \qquad (2)$$

θ_0 = Bragg angle corresponding to peak position of intensity distribution.
θ_1, θ_2 = Bragg angles corresponding to positions in the intensity distribution where the tails merge into the background.
λ = wavelength used,
p = size of the particle in the direction considered.

Mitra (1965) has shown that the equation (1) is valid for Gaussian strain distribution only. Thus the particle size on the basis of the Gaussian strain-distribution hypothesis can be determined by drawing a tangent to the (A_L-L) curve at $L=0$.

It is well known that

$$A_L=A_L^P A_L^S \qquad (3)$$

where A_L^s is the Fourier coefficient of the line profile due to strain only corresponding to A_L and A_L^P and also that

$$A_L^s = \exp(-2n^2 l^2 z_L^2) \quad (4)$$

$$z_L = Ls/d \quad (5)$$

s=r. m. s. strain.
l=order of the reflexion considering the reflexion to be of type 00l.

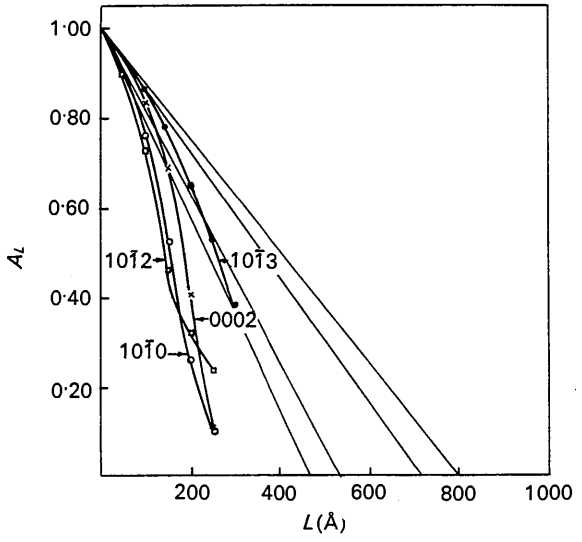

Fig. 1. Plot of Fourier coefficients A_L against distance L.

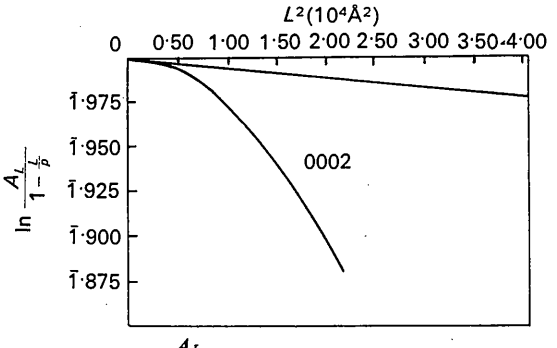

Fig. 2. Plot of $\ln \dfrac{A_L}{1-L/p}$ against L^2 for the $10\bar{1}0$ reflexion.

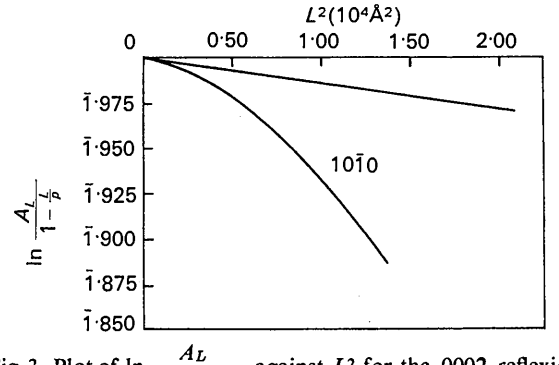

Fig. 3. Plot of $\ln \dfrac{A_L}{1-L/p}$ against L^2 for the 0002 reflexion.

Equation (4) is well known to correspond to the Gaussian strain-distribution hypothesis. Combining (3), (4) and (5) we have

$$\ln \frac{A_L}{A_L^P} = -2\pi^2 l^2 \frac{L^2 s^2}{d^2}. \quad (6)$$

If s is independent of L, then for a given value of L, the plot of $\ln (A_L/A_L^P)$ against L^2 will be a straight line passing through the origin, and the slope of this line will give an estimate of s. If on the other hand s is dependent on L, the plot will be a curve and s can be determined from the slope of the curve at $L=0$.

For reflexions of the type 00l, the crystallite can be treated as one-dimensional with a particle-size line profile of the type $\sin^2 N\varphi/\sin^2 \varphi$, which can be expanded into a cosine Fourier series having coefficients $(N-n)$. If we set A_0 to be 1, as is actually done, then

$$A_n = 1-n/N \quad \text{and} \quad A_L^P = 1-L/p\,, \quad (7)$$

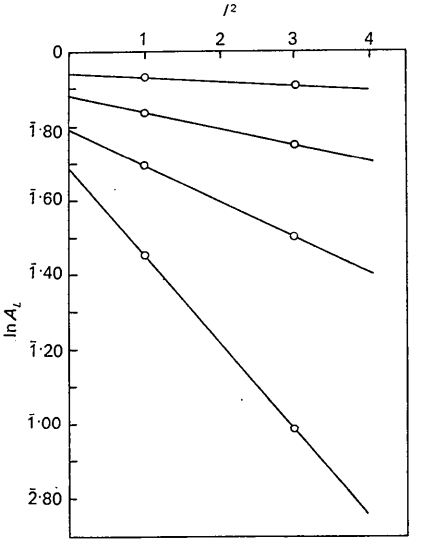

Fig. 4. Plot of $\ln A_L$ against l^2.

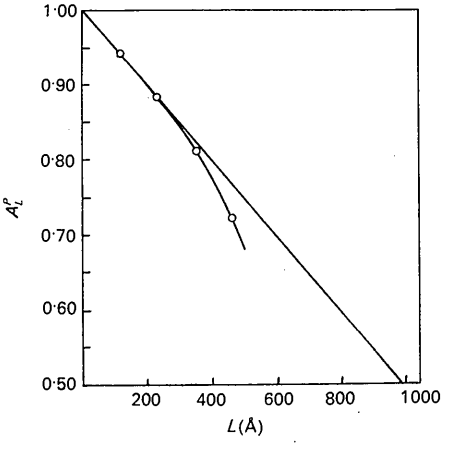

Fig. 5. Plot of particle size coefficient $A_L{}^P$ against distance L.

Table 1. *Particle size and lattice strain*

	Particle size (Å)			(r.m.s. strain) $\times 10^3$		
	Gaussian	Cauchy	Averbach–Warren Method	Gaussian	Cauchy	Averbach–Warren method
$10\bar{1}0$	535	579		9·23	10·11	
0002	720	759	990	7·58	8·30	18·32
$11\bar{2}0$	880	917		7·89	8·64	
$10\bar{1}3$	800	832		7·95	8·71	

assuming of course, that all the crystallites are of equal size. It is clearly seen that equation (7) is consistent with equation (1). Thus by determining p from equation (1), substituting in equation (7), and then using equation (6), s can be determined. Thus from a single reflexion only, particle size and strain values can be determined on the hypothesis that the strain distribution is Gaussian.

Corresponding quantities on the basis of the Cauchy strain distribution hypothesis can be determined from

$$s_c = \sqrt{6\sigma \frac{lL}{d}} \cdot s_g \qquad (8)$$

and

$$p_c = \frac{p_g}{1-(\pi^2 l s_c^2/\sigma d)\,.\,p_g} \quad \text{with } \sigma = 0{\cdot}2 \qquad (9)$$

where suffixes c and g denote the corresponding quantities for the Cauchy and Gauss strain distribution hypotheses. Equations (8) and (9) follow directly from equations (12) and (8) respectively of Mitra (1965).

Results and discussion

The method described above has been used in the present case. Figs. 1, 2 and 3 show the determination of particle size and strain for several reflexions on the basis of the Gaussian strain-distribution hypothesis. Table 1 shows the particle size and strain for different reflexions on the basis of the Gaussian as well as the Cauchy strain-distribution hypothesis. The strain is found to be of the same order of magnitude as observed by Lele & Anantharaman (1964), although several times as great. This is consistent with the observations made by Mitra (1964) for cold-worked aluminum, who found that while the measurement of integral breadth yielded a strain of magnitude $1{\cdot}02 \times 10^{-3}$, that by line shape analysis on a Gaussian strain-distribution hypothesis yield a value $6{\cdot}51 \times 16^{-3}$. While strain values show remarkable isotropy which is to be expected, since magnesium is elastically isotropic, the particle size is found to be different for different directions, the particle size in the $10\bar{1}0$ direction being almost half of that in other directions. The particle sizes found in the other three directions are of the same order of magnitude, although there is considerable variation between them. This perhaps shows that the particles are neither cubes nor spheres but are of irregular shapes. Similar results have also been obtained for uranium by Krishnan, Arunachalam & Asundi (1962). In the usual Averbach–Warren (1949) technique the particle size is considered to be identical in the various directions. To find out the effect of such a hypothesis, the Averbach–Warren (1949) analysis was applied to the $10\bar{1}0$, $11\bar{2}0$, pair of reflexions. Fig. 4 shows plots of $\ln A_L$ against l^2 for several values of L. The intercept of the linear plot so obtained gave the value of $\ln A_L^P$, the slopes the values of z_L^2. From z_L, s was determined. Fig. 5 shows the plot of A_L^P against L from the slope of which at $L=0$ the particle size was determined. Table 1 shows these values. It is observed that the strain value so obtained is somewhat unexpectedly high, being about twice the strain obtained from the $10\bar{1}0$ reflexion on the basis of the Cauchy strain-distribution hypothesis. The particle size thus obtained is of the same order as obtained for the $11\bar{2}0$ reflexion by the Pines (1953) method. The high value of strain shows that the hypothesis that the particle size in the $10\bar{1}0$ and $11\bar{2}0$ directions are the same is not perhaps correct. Thus, there appears to exist a real particle-size anisotropy.

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