Acta Cryst. (1964). **17**, 177

Texture of Crushed Coarse Crystalline Powder Specimens

BY S. B. BRODY AND G. G. WOOTAN*†
St. John's University, New York 32, N.Y., U.S.A.

(*Received 27 August 1962 and in revised form 26 March 1963*)

In pinhole powder diagrams of crushed coarse powder samples of fluorite and other insulators, studies of the number and appearance of the diffraction spots indicate that the particles are single grains.

Introduction

Freshly abraded single crystals and freshly crushed coarse crystalline powders show similar diffraction changes. (D'Eustachio & Brody, 1945). Very thin fabricated quartz wafers tend to become polycrystalline throughout when cold worked (D'Eustachio, 1946). These two phenomena suggested an investigation, first, to find out if coarse powders of size 30–40 μ, made by crushing up good single crystals, are single-crystal or polycrystalline; and second, to see if they develop a surface layer of damaged "mosaic" material of the order of a few microns thick, which is the depth of the surface layers observed on abraded slabs (Gay, Hirsch & Kellar, 1952).

The experiment to determine the average number of grains per powder particle consists essentially in counting the number of diffraction spots from a roughly uniform coarse powder sample and comparing with the predicted number. In the experiments to see if there is a damaged layer, the number and intensity of spots as a function of exposure time are studied.

Theory

Number of grains per particle

If a monochromatic beam of convergence $d\theta$ illuminates n grains of a powder or polycrystalline sample, the number, N, of diffraction spots from planes at Bragg angle θ is given by

$$N=\tfrac{1}{2}pn(d\theta+\Delta)\cos\theta \qquad (1)$$

where p is the multiplicity, and Δ is a lumped angular spread due to all causes other than the lack of parallelism in the beam. An experimental plot of N versus $d\theta$ should give a straight line from which n may be determined. Very thin powder samples were used, one layer of particles thick. The number of powder particles, n', was determined by counting, and n/n' is the number of grains per particle.

Equation (1) represents an adaptation of the method used by Hirsch & Kellar (1952) for measuring the grain size of polycrystalline aluminum. We could not use their reflection techniques because the particles we used were too large compared with the beam penetration.

Depth of damage

Suppose the powder sample consists of approximately cubical blocks of a highly absorbing material; say $\exp(-\mu b_1)\simeq 0{\cdot}01$, where μ = linear absorption coefficient and b_1 = length of edge of a block. Assume a polycrystalline damaged layer of depth b_2 cm. Then if t_1 is the exposure time to produce faint spots from large blocks of volume b_1^3, a small increase in exposure time will produce little or no increase in the number of spots. However, new faints spots from the surface layer will appear after prolonged exposure time t_2 such that

$$t_1/t_2=(b_2/b_1)^2[\exp(2\mu b_2)-1]\,. \qquad (2)$$

Results and conclusions

In the first experiment coarse fluorite powder of approximately homogeneous size was prepared from a good single-crystal specimen. Spotty transmission powder diagrams were obtained, at four different values of the beam convergence, with Ni-filtered Cu radiation. Values of N obtained by counting spots are given in Table 1. From the slopes of the graphs of N versus $d\theta$ in Fig. 1, n was determined (see equation 1). Then n' was found by direct count and finally n/n' was computed. The results are given in Table 2. All results indicate one grain per particle.

If an estimate of Δ can be made, equation (1) can be used to find n from a single value of $d\theta$. In Table 3

Table 1. *Number of spots, N, vs. convergence, dθ, for the three curves of Fig.* 1

	N		
$d\theta$	I	II	III
$3{\cdot}96\times10^{-3}$ radians	283	478	502
$5{\cdot}85\times10^{-3}$ radians	331	518	533
$8{\cdot}00\times10^{-3}$ radians	407	641	643
$9{\cdot}22\times10^{-3}$ radians	430	638	748

* Submitted in partial fulfillment of the requirements for the degree of Master of Science in Physics.

† Present address: Keuka College, Keuka Park, N.Y., U.S.A.

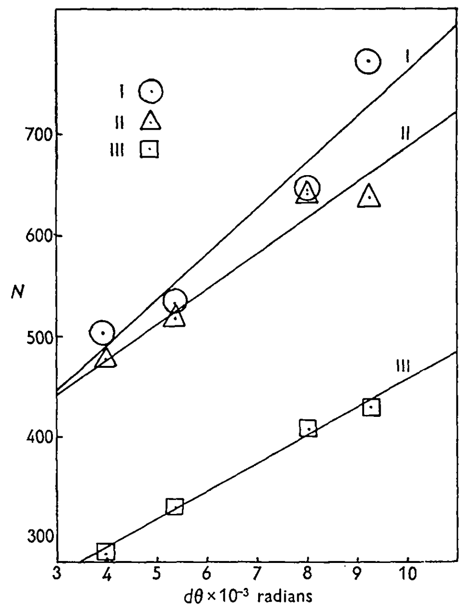

Fig. 1. Number of spots, N, plotted as a function of beam convergence, $d\theta$. Data from Table 1. Fluorite.

Table 2. *Determination of n/n' for fluorite by using the graphical method*

	Sieve size*	Indices of ring used	n	n'	n/n'
I	200–270	(220)	$5{\cdot}2\times10^3$	$5{\cdot}2\times10^3$	1·0
II	325–400	(220)	7×10^3	12×10^3	0·6
III	325–400	(111)	12×10^3	12×10^3	1·0

* The powder passed through the larger sieve but not the smaller.

Table 3. *Determination of n/n' for various materials using only one value of $d\theta$*

Material*	Fluorite	Quartz	Wulfenite
Indices of ring	(444)†	(101)	(116)
$d\theta$ (radians)	8.43×10^{-3}	$17{\cdot}3\times10^{-3}$	$11{\cdot}2\times10^{-3}$
Δ (radians)	4×10^{-3}	0‡	—
N	147	80	84
n	$1{\cdot}3\times10^4$	10^3	$2{\cdot}1\times10^3$
n'	$1{\cdot}2\times10^4$	$0{\cdot}7\times10^3$	$1{\cdot}1\times10^3$
n/n'	1·1	1·4	1·8

* All three materials are of sieve size 270–325.
† This is a back reflection ring.
‡ Small enough to be negligible.

are results for fluorite, wulfenite and quartz. The estimates for Δ were made from line broadening. In the calculation for wulfenite this information was not available and Δ was neglected. Thus, the number 1·8 is an upper limit on the value of n/n' for wulfenite. Again all results indicate one grain per particle.

Experiments using equation (2) were made with barite and wulfenite powders of nominal 325–400 mesh size. No increase in the number of spots was found with an exposure time $t_2=100t_1$. This established the non-existence of any "mosaic" layer thicker than about 2–3 microns. The value of b_1 used in equation (2) was 31 microns (microscopic measurement).

When exposure times t_2 were extended to 1000 t_1 in attempts to find a thinner layer, the background got too dense for useful counts. Diffuse rings were then observed. The area of a diffuse ring may be expressed as the product of the number of individual spots in the ring and the average area of each spot. An analysis showed that the amount of material in the beam was far too scanty for the diffuse rings observed to be produced by a polycrystalline surface layer. On moderately long transmission powder diagram, the 'powder effect' noted by Trigunayat & Verma (1962) was usually observed. These effects are rare on back reflection rings and common on low-index transmission rings; therefore they too come from the body rather than the surface of the particles. Also, measurements of line broadening indicate that there is more angular distortion relative to their size in the powder particles than in the large crystal the powder was obtained from.

Some further experiments, taking Laue diagrams of individual grains, are expected to yield more information about the kind and amount of the damage in the powder particles. All results so far are consistent with the model of a single grain per particle with no damaged surface layer of thickness 3 microns or greater.

This investigation was supported in part by a grant from the Research Corporation of America. The authors also wish to thank S. Gay, P. B. Hirsch, and I. Fankuchen for suggesting the method of attack on the problem.

References

D'Eustachio, D. (1946). *Phys. Rev.* **70**, 522.
D'Eustachio, D. & Brody, S. B. (1945). *J. Opt. Soc. Amer.* **35**, 544.
Gay, P., Hirsch, P. B. & Kellar, J. N. (1952). *Acta Cryst.* **5**, 7.
Hirsch, P. B. & Kellar, J. N. (1952). *Acta Cryst.* **5**, 162.
Trigunayat, G. C. & Verma, A. R. (1962). *Acta Cryst.* **15**, 499.