

Figure 4 Circuit for Hall current measurement,

conditions (especially applied field strength), may be significant factors determining the conduction mechanism.

Provided single crystals of suitable size can be prepared the Hall current technique provides a powerful method of investigating the electronic properties of high resistivity single crystals. It may also prove of value in checking the microwave Hall mobility measurements made on polycrystalline protein samples [8, 9].

Note on X-ray line Broadening and Electron Microscopy Study of the Effects of Milling and Subsequent Annealing of Alumina Powders

Various workers have observed the effects of milling and explosive shock in ceramic powders by X-ray line broadening [1-4], but no investigations have correlated the results with electron microscopy in deformed oxide ceramic powders, and the effect of annealing. The present work describes the observations made by X-ray line broadening and electron microscopy on the effects of impact milling and subsequent annealing on high purity submicron alumina powders.

B.D.H. Analar grade α alumina of average 1 μ m particle size and purity 99.988% was used for the present investigation.

5g charges of powder were impact milled for various times up to 48 h in an alumina cylinder with an alumina cretoid, using a Glen Creston vibratory mill.

X-ray powder profiles were obtained using a Philip's diffractometer with Ni filtered Cu K α radiation by step scanning at intervals of $1/8^{\circ} 2\theta$. 182

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References

- 1. V.N. DOBROVOLSKII and YU.I. GRITSENKO, Soviet Phys. Solid State 4 (1963) 2025.
- 2. R. PETHIG and K. MORGAN, Nature 214 (1967) 266.
- 3. K. MORGAN and R. PETHIG, ibid 223 (1969) 496.
- 4. A. SCHMILLEN and W. W. FALTER, Z. Physik 218 (1969) 401.
- 5. L. FRIEDMAN, Phys. Rev. 133 (1964) A1668.
- 6. M. SCHADT and D. F. WILLIAMS, *Phys. Stat. Sol.* **39** (1970) 223.
- 7. R. W. MUNN and W. SIEBRAND, Chem. Phys. Letters 3 (1969) 655.
- 8. E. M. TRUKHAN, Pribory tekhn. eksper 4 (1965) 198.
- 9. D. D. ELEY and R. PETHIG, *Bioenergetics* 1 (1970) 105.

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The profiles were analysed for crystallite size and strain by using the integral breadth method of Wagner and Aqua [5], the separation of α_2 component was obtained by Rachinger's method corrected for instrumental broadening by the parabolic relation.

The shape and size of the powder was examined in an EM6G electron microscope.

Milling produces broadening of the X-ray line profiles, which on analysis measured increase in strain and decrease in crystallite size of the



Figure 1 Variation of strain, crystalline size and particle size with milling. $\bigcirc =$ strain. $\bigcirc =$ crystallite size. $\bigcirc =$ particle size.

powdered samples. On separating the two components, the strain was found to increase with milling to a saturation value of 4×10^{-3} whilst the crystallite size decreased to a limiting value of 400 Å. Electron microscopy study showed that the particle size decreased to a limiting value with milling. The results are shown in fig. 1.

Observations on the shape of the unmilled particles were inconclusive; however, after about 4 h milling the majority of the particles appeared to be thin platelets. The few electron diffraction photographs that could be obtained from the unmilled particles showed typical single crystal spot patterns, whilst milled particles showed arced spots, the angular spread of the arcs increasing with milling time. On annealing the arcing gradually disappeared finally developing into single crystal spot patterns at temperatures above 1300° C. Typical results are shown in fig. 2. Dislocation densities were calculated from the arcing of the electron diffraction spots [6], assumed to be due to blocks of small crystals within the particle separated by dislocation arrays, depicted diagrammatically in fig. 3.

Thus
$$\rho = \frac{1}{r\mathbf{b}}$$
 [7] where $\theta = \underset{\text{the spots}}{\text{angular arcing of}}$
 $t = r\theta$ $\rho = \underset{\text{dislocation}}{\text{density}}$
 $\rho = \frac{\theta}{t\mathbf{b}}$ $r = \underset{\text{curvature}}{\text{radius of}}$
 $t = \underset{\text{crystallite size}}{\text{b} = \underset{\text{burgers vector}}{\text{surgers vector}}}$
 $= 1.586 \text{ Å for unit}$
 $\dim(11\overline{2}0)$ direction
[8].

Dislocation densities were also calculated from



Figure 2 Electron transmission micrographs and selected area electron diffraction of milled and annealed alumina particles (a and b milled for 48 h, c and d milled and annealed for 1 h at 1300° C).

the crystallite size and strain values obtained from X-ray line broadening by the method described by Williamson and Smallman [9]. The results are tabulated in table I.

The measure of increasing dislocation densities and the reduction in crystallite size with increasing milling time, implies that the crystallites are created by dislocation arrays separating the



Figure 3 Schematic diagram of the arcing of electron diffraction spots by crystallites with dislocation arrays.



Figure 4 Effect of 1 h anneal at temperature on strain and crystallite size of milled powders. $\bigcirc =$ strain. $\bigcirc =$ crystallite size.

TABLE I	Calculated	dislocation	densities	(cm ²)
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individual crystallites (fig. 3). The dislocation densities calculated are comparable to a deformed metal. This suggests that the strain measured is the measure of non-uniform deformation, i.e. plastic bending of the crystals.

X-ray line broadening measurements of the annealing effects of the heavily strained powder for 1 h in air (fig. 4) showed that the strain removal began at temperatures around 700° C and was virtually complete by 1300° C, whilst the crystallite size remained unchanged. Electron microscopy examination of the annealed powders at various temperatures showed disappearance of the arcing with increasing annealing temperatures and the appearance of multicrystalline spot patterns. Particles examined above 1300° C showed single crystal patterns suggesting some growth of the crystallites.

This gradual disappearance of strain and arcing of the crystals with increasing temperatures suggests an occurrence of a polygonisation process at lower temperatures and a subgrain growth type of recrystallisation at higher temperatures analogous to the annealing processes observed in metals.

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References

- 1. D. LEWIS and M. W. LINDLEY, J. Amer. Ceram. Soc. **49** (1966) 49.
- 2. E. J. WHEELER and D. LEWIS, J. Mater. Sci. 4 (1969) 681.
- 3. O. R. BERGMANN and J. BARRINGTON, J. Amer. Ceram. Soc. 49 (1966) 502.

Milling time h	Crystal size Å	Average strain	θ degrees	Dislocation densities calculated from		
				Crystallite size	Strain	Spread of arcs
0	7000	3 × 10 ⁻⁴	0.7	$1.02 imes10^9$	2.65×10^{8}	3.5 × 10 ⁹
1	5000	$4.8 imes10^{-4}$	1	$1.2~ imes 10^9$	$4.28 imes10^{8}$	$7.4 imes10^{9}$
4	1000	$2 imes 10^{-3}$	2	3×10^{10}	$3.05 imes10^{10}$	7.3 \times 10 ¹⁰
8	700	$3.7 imes10^{-3}$	4	6.3 × 10 ¹⁰	5.7 × 1010	2.11×10^{11}
48	400	4.0 × 10 ⁻³	5	1.18×10^{11}	6.5 × 10 ¹⁰	$4.72 imes10^{11}$

- 4. I. CUTTER and R. MCPHERSON, *Phil. Mag.* 9 (1969) 489.
- 5. C. N. WAGNER and E. N. AQUA, Adv. X-ray Anal. 7 (1963) 46.
- 6. D. LEWIS, private communication.
- 7. A. H. COTTRELL, "Dislocations and Plastic Flow in Crystals" (Oxford and the Clarendon Press, 1953) p. 29.
- 8. M. L. KRONBERG, Acta Metallurgica 9 (1957) 507.
- 9. G. K. WILLIAMSON and R. E. SMALLMAN, *Phil. Mag.* 1 (1956) 34.

Short Notices

Modern Physical Metallurgy

R. E. Smallman 544 p. 3rd edition (Butterworths) £3.00.

This book represents an expanded and considerably updated version of the previously wellreceived edition. Much has been accomplished in the science of metals in the past decade and this is reflected in the extended treatment of the microstructural aspects of metals and the influence they have upon the mechanical properties. The book unashamedly deals only with metals, but the author's style and his overall plan of relating the fundamental principles to the properties, use and problems encountered with metals, together with its price and breadth of contents will appeal to students who are taking degrees and advanced courses in metallurgy.

The initial chapters are devoted to atomic and crystal structures, metallurgical tools, thermodynamics, and the structure of metals and alloys. Mechanical properties are fully covered in terms of the dislocation theory with great emphasis being placed on the application of this theory. The final chapters of the book cover precipitation and eutectoid transformations, the failure of metals by fracture, fatigue and creep and a new section devoted to corrosion and oxidation reactions. The omission of examples from this edition is a pity as their inclusion would have added to the value of the book for teaching purposes.

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Elements of Advanced Quantum Theory

J. M. Ziman

(Cambridge University Press, 1969). 55s

Professor Ziman's concern in writing this book has been to help research workers in physics to comprehend more adequately the research activities and techniques centred upon quantum mechanics used by the more mathematical of their colleagues. The starting point assumed is that of a good physics graduate of a British university in his first or second year of research, having some acquaintance with quantum mechanics in the Dirac von Neumann formalism – matrix representations, orthogonal functions, operators, eigenvalues, and the like.

From this basis, the book attempts to explain the essentials of a variety of advanced quantum mechanical concepts, ranging over field operators graphs, propagators, Green's functions, Smatrices, irreducible representations and so on. The path is not an easy one and calls for quite a high degree of application from the graduate student. However, given a field of physics in which the theoreticians express their results and analyses in such advanced terms, the student needs such a text as this to follow exactly what methods have been used and what assumptions made if he is not to be reduced to the level of merely lifting the apparently relevant equation from the end of the theoretician's paper. Even one hard read through the book gives an experimentalist confidence that on the next time around he will understand more of the arguments. In the context of other available texts on advanced quantum theory, this is indeed praise for the work that Professor Ziman has done here.

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