$$L_a = K\lambda/\beta\cos\theta \tag{3}$$

$$K = 0.9 - (1.84 - 0.9) p [3]$$
 (4)

$$1 - p^2 = (3.440 - c_0/2)/0.086 [4]$$
 (5)

where K is the Scherrer constant, λ is the wavelength of the X-radiation, θ is the Bragg angle, p is Franklin's parameter [4], and $c_0/2$ is the inter-layer spacing. The values of $c_0/2$ for various samples were measured from the (00.4) reflection.

The values of L_a of various samples employed in the present experiment are shown in fig. 4 and listed in table II. Fig. 4 also includes values



Figure 4 Crystallite size L_a of various samples.

TABLE II Crystallite size L_a of various graphites

Sample	$L_a(\text{\AA})$	
	190	
HT	> 1000	
G	750	
NG	> 1000	

obtained by means of a Fourier analysis by Guentert [1], and from the measurement of diamagnetic susceptibility by Fischbach [2]. The difference between the results of the present experiment and the earlier values may be due to variation in structural features which depend on the conditions of preparation.

Acknowledgement

The author would like to thank Professor Seishi Yajima for his guidance and encouragement, and the Nippon Carbon Co for supplying the pyrolytic graphite samples.

References

- 1. O. J. GUENTERT, J. Chem. Phys. 37 (1962) 884.
- 2. D. B. FISHBACH, NASA-TR-32-532 (1966).
- 3. H. AKAMATSU, H. INOKUCHI, H. TAKAHASHI, and Y. MATSUNAGA, Bull. Chem. Soc. Japan, 29 (1956) 574.
- 4. R. E. FRANKLIN, Acta Cryst. 4 (1951) 253.

8 November 1968 T. HIRAI Research Institute for Iron, Steel and other Metals Tohoku University Sendai, Japan

Twinning in Lead

Deformation-twinning does not ordinarily occur in fcc metals. In the case of pure fcc metals tested at normal rates of strain, deformationtwinning has been observed only at very low temperatures. However, very high strain rates, such as those associated with explosive loading or high velocity impact, may lead to the production of twins at room temperature [1]. Early attempts [2-5] to detect twinning in aluminium 646 and lead were unsuccessful, even at temperatures as low as 4.2° K. Bolling *et al* [6] report the production of mechanical twins by the high compression of zone-refined lead single crystals at 77° K.

We were able to produce large numbers of twins in high purity lead single crystals by spark-machining and annealing. Lead of 99.999% purity, obtained from ASARCO, was melted and cast into square bars which were about 15 cm long and 1.3 cm on a side. Each bar was pointed

at one end so that a single crystal could be grown by a modified Bridgman technique. The crystal was grown in a split graphite mould at the rate of about 1.8 cm/h, and after growth the long crystal was spark-planed on two opposite faces to get rid of the fins at the mould joint. The spark-planing was done with a Servomet type SMC spark-machine set at a low speed. Kerosine (paraffin) oil at room temperature was used for the dielectric. The spark-planed crystal was cut at intervals of 1.3 cm to produce 1.3 cm cube single crystals of lead.

The cut crystals were cleaned and annealed at $300 \pm 2^{\circ}$ C for 24 h in a stream of purified argon gas, then furnace-cooled. X-ray examination showed that the growth axis of the crystal was 9° off the <100> axis and that the sides of the crystal were 9° off the $\{110\}$ planes. So the spark-planing was done on planes which were 9° off the $\{110\}$ planes. The annealed crystals were cleaned with 25% HCl and chemically polished with a solution containing 5 ml of 30% H₂O₂ in 45 ml of glacial acetic acid. The twins observed on the adjacent $\{110\}$ planes are shown in figs. 1 to 3. The position of the twins of fig. 1 is shown in fig. 4.

Fig. 3 shows the different directions of slip bands on the two sides of the longer twin trace, especially near the corner, where it meets the edge of the specimen. X-ray Laue patterns of the twin and the matrix were taken separately as well as on the same film by shifting the specimen during exposure. Stereographic analysis showed the twin plane to be a $\{111\}$ plane. The angles shown on fig. 4 were measured with an optical microscope and a rotating stage. A two-surface analysis of the twin traces in the sample of fig. 1 showed the twin plane to be the $(11\overline{1})$ plane. When the Laue pattern was rotated to the {111} standard projection a six-fold symmetry was observed, rather than a three-fold symmetry about the $\langle 111 \rangle$ axis. This higher symmetry results from the twin relation adding a 180° symmetry around the <111> axis.

The favourable twinning systems in fcc metals in compression along the [011] axis [7] are: (111), $[11\overline{2}]$ and $(\overline{1}11)$, $(\overline{1}1\overline{2}]$. We presume that the twins observed in our samples were nucleated by compressive shock waves generated by sparkplaning and propagated along the $[0\overline{1}1]$ axis. The annealing treatment that followed the sparkplaning caused these nuclei to grow. These twins were observed only in the crystals subjected to spark-planing. Although many further crystals



Figure 1 Twins observed on sample Pb A-4 on (011).



Figure 2 Twin observed on sample Pb A-5 on (0 11).



Figure 3 Twin observed on sample Pb A-5 on $(0\overline{1}1)$.



Figure 4 Position of the twin of fig. 1.

were grown, cut by a wire saw in the sparkmachine and then annealed, no such large twins were observed. But spark-cutting was found to generate small twinned areas very near the cut edges.

Acknowledgement

This research was supported by the Advanced

Dendritic Surface Patterns on Alumina Spheroids formed in a High Temperature Plasma

Dendritic patterns have been observed on spheroidal particles of alumina made in a high temperature induction plasma [1] and applications for the particles based on their enhanced surface area and roughness are being investigated. Examples of the dendritic patterns on spheroidal particles of about 150 μ m diameter can be seen on the scanning electron micrographs in figs. 1 and 2. These were obtained using a Stereoscan Mark II scanning electron microscope.

Particles with enhanced surface areas are of interest as catalyst supports in the chemical industry. For example, particles of materials such as activated alumina with very high specific surface areas and pore sizes down to the order of 10 Å diameter are used extensively as catalyst supports, particularly for vapour-phase processes However, particles with such fine pores are not so suitable for some liquid-phase processes because of the difficulty of diffusion of reactive species into the interior of the particles. There is then a

Research Projects Agency of the Department of Defense through the Northwestern University Materials Research Center.

References

- 1. C. S. SMITH, Trans. AIME 214 (1958) 574.
- 2. P. R. THORNTON and T. E. MITCHELL, Phil. Mag. 7 (1962) 361
- 3. T.H. BLEWITT, R.R. COLTMAN, and J.K. REDMAN, J. Appl. Phys. 28 (1957) 651.
- 4. P. HAASEN, Phil. Mag. 3 (1958) 384.
- 5. Z. S. BASINSKI, Proc. Roy. Soc. A240 (1957) 229.
- 6. G. F. BOLLING, K. W. CASEY, and R. H. RICHMAN, Phil. Mag. (1965) 1079.
- 7. R. J. DE ANGELIS and J. B. COHEN, "Deformation Twinning", edited by R. E. Reed Hill (Gordon and Breach Science Publishers, New York, 1964).

16 December 1968 V. R. PARAMESWARAN J. WEERTMAN Department of Materials Science Northwestern University Evanston, Illinois, USA



Figure 1 Scanning electron micrograph of alumina spheroid.

need for particles with enhanced areas but larger pore sizes, sometimes of the order of 1 μ m and it can be seen from fig. 2 that the present particles have pores of this order of size. A somewhat