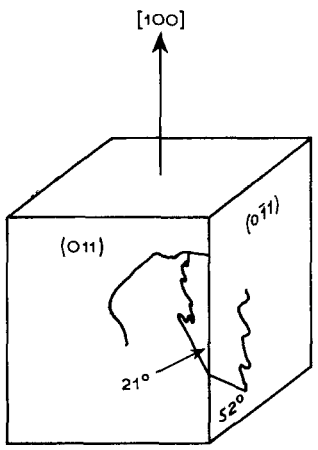

Figure 4 Position of the twin of fig. 1.

were grown, cut by a wire saw in the spark-machine 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

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

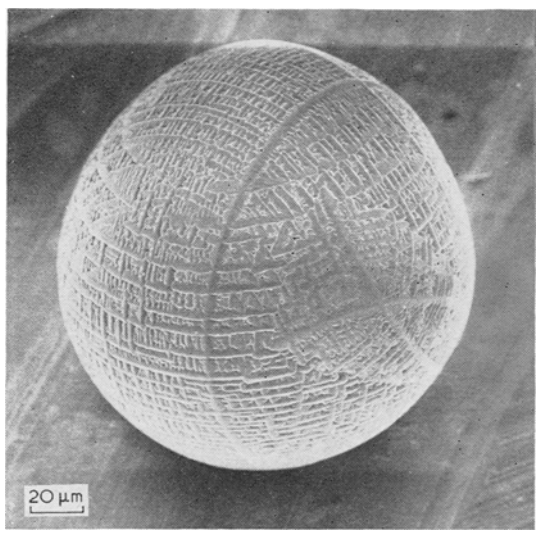

Figure 1 Scanning electron micrograph of alumina spheroid.

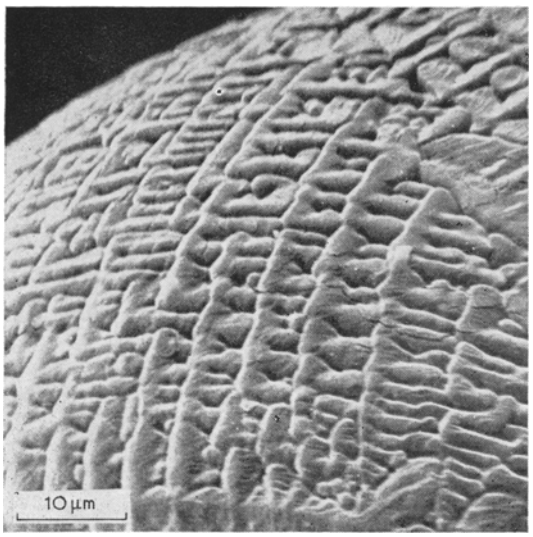

Figure 2 Scanning electron micrograph of part of alumina spheroid.

similar potential application for the particles is in the recently-developed fluidised bed electrode [2-4] used in liquid-phase electrochemistry. Here increased electrode area is obtained by using a fluidised bed of metal-coated particles as an electrode. Further increases in area should be possible by using coated particles of the present type in place of smooth-coated particles. This application would probably be restricted to synthesis cells, as the outer parts of the particles used in batteries [4] already tend to become porous.

The present particles are made by melting alumina particles in an argon plasma and allowing them to solidify during their fall from the plasma into a receiver. The plasma was of the radio frequency induction type [1], formed in a vertical quartz tube of 25 mm diameter. The particles and argon passed through continuously, entering near the top of the tube. A tangential argon inlet was used to help stabilise the plasma. Plasma temperatures in such an arrangement [1] can be as high as 10 000 to 15 000° K, well above the melting point of alumina. Electrical power at 34 to 38 MHz frequency was supplied by a generator consuming 2 to 2.4 kW, via a copper coil surrounding the quartz tube. The feed particles contained nominally 98.5 to 99.5 wt % alumina (Thermal Syndicate grade REHT). They were irregular in shape and in the sieve range -85 to $+120$. (Mesh openings 180 to 125 μm.)

About 80% by number of the particles were converted into spheroids. When viewed by reflected light in an optical microscope, roughly 60% of the spheroids appeared opaque and white, the remainder having various degrees of transparency. In this respect the particles appear to be similar to the alumina spheroids produced by Bildstein [5] with a plasma jet. For examination in the scanning electron microscope, the particles were mounted on aluminium sample stubs with collodion and then metallised with a gold/palladium mixture according to the method described by Boult and Brabazon [6]. The surface patterns shown on the resulting electron micrographs in figs. 1 and 2 are thought to be due to dendritic growth during crystallisation from the molten alumina. The prominent band on fig. 1 appears to be a main stem and primaries can be seen branching from it, often at right angles. Secondary branches at right angles to the primary branches can also be seen. Photomicrographs of alumina spheroids made in plasma jets have been presented by Meyer [7]. Surface patterns were evident but no explanation for them was given. The patterns appeared to be much simpler than those on fig. 1 and did not have such markedly dendritic characteristics. Their apparent simplicity could to some extent be due to the lower magnification used and to the shorter depth of field of the optical microscope. One factor which may have affected the surface structure of the spheroids made by Meyer was the evolution of nitrogen which made the particles porous [7].

Not all of the alumina spheroids made in the present experiments had dendritic patterns, possibly because of differences in the temperature histories of the particles. That the temperature histories of particles can differ is shown by the presence of some unmelted particles in the receiver. Differences in temperature history could arise owing to the steep radial temperature gradients which exist [1] in induction plasmas due to non-uniform gas velocities, and to differences in the sizes and shapes of particles. Work is in progress to assess the temperature histories of particles and to establish the conditions most favourable for the formation of dendrites and pores.

A fuller account of the preparative technique and the properties of the particles will be presented at a later date.

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On the Reversion of GP Zones in Al-10% Zn and Al-10% Zn-0.1% Mg Alloys

Most of the previous studies on spherical GP zones, which characterise the pre-precipitation process in the Al–Zn alloys, were related to their formation and growth [1-5]. However, very few of them have been concerned with reversion, i.e. dissolution of the GP zones that results when the solid solution containing them is heated above a given temperature. This temperature is determined, for a given composition of the alloy considered, by the metastable miscibility gap which seems to control the formation of such zones [3, 5].

The aim of this note is to present some results of small angle X-ray scattering during reversion of GP zones in Al–10% Zn and Al–10% Zn–0.1% Mg (wt %) alloys. The latter system has become interesting through the study of the atomic diffusion mechanisms by which the zones are formed [6-8]. Structural analogies and differences between those two alloys were recently pointed out; particularly it has been shown that segregation in the ternary alloy may be assumed to be also controlled by a metastable miscibility gap [9]. No data are available, however, on the reversion of this alloy.

The measurements were carried out with a device already described elsewhere [9-11] under such conditions that the "infinite beam" approximation was justified. The scattered intensity is expressed on an absolute scale in terms of the function $j_n(s)$ [5], where $s = 2\sin\theta/\lambda$, 2θ being the scattering angle and $\lambda = 1.54$ Å the X-ray wavelength (Cu $K\alpha_1$). The exact compositions and characteristics of the alloys were given in a previous work [9]. After a solution-treatment at 300° C the specimens were quenched in water at 0° C and aged for several days at room temperature. The scattering curves were then measured for both alloys (time $t = 0$ of the reversion treatment). Subsequently, the samples were given successive reversion treatments in a glycerine bath at 110 ± 2° C. After each treatment the scattering curve was measured at room temperature. Although there is no appreciable change in the alloys during holding at room temperature, nevertheless the specimens were stored in liquid nitrogen between the measurements. Ten scattering curves were obtained for each alloy. Some of the curves are shown in fig. 1a and b. Other curves have been

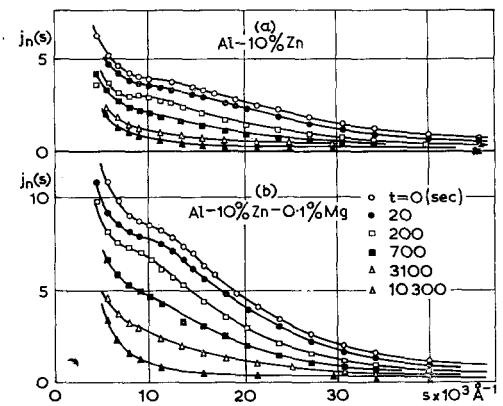

Figure 1 Scattering curves observed during reversion at 110° C. In order to avoid confusion, not all the measured curves are shown. For the same reason the figure is limited to scattering angles equivalent to $s = 45 \times 10^{-3}$ Å^{-1}, though measurements were made up to $s = 60 \times 10^{-3}$ Å^{-1} ($s = 2\sin\theta/\lambda$).