

Fig. 7 shows a group of liquid Pb inclusions of different sizes. The smaller particles are nearly spherical while the large particle displays distinct facets parallel to the  $\{111\}$  planes of the Al matrix. It was found that large particles remained faceted up to about 500°C, when they were observed to round off quickly. During subsequent cooling, the rounded shape remained stable until the particle solidified at about 300°C. This shape hysteresis was found to be due to a kinetic limitation that prevented the faceted particles from approaching their equilibrium shape (Gabrisch et al. 2001). For a particle to change to its equilibrium shape at constant volume, it is necessary to nucleate ledges on the  $\{111\}$  facets. Since the driving force for ledge nucleation is inversely proportional to size, smaller particles can equilibrate at a lower temperature than larger particles.

From a careful measurement of the local equilibrium shape, it was possible to derive the anisotropy of the interfacial energy between liquid Pb and solid Al. Contrary to earlier reports, this anisotropy was found to be only about 2%. From these observations it was also possible to measure a step energy in the  $\{111\}$  facets of  $1.9 \times 10^{-11}$  J/m. The disappearance of this step energy at about 550°C indicates a roughening transition at about 100K below the monotectic temperature. In-situ observations also indicate that small liquid Pb inclusions undergo Brownian motion, which again depends strongly on particle size. The same kinetic limitation that prevents large particles from reaching their equilibrium shape also inhibits their migration, whereas very small inclusions can reach shape equilibrium and thus overcome the kinetic barrier posed by extended  $\{111\}$  facets. A detailed investigation of the Brownian motion of these particles is currently underway.

### 3.2 Inclusions at Grain Boundaries

When Pb nucleates at grain boundaries, it forms compound shapes that are more complex than those in the bulk. Fig. 8 shows typical inclusions at a 90°  $\langle 110 \rangle$  tilt grain boundary in a "mazed" bicrystal grown by vapour deposition on a single crystal substrate. Although it appears that the inclusions are made of two distinct parts joined along the interface, each of these particles is actually a single crystal. The different moiré patterns in the two grains result from interference between a single crystal of Pb with a bicrystal of Al. Thus, in the faceted part of the inclusions the Pb lattice is in parallel orientation with the Al lattice, while in the rounded part the Al lattice is rotated by 90° relative to the same Pb lattice. As a result, the interface structures in the two grains are very different, leading to correspondingly different interfacial energies and anisotropies.

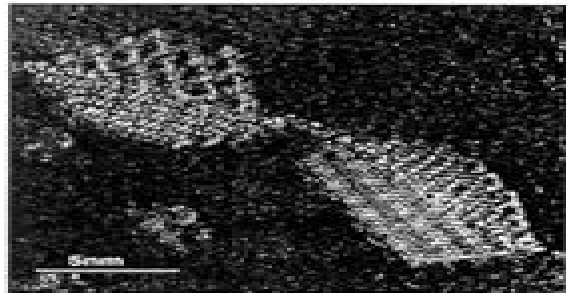


Figure 8 High resolution micrograph of typical Pb grain boundary inclusions in Al showing the effect of boundary inclination on the inclusion shape. The boundary shown is a 90°  $\langle 110 \rangle$  tilt boundary in a "mazed" bicrystal film. The images were recorded under dark field imaging conditions to emphasise the inclusion lattice. Moiré fringes are due to overlapping lattices of Pb and Al. Note faceting toward opposite grains.

Inclusion shapes at grain boundaries were found to depend strongly and reproducibly on the boundary orientation (Johnson et al 1996). The equilibrium shape of such an inclusion can be determined using a variation on the Wulff construction, first proposed by Winterbottom (1967) for crystals on surfaces, and subsequently modified by Lee and Aaronson (1975) for precipitates at grain boundaries. A more general treatment of the equilibrium shape of such inclusions is given by the  $\chi$ -vector construction of Cahn and Hoffman (1974). By comparison with inclusions in the bulk, those at grain boundaries are of a more complex shape, being composed of two segments joined along the interface. As these particles are single crystals with two different interfaces, they can be used as test objects to investigate the effect of inter-

# Electron Microscopy Analysis 2001

**Terry C. Jones**



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