Figure 2 Splat-cooling apparatus associated with a solar furnace.

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Growth of alumina by oxidation of amalgamated aluminium sheets

The work reported in this note developed from studies on aluminium amalgam to develop a technique for making ohmic contacts on ceramics and semiconductors. Various workers have reported the growth of alumina powder by exposing aluminium sheet wetted by mercury to moist air or to water. Usually mercury does not wet aluminium sheet. Watson *et al.* [1] have used 1% aqueous mercuric chloride solution for wetting aluminium sheet with mercury, while Bennett *et al.* [2] wet the aluminium sheet first with dilute HCl and then treated the HCl-wetted portion with a drop of mercury. Dilute hydrofluoric acid [3] has also been used to wet the aluminium sheet with mercury. Peri and co-workers [4-6] used a solution of glacial acetic acid and red mercury oxide in water to grow alumina powder from aluminium sheet. Bruce *et al.* [7] reported the instantaneous formation of alumina by wetting aluminium sheet with gallium and then reacting this gallium-wetted sheet with water. Numata [8] reported the growth of alumina on AlSb crystals in moist air or water. In the case of magnesium, Bennet *et al.* [2] observed an instantaneous formation of magnesium oxide powder by wetting magnesium with mercury and by exposing the mercury wetted magnesium sheet to moist air.

In the work reported here, we used both dilute HF and dilute HCl to wet the surface of aluminium with mercury and then left the amalgamated aluminium in moist air. Voluminous growth of fibrillar alumina was then observed. It was found that the completion of growth of alumina in moist air from the aluminium sheet wetted with dilute HF was faster than that from the aluminium sheet wetted with mercury with dilute HCl. The rate of growth at a particular humidity and temperature is shown in Fig. 1.

The growth of alumina powder for both the wetting agents HF and HCl, stopped after some time. Bennett *et al.* [2] attributed this to the formation of an intermediate phase at the
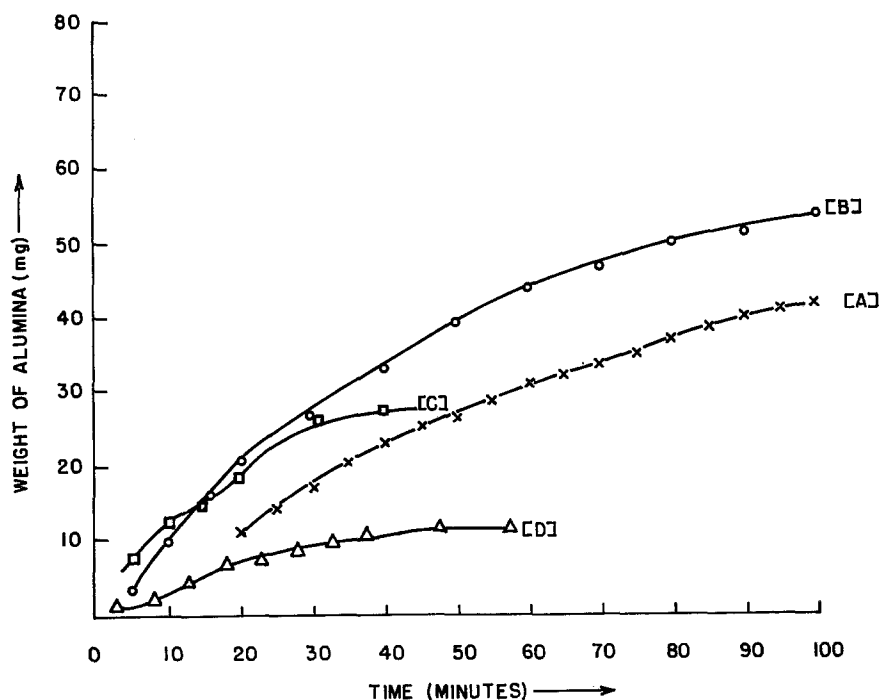
Figure 1 Rate of growth of alumina powder from a mercury-wetted aluminium sheet when exposed to moist air. Curves A and B are for two different aluminium sheets which have been wetted with mercury with the help of dilute HCl. Curves C and D are for another two aluminium sheets wetted with mercury with the help of dilute HF.
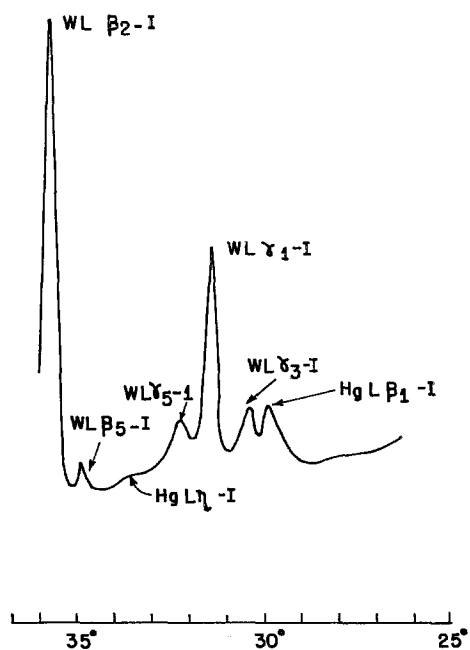

Figure 2 X-ray fluorescence spectra of alumina powder showing the presence of mercury.

mercury–aluminium interface. Watson *et al.* [1] reported continued growth of alumina from aluminium sheet which had been exposed to air only, with growth of alumina stopped, by immersing the specimen in water.

It was found by gravimetric analysis that the amount of mercury needed for the reaction to take place in moist air (using dilute HCl or HF) was 7 to 12% by weight of the quantity of alumina produced. An X-ray powder pattern gave broad and diffuse bands, indicating a very small crystallite size for the alumina powder. X-ray fluorescence spectra showed that mercury was present in traces of a few parts per million in the alumina powder grown from the amalgamated aluminium sheet, as shown in Fig. 2. This was confirmed by a spectrophotometric method with Dithizone, which gave a mercury content of $\sim$ 0.0005% by weight. Electron microscope studies of the powder revealed that the diameter of the fibrils was $\sim$ 50 Å.

Further experiments on the dielectric constant of the alumina produced by this method are in progress.

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Crystal imperfections in silicon epitaxial layers grown on ion-implanted substrates

Ion implantation is now being introduced into many semi-conductor manufacturing processes. Generally, it is extremely successful, but recently Moline et al. have observed that crystal defects can be generated in an epitaxial layer grown on an ion-implanted substrate [1]. They grew epitaxial layers by silane pyrolysis on substrates implanted with arsenic ions at 150 kV. The general perfection of growth was monitored by Berg-Barret X-ray topography but the individual defects were not identified. We have investigated a similar experimental system and have found the same imperfect growth. Further, we have used transmission electron microscopy (TEM) to identify the defects present as edge dislocations and epitaxial stacking faults.

2 in. diameter 5 Ω-cm p-type Si slices were implanted with arsenic ions at 100 kV and 1 mA through a thermal oxide mask. This mask consisted of fingers of oxide 650 μm wide with 650 μm spacing which was aligned parallel with the long axis of the ribbon beam of ions. The beam was held stationary while the slice was passed under the beam, moving perpendicular to the fingers of the mask. After each pass the slice was moved ~ 1 mm parallel with the fingers of the mask and then passed under the beam in the opposite direction. The total dose received by the slice was ~ 10^{16} ions cm^{-2}. After implantation the slices were oxidized at 1200°C for 2 h. We examined the slices before and after epitaxial growth using Lang X-ray topography (XRT), platinum/carbon replicas and TEM.

The conditions of epitaxial growth and the observations made by the various techniques are shown in Table I.

Fig. 1 is a transmission X-ray topograph of part of a slice with 0.5 μm epitaxial layer grown from silane at 1050°C. The ion implantation gives rise to bands of contrast running approximately parallel to the direction of movement of the slice as it passed under the ion beam. These bands are cut by the fingers of the oxide mask

TABLE I

Material	Visual inspection	XRT	Pt/C replica	TEM
Substrate after implantation and oxidation	Smooth polished surface	Bands of contrast, slip, "swirls"	Very smooth	Edge dislocation loops
Epitaxial layer grown from silane at 1050°C	Bands showing diffuse reflection	Bands of contrast, slip	Shallow circular depressions with pits at bottom	Epitaxial stacking faults ~ 10^4 cm^{-2}; edge dislocation segments ~ 10^7 cm^{-2}
Epitaxial layer grown from SiCl₄ at 1140°C	Smooth polished surface	Bands with slight contrast	Very smooth	Epitaxial stacking faults ~ 10^4 cm^{-2}