

FIG. 2. Gray tin crystals typical of the largest crystals that have been grown.

the transformation to the gray phase, an amalgam containing a few percent mercury was maintained at reduced temperature the mercury was expelled forming droplets on the surface. This indicated a low solubility of mercury in gray tin and therefore that crystals, if they could be grown, would be quite free of mercury.

CRYSTAL GROWTH

The crystal growing apparatus was designed to permit continuous crystallization from a mercury solution locally supersaturated by cooling to -30°C , while the tin concentration was maintained by continuous dissolution of the metal in a warmer portion of the mercury. The original design has been modified repeatedly in an effort to concentrate the growth on a small number of crystals beneath the mercury surface. The apparatus being used at present is shown in Fig. 1. This apparatus

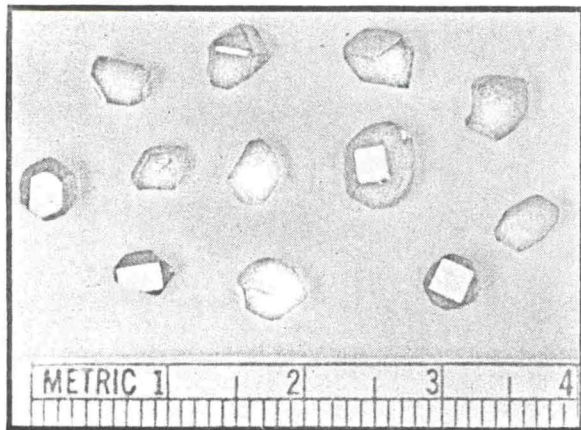


FIG. 3. Gray tin crystals selected and oriented to show external perfection.

is placed in a refrigerator at -55°C . The charge of tin and surrounding mercury in the lower constricted part of the beaker are maintained at -20°C by means of the heating coil. As tin dissolves a density gradient is established which causes convection and the liquid amalgam rises at nearly constant temperature in the Dewar-type tube. As the solution flows radially outward at the top and downward along the walls it is cooled to a minimum temperature of about -30°C . In this particular apparatus crystal growth occurs only at the vertical walls beneath the mercury surface. Tin is supplied to the apparatus in the form of rods 4 mm in diameter and 2.5-cm long at 24 hour intervals.

Intentional seeding with gray tin powder is unnecessary. The initial crystallization product consists of hexagonal plates, presumably the compound HgSn_{12} .⁸ If, after a week's growth these crystals are removed, further crystallization invariably produces a great number of gray tin crystallites. By removing these repeatedly at one week intervals the number of nuclei is sufficiently reduced to permit reasonably rapid growth

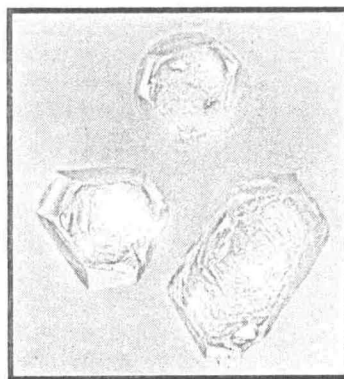


FIG. 4. Crystals oriented to show ring structure of imperfect faces.

of the individual crystals. This growth rate is such that crystals of the size shown in Fig. 2 are grown in a period of a month.

CRYSTAL PROPERTIES

The crystals shown in Fig. 2 are the largest that have been grown thus far. The present limitation on the size is that imposed by the dimensions of the apparatus. Though there is no inherent limitation on size, the larger crystals tend to be less perfect as may be seen by comparison with Fig. 3 which shows somewhat smaller crystals selected and oriented to display the external symmetry. Most crystals, regardless of size, have one imperfect face characterized by the ring structure shown in Fig. 4.

Gray tin is brittle and the crystals are readily fractured to expose surfaces of high luster and having the general appearance of broken germanium. This is in

⁸ See, for example, M. Hansen, *Aufbau der Zweistofflegierungen* (Julius Springer, Berlin, 1936), p. 809. These hexagonal crystals will always be produced if the minimum mercury temperature is raised to -10°C .

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