A Cold Crucible for High-Temperature Melting Processes

EXPLORATORY research in the solid-state field has resulted in the development of a new melting technique for the processing of silicon. Although this method has been associated primarily with the purification of silicon and its growth as single crystals. it has important applications in the more general field of metallurgy.

In our earlier work[1], a container was made from a silver tube by pressing a longitudinal dent into its surface for most of the length. This silver 'boat' was internally water-cooled and contained a silicon bar in the depression. When power was applied to a short radio-frequency coil surrounding a small part of the boat, a narrow molten zone was formed in the bar. This zone was traversed along the bar to zone-refine the silicon and leave only the impurity boron, which does not segregate appreciably in silicon. The silver boat acts as a radio-frequency transformer, and current is induced into the silicon bar from the silver underneath it, as well as from the primary coil above it. The liquid silicon does not wet the cold silver surface; it should be emphasized, however, that melting is complete, without any skin or shell of solid silicon.

The results of pinch effect caused by mutually opposing electric currents can be observed following changes in the power setting or movements of the coil. Partial levitation of the melt can be obtained with suitably designed coils, particularly at a frequency of 500 kc./s. or lower; but at frequencies of 2 Mc./s. and above, these magnetic effects are very much reduced.

Another form of horizontal boat which has certain advantages in special cases can be made by using several straight parallel tubes mounted in a semicircle and closely spaced, so that the melt is confined in the boat by its surface tension.

The growth of silicon single crystals has been accomplished in the past by pulling from silica crucibles or by means of a crucible-free method (see, for example, ref. 2). The methods suffer from some disadvantages; in the former method, oxygen is introduced into the growing crystals due to chemical reaction between molten silicon and silica; in the latter procedure the density of dislocations tends to be high, and the diameter of the crystal is limited in size. Some attempts were made to grow single crystals horizontally in the silver boat, but these failed mainly on account of the large temperature gradient at the solid/liquid growing interface.

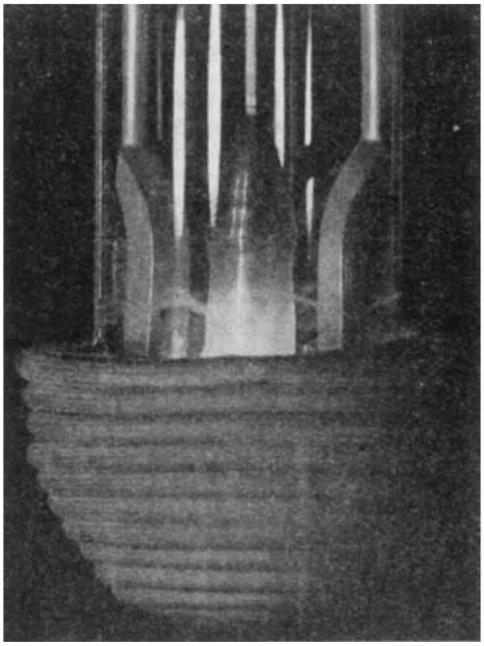

Fig. 1. A silicon single crystal grown from a cooled silver crucible

In order to obtain a more favourable thermal symmetry and a correct temperature distribution for the growth of single crystals, several novel types of crucible[3] have been designed. In one particular form (Fig. 1) a number of tapered and water-cooled silver tubes is arranged vertically, and curved inward at the bottom to join at their narrow ends and form a basket. This crucible is water-cooled internally, and mounted inside a radio-frequency primary coil which has the shape of an upturned skep. Power is induced through the spaces between the tubes, and also by induction due to the current which flows circumferentially in each tube. The tubes are placed sufficiently close together to prevent the melt, when formed, from escaping. Again it is important to note that melting is complete, and that no thin skin of silicon remains as in 'skull melting'.

Single crystals of silicon which are oxygen-free and dislocation-free have been grown by the Czochralski method from crucibles of this and similar types, using silicon which was obtained by the thermal decomposition of pure silane gas[4]. Life-times up to 1 msec. have been measured and resistivities greater than 1,000 ohm cm. have been preserved. Electrical and radiochemical tests have not detected the presence of silver in these crystals.

The cold-metal crucible has been also successfully used as a non-contaminating container for the melting and processing of such materials as ferrites and allied oxides, rare earth metals, molybdenum, titanium, uranium and boron; it is not restricted to high-temperature melting applications only.

We wish to acknowledge the work of Mr. F. J. Raymond, who is a co-inventor, and also other assistance given by colleagues of Standard Telecommunications Laboratories, Ltd., who are associated with this project. We thank the management of these Laboratories for permission to publish this communication.

H. F. STERLING
R. W. WARREN

Standard Telecommunication Laboratories, Ltd.,
London Road, Harlow, Essex.

[1] Hannay, N. B., edit., *Semiconductors*, 124 (Reinhold Pub. Corp., New York, 1959). Brit. Pat. 827676 (March 7, 1957).

[2] Brit. Pat. 829422 (November 14, 1957).

[3] Brit. Pat. 871156/7/8 (November 21, 1957).

[4] Brit. Pat. 826575 (September 17, 1956).

RADIOCHEMISTRY

Organic Extraction from Fused Uranium Chlorides

FUSED plutonium and uranium chlorides may be a good nuclear fuel for a fast breeder reactor[1–4]. The aim of the present work is to investigate some chemical properties of fused chloride components, in particular, the presence of dissociated or associated or non-ionic molecules, and the possibility of using the liquid salt–liquid organic extraction. Some recent results concerning the extraction processes with fused