



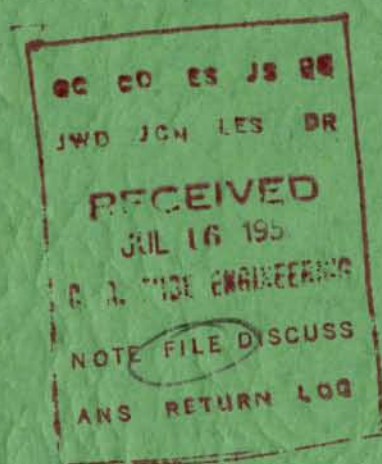
LB-1036

CRYSTAL PULLER OF CZOCHRALSKI

TYPE FOR GERMANIUM OR

SILICON WITH 60-CYCLE

3-PHASE HEATER



RADIO CORPORATION OF AMERICA
RCA LABORATORIES
INDUSTRY SERVICE LABORATORY

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Approved



Crystal Puller of Czochralski Type for Germanium or Silicon with 60-Cycle 3-Phase Heater

This bulletin describes a relatively inexpensive crystal puller of the Czochralski¹ type for use with germanium or silicon. The advantages of this puller are fast heating, clean atmosphere, excellent temperature control, and self stirring of the melt.

Silicon crystals, germanium crystals, germanium-silicon alloy crystals containing up to 8 atomic percent of germanium, germanium p-n junctions, and rate-pulled germanium crystals have been made in this puller.

Principle of Operation

The novel part of this furnace is the use of three-phase 60-cycle currents to supply the heat. The heater consists of a split carbon cylinder. At one end the three sections of the cylinder are joined in a stub. At the other they are joined to individual flanges (see Figs. 1 and 2). Three 1000 ampere currents go down the legs of the heater and join in the stub. The effects of this method of operation are three:

¹The Czochralski process of crystal growing is well known and is described fully in LB 892.

(a) Fast heating because a large power excess can be utilized in this type of heater.

(b) Stirring of the melt by the induction motor effect of the three-phase 1000 ampere currents. The melt of silicon or germanium rotates at about 100 rpm in its retaining quartz crucible.

(c) Fast cooling because the heater is fastened to water cooled leads.

The conjunction of these three effects with the precise temperature control described later yields a very smooth and uniform crystal.

SCHEMATIC CROSS SECTION OF CHAMBER OF CRYSTAL PULLER

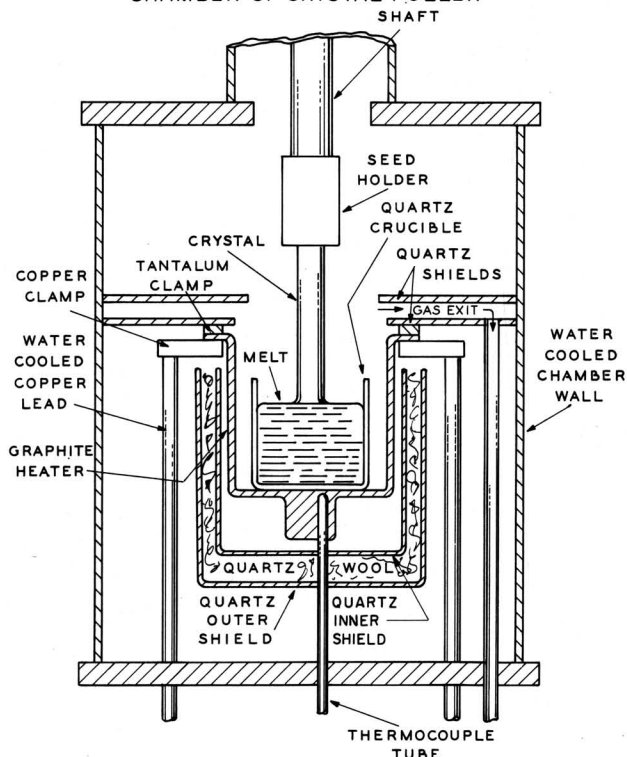


Fig. 1 - Schematic cross section of chamber of crystal puller.

Construction of the Puller

Fig. 1 shows a cross section of the crystal puller

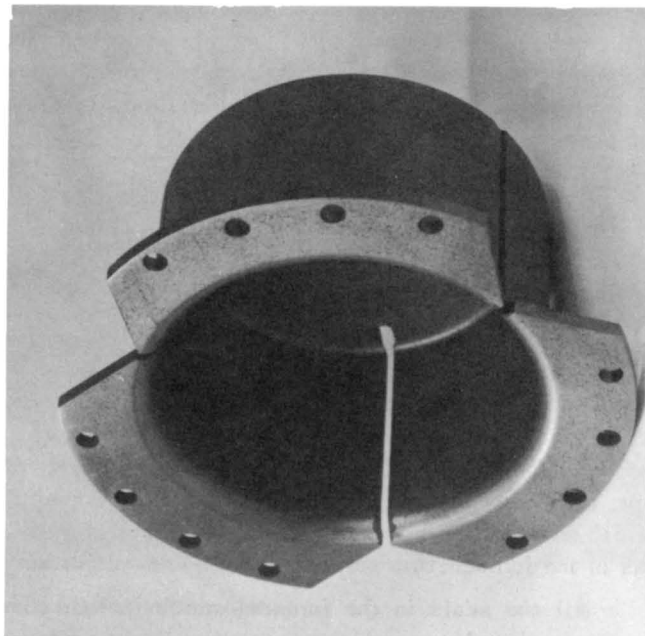


Fig. 2 - Carbon heater unit.

heating chamber. The crux of this design is the heater, machined out of a 4 inch rod of pure carbon and shown in Fig. 2.

A quartz crucible containing the charge is placed inside this heater. The heat is produced by passing large 60-cycle currents directly through the legs of this heater. These currents can be as large as 1000 amperes at a voltage of 5 volts per leg. Currents are supplied through six water cooled copper leads. The flange on the carbon heater is clamped directly against the copper underneath and is held on the top with a tantalum clamp. The use of six leads makes it possible to join them in pairs for use with three-phase currents resulting in a strong rotational force on the melt, or with six-phase currents connected as two opposing three-phase arrangements, so that there is no net rotational force.

The same rubber hose that insulates the electric cables also supplies the cooling water. These hose-enclosed cables are wound on the transformers as secondaries and each one is continuous from the Y connection of the secondaries to the connection to the furnace. Water cooling of the leads and transformers accomplished in this manner allows the use of smaller copper and yields a moderately flexible power lead.

To conserve power somewhat the heater is shielded by two quartz cups into which it is suspended from its leads. The space between these quartz shields is packed with quartz wool. Quartz is used because of the temperature that must be withstood when silicon is being pulled. The chamber in which heater, shields and leads are enclosed is six inches in diameter. It is water cooled. This eliminates any possibility of vaporization from the walls of the chamber, or of passage of oxygen through the walls, such as happens with hot quartz. Since the chamber and pulling mechanism are gas tight, a very small change in the design would make it possible to operate in a vacuum.

Gas Supply

Gas is fed into the chamber from the top and from an entrance in the bottom plate. The exit for the gas is between two quartz plates located immediately above the heater and crucible. The purpose of this arrangement is to sweep out immediately, as they are produced, vapors from the melt. This applies especially to the production of SiO from an Si melt. Because of the turbulence of the gas in the furnace, this purpose is not achieved perfectly.

All the seals in the furnace, which include those for water cooled power leaks, gas-exits, main seal, etc. are formed by the compression of a silicone rubber gasket. This includes the seal through which the pulling rod

enters the furnace chamber. This type of seal is gas tight.

Temperature Control System

The temperature control is based on the Brown Elektronik Amplifier and a power amplifier. The power amplifier controls a saturating reactor in the furnace supply thus giving a continuous control of power input (see Fig. 3).

The input to the Brown Elektronik Amplifier consists of two parts: thermocouple voltage from a thermo-

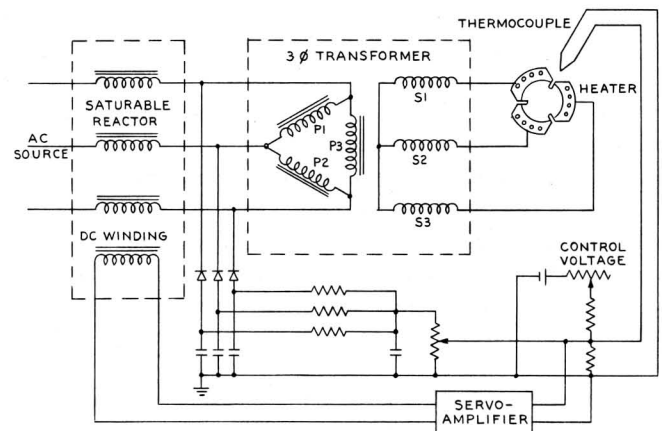


Fig. 3 - Simplified circuit diagram.

couple inserted in the stub of the heater, and rectified heater voltage reduced to thermocouple level. These two are added and compared to a d-c control voltage derived from batteries. The effect of a small increase in control voltage is to actuate the Brown Elektronik Amplifier and power amplifier until the increase is matched by a like increase in the rectified and reduced heater voltage. This happens at once. Then the temperature of the heater starts to increase and the thermocouple voltage with it. As the thermocouple voltage increases the control circuit will gradually decrease the heater voltage until temperature and heater power come into balance.

On the other hand, if line voltage varies, the effect of the control circuit is to restore the heater voltage to its proper value immediately, before the temperature changes. The power control, obtained in this way, is sufficient to operate the puller without the thermocouple if the thermocouple fails, and this has actually happened.

Results

This puller has been used primarily for silicon, for

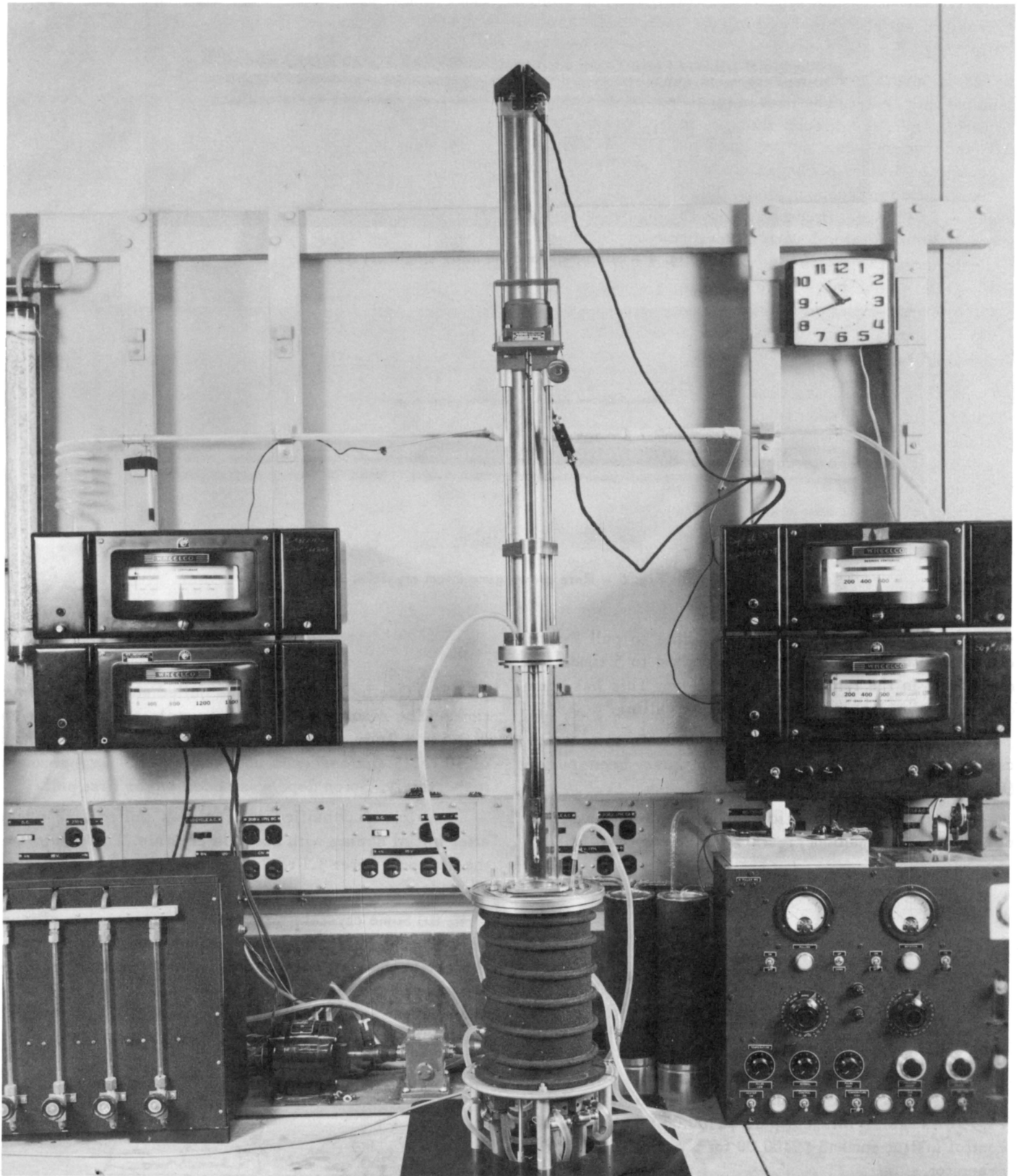


Fig. 6 – Complete Czochralski type crystal puller.

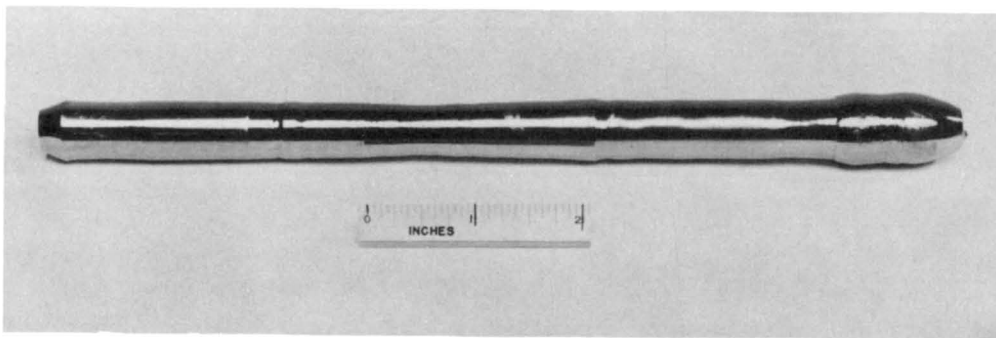


Fig. 4 – Silicon-germanium alloy single crystal.

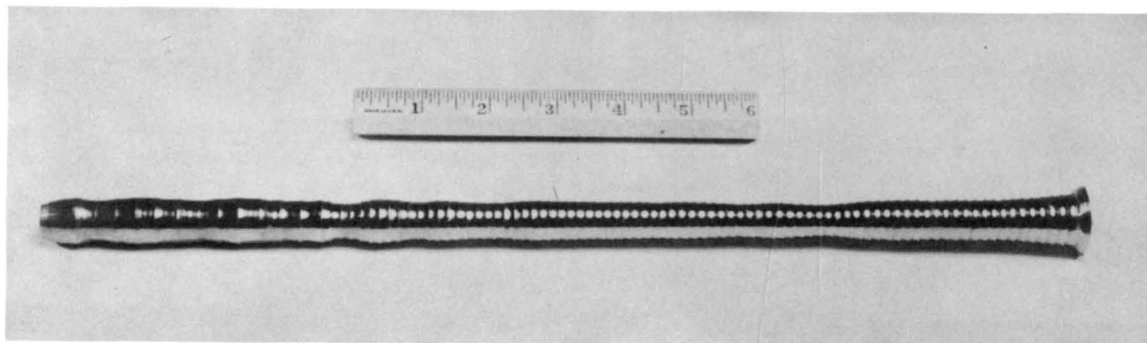


Fig. 5 – Rate grown germanium crystals.

which it was built. It has been possible to pull P-type crystals with lifetimes in microseconds 4 to 5 times the resistivity in the range $1/2$ to 5 ohm-cm. This has been done by adding boron and then multiple pulling.

Single silicon-germanium alloy crystals up to 8 percent atomic weight of germanium have been pulled (Fig. 4).

By use of an automatic scheduling device, a series of rate grown germanium crystals were made (Fig. 5).

Costs

The cost of the control, power reactor and transformer for this unit is about \$2000.00 including installation. A temperature indicating device (not necessary when working from the freezing of the melt) costs about \$300.00 more. (The complete installation is shown in Fig. 6). As against this, compare \$2300.00 for a commercial control instrument and \$9400.00 for a purchased 25 kw rf. generator.

Data

(a) One lot of material was pulled successively four times. The resulting two final crystals had resistivities of 3.5 to 4.0 ohm-cm, and 2.5 to 3.5 ohm-cm, and lifetimes of 10 to 15 microseconds and 12.5 to 17.5 microseconds respectively. Boron dope was added on the first pull.

A lot of densified silicon was pulled five times after initial doping with boron in crystals. The results on the fifth pull were: 3.1 to 2.7 ohm-cm, and 10 to 15 microseconds.

(b) Some crystals were made of a germanium-silicon melt. The highest germanium content was about 8 atomic percent in the melt. The crystal grown with this percentage started to grow on a silicon seed. At the start it was about 2 atomic percent of germanium. When it reached about 8 atomic percent it went poly-crystalline.

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