A Vertical, Vacuum, Split-Tube, Graphite-Resistance Furnace

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By splitting the graphite resistor tube of a vertical, evacuated, high temperature furnace, the construction is simplified, the possibility of resistor breakage is circumvented and the zone of highest temperature is brought deeper in the furnace than in the spiral or solid tube types of vertical furnace. The construction of the furnace cover permits crucibles to be put in or removed from the furnace without breaking the main vacuum seal. The melt can be observed visually, or the gases from the melt can be drawn off to other apparatus, without having to pass through the main furnace volume. The furnace reaches 2000°C on 7 kw of power input.

IN some recent experiments on the de-emanation of radioactive solids, a vacuum furnace was required in which there was not the necessity of breaking the main vacuum seal each time a new crucible was used or a new material tested in the furnace, and in which gases given off from the melt could be withdrawn from the crucible without their entering the main furnace volume.

Scale drawings of the furnace are shown in Fig. 1, while Fig. 2 shows a photograph of the cover assembly. The case is of chromium-plated steel, 7 inches in diameter and 9 inches in height and stands $\frac{1}{2}$ inch under the surface of flowing, cooling water in a larger, steel vessel. The cover plate is bolted to the case and is made vacuum tight by a groove and lead-wire-gasket joint. The cover plate carries four hollow posts, at the top of each of which is a fiber-insulated packing gland through which passes a $\frac{1}{2}$ inch copper tube carrying cooling water and electric current to the hollow copper electrodes. This construction affords sufficient length to the tubes to permit their flexing to accommodate the clamping in or removal of graphite resistors from the semi-circular jaws of the electrodes. The electrodes form the two halves of a split circle and are drawn together by two mica-insulated bolts, clamping between themselves the graphite resistor. The two gas outlets may be seen in the furnace cover in Fig. 2 (not shown in Fig. 1). These permit either evacuation of the furnace interior, flowing of gas through the furnace, or the application of pressure.

The principal novelty of the furnace is the form of the graphite resistor and the copper electrodes. These may be seen in Figs. 1 and 2. The resistor tube is cut from Acheson graphite. It is 7 inches long, with $\frac{1}{8}$ inch thick walls, and is split along a diameter for all but the last (bottom) inch of its length. The width of this gap between the two sections of the resistor is $\frac{1}{4}$ inch, as is also the

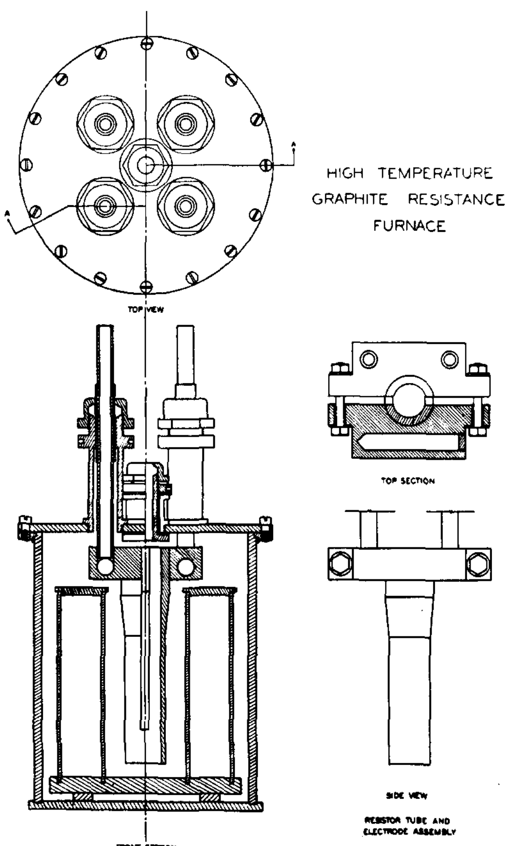

FIG. 1. Details of construction of the split tube furnace.

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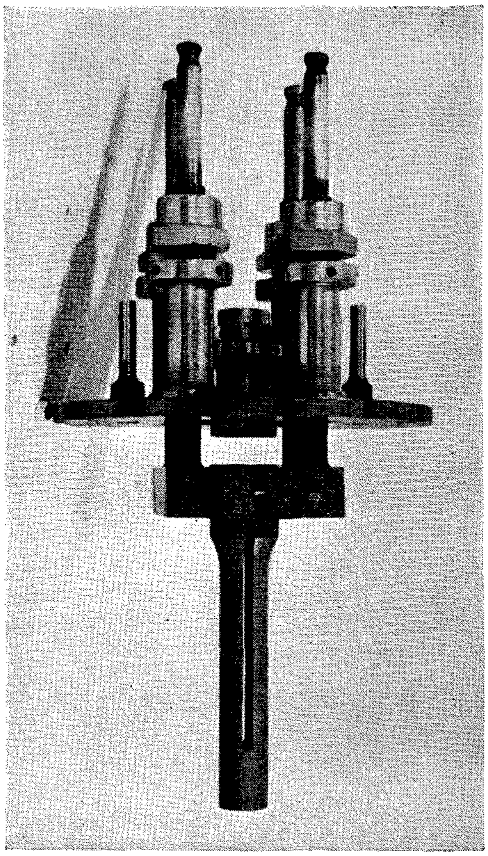

FIG. 2. Details of furnace cover, electrodes and resistor.

gap between the copper electrodes. At the upper end of the split tube, where the resistor is clamped between the semi-circular electrodes, two blocks of lavite (baked soapstone) fit into the gaps between the two halves of the resistor tube in order to insulate them from each other and yet provide mechanical reinforcement against the pressure of the electrode clamps.

The electric current enters through one electrode, passes downward in one side of the split tube, across the intact lower end of the tube, up through the other side of the tube, and out through the second electrode. This construction completely eliminates any danger of resistor fracture due to thermal expansion of the graphite in ordinary rigid electrodes, for the lower end of the resistor tube is perfectly free to expand. A second advantage of this construction is that only one end (upper) of the resistor loses heat by conduction to the water-cooled electrodes.

The resistor shown in Figs. 1 and 2 has a shoulder fitting into the electrodes. Its purpose is to provide a resistor diameter which is smaller than the electrode clamp diameter. Straight, shoulderless resistors are also used and are easier to make. The shouldered resistor of Fig. 1 has a resistance of 0.024 ohm when new; this gradually increases to about 0.048 ohm before failure. The average resistor life when the furnace is not evacuated is 3 hours, representing about 20 heatings and coolings. The furnace can be operated without evacuating because the graphite resistor soon burns up all the oxygen in the furnace volume, and thereafter no air enters the furnace, hence inactive gases only are present. Evacuation, however, greatly lengthens the life of the resistor.

The copper electrodes are cut from solid stock. The water conducting circuits are put in them by drilling in from the outside, then plugging the outer end of the drill hole with a threaded, copper plug, silver-soldered in place. The copper tubes which carry the cooling water and electric current are also silver-soldered into the electrodes.

In the center of the cover plate, on the vertical axis of the furnace, is a packing gland, $\frac{1}{2}$ inch in diameter. The crucible is introduced into the furnace through this gland. This construction permits exchanging crucibles without opening the furnace. If desired, a larger or smaller gland can be used in order to accommodate crucibles of different diameters.

The crucible is a tube, closed at the lower end, of quartz, graphite or other material, as is required by the experiment at hand. The crucible is 11 inches long and extends along the axis of the furnace to the highest temperature zone, near the bottom of the split tube resistor. The melt can be directly observed from above the furnace, or, if desired, the outside end of the crucible can be connected to any other apparatus in which the gases from the melt are to be examined. Even in prolonged runs at 2000°C, the upper end of a graphite or quartz crucible is easily kept within 10°C of room temperature by a small jet of compressed air blowing on a rubber tubing sleeve joining the crucible to other apparatus.

Because of the difficulty of outgassing graphite, this furnace is not intended for high vacuum work. There is, however, no difficulty in operating at pressures of 1 to 5 mm Hg in the main furnace

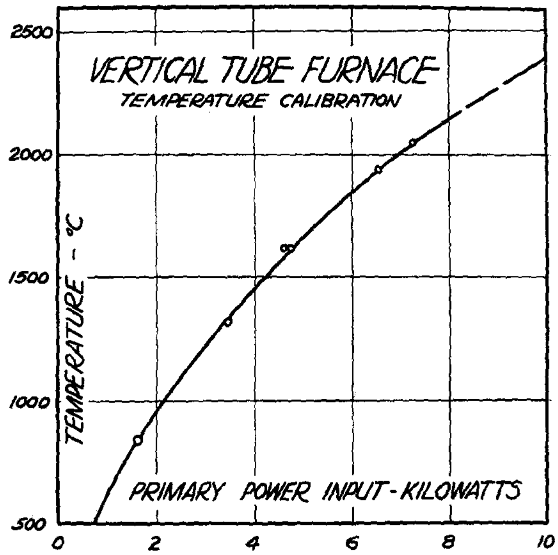

FIG. 3. Equilibrium temperature as a function of transformer primary power input.

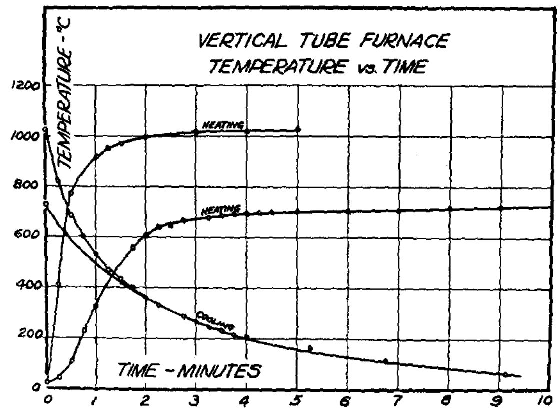

FIG. 4. Typical heating and cooling curves showing time-response characteristics.

volume without continued pumping, or at slightly lower pressures by continuous pumping during heating. On the other hand, inside the quartz crucible, high vacuum is readily obtained. When graphite crucibles are used and vacuum conditions are desired, the crucible end exposed outside the furnace should be painted with water-glass to prevent vacuum leaks through the very porous graphite.

A hollow graphite radiation screen, filled with graphite powder, surrounds the electrode and retards heat transmission from the resistor to the cold metal walls of the furnace case. Arsem[1] has shown that a similar screen in his furnace increased the furnace efficiency nearly fourfold.

Furnace temperatures up to 1100°C were measured with a chromel-alumel thermocouple in a quartz crucible. From 900 to 2000°C a Leeds and Northrup optical pyrometer was used, temperature observations of the inside wall of the resistor being made through the $\frac{1}{2}$ inch crucible gland in the furnace cover. Characteristic curves are given in Figs. 3 and 4, showing (1) equilibrium temperature against watts input and (2) temperature *vs.* time curves for both heating and cooling, with the furnace containing one atmosphere of nitrogen. As will be seen, the furnace attains 2000°C on only 7 kw power input.

The power supply[2] is a 220 volt to 22 volt step-down, 10 kw transformer, and is controlled by a water immersed resistance in series with the primary. This resistance is tapped into eight sections bearing ratios to each other similar to those between members of a set of analytical balance weights. By shorting out the proper sections all resistances between 0.025 and 4.025 ohms may be reached by 0.025 ohm steps.

I am indebted to Dr. Alexander Goetz for the suggestion that the split tube construction here adopted could be extended to a similar resistor split into three sections, and fed from the three terminals of a three phase transformer, the lower end of the crucible, where the three sections join each other being the neutral point of the circuit.

This furnace was constructed during the course of radioactive investigations in connection with Professor R. A. Millikan's cosmic-ray research program supported by a grant from the Carnegie Corporation of New York. Messrs. Julius Pearson and Bruno Merkel have received my cordial thanks for the interest and care they took in constructing this furnace.

[1] Arsem, J. Am. Chem. Soc. **28**, 921 (1906).

[2] Evans, Rev. Sci. Inst. **4**, 223 (1933).