

of other arrangements have been tried with varying success, but only the sapphire rod has proven to be completely satisfactory and reliable. The high thermal conductivity of sapphire in this low temperature region plus the intimate nature of the solder bond are the properties which make this holder satisfactory.

The liquid helium consumption rate of the apparatus is somewhat less than one liter per hour, independent of whether radiation is proceeding or not. The major cause of consumption is conduction down the necessary electrical wiring connecting to all four samples. Since one effectively is performing four irradiations at one time, not to mention the advantage of simultaneous comparison of different samples, this loss rate is considered very reasonable.

Liquid helium is transferred from a standard storage container with two conventional helium transfer tubes,

one inserted into the storage container, the other inserted into the top of the helium can H. A joint is made between the two transfer tubes by joining the inner, helium-transferring tubes, which extend beyond the outer jacket slightly, with a short length of rubber tubing, then swathing the joint with Fiberglas padding. This has proven to be a simple and efficient manner of forming a temporary joint.

The liquid helium level is monitored whenever desired by probing with a $\frac{1}{8}$ -in. thin-walled stainless tube, terminated by a $\frac{1}{2}$ -in. tube at the top. This device is inserted, after precooling in liquid nitrogen, into H. Pressure vibrations characteristic of the vapor above the liquid level die out when the interface is penetrated; these vibrations are sensed by the experimenter's finger capping the $\frac{1}{2}$ -in. tube.

Compact High-Temperature Vacuum Furnace

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A small furnace design is described. The furnace is heated by electrical resistance in a refractory metal tube which is surrounded by radiation shields and a water-cooled housing arranged in a compact assembly. The heater tube can be replaced readily without disassembling the furnace. The furnace can be heated to a temperature of 2200°C or higher in vacuum or nonoxidizing atmosphere.

INTRODUCTION

ALTHOUGH a number of furnace designs¹⁻⁵ have been proposed for high temperature use in the laboratory, the furnaces in general are not compact and require a significant amount of disassembly to replace damaged heater elements. The present article describes a furnace design whose compactness and ease of heater element replacement with minimum furnace disassembly makes it very convenient for laboratory use.

FURNACE DESIGN

The furnace is heated by a tubular resistance element surrounded by metal radiation shields, the entire assembly contained within a water-cooled housing. The overall configuration and some of the major dimensions are shown in Fig. 1.

As can be seen in Fig. 1, the furnace is a compact unit which can be quickly placed upon or removed from a vacuum system. It is small enough that, with electrical and water connections removed, it can be easily handled and stored. The housing requires disassembly only infrequently since the heater tube, which needs the most frequent attention, is accessible from the open ends of the furnace.

For heater tube replacement, the furnace is removed from the vacuum system, thereby exposing the clamping rings. Removal of the clamps allows replacement of the old heater tube with a new one. The ends of the heater tube are slit into a number of narrow tabs to be spread for clamping. The tabs at the lower end are long enough to leave $\frac{1}{4}$ in. between the tube diameter and point of clamping. This latter feature permits sufficient flexibility to accommodate vertical thermal expansion of the heater. Details of the clamping at top and bottom are shown in Fig. 2.

The housing consists of two parts electrically separated except through the heater tube. These two parts are joined at the upper and lower assembly flange with an O-ring serving as vacuum seal. Electrical insulation between the flanges is provided by an insulating layer between the flanges and by using fibre bushings on the screws.

¹ H. G. Sowman and A. I. Andrews, *J. Am. Ceram. Soc.* **33**, 365 (1950).

² R. F. Domagala and D. J. McPherson, in *Vacuum Metallurgy*, edited by J. M. Blocher (The Electrochemical Society, Inc., N. Y., 1955), p. 67.

³ J. M. Dickinson, in *1958 Fifth National Symposium on Vacuum Technology Transactions*, edited by W. G. Matheson (Pergamon Press, New York, 1959), p. 192.

⁴ E. V. Kornelsen and J. O. Weeks, *Rev. Sci. Instr.* **30**, 290 (1959).

⁵ J. Cohen and W. Eaton, *Rev. Sci. Instr.* **31**, 522 (1960).

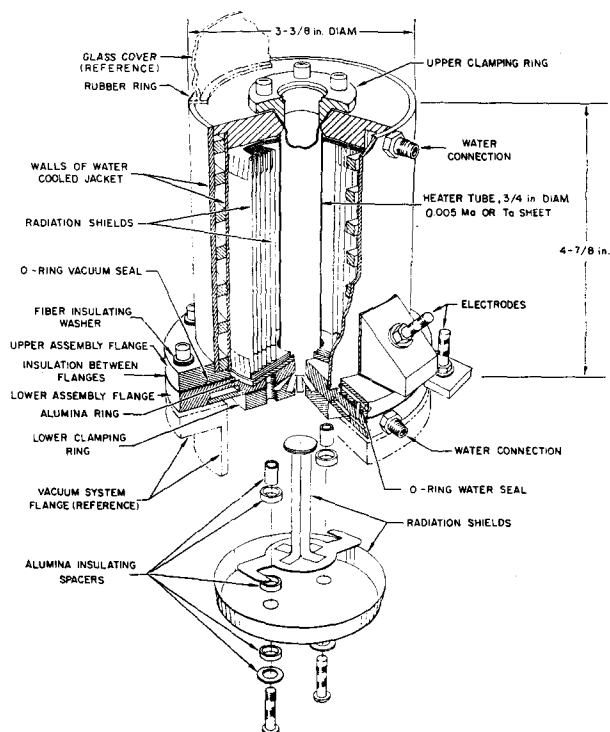


FIG. 1. Exploded view of furnace assembly.

The entire assembly is cooled by a continuous flow of water. The cooling water path starts at the upper water connection, spirals downward through the double wall, passes from the upper to the lower flange through an O-ring seal, goes around an enclosed and sealed passage in the lower assembly flange, and finally emerges from the assembly at the lower water connection. Thus, a single passage of water cools both end plates to which the heater tube is clamped, as well as the vertical walls. The end plates to which the heater tube is clamped at each end are made of copper to facilitate cooling of the clamping points.

The radiation shielding is all molybdenum. This permits the use of hydrogen in the furnace. (In contrast, tantalum

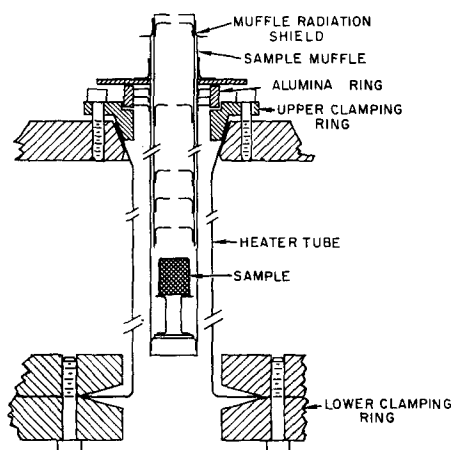


FIG. 2. Details of heater tube and muffle assembly.

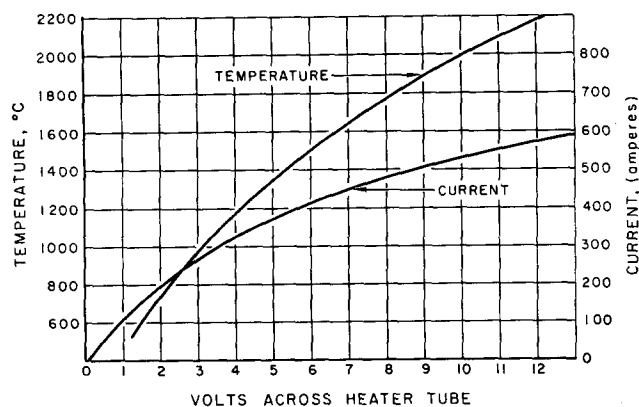


FIG. 3. Typical power and temperature characteristics.

cannot be used in hydrogen at lower temperatures because a tantalum hydride forms, leading to disintegration of the tantalum components.) Radiation shields are placed inside the heater tube from the top and the bottom to achieve maximum uniformity of temperature in the center of the furnace. Care must be taken, here, to avoid short circuiting of the heater tube by these inserted radiation shields. Therefore, they are supported at the outside ends by alumina insulators.

The radiation shield surrounding the heater tube is made entirely of 0.005-in sheet. The vertical portion is simply a multiple roll with successive turns separated by a narrow corrugated strip at top and bottom. A number of broad rings and retaining caps were placed at each end, and the entire assembly spot-welded sufficiently to give reasonable sturdiness. The alumina ring at the bottom of the shield assembly prevents electrical short circuiting to the lower assembly flange.

The bottom closure for the furnace is the vacuum system manifold. The vacuum seal at this point is a gasket or O-ring against the bottom face of the lower assembly flange. The top closure rests on the upper plate with a gasket for vacuum seal. Since the top closure is not itself a part of the furnace structure, it can be specially made for various experiments (i.e., to provide electrical leads or mechanical devices). In general, only a simple enclosure is necessary. For most of our work, a short Pyrex tube with an optically flat end-window was used.

A convenient method of placing a sample in the furnace also is shown in Fig. 2. The sample is contained within a deep closed-bottom tube, referred to here as a "muffle," suspended within the heater tube from an insulating alumina ring. The muffle in turn is closed at the top by a radiation shield assembly pierced by a series of holes. Temperature measurement is obtained by pyrometer-sighting through the holes.

Power requirements for this furnace are shown in Fig. 3. As indicated in the figure, temperatures of up to 2200°C are readily attainable with either Ta or Mo as the heater tube in argon or vacuum (or Mo in hydrogen).