High temperature laboratory furnaces

HIGH TEMPERATURE LABORATORY FURNACES. By

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ABSTRACT. Two laboratory furnaces for temperatures up to 1900° C are described. The preparation of the necessary ceramics is discussed.

The production of temperatures from 1500° C to 1900° C in the laboratory with simple means is still a matter of some interest. A laboratory furnace should be straightforward to assemble and to handle, and all parts which are likely to wear out should be easy to replace. For these temperatures, the only readily obtainable heater materials are molybdenum and tungsten and for these a non-oxidising atmosphere either of hydrogen or of a mixture of nitrogen and hydrogen has to be provided. There is also no great scope in the choice of an insulating material since (*a*) its melting-point should be well above 2000° C, (*b*) its dissociation pressure should still be low at this temperature, and (*c*) its reaction with the heater material should be negligible. Assuming the existence of materials which fulfil the conditions (*a*) and (*b*), there are two ways of fulfilling the condition (*c*). The one is to choose a material which does not react with molybdenum or tungsten at the highest temperatures, the other to construct the heaters in such a way that they shall not touch the insulating materials. One example of each of these solutions will be given.

For the first solution the only material available with a melting point well over 2000° C, which is chemically sufficiently inert at high temperatures, is beryllium-oxide (BeO). A tube of 20 mm. external diameter and 100 mm. long of this material was prepared in the following way. Fine beryllium-oxide powder, as obtainable on the market 99·5 per cent pure, was stamped into an aluminium-oxide crucible and fired to 1350° C in air. It was then crushed and sieved through a 100-mesh sieve (maximum particle diameter about 0·10 mm.) and mixed with 10 per cent by weight starch paste (containing 15–20 per cent of starch powder) to form a plastic dough. This was wrapped around a metal tube previously painted with paraffin wax so as to form a tube of the oxide which was 1 mm. to 2 mm. in thickness. The ends were cut clean with a razor blade, and after the tube had been allowed to dry for some hours, it was slipped off the mandrel by heating slightly above the melting point of the wax. To prevent the tube from collapsing, it was loosely filled with beryllium-oxide powder or was supported on a mandrel of aluminium-oxide; as the tube shrinks about 20 per cent, this mandrel must be that much less in external diameter than the internal diameter of the tube.

For the heating element, 35 turns, of 0·8 mm. diameter molybdenum wire was wound on an iron mandrel in which a screw thread had been cut. The spiral was 90 mm. long and had an internal diameter of 20 mm. Molybdenum wire was wound on the ends spirally to convey the current to the heater in order to keep their temperature lower than the temperature of the heater. The length of the end wires was 10 cm. The heater was annealed at a temperature of 1000° C. This heater enclosed the beryllium oxide tube *B* (Fig. 1). As will be seen in Fig. 1, the heater is contained in an iron outer case into which the hydrogen is led by the iron pipe on the right. The case is lined with porous silica bricks. Within this lining further insulation is provided by a layer of aluminium-oxide tubes, 8 mm. in diameter, which had a wall thickness of 1 mm. to 2 mm. This layer was built up to a thickness of about 2 cm. on all sides. Between these tubes and the heater are thin slabs *C* of beryllium oxide, forming a sort of channel and cover to protect the aluminium-oxide tubes from the hot wires. The

electrical connexions are taken through the lid (which, with the cover and the upper tubes, have been removed in the view given in Fig. 1). The casing is made airtight with Alundum cement. This furnace, with the molybdenum spiral, can be successfully raised to a temperature of 1950° C. The beryllium oxide has a high conductivity when hot and the part of the inner wall of the beryllium oxide tube, at the end of which the temperature drop was of
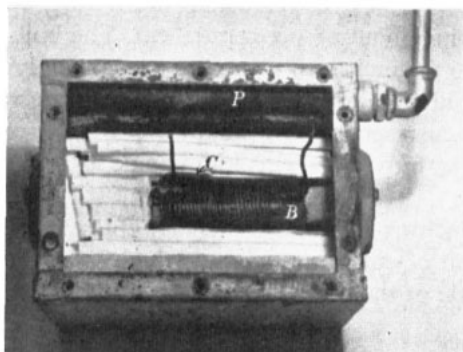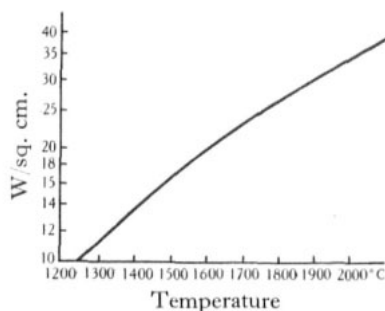

Fig. 1. Beryllium oxide furnace

Fig. 2. Energy temperature curve at equilibrium conditions, for furnace with molybdenum spiral employing beryllium oxide tube and aluminium oxide tube insulation

the order of 0·2 per cent, had a length of about 7 cm. Fig. 2 shows the energy temperature curve for equilibrium conditions at each individual temperature in terms of watts per sq. cm. (log scale) of heated inner surface of 7 cm. length. Though this furnace was satisfactory it would probably be difficult to construct on a larger scale, and therefore for a larger furnace the second solution was adopted in which the heater is separated from the insulator.
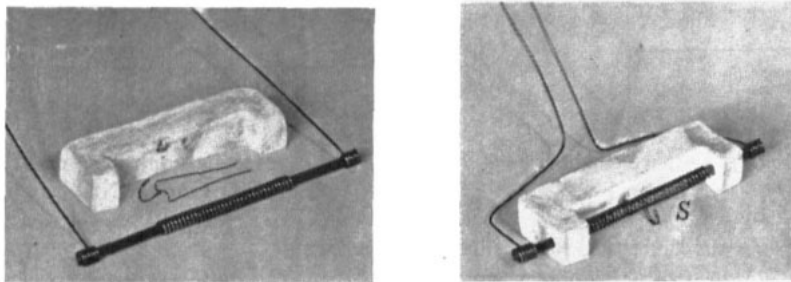

Fig. 3. Furnace elements consisting of zirconium silicate ceramic with tungsten heater and molybdenum end-pieces

Elements made in the form shown in Fig. 3 are used to build up the walls of the chamber in which the high temperature is required.

The heater is a spiral of 34 turns of 1 mm. tungsten wire wound and annealed on a 4 mm. mandrel. It is supported at its ends on short 4 mm. rods of molybdenum which are pushed into a few turns of the ends of the spiral and which themselves are held in the insulator. On the outer ends of these rods are wound a few turns of molybdenum wires, 1·2 mm. in diameter; the far ends of these wires are carried to a cooler part of the furnace and there connected as required, i.e. in parallel or in series or in groups. The middle of the heater spiral is supported by a piece of 0·5 mm. tungsten wire which passes through holes in the insulator. Fig. 4 is a sketch of a furnace chamber consisting of four heater elements, a top lagging and a bottom lagging made of small bricks B. The end-gap between the heater

elements can also be closed by small bricks. The furnace chamber was surrounded by two
1 cm. layers of zirconium silicate plates. These were surrounded by porous fireclay plates
(e.g. Morgan), which formed the lining of a metal box. In order to secure a better heat
insulation thin-walled zirconium silicate tubes were used in a later model instead of the above-
mentioned plates. The necessary connexions to the individual elements were made on top of
the insulation in the coolest part of the furnace. The material chosen for the insulating
supports and for the lagging was a mixture of 50 per cent zircon sand (ZrO_2. SiO_2) with
50 per cent commercial zirconium oxide (containing about 18 per cent silica). The bulk of
the zircon sand had a grain size of 50–200 mesh to the inch with the major proportion lying
between 100 and 150, i.e. 0·08 mm.–0·12 mm., while the commercial zirconium oxide was
a very fine powder (unfired). The composition corresponds to a mixture of 74 per cent
zirconium oxide and 26 per cent silica (where 33 per cent of the zirconium oxide is from the
sand).

It will be seen from the zirconium oxate silica melting-point diagram (Fig. 5) that smaller
additions of either would lower the melting point of zirconium silicate which is at about
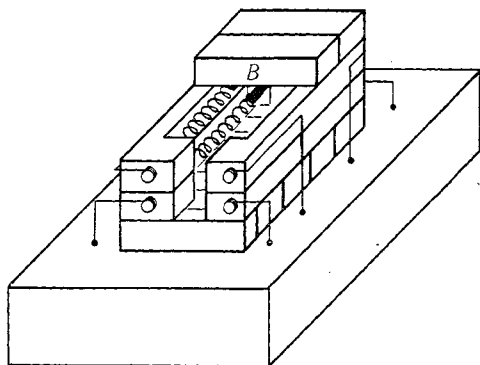2430° C, while larger additions of zirconium oxide of the order of 40 per cent would tend
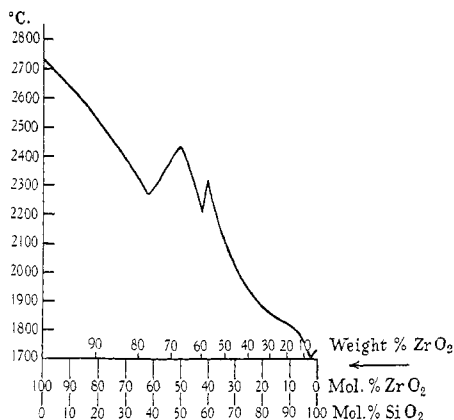

Fig. 4. Furnace chamber

Fig. 5. Melting-point diagram of ZrO_2/SiO_2
mixtures (after N. Zhirnova)

to raise it.* However, as zirconium silicate dissociates at very high temperatures, the silica
tends to evaporate from the hotter parts of the furnace if temperatures over 1850° C are
maintained over prolonged periods. Thus the melting temperature of the ceramic body
becomes automatically higher, while at the same time cracks develop, due to the shrinkage
connected with the dissociation.

It is not considered necessary to stress the point that the grain size distribution of a
ceramic product determines its workability when formed, e.g. pressed or moulded, its
shrinkage and its porosity. A few suggestions are now given about the preparation of the
zirconium silicate insulators in the laboratory. The above-mentioned mixture of zircon
sand and zirconium oxide is wetted with a somewhat sticky 10 per cent solution of an
artificial resin (Paralac, Diakon, Patrex, etc.) in a non-aqueous solvent, e.g. acetone or cello-
solve, and agitated for 1–2 hr. in a glass bottle in order to get the powders well mixed. About
6 per cent starch paste is added in a mortar (in terms of volume the starch paste is about 1/3),
so that a plastic dough results. After 15 min. thorough mixing and kneading, it is tamped
by hand with the aid of a tamper into a wooden form like the one shown in Fig. 6, which

* N. Zhirnova, *Chem. Abstr.* p. 1902 (1935).

consists of five pieces screwed together. Before starting to tamp, the box is painted with paraffin wax (the parts having been soaked in paraffin wax to avoid swelling). When the mould is full, the screws are taken out and it is put in a dry atmosphere for 12 hr.; then it is put into a furnace and heated to about 60° C so that the paraffin wax melts. The wooden form pieces are removed and the ceramic taken off and allowed to dry for a further day.

It is then drilled or machined with any tools, using water as a lubricant. After that, the completed insulators are fired to about 1350° C, when they shrink about 5 per cent, depending on the amount of sand used in relation to the fine powder. Should any of the pieces develop cracks after drying, they can easily be mended with some wet mixture of the same kind. If production of a large number of similar pieces of the same dimensions is intended and metal moulds and pressing tools are available, they can obviously be pressed. But it should be mentioned that the drilling and machining is easier with tamped material. A certain grain size adjustment has then to take place to get good results.

Fig. 6. Tamping form for furnace elements

In cases where the temperature requirements are less severe, an addition of 30 per cent commercial zirconium oxide is sufficient and even fine quartz-meal may be used up to 15 per cent without lowering the melting point more than about 150° C. The insulation plates mentioned above are made by tamping or rolling the dough with a wooden roller. The dimensions of each plate should not exceed 5 cm. × 10 cm.; a thickness of 5 mm.– 6 mm. has proved satisfactory. The chamber had 6 cm. × 7 cm. × 4 cm. inner wall dimensions, i.e. 168 sq. cm. of surface. It ran at 22 W per sq. cm. at 1900° C, using the insulation described.

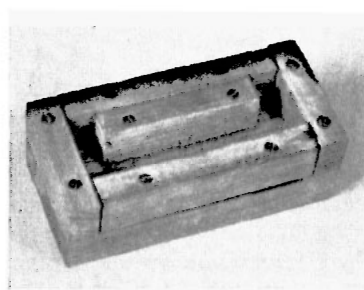The four tungsten spirals of the dimensions given above connected in series dropped 100 V at the current used.
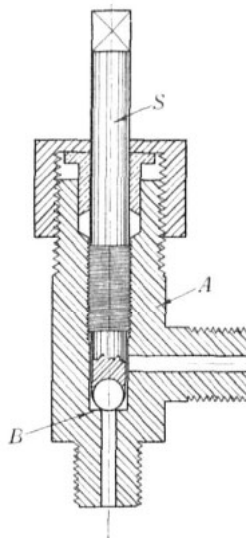

LABORATORY AND WORKSHOP NOTES

A SELF-SEATING VALVE

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THE valve shown in the accompanying sketch may be of interest to those engaged in work on gases either under vacuum or at relatively high pressures. It consists essentially of a screwed spindle S having a very fine thread and carrying at its lower end a hardened rustless steel ball spun into the suitably bored ball seat. The ball is left free to rotate in order to prevent localized wear on the surface. The housing A has a sharp-edged seat at B so that the ball on its first impression moulds the seat to suit. The spindle gland may be of any approved type suitable for the desired purpose.

Experience with this type of valve over a number of years has shown it to have many advantages over most types of gas valves in general use. It has been found to be perfectly tight under the

A self-seating valve

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