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CENTRAL FILES NUMBERCF-56-8-133

DATE: August 17, 1956

COPY NO. 21

SUBJECT: A Small High Temperature Laboratory Furnace

TO: Distribution

FROM: C. E. Curtis

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A SMALL HIGH TEMPERATURE LABORATORY FURNACE
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ABSTRACT

A small high temperature furnace is described which is simple and comparatively cheap in construction and in which temperatures up to 2000°C or higher can be attained in 2 to 4 hours. Zirconia is employed as the refractory material; heat is supplied by oxyacetylene.

Construction procedures, drawings and operational details are given.

INTRODUCTION

A small inexpensive furnace capable of rapid heating to temperatures of 1800° to 2200°C in air is often a handy tool in research. Apparently such furnaces are not readily available commercially and, although their use is not new, experience with their performance and operation seems to be limited to comparatively few people.

A furnace of this type was built and has been operated usually at temperatures of 1700° to 1800°C over a period of five years in the Ceramic Laboratory of the Oak Ridge National Laboratory. This report is intended to make the experience thus obtained available to those interested in building or operating such a furnace, whether they are experienced in ceramic techniques or not.

CONSTRUCTION

General

A furnace of the type to be discussed is shown in the photograph accompanying. It consists essentially of a cylindrical refractory liner (A), supporting casing (B), insulation (C), and cover (D); heat is

supplied by an oxyacetylene torch as shown. Temperatures are controlled manually and are read by means of an optical pyrometer sighted through a sight tube and through the flame and directly on the ware being fired; or, alternatively, on the bottom of a closed-end tube extending well inside the furnace.

A feature of this furnace that is somewhat unique is its portability as can be seen in the photograph.

Construction Details

Liner

The liner in the furnace under discussion is six inches in inside diameter, seven inches high and one inch thick. It is made preferably of "stabilized" zirconia because this oxide, although it is rather expensive, is the most refractory (mp = approximately 2800°C, softening temperature (when unsupported) approximately 2100°C) and has the lowest thermal conductivity (5 BTU/ft²/hr/°F inch thickness at 2400°F⁽¹⁾) of the more available ceramic materials. Zirconia liners of the type needed can be obtained on special order from refractory suppliers* or they may be made by the user if he has the proper equipment (particularly a furnace of sufficient size in which to fire the liner).

(1) S. M. Phelps, Props. Cer. Mats., Refractories Fellowship, Mellon Institute, Pittsburgh, Pa. Copyright 1918.

* Zirconia is obtainable from the following concerns:

Norton Co., Worcester, Mass.

Titanium Alloy Mfg. Division of National Lead Co.,
111 Broadway, New York, N. Y.

Zirconium Corp. of America, Solon Center Bldg., Solon, Ohio.

Liner Compositions

The data from this point to page 6, "Liner Preparations", are given for the benefit of those who intend to fabricate their own liners and covers. Commercial "stabilized"* zirconia ramming mixes are convenient for this purpose in that they usually contain a combination of different grain sizes designed to reduce shrinkage and a mixed-in binder to produce dry strength in the ware.*** If it is desirable to reduce the thermal conductivity of the commercial composition further, this may be done by mixing a fine-grained combustible material, such as wood flour, to the ramming mix. Wood flour up to 20 per cent by volume of the mixture may be added (preferably to the dry batch) without undue weakening of the fired liner (Table 1, Composition No. 1).

* "Stabilized" zirconia usually contains five to eight weight per cent of calcium oxide which is added to convert at least a portion of the monoclinic zirconia to a cubic solid solution phase and thus to reduce the amount and destructiveness of the monoclinic-to-tetragonal inversion occurring at 1050°C. In one case, at least, completely cubic zirconia is also sold as "stabilized".

*** Commercial zirconia usually contains three to five per cent of hafnium oxide which is present in most zirconium ores. Its separation from ZrO_2 is considered as not economical for most purposes. However, the presence of HfO_2 in the refractories is probably beneficial rather than otherwise. See for instance: C. E. Curtis, L. M. Doney, J. R. Johnson, Some Properties of Hafnium Oxide, etc., Jour. Amer. Ceram. Soc. 37 (10), 458-465 (1954).

Suitable ramming mixes can be made up readily in the laboratory, using formulas such as Compositions No. 2, 3 and 4 in Table 1. Compositions 2 and 3 offer an economy in the use of ZrO_2 in that their main ingredient is a grog made from used liners. The latter are crushed to pass an eight-mesh screen and the resulting grain is blended with a portion of unfired, fine-grained, stabilized zirconia to produce strength in the fired liner. The dextrine and wood flour are preferably mixed into the dry batch. Water is then added until the consistency is such that the mass slowly crumbles after compaction in the hand. It should be noted that the proportions given in the table are not critical.

Liner - Ramming Procedure

The liner is most conveniently formed by "ramming". For this purpose a suitable mold consists of an outer steel shell (8" i.d.) of approximately 14 gauge steel, a removable core of wood (6" o.d.); a one-inch space separates shell and core; a wood base is designed to hold the shell and core in place in ramming. Ramming procedure consists in filling the one inch space to a depth of about an inch with the damp mix and tamping this layer thoroughly and evenly with a flat-end metal or wooden tool. The tamped surface is roughened each time to give better adhesion between it and the next layer. The liner is built up thus to a height of seven or eight inches (to allow for six inches in the clear after it is trimmed and set in the furnace and a layer of zirconia, approximately one inch deep, is placed in the bottom).

The wooden core is then extracted, this being more easily accomplished if the core is tapered toward the bottom and a handle is provided; it may be found necessary to precoat the core with a lubricant such as paraffin or stearic acid for easier extraction.

Liner - Drying Operation

With the core extracted, the liner may be allowed to dry in the open room until fairly strong. The outside shell is then removed and drying can be completed in the open room or in a dryer at 100°C.

Openings for the inlet and outlet tubes of the furnace can be dug out at this stage of fabrication but with more risk of breaking the liner than if the operation is performed on the fired liner as detailed later (page 6).

Furnace Cover - Forming and Drying

The furnace cover is conveniently rammed in the bottom of the liner mold (with the wooden core removed); or it may be first rammed and then pressed in the mold to give better uniformity of structure. In the latter operation a pressure of 1000 psi has been used with good results. The remarks above concerning liner compositions, ramming and drying procedure, apply as well to the cover.

Liner and Cover - Firing Procedure

It has been found to be good practice in firing to bed the liner (standing upright) and the cover in coarse (4-8 mesh) zirconia or alumina. (Alumina (90% pure) and zirconia do not react below approximately 1750°C).*

Firing is conducted in an oxidizing atmosphere. A fairly slow rate in heating is desirable, especially in the interval from 1000° to 1150°C, the range in which monoclinic zirconia inverts to tetragonal with considerable shrinkage. The firing schedule can be varied to suit the kiln and conditions but the following may serve as an example:

* C. E. Curtis, D. Laurie, Investigation of Various Properties of Zircon and of Stabilized Zirconia at Elevated Temperatures, J. Amer. Ceram. Soc. 33 (6) 198-207 (1950).

Room temperature to 1000°C	4 hours
1000 to 1150°C	4 "
1150 to 1500°C	4 "
Held at 1500°C	2 "

Cooling at natural rate with the kiln well closed; cooling should occupy 8 hours at least.

A firing temperature of 1500°C is given because it is convenient for laboratories which possess furnaces heated by silicon carbide resistors. A liner fired at 1700 or 1800°C will have greater strength but this will not usually prolong its life appreciably. The produce fired at 1500°C is rather friable and must be handled carefully. This "punkiness" has some advantage, however, in increasing the ability of the liner to endure the stresses caused by rapid temperature changes. It should be noted that otherwise this lining is subjected to very little stress in service. Liners produced as described have been known to remain usable after two years of service during which they were heated to 1800°C on an average of once a week.

The covers withstand only six or seven firings to 1800°C since they are unsupported at the center. It is well to keep a supply on hand.

(Cracked covers can be broken up and used as grog in making more ramming mix according to the procedure described on page 4).

Fired Liner Preparation

Holes for the gas outlets, etc., can best be drilled at this time. Since the liner is usually rather friable, a chisel-shaped tool one inch wide is all that is needed as a drill. The openings in the wall of the liner for the outlet tubes and burner port are designed to permit the

zircon tubes to extend only about half-way through the lining. (Figures 1, 2 and 3). Beyond that point the openings should be tapered toward the inner surface of the liner as shown in Figures 1, 2 and 3; in the case of the outlet ports the opening in the inner surface of the wall is three quarters of an inch in diameter and for the burner port it is 1/2 inch. The temperature sight tube opening goes straight through into the furnace without a taper. Positions of the various openings are shown in the figures cited.

The size and position of the temperature observation port will depend upon the size and type of sight tube available. If this tube is open at both ends the position and slope of opening in the liner will be such as to give a good view of the ware. A closed end tube is preferable when it is desirable to eliminate flame effects in reading temperatures with an optical pyrometer. In this case the closed end should extend into the furnace and as close as possible to the material that is being fired.

Tube Composition

Inlet and outlet tubes and the open-end temperature observation tube are protected from the direct heat of the furnace in that they do not extend through the liner. Since they probably will seldom be heated above 1500°C (see Figures 1, 2 and 3), tubes of alumina or zircon have been found suitable for the purpose.

For the temperature reading tube, in the case of a closed-end tube which extends inside the furnace, stabilized zirconia is best. The tube is unsupported in this case and breaks rather readily, hence it must be replaced frequently; further, since zirconia is a good heat insulator, the temperature inside the tube may lag considerably behind that inside the

furnace unless a thin-walled tube is selected or a slow rate of heating is followed. Fortunately, an open-end tube serves for temperature reading very well provided the flame inside the furnace is not highly colored, particularly by carbon from the acetylene.

Tube Preparation

Commercial alumina, zirconia or zircon tubes* can usually be cut to size with a silicon carbide wheel. The inlet tube is cut to meet the curvature of the liner at an angle that will produce a swirling motion to the burning gases as mentioned above. The gas outlet tubes are cut squarely off since they enter perpendicular to a tangent. The temperature observation tube, if open at both ends, can be cut squarely also. (Figures 2 and 3).

Furnace Housing

A 16-gauge sheet steel shell 10 to 13 inches high and 10 to 11 inches i.d. is needed to house the furnace. (A paint pail of suitable size has been found convenient for the purpose). Openings in the metal must be cut to match the various tube holes in the liner.

Assembling the Furnace

The bottom of the furnace is built up with one or two layers of high temperature insulating bricks cut to fit the shell. On these the zirconia furnace liner is set and centered. The inlet and outlet tubes and the temperature sight tube are set in place and sealed into the outer steel shell with a refractory cement to prevent escape of the insulating grain. For cementing most commercial refractory cements are suitable** since resistance only to about red heat temperatures is required.

* Usually available from producers of zirconia (page 2).

** Cements of this type are obtainable from a number of sources; say, for instance, the list of makers of refractory cements in current issues of the Bulletin of the American Ceramic Society and trade journals.

The space between shell and liner is then filled to the top with an insulating refractory material, which may be coarse zirconia grain (unstabilized) or cheaper materials such as bubble alumina or fireclay grog. The bottom of the firing chamber is filled with the zirconia grain to a depth of one to two inches.

Containers

Materials to be fired at temperatures from 1400° to approximately 1600°C may be placed on or in alumina containers. For higher temperatures they may be bedded in the zirconia grain of the furnace bottom or fired in zirconia containers.

Burner Equipment

An ordinary oxyacetylene welding outfit is employed, special attention being given to the burner tip. For this a welding tip with an opening approximately 0.040 inches in diameter is convenient. The size and condition of the opening are especially important because of the tendency of the flame to backfire with the rather "slow" speed of gas flow employed. For this reason a cutting tip or multiple burner tip is unsuitable since a small opening is necessary to give a rate of gas flow that is faster than the rate of flame propagation. The flow may be slowed and backfiring result from roughened or scaled surfaces inside the tip also. Thus trouble has been encountered from a tip which was cleaned with wire brush sometimes provided for the purpose.

The tip must be water-cooled since it is "looking" directly into the hot furnace. For this purpose a 1/8 inch i.d. copper tube wrapped 6 to 7 times as closely as possible around the tip and anchored in place with silver solder (see photograph) does very well. The small overall diameter

of such a tip permits easy insertion into the inlet port. In soldering the tube to the tip care must be taken not to heat the tip any more than can be avoided because of the danger of scale formation as mentioned above.

Experience has shown that one burner is preferable to two or more in a furnace of the size in question. This is true because control of the heating is based on the size and appearance of the flame from the exit ports and with two burners in use it is possible to have one badly out of adjustment without the operator's knowledge. With one burner, control is simpler and although the heat distribution at the lower temperatures is apt to be uneven, at radiant temperatures it has been found to be surprisingly uniform.

Gas Supply

In order to reach and hold the furnace at temperatures of 1800°C for four hours a full tank of oxygen (approximately 2200 psi) and of acetylene (approximately 220 psi) are required. Exact operating pressures from the gauges will depend on the operator and the furnace and are learned mainly by experience.

Flame adjustment can be at the torch or at the gauges, depending on convenience.

OPERATION

Starting

The oxygen regulator gauge is maintained at about 35 psi and the acetylene gauge at approximately 25 psi.

With the furnace lid covering only about 3/4 of the furnace opening, the torch is lighted, adjusted to give a long flame and inserted in the inlet tube; the tip of the torch should not extend inside the inner surface of the liner. It may be necessary to adjust the torch position

in order that the flame may travel horizontally and strike the opposite wall at an angle; this is essential to give a swirling effect to the gases. The flame is then shortened as much as it is possible to do without backfiring (it may still be yellow and may burn partially outside the furnace). It is left at this setting for perhaps 10 or 15 minutes or until the furnace wall is white hot over a considerable area. If the wall is not well heated, backfiring may be encountered when the furnace is covered.

When it is safe to do so, the cover is slid into place and the flame is adjusted to give a slightly yellowish flame extending approximately an inch from the outlet ports. After further heating, the top of the heating chamber can be more or less sealed by heaping on more zirconia grain around the edges of the lid. (Figures 2 and 3). A layer of grain on the lid itself may be necessary to decrease heat losses in order to attain the higher temperatures.

With the furnace closed, the flame should be practically noiseless. Backfiring or a hissing sound indicate that the furnace wall is too cool for complete combustion or that the flame is not adjusted properly or that the tip is defective. If the latter is found to be the case a new tip is often the only cure. Control in either heating up or in holding a steady temperature will be found to be quite easy after short experience provided the tip is not defective.

The above directions are for general guidance only since furnaces differ widely in their characteristics. Experience will determine the best operational procedure. Probably the most important considerations are: Plenty of time for the furnace to warm up before the cover is completely in place, quiet combustion inside the furnace at all times and a small flame visible outside the outlet ports at all times when the furnace is closed.

A typical heating curve is given in Figure 4 which shows a rapid heating rate all the way to 1800°C. This rate is about maximum and can be slowed down, if desirable. If it is desirable to know the rate of heating below radiant temperatures a thermocouple is necessary; an optical pyrometer is used above that temperature.

Acknowledgments:

The building of this furnace and its operation for the most part has been in the hands of J. A. Griffin.

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TABLE 1

COMPOSITIONS FOR RAMMED ZIRCONIA LINERS

Composition No. 1

Commercial stab. zirconia ramming mix	100%
Water	6-8
Wood flour (dry volume)	20

Composition No. 2

Re-used zirconia Liner, 8 mesh (wt)	50%
Electrically fused lump zirconia, 8 mesh (wt)	50
Wood flour (dry volume)	20
Dextrine (wt)	4
Water	6-8

Composition No. 3

Re-used zirconia Liner, 8 mesh	33-1/3%
Electrically fused zirconia lump, 8 mesh	33-1/3
Stab. zirconia, 200 mesh or finer	33-1/3
Dextrine	4
Water	7-8

Composition No. 4

Electrically fused zirconia lump, 8 mesh	66-2/3%
Stab. zirconia, -200 mesh or finer	33-1/3
Dextrine	4
Water	7-8

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Oxyacetylene Furnace

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FIGURE 1. PLAN OF FURNACE - ONE HALF SIZE

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FIGURE 2. SECTION ON ABC

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FIGURE 3. SECTION ON EDF

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FIGURE 4. TYPICAL FIRING AT 1800°C FOR 3 HOURS

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