Dec. 8, 1964 H. R. PALMER 3,160,693

FURNACE FOR DETERMINING MELTING POINTS OF METALS

Filed April 26, 1962

FIG. 1

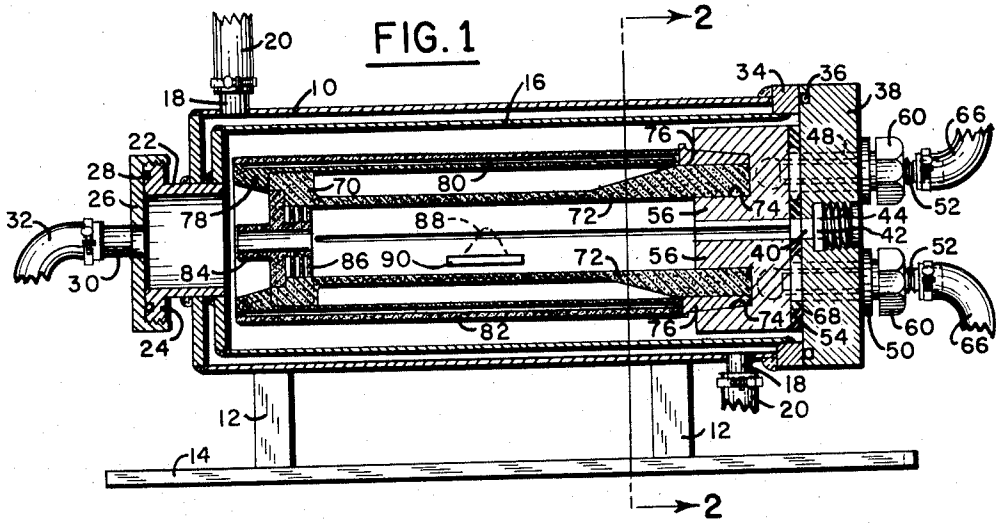


FIG. 3

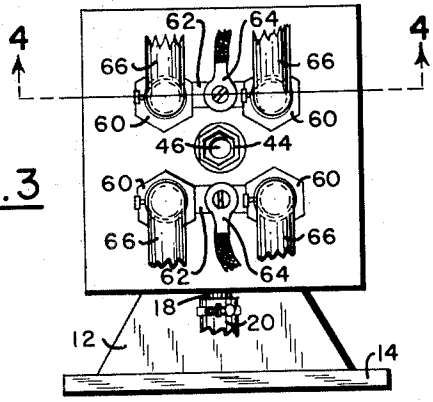


FIG. 4

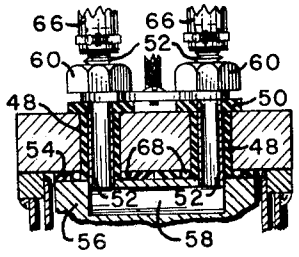
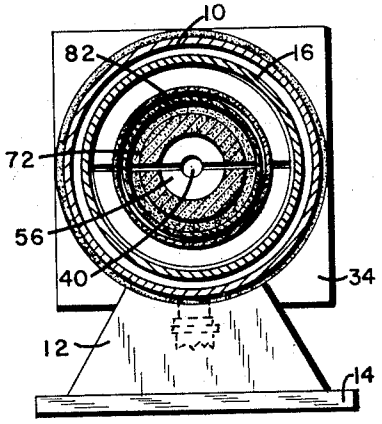


FIG. 2



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3,160,693
Patented Dec. 8, 1964

3,160,693

FURNACE FOR DETERMINING MELTING POINTS OF METALS

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Filed Apr. 26, 1962, Ser. No. 190,312
4 Claims. (Cl. 13—22)

This invention relates to a furnace and more particularly to a furnace adapted for use in the determination of the melting points of metals and other materials.

A furnace for use in determining melting points of metals must be capable of being operated at high temperatures in order that the melting points of refractory metals, for example, may be properly determined. Such a furnace must be conveniently charged with a sample whose melting point is to be determined and must also be operable under high vacuum since many metals and materials decompose and oxidize in an air atmosphere at a high temperature.

It is therefore a principal object of this invention to provide an improved furnace useful in the determination of melting points of metals and other materials. Another object of this invention is to provide a melting point determination furnace which can be operated under high vacuum. Another object of this invention is to provide a melting point determination furnace which may conveniently be charged with a sample whose melting point is to be determined and yet may readily be evacuated to provide for determination of melting points under high vacuum. These and other objects of this invention will be apparent from the following description thereof and from the annexed drawings in which:

FIG. 1 shows a longitudinal section taken centrally of a furnace embodying features of this invention.

FIG. 2 shows a transverse section of the furnace of FIG. 1 taken along the line 2—2.

FIG. 3 shows an end view of the furnace of FIG. 1.

FIG. 4 shows a more detailed view of the end plate of the furnace of FIG. 1, the section being taken along the line 4—4 of FIG. 3.

Referring now more particularly to FIGS. 1, 2 and 3, the furnace comprises an outer tubular shell 10 mounted in horizontal position on support members 12 which are in turn attached to base plate 14. For high temperature operation, shell 10 is preferably cooled by provision of inner shell 16 which is diposed within outer shell 10 and spaced apart therefrom to form a double wall and to provide a jacket through which is circulated a cooling fluid, this being introduced and exhausted from this cooling jacket through pipe connectors 18 to which are attached suitable means for transferring a cooling fluid such as water, as for example, hoses 20.

At one end, identified by reference to FIG. 1 as the left hand end, outer shell 10 and inner shell 16 are joined as by welding to tube section 22 whose outer end is provided with flange 24. A cap 26 is removably attached to tube section 22 as by threads around flange 24, a suitable gasket being arranged between flange 24 and cap 26, for example, O-ring 28. Cap 26 thus seals this end of shell 10. The face of cap 26 is perforated and communicates with short pipe 30 to which is attached a suitable hose or other pipe connection 32, to provide means

for connection of interior of the furnace to a vacuum pump (not shown).

At their other ends, identified by reference to FIG. 1 as the right hand end, outer shell 10 and inner shell 16 are joined as by welding to annular flange 34. Engaged in abutting relationship with flange 34 and sealed thereto as by O-ring 36 is end plate 38 which thus seals this end of the furnace. An aperture 40 is provided centrally in end plate 38 which is covered by a transparent window, preferably of heat resistant glass 42, which is maintained in tightly sealed relationship with the shoulder of aperture 40 by nut 44 which itself is provided with a central bore 46.

Passing through end plate 38 and insulated therefrom by bushings 48 and washers 50 are cooling pipes 52, preferably made of brass or copper. Inside the furnace and insulated from end plate 38 by gaskets 54 are contact blocks 56, preferably of copper, which as will be seen, are of generally semi-circular configuration and are spaced apart by a horizontal split. Contact blocks 54 are each provided with an inner cooling fluid channel 58 and pipe 52 are suitably fixedly attached as by soldering or brazing to blocks 54 in pairs as shown, one of pipes 52 communicating with each end of cooling fluid channels 58.

An outer portion of each of pipes 52 is threaded to receive nuts 60 under horizontal pairs of which are placed contact bars 62 to which are attached the terminals 64 of power leads connected to a suitable source of electric power (not shown).

The outer ends of pipes 52 are smooth, that is not threaded, so that they may snugly fit into clamped on hoses 66 which can supply circulating cooling fluid, such as water.

It will be seen that by adequately tightening nuts 60 on pipes 52, contact blocks 52 can be firmly maintained in position insulated from end plate 38 by gaskets 54, and preferably further sealed by provision of O-rings 68 around each of the passages of pipes 52 and their bushings 48 through end plate 38.

The heating element of the furnace of this invention is preferably fabricated of graphite in the form of an end ring 70 having a bifurcated skirt 72, this skirt being split horizontally and centrally to form a top segment and a bottom segment thereof, the split being aligned with the space between contact elements 56. The free ends of the skirt segments are somewhat thickened, for strength and current carrying capacity, and are inserted into semi-circular grooves 74 in contact blocks 56. Wedges 76 are jammed also into grooves 74 to hold the skirt ends firmly locked in place. The resistor element is thereby mounted coaxially in shell 10 by attachment of the ends of the segments of skirt 70 by means of contact blocks 56 to end plate 38.

A radiation shield, preferably also of graphite, having an end ring as at 78 and a tubular skirt 80 is placed over resistance element end ring 70 and the enlarged skirt ends. Outer tubular graphite radiation shield 82 also provides an additional heat loss barrier.

The open end of the resistance element at ring 70 is provided with removable graphite plug 84 which is formed with a central bore and heat shielding fins as at 86.

In operation of the furnace for determining the melting point of a metal, for example, a sample of such metal as

at 88 is placed in a suitable open top container or boat 90. The furnace is then opened by removal of cap 26, plug 84 is removed and the boat 90 with sample 88 is placed in the furnace supported on the bottom segment of skirt 72 of resistor element 70. Since the inner surface of skirt 72 is rounded, contact will be made with the edges of boat 90 which will thereby be elevated somewhat, and the sample 88 will be aligned with the opening between the contact blocks 56 and also the window 40 in end plate 38. Plug 84 is replaced and the furnace is then closed by replacing cap 26 and screwing in place tightly to make an effective seal, and pumping means connected to hose 32 and started to evacuate the furnace interior. Water flow is arranged through hoses 20 and through hoses 66 to circulate through the shell cooling jacket and the contact block cooling channels. Electric power is then turned on to supply current through leads 64 to the resistor element 70 and its skirt 72 which begin to heat up and in turn heat boat 90 and sample 88. Direct current or single phase alternating current may be employed, as will be apparent.

When the pressure in the furnace has been reduced to the required level (this may, of course, be determined by a vacuum gauge connected, for example, into hose 32 in conventional manner) the electric power input is raised so that the temperature of the resistor element 70 and the boat 90 and sample 88 is gradually raised. Meanwhile, the sample 88 is carefully watched by means of an optical pyrometer through window 40 in end plate 38. When the sample 88 is seen to show signs of melting as by slumping in boat 90, its temperature determined by the optical pyrometer is noted and taken as its melting point.

The furnace of this invention, arranged in horizontal position and penable at one end is ideally suited for charging and discharging sample boats. These may be simply pushed in from the open end onto a central portion of the heating element where they will be exposed to the desired heating effect. The horizontally aligned viewing window provides convenient observation of the sample during heating for melting point determination.

The resistor type heating element provides efficient and rapid heating and its use in the simple and clean design shown permits quick pump-down to required low operating pressure without degassing problems associated with packings or other subdivided materials. The resistor element is supported at one end and the graphite heat shields preserve a satisfactory temperature gradient to the shell. Experimental operation of such a furnace has shown that comparatively long life can be expected from the resistor element and it does not tend to sag or deform in spite of its one-end support, and high temperature operation.

The design of the heating element is an important feature of the furnace. Use of direct current or single phase alternating current is convenient, and the end ring and bifurcated skirt provide an efficient organization for resistance heating. The division of the skirt horizontally provides a single and continuous bottom segment which also serves as a hearth on which the sample or sample container may be placed for test heating. The hearth extends laterally almost up to the center of each side of heating element and this is important since a flat bottom boat or container will rest on portions of the hearth part way up each side due to the curvature of the hearth bottom. It will be appreciated that a smooth continuous hearth surface is advantageous for charging, placing and discharging sample containers, and also that placement of such a container over any skirt element or segment split could cause short circuiting of the current flow and disruption of the electrical system.

The shell and contact plate end of the furnace are efficiently cooled without affecting the high temperature capability of the heating zone inside the resistor element. The contact block and end plate assembly provides a compact organization with cooling pipes 52 actually fulfilling a triple function; that is, they tie the contact blocks and end plate together, they act as pipes for circulating cooling fluid and they also act as means to conduct electric current through the end plate and the contact blocks to the resistor element skirt segments inside the furnace.

The furnace of this invention is useful for determination of the melting point of many materials, and particularly of refractory and reactive metals such as titanium and zirconium and their alloys.

I claim:

1. A furnace for use in determining the melting point of a material comprising:
   (*a*) a tubular horizontal shell;
   (*b*) a removable cap sealing one end of said shell;
   (*c*) a plate sealing the other end of said shell;
   (*d*) a tubular resistor element of graphite having open ends and a skirt split horizontally and centrally to form a top segment and a bottom segment thereof, said resistor element being mounted inside said shell coaxially therewith only by attachment of said skirt segments to said plate;
   (*e*) means for connecting electric current to said resistor element skirt segments through said plate;
   (*f*) means for connecting a vacuum pump to communicate with the interior of said shell;
   (*g*) a window in said plate aligned with the interior of said resistor element; and,
   (*h*) a container supported on the bottom segment of said resistor element so that a sample contained therein can be viewed through said window in said plate, said container being removable through the end of said shell sealed by said cap.

2. A furnace for use in determining the melting point of a material comprising:
   (*a*) a tubular horizontal shell;
   (*b*) a removable cap sealing one end of said shell;
   (*c*) a plate sealing the other end of said shell;
   (*d*) a tubular resistor element of graphite having open ends and a skirt split horizontally and centrally to form a top segment and a bottom segment thereof, said resistor element being mounted inside said shell coaxially therewith only by attachment of said skirt segments to contact blocks which are attached to said plate but insulated therefrom;
   (*e*) means for connecting electric current to said resistor element skirt segments through said plate;
   (*f*) means for connecting a vacuum pump to communicate with the interior of said shell;
   (*g*) a window in said plate aligned with the interior of said resistor element; and,
   (*h*) a container supported on the bottom segment of said resistor element so that a sample contained therein can be viewed through said window in said plate, said container being removable through the end of said shell sealed by said cap.

3. A furnace for use in determining the melting point of a material comprising:
   (*a*) a tubular horizontal shell;
   (*b*) a removable cap sealing one end of said shell;
   (*c*) a plate sealing the other end of said shell;
   (*d*) a tubular resistor element of graphite having open ends and a skirt split horizontally and centrally to form a top segment and a bottom segment thereof, said resistor element being mounted inside said shell coaxially therewith only by attachment of said skirt segments to a pair of contact blocks each having a cooling fluid passage communicating with pipes which are fixedly attached to said blocks and which pass through said plate, said pipes providing means for attaching said blocks to said plate, the said contact blocks and pipes attached thereto being insulated from each other and from said plate;
   (*e*) means for connecting electric current to said pipes to conduct current to said resistor element skirt segments through said contact blocks;

(*f*) means for connecting a vacuum pump to communicate with the interior of said shell;

(*g*) a window in said plate aligned with the interior of said resistor element; and,

(*h*) a container supported on the bottom segment of said resistor element so that a sample contained therein can be viewed through said window in said plate, said container being removable through the end of said shell sealed by said cap.

4. A furnace for use in determining the melting point of a material comprising:

(*a*) a cooled tubular double wall horizontal shell;

(*b*) a removable cap sealing one end of said shell;

(*c*) a plate sealing the other end of said shell;

(*d*) a tubular resistor element of graphite having open ends and a skirt split horizontally and centrally to form a top segment and a bottom segment thereof, said resistor element being mounted inside said shell coaxially therewith only by attachment of said skirt segments to said plate;

(*e*) means for connecting electric current to said resistor element skirt segments through said plate;

(*f*) means for connecting a vacuum pump to communicate with the interior of said furnace through said removable cap;

(*g*) a window in said plate aligned with the interior of said resistor element; and,

(*h*) a container supported on the bottom segment of said resistor element so that a sample contained therein can be viewed through said window in said plate, said container being removable through the end of said shell sealed by said cap.

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