United States Patent

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[54] **TRANSPARENT FURNACE**
15 Claims, 8 Drawing Figs.

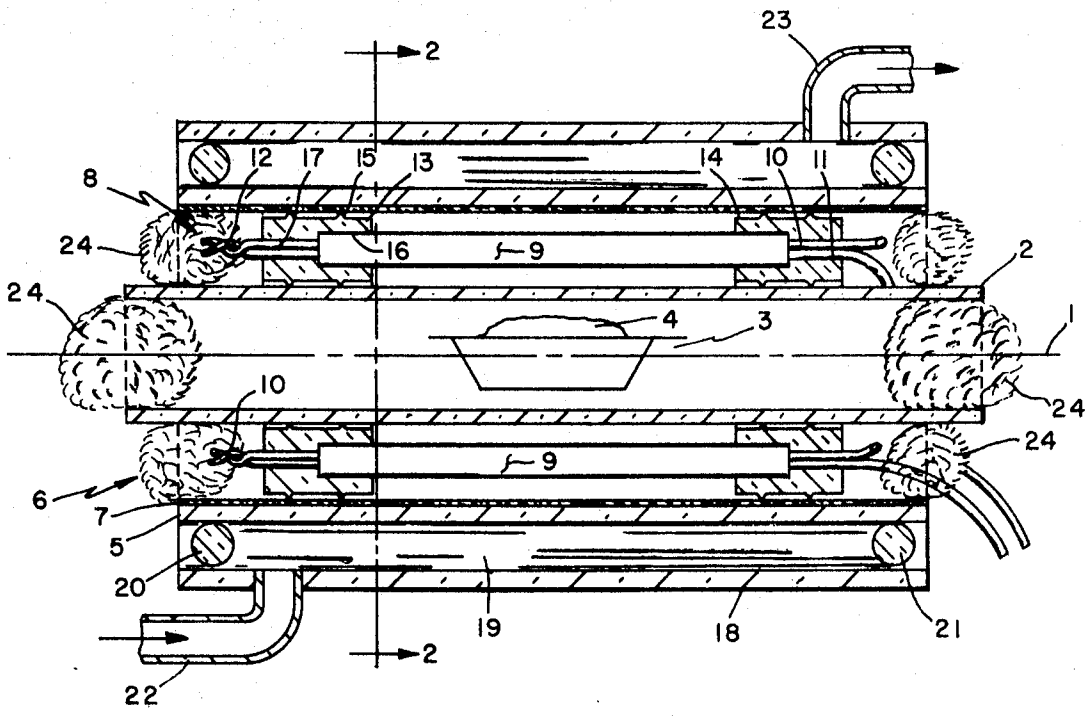
[52] **U.S. Cl.** **219/411,** 99/447, 126/200, 219/354, 219/405, 219/531, 219/553
[51] **Int. Cl.** **F27d 11/02**
[50] **Field of Search** 219/405, 410–411, 353–354, 352, 553, 543, 531, 388; 52/171; 99/447; 126/200

[56] **References Cited**
UNITED STATES PATENTS

2,864,932	12/1958	Forrer	219/411 X
2,954,826	10/1960	Sievers	219/553 X
3,192,575	7/1965	Rosenau, Jr. et al.	52/171
3,249,741	5/1966	Mills	219/388
3,304,406	2/1967	King	219/411
3,307,017	2/1967	Horstmann	219/553
3,363,090	1/1968	Langley	219/353 X
3,445,662	5/1969	Langley	219/354 X
3,541,293	11/1970	MacDonald et al.	219/390

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ABSTRACT: A furnace which heats by infrared radiation includes a wall portion of material transparent to visible radiation having a layer of selected material on the inside thereof, which reflects substantially all infrared radiation and yet transmits sufficient visible radiation so that the inside of the furnace can be viewed from the outside.



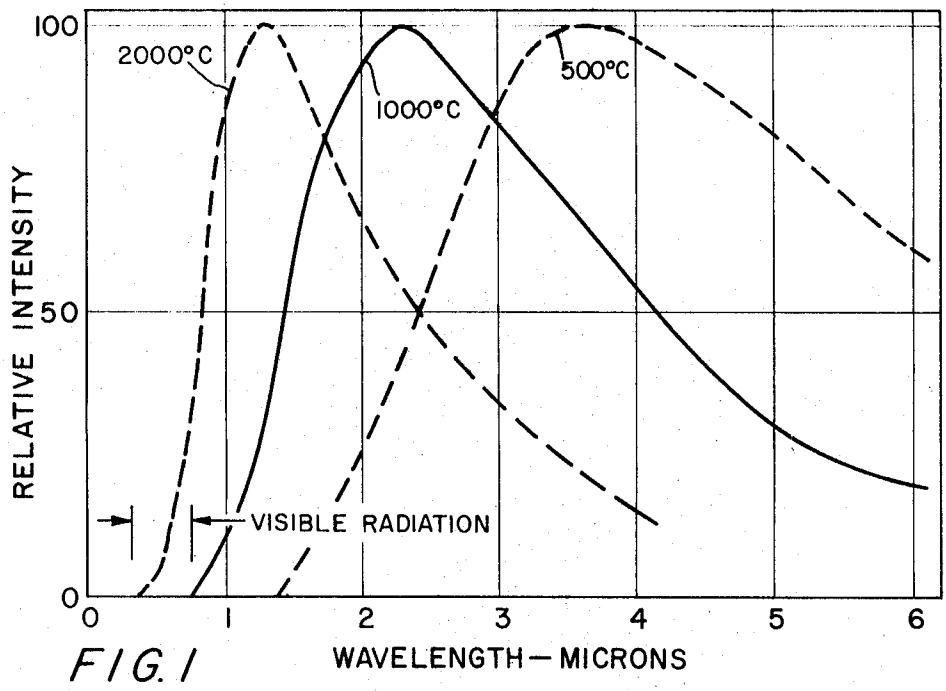


FIG. 1

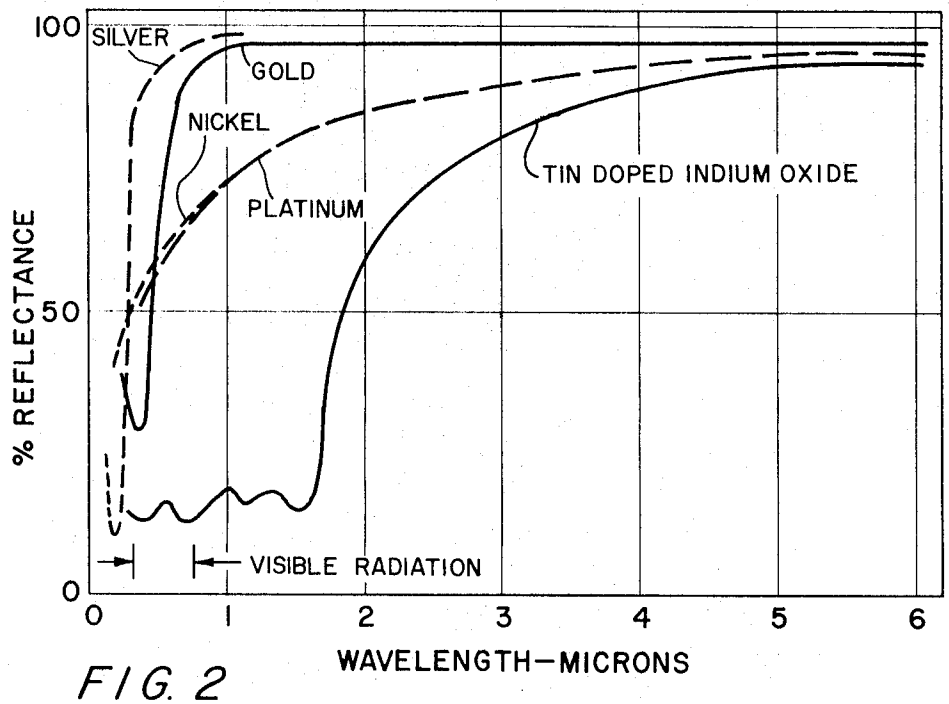


FIG. 2

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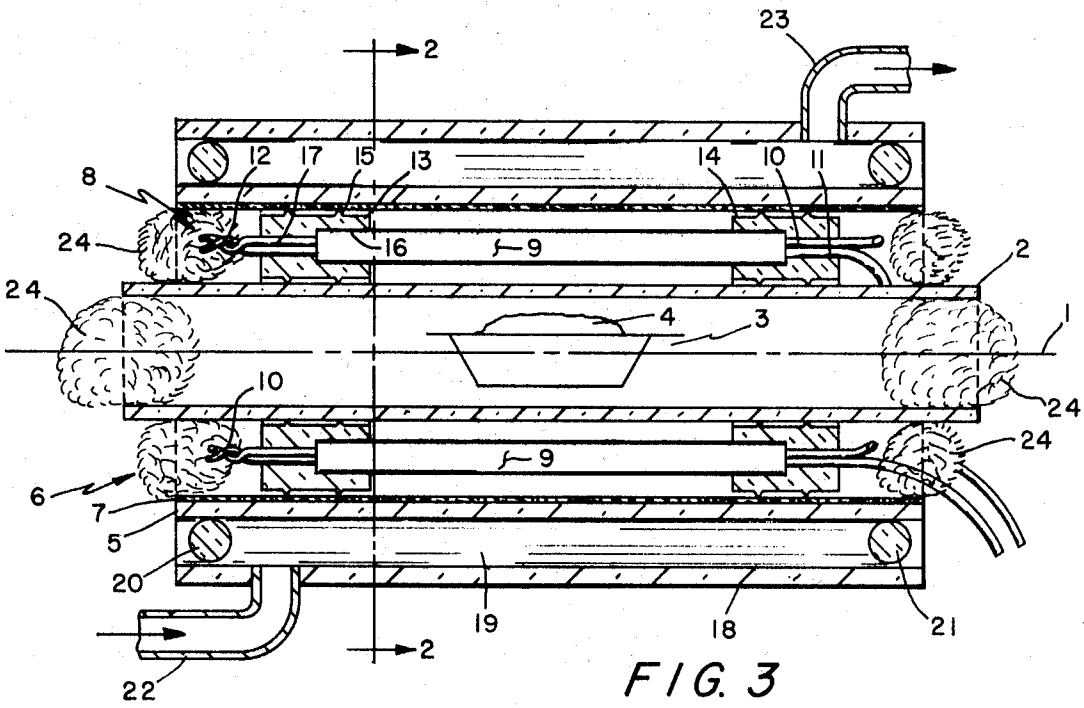


FIG. 3

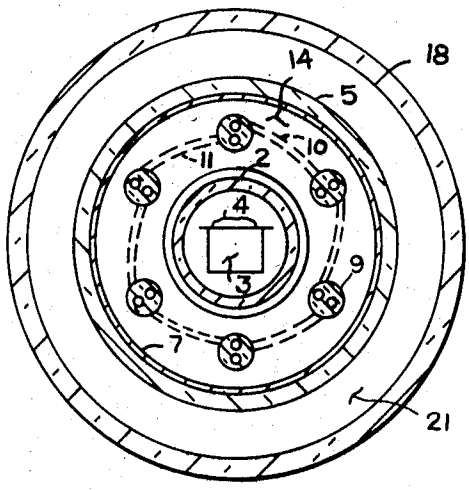


FIG. 4

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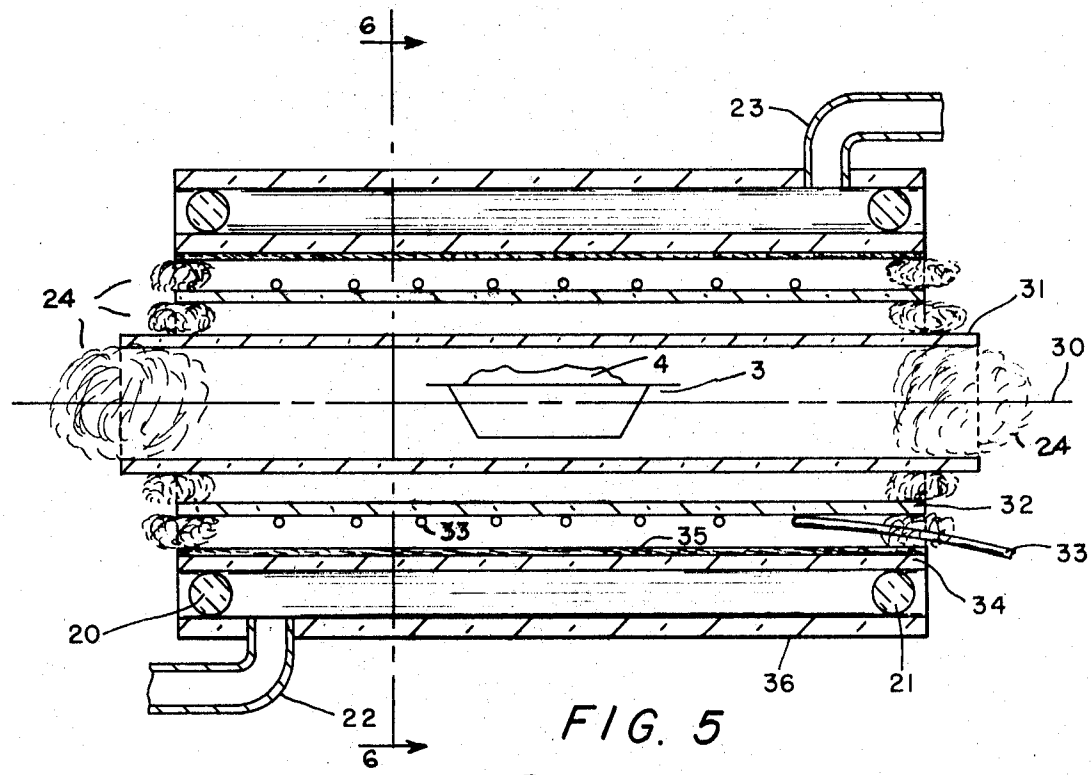


FIG. 5

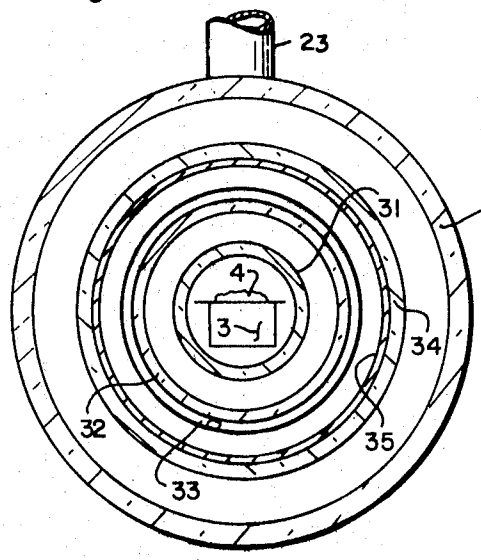


FIG. 6

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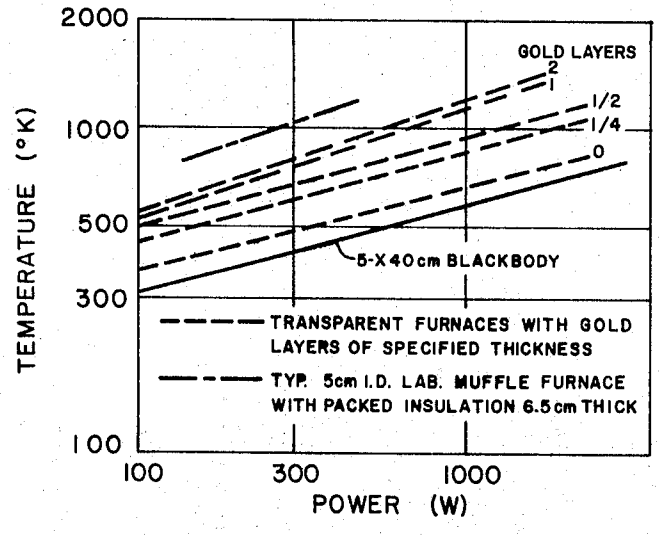


FIG. 7

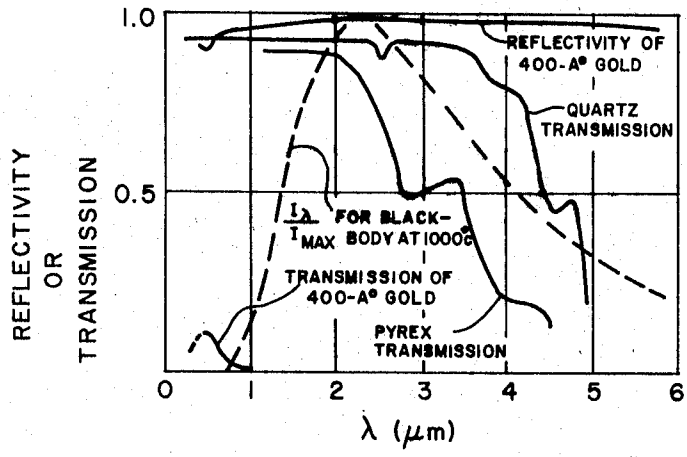


FIG. 8

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TRANSPARENT FURNACE

This invention relates to furnaces and ovens heated by infrared radiation, and more particularly to a muffle furnace.

A conventional muffle furnace usually consists of an enclosure containing the body to be heated all within another enclosure. Heat generated in the space between the two enclosures is conducted to or radiates to the body to be heated and so the body is separated from the source of heat by the first enclosure. Conventional muffle furnaces, as well as other types of furnaces, use asbestos or similar materials for insulation around the outside of the furnace to reduce heat losses. In high-temperature muffle furnaces operating above 2,000° C., radiation heat shields are often employed instead of the packed insulation. These shields surround the hot zone of the furnace with a very thin sheet of metal. A multitude of such shields are employed and it can be shown thermodynamically that under ideal conditions, each heat shield comes to an intermediate temperature such that it reduces radiation loss by a factor of two. Thus n such shields reduce the radiation loss by $\frac{1}{2}^n$.

It is one object of the present invention to provide a furnace which requires neither conventional insulation nor a multitude of conventional heat shields.

Heretofore, with muffle furnaces and with other types of high-temperature furnaces employing heat shields, it has not been possible to watch the progress of an experiment within the furnace except through a special window provided for that purpose. However, the window is unsatisfactory in furnaces used for vapor crystal growth, because the furnace window allows infrared radiation to escape and becomes a cold spot in the furnace where the vapor can condense, obscuring the view.

The same problem arises with commercial self-cleaning ovens. These ovens heat to around 400° C. to clean, and heretofore, have not had windows, because an ordinary Pyrex window would transmit much infrared and become a cold spot on the inside of the oven on which vapors would condense and eventually obscure the view.

It is another object of the present invention to provide in a furnace or oven a wall portion which is substantially transparent, so that the progress of an experiment can be observed, but without incurring the aforementioned vapor condensation which obscures observation.

It is another object of the present invention to provide a muffle furnace of relatively light weight, which heats to temperature rapidly and also cools rapidly.

It is another object of the present invention to provide a furnace for operation in the range of 1,000° C. and above, which heats to temperature rapidly, and is at least partly transparent so that the progress of an experiment within the furnace can be viewed from the outside.

It is another object of the present invention to provide a furnace which heats principally by infrared radiation and is brought to temperatures in excess of 1,000° C. in reasonably short periods, with efficiency comparable to that of a conventional infrared heated muffle furnace encased by conventional packed insulation.

A transparent furnace and a transparent oven window are applications of the present invention. The invention includes a wall portion which is transparent, coated with a material, which (a) reflects as much of the infrared as possible, (b) transmits as much of the visible radiation as is required for good visibility of the contents of the furnace, and (c) is inert at the temperature of operation.

Other objects and features of the invention will be apparent in view of the specific description, taken in conjunction with the Figures, in which:

FIG. 1 shows curves of black body radiation intensity vs. wavelength;

FIG. 2 shows curves for a number of metals and a semiconductor of reflectance vs. wavelength;

FIG. 3 is a cross section view taken through the axis of a muffle furnace incorporating features of the present invention;

FIG. 4 is a sectional view of the furnace, taken transverse to the axis;

FIG. 5 is a sectional view taken through the axis of another embodiment of the muffle furnace, incorporating features of the invention;

FIG. 6 is a sectional view of the furnace shown in FIG. 5, taken transverse to the axis;

FIG. 7 shows plots of relative transmission and reflectivity, versus wavelength for a film of gold, quartz and Pyrex; and

FIG. 8 is a family of curves of temperature versus power for a muffle furnace, in accordance with the present invention, with different thicknesses of the gold layer for comparison with a conventional muffle furnace with packed insulation.

The relative intensity of radiation as functions of wavelength for a black body (which simulates the radiation from materials in a furnace) at 500° C., 1,000° C. and 2,000° C. are shown in FIG. 1. The eye is only sensitive to radiation in the range 0.4–0.7 microns, and at these temperature 10^{-6}, 0.01 and 2 percent, respectively, of the total radiation lies in the visible.

The reflectance of a number of materials as a function of wavelength is shown in FIG. 2. The transmission of thin films is correspondingly high where the reflectance is low and vice versa. A thin film of material which exhibits low reflectance of visible radiation will correspondingly exhibit high transmittance of visible radiation. The thinner the film, the higher will be the transmittance. The thickness of the film, however, does not affect reflectance and so the film thickness of the selected material can be tailored to transmit visible radiation without substantially reducing its reflectance of infrared radiation.

At sufficiently long wavelengths, metals have a high reflectance when the wavelength exceeds the plasma wavelength, λ_p
where

$$\lambda_p = Ne^2/m^*\epsilon$$

Here N is the number of free electrons/cm.^{3}, e the charge on the electron m^* the effective of the mass of the electron and ϵ is the dielectric constant. However, at shorter wavelengths than λ_p, loss mechanisms come into play, which inhibit reflection and increase transmission.

Several materials are shown in FIG. 2. The curve for nickel is typical of most metals, having a moderately high reflectance at all wavelengths with little distinction between visible and infrared wavelengths. The curve shown for gold (copper is very similar) is ideal for a furnace operating at 1,000° C., as it can be seen that the reflectance is very high where most of the energy lies, and falls off rapidly in the visible, giving rise to the "gold" color. Silver even surpasses gold in its infrared reflectance, but is not as good a transmitter in the visible. However, at 2,000° C. higher reflectance in the visible is required, because there would be so much visible light available that it would be advantageous to reflect most of it for the comfort of the observer. This may be called the "sunglasses" effect.

Semiconductor materials as well as metals are useful infrared reflectors. Tin-doped indium oxide is a good example and its reflectance is shown in FIG. 2. In this case, the plasma wavelength is longer than that of most metals so that the infrared reflection is not as high as that of gold, but the transmission in the visible is much better. In constructing a furnace to operate at 500° C., this type of material should have adequate reflectance for the longer wavelengths and at the same time the high transmission in the visible is needed because there is no visible light generated by the heat of the furnace and one must bring light in from the outside.

Specific embodiments of the present invention described hereinbelow employ gold as the material for reflecting infrared while transmitting visible radiation. A thin layer of gold a few hundred angstroms thick will reflect substantially all incident infrared and will, at the same time, transmit sufficient

visible radiation so that the visible radiation can be observed with the naked eye. The muffle-type furnaces described herein consist of a clear quartz muffle in which the body to be heated is placed. Outside of the quartz muffle is a transparent (Pyrex) enclosure coated on the inside with a thin film of gold, a few hundred angstroms thick. A source of infrared is located between the quartz muffle and the Pyrex enclosure. The Pyrex enclosure may be cooled with a suitable transparent cooling fluid and no packed insulation is required. The cooling apparatus around the Pyrex enclosure is preferably transparent to visible radiation, so that an observer can view the body inside the muffle with the naked eye, there being sufficient visible radiation transmitted through the thin layer of gold so that the body can be observed. The gold, however, reflects substantially all infrared radiation incident upon it and so very little of the infrared radiation escapes past the gold layer. Heating is rapid and efficient. The coolant fluid (which may be gas or liquid) flows directly against the gold layer and so is most effective to hold down the temperature of both the gold and the Pyrex enclosure.

It is important in a furnace heated by radiation to maintain black body conditions within the furnace, because then it can be ascertained with reasonable assurance that the temperature throughout is uniform and this temperature can be determined by optical pyrometer readings. At the same time, it is desirable to watch the progress of an experiment performed in the furnace when a material is heated. This is especially true in vapor crystal growth, where large crystals are grown in the vapor phase which requires a rather long period of time. It would be of considerable advantage to be able to monitor the process of crystal growth in order to modify the growth conditions or terminate an experiment in case of failure. Hence, to be able to watch the progress of the crystal growth would be most advantageous. Accordingly, the ideal furnace for this would be a transparent furnace, in which black body conditions are maintained within the furnace and which can be heated to temperature rapidly and cooled rapidly. With such a furnace, the growth of crystals can be observed and an experiment can be terminated quickly and then resumed again quickly, as desired by the operator. Embodiments of the present invention provide just such a furnace.

Specific embodiments of the invention shown in FIGS. **3** to **6** reveal a muffle-type furnace. The muffle furnace is generally a furnace in which the material to be heated is enclosed within a chamber and outside of this chamber a heating element is provided. The heating element is enclosed and insulated from the surroundings to prevent heat loss to the outside. In operation, heat from the element radiates through or is conducted through the chamber enclosing the body to be heated. Heretofore, laboratory muffle furnaces of this sort have had a packed insulation surrounding the heating element to prevent a window in the furnace so that crystal growth within could be observed. Such attempts have not been successful, particularly with regard to vapor crystal growth, because the window allowed infrared radiation to escape and became a cold spot on which the vapor would condense, obscuring the view. This disadvantage is avoided in muffle furnaces incorporating features of the present invention.

Turning first to FIG. **3**, there is shown a muffle furnace which can be used in the horizontal or vertical position. Many of the parts of the furnace are Figures of revolution about the axis **1**. The innermost part is a quartz muffle **2**, which is a cylinder of clear quartz. Within the quartz muffle **2** is placed the item to be heated and which may consist of the crucible **3**, containing a material **4**.

Enclosing the quartz muffle **2** is a Pyrex tube **5** concentric with the tube **2** and of larger diameter, defining an annular space **6** in between. The inside surface of the Pyrex tube **5** is coated with a thin film of gold **7**, of thickness preferably greater than 100 angstroms and less than 1,000 angstroms. The thickness of the gold film is selected in view of the temperature that the furnace is to be operated at. The higher the operating temperature, the greater the thickness.

Heater elements **8** are located in the annular space between the muffle **2** and Pyrex tube **5**. These heating elements may consist of a number of rods **9**, of alumina each drilled lengthwise with two holes, through which lengths of Kanthal A–1 wire, such as **10** and **11** are inserted. The ends of the Kanthal wires extending from one end of each rod are connected together as at **12**, and the other end of each length of Kanthal wire is inserted through a hole in the next adjacent aluminum rod. In this manner, the lengths of wire are connected in series, each length being fed through two adjacent alumina rods.

The alumina rods are suspended in the annular chamber **6** by rings **13** and **14** located in the annular chamber. The rings are equipped with protuberances, such as **15**, on the inside and out, which contact the outside of the muffle **2** and the inside of the Pyrex tube **5**, and so suspend the rings in the annular space **6**. The contact area is a minimum to reduce conductive heat flow. Holes such as **16** are located in the rings **13** and **14**, into which the alumina rods **9** are inserted and, thus, the rods are suspended in the annular chamber **6**. From the holes **13**, a smaller hole such as **17** extends through the ring to accommodate passage of the ends of the Kanthal wires and these ends are joined by, for example, twisting as at **12**. The end rings **13** and **14** may be made of boron nitride.

The coolant jacket **18** encloses the Pyrex tube **5** and is concentric with the axis **1** and of larger diameter than the Pyrex tube, defining the annular coolant space **19** therebetween. "O"-ring seals **20** and **21** seal the ends of the coolant space and a cooling fluid, such as water, is conducted to the space via inlet and outlet tubes **22** and **23**. The jacket **18** is preferably transparent, or at least a portion of it is transparent, so that the crucible and heated material **4** within the quartz muffle **2** can be observed from outside by looking through the water jacket, Pyrex tube **5**, gold layer **7** and the quartz muffle **2**. A high-temperature insulating material **24**, such as glass wool, is stuffed into the ends of the quartz muffle **2**, after it is loaded with the crucible and some is also stuffed into the ends of the annular space **6** to prevent convective heat loss from the heater element and from within the quartz muffle **2**.

The muffle furnace shown in FIGS. **3** and **4** is capable of operation in either the vertical or horizontal position. The alumina rods **9** expand only very little over extremes of temperature and so they can fit loosely within the holes **13** in the end rings **13** and **14**, which permits expansion without causing the rings to move. The Kanthal wire expands more than the rods and so the ends of the Kanthal wires, which extend from the rods elongate when the furnace is used, but this causes no problem because the wires fit loosely within the holes in the rods.

Another embodiment of the invention, shown in FIGS. **5** and **6**, employs a helical heater wire, rather than the alumina rods loaded with Kanthal wire, as shown in FIGS. **3** and **4**. In FIG. **5**, the numerous parts of the muffle furnace shown in this embodiment are figures of revolution about the axis **30** and concentrix therewith. This includes a quartz muffle **32** in which the crucible is loaded, a second quartz muffle **32** concentrix with muffle **31** and of larger diameter, a heater wire which may be Kanthal A–1, wound in the form of a helix around the outside of the tube **32**, a Pyrex tube **34** enclosing that, and including a thin layer of gold **35** on the inside thereof, and a cooling jacket **35** enclosing the Pyrex tube. This furnace has a double muffle (quartz tubes **31** and **32**). The purpose of the quartz tube **32** being to provide a mandrel for the heater wire **35** and so it serves substantially only to support the heater wire. This furnace is preferably used only in the horizontal position, because the helical wire expands considerably as the furnace heats and so the leads **37** and **38**, which extend from opposite ends of the wire must be able to expand outward. Such expansion is easily accommodated in the horizontal position, but not on the vertical.

The thin gold layer in either of the ovens shown in FIGS. **3** or **5** is formed on the inside of the Pyrex tube by, for example, evaporating gold from a tungsten wire which is hung inside the

tube, along the axis of the tube. The wire is electroplated with sufficient gold to form the film on the inside of the Pyrex. A 200 A. film of gold is produced on the inside of the Pyrex with a tungsten wire plated with gold for 40 milliampere-minutes per foot of wire. In the process of forming the film, a vacuum pump is attached to the tube and the ends of the tube are sealed, as in vacuum deposition apparatus. First, a very thin layer of chromium is flashed on the inside of the tube. This is accomplished by, for example, heating the tube to about 300° C. and evaporating chromium from a tungsten wire within the tube, employing the vacuum deposition apparatus. The thin flash of chromium provides an even surface to use to which gold will adhere more readily than to bare Pyrex. The gold is evaporated from tungsten wire under controlled conditions by varying the magnitude and duration of current conducted by the wire.

The gold layer can also be formed on the inside of the Pyrex tube by coating the inside with a commercially available gold resinate solution, such as "liquid bright gold No. 6854" supplied by the Hanovia Liquid Gold Division of Englehart Industries, located in New Jersey. First, the Pyrex is scrubbed with hot alconox. One end of the tube is stoppered and a small of gold resinate solution is poured in. Then the tube is rolled to coat evenly the inside, the stopper is removed and the excess is drained off. Immediately thereafter the tube is fired by passing it through a furnace at about 600° C. at a rate of 1 centimeter per minute, while blowing air gently through the tube. Then the tube is annealed at 600° C. for several hours to form a very uniform, extremely adherent, layer of gold on the inside of the Pyrex tube. The thickness of the layer of gold formed in this manner can be controlled by diluting the gold resinate solution with a solvent to make the layer of gold thinner, or by laying down a layer upon a layer of gold to form a thicker layer.

For purposes of nomenclature herein, a one-layer thickness of gold is formed as just described, using the commercial gold resinate solution No. 6854, mentioned above, at full strength without any dilution. This full thickness layer measures about 400 angstroms thick. Similarly, a two-layer coating is formed by laying down two layers of the gold using the commercial gold resinate solution at full strength. A half-layer is laid down, using the commercial gold resinate solution diluted 50 percent and a quarter-layer is laid down using the solution diluted 75 percent. Thus, the thickness of the gold layer is easily controlled to produce gold layers of 800, 400, 200 and 100 angstroms for the designated 2, 1, one-half and one-quarter layers, respectively, formed in the manner described.

Plots of relative transmission and reflectivity versus wavelength are shown in FIG. 8 as an aid in understanding the operation of the gold layer furnaces. As can be seen, the reflectivity of a 400 angstrom thick layer of gold is very high in the infrared and begins to drop off rapidly in the visible region. The transmission of the quartz muffle is relatively high in the infrared and is even higher in the visible. This is desirable, as the infrared from the heating elements must be transmitted to the heated material through the quartz muffle. The transmission of infrared by the Pyrex is substantially lower than for quartz; however, this is no problem, because the gold layer is on the inside of the Pyrex and it reflects over 95 percent of the infrared and so very little infrared penetrates through the gold layer to the Pyrex. On the other hand, the Pyrex, like quartz, transmits visible quite readily. The plots in FIG. 8 illustrate why quartz is most suitable for the muffle and Pyrex is a suitable transparent material for carrying the layer of gold. Quite clearly, other materials besides Pyrex could be used, however, Pyrex is a suitable relatively low-cost material that performs satisfactorily. The muffle, on the other hand, is preferably quartz and there is no other material quite as good as quartz for this purpose. A suitable muffle of opaque refractory material could be substituted for the quartz muffle having a portion thereof equipped with a quartz window. However, this would require considerable fabrication. It is convenient to use commercial stock quartz tubing for the quartz muffles 2 and 31.

If no vapors are produced in the furnace, of if the vapors produced are not harmful to the heating element and the Pyrex tube, no muffle is needed and the furnace can consist of only the gold-coated Pyrex tube enclosing the heating element and the body heated.

Generally, furnaces of the type shown herein designed to operate at temperatures lower than 1,000° C. or a window in a commercial self-cleaning oven need less infrared reflectance and more visible transmittance and so a thinner gold layer is appropriate. Conversely, furnaces for higher temperature operation where visible radiation may be too intense for the naked eye need a thicker gold layer to cut down visible light transmittance.

The effectiveness of the gold layer as an infrared insulator is demonstrated by the plots in FIG. 7. A family of plots are shown of temperature versus power for muffle furnaces such as shown in FIG. 1 with gold films denoted 2, 1, one-half and one-quarter layers thick. As already described, this designation of layer thickness signifies about 800, 400, 200 and 100 angstroms, respectively. The plots show furnace temperatures vs. input power for the muffle furnace using Pyrex tubes coated with gold film of the thickness indicated. The additional plot is for a typical 5 centimeter inside diameter laboratory muffle furnace with conventional packed insulation 6-½ centimeters thick and is shown for comparison. Clearly, the muffle furnaces with a gold layer compare favorably in performance with the conventional muffle furnace. There is little increase in temperature obtained with a given power input when a one-layer gold film Pyrex tube is replaced by a two-layer gold film Pyrex, and it can be seen that the input power for each furnace increases very nearly as temperature to the fourth power.

The embodiments of the invention described herein are the best known current uses of the invention and are described by way of illustration. The scope of the invention is set forth in the appended claims, as it is desired to protect all uses of the invention apparent to those skilled in the art, in addition to those described herein.

What is claimed is:

1. A furnace for heating a body by infrared radiation comprising,
   - a first transparent cylinder containing the body,
   - a second transparent cylinder concentric with and enclosing the first cylinder and defining an annular space therebetween,
   - a layer of material on the inside of the second cylinder, the plasma wavelength of the material being less than the wavelength of the infrared radiation,
   - an electrically energized infrared radiator in the annular space, the radiating surfaces thereof being distributed throughout the annular space,
   - the thickness of the layer of material being such that it substantially reflects infrared and transmits visible radiation,
   - whereby the body is heated and is visible through the transparent cylinders between said radiating surfaces and
   - thermal insulating means closing the ends of said first transparent cylinder.

2. In a furnace or oven as in claim 1 and in which, the material is substantially inert to gases and vapors in the furnace or oven.

3. In a furnace or oven as in claim 1 and in which, the material is a metal.

4. In a furnace or oven as in claim 1 and in which, the material is a relatively heavily doped semiconductor.

5. In a furnace or oven as in claim 1 and in which, the metal is gold.

6. In a furnace as in claim 5 and in which, the thickness of the gold layer is dependent on the operating temperature of the furnace.

7. In a furnace as in claim 5 and in which, the thickness of the gold layer is less than 1,000 angstrom units.

8. In a furnace as in claim 5 and in which, the gold layer is between 100 and 1,000 angstrom units thick and the infrared radiation temperature is about 1,000° C.

9. In a furnace or oven as in claim 1 and in which, the metal is silver.

10. In a furnace or oven as in claim 1, and in which, the semiconductor is indium oxide.

11. In a furnace or oven as in claim 1 and in which, the semiconductor is indium oxide doped with tin.

12. A furnace as in claim 1 and in which, the source of infrared radiation is an electrically heated radiator.

13. A furnace as in claim 1 and in which, means are provided for cooling the second cylinder.

14. A furnace as in claim 13 and in which, the material is gold, the first mentioned transparent cylinder is quartz and the second is Pyrex.

15. A furnace as in claim 14 and further including, a transparent jacket enclosing the second cylinder and a transparent fluid coolant in the jacket.

* * * * *