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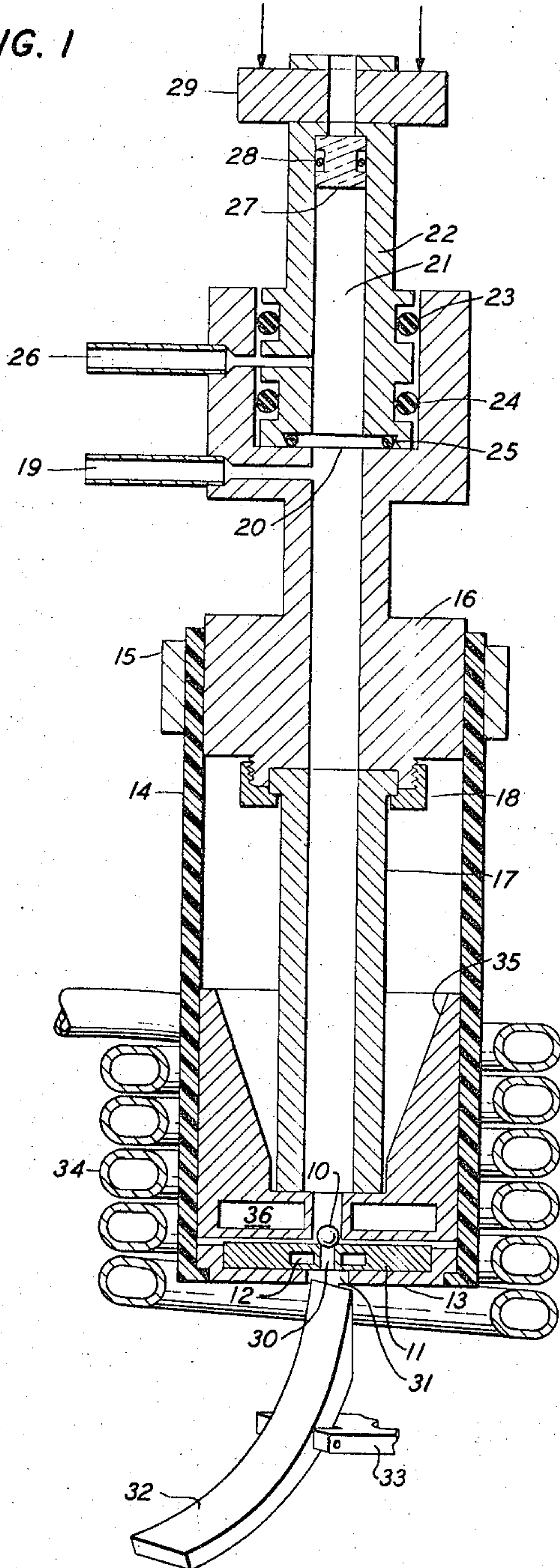
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MELTING APPARATUS

3,365,184

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2 Sheets-Sheet 1

FIG. 1



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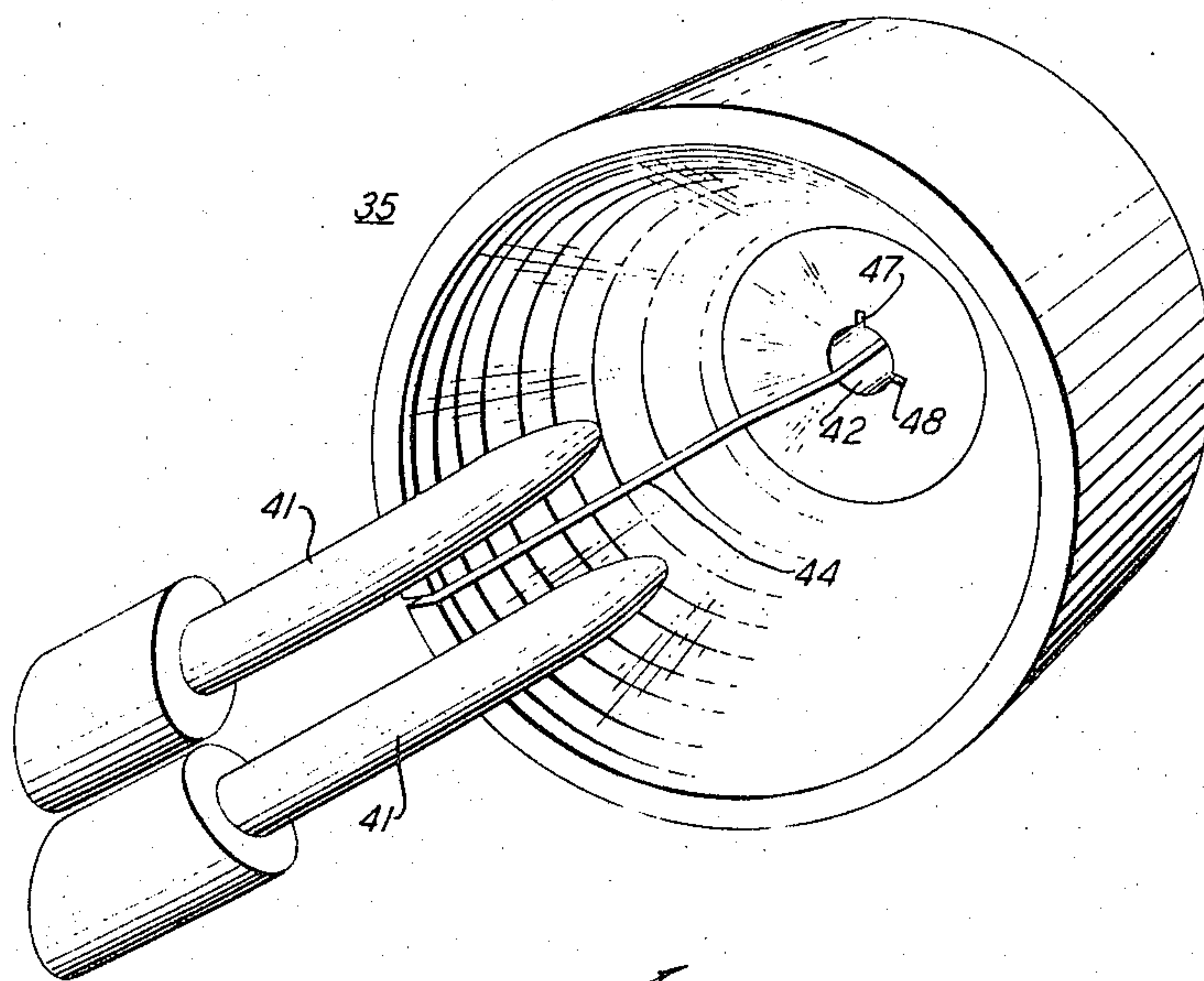
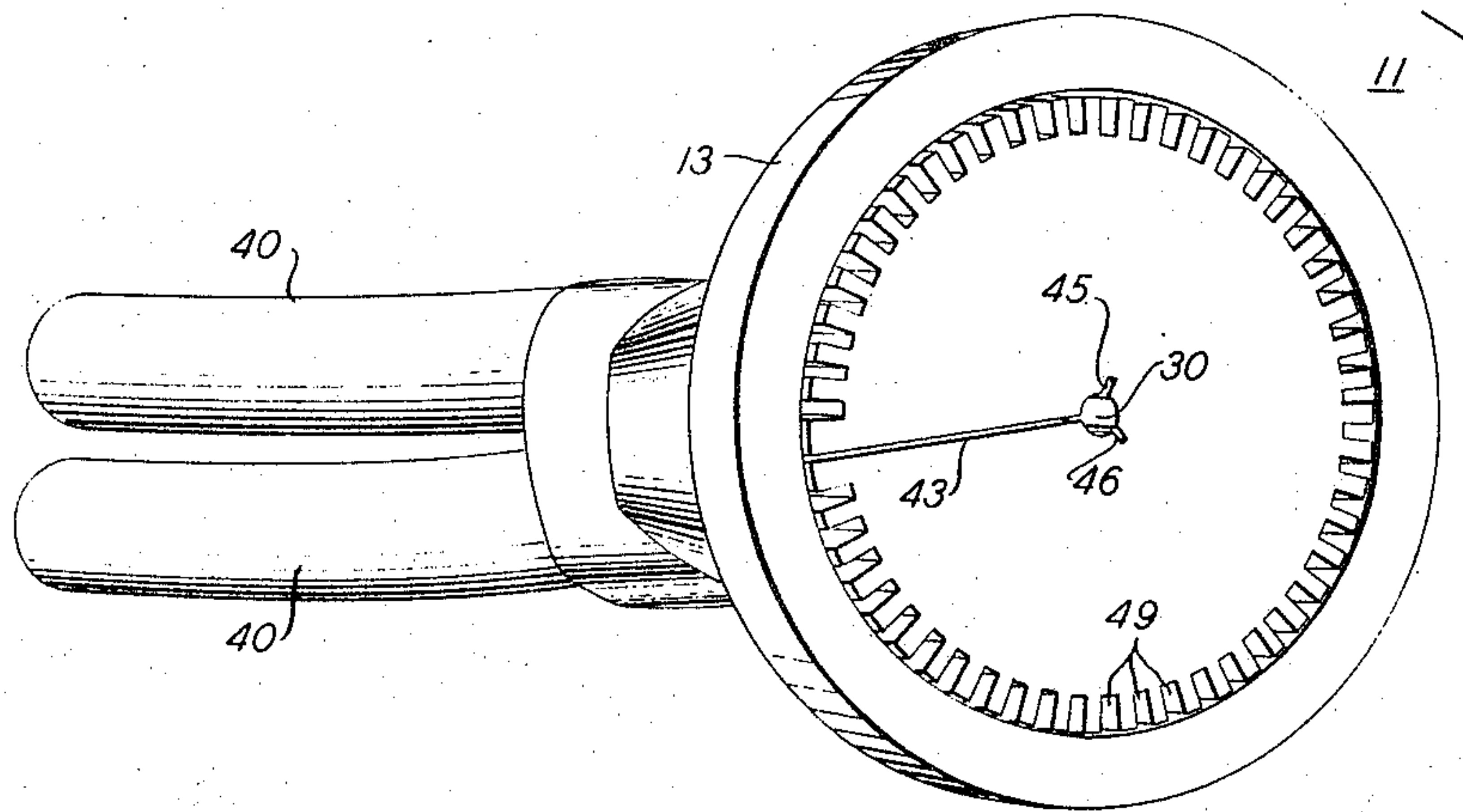


FIG. 2

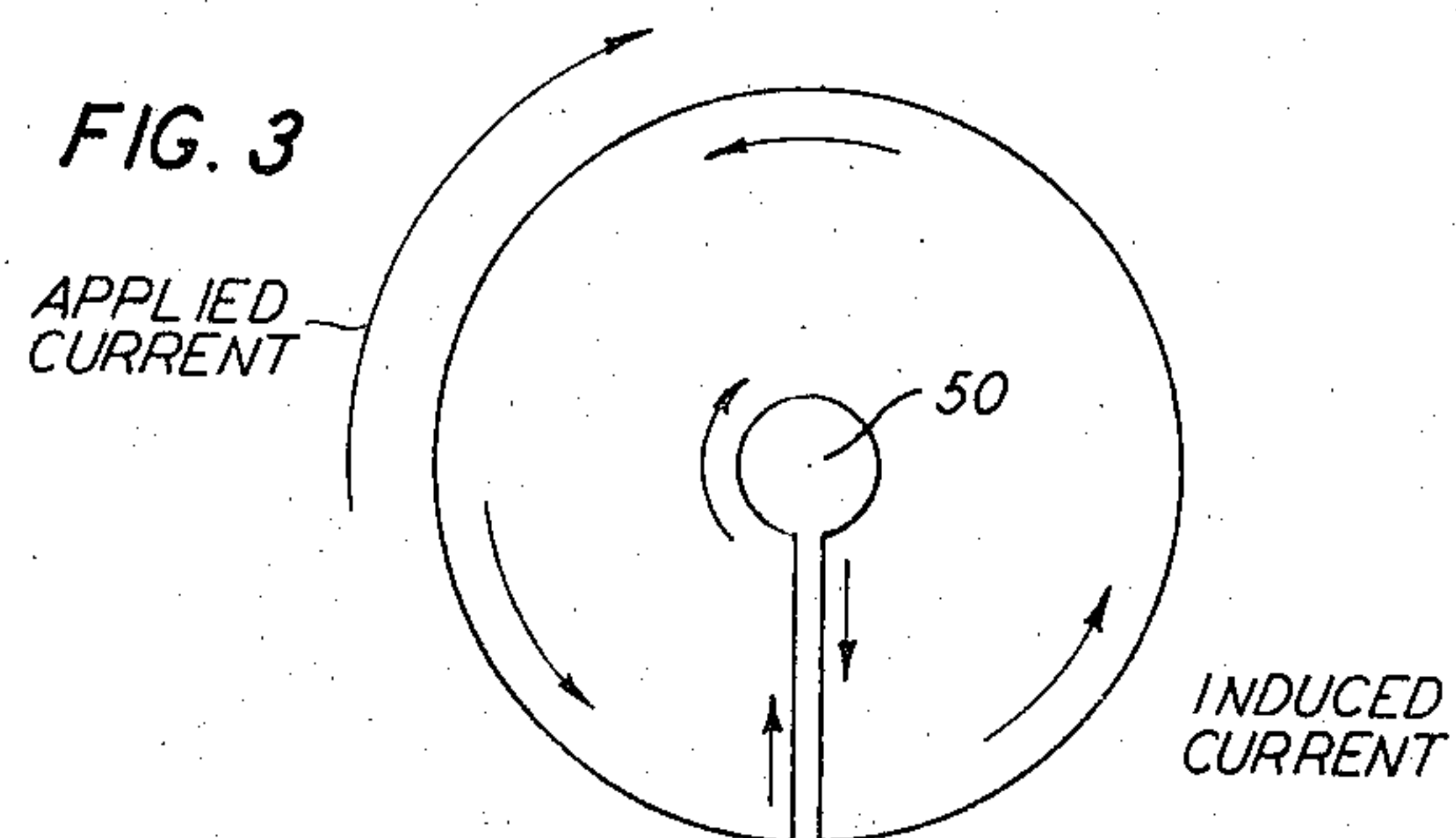


FIG. 3

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3,365,184

MELTING APPARATUS

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6 Claims. (Cl. 266-33)

This invention relates to a heat treating apparatus especially useful for melting refractory material and materials with high vapor pressure constituents without contamination or significant loss of the volatile ingredient.

There are several prior art techniques used to melt materials without crucible contamination. These include arc melting, levitation melting, electron beam melting, plasma beam melting and silver boat melting. All of these methods suffer various deficiencies when compared to the technique utilizing the apparatus of this invention. Through the use of this new melting apparatus the most refractory materials known, such as tantalum carbide, M.P. 3880° C., can be melted. Furthermore, the melting is so instantaneous that there is little opportunity for volatile ingredients to boil off. This feature is important when the melting technique is used in combination with the rapid quench procedure for analytical phase studies as hereinafter described.

The melting apparatus consists basically of a fluid-cooled hearth which also acts as an induction heat source, and a flux concentrator in combination with the hearth for confining RF energy to the area where the sample is being melted. Both of these components are shaped and combined in a particular manner to concentrate the RF field and produce a partial levitation of the sample during melting.

These and other aspects of the invention will become apparent with the aid of the following detailed description. The apparatus will be described in combination with a rapid quenching apparatus to demonstrate an important area of possible application. Other commercial applications of the melting method will become apparent to those skilled in the art.

In the drawing:

FIG. 1 is a front elevation mostly in section of a rapid quenching apparatus embodying the melting apparatus which forms the basis for this invention;

FIG. 2 is a perspective view of two subcomponents of the assembly of FIG. 1 which comprise the essence of the melting apparatus; and

FIG. 3 is a schematic representation of the current flow in the components of the melting apparatus shown in FIG. 2.

FIG. 1 shows the melting apparatus combined with a rapid quench device. Such a device can be used for creating new metastable phases in various alloy systems. For instance, tungsten carbide, which normally possesses a hexagonal crystal structure and is not superconducting, has been quenched using this apparatus to produce a face centered cubic structure. This form of tungsten carbide is superconducting with a transition temperature of 10° K. Another superconductor, Nb₃Ge, normally does not form stoichiometrically. When it is rapidly quenched with the apparatus described here it forms stoichiometrically with a critical temperature of 17° K. as compared with its normal critical temperature of 60° K. Such applications of the invention have considerable technical importance.

The obtaining of metastable states by melting and then cooling is generally contingent upon the quenching rate. The apparatus described is used to fuse a multicomponent or alloy sample and drive the molten globule against a cool, curved surface so as to instantaneously spread the globule on the cool surface and thereby rapidly quench the material over a wide surface area.

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In FIG. 1 the sample 10, typically weighing about 0.1 gram, is shown supported on a silver hearth 11. The silver hearth is cooled with water through a circumferential duct 12. The silver hearth is supported by an insulating support 13 which may be of boron nitride. The support 13 is contained in the lower end of the support tube 14 which is made of Bakelite. The tube is preferably nonconducting and can be ceramic. Clamped onto the upper end of the support tube 14 with clamp 15 is a gas tube assembly 16 used for flushing the melting zone with inert gas and for introducing a high pressure gas shock wave to propel the molten sample against the cool surface. The gas tube assembly 16 communicates with the melting zone through a nonconducting extension tube 17 which is fixed to the lower end of the assembly 16 with a flange held by threaded cap 18. The extension tube may also be boron nitride or any appropriate nonconducting material. The upper region of the gas tube connects first to an inert gas inlet 19 and is sealed from the upper end of the tube with a Mylar diaphragm 20. In the region of the gas tube above the diaphragm 20 is a high pressure chamber 21 defined by the sleeve 22 which is fitted and sealed to the assembly 16 by O-rings 23, 24 and 25. Gas, such as helium, is admitted to the high pressure chamber 21 through gas inlet 26. The upper end of the high pressure chamber 21 is sealed with a glass window 27 and O-ring 28. The clamping pin 29 biases the O-rings against their bearing surfaces. At the lower end of the apparatus there is provided a hole 30 through the silver hearth 11 having a diameter of approximately 1/16 inch. There is a larger opening 31 in the support 13. Disposed adjacent the holes 30 and 31 is a curved copper strip 32. The strip is held in place by clamp 33. Heat for melting the sample is provided by the induction coil 34 which couples to both the silver hearth 11 and an RF concentrator 35. The construction of the silver hearth and the RF concentrator are such that the RF field is concentrated in the region of the hole 30. The RF concentrator 35 has a circumferential channel 36 for accommodating a coolant such as water to cool the metal adjacent the melting region. The RF concentrator is constructed of silver also. The cooled hearth 11 and the cooled RF concentrator are the basic units of the melting apparatus to which this invention is principally directed. The details of design and construction of these elements will be described subsequently in connection with FIGS. 2 and 3.

The operation of the apparatus of FIG. 1 is essentially as follows:

The interior of the apparatus is continuously flushed with argon or other appropriate inert or noncontaminating gas. The gas is admitted through inlet 19, courses through tube 17 and exits through hole 30. This gas reduces oxidation of the sample during melting. The copper strip 32 can be bathed in a stream of inert gas to provide an inert surface upon which to splat the molten sample. The induction coil 34 is energized and the RF field couples to both the silver hearth 11 and the RF field concentrator 35. Water is circulated continuously through both elements to prevent melting of the silver under the high fields induced. The generator used with this particular apparatus was rated at 20 kw., 450 kc./s. although the power source is not critical. The size of the power unit will vary according to the construction details of the apparatus which determine the coupling efficiency of the power input to the sample to be melted. The temperature required to heat the sample will also affect the amount of power required to melt a given sample.

Gas is then admitted through inlet 26 into the high pressure chamber 21 until it ruptures the Mylar diaphragm 20 and sends a shock pulse through the gas tube 17 to the molten sample 10, driving the molten sample through

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the hole 30 in the hearth 11 and through the opening 31 in the support 13. The globule then impinges on the curved copper strip 32. The radial acceleration given to the molten material by the strip tends to maintain good thermal contact between the melt and the copper strip. The sample cools almost instantaneously. The entire operation is very quick, usually requiring less than a second. Quenching rates are of the order of several million degrees per second.

The application of pressure to the chamber 21 and rupture of the diaphragm 20 are almost simultaneous. The chamber 21 is most conveniently valved to a high pressure source with a trigger-like mechanism so that when the trigger is "fired" the diaphragm ruptures and the molten sample immediately splats onto the strip. The manipulation of the apparatus involves simply energizing the coil, visually observing through the window 27 until the sample is molten and then "firing" the molten sample onto the copper strip. More elaborate methods could be used to automatically operate the apparatus.

The basic elements of the melting apparatus are shown in FIG. 2 which is a perspective view of the hearth 11 and the RF flux concentrator 35. The cooling tubes are shown at 40 and 41. The essential features of the melting apparatus are the use of a combined heat source and a noncontaminating hearth for supporting the sample during melting and the particular manner of applying RF energy to the sample. The noncontaminating hearth must be of an electrically conductive material to permit induced electrical fields and should also be thermally conductive to permit effective cooling. Accordingly, the hearth should be constructed of a material having a room temperature resistivity of less than 5×10^{-6} ohm cm. and a thermal conductivity measured at 0° C. of at least 0.2 cal./cm. sec. degree C. Metals which meet these limitations are aluminum, copper, magnesium, molybdenum, gold and silver. Various alloys such as brass are also suitable.

The effective coupling of RF energy from the external coil to the sample to be melted is due in part to the peculiar geometry of the hearth 11 and the RF concentrator 35. The concentrator 35 should be of a material having the same properties as those prescribed above in connection with the hearth. The shape of the concentrator 35 provides a tapered interior wall which terminates in a small central opening 42 at the base of the cylinder. The hearth 11 also has a small hole 30 at its center. An essential feature of each member are the radial slots 43 and 44. The RF currents induced in these members are confined to the surface regions of the silver and flow as illustrated in the diagram of FIG. 3. The current which passes around the center hole 50 (which corresponds to holes 30 and 42 of the hearth 11 and concentrator 35, respectively) is approximately in phase with the current in the RF coil. The volume in the center hole is then a high RF flux region which is uncompensated (i.e., non-cancellation of flux lines from applied and induced RF currents).

The ratio of the diameter of the hole to the over-all diameter of the hearth and flux concentrator is not critical if the slot is present. However, the effective field concentration is a function of the ratio of the area of the outer surface exposed to the external RF field to the area of the internal hole. For the purposes of this invention it is preferred that this ratio exceed five and preferably ten. For a cylindrical element, such as that shown at 11 in FIG. 2, this limitation prescribes a ratio of the over-all diameter of the element to the diameter of the hole. Other geometries may be used to achieve the same result. For instance, the hearth 11 could assume a shape similar to the concentrator 35 whereby the outer area is multiplied relative to the internal area by tapering the internal wall to a small area at the hole. The upper concentrator 35 in this apparatus is tapered so as to couple a larger portion of the external field into the upper element. The

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hearth should be more weakly coupled. The proper balance is important for achieving the levitating effects mentioned hereinafter. In most cases the ratio of the energy effectively coupled to the hearth to the energy coupled to the upper field concentrator should be approximately within the range 0.3 to 0.9. The relative proportion of energies coupled to the two elements is most conveniently adjusted by varying the position of the external field relative to the two elements.

The presence of both elements 11 and 35 is essential. The RF currents necessary to melt refractory materials are so high that a significant levitating force is applied to the sample from induced currents in the hearth 11. If the hearth is used alone the sample is often forced away from the hearth by the levitating field and heating is ineffectual. When the RF field is coupled to the RF concentrator 35 also, which is disposed over the hearth and the sample as shown in FIG. 1, the downward magnetic and gravitational force tends to equalize the upward levitating force of the hearth and the sample is stabilized. For this reason it is important to couple the RF field to both the hearth and the concentrator. It should be pointed out that the field induced in the hearth does partially levitate the sample and this aids significantly in reducing the wasteful heat flow from the sample, as it is heating, back into the heat-conductive hearth. If a large contact area is permitted between the sample and the hearth, heating will be less effective. With the sample supported only by the edges of the hole 30 and then partially levitated by the field, significant heat transfer back into the hearth is virtually eliminated. These considerations are of less consequence where only moderate temperatures are required for the heat treatment.

It should be pointed out that there is cross-coupling between the two silver pieces. Thus the external field may couple partially to the lower hearth piece through the top concentrator. The proximity of the material to be melted, to the top concentrator might suggest the possibility of melting without slotting the lower silver hearth. However, it has been found that this can not be done effectively. Even supporting the sample on top of a small diameter silver tube just below the top concentrator leads to ineffectual heating. The success of the method depends, in part, upon the radial slot in the lower hearth. Since the RF field is distorted in the region of the radial slots 43 and 44, two additional notches 45, 46 and 47, 48 (FIG. 2) can be put in the center hole to make the RF field symmetrical about the holes. This latter expedient is helpful but not essential. An improvement of the efficiency of melting some materials was noted when notches 49 were milled around the circumference of the lower hearth. This notching increases the resistive component of the RF voltage drop on the lower hearth.

The apparatus has been described as a melting apparatus but in its broadest aspects is a heat treating apparatus. These same principles can be applied to the design of an apparatus employed for annealing refractory materials or other heat treatments which require extremely high temperatures but do not necessarily involve melting.

Various additions and modifications of this invention will become apparent to those skilled in the art. All such variations and deviations which basically rely on the teachings through which this invention has advanced the art are properly considered within the spirit and scope of this invention.

What is claimed is:

1. A melting apparatus comprising a metallic hearth and an RF field concentrator each composed of a material having a room temperature resistivity of less than 5×10^{-6} ohm-cm. and a thermal conductivity measured at 0° C. of at least 0.2 cal./cm. sec. degree C., said hearth having a cylindrical shape and a hole extending through the approximate axis of the cylinder having a diameter sufficient to support a sample to be melted on its edge portions, a radial slot extending from said hole to the exterior

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wall of the cylinder said slot having a width less than the diameter of the said hole, cooling means associated with said hearth for fluid cooling the portion of the hearth surrounding the hole, the said RF field concentrator disposed adjacent said hearth for concentrating an external RF field to the region of the hole in said hearth said field concentrator mounted atop the hearth and having means for fluid cooling the region of high field concentration and an RF coil encircling the hearth and field concentrator for inducing a high current flow in the hearth and field concentrator, the ratio of energy coupled to the hearth to the energy coupled to the upper field concentrator being in the range of 0.1 to 0.9.

2. The apparatus of claim 1 wherein the hearth comprises silver.

3. The apparatus of claim 2 wherein the RF concentrator comprises silver.

4. The apparatus of claim 1 wherein the RF flux concentrator has an essentially cylindrical shape having a hollow portion extending axially through said cylinder the hollow portion having sloping walls beginning at the outer edge of the upper portion of the cylinder and terminating in an axial hole at the lower portion of the cylinder said internal area of the hole having a diameter of less than

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half the over-all diameter of the RF concentrator, and a slot extending from said hole to the exterior surface of the cylinder and extending over the entire height of the cylinder.

5. The apparatus of claim 1 in combination with means for expelling the molten sample through the hole in the hearth and a clean, cool, curved surface disposed at the exit of said hole upon which the expelled sample can impinge.

6. The apparatus of claim 5 wherein the means for expelling the molten sample comprises a means for applying a gas shock wave to the region of the hole at the surface of the hearth.

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