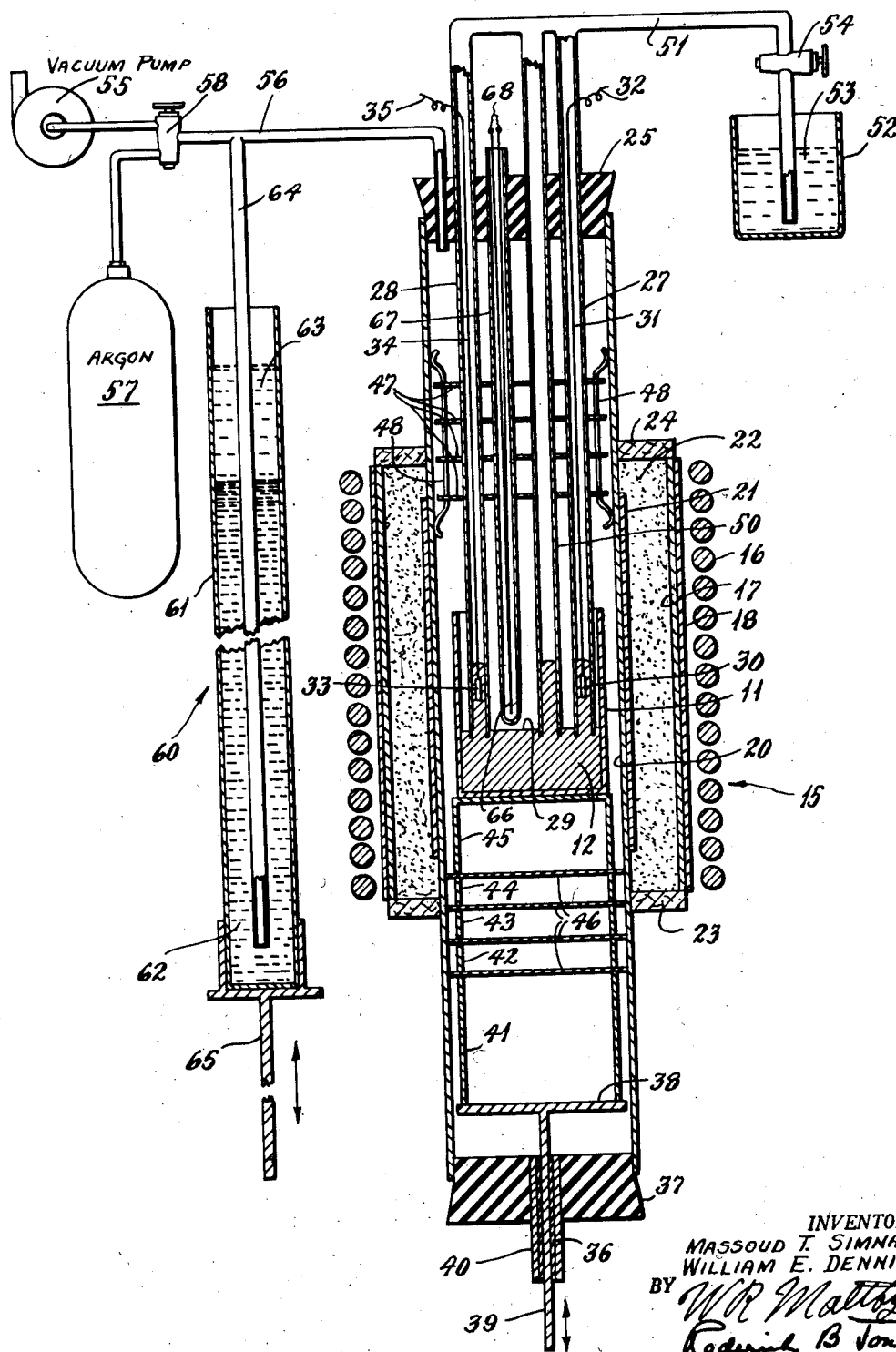


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FURNACE, ADAPTED PARTICULARLY FOR ELECTROCHEMICAL
STUDIES OF MOLTEN MATERIALS AT HIGH TEMPERATURES
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FURNACE, ADAPTED PARTICULARLY FOR ELECTROCHEMICAL STUDIES OF MOLTEN MATERIALS AT HIGH TEMPERATURES

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5 Claims. (Cl. 13—31)

The present invention pertains to the art of making electrochemical studies of materials at high temperatures, and in the molten state, and has been used particularly to study salts and slags that occur in the compositions of melted iron during production of steels. The invention is embodied in a furnace for containing the molten material, and which supports instruments and appurtenances that are used for making the aforementioned studies.

The furnace of the invention comprises a refractory housing that contains a crucible in which a melt of the material in molten condition is supported inside a furnace. The housing extends from within the furnace upwardly out of the furnace to the atmosphere exteriorly thereof, where it is closed and sealed from the outside atmosphere.

A set of compartments or containers project downwardly into the housing, and inside the housing into the crucible. Each container is open at its lower end, and the open end is dipped into the molten material to a predetermined level below the surface level of the melt. An electrode is positioned inside each container, and is contained thereby to be in contact with the melt at a point separated from the other electrode. Each electrode comprises an electrical lead that extends upwardly within the container to a position outside the housing, and extends to the outside of the furnace where it is away from the intense heat of the furnace. Any suitable electrical measuring instrument may be connected to the electrodes through their leads, to measure electrical characteristics of the melt in the crucible at the high temperature of its molten condition.

By means of the described structure, the cathode and anode are separated from each other, and the cathodic and anodic portions of the melt are separated and isolated from each other. This enables electrical measurements to be made at high temperatures, with a high degree of accuracy.

The accompanying drawing presents one practical embodiment of the invention, and affords fuller understanding of the advantages and principles thereof. The single view of the drawing illustrates apparatus embodying the invention in cross-sectional elevation on a lengthwise center plane, with some parts shown schematically.

Crucible 11 contains material 12, and is positioned in furnace 15 at the center of the hot zone, where the crucible is surrounded completely by heat of the furnace, and material 12 is subjected to temperature that is constant throughout its mass.

Furnace 15 is the electrical induction type, and comprises induction coil 16, the windings of which are positioned around the shell 17 of the furnace. A tube of fireclay, or the like, forms the shell 17, and is wound with mica sheet 18 contained inside coil 16. Furnace 15 is lined with housing 20 of refractory material, in which crucible 11 is housed.

Housing 20 also is tubular, and is positioned inside the tube of shell 17 coaxially therewith. Graphite sleeve 21 is positioned around housing 20 coaxially therewith, and inside shell 17 where it is heated by induction coil

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16. The rest of the space inside shell 17, and around housing 20, is filled with lamp black 22 in granular form, and discs 23 and 24 constitute end plates of asbestos, which close the respective opposite ends of shell 17 and confine its contents.

Power is supplied to coil 16 from any suitable source of electricity, not shown. A 2 kva. unit of a high frequency generator has been used and is suitable.

Housing 20 is extended out of furnace 15, from its position inside the furnace and around crucible 11, upwardly through end plate 24 to the atmosphere exteriorly of the furnace. Housing 20 is closed and sealed at its upper end outside furnace 15, by means of a bung or stopper 25 of rubber or the like material. Containers 27 and 28 project from the atmosphere outside furnace 15 downwardly through stopper 25, and inside housing 20 to a position in crucible 11 proximate to the surface level 29 of material 12. In the preferred structure shown, containers 27 and 28 consist of refractory tubes, which are open at their lowermost ends. In crucible 11, containers 27 and 28 are spaced apart at intervals along surface 29, and their open ends dip into material 12 under its surface level.

Electrode 30, which is the cathode, is contained in container 27, positioned to contact material 12, and comprises lead 31 extending upwardly in container 27 and through its wall to terminal 32, which is located outside housing 20 and exteriorly of furnace 15 where it is away from the intense heat of the furnace. Similarly, electrode 33, which is the anode, is contained in container 28 in position to contact material 12, and comprises lead 34 extending upwardly in container 28 and through its wall to terminal 35, which is located outside housing 20 and exteriorly of furnace 15 where it is away from the intense heat of the furnace. Any suitable electrical measuring instrument, not shown, may be connected with terminals 32 and 35 to measure electrical characteristics of the melt of material 12. A Wheatstone bridge has been used, with an 100 cycle A. C. current supply.

The cathode portion of melt 12 inside container 27, and the anodic portion inside container 28, are separated from each other and isolated by the containers, and this structure enables electrical readings of material 12 to be made with a high degree of accuracy, and at the high temperatures that occur in practice of the invention, between 1000° and 1400° C.

Housing 20 extends downwardly also, from inside furnace 15 to the outside where it is closed and sealed at its lower end by means of bung or stopper 37, of rubber or the like material. An elevator comprises the platform 38 on which crucible 11 rests, and the stem or rod 39 which travels in slide bearing 40, the bearing being secured in stopper 37. Stem 39 projects downwardly to the outside of housing 20 and exteriorly of furnace 15, where it may be manipulated manually to raise or lower crucible 11. By means of the described structure, melt 12 is positioned alternatively into and out of engagement with the electrodes 30 and 33, and the depth is adjusted to which the lower open ends of tubes 27 and 28 are dipped into the melt 12 and below the surface level 29 thereof. Grease 36 on stem 39 serves as a lubricant, and also seals bearing 40 against leakage of gas from inside housing 20 to the atmosphere outside of furnace 15.

A set of spacer members of heat resistant material, 41, 42, 43, 44 and 45, rest on top of each other on platform 38, and crucible 11 rests on top-most spacer member 45. Platform 38 is positioned in housing 20 beyond end plate 23 and remote from the hot zone of furnace 15, and the spacer members 41, 42, 43, 44 and 45 locate crucible 11 at the center of the hot zone of furnace 15.

A set of heat reflectors of stainless steel 46 are posi-

tioned severally between adjacent spacer members 41, 42, 43, 44 and 45, and are located in succession from a position beyond end plate 23 and outside furnace 15 to a position inside the furnace where the uppermost spacer member 45 holds crucible 11 in the hot zone. Heat reflectors 46 operate to reflect heat upwardly towards the hot zone, and constitute an insulator. A similar set of stainless-steel heat reflectors 47 are held inside housing 20 by means of frame members 48, which hold them at spaced intervals and located in succession from a point outside furnace 15 beyond upper end plate 24 to a position inside the furnace. Reflectors 47 reflect heat downwardly towards the hot zone of furnace 15, and also function as an insulator.

By means of the described structure, the temperature in the hot zone at the center of furnace 15 is controlled within the limits of $\pm 3^\circ \text{C}$. over a distance of six inches, at the highest operating temperature of 1400°C . Any position of crucible 11 in furnace 15 is within the hot zone.

In addition to tubes 27 and 28, the tube 50 of refractory material also is projected through stopper 25, and extends downwardly inside housing 20 into crucible 11. At their lower ends, all the tubes 27, 28 and 50 are at the same level with reference to the surface level 29 of melt 12. Tube 50 is open at its lower end also, in a manner similar to tubes 27 and 28, and serves to take samples of melt 12. At their upper ends, tubes 27, 28 and 50 are connected to each other by means of fluid line 51.

Fluid line 51 is open at its lower end, and is extended to project downwardly into a vessel 52 of liquid 53. Vessel 52 constitutes a beaker, and the liquid 53 that it contains is a low vapor-pressure oil, which serves as a trap to prevent atmospheric air from entering the system of furnace 15 through fluid line 51 and the several tubes 27, 28 and 50. Trap 52 permits gas under pressure in furnace 15 to escape to the exterior atmosphere through tubes 27, 28 and 50, fluid line 51, and oil 53 in beaker 52. Petcock 54 in line 51 serves to close or open the line 51 at will.

Vacuum pump 55 is connected with the atmosphere inside housing 20, and in crucible 11 above material 12, through fluid line 56, which projects into housing 20 through stopper 25. A container or bottle 57 of gas under pressure is connected through fluid line 56 with the interior of housing 20. An inert pressure gas is preferred, and bottle 57 contains argon. The three-way petcock 58 is operable as a valve to connect the vacuum pump 55 or the argon supply 57 with the inside of housing 20 alternatively, or to disconnect both and close the line 56.

A fluid trap 60 is connected with line 56 between petcock 58 and housing 20, and comprises the elongated vessel 61 containing a column of mercury 62 topped by a depth of oil 63. Tube 64 is open at its lower end, and is connected with line 56 at its upper end. The lower end of tube 64 is projected downwardly into vessel 61, which may be raised or lowered by means of elevator 65 to dip the tube 64 into the liquid of vessel 61, or to remove it, alternatively. Vessel 61 may be raised or lowered for adjustment, to dip the open lower end of tube 64 to any desired depth in oil 63, or to any desired additional depth in mercury 62. Trap 60 serves to control the magnitude of pressure of the gaseous atmosphere inside of housing 20. Tube 64 is at least 76 cm. in length, and vessel 61 is correspondingly deep, to afford a sufficient column of mercury for the magnitude of atmospheric pressure desired in furnace 15.

Tube 67 is closed at its lower end, and is projected through stopper 25 and downwardly in housing 20 into crucible 11. Thermocouple 66 extends downwardly in tube 67 to a position at its lower end, which is located in the hot zone of furnace 15 positioned near and adjacent to the surface level 29 of material 12. Thermocouple 66 comprises the terminals 68 located outside housing 20 and in the atmosphere exteriorly of furnace 15, where

the thermocouple may be connected to an instrument, not shown, for measuring the temperature in the furnace in a manner that is well understood in the art.

The apparatus of the disclosure has been used to study the electrochemistry of the molten FeO-SiO_2 system in equilibrium with iron, using iron crucibles 11, and also iron electrodes 30 and 33, at temperatures between 1000° and 1400°C .

To prepare the apparatus for a study of a melt of material 12, the elevator of platform 38 is lowered to position the material 12 below the lower open ends of tubes 27, 28 and 50. Petcock 54 is closed. Trap 60 is raised to dip the open lower end of tube 64 into mercury 62. Now petcock 58 is opened to the vacuum pump 55, which is operated to evacuate the furnace 15.

When furnace 15 is completely evacuated, petcock 58 is turned to connect the gas supply 58 with the furnace, whereby the vacuum pump 55 is disconnected. Vessel 61 is lowered to adjust trap 60 to position the lower end of tube 64 about $1\frac{1}{2}$ " below the surface level of oil 63. Petcock 54 is opened, and the argon gas bubbles out of oil 63 in beaker 52. The atmosphere of furnace 15 thereby consists of inert argon gas.

The power source of induction coil 16 is turned on, and furnace 15 is heated to the desired temperature at which the material 12 is molten. Now stem 39 is raised in bearing 40 to raise crucible 11 until the tubes 27, 28 and 50 dip into the melt 12, and the surface level 29 just covers the open ends of the tubes. This is indicated when argon bubbles out of the oil 63 of trap 60 instead of through the oil 53 of beaker 52.

The elevator of platform 38 is raised to a higher level to keep the lower open ends of tubes 27, 28 and 50 below the surface 29 of melt 12 when the tubes become filled with material of the melt. Vessel 61 of trap 60 is raised with reference to tube 64, to project the tube into the mercury column 62. This increases the atmospheric pressure of argon gas in housing 20 and exteriorly of containers 27 and 28, and raises material 12 in the tubes 27, 28 and 50. Vessel 61 is raised gradually, to build up the atmospheric pressure gradually in furnace 15, and the process is continued until material 12 rises in tubes 27, 28 and 50 sufficiently to cover electrodes 30 and 33 to a depth of about one centimeter. The location of electrodes 30 and 33 lengthwise of containers 27 and 28 establishes the pressure in furnace 15 at the desired magnitude when good electrical contact is made between the melt 12 and the electrodes 30 and 33. When this condition is reached, argon gas continues to bubble out of oil 63 of trap 60.

The described structure has been used to make electrochemical studies of slag and fused salts at the high temperatures of the slag in molten condition, and to obtain accurate measurements of conductivity, transference number, and ionic mobility of molten slag and fused salts.

The scope of the present invention is determined by the accompanying claims.

We claim:

1. In combination with a crucible of molten material in a furnace, a refractory housing containing the crucible, the housing extending from inside the furnace upwardly out of the furnace and being closed to the atmosphere exteriorly of the furnace, a set of containers projecting downwardly in the housing, each container comprising an opening at its lower end that dips into the crucible below the level of the molten material, the several containers engaging the molten material at points spaced apart along the surface of the molten material, an electrode in each of the several containers and an electrical lead from each of the electrodes to the atmosphere exteriorly of the furnace, pressure-control apparatus operable to vary and control the pressure differential of atmospheric pressure between the inside and outside of the containers above the level of molten material.

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2. In the combination of claim 1, the pressure-control apparatus comprising a pump and a fluid line from the pump to the atmosphere inside the housing and above the molten material exteriorly of the electrode containers.

3. In the combination of claim 2, the pressure-control apparatus comprising a valve operable to control fluid flow one-way to the atmosphere exteriorly of the furnace and a connection from the electrode containers above the molten material to the atmosphere exteriorly of the furnace through the one-way valve, a container of gas under predetermined pressure, the pump constituting a vacuum pump, and a valve between the vacuum pump and the gas container and in the fluid line to the housing exteriorly of the containers to connect the vacuum pump or the gas container with the housing alternatively.

4. In the combination of claim 1, the housing extending from within the furnace to the atmosphere exteriorly thereof, an elevator supporting the crucible in the housing and operable from below the furnace to raise and lower the crucible for adjusting its position within the furnace with reference to the electrode containers.

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5. In the combination of claim 3, the housing extending downwardly from within the furnace to the atmosphere exteriorly thereof, an elevator supporting the crucible in the housing and operable from below the furnace to raise and lower the crucible for adjusting its position within the furnace with reference to the electrode containers, the pressure-control apparatus comprising a valve to vary the atmospheric pressure of the housing exteriorly of the electrode containers and operable for fluid flow one-way to the atmosphere exteriorly of the furnace, the one-way valve comprising an elongated container and a column of liquid therein, a pressure-control line extending into the column of liquid at a predetermined depth and from the fluid line between the valve of the pump and gas container to the housing, and an adjustment for controlling the depth of the pressure-control line in the liquid column.

No references cited.