United States Patent

Sitek et al.

[15] **3,636,229**

[45] **Jan. 18, 1972**

[54] **ELECTRICALLY RESISTIVE CRUCIBLE**

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[52] **U.S. Cl.** **13/25,** 263/47

[51] **Int. Cl.** **F27d 11/02,** H05b 3/10

[58] **Field of Search** 13/20, 22, 25, 31; 263/47, 263/48, 49; 266/39

[56] **References Cited**

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[57] **ABSTRACT**

An electrically resistive carbon crucible for an impulse or resistance furnace designed to have improved heating characteristics.

2 Claims, 3 Drawing Figures

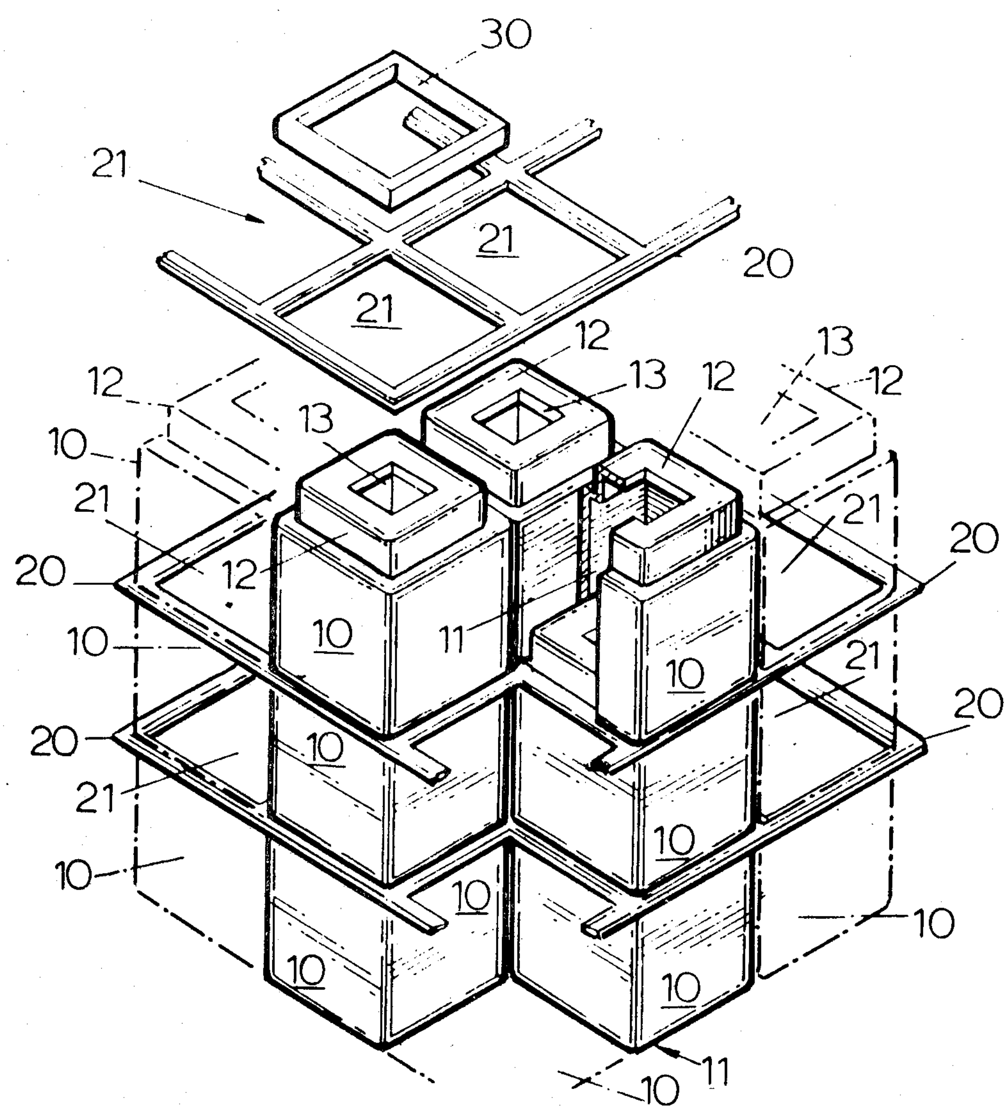

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Fig. 1.

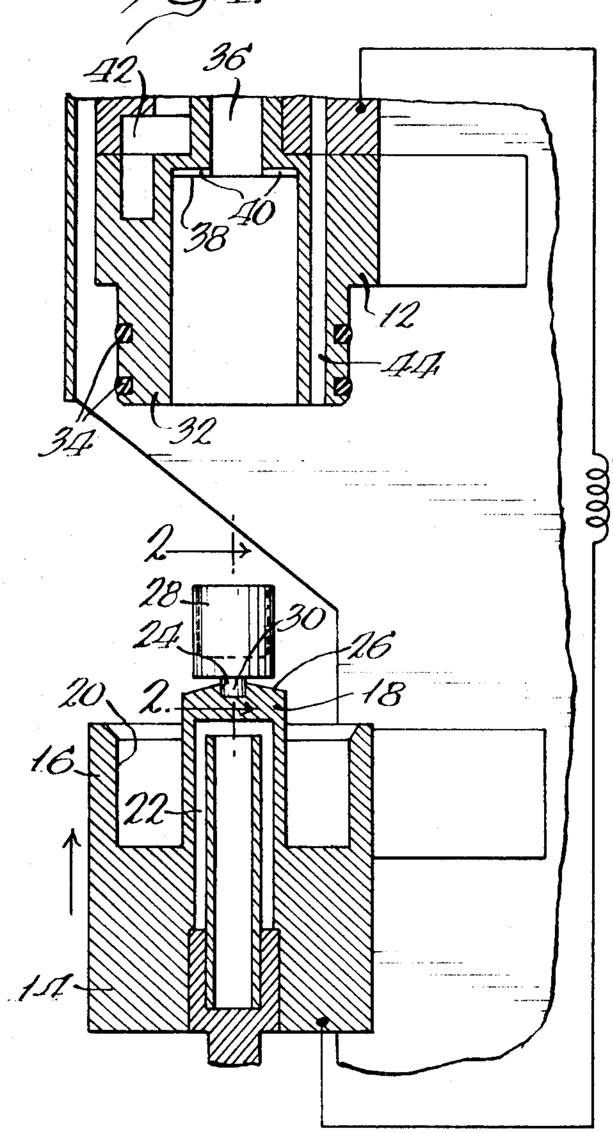


Fig. 2.

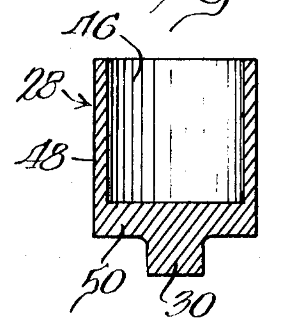
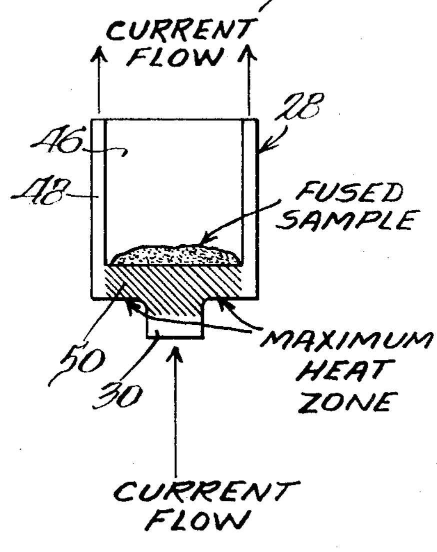


Fig. 3.



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ELECTRICALLY RESISTIVE CRUCIBLE

BACKGROUND OF THE INVENTION

In the analysis of metals for such components as oxygen, hydrogen, nitrogen, etc., it is common to raise the metal to extreme elevated temperatures to fuse it and release the gaseous or volatile matter for subsequent analysis. Such technique is applicable generally to any composition. Induction heating has frequently been employed in the past to obtain this elevation of temperature. Now, however, resistance heating (DC) or impulse heating (AC) is preferred. Either of these consists of passing an extremely high current through a carbon or graphite crucible contained between water cooled electrodes. The crucible constitutes a resistance element which achieves the desired temperature of 2,700° C. or higher. The crucible not only constitutes the resistance element in such a usage; it also provides the reactive material in the event that oxygen is the subject of the analysis. Upon heating of the sample, the oxygen will react with the carbon of the crucible to form carbon monoxide which may be swept from the furnace by a nitrogen stream for subsequent analysis.

The crucible hitherto employed for this purpose has been a simple cylinder having an axial bore extending partially therethrough from one end so as to leave a relatively heavy base. The heating in a resistance device will, of course, be greatest where the section is smallest. Therefore, with the conventional crucible, the maximum heating will occur in the wall portion of the crucible above the base. Since, however, there is heat loss to the cooled electrodes, the areas of highest temperature will be about midway along the length of the crucible.

To withstand the clamping of the crucible between the electrodes for satisfactory conductivity and to withstand handling generally, such a crucible must obviously have a certain minimum wall thickness. With an increase in the diameter of the crucible, therefore, such as might be desired for the analysis of larger samples or solid samples of appreciable length, the area of the minimum section will increase and the heating effect be diminished for any given current. Thus, with a given power supply, there is a highly restrictive limit to the diameter of the crucible employed. The time for heating could, of course, be extended, but this is undesirable in such analytical apparatus, and by virtue of the heat-sinking effect of the electrodes, even the extension of heating time will run into a limit.

Also, since the heat is generated primarily in the walls, the floor of the crucible must receive its heat by thermal conduction from the walls. At the same time there is the heat loss to the electrodes. The latter effect can be minimized by making the crucible bottom thick, but as the diameter of the crucible increases, the loss to the electrode becomes proportionately greater to a point where, again, the temperature achieved at the floor is inadequate.

SUMMARY OF THE INVENTION

This invention teaches a carbon crucible having an integral stud on the exterior of the bottom thereof through which the crucible is connected into the electrode circuit whereby the size limitations of the conventional crucible discussed above are avoided and whereby the point of maximum heating occurs on the bottom of the crucible rather than a distance up the sides thereof.

DESCRIPTION OF THE DRAWINGS

FIG. 1 is a vertical, somewhat diagrammatic, section through an impulse furnace adapted to use the graphite crucible of the invention, shown with a crucible mounted therein;

FIG. 2 is a vertical section through the crucible of FIG. 1 taken along the line 2—2 of FIG. 1; and

FIG. 3 is a somewhat diagrammatic representation of the section of FIG. 2 illustrating the zone of maximum heat.

DESCRIPTION OF A PREFERRED EMBODIMENT

In FIG. 1 there is shown a portion of combustion apparatus to be used in conjunction with analytical equipment, the portion illustrating an impulse furnace particularly. The furnace includes a stationary top electrode **12** and a lower support electrode **14** movable toward and away from the top. The support electrode is cup-shaped with an annular rim **16** and a post **18** extending upward from the center of the bottom of the cup, defining an annular groove **20** between the rim and the post. The post is hollow as at **22** to provide for a flow of liquid coolant therethrough. The upper end of the post has a cuplike socket **24** in the center thereof, and from the socket, the upper end slopes broadly conically downward as at **26**. A crucible **28**, the subject of this invention, will be mounted to the lower electrode post **18** with the stud **30** thereof received in the socket **24**.

The top stationary electrode **12** is likewise cup-shaped, but downwardly facing. It includes a rim or skirt **32** fitting closely telescopically within the rim **16** of the lower electrode, and O-rings **34** embedded in the outer periphery of the skirt make a gastight seal with the rim **16**. The top electrode has a large diameter passage **36** extending upward from the cup floor **38** for sample introduction and carrier gas admission. The passage **36**, while large, is smaller than the mouth of the crucible **28**. Radial grooves **40** are formed in the floor of the electrode extending from the passage **36** to the upper end of the skirt **32**. Liquid passages **42** are provided within the body of the electrode for the flow of coolant. A gas exhaust duct **44** extends from the bottom face of the skirt **32** upwardly through the electrode to be connected to analysis apparatus.

The crucible **28** is formed of a short length of cylindrical graphite rod which is machined to provide an interior cavity **46** defined by upstanding walls **48**, a base **50** somewhat heavier than the walls, and the cylindrical stud **30** projecting centrally from the bottom **50** of the crucible. The stud will be received in the socket **24** of the support electrode as stated above.

The electrodes are connected across a source of low voltage high current power **52** and desirably are made of copper.

The relation of parts in the furnace is such that, when the support electrode **14** is raised to its uppermost position with the crucible attached for sample heating, the top edge of the crucible walls **48** engages the floor **38** of the top electrode **12** concentrically with the gas passage **36**. The crucible is thus clamped under pressure between the support electrode **14** and the top electrode **12**. In such position, the sleeve **32** slides telescopically into the groove **20** but stops short of the bottom of the groove **16**. The O-rings **34**, as stated above, effect a gas seal between these telescoping surfaces and likewise serve to space the sleeve from the rim **16** in electrically insulating relationship under the low voltage of heating. The sleeve **32** is also thin enough such that it stands well clear of the center post **18** of the support electrode. Since, as illustrated, the crucible has a smaller diameter than the center post, it will be appreciated that raising the crucible into heating position will define an annular chamber about the outside of the crucible.

When the support electrode is in raised position, the bottom edge of the skirt **32** stands well above the bottom of the groove **20**, and the outlet passage **44** is thus in communication with the above-defined annular chamber. The radial grooves **40** in the top electrode open the interior of the crucible to the outside, annular chamber.

In operation, a sample will be placed in a conventional loading device above the passage **36**, not shown, and there purged by a flow of carrier gas. At the same time, the crucible will be heated in the same gas stream to eliminate occluded and absorbed gases. Thereafter, the sample will be dropped by the loading device into the crucible and the crucible heated to fuse the sample. Evolved gases from the sample will be swept by the carrier gas stream, entering through passage **36**, through radial grooves **40** and out the gas outlet **44** to analytical apparatus.

A representative crucible made in accordance with the teachings of this invention will have an outside diameter of 0.56 inch and a wall thickness of 0.04 inch. The floor of the crucible has a thickness of 0.12 inch, and the stud has a diameter of 0.187 inch and a projection of about 0.13 inch. The stud is filleted at its juncture of the bottom of the crucible **50** for reasons of strength. The socket **24** is 0.08 inch deep, thus leaving a separation of 0.05 inch between the bottom of the crucible and the upper end **26** of the post **18**.

As is common with carbon conductors, some pressure is necessary to make a good conductive contact with an electrode. Since the crucible is held between the top edges of the walls and the base of the stud **48**, primary conduction will be through the bottom surface **54** of the stud. The stud of the crucible detailed above has a cross section of about 0.025 square inch. The walls have a section equal to about 0.06 square inch. Therefore, with the passage of a high amperage current through the crucible, the stud will offer the greater resistance and will heat to a correspondingly higher degree. The heating, of course, will be sinked away to a degree by the water cooled electrodes, but by thermal conduction, the floor **46** of the crucible will heat to the greatest degree as particularly illustrated in FIG. 3. The areas of pressure contact, as well as providing conductive contact, are also the principal areas of heat loss to the electrodes. The increment of stud nearest the bottom of the crucible will therefore be the hottest portion of the system. This is in notable contrast to the previously employed studless crucible, where the outer periphery of the base of the crucible is in contact with the lower electrode (through a tripodal electrode configuration), current flow is primarily through the annular portion of the base aligned with the walls, and wherein the point of maximum heating occurs about halfway up the walls of the crucible.

By virtue of the heating of the floor of the crucible, the diameter of the crucible can be made substantially larger and thus be receptive to larger samples including pin samples which can lie flat on the floor of the crucible rather than be held in an inclined position therewithin as would be the case with the smaller diameter crucible.

The stud not only provides a small section conductor whereby the heating effect is augmented; it also serves as a stand-off insulator whereby contact of the base of the crucible with the water cooled electrode is avoided thus preventing loss of heat from the base to the electrode. Also, since the floor alone is the matter of primary interest in the crucible heating, there is less bulk of crucible to be outgassed, and accordingly, the outgassing time may be substantially reduced.

We claim:

1. An electrically resistive graphite crucible for fusion apparatus having cylindrical sidewalls, a bottom, and a stud projecting downwardly from said bottom, said stud having a cross-sectional area less than the cross-sectional area of said wall.

2. The crucible as defined in claim **1** wherein said stud has a cross-sectional area less than half the cross-sectional area of the walls of said crucible.

* * * * *

Disclaimer

3,636,229.—*George J. Sitek*, Stevensville, and *Robert N. Revesz*, Berrien, Mich. ELECTRICALLY RESISTIVE CRUCIBLE. Patent dated Jan. 18, 1972. Disclaimer filed Jan. 12, 1978, by the assignee, *Leco Corporation*.

Hereby enters this disclaimer to claims 1–2 of said patent.

[*Official Gazette March 21, 1978.*]

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