April 7, 1936. D. W. RANDOLPH 2,036,496

METHOD OF MAKING GAS FREE COMPOSITIONS OF MATTER

Filed Sept. 29, 1934 2 Sheets-Sheet 1

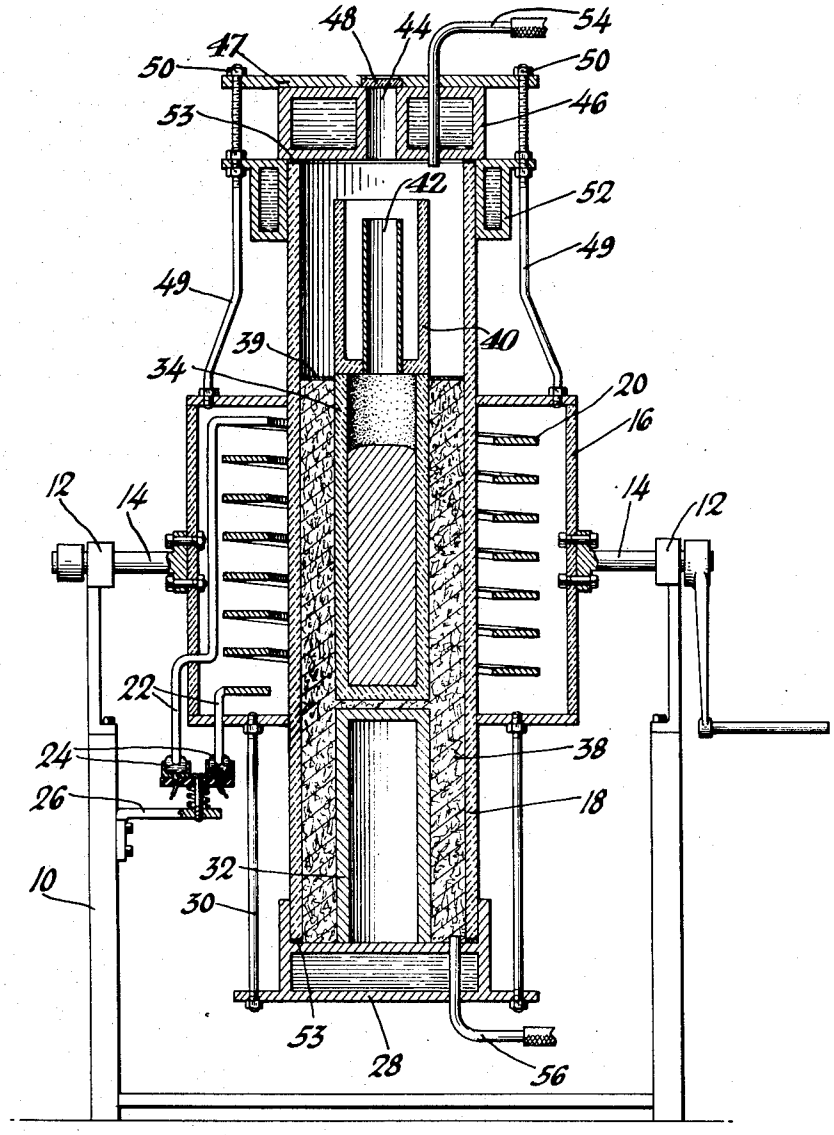


Fig. 1

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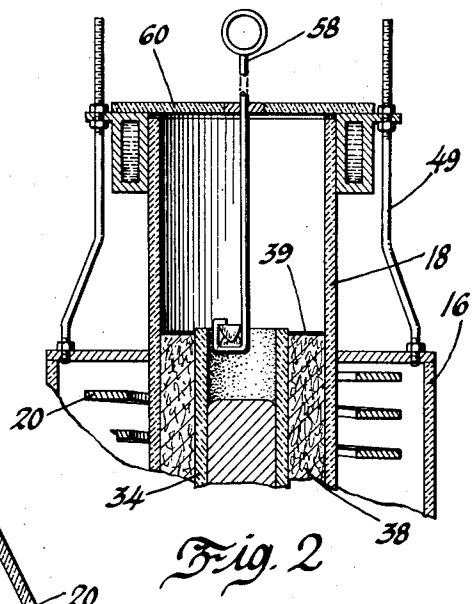


Fig. 2

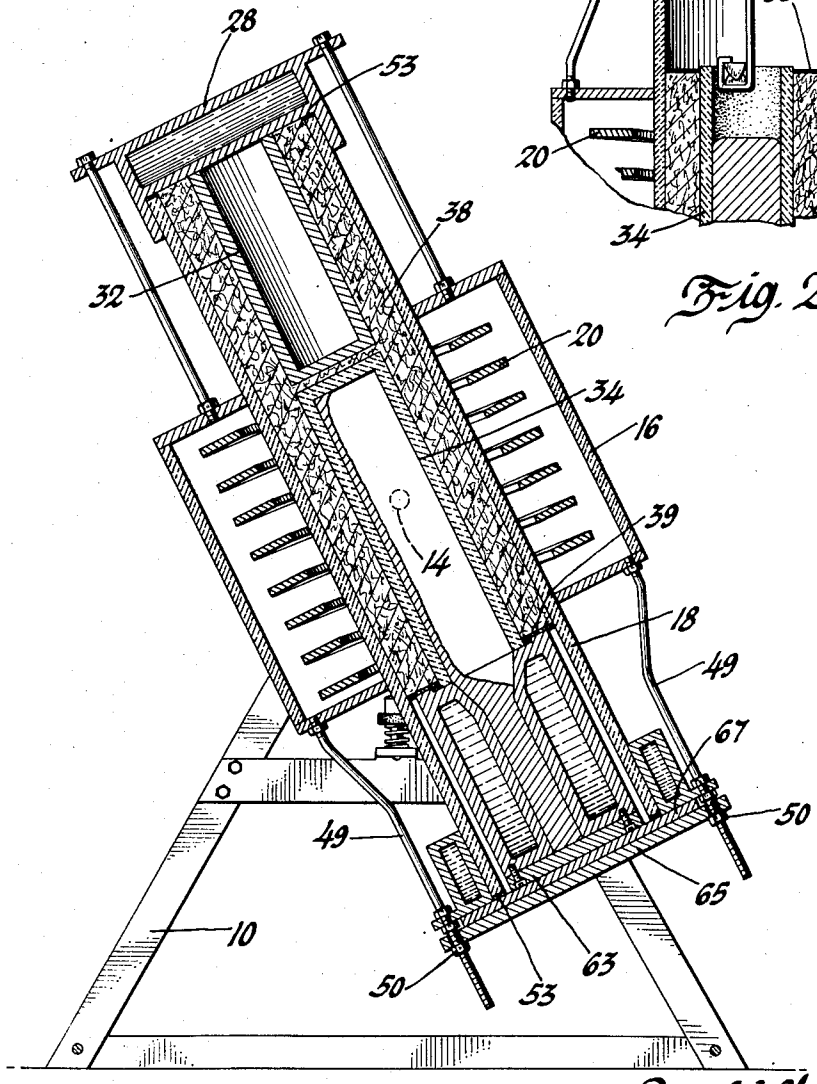


Fig. 3

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Patented Apr. 7, 1936 2,036,496

UNITED STATES PATENT OFFICE

2,036,496

METHOD OF MAKING GAS-FREE COMPOSITIONS OF MATTER

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Application September 29, 1934, Serial No. 746,153

4 Claims. (Cl. 75—135)

This application is a continuation in part of my prior application Serial No. 713,520, filed March 1, 1934.

This invention has to do with a method of making compositions of matter, especially alloys including in their composition an element that is highly volatile and chemically active, such as barium.

It has been found that the ductility and other properties of such alloys are adversely affected by gas contained within the melt. To eliminate the gas content it is proposed to melt the base metal or metals under vacuum, and to follow the melting with further heat treatment under vacuum at temperatures several hundred degrees above the melting point to further reduce the gas content.

The next problem consists in adding the volatile metal to the melt in such a way that little or no gas is introduced. This has been accomplished by surrounding the melt with an atmosphere of inert gas just prior to the introduction of the volatile metal so that when such addition is made there is little or no opportunity for absorption of gas by the melt.

With these precautions taken there remains, nevertheless, the opportunity for gas to enter the metal upon pouring into the molds. This difficulty has been overcome by placing the mold in the furnace so that it, too, is immersed in the atmosphere of inert gas. The furnace is preferably mounted so that it may be inverted to discharge the melt into the mold. The pig is allowed to remain in the mold under the inert gas until it hardens.

Alloys produced by this method are found to be very low in gas content, and as a consequence the material is of greatly improved ductility and homogeneity, and may be readily drawn into fine wire and ribbons and otherwise worked. In the case of emitters for vacuum tubes made of alloys treated in this manner there is the further advantage that it is not necessary to outgas the electrodes after assembly in the tube for the metal of which they are made is substantially free from gas.

The furnace used in this process is likewise of novel design as will be pointed out in the accompanying specification and claims.

In the drawings:

Figure 1 is a front view of the furnace and its mounting, the furnace itself being shown in section.

Figure 2 is a vertical section through the top of the furnace showing the arrangement of parts when adding material to the melt.

Figure 3 is a vertical section through the furnace taken at right angles to the section of Figure 1 but showing the furnace tilted to pour the charge into the mold.

10 indicates a suitable supporting framework having bearings 12 to receive trunnions 14 carried at the opposite sides of the casing 16, preferably formed of heat resisting and electrical insulating material such as Transite, a form of asbestos board. Within the casing 16 is mounted a tube 18 of refractory insulating material, such as quartz. Surrounding the central portion of the tube 18, and contained within the casing 16, is induction coil 20. This coil may be water cooled or not as desired. If water cooled it will preferably consist of hollow copper tubing through which water is circulated in the usual manner. Coil 20 is provided with spaced terminals 22 which are received in mercury cups 24 when the furnace is in upright position. Mercury cups 24 are the terminals of a power supply line, and provide ready means of disconnection of power when tilting the furnace. They may be supported in any suitable way as by means of bracket 26 secured to framework 10.

The bottom of the quartz tube 18 is covered by means of hollow closure 28 of suitable material through which water may be circulated for cooling. Closure 28, like the other water cooled parts of the furnace, may be made of brass. It is held in place by suitable supporting rods 30 and clamping nuts as shown.

There is supported on the closure 28 within the tube 18 a stand 32 which may be in the form of an inverted cup, and may be filled with insulating material if desired. Upon the stand is supported crucible 34 for receiving the material to be melted.

The stand locates the crucible at the proper position within the length of the induction coil 20 so that its contents are heated by the eddy currents produced by the inductive effects of the current flowing through the coil. Stand 32 and crucible 34 are made of refractory material such as magnesia, and are spaced from the quartz tube by a filling of insulation indicated at 38. 39 indicates a loose fitting or perforated annular cover over the insulation to prevent its entering the mold when the furnace is inverted upon pouring the charge.

In Figure 1, 40 indicates a cover for the crucible 34, having a central aperture 42 alined with aperture 44 in water-coooled closure 46 covering the top of the quartz tube 18. The cover prevents material from being thrown out of the crucible, and limits the loss of heat by radiation. The aperture 42 permits inspection of the melt through the window 48, and also makes it possible to insert addition agents without removing the cover. Aperture 44 is closed by window 48 permitting inspection of the melt. Window 48 is carried by clamping member 47 secured in position by supporting rods 49 and clamping nuts 50.

Clamping member 49 holds the cover 46 tightly closed.

As shown in Figure 1 rods 49 likewise support water jacket 52 surrounding the upper part of the quartz tube 18.

In order to create a vacuum within the furnace, suitable sealing means, such as gaskets 53, must be provided between the quartz tube and the top and bottom closures, and provision is made for exhausting the furnace chamber through pipe 54 which may be connected to any suitable type of vacuum pump.

To permit the introduction of inert gas such as CO_2, into the furnace chamber the bottom closure 28 is provided with pipe 56 discharging into the loose filling 38 of insulation and thence escaping past cover 39 into the upper part of the furnace chamber.

During the melting and degassing of the base metals the furnace will be as shown in Figure 1. At this time the ingredients will be heated by the inductive effect of the current passing through the coil 20, and at the same time vacuum will be maintained within the furnace chamber through suction applied to the pipe 54. In the making of alloys of nickel, barium and copper, or nickel, barium, copper and chromium, the ingredients other than the barium are melted in this manner, and after melting, the temperature is raised several hundred degrees, and vacuum is maintained for a sufficient length of time to thoroughly outgas the material.

The next step consists in cutting off the connection to the vacuum pump and introducing inert gas through the pipe 56. When the entire furnace chamber has been filled with inert gas at atmospheric pressure the top closure 46 is removed, and barium is introduced into the melt in the manner shown in Figure 2; i. e., the barium, covered with pyroxylin lacquer, foil, or the like, to exclude air is secured to rod 58 slidable in a central opening in temporary cover 60, which is now placed over the melt, and the rod is slid downward until the barium is immersed in the melt. Cover 60 is preferably made of asbestos board and is simply laid on. It prevents air reaching the melt through convection currents and confines to the furnace the disturbance caused by the introduction of barium into the melt. It is preferable to add at the same time to the melt a deoxidizer, such as magnesium, the magnesium constituting on the order of one-tenth of one percent of the mixture.

The next step consists in removing cover 60 and placing mold 63, shown in Figure 3, in inverted position over the crucible 34. The mold may be water cooled as shown, if desired. The mold is held in place by means of cover 65 from which it may be spaced by suitable insulating material such as Transite board 67. By tightening nuts 50 on rods 49 the cover and mold are held securely in position. All of this is done with the furnace in upright position.

Thereafter a sufficient time is allowed to elapse to insure the diffusion of the added ingredients throughout the melt, and then the furnace is rotated on the trunnions 14, and the metal is poured from the crucible into the mold 63, where it is allowed to remain until it hardens, whereupon the furnace may be opened and the mold removed.

Alloys made by this process have been found to be quite free of gas so that when used in such devices as vacuum tubes, where even a little absorbed gas is very undesirable, it has proven to be unnecessary to use the long degassing treatment heretofore customary with electrodes of conventional materials. The alloys so made have superior homogeneity and ductility, and exhibit a minimum of oxide inclusions. The product is superior to alloys protected from the atmosphere by slags in that there is no opportunity for silicon to enter the melt. Silicon has been found to be objectionable in its effects on emission of electrons.

It is apparent that this process and apparatus will also be found useful in the making of many alloys and compositions. I have found that the manufacture of common materials such as steel by this method results in a product having very different physical properties and micro-structure from what it would have if made the usual way. It is to be expected that the process here described may find wide application in the manufacture of materials now in wide use in order to obtain the new properties resulting from reduction of gas content. Experiments also indicate that the properties of the metal vary with the degree of vacuum as well as with the degree of pressure to which the material is subjected and these factors can be easily controlled with this process.

I claim:

1. The method of preparing substantially gas-free alloys and the like which consists in melting the base material under vacuum to remove gas therefrom, superheating the melt while continuing the vacuum treatment for a length of time sufficient to remove substantially all of the occluded gases, discontinuing the vacuum treatment and simultaneously surrounding the melt with an atmosphere of inert gas, introducing an addition agent into the melt while so immersed and pouring the melt into a mold immersed in the same atmosphere so as to prevent contamination of the casting.

2. The method of preparing substantially gas-free alloys and the like which consists in melting the base material under vacuum to remove gas therefrom, continuing the vacuum treatment until substantially all of the occluded gases are removed, discontinuing the vacuum treatment and simultaneously surrounding the melt with an atmosphere of inert gas, introducing an addition agent into the melt while so immersed, and pouring the melt into a mold immersed in the same atmosphere so as to prevent contamination of the casting.

3. The method of preparing substantially gas-free alloys and the like which consists in melting the base material under vacuum to remove gas therefrom, superheating the melt while continuing the vacuum treatment for a length of time sufficient to remove substantially all of the occluded gases, discontinuing the vacuum treatment and simultaneously surrounding the melt with an atmosphere of inert gas, and pouring the melt into a mold immersed in the same atmosphere so as to prevent contamination of the casting.

4. The method of preparing substantially gas-free alloys and the like which consists in melting the base material under vacuum to remove the gas therefrom, discontinuing the vacuum treatment and simultaneously surrounding the melt with an atmosphere of inert gas, and pouring the melt into a mold immersed in the same atmosphere so as to prevent contamination of the casting.

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