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DETERMINING GAS CONTENT IN METALS

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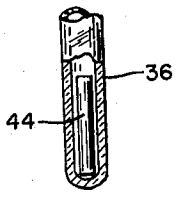


FIG-2

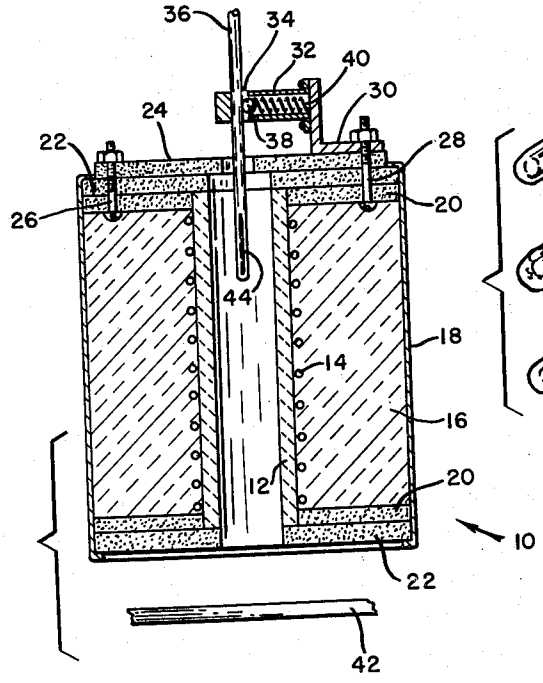


FIG-1

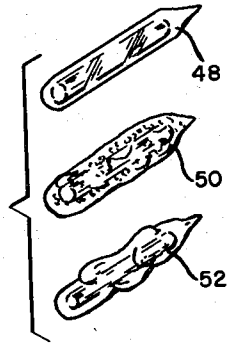


FIG-5

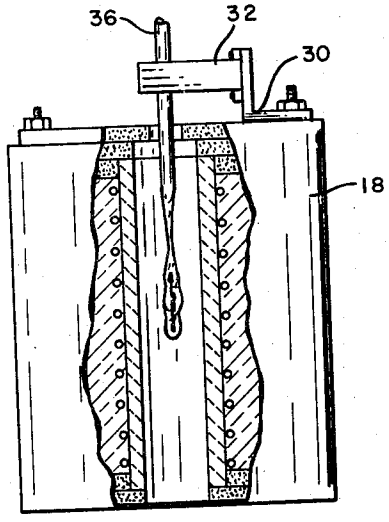


FIG-3

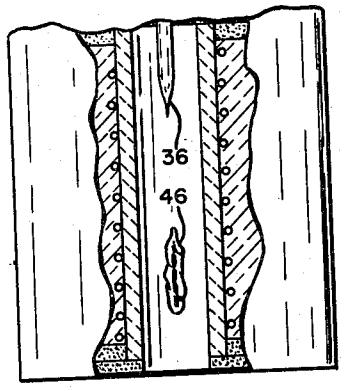


FIG-4

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DETERMINING GAS CONTENT IN METALS

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5 Claims. (Cl. 73—19)

The invention relates to methods for ascertaining the gas content of metals.

It is an object of the invention to provide a process for quickly and cheaply running tests on metals in order to learn, qualitatively, the gas content and to enable such tests to be run by individuals with very little training. Such tests may be performed on metals whether they be in the form of wire, ribbon, strip or tube.

In accordance with the invention a sample of metal to be tested qualitatively for its gas content is placed within a glass tube sealed at only its lower end and heat is applied to the assembly in such fashion that the lower part of the tube fuses before the upper end. As a result the metal is sealed into the glass in a progressive upward zone of travel of the fused glass until finally the entire sample has been enveloped and sealed into the glass, thus forming an ampule of glass tubing with entrapped metal and gas. Due to the application of heat to the assembly, gases contained within the metal will be driven out and some will escape out of the upper end of the tube as the zone of fusing progresses upwardly and some will be trapped in the fused glass, mostly adjacent the metal. The amount of gas evolved depends upon the character of the metal, the temperature to which the metal is subjected, the duration of heat application and other factors. The amount of gas trapped in the glass depends upon these factors and also the softening temperature of the glass. However, for practical purposes, should a comparative qualitative quick test of the gas content of a sample of metal with a standard be desired, it is merely necessary to fix a set of conditions involving the kind of glass used and the temperature of the heat applied. Then ampules will form in about the same time. Ampules with different samples of metal will show, by mere inspection, the relative amount of gas content in the specimens compared to a standard or standards.

For a better understanding of the process and for an exemplification of apparatus which may be utilized to carry out the process, attention is called to the accompanying drawing in which:

Fig. 1 is a cross section through a furnace which may be utilized in carrying out the process and showing a sample of metal in a glass tube ready for being sealed within the tube.

Fig. 2 is an enlarged view of the lower end of the glass tube with metal sample therein, the glass being shown partly in section.

Fig. 3 is a view of the furnace with the sealed sample in a transient state.

Fig. 4 is a similar view showing the sealed sample dropping away from the remainder of the glass tube, and

Fig. 5 shows various samples of completed ampules.

In more detail, at **10** there is indicated an electric furnace having a heat resistant core **12** as of ceramic material. This core is surrounded by a helical electric heating element **14** of uniform pitch, the element being enclosed in an insulating covering **16** of asbestos or the like, all encased in a metal or other suitable jacket **18**.

Hard heat and electrical insulating washers **20** of any suitable material serve to space the ceramic core **12** centrally of the oven and end washers **22** confine the core against longitudinal displacement.

A top plate **24** of material which may be similar to the material of washers **20** and **22** is fastened to the top of the oven as by bolts **26** and **28**, the bolt **28** additionally bolting down an angle bracket **30** to which is secured a horizontal arm **32** with a vertical passageway **34** coaxial with the core **12** and adapted to loosely receive a glass bulb **36** with bottom end closed. A ball **38** is urged against the cylindrical wall of the bulb to hold it in any adjusted vertical position by a coil spring **40** within a horizontal bore in the arm. Any suitable part, such as an asbestos pad **42**, is placed beneath the furnace to arrest metal loaded droppings or ampules as they separate from the tube **36**. The opening in plate **24** is made large enough to easily accommodate the tube **36** but is smaller than the interior diameter of the core **12** to conserve heat within the furnace.

In use a long soft glass tube with closed bottom is inserted into the frictional holding means afforded by the ball **38** and associated parts and is adjusted so that the lower end is near the upper end of the heated furnace, about one and one-eighth inches down into the ceramic tube. A sample of metal, as bar **44**, has either previously been placed in the tube or is now dropped into it. The glass is heated to its softening temperature and collapses around the metal sample progressively from the lower to the upper end. This eliminates the possibility of ambient air being trapped around the sample. Any gas given off by the metal after the glass encloses the sample is trapped in the soft glass. As the glass softens, the tube gets longer, as shown in Fig. 3, allowing the sample to move to a hotter zone in the center of the furnace. Finally, as shown in Fig. 4, when the glass has become so soft that it will no longer support the weight of the sample, the ampule **46** falls out of the furnace onto the pad **42**. The bottom of the tube is tipped off by this operation and another slug of metal may be dropped into the tube; the tube is then lowered to proper position within the furnace. The ampule forming operation will then ensue. In some cases it is desirable to use short tubes, about three inches in length, one for each sample to be formed, since with certain metals condensations from substances given off from previous samples will form in upper cooler portions of a long tube, such as previously described. The choice of glass and of furnace temperature is important. A glass with a high softening point will allow more gas to escape before enclosing the metal than will a glass with lower softening point. Also a high softening point glass requires the sample to remain in the furnace longer than a low softening point glass with consequent greater evolvement of gas before sealing within the ampule. Various furnace temperatures may be used which will result in different time in the furnace for the same glass. It has been found, that at temperatures of about 1100° C. there is very little variation in time in the furnace for the same glass and the time is so short that a number of tests can be made very rapidly. With a potash soda lead glass whose softening point is about 630° C. it has been found that a time of 38 seconds is sufficient to form the ampule and make the test. A borosilicate glass whose softening point is 708° C. requires about 50 seconds to form the ampule; obviously the higher the softening point, the longer the time required to form the ampule. Fig. 5 shows samples of ampules **48**, **50**, and **52** formed with the same glass and temperature but with different metals. Note that ampule **48** shows very little gas, sample **50** shows more of it but with the gas distributed in small bubbles mostly concentrated about the metal sample, while ampule **52**

shows the glass distended by large bubbles of gas. It has been found best, at 1100° C. to use tubing of .146 inch to .152 inch outside diameter with a wall thickness of between .032 inch and .038 inch, with the samples of sufficiently small diameter to fit within the bores of the tubing and about three-quarter inch long. Other tubing sizes may be provided, provided the ampules formed be compared with a standard ampule made from the same size and grade of tubing.

Having thus described the invention, what is claimed as new is:

1. A method for forming glass embedded metal samples from which the gas content of a metal may be determined which comprises vertically supporting a glass tube with closed bottom, placing within the tube a sample of metal whose gas content is to be determined, applying heat to the glass tubing in an upward direction of the tubing to cause the glass to enfold about the metal in an upwardly progressive zone and to eliminate any gasses existing between the glass and metal, continuing this heating until the metal is completely enclosed within the glass, and maintaining the application of heat to the tubing to enable gasses evolved from within the metal to be occluded within the glass.

2. A method for forming glass embedded metal samples from which the gas content of a metal may be determined which comprises vertically supporting a glass tube with closed bottom, placing within the tube a sample of metal whose gas content is to be determined, applying heat to the glass tubing in an upward direction of the tubing to cause the glass to enfold about the metal in an upwardly progressive zone and to eliminate any gasses existing between the glass and metal, continuing this heating until the metal is completely enclosed within the glass, and maintaining the application of heat to the tubing to enable gasses evolved from within the metal to be occluded within the glass, and until the weight of softened glass and metal is sufficient to cause separation of the glass enclosed metal from the remainder of the glass tubing.

3. A method for forming glass embedded metal samples from which the gas content of a metal may be determined which comprises vertically supporting a closed bottom glass tube in a first region, applying heat at that region to the glass and the metal therein sufficient to cause the glass to soften, enfold about the metal to expel any gas

between the glass and metal and to elongate so that the softened portion may by gravity move to a second and hotter region, and continuing the application of heat at said second region until the glass has completely embedded the metal and gases from the metal have permeated the glass and until the glass has become sufficiently soft to separate, by reason of its own weight and that of the contained metal, from the remainder of the tube.

4. A method for forming glass ampules containing embedded metal samples and amounts of air which comprises supporting a closed bottom glass tube with its lower end enclosed within the upper end of an open bottomed furnace, the temperature of the furnace being less at the upper end than further down, inserting a metal sample less in height than the length of the tube within the tube and maintaining the closed bottom tube in the furnace until an ampule is formed completely enclosing the metal sample, with gas from the metal sample occluded within the glass and until the ampule drops off from the remainder of the tube and out of the furnace.

5. A method for forming glass ampules containing embedded metal samples and amounts of air discharged from said samples which comprises supporting a closed bottom glass tube within a heated atmosphere, creating a hotter atmosphere at a level below said first heated atmosphere, inserting a metal sample less in height than the length of the tube within said tube and maintaining the closed bottom tube in said first atmosphere until an ampule is formed completely enclosing the metal sample, maintaining the first atmosphere while the ampule with enclosed sample moves to the hotter atmosphere and finally drops off from the remainder of the tube and out of the regions of the heated atmospheres, said ampule accumulating within the glass any gases evolved from the metal subsequent to its envelopment by the ampule.

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