Oct. 27, 1964 J. E. LINDBERG, JR 3,153,847

METHOD OF MAKING HEAT SENSORS

Filed Aug. 2, 1963 2 Sheets-Sheet 1

Fig. 1

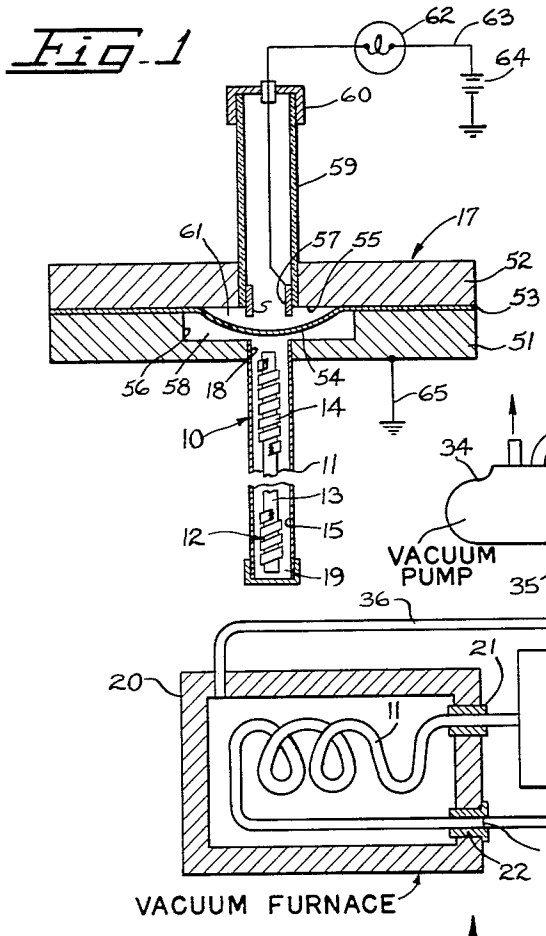


Fig. 2

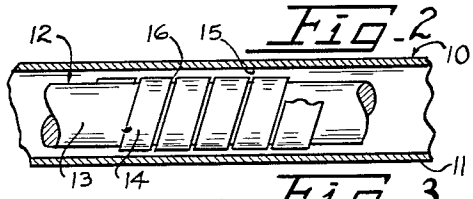


Fig 3

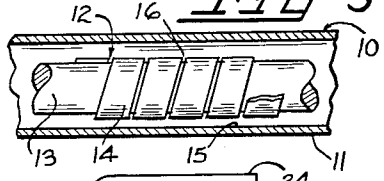
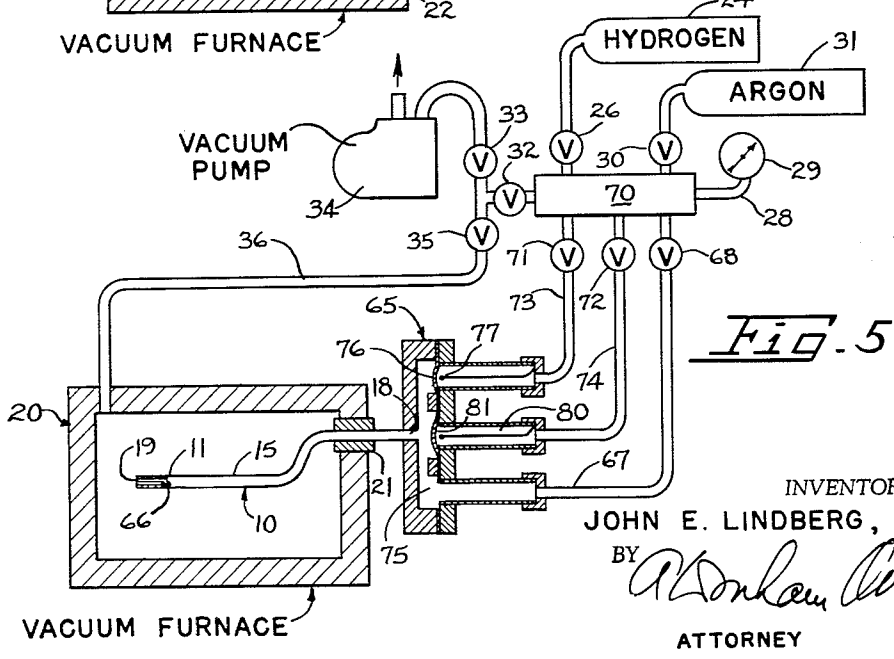


Fig. 4

Fig. 5



INVENTOR.
JOHN E. LINDBERG, JR.
BY
ATTORNEY

Oct. 27, 1964 J. E. LINDBERG, JR 3,153,847

METHOD OF MAKING HEAT SENSORS

Filed Aug. 2, 1963 2 Sheets-Sheet 2

Fig. 6.

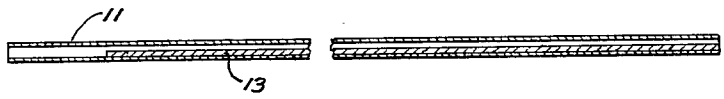


Fig. 7.

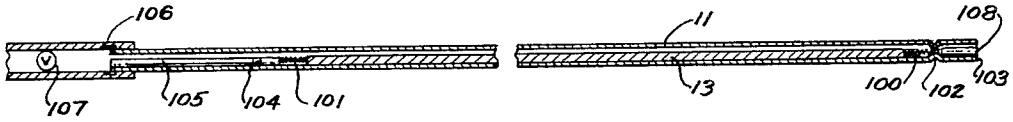
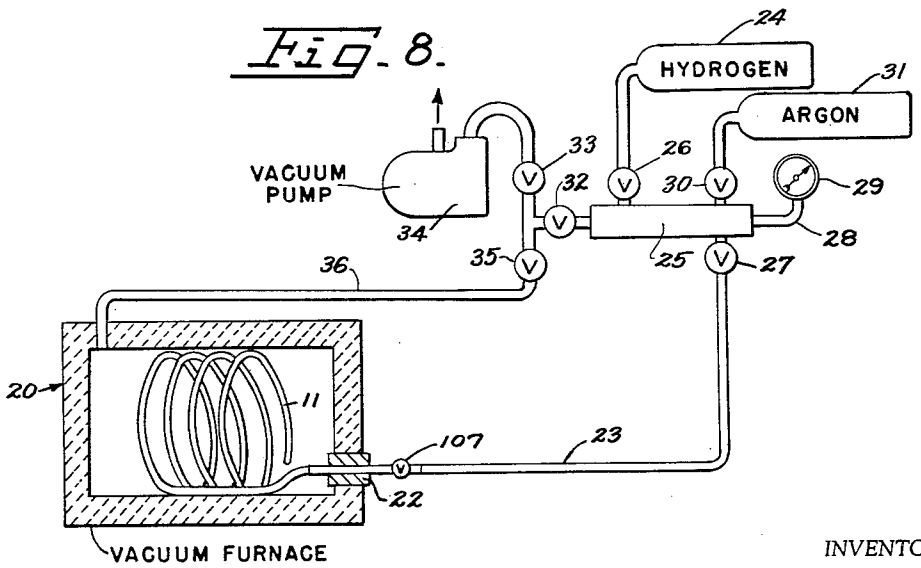


Fig. 8.



INVENTOR.
JOHN E. LINDBERG, JR.
BY
A. Donham Owen
ATTORNEY

United States Patent Office

3,153,847
Patented Oct. 27, 1964

3,153,847

METHOD OF MAKING HEAT SENSORS

John E. Lindberg, Jr., 1024 Adrienne Drive, Alamo, Calif.

Filed Aug. 2, 1963, Ser. No. 299,672
37 Claims. (Cl. 29—400)

This application relates to a method of making heat sensors, especially heat sensors of the type disclosed and claimed in my application Serial No. 815,406, filed May 25, 1959, now U.S. Patent No. 3,122,728, issued February 25, 1964. This application is a continuation-in-part of application Serial No. 180,121, filed March 16, 1962, now abandoned, which was a continuation-in-part of application Serial No. 66,221, filed October 31, 1960, now abandoned, which was a continuation-in-part of application Serial No. 815,406.

Such a heat sensor comprises an enclosure, preferably a narrow-diameter metal tube of constant cross-sectional area and of any desired length, within which is a metallic hydride, there being means to provide passage for the hydrogen gas that is emitted from the hydride upon the sensor's being subjected to certain temperatures. The emission or taking up of the hydrogen acts to vary the pressure inside the enclosure. The enclosure is gas-tight and its only opening is connected to a device called a responder, which itself defines a closed chamber connected only to the sensor enclosure. An alteration of the internal pressure within the enclosure therefore affects the responder, which then acts on an electrical circuit or other device to actuate a signal.

An object of this invention is to provide a method for making a completely hermetically sealed heat-detection transducer, completely free from environmental errors caused by such things as pressure and altitude changes, moisture condensation, and so on.

A difficult problem is to provide a method for making sensors which will assure that the sensor will give accurate results, and will give the same results each time, over and over. Another object of the invention is to solve this problem.

Other objects and advantages of the invention will become apparent from the following description of a preferred example.

In the drawings:

FIG. 1 is an enlarged view in elevation and in section of a simplified form of heat-detection device, the sensor thereof being broken in the middle in order to conserve space.

FIG. 2 is a greatly enlarged view in elevation and in section of the sensor in its fully ingassed state.

FIG. 3 is a view similar to FIG. 2 showing the same sensor in its fully outgassed state, or as it is before the metal is ingassed with hydrogen.

FIG. 4 is a diagrammatic view of apparatus that may be used in performing the method of this invention.

FIG. 5 is a view similar to FIG. 4 of a modified form of apparatus.

FIG. 6 is a somewhat diagrammatic view of the sensor (broken in the middle and mostly omitted there, to conserve space) with its thickness greatly exaggerated to illustrate one stage of treatment according to one form of the invention.

FIG. 7 is a view like FIG. 6 showing a later stage of the same process.

FIG. 8 is a view like FIG. 4 illustrating a modification of that method.

The invention deals with the use of certain metallic hydrides as transducing agents, for release or emission of large volumes of gases or vapors when elevated to a temperature sought to be detected. When these metallic hydrides are enclosed in a constant-volume container or enclosure and subjected to temperature changes, the resultant alteration of pressure within this container or enclosure is employed to actuate the responder.

With the elements of Groups III–B (including the rare earth and actinide elements), IV–B and V–B, hydrogen forms pseudohydrides. Elements of these groups compose the class consisting of scandium, titanium, vanadium, ytterbium, zirconium, niobium, hafnium, tantalum, the rare earth metals (atomc numbers 57 through 71), and the actinide metals (atomic numbers 89 through 103). The solubility of hydrogen in elements of these groups varies as the square root of the pressure, and it decreases with increase in temperature. This solution is commonly termed a "hydride," though it is not a stoichiometric compound. Examples of the sorptive capacities as a function of temperature of some of these materials are given in Table I.

TABLE I

Sorption of Hydrogen by Certain Metals

[In cm.^{3} (S.T.P.) per gm. at 1 atm.]

Temperature, ° C.	Titanium	Vanadium	Zirconium
20	407.4	150	235.5
400	387.7	38	
600	334.7	10	184
800	140.9	4.4	165
1000	66.1	2.5	78

FIGS. 1 to 3 illustrate how a sensor **10** may be constructed. A sensor tube **11** may be a non-porous tube of constant cross-sectional area. In applications where the tubes **11** are to be bent or curved around corners, metal is the preferred material. Suitable metals are nickel, stainless steel, other nickel alloys, and molybdenum, for example. In applications where bending is not required and minimum diffusion is desired, the tube **11** is preferably made from non-porous quartz or ceramic. In any event, the inner surface of the tube **11** should not react with the materials it contacts, including the gas involved. Where the tube **11** is reactive with the metallic hydride transducing agent **12**, a special problem is created, which I solve as described below. A typical sensor tube **11** is preferably about 0.040" to 0.060" outside diameter with a wall thickness of preferably about 0.005" to 0.015". Such tubes **11** are preferably about two to forty feet long, although they may be longer or shorter.

FIGS. 2 and 3 show a sensor **10** having a preferred form of transducing agent **12** enclosed in the sensor tube **11**. Here the transducing agent **12** is a filament **13**, such as zirconium wire about 0.005" to 0.030" in diameter, for example. The filament **13** may be titanium, vanadium, tantalum, or other of the previously mentioned materials. The more nearly the filament **13**, when ingassed,

fills the tube **11**, the less the remaining space; hence the greater the increase in pressure when the same quantity of hydrogen is emitted at any temperature. Preferably, a ribbon **14** of suitable material, such as molybdenum or tungsten, preferably about 0.020" wide and 0.002" thick, is wrapped tightly around the filament **13** and fits snugly within the tube **11**. The ribbon **14** physically spaces the filament **13** from the walls **15** of the tube **11** and prevents certain transducing agents **13** such as zirconium and titanium from fusing or welding to the tube walls **15**, even when the sensor **10** is exposed to extreme heat.

A typical example of the sensor **10** embodies a stainless steel tube **11** containing a zirconium or titanium wire **13** separated from the tube walls **15** by a thin closely spiral-wound ribbon **14** of molybdenum or tungsten.

SENSOR CLEANILINESS

In order to get consistent results, the sensor interior must be very clean; the interior walls of the tube **11**, the wire **13**, and the ribbon **14** must be free from oxide coatings, from grease, from reactive gases such as oxygen, and from other impurities. The initial steps taken in this invention are taken to insure such cleanliness. These steps include one or more of the following: wire pickling, degreasing, purging, evacuation, and heat treatment.

Raw zirconium wire—even though of a high quality and substantially chemically pure—tends to become coated, upon exposure to the atmosphere, with some zirconium oxide. One way of treating it is to wash the zirconium wire **13** in a dilute solution of hydrofluoric acid, which gets rid of most but not all of the oxide; then the wire **13** is washed and rinsed. The process should in this instance proceed to the succeeding steps without delay so that no oxide coating will be renewed by contact with the atmosphere.

Another way of preparing the sensor essembly starts with clean, annealed, pickled ductile zirconium (or titanium) wire **13**, preferably of the purest grade and preferably about 0.010" in diameter.

The clean wire is closely wrapped with a ribbon **14** of molybdenum or tungsten, preferably pure and preannealed at 1900° F. for one hour. The ribbon **14** may be closely wound around the wire **13**, for hydrogen is extremely mobile and will find its way out via the spiral passage **16** between successive parts of the ribbon even though that passage **16** seems to be non-existent to the eye. Tight winding prevents particles of the wire **13** from breaking off and fusing to the tube wall **15**. At each end the ribbon **14** is cut off roughly in order to leave a little to cover the ends of the wire **13**.

The wrapped titanium or zirconium filament **13** may then be spot welded to a draw wire and drawn into the sensor tube **11**. Alternatively, the filament **13** may be pushed into the tube **11**, as by a conventional wire feeding device.

Degreasing may be done by submerging one end of the tube **11** in acetone (which may be in a bottle), and applying compressed air to the surface of the acetone to blow the liquid acetone through the tube **11**, flushing it out and degreasing the wire **13**, ribbon **14** and the tube **11**. Other degreasing solvents that do not react with the metal involved may be used. Also, instead of flushing after insertion of the filament **13**, the tube **11** may be flushed with acetone before inserting the wrapped filament **13**, the wrapped filament being degreased before insertion by immersion in acetone (or other degreasing solvent).

Preferably (as illustrated diagrammatically in FIG. 6, where, unfortunately, thickness had to be exaggerated with respect to length), the tube **11** (typicaly five to forty feet long) is about four to six inches longer than the wrapped filament **13**, and the filament **13** is preferably inserted flush with one end of the tube; then the tube **11** is preferably stretched about four inches—about two inches at each end, the end that was formerly flush then extending about ten inches beyond the filament **13**. Then, as illustrated in FIG. 7, at each end of the tube **11** a short piece **100**, **101** (e.g., 1" long) of molybdenum wire or twisted strands thereof is inserted snugly against the filament **13**, to insure that there be no place where the wire **13** or particles thereof could come into contact with the tube **11** (such precautions are not necessary with some materials, such as vanadium). At the formerly flush end, the walls of the tube **11** are then crimped at **102** around the end of the added one-inch length **100** of molybdenum wire, about one inch from the end **103** of the tube **11**. In the other end, a one or two inch length **104** of high-temperature brazing wire, such as Cuplat, is inserted loosely, and then a four-inch length **105** of molybdenum wire. This end **106** may now be brazed to a valve **107** that is used only during processing and is later detached.

In order to assure stable operation of the completed fire detector, all atmospheric gases and vapors, which during their construction entered the sensor **10** (and the chamber in the responder **17** to which the sensor **10** is connected) are removed before forming the hydride. Preferably, this purging is done by connecting the processing valve **107** to a tank of hydrogen, and the tube **11** is flushed at room temperature with hydrogen under pressure (about 25 p.s.i.a. to 250 p.s.i., preferably 125 p.s.i.) for ten minutes or so, at least five minutes. During this time, the acetone apparently also acts to carry away moisture with it, thereby helping to prevent oxidation when the temperature is later raised. This purging (which may, if desired, be done with argon instead of hydrogen, though hydrogen is preferred generally) is not always required, but it does help and adds a higher degree of certainty to the process.

This purging may be done as shown in FIGS. 4 and 8 by putting the sensor **10** into a vacuum furnace **20** and purging it there with the hydrogen. FIG. 4 shows the responder **17** mounted outside a vacuum furnace **20** with the sensor **10** extending into the furnace through a vacuum-tight fitting **21**. The open end **19** of the sensor tube **11** (the valve end, if the valve is on) is brought out from the furnace **20** through another vacuum-tight fitting **22** and is connected then to a tube **23**, which may be connected directly to a cylinder **24** of hydrogen, or may, as shown, be connected to a manifold **25**, which in turn is connected to the cylinder **24** through a valve **26**. FIG. 8 shows the entire sensor **10** with no responder attached, connected to the tube **23** through the valve **107**. There is also a valve **27** between the tube **23** and the manifold **25**. The manifold **25** is connected (1) through the valve **27** and the tube **23** to the tube **11**; (2) through a tube **28** to a pressure/vacuum gauge **29**; (3) through a valve **30** to a tank **31** of argon; (4) through the valve **26** to the tank **24** of hydrogen; (5) through valves **32** and **33** to a vacuum pump **34**; and (6) through valves **32** and **35** and a tube **36** to the vacuum furnace **20**.

With this apparatus the system may be purged at room temperature with hydrogen, as follows: The valves **30** and **32** are closed, and the valves **26** and **27** are opened to send hydrogen into the tube **11** at a pressure of preferably about 125 p.s.i. (or 50 to 250 p.s.i.) as indicated by the gauge **29**.

For processing according to FIG. 4, the connection between the valve **107** and the tube **11** may be cut, or the tube **11** otherwise detached, and a responder **17** is brazed thereto, as by a Cuplat braze in a hydrogen furnace. However, it is often preferable to leave the valve **107** on for a few more steps, as will be explained below in connection with FIG. 8. If it is left, and while the hydrogen is flowing, a brazing wire **108** (e.g. a one-inch length of Cuplat) is inserted in the formerly flush end **103** (see FIG. 7), and, turning off the flow of purging gas, that end **103** is brazed closed at once to help to keep the surfaces inside the tube **11** free from oxides. The brazing may be done

in an argon atmosphere. Then the seal at the wire **108** may be checked by passing hydrogen into the other end of the tube **11** at 250 p.s.i., to make sure the tube end **103** is sealed.

After purging, the valve **26** is closed and the valves **32** and **33** are opened, and a vacuum, preferably of about one micron of mercury is drawn on the sensor tube **11**. If desired, purging and this operation may be repeated several times in order to assure the sweeping of any remaining gases and vapors which might have been trapped on the surfaces of the sensor tube **11** or the transducing agent **12**. If purging is omitted, special care should be taken to draw a good vacuum.

While maintaining in the tube **11** the pressure at a vacuum, preferably of about one micron (0.001 mm.) of mercury, the furnace **20** of FIG. 4 or FIG. 8 is then heated slowly (e.g., over a 30 or 40 minute period) to a top of about 1900° F. to 2200° F. (or as hot as the brazing wire can go without melting; if Cuplat is used, 2150° F. is the maximum safe temperature) and held at that temperature about 30 minutes. Time may be saved by doing several tubes at once. If the responder **17** is attached, it is also heated at the same time, though usually not quite as hot, depending on the materials from which it is made. This heating cleans up the tube **11** and also apparently dissolves the remaining filament oxides into the filament metal.

INGASSING WITH HYDROGEN

Cleanliness having been assured by the removal or neutralization of impurities, according to the preceding steps, we come to the actual preparation of the hydride by causing the filament **13** to ingas hydrogen. The metals of the zirconium-titanium type do not readily take in hydrogen at room temperature; I have found that it is important to ingas them by starting at a relatively high temperature—e.g., 1500° F. to 1800° F. for zirconium—and cooling them in an atmosphere of hydrogen under pressure to a temperature preferably below a threshold temperature, where the metal substantially ceases to take in gas. The threshold temperature differs from metal to metal, and it also varies according to the hydrogen pressure at that temperature and the amount of hydrogen remaining in the metal. These variations call for careful treatment in order to obtain reliable, completely repeatable results.

For example, with the sensor **10** in the furnace **20** at 1900° F. to 2200° F., I prefer to cool the tube **11** to a starting ingassing temperature of about 1500° F. to 1850° F. for zirconium wire **13** (to about 1200° F. to 1500° F. for titanium wire **13** and to other temperatures for different materials), and hydrogen from the valve **26** is then added at about 50 to 250 p.s.i.g., e.g., 150 p.s.i., as indicated by the gauge **29**. This pressure level of hydrogen is then maintained while lowering the temperature slowly (e.g., over about an hour and a half) in the furnace **20** below 1200° F., e.g., about 550° F. for zirconium and there or even to room temperature for titanium. Under these conditions, the threshold temperature of zirconium is about 660° F. (650° to 675° F., depending on the exact pressure conditions etc.). The cooling to 550° F. is a precaution to insure a complete, and actually in some ways an overcharged, ingassing. Under these conditions of temperature and pressure, the zirconium filament **13** ingasses and becomes a hydride, as explained above, enlarging in size as indicated by the change from FIG. 3 to FIG. 2. The heating and cooling may be repeated several times, to assure stability later. Then the hydrogen valve **26** is shut off.

As an alternate method, the temperature may be lowered from the 1900° F. to 2200° F. cleaning temperature to about room temperature, and at a later time, preferably within a day or so, hydrogen put into the tube **11** at about 200 p.s.i.g. In this instance the furnace **20** is then heated above 1200° F., e.g., to about 1200° F. to 1500° F. for titanium, say, or 1500° F. to 1800° F. for zirconium, and cooled, as before below the threshold temperature, to cause the filament **13** to ingas the hydrogen, the cycle preferably being repeated several times.

With the furnace **20** then below the threshold temperature of the hydride, the excess hydrogen is pumped out from the sensor tube **11**, preferably by pumping it out to about one micron of mercury by means of the pump **34**, as the tube cools from say 550° F. slowly to about 250° F. or 300° F. or even to room temperature. This may take several hours or even overnight. Preferably, when the pump **34** is disconnected from the sensor **10** or turned off, the interior of the tube **11** is given a charge of helium or argon or other noble gas from the tank **31** and valve **30**, the charge preferably being greater than atmospheric, e.g., 20 p.s.i.g. This helps to prevent leaks from outside in. (For some uses a hydrogen overcharge is desired; so in those circumstances the hydrogen is pumped out to a predetermined pressure level which may be greater than atmospheric.)

After putting in the helium or argon charge (or obtaining the desired hydrogen overcharge), the valve **107** is closed and the sensor **10** removed from the vacuum furnace. Then, to condition the hydride and help to assure repeatability later on, I prefer to pass the sensor **10** slowly through a flame or other hot zone of, say, 1500° F. It may be fed through the flame at about three or four feet per minute. While once is adequate, it may well be done several times. Care is taken not to let the tube **11** get too hot; a dark or cherry red heat is good, bright red is usually too hot. The cooled sensor **10** may then be stored indefinitely, if desired, or processing may continue.

ADDING THE RESPONDER AND THE CHARGE OF INERT GAS

It is now time to connect the sensor **10** to its responder **17**, at its end **18**, if it has not been added earlier, as in FIG. 4. The responder is preferably made from molybdenum, or tungsten. A "Cuplat" (copper platinum) alloy having a melting point of 1250° C. and a freezing point of 1200° C. may be used as the brazing material. When using the procedure illustrated in FIGS. 6–8, the tube end **103** and braze wire **108** are cut off beyond the crimp **102** and the responder **17** brazed thereto, the crimp **102** remaining to prevent relative displacement of the filament **13**.

Then the sensor **10** is put into an air furnace (like the furnace **20** in FIGS. 4 and 8) and heated there to a temperature near or at a desired all-point operating temperature. ("All-point" operation is based on a charge of inert gas such as argon; "discrete" operation is obtained from the hydride.)

Since the responder interior carries a little atmospheric gas, usually, and in order to clean this out, the system is again evacuated through the valve **107**. Of at least equal importance, this step also removes any overcharge of hydrogen that may have resulted from the flame treatment and is mixed with the helium or argon, assuring that the sensor is now stabilized and will thenceforward act uniformly. (If the responder **17** was attached previously, this step may in some instances be omitted.)

Hydrides appear to take in hydrogen in different amounts that depend on the hydrogen partial pressure and the hydrogen content already present. The action is almost like a change in state in that it acts in stages, alpha, beta, and even gamma stages being recognized in some metals. When treated according to this invention, the hydride first formed is apparently in a beta or gamma state which is overcharged and not repeatable after taking out the excess hydrogen, or indeed unrepeatable except at high hydrogen pressures. Maintenance of such high pressures of hydrogen in the tube **11** is not feasible. The minute hydrogen molecules seep out through the tube walls, when the tube **11** is heated in tests or in operation, passing between the metal molecules or crystals.

Hence, such hydrogen high over-pressure should be removed for several reasons: if left, the pressure will drop after each operation, and the all-point operating temperature of the sensor will change, the gradual depletion of the hydrogen overcharge will affect the ability of the metal to reingas its hydrogen after each heating, changing the discrete point operating temperature, and the responder **17** is preferably made to operate at a relatively low pressure such as two atmospheres. By removing all hydrogen from the tube atmosphere before passing the sensor through the flame and then removing from the sensor the hydrogen that fails to reingas, one finally gets an atmosphere having no significant amount of hydrogen; thence, the filament **13** ingasses and outgasses uniformly. The objective is to get the hydride where it reingasses the free outgassed hydrogen down to a hydrogen partial pressure of substantially zero, so that the free hydrogen does not affect the "all-point" operation of the added argon. Hence, the "discrete" operating temperature and the "all-point" operating temperature are both made invariable and stable. It can now be seen how important this stabilization is, by whatever combinations of time-temperature and vacuum pumping steps it is achieved.

The vacuum pump **34** is used for this evacuation, and once the desired pressure level—e.g., one micron—is reached, the pump **34** is stopped, and the valve **30** used to introduce argon at a very slow rate. The argon preferably contains 5% helium for use in checking for leaks in a helium leak detector. The sensor **10** is kept at a desired set point, e.g., 310° F., and argon is admitted until the responder diaphragm snaps to its actuated position. Then the argon valve **30** is shut off, and the valve closed, and the sensor again heated to see whether it operates at the same point. Then the sensor is taken out of the furnace and checked by the discrete test, e.g., by exposing a twelve-inch length in an open flame; it should not come on at (say) 750° F. but should come on within one minute at, say 850° F. Three places are usually checked—e.g., at two feet from each end and at the middle. These tests also help to check stabilization of the operation of the sensor, so that results from then on are known to be repeatable and identical. After being left overnight for recovery, it may be tested again. If satisfactory, the process is complete. If it does not operate satisfactorily, it is pumped out and reset. Sealing with Cuplat may result in a change of its operating temperature, adjusting it to the operating point by shortening the length of the tube; such effects may easily be compensated for in advance. The seal may be checked at 500 p.s.i. or other high pressure.

The preferred procedure just described is not the only one feasible, of course. The invention is capable of many variations. For example, in determining the discrete or hydrogen operating point, the operating point of the responder **17** may be adjusted by holding the sensor **10** at that desired temperature and removing hydrogen until there is just enough to effect the desired operation and then cooling the sensor **10** to enable that amount of hydrogen to become ingassed into the filament **13**. Or experience tables can be compiled and the hydrogen excess withdrawn at a different temperature but in an amount shown by the experience tables to accomplish the same thing as the desired removal at the operating temperature. Thus, there is then a controlled vacuum in the tube **11**. The sensor **10** is now tested by heating it and seeing whether the responder **17** operates properly. If not, the hydriding process is repeated before going on to the next step. In fact, there need be no next step for some applications, except for sealing the tube end **19**. But for many uses, it is preferred to add the overcharge of inert gas.

This overcharge of inert gas may be added in an alternative manner also. With the furnace **20** cooled to a desired temperature (unless it is at that temperature), the noble gas, such as argon, may be added from the cylinder **31** at from 10 to 25 p.s.i.a., preferably above 15 p.s.i.a. It may be added at the desired "all-point" operating temperature, say 600° F. or 400° F. or 300° F. in an amount that barely achieves operation of the responder **17**. Or a chart may be used to determine the room temperature and pressure corresponding to the actuating pressure at any desired temperature, and that amount is then added at room temperautre. The sensor is preferably then tested again for temperature response, as by passing it through a 1500° F. frame, or at any event above the transition point of the hydride.

OPERATION

Let us now consider the operation of a responder, to see how the finished product works.

FIG. 1 shows a responder **17** comprising two circular plates **51** and **52**, preferably of non-porous metal, between which is bonded (as by brazing) a thin metal flexible disc or diaphragm **53**. The plates **51** and **52** are hermetically sealed together and are in electrical contact for their full peripheries and over a substantial margin, but in the center the diaphragm **53** preferably has a spherical depression **54** called a "blister," which is free to move relative to the plates **51** and **52** and constitutes the active or movable part of the diaphragm **53**. Use of a diaphragm with a blister **54** makes possible the use of an upper plate **52** with a planar lower surface **55** and gives a more predictable response, but other diaphragm structures may be used where feasible. The lower plate **51** is formed with a recess **56** in its upper surface.

The force necessary to deflect the blister **54** against an electrode **57** can be chosen to accommodate a wide range of values by a suitable choice of mechanical parameters. Once this force is determined, the dimensions of the sensor tube **11** and the amount of transducing agent **12** may be chosen by design to provide the force necessary to obtain contact between the blister **54** and electrode **57** at a certain temperature.

In addition to mechanical design consideration, the necessary deflecting force may also be altered by precharging the anti-sensor chamber **58** with a gas under pressure or by partially evacuating it. To accomplish this, gas is forced into (or withdrawn from) the tube **59** after its attachement to the plate **52** and before it is closed by its cap **60**. The required deflecting pressure against the blister **54** becomes greater as more gas is present in the chamber **58**.

Alternatively, the deflecting pressure may be effectively lowered by precharging the inside of the sensor tube **11** and the sensor chamber **61** with gas. In this case, if the ambient pressure in the sensor chamber **61** is greater than normal, less than normal gaseous emission from the transducing agent **12** is required to deflect the blister **54** against the electrode **57**.

More gases may be employed for this purpose; however, ideally the gas should not react chemically with its surrounding materials. Particularly suitable are the inert gases, such as helium, argon, neon, and xenon, especially since they do not readily diffuse through most materials. As a consequence, a precharged pressure of argon, for example, may be maintained for an indefinite length of time to retain a desired biasing of the diaphragm **53**, as described.

The responder **17** may be connected to an alarm circuit which, as shown in FIG. 1, is a simple visual indicator consisting of a lamp **62** in series with the conducting wire **63** and a source **64** of electrical current, which may be a battery, as shown, or may be a source of alternating current. A return path for the electrical circuit may be provided by grounding either one of the plates **51** or **52** and is shown as a ground wire **65**.

In operation, when the sensor **10** is exposed to heat at a level high enough to cause the argon gas to expand and increase in pressure, in the sensor chamber **61**, it

exerts pressure upon the blister 54. This pressure tends to move the blister 54 away from the plate 51 and toward the plate 52. The pressure in the sensor chamber 61 is a function of the temperature of the sensor 10, and in general there will be a one-to-one correspondence between the temperature of the sensor 10 and the pressure within the sensor chamber 61. This pressure, if great enough, will cause the blister 54 to make contact with the electrode 57, but no contact will be made unless the temperature of the sensor 10 is at or above a definite level.

A similar action takes place at a higher temperature when a portion of the transducing agent is heated above its threshold temperature and emits hydrogen. Thus, in this embodiment, the argon serves for actuation at a predetermined *over-all* temperature of the tube 12, and the hydride causes actuation at a predetermined *spot* temperature.

When the sensor 10 is exposed to heat at a level high enough to cause the blister 54 to make contact with the electrode 57, current flows from the battery 64 through the lamp 62, the conductor 63, the electrode 57, and the blister 54 to the plates 51 and 52 and returns to the battery through ground line 65. This current flow causes the lamp 62 to light and provides a visual indication that the temperature of the sensor 10 is at or above a certain level. In this sense, the device shown in FIG. 1 functions both as an all-point and as a threshold temperature indicator. When heat is removed from the sensor 10, the transducing agent 12 cools and reabsorbs its previously emitted gas, resulting in reduction of the pressure exerted upon the blister 54. The blister 54 moves away from the electrode 57, breaking the electrical circuit, and the lamp 62 goes out.

In practice, the sensor 10 is placed in the area whose temperature is to be monitored, while the responder 17 may be located upon or behind a shielded wall or at some easily accessible area. Thus only the sensor 10 itself need be exposed to possible heat sources, and it contains no element of the electrical circuit. In this manner, protection for the responder 17 and its associated alarm circuit may be provided.

The preparation of a unit having a responder 65 more complex than the responder 17 is shown in FIG. 5 by way of example. The basic method remains the same whatever type of responder is used but there are of course some changes in procedure.

In this process, a short piece, perhaps one inch long, of brazing wire 66 is initially inserted into the open end 19 of the sensor tube 11. The brazing wire 66 may be made of Cuplat (melting point 1250° C.) or of Nioro, an alloy of nickel and gold (melting point 950° C.). The diameter of the wire 66 should be chosen so that the wire fits snugly in the sensor tube 11. The wire 66 will be used later to seal the end 19 of the sensor tube 11 at the appropriate time.

FIG. 5 shows the responder 65 mounted outside the vacuum furnace 20 with the sensor 10 extending into the furnace through the vacuum-tight fitting 21. (The fitting 22 is plugged.) The end 19 of the sensor tube 11 is left free in the furnace 20, while the other end 18 of the sensor tube 11 is connected to the responder 65, through which a passageway extends to a tube 67 and a valve 68 to a manifold 70. The manifold 70 is connected (1) through valves 68, 71 and 72 and tubes 67, 73, and 74 to the responder 65; (2) through the tube 28 to a pressure/vacuum gauge 29; (3) through the valve 30 to the tank 31 of argon; (4) through the valve 26 to the tank 24 of hydrogen; (5) through the valves 32 and 33 to the vacuum pump 34; and (6) through the valves 32 and 35 and the tube 36 to the vacuum furnace 20.

With the valves 30 and 26 closed and all other valves open the system is evacuated at room temperature (15 to 25° C.), to about one micron of mercury by means of a vacuum pump 34. Then, the system is purged at room temperature with hydrogen, as follows: The valves 71, 72, and 32 are closed, and the valve 26 is opened to maintain a pressure of about fifty p.s.i.g. in the manifold 70, as indicated by the gauge 29. Hydrogen flows through the valves 26 and 68 and the tube 67 into the responder 65 and thence into the sensor tube 11. Flowing through the sensor tube 11, the hydrogen sweeps away with it any remaining gases and vapors which might have been traped on the surfaces of the sensor tube 11 or the transducing agent 12. Finally, the hydrogen seeps between the brazing wire 66 and the wall of the sensor tube 11 into the vacuum furnace 20 and is pumped away through the tube 36 and valves 35 and 33 by the vacuum pump 34. This purging is repeated several times, ending with the system evacuated, the pressure being about 0.001 mm. (i.e., one micron) of mercury, or less. While maintaining the pressure of 0.001 mm. of mercury in the system, the furnace 20 is then heated to about 2200° F. and the process from then on is basically like that already described.

The heat in the furnace 20 melts the brazing wire 66, and when the furnace 20 is cooled to about 1700° F., the melted brazing wire 66 solidifies and seals the end 19 of the sensor tube 11.

Another step that may be taken later is to remove the sensor 10 from the vacuum furnace after the hydride is ingassed and put it in an air furnace (not shown). The purpose of removal from the vacuum furnace to the air furnace is as follows: the tube walls 15 are at this stage somewhat permeable to hydrogen, and hydrogen could therefore be pulled through them by the vacuum pump. By putting the tube 11 in an air furnace and heating it back up to about 800° F. an exterior coating is formed on the outer wall of the tube 11, and this coating is substantially impermeable to hydrogen, so there will be no seepage through the barrier formed by the coating resulting from reaction with the air. This step may come before adding the inert gas.

When the inert gas has been added, the valve 30 is closed, and the chamber 75 closed, as by squeezing the tube 67 closed. This operation simulates a fire condition by pressurizing the sensor 10 as it would be pressurized in an actual fire. The valve 68 is then closed in order to maintain the correct pressure in the sensor 10. Since a simulated fire condition exists in the sensor 10, the responder 65 should be at the point of just responding to the fire condition; i.e., the diaphragm blister 76 should be just touching the contact 77. If it is not, then the pressure in the chamber 75 is adjusted, either by opening the valve 30 and admitting more argon, or by opening the valve 32 and pumping out some argon, until the diaphragm blister 76 is at the point of just touching the contact 77. (This point may be indicated by attaching an ohmmeter or by a light or by another suitable low-current indicating device between the diaphragm 76 and the contact 77.) Once the pressure in the chamber 75 is correct for the fire detector to respond to the selected temperature, the chamber 75 is sealed by some suitable means, such as squeezing the tube 73 with an appropriate tool.

The air furnace is then cooled to the temperature selected for overheat response and a procedure similar to that just outlined is followed, to provide the correct pressure in the chamber 80 behind the other diaphragm blister 81, thereby completing the processing of the fire detector of FIG. 5.

To those skilled in the art to which this invention relates, many changes in construction and widely differing embodiments and applications of the invention will suggest themselves without departing from the spirit and scope of the invention. The disclosures and the description herein are purely illustrative and are not intended to be in any sense limiting. For example, the responder may also be put into the vacuum furnace; or the sensor may be heated electrically in a vacuum

by using the resistance of its own tube walls, without using a furnace.

I claim:

1. A method of making a heat sensor, comprising the steps of:

inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,

evacuating said tube to withdraw substantially all atmospheric gases therefrom,

then forcing hydrogen at high pressure into said tube and maintaining said hydrogen there under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingas said hydrogen,

then withdrawing some of said hydrogen from said tube; and

sealing off the tube interior from the atmosphere.

2. The method of claim 1 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube.

3. A method of making a heat sensor, comprising the steps of:

inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,

heating said tube to a temperature and for a time sufficient to rid the inner tube surfaces of substantially all atmospheric gases;

cooling said tube to a desired temperature,

then forcing hydrogen at high pressure into said tube and maintaining said hydrogen there under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingas said hydrogen,

then withdrawing some of said hydrogen from said tube; and

sealing off the tube interior from the atmosphere.

4. The method of claim 3 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube.

5. A method of making a heat sensor, comprising the steps of:

inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,

evacuating said tube to withdraw substantially all atmospheric gases therefrom,

heating said tube to a temperature high enough and for a time long enough to substantially free the surface of said tube of components that react with hydrogen,

then forcing hydrogen at high pressure into said tube and maintaining said hydrogen there under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingas said hydrogen,

then withdrawing some of said hydrogen from said tube; and

sealing off the tube interior from the atmosphere.

6. The method of claim 5 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube.

7. A method of making a heat sensor, comprising the steps of:

inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,

purging said tube by sending hydrogen under pressure into said tube and removing said hydrogen therefrom,

then forcing hydrogen at high pressure into said tube and maintaining said hydrogen under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingass said hydrogen,

then withdrawing some of said hydrogen from said tube; and

sealing off the tube interior from the atmosphere.

8. The method of claim 7 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube.

9. The method of claim 7 in which said purging is accomplished by alternately forcing hydrogen into said tube and then pulling a vacuum on said tube, to free said passage from substantially all gases.

10. The method of claim 9 wherein said hydrogen is withdrawn to an amount providing a desired hydrogen pressure at a preselected temperature above the outgassing temperature of the hydride formed with said material.

11. The method of claim 9 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube.

12. The method of claim 11 wherein said gas is added to an amount providing a desired gas pressure in said tube at a preselected temperature below the outgassing temperature of the hydride.

13. The method of claim 7 wherein said tube is open at both ends during purging and wherein the purging is accomplished by forcing said hydrogen under pressure through said tube from one end while pulling a vacuum on the other end of said tube, to free said passage from substantially all gases.

14. The method of claim 13 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube to an amount that creates a preselected pressure at a preselected temperature below that at which said material begins to outgas hydrogen.

15. The method of claim 13 wherein the hydrogen that is forced in at high pressure after the purging step and in the forcing step is unmixed with other gases and wherein at the step of withdrawing hydrogen from said tube an amount of hydrogen is left that creates a preselected pressure at a preselected temperature above the point at which said material begins to outgas hydrogen.

16. A method of making a heat sensor, comprising the steps of:

inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,

purging said tube by forcing hydrogen under pressure into one end of said tube while pulling a vacuum on the other end of said tube to free said passage from substantially all gases,

closing one end of said tube,

forcing hydrogen at high pressure into the open end of said tube,

cooling said tube from a high temperature to a lower temperature while said material ingasses said hydrogen,

cooling said tube to a preselected temperature,

withdrawing said hydrogen that did not ingas into said material to a desired pressure level, and
sealing off its interior from the atmosphere.

17. A method of making a heat sensor, comprising the steps of:
inserting a wire of metal that dissolves hydrogen therein in a tube of metal that does not dissolve hydrogen therein,
degreasing the tube interior and wire by flushing with solvent,
evacuating the tube and raising the temperature to at least 1900° F. while holding a vacuum of about one micron of mercury,
cooling said tube to a desired lower temperature,
pumping hydrogen under pressure into said tube,
changing the temperature of said tube while maintaining said hydrogen in said tube under pressure to cause said wire to ingas hydrogen,
cooling said tube to a preselected temperature,
withdrawing excess hydrogen from said tube, and
then sealing off the interior of said tube from the atmosphere.

18. A method of making a heat sensor, comprising the steps of:
washing a wire of metal that dissolves hydrogen therein in hydrofluoric acid to dissolve away oxides,
rinsing said acid from said wire,
inserting said wire in a tube of metal that does not dissolve hydrogen therein,
purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and (*b*) evacuating said tube,
raising the temperature to at least 1900° F. while holding said tube evacuated,
cooling said tube to a desired lower temperature,
pumping hydrogen under pressure into said tube,
changing the temperature of said tube while maintaining said hydrogen in said tube under pressure to cause said wire to ingas hydrogen,
cooling said tube to a preselected temperature,
withdrawing excess hydrogen from said tube, and
then sealing off the interior of said tube from the atmosphere.

19. A method of making a heat sensor, comprising the steps of:
tightly spiraling wrapping metal wire that ingasses hydrogen with a protective strip of metal that does not ingas hydrogen,
inserting the wrapped wire in a tube of metal that does not ingas hydrogen,
degreasing the tube interior and wrapped wire by flushing with solvent,
evacuating the tube to a vacuum of about one micron of mercury,
then purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip and (*b*) evacuating said tube,
raising the temperature to at least 1900° F. while holding a vacuum of about one micron of mercury,
cooling said tube to a desired lower temperature,
pumping hydrogen under pressure into said tube,
changing the temperature of said tube while maintaining said hydrogen in said tube under pressure to cause said wire to ingas hydrogen,
cooling said tube to a preselected temperature,
withdrawing hydrogen from said tube to a desired pressure level,
then putting into said tube an inert gas chosen from the group consisting of helium, argon, neon, krypton, and xenon to a desired pressure, and
then sealing off the interior of said tube from the atmosphere.

20. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping metal wire chosen from the group consisting of scandium, titanium, vanadium, ytterbium, zirconium, niobium, hafnium, tantalum, the rare earth metals, and the actinide metals, with a strip of metal chosen from the group consisting of molybdenum and tungsten,
inserting the wrapped wire in a tube of metal chosen from the group consisting of nickel, iron, and alloys of nickel and iron to enclose the spiral wrap,
degreasing the tube interior and wrapped wire by flushing with solvent,
evacuating the tube to a vacuum of about one micron of mercury,
then purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip and (*b*) evacuating said tube,
raising the temperature to at least 1900° F. while holding a vacuum of about one micron of mercury,
then cooling said tube to a desired lower temperature,
then pumping hydrogen under pressure into said tube,
changing the temperature of said tube while maintaining said hydrogen in said tube under pressure to cause said wire to ingas hydrogen,
cooling said tube to a preselected temperature,
withdrawing excess hydrogen from said tube,
then putting into said tube an inert gas chosen from the group consisting of helium, argon, neon, krypton, and xenon to a desired pressure, and
then sealing off the interior of said tube from the atmosphere.

21. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping a strip of molybdenum around a zirconium wire,
fully inserting the wrapped wire into a stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
purging the tube by alternately (*a*) flushing it with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip; and (*b*) evacuating said tube,
raising the temperature of the tube to at least 1900 ° F. while holding the tube interior at a vacuum of about one micron of mercury,
cooling said tube to between 1500° F. and 1850° F.,
pumping hydrogen under pressure into said tube,
lowering the temperature of said tube to about 1200° F. to cause said wire to ingas hydrogen,
cooling said tube to a preselected temperature and withdrawing the hydrogen therefrom to a desired pressure level in said tube, and
sealing off the interior of said tube from the atmosphere.

22. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping a strip of molybdenum around a zirconium wire,
fully inserting the wrapped wire into a stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
evacuating the tube to a vacuum of about one micron of mercury,
then purging the tube by alternately (*a*) flushing it with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip; and (*b*) evacuating said tube,
raising the temperature of the tube to at least 1900° F. while holding the tube interior at a vacuum of about one micron of mercury,

then cooling said tube to between 1500° F. and 1850° F.,
then pumping hydrogen under pressure into said tube,
then alternately for several cycles (*a*) lowering the temperature of said tube to about 1200° F. to cause said wire to ingas hydrogen, and (*b*) heating said tube again to between 1500° F. and 1850° F.,
cooling said tube to a preselected temperature and withdrawing the non-ingassed hydrogen therefrom to a desired pressure level,
then putting into said tube an inert gas chosen from the group consisting of helium, argon, neon, krypton, and xenon to a desired pressure, and
then sealing off the interior of said tube from the atmosphere.

23. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping a strip of molybdenum around a titanium wire,
fully inserting the wrapped wire into a stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
purging the tube by alternately (*a*) flushing it with hydrogen, to drive out subtsantially all other gases from said tube and from the wire and strip; and (*b*) evacuating said tube,
raising the temperature of the tube to at least 1800° F. while holding the tube interior at a vacuum of about one micron of mercury,
cooling said tube to about 1200° F. to 1500° F.,
pumping hydrogen under pressure into said tube,
lowering the temperature of said tube to about room temperature to cause said wire to ingas hydrogen,
cooling said tube to a preselected temperature and withdrawing the non-ingassed hydrogen therefrom to a desired pressure level, and
sealing off the interior of said tube from the atmosphere.

24. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping a strip of molybdenum around a titanium wire,
fully inserting the wrapped wire into a stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
evacuating the tube to a vacuum of about one micron of mercury,
then purging the tube by alternately (*a*) flushing it with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip; and (*b*) evacuating said tube,
raising the temperature of the tube to at least 1900° F. while holding the tube interior at a vacuum of about one micron of mercury,
then cooling said tube to about 1200° F. to 1500° F.
then pumping hydrogen under pressure into said tube,
then lowering the temperature of said tube to cause said wire to ingas hydrogen,
alternately raising and lowering said temperature several times,
cooling said tube to a preselected temperature and withdrawing the non-ingassed hydrogen therefrom to a desired pressure in said tube,
then putting into said tube an inert gas chosen from the group consisting of helium, argon, neon, krypton, and xenon to a desired pressure, and
then sealing off the interior of said tube from the atmosphere.

25. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping zirconium wire with a strip of molybdenum,
fully inserting the wrapped wire in a stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip and (*b*) evacuating said tube,
raising the temperature of said tube to at least 1900° F. while holding a vacuum of about one micron of mercury,
cooling said tube to room temperature,
then pumping hydrogen under pressure into said tube,
raising the temperature of said tube to about 1500–1800° F. and then cooling said tube to about room temperature to cause said wire to ingas hydrogen,
withdrawing the excess hydrogen therefrom, and
sealing off the interior of said tube from the atmosphere.

26. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping titanium wire with a strip of molybdenum,
fully inserting the wrapped wire in a stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip and (*b*) evacuating said tube,
raising the temperature of said tube to at least 1900° F. while holding a vacuum of about one micron of mercury,
cooling said tube to room temperature,
then pumping hydrogen under pressure into said tube,
raising the temperature of said tube to about 1200° F.–1500° F. and then cooling said tube to about room temperature to cause said wire to ingas hydrogen,
withdrawing the excess hydrogen therefrom, and
sealing off the interior of said tube from the atmosphere.

27. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping zirconium wire with a strip of moylbdenum,
fully inserting the wrapped wire in an open end stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
evacuating the tube to a vacuum of about one micron of mercury,
then purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip and (*b*) evacuating said tube,
raising the temperature of said tube to at least 1900° F. while holding a vacuum of about one micron of mercury,
then cooling said tube to room temperature,
then pumping hydrogen under pressure into said tube,
raising the temperature of said tube to about 1500–1800° F. and then cooling said tube to about room temperature to cause said wire to ingas hydrogen,
raising and lowering the temperature several times between 1500°–1800° F. and a substantially lower temperature,
then withdrawing the non-ingassed hydrogen therefrom to a desired pressure at a preselected temperature,
then putting into said tube an inert gas chosen from the group consisting of helium, argon, neon, krypton, and xenon to a desired pressure, and
then sealing off the interior of said tube from the atmosphere.

28. A method of making a heat sensor, comprising the steps of:
tightly spirally wrapping titanium wire with a strip of molybdenum,
fully inserting the wrapped wire in an open end stainless steel tube,
degreasing the tube interior and wrapped wire by flushing with solvent,
evacuating the tube to a vacuum of about one micron of mercury,
then purging the tube by alternately (*a*) flushing with hydrogen, to drive out substantially all other gases from said tube and from the wire and strip and (*b*) evacuating said tube,
raising the temperature of said tube to at least 1900° F. while holding a vacuum of about one micron of mercury,
then cooling said tube to room temperature,
then pumping hydrogen under pressure into said tube,
raising the temperature of said tube to about 1200°–1500° F. and then cooling said tube to about room temperature to cause said wire to ingas hydrogen,
raising and lowering the temperature several times,
then withdrawing the non-ingassed hydrogen therefrom to a desired pressure at a preselected temperature,
then putting into said tube an inert gas chosen from the group consisting of helium, argon, neon, krypton, and xenon to a desired pressure, and
then sealing off the interior of said tube from the atmosphere.

29. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
freeing said passage from substantially all gases,
forcing hydrogen at high pressure into said tube and then cooling said tube from a high temperature to a lower temperature while said material ingases said hydrogen,
cooling said tube to a preselected temperature,
withdrawing said hydrogen that did not ingas into said material to a desired pressure level, and
sealing off its interior from the atmosphere.

30. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
purging said tube with gas under pressure,
heating said tube to at least 1900° F.,
closing one end of said tube,
forcing hydrogen at high pressure and high temperature into the open end of said tube,
cooling said tube from a high temperature to a lower temperature while said material ingasses said hydrogen,
cooling said tube to a preselected temperature,
withdrawing said hydrogen that did not ingas into said material to a desired pressure level, and
sealing off its interior from the atmosphere.

31. The method of claim 30 wherein before sealing off the interior of said tube from the atmosphere said passage is filled with gas that is chemically inactive with respect to hydrogen, to said material, and said tube.

32. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
freeing said passage from substantially all gases,
forcing hydrogen under pressure into said tube and then cooling said tube from a high temperature to a lower temperature while said material ingasses said hydrogen, and
sealing off the interior of said tube from the atmosphere.

33. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
purging said tube with gas under pressure,
heating said tube to at least 1900° F.,
closing one end of said tube,
forcing hydrogen at high pressure and high temperature into the open end of said tube,
cooling said tube from a high temperature above the threshold temperature of said material to a lower temperature below said threshold temperature, while said material ingasses said hydrogen, and
sealing off the interior of said tube from the atmosphere.

34. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
evacuating said tube to withdraw substantially all atmospheric gases therefrom,
then forcing hydrogen at high pressure into said tube and maintaining said hydrogen there under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingas said hydrogen, and
sealing off the tube interior from the atmosphere.

35. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
heating said tube to a temperature and for a time sufficient to rid the inner tube surfaces of substantially all atmospheric gases;
cooling said tube to a desired temperature,
then forcing hydrogen at high pressure into said tube and maintaining said hydrogen there under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingas said hydrogen, and
sealing off the tube interior from the atmosphere.

36. A method of making a heat sensor, comprising the steps of:
inserting a material that readily and reversibly forms hydrides by ingassing and outgassing above a threshold temperature into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,
evacuating said tube to withdraw substantially all atmospheric gases therefrom,
heating said tube to a temperature high enough and for a time long enough to substantially free the surface of said tube of components that react with hydrogen,
then forcing hydrogen at high pressure into said tube and maintaining said hydrogen there under pressure

while reducing the temperature of said tube from a temperature above the threshold temperature to a temperature below said threshold temperature to cause said material to ingas said hydrogen, and

sealing off the tube interior from the atmosphere.

37. A method of making a heat sensor, comprising the steps of:

inserting a material that readily and reversibly forms hydrides by ingassing and outgassing into a tube of a substance that does not readily form hydrides, while providing free passage for gas in contact with said material,

purging said tube by sending hydrogen under pressure into said tube and removing said hydrogen therefrom,

then forcing hydrogen at high pressure into said tube and maintaining said hydrogen under pressure while reducing the temperature of said tube from a high temperature to a low temperature to cause said material to ingas said hydrogen, and

sealing off the tube interior from the atmosphere.

References Cited in the file of this patent

UNITED STATES PATENTS

2,279,611	Barnhardt	Apr. 14, 1942
2,535,477	Andrae	Dec. 26, 1950
2,868,017	Beard	Jan. 13, 1959
3,064,245	Lindberg	Nov. 13, 1962