Oct. 30, 1945. W. D. BROWN 2,387,878

APPARATUS FOR DETERMINING HYDROGEN IN STEEL

Filed Nov. 17, 1944

FIG. 1.

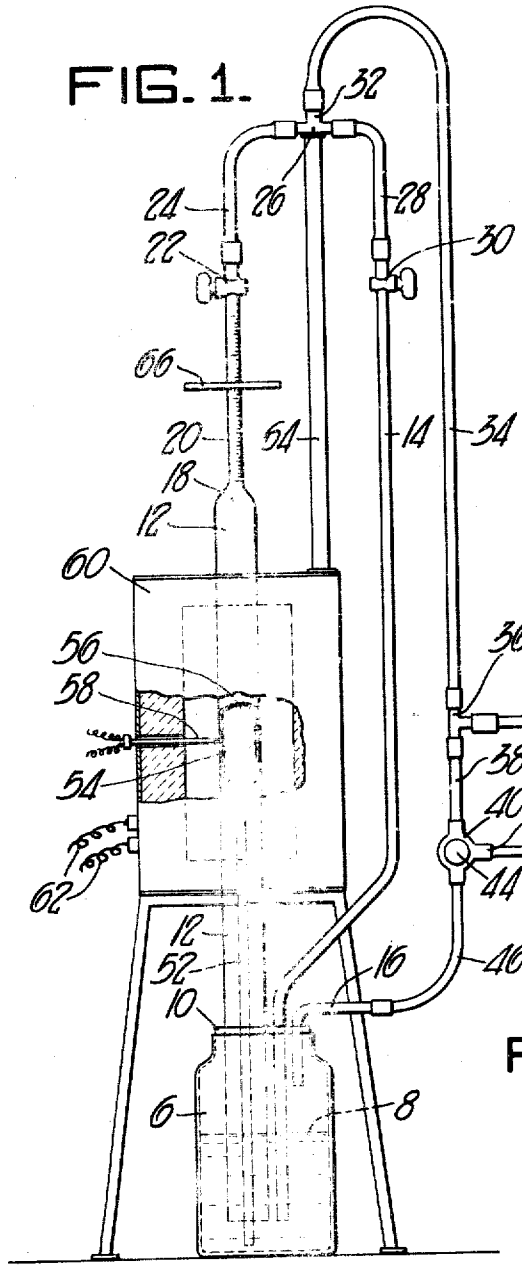


FIG. 2.

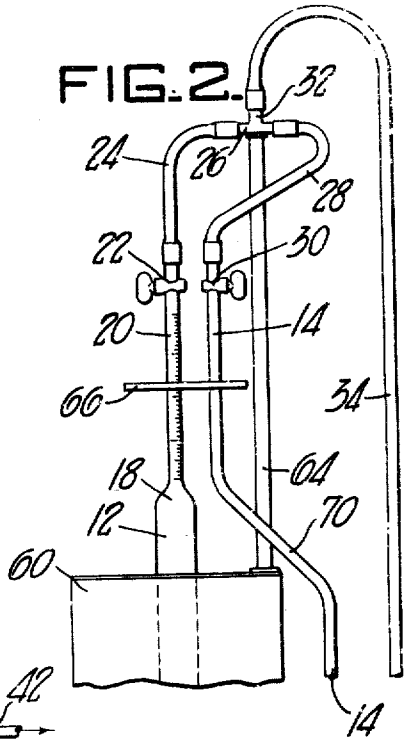


FIG. 3.

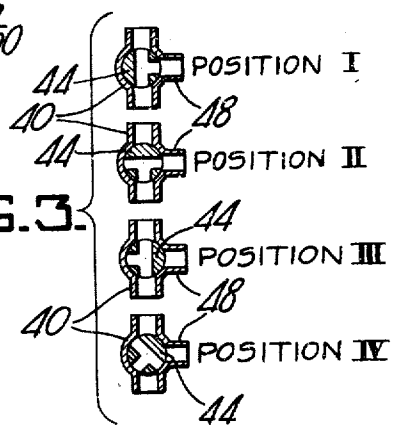
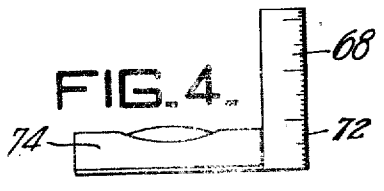


FIG. 4.



INVENTOR:
WILLIAM D. BROWN,
BY: John E. Jackson
HIS ATTORNEY.

Patented Oct. 30, 1945 2,387,878

UNITED STATES PATENT OFFICE

2,387,878

APPARATUS FOR DETERMINING HYDROGEN IN STEEL

William D. Brown, Pittsburgh, Pa., assignor to Carnegie-Illinois Steel Corporation, a corporation of New Jersey

Application November 17, 1944, Serial No. 563,942

5 Claims. (Cl. 73—15)

In accordance with the present invention there are provided certain improvements in an apparatus for determining hydrogen in steel, wherein the determinations are made with speed and accuracy.

The present invention includes means for heating a steel specimen to a known temperature at which any hydrogen contained in the steel will be evolved therefrom, together with a system wherein a vacuum pump is employed in conjunction with a Torricellian vacuum for enabling the evolved hydrogen to be collected and measured. The apparatus also involves the employment of a second measuring tube by means of which the pressure of the hydrogen, which has been removed from the steel sample, may be measured while in the extraction tube.

The invention will be understood by reference to the accompanying drawing, wherein:

Figure 1 represents a side elevation, partly in section, of the improved apparatus;

Figure 2 is an upper fragmentary elevation of the apparatus of Figure 1, showing a modification;

Figure 3 is a diagrammatic view in section showing various positions taken by a main control valve during operation of the apparatus; and

Figure 4 represents a side elevation of a combined level and gage employed in connection with the present invention.

Referring more particularly to the drawing, the reference numeral 6 represents a glass jar which is about half filled with mercury 8. The jar 6 has a wide mouth which receives a three-hole rubber stopper 10. Inserted through this rubber stopper 10 are three tubes made of refractory high melting point glass, these tubes being an extraction tube 12, of large diameter, a mercury tube 14, provided with a reverse bend therein, as shown, and a right-angle connecting tube 16. The extraction tube 12 and mercury tube 14 dip into the pool 8 of mercury contained in the jar 6, while the connecting tube 16 merely extends through the stopper 10.

The top of the extraction tube 12 is drawn down as shown at 18, and sealed to the end of a refractory gas measuring tube 20 which is graduated in millimeters. The other end of the measuring tube 20 is provided with a stop cock 22 and is connected by means of a rubber tube 24 to one end of a T-connection 26. This T-connection is made of a suitable metal, such as bronze, for example. The other end of the T-connection 26 is connected through rubber tube 28 to one end of the mercury measuring tube 14, adjacent in which there is mounted a stop cock 30, this being similar to stop cock 22.

The branch 32 of the T-connection 26 is connected through rubber tube 34 to a second T-connection 36, similar to the T-connection 26, the tube 34 being connected to one end of the T-connection 36, the other end of the said connection receiving a connecting tube 38 to one end of a third T-connection 40.

The branch of the T-connection 36 connects through a tube 42 to a vacuum source, such as an air pump, not shown.

The T-connection 40 has a three-way valve 44 mounted therein, this being the control valve for the system. One end of the T-connection 40 receives the tube 38, the other end of the T-connection 40 being connected through rubber tube 46 to the glass tube 16. The branch 48 of the T-connection 40 is connected to the air through suitable drying means, such as the calcium chloride tube 50.

A glass rod 52 is positioned in the extraction tube 12. One end of the rod dips in the mercury 8, which floats the rod. The other end of the rod 52 is enlarged into a flat-topped button 54, which is somewhat similar than the inside diameter of the extraction tube, so that the inside wall of the tube will support the top of the rod and continuously maintain this top approximately level for receiving and retaining a specimen 56 of steel to be tested, while allowing the top of the rod with the specimen thereon to be freely inserted in the extraction tube. The length of the rod 52 is made so that during the testing, the sample 56 will be positioned on the top 54 opposite to the pyrometer 58 mounted in a split electric resistance furnace 60, current to which is supplied through leads 62.

A support 64 for the tubing of the apparatus is turnably mounted on the top of the furnace 60, this support suitably holding the T-connection 26 in clamped relation thereto. An asbestos shield 66 is interposed between the furnace 60 and valve 22 of graduated tube 20 for protecting the valve against overheating from the furnace. The valve 30 is preferably at the same height as the valve 22, but, in Figure 1, is sufficiently far removed from the furnace so as not to require the use of such a shield, although such may be provided should it be desired.

The extraction tube 12 is sufficiently large to admit sample 56 which is of suitable size for the determination. It may be a cube, say for example, one-half inch in each dimension, or it may be specimens of steel strip. Consequently, the clear-

ance between the perimeter of the button head 54 of the rod 52 and the inside wall of the tube 12 is only sufficient to enable the free insertion of the rod in the tube. In practice, it is found that the extraction tube 12 may have a diameter of one inch with a diameter of seven-eighths inch for the button head 54, having a clearance of one-sixteenth inch between the button head 54 and the inside wall of the tube. Stop cocks 22 and 30 are provided with a suitable vacuum lubricant for preventing leakage.

The operation of the device of Figure 1 is as follows:

A suitable steel sample, for example, a one-half inch cube is prepared by filing off sharp corners, directly washing with ether, and placed in a desiccator, using tweezers therefor. The rubber stopper 10, which holds the extraction tube 12, the mercury measuring tube 14, and the right angle connecting suction tube 16, is removed from the jar 6. The sample 56 is placed on the head 54 of rod 52 and inserted on the rod into the extraction tube 12, and the length of the rod 52 is adjusted if necessary so as to bring the sample in opposition to the thermocouple 58, when the stopper 10 has been replaced in the jar 6.

After the stopper 10 has been replaced in the jar 6, the three-way valve 44 is turned from position I in Figure 3, wherein the entire system is open to the air through drying tube 50, to position II in Figure 3, which closes the tube 38 and, through tube 34, the extraction tube 12 and the measuring tube 14 to the outside air, and places tube 16 in communication with the air through the said drying tube 50. The stop cocks 22 and 30 are opened and the vacuum pump (not shown) is started, and when the mercury from the pool 8 thereof in the jar has risen in the extraction tube 12 and measuring tube 14 to a level above the stop cocks 22 and 30 (which stop cocks are located in practice at a height of substantially twenty-nine inches from the lower, immersed ends of the tubes 12 and 14), the stop cocks are closed, and the valve 44 is turned to position III on Figure 3, so that vacuum is applied to the interior of the jar 6 and the outlet to the outside air through the calcium chloride tube 50, is cut off. The mercury then drains from the tubes 12 and 14, creating a Torricellian vacuum therein.

Any traces of residual air remaining in the tubes are withdrawn by repeating the foregoing, viz. turning valve 44 to position II in Figure 3, thereby allowing dry air to enter the jar 6, and mercury to rise in tubes 12 and 14. The stop cocks 22 and 30 are opened, when the suction created by the vacuum pump causes mercury to replace any air in the tube 20, through the stop cock 22, and in tube 14 through stop cock 30. When the mercury level has risen slightly above these stop cocks, they are closed and valve 44 moved to position III, thereby drawing the mercury from the tubes into the jar 6. This operation may be repeated, but repeated practice has shown that one gassing out is sufficient.

The extraction tube 12 is moved into the groove in split furnace 60 by moving the jar 6. The furnace cover is closed and the heating current supplied to the furnace. The sample is heated to from approximately 950° F. to approximately 1000° F. as shown by the thermocouple 58, and maintained for two hours at this temperature.

After this time, the current is cut off, and the jar 6 is moved so that tube 12 is out of the groove in the furnace. The turning of the support 64 enables this action to be accomplished. Asbestos shields are placed between the tube 12 and the halves of the furnace. Shield 66 is maintained in place around the graduated tube 20 throughout the heating to protect the stop cock 22, and if necessary, a similar shield (not shown) may be placed around tube 14 for the protection of stop cock 30. When the tube 12 has cooled sufficiently, air may be blown on it until cool to the hand.

The valve 44 then is turned slowly to position II of Figure 3. Mercury will rise in the tubes 12, 20 and 14, without filling tube 20 owing to the hydrogen retained in the tube, this being the hydrogen evolved from the sample. When the mercury in tube 20 has risen to some specified graduated number of millimeters such that the mercury in tube 14 has not risen to stop cock 30, the mercury levels in tubes 20 and 14 are held by turning the valve 44 to position IV in Figure 3. The difference in height between the mercury in tube 20 and the mercury in tube 14 being the pressure of the gas collected in the tube 20.

The difference in the said mercury levels is ascertained readily by the use of the level 74 and millimeter scale 68, as will be apparent. See Figure 4 for these elements, which are carried by a square 72. The volume of collected hydrogen is found by reading the milliliter scale on tube 20 from the stop cock 22 downwardly.

It will be understood that when the tubes are filled with mercury, the rod 52 and sample 56 float upwardly in the tube 12 to the constricted end thereof. However, the internal diameter of tube 20 is too small to allow passage of either the sample or the rod into the tube 20, so that the volume of the gas measured therein is not affected by this action. As the mercury empties from the tube, the rod and sample correspondingly drop, so that during the heating, the sample is level with the thermocouple 58, the inner wall of the tube 12 guiding the rod in the movements thereof.

The percentage of hydrogen may be determined from the foregoing observations as follows:

The weight of one liter of hydrogen at 60° F. and 30 inches pressure is 0.08526 gm., from which the weight at 80° F. and 760 mm. pressure is 0.08188 gm., and 1 ml. therefore weighs 0.00008188. From this 1 ml. at 1 mm. pressure weighs 0.000000108 gm. At 70° F., 1 ml. weighs 0.00000011 gm. Therefore, to obtain the weight of the hydrogen collected, multiply the number of milliliters of gas in the tube 20 by the millimeter pressure (difference between the mercury levels in the tubes 20 and 14), and by 0.00000011 and divide by the weight of the sample, then multiply by 100.

For example, suppose 6 ml. of gas collect in tube 20 and that the sample 56 weighs 16.5 gms. and the pressure reading is 53 mm. Then the amount of hydrogen, by weight in the sample would be, under the assumed values.

$$\frac{0.00000011\times 6\times 53\times 100}{16.5}=0.00021\%$$

Precautions must be taken, obviously, to adjust the size of the sample so that all of the hydrogen will be retained in the volume of the graduated tube 20. Experience will teach closely what such relationship will be in any given case. In addition to a cubical specimen, other shapes of known dimensions may be used for the test, such as strips, or the like.

In the apparatus of Figure 1, it is necessary to use the level and millimeter scale shown in Fig-

ure 4 for obtaining differences in mercury levels in the tubes 20 and 14. The modification of Figure 2 is designed to eliminate this necessity, the tube 14 being provided with a reverse bend 70 in its upper portions so as to bring it sufficiently close to the graduated tube 20 so that the differences in mercury level may be ascertained directly by the use of a millimeter scale alone. In this modification of the apparatus, the asbestos shield 66 is extended so as to protect both stop cocks 22 and 30 from overheating.

Except for the reverse bend 70 in the tube 14 as shown in Figure 2, the parts are the same as shown in Figure 1, and the same reference numbers are employed and applied as in Figure 1; and the operation of the apparatus is the same as described above in connection with Figure 1, except that the use of the square 72 shown in Figure 4 may be dispensed with, and replaced by an ordinary meter stick, as will be well understood.

All of the glass tubing in the present construction is highly refractory in composition and resists the temperature of the furnace without tendency to soften. Tubing known to the trade under the trade-mark "Pyrex" of the Corning Glass Company, Corning, New York, is a suitable example of a satisfactory type of glass which may be employed. As a further illustration of dimensions, which obviously may be varied as may be desired, and are illustrative only in character, graduated tube 20 may be 8 mm. in inside diameter, while tube 14 may be 8 mm. inside diameter. Stop cocks 22 and 30 may be 4 mm. bore or larger, each. The extraction tube 12 may be 1 inch inside diameter. Stop cocks 22 and 30 are 29 inches above the bottom ends of tubes 12 and 14, respectively.

I claim:

1. Testing apparatus for determination of hydrogen in steel, which comprises the combination with a heating furnace, of means for containing a mercury reservoir, a closure for the said means, an extraction tube extending through the said closure to a point of immersion in the mercury reservoir, the said extraction tube having an extraction section and a graduated gas measuring section, a second measuring tube extending through the said closure to a point of immersion in the mercury reservoir, means connecting the graduated gas measuring section of the extraction tube to the second measuring tube, valve means in the said gas measuring section and in the second measuring tube, means enabling evacuation of air from the entire extraction tube and second measuring tube, means for admitting dry air through the closure and into the tubes, means for introducing a sample into the extraction tube for a predetermined distance, and in the zone of the heating furnace, control means for controlling evacuation of the extraction tube and the second measuring tube, and means for determining difference in level of mercury rising in the extraction tube and measuring tube.

2. Testing apparatus for determination of hydrogen in steel, which comprises the combination with a heating furnace, of means for containing a mercury reservoir, a closure for the said means, an extraction tube extending through the said closure to a point of immersion in the mercury reservoir, the said extraction tube having an enlarged extraction section and a constricted gas measuring section, a second measuring tube extending through the said closure to a point of immersion in the mercury reservoir, means connecting the graduated gas measuring section of the extraction tube to the second measuring tube, valve means in the said gas measuring section and in the second measuring tube, means enabling evacuation of air from the entire extraction tube and from the second measuring tube, means for producing and maintaining a Torricellian vacuum in the said tubes, a holder for the sample being tested extending for a predetermined distance into the extraction tube, the said holder being freely floating in the extraction tube and supported thereby for maintaining the sample being tested at a predetermined level in the tube, and a three-way control valve for controlling evacuation of the tube.

3. Testing apparatus for determining hydrogen in steel, which comprises the combination with a heating furnace for heating a sample of steel being tested, of a mercury reservoir, interconnected measuring tubes immersed in the mercury reservoir, one of the said tubes being adapted to receive a sample being tested and to measure gas evolved therefrom during the testing, means enabling air to be evacuated from the measuring tubes, and means for maintaining a Torricellian vacuum in the said tubes.

4. Testing apparatus for determining hydrogen in steel, which comprises the combination with a heating furnace for heating a sample of steel being tested, of a mercury reservoir, interconnected measuring tubes immersed in the mercury reservoir, one of the said tubes being adapted to receive a sample of steel being tested and to measure gas evolved therefrom during the testing, means enabling air to be evacuated from the measuring tubes, a freely floating mounting for the sample extending for a predetermined distance in the sample-receiving tube for maintaining the sample at the said distance in the said tube, the mounting being supported by the tube, means for maintaining a Torricellian vacuum in the said tubes, and control instrumentalities adapted to enable the apparatus to become filled with air and to control evacuation of the said tubes for causing mercury to rise therein at least as far as the Torricellian vacuum retaining means, operation of the said means in conjunction with predetermined selected operation of the control instrumentalities producing a predetermined sequence of operations, namely, first, the filling of the apparatus with dry air responsively to operation of the control instrumentalities to a first position and opening of the said vacuum retaining means, then mechanical evacuation of the said apparatus responsively to operation of the control instrumentalities to a second position with accompanying rise of mercury in the said tubes to a level at least equal to that of the vacuum retaining means, then production of a Torricellian vacuum in the said tubes responsively to closing the vacuum retaining means and movement of the control instrumentalities to a third position with return of the mercury from the said tubes into the said reservoir, and finally, after evacuation of the sample, operation of the control instrumentalities to a fourth position causing the mercury to rise in the said tubes to different levels corresponding to the amount of gas evolved from the sample.

5. Testing apparatus for determining hydrogen in steel, which comprises the combination with a heating furnace for heating a sample of steel being tested, of a mercury reservoir, interconnected measuring tubes immersed in the mer-

cury reservoir, one of the said tubes being adapted to receive a sample of steel being tested and to measure gas evolved therefrom during the testing, means enabling air to be evacuated from the said tubes, valve means in the tubes for maintaining a Torricellian vacuum in the tubes and control instrumentalities for enabling the apparatus to be put through a predetermined sequence of operations responsively to predetermined positioning of the control instrumentalities, the first of the said sequence of operations being the filling of the apparatus with air responsively to operation of the control instrumentalities to a first position and opening of the valve means in the tubes, the second in the sequence of operations being mechanical evacuation of the tubes responsively to movement of the control instrumentalities to a second position with accompanying rise of mercury in the said tubes to a level at least equal to that of the said valve means in the tube, the third of the sequence of operations being the production of a Torricellian vacuum in the said tubes responsively to closing the valve means in the said tubes and movement of the control instrumentalities to a third position to cause return of the mercury in the tubes into the mercury reservoir, the fourth in the sequence of operations being movement of the control instrumentalities into a fourth position following completion of test heating of the sample, thereby causing the mercury to rise in the Torricellian vacuum tubes to a level corresponding to the volume of gas extracted from the sample during the heating thereof.

WILLIAM D. BROWN.

CERTIFICATE OF CORRECTION.

Patent No. 2,387,878. October 30, 1945.

WILLIAM D. BROWN.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, line 48, for "millimeters" read --milliliters--; and second column, line 26, for "similar" read --smaller--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 12th day of February, A. D. 1946.

Leslie Frazer

(Seal) First Assistant Commissioner of Patents.

cury reservoir, one of the said tubes being adapted to receive a sample of steel being tested and to measure gas evolved therefrom during the testing, means enabling air to be evacuated from the said tubes, valve means in the tubes for maintaining a Torricellian vacuum in the tubes and control instrumentalities for enabling the apparatus to be put through a predetermined sequence of operations responsively to predetermined positioning of the control instrumentalities, the first of the said sequence of operations being the filling of the apparatus with air responsively to operation of the control instrumentalities to a first position and opening of the valve means in the tubes, the second in the sequence of operations being mechanical evacuation of the tubes responsively to movement of the control instrumentalities to a second position with accompanying rise of mercury in the said tubes to a level at least equal to that of the said valve means in the tube, the third of the sequence of operations being the production of a Torricellian vacuum in the said tubes responsively to closing the valve means in the said tubes and movement of the control instrumentalities to a third position to cause return of the mercury in the tubes into the mercury reservoir, the fourth in the sequence of operations being movement of the control instrumentalities into a fourth position following completion of test heating of the sample, thereby causing the mercury to rise in the Torricellian vacuum tubes to a level corresponding to the volume of gas extracted from the sample during the heating thereof.

WILLIAM D. BROWN.

CERTIFICATE OF CORRECTION.

Patent No. 2,387,878. October 30, 1945.

WILLIAM D. BROWN.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, line 48, for "millimeters" read --milliliters--; and second column, line 26, for "similar" read --smaller--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 12th day of February, A. D. 1946.

Leslie Frazer

(Seal) First Assistant Commissioner of Patents.