

Jan. 13, 1931.

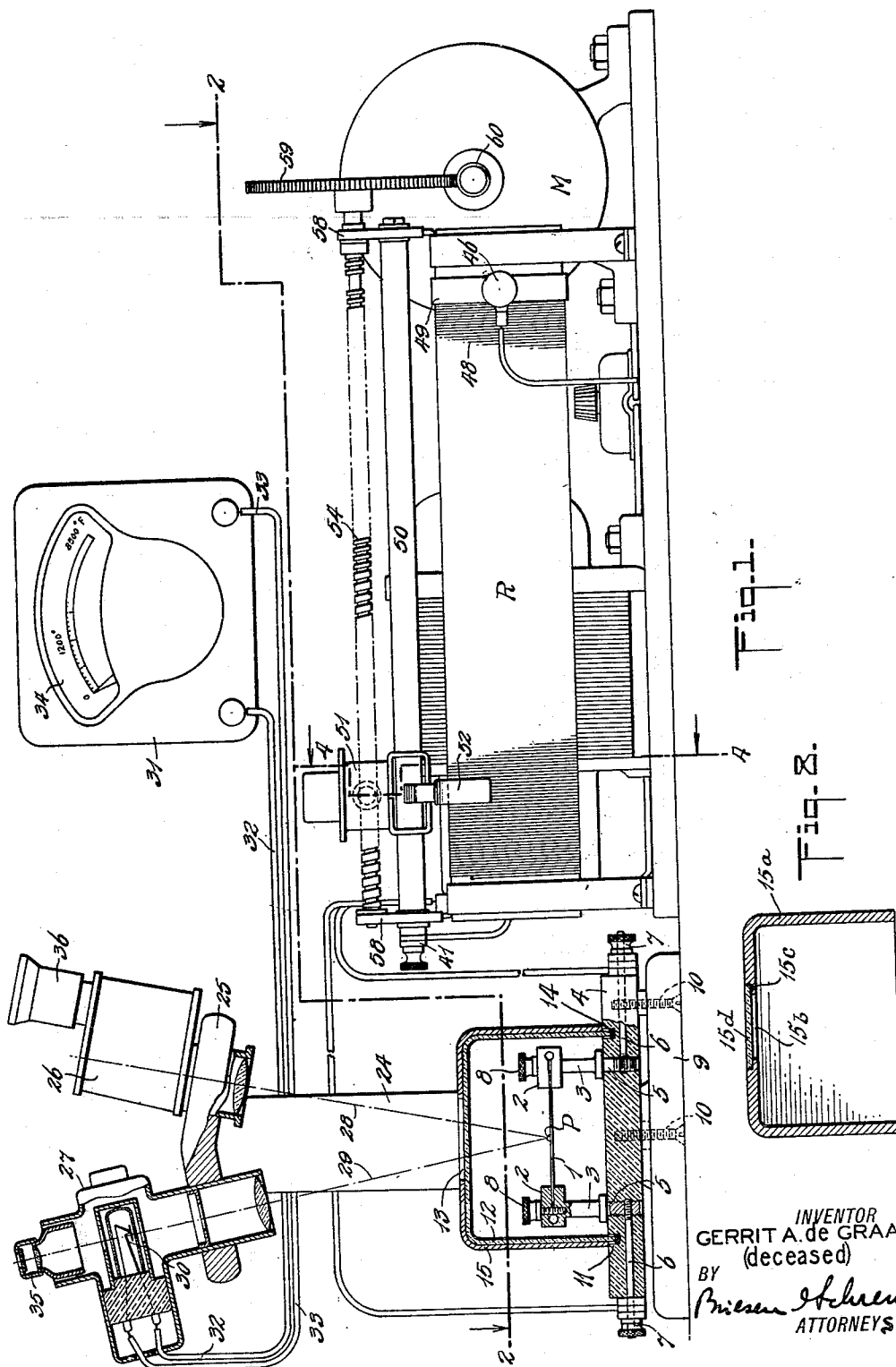
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1,789,098

PROCESS FOR THE DETERMINATION OF MELTING TEMPERATURES OF MATERIALS
AND APPARATUS FOR CARRYING OUT THE AFORESAID PROCESS

Filed Jan. 27, 1928

3 Sheets-Sheet 1



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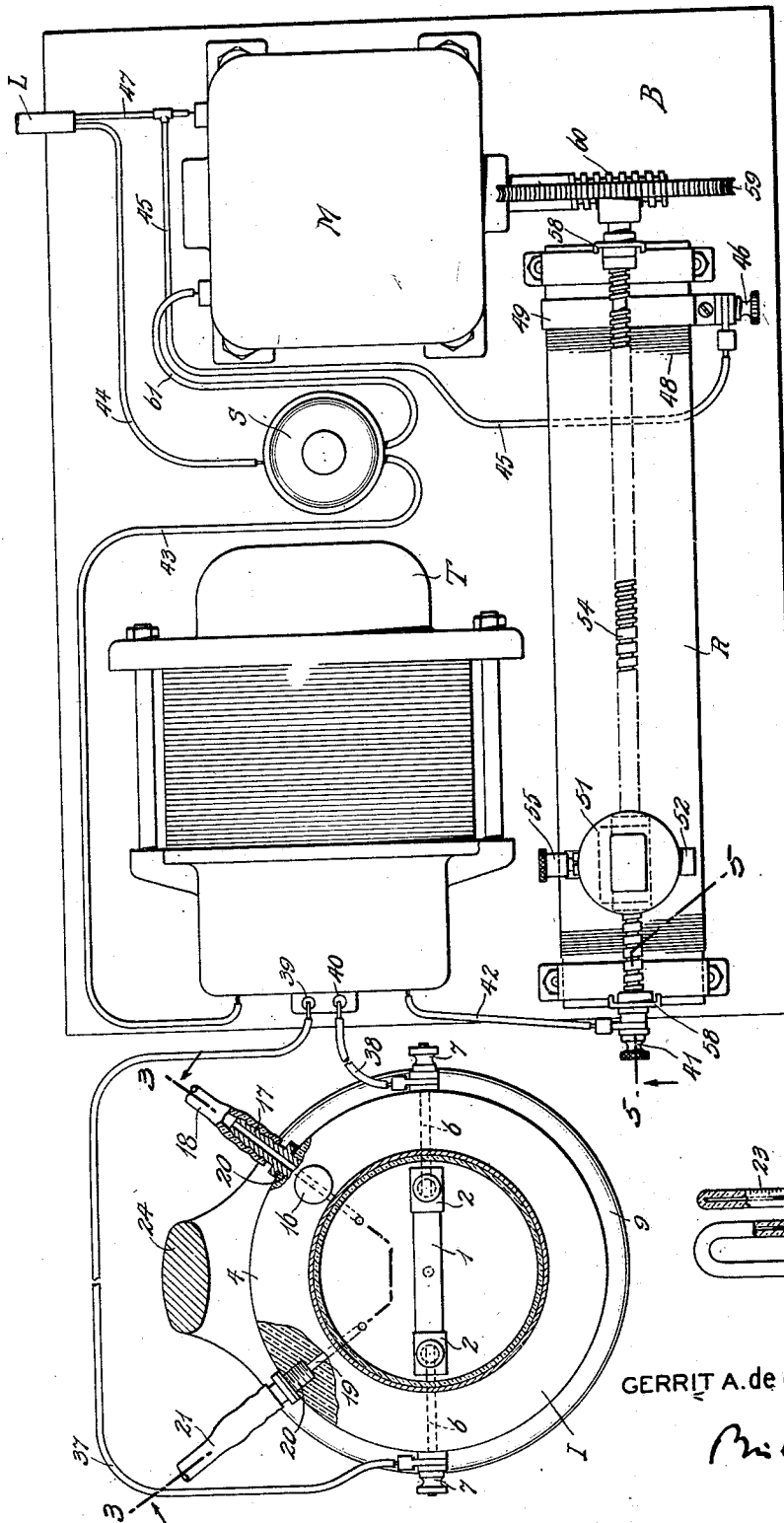
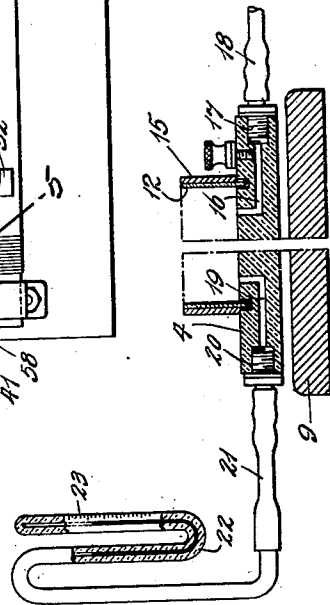


Fig. 2.

Fig. 3.



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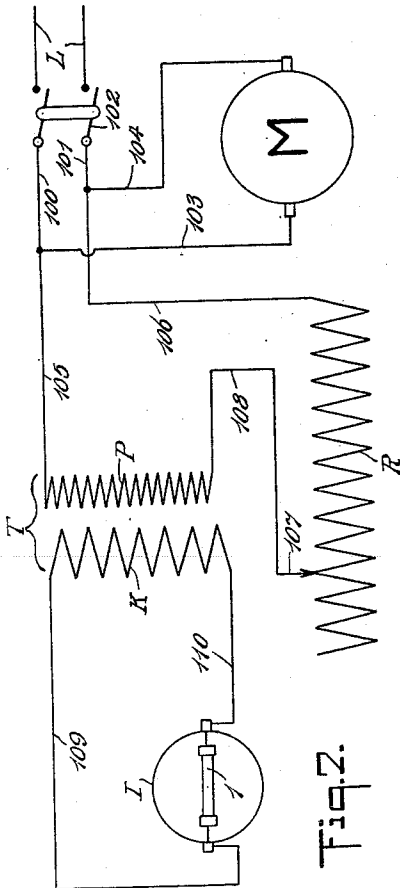
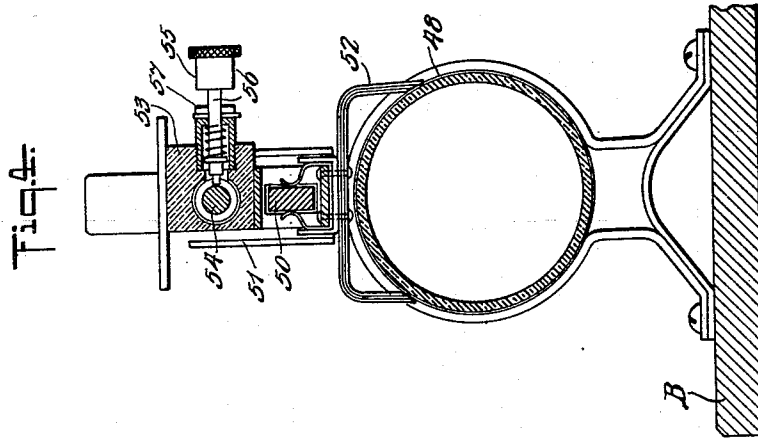


Fig. 3.

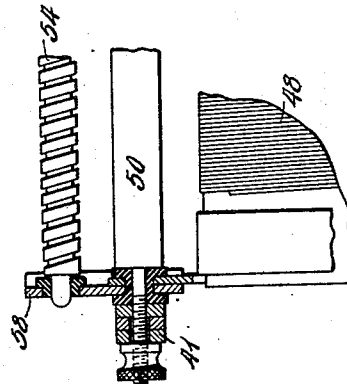
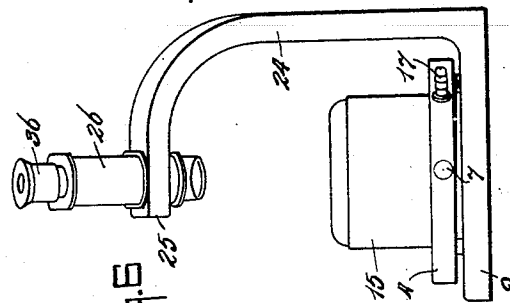


Fig. 4.



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PROCESS FOR THE DETERMINATION OF MELTING TEMPERATURES OF MATERIALS AND APPARATUS FOR CARRYING OUT THE AFORESAID PROCESS

Application filed January 27, 1928. Serial No. 250,013.

This invention relates to a process for the determination of the melting or fusing temperatures of material and to apparatus for carrying out the aforesaid processes, and more particularly to a process of determining the melting or fusing temperature of coal ash and to an automatic instrument for effecting the said determinations.

The invention has many applications but in this specification it will be described in connection with the determination of the melting or fusing temperature of coal ash. This determination has a very important economic and industrial importance and must be made with considerable accuracy. In the sale of coal, for example, the purchaser buys the coal on the basis that the coal ash is only present to a certain amount and has a certain fusing temperature. The seller of the coal agrees, among other things, that if the ash fuses below a certain temperature he will reimburse the purchaser a certain amount of money depending upon the difference in degrees of temperature between the contracted fusing temperature and the actual fusing temperature of the coal ash. At the present time the seller of coal often has to make enormous reimbursements to the purchaser because of the difference between the contracted and the actual fusion temperatures. As the margin of profit in selling coal to large consumers, such as public utilities, is very small, the seller is often subjected to serious losses. On the other hand, the purchaser of coal who is operating a power plant, for instance, is subjected to great trouble and expense if the coal contains an ash having a melting temperature lower than a safe fusing temperature for which he contracted. For example, in firing boilers troublesome clinkers form on the grates. The clinker formation has many objections among which may be mentioned the improper distribution of fuel over the grate surface, the formation of a more or less complete blanket over air openings and through the fuel bed, the interference with fuel movement on the grates, the clogging up of grate openings, and the adherence of the clinker mass to the stoker in such a way

that fresh fuel is pushed down by the lower ram and is caused to ride over the clinker mass on to the dumping grate where it is wasted. All of these objections, disadvantages, and troubles arise from the fact that it is difficult to determine the fusing or melting temperature accurately by any commercial or practical method.

Heretofore, the most general method used and the one adopted by the American Society for Testing Materials was the so-called cone method. In this method, as is well known, a representative sample of the ash contained in the coal is mixed with a binder such as dextrine and is then moulded into the form of a cone which has a height of $\frac{3}{4}$ of an inch and base of an equilateral triangle having a side of $\frac{1}{4}$ of one inch. These cones after being properly prepared are placed on a plate which is supported in a gas muffle furnace. After applying the heat for a sufficient time in the furnace, an observer looking through an observation hole can see the cones begin to melt. Upon the fusion of the cone into a sphere the observer makes an observation of the temperature indicating pyrometer, either a thermocouple or an optical pyrometer, and records the temperature. This temperature is then arbitrarily considered to be the softening or fusion temperature of the ash. There are several errors involved in this temperature determination, the more important of which are, first, that the thermocouple gives an inaccurate indication because it passes through and is heated by the flames of the burners which heat the muffle containing the cones; and second, that the atmosphere in the crucible is not truly reducing, and consequently, the iron contained in the ash is oxidized to a higher state of oxidation with the liberation of heat within the cone itself. It is estimated that the temperature measured and indicated by a thermo-couple pyrometer is in error by about 50° C. A difference of this sort, however, may be very important when considered from the economic point of view of a purchaser or seller of coal. In addition to the inaccuracy of the temperature measurements by the same and different ob-

servers, the cone method is a cumbersome and time-consuming procedure which requires complicated equipment.

Many attempts have been made to replace the present cone method with a satisfactory, reliable, accurate and practical method but nothing so far has been successful. An electrical method which was proposed and discarded, contemplated manual adjustments and operations for effecting the melting or softening temperature of coal ash and for determining the temperature at which the melting occurred. This method has many objections, the principal among which are that the manual operations do not give uniform heating, treatment and results; that two operators are required in order to make a temperature determination, one operator manipulating the electrical heating and the other making the pyrometer adjustments and observations; and that the procedure is impractical for commercial and industrial purposes and is not absolutely dependable and reliable.

An object of the present invention is to provide a simple, reliable, and practical procedure and a compact, substantially automatic, and electric apparatus for carrying out the aforesaid procedure.

Other objects and advantages of the invention will become apparent from the following description taken in conjunction with the accompanying drawings, in which

Fig. 1 is an elevational view, partly in section for purposes of clarity, of a preferred embodiment of the invention;

Fig. 2 is a sectional view taken on the line 2—2 of Fig. 1;

Fig. 3 is a sectional view taken on the line 3—3 of Fig. 2;

Fig. 4 is a sectional view taken on the line 4—4 of Fig. 1;

Fig. 5 shows a fragmentary view, partly in elevation, of the rheostat used in my apparatus;

Fig. 6 illustrates a side elevation of the instrument for melting the materials and for observing and measuring the melting temperatures;

Fig. 7 is a diagrammatic illustration of the electrical connections; and

Fig. 8 depicts a modified cover used for covering the heating element of the instrument shown in Fig. 6.

Referring more particularly to Figs. 1 and 2, the letter M designates a motor which actuates an automatic rheostat R. This rheostat controls electric current flowing through transformer T from supplying lines L when switch S is turned on, to instrument I. In order to make a compact structure capable of being handled as a unit, it is preferable to mount the motor, the switch, the rheostat and the transformer on a solid base B.

Within instrument I a heating element 1 constituted of platinum or tantalum for melting the materials to be tested is incorporated. This heating element is preferably held at its ends by clips 2 which may be integral with posts 3. Any desired way of mounting posts 3 on insulating foundation 4 may be employed but the posts are herein shown as being secured to the foundation by means of threads 5. Rods 6 screw into or connect in some other suitable manner with posts 3 so that electricity can flow from terminal connectors 7 to posts 3 and thus to heating element 1. In order to establish a good electrical connection between the heating element 1 and the post 3, a clamping screw 8 is provided on each clip 2.

The foundation 4 may constitute the base of the instrument 1 or, as in the illustration, may be joined to a metal frame 9 by a plurality of screws 10 or the like. As the foundation 4 has to withstand heat as well as electricity it is preferably made of a material such as asbestos wood or the like. Cut or otherwise formed in foundation 4 is a channel 11 for receiving a refractory cover 12 which has a transparent window 13 provided in its upper face. Between the lower edge of cover 12 and the bottom of the channel, a gasket 14 is provided so as to maintain a gas-tight joint. In practice it is desirable to make the cover 12 of high-temperature glass (e. g. pyrex glass) and to protect the glass with a metal shell 15 having an opening for window 13.

When a material is being tested that should be protected against oxidation, the air trapped under cover 12 is withdrawn through channel 16, connection 17, and tube 18 leading to an evacuating pump (not shown). Any suitable pump which gives a practically perfect vacuum may be employed as one skilled in the art will readily understand. In order to measure the pressure existing in the space under cover 12, a channel 19 leading to the said space is provided in foundation 4 which is connected via inlet 20 and tube 21 to an absolute gauge 22. This gauge has a scale 23 from which the pressures may be read directly. Of course, neutral, reducing or other gases can be sucked into the space under the cover so that the heating and melting operation can be conducted in any desired atmosphere.

Projecting upwardly from metal frame 9 is an arm 24 which carries a branched bracket 25. This bracket has two sockets for mounting a monocular telescope magnifier 26 (e. g. a Carl Zeiss telescopic magnifier) and a radiation pyrometer 27 known commercially as an "Ardometer." The magnifier 26 and the pyrometer 27 are mounted in the bracket at such an angle to each other that their optical axes 28 and 29 respectively intersect each other at about a point P on the

heating element where the material to be tested is placed. On axis 29 and at the focus of the radiation pyrometer, a thermo-element 30 is located. All the heat radiation received by the pyrometer is concentrated and focused on thermo-element 30 which generates a small electric current. By passing the generated current to a galvanometer 31 via conductors 32 and 33 the temperature existing at point P may be measured and indicated on scale 34. This scale may be graduated in any suitable units, as for instance, in degrees Fahrenheit. In order to be able to focus the pyrometer and the telescopic magnifier, each is provided with an eye lens 35 and 36 respectively.

Electricity is supplied to the heating element of instruments I through conductors 37 and 38 which are connected respectively to the outlet terminals 39 and 40 of the secondary side of transformer T. The primary side of the transformer is connected to a binding post 41 at one end of rheostat R by conductor 42 and to switch S by conductor 43. The switch is also provided with a connection 44 to main electrical supply lines L whereas the rheostat has a connection 45 extending from binding post 46 to a conductor 47 which leads to supply lines L.

The rheostat has the usual electrical connections in that binding post 46 is united to the right end of helical resistance coil 48 by means of ring 49 and binding post 41 is electrically secured to the left end of slide bar 50 (see Fig. 5). Mounted on the slide bar is a carriage 51 which is provided with spring contact clips 52. These clips bear against the turns of the resistance coil and thus establish a connection between the slide bar and the resistance coil (see Fig. 4). The upper part of the carriage has a member 53 slidably positioned on a rotatable helically threaded rod 54. By providing a pin clutch 55 of well known construction, the member 53 and thus the carriage 51 may be either coupled with or uncoupled from rod 54. When the pin 56 of clutch 55 is in mesh with the threads of rod 54 as shown in Fig. 4, the carriage is coupled with rod 54. By pulling the pin out and turning it 90° across lug 57, it bears against the face of member 53 and thus maintains an uncoupled condition between the carriage and the rod. In this condition the carriage may be moved back and forth on the slide bar.

The threads on rod 54 may be of any particular type but I have found that it is preferable to make the pitch of the threads on the right hand portion of the rod about one half of the pitch of the threads on the left hand portion. This means that it will take about twice as long for the carriage to move across the right portion of the rheostat as it will to move across the left portion

of the rheostat. In order to provide for the rotation of the rod it is appropriately mounted in plates 58 which are suitably attached to the sides of the rheostat and it has a gear wheel 59 secured to one end, herein shown as the right end. The gear wheel 59 is driven by a worm gear 60 on motor M which is electrically connected to supply lines L via conductors 47 and 61. By selecting a motor having a suitable speed and by using a worm and gear having appropriate construction, as one skilled in the art understands, the rod may be rotated at a speed sufficient to move the carriage across the right side of the rheostat in about five to six minutes and across the left side in about three minutes.

The electrical connections for the motor and for the transformer rheostat and heating element may be of any appropriate arrangement. In Fig. 7 a diagrammatic illustration of a preferred arrangement is depicted. The supply lines L may be those of an ordinary light circuit of 110 volts or of any other suitable source of electric current. Interposed between main supply lines L and circuit lines 100 and 101 is a switch 102. Conductors 103 and 104 lead from circuit lines 100 and 101 respectively to motor M. The circuit line 100 is attached to one end of the primary side P of transformer T by conductor 105 and the other circuit line 101 is connected to one end of rheostat R by conductor 106. The rheostat is provided with a movable slide 107 which has an electrical connection 108 extending between it and the other end of the primary P. The secondary side K of the transformer is connected directly with the heating element 1 of instrument I by means of conductors 109 and 110. When switch 102 is thrown in so as to connect supply lines L with circuit lines 100 and 101, the motor is actuated and the heating element is supplied with electrical energy. By this simultaneous action the heater begins to heat and the slide begins to move from the left end of the rheostat to the right end and thus cuts out the resistance of the rheostat and increases the electric current flowing to the transformer to a maximum.

In the process of determining the melting or fusing temperature of coal ash a specimen of the ash resulting from either a proximate or an ultimate analysis of a sample of coal weighing about one gram is first placed in a small agate mortar. Sufficient water, about one or two drops, is added to the ash or other refractory material which is then rubbed for a few minutes with an agate pestle to break up any coarse particles. The mortar and its contents are dried with the pestle and when dry, the mass is loosened up by rubbing it with the pestle. The material (ash or the like) to

be tested is then ready to be placed on the heating element in the instrument I to determine its fusion point. With this mode of preparing a sample all the complicated and elaborate work required for the preparation of the sample in the cone method is eliminated. This saves considerable time, labor and money.

After the specimen of material is placed upon the heating element as shown at P in Fig. 1, and the telescopic magnifier and the radiation pyrometer are focused on material P, the switch S is turned on so as to supply electricity simultaneously to motor M and to the circuit including the transformer, the rheostat and the heating element. The motor immediately begins to move carriage 51 on the rheostat from its initial position at the left end of the rheostat where all of the resistance of coil 48 is in the circuit to the right end where all of the resistance is cut out of the circuit.

When an electric current of 110 volts and 60 cycles is used in the supply lines L, a transformer stepping down the voltage to 10 volts is employed, and a resistance of 10.8 ohms is used in the rheostat, a temperature of about 900° F. will be available in a heating element constituted of a platinum strip $2\frac{1}{2}$ inches long, $\frac{3}{8}$ of an inch wide and $\frac{2}{1000}$ of one inch thick when the carriage traverses about one half of its travel on the slide bar, i. e. when the carriage reaches the junction between the coarse and the fine threads. The time consumed in traversing one half of the slide bar is approximately three minutes.

During the remainder of the travel of the carriage, the operator observes the material being heated on element 1 through the telescopic magnifier 26. As the carriage nears the end of its travel sufficient electricity has passed through the heating element to generate heat enough to cause softening of the material or specimen under test. Upon initial fusion of the specimen a white border appears around the edge of the specimen or pellet of ash. As the temperature increases the border spreads over the surface of the pellet and finally covers the entire surface. When the black spot disappears from the center of the pellet, the final fusion is reached. Since ash has no true melting point, it is desirable to obtain the initial fusion temperature as well as the final fusion temperature. With the cone method it has been very difficult, if not impractical, to obtain the initial fusion temperature.

Throughout the testing process, the pyrometer is focused on the specimen or pellet and is affected by the heat radiated therefrom. Since cover 15 envelops practically the entire pyrex jar black body conditions are approximated in the space under the

cover. The electric current generated in the thermo couple 30 by the heat is transmitted to the galvanometer so that the temperature of the pellet is continually and automatically being indicated. When the operator first observes the appearance of the white border around the edge of the pellet, he looks at the galvanometer and notes the temperature. Then he continues to observe the specimen through the magnifier and when the black spot disappears from the center of the pellet he observes and notes the temperature indicated by the galvanometer. In this manner the initial and final fusion temperatures of the specimen of ash or other material are obtained practically automatically. No manual adjustments are required during the test and one operator can determine the initial and final fusion temperatures in about eight minutes. With the cone method the time required to make one test is about three to four hours of laborious, hot and inconvenient work. The present method and apparatus completely eliminates these disadvantages.

When temperatures under about 2800° F. are to be measured, a strip of platinum is to be used for the heating element, whereas when temperatures over 2800° F. under about 3500° or 3600° F. are to be measured a strip of tantalum is used as the heating element. It is necessary that the atmosphere surrounding the tantalum strip at high temperatures must be absolutely free from oxygen otherwise the tantalum will be oxidized. If a high vacuum is obtained under the glass jar the atmosphere is safe enough to heat the tantalum. Of course, it is only necessary to use tantalum for very refractory materials which fuse above the melting temperature of platinum.

The temperature indicating device or galvanometer may be calibrated by melting various metallic pellets on the heating element in a well known manner. Thus, for example, tin, zinc, copper, nickel, and other appropriate metals may be used to calibrate the temperature scale 34. When the galvanometer is properly calibrated the improved apparatus gives extremely accurate results which can be reproduced by the same or different observers within a remarkably close range. Of course, other materials such as non-metallic substances may be used in the calibration of the scale as one skilled in the art will readily understand.

In Fig. 8, has been depicted a modified cover for the envelopment of the heating element 1 of instrument I. This cover consists of a metal shell 15a which has a window opening 15b provided with a recess 15c. This recess is ground smoothly to provide a seat for window 15d which is finely polished and made of a uniform thickness so that it will not interfere with the passage of light

and heat radiations. Ordinarily the window is only given a frictional fit in the window opening because the vacuum will cause a tight joint to be made. In some instances it may be well to seal the window in place by appropriate means such as by cementing with glycerine and litharge as is well known.

It is to be observed that the invention provides a process and apparatus whereby the fusing or melting point or temperature of refractory or non-refractory materials can be very accurately observed without the usual inconvenience, the elaborate apparatus required in the present methods of fusing or melting point determinations are eliminated, an accurate and uniform temperature control and uniform temperature increase over a definite length of time can be obtained, an accurate measurement of the temperature at which the fusion of the refractory or non-refractory material takes place can be made, only very slight quantities of substances are required for the determination of the fusing or melting points of such substances, and the fusing or melting points of a number of specimens may be determined simultaneously.

It is to be noted that the invention provides a process and an apparatus which eliminates a cumbersome gas furnace requiring a motor-driven blower, the consumption of considerable volumes of gas and air and quantities of electricity, a large floor-space, the use of a separate room required for a gas furnace liberating a large amount of heat, and the difficulty of fusing highly refractory ashes in a temperature beyond 2800° F.

Claims:

1. In an apparatus for the determination of melting temperatures of materials, the combination which comprises a container, means associated with said container for controlling the atmosphere therein, electrical means incorporated in said container for conducting electricity, said means including a conductive element capable of being heated to melting temperature by electricity and of bearing specimens of materials to be melted, a window provided in said container for observing a specimen carried on said conductive element, a pyrometer focused through said window on the specimen in the container, and a magnifier also focused on said specimen.

2. In an apparatus for the determination of melting temperatures of materials, the combination which comprises an instrument for electrically melting materials consisting of an electrical heating element enclosed in a container whose atmosphere can be controlled, an observation window in said container, and a pyrometer and telescopic magnifier capable of being focused on said heat-

ing element; automatic means for controlling the electricity flowing to the heating element contained in the said instrument; and a meter associated with the pyrometer for indicating the temperature of the heating element.

3. In an apparatus for the determination of melting temperatures of materials, the combination which comprises an instrument for electrically melting materials consisting of an electrical heating element enclosed in a container whose atmosphere can be controlled, an observation window in said container, and a pyrometer and telescopic magnifier capable of being focused on said heating element; means for controlling the supply of electricity to said heating element comprising a step-down transformer, a rheostat having an automatically movable carriage, a motor operatively associated with said rheostat for moving said carriage, and a switch for simultaneously controlling the electricity flowing to said transformer and to said motor; electrical connections between the secondary side of said transformer and the heating element of said instrument for conducting the electricity thereto; and a meter electrically connected to the aforesaid pyrometer for automatically indicating the temperature of the heating element.

4. In an apparatus for the determination of melting temperatures of materials, the combination which comprises an instrument for electrically melting materials including a pyrometer and a telescopic magnifier capable of being focused on a specimen of the materials undergoing melting; means for controlling the supply of electricity flowing to said instrument consisting of means constituting a source of electricity, control means electrically connected with said source to vary the flow of electricity going to the instrument, means for automatically operating said control means to cause a substantially uniform increase in the quantity of electricity going to the instrument, and means for switching the electricity on and off; and a meter electrically connected to said instrument for indicating the temperature of a specimen undergoing a determination.

5. The combination set forth in claim 3 in which the carriage is supported on a rod having threads of such a character that the carriage is moved twice as fast on the starting portion of the rheostat as on the finishing portion.

6. An instrument for the determination of the melting temperatures of materials comprising in combination a supporting frame and arm carrying two brackets, an insulating foundation secured to said supporting frame and having electrical and gas connections incorporated therein, an electri-

cal heating element mounted on said foundation and connected to said electrical connections, a refractory cover seated in a channel in said foundation so as to enclose said heating element said cover having an observation window, a gasket fitted in said channel under the edge of said cover and adapted to make a gas-tight joint, a pyrometer mounted in one of said brackets so as to be capable of being adjusted and focused through the observation windows on the heating element, and a telescopic magnifier mounted in the second bracket so as to be capable of being focused on said heating element.

7. An instrument such as set forth in claim 6 in which the refractory cover is entirely enveloped except for its observation window in a metallic shell whereby black body temperatures are approximated in the space under the cover.

8. The process of determining the melting temperatures of materials which comprises establishing a chamber within which black body conditions are approximated, heating a surface supporting a specimen of the material whose melting temperature is to be determined within the aforesaid chamber, automatically regulating the heating of the said surface and specimen to increase the temperature thereof at a substantially definite rate within a given restricted time limit, continuing the application of heat to cause the melting of the specimen of material under determination, maintaining an element thermally sensitive to the radiations emitted by said specimen and heated surface within such a range of the latter that the radiant energy emitted from the heated surface is effective thereon, and measuring the temperature at which said specimen melts with the aid of said thermally sensitive element.

9. The process of determining the melting temperatures of materials which comprises establishing a chamber within which black body conditions are approximated, causing electricity to flow through a metallic strip supporting a specimen of the material whose melting temperature is to be determined within the aforesaid chamber, automatically controlling the electricity flowing through said strip to increase the temperature thereof at a substantially definite rate within a given restricted time limit, continuing the application of heat to cause the melting of the specimen of material under determination, maintaining an element thermally sensitive to the radiations emitted by said specimen and heated surface within such a range of the latter that the radiant energy emitted from the heated surface is effective thereon, and measuring the temperature at which said specimen melts with the aid of said thermally sensitive element.

10. The process as set forth in claim 8 in

which the increased heating is temporarily discontinued during the step of measuring the melting temperature of the specimen.

In testimony whereof I have hereunto set my hand.

LEA FRANK DE GRAAF,
Executrix of Gerrit A. de Graaf, deceased.

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