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THE CONSTRUCTION AND OPERATION OF A MICRO-CONE-SOFTENING APPARATUS

A. JONGEJAN

MINERAL SCIENCES DIVISION

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THE CONSTRUCTION AND OPERATION OF A MICRO-CONE-SOFTENING APPARATUS

by

A. Jongejan*

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SYNOPSIS

The construction and operation of an apparatus with with which cone-softening temperatures can be determined are described. The temperature of the furnace, which contains the cone, is controlled and can be recorded. The silhouette of the cone can be photographed with a standard grid as background, so that its extent of deformation can be determined quantitatively. The apparatus is built from components that can also be used in other combinations for other purposes.

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Direction des mines

Bulletin technique TB 89

LA CONSTRUCTION ET LE FONCTIONNEMENT D'UN APPAREIL DE DÉTREMPE DES MICRO-CÔNES

par

A. Jongejan*

RÉSUMÉ

L'auteur décrit la construction et le fonctionnement d'un appareil qui sert à déterminer les températures de détrempe des micro-cônes. La température du four qui contient le cône est contrôlée et peut être enregistrée. On peut photographier la silhouette du cône en se servant d'un quadrillage régulier, de sorte que le degré de déformation peut être déterminé quantitativement. L'appareil est fait de pièces qui peuvent aussi être utilisées dans d'autres montages servant à d'autres fins.

** Préposé aux recherches scientifiques, Section de la chimie physique, Division des sciences minérales, Direction des mines, Ministère de l'Énergie, des Mines et des Ressources, Ottawa, Canada.

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FIGURES

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INTRODUCTION

The procedure for determining furnace temperatures by the "cone-bending" method was developed at the turn of the century. It is still used for the same purpose, particularly in the ceramic industry, but it is also used in the study of melting phenomena of mixtures of inorganic compounds, such as ceramic materials, slags, fuel ashes and glasses. In addition, it is used in the study of sintering phenomena, since the change in volume of a sample can be correlated with change of temperature. The reactions involved in melting phenomena are those that occur in multi-component phase systems when a change of state takes place on heating.

A triangular pyramid - the so-called cone - which normally has one face perpendicular to the base, is made from the pulverized sample. In order to increase the cohesion of the sample, it is usually mixed with a small quantity of a binder. The cone is then gradually heated. It starts to soften and bends down at a certain temperature due to the formation of liquid in it. The temperature at which the apex of the specimen touches the base is considered to be the "cone-bending point". This phenomenon, therefore, also indicates a specific temperature, when cones of standard materials are used.

The temperature at which the cone shows any rounding of edges has been considered to be the "cone-softening point". At this point, the smallest amount of liquid that is needed to show the first observable sign of melting has been formed.

The cone-softening point is only an approximate measure of the solidus temperature of the sample, since it is influenced by factors such as viscosity of the liquid, the slope of the liquidus curve, the grain size of the sample, etc. In addition, it is also evident that many factors in the procedure depend on subjective observation of the deformation of the cone.

Except for the size and the shape of the cone, little more has been standardized in British and American "Standard" procedures. Both the British (B.S.S. 453/1932), and the American (A.S.T.M. D271/33), standard procedures recognize the initial deformation temperature or "softening point" as the temperature at which the first signs of rounding of the edges or apex of the cone are observed.

Several designs of apparatus to study the above phenomena have been developed in the course of time. Attempts have been made to improve upon the subjective observation methods [1]. A heating microscope has been manufactured by the firm of Leitz for several decades. This consists essentially of a furnace in which a small cylindrical pellet is heated, together with a photographic microscope by means of which enlarged pictures of the pellet can be taken during its various stages of deformation.

It was felt that the recording system used in the Leitz instrument could be improved upon, and that much could be gained economically by building an apparatus from components that could also be used in combination with other instruments.

The apparatus that is described in this report will produce a photograph of the silhouette of a small pellet at elevated temperatures with a standard grid as background. In addition to the shape of the specimen, the temperature at which the photograph is taken is also recorded, together with a reference to other information about the sample.

The EMF of a thermocouple inserted in the furnace can be measured to an accuracy of $\pm 1\ \mu V$ and the temperature of the bottom of the cone can thus be determined to an accuracy of $\pm 1\ °C$, using calibration standards. However, the thermal gradient from the bottom of a 3-mm-high pellet to its top is approximately 3 deg C.

The maximum temperature at which the furnace can be operated for any convenient length of time is 1600°C. The furnace design leaves open the possibility of using other resistor elements that may reach higher temperatures. The possibility of using other heating procedures could also be considered. Instead of the heat being developed by electrical resistance in the furnace, gas flames, carbon-arc image or other electrical methods are feasible. The sample's comparatively small size and its considerable distance from the optical system leave ample room for the installation of devices other than the furnace described in this report.

PRINCIPAL FUNCTIONS OF THE APPARATUS

A small cylindrical pellet, which is pressed from the powdered sample, is heated in a horizontal tube furnace having a platinum-wound resistor as heating element. The temperature of the furnace is regulated by an improved type of Roberts controller. In this type of controller, the platinum winding of the heating element of the furnace forms a part of a Wheatstone bridge circuit from which the signal is amplified and fed back into the power circuit [2].

The temperature of the furnace is measured with a Pt-Pt/10% Rh thermocouple which is connected to a potentiometer. The null-detector for the potentiometer is a microvoltmeter, whose signal can also be recorded. Several voltage ranges can be indicated by the microvoltmeter and/or the recorder, from the smallest range of 100 μV for a full-scale deflection on the scale of 100 divisions, to the largest EMF produced by the thermocouple.

The recorder is used to judge whether the temperature of the furnace has reached equilibrium conditions. The record obtained also serves as a check on possible temporary changes in temperature during long runs. This is essential since the shape of the pellet reflects its condition at the highest temperature that has been reached during any time of the run.

The furnace forms a part of an optical system which serves to produce a silhouette picture of the specimen on the film of a camera. The optical system branches out to the scale of the microvoltmeter and to a card upon which a reference to other information relating to the sample is recorded. The scale of the microvoltmeter, the reference number on the card and the specimen in the furnace are, therefore, photographed on the same picture.

The physical arrangement of the apparatus is shown in Figure 1.

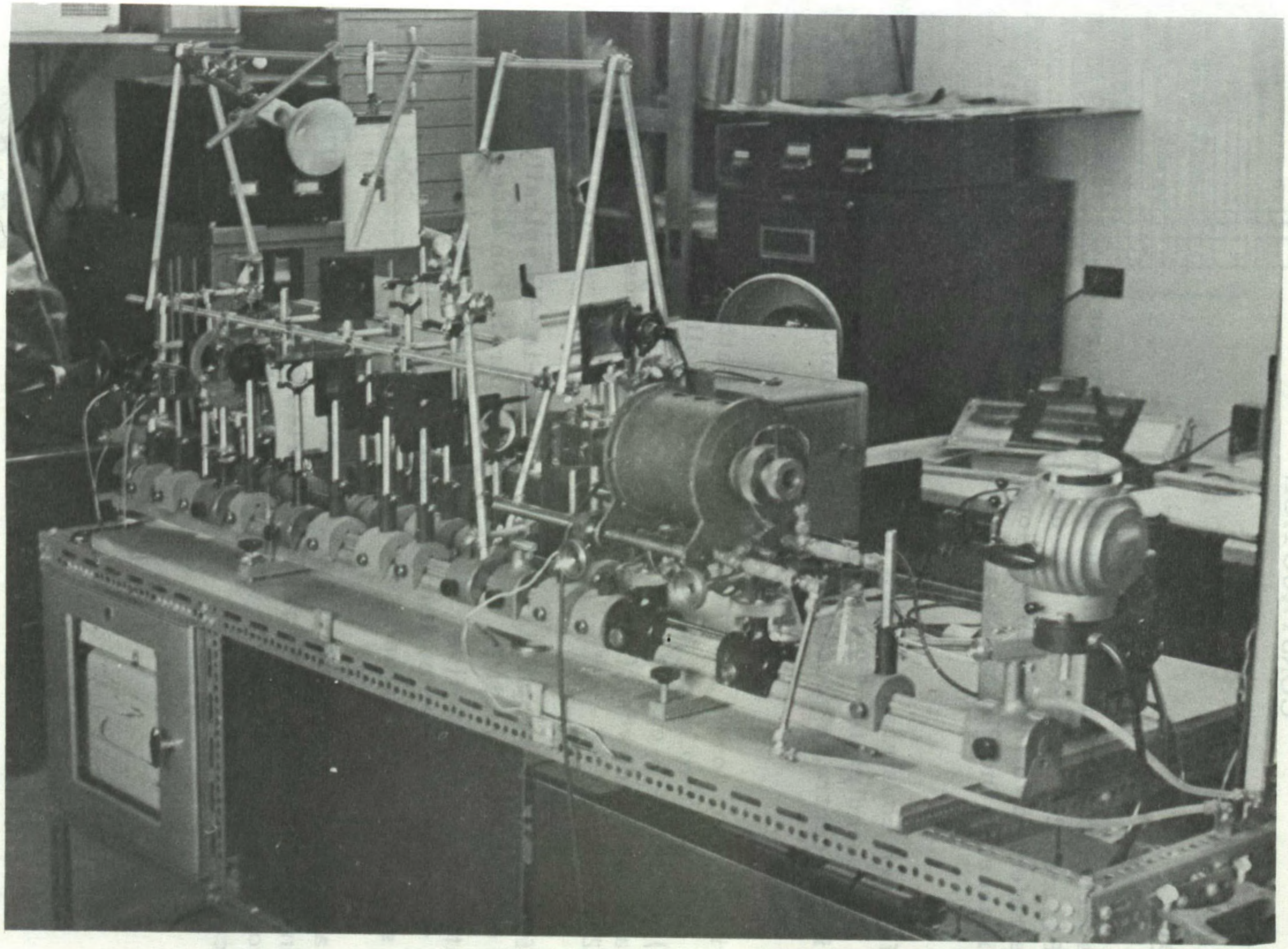


Figure 1. Arrangement of the Micro-cone-softening Apparatus.

From the front to the far end, respectively, of the optical bench, can be recognized the general-purpose microscope lamp, the furnace, and the optical train leading to the camera assembly.
At the left side, below the table top, is the recorder, which can be connected to the microvoltmeter, a part of which is visible behind the optical benches.

CONSTRUCTION

A. The Furnace

The specifications for machining the metal parts of the furnace are shown in Figures 2a, 2b and 2c. The numbers indicated in these figures will be referred to in the description of the furnace and are also indicated in Figures 3a and 3b, which are photographs of the assembled furnace.

The furnace consists of a shell (2) made from 0.125-in.-thick sheet brass. Brass end plates (3) 0.25-in.-thick are attached to it, each having a hole in the centre, to accommodate the ceramic tube, and a cooling chamber (4).

Provided in the lower part of the plates are attachments to the saddle stands (23), and two holes in each of which a hollow stainless steel pipe (12) is fitted. A carrier (9a, Detail 38) for the thermocouple-sheathing (9) and one windowcap (7) can be moved along these stainless steel pipes (12).

The furnace shell proper is cooled by 0.125-in.-ID, spirally-wound copper tubing (Figure 2b, Detail 2). Two of such spirals run side by side, carrying countercurrent water-flows. The endpieces are cooled by cooling chambers (4), which are connected through tubing (35) to the same water supply as the spirals on the furnace shell.

The adjusting mechanism of each stand (23) enables the furnace to be moved in any desired direction. The knurled knobs (24) move the furnace horizontally on the brackets (26), while the knurled knobs (25) move it vertically.

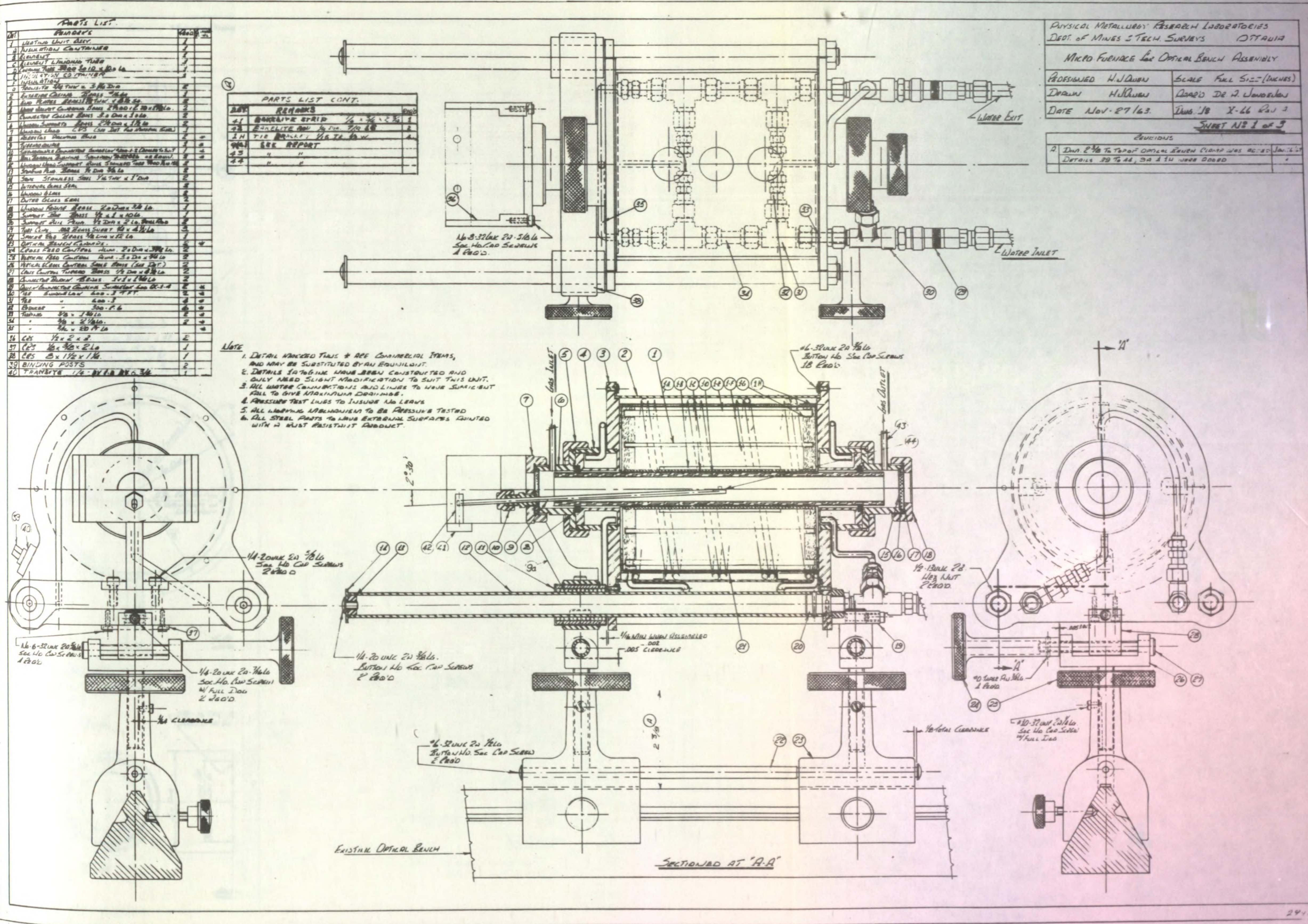

PARTS LIST
PARTS LIST CONT.
NOTE
1. DETAIL MARKED THUS * ARE COMMERCIAL ITEMS, AND MAY BE SUBSTITUTED BY AN EQUIVALENT.
2. DETAILS 1A TO 6 INC HAVE BEEN CONSTRUCTED AND ONLY NEED SLIGHT MODIFICATION TO SUIT THIS UNIT.
3. ALL WATER CONNECTIONS AND LINES TO HAVE SUFFICIENT FALL TO GIVE MAXIMUM DRAINAGE.
4. PRESSURE TEST LINES TO INSURE NO LEAKS
5. ALL WORKING MECHANISM TO BE PRESSURE TESTED
6. ALL STEEL PARTS TO HAVE EXTERNAL SURFACES PAINTED WITH A RUST RESISTANT PRODUCT.
PHYSICAL METALLURGY RESEARCH LABORATORIES
DEPT. OF MINES & TECH. SURVEYS OTTAWA
MICRO FURNACE FOR OPTICAL BENCH ASSEMBLY
DESIGNED H.J.OWEN
DRAWN H.J.OWEN
DATE NOV. 27/63.
SCALE FULL SIZE (INCHES)
SHEET NO 1 OF 3
REVISIONS
WATER EXIT
WATER INLET
GAS INLET
GAS OUTLET
EXISTING OPTICAL BENCH
SECTIONED AT "A-A"

Figure 2a.

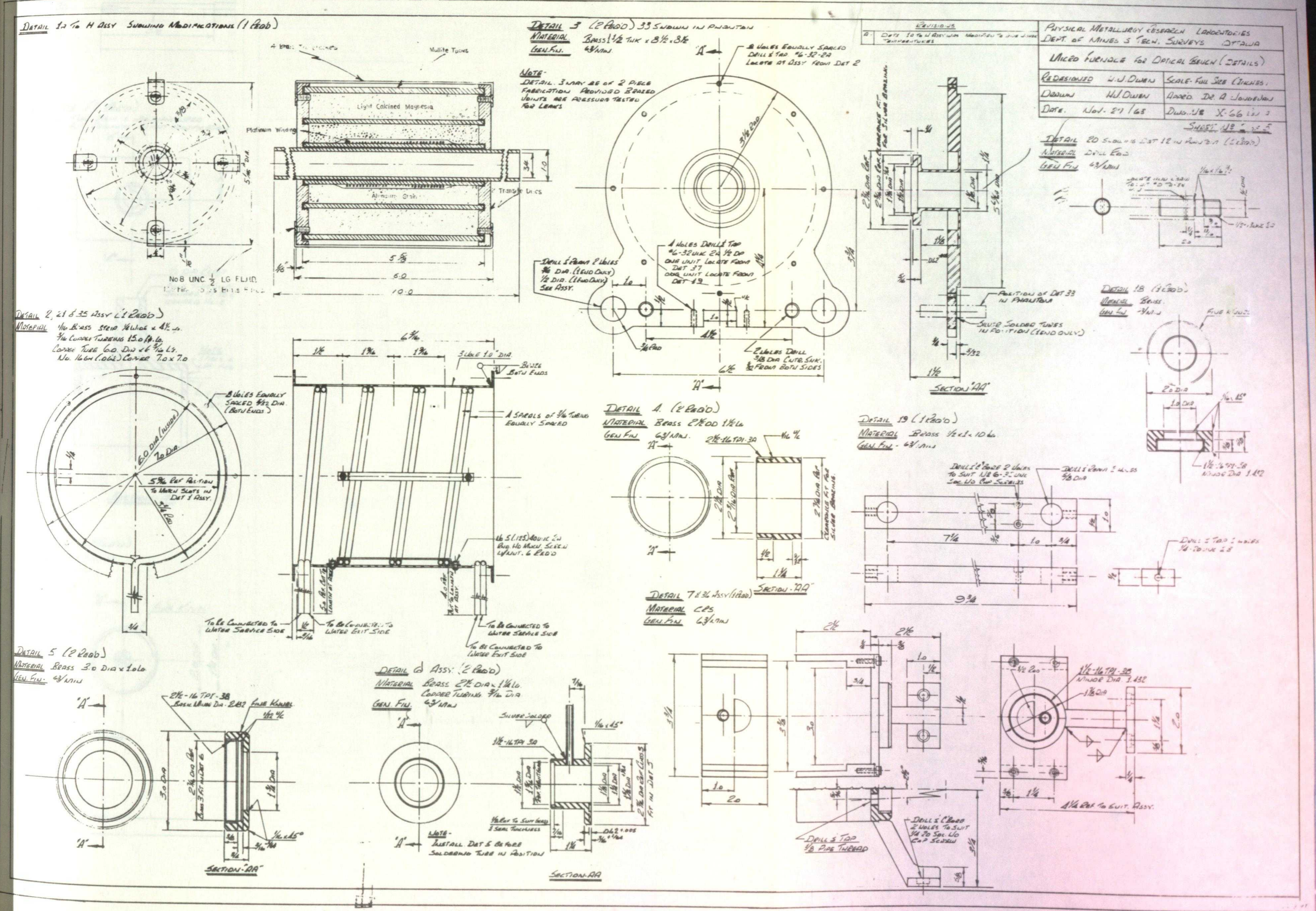

Physical Metallurgy Research Laboratories
Dept. of Mines & Tech. Surveys Ottawa
Micro Furnace for Optical Bench (Details)
Redesigned W.J. Owen
Drawn W.J. Owen
Date. Nov. 21 /63
Scale - Full Size (Inches)
Detail 1a to H Assy Showing Modifications (1 Req'd)
Mullite Tubes
Light Calcined Magnesia
Platinum Winding
Alundum Grain
No 8 UNC 1/2 LG FLHD
Detail 2, 21 & 35 Assy (1 Req'd)
Detail 3 (2 Req'd)
Detail 4 (1 Req'd)
Detail 5 (2 Req'd)
Detail 6 Assy (2 Req'd)
Detail 7 & 36 Assy (1 Req'd)
Detail 18 (1 Req'd)
Detail 19 (1 Req'd)
Detail 20 Showing Det 12 in Phantom (1 Req'd)
Section "AA"
Section-AA
To Be Connected To Water Service Side
To Be Connected To Water Exit Side
Silver Solder Tubes in Position (1 End Only)
Install Det 5 Before Soldering Tube in Position

Figure 2b.

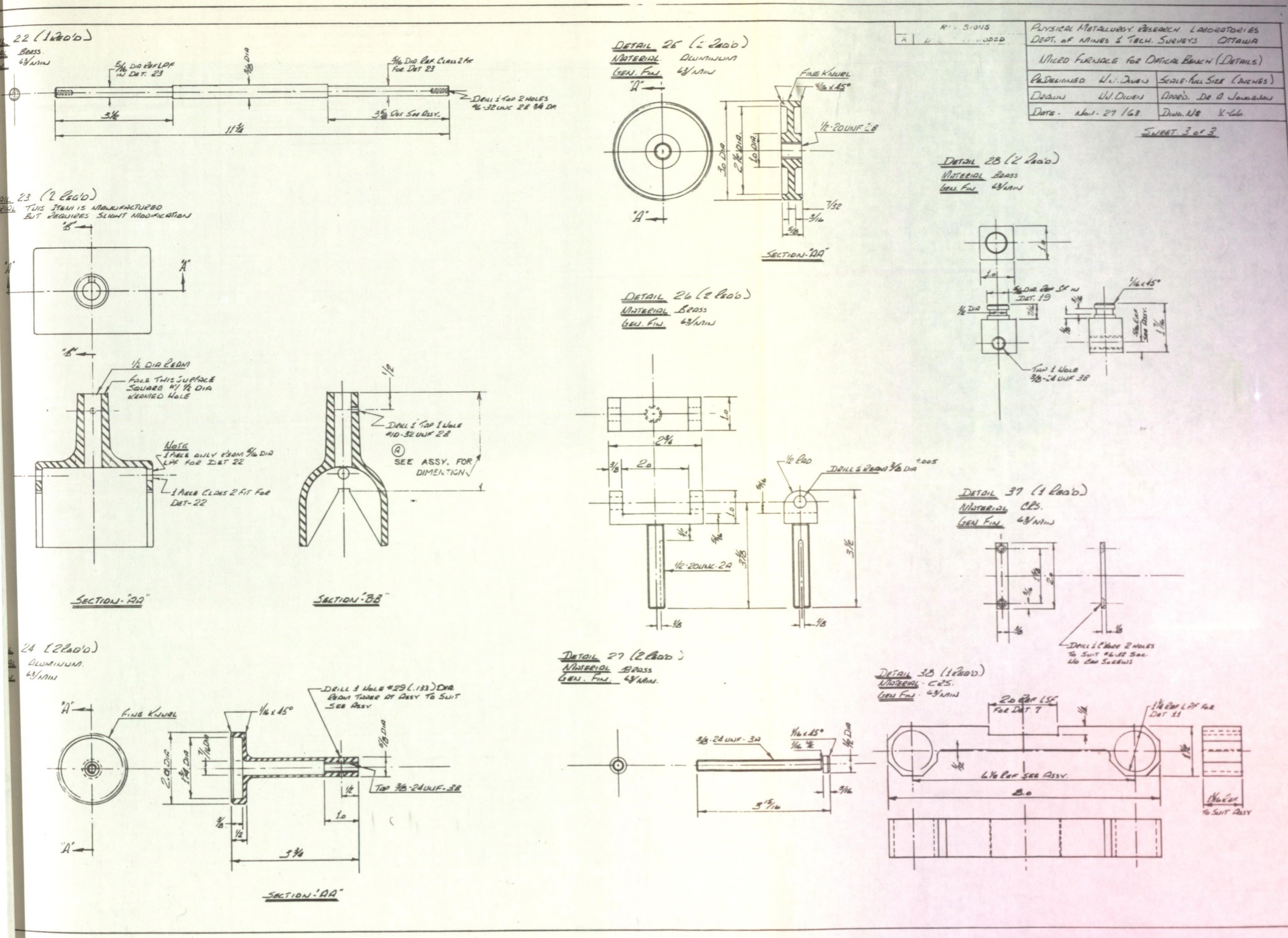


Figure 2c.

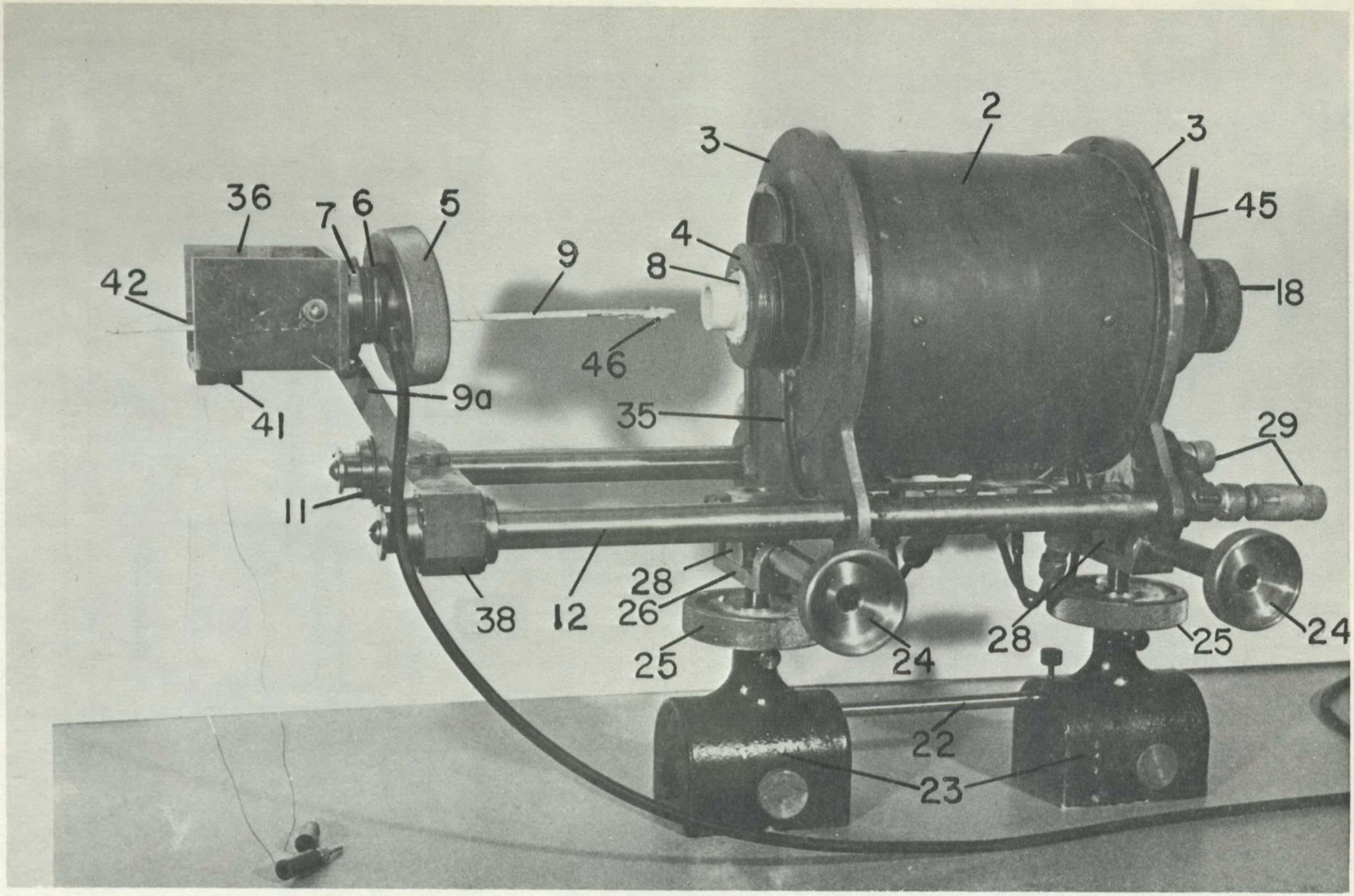


Figure 3a. Micro-cone-softening Furnace with Sample Carrier in Loading Position. The numbers refer to parts described in the text.

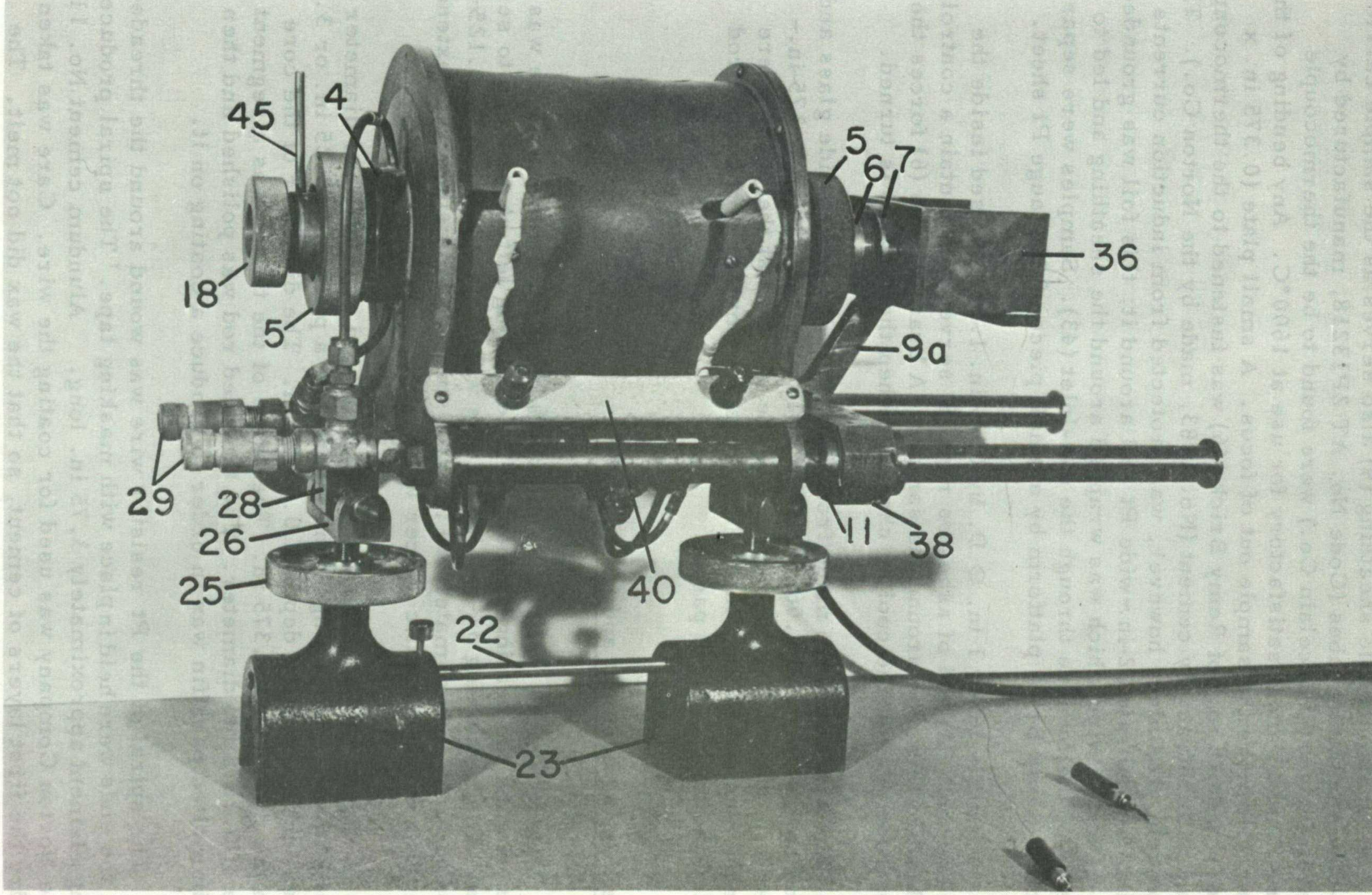


Figure 3b. Micro-cone-softening Furnace in Operating Arrangement.

The thermocouple sheathing (9) also serves as the sample carrier. Double-bore insulating tubes (Code No. AT 2P13218, manufactured by McDanel Refractory Porcelain Co.) were found to be the thermocouple sheathing that was most satisfactory for use at 1600°C. Any bending of the sheathing will move the sample out of focus. A small plate (0.375 in. x 0.375 in. x 0.0625 in.) of Remy Brick (46) was fastened to the thermocouple sheathing with refractory cement (No. 1183, made by the Norton Co.). The thermocouple sheathing, however, was protected from induction currents by winding a perforated 2-in.-wide Pt foil around it; this foil was grounded by a 32-gauge Pt wire which was wrapped around the sheathing and led to the outside of the furnace through the gas-inlet (43). Samples were separated from the Remy Brick platform by a small piece of 22-gauge Pt sheet.

A mullite tube, 1 in. O.D. by 0.75 in. I.D., is used inside the heating element. A seal of asbestos rope (8) serves to maintain a controlled atmosphere in this tube around the sample. A brass sleeve (6) forces the asbestos rope against the cooling chamber when the ring (5) is turned.

The windows were made from 2-in. by 3-in.-microslide glass and sealed on both sides with 0.0625-in.-thick silicone rubber. A 0.375-in.-diameter hole was drilled through one of the windows at the place where the thermocouple sheathing enters the furnace, by means of a copper rod embedded in boron carbide paste.

B. The Heating Element and its Insulation

A length of 40 feet of 20-gauge 60% Pt-40% Rh resistor wire was used for winding the heating element, 1.5 feet being left at each end to serve as leads. The remaining 37-foot length was wound tightly around a 0.125-in. drill rod to form approximately 30 in. of a spiral. This spiral was extended to 60 in. by moving a plastic spacer in between the wire.

A thread was cut in a piece of Lucite rod of 1.4375 in. diameter and approximately 1 foot long. The thread had a pitch of 0.3125 in. or 3.2 turns per inch, and had a depth of 0.03125 in. The diameter of the core between the thread was 1.375 in. The shape of the thread was a segment of a circle of 0.1875 in. diameter. The threaded rod was polished and then dipped in molten paraffin wax in order to produce a coating on it.

The spiral of the Pt resistor wire was wound around the threaded rod and the ends were held in place with masking tape. The spiral produced a heating element approximately 3.75 in. long. Alundum cement No. 1139 from the Norton Company was used for coating the wire. Care was taken to air-dry the first layers of cement, so that the wax did not melt. The Lucite rod was then twisted out of the cement cylinder, and two Lucite plugs, provided with a rim at one end, were inserted in order to finish the tube to

the desired length of approximately 5.25 in. After the plugs were removed, the inside of the tube was coated with Alundum cement, so that the resistor wire was well embedded. The whole tube was pre-fired in a furnace to 1500°C.

The "ceramic" part of the furnace, containing the heating element, is shown in Figure 4. It consists of a mullite protection tube, 4.375 in. I.D. and 4.75 in. O.D., which encloses an insulating cylindrical space filled with magnesium oxide. This, in turn, encloses another mullite protection tube, 2.5 in. I.D. and 2.75 in. O.D. The space between this tube and the heating element was filled with Alundum insulating grain. These tubes, as well as the heating element, fit into slots cut in 0.375-in.-thick Transite disks, which are held together by 4 brass tie-brackets. The same technique, as was described on page 12, was used for drilling the holes in the side of the 2.75-in.-and 4.75-in.-diameter mullite tubes, in order to lead the resistor wires to the outside of the furnace shell. The ceramic portion of the furnace, as shown in Figure 4, could be assembled outside the furnace, and slid into the shell as one unit when any repacking of insulation or a replacement of the heating element was desired.

C. Furnace Power Controller

A controller, incorporating a circuit based on that described by Roberts [2], maintained the temperature in the furnace at any desired level.

It had been the intention to use another type of controller, consisting of a transistorized circuit. However, the development of a satisfactory form of this type of controller was not completed at the time of the preparation of this report.

The elaborate design of the heating element was necessitated because of the limitations of the Roberts controller insofar as the magnitudes of required current and potential were concerned. With a controller having greater flexibility in output, a closely-wound "non-spiral" resistor winding would be sufficient to reach the required 1600°C temperature.

D. Instruments for Temperature Measurement

The thermocouple wires, which come out of the 0.125-in.-diameter double-hole ceramic tubing, are fed through small holes in bakelite posts (42) which are attached to a 0.25-in.-thick bakelite strip (41), fitted to two flanges (36) of the carrier (9a). The wires are wound once around the posts, so that no tension can be exerted on the thermocouple sheathing when the carrier is moved back and forth along the stainless steel pipes (12). Each

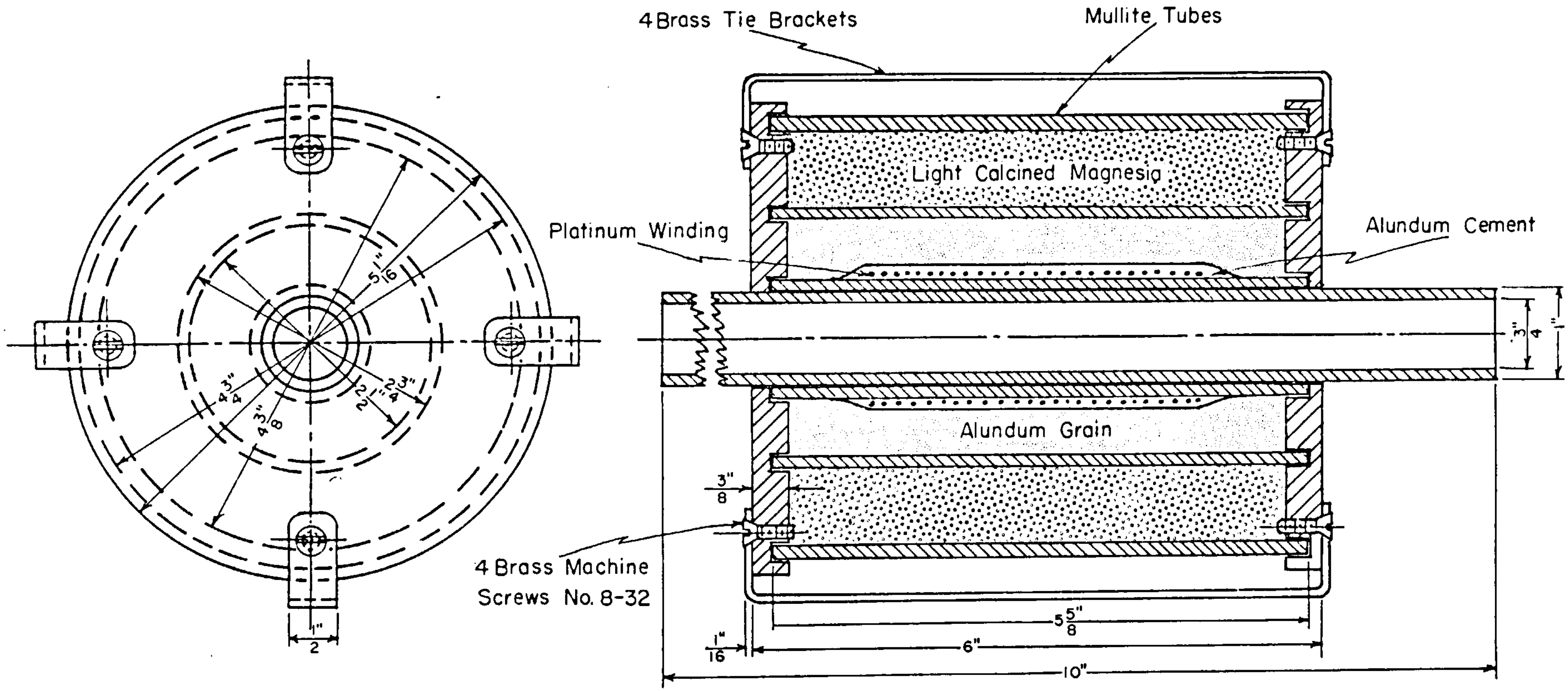


Figure 4. The Ceramic Part of the Furnace Containing the Heating Element.

of the thermocouple wires, which can be insulated by pieces of ceramic tubing, is connected to a gold wire and forms a cold junction in a vacuum bottle filled with melting ice.

The EMF of the Pt-Pt/10%Rh thermocouple is measured by means of a Leeds and Northrup Type K3 Universal potentiometer in conjunction with a Philips D.C. microvoltmeter, Model GM 6020/03. In this system, the potentiometer can be used as a "bucking" potential, and the deviation from the null-point read by the microvoltmeter. The smallest voltage range that can be covered by a full-scale (100 div.) deflection of the microvoltmeter is 100μV.

This system has the advantage that it leaves the operator free to spend time on the photography. Since the scale of the microvoltmeter is recorded on the photograph, together with the silhouette of the sample, the temperature of the sample need not be read by the operator while he is engaged in taking the photographs.

The microvoltmeter can also be connected to a Philips recorder, Model PR 2216A/21 with automatic compensator, which is similar to Model PR 2210/21 but is intended for use with AC voltage of 110 V, 60 c/s. The recorder is required for checking any possible temporary change in temperature during the run.

It should be noted that, in the circuit shown in Figure 5, the microvoltmeter is connected in series with the thermocouple circuit of the potentiometer. Due to its high impedance, it cannot be connected to the galvanometer posts of the potentiometer. When the potentiometer has to be calibrated against the standard cell, the microvoltmeter connections have to be switched to the galvanometer posts. In that case, it serves only as a null indicator.

The whole system is guarded against leakage currents and is grounded as is shown in Figure 5.

Gold, diopside, pseudowollastonite and palladium were used periodically to calibrate the thermocouple.

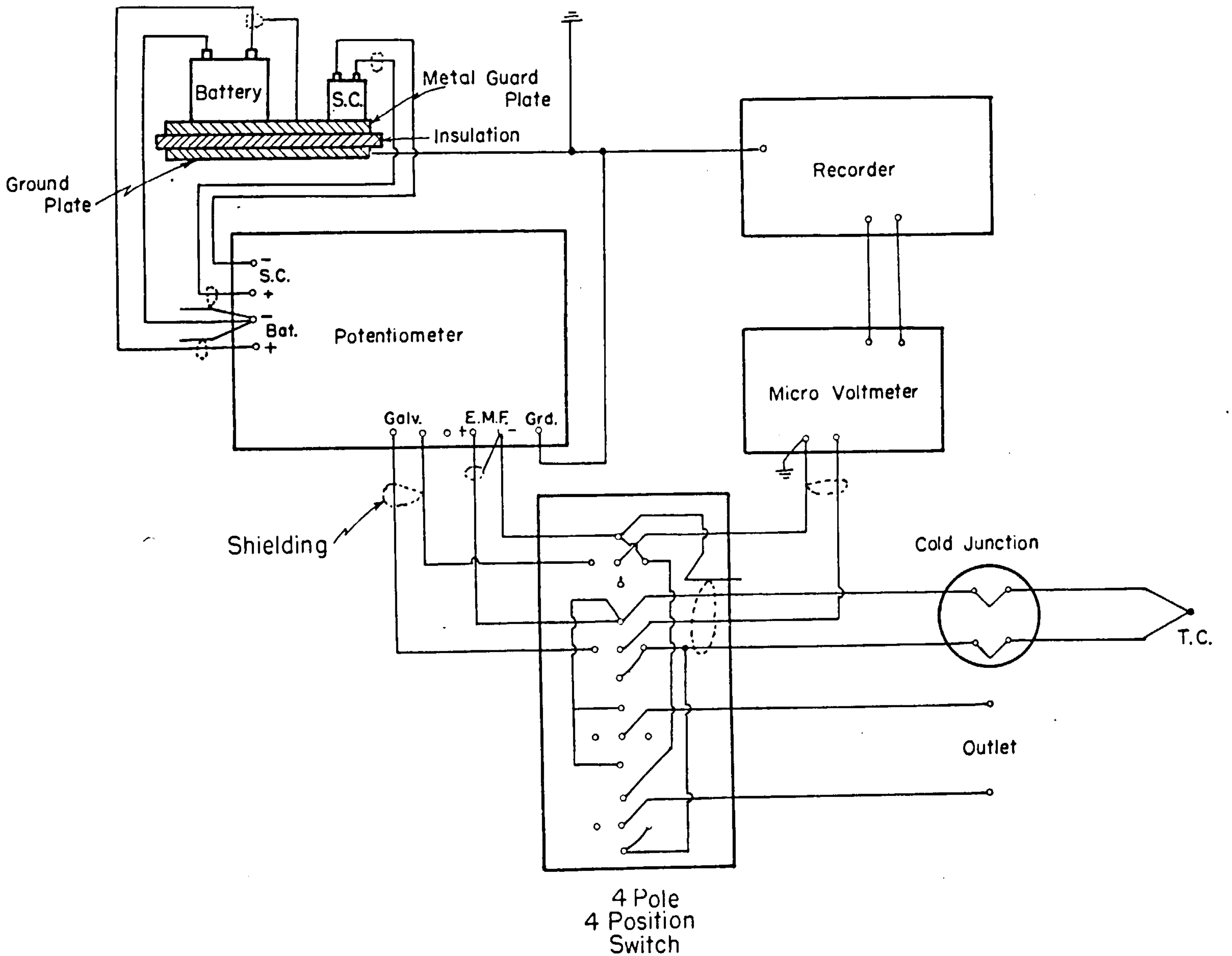


Figure 5. The Electrical Circuit for Determining the Temperature in the Furnace. (A junction board consisting of pinjacks can be used instead of the switch.)

E. The Optical System and its Alignment

The optical system consists of a 2-metre optical bench, carrying the furnace, the light source, the camera and the necessary optical equipment. A 1-metre-long optical bench serves to project an image of the scale of the microvoltmeter and the reference number of a card, on to the photographic film, below the image of the specimen in the furnace. The section of the optical system used to project the reference number is fastened to a bracket of Flexaframe construction, which is supported by saddlestands on the optical benches. The card carrying the reference number is illuminated by a system consisting of a 300-watt reflector floodlight, two lenses and three screens. It is essential that stray light, either from the outside or from the floodlight, should not interfere with the rest of the optical system.

The details of the optical system are shown schematically and photographically in Figures 6a and 6b, respectively.

For the purpose of alignment, the optical benches are placed on a table top parallel to each other and 6.5 in. apart. The top of the 2-metre-long optical bench is 3.625 in. above the table top, and that of the 1-metre-long bench, 3.125 in. The axis of the furnace tube is 9 in. above the top of the 2-metre-long optical bench, and the centre of the microvoltmeter scale is 10.125 in. above the table top. The axis of the optical system connected with the scale of the microvoltmeter is, therefore, 7 in. above the top of the 1-metre-long optical bench.

The optical system is first aligned geometrically and then is adjusted optically. Diaphragms and cards, on which cross lines are drawn, are used for the "geometrical" adjustment. The alignment of each part of the optical system is described in the following sections of this report, which also indicate the order of the alignment.

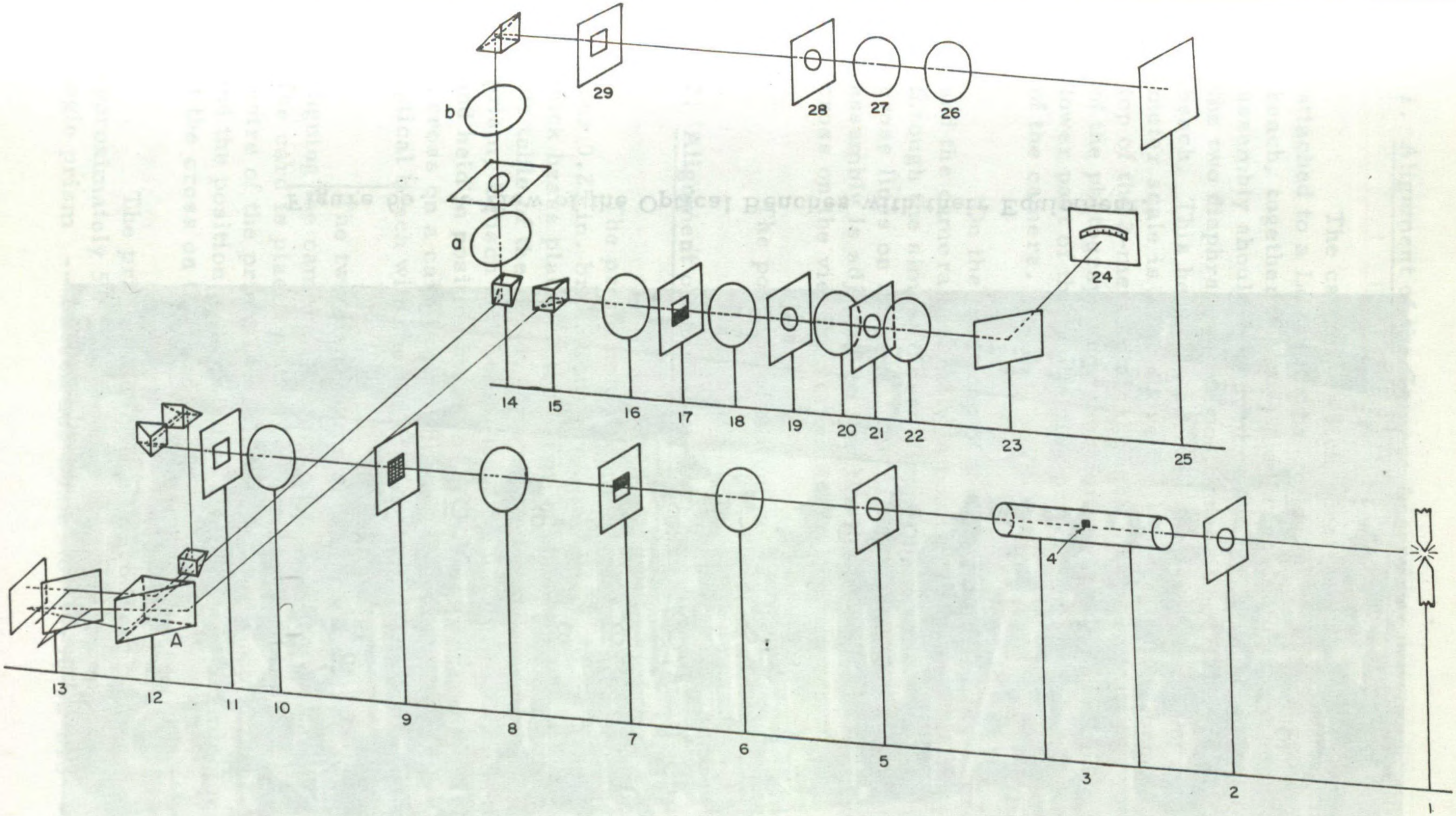


Figure 6a. Schematic Representation of the Optical System of the Apparatus.

1. microscope lamp
2. iris diaphragm
3. furnace
4. specimen
5. iris diaphragm
6. lens, f 12 cm
7. half-field screen
8. lens, f 5.58 cm
9. grid
10. lens, f 5.58 cm
11. filter holder and iris diaphragm
12. prism holder (A)
13. camera assembly
14a. lens f 3.61 cm
14b. lens, f 5.58 cm
14. prism holder
15. prism
16. lens, f 13.14 cm
17. half-field screen and filter holder
18. lens, f 7 cm
19. iris diaphragm
20. lens, f -10 cm
21. iris diaphragm
22. lens, f -15.84 cm
23. mirror
24. scale of micro-voltmeter
25. card with reference number
26. lens, f -11.10 cm
27. lens, f -6.69 cm
28. iris diaphragm
29. filter holder

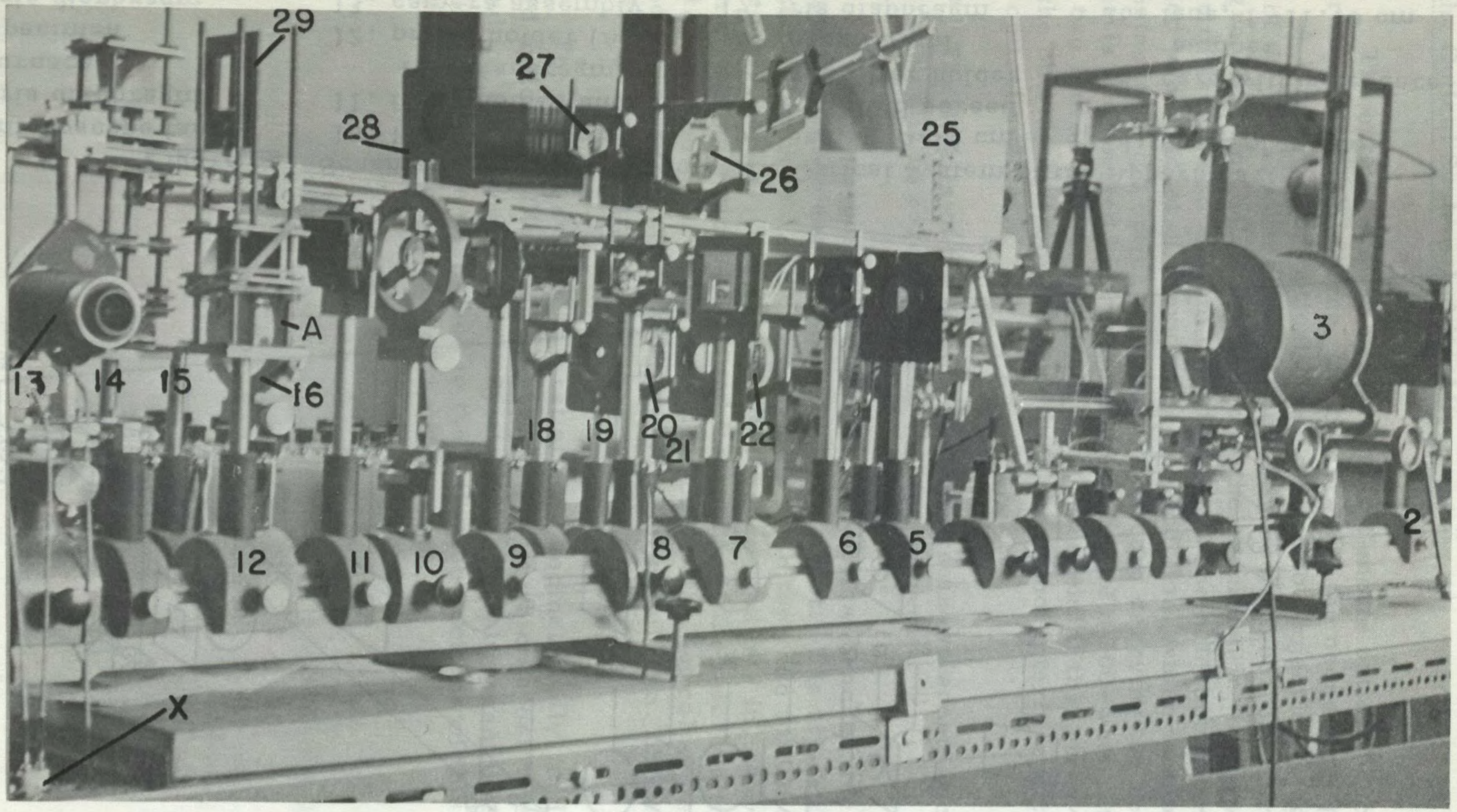


Figure 6b. A View of the Optical Benches with their Equipment.

1. Alignment of the Camera Assembly

The camera, which consists of a suitable Leica camera-housing attached to a Leitz mirror-reflex unit, is arranged on the 2-metre optical bench, together with two diaphragms and a light source. The camera assembly should be placed on the far left end of the bench. The centres of the two diaphragms are positioned at 6.875 in. above the top of the optical bench. This height is arrived at as follows. The axis of the microvoltmeter scale is 7 in. above the 1-metre optical bench, or 6.5 in. above the top of the 2-metre optical bench. This axis should centre in the lower half of the photograph and, therefore, should pass through the centre of the lower part of the "collecting prism" (A), which is 0.375 in. below the centre of the camera.

On the front cap of the mirror-reflex unit, cross lines are drawn and the camera assembly is positioned so that the light beam, passing through the almost closed diaphragms, coincides with the centre of the cross lines on the cap. The cap is then removed and the position of the assembly is adjusted so that the light beam coincides with the centre of the cross on the viewing screen as well as that on the cap.

The position of the camera assembly (13) is then left undisturbed.

2. Alignment of the Collecting Prism Holder

The prism holder consists of a 2.5-in.-square table-stand to which four 0.25-in. brass rods are attached, 1.75 in. apart. The 0.0675-in.-thick brass plates of the prism holder can be positioned at any height above the table of the stand. The collecting prism (A) -- a 1.5-in. right-angle prism, Bausch and Lomb No. 31-90-02-019 -- is placed in a prism holder and held in position by the brass plates, covered with felt. The centre of a cross on a card is positioned at 7.375 in. above the top of the 1-metre optical bench with the aid of two diaphragms and a light source.

The two diaphragms and the light source, which were used for aligning the camera, are placed in between the camera and the prism holder. The card is placed on the 1-metre bench, exactly opposite the prism. The centre of the prism is 6.875 in. above the top of the 2-metre optical bench and the position is adjusted so that the light beam coincides with the centre of the cross on the card.

The prism holder with the collecting prism is then positioned approximately 5 in. in front of the camera assembly. One 0.75-in. right-angle prism -- Bausch and Lomb No. 31-90-02-016 -- is placed against the upper half-side of the prism (A) according to Figure 6a. It is held in place by an elastic band and is brought into optical contact with the prism (A) by use of a drop of immersion oil.

The centres of two diaphragms are positioned at the 9-in. height. The diaphragms and a light source are placed on the right side of the prism holder. The 1.25-in. right-angle prisms -- Bausch and Lomb No. 31-90-02-018 -- are positioned in the prism holder at the 9-in. level. One faces the 9-in.-high optical axis and reflects the beam towards the side into the other prism, which then reflects the beam downwards into the 0.75-in. prism attached to the side of the collecting prism. A brass clip prevents the second 1.25-in. prism from tilting. The position of each prism is adjusted until one light spot coincides with the centre of the upper half of the viewing screen in the camera assembly.

3. Alignment of the Other Prisms

The light beams coming, respectively, from the scale of the microvoltmeter and from the card with reference number, are each reflected by a 0.75-in. prism on the 1-metre optical bench into the collecting prism (A). The prism connected with the optical system containing the card with the reference number is attached with wax to a horizontal rod in a prism holder. The other is attached with wax to a separate stand. Both are positioned as closely together as possible on the 1-metre bench opposite the collecting prism (A). The prism holder which forms part of the optical system of the card with the reference number contains, in addition to the 0.75-in. prism at the 7-in. level, a 1.5-in. right-angle prism, which has its centre 14.25 in. above the top of the 1-metre bench. Each of them is then centered on the viewing screen of the camera assembly with the aid of two iris diaphragms and a light source, forming the required optical axis above the 1-metre optical bench.

4. Alignment of the Lenses

The lenses are aligned using two diaphragms, a mirror, and a light source. The centres of the diaphragms are positioned at the height of the optical axis, respectively 7 in. above the top of the optical bench for the system of the scale of the microvoltmeter, and 9 in. above the top of the optical bench for the system of the furnace.

The position of the mirror is adjusted so that the reflected and the incident beams, passing through the centre of the diaphragms, coincide. The lens is placed between the mirror and the diaphragm system. It is adjusted in a similar way. Although each of the lenses can be centered in this way individually, the combination of the three which make up each system, should also be checked by this autocollimation method.

The centering of the lenses belonging to the optical system of the card with the reference number, should be done by viewing the screen in the camera assembly rather than by autocollimation. A light source is placed in the position of the card and two diaphragms are positioned at the 14.25-in. level. The lenses are centered in the following order: f 3.61 (14a), f 5.58 (14b), f 6.69 (27), f 11.10 (26).

5. Alignment of the Light Source

A universal microscope lamp from Wild of Canada Ltd., which contains a magnesium ribbon-filament bulb, is used as the light source on the far right end of the 2-metre optical bench. An image of the filament can be formed on a card which is placed in front of the collecting prism. An iris diaphragm is placed a couple of inches in front of it.

The lamp should be placed in such a position that the image of the filament is symmetrical with reference to the centre of the iris diaphragm, and that the intensity on the viewing screen in the camera assembly is the brightest obtainable.

6. Alignment of the Furnace

A disk of 0.0625-in. brass sheet, which has a small hole in the centre, is put in place of the circular window of the furnace. The universal microscope lamp and two iris diaphragms, which are centered on the 9-in.-high optical axis, are used to form an image of the hole in the brass disk on a card. The distance between the two diaphragms should be greater than the length of the furnace. The position of the furnace is adjusted so that the image of the hole in the brass disk is centered on the card; then the front of the furnace with its half-circular window is centered. The positioning in the horizontal plane is done by turning the knurled knobs (No. 24 in Figure 2a), and the positioning in the vertical direction by turning the knurled knobs (No. 25 in Figure 2a).

7. Final Arrangement of the Optical Equipment

Several pieces of equipment such as the camera assembly, the prism holders with prisms, the universal microscope lamp and the furnace are positioned according to the procedures detailed in the foregoing sections.

When the lenses and the diaphragms have been aligned, the remainder of the equipment should be positioned on the optical benches according to the arrangement shown in Figure 6a. The position of the lenses depends on the magnification desired. It should be emphasized that the positions of the prisms on the optical benches should be left undisturbed once they have been ascertained. Readjustment of the image of the specimen, the scale of the microvoltmeter or the reference number should be attempted with the "least dependent" item. This is the mirror for the microvoltmeter, the furnace itself for the specimen, and the f 11.10 lens for the reference number.

The half-field screens, which are actually focussed by the f 5.58 and the f 13.14 lenses, serve to separate the three branches of the system. Screen No. 7 (Figure 6a) blocks the lower half of the field of view and screen No. 17 (Figure 6a) the upper half. The semicircular part under the scale of the microvoltmeter is also blocked by screen No. 17, so that the number of the card (25) can be focussed upon this blocked part of the field of view.

The brightness of illumination of the specimen, of the scale of the microvoltmeter, and of the reference number should be equalized by using grey filters and proper apertures of the diaphragms. This is done by using, as far as possible, diaphragm No. 19 (Figure 6a) for the brightness of the scale of the microvoltmeter. The brightness of the reference number can be equalized by placing a 0.3 neutral filter in holder No. 17 (Figure 6a) and that of the furnace by placing a 0.6 neutral filter and a green filter in the holder No. 11 (Figure 6a). It did not appear to be necessary to adjust the voltage of the 300-watt reflector floodlight illuminating the reference number.

OPERATION

A. Preparation of the Pellet

The sample to be studied is ground to -300 mesh size under alcohol in an agate or boron carbide mortar. A small amount of the sample is then transferred to a Leitz handpress (Code No. AKIIF), which was fitted with a heavier spring than that supplied by the manufacturer. The pressure on the sample is approximately 3,500 psi. Before pressing the pellet, a drop of 10% alcoholic stearic acid solution is placed on the powder in order to serve as a binder.

Pellets with a round or a square section of 2 or 3 mm can be formed in the press. Pellets with 2-mm-square section and 3-mm-high sides are mostly used.

The pellet is placed on the sample holder of the furnace, which is covered with a piece of 22-gauge platinum foil. The carrier is pushed forward into the furnace and locked in place by turning knurled ring No. 5 (Figure 2a).

The flow of cooling water is regulated by a micro-regulating Whitney valve. The gas lines are attached to 0.125-in.-I D gas inlet and outlet pipes (No. 43, Figure 2a), when the sample is to be heated in an atmosphere other than air.

B. Recording the Deformation of the Pellet

During the procedure of reaching a certain temperature level, the pellet is centered in the viewing screen of the camera by adjusting the position of the furnace. When the pellet coincides with the optical axis, the image of the pellet is brought into focus by moving the f 12-cm lens (6) or the furnace along the 2-metre optical bench. The half-field screen (7) is then adjusted so that it defines the position of the bottom of the sample sharply.

When the pellet has been at a particular constant temperature for a certain period, as is indicated on the recorder, the recorder is disconnected from the microvoltmeter in order to read the EMF directly from the scale of the microvoltmeter. The equality of brightness of the sample, of the scale of the microvoltmeter and of the reference number is checked.

Since the Leica camera does not permit double exposures, its shutter should be kept open with the "double-cable" connector ("x" in Figure 6b), which also moves the viewing prism in the mirror-reflex unit out of the optical axis. The exposures are made using the shutter in front of the mirror-reflex unit. An example showing a few typical photographs is given in Figure 7.

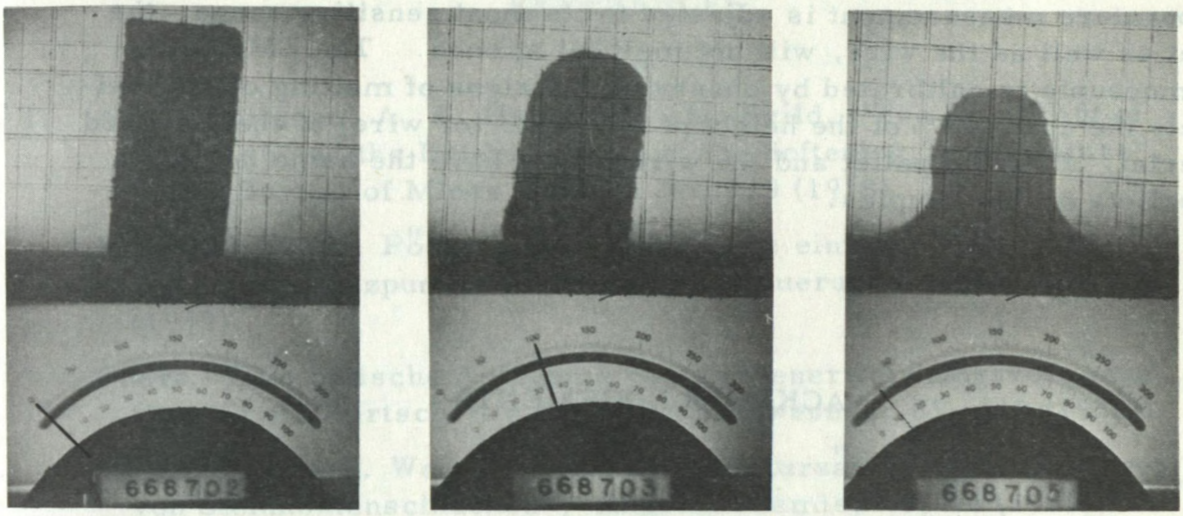
C. Attainment of Temperature Level

The temperature range that is of interest is determined in a preliminary run. This is done by gradually increasing the temperature of the furnace and determining the approximate cone-softening temperature by observing the deformation of the pellet.

When the lowest temperature of the melting range has been established, the furnace is heated to that temperature and maintained at it by means of the controller, so that the sample can reach equilibrium conditions. The temperature is then maintained at each level at which a photograph is to be taken. At no time should the furnace overshoot the temperature level. Photographs are taken after the furnace has been kept at each level for the period required by the composition of the sample. The times necessary for these operations vary widely, depending on the type of sample under investigation.

D. Calibration of the Thermocouple

Due to the thermal gradient between the centre of the 1-in. mullite tube and its walls, a calibration of the thermocouple to indicate the temperature of the upper part of the pellet is a necessity. In spite of the fact that the thermocouple is positioned as closely to the pellet as possible, the horizontal orientation of the furnace tube creates a gradient of approximately 3 deg C between the top and the bottom of the pellet. The correction that should be applied is, therefore, larger than that in the case of quench furnaces with a vertically-oriented tube.



(a) (b) (c)

Figure 7. Three Stages in the Deformation of a Pellet Made from a Mixture of Components in the System $CaO-Nb_2O_5-TiO_2$:

(a) No deformation at 1372°C;

(b) Formation of a solid solution at 1400°C;

(c) Formation of a liquid at 1463°C.

NOTE:- Each picture is an enlargement at a magnification of X 3 of one frame of 35-mm film, as used in the Leica camera. In each frame, as originally photographed, the relation between the size of the image and the size of the object photographed was as follows:-

(a) pellet: X 2.5

(b) scale of millivoltmeter: X 0.1

(c) identification number: X 0.1

Gold wire, diopside pellets, pseudowollastonite pellets and palladium wire are used as standard materials for the procedure of calibrating. When the temperature is raised sufficiently slowly and the temperature measurement is adjusted to its most sensitive range, the pellet, as well as the wire, will not melt all at once. The EMF of the thermocouple is calibrated by observing the signs of melting of approximately the upper 25% of the height of the pellet (or wire) of the standard material. Both the pellet and the wire should have the same height as the pellets of the samples.

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SPECIAL NOTE

In numerous places throughout this report, specific products made by various manufacturers have been mentioned. No sponsorship or advertisement of these materials is implied. The mention merely indicates that they were used in the work described. It is not the intention to convey the impression that the items used were either the only or, necessarily, the best such materials on the market.

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