NBS TECHNICAL NOTE 1183

U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Techniques in High-Temperature Resistance Thermometry

- Construction of the NBS-Design High-Temperature Platinum Resistance Thermometer
- Toroidal Resistor for High-Temperature Platinum Resistance Thermometers

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• Toroidal Resistor for High-Temperature Platinum Resistance Thermometers

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Foreword

The NBS Technical Note format has been chosen for the two papers included here because Technical Notes can accommodate more technical detail than would be appropriate in other publications emphasizing results and analyses. This presentation of full detail has at least two distinct benefits. First, detailed descriptions of apparatus or procedures can aid in the understanding and analysis of complex behavior, and thus they can serve as useful background and support references for other papers. Second, the "how-to-do-it" flavor distilled from abundant detail can be of assistance to those wishing to duplicate or emulate the effort described.

The two papers in this Technical Note deal with design and construction aspects of some specific high-temperature platinum resistance thermometers. Performance experience with these thermometers has been or will be described elsewhere; the papers here are intended to provide the needed backup. It is also intended that these papers give enough detail so that workers generally familiar with the arts and crafts of thermometer making will have little difficulty in constructing similar devices.

The first paper describes the construction steps of thermometers whose design evolved at the National Bureau of Standards during the period 1976-1981. Experiences with many of these thermometers were presented at the Sixth International Temperature Symposium in March, 1982 (see reference 3 of the first paper), along with some information about the details of construction. This present paper completes and brings up to date the construction information. It is based on the work done by N. Bass during his tenure as a guest worker at NBS in 1981, in which he significantly improved the techniques developed in prior years by refining, modifying, and adding to them.

The second paper describes a new type of thermometer resistor. This resistor was also mentioned at the Sixth Temperature Symposium, but at that time little was known about its behavior. Subsequently, several new thermometers were made using the resistor design, and the data obtained with the thermometers may prove to be useful in future temperature scale considerations. The second paper is presented in anticipation that this will be the case.

John P. Evans

Construction of the NBS-design High-temperature Platinum Resistance Thermometer

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The construction of a high-temperature platinum resistance thermometer having a resistance at 0 °C of 2.5 ohm, suitable for use as a defining instrument up to the freezing point of gold (1064.43 °C, the "gold point"), is described. The thermometer is made with high-purity fused silica supports and insulators. The resistor is reference-grade platinum wire wound in a single-layer, bifilar-helix. The necessity of using the highest purity materials and of scrupulous cleaning procedures of all component parts, subassemblies and assemblies is stressed. To minimize the effect of electrical leakage, a fifth or guarding lead is incorporated. The stability and other characteristics of this type of thermometer have been evaluated at temperatures up to 1064.43 °C.

Key words: Construction; electrical guarding; gold point; high temperature; IPTS; platinum resistance thermometer; standard interpolating instrument.

1. Introduction

For more than 50 years the National Bureau of Standards, in order to meet the needs for improved temperature standards in science and industry, has been involved in the development of platinum resistance thermometers [1]. In recent years, the effort has been directed towards the design and construction of high-temperature platinum resistance thermometers. Because modern techniques for resistance measurements often require the use of a guarded system for highest accuracy, the NBS design now incorporates a fifth wire [2] to permit such guarding of the thermometer leads. The results of a recent evaluation [3] suggest that a high-temperature thermometer of this design can replace the platinum-10% rhodium/platinum thermocouple as the standard interpolating instrument on the IPTS for temperatures up to the gold point, 1064.43 °C.

Participation of invited guest workers in its programs is one of the means by which the National Bureau of Standards shares its expertise with other laboratories. The author was fortunate to be able to take part in the high-temperature resistance thermometer program. As a result of the exchange of experience through this participation, techniques used at both the National Bureau of Standards and the Commonwealth Scientific and Industrial Research Organization have been developed and improved.

2. Component Parts

The essential high temperature parts of the thermometers are made of high-purity platinum and a type of silica glass known as clear fused quartz. Thermometric quality platinum wire is used for the thermometer resistor, internal leads, and guard rings, while clear fused quartz is used for all protecting and insulating parts of the thermometers that are to be exposed to temperatures above room temperature.

The quartz components include parts made of clear fused quartz tubing and parts made by laser machining. The tubing is used for thermometer protecting sheaths and lead insulating tubes. It is selected from the manufacturer's standard production material to be round and straight, and to have the desired inner and outer diameters. The laser-machined parts are made by specialists to close tolerances from flat stock of the same clear fused quartz material. They include cross blades (figure 1) and four-hole disks. The disks can also be made by slicing sections from a four-bore quartz tube that has been ground to the desired diameter.

The manufacturer specifies that the clear fused quartz has a metallic impurity concentration of less than 50 ppm (parts per million), mainly aluminum oxide; the iron content is less than 4 ppm. The material is stated to have a low hydroxyl-ion content, an attribute believed to enhance its high-temperature durability. The quartz components are subjected to stringent surface cleaning procedures throughout the construction steps. As a final measure, where possible, they are fired at 1100 °C in flowing O_2 to oxidize any remaining impurities.

Platinum wire of the highest available purity (reference grade) is used for the resistor and leads. The manufacturer claims that the metallic impurities are less than 10 ppm in this grade of platinum and that its alpha coefficient is greater than 0.003926 K^{-1}. The wire is received from the manufacturer in the hard drawn state.

Room temperature parts of the thermometers contain ordinary glasses, plastics, and metals that are suited to the intended purpose. These include a borosilicate glass header, epoxy sealant, external copper lead extensions with plastic insulators and connector, and an external aluminum head.

3. Resistor Support Cross

The resistor support cross is fabricated from two fused quartz blades (figure 1), laser-machined from 0.4-mm thick fused quartz stock. The blades are cleaned by ultrasonic agitation in a 5% hydrofluoric acid solution to remove

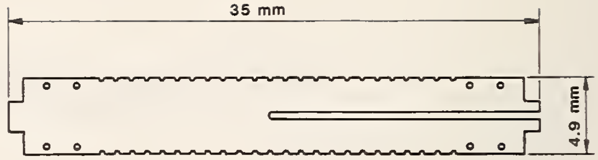

Figure 1. Fused silica cross blade. Two blades assembled together and fused at ends form resistor support cross.' The notches are spaced 1 mm apart.

contaminants adhering to or imbedded in the surface of the silica glass. They are then washed several times in distilled water, washed in ethyl alcohol, and allowed to air-dry.

After cleaning, the longitudinally slotted blades (figure 1) are carefully fitted together to form a cross. The assembled blades are held in a four-jaw pin vise, and one end of the cross is fused using an oxy-hydrogen flame. The cross is then inverted in the vise and the other end is fused. All edges, notches and holes are fire polished to a smooth glazed finish to allow abrasion-free movement of the platinum wire. The diameter of the finished cross is such that it fits nicely inside the quartz protecting sheath, ensuring that the resistor wires are held close to the sheath. The notches in the side of the cross, spaced one millimeter apart, allow the wire to be wound in a double helix with a pitch of 2.0 mm. After assembly, the cross is again etched in the 5% HF solution and rinsed in distilled water and ethyl alcohol. It is then fired at 1100 °C for 1 hour in flowing O_2.

4. Resistor and Guard Ring Construction

Resistor fabrication begins with preparation of the platinum resistor wire. A 510-mm length of reference grade wire, 0.13 mm in diameter, is washed in trichloroethylene, wiped with a clean tissue, then washed in acetone and again wiped with a clean tissue. This removes any surface lubricant remaining from the drawing process during manufacture. The wire is then annealed in air at approximately 750 °C by passing an alternating electric current through it for five minutes.

The wire is next cut into two equal lengths, and a suitable weight (approximately 2 g) is attached to one end of each of the lengths. The cross is mounted in a special coil winder (see below) and the free end of each wire threaded through appropriate holes in one end of the cross with the weighted end hanging free. The two coils are wound simultaneously, and upon completion the weights are removed and the ends inserted into holes in the opposite end of the cross. Excess platinum is removed by flame cutting, and the ends of the coils are anchored by fusing the wire ends into small balls that cannot pull back through the holes. Additional short lengths of reference-grade platinum wire (the same wire as used for the resistor) are welded to the upper ends of the resistor to form a four-lead resistor (figure 2). These lead branches are anchored in the other holes in a similar manner to the resistor wires, and all four leads are extended beyond the end of the cross with larger diameter reference-grade wire for later joining to the leads. A short piece of resistor wire is used to join the bottom ends of the coils of the resistor to complete the circuit (figure 3).

The coil winder, designed at NBS by L. A. Guildner, facilitates winding of the platinum resistor wire directly onto the cross under the tension of two-gram weight. The cross is supported at both its ends in the winder. At one end the support is provided by a four-jaw platinum chuck, which also serves as a round mandrel on which the resistor is formed. As the cross is rotated in the winder, and as winding progresses, the chuck withdraws from the cross so that only two formed turns are in contact with the chuck at any one time. The winder produces near-perfect round coil turns and places them precisely in the notches.

The guard rings are made of annealed reference-grade platinum wire 0.3 mm in diameter. They are formed on a glass forming tool (figure 4a) in a clover-leaf pattern (figure 4b). Each ring consists of a single full pattern as shown.

Figure 2. Two views of upper end of resistor showing connection of lead branch to resistor wire and method of anchoring wire.

The resistor and guard rings are cleaned in high-purity 2 N HCl for four hours at 50 °C, rinsed several times in fresh distilled H_2O and then in high-purity 6 N HNO_3 for four hours at 95 °C. They are then agitated several times in fresh distilled H_2O, boiled in distilled H_2O for four hours, rinsed in ethyl alcohol, and finally air-dried.

5. Preparation of Lead Insulating Tubes and Protection Sheath

The lead insulating tubes and the protection sheath require special preparation procedures because of their dimensions. The lead insulating tubes are made of fused quartz 1.5 mm diameter x 0.5 mm bore x 760 mm long. To minimize heat losses caused by radiation piping, the outer surface of the tubes is

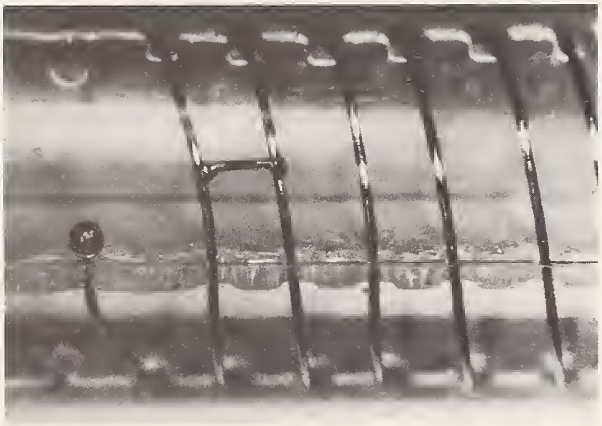

Figure 3. Lower end of resistor showing platinum link that joins the two coils to form a continuous resistor circuit.

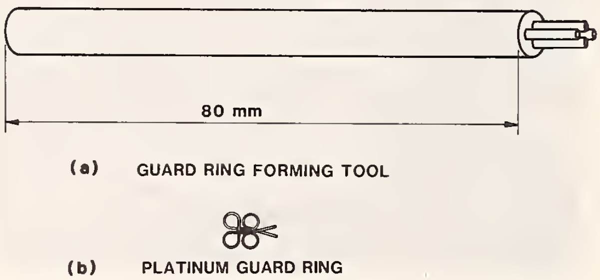

Figure 4. Guard ring and forming tool.

roughened by blasting with Al_2O_3 powder over a length of 500 mm, leaving about 10 mm at the bottom end clear. The protection sheath is made from fused quartz tubing 7.25 mm diam. x 5.00 mm bore x 800 mm long, closed round at one end. Since both types of tubes are too long to be cleaned conveniently by ultrasonic agitation, two alternative methods have been developed.

In the first method, the lead insulating tubes are placed in a burette-type tube clamped in a vertical position. To prevent the tubes from falling into the stop-cock hole, a small perforated glass disc is placed at the bottom of the burette. The following cleaning steps are then taken:

(i) The burette is filled with a 5% HF solution to cover the tubes. Air bubbles are displaced from the tubes by heating the burette with a hot-air gun and tapping the side of the burette. The tubes are allowed to etch for several minutes.

(ii) The HF solution is drained off and the top of the burette is connected to a cold-water tap with flexible tubing. The system is thoroughly flushed with the stop-cock open. Tap water is used in this step for convenience.

(iii) The burette is allowed to drain and the tubes are rinsed with distilled water by filling and draining the burette. The rinsing is repeated several times.

(iv) The tubes are rinsed with ethyl alcohol, then removed from the burette and allowed to air-dry.

The second method requires the use of a "peristaltic" type pump. The lead insulating tubes are placed in a horizontally-mounted glass tube about 1 cm in diameter and several cm longer than the lead tubes. The glass tube is connected to the pump (and to liquid reservoirs) with flexible plastic tubing that will withstand the acids and solvents to be circulated through it. Liquids are forced through the tube, in the sequence described above, with enough pressure to ensure that they flow through the small bores of the lead tubes; the flowing is easy to detect by observing the movement of small air bubbles in the bores.

The inside of the protection sheath is chemically cleaned in a 5% HF solution for several minutes, and it is then rinsed with tap water, with distilled H_2O several times, and with ethyl alcohol. The sheath may also be cleaned by an adaptation of the above pump method, either before it is closed at one end, or afterward, or both.

After the lead insulating tubes have been dried, the upper end of each tube is fire polished. The purpose of this is to provide a smooth entrance and exit for the lead wires as they expand and contract. By directing a small oxy-hydrogen flame directly into the bore, the inner edge is polished without shrinking the bore of the tube.

As a final preparation step, both the lead insulating tubes and the sheath are fired at 1100 °C in flowing O_2 for one hour. Oxygen is introduced into the closed-end sheath through a small diameter quartz tube extending nearly to the bottom of the sheath. This firing is intended to oxidize any remaining impurities and to flush away the volatile oxides.

6. Lead Construction

The internal leads are made from 0.25-mm diameter unannealed reference-grade platinum wire. Four wires, each 800 mm long, are cleaned with trichloroethylene-soaked tissue then acetone-soaked tissue, straightened by stretching gently, and inserted into the cleaned lead insulating tubes. A 10-mm length of 0.3-mm reference-grade platinum wire is welded to the bottom end of each platinum lead, the weld bead being small enough to fit nicely inside the bore of the tube. The small bead is pulled about 2 mm into the tube, and then the end of the tube is heated enough to fuse the quartz, but not enough to melt the platinum. This constricts the end of the tube bore so that the small bead cannot be pulled back out.

The four lead tubes are held in place by the platinum guard rings so that the tubes cannot touch each other directly nor touch the wall of the protecting sheath. Three of the clover-leaf shaped rings are spaced closely together at the resistor end to provide axial radiation shielding and radial thermal coupling of the lead tubes to the sheath. Three additional single rings are spaced along the tubes at about 200, 400, and 600 mm from the top of the resistor. The rings are all connected electrically by a wire welded to them. This fifth guard lead is made of the same cleaned reference-grade platinum wire as is used for the thermometer leads. It is uninsulated, and it exits from the thermometer at the borosilicate glass header. The fifth wire can be connected to an active electric guard circuit in the external measuring system.

The lead assembly is mechanically terminated at the resistor end with a fused quartz spacer disk, 4.8 mm diam and 1.0 mm thick, containing four equally spaced holes. The four thermometer lead wires can protrude through the holes, but the lead tubes cannot. The protruding ends of the lead wires are fused into balls, locking the leads and the disk together (figure 2). A four-bore glass spacer is placed temporarily over the upper ends of the leads, and the lead wires are welded together at their upper end to form a loop. This loop, later removed, is used to suspend the lead assembly in a water refluxing washer.

7. Assembly of the Resistance Thermometer

The resistor is joined to the lead assembly by fusing its lead extensions to the balled ends of the leads. The assembly is then suspended in the water refluxing unit and washed for a minimum of 100 hours. The purpose of this step is to remove unwanted materials inadvertently introduced during assembly. After washing, the resistor and lead assembly is placed in a clean glass drying tube and dried in flowing O_2 in a furnace at 300 °C.

The thermometer is next inserted into the cleaned protection sheath, and the temporary glass disk is removed. The thermometer is positioned in the sheath so that there is a gap of a few millimeters between the end of the resistor and bottom of the sheath, and so that the upper end of the lead tubes protrude slightly out the sheath. The lead tubes are then fused to the end of the sheath, or to a small quartz ring the same diameter as the sheath. Care is taken to space the lead tubes equally around the sheath or ring, and to avoid constricting the bore of the lead tubes. The result of this procedure is that the lead tubes and sheath become a connected fused quartz structure, and that the platinum leads are fixed relative to the structure at the resistor end of the leads, permitting little, if any, movement of the resistor upon expansion and contraction of the lead wires.

The expansion of the leads is accommodated by platinum expansion coils located at the upper end of the thermometer. The four expansion coils are made by close winding 8-1/2 turns of 0.25-mm diameter platinum wire on a 4-mm diameter mandrel, leaving a straight tail 40 mm long on one end of the coil. The platinum need not be reference grade, since the expansion coils remain at room temperature, and it is advantageous to keep the coils in the unannealed, hard drawn state. The coils are stretched to a length of 25 mm, and they are then welded to the trimmed platinum thermometer leads as close to the lead tubes as practicable.

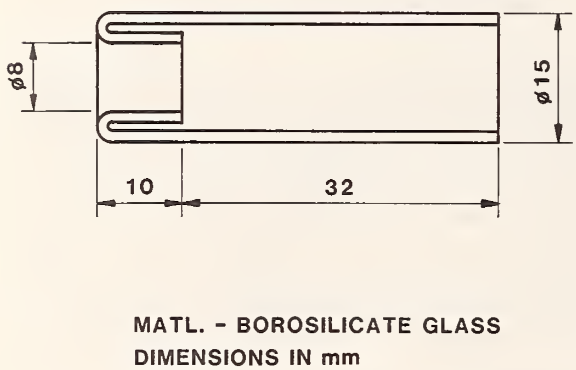

Figure 5. Body of glass thermometer header. The small end of the header is joined to the thermometer protection sheath.

The expansion coils are housed in a borosilicate glass header (figure 5), which is attached to the upper end of the protection sheath with low vapor-pressure epoxy resin. A cross-shaped glass divider (figure 6) is placed inside the header to keep the coils separated. The coil tails are threaded through holes in the borosilicate glass header cap (figure 7) where they are sealed with the same epoxy, and a glass tubulation on the cap serves as an evacuating and filling tube. The guard lead emerges from the thermometer through the epoxy seal between the header and sheath.

The assembled thermometer is now placed in a horizontal bake-out furnace and attached to a suitable oil-free vacuum system via the glass tube (see figure 7). To oxidize any remaining

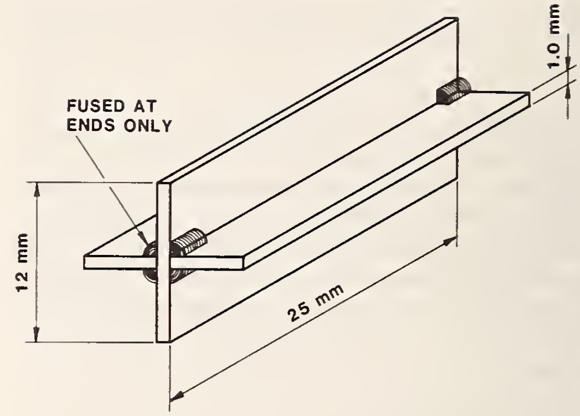

Figure 6. Cross-shaped divider. The divider, made from clear fused quartz microscope slides, is inserted into the header to keep the platinum expansion coils separated.

organic material, the system is flushed and filled to atmospheric pressure with O_2 and the furnace temperature raised to 1100 °C for 20 minutes. The temperature is then reduced to 800 °C and the thermometer evacuated. The furnace is maintained at this temperature and the pumping continued until the pressure becomes less than 5×10^{-6} Pa, but for at least 50 hours. The temperature is then reduced to ambient, and the thermometer is filled to a pressure of 40 kPa with pure argon containing 10% O_2 and sealed at the constriction in the glass tube.

Outside the glass header the four platinum thermometer leads are trimmed to the same length and then welded to copper

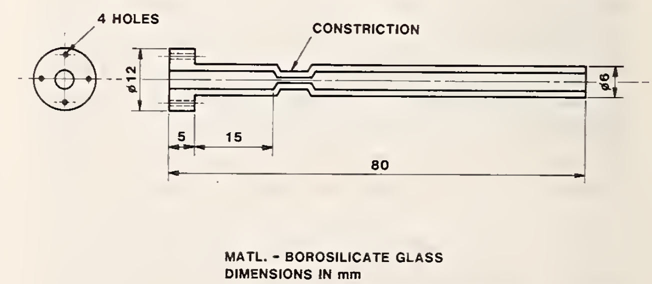

Figure 7. Header cap. The header cap is inserted into the large end of the header body and sealed with epoxy. Thermometer leads exit through the four holes. The tube is used for evacuating, filling, and sealing the thermometer.

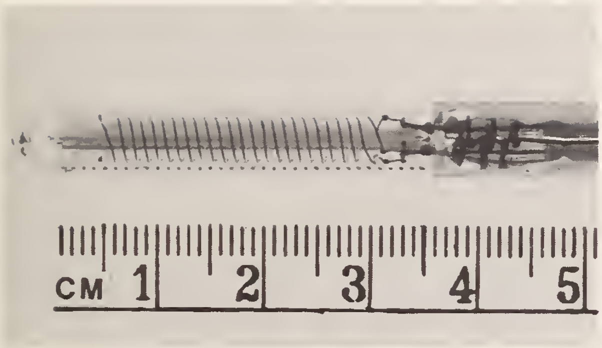

Figure 8. Resistor end of completed thermometer. Oil has been placed on the roughened protection sheath so that the guard rings may be seen in the photograph.

extension leads using a plasma needle arc welder. The leads are insulated with PTFE tubing, and the four platinum-copper junctions are clamped together with a heat shrinkable plastic tubing to minimize thermal emfs. A copper lead extension is also welded to the guard lead, and all five copper extensions are then soft-soldered to a multi-contact electrical connector. A thin-wall aluminum head (20 mm dia. x 125 mm long), attached to the sheath at its upper end with silicone rubber, completes the construction. This protects the glass header and supports the connector. As a final step, the outside of the sheath is roughened by blasting with Al_2O_3 powder for 500 mm above the resistor position. The resistor end of the completed thermometer is shown in figure 8.

The author wishes to thank the Executive of the Commonwealth Scientific and Industrial Research Organization for granting him a study award, the National Bureau of Standards for guest worker status and hospitality during the tenure of this award, and Dr. J. F. Schooley, whose efforts made it all possible. Special thanks are due to J. P. Evans, L. A. Guildner, S. Tillett and R. E. Edsinger for their invaluable assistance with the project, and to all the staff at NBS for the friendship and courtesy they extended to the author during the period of his visit.

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Toroidal Resistor for High Temperature Platinum Resistance Thermometers

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A new type of resistor for high temperature platinum resistance thermometers, called the "toroidal" resistor, is described. It is designed to be easy to make from readily available materials, and it features robustness, small size, and freedom from strain. Limited experience indicates that the resistor performance meets these design expectations, and that the resistor may be a useful alternative for high temperature thermometers.

Key words: Construction; design alternatives; high temperature; platinum resistance thermometer; practical temperature scale; resistor; silica glass; stability

1. Introduction

We have designed a new type of thermometer resistor as part of a continuing effort to develop high temperature platinum resistance thermometry. Our purpose has been to explore resistor design alternatives that can foster and enhance the use of such thermometry to define a practical temperature scale.

The use of a platinum resistance thermometer as a scale defining instrument at high temperatures imposes certain requirements on the temperature sensing platinum resistor. The platinum must be very pure and it must be in a "strain free" condition at all temperatures. It must be capable of reaching the temperature of the environment to be measured as closely as possible. It must be protected from chemical change and permanent dimensional change. And it must be configured to permit precise determination of its electrical resistance.

In designing a thermometer resistor one attempts to meet the requirements cited above. The customary design approach is to use platinum in the form of wire, to support the wire on an electrically insulating structure of metal oxide, to equip the resulting wire-wound resistor with four lead terminations, to encapsulate the resistor in a close-fitting envelope, and to fill the envelope with a benign gas (containing some oxygen). The resistor design must take into account the properties of the materials used, such as potential sources of contamination for the platinum and the electrical characteristics of the insulators at high temperatures. Ease and cost of construction and use must also be considered. The various requirements are not always compatible, and design details are therefore often the result of compromises between conflicting requirements.

A number of resistor designs have evolved from efforts to embody the conceptual requirements into useful high temperature platinum resistance thermometers. Most of the designs incorporate circular coils of platinum wire in one form or another. Tightly wound coils have been supported on notched cruciform cadres (in helical spirals), on twisted silica-glass ribbons, and around small insulating tubes. In other designs, single-layered helices of wire have been mounted on notched crosses, on grooved rods, or, if the wire diameter is large enough, in virtually unsupported free suspension. A recent design has a single-layered bifilar helix of relatively heavy platinum wire supported on a flat notched blade [1, 2]. In another design, the "birdcage" resistor [3], the platinum wires are kept straight and parallel to the axis of the resistor over most of their length.

In this paper we describe the design details and construction of the new resistor, which we call the "toroidal" resistor because of its shape. Our goal has been to design a resistor that is relatively easy to make from readily available material. Design compromises have favored robustness, small size, and freedom from strain at the possible expense of optimum thermal communication between the resistor wires and their surroundings. Limited experience with the resistor indicates that it does indeed meet the design objectives, but considerably more experience would be needed to evaluate fully its efficacy in a variety of temperature measuring situations.

2. Design Approach

The toroidal resistor is shown in figure 1. It consists of a silica-glass former on which is wound the platinum resistor wire. The former is a piece of tubing slotted at both ends, and the resistor wire, wound in and out of and around the tube, is held in position by the slots. The resulting coil is an elongated, flattened toroid -- hence the resistor's name. A four-hole silica glass disk fused to one end of the tube serves to support and separate the four resistor lead branches.

The use of silica-glass tubing for the former has considerable advantage. High-purity silica glass is readily available as clear fused quartz or as synthetic fused silica, and it is relatively inexpensive. Its electrical, thermal, and mechanical properties are well suited for high temperature resistance thermometers. The material can be cut and ground by conventional techniques, it is easily flame-worked, and it can be cleaned by simple treatment. Silica-glass tubing is manufactured in a wide range of sizes; suitable diameters can be obtained from stock without resorting to special fabrication, and formers of any practicable length can be cut from ordinary production tubing.

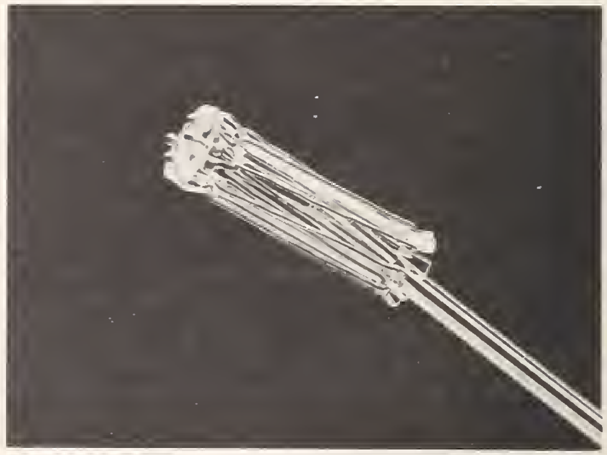

Figure 1. The toroidal resistor (supported on a rod for illustration). The silica-glass former is 15 mm long and 4-mm diameter. The resistor wire is 0.25-mm diameter. Protruding from the four-hole silica-glass disk at the top of the resistor are balls of platinum that secure the four lead branches in place.

The tools needed for construction of the toroidal resistor are often found in a well equipped laboratory. All cutting of the silica glass, including the end slots, can be done with a type of low-speed diamond saw commonly used for the preparation of metallurgical specimens. Disks cut from silica-glass rods can be drilled by means of an air-propelled abrasive device that is also useful in other stages of thermometer construction. Alternatively, disks can be cut from four-bore silica-glass tubing of the proper diameter, a product that is sometimes available commercially. Flame working of the glass and welding of the platinum wire are easily accomplished with miniature oxy-hydrogen or oxy-gas torches. The platinum wire is wound on the former by hand; only small electronic-type hand tools are required for final adjustment and positioning of the wires. Finally, a small ultrasonic bath provides a convenient and effective means of cleaning the various parts.

The resistor shown in figure 1 is only about 15 mm long. It is wound with platinum wire 0.25-mm diameter, and it has a nominal resistance at 0 °C of

0.37 ohm. The resistor former is made of 4.0-mm diameter tubing; the eight "fingers" at each end, formed by the slots, are flared out uniformly so that the former fits nicely in the center of a protecting sheath (the envelope) having an inner diameter of 5.0 mm. Since there is enough radial clearance between the former and the sheath wall, and since end slots are wide enough and flame polished, the resistor wire is free to move without constraint (if the resistor axis is vertical) as it expands and contracts upon temperature change, and the only tension on the resistor wire is due to its own mass. Furthermore, expansion and contraction of the wire are not likely to alter permanently the resistor wire geometry in any way. These features constitute the main performance merits of the resistor design.

3. Preparation of Silica-glass Parts

Preparation of the silica-glass parts begins with the selection of tubing and rod. The former tubing is selected to be round and straight, and to be free of cracks, striations, air lines, or other flaws that might weaken the tubing and cause it to break during the fabrication steps. Inner and outer diameters are not critical so long as the finished former will fit inside the protecting sheath with sufficient clearance on its outside and enough room on its inside to accommodate the resistor wires. The disk rod or four-bore tubing must be small enough in diameter to fit inside the protecting sheath but large enough to be attachable to the former. The former for the resistor shown in figure 1 utilizes nominally 4.0-mm o.d. x 2.0-mm i.d. clear fused quartz tubing selected from the manufacturer's standard tolerance production. The four-hole disk is cut from four-bore silica glass tubing ground to a diameter of 4.8 mm.

The next step is to support the silica-glass stock for sawing. This is done by "potting" or "blocking" the tubing or rod in a larger supporting glass tube with a suitable compound in order to minimize breaking, cracking, or chipping of the fragile parts. Wax and shellac based products are commercially available for this purpose. We have used a hard wax compound made of rosin, beeswax, and carnauba wax (in the weight ratio 16:2:1); it melts at about 150 °C and it is easy to remove with solvents. The glass parts are potted either by immersing them in a container of molten wax, or by filling the support tube with molten wax. After the potting wax hardens, the material on the outside of the support tube is removed by scraping and wiping. Several disk rods or tubes may be potted in the same large size support tube for efficient production, but each piece of former tubing must be supported by its own close-fitting glass support tube (see figure 2). This is because the former, after it has been cut to length, must be slotted at each end precisely on diameters, a step more easily accomplished if the axes of the former tubing and the support tube coincide. The 4.0-mm former tubing used here fits nicely inside selected 1/4-inch standard wall borosilicate-glass tubing. Tube lengths of about 50 mm are convenient for potting and cutting into the finished former length.

All glass cutting is done with a thin circular diamond saw, operated at low speed, and cooled and lubricated with distilled water. The width of the saw blade is determined by the desired width of the former end slots, which in turn is

Figure 2. Silica-glass former tubing potted in close-fitting borosilicate glass holding tube with hard wax compound.

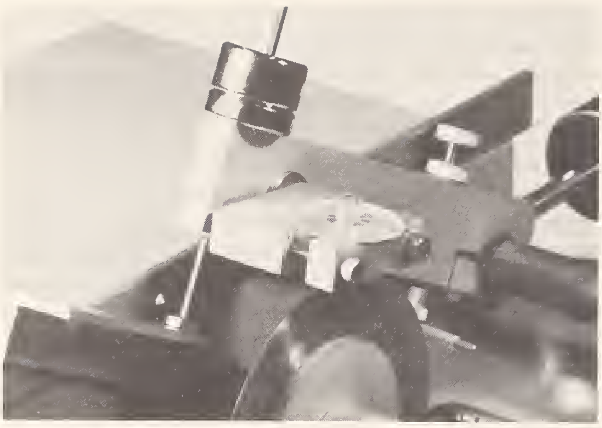

Figure 3. Silica-glass former in holding tube mounted on saw for cutting end slots. The circular disk is an indexing device.

related to size of the resistor wire. The saw used here produces a kerf of about 0.3 mm. Disks are cut by slicing thin sections from the potted multi-bore tubing or rod perpendicular to its axis. The disks should be thick enough to avoid distortion when they are fused to the former end -- a thickness of 1.0 mm is about right. The former tube is cut to length (15 mm for the resistor shown in figure 1), and it is then mounted in a holding fixture to cut the end slots (see figure 3). The fixture must be capable of producing centered, uniformly-indexed, controlled-depth slots. Each end of the resistor has eight peripheral slots 2.5 mm deep produced by four cuts.

After the glass parts have been cut, they must be removed from their temporary supports, freed of potting wax, and thoroughly cleaned. The disks may be separated by simply dissolving away or melting away the supporting wax. The former should be removed by melting the wax, carefully separating the former from the holding tube, and then draining off the molten wax. The remaining wax film is removed from the parts by gentle agitation in appropriate solvents; care should be taken because the former is especially fragile at this stage. After the parts are rinsed in ethyl alcohol and distilled water to remove the solvents, they are lightly etched by agitation for 10 minutes in a 5% solution of HF, and then rinsed again in distilled water. The etching is intended to remove surface contaminants, but it also seems to lessen fragility of the parts, probably by relieving strains at sharp corners and microscopic cracks. The parts are air-dried in preparation for flame polishing, and they are not touched again by bare hands until that step has been completed.

Flame polishing of the glass parts produces smooth, glazed edges, and it relieves any strains remaining from the cutting operation. The polishing should be carried out using a torch flame only big enough to glaze edges and fuse corners without distorting the part, and it should be observed with enough optical magnification to assure the desired result. The former is conveniently supported on a holder made of silica-glass tubing or rod clamped in a maneuverable vise (see figure 4). During flame working of the former slots, the eight "fingers" formed by the slots must be flared out uniformly to the diameter of the inside of the protecting sheath. This can be done by tilting the former slightly and applying the torch flame to the proper spot near the base of the finger. The procedure is not difficult, but it does require a little practice. When all fingers have been so worked, and the flare appears to be symmetric, the former should be gaged for proper fit in

Figure 4. Silica-glass former on holding jig for flame working. Some of the "fingers" have been flame polished and flared.

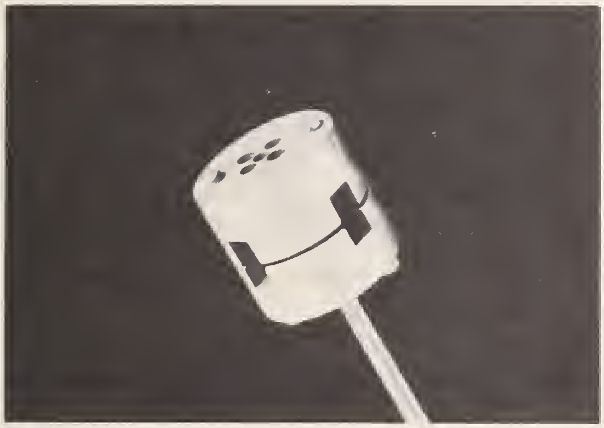

Figure 5. Stainless steel hole locating jig (supported on a rod for illustration). A silica-glass disk is clamped between the two halves of the jig, and holes are drilled with abrasive powder propelled by air from a nozzle inserted into the locating holes in the jig. The rectangular openings are exhaust ports for the spent abrasive.

the sheath. The four-hole disk may be flame polished at this stage, or it may be polished later when the disk is attached to the former.

The disk used in the resistor shown in figure 1 was made from commercially available four-bore tubing, but a four-hole disk may be made in other ways. Disks cut from silica-glass rod of the correct diameter can be drilled by air propelled abrasive powder (e.g. aluminum oxide) with the aid of a simple hole-locating jig (see figure 5). The holes may be somewhat tapered, but they serve the intended purpose adequately. Of course, a disk so drilled would have to cleaned after the drilling. Disks may also be drilled by ultrasonic cavitation or by laser, and commercial establishments can provide these services.

4. Assembly of the Resistor

The assembly of the prepared parts into a finished resistor is a hand operation involving only the use of hand tools, holding jigs, and the torch. Handling of the parts is kept to a minimum, but it is not possible to eliminate all contact between the parts and hands or tools. If an operation has the potential for contaminating the silica glass or platinum, it is conducted so that the contamination may be removed after assembly.

The platinum wire is made ready for assembly by cleaning its surface with solvents and annealing it electrically until it is soft enough to work easily. Two different wire sizes may be used -- one diameter for the resistor and another for the lead connections; or the same size wire may be used for both purposes. In the resistor shown, 0.25-mm diameter wire is used for both the resistor and the lead connections. In any case, all of the wire must be reference grade of the highest available purity.

The first step is to install platinum lead connections in the four-hole disk (see figure 6). Two platinum "U's" are fashioned from the lead connection wire to fit into disk holes by pairs. At the middle of each "U" bottom is welded a piece of resistor wire several millimeters longer than the length of the former. Both "U's" are then inserted fully into the holes from the same side of the disk, and the ends of the platinum wire on the other side of the disk are trimmed to equal lengths. The trimmed ends are then fused with the torch to form a platinum ball on each of the four lead connections that cannot be pulled back through the hole. The lead

Figure 6. Lead connection branches before (left) and after installation in four-hole silica-glass disk. The branches are secured to the disk by the fused platinum balls. The wires extending down from the branches are part of the resistor wire.

connections are thereby locked onto the disk and can move very little with respect to it. The length of platinum wire required to form the balls depends on wire and hole size; it is readily determined by trial.

Next the four-hole disk is attached to the former. The former is positioned vertically in a holding fixture, and the disk is placed on top of it with one resistor wire running down through the center of the former and the other wire extending out through one of the slots at the top. The tip of the "fingers" are then fused to the disk one at a time with the torch, taking care not to melt the platinum, not to close the slots, and to keep the structure undistorted and symmetric. Sometimes it is necessary to pull a finger, when it is hot enough to flow, into contact with the disk with a small silica-glass puller rod. After all of the fingers are fused to the disk, the former is again gaged to make sure that it will fit properly in the sheath.

The former is now ready to accept the resistor winding. To one end of an appropriate length of cleaned resistor wire is welded a piece of scrap platinum wire, later removed, that serves as a "leader". The other end of the resistor wire is welded to the wire emerging from the lead connections through a slot at the top of the former. This must be a butt weld, and, in order to avoid problems such as shorting or binding, the weld diameter must be not much larger than the wire diameter. The leader is threaded up the center of the former and out through an adjacent upper slot creating a loop around the former wall. The wire is pulled until the loop bottom is positioned in the proper slot at the bottom of the former. The loop is then flattened, straightened, and adjusted with tweezers or other suitable tools until the wires lie flat against the inner and outer walls of the former. The sharp bend at the bottom of the loop must not touch the former, but also the loop must not be so long that it will come out of the notch upon maximum expected thermal expansion. At the top of the former the resistor wire is bent down sharply to start the next loop, and the process is continued around the former until all slots have been filled. The leader is removed, and the resistor wire is welded to the other piece of wire extending down from the lead connections through the center of the former. A final adjustment of all wires to make sure that they cannot touch each other completes the resistor assembly.

As a final step, the resistor must be cleaned. It is first agitated in ethyl alcohol and distilled water to remove surface soils that accumulated during assembly, and then it is etched by agitation for 10 minutes in a 5% HF solution. This etching accomplishes two important things: (1) it removes from the surface of the platinum wire finely divided silicon dioxide that is vaporized onto the wire during the fusing of the silica-glass, and (2) it removes a minute layer of silica from the glass parts and along with it any remaining surface contaminants. The cleaning is completed by thorough rinsing in distilled water and an alcohol rinse to aid drying.

5. Evaluation of the Resistor

Toroidal resistors have been incorporated in several high temperature resistance thermometers. These thermometers employ guarded leads and they are sheathed in long silica-glass tubes 7.25-mm o.d. x 5.00-mm i.d. The assembly of thermometers of this type is reported elsewhere [4].

Three thermometers were built with the 0.37-ohm resistor described here and tested at high temperature. All three proved to be just as stable during a 500-hour exposure at 1100 °C as any other thermometers made at NBS, and they retained values for the alpha coefficient greater than 0.0039269 K^{-1}, indicating that the resistor platinum remained pure and strain free during and after exposure to the high temperature. Measurements at thermometric fixed points show that the thermometers exhibit the same resistance-temperature relation as other thermometers, and that they can be used to make precise temperature measurements. In contrast, the self-heating of the toroidal resistor due to its measuring current is somewhat greater than for some other resistors, and immersion characteristics of thermometers using the toroidal resistor may be somewhat less favorable than those of thermometers using some other types of resistors.

Two other thermometers were built with resistors having formers 30 mm long. One resistor was wound with 0.125-mm diameter wire, giving a resistance at 0 °C of about 2.90 ohms, and the other resistor was wound with 0.25-mm diameter wire, giving a resistance of about 0.71 ohms. The resistor made with the heavier wire was nearly as stable as the ones described above upon exposure for several hundred hours at 1100 °C, and its alpha value remained greater than 0.0039272 K^{-1}. The resistor made with the smaller wire, however, exhibited a continuing increase in resistance during the same exposure, though its alpha value stayed relatively constant at 0.0039257 K^{-1}.

The experience cited above is consistent with design expectations. The toroidal resistor appears to be "strain free" when used in a vertical orientation (as was done), and there has been no evidence of a tendency for the resistor turns to become distorted or shorted together. The continuing resistance increase observed in the resistor made with fine wire has been attributed to creep of the platinum wire at high temperature; evidently resistor stability is favored by the use of a short former and large diameter wire. The greater heating effect and increased required depth of immersion are to be expected since half of the resistor wire is remote from the wall of the thermometer sheath. However, there is partial compensation because its short length lets the resistor be immersed farther into the measured environment.

This preliminary evaluation of the toroidal resistor has established the simplicity and flexibility of its design and some of its favorable performance characteristics. These features make it an attractive possible alternative for use in precise high temperature platinum resistance thermometers. However, a complete evaluation of the resistor will require additional work. It should be tested at high temperatures in a horizontal orientation, its intrinsic electrical leakage across the former should be determined, investigations should be made of its reactance and electrical coupling to its environment, and more should be learned about its immersion characteristics. An important design enhancement would be the inclusion of means for electrical guarding. When such work is done, thermometer makers desiring to construct resistors by hand with simple tools will have a solid basis for assessing the merits of the toroidal resistor.

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This Technical Note consists of two papers dealing with design and construction aspects of high-temperature platinum resistance thermometers intended for use as standard interpolating instruments up to the gold point (1064 °C). Performance experience with the thermometers has been or will be presented elsewhere.

The first paper describes the construction of a thermometer utilizing a resistor of reference-grade platinum wire wound in a single-layer, bifilar helix. The supports and insulators are made of high-purity fused silica, and the thermometer incorporates an electrical guard system to minimize the effects of electrical leakage.

The second paper describes a new type of resistor for high-temperature platinum resistance thermometers, the "toroidal" resistor. The resistor is designed to be easy to make from readily available materials, and it features robustness, small size, and freedom from strain.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

Construction; design alternatives; electrical guarding; gold point; high temperature; IPTS; platinum resistance thermometer; practical temperature scale; resistor; silica glass; stability; standard interpolating instrument.

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