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MARTIN MARIETTA

The Method of Construction of a Dual Johnson-Noise-Power and Resistance Thermometer for 1375 K (1100°C) Service in a Vacuum Environment

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INSTRUMENTATION & CONTROLS DIVISION

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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APPLIED TECHNOLOGY

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3. Retired.

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ACKNOWLEDGMENTS

Delta M Corporation, located in Oak Ridge was formed by a group of engineers and scientists who, while working for Oak Ridge National Laboratory (ORNL), developed techniques for making sensors (for high-temperature fuel studies) that could not be obtained elsewhere. It was difficult to obtain bench space at ORNL long enough to accommodate the 10-m length of the secondary cover system for the sensor assembly facility. For that reason, the assembly of sensor components within the long glove box was performed at Delta M, where long benches are used for thermocouple manufacturing.

A development subcontract was placed with Delta M wherein ORNL supplied all materials and performed chemical and metallurgical tests. Special equipment such as the glove boxes and video close-up viewing were loaned by ORNL to Delta M, which completed much of the sensor fabrication.

ABSTRACT

The design and methods of fabrication are described for resistance thermometers that will operate at 1375 K (1100°C), unattended in a vacuum for a 10-year service life, with high reliability. The expected dc calibration drift of the thermometers is corrected by concurrent measurements of the Johnson noise temperature of the thermometers.

These thermometers were to be used on the SP-100 space reactor, which requires a diverse temperature measuring system to complement the high-temperature thermocouple. A major problem was that the thermometer's long-term durability might be compromised by chemical reactions between the thermometer components. To avoid this problem, thermometers were constructed by using ultrahigh-purity, crushable ceramic insulation and metallic components which were cleaned and handled so they would retain their purity during the construction of the thermometer. A unique method of fabrication in an inert atmosphere was developed.

Two prototype thermometers were constructed and successfully operated at 1375 K in bench tests. By eliminating moisture adsorption in the ceramic insulation, the thermometer showed high enough resistivity at operating temperatures to avoid electrical shunting errors. Although the SP-100 program has been canceled, the techniques developed in the construction of these thermometers can be applied to other high-temperature thermometers.

1. INTRODUCTION

The SP-100 project objective was to design and construct a 100-kW nuclear reactor to operate unattended in space. Low weight and compact design were necessary to allow the completed reactor, along with associated remotely controlled instrumentation, to be raised into orbit. The compact design required a small, high-power core to be cooled with liquid lithium metal at a reactor outlet temperature of 1375 K. The lithium coolant was to be contained in niobium-1%zirconium (Nb-1%Zr) tubing having a wall thick thickness of 0.5 mm (0.020 in.).

The objective of the diverse temperature measurement component of the SP-100 project was to design and construct a thermometer diverse from those using the Seebeck effect so that its failure or drift rate would be independent of the rates expected in the W-Re thermocouples. The thermometer would measure the reactor outlet temperature with an accuracy of ±1.4% or ±10 K (whichever was greater) from 150 to 1650 K, with a normal operating temperature of 1375 K.

Although the refractory metal (W-5%Re/W-26%Re) thermocouples would operate at the specified temperatures, very significant drift in the temperature-to-electromotive force (emf) relation would occur with time at 1375 K and accumulated neutron dose. As a consequence, reliance could be placed on the thermocouple signals only during the early operating history of the reactor.

The requirements for the diverse temperature thermometer were that the thermometer sheath was to be a continuous (without joints) length of at least 3 m (9.8 ft), with an outside diameter of no more than 5 mm (0.2 in.). The thermometer could have a sensitive length of only about 100 mm (4 in.). The thermometer sheath had to be Nb-1%Zr to be compatible with the reactor tubing. The thermometer had to operate unattended for 10 years, maintaining its accuracy, while absorbing a dose of 10^6 Gy (100 megarad) of gamma and 3×10^{-15} neutron/cm^2. The temperature-response time constant of the thermometer was required to be no more than 8 s.

No industrial resistance thermometers had previously operated successfully at a temperature as high as 1375 K (1100°C). At high temperatures, the electrical resistance of the insulators used in most thermometers decreases and produces shunting errors in the temperature measurements. In addition, the sensing element gradually reacts with the surrounding materials to change the resistance-vs-temperature calibration. Resistance changes of industrial platinum resistance thermometers (IPRTs) at 1100°C that were tested in ideal conditions with no radiation effects would be much larger than 1–10°C/year, on the basis of extrapolation of drift rates observed at lower temperatures.[1]

A Johnson Noise Thermometer (JNT) can measure the temperature of a resistance element without drift and largely independent of shunting errors. Unlike all other temperature measuring systems that measure the change of a voltage or a physical property and relate the change to temperature, the JNT is an absolute measurement of temperature since it measures electron fluctuation in a resistor. These measurements are independent of the resistor material and resistivity over a wide range of electrical resistivity. To meet the SP-100 lifetime goal, the resistor must remain intact and have an electrical resistance that is small compared to its surrounding insulation. The JNT must integrate noise signals over a period of time (tens to hundreds of seconds) to obtain an accurate temperature reading, so prompt temperature measurements are not available. A JNT cannot be used to measure rapid temperature transients.

Combining the functions of a dc resistance thermometer with a JNT uses a sensor configuration that is suitable for both types of thermometers. The dc resistance thermometer mode can be used for prompt temperature measurements by using the known resistance-vs-temperature relation for the sensing coil. At the same time, the high-frequency noise signals processed by the JNT can be used to measure the absolute temperature of the sensing coil. In this manner, changes of electrical resistance caused by chemical or microstructure changes in the resistor material or by neutron-induced transmutation of the resistor material can be detected and the resistance-vs-temperature calibration corrected for future resistance thermometer measurements.

Another problem with the JNT is that mechanical vibration can produce microphonic electrical noise that can interfere with the temperature measurements. To avoid this problem, the refractory insulation surrounding the thermometer components is packed firmly enough that the components do not rattle. This process means that high-density, hard-fired, insulators cannot be used in the thermometer; instead, crushable insulators have to be used, which are much more prone to adsorb impurities and must be thoroughly dried and outgassed.

2. METHODS OF CONSTRUCTION

2.1 CONSTRUCTION PROCEDURE

The following sections describe the procedures used to fabricate a reliable resistance thermometer suitable for operation in contact with the Nb-1%Zr pipe containing molten lithium at 1375 K (1100°C). Further details of the individual steps and procedures are contained in the appendices.

2.1.1 General Principles

All the metallic components were required to be Nb-1%Zr to avoid compatibility problems. The electrical resistance of the sensing element required for best operation of the JNT is about 100 Ω at the operating temperature of 1375 K. The optimum electrical resistance for the lead wires is < 0.1 Ω to minimize errors in the JNT measurements. These requirements mandate the smallest-possible-diameter wire for the sensing element and largest-possible-diameter wire for the lead wires.

If small-diameter Nb-1%Zr wire is to retain continuity through a 10-year service life, the level of reactive substances within the sheath must be minimized when it is sealed. The niobium alloy required for the lead wires and the sensing element is especially prone to rapid oxidation at the design operating temperature.

The crushable insulation that must surround the lead wires and the sensor element is by far the largest potential source of oxygen and moisture. The large surface area of the insulation provides an adsorption site for moisture and gases. The crushable insulation can be outgassed initially by vacuum heating, but the insulation must not be in the same container with the sensing element during the outgas procedure so as to prevent a reaction between the desorbed gas and the Nb-1%Zr sensing element. The insulation preforms must be outgassed before the preforms are installed on the lead wires or the sensor. Moreover, the insulation preforms cannot be subsequently exposed to air or moisture after they are outgassed. To meet these objectives, the thermometer must be assembled and sealed in an inert atmosphere.

2.1.2 Construction Flow Plan

The assembly construction procedure is shown in Fig. 1. Descriptions of the testing and the assembly procedures are given in the main body of the document, with additional details provided in the appendices.

2.2 INERT ATMOSPHERE FACILITY

2.2.1 Basic Plan

A primary and secondary cover system of inert gas is used to prevent air and moisture from contacting the insulation. The primary system is heavy-walled quartz tubing, 8-mm ID (0.31-in. ID), 3.2 m (10.5 ft) long, closed on one end and with "3/8-in." compression fittings sealing the open end (see Figs. 1A, 1B, and 2). During fabrication, each assembly is stored and transported in its individual quartz tube. The primary cover system for the insulators are sealed widemouth bottles. The sensor materials are removed from the primary containment only within the secondary containment of a glove box containing a positive pressure of dry, inert gas.

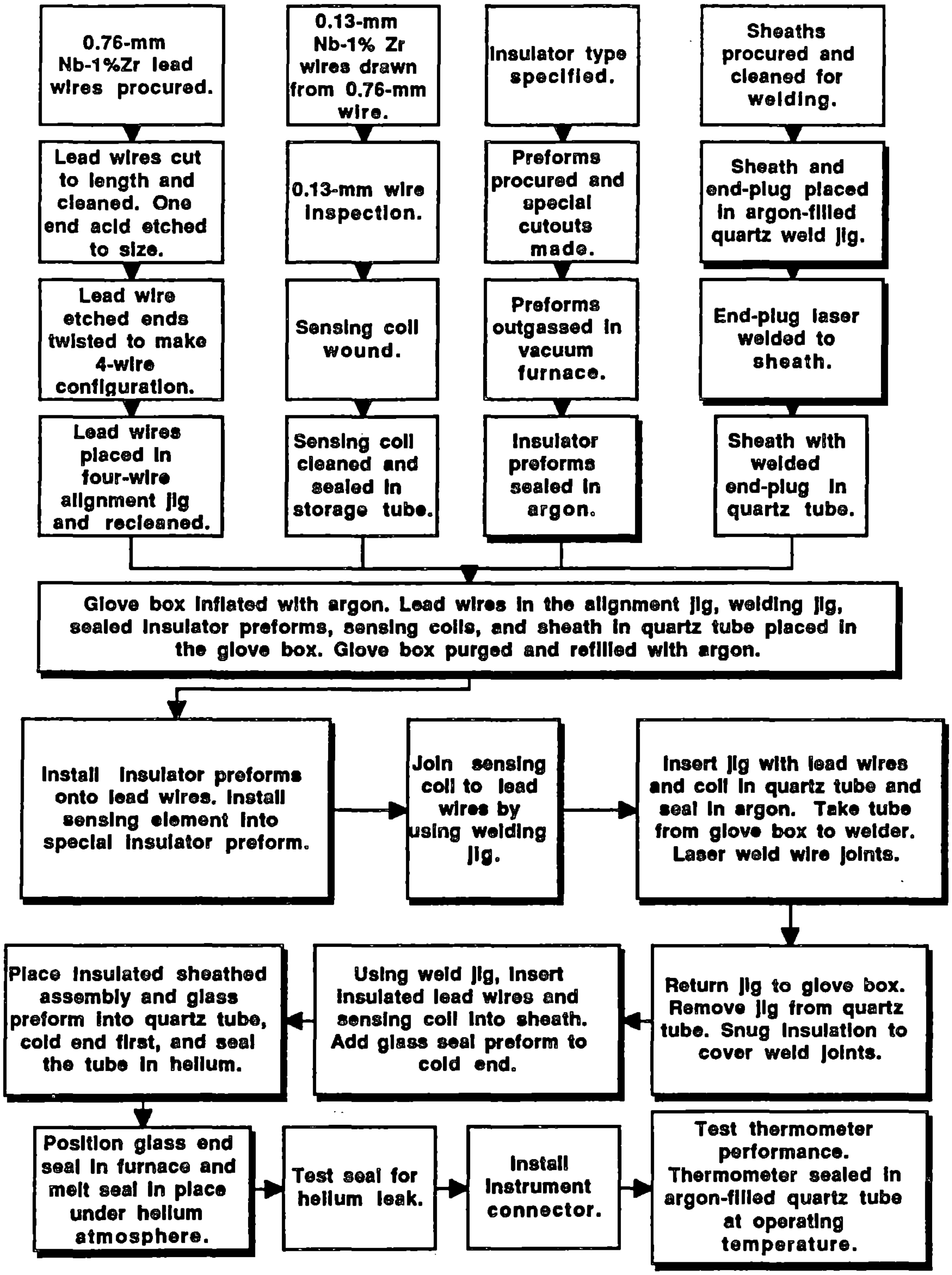
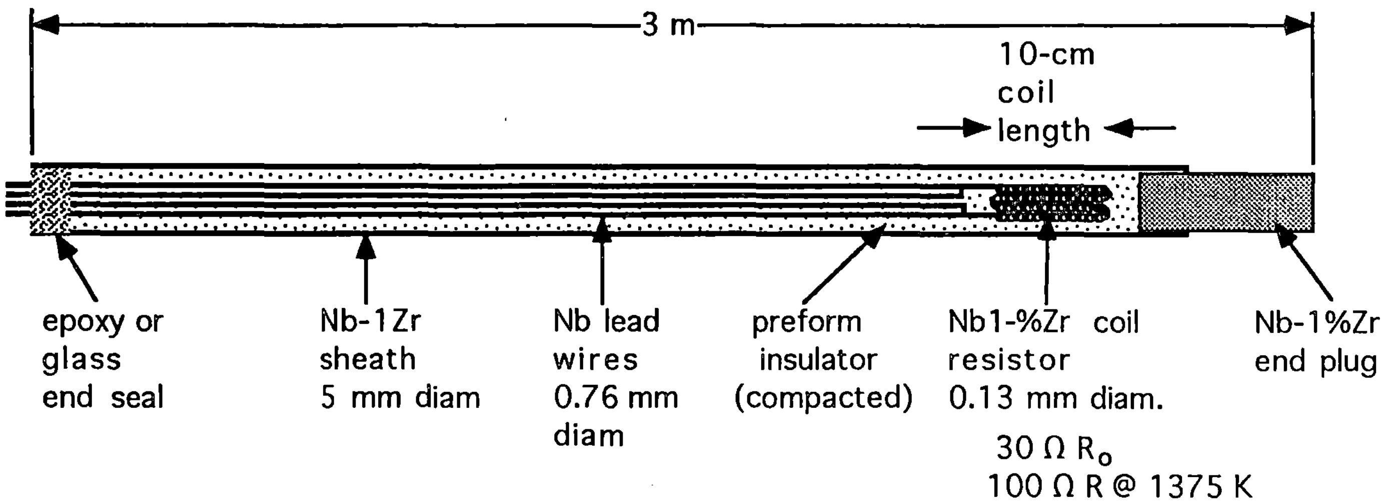


Fig. 1. Thermometer construction procedure (box shadow indicates that the work is done in an inert gas cover).



- Designed for use up to 1700 K
- Materials compatible with SP-100 coolant pipes
- 4-lead design can be used as a noise or as a resistance thermometer

Fig. 1A. Johnson Noise Thermometer design.

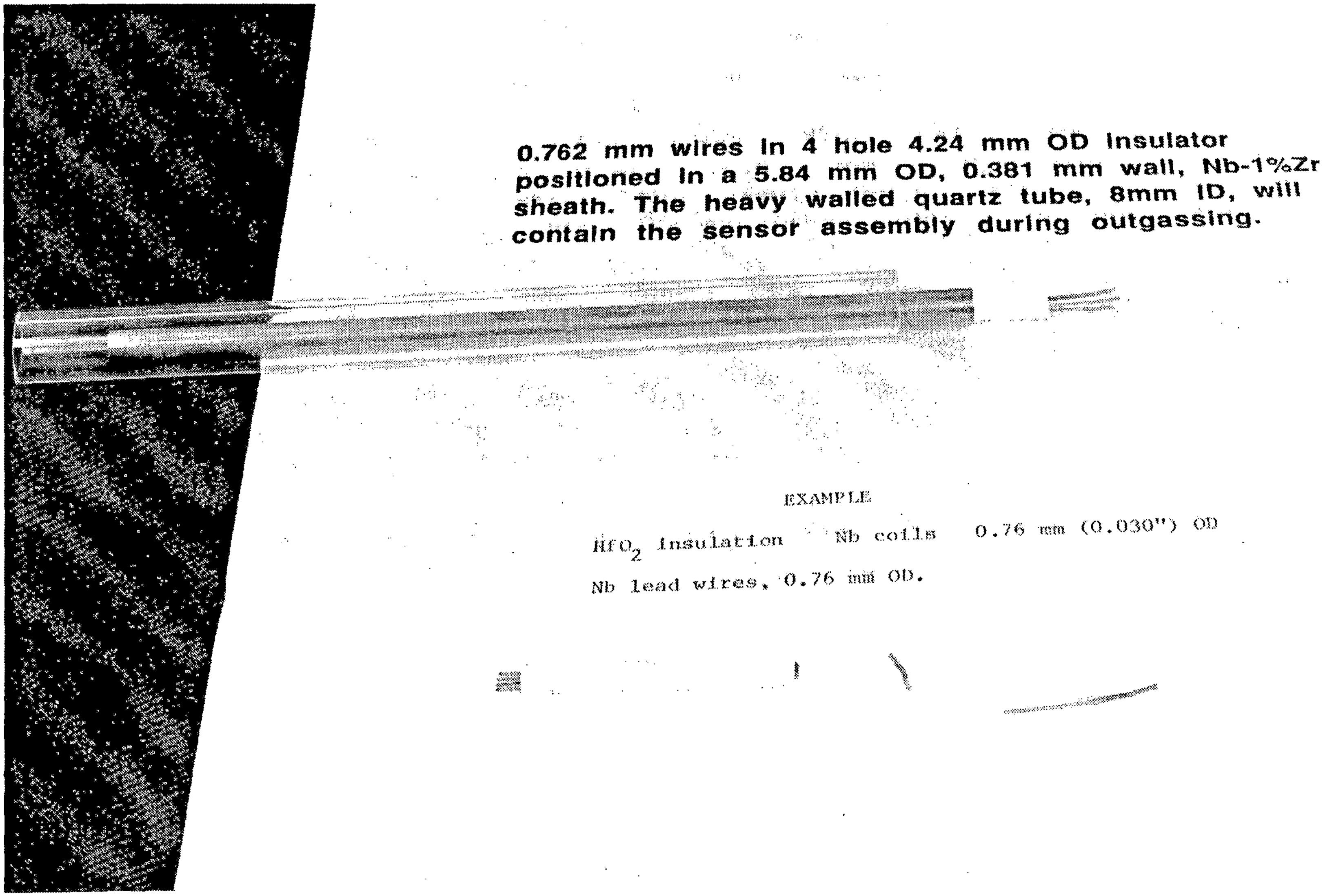


Fig. 1B. Photo showing thermometer components.

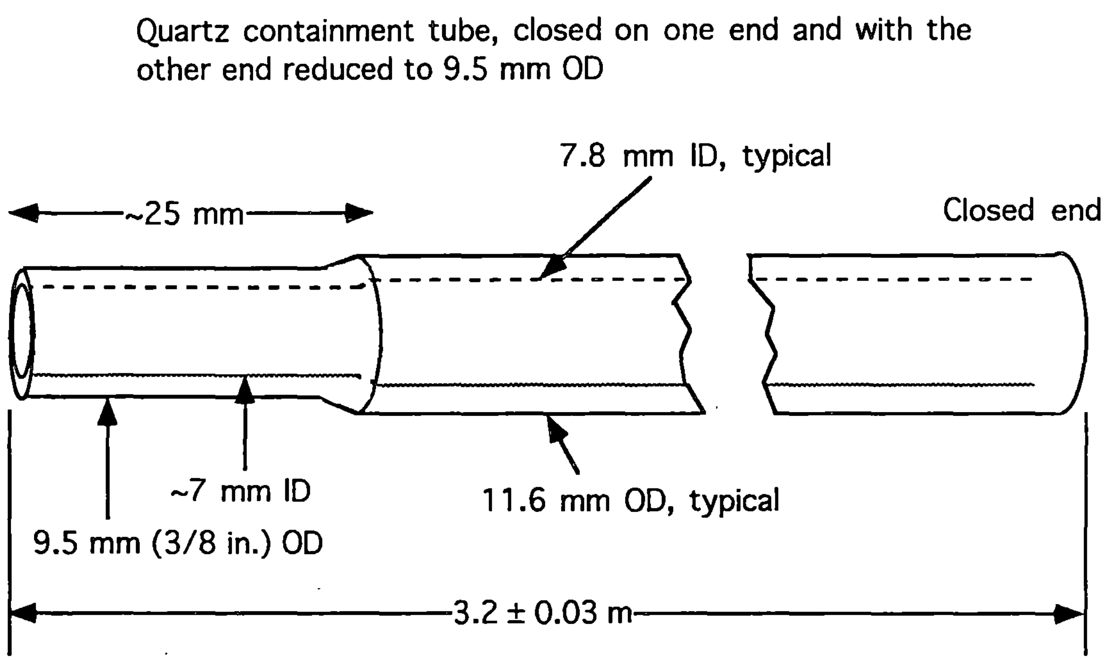


Fig. 2. Quartz tubes used for primary containment.

A glove box is necessary to provide an assembly area with an inert atmosphere. Conventional glove boxes are not long enough to allow a 3-m-long sensor to be assembled unless special extensions are fitted to the entrance ports. A 6-m-long glove box is required to allow 3-m-long components to be placed in a 3-m-long sheath. No glove boxes of this size were available at Oak Ridge National Laboratory (ORNL), and a custom-built conventional glove box would be prohibitively expensive.

A conventional glove box is expensive mainly because a rigid design is required for the containment of toxic materials. Our specific requirement, however, was only to contain an inert atmosphere around a nontoxic system. An additional requirement was to be able to see the material within the glove box and to be able to manipulate the components. These requirements were met by a secondary cover system consisting of an inflatable 10-m-long (32-ft-long) glove box of clear, 0.32-mm-thick (0.0125-in.-thick), polyvinylchloride (PVC) plastic that was fabricated on site and fitted with surgical gloves (see Fig. 3). The box was made long enough so that the internal components of the 3-m-long thermometer could be slid into the sheath without bending.

2.2.2 Plastic Glove Box

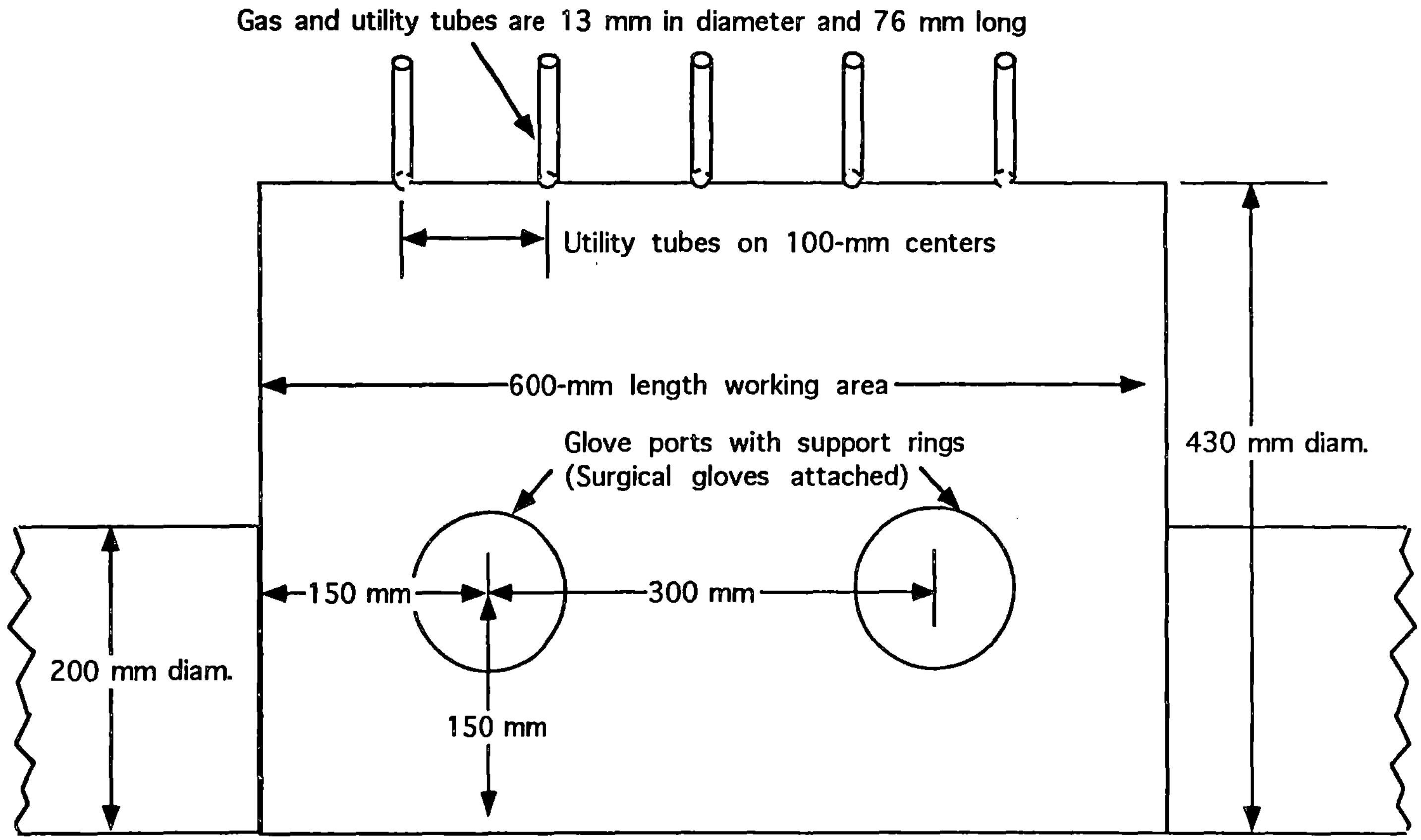
A low-cost, flexible, plastic bag-type glove box was designed and constructed of 0.32-mm-thick (0.0125-in.-thick) PVC clear plastic sheet, which is easily heat sealed and is compatible with niobium alloys at room temperature. The working section is a tube 0.43 m (17 in.) in diameter and 0.6 m (24 in.) in length, with two sleeved openings that accommodate surgical gloves. Five openings of 13-mm (0.5-in.) diameter allow vacuum, gas, electrical, and instrument line access (see Fig. 4).

Plastic access tubes 0.2 m (8 in.) in diameter and 4.6 m (15 ft) in length are joined to each end of the working section (see Fig. 5). The access tubes have openings for vacuum and gas fill. Entrance to the inert gas-filled working section is obtained by first clamping the access tube closed near the working section and then opening the end of the access tube and inserting the item, moving it along the tube by grasping through the flexible, tear-resistant plastic. The end of the access tube is clamped or heat sealed and evacuated. The plastic tube will simply collapse around the item, so the item had to be sturdy enough to withstand the pressure or else be protected. The evacuated access tube is then filled with inert gas. The evacuation and filling procedures are repeated several times to ensure that the access tube gas is sufficiently pure before unclamping the section between the access tube and the working section. When not in use, the plastic glove box can be collapsed or folded and is easily transported and stored since it weighs only a few kilograms.

2.3 CONSTRUCTION CONSTRAINTS

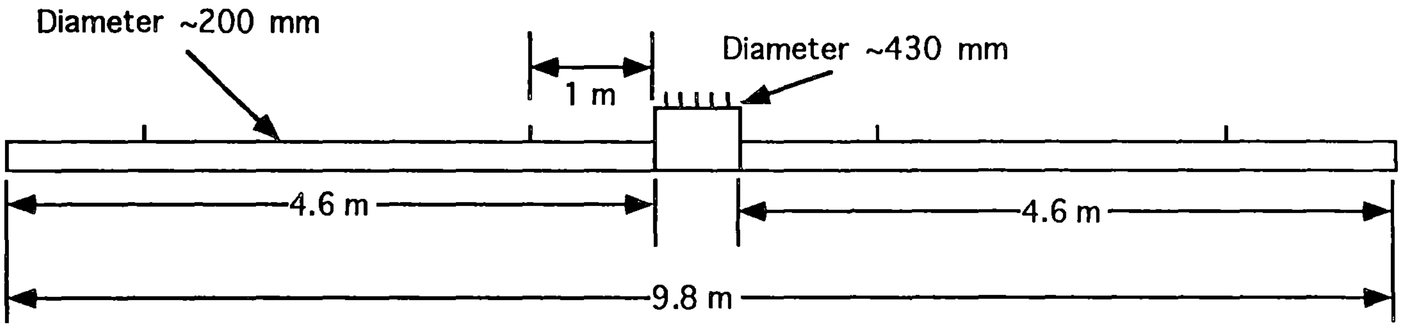
Long-term reliability of the sensor at moderately high temperatures is required, although freedom from resistance drift is not, since drift can be corrected by the JNT measurements. Reliability consists of maintaining electrical circuit continuity. The choice of relatively large diameter [0.127-mm (0.005-in.)] wires for the sensing element, minimizing the variety of different materials within the sheath, and optimum fabrication and joining techniques are expected to provide long-term circuit continuity. Long-term, high-temperature durability of the sensing element may also be affected by the grain size, annealing condition, and thermal processing of the sensing element. The sensing element and lead wires should have grain sizes small enough that no grain boundary will bridge the wire diameter.

Fig. 3. PVC plastic glove box for secondary containment. Working section is 0.6 m long, 0.4 m in diameter, and has 0.2-m-diam wings, 4 m long, attached to each side of the working section.



Note: 13-mm-diam gas and utility entrance tubes are also located on the 200-mm-diam side tubes. One tube is located 1 m from the center section, and another is 3 m from the center section. All entrance tubes are on the top side.

Fig. 4. Center section of glove box.



Material is PVC clear plastic sheet, 1.37 m wide. Tube 200 mm in diameter results from joining 1/2 width of the 1.37-m-wide sheet. Center tube results from joining a full width of the sheet. The diameters are approximate, depending on the size of the lap joint.

Tubes are to be joined so the lower ends are aligned.

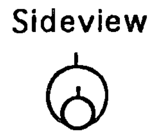


Fig. 5. Overall side view of plastic glove box.

2.3.1 Materials Compatibility

The thermometer was intended to be clamped to the reactor coolant tubing, and it was essential that the thermometer sheath not react with the thin-wall Nb-1%Zr tubes containing the lithium coolant of the SP-100 reactor. For compatibility, Nb-1%Zr was also selected for the thermometer sheath material. Since this alloy was available in the form of high-purity tubing and could be easily worked, no alternative materials were considered for the SP-100 application.

Compatibility of materials within the Nb-1%Zr sheath was optimized by using either niobium or Nb-1%Zr for the sensing element and lead wires. Any other metals would have introduced additional chemical constituents into the sheath interior that might migrate with time at the operating temperature and cause early failure in the sensing element. The alloy Nb-1%Zr is favored because it is less susceptible than pure niobium to the growth of large grains in the small-diameter sensing element wire.

Insulator materials considered included alumina, beryllia, hafnia, magnesia, and yttria. Magnesia was rejected since it was expected to be more reactive with niobium than alumina and has an insulation resistance that is sensitive to impurities. Alumina was one insulator that combined availability as high-purity, low-density, crushable preforms with good, high-temperature electrical resistivity characteristics. Hafnia was difficult to obtain in low-density, crushable preforms of adequate purity. Beryllia was avoided because of its apparent toxicity. Yttria could be obtained in high-purity, crushable preforms and, thermodynamically, yttria appeared to be more stable in the niobium environment than alumina, but it was expected to have a lower insulation resistance at high temperatures than alumina. One prototype thermometer was by constructed by using alumina insulation, and another was constructed by using yttria insulation.

2.3.2 Johnson Noise Thermometer Requirements

One of the problems with the JNT measurements is that mechanical vibration can produce microphonic electrical noise that can interfere with the measurements. The microphonics can be reduced by compacting the insulation about the sensing element and the lead wires. The construction plan was to insulate the sensor with a crushable metal-oxide ceramic and, once the sensor was constructed, swage the sensor sheath to a smaller diameter, crushing and compacting the insulation about the sensing element and the lead wires. An alternative method might use vibratory compaction of insulator powder.

A four-lead design, with two lead wires connected to each end of the sensing element, provides redundancy and allows the lead wires to be parallel to reduce lead resistance for JNT measurements. The four-lead design also permits dc measurement of the coil resistance, independent of lead resistance.

2.3.3 Thermometer Configuration

The thermometer design selected used conventional fabrication methods—drawing, swaging, welding—for which commercial sources are available and problems of contamination control have already been solved. Alternative designs would have required extensive fabrication method investigation and qualification.

Materials selected for the thermometer were not constrained to those having low thermal neutron ($E < 1$ MeV) cross sections (Nb = 0.1 b, Zr = 0.1 b, Hf = 104 b, Be = 0.009 b, Al = 0.24 b), since the total neutron fluence for the sensors in the SP-100 application below the shield was to be 3×10^{15} neutrons/cm^2 (nvt). These thermometers will experience much less radiation than fuel irradiation testing thermometers, which are tested to thousands of hours, receive in the first few minutes of operation.

2.3.4 Thermometer Design Requirements

Accuracy: The temperature accuracy of the thermometer is to be ±10 K up to 715 K and then to be ±1.4% of the temperature up to the design maximum of 1650 K.

Response Time: The attached thermometer must have an overall response time of <8 s seconds at operating temperature.

Size: The thermometer is to be 3 m (9.8 ft) long, have a 5-mm (0.2-in.) diameter, and have a 100-mm (4-in.) sensitive length.

Durability: The SP-100 reactor is required to run at rated power for seven years and operate at standby power for three years, for a total reactor lifetime of ten years. The thermometer system is to operate within the required accuracy during the reactor lifetime. Since the JNT measurements are largely independent of resistance changes, the requirement is primarily that the sensing element retain continuity.

Drift: There are no drift limitation requirements (in the classical sense) that changes of the sensor resistance-vs-temperature relation should be less than a specified amount. If the JNT measurements should verify that there is no drift, or only very gradual drift, the sensing element can be used as a resistance thermometer to give continuous prompt temperature measurements.

Radiation: The fluence of 3×10^{15} neutrons/cm^2 (nvt) and the gamma dose of 1×10^4 Gy (1×10^6 rad) should have no significant effects on the sensor materials.

Temperature Range: The nominal operating temperature is 1375 K (1100°C), ranging to a low of 150 K (–123°C) if in deep space without the reactor operating. The upper temperature limit of 1650 K (1375°C) is the maximum predicted during a design-basis-event transient. The thermometer design will withstand these temperatures except for the cold seal region, which is not expected to be exposed to high temperatures.

Vibration Environment: The thermometer must withstand a 32-g peak gross dynamic load and a sustained vibration at 200 Hz and 0.2 g for 20 days.

Emplacement: A clamp or wire must be used to attach the thermometer to the thin-wall reactor coolant tubing since spot-welding of attachment devices to the thin-walled lithium containment tubing is prohibited.

2.3.5 Thermometer Materials Compatibility

Sheath: The thermometer sheath must be Nb-1%Zr, the same material as the reactor coolant containment tube to which it will be attached. The thermometer sheath is compatible with the proposed metal-oxide insulation over the temperature range of operation.

Sensing Element and Lead Wire: The first-choice material for lead wire and sensing element is pure niobium, which is even more compatible (i.e., less reactive) with the metal-oxide insulation than is the Nb-1%Zr alloy . However, grain growth problems in pure niobium require the use of Nb-1%Zr alloy for the small-diameter sensor element wires. (The first prototype sensing element used pure niobium because of the unavailability of Nb-1%Zr alloy wire in small diameters.)

2.3.6 Sensing Element Electrical Resistance

The target resistance for JNT measurements was ~100 Ω at the design operating temperature of 1375 K (1100°C). For 100-Ω resistance at 1375 K, a 0.127-mm-diam (0.005-in.-diam) niobium wire must be about 3 m in length. If coiled on a 0.51-mm (0.020-in.) mandrel, with one wire diameter between turns, the coil is about 38 cm (15 in.) long. A sensing element 107 mm (4.2 in.) long can be formed by looping the coil in a 4-hole insulator (see Fig. 6). A smaller diameter wire would make a shorter coil, but one more likely to fail.

2.3.7 Materials Restrictions

The thermometer sheath was to be pressed against the Nb-1%Zr tubing containing the reactor coolant, and to avoid any compatibility problems, the sheath of the thermometer should be the same alloy as the reactor tubing.

At high temperatures, especially in the presence of a temperature gradient, a driving force exists for the migration of the elements from one alloy to an alloy of different composition. In this situation, there can be a mass transfer from one component to another. The mass transfer in the thermometer could be reduced by using the same alloy for the sensing element, the lead wires, and the sheath.

The hot end closure had to be welded to the thermometer sheath, and the connections between the sensing coil and the lead wire had to be welded. Brazing could not be used since the braze material would introduce extraneous elements which might cause early failure. Welding the small wire joints and the sheath had to be done under inert gas and without an electrode or any type of flux. The welding was done by passing a laser beam through a quartz window onto the components located in an argon-filled chamber. The edges of the niobium alloy parts were melted together to form a weld without the use of fill material. This welding method required tight fits for all components since no fill metal was used. All the welds were ductile and leaktight. The good ductility proved the purity of the inert gas since the first sign of oxidation is the embrittlement of niobium.

No organic materials could be used in the thermometer because of both the high temperatures and irradiation levels. Delays in obtaining suitable glass seals for the cold end seal required the cold ends to be sealed with epoxy for the demonstration thermometers.

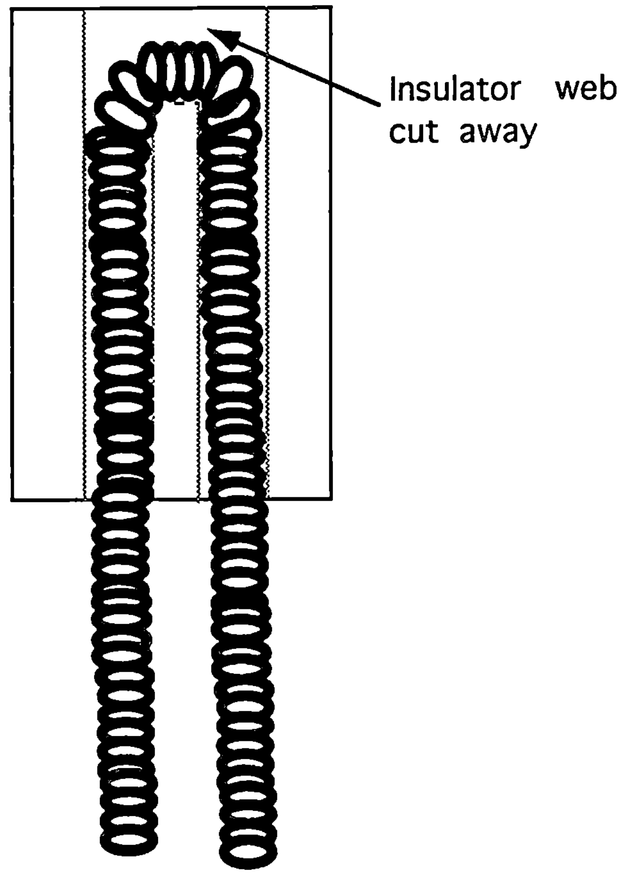
2.3.8 Size Restrictions

The thermometer was specified to have an uninterrupted length of about 3 m (10 ft) and a diameter no larger than 5.08 mm (0.20 in.). The tubing for the sheath was purchased in 3-m lengths with a diameter of 5.82 mm (0.23 in.) and a wall thickness of 0.38 mm (0.015 in.). The tubing was to be reduced to 5.08-mm diam by swaging the sheath after the sensor was constructed. The alumina-insulated thermometer was constructed to the specified 3-m length, but for demonstration purposes, an yttria-insulated thermometer was fabricated only 1 m (3 ft) long. Because of curtailment of the program, only the compatibility capsules were swaged to the final diameter.

2.3.9 Response Time Restrictions

The requirement for an 8-s time constant for a resistance thermometer with a 5-mm OD could be met only by filling the sheath of the thermometer with helium to improve the heat transfer through the crushed insulation. There were no response time tests made since these tests could be made only after the thermometer was swaged to the final size.

Embedded Coil Turn



Webbing cutouts for sensor insulator

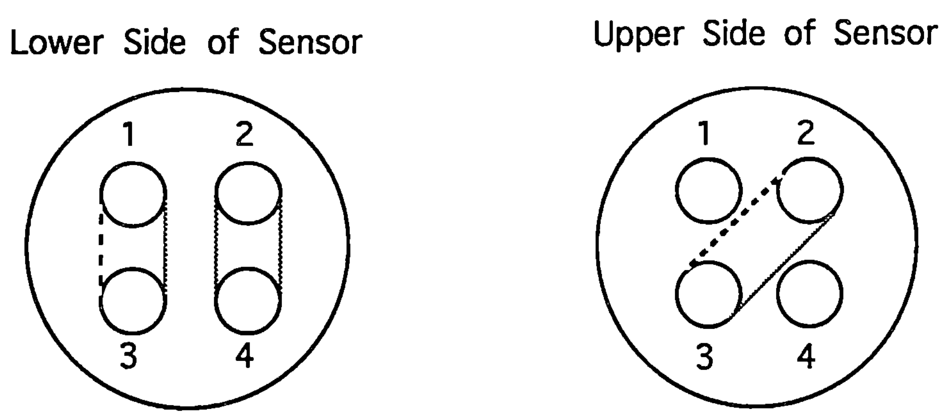


Fig. 6. Sensing element coil snakes through insulation.

2.3.10 Handling Restrictions

The niobium and niobium-based alloys are subject to damage from minute amounts of contaminating materials. Just the touch of nickel, copper, gold, or platinum during room-temperature handling can result in high-temperature failure of the alloy. All of the construction techniques had to ensure that the niobium was cleaned properly and never touched except with carbon steel or plastic tools or with surgical gloves. Niobium and Nb-1%Zr are particularly sensitive to attack from oxygen, so none of the metallic elements of the thermometer could be exposed to air at higher than room temperatures.

The crushable ceramic insulation was outgassed in a vacuum furnace at temperatures just below the densification level (800°C for the high-purity, ultrafine-grain alumina; 1250°C for hafnia). The outgassed ceramic was sealed in argon-filled jars. Care was taken that the ceramic preforms were never exposed to air after the outgas procedure.

2.4 COMPATIBILITY TEST CAPSULES

The purpose of the compatibility test was to determine whether the combination of materials and fabrication technique would result in material interactions that would compromise the integrity of the thermometer wires and sheath.

Test specimen capsules were constructed by using the materials and techniques proposed for constructing the thermometer. The concept was to fabricate capsules from the thermometer sheath material and enclose the insulation, lead wires, and sensing element wire. The process of constructing the capsules included all the techniques that would be used for construction of the thermometer. The procedures included the construction under inert gas, remote laser welding, helium leak testing, swaging the capsule from 5.82- to 5.08-mm diam, and even moving the capsules between fabrication facilities in the same manner as the full-sized thermometer. The dimensions of the capsule are shown in Figs. 7 and 8.

2.5 CONSTRUCTION OF A PROTOTYPE THERMOMETER

One 3-m-long prototype thermometer, with Nb lead wires and sensing element coil and insulated with fine-grain, high-purity alumina, was completed except for swaging. The prototype thermometer was coded S/N 52 by the serial number engraved on the hot end closure of the sheath. Pure Nb lead wire and sensing element wires were used because the Nb-1%Zr wire had not been obtained. The purpose of the prototype was to test the construction technique and to produce a thermometer that could be used for electronic evaluation. For that reason, only one full-length thermometer was constructed. The plan was for subsequent sensors to use certified Nb-1%Zr wire.

A second thermometer, S/N 33, with dimensions identical to the prototype except for a 1-m length, was constructed by using certified Nb-1%Zr lead wires and sensing coil and yttria insulation. The shorter thermometer (S/N 33) was fabricated by using the same techniques as those for the prototype (S/N 52). The thermometer S/N 33 was constructed primarily to compare the insulation resistance of yttria with that of alumina. There was some concern that alumina insulation might have a long-term high-temperature reaction with the zirconium to form Al-Zr bimetallics and decrease the insulation resistance.

The full-length, alumina-insulated thermometer (S/N 52) with a pure niobium sensing element was placed in an argon-filled quartz tube and inserted into a furnace as shown in Figs. 9 and 10. The furnace was brought to equilibrium temperature, determined when the three monitoring thermocouples and the sensing

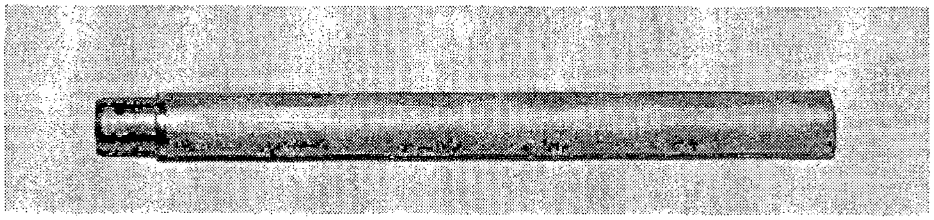


Fig. 7. Nb-1% Zr end plug laser welded into Nb-1% Zr sheath tube.

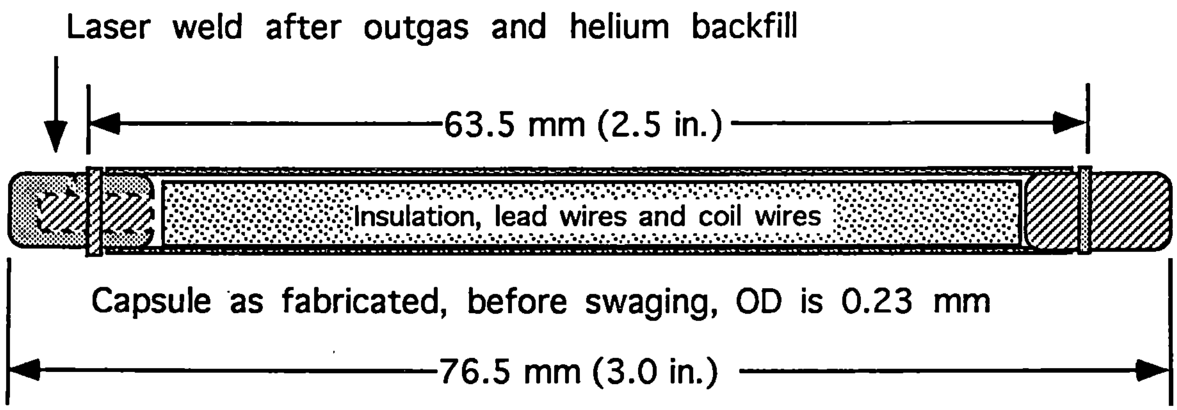


Fig. 8. Compatibility capsule dimensions.

element resistance were stable. The resistance of the sensing element was measured with the 4-wire potential-drop method to ±1 mΩ. The insulation resistance between conductors and sheath was measured with a megohm bridge at 10-Vdc potential. The Johnson noise measurement was made with a tuned-circuit signal processor. The equilibrium temperature was measured by a Pt-10%Rh/Pt (type S) thermocouple. The type S thermocouple measurement resolution was ±1°C; however, the absolute accuracy of the thermocouple is not better than ± 2°C and may be as bad as ±1 to 2%.

The 1-m-long, yttria-insulated thermometer (S/N 33) with an Nb-1%Zr sensing element and lead wires was tested in the same manner as that used for the 3-m-long, alumina-insulated thermometer with an Nb sensing element (S/N 52).

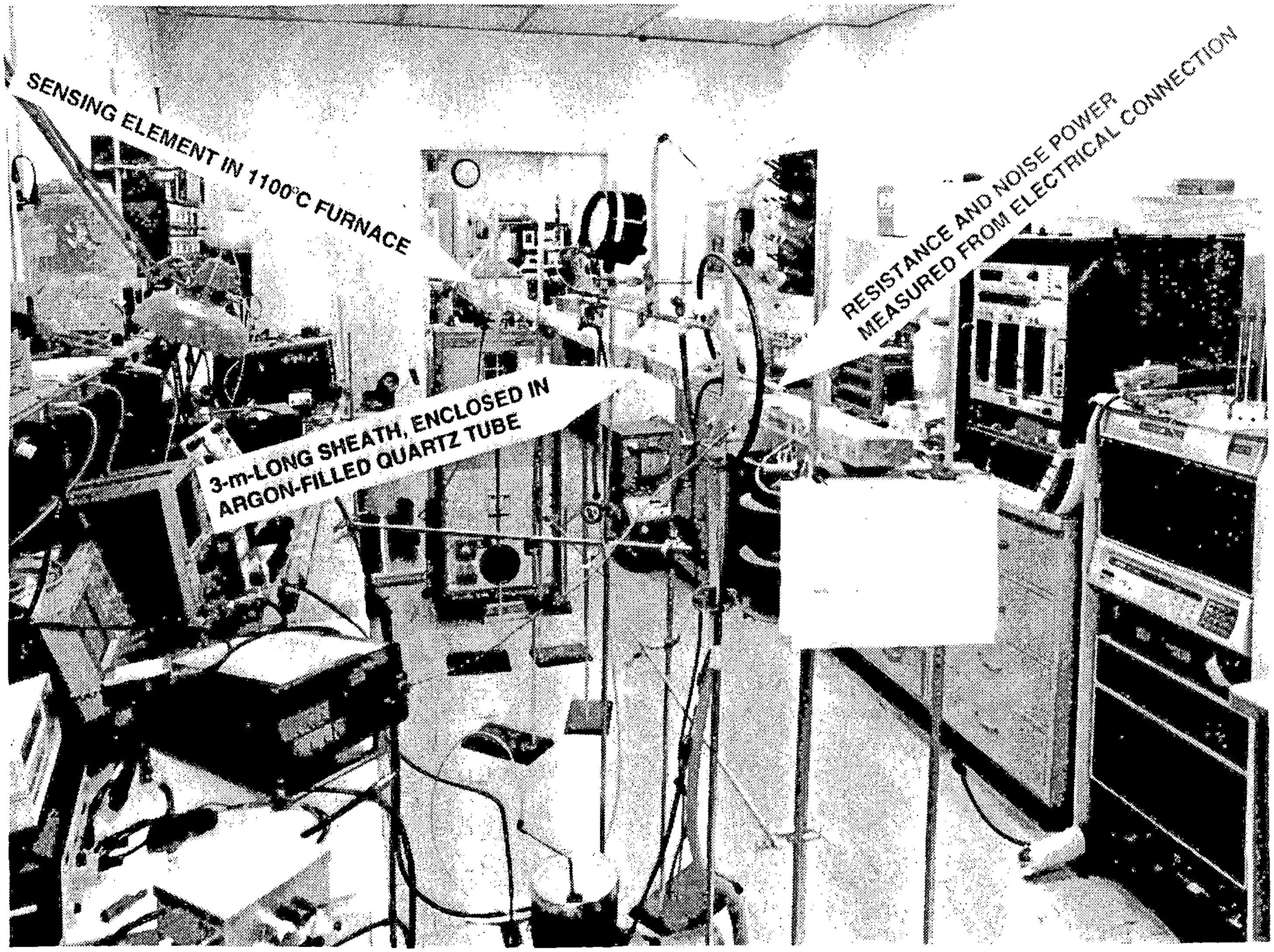


Fig. 9. Prototype Johnson Noise Thermometer.

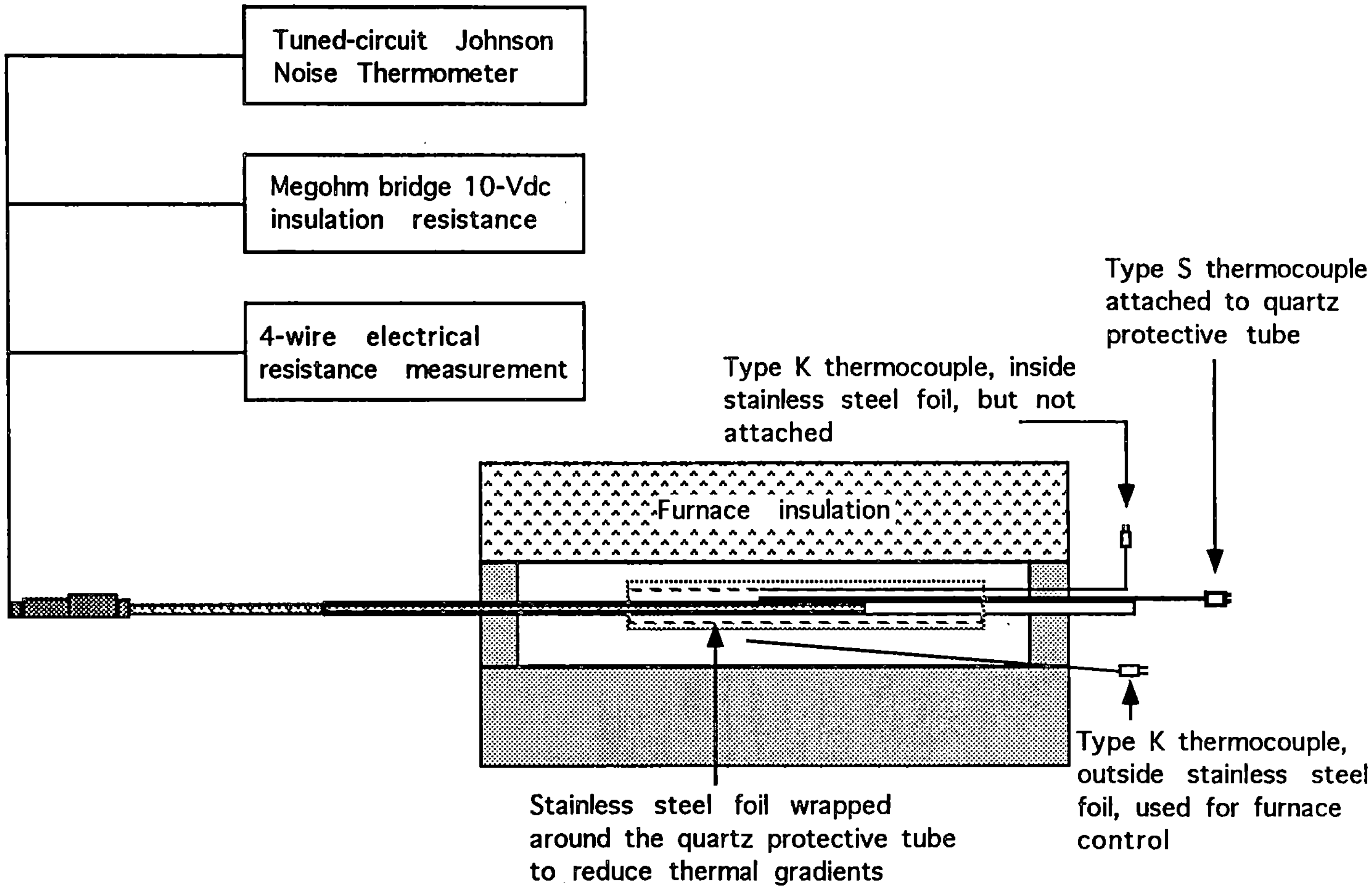


Fig. 10. Electrical measurements made during furnace testing of thermometer S/N 52.

3. PERFORMANCE TEST RESULTS

3.1 Nb AND Nb-1%Zr SENSING ELEMENT RESISTANCE VS TEMPERATURE

The Nb thermometer, insulated with alumina (S/N 52), was tested during repeated temperature cycles. The sensing element resistance showed very little hysteresis with temperature cycling (see Fig. 11) or with short-term but repeated exposure to 1373 K (see Fig. 12A). The Nb-1%Zr, yttria-insulated thermometer (S/N 33) was tested only briefly because of funding limitations. Results of the measurement of the resistance-vs-temperature relationship of the two sensors are shown in Fig. 12B.

The sensing element resistances of both thermometers are a smooth reproducible function of temperature from room temperature to 1373 K (1100°C). The resistance of the Nb-1%Zr (S/N 33) has a slightly higher resistivity and temperature dependence than the pure niobium (S/N 52). Either sensor could be used as a conventional dc resistance thermometer. The electrical resistivity of niobium and Nb-1%Zr is compared to that of molybdenum, tungsten, platinum, rhenium, and Nichrome V and is shown in Fig. X.1.1 in Appendix X.1.

3.2 INSULATION RESISTANCE VS TEMPERATURE

The alumina-insulation resistance (measured at 10 Vdc) used in sensor S/N 52 decreased exponentially as the temperature increased above ~600 K (Ref. 2). The alumina insulation resistance measurements were very stable and reproducible on heating and cooling, showing none of the drift that is usually associated with compacted, ceramic-insulated temperature sensors (see Fig. 13).

The yttria-insulation resistance used in sensor S/N 33 was about an order of magnitude lower than that of alumina over the entire temperature range, which is to be expected because of the smaller band gap of the yttria crystal, which is about half the band gap of the alumina crystal (see Fig. 14). The problems associated with electrical shunting may be more serious with yttria at the 1375-K operating temperature than with the alumina-insulated sensor.

3.3 JOHNSON NOISE POWER VS TEMPERATURE

The Johnson noise signals from the alumina-insulated thermometer S/N 52, using a least-squares fit of the noise voltage-vs-temperature data were linear with absolute temperature. The data obtained at temperatures above room temperature extrapolated to an intercept at zero noise power of <0.2 K (see Fig. 15). An ideal measurement would be linear with absolute temperature, and the noise power would be zero at absolute temperature zero.

The temperatures used to compare the Johnson noise measurements in Fig. 15 were measured by a type S thermocouple. The deviations of the noise measurements from an ideal linear relation can be attributed to the behavior of the thermocouple measurements. Figure 16 shows the differences between the thermocouple temperatures and those obtained from a linear fit of the noise temperatures. The variations are typical of the second- or third-order behavior of thermocouples. The uncertainty of the type S thermocouple measurement (which was not individually calibrated) is estimated to be at least ±4 K at 1375 K. Since the temperature deviations shown in Fig. 16 are much less than ±4 K, the differences between thermocouple and Johnson noise temperatures are probably due to uncertainties in the thermocouple calibration.

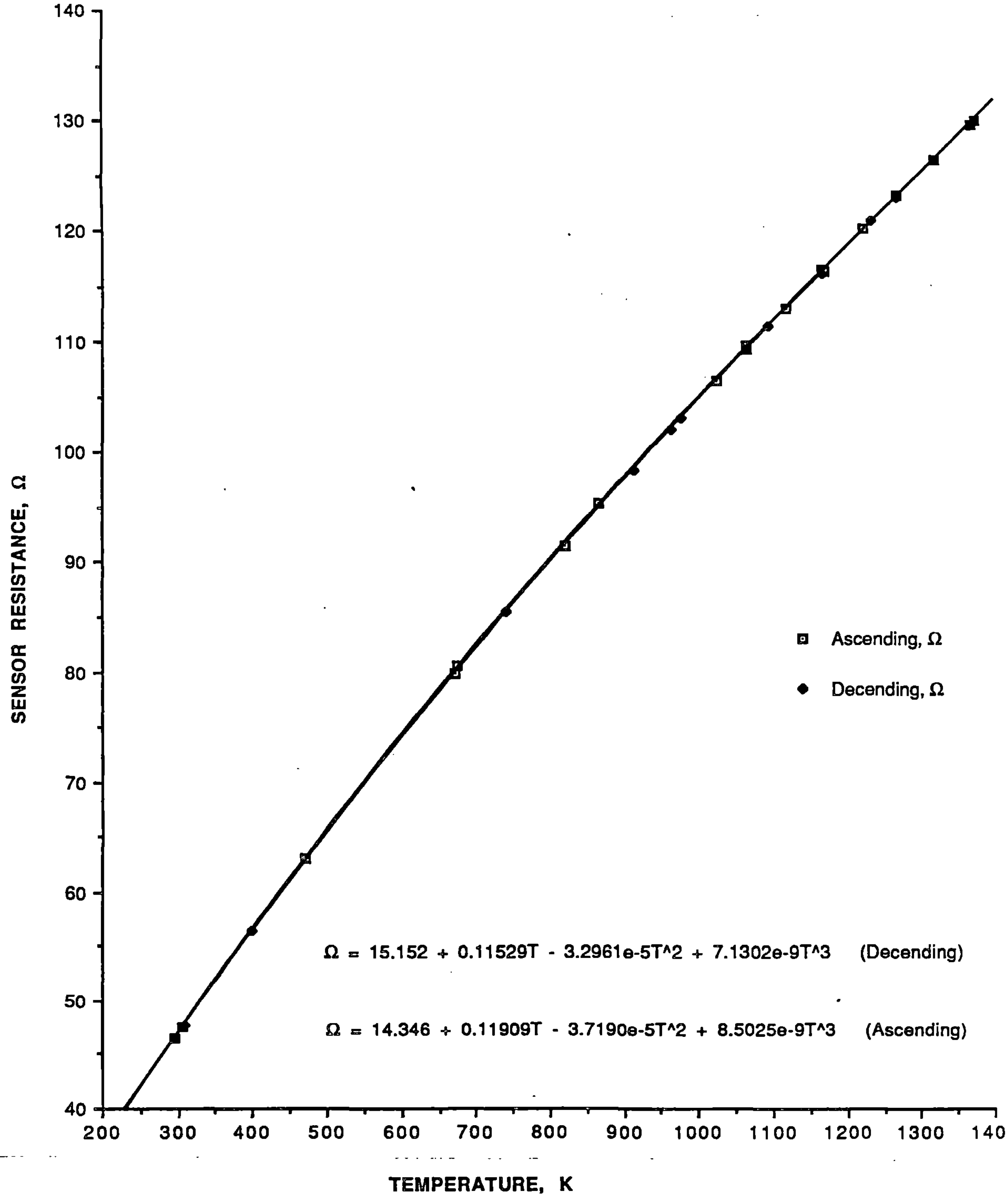


Fig. 11. Niobium sensing element S/N 52 has very little hysteresis.

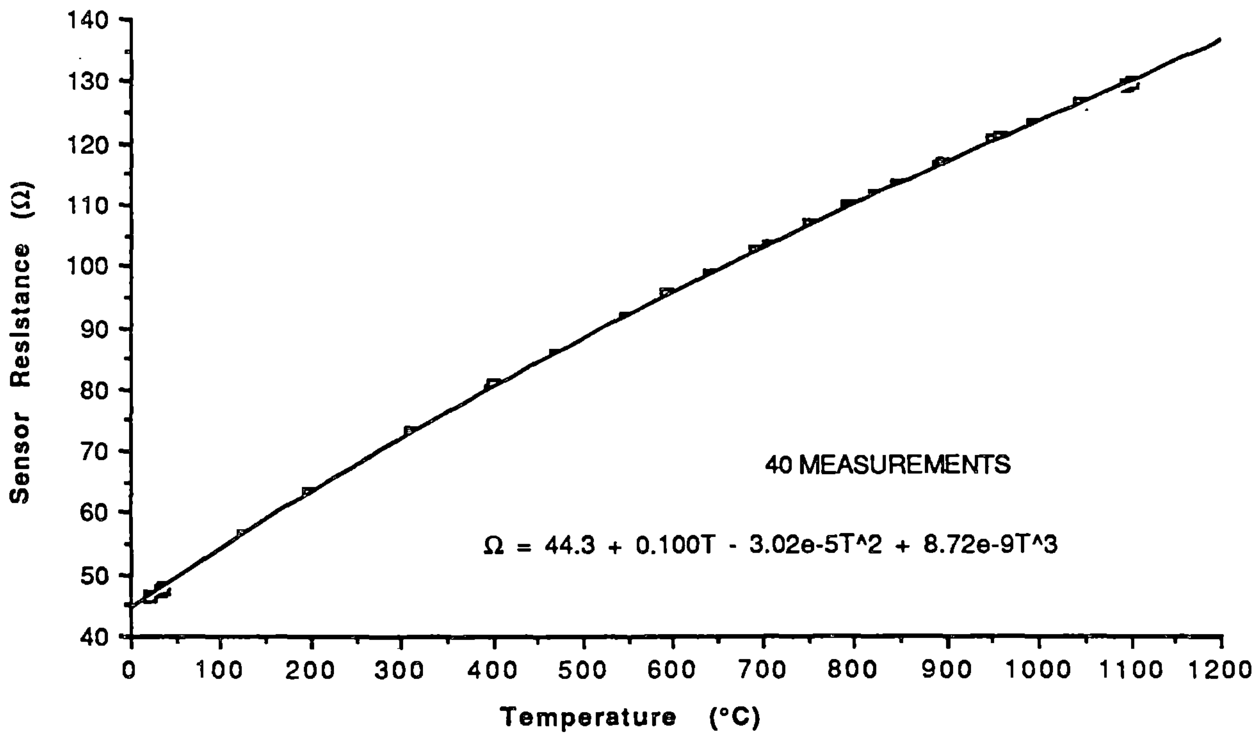


Fig. 12A. The diverse temperature sensor can serve as a high-temperature resistance thermometer.

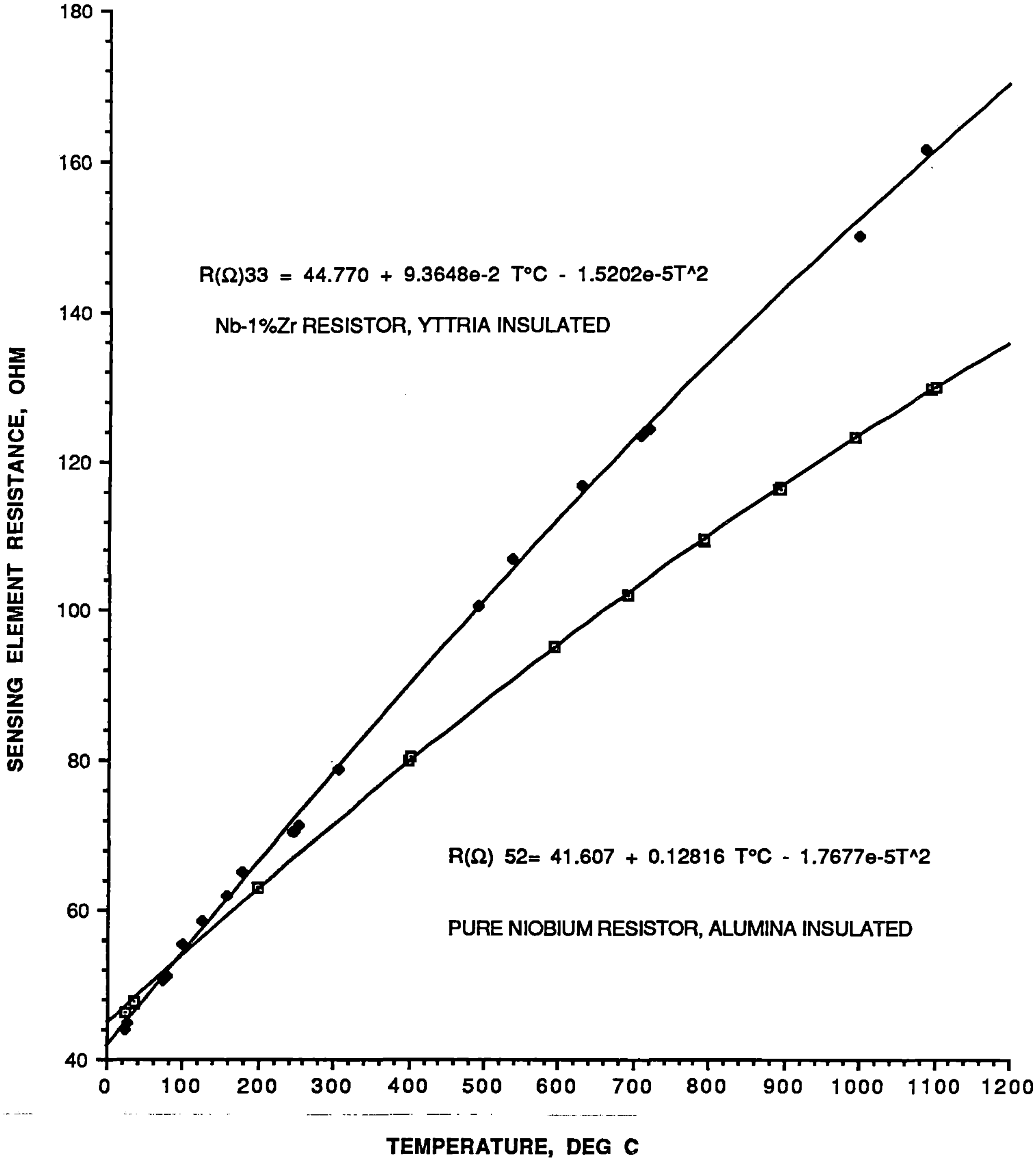


Fig. 12B. The sensing elements of thermometers S/N 33 and S/N 52 have different temperature-vs-resistance relations.

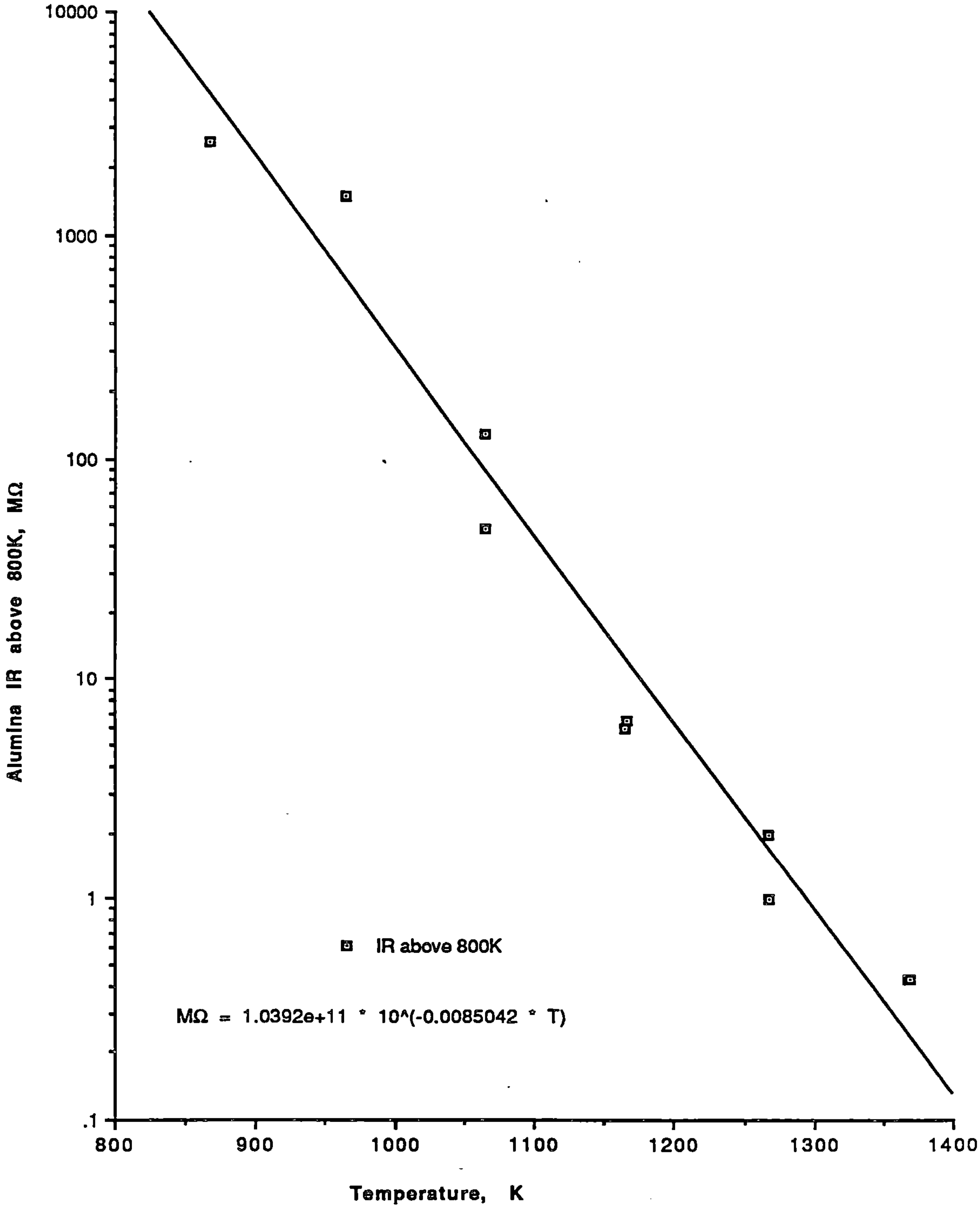


Fig. 13. Thermometer S/N 52 insulation resistance (IR), ultrahigh purity, ultrafine grain, alumina.

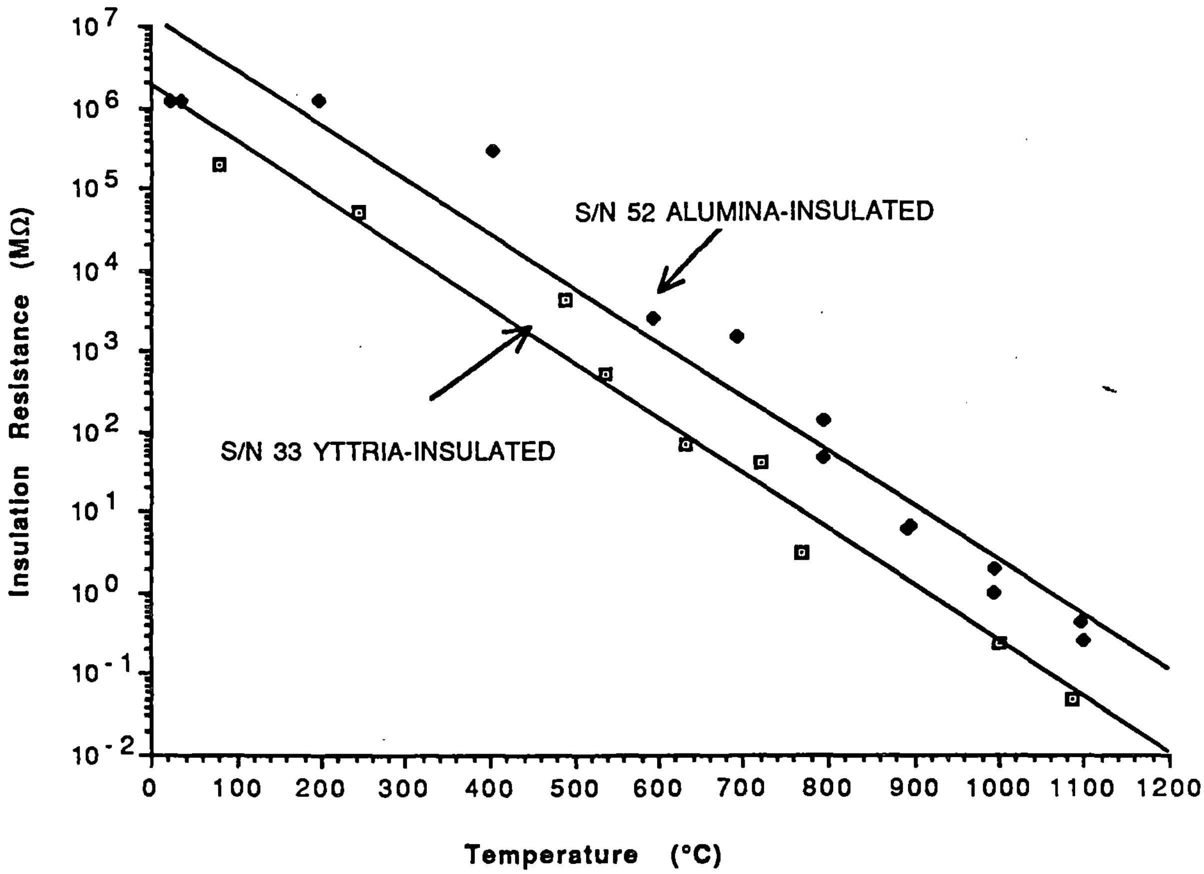


Fig. 14. The resistance of alumina is about 10 times greater than that of yttria.

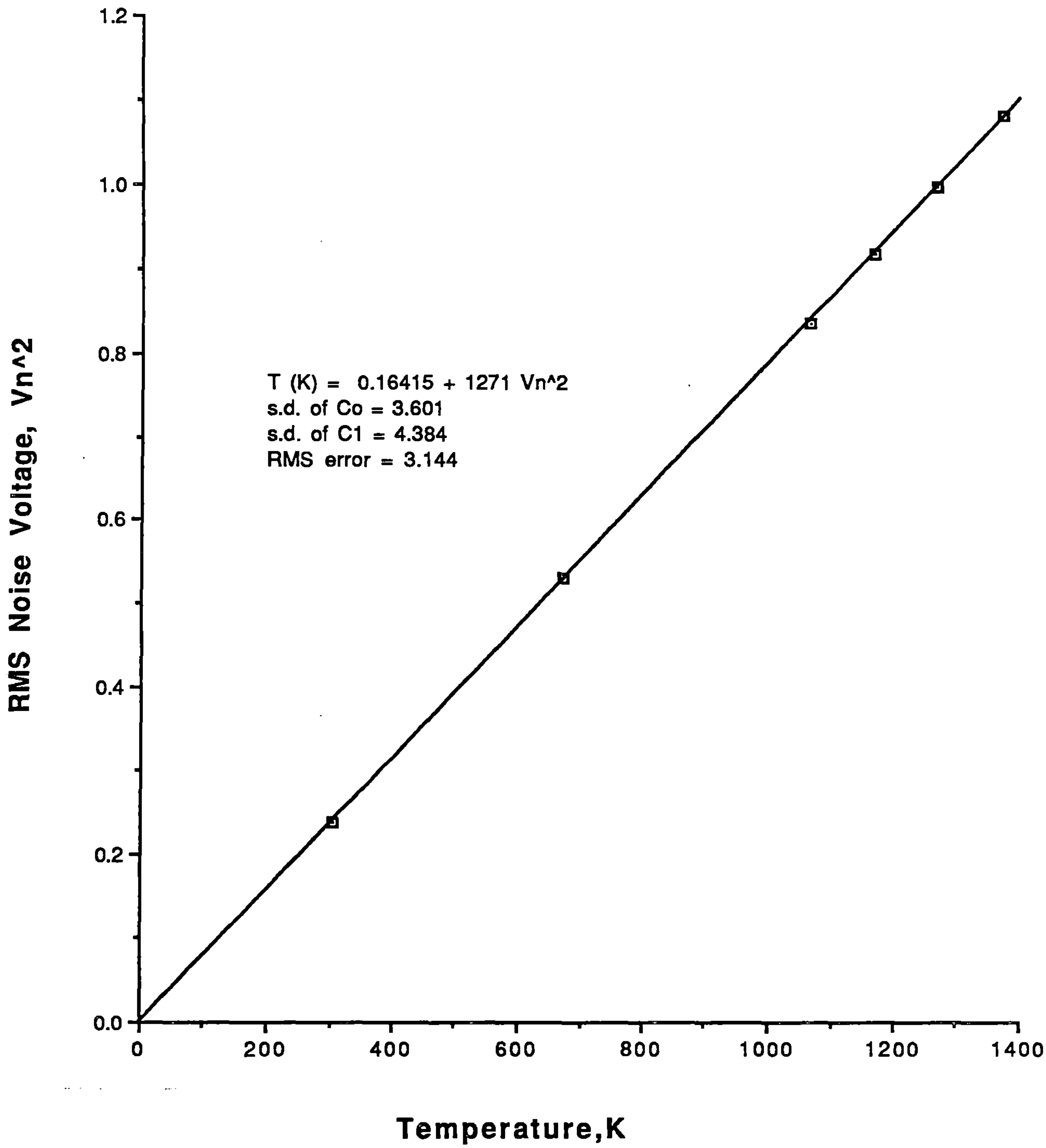


Fig. 15. Johnson noise measurements are linear with absolute temperature.

The Johnson noise signals obtained from thermometer S/N 33 (Nb-1%Zr, yttria-insulated) were also linear with absolute temperature, but the data showed considerably more scatter than for thermometer S/N 52 (Fig. 17). Because of time limitations, some sources of extraneous noise (pickup from the furnace current) were not eliminated and the data obtained from tests on S/N 33 show larger variations than do those from S/N 52. We do not believe these differences are attributable to the difference in materials used.

3.4 JOHNSON NOISE SIGNAL PROCESSING

In tests performed after termination of the sensor fabrication project, ORNL demonstrated that the dc resistance and the Johnson noise of the 4-wire sensor could be measured simultaneously by using the circuit shown in Fig. 18. The use of the tuned series-coupled resistor-inductor-capacitor (RLC) circuit to measure noise temperatures was also demonstrated. Tests were conducted by using sensor S/N 33 at temperatures up to 1400 K to confirm the capability of a sensor and signal processor system that does not require commutation of the low-level noise input signal during routine operation of the system.

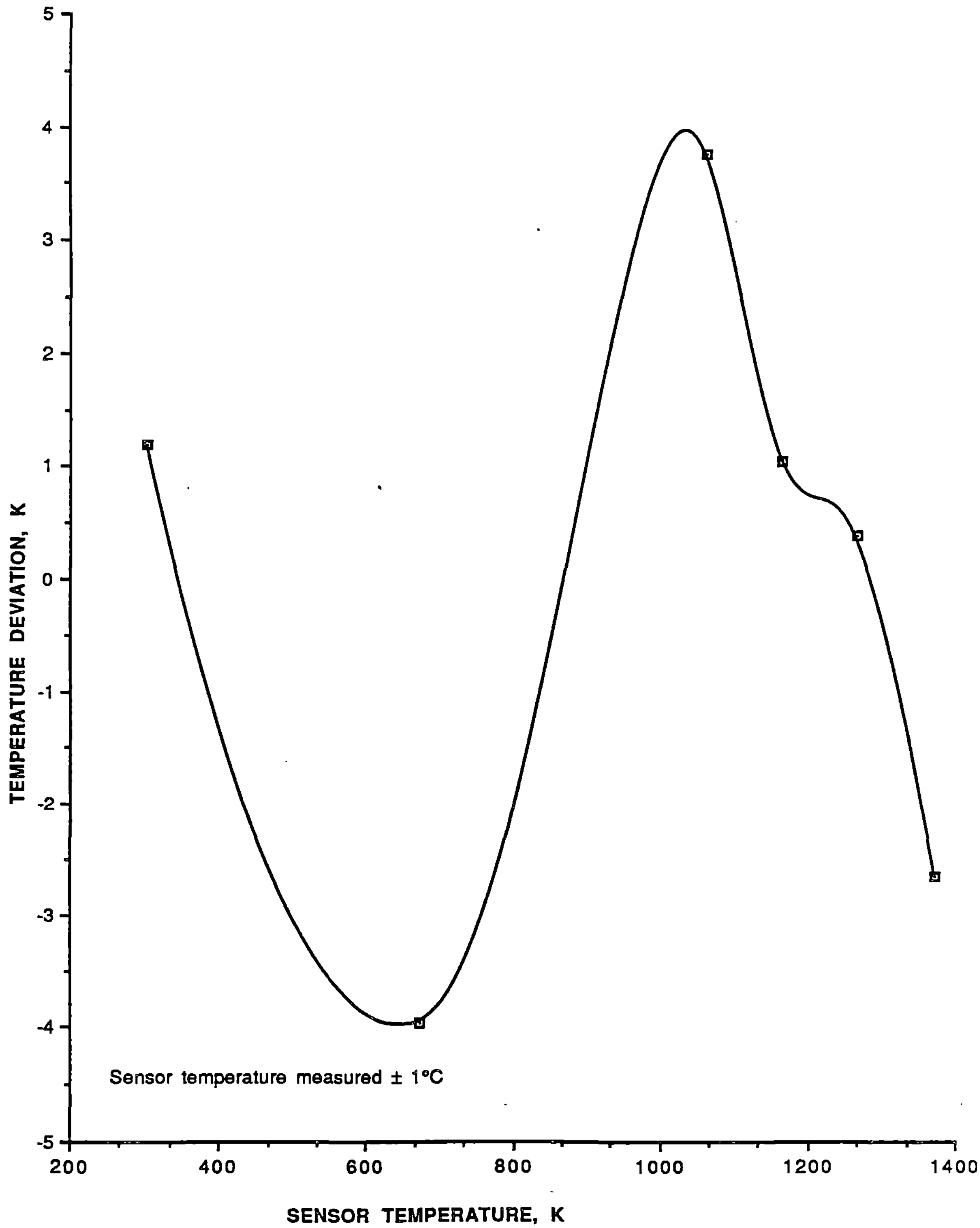


Fig. 16. Deviation of sensor S/N 52 fitted noise curve from the temperature measured by thermocouple.

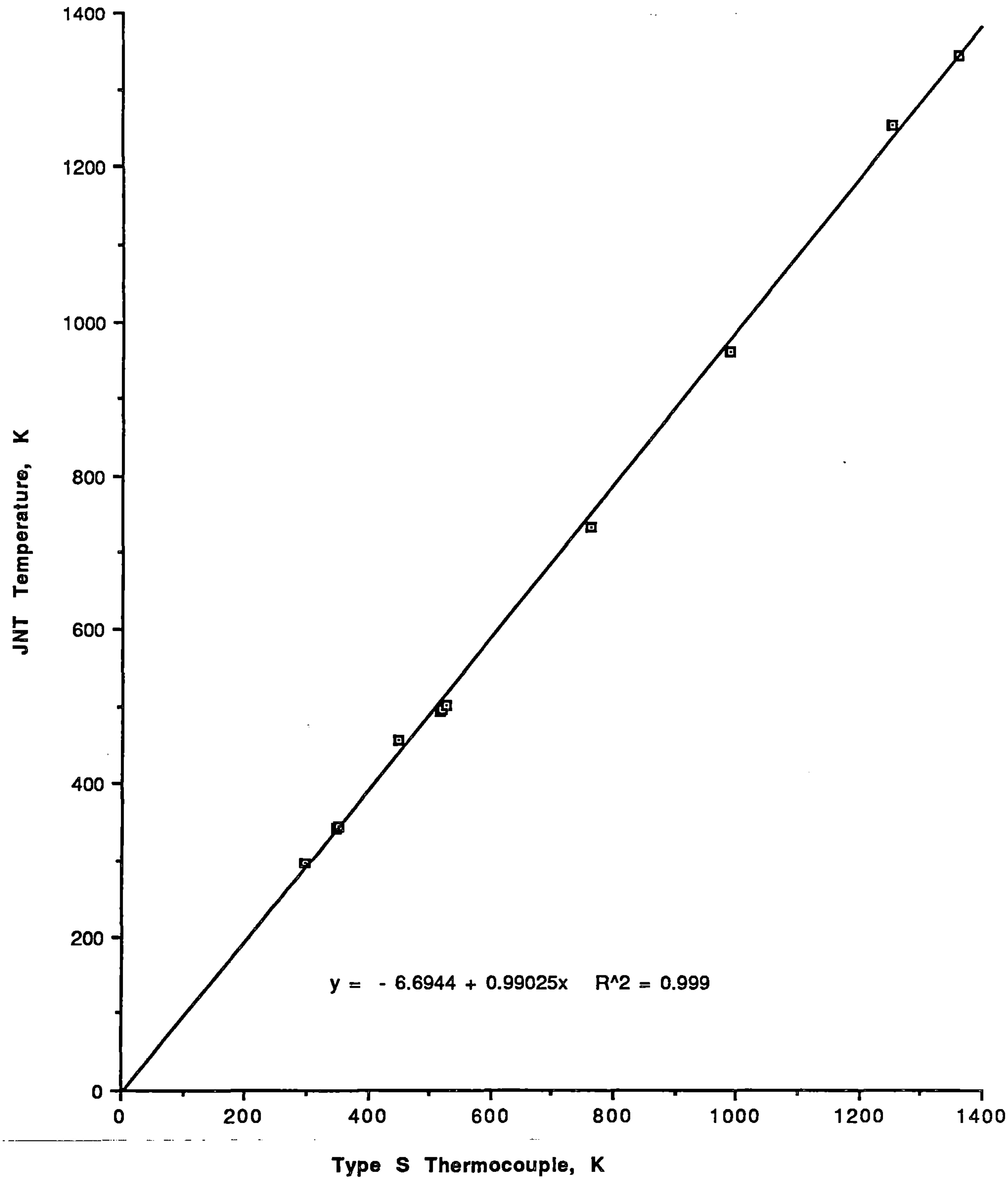


Fig. 17. Johnson noise temperature measurement compares with a thermocouple measurement for sensor S/N 33.

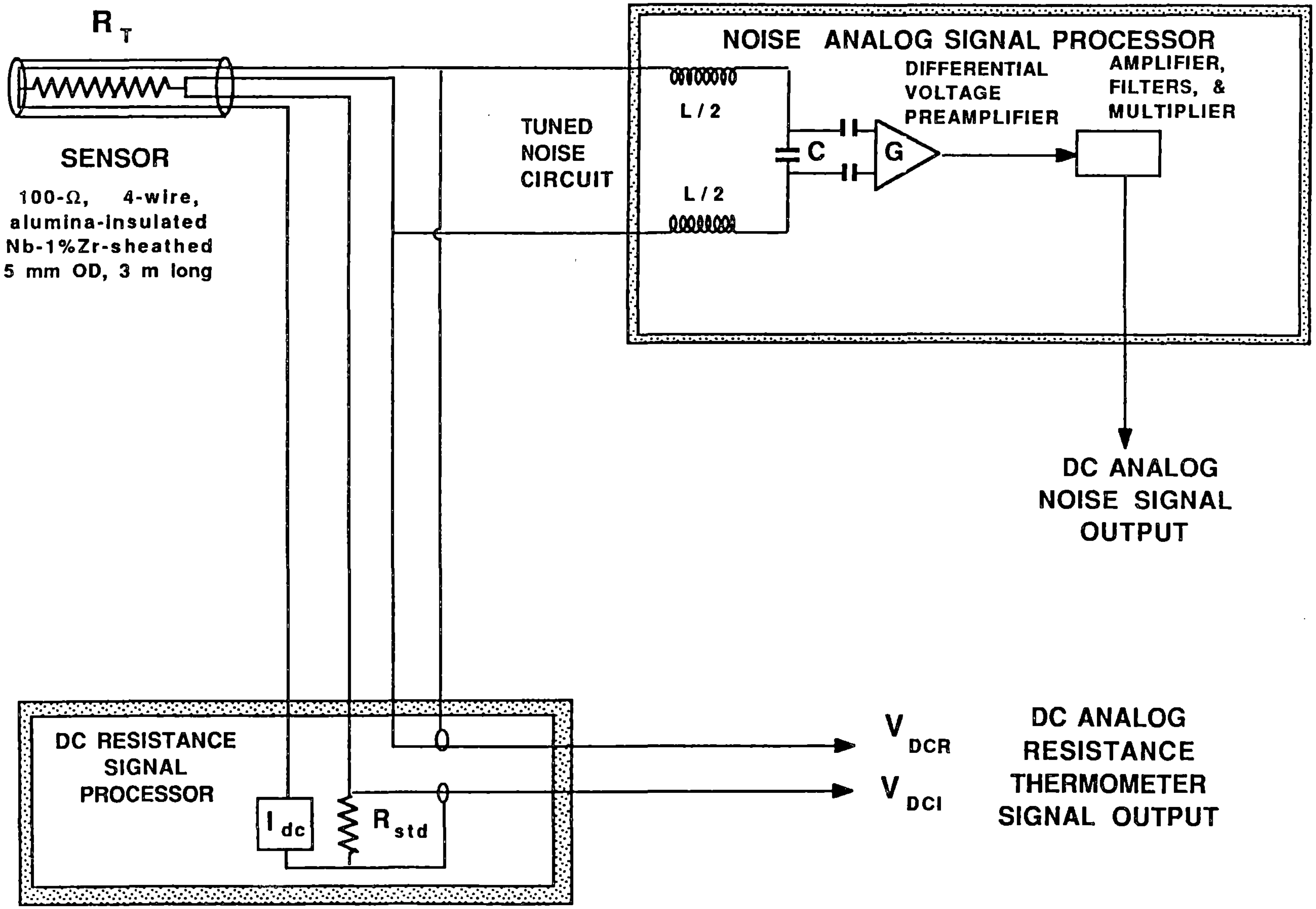


Fig. 18. SP-100 single-sensor combination Johnson noise measurement and dc resistance temperature detector.

4. CONCLUSIONS

The capsule experiments proved that the inert-gas–contained construction technique prevented the contamination of the thermometer components. The prototype thermometer with alumina insulation had a much higher insulation resistance than would be expected for a low-fired, crushable alumina preform; this result is attributed to keeping the preform free of moisture. The thermometer with yttria insulation showed a higher insulation resistance than expected, but the insulation resistance was still an order of magnitude less than that of alumina.

Johnson noise power measurements indicated a precise and absolute measurement of temperature for the alumina-insulated thermometer (S/N 52). The thermometer insulated with yttria (S/N 33) had a good linear relation of Johnson noise power with absolute temperature, but the thermometer data were more scattered than those of S/N 52. Both prototype sensing elements showed excellent potential for a high-temperature resistance thermometer in an inert atmosphere.

Because of the curtailment of the program, no compaction tests and thus no response-time measurements could be made on the prototype thermometers. Nor were tests made for long-term durability, the effects of microphonics on Johnson noise measurements, or for long-term high- temperature drift and durability.

APPENDICES

X.1. APPENDIX 1. SENSING ELEMENT—SPECIFICATIONS AND MATERIALS PROCUREMENT

X.1.1 FORM REQUIREMENTS

A resistor of about 100 Ω at 1375 K was desired for the Johnson noise measurements. Wire of 0.127 mm (0.005 in.) was selected as the smallest wire consistent with high durability. Since it could be used at 1375 K, Nichrome V had a desirably high electrical resistivity, but its limited temperature range indicated that it would not have long-term reliability (see Fig. X.1.1). The niobium and Nb-1%Zr alloy have the advantage of

1. being chemically compatible with the sheath material,
2. having a smooth function of resistivity vs temperature,
3. having a higher electrical resistivity than the other materials (except rhenium), and
4. being ductile and easily shaped.

Using 0.127-mm diam wire, a 4-m length of either the niobium or the Nb-1%Zr is necessary to obtain the desired 100-Ω resistance (see Fig. X.1.2).

The thermometer sheath was specified to have a diameter of no more than 5 mm (0.2 in.), and this specification determined the diameter of the other internal components.

To compact the insulation and still have the maximum allowed diameter, the starting diameter of the sheath became 5.8 mm (0.23 in.). The plan was to swage the sheath to 5.0-mm diam, barely crushing the insulation, and then, if necessary to prevent microphonics, the sheath would be swaged to smaller diameters producing additional compaction (see Fig. X.1.3).

The crushable ceramic preform can have a maximum diameter of 4.2 mm (0.167 ± 0.003 in.) and still fit within the sheath. The largest diameters of the holes in a four-hole preform of 4.2-mm diam is 0.86 mm (0.034 in.). These dimensions mean that the lead wire and the sensing element diameters can not be larger than 0.76 mm (0.030 in.) to fit within the holes in the preform (see Fig. X.1.4).

To fit the required 4-m length of wire within the smallest length of the sensor sheath, the wire is coiled into a helix of 0.74-mm (0.29-in.) diam and 0.43 m (17 in.) long. There are 3.94 turns/mm (100 turns/in.) with a 0.127-mm spacing between the turns. The plan was to snake the sensing coil through a insulator preform to make a sensing element just over 100 mm (4 in.) long (see Fig. X.1.5). After an exhaustive search, the RdF Corporation,[1] was the only manufacturer located that was able to form long coils of this diameter.

X.1.2 WIRE REQUIREMENTS

1. Niobium-1% Zirconium alloy for the wire, the same alloy as the thermometer sheath and the lithium containment tubes.

2. Grain size < 0.1 of the wire diameter.

3. Wire in the cold-worked condition, to minimize grain growth at the annealing temperature.

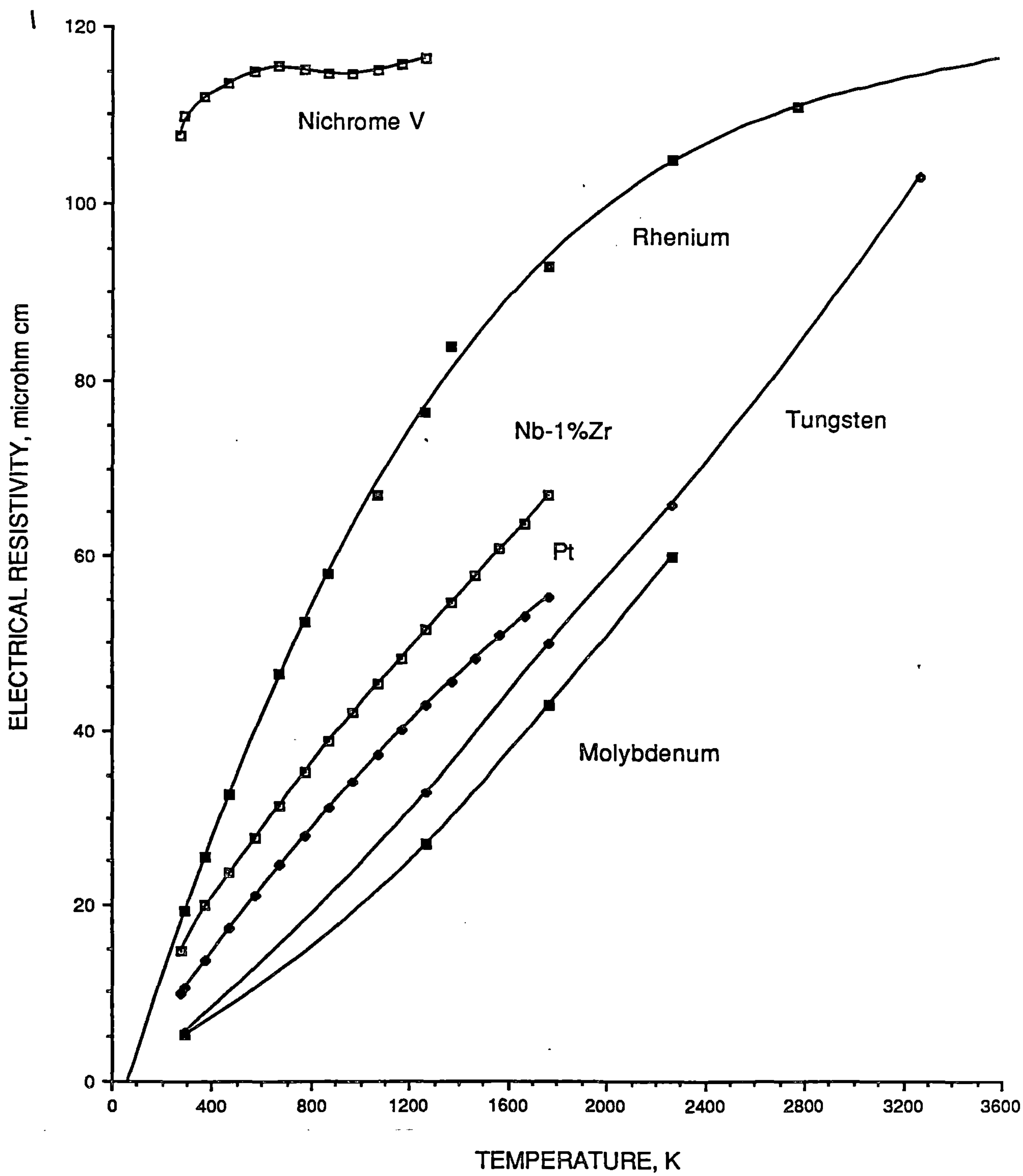


Fig. X.1.1. Electrical resistivity of refractory metals.

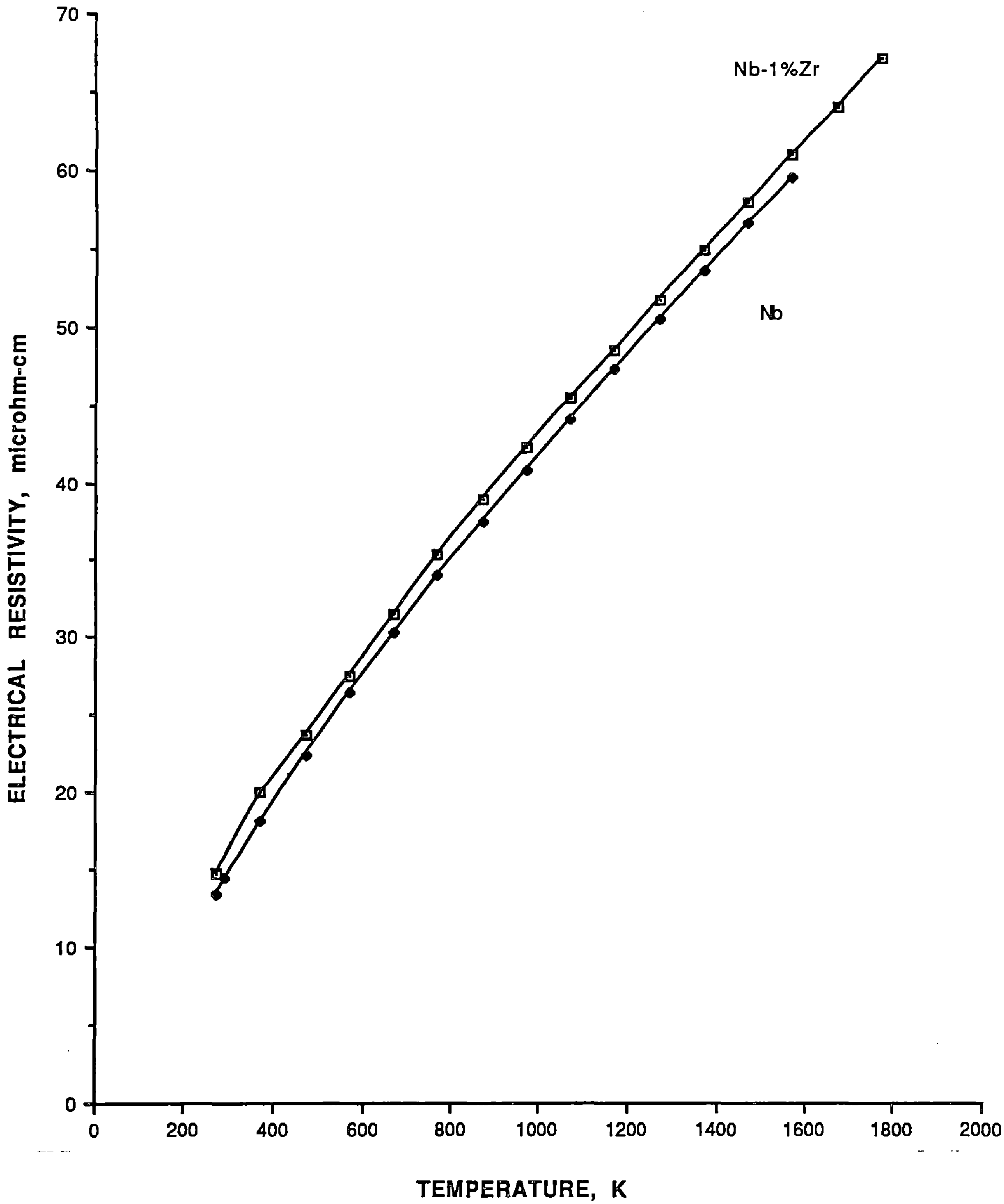


Fig. X.1.2. Niobium and Nb-1% Zr electrical resistance.

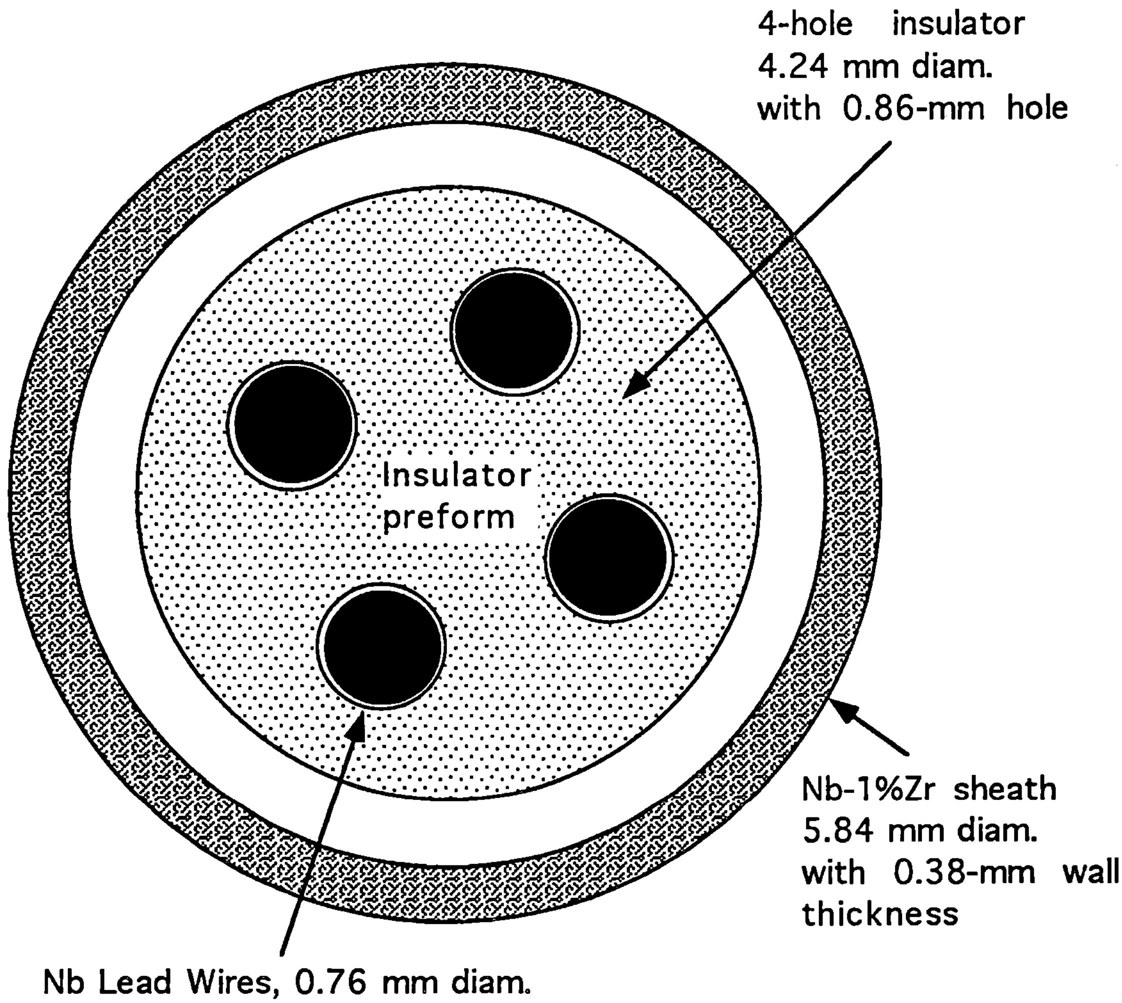


Fig. X.1.3. Thermometer cross section before swaging.

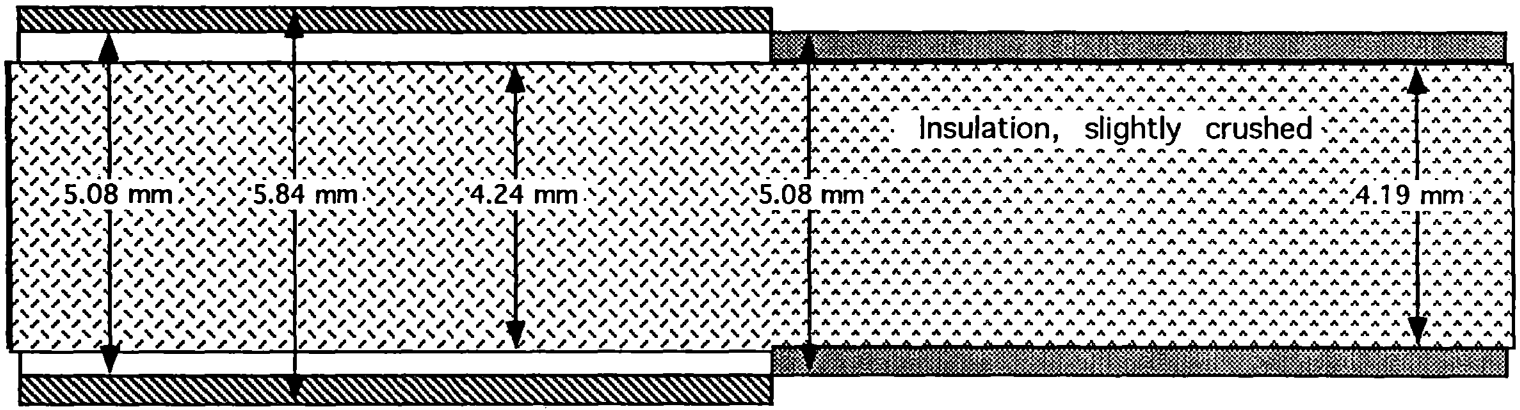


Fig. X.1.4. Cross section of tubing and insulation during swaging.

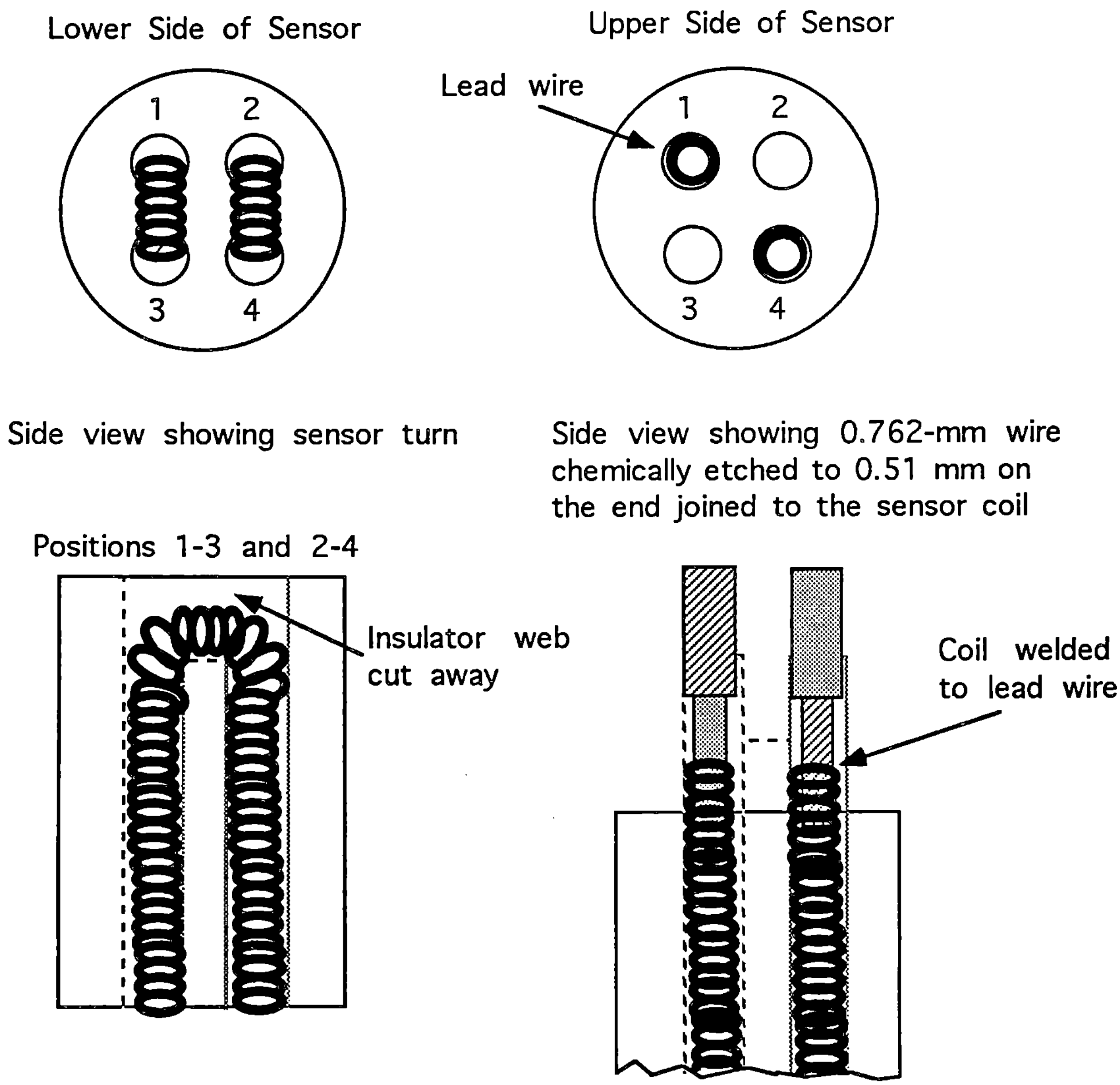


Fig. X.1.5. Diverse temperature-sensing element assembly.

Tests have shown that after a small amount of cold work such as winding the sensing coil was imposed, pure niobium wire exhibited extreme grain growth at the operating temperature of 1375 K. A test on 0.25-mm-diam (0.010-in.-diam) wire of Nb-1%Zr alloy did not show grain growth under the same type conditions that had produced grain growth in the pure niobium wire. Consequently, the sensing element wire material specification was changed to Nb-1%Zr. Cold-worked 0.13-mm-diam (0.005-in.-diam) wire for the sensor and 0.76-mm (0.030-in.) wire for the leads had to be procured.

X.1.3 WIRE PROCUREMENT

With the decision to use Nb-1%Zr wire (of the same specification as the sheath) for the primary sensor material, a source of 0.76-mm (0.030-in.) and 0.13-mm (0.005-in.) wire of certified composition had to be located. The Metals & Ceramics Division of Oak Ridge National Laboratory (ORNL) supplied SP-100 certified 1.52-mm (0.060-in.) wire (from SP-100 approved ORNL Mett lot 531029) from their stock for the source of the wire. A purchase order was given to California Fine Wire[2] to draw 0.76-mm and 0.13-mm wire from the supplied 1.52-mm wire.

The Nb-1%Zr wire had to be annealed in a vacuum furnace after drawing produced 100% cold work because subsequent diameter-reducing drawing would break the wire. Several sequences of drawing and vacuum annealing were required to reduce the wire from 1.52 mm (0.060 in.) to 0.13 mm (0.005 in.). California Fine Wire discovered that a special lubricant was needed for drawing fine-wire Nb-1%Zr. The final 0.13-mm product was cold-worked wire rather than fully annealed wire. A high degree of cold work is desired in the completed sensing element, so a fine-grain structure will result after the sensing element anneals during use.

X.1.4 RATIONALE FOR PROTOTYPE THERMOMETER

All of the components were on hand to construct the first prototype thermometer except for the 0.13-mm-diam (0.005 in.-diam) Nb-1%Zr wire for the sensing element. To avoid delay, the first prototype sensing elements were constructed from pure niobium wire. The reasons for using the niobium were:

1. Ultrapure fine-grain niobium wire of the correct 0.13-mm diam was immediately available from a superconductor program surplus.

2. Niobium is compatible with the same material as the Nb-1%Zr.

3. Niobium has a temperature coefficient of resistivity suitable for resistance thermometry that is not much different from Nb-1%Zr (see Fig. X.1.2).

4. Niobium has high resistance and good ductility compared to other refractory metals (see Fig. X.1.1).

The main problem with the use of pure niobium is that if it is slightly cold worked before use at annealing temperatures (~1100°C), there will be dramatic grain growth. The process of coiling the wire into the sensing element would produce the unwanted slight cold working. The grain growth was not considered to be a major problem for the first prototype, since the prototype was mainly intended to test the construction techniques and the instrumentation.

X.1.5 WIRE INSPECTION

1. Measure the grain size both transverse and longitudinal, using the method of ASTM E-112 (Ref. 3).

2. Inspect a length of the wire with a microscope to look for uneven diameter, cracks, gall marks, etc.

3. Measure the wire diameter to ±0.0025 mm (0.0001 in.).

4. The sensing element wire is 0.13-mm (0.005-in.) diam and is too small to be covered by ASTM B-392-84, "Standard Specification for Niobium and Niobium Alloy Bar, Rod and Wire,"[4] but chemical requirements shall apply.

X.1.6 SHIPPING AND STORAGE

The fragile coils (0.74-mm diam and 0.43 m long) were placed in 0.55-m (22-in.) lengths of 2-mm bore, heavy-wall capillary glass tubes closed on each end with plastic shrink tubing. Care must be taken to avoid crushing, kinking, or exerting excessive stress on the coiled wire. The protective glass tubes were used as shipping, storage, and cleaning containers.

X.2. APPENDIX 2. LEAD WIRE MATERIAL AND SIZE

The lead wire was either (1) niobium to match the niobium sensing element or (2) Nb-1%Zr if the sensing element was Nb-1%Zr. The lead wire should always be the same material as the sensing element to ensure good weld joints and to prevent a Seebeck voltage from the joining of dissimilar metals. For high-temperature operation, it is necessary to consider problems of mass transfer between the components of the thermometer; both niobium and the Nb-1%Zr are compatible with the Nb-1%Zr sheath.

The lead wire was selected to be the largest diameter (0.76 mm or 0.030 in.) that would allow insertion in the 0.864-mm-diam (0.034-in.-diam) bore holes of the crushable insulation (as shown previously in Fig. X.1.3). The main purpose of the large lead wire was to make the lead electrical resistance small compared to that of the sensing coil and reduce the influence of the leads on the Johnson noise measurements. A four-wire design was necessary for the resistance thermometer measurements, or the two leads on either end of the sensing coil could be paired in the Johnson noise measurements to minimize lead resistance.

X.2.1 LEAD WIRE INSPECTION

1. Measure the wire diameter on a 0.3-m (12-in.) sample cut from the end of the spool. Use a micrometer capable of measuring to 0.003 mm (0.0001 in.). Measure three locations, making two measurements at 90° angles at each location.

2. Record the mean and the variation from the mean diameter. If the diameter has a variation more than ± 0.03 mm (0.001 in.), cut samples from another segment of the spool and repeat the test.

3. Inspect the wire sample microscopically (50 ×) for galling, cracks, or other surface defects. If the surface has any visible cracks or any pitting or galling defects more than 1% of the wire diameter, cut samples from another segment of the spool and repeat the test.

4. Wrap a segment of the wire sample twice around a mandrel the same diameter as the wire. Inspect the outside of the wrapped wire microscopically (50 ×) for cracks. Section the wrapped wire, and do a metallographic examination of a cross section of the wire for evidence of cracking.

5. Cut about 50 mm (2 in.) from one end for metallographic grain-size measurement. Measure the grain size of the wire both radially and longitudinally according to ASTM E-112 (Ref. 3).

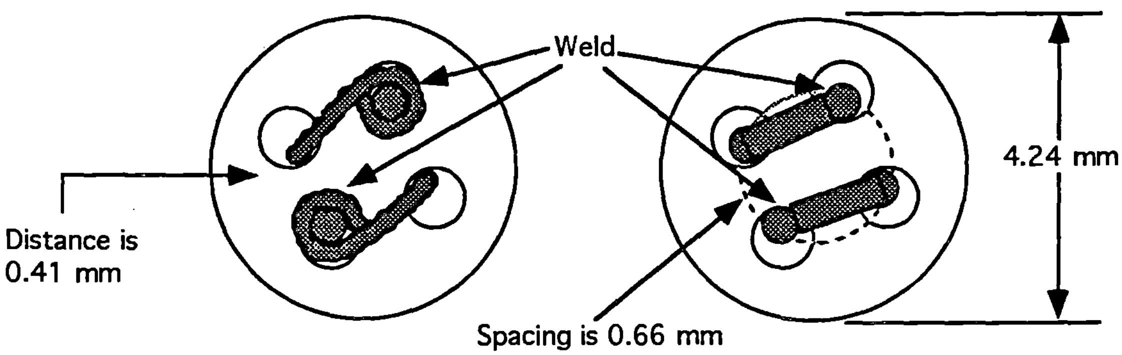
6. Use remainder of wire sample for chemical analysis. The 0.762-mm (0.030-in.) diam of the lead wire is large enough to conform to ASTM B-392-84 "Standard Specification for Niobium and Niobium Alloy Bar, Rod and Wire".[4]

X.2.2 CHEMICAL MILLING OF THE LEAD WIRE ENDS

The lead wire ends must be reduced in diameter to fit inside the sensing element coil and to make the four-wire twist junction (see Figs. X.1.5 and X.2.1). The reduction in diameter is done by chemical milling. Niobium and Nb-1%Zr are resistant to acid attack except hydrofluoric (HF) acid. Etch the lower 50 mm (2 in.) of two of the lead wires with a solution of 40% water, 40% hydrochloric acid (HCl), and 20% HF until the outside diameter (OD) of the wire is 0.46 ±0.03 mm (0.018 ±0.001 in.). Acid etch the lower 50 mm (2 in.) of the other two lead wires until the OD is 0.23 ±0.03 mm (0.009 ±0.001 in.).

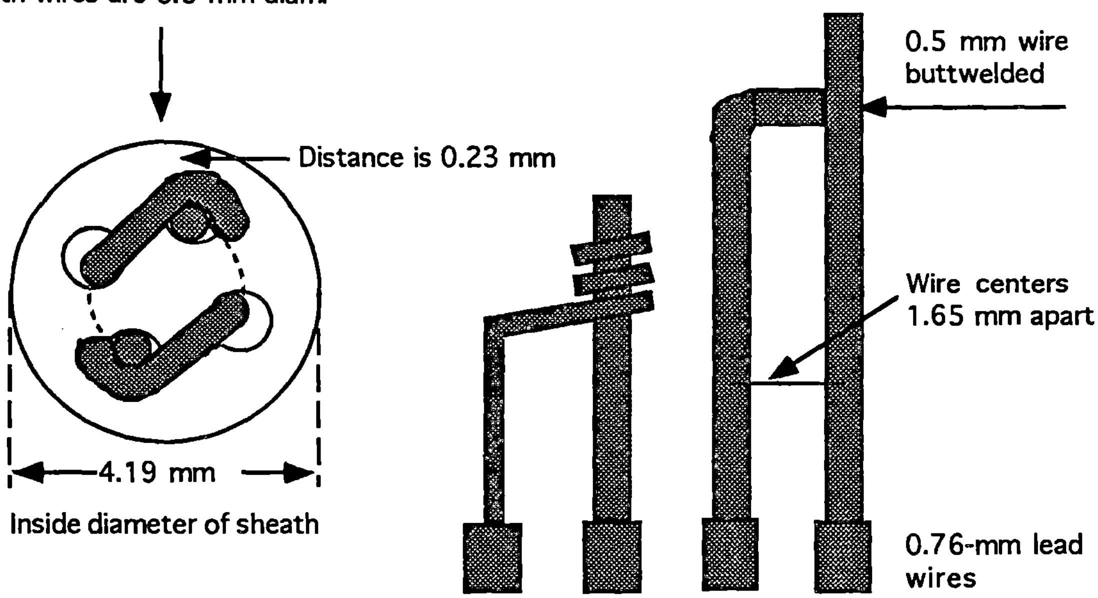
CAUTION: THE ETCHANT USED TO MILL THE NIOBIUM ALLOY IS EXTREMELY CORROSIVE AND DANGEROUS TO HANDLE. MINIMUM REQUIREMENTS INCLUDE OPERATIONS IN A FUME HOOD, GLOVES, AND FACE PROTECTION.

Ends of lead wire must be reduced to 0.5 mm to fit within sensing coil



This shows that the wire can be 360° wrapped around for strength only if one of the wires is reduced to 0.25 mm at the end.

Even a 90° wrap will bring the wire too close to the sheath if both wires are 0.5 mm diam.



Conclusion: the lead wires must be either butt welded, or one wire must be reduced to 0.25 mm at the joint. The wrap method is more reliable, so the end of one of the lead wires must be acid etched to 0.25 mm diam.

Fig. X.2.1. Comparison of spacing when wrapping and butt welding.

X.3. APPENDIX 3. SHEATH REQUIREMENTS AND HOT END CLOSURES

The sheath material was Nb-1%Zr tubing 3 m (10 ft) long, 5.84-mm (0.230-in.) diam with 0.38 mm (0.015-in.) wall thickness. The sheath tubes were drawn by Superior Tube Company,[5] using thick-walled tubing supplied by ORNL. The thick-walled tubing was already certified for SP-100 use.

The manufacturing standards for the sheath tubes were corresponded to ASTM B-394-86 (Ref. 6) and included:

1. Pneumatic test on sheath by vendor after manufacture as per ASTM B-394-86 (13.2.2).

2. Dimensional requirements are as per ASTM B-394-86, Table 4.

3. Tubing supplied as hard drawn rather than fully annealed.

4. Chemical and mechanical requirements of ASTM B-394-86 were waived since the sheath tubes were manufactured from stock already certified for SP-100 use.

X.3.1 INCOMING INSPECTION AT ORNL

1. Visual inspection of all tubes for warps, kinks, etc.

2. Measurement of outside diameter of selected tubes and measurement of inside diameter of tube segments used to make compatibility capsules. No 100% measurement of diameter is required at this point, since the tubing will be swaged to the final dimension.

3. No initial helium leak testing of the tubing, since it would be helium leak tested during construction.

X.3.2 HOT END CLOSURE (END PLUG) REQUIREMENTS

1. The hot end plug must be of the same material as the thermometer sheath. It must be capable of being welded to the sheath with a ductile weld so that the end plug and sheath together can be swaged to a smaller size without breaking the weld.

2. The end plug must have an identifying number and a fitting that will allow attachment to devices for handling during fabrication and attachment to the SP-100 piping.

3. The same composition and cleaning standards must apply to the hot end plug as to the sheath tubing.

X.3.3 HOT END CLOSURE (END PLUG) FABRICATION

The hot end plugs were fabricated at ORNL from SP-100 certified Nb-1%Zr, Heat 530126 (see Fig. X.3.1). A 0.89-mm-diam (0.035 in.-diam) hole is drilled through the end plug so that the thermocouple can be tied with wire to the pipe whose temperature it will measure. These plugs are to be laser welded to the thermometer sheath, using remotely controlled welding. The welding system rotates the sheath under the laser beam to melt the junction of the sheath and the welding lip. Therefore, it is very important that the plugs fit snugly within the sheath (a light press fit) and the welding lip fits squarely against the sheath end. The sheath end must also be square. The protrusions of the end plug are used to support the sheath on the one end and to attach to a handling tool on the outer end (see Fig. X.3.2).

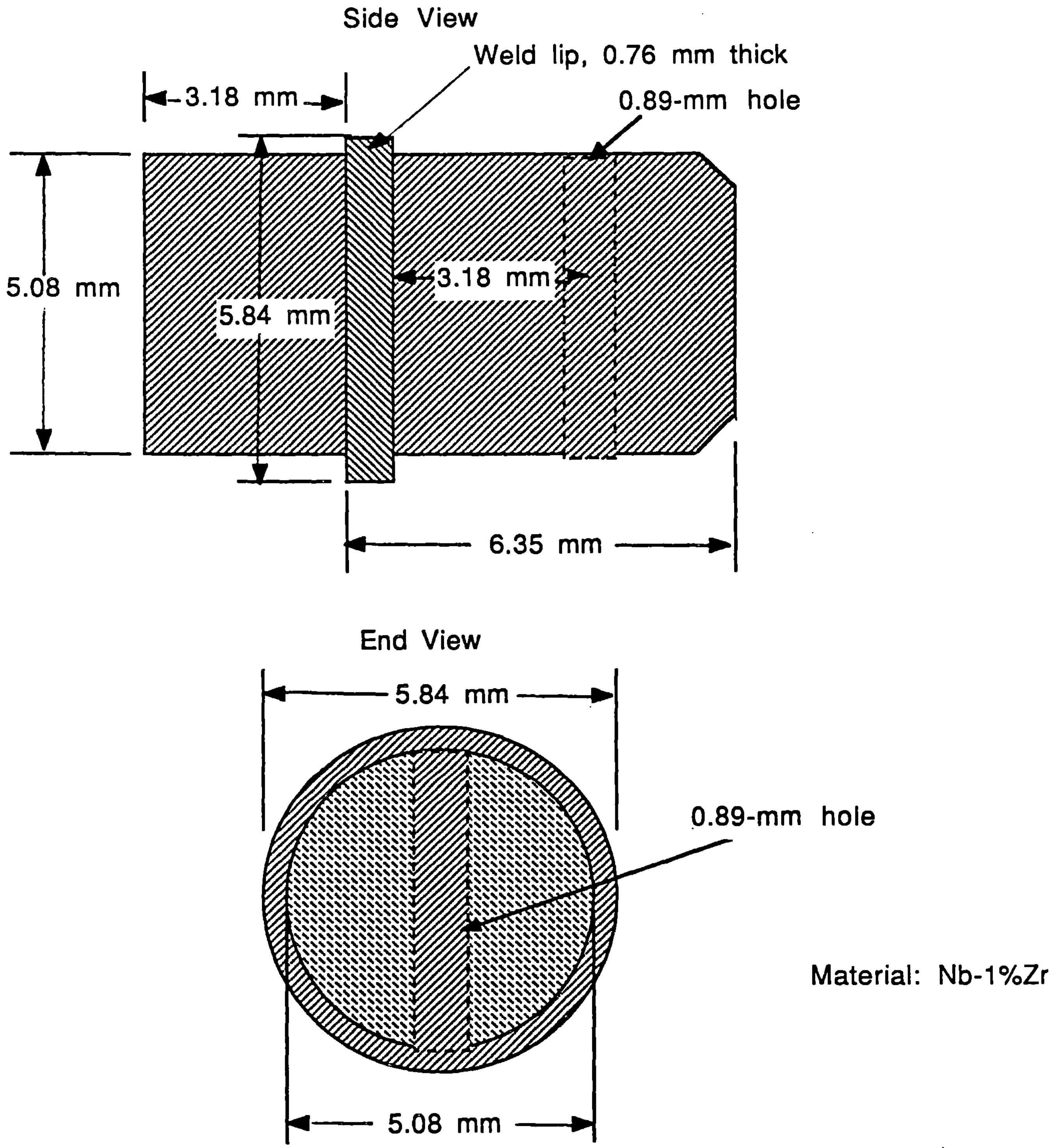


Fig. X.3.1. SP-100 end plug.

The hot end plugs have enough thickness that an identifying number can be engraved on the protruding end. Since the sensor sheath might be damaged by an engraved number, the number on the hot end plug welded into the sheath was used as a serial number for the thermometers.

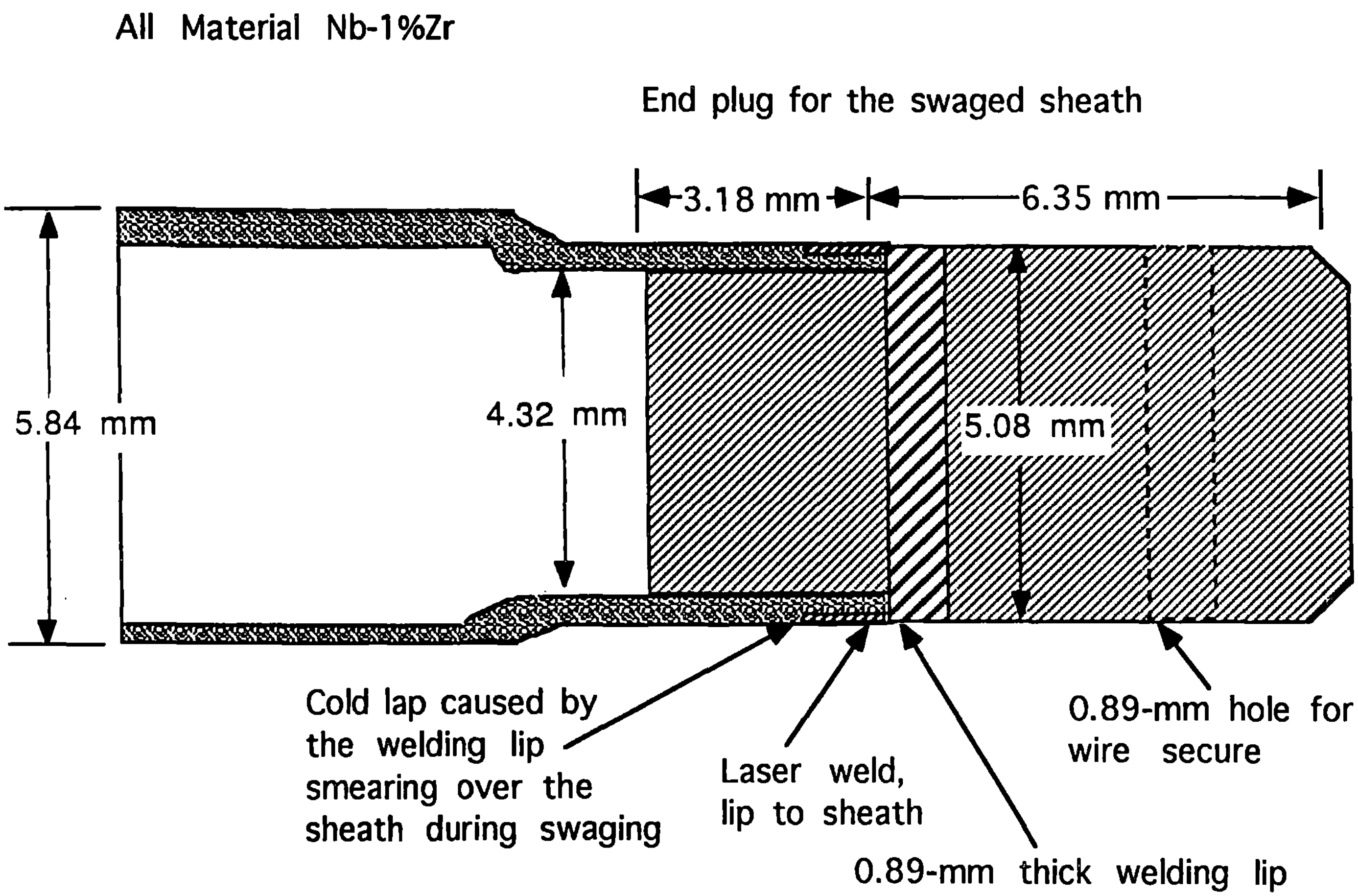


Fig. X.3.2. SP-100 End plug after sheath swaging.

X.4. APPENDIX 4. INSULATORS

X.4.1 INSULATOR REQUIREMENTS

The thermometer insulators must be high purity, crushable, 4-hole preforms that will withstand 1375 K (1100°C) for long times without loss of insulating properties or reacting chemically with the sensor or lead wires. The preforms must be made to precise dimensions and fired hard enough to allow handling, yet soft enough to crush without undue stress on the thermometer components. (see Fig. X.1.1). The material must be available commercially and be one that insulator producers have experience in handling. All of the preforms used in these experiments were fabricated by Ozark Technical Ceramics.[7]

Alumina, hafnia, and yttria were selected as the insulators best able to meet the requirements for the SP-100 thermometer. High-toxicity materials such as beryllia, or radioactive materials such as thoria, require very restrictive handling and can be used only as a last resort.

X.4.2 ANALYSIS OF ALUMINA

An analysis of the ultrafine-grain, ultrahigh-purity alumina used on prototype thermometer 52 was made to determine whether the fabrication of the preforms had degraded the purity of the supplied powder; all measurements are in parts per million.

Table X.4.1. Analysis of alumina

Element	Vendor analysis of alumina powder used to make the preforms	ORNL analysis of preforms used in compatibility capsules
Ca	13	5
Cr	2	2
Cu	<1	5
Fe	24	20
Ga	<4	<1
K		10
Li	<1	
Mg	2	20
Mn	1	5
Mo	<4	<2
Na	18	20
Ni	2	2
Si	28	80
Ti	1	2
Zn	2	5
Zr	<1	<1

Other elements were either not detected or calculated <5 ppm in the ORNL analysis.

Except for magnesium, the ORNL analysis by spark source mass spectrometry agrees with the vendor's analysis using inductively coupled argon plasma/atomic absorption, within the uncertainty limits.

X.4.3 ANALYSIS OF HAFNIA

The analysis was made of hafnia powder and of crushable preforms made from the powder. The ORNL analysis of the powder was compared to the Teledyne[8] analysis furnished to the vendor, Ozark Technical Ceramics, who made the crushable preforms.

The ORNL analysis was made by using a spark source mass spectrometer that has a precision of +100% and –50%, that is, you can double or half the given number and be within the precision of the machine. The analysis precision of the method used by Teledyne/Wah Chang Albany [8] is unknown.

All the values are in parts per million by weight.

X.4.4 ANALYSIS OF YTTRIA

Yttria insulator preforms were obtained from Ozark Technical Ceramics. The main questions were:

1. Is the starting material really of the purity claimed by the powder vendor?
2. Can the insulators be fabricated without degrading the purity?

To answer these questions, Ozark Technical Ceramics made up a crushable sample of test insulator and sent it along with the manufactured insulator made from the same source powder to ORNL for chemical analysis.

The test samples were submitted for spark source mass spectrometer analysis, and the analysis verified the ultrahigh purity required for the fabricated insulator.

X.4.5 APPLICABLE STANDARDS FOR INSULATOR PREFORMS

Chemical standards are ASTM E-235, paragraph 5.1.3.2 (Ref.9), for alumina; same levels of sulfur and carbon as for the hafnia. There are no accepted standards for crushability or density/porosity of soft-fired insulators. Preliminary tests were made to evaluate the amount of crushing and the density of the insulation when the sheath was swaged. The insulators were not outgassed or handled in an inert system for this test.

Sheath Swaging: The fabrication included swaging a 3 m-long Nb-1%Zr sheath containing hafnia- and alumina-insulated lead wires to the final 5-mm (0.20-in.) diam; the hafnia-insulated sheath elongated 2.3% in swaging, and the alumina-insulated sheath elongated 2.2%.

Crushed Insulation: Air flow tests made on the swaged sheaths (to get a concept of how tightly the insulation was packed) showed the hafnia was more tightly packed than the alumina. The hafnia in the sheath was 60% of the theoretical density (9.68 gm/cm^3), and the alumina in the sheath was 55% of the theoretical density (3.965 gm/cm^3).

Insulation Electrical Resistance: At 50 Vdc, the room temperature electrical resistance of the hafnia was a satisfactory $2 \times 10^{11}\ \Omega$, but the alumina was only $7 \times 10^{6}\ \Omega$. The low electrical resistance of the alumina insulation is attributed to the adsorption of moisture while the preforms were handled in air. When alumina was protected from moisture, the insulation values were much higher (as shown previously in Fig. 14).

Table X.4.2. Analysis of hafnia

Powder used to make preforms			Preform used in compatibility capsule
Element	Teledyne	ORNL	ORNL
Ag		23	17
Al	370	1600	1000
As		<88	120
B		2200	2300
Ba		56	66
Be		38	43
Ca		<150	<170
Cd		26	33
Co		25	31
Cr		<7	<8
Cu		320	520
Fe	<200	280	180
Ga		<440	<500
Li		<300	<340
Mg		3000	3100
Mn		49	56
Mo		390	260
Na		<4400	<5000
Ni		250	290
P		<440	<500
Pb		<74	<84
Sb		<250	<230
Se		170	170
Si	<200	<200	<200
Sn		580	710
SO_4		1200	
Sr		<7	<8
Ti	<50	68	140
V		<6	<7
U	<50		
Zn		39	37
Zr	12,000	9,700	13,000

Table X.4.3. Analysis of yttria

Element	ORNL analysis of yttria powder used to make the preforms (ppm)	ORNL analysis of insulator made from yttria powder (wt ppm)
Al	1	<1
B	<1	<1
Ca	1	<1
Cl	1	1
Co	<1	<1
Cr	<1	<1
Cu	<1	<1
Fe	1	<1
K	1	<1
Mg	1	1
Mn	<1	<1
Mo	<1	<1
Na	3	3
Ni	<1	<1
S	1	1
Si	1	1
Ti	<1	<1
Zn	1	<1
Zr	<1	<1

(Each of the rare earths <1 ppm)

X.4.6 INSULATOR SIZE SPECIFICATIONS

Ultrahigh-purity, ultrafine-grain alumina, 4-hole insulators 108 mm (4.25 in.) long, 0.84-mm (0.034-in.) hole, and 4.2-mm (0.167-in.) diam were ordered from Ozark Technical Ceramics. Hafnia and yttria insulators of the same dimensions were also obtained from Ozark Technical Ceramics.

X.5. APPENDIX 5. THERMOMETER ASSEMBLY EQUIPMENT

To handle the thermometer components without compromising their cleanliness, and especially to weld and assemble the thermometer in an inert atmosphere, special equipment and jigs had to be constructed and used.

X.5.1 INERT GAS CONTAINMENT

The cover gas was contained in a primary and secondary system. The primary system enclosed a very high purity argon or helium atmosphere. The primary cover gas was contained in quartz or glass containers. For primary containment, the system was evacuated to high vacuum and filled with high-purity argon or helium. This process was repeated at least five times. With primary containment, the components could be welded or operated at high temperatures.

The secondary containers were plastic, 0.32-mm-thick (0.0125-in.-thick) PVC for the inflatable glove box and furnace shrouds and polypropylene sealable jars for the insulator preforms. The secondary containers were evacuated to a low vacuum and then back-filled with argon. This process was repeated at least three times. Only room temperature operations were conducted in secondary containment.

X.5.2 PRIMARY INERT GAS CONTAINMENT

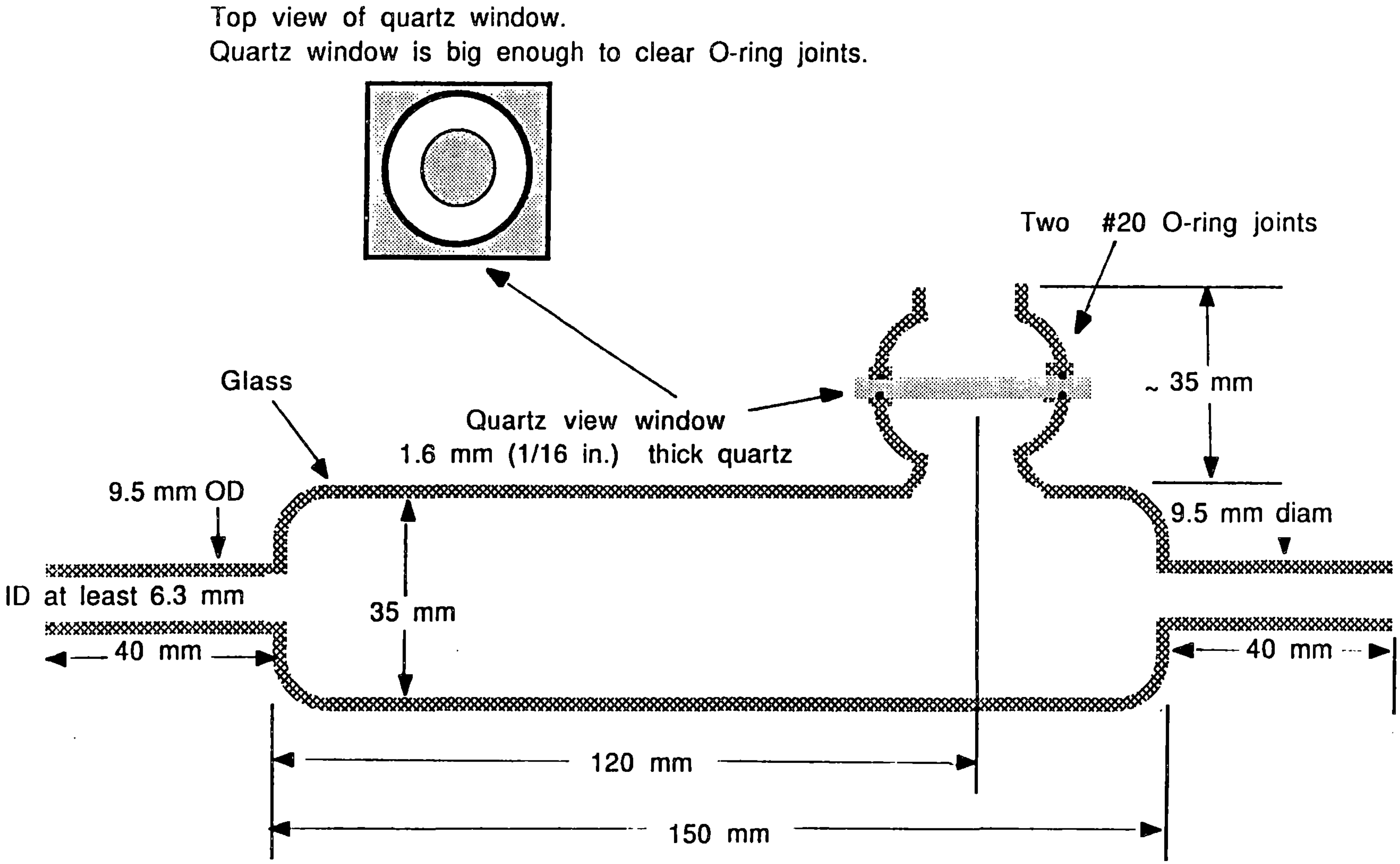
The usual primary container was a 3.2-m-long quartz tube, 8-mm (0.31-in.) ID (as shown previously in Fig. 2) that could contain the 3-m-long prototype thermometer. The quartz tube was fused shut on one end and reduced on the other end to accept a "3/8-in." teflon and neoprene compression seal fitting that would allow the quartz tube to be sealed gas tight. The quartz tube was both fragile and expensive, but it allowed the thermometer to be placed directly into 1375-K temperatures without removing it from the primary container. Also, laser welding could be done directly through the quartz tube but not through glass or metal.

The quartz tube required careful handling. The tube must be supported over its entire length, and mechanical shocks have to be avoided. It is especially important that the tube not be tilted to prevent the 3-m-long thermometer from sliding to the end of the 3.2-m tube and breaking out the bottom. A segment of sheath material was inserted in the quartz tube so the thermometer plus the sheath segment filled the length to prevent sliding.

For transport, the quartz tube is shock mounted in segments of thick-walled gum rubber tubing positioned about every 150 mm (6 in.) along the length of the quartz tube. The gum rubber tubing has an inside diameter slightly smaller than the quartz tube so segments of the tubing can be cut with scissors and slit so the segments of gum rubber tubing can be easily snapped on and off the quartz tube. The shock-mounted quartz tube is then inserted into a PVC heavy-wall pipe that contains plastic foam packing on each end to inhibit longitudinal movement. The PVC pipe, capped on both ends, still has some flexibility, so a heavy-gauge aluminum angle is used to transport the PVC pipe containing the quartz tube. This has been a successful system; the quartz tubing was never broken during transport.

The primary containment system formed from glass is used for the hot end plug welding to the thermometer sheath. A removable quartz window is included to allow welding with the high-power laser beam; when the window became damaged from the beam, it was replaced (see Figs. X.5.1 and X.5.2).

In all cases, the primary containment system is connected to an evacuation, inert gas supply, system for furnace experiments and welding. The vacuum system is used only during the establishment of an inert atmosphere; a positive pressure of inert gas is maintained during high-temperature operation or welding (see Fig X.5.3). The primary containment system was also used for full-length helium leak tests for the sheath and hot end closure weld (see Fig X.5.4).



Dimensions approximate except for 9.5-mm (3/8 in.) connection ends

Fig. X.5.1. Inert atmosphere jig for laser welding SP-100 end plug.

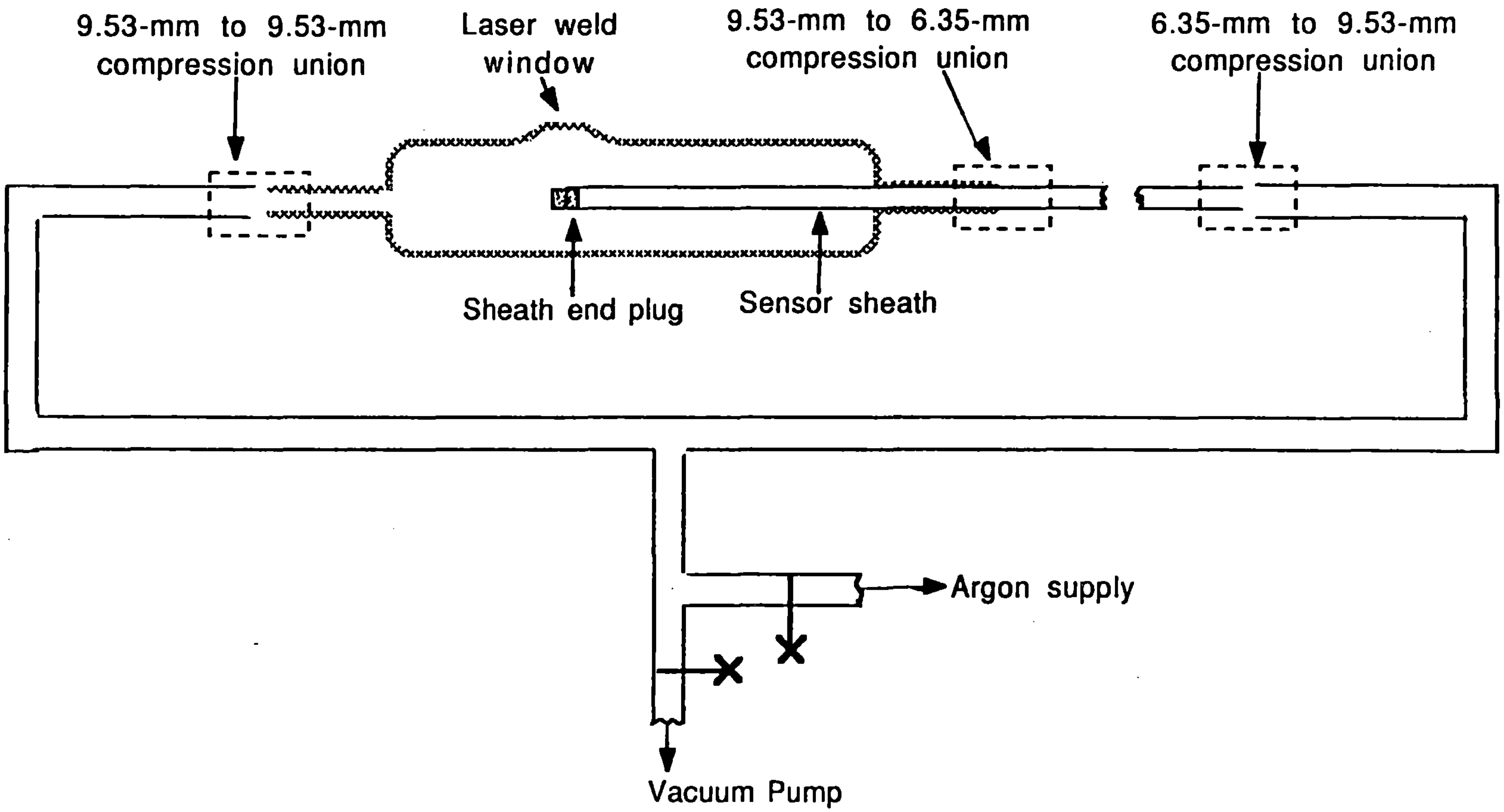


Fig. X.5.2. Jig for end plug laser weld: gas and vacuum connection arrangement.

X.5.2.1 Polypropylene Sealable Jars

The jars are used to hold insulators in an argon atmosphere after the preforms are outgassed with a high-temperature vacuum bake. The jars are first filled with argon before the insulators are put in the jar. To fill the jars with argon, the jars and lids were put into a desiccator with a top lid. The desiccator was evacuated and back-filled with argon several times. The desiccator was filled with a positive pressure of argon, the top was removed, and reaching down into the argon-filled desiccator, the lids were sealed to the polypropylene jars.

If the insulators are outgassed in a vacuum furnace with a top lid, a shroud is not needed. The cool furnace is put under a positive pressure of argon, and the top lid is opened. Then, the argon-filled jars are placed in the argon cover, the lid is opened, the niobium foil-wrapped insulators are placed in the jars, and the jars are resealed. This operation can be done very rapidly.

CAUTION! ARGON DISPLACES AIR, AVOID BREATHING ARGON.

X.5.2.2 Inert Gas Storage

The storage container for the long thermometer components is a 3.2-m-long quartz tube, 8-mm (0.31-in.) ID (as shown previously in Fig. 2), that can contain the 3-m-long prototype thermometer. The quartz tube is fused shut on one end and reduced on the other end to accept a "3/8-in." teflon and neoprene compression seal fitting that allows the quartz tube to be sealed gas tight. When the thermometer assembly has to be removed from the glove box (e.g., to have the welding done), the thermometer assembly is sealed into the quartz tube under an argon atmosphere. Even if the thermometer assembly is in the glove box but not being worked on, it is sealed into an argon-filled quartz tube for protection.

The storage containers for the insulators are argon-filled plastic jars. These sealed jars are not opened except in an argon atmosphere. The needed insulators are removed, and the jar is resealed at once.

The sensing element coils are not stored under inert gas, since room temperature reactions with the atmosphere are not a problem. To protect the coils from physical damage and to maintain their cleanliness, the coils are kept sealed in their glass protective tubes until they are ready to be placed in their insulator prêforms. To keep the preforms from contacting air, the sensing coil protective tubes are opened only in the argon-filled glove box.

X.5.3 SECONDARY INERT GAS CONTAINMENT

The secondary containers were plastic, 0.32-mm-thick (0.0125-in.-thick) PVC for the inflatable glove box and furnace shrouds.

X.5.3.1 Furnace Shrouds

Each vacuum furnace had to have a shroud made to fit its size and door configuration. We were not permitted to modify the valuable furnace, and no operation was allowed that might compromise the purity level of the furnace.

One type of furnace shroud, equipped with a surgical glove port, was used to install epoxy seals on a thermometer assembly while it was being contained under a helium atmosphere and after a vacuum bake-out. The shroud, containing epoxy and tools, was fitted around the end of a horizontal vacuum furnace, and the open edges of the shroud were taped to the cold furnace outer surface to form a seal.

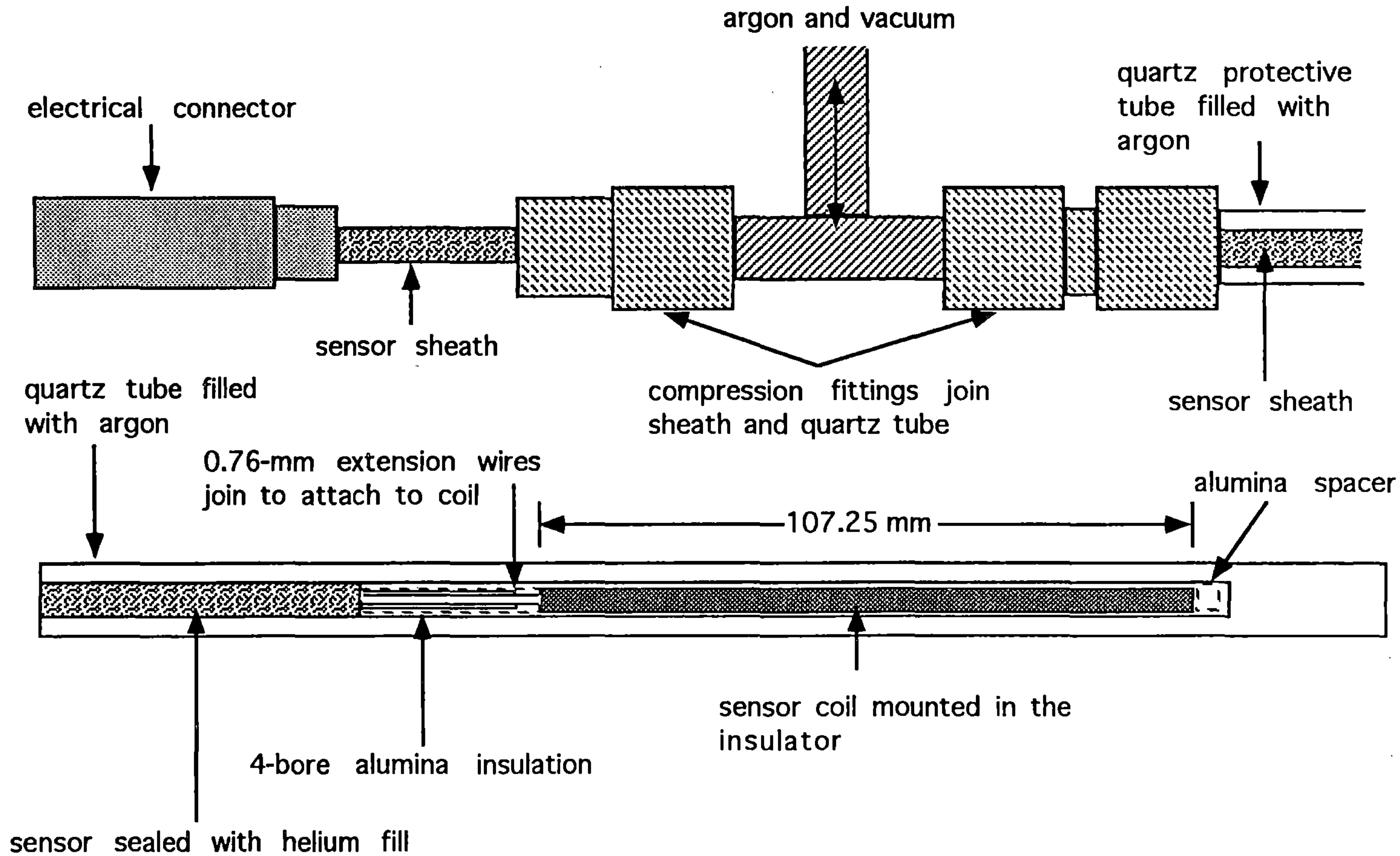


Fig. X.5.3. System for protective atmosphere for furnace tests.

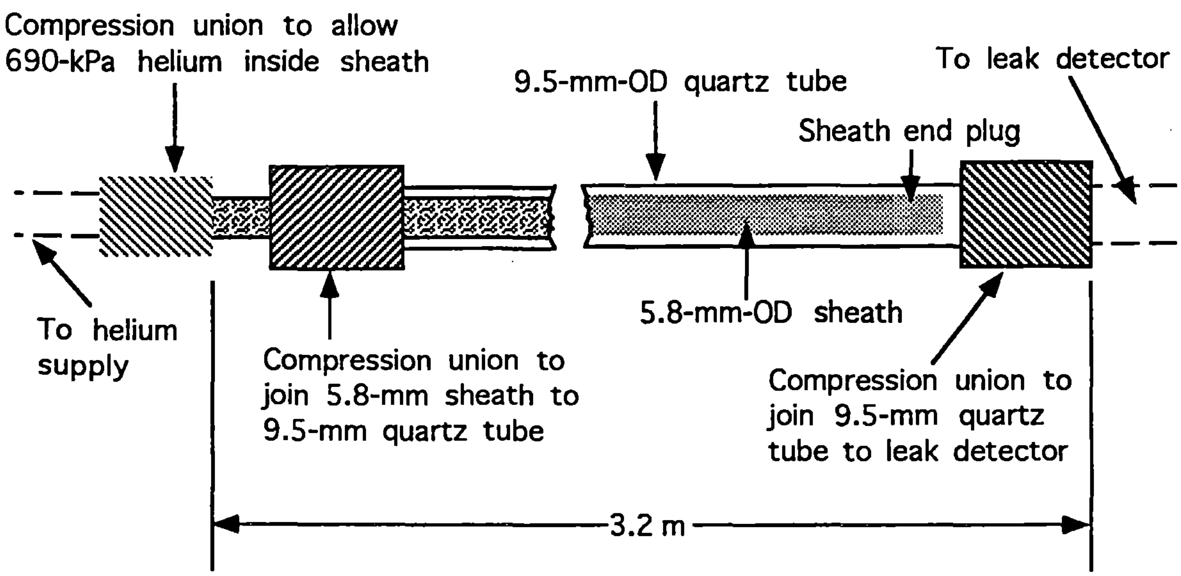


Fig. X.5.4. Arrangement for sheath helium leak test.

The furnace was placed under a positive pressure of helium, and with the use of the gloves in the shroud, the furnace door was opened slightly to allow helium to escape and fill the shroud, venting through the vent tube of the shroud (see Fig. X.5.5). The shroud was allowed to purge for several minutes, and then the vent was throttled to place a slight positive pressure on the shroud. With the use of the gloves, the epoxy seal was placed on the cold end of the thermometer while the cold end of the thermometer was just outside the furnace and the rest of the thermometer was still in the furnace.

Another type of shroud was used to remove insulator preforms that were outgassed in a horizontal vacuum furnace. The insulators were wrapped in niobium foil and tied with niobium wire for the bake-out. The insulators were packaged so they would fit within a widemouth polypropolene sealable jar. The wire wrap on the package was arranged so it could be hooked to remove the package from the furnace.

After the vacuum outgas procedure was performed and the furnace cooled, the furnace was filled with a positive pressure of argon. The shroud, containing sealed jars filled with argon and a long wire hook was taped to the furnace (see Fig. X.5.6). The large end of the shroud was taped to the furnace, and the operator, wearing surgical gloves, placed one hand through the 75-mm (3-in.) end of the shroud and taped the shroud around his arm. The shroud was purged with argon, the door of the furnace was opened, the foil-wrapped packages were hooked and dragged into the shroud filled with a positive pressure of argon. With one hand grasping through the flexible plastic along with the other hand inside the shroud, the jar lids were removed, the packaged insulators were placed in the jars, and the jars were resealed.

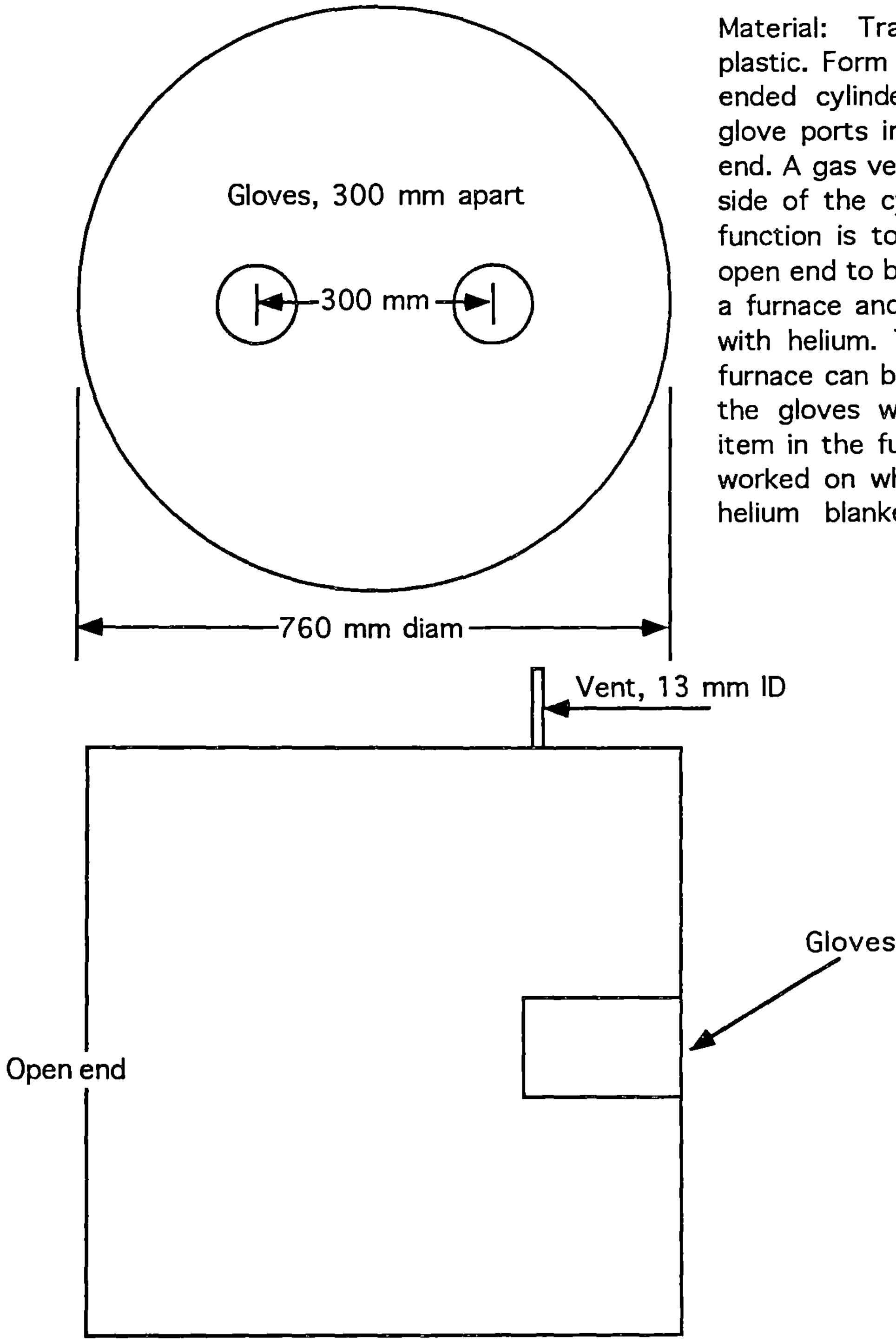
X.5.3.2 Plastic Film Glove Box

A glove box is necessary to provide an assembly area with an inert atmosphere. Conventional glove boxes are not long enough to allow a 3-m-long sensor to be assembled unless special extensions are fitted to the entrance ports. To allow 3-m-long components to be placed in a 3-m-long sheath, a 6-m-long glove box is required. An additional requirement is to be able to see the material within the glove box and to be able to manipulate the components. This was accomplished by constructing a secondary cover system consisting of an inflatable 10-m-long (32-ft-long) glove box of clear, 0.32-mm-thick (0.0125-in.-thick) PVC plastic fitted with surgical gloves, as shown previously in Fig. 3. The working section is 0.43-m-diam (17-in.-diam), 0.6-m-long (24-in.-long) tube with two sleeved openings that accommodate surgical gloves. Five openings of 13-mm (0.5-in.) diam will allow vacuum, gas, electrical, and instrument line access (as shown previously in Fig. 4).

Plastic access tubes 0.2 m (8 in.) in diameter and 4.6 m (15 ft) in length are joined to each end of the working section (as shown previously in Fig. 5). The access tubes have openings for vacuum and gas fill. Entrance to the inert gas-filled working section is obtained by first clamping the access tube closed near the working section and then opening the end of the access tube and inserting the item, moving it along the tube by grasping through the flexible plastic.

After the item is inserted in the access tube, clamp shut the open end of the access tube and evacuate the access tube. The plastic tube will simply collapse around the item, so the item must be sturdy enough to withstand the pressure or else be protected. The evacuated access tube is then be filled with argon. The evacuation and filling procedure should be repeated several times to ensure that the access tube gas is at an acceptable level of purity before unclamping the section between the access tube and the working section.

Plexiglas and glass plate strips are inserted into the glove box through the access tubes to provide a working surface and to allow the jigs, quartz containment tubes, sealed bottles, etc., to slide easily into and out of the working area.



Material: Transparent plastic. Form is an open-ended cylinder with glove ports in the closed end. A gas vent is on one side of the cylinder. The function is to allow the open end to be taped around a furnace and the bag filled with helium. The cold furnace can be opened, and the gloves will allow an item in the furnace to be worked on while under a helium blanket.

Fig. X.5.5. Plastic bag connection to outgas furnace, end view.

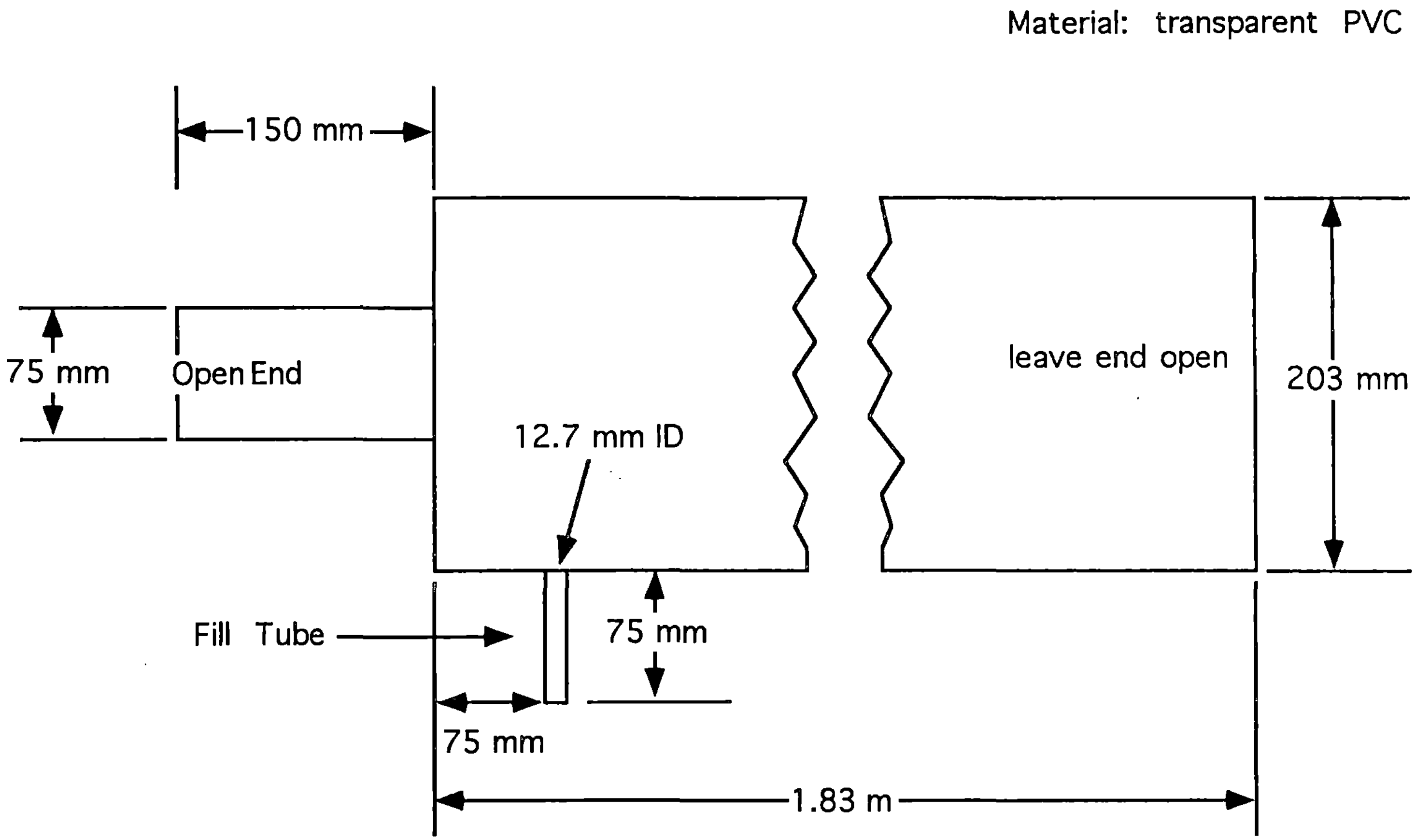


Fig. X.5.6. Insulator outgas furnace bag for horizontal furnace.

X.5.4 SENSOR HANDLING JIGS

Most of the equipment and tools used in assembling the thermometer are readily available. It is necessary only to ensure that the tools are free of grease or dirt. There are some tools that must be constructed, and these are jigs to hold the thermometer components in alignment while the thermometer is being assembled in an argon atmosphere.

One necessary item is a angle bed-plate that can align components. The angle must be long enough to allow the thermometer sheath to align with the insulated lead wires as the sensing element and the lead wires are being inserted into the sheath; for a 3-m-long thermometer, the angle must be at least 6 m long.

The assembly work is done in the working section of the glove box with the attached gloves (as shown previously in Fig. 3), so the work done on the angle, or the angle itself, must slide into and out of the extension tubes of the glove box (as shown previously in Fig. 5). Plexiglas or glass plate strips in the bottom of the glove box provide a sliding work surface. An aluminum angle is used in this work because of its lightness. The interior of the angle is covered with plastic tape, so the thermometer components never touch aluminum and the plastic surface allows components to slide easily in the angle. The aluminum angle jig is illustrated in Fig. X.5.7.

Another necessary item is a device to hold the four lead wires in alignment while the four-hole preforms are being threaded onto the wire inside the glove box. The wires are rather soft and are a tight fit within the insulator preform holes. The tight fit means that, as the preforms are being strung on the wires, the axis of the preforms must have an exact alignment with the wires. The preforms are pushed onto the wires, and all the wires must pass into the preform at the same rate. Otherwise, one of the wires might kink and form a lump that would require a new set of lead wires.

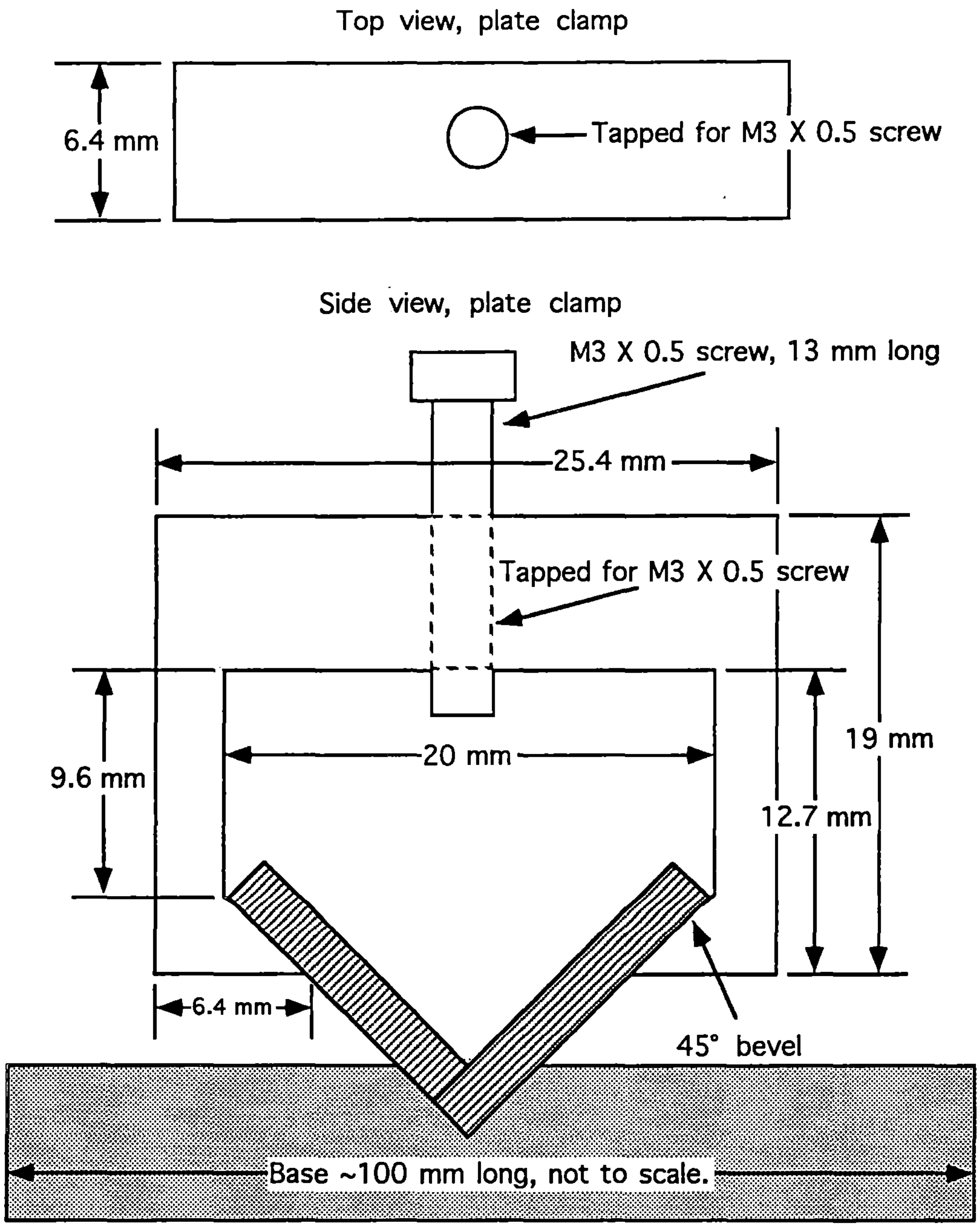
To avoid kinking, the wires must be held in tension and in alignment with the preform holes while the insulators are installed. A plate clamp can be attached to the aluminum angle (as shown in Fig. X.5.7) to hold a wire clamp block to the aluminum angle (Fig. X.5.8). The clamps can be placed on, or removed from, the lead wires by removing the plate clamp and loosening the tension screws. The tension screws keep the wires locked in alignment while the plate clamp locks the aligned wire to the aluminum angle.

Tension in the wires is obtained by sliding the plate clamp along the aluminum angle until the wires are taunt and uniformly tight and then locking the plate clamp in place. There are four clamp assemblies which are removed and replaced one at a time to allow the insulators to slide along the wires as the insulator are being installed. Details of the clamp block are shown in Figs. X.5.9A and X.5.9B.

The sensing element, installed in the preform (as shown in Fig. X.1.5), must be attached to the lead wires, and the junction of lead wires and sensing element must be welded before the insulation can finally be snugged into place, covering the connecting junction. The laser welding equipment and glove box are in different buildings, so the insulated lead wires, attached in a slip fit to the sensing element, must be transported in an argon atmosphere to the welder. A special jig is necessary to align, support, and transport the insulated wires and sensing element.

The jig is made by cutting 25-mm segments from a 3-m length of Nb-1%Zr sheath tubing as shown in Fig. X.5.10. The cutting of the soft, thin-walled tubing could not distort the tubing diameter or leave burrs, so the segments of the tube were cut with an electric discharge machine (EDM). Slivers of the segments removed from the sheath were retained to use as shims to lock the insulation within the jig while it was being transported.

Other tools that must be used in the assembly include a wire wrap tool to wrap the etched end 0.23-mm (0.009-in.) wire around the etched end 0.46-mm (0.018-in.) wire to form a two-wire connection to the sensor element (detailed in Appendix X.7).



Aluminum base for aluminum angle, 6 mm thick, ~ 25 mm wide, and 100 mm long. Base glued to angle every 0.3 m of length.

Fig. X.5.7. Aluminum angle with wire jig clamp block.

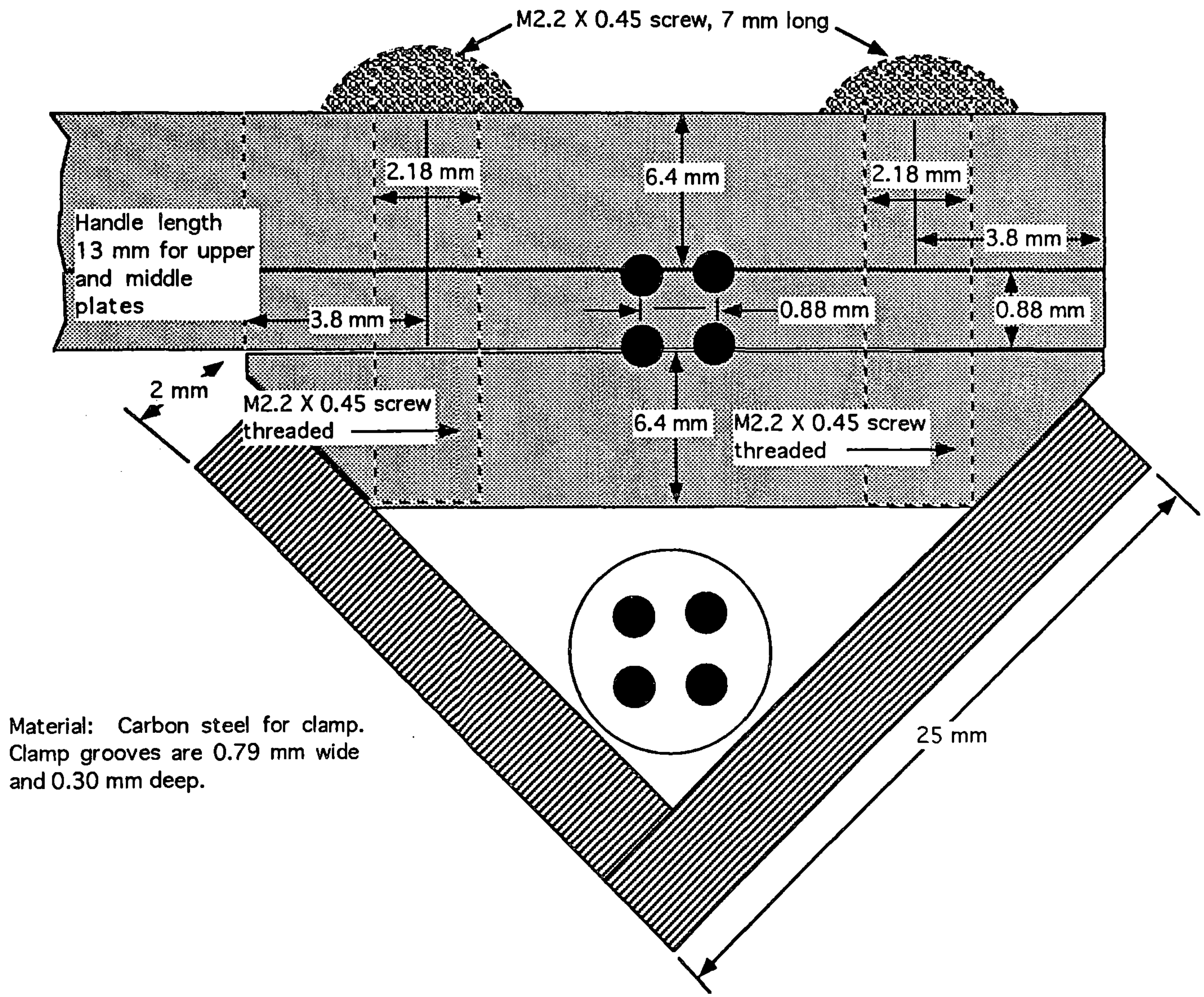


Fig. X.5.8. Clamp block in aluminum angle.

Commercially available tools are needed within the glove box. These include:

- fine-point carbon steel tweezers (no plated tweezers),
- end cutter for wire,
- scalpel,
- glass plate for work platform,
- high-intensity light, and
- platform magnifying glass.

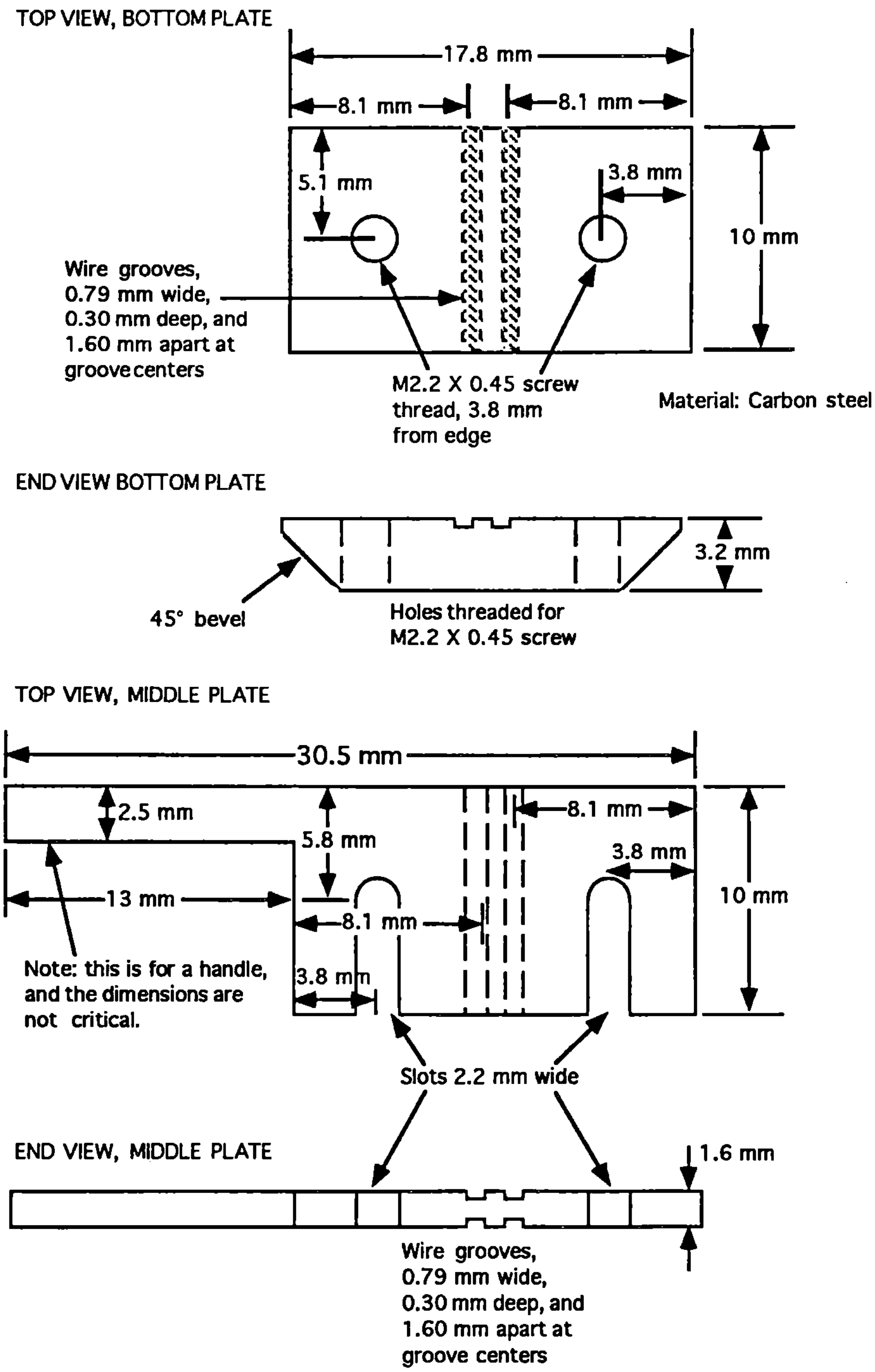


Fig. X.5.9A. Wire jig, clamp block details.

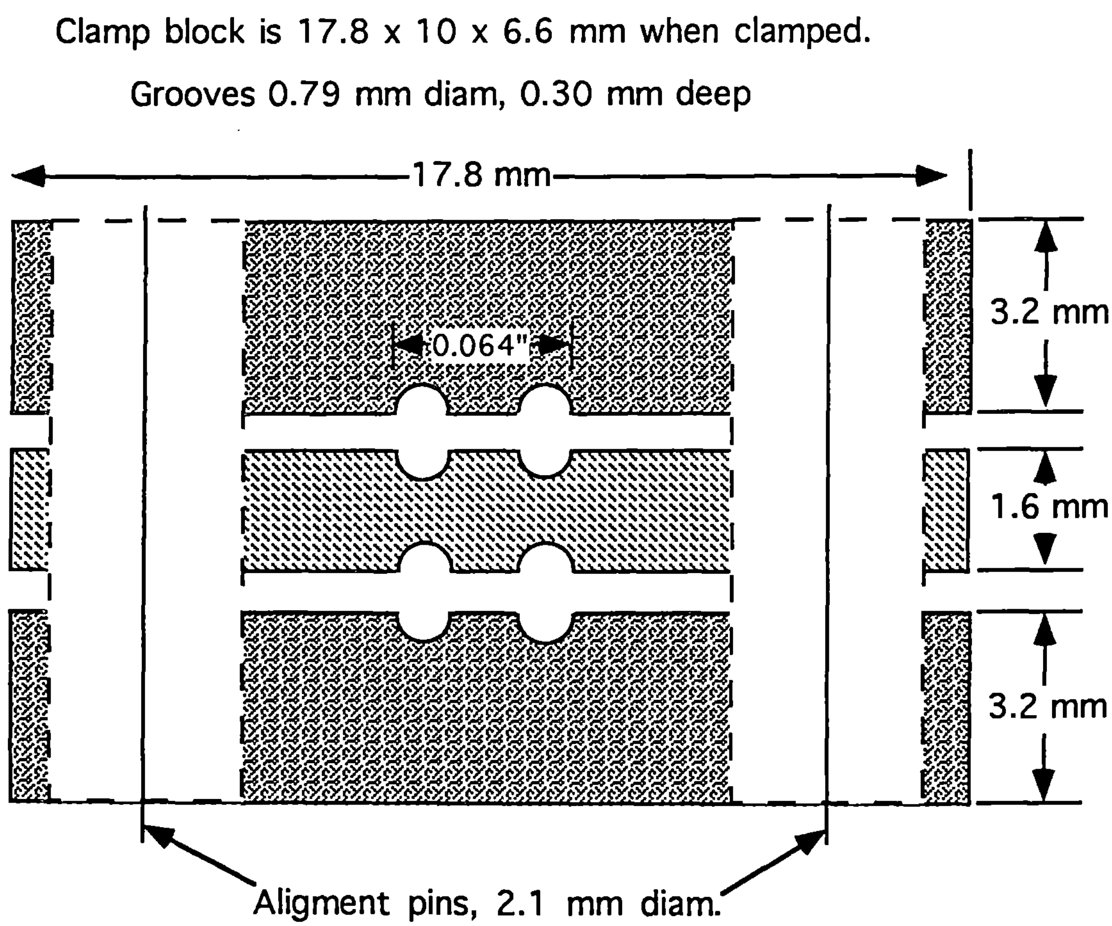
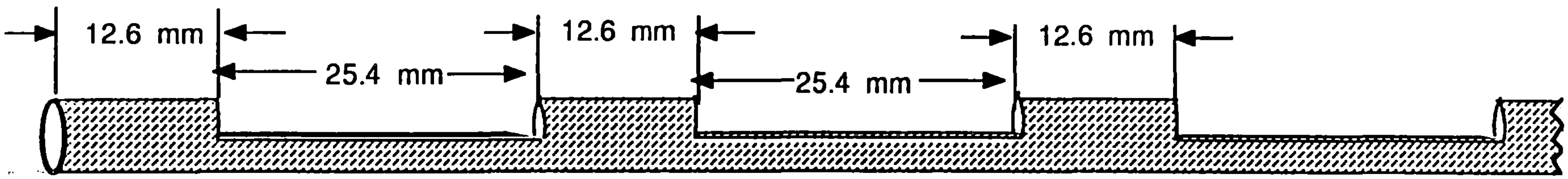


Fig. X.5.9B. The clamp block holds the lead wires aligned.



Nb-1%Zr sheath tubing, 3 m long, 5.82 mm diam. Sections 25.4 mm in length cut to half the diameter with 12.6-mm intervals of intact tubing for the full length.

Fig. X.5.10. Jig for holding insulated wires and joining the sensing element to the lead wires.

X.6. APPENDIX 6. CLEANING, OUTGASING, AND STORAGE

X.6.1 PREPARE INSULATORS FOR OUTGAS

1. Remove the insulator preforms from the factory packing by using plastic or rubber gloves—at no time is the insulation to be touched by bare hands.

2. Make the required cuts in some of the 108-mm-long (4.25 in.-long) preforms to be used for the sensing element as shown previously in Figs. 6 and X.1.5. The cuts are better made with air abrasive but can be made with carbon steel tools as long as the cuts are rounded and deep enough to allow the sensing element to make the turns without protruding above the surface of the preform.

3. Make separate bundles of the insulator preforms and those cut away for the sensing element. Loosely wrap the bundles with Nb or Nb-1%Zr foil as a surface barrier between the furnace lining and the insulator preforms, and tie the bundles with Nb wire. The finished bundles are to be no larger in diameter than the mouths of the jars that will be used to store them—in this case, 38-mm (1.5-in.) diam. If a horizontal furnace is to be used for the outgas, attach a niobium draw wire to the bundle to help in the removal from the furnace.

X.6.2 OUTGASING AND STORING INSULATORS

X.6.2.1 Vacuum–Temperature Schedule for a Vertical Furnace

1. With the insulators in the furnace, evacuate the furnace at room temperature until the vacuum level attains 10^{-3} Pa (10^{-5} torr) for at least 30 min.

2. Fill evacuated system with argon or helium to about one-half atmospheric pressure.

3. Repeat steps 1 and 2 twice.

4. Evacuate the furnace at room temperature until the vacuum level attains 10^{-3} Pa. The crushable preforms shrink when they are fired. The preforms are fired at a time-temperature schedule during their manufacture to obtain the specified dimensions and hardness. Heating at higher temperatures during an outgas procedure will cause additional shrinkage and change the dimensions and hardness of the preforms. For the outgas, it is desirable to heat the preforms to a temperature greater than the operating temperature but below that which will cause shrinkage. Heat the evacuated system to the highest safe temperature for the preforms used (800°C for ultrahigh-purity, ultrafine-grain alumina; 1200°C for hafnia and yttria), and maintain the temperature and vacuum for at least 16 h.

5. Cool to room temperature under vacuum.

6. Fill the furnace system with high-purity argon and open the furnace, maintaining a positive pressure of argon. Place a sealed bottle that was earlier filled with argon into the furnace and open the bottle. Place the container of insulators in the bottle, but do not close the bottle.

CAUTION! ARGON DISPLACES AIR; AVOID BREATHING ARGON.

7. Repeat steps 1 and 2 twice, and then fill the furnace with a slightly positive pressure of argon. Open the furnace and quickly seal the bottle before removing it from the argon blanket in the furnace. Hereafter, the bottle is to be opened only in an argon atmosphere.

X.6.2.2 Vacuum–Temperature Schedule for a Horizontal Furnace

Perform steps 1 through 5, the same as in Sect. X.6.2.1.

6. Fill cooled furnace with argon gas to slightly more than atmospheric pressure.

7. Place the plastic bagging device (as shown in Fig. X.5.6) containing cutting tools and plastic storage bottles, with lids off, over the furnace entrance and connect bubbler filled with vacuum pump oil to the bagging device.

8. With the far end of the bagging system open, and working through the flexible bag, remove the furnace plug so that argon flushes through the bagging device. Flush for 1 min with a vigorous argon flow; then seal the large open end of the bagging system and the exhaust tube.

9. Using the furnace roughing pump, evacuate the furnace and the bagging system and refill with argon. Repeat the cycle three times.

10. With argon at about 50 mm of oil above atmospheric pressure, draw the bundled insulators into the plastic sleeve by the attached wires.

11. Working through the plastic sleeve, place the bundled insulators into the plastic bottles, cutting the draw wires if necessary, and screw the bottle caps on tightly.

12. Remove the sealed bottles from the bagging device and place a tape seal about the screw cap and the bottle as a secondary seal. Label the bottles for the type and number of insulators.

13. The insulators are to be removed from the bottle only in an argon or helium atmosphere.

X.6.3 CLEANING SENSING ELEMENT COILS

NOTE: THE COIL IS NOT TO BE TOUCHED EXCEPT WITH CLEAN SURGICAL GLOVES, GLASS, OR APPROVED TOOLS.

The sensing element coil-winding equipment requires lubrication and so will not allow superclean operation. Therefore, the wires were not cleaned before the coils were wound and must be cleaned after the coils are fabricated. The small coils of 0.127-mm-diam wire are not suitable for cleaning by the acid etch methods of ORNL MET-SR-TS-4, R 2, or the abrasive method of MET-NbA-SOP-10.

The coils are to be cleaned by solvent and nitric or hydrochloric acid immersion while still in the protective glass tube. (The niobium and Nb-1%Zr alloys are not attacked by the acids.) The coils are to remain in the glass tube during cleaning and drying and are not to be removed from the tube until they are installed in the insulator.

1. With one end of the protective glass tube immersed in a container of acetone or perchlorethylene, apply a vacuum to the other end using a suction bulb (NOT BY MOUTH!!) so the fluid is drawn up into the glass tube, totally immersing the coil. Vary the vacuum level so the fluid surges back and forth through the tube for 5 min.

IMPORTANT! The small coils are very flexible and slide easily in the fluid- filled glass tubing. Place glass fiber plugs as filters on either end of the protective glass tube to avoid expelling the coil during the cleaning and drying operation.

2. With one end of the protective glass tube immersed in a container of distilled water, apply a vacuum to the other end so the water is drawn up into the glass tube, totally immersing the coil. Vary the vacuum level so the water surges back and forth through the tube for 5 min.

3. Repeat steps 1 and 2.

4. Repeat step 1 except use 50% distilled or deionized water and 50% nitric acid (50% water, 25% nitric and 25% HCl can be substituted).

5. Repeat step 2 three times to rinse the acid from the coil.

6. Dry the coil by flowing argon or helium through the protective glass tube.

7. Seal the coil in the protective glass tube using heat shrink tubing on both ends until it is ready for use.

X.6.3.1 Basis for Wire Cleaning Procedure

Niobium-1%Zr wire of 0.25-mm-diam (0.010-in.-diam) wire [stock on hand at Westinghouse Hanford Company (WHC) of unknown pedigree] was drawn to 0.13-mm (0.005-in.) diam. Chemical analysis of the wire was made after two preparation treatments: first, after degreasing the wire with acetone and, second, after etching the wire with a 50% nitric acid solution. The chemical analysis, listed in weight ppm, is compared to the ASTM specification for reactor grade Nb-1%Zr. (Ref. 4)

Table X.6.1. Surface contamination removed by acid etching

Element	Degrease	Acid etch	ASTM B-392[7]
Al	5	10	20
B	5	5	2
Ca	10	3	40
Cl	5	5	
Co	<1	<1	20
Cr	<3	<3	20
Cu	300	20	40
Fe	200	100	50
K	10	5	40
Mg	3	3	40
Mn	<3	<3	40
Na	20	10	40
Ni	100	20	50
S	<3	<3	
Si	<5	<5	50
Ta	800	800	1000
Ti	<3	<3	40
V	<1	<1	40
W	<3	<3	300
Zn	<1	<1	40

The analysis shows that the 0.127-mm (0.005-in.) wire has a surface contamination, mainly from iron and copper that probably was caused by the drawing process. The surface contamination cannot be removed by degreasing but will be removed by an acid bath (niobium and zirconium are not attacked by hydrochloric or nitric acid).

The wire had an additional analysis for gas and alloy content. The results are listed in weight ppm.

Table X.6.2. Niobium sensing element wire gas and alloy content

Element	ORNL analysis	ASTM B-392 specification
Carbon	371	100
Hydrogen	89	10
Nitrogen	113	100
Oxygen	695	250
Zirconium	11,000	8,000 to 12,000

The results show that the 0.127-mm (0.005-in.) wire is out of specification, probably because the melt was made before process improvements were adopted.

Samples of the acid-cleaned 0.127-mm wire were submitted for high-temperature grain growth and embrittlement tests. Specimens of the 0.127-mm wire, unstressed and stressed by tight coiling, were aged at 1200°C for 1 h and 1100°C for 100 h. The wire was ductile after the heat treatment and had small (ASTM 8–10) grain sizes.

SUBSTITUTE CLEANING PROCEDURES USING A HIGHER LEVEL OF CLEANING CAN BE USED.

X.6.4 CLEANING LEAD WIRES

1. Cut the wire into the length needed for the sensor assembly, using clean carbon steel tools.

2. Do not kink or make sharp bends in subsequent handling. Place the wire in moderately flexible plastic tubing that will allow flexibility but not kinking.

3. Clean wire according to Sect. I of MET-NbA-SOP-10, "Cleaning Procedures for Niobium-Base Alloys." Alteratively, clean wire with abrasive soap and wash in distilled or deionized water.

4. Rinse wire in ethanol, and air dry.

5. Do not touch wire except with clean gloves after cleaning. Keep the wire in plastic tubing for storage.

X.6.5 CLEANING THE SENSOR SHEATH

The 3-m-long Nb-1%Zr tubing is placed in quartz tubing that is closed on one end and slightly longer than the Nb-1%Zr tubing (as previously shown in Figs. 1 and 2). The quartz tubing serves to protect the sheath during storage and assembly of the sensor.

1. Fill the glass tube with perchlorethylene, totally immersing the Nb-1%Zr tubing. Close the open end of the quartz tubing with a clean plastic stopper. Allow the tube to remain filled for 30 min and gently agitate by rocking the tube every 10 min (or by sonic agitation).

2. Pour the perchlorethylene from the tube and add fresh perchlorethylene to immerse the tube as before. Allow the tube to remain filled for two hours and gently agitate every 30 min.

3. Pour the perchlorethylene from the tube and add fresh perchlorethylene to immerse the sheath tubing as before. Allow the tube to remain filled overnight—at least 16 h. Agitate before and after the overnight soak.

4. Fill the tube with distilled or deionized water and allow the tube to stand for 5 min with gentle agitation.

5. Repeat step 4 except for 10 min.

6. Repeat step 5.

7. Repeat steps 4–6, using ethyl alcohol.

8. Dry by vacuum or by flowing inert gas through the glass tube.

Seal the open end of the protective glass or quartz tube by using a teflon plug and a neoprene O ring compression seal until ready to use the sheath tubing. Touch the sheath hereafter only with gloves or approved materials.

The end of the tubing where the end plug is to be welded is cleaned by acid etch according to the method of MET-RS-TS-4, R2.

X.7. APPENDIX 7. THERMOMETER CONSTRUCTION SEQUENCE

Removing and inserting 3-m-long sheaths and sensor assemblies from 3.2-m-long quartz containment tubes require a glove box that is at least 7 m (23 ft) long. Provision for gas locks and working areas increase the glove box length to 9.7 m (32 ft). Space to load the containment tubes into the glove box increases the required work bench length of at least 14 m (46 ft).

A flow diagram of the construction procedure is given in Fig. X.7.1.

X.7.1 INFLATING GLOVE BOX AND PREPARATION

This procedure only prepares the glove box for work and ensures that all the equipment is in place.

The inflatable 10-m-long (32-ft-long) glove box of clear, 0.32-mm-thick (0.0125-in.-thick) PVC plastic fitted with surgical gloves (see Figs. 3 and 5, shown previously) is unrolled and placed on a work bench so there is 3-m clear bench space at the end of one of the sleeves. A plastic argon supply tube is taped into one of the five openings of 13-mm (0.5-in.) diam for vacuum, gas, electrical, and instrument line access in the working section (as shown previously in Fig. 4), and the other openings are plugged or clamped shut. An easy clamp is made by using two strips of thick wood long enough to bridge the sleeve and using C clamps to squeeze the sleeve between the two strips. The glove box is inflated to a soft level to assume its inflated shape. Pressure is controlled by an oil bubbler.

After unclamping one of the sleeves, Plexiglas strips about 150 mm (6 in.) wide and 1.2 m (4 ft) long are inserted into both sleeves and the working section to form a floor. Spaces of about 0.3 m (1 ft) are left in the sleeves to allow the sleeves to be clamped shut if necessary. The hand tools listed in Appendix X.5 are then inserted into the sleeve, and moving them along the sleeve by grasping through the flexible plastic, the tools are placed in the working section. [The working section is a tube 0.43 m (17 in.) in diameter and 0.6 m (24 in.) long, with two sleeved openings that accommodate surgical gloves.]

The sealed jars containing the outgassed crushable preforms and the sealed glass tubes containing the cleaned sensing elements are then moved into the working area and stored. The sealed quartz tubes containing the cleaned sheaths with a welded end plug are moved past the working area and stored in the far sleeve, where they can be reached from the glove ports.

The procedure is then to clamp the end of the access tube and evacuate the glove box; the plastic will simply collapse. The collapsed glove box is then refilled with argon. The evacuation and filling procedure is repeated several times. The argon pressure in the glove box is controlled to a positive pressure by simply venting the excess argon through an oil filled bubbler.

X.7.2 PREPARING THE LEAD WIRES

This procedure is performed in air on a clean bench.

The lead wires have already been cleaned, and one end has been acid-etched to a smaller diameter. The lead wires are stored in plastic tubing to keep them clean and to prevent kinking. The wires are removed one at a time from the plastic tubing and placed on a clean paper surface.

As each wire is removed from the tubing, the end of the wire next to the acid-etched stub is clamped in a bench vise with smooth clean jaws. The other end of the lead wire is grasped with a pair of pliers. The wire is pulled straight, and then enough tension is applied that a slight "give" is felt. The inelastic stretch given to the wire is important to remove the tendency of the wire to curl; however, no further elongation should be made.

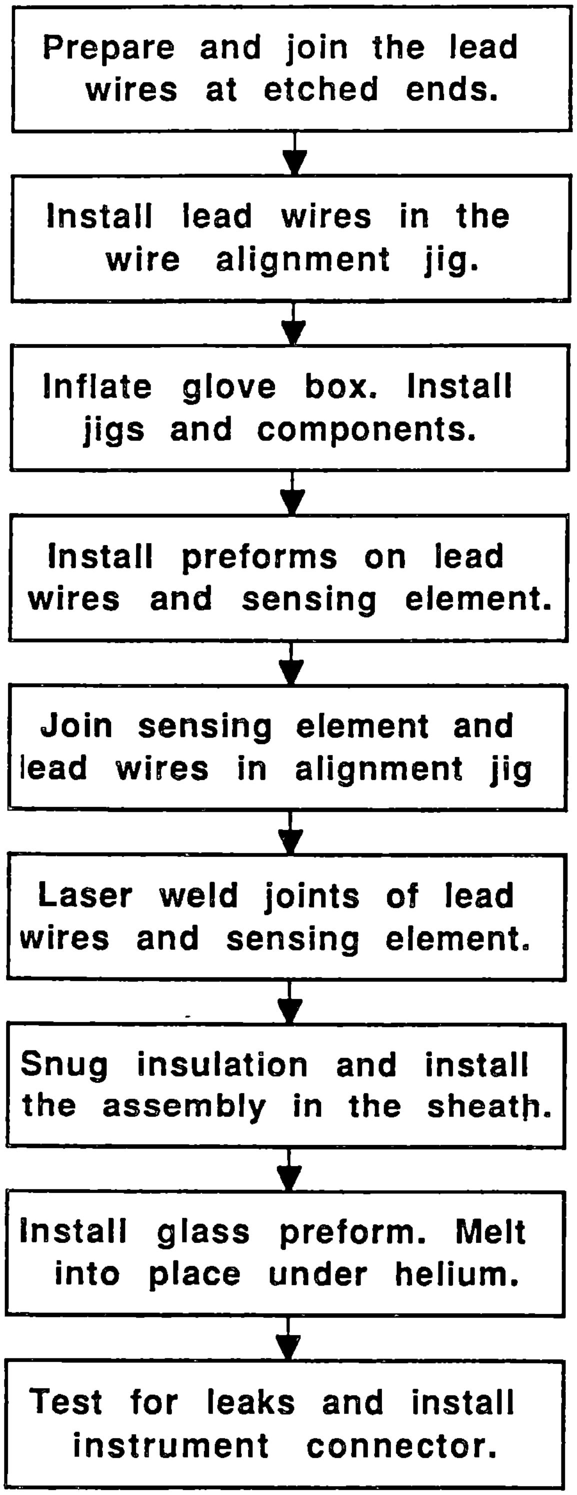


Fig. X.7.1. Assembly sequence for thermometers after components have been cleaned and prepared for assembly.

X.7.2.1 Joining The Lead Wires

This procedure is performed in air on a clean bench.

The lead wires are stretched to remove curves and bends before the 0.23-mm-diam (0.009-in.-diam) wire stub is wrapped around the 0.46-mm-diam (0.018-in.-diam) wire stub. The 0.23- and 0.46-mm wire ends of two leads are clamped in the wire wrap joining clamp (see Fig. X.7.2). Using the wire wrap tool (see Fig. X.7.3), insert the 0.46-mm wire into the 0.51-mm-diam (0.020-in.-diam) central annulus. A shorter length of the 0.23-mm wire is inserted in the annular slot, and the wrapping handle is turned to make several tight turns of the 0.23-mm wire around the 0.46-mm wire.

Some practice is necessary to learn the correct length of wire to put into the annular slot to avoid excessive turns. The length of the stub of 0.46-mm wire protruding below the wrap is not important, since it will be trimmed to length when the sensing coil is installed. The wire wrap will be back-welded to secure good electrical connection and prevent any possible slippage of the tight wrap. The strength of the connection is, however, in the wire wrap.

X.7.2.2 Placing the Lead Wires in the Wire Jig

This procedure is performed in air on a clean bench.

The lead wires, wrap joined at the coil end, are placed in the wire clamp block (as shown previously in Fig. X.5.8) to hold the four wires parallel. The center plate (see Figs. X.5.9A and X.5.9B) is first placed between the two upper and the two lower wires, and then the upper and lower plates are positioned to hold the wires in the grooves. The clamp block screws are tightened to hold the wires in place, and then the clamp block is attached to the aluminum angle with the plate clamp (as shown previously in Fig. X.5.7).

Additional clamp blocks are applied, starting close to the preceding clamp, and with the clamp block loosely screwed together, the wires are combed to bring the wires parallel as the clamp block is moved away. It is necessary to release the clamps, one at a time, and adjust the positioning of the individual wires to ensure that all the wires are under the same tension.

Finally, the lead wires should be under tension, without slack in any wire, and positioned properly for the preform holes. The clamp block nearest the cold end should have about 100 mm of wire protruding. The cold ends of the lead wires should be cut so they are slightly uneven in length, and the wires thereafter can be identified by the length.

After a final cleaning of the wires with an alcohol spray to remove any paper fiber dust, the aluminum angle, mounted on its base, with the wires clamped on, is inserted into the argon-filled glove box.

With the glove box filled with argon, the sleeve is unclamped and the aluminum angle jig with the lead wires is slid down the sleeve, with the cold end of the lead wires entering the working area first. The sleeve of the glove box is clamped shut, and the glove box is allowed to vent for a while before the vent tube is attached to the bubbler (see Fig. X.8.3 in Appendix X.8).

X.7.3 INSTALLING INSULATOR PREFORMS

This procedure is performed in the inert atmosphere in the glove box.

Installing the insulator preforms is done in the argon-filled glove box after the lead wires mounted in the supporting jig (as shown previously in Fig. X.5.8) have been moved into the glove box. The jig for holding the insulated wires (as shown previously in Fig. X.5.10) has been cleaned and is in a protective quartz tube in the glove box.

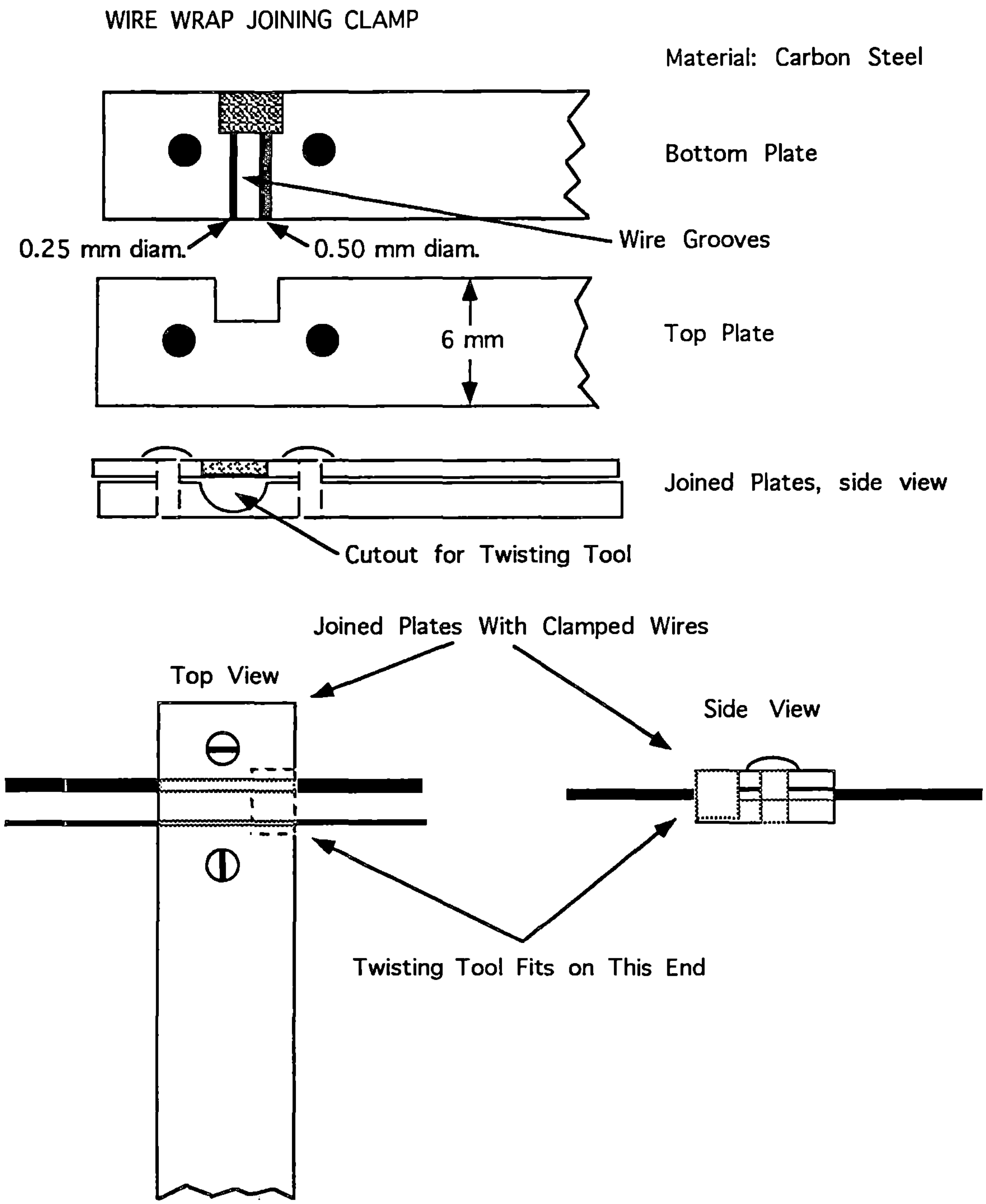


Fig. X.7.2. Lead wire twist tools (clamp).

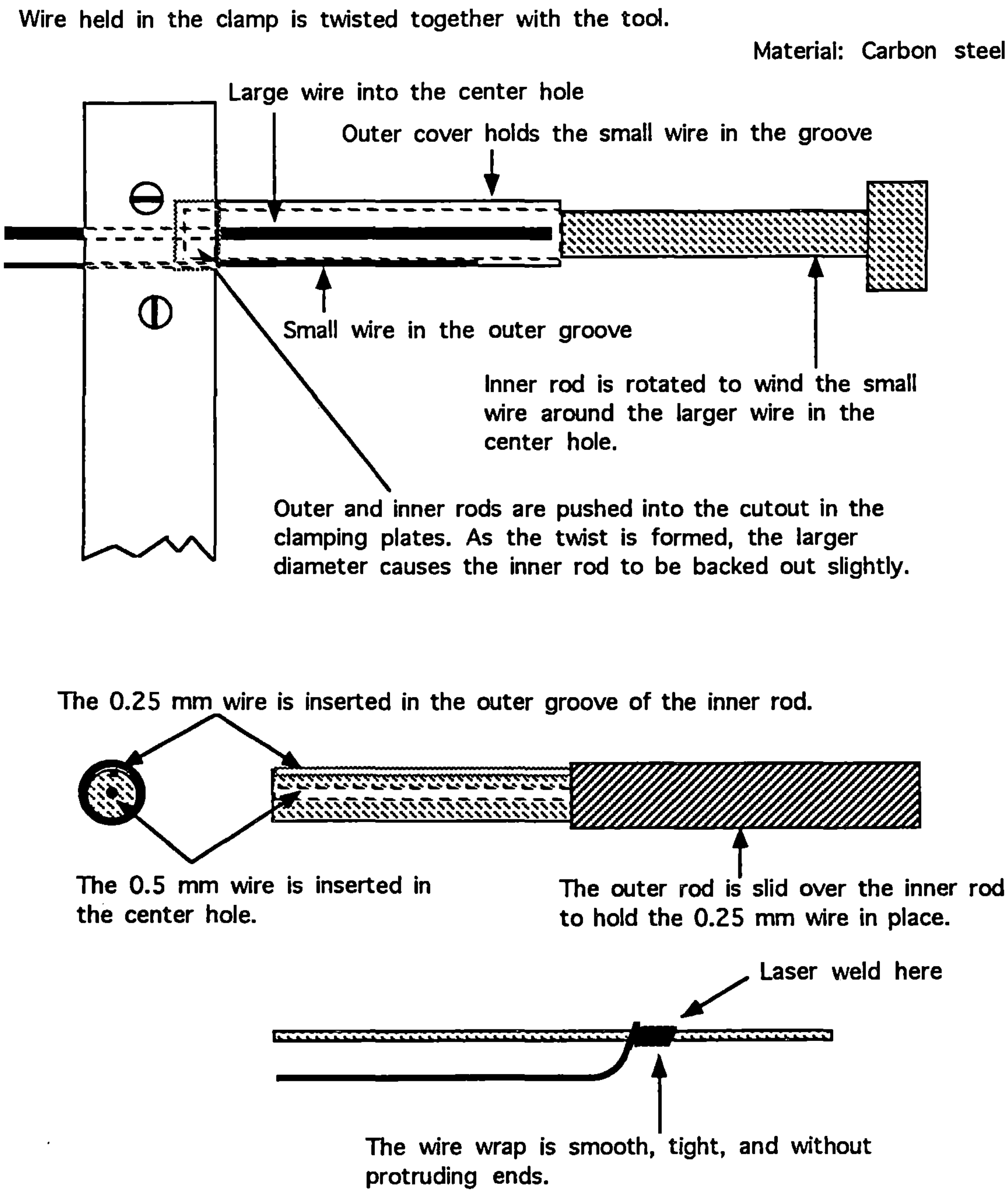


Fig. X.7.3. Lead wire twist tool.

The sealed insulator jar containing the special cutout insulator preforms is opened. The niobium wire holding the niobium foil around the bundle is clipped, and the foil is unwrapped. It is likely that the foil will be brittle after reacting with the gases baked from the insulators. Select a preform that has a web cutout on one end only; this cutout is to allow the wrap joints to be drawn into the insulation (Fig. X.7.4). Replace the other preforms in the jar, and seal the jar.

Move the wire jig into the working area so the cold ends of the wires are in reach. Thread the cutout preform, cutout end first, onto the protruding four wire stubs. Be careful that the preform is oriented so the web cutouts will match the wire wrap joints. [The threading will be easier if preform bores are first cleared of protruding particles by passing a 0.76-mm-diam (0.030-in.-diam) niobium alloy wire probe through each hole.] Slide the preform up the wires, using a gentle back and forth rotary motion of a few degrees while pushing gently. The preforms are crushable, so they must be handled with a soft touch.

When the preform is at the first wire clamp, remove the clamp and slide the preform to the second clamp, and then replace the first clamp. Open the sealed jar containing the unmodified preforms, and remove several preforms to make a working supply (placed on a glass plate) before resealing the jar. The insulators are then to be worked down the four wires, always ensuring that the wires are taunt so as to avoid kinking them. The wire clamp jigs are moved as required when the insulation slides down the wires. The procedure is repeated until preforms are spaced along the wires about 150 mm (6 in.) apart. When preforms are spaced along the wires so that the wires are held in alignment, the wire clamps can be removed. Preforms are added and gradually moved down until there are no gaps between the preforms. NOTE: The cutout preform is not moved down over the wrapped wire joint, since this is yet to be welded.

At this point in the assembly, the insulated lead wires are laying in the plastic-coated aluminum angle with the clamping blocks removed (as shown previously in Fig. X.5.7). The cutout sheath (as shown previously in Fig. X.5.10) is removed from its protective quartz tube and placed in the aluminum angle. The cutout sheath is then moved up the angle as the insulated wires are slid into the sheath. When the insertion is completed, the joined wire wraps are about 125 mm (5 in.) from the end of the sheath in the center of the cutout, with the wrap joints aligned horizontal.

X.7.4 INSERTING SENSING COIL INTO INSULATOR PREFORM

This procedure is performed in the inert atmosphere in the glove box.

The sealed insulator jar containing the special cutout insulator preforms is opened. A preform containing cutouts on both ends (as shown previously in Fig. 6) is removed from the jar, and the jar is resealed. The bores on the preform are cleared of protrusions by passing a 0.76-mm-diam (0.030-in.-diam) niobium wire through the preform. This bore reaming is important since the sensing coil must move through the bore without friction.

The sealed tube containing the cleaned sensor coil is opened, and the coil is slid onto a clean glass plate. The coil is straightened, and the length is measured. Handling is done with the surgical rubber gloves of the glove box, and a very delicate touch is required for the fragile coil. The length of the coil should be 0.43 m (17 in.) to make the four loops in a preform 108 mm (4.25 in.) long with cutouts on both ends. The coil ends are inspected under magnification to see whether the final turns are of the correct diameter and without a protruding wire end. If necessary, the coil end can be trimmed with the sharp point of a craft knife pressing against the glass plate. The coil can be stretched slightly if it is not long enough and trimmed if it is too long.

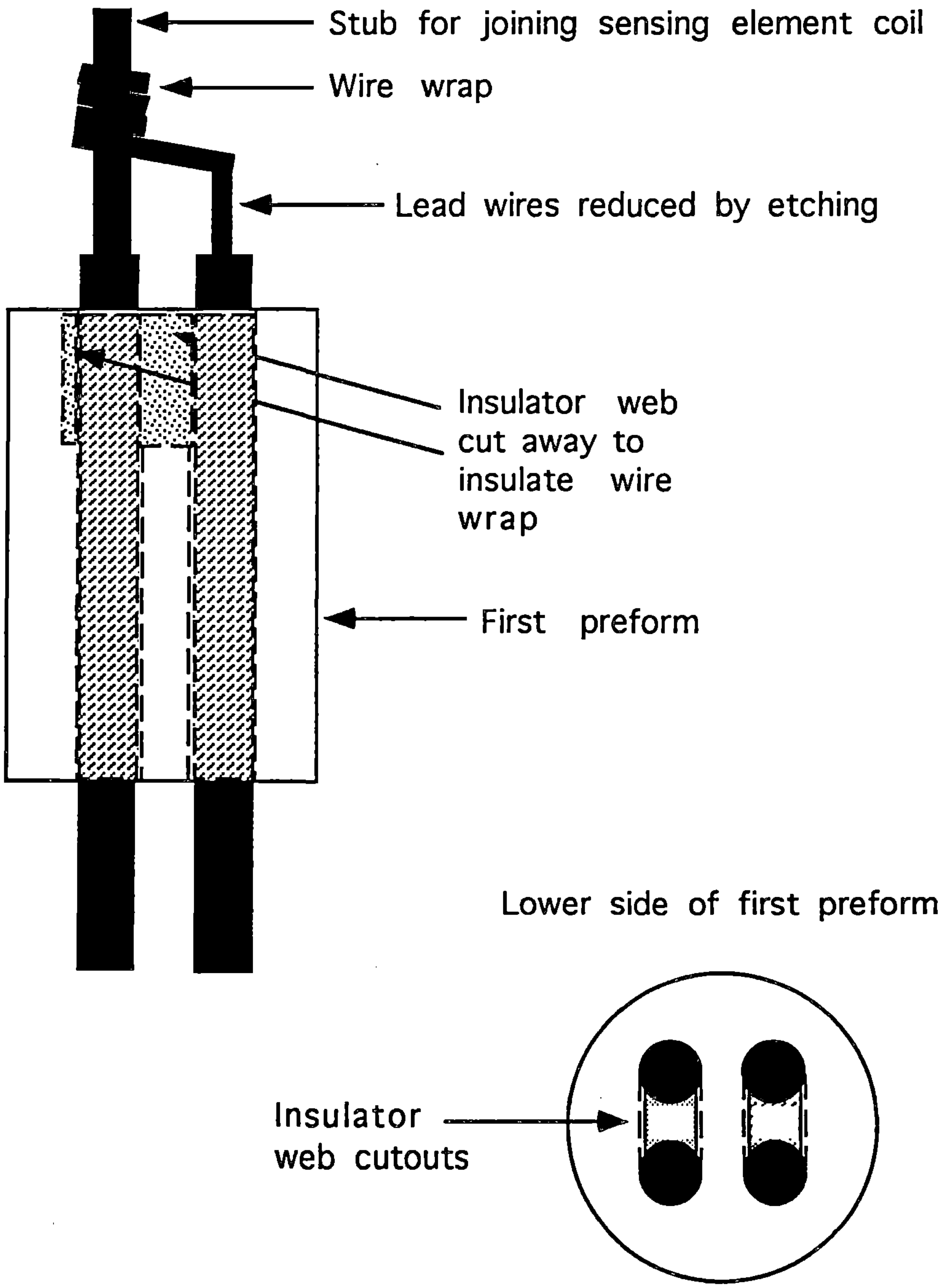


Fig. X.7.4. Webbing cutouts for joined lead wire installation.

The preform is clamped (gently) in a vertical position so the cold end (as shown previously in Fig. 6) is upper and there is about a 150-mm (6-in.) clearance below the preform. The sensing coil is lifted gently at the center (using a thin segment of niobium foil to scoop it up—tweezers would crush the coil) and draped over the operator's finger. The rubber glove has enough friction to prevent the coil from sliding. The two ends of the coil are lowered into holes 2 and 3 of the preform (as shown previously in Fig. 6) and should slide freely through the preform. If the two ends of the coil are even, the coil turn can be gently pressed into the cutout to provide friction that will hold the coil in place.

The preform is unclamped and reclamped inverted so the lower side of the sensor is up (Fig. 6, shown previously, and Fig. X.1.5). The end of the coil protruding from hole 2 is inserted into hole 4, and the end of the coil protruding from hole 3 is inserted into hole 1. After inspecting to see that the ends of the coils just emerge from the preform, the coil turns can be gently pressed into the cutouts. If the coils do not protrude enough, they can be pulled back and stretched to have the correct length.

X.7.5 JOINING SENSING COIL TO LEAD WIRES

This procedure is performed in the inert atmosphere in the glove box.

This procedure is done under argon atmosphere. Insert the insulated lead wires into the welding jig (made from Nb-1%Zr tubing with segments cut from half the upper section of the tuning to allow access). Insert the insulated sensing coil into the jig so the open ends of the of the sensor coil are aligned with the protruding stubs of the 0.46-mm-diam (0.018-in.-diam) lead wire.

Using the tip of a craft knife, or some other sharp probe, tease the coil onto the stubs so that several turns are onto the stub (as shown previously in Fig. X.1.5). Push the sensor toward the leads so the protruding coils are under compression but not into the preform; to allow welding to the lead wire stub, the coils must be visible.

Using slivers of Nb-1%Zr foil, wedge the insulators of the sensing element and the lead wires into the welding jig so the insulators will not shift or rotate during transport of the jig. Be sure that the joints to be welded are clearly visible through the cutout sections of the sheath tubing.

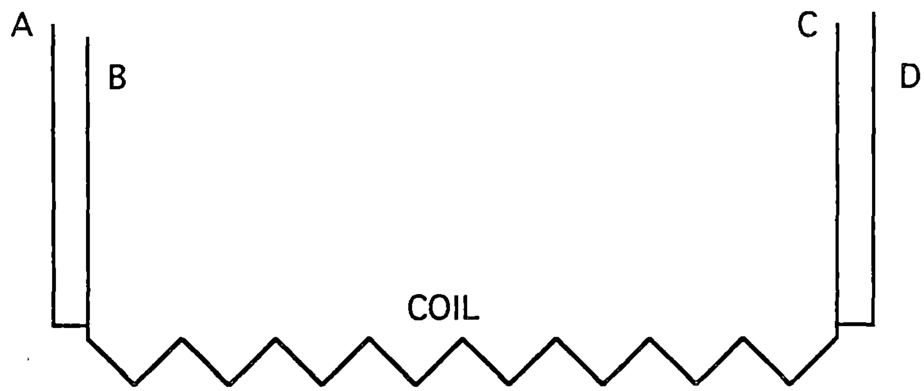
Measure the loop resistance of the 4 wires A, B, C and, D in the following manner: A-B; A-C; A-D; B-C; B-D; C-D. Record the ambient temperature. Calculate (Fig. X.7.5) the lead wire and sensing coil resistance and record to compare to that measured after welding. No standard is established for rejection, since temperature may vary between measurements, but any variation should indicate an irregular weld contact.

Slide the welding jig (containing the insulated lead wires and sensing coil) into the quartz protective tube with the sensor end toward the closed end of the tube. While still in the glove box, evacuate the quartz tube and back-fill with argon, repeating the process 10 times. Use tubing attached directly to the quartz tube; it is not necessary to do this procedure on the entire glove box. Seal the quartz tube with a compression fitting to retain the argon atmosphere. Remove the quartz tube from the glove box, and place the quartz tube into the plastic pipe storage tube.

X.7.6 WELDING THE SENSING COIL TO THE LEAD WIRE

This procedure is performed in the inert atmosphere in the quartz protective tube.

Place the sealed quartz tube under the laser welder, and rotate the tube so that the joints of the lead wire wrap and the sensing coil connection are visible through the quartz tube. Using the laser positioning



A,B,C,D are the lead wires resistances, and COIL is the sensing element.

The resistance measurements are between the given wires.
R1 = A to B, R2 = A to C, R3 = A to D
R4 = B to C, R5 = B to D
R6 = C to D
All measurements except R1 and R6 include the coil resistance.

R1 = A + B, R2 = A + COIL + C, R3 = A + COIL + D

R4 = B + COIL + C, R5 = B + COIL + D

R6 = C + D

R2 - R4 = A - B and R1 = A + B, so R1 + R2 - R4 = 2A.
B = R1 - A ; there are now numerical values for A and B.

R4 - R5 = C - D and R6 = C + D, so R6 + R4 - R5 = 2C; a value for C.
D = R6 - C; there are now numerical values for A, B, C, and D.

The coil resistance is measured directly by the four-wire potential drop method. The direct measurement should agree with
COIL resistance = R2 - A - C.

Fig. X.7.5. Sensing element resistance measurement method.

equipment, spot weld the lead wire wrap to ensure good electrical contact and to prevent possible slipping of the wrap. Spot weld the upper turns of the sensing coil to the lead wire stubs. Inspect the welds (microscope used for the laser beam positioning) to see that wells are bright and clean.

Return the sealed quartz tube to the glove box, but do not remove the welding jig from the sealed quartz tube until ready to install the assembly into the sheath.

X.7.7 WELDING THE HOT END PLUG TO THE SHEATH

This procedure is performed in the inert atmosphere in the glass welding chamber.

The welding, before the tube is to be swaged, is done with a Raytheon SS 500 YAG Laser Welder. The welding of the Nb-1%Zr end plug (in previously shown Figs. 7, 8, and X.3.1) to the 5.8-mm-OD (0.230-in.-OD) by 5-mm-ID (0.200-in.-ID) Nb-1%Zr tube is performed by using the procedure listed below.

1. Acid clean the end plug and the end of the tube to be welded. Handle thereafter only with clean gloves.

2. Insert the end plug into the tube by using clean gloves or approved tools. The plug must be a tight fit in the tube end for good heat transfer during welding. The end plug must fit square to the sheath end so there is minimum gap between the tube end and the end plug weld lip.

3. Insert the end of the tube to be welded into the quartz window welding chamber, and position the joint under the window (as shown previously in Figs. X.5.1 and X.5.2).

4. Tighten all connections, and evacuate five times with a mechanical vacuum pump, purging with argon after each evacuation. Weld with 3.2×10^4-Pa (3-psig) argon pressure in the chamber. Compression unions allow the sheath to be rotated under the laser beam while maintaining argon inside the sheath and outside in the region where the sheath is being welded.

5. Welding parameters:

Focusing lens	100 mm (4 in.)
Pulse length	5 ms
Pulse mode	Repetition
PFN adjust	600
Shutter mode	Automatic
Weld time	20 s
Ramp time	2 s
Slope adjust	900
Post ramp time	2.0 s
Shutter time	2.0 s
Focal point	Sharp
Part travel speed	18 s/revolution (This amounts to 1 mm/s (0.04 in./s))

X.7.8 INSPECT WELDS, VISUAL AND X-RAY

Test weld sections were submitted for metallographic examination for grain structure and microhardness traverse. Metallography showed good weld penetration and good weld microstructure. Hardness tests showed the weld to be softer than the surrounding structure, indicating no contamination by oxygen during welding, which would have produced a brittle weld.

X.7.9 INSERTING SENSING ELEMENT AND LEAD WIRES INTO SHEATH

This procedure takes place in a glove box filled with argon.

From the sealed quartz tube, remove the welding jig containing the insulated lead wires that have been welded to the sensing element. Place the welding jig in the aluminum angle, and removing the locking shims as necessary, snug the insulation preforms so the joined ends of the lead wires are snugged into the cutaway webbing and there are no gaps between the preforms. It may be necessary to add another preform after the insulation is snugged together.

After the insulation is snugged into place, measure the loop resistance of the four wires A, B, C, and D in the following manner: A-B, A-C, A-D, B-C, B-D, C-D. Record the ambient temperature. Calculate (Fig. X.7.5) the lead wire and sensing coil resistance, and record to compare to that measured before welding.

The process of inserting the thermometer components into the sheath is performed in the following steps:

1. Remove the sheath with a welded end plug from its protective tube. Place the sheath in the aluminum angle alignment jig, and clamp into place.

2. Place the insulated sensor and lead wires (still clamped in the welding jig) in the alignment jig so the sensor is aligned with the open end of the sheath. Remove the locking shims from the sensor and insulators in the welding jig. Put a small section of empty preform, about 4 mm long, into the sheath entrance; it will be pushed into the sheath by the sensor and prevent the sensing element from possibly grounding to the end closure.

3. Carefully pushing on the insulated lead wires—NOT THE SENSING ELEMENT!—push the sensor assembly from the welding jig into the sheath until the sensor assembly bottoms in the sheath.

4. Measure the sensor resistance and insulation resistance (Ref. 10), using the same techniques as for earlier measurements. Compare to earlier measurements for evidence of sensor or lead wire damage or inadequate insulation.

5. Unclamp the sheath from the alignment jig and carefully inset it into a quartz carrying tube. Use a niobium coil spring to fill the empty length in the quartz tube to prevent the sheath assembly from sliding in the quartz tube. Seal the tube within the argon atmosphere by using compression fittings.

6. Remove the sealed assembly from the glove box, and tag it with the number assigned to the sensor assembly (the number engraved on the hot end plug).

7. Place the sealed assembly in the plastic pipe protective tube used for the quartz, using foam padding to minimize the quartz tube rattling. Tag the protective tube with the number of the sensor.

X.7.10 FINAL OUTGAS ON THE UNSEALED THERMOMETER

A final outgas was given to the thermometer S/N 52; a process that is now believed to be unnecessary.

The 3-m-long sheathed assembly S/N 52, sealed in a quartz tube filled with argon, was placed in a 5.5-m-long (18-ft-long) vacuum furnace. The teflon and rubber compression seal was removed from the quartz tube, the furnace door was shut immediately, and the furnace was evacuated at once. The assembly was maintained at room temperature under 10^{-4}-Pa (10^{-6}-torr) vacuum for the weekend; then, the furnace temperature was raised to 800°C for ~6 h. The furnace was allowed to cool under vacuum overnight.

A plastic hood equipped with glove ports (as shown previously in Fig. X.5.5) was taped to the end of the furnace and filled with helium. The furnace was back-filled with helium to 250 Pa (1-in. water pressure gauge), the furnace door was opened, and an epoxy seal was applied to the open end of the sensor while under a helium cover. An infrared lamp was used to accelerate curing the gel. The epoxy seal was allowed to cure for 48 h, and the sensor S/N 52 was transported to ORNL.

X.8. APPENDIX 8. COLD END SEAL

X.8.1 REQUIREMENTS

1. The cold end seal must be capable of withstanding 150°C for 10 years while one side is exposed to hard vacuum without a leak > 10^{-10} std cc/s of helium.

2. Helium leak test on end seal is to be done as described by ASTM E-839, 7.1.3, "Sheath Integrity-Mass Spectrometer Method".[11]

3. Prototype sensors will have epoxy end seals until the connection configuration has been specified.

4. Prototype connectors will be 4-pin Lemo[12] F2.304NYL S/7.7 female shell, which is to be connected to the sensor sheath or to the extension sheath (Fig. X.8.1).

X.8.2 PURPOSE

There are two types of seals used on each sensor:

1. a temporary seal to keep out contaminants during fabrication and preliminary testing and

2. a permanent seal to be used in calibration tests and to remain in place for the subsequent life of the sensor.

X.8.3 MATERIAL, APPLICATION, TYPE

This procedure is performed in an inert atmosphere.

1. The temporary seal is impervious to gas and can be removed by peeling or cutting without introducing contaminants into the thermometer assembly. Examples are heat shrink tubing, etc.

2. A permanent seal of epoxy can be used for prototype bench testing when the cold end is below 100°C. A leaking seal can be repaired by the application of more epoxy.

3. The permanent high-temperature cold-end seal is either a glass or a metal-ceramic seal and connector combination. The metal-ceramic seal must be welded or brazed to the sheath, and the wires must be welded or brazed to the feed-through under a helium atmosphere. The insulating portion of the seal must be ceramic, either metal oxide or glass. A glass seal can be melted in place under a helium atmosphere, and the electrical connector (Lemo F2.304NYL S/7.7) female shell can be fixed on the sensor or extension sheath by using the compression attachment.

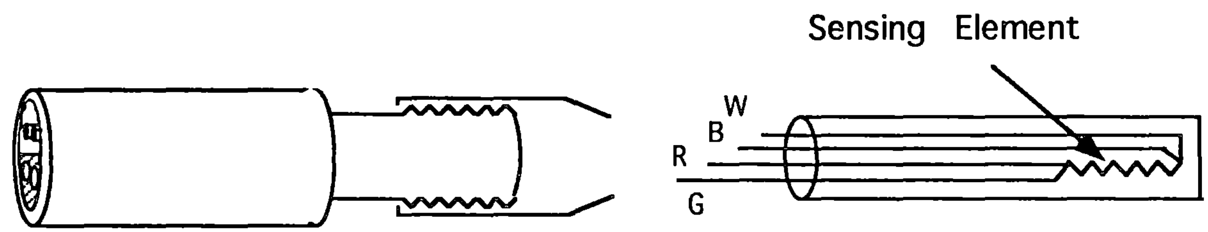
X.8.4 TESTING AND INSPECTION

The seal is tested by:

1. a visual check with a 10-power ocular for evidence of cracks and

2. measuring, with a helium leak detector, for a helium leak from the helium-filled sensor. An indication of helium leakage of more than 10^{-10} std cc/s shall be taken as evidence of a faulty seal, and the seal must be repaired or replaced.

Connector Side View



Lemo Model F2.304 NYL S/7.7

Connector End View

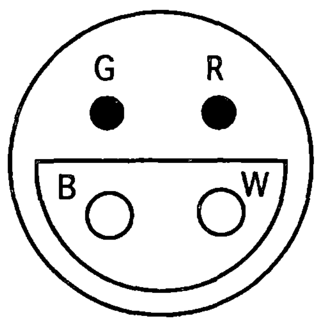


Fig. X.8.1. Lead wire connections for thermometers.

X.8.5 PERMANENT-SEAL, LOW-TEMPERATURE EPOXY

This procedure is performed in the inert atmosphere in the glove box.

1. Apply the epoxy seal after final outgassing and while the sensor is in a helium-filled glove box.

2. With the mineral insulation level just beneath the sheath termination, fix the sensor in a holding jig so the sensor is slanted with the open end uppermost.

3. Mix the epoxy [Scotch Cast 5 (Ref. 13) or equivalent].

4. Heat about 0.3 m of the sensor sheath nearest the open end to about 100°C. The purpose of the heating is to impose a slight negative pressure on the epoxy seal as the sensor cools. The heating can be by radiant heat, a hot gas blower, or a flexible heater.

5. Stop the heating and immediately apply the mixed epoxy to the open end of the cooling sensor, making sure that the wires and sheath edges are wetted. Rotate the sheath so the epoxy flows evenly on both sides. After about 10 min, the cure rate of the epoxy can be increased by careful warming of the epoxy—DO NOT HEAT THE SENSOR SHEATH AS BEFORE, SINCE BUBBLES MAY OCCUR IN THE EPOXY.

X.8.6 PERMANENT-SEAL, HIGH-TEMPERATURE GLASS

Corning 0211 (Ref. 14), a glass composition used to make microscope slide cover glasses, has been found to have a coefficient of thermal expansion (74×10^{-7}/°C) close to that of Nb-1%Zr. A supply of this glass was obtained and powdered in preparation for melt-in-place glass end seals for the noise sensors. Some glass seals applied with a torch looked satisfactory, but all leaked under the helium leak test. The seal had cracked during cooling because the torch melting did not provide the proper annealing process.

A facility was prepared to allow glass seals to be made and annealed in a high-purity helium environment, as required in the fabrication of the noise sensors. The procedure requires preforms of Corning 0211 (see Fig. X.8.2), and the process is as follows:

This procedure is performed in the inert atmosphere of the glove box and the quartz tube.

1. The thermometer assembly has been completed, with the ceramic preforms ending about 6 mm (1/4 in.) below the top of the sheath. A glass preform, 13 mm (1/2 in.) long, is slipped over the lead wires and bottomed onto the top of the preforms. The thermometer assembly is then inserted into the quartz protective tube with the thermometer cold end inserted first. The quartz tube is closed with a compression fitting, and the interior of the quartz tube is evacuated and filled with helium. After several flushes and refills, the quartz tube is valved closed and removed from the glove box.

2. The quartz tube containing the thermometer is fastened vertically so the end of the thermometer with the glass preform is inside a clamshell heater (Fig. X.8.3). The other end of the quartz tube is fastened to a vacuum/helium supply system, as shown in Fig. 8.4. At room temperature, the quartz tube is evacuated and back-filled with helium five times. The vacuum pump is valved off, and the helium supply is throttled so the positive pressure in the quartz tube is just sufficient to blow a small stream of bubbles through the oil bubbler shown in Fig. X.8.4. The pressure can be adjusted over a small range by inserting or withdrawing the bubbler pipe to have different depths in the oil.

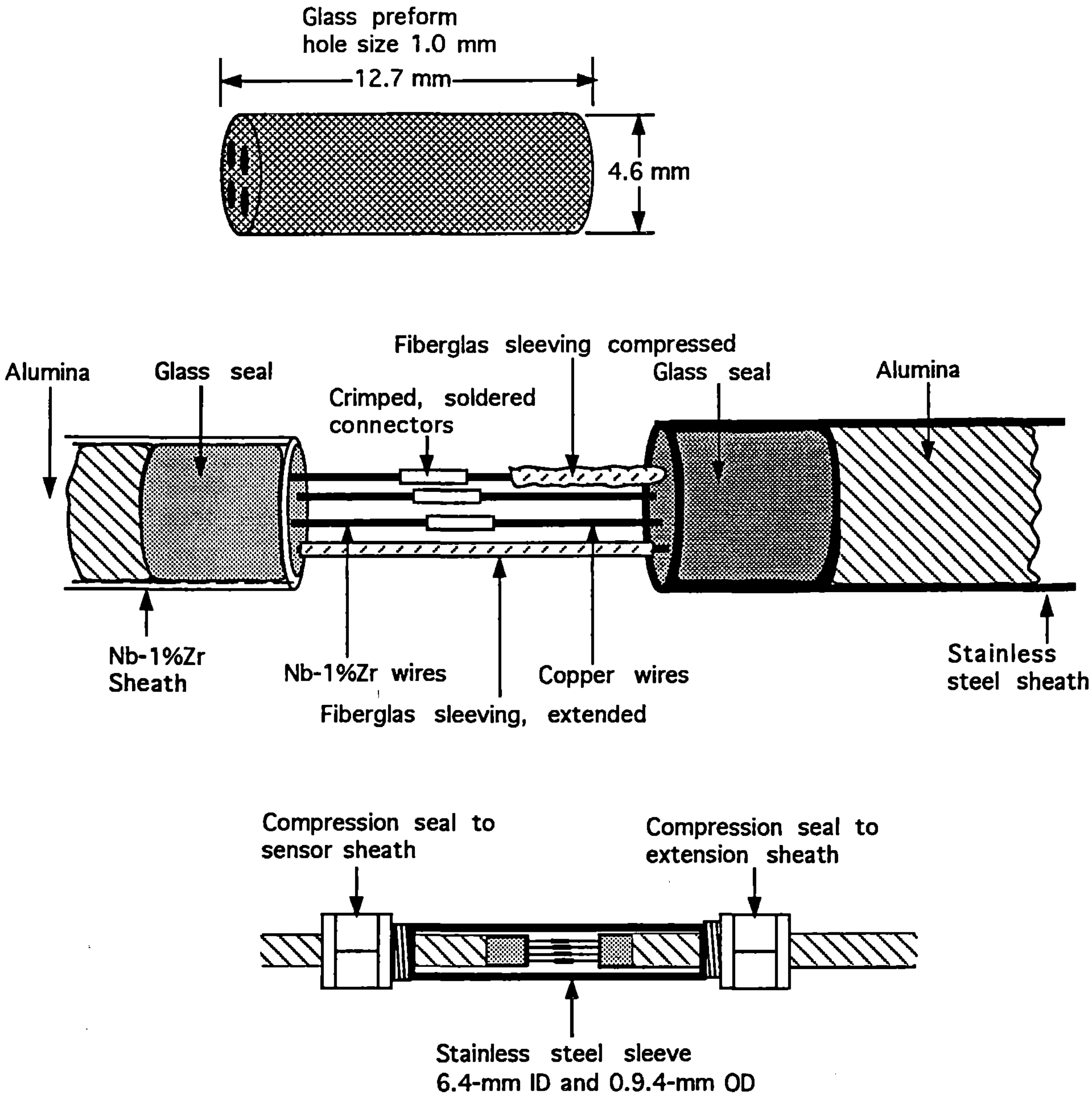


Fig. X.8.2. The glass preforms are melted to make the glass seal, and the thermometer is connected to the extension sheath.

3. The heater temperature is controlled and monitored to produce a furnace time-temperature schedule as follows:

   - Heat the furnace to 1008°C, and hold the temperature for 10 min.
   - Reduce the furnace set point to 720°C, allowing the furnace to cool at its natural rate.
   - Cool the furnace at 5°C/min until it is at 550°C, and then hold for 30 min.
   - Cool the furnace at 5°C/min until it is at 485°C, and then cool at 10–15°C/min to room temperature.

Several glass end seals have been formed by using the system and procedure. The program was terminated before the technique could be fully developed, but the initial results were promising.

X.8.7 EXTENSION SHEATH

In the event that the plug-in electrical connection had to be made further than the 3-m length of the thermometer, an extension cable could be added to the thermometer by using the connection shown previously in Fig. X.8.2.

The extension cable has a 6.35-mm-OD (0.25-in.-OD) stainless steel sheath with four 0.762-mm-diam (0.030-in.-diam) copper wires, alumina insulated, with a glass end seal. The protruding copper lead wires are insulated with fiberglass sleeving compressed to a fraction of its normal length. The Nb-1%Zr wires are attached to the copper wires with crimp sleeves and soldered with tin solder. (The Nb-1%Zr wires are first wetted with a Ga-In-Sn alloy to allow soldering.) The compressed fiberglass sleeves are allowed to expand and provide electrical insulation to the wires after the crimp and solder joint.

Strength and a secondary gas seal are provided by a 6.35-mm-ID stainless steel sleeve with a compression seal on both ends. The sleeve is to be inserted over the sensor cold end before the lead wires are joined. The sleeve is then slipped into place, and the compression fittings are tightened to make a gas-tight seal. The sleeve serves to stiffen the sensor-to-lead wire connection and to provide a secondary gas seal.

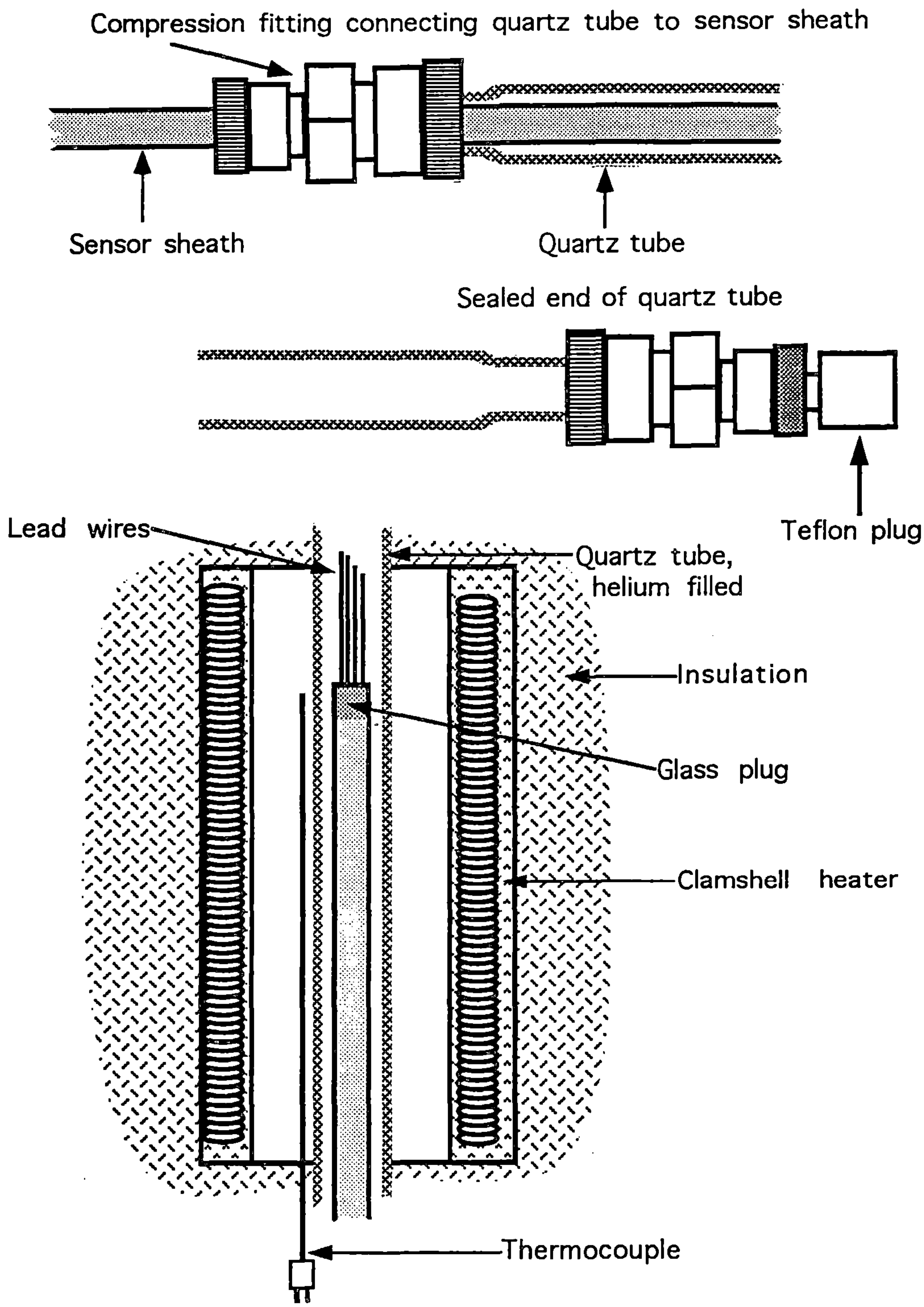
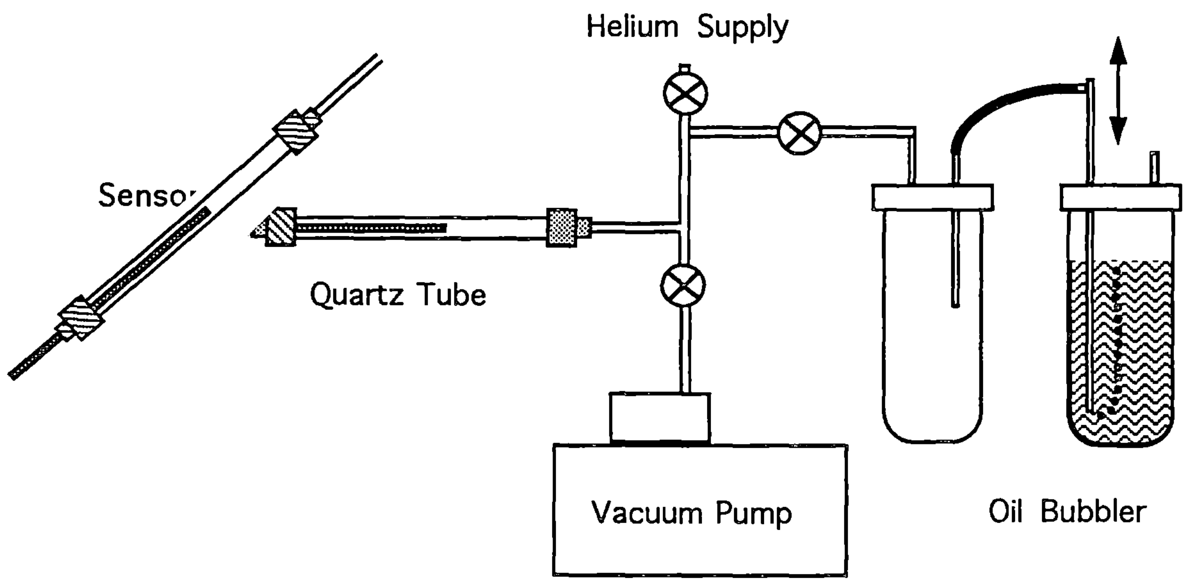


Fig. X.8.3. Glass seals are melted in place under a helium blanket.



Helium presssure in the quartz tube is regulated by raising or lowering the gas exit tube in the oil bubbler.

Fig. X.8.4. The oil bubbler controls the helium pressure in the quartz tube.

X.9. APPENDIX 9. COMPATIBILITY CAPSULE

X.9.1 CONSTRUCTION SEQUENCE

The sequence of the construction of the compatibility capsules was as follows.

1. The insulators were outgassed and stored under inert gas cover. (Note: The alumina was outgassed overnight at 800°C and 10^{-4} Pa (10^{-6} torr). The hafnia was outgassed 100 h at 1200°C and 10^{-3} Pa (10^{-5} torr). The ultrapure, fine-grain alumina was found to shrink and harden at 1200°C, so it had to be outgassed at a lower temperature.)

2. Sections of sheath were cut, acid cleaned, and stored in sealed containers.

3. Segments of the lead wire and sensing element wire were acid cleaned and stored in sealed containers. The 0.13-mm (0.0050-in.) Nb-1%Zr wire was cleaned with 50% nitric acid.

4. End plugs of Nb-1%Zr were fabricated to be press fits on the ends of the sheath segments (shown previously in Figs. 7 and X.3.1). It was important to the welding procedure that the plugs fit squarely and tightly. The end plugs were numbered, and these numbers were used to identify the test capsules and the prototype thermometers. One end plug, special to the capsules, had a vent hole to allow a final filling of helium .

5. The end plugs were welded into the sheath sections under an inert gas cover by using a laser welder shooting through a quartz window (see Fig. X.5.1, shown previously). Because the gas cover equipment was designed for the full-sized thermometer, the test capsule had to have a temporary extension tube attached to allow the capsule to be rotated under the laser beam (see Fig. X.5.2, shown previously).

6. This procedure is performed in the inert atmosphere in the glove box. The capsules were loaded under inert gas cover, and the vented end plug was inserted (but not welded) (see previously shown Fig. 8). The capsules were stored under inert gas cover. The contents of the compatibility capsules were as shown in Table X.9.1.

The sequence of closing the compatibility capsules is as follows:

7. The capsules had a vented plug laser-welded into the capsule end while under an inert gas blanket (see Figs. X.5.1 and X.5.2, shown previously). Capsule identification was maintained by a serial number etched on both the vented and unvented plug ends.

8. The capsules were wrapped in Nb-1%Zr foil and placed in a vacuum furnace. The capsules were outgassed (through the plug vent) at 10^{-4} Pa (10^{-6} torr) at room temperature for 24 h. Then, the furnace temperature was raised to 800°C, and the capsules were outgassed in a vacuum of 10^{-4} Pa (10^{-6} torr) for 96 h.

9. The furnace was back-filled with argon; while flooded with argon at a positive pressure, the furnace was opened; and a sealed jar filled with argon was placed in the furnace. While under the argon blanket, the jar was unsealed and the capsule container (made from Nb-1%Zr foil) was placed in the jar. The furnace was sealed again, evacuated at room temperature to 10^{-4} Pa (10^{-6} torr), and then filled with argon to a positive pressure. The furnace (a vertical furnace with a top lid so that argon would not tend to flow out) was opened, and the jar was sealed.

Table X.9.1. Compatibility capsule contents

Capsule	Insulator	Wire material	Form
32V51	Alumina	Nb-1%Zr	0.13-mm (0.005-in.) wire
33V3	Hafnia	W-25%Re	0.13-mm wire in 0.76-mm (0.030-in.) coil
35V13	Alumina	W-25%Re	0.13-mm wire in 0.76-mm coil
38V4	Hafnia	Nb-1%Zr	0.13-mm wire
41V28	Hafnia	Nb-1%Zr	0.13-mm wire
42V14	Alumina	W-25%Re	0.13-mm wire in 0.76-mm coil
43V6	Hafnia	W-25%Re	0.13-mm wire in 0.76-mm coil
45V5	Hafnia	Nb-1%Zr	0.13-mm wire
46V11	Alumina	Nb-1%Zr	0.13-mm wire
48V24	Alumina	Nb-1%Zr	0.13-mm wire
49V18	Hafnia	Nb-1%Zr	0.13-mm wire
50V17	Hafnia	Nb-1%Zr	0.13-mm wire
51V21	Hafnia	Nb-1%Zr	0.13-mm wire
56V23	Alumina	Nb-1%Zr	0.13-mm wire

10. The sealed jar was placed in a metal glove box which was evacuated overnight to 1.5×10^{-3} Pa (1.5×10^{-5} torr) and then back-filled with argon. The sealed jar was opened in the argon atmosphere, and the capsules were removed from the jar and the foil container. The glove box was then evacuated overnight to 3.2×10^{-4} Pa (3.2×10^{-6} torr) and filled with helium. The vent holes in the end plugs were sealed in the glove box by using tungsten inert gas (TIG) welding.

11. The capsules were tested for leaks of helium by placing them in a chamber connected to a helium leak detector calibrated with a 2.4×10^{-10} cc/s standard leak. No leaks were detected, proving that the laser welds on the end plugs and the TIG welding seal of the vent was successful.

12. The capsules were swaged along their full length to 5-mm (0.20-in.) diam. The solid plug end was about 0.03 mm (0.001 in.) larger in diameter than the end with the vented plug and the body of the capsule.

13. Dye penetrant tests, made after the capsules were swaged, showed an apparent circumferential crack where the sheath joined the end plug. Metallography showed that the apparent crack was just a cold lap that resulted from swaging the weld ring and did not constitute a flaw (shown previously in Fig. X.3.2). The dye penetrant tests serve no useful purpose and will be discontinued.

14. The capsules were again tested for helium leaks by placing them in a chamber connected to a helium leak detector calibrated with a 2.4×10^{-10} cc/s standard leak. No leaks were detected, proving that the swaging process did not crack the welds.

15. The capsules were then degreased and hydrofluoric acid cleaned to remove surface contamination. The capsules were placed in individual plastic envelopes, with the identifying number written on the envelopes.

X.9.2 COMPATIBILITY CAPSULE HEAT TREATMENT AND EXAMINATION

At Westinghouse Hanford Company (WHC), four of the helium-filled capsules were heated to 1100°C for 1009.3 h in a vacuum ranging from 10^{-4} to 10^{-5} Pa (1×10^{-6} to 1×10^{-7} torr). The capsules were shipped to ORNL, where they were sectioned and examined for structure damage. The examined regions were as shown in Table X.9.2.

Table X.9.2. Examination of compatibility capsules

Capsule	Insulator sample	Wire and sheath material, 2 mounts
38V4	Hafnia	Nb-1%Zr, longitudinal and transverse
41V28	Hafnia	Nb-1%Zr, longitudinal and transverse
32V51	Alumina	Nb-1%Zr, longitudinal and transverse
35V13	Alumina	W-25%Re, 0.13-mm (0.005-in.) wire in 0.76-mm (0.030-in.) coil
Nb-1%Zr	Sheath	Longitudinal and transverse sections

Metallographic examination of the wires and sheath showed no evidence of reaction with any components of the capsules.

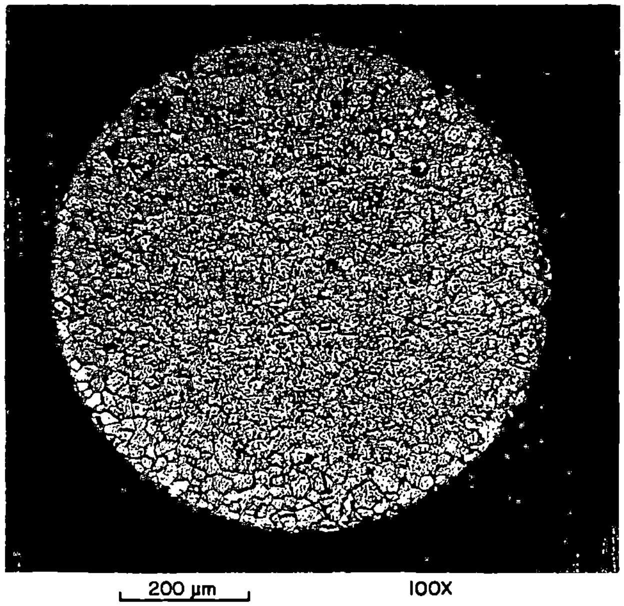
Microprobe analysis of the wire and inside sheath surfaces showed no gradients of oxygen, aluminum, or hafnium concentration between the surface of the wire or sheath and the interior.

The result from the microprobe supported the conclusions from the examination of the microstructure. Namely, there was no indication of insulator diffusion of or fill gas components into either the wire or the sheath.

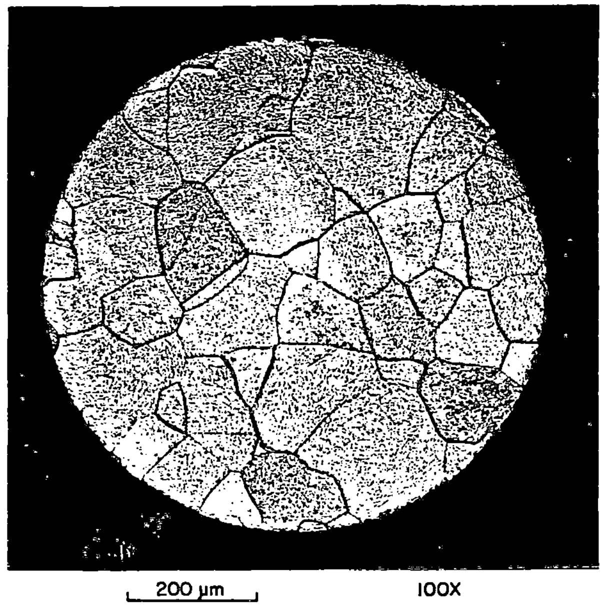
The complete absence of any deterioration of the capsule components constitutes proof that the handling techniques that were employed in constructing the compatibility capsules are also suitable for constructing the thermometer.

X.9.3 SENSOR WIRE GRAIN STRUCTURE

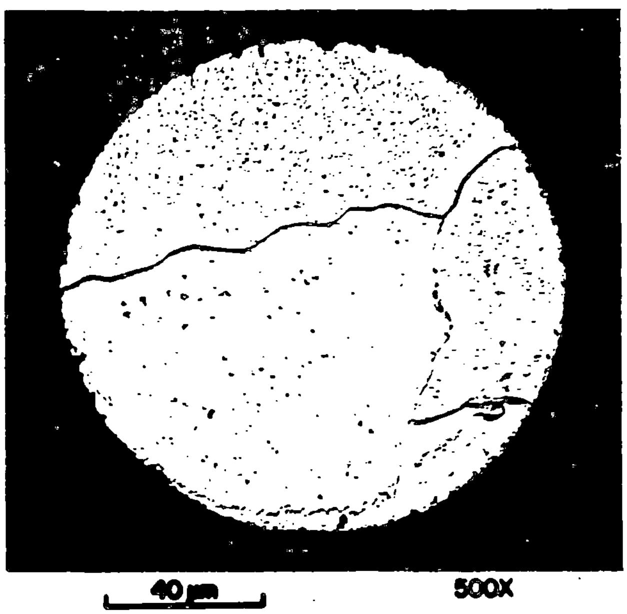
Representative samples of 0.76-mm-OD (0.030-in.-OD) pure niobium wire and 0.25-mm-OD (0.010-in.-OD) Nb-1%Zr wire were heated at 1100°C for 100 h in vacuum, and the cross sections of the wire were examined for grain structure. The metallurgical photographs of the samples (a) the as-received condition, (b) the condition after heating, and (c) the condition when first coiled and then heated were compared and are shown in Figs. X.9.1 and X.9.2. It is apparent that the trace amount of zirconium in the Nb-1%Zr wire inhibited the growth of grains, particularly in the wire that was first coiled and then heated, as it would be in the operation of the temperature sensor. The slight amount of cold work introduced in the dead-soft annealed pure niobium wire promoted extensive grain growth as shown in Fig. X.9.1.



a. 0.76-mm-diam pure niobium wire, as received (100 X).

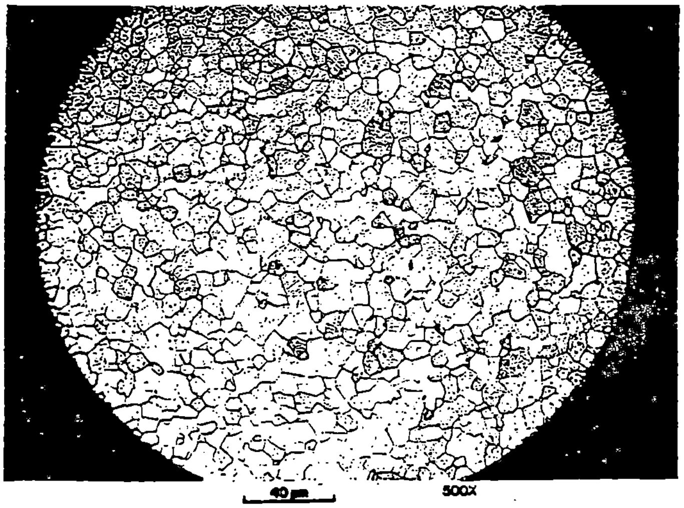


b. 0.76-mm-diam pure niobium wire heated at 1100°C for 100 h (100 X).

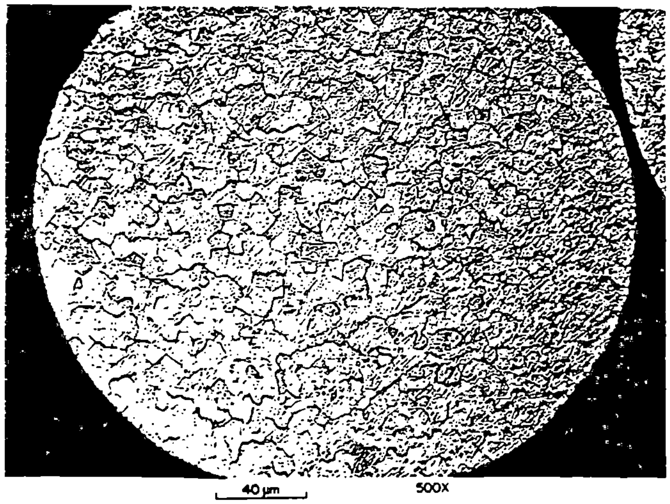


c. 0.76-mm-diam pure niobium wire, coiled and then heated at 1100°C for 100 h (500 X).

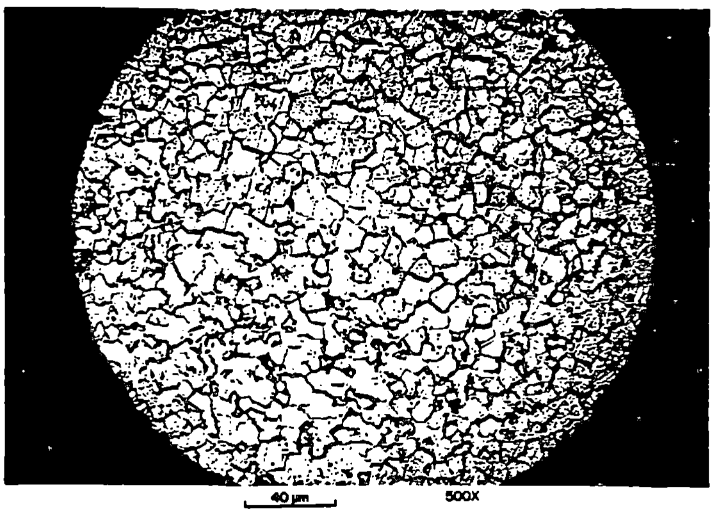
Fig. X.9.1. Grain growth in pure niobium wire.



a. 0.25-mm-diam Nb-1%Zr wire as received (500 X).



b. 0.25-mm-diam Nb-1%Zr wire heated at 1100°C for 100 h (500 X).



c. 0.25-mm-diam Nb-1%Zr wire, coiled and then heated at 1100°C for 100 h (500 X).

Fig. X.9.2. Grain growth in niobium + 1% zirconium wire.

X.10. APPENDIX 10. JOHNSON NOISE POWER PRINCIPLE

A prototype Johnson noise thermometer (JNT) tuned-circuit signal processor was developed and tested. The single-ended (one-side-grounded) preamplifier previously used was replaced with a differential (both-sides-floating) input, which has markedly reduced the susceptibility of the signal processor to noise pickup. Simultaneous dc resistance and Johnson noise measurements with a single sensor were demonstrated at room temperature and were confirmed at high temperatures where insulator shunting occurs.

Effects of insulation resistance on the accuracy of the JNT and on its susceptibility to electromagnetic interference (EMI) were analyzed, and the results indicate that the insulator resistance may decrease to as low as 1000 Ω at the operating temperatures. This reduction may not affect the noise temperature measurement significantly, if the temperature distribution along the sensor increases monotonically, approximately following the design proposed for the SP-100 reactor. The measured dc resistance value of the sensing element would, of course, be affected by shunting at a 1000-Ω insulation resistance, but the resistance thermometer error could be corrected by noise measurements.

Some attention needs to be directed at the sensor lead resistance, which can introduce a significant noise temperature error. A lower lead resistance reduces the error from the leads, which is the rationale for using the largest possible lead wires and combining the four leads of the resistance thermometer system to make a low-resistance, two-lead system for the JNT measurements. The sensor lead resistance can be measured at any time by using the dc resistance thermometer data.

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