Fabrication of a Guarded-Hot-Plate Apparatus for Use Over an Extended Temperature Range and in a Controlled Gas Atmosphere

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ABSTRACT

This paper describes a guarded-hot-plate apparatus suitable for determining the steady-state thermal transmission properties of insulation specimens from 90 K to 900 K in a controlled gas atmosphere from 10^{-5} kPa (10^{-4} torr) to 105 kPa (790 torr). The apparatus accommodates flat-slab specimens 500 mm in diameter and from 13 mm to 100 mm in thickness. Fabrication and assembly details are presented for the apparatus hot and cold plates, guard system surrounding the plates, temperature sensors, metal-sheathed heaters, and vacuum/gas pressurization system. An initial assessment of the calibration uncertainties for the primary measurement sensors is presented.

INTRODUCTION

Laboratory comparisons [1-3] of high-temperature guarded-hot-plate apparatus have revealed a high level of scatter, on the order of ±20 %, in the test data of different insulation materials. Further, these studies have found that the level of variability increases as a function of temperature. In particular, the study conducted under the auspices of the ASTM C16.30 Subcommittee on Thermal Measurement [3] recommended that the National Institute of Standards and Technology (NIST) develop a high temperature testing capability for the purpose of developing reference materials. As a result of these studies, NIST has initiated a program to design and fabricate a 500 mm diameter guarded-hot-plate apparatus for the eventual development of thermal insulation reference materials for greater temperature and pressure ranges than are currently available.

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The new apparatus builds on the concept of using line-heat-sources in circular metal plates. The concept was first proposed by Robinson [4] in 1964 and later implemented by Hahn, Powell, Siu, and others [5-8] at the National Bureau of Standards (now NIST). The design approach for the new apparatus has been summarized previously by Flynn et al. [9] and includes mineral-insulated, metal-sheathed heaters that have been vacuum brazed into a metal guarded hot plate. The locations of these heaters are based, in part, on ASTM guidelines [10, 11] and optimized by finite-element analyses conducted by Healy and Flynn [12]. The apparatus has been designed to meet the current requirements of ASTM and ISO standard test methods for guarded-hot-plate apparatus [13, 14].

APPARATUS DESCRIPTION

The NIST 500 mm guarded-hot-plate apparatus has been designed primarily for thermal measurements on industrial insulation materials having thicknesses of 13 mm to 100 mm. The plate size of 500 mm was selected based on current recommendations in ISO 8302 [14]. Figure 1 illustrates the main components of the test facility. The apparatus is enclosed by a vacuum chamber for testing in controlled atmospheres at different pressures. To facilitate installation of test specimens, the bell jar is removable by means of a wheeled support cart.

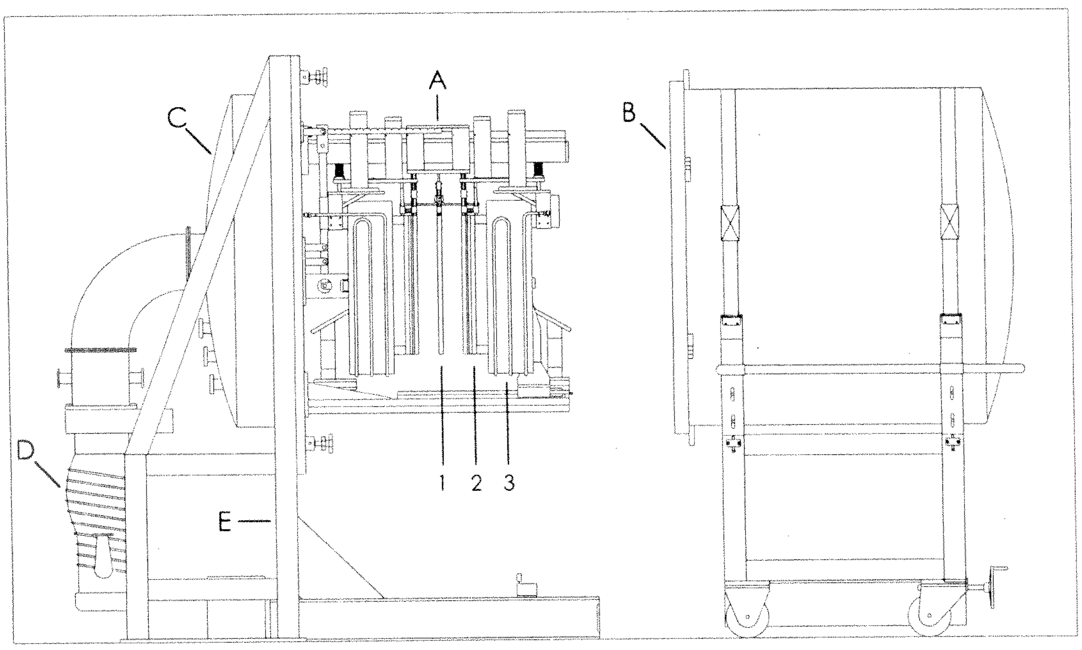

FIGURE 1. NIST 500 mm Guarded-Hot-Plate Facility: A – apparatus hot plate (1), cold plates (2), edge guards (3), thickness system, force application, and structural support; B – bell jar (and removable support cart); C – vertical baseplate weldment; D – diffusion pump; E – aluminum support frame superstructure (bolted to laboratory floor).

The structural support for the plates, guards, thickness measurement system, and force application system are bolted to a vertical baseplate weldment, which is bolted to the support frame superstructure. The plates and edge guards are suspended vertically from an overhead rail system and can translate independently of each other in the horizontal direction using pillow-block bearings mounted on precision shafts. The outermost layer of the apparatus edge guard has a water jacket to maintain exposed surfaces at room temperature.

Figure 2 illustrates a close-up of the plates (without edge guards), thickness measurement system, force application system, and structural support. In the two-sided mode of operation, a pair of specimens is placed between the hot plate and cold plates and clamped to maintain specimen thickness. The clamping force is transmitted axially to the inboard cold plate (nearest to the vacuum baseplate) by a lever mechanism connected to a hanging beam scale. A dead weight(s) of known mass is suspended from the V-notches of the beam scale providing a fixed axial clamping load that is measured by a strain-gage load cell. The specimen thickness is measured at the centerline of the apparatus plates using quartz rods in contact with the back surfaces of the cold (thermometry) plates (see Figure 4, below) and aligned with adjoining high-accuracy length transducers. The length transducers are clamped to stands manufactured from an iron-nickel (36 %) alloy, UNS[1] K93601. The length stands are removable to facilitate specimen installation and are re-positioned on pedestals by means of precision kinematic coupling mounts.

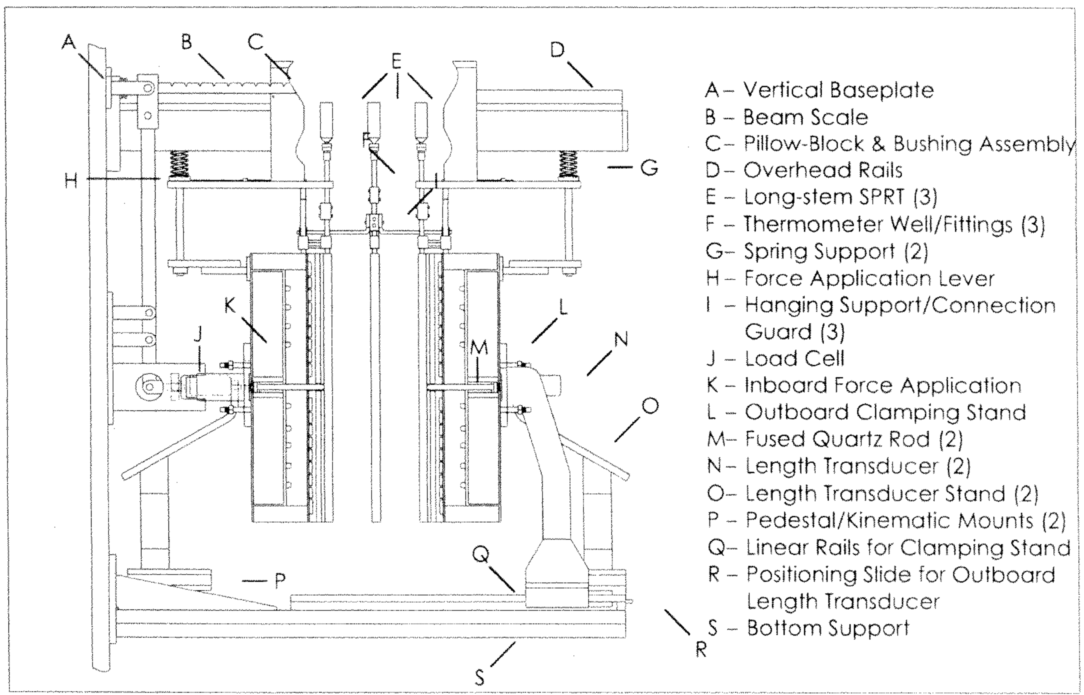

FIGURE 2. Side view of the apparatus plates (without edge guards), thickness system, force application, and structural support. Cold plate assemblies are shown in cross section view.

[1] Unified Numbering System for Metals and Alloys K93601 has a low thermal expansion coefficient (on the order of 1.6×10^{-6} /K near ambient temperature).

All of the apparatus plates, except the exterior water-jacket plates, have been manufactured from commercially pure nickel (99.6 % minimum). Nickel was selected primarily because of its good oxidation resistance at elevated temperatures. The plates in contact with the specimens have thermometer wells brazed in their respective centerplanes for insertion of long-stem standard platinum resistance thermometers (SPRTs). To minimize radial heat flow, the circular plates are surrounded by a multi-layer edge guard (described later). A U-shaped opening at the top of the edge guard provides access for translational movement of the plate suspension system. To compensate for the absent portion of the edge guard, a connection-guard system (Figure 2) has been brazed above each thermometry plate to minimize conductive heat flows along the thermometer well and metal-sheathed components. If necessary, additional guarding is provided for each thermometry plate by a metal-sheathed heater brazed in a groove around the plate perimeter.

Hot Plate

As illustrated in Figure 3, the hot plate consists of a meter plate (central disk) 198.6 mm in diameter, a 500 mm diameter guard ring (primary guard), and a secondary guard system consisting of a temperature controlled connection guard and metal-sheathed perimeter heater. The ratio of guard to meter radii is 2.5. The plate and secondary guards are made of commercially pure nickel (UNS[2] N02201) vacuum brazed (described later) to form a solid metal plate nominally 16 mm thick. The width of the guard gap at the plate surface is 1.4 mm and, to minimize lateral heat flow, the gap profile is diamond shaped (having an 11.4 mm diagonal dimension). The meter plate is supported mechanically by three inserts brazed 120° apart and manufactured from a high-strength nickel-chromium-cobalt alloy (UNS N07001).

The primary temperature measurement for the guarded hot plate is a long-stem SPRT inserted in a thermometer well brazed at the centerplane of the plate. The grooves for the meter plate and guard ring heaters are also located at the centerplane and have been cut in precision circular meander patterns (Figure 3). The four-fold and five-fold designs for the meter plate and guard ring heaters, respectively, are based on finite-element analyses that minimized temperature variation (40 mK) across the plate surfaces [12]. The heater grooves adjoining the SPRT are designed to minimize the temperature variation (less than 10 mK) in the immediate vicinity of the SPRT [12]. Metal-sheathed heaters, thermocouples, and thermopiles were selected for this application because their rugged characteristics are well suited for the high temperatures required for manufacture (especially during vacuum brazing) and for the intended temperature and vacuum conditions anticipated in operation. Note that the meter plate heater has a special 2-to-4 wire connection at the guard gap (Figure 3), providing voltage taps for measuring the voltage drop of the meter plate heater.

To control undesired lateral heat flows across the guard gap, metal-sheathed thermopiles have been pressed in mating grooves on both surfaces of the hot plate. The grooves are cut in a zigzag pattern (Figure 3) and cross the guard gap every 30°.

[2] N02201, commonly known as nickel 201, a low carbon (0.02 % maximum) nickel alloy.

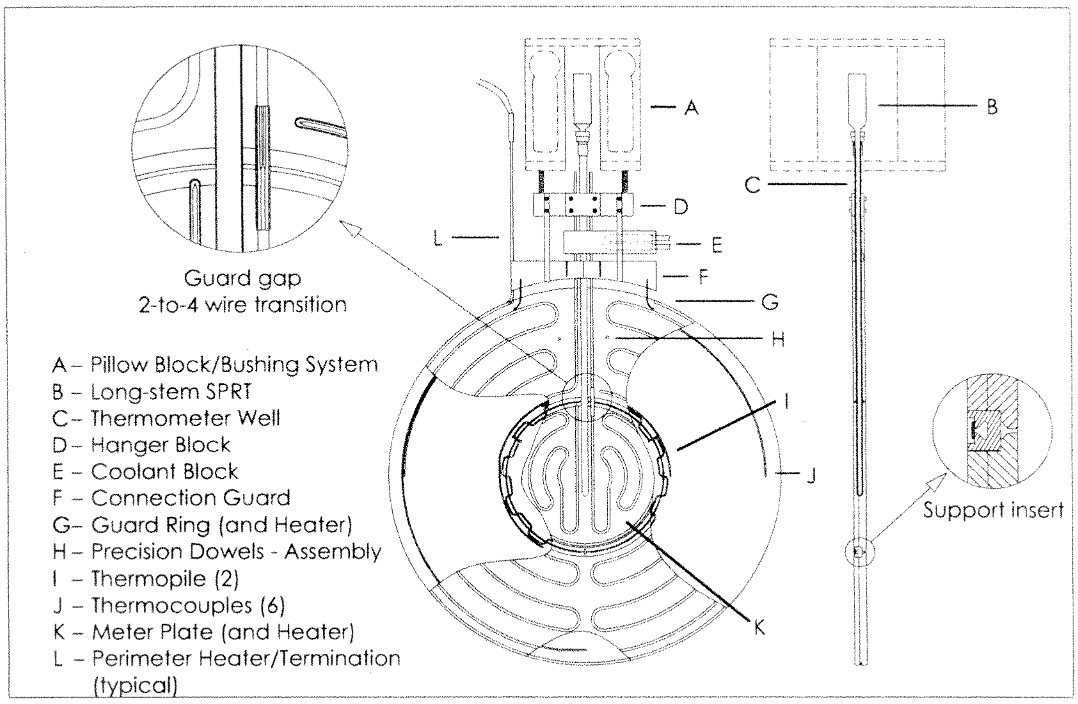

FIGURE 3. Cutaway view of hot plate illustrates the internal thermometer well and heaters located at the mid-plane of the meter plate and guard ring and the thermopile and 6 thermocouples near the surface of the plate. Close-up schematic (left) shows heater and voltage tap wires (2-to-4 junction) for the meter plate and close-up (right) illustrates support insert for meter plate in guard gap. Exit paths for metal-sheathed thermocouples and thermopiles from the plate are not shown.

The inner and outer diameters of the thermopile are 5.2 mm from the centerline of the guard gap (100 mm radius). Each thermopile has 12 pairs of alternating Type K elements[3] junctions (KP: nickel-chromium versus KN: nickel-aluminum-silicon). The sensitivity of the two thermopiles when connected in series is approximately 980 μV/K at room temperature, providing control capability of roughly 1 mK for a digital voltmeter capable of resolving 1 μV. Additional (secondary) temperature measurements are provided by six Type N metal-sheathed thermocouples brazed in grooves on one face of the guard ring. The inner 3 thermocouples (108 mm radius) are 120° apart, and the outer thermocouples (231.3 mm radius) are 90° apart on the lower half of the plate. The outer three thermocouples are intended as control sensors for the perimeter guard heater.

Long-Stem Standard Platinum Resistance Thermometers

Standard platinum resistance thermometers (SPRTs) are defining instruments of the International Temperature Scale of 1990 [15]. The long-stem SPRT (Figures 2

[3] The original thermopiles that were brazed in the hot plate were a Type KP/gold-palladium alloy construction selected for its high temperature sensitivity and having an outer sheath diameter of 1 mm [9]. For unknown reasons, both of the original thermopiles, unfortunately, did not survive manufacture. The original thermopiles were subsequently removed by cutting from the plate and replaced by (pressing in grooves) the Type K thermopiles described above.

and 3) has a 4-wire sensing element near the tip that is 50 mm in length and is manufactured from annealed platinum wire. The wire sensing element is non-inductively wound in a bird-cage design and the nominal resistance of the SPRTs is 25.5 Ω at 273.16 K. The sensor is housed in a nickel-chromium-iron alloy (UNS N06600) sheath, 5.6 mm in diameter and 457 mm in length (excluding the termination housing). As described above, the long-stem SPRTs are inserted into nickel-chromium-iron alloy tubes that have been brazed in the centerplanes of the hot and cold thermometry plates so that the 50 mm sensing portion is bisected by the center of the plate. After insertion in the thermometer well, the SPRTs are sealed and, if necessary, the well is pressurized with either air or helium gas.

Metal-Sheathed Heaters, Thermocouples, Thermopiles

The heaters, thermocouples, and thermopiles are mineral insulated, metal-sheathed cables manufactured by a cold draw and anneal process and, later, formed using templates having precision-cut grooves machined by the NIST Fabrication Technology Division. The sheath tube is a nickel-chromium-iron alloy (UNS N06600) and the internal wires in the cable are electrically isolated by high-purity magnesium oxide (MgO) insulation, which is compacted around the wires during the draw process. Because the MgO insulation is hygroscopic, the open cable end is temporarily welded. Subsequent electrical connections are sealed in an epoxy potting compound at a curing temperature near 100 °C.

The metal-sheathed heaters for the hot plate were 3.2 mm in diameter and custom manufactured using an internal bifilar design of nickel 201 wires butt-welded to gold (99.99 % nominal purity) wire leads. The heaters were formed such that the nickel-gold weld terminations were within 3 mm (or less) of the plate edge and the 2-to-4 transition of the meter-plate heater was within 1 mm of the centerline of the guard gap (Figure 3). These locations were later verified by radiographic images taken by the NIST Ionizing Radiation Division.

The metal-sheathed thermocouples [16] for the hot plate were 1.6 mm in diameter and were Type N construction (NP: nickel-chromium-silicon versus NN: nickel-silicon). The two metal-sheathed thermopiles (1.6 mm in diameter) were custom manufactured using a co-axial design of alternating segments of Type K construction. The wire segments were butt-welded together, strung with single-bore MgO pellets, drawn, annealed, and formed in the pattern shown in Figure 3 using a template.

Fabrication

Fabricating the hot plate required the following steps: 1) component fabrication; 2) assembly of components by vacuum brazing; 3) surface machining to remove excess braze alloy and control dimensional variations introduced in brazing; 4) application of a high-emittance coating (both faces); and, 5) installation of electrical terminations. Steps 1) and 3) were performed by NIST; the other steps required external vendor services. Unfortunately, the failure of the original thermopiles[3] required two additional steps – removal of the original thermopiles and installation of replacements by press-fitting.

The major components of the hot plate – the base plate and cover – were fabricated on a high-speed, horizontal-spindle, numerically-controlled milling machine. Precision locating (nickel 201) dowels and threaded holes were placed in both components for assembly. Grooves for the metal-sheathed heaters, thermocouples, thermopiles, and thermometer tube were cut in the base plate with clearance tolerances ≤ 0.05 mm. The locations of the meter plate and guard-ring heater grooves were verified with a coordinate measuring machine (CMM) to confirm location agreement with finite-element analyses [12]. The (guard) gap, separating the meter plate and guard ring, was cut using electric-discharge machining and the diamond-shape profile was cut in a lathe. The assembled plate thickness was oversized by 0.5 mm (0.25 mm per face) to allow for dimensional changes anticipated due to vacuum brazing (described below).

The hot-plate components were assembled and subsequently joined by brazing in a vacuum furnace using a nickel-phosphorus (11 %) eutectic alloy. Prior to brazing, the metal-sheathed cables were grit blasted to remove oxidation and methodically "staked" in their respective grooves. The measured braze filler metal was applied (by syringe) as a water-base paste to the grooves and to interface joints between the base plate and cover. A water-base ceramic paste was applied, as needed, to prevent unwanted braze alloy from flowing into the guard gap and other areas. The assembly was placed in a furnace, evacuated $< 1.3\times10^{-5}$ kPa (10^{-4} torr), and heated to 920 °C for about 1.5 h. At the joining temperature, the liquid braze alloy is drawn into clearance joints by capillary action and diffuses in the solid nickel matrix. During vacuum cooling, the new braze alloy solidifies, providing a metallurgical bond for good heat transfer between the heater and metal plate.

The brazing vendor was directed to overfill all external surface grooves with braze filler metal 0.25 mm above the surface of the nickel substrate. After brazing, the plate surfaces were ground with segmented aluminum-oxide abrasives to remove excess braze alloy and establish a smooth, flat plate surface. Grinding to a desired surface condition was accomplished by the complete removal of the old surface on one face. The distance from the ground surface to the thermometer well was measured and the process repeated by grinding the other surface to the same dimension (i.e., equal distance from each surface to the thermometer well). The total thickness of material removed by grinding was 0.69 mm.

After grinding, a ceramic coating ($Cr_2O_3 - SiO_2$ blend), approximately 0.05 mm to 0.08 mm thick, was applied to each plate surface by a vendor to obtain a high emittance. The surfaces were prepared by blasting at low pressure with fine alumina grit. The initial base coating was spray-applied as a water-base slurry, air-dried, and fired at 510 °C (950 °F) creating a ceramic-to-metal bond (i.e., $NiO{\cdot}SiO_2$-Cr_2O_3 spinel). The open-pore structure of the coating was gradually filled by repeated applications of a Cr_2O_3 solution at room temperature followed by heat treatment at 510 °C (950 °F). The densification process required 14 applications and 2 sequences of polishing. The finished surface roughness was about 0.38 μm (15 μin.) and the surface emittance ranged from 0.80 to 0.81 as measured by a commercial infrared reflectometer at room temperature.

Cold Plate Assembly

As shown in Figure 4, the primary components are a sandwich of metal plates and layered insulation, consisting of: 1) thermometry plate; 2) 0.5 mm woven S-glass[4] fabric (not shown); 3) heater plate; 4) 9.5 mm flexible microporous thermal insulation enclosed in quilted woven S-glass; 5) coolant plate; 6) 38 mm rigid alumina insulation; and, 7) water-cooled back plate. These metal layers are mechanically connected and translate on the overhead rails as an integrated unit. At temperatures near or below ambient, liquid ethanol (or for low temperature, liquid nitrogen) is circulated through the coolant plate. Temperature control of the thermometry plate surface is maintained by the heater plate. The primary temperature measurements for the thermometry plates are long-stem SPRTs inserted in thermometer wells brazed at the centerplane of each plate. The working surface of the thermometry plate has been coated with a ceramic coating (Cr_2O_3 – SiO_2 blend) having an emittance of 0.81. Each thermometry plate has a perimeter cable heater and also a connection guard to minimize conductive heat flows due to the thermometer well and metal-sheathed cables.

Edge Guard Assembly

Figure 5 shows the following primary components of the edge guard assembly: 1) 10 mm air space that can be filled with insulation, if necessary; 2) heater ring (nickel 201); 3) and 5) microporous thermal insulation enclosed in quilted woven S-glass; 4) coolant ring (nickel 201); and, 6) water jacket (copper plated with electroless nickel). The edge guard assemblies are suspended from the overhead rail system and translate in tandem with their respective cold plate assemblies. Each

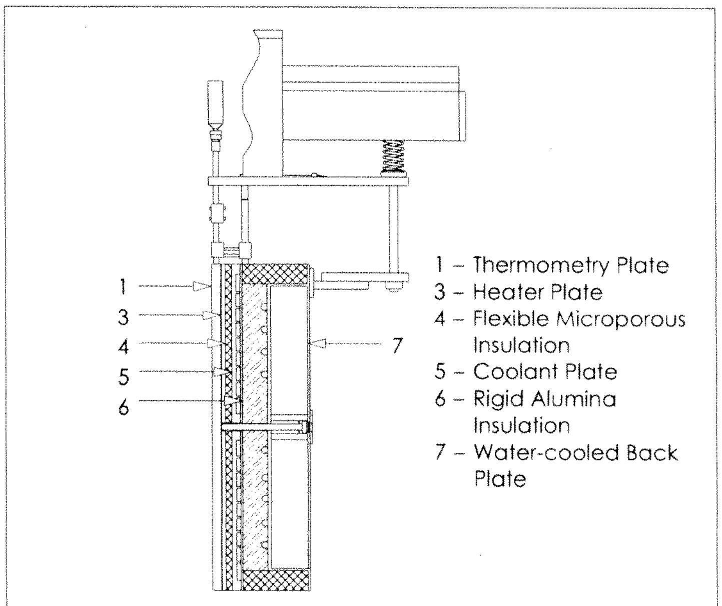

FIGURE 4. Side view of a cold-plate assembly (section view).

[4] Family of magnesium-alumina-silicate glasses.

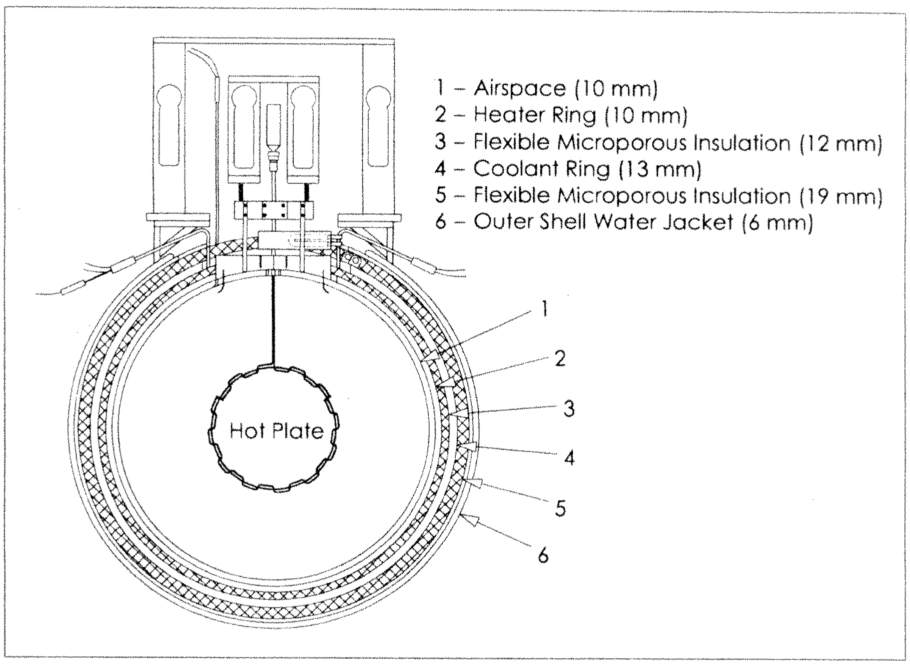

FIGURE 5. End view of cylindrical edge guard encompassing hot plate. Nominal thicknesses are given for each layer.

guard is an integrated assembly providing cooling and/or heating to maintain the internal surface of the guard near the mean temperature of the test specimens, thereby promoting one-dimensional heat flow through the meter plate test section [17].

Vacuum Chamber and Equipment

The vacuum vessel (constructed of Type 304 stainless steel) consists of a vertical baseplate weldment and a horizontal bell jar supported on a movable cart (Figure 1). The weldment, which has been shimmed and bolted to a support frame, consists of a vertical baseplate (32 mm × 1370 mm × 1520 mm), a right circular cylinder (1170 mm diameter by 390 mm), and a torispherical dome. A 200 mm knife-edge flange for right elbow and diffusion pump is located at the centerline of the dome and fourteen 70 mm knife-edge flanges for fluid and electrical feedthroughs are located in the lower portion of the dome.

The bell jar is a flanged (with captive O-ring) right circular cylinder (1190 mm diameter by 1240 mm) with a torispherical dome. In operation, the movable bell jar is aligned with the vertical baseplate using a floor-level guide rail and fastened to the baseplate with six hold-down clamps located 60° apart (Figure 1). After delivery, the helium leakage rate of the bell jar seal was found to be approximately 2×10^{-8} standard cm^3/s (as determined by the NIST Vacuum and Pressure Group).

CALIBRATION UNCERTAINTIES

For the primary measurements of temperature, power, meter area, and thickness, considerable advice and calibrations were provided by the NIST laboratories responsible for realizations of *The International System of Units (SI)*. The primary temperature sensors – long-stem SPRTs – have been calibrated by the NIST Thermometry Group with an AC bridge at 30 Hz and measuring currents of 1.0 mA and 1.414 mA at the following points: Ar, Hg, H_2O, Sn, Zn, and Al. These triple points and fixed points cover a temperature range of 83.8058 K to 933.473 K (-189.3442 °C to 660.323 °C). In the worst case, at the freezing point of Al, the expanded uncertainty (coverage factor of $k = 2$) is 0.64 mK.

Three ancillary metal-sheathed Type N thermocouples from the same lot as those brazed in the apparatus components were calibrated by the NIST Thermometry Group. The thermocouples were heated approximately 15 min to 120 °C and held for 2 h and then heated to 925 °C (i.e., maximum temperature during brazing) for approximately 30 min. From 0 °C to −195.7 °C, the thermocouples were calibrated by comparison with a calibrated SPRT and from 0 °C to 700 °C the thermocouples were calibrated by comparison with a calibrated Type S thermocouple. The expanded uncertainties ($k = 2$) were 0.004 mV and 0.030 mV for measurements < 0 °C and > 0 °C, respectively.

The electric power to the meter plate is determined by the product of the voltage drop of the meter heater (measured at the 2-to-4 junction described above) and the current. For determining the current, a precision 0.1 Ω standard resistor was calibrated at 25 °C by the NIST Electricity Division by comparison with working standards calibrated in terms of the Quantum Hall effect used as the U.S. representation of the ohm. The expanded uncertainties ($k = 2$) of the calibration at test currents of 0.316 A and 1 A are 2×10^{-6} Ω, respectively.

The meter area is defined as the summation of the surface areas (normal to heat flow) of the meter-plate and one-half the guard gap. The dimensions of the meter plate diameter and inner diameter of the guard ring were determined by dimensional data sampled around the gap circumference using an extremely accurate coordinate measurement machine [18].

The length transducers for determining specimen thickness (Figure 3) are set to known positions using a system of three coplanar precision metal spheres situated in a fixture 120° apart at a radius of 95 mm. The fixture is suspended from the overhead rails and the three spheres provide precision kinematic coupling between the hot plate and a cold plate (i.e., a known offset distance). Four sets of precision metal spheres, having nominal diameters of 13 mm, 25 mm, 38 mm, and 50 mm, were calibrated by a mechanical calibrator retrofitted with a laser interferometer [19].

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