Fabrication of High-Value Standard Resistors

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***Abstract*—The National Institute of Standards and Technology (NIST) has fabricated stable, transportable 10 MΩ and 1 GΩ standard resistors for use in an international comparison of high resistances. This fabrication process is being applied to the construction of standard resistors of values up to 10 TΩ, with initial results indicating significant improvements in stability and fewer adverse effects induced by mechanical shock and vibration.**

***Index Terms*— Film-type resistors, resistance, resistance measurements, resistors, standards, wirewound resistors.**

I. Introduction

THE highest quality standard resistors are usually the wirewound type, constructed with wire made from special high-resistivity alloys [1]. The highest value commercially available wirewound resistor is a 100 MΩ unit. It is impractical to construct wirewound resistors of higher values because of the limiting maximum resistivity and minimum wire diameter available with these special wire alloys. For example, the length of wire needed to construct a 100 MΩ unit is approximately 18.7 km. Consequently, single standard resistors above a 100 MΩ are constructed using film-type resistance elements. Materials in film form have higher resistivities than the wire alloys because of the additional resistivity due to scattering of the conduction electrons at the boundary of the film [2].

Generally, film-type resistors are not as stable as wirewound resistors, and the film types exhibit higher temperature coefficients of resistance (TCR's) and voltage coefficients of resistance (VCR's). If the film resistance elements are not hermetically sealed, moisture can produce two reversible effects: resistance decrease due to surface leakage across the element, and resistance increase caused by the swelling of the protective insulator, resulting in pressure being exerted on the resistance element. Also, mechanical shock and vibration can cause resistance shifts and instabilities in film-type resistors. NIST has developed a fabrication process to alleviate or control some of these problems with high-value standard resistors.

Using this process, NIST, as the pilot laboratory, has constructed several 10 MΩ and 1 GΩ standard resistors for use in an international comparison of high resistances. Repeated measurements of these transport standards after traveling to various countries indicate that these standards have improved stabilities and are less susceptible to changes of resistance induced by mechanical shock and vibration than standard commercially available units. These encouraging results have led to the production of additional film-type resistance standards of nominal values 1 GΩ, 10 GΩ, 1 TΩ, and 10 TΩ using the same or a slightly modified fabrication process.

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II. Fabrication Process

This section describes the NIST fabrication process for the different levels of resistance. The 10 MΩ wirewound resistance elements obtained commercially were hermetically sealed and were not subjected to heat treatment. All film-type resistance elements were heat treated and hermetically sealed in specially designed containers. The sizes of the containers varied with the physical dimensions of the resistance elements.

A. Transport Standards

The resistance elements of the 1 GΩ transport standards consist of precious-metal-oxide (PMO) film resistors that are commercially available. To improve their stabilities, these PMO film resistors are pre-aged by external heating [3]. This is accomplished by heat treating the resistors in an air oven at about 125 °C for over 100 h. Either a single resistor or a series/parallel network of four resistors are mounted in a brass cylinder (15 cm long $\times\, 3$ cm OD) with a wall thickness of 6.35 mm. The resistance element is mounted in the brass cylinder using glass-to-metal seals that are soldered on brass end plates as shown in the photograph of Fig. 1. The container is sturdy, so that any variations of atmospheric pressure do not affect the value of the internal resistance element.

To reduce the number of dissimilar metal junctions, which aids in diminishing thermoelectric effects, the resistance element leads pass through center tubes of the glass-to-metal seals prior to being sealed in place with solder. Also soldered on the brass end plates are soft copper tubes that are used to purge the resistor can with dry nitrogen gas. After mounting the resistance element and before purging, the assembly is heated in an air oven at about 100 °C for approximately 4 h to completely dry out the internal components of the container. Immediately upon removal from the oven, the resistor can is purged with dry nitrogen gas, allowed to cool to room temperature, and then hermetically sealed by crimping and soldering the ends of the soft copper tubes. The resistor container assembly is mounted in an aluminum enclosure, as shown in the photograph of Fig. 2, to provide increased electrostatic shielding. The assembly is shock-mounted in the enclosure using two isolation pads made of a highly damped visco-elastic material, which is electrically insulated from the brass container by polytetrafluoroethylene (PTFE)

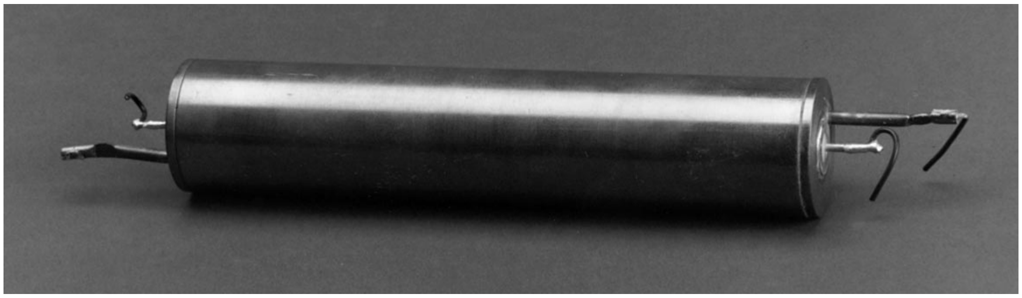

Fig. 1. Hermetically-sealed resistor container assembly with glass-to-metal seals and copper purging tubes soldered to end plates.

Fig. 2. Completed 1 GΩ standard resistor with coaxial terminals mounted on circular PTFE plates.

tape. The resistor terminations are coaxial connectors mounted on grooved PTFE circular plates on the top panel of the enclosure. The grooves in the PTFE plates extend the surface leakage path between the aluminum enclosure and the coaxial connectors. The resistor container is electrically isolated from the enclosure and electrically connected to the shield of one of the coaxial connectors. This allows the resistor container to be operated either in a floating mode, a grounded mode, or to be driven at a guard potential. A calibrated thermistor, terminated to a two-conductor shielded connector on the top panel, is mounted on the resistor can to monitor its temperature.

The resistance elements of the 10 MΩ transport standards consist of wirewound, hermetically sealed resistors that are commercially available. In contrast to the PMO film resistors, there was no need to heat treat these wirewound resistors to improve upon their stabilities. The 10 MΩ resistance elements were also shock mounted in aluminum enclosures in a similar fashion as described above for the 1 GΩ standards.

B. 1 GΩ and 10 GΩ Resistors

Ten 1 GΩ and ten 10 GΩ resistance units were completed and have been used as the main elements of a 10×1 GΩ and a 10×10 GΩ guarded resistance transfer standard, respectively, for establishing accurate 10:1 and 100:1 resistance ratios. These PMO film-type resistors were heat treated and sealed in similar fashion as the elements of the 1 GΩ transport standards described above. However, smaller diameter brass cylindrical containers (17 cm $\times$ 1.9 cm OD) were used for these two sets of resistors for compactness, and to eliminate the need for brass end plates. The glass-to-metal seals form the end plates; this necessitated that the soft copper purging tubes be located along the perimeter of the brass cylinder and near each end. No special shock mounting of the completed resistance assembly was employed, since these transfer standards were designed to be used *in situ* and not transported.

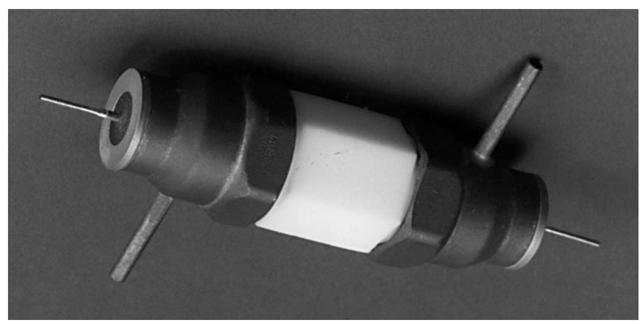

Fig. 3. Metal-insulator-metal resistor container of end copper fittings attached to middle PTFE sleeve.

C. 1 TΩ and 10 TΩ Resistors

These film-type resistors were heat treated and hermetically sealed similarly to the process used for the 1 GΩ transport resistance elements. A main difference in the process of fabricating these resistance standards was sealing the elements in a metal-insulator-metal container as shown in the photograph of Fig. 3. The specially designed container consists of two threaded (NPT-1/2) copper pipe fittings attached to either end of a threaded PTFE sleeve. Prior to assembly, glass-to-metal seals are soft-soldered to the ends of the copper fittings and a copper purging tube is hard-soldered along the perimeter of each fitting. This metal-insulator-metal container design allows the copper fittings to be driven at separate guard potentials

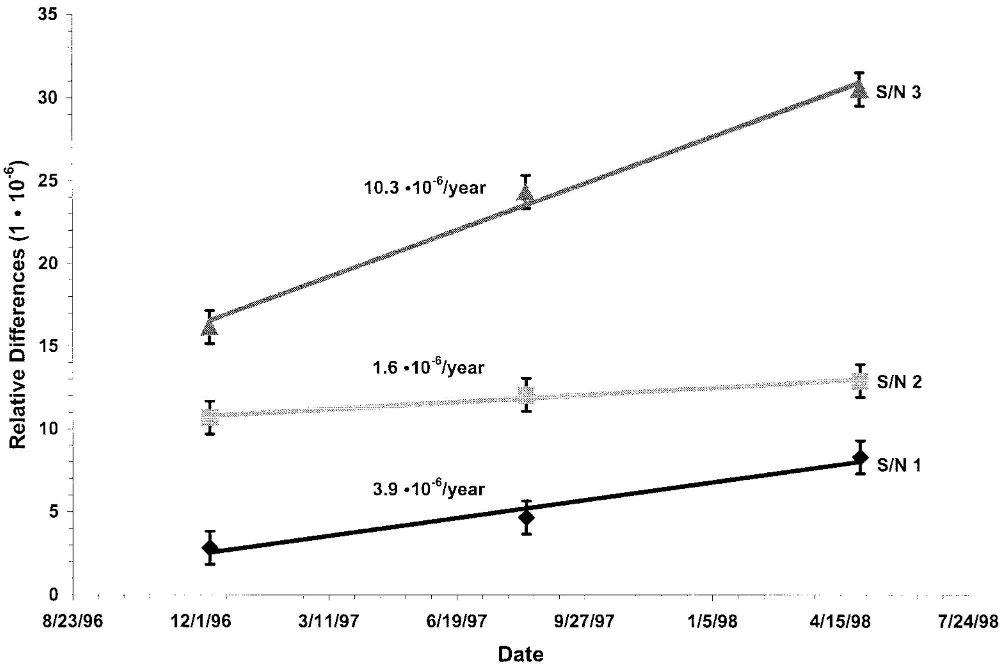

Fig. 4. NIST measurements of 10 MΩ transport standards before and after shipping standards to LCIE, NPL, PTB, CSIRO, MSL, and NML.

nominally equal to the potentials at the resistor terminations at the respective inner conductors of the glass-to-metal seals. This guarding method greatly suppresses leakage currents flowing across the glass insulator of the seals. The resistance of the glass insulator is on the order of 100 TΩ and, if the remainder of the container was all metal, the insulation resistance would shunt a 10 TΩ standard and reduce its value by up to 10%. Another factor to consider is the effective resistance of the atmosphere, which is on the order of 10 PΩ [4] in parallel with the 10 TΩ standard, which would reduce its value by approximately 0.1%. Guarding would also help to reduce the leakage resistance effect caused by the atmosphere.

III. Measurements

Three each of the 10 MΩ and 1 GΩ standards are currently being used in an international comparison of high resistances. NIST is the pilot laboratory for this comparison, and repeated measurements have been made on these standards after traveling to various countries. These standards are measured at NIST using either a guarded Wheatstone bridge system [5] or a guarded active-arm bridge system [6]. A control standard at each of these resistance levels remains at NIST and is measured along with the transport standards. These control standards were constructed at the same time and using the same fabrication process as for the transport standards. Measurements indicate no significant behavior differences between the control standards and the transport standards.

Figs., 4 and 5 indicate NIST measurements of the 10 MΩ and 1 GΩ transport standards, respectively, before and after shipping the standards to other national laboratories. From January to July 1997, the transport resistors were at the Laboratoire Central des Industries Electriques (LCIE) in France, the National Physical Laboratory (NPL) in the United Kingdom, and the Physikalisch-Technische Bundesanstalt (PTB) in Germany. From September 1997 to March 1998, the resistors were at the Commonwealth Scientific Industrial Research Organization (CSIRO) in Australia, the Measurements Standards Laboratory (MSL) in New Zealand, and the National Measurement Laboratory (NML) in South Africa. Linear regression analysis of the NIST data indicate a mean drift for the 10 MΩ and 1 GΩ transport standards of 5.3×10^{-6}/year and 11.0×10^{-6}/year, respectively. The relative standard uncertainties are estimated to be 1×10^{-6} for 10 MΩ measurements and 3×10^{-6} for 1 GΩ measurements. In contrast, resistors of this type that are commercially available specify drifts of 15×10^{-6}/year and greater than 100×10^{-6}/year for the 10 MΩ and 1 GΩ resistors, respectively. The TCR's of the film-type 1 GΩ transport standards range from -25×10^{-6}/ °C to -30×10^{-6}/ °C. Their VCR's are all less than -0.1×10^{-6}/V.

The shipping container for the transport standards contains a portable recorder to monitor the temperature and humidity of the standards during transit. A temperature recording for the period from September 1997 to March 1998 is shown in Fig. 6. This recording indicates that during transport, the standards were subjected to temperature excursions from 15 °C to 30 °C. Unfortunately, the recorder's memory was exhausted while in transit from NML to NIST and the resistors may have been subjected to temperatures below 15 °C. It appears the transport standards were not affected by these temperature changes. In contrast, the control standards residing at NIST remain in a laboratory environment of (23.0 ± 0.5) °C.

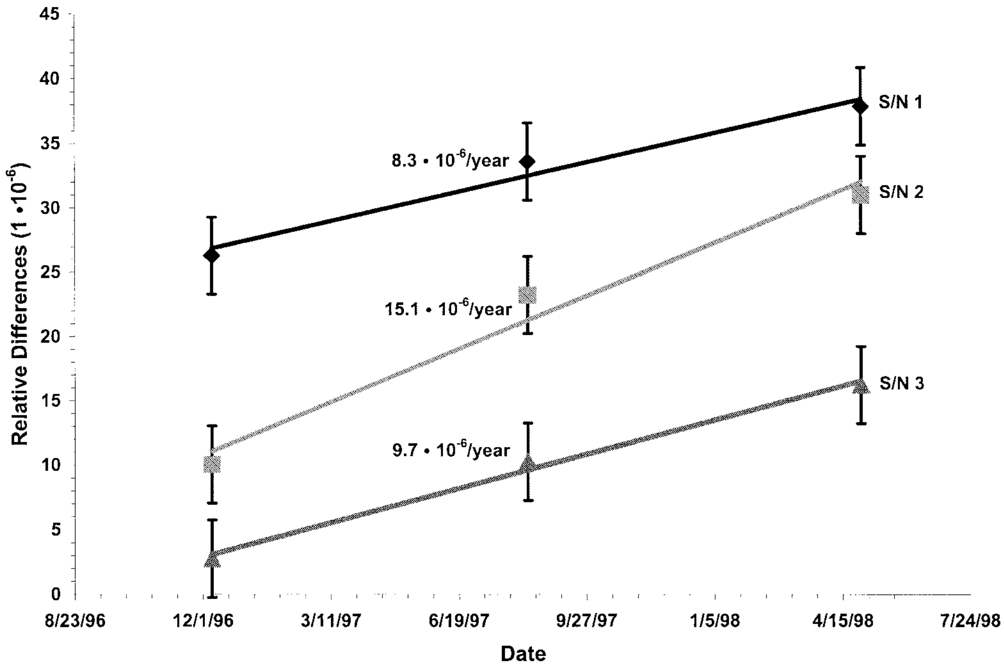

Fig. 5. NIST measurements of 1 GΩ transport standards before and after shipping standards to LCIE, NPL, PTB, CSIRO, MSL, and NML.

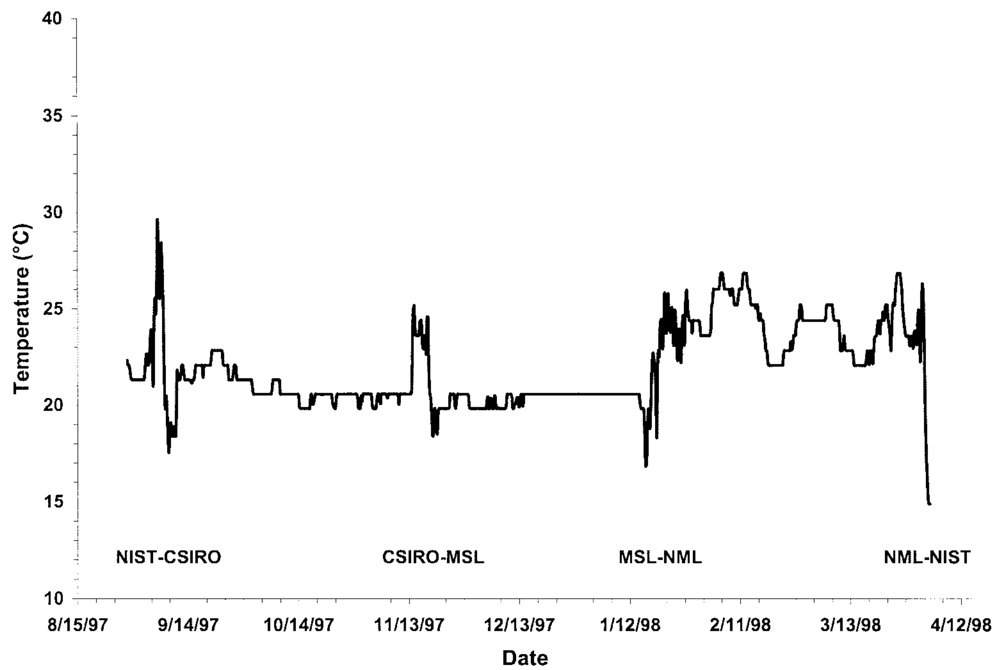

Fig. 6. Temperature recording of transport standards during transit from NIST to CSIRO to MSL to NML and return to NIST.

IV. Future Plans

NIST plans to further pre-age the film-type resistance elements by applying continuous and/or pulsing high voltages (>10 kV) to the resistance elements in addition to the heat-treatment process. It is expected that this conditioning will further relieve stresses and strains in the films and result in resistors having improved stability [7]. Also, such conditioning may improve the continuity of the films and possibly reduce their VCR's. A guarded 100 TΩ resistance transfer standard is under construction that will consist of ten 10 TΩ units hermetically sealed in special metal-insulator-metal containers.

V. Conclusion

NIST has developed a fabrication process for the construction of high-value standard resistors. Significant improvement in stability and the elimination of humidity and pressure effects is achieved by pre-aging and hermetically sealing the film-type resistors. The shock-mounting technique appears to have eliminated any effects due to transport for both the wirewound and film-type standards.

References

[1] R. F. Dziuba, "Resistors," in *Encyclopedia of Applied Physics* New York: VCH, 1966, vol. 16, p. 433.
[2] L. I. Maissel, "Thin-film resistors," *Handbook of Thin-Film Technology*. New York, McGraw-Hill, ch. 18, pp. 18–5, 1970.
[3] R. W. Berry, P. M. Hall, and M. T. Harris, *Thin-Film Technology*. New York: Van Nostrand Reinhold, 1968, ch. 7, pp. 359–364.
[4] O. H. Gish, "Universal aspects of atmospheric electricity," in *Compendium of Metrology*, T. F. Malone, Ed. Boston, MA: American Meteorological Society, 1951.
[5] R. F. Dziuba, P. A. Boynton, R. E. Elmquist, D. G. Jarrett, T. P. Moore, and J. D. Neal, "NIST measurement service for dc standard resistors," *NIST Tech. Note 1298*, pp. 20–21, Nov. 1992.
[6] D. G. Jarrett, "Automated guarded bridge for calibration of multimegohm standard resistors from 10 MΩ to 1 TΩ," *IEEE Trans. Instrum. Meas.*, vol. 46, pp. 325–328, Apr. 1997.
[7] T. Seino, M. Watanabe, and K. Sato, "Analysis of resistance change of thick-film resistor by electric pulse loading," *Electron. Commun. Jpn. 2, Electron.*, vol. 80, pp. 47–55, Dec. 1997.

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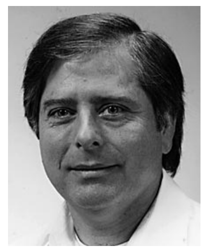

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