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DIELECTRIC AND MAGNETIC MEASUREMENTS FROM −50°C TO 200°C AND IN THE FREQUENCY BAND 50 MHz TO 2 GHz

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Contents

List of Figures

Dielectric and Magnetic Measurements From -50^oC to 200^oC and in the Frequency Band 50 MHz to 2 GHz 1.5in

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Abstract

This is an overview of techniques for dielectric and magnetic measurements of low-loss through high-loss materials in the frequency range from 50 MHz to 2 GHz and over a temperature range of -50°C to 200°C. We conclude that a single fixture is not adequate to satisfy the measurement objectives. The necessary measurements can be met using a combination of reentrant cavity, coaxial line, and dielectric resonator fixtures. In order to minimize heat loss, the coaxial line fixture should be milled from stainless steel stock and then gold plated. The reentrant cavity and split post resonator fixtures should be fitted with high temperature coaxial cables and temperature control obtained from an environmental furnace.

Key words: cavity; coaxial line; dielectric constant; high temperature; loss factor; microwave measurements; open circuit; permeability measurement; permittivity measurement; reentrant cavity; reflection method; transmission; uncertainty; waveguide

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1. Introduction

This is an overview of techniques for dielectric and magnetic measurements of unbiased, low to high-loss materials in the frequency range from 50 MHz to 2 GHz and over a temperature range of -50°C to 200°C.

Dielectric and magnetic properties depend on frequency, sample homogeneity and anisotropy, temperature, sample surface roughness, and applied bias field. Since the dielectric parameters depend on many variables, a large number of fixture designs have been developed over the years.

Each dielectric measurement fixture subjects the sample to a specific modal field [1]. Measurement fixtures, such as TE_{01} cavities, in which electric fields are tangential to the air-material interfaces, generally yield more accurate results than fixtures in which the field orientation is normal to the interface. Unfortunately, for many applications, it is not always possible or preferable to measure in-plane field orientations. For example, characterization of anisotropic materials must include measurements both in plane and out of plane.

Over the years, many techniques have been used for elevated temperature measurements. Westphal used resonant transmission lines, reentrant cavities, electroplated sample resonators, capacitors, and TE_{01} cavities [2]. Of these fixtures only the reentrant cavity satisfies our requirements. The American Society for Testing and Materials (ASTM) standard techniques [3] are short-circuited waveguide and resonant rectangular cavity. These techniques generally fall outside the frequency band of the present study. Moore [4] has used resonant rectangular waveguides, free-space techniques, and circular waveguides. The operation frequencies for these fixtures are too high for the present study. Bringhurst has used coaxial probes at elevated temperatures [5]. Coaxial probes and coaxial lines operate in the required frequency band and must be considered in this study. Tinga [6] has used reentrant cavities for measurements up to 1000°C. Tinga's approach uses an oven to heat the sample; the specimen is then quickly inserted into the reentrant cavity for measurement. Techniques that isolate the sample heating from the fixture have distinct advantages. Thermal expansion and thermal degradation are kept to a minimum. If the fixture itself is not heated, then the surface resistance of the conductors does not increase. However, these techniques have the disadvantage of less control on sample temperature.

2. Measurement Techniques

2.1. Transmission-Line Techniques

Due to their relative simplicity, the on-resonance or off-resonance transmission-line methods are widely used broadband measurement techniques [2-4]. The types of transmission lines used include coaxial lines, rectangular and cylindrical waveguide, stripline, and microstrip. Transmission line measurements use various terminations which produce different field patterns. For dielectric measurements, it is advantageous to have a strong electric field in the vicinity of the sample. This can be achieved by an open-circuited termination. For magnetic measurements, a strong magnetic field is required. This can be achieved by a short-circuited termination. The model of a coaxial open-circuited termination can involve a full-mode solution or a simpler length extension approach. In these methods a precisely machined sample is placed in a section of transmission line, and the scattering parameters are measured. The relevant scattering equations relate the measured scattering parameters to the permittivity and permeability of the material. For the frequency range of interest in this study, stripline and microstrip resonator systems are too large. Coaxial line techniques are attractive since they are broadband; however, air gaps between sample and holder severely limit their usefulness for permittivity determination [7]. Corrections for air gaps between the sample and holder can be made by analytical formulas or mitigated by use of conducting pastes or solder applied to the external surfaces of the sample before insertion into the sample holder [8, 9]. Coaxial lines allow accurate determination of permeability since the magnetic field is tangential to sample-holder air gaps. Two-port [10, 11] and one-port [5] coaxial devices, as depicted in figures 1 through 3, have been used for elevated temperature measurements. Typical uncertainties are $\Delta\epsilon_r'/\epsilon_r' \approx \pm 5$ to 10 %, $\Delta\mu_r'/\mu_r' \approx \pm 1$ %.

There are two major problems with transmission line methods at elevated temperatures [2, 12, 13]. First, the surface resistance of the transmission line walls increases as temperature increases. This increase in surface resistance has its origin in highly temperature dependent electron-phonon interactions. Secondly, thermal expansion can deform samples and holders causing air gaps.

Transmission lines are usually constructed of copper-plated or gold-plated stainless steel for temperatures up to about 1000°C. Above 1000°C, platinum-coated ceramics are usually used.

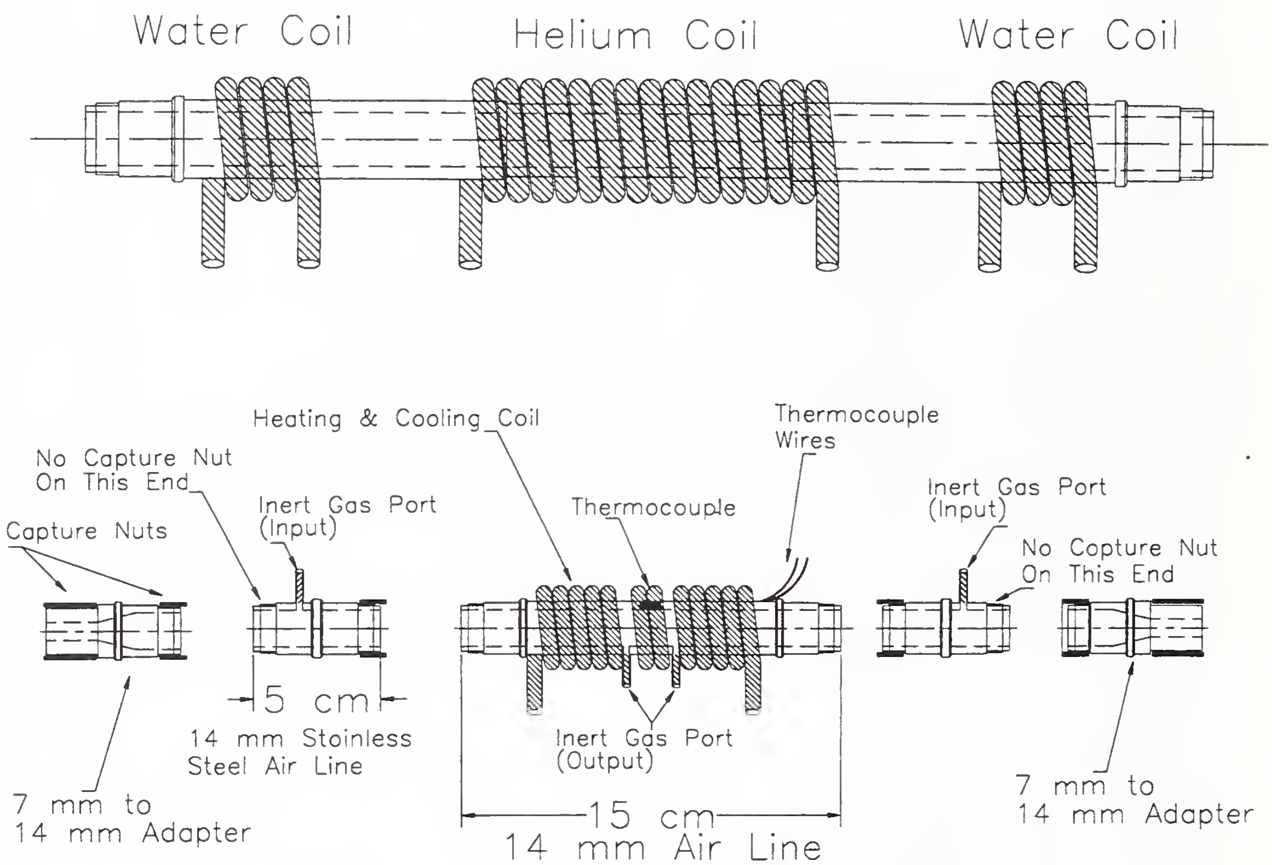

Figure 1: Two-port coaxial fixture for elevated temperatures. The fixture maintains temperature control by heating and cooling coils. Also depicted is the coaxial line setup with feedlines and connectors.

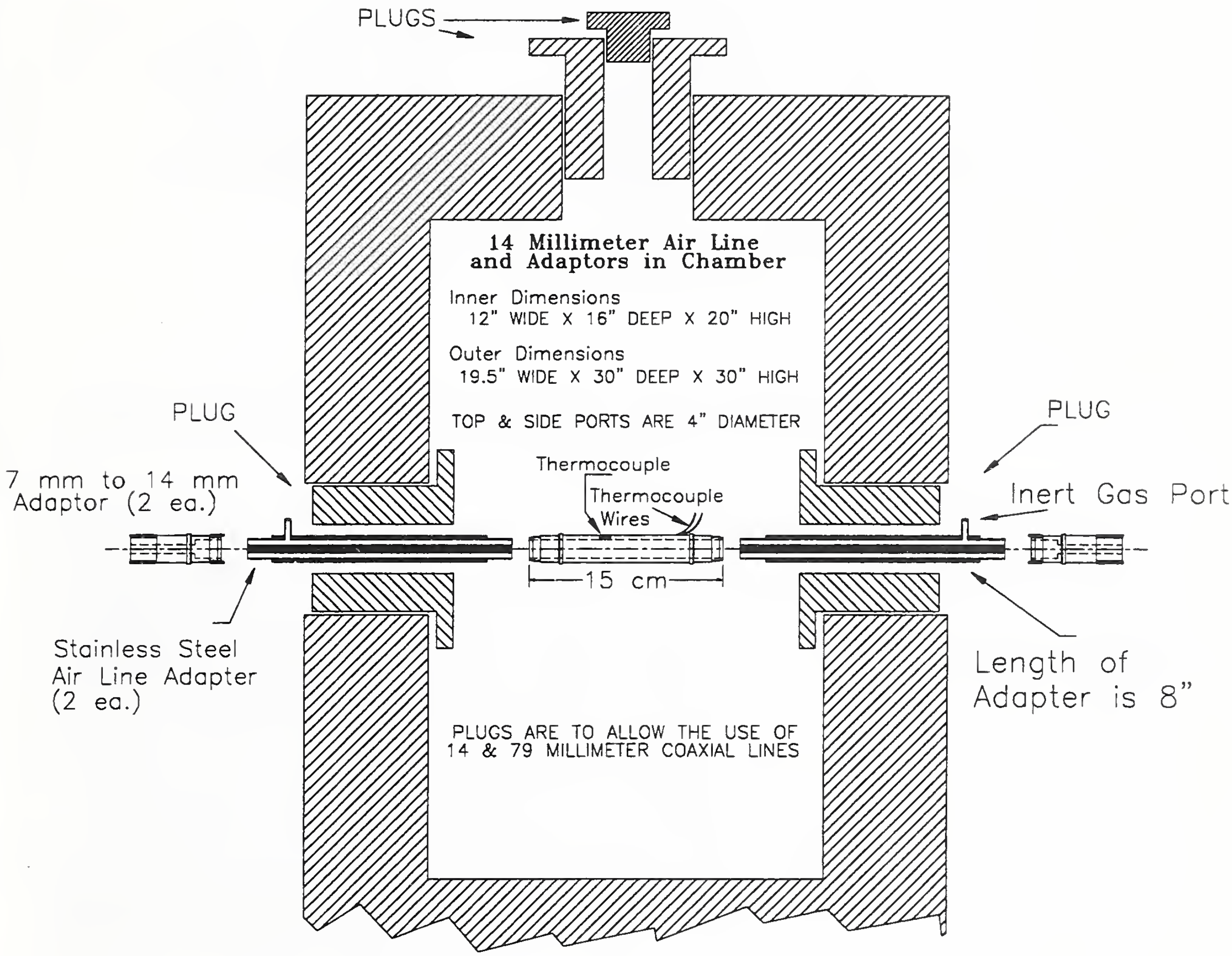

Figure 2: Two-port system with environmental chamber.

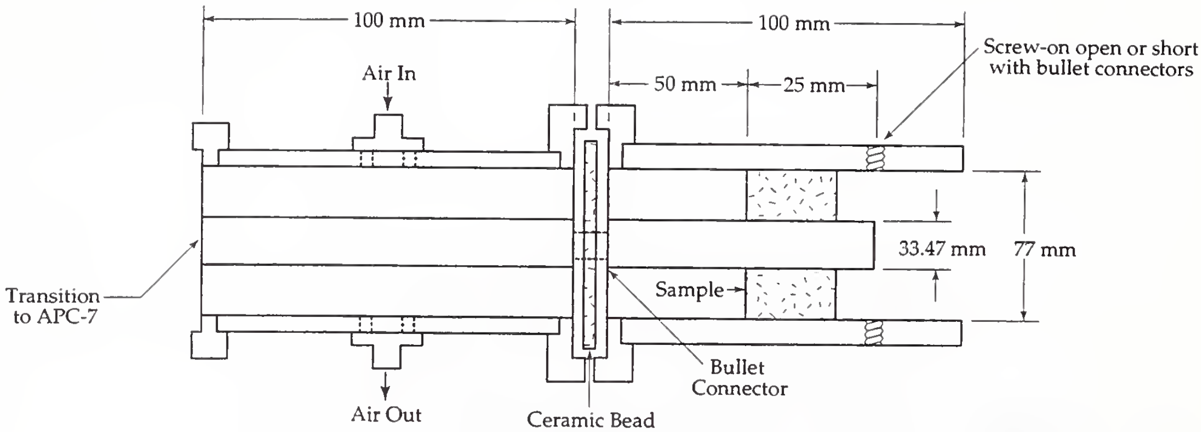

Figure 3: One-port coaxial fixture for elevated temperatures. The end of the fixture would be inserted into an access hole in an environmental chamber.

2.2. Coaxial and Waveguide Apertures

Open-ended coaxial lines and waveguides have been used for years as nondestructive testing tools [5, 14] both at ambient and elevated temperatures. In the open-ended coaxial or waveguide measurement the probe is pressed against a sample and the reflection coefficient is measured and used to determine permittivity [5]. Since the coaxial probe has electric field components in both axial and radial directions, the measurement contains elements of both the axial and radial permittivities. Although nondestructive, the method has definite limitations. The method is sensitive to air gaps since the probe has a E_z electric field component. At low frequencies, there is little field interaction with the material; field interaction increases as frequency increases. In high-temperature applications, a noncontacting probe may be required [15, 16]. For this reason, it is important to have a model of a coaxial probe which includes *lift-off*, that is, inclusion of an air gap between sample and probe [14]. Typical uncertainties for are $\Delta\epsilon_r'/\epsilon_r' \approx \pm 5$ to 10%.

2.3. Free-Space Techniques

Free-space measurements have been carried out using antennas from 50 MHz to 20 GHz. A typical setup is depicted in figure 4. Numerical reduction algorithms for frequency-domain free-space techniques are very similar to transmission-line techniques [17, 18]. The calibration

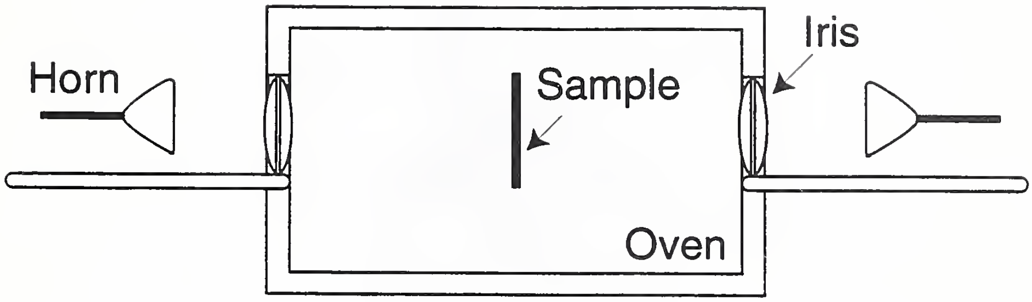

Figure 4: Free-space measurement setup.

is generally a variation of transmission-reflection-line (TRL). The transmission standard is accomplished by setting the antennas a known distance apart, usually twice the focal length. The short-circuited termination is a metal plate at the reference plane. The delay measurement is obtained by separating the antennas by another, different known distance. Although the wave front is spherical, to a good approximation the electromagnetic fields can be assumed to be plane waves. This assumption simplifies the inversion algorithm. The measurement may use a metal-backed sample or an open sample. For electrically thin samples, a short-circuit termination is not useful since the tangential electric field component approaches 0 on the metal. For electrically thin materials, a transmission measurement is preferred. In this case, both scattering parameters S_{11} and S_{21} can be used. However, at frequencies where the reflected signal becomes small, the phase of S_{11} becomes inaccurate. The technique lends itself well to elevated-temperature measurements [19].

2.4. Capacitance Methods

Capacitance techniques can be used at frequencies from 1 Hz to 100 MHz [15,16]. A typical capacitor device is displayed in figure 5. In these techniques the electric fields are nearly normal to the sample plane. The field in this fixture is normal to the sample plane. The difficulty with these measurements resides in minimizing fringing-field effects. The fringing field is usually partially eliminated by measuring the capacitance of the fixture with and without sample

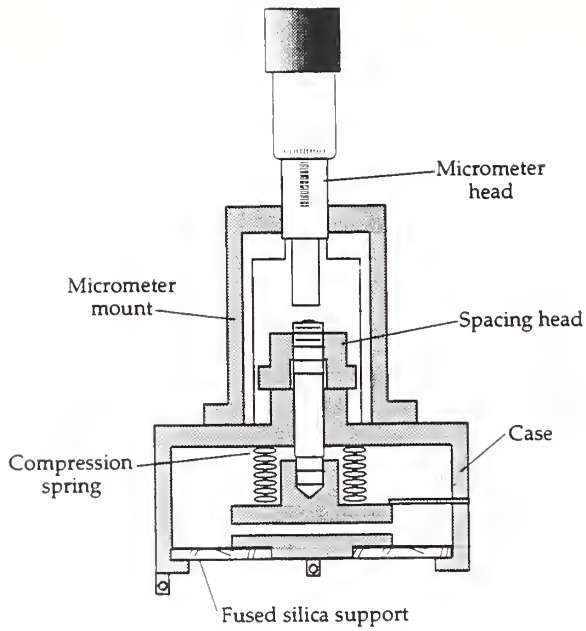

Figure 5: Capacitance dielectrometer.

and subtracting the results. Guards also help mitigate the influence of the fringing fields. Permittivity measurements using capacitor fixtures are strongly influenced by air gaps between capacitor and sample. An air gap at the sample-capacitor interface will yield a value lower than the actual permittivity. Liquids and solids have been successfully measured at elevated temperatures with capacitors [20]. Typical measurement uncertainties are $\Delta\epsilon_r'/\epsilon_r' \approx \pm 1$ to 5%.

2.5. Cavity and Dielectric Resonator Methods for Low-Loss Materials

Resonant measurement methods are the most accurate ways of obtaining permittivity and permeability. Resonant structures can be advantageous due to their accuracy; however, the frequency coverage and limitation to low-loss materials restricts their usefulness in this study. Cavity resonators operate primarily in the X-band region for materials of low loss. Dielectric resonators can operate in the 1 to 10 GHz region, but are limited to low-loss materials. The fields in these devices are usually tangential to the sample plane. Typical uncertainties are $\Delta\epsilon_r'/\epsilon_r' \approx \pm 0.20$ to 0.5%.

2.6. Reentrant Cavities

Many applications require an accurate measurement of the component of the permittivity normal to the plane of the material in the radio-frequency range. The reentrant cavity, as shown in figure 6, is an attractive alternative since it allows accurate measurement of materials

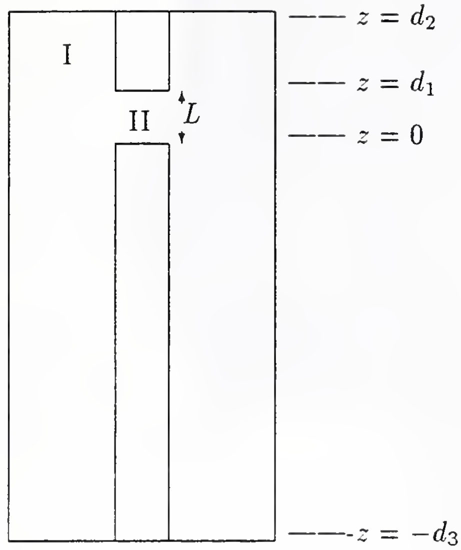

Figure 6: A doubly reentrant cavity. The sample resides between the parallel plates in region II.

at frequencies from 100 MHz to 1 GHz with the electric field perpendicular to the sample face [21-26]. The reentrant cavity consists of a coaxial line or other transmission line with a gap between electrodes where a sample is positioned. The cavity is then resonated and the capacitance of the gap produces a frequency shift from an air measurement. If the sample gap region is at the very top or bottom of the cavity then the system is called a *singly reentrant cavity*, whereas if the sample is in between the parallel plates then the cavity is called *doubly reentrant*. The loss tangent is determined from Q measurements with and without sample. Typical uncertainties for a well-characterized system on low to medium permittivity values are $\Delta\epsilon_r'/\epsilon_r' \approx \pm 1$ to 3 %. There are two approaches to modeling the reentrant cavity. The first is a lumped-circuit approximation [21, 22]. The other approach is to solve for the fields by a rigorous mode-matching technique [23, 24].

High-temperature measurements have been performed by Xi and Tinga [25, 24] using a singly reentrant cavity into which a heated specimen can be quickly inserted through a hole in the lower end plate. They have developed a full-field analysis for this problem using the same approach as [23]. The method appears to be viable for high-temperature applications.

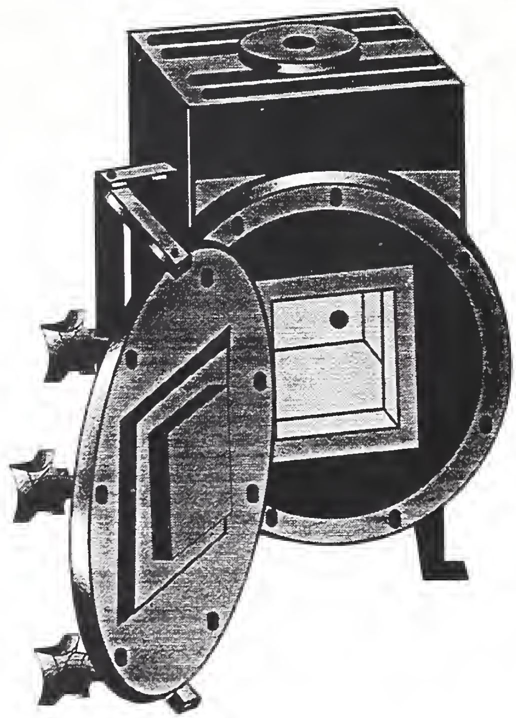

Figure 7: Typical oven for use up to 2000°C.

3. Ovens, Heat Baths, and Environmental Chambers

Dielectric measurement fixtures and samples can be brought to temperature in various ways. Environmental chambers operate from low to elevated temperatures, whereas ovens usually operate only at elevated temperatures. Environmental baths maintain a liquid at a specified temperature. This liquid can be circulated around fixtures to maintain a specified temperature.

For heating, the fixture is most commonly inserted into an oven or environmental chamber. This approach is complicated by the fact that microwave connectors and cables usually cannot withstand high temperatures. Special fused-silica filled cables can be used to minimize this problem. In addition, the entire measurement fixture must be built to withstand high temperatures, and the surface resistance of the walls of the fixture increases with temperature. Another approach is to heat the sample by an environmental bath that recirculates liquids around the fixture; copper tubing surrounded by insulation can accomplish the temperature control. This approach minimizes connector and cable heating. Another approach is to heat the sample in isolation and then quickly insert the sample into a fixture and measure the electrical response [6]. The difficulty with this approach is that the sample must be measured quickly to minimize cooling.

For this study the goal in measurement thermal stability is ± 0.2°C. Sample surface reactivity and condensation can be kept to a minimum by purging with dry nitrogen gas or operating in a vacuum. The vacuum approach is viable at low temperatures. However, as temperature

Table 1: Material properties: thermal conductivity, k, the thermal expansion coefficient, α, and electrical conductivity, σ.

Material	k (J/s.m.°C)	α (10^{-6} K^{-1})	σ (10^7 S/m)
aluminum	201	24	3.72
brass	108	19	1.57
copper	385	17	5.80
fused silica	2	2×10^{-3}	1×10^{-18}
silver	423	18	6.17
steel	46	12	1

is increased, the evolved gases also increase. In addition, heat loss due to thermal radiation increases as temperature increases. Therefore above ambient conditions, sufficient insulation cannot be maintained by vacuum alone.

4. Materials Used In Fixture Construction

At high temperatures, materials used for fixture construction must be picked judiciously. It is important to understand the thermal conductivity and thermal expansion of the fixture construction materials. A high thermal conductivity could allow connectors to overheat. The temperatures in this study are in a range where copper and stainless steel can still be used for fixture fabrication. In table 1, the thermal conductivity, thermal expansion, and electrical conductivity of common fixture materials are listed. Stainless steel has the advantage of low thermal conductivity. Copper is generally the preferred material at ambient temperatures; however, as temperature increases the thermal conductivity of copper increases. For this reason, fixtures are commonly constructed of gold or platinum-plated stainless steel. Platinum has a high melting temperature and good electrical resistance. In very high-temperature applications, platinum is coated on ceramic coaxial lines.

5. Conclusions and Recommendations

Table 2 summarizes frequency band, field orientation, temperature range, and loss characteristics of measured materials for the various techniques. One technique alone is not sufficient to characterize dielectric materials over the frequency and temperature region of interest. TE_{01} cavities generally fall outside the frequency band of interest. Capacitors operate at too low

Table 2: Fixture operating ranges.

Fixture	Frequency Range	Accuracy	Loss	Temperature(°C)
Capacitor	1 Hz - 100 MHz	medium	low to high	1500
Coaxial line	1 MHz - 18 GHz	medium	low to high	1500
Waveguide	3-30 GHz	medium	low to high	1000
Reentrant cavity	100 MHz - 1 GHz	medium-high	low to high	1500
Dielectric resonators	1-30 GHz	high	low	1500
Cavity resonators	6-30 GHz	high	low	1500

a frequency for the present study. Free-space techniques are not accurate enough for our purposes. The geometry of the materials under test also require specific fixture design. For example, thin materials can be measured in reentrant cavities or dielectric resonators, whereas thick materials are more amenable to transmission-line techniques.

In order to satisfy our frequency, temperature, and temperature stability demands we recommend the following strategy:

- a two-port 14 mm coaxial line system used with an environmental chamber
- a 79 mm one-port coaxial line system with environmental chamber
- a reentrant cavity with fused-silica-filled cables in an environmental chamber
- a dielectric resonator operating at 2 GHz for low-loss thin materials

The following heating equipment is required:

- an environmental furnace
- a heat bath
- a thermocouple system
- high-temperature cables

In the reentrant cavity technique, either the fixture can be heated or the sample can be preheated and then inserted. We believe the complications encountered by the sample insertion technique outweigh the problems of heating the reentrant cavity directly. Since the sample must be both raised above and below room temperature, the insertion technique loses some of its appeal. Use of ovens and environmental baths cannot be avoided for coaxial-line and dielectric-resonator systems.

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