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RF Materials Characterization Metrology at NBS/NIST: Past and Recent Work, Future Directions and Challenges*

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Abstract: We summarize the RF materials characterization programs at NBS/NIST. We also project what we believe will be the most important measurement problems for the future.

Introduction

Over the last 75 years, measurements of the electrical permittivity and magnetic permeability of materials as functions of frequency and temperature have been crucial for a wide array of applications. Characterization of these parameters has been instrumental in the development of electromagnetic shielding materials, wireless communications circuitry, medical applications, and microelectronics. The need for broadband dielectric measurements has been steadily increasing over the years. Interestingly, both the present and past impetus for characterization data on dielectric materials has been driven by the development of wireless technology. As electrical components are miniaturized, there is a critical need to accurately measure low-loss dielectric materials. For example, new packaging and printed wiring board (PWB) technology requires materials with low permittivity. The use of fine-line signal conductors requires thinner, possibly laminated, low-permittivity printed wiring boards, thin films, and substrate materials. There is also an increasing need to characterize ceramic substrates that are used in low-temperature co-fired ceramic applications (LTCC). The need for metrology, standard measurement methods, and materials to support the development of these novel materials is steadily increasing.

Past Research:

Research into dielectric and magnetic material properties has been a perennial focus area of both the original National Bureau of Standards (NBS) and the National Institute of Standards and Technology (NIST). The first papers in the area of dielectric measurements by Dellinger and Preston, dated back to 1922. These measurements pertained to characterizing power loss and dielectric constants [1]. The early NBS program used bridges, capacitors, RF generators, and Q-meters. In the late 1940s, George Birnbaum and Samuel Kryder developed a recording microwave refractometer and other methods for measuring the dielectric constant of gases, water vapor, and solids [2]. In 1946, John Dalke and others developed a measurement service for dielectric constant and loss tangent. In this service they used a reentrant cavity from 50 to 300 MHz, based on a design from MIT Laboratory of Insulation Research. Reentrant cavities consist of resonate coaxial short-circuited waveguides with the sample placed in a gap in the center conductor. James Beardsley developed a reentrant cavity in 1950 that operated at 150 MHz. This cavity is currently exhibited in the NIST, Boulder Museum. Robert Powell joined the project in 1948 and worked on the reentrant cavity and permeability meter (permeameter) for measuring permeability [3].

The first round robin to measure the permeability of magnetic materials was conducted in 1948 over frequencies from 1 to 500 MHz. In the 1950s magnetic materials became a focus of the radio-frequency project. P. H. Haas and R. D. Harrington developed the radio-frequency permeameter in 1952 and published results in the NBS Journal of Research [3].

The permeameter became the NBS primary standard for low-frequency magnetic materials. In this device, the sample served as a secondary in a transformer. Powell and Rasmussen developed a radio-frequency permittimeter in the early 1950s [4]. In 1958 Bussey and Birnbaum applied the refractometer method to measuring the refraction index of the atmosphere. Howard Bussey and Edwin Bamberger introduced a number of new techniques in the microwave spectrum. Bussey used the rod-cavity technique extensively for

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measuring both the dielectric and magnetic material properties. In 1964 and in 1972 Bussey conducted round-robin intercomparisons on fused silica and glass [5,6]. The participants were NBS the National Physical Laboratory (NPL), UK, and National Research Council (NRC) in Canada. Howard Bussey and Ramon Jesch developed a simple model for the shielded open-circuit holder for liquid measurements. The NBS materials project ceased work in the late 1970s, but was restarted in 1986. Goldfarb and Bussey developed a coaxial-line permeameter for use at radio frequencies [7].

From 1986 to 1988 with Air Force funding an extensive literature review was performed in the area of radio-frequency dielectric measurements. William Kissick led the electromagnetic properties of materials project (EPM) from 1988 to 1990 with an emphasis on characterizing radar-absorbing materials using transmission-line methods, and a mode-filtered TE_{01} cylindrical cavity. This cavity currently serves as a standard for characterization of reference materials. A novel procedure for obtaining complex permittivity from transmission-line scattering equations was developed that is numerically more stable than the commonly used Nicolson-Ross method. This procedure allows samples of arbitrary length to be measured [8]. Claude Weil led the project from 1991 to 1999. During this era, the reentrant cavity, stripline resonator, coaxial probe, bio-liquids measurement techniques, and various dielectric resonators were developed. A full-mode model for the open-ended probe was developed by the EPM project that includes the effects of liftoff above the sample [9]. The EPM project also developed a Fabry-Perot resonator that operates from 60 to 70 GHz [9]. In an effort to broaden the frequency range and increase measurement sensitivity for thin materials, Janezic and Williams developed a thin-film measurement method that uses planar transmission lines, such as microstrip and coplanar waveguide where the thin film is incorporated as part of the transmission line. This method uses measurements of both the propagation constant and characteristic impedance of the planar transmission line to find the permittivity of the thin film. The advantage of this method is the ability to separate the electrical properties of the metal conductors from the electrical properties of the thin film. A full-mode model was also developed for the shielded open-circuited holder that is used to measure liquids and high-loss semi-solids. The whispering-gallery and parallel-plate methods for measurement of ultra low-loss materials were developed through collaborations with Jerzy Krupka [8]. With this method, quality factors up to 10^6 have been measured on sapphire at cryogenic temperatures. The EPM project also conducted a number of intercomparisons. These intercomparisons used coaxial 7 and 14 mm lines on dielectric and magnetic samples and a stripline intercomparison [9-12]. The coaxial-line intercomparisons included measurements from various companies. There were 15 participants for the magnetic and 10 for the dielectric intercomparisons. In these intercomparisons a large variability in measurements was demonstrated. The EPM developed a full-mode model and software for the reentrant cavity for measurement of printed wiring board materials and other substrates. This new model is significantly more accurate than the previously used lumped-circuit model [9-12]. The project also developed a variation of the reentrant cavity for the measurement of biased ferroelectrics. In this method a dc-bias field is applied between the insulated bottom post and the base.

Currently, the main focus areas of the EPM project are: wireless, low-temperature co-fired ceramics (LTCC), printed wiring board, thin films, composites, biomaterials, and relaxation theory [12,13]. The project uses various resonators for the measurement of low-loss bulk and substrate dielectrics. New full-mode software for the split-cylinder and sleeve resonator methods has been developed and software for the split-post resonator is near completion. Our new full-mode theory and software for the split-cylinder rigorously models the fringing fields [14]. We use four split-post resonators for measurements from 1 to 10 GHz. We also use the full-sheet resonance and reentrant cavity techniques for clad substrates [13]. In addition we use TE_{01} cavities and dielectric resonators for thin-material measurements. The project has also developed a thin-film method based on deposition on substrates with known dielectric properties. In this method a TE_{01} cavity or split-post dielectric resonator is used to measure the substrate with and without the deposited film. Then software based on a theoretical layered-model for the cavity makes it possible to determine the film properties. A Standard Reference Material (SRM) service has been developed. The first dielectric SRM is cross-linked polystyrene.

Future Directions

The trend for the future is increasing use of ferroelectric, ferromagnetic, and low-permittivity substrates and films. The use of bias-field tunable ferrite and ferroelectric materials should steadily increase. Wireless technology is expected to gradually expand its frequency range from 1 GHz up to 200 GHz. We also see a strong increase in applications of artificial dielectrics such as photonic band-gap materials. These new materials will be engineered for specific needs and applications. Some microelectronic materials will gradually be replaced by these artificial dielectrics. New materials such as the recently reported metamaterials with negative permittivity and permeability will be developed for new applications. There will also be a need for fundamental metrology on new artificial dielectrics. There will be a continual need for reference materials and measurements on bulk and thin-film materials and liquids.

Summary

Frequency-dependent electrical material property measurement at NBS/NIST has been an important research area for over 75 years. Over the years, applications to radio and wireless has driven the metrology. Previous NBS efforts developed metrology for the reentrant cavity, permeameter and various cavities. Various intercomparisons were made over the years. These intercomparisons are important in standardization of measurements. The EPM project has greatly expanded the number and accuracy of methods. The improvement in measurement accuracy is tied to the use of full-mode models and the use of dielectric resonator methods for low-loss materials. The EPM project has compared measurements on the same materials used in past intercomparisons. These measurements have produced continuity in the evolving project.

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