Quantum Design

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Resonator Techniques to Characterize Material and Device Properties at Microwave Frequencies in the Quantum Design PPMS Measurement System

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Abstract

We describe resonator techniques that can be used to characterize microwave material and device properties in the Quantum Design PPMS measurement system over a wide range of temperatures (1.9 - 400 K) and magnetic-fields (up to 14 Tesla). In this application note, we provide the methodology used and several measurement examples, including determinations of superconductor-film surface resistance, dielectric loss tangent and Electron Paramagnetic Resonance (EPR) spectra. To perform these experiments, a ~1 cm^2 resonator is placed inside a gold-plated copper RF cavity (2.03 cm x 0.61 cm x 1.52 cm) that is bolted to the bottom end of a cryogenic dipping probe. A microwave network analyzer is utilized to excite transverse electromagnetic modes and measure the quality factor in a non-contact configuration with open circuit boundary conditions. For measurements at ~10 GHz, the technique has a sensitivity of better than 5 $\mu\Omega$ for superconductor surface resistance measurements and 5 x 10^{-7} for loss tangent measurements of materials with a dielectric constant, ε_r, of 10 and even lower for higher ε_r materials. The RF cavity can also be used to make filter and other device measurements in the PPMS.

Section I: Introduction

We also have found that this method with minor modifications can be used to measure the loss tangent of dielectric samples. This technique has advantages over the commonly used $TE_{01\delta}$ cavity loaded dielectric resonator (DR) method [9-12]. Besides the high sensitivity, the sample volume of the interlayer dielectric used in the PPR is almost two orders smaller than that needed to make dielectric-resonator loss-tangent measurements in the same frequency range. Thus, the PPR method can minimize the volume of material needed. Also, since a single small high-loss defective region can skew the Q measurement of a conventional industry-standard $TE_{01\delta}$ dielectric resonator [9-12], this method can determine the material uniformity by measuring many samples from a single $TE_{01\delta}$ resonator. The PPR technique can use high-quality Nb films (for measurements up to ~7 K) and $YBa_2Cu_3O_{7-\delta}$ (YBCO) films (for measurements up to ~70 K) with low surface resistances to determine the loss tangent as a function of temperature.

The superconductor properties degrade in the presence of magnetic fields and at temperatures approaching their critical temperature, so the accuracy of the PPR technique is unacceptably poor under these conditions. However, using PPR loss tangent determinations at low temperatures and in the absence of an applied magnetic field as a standard, we have been able to show that DR measurements are accurate. Since the DR technique is essentially unaffected by temperature or magnetic field, it can thus be used to accurately measure the loss tangent over the wide range of temperatures (1.9-400 K) and magnetic fields (0 - 14 T) that the PPMS system facilitates. The DR also enables Electron Paramagnetic Resonance (EPR) measurements. The improved fill factor and higher Q also makes this technique potentially orders of magnitude more sensitive than a conventional EPR system.

Both the PPR and DR techniques have the advantage of using well-defined principle and higher order dielectric resonances, a near-unity fill factor and well-understood relatively-uniform electric and magnetic field distributions. This enables accurate measurements with these techniques without the use of standards or other methods of calibration.

Section II: Experimental setup

The methods we described here are based on our collaborative work investigating high temperatures superconductor properties with Bob Taber of HP/Agilent [1-3] and our own more recent work on high performance dielectrics[13].

PPR [1,5,6,15] and DR measurements are performed by placing the resonator inside a gold-plated copper RF cavity (2.03 cm x 0.61 cm x 1.52 cm). The cavity is bolted to the bottom end of a cryogenic dipping probe, as shown in Figure 1 The details of the structure of the resonator are described in Taber and other researcher's work [1,6]. The fixtures we used to affix the microwave cavity and its surrounding isolation enclosure to the stainless steel tube were modified to have less than a 2.5 cm cross-sectional diameter to fit into the Physical Properties Measurement System (PPMS) (Quantum Design, San Diego, CA).

A gas-tight seal for the probe is crucial to eliminate contamination from entering the cryogenic region (most importantly, icing). On the top part of the dipping probe, two compression O-ring fittings are used to make an air-tight sliding seal where the 2.16 mm OD microwave coaxial cable enters the 1.27 cm OD stainless steel support tube (i.e. 1.27 cm OD and 0.8 mm thick). The O rings have an ID of 2.1mm and an OD of 5.1mm. To make the microwave coupling probes, the wire at the end of the coax nearest the sample is stripped back and then soldered to the ground sheath (to produce magnetic field probes) or left unsoldered (to produce electric field probes). In the earlier work, Taber [1] used a small piece of microstrip to make electric field probes.

Transmission measurements are made using an HP8510C microwave vector network analyzer (VNA) to excite fundamental electromagnetic and higher order resonant modes and record the associated S_{21} vector values. The microwave signals are transmitted through microwave coaxial cables (silver plated copper inner conductor, stainless steel outer conductor, 50Ω, 4.8 dB/m attenuation @10GHz, UT-085-SS, Micro Coax, Pottstown, PA). Micrometers (1/2 inch, Non-Rotating, Item No. 2ZUD6, Grainger Inc., Lake Forest, IL) and their associated clamps are used to grip and move the coaxial cable so that the distance between the microwave coupling probes and sample (see Figure 1) results in a measurable signal that is weakly coupled to the resonator [1,6].

There are a few important details that must be attended to in order to achieve a reliable and accurate loss tangent measurement. Firstly, the resonator should be well centered in the cavity and its edges aligned parallel to the metal cover plate. To consistently place the sample in the proper position, a centering L-shaped fixture can be affixed with machine screws to the tapped cover plate holes. Once the sample is located in the center, the set screws that press on the dielectric support posts to hold the sample in the cavity can be tightened and the fixture removed. Secondly, after centering the resonator, we need to refine the loading position and the distance between the coupling loop and the sample to obtain a strong S_{21} signal intensity. We have found that when the peak intensity is adjusted to between 25 and 35 dB by adjusting the probe positions, the error in Q can be consistently kept to less than 2%. When we measured a commercial BZT sample 12 times at an S_{21} intensity maintained at 30 ± 1 dB, the standard deviation of the error was below 1%. When the S_{21} intensity is only 10 dB, the error typically is found to increase to ~15%. The S_{21} values are then fit to a circle in the Smith chart [10] to infer the quality factor. Because only the value of the quality factor is sufficient to infer the loss tangent, a calibrated measurement is not typically required.

A LabVIEW (National Instruments, Austin, TX) program was made to (a) automatically set the magnetic field and temperature in the PPMS, (b) set the measurement range and initiate the measurement in the VNA and then (c) retrieve the resulting data from the VNA. LabVIEW drivers to interface with Quantum Design MultiVu software are available from Quantum Design at www.qdusa.com (see application note 1070-210). A Matlab script for fitting a circle on the Smith chart to determine the unloaded Q [14] was inserted into the LabVIEW computer module.

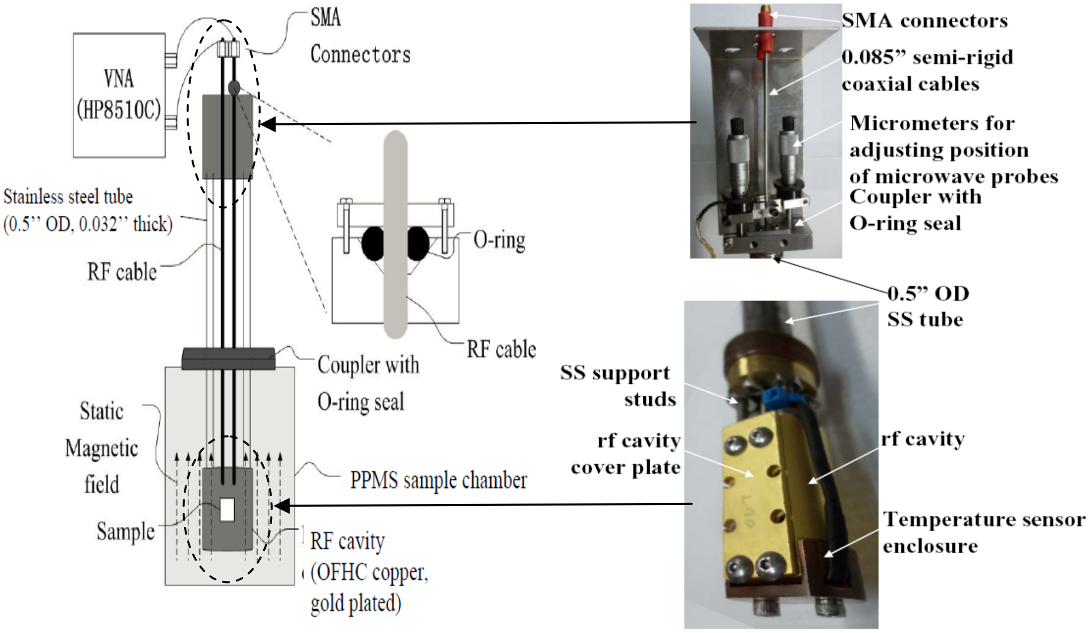


Figure.1 Experimental schematic for measuring the loss tangent and EPR spectra of small dielectric samples over a range of temperatures and magnetic fields.

In our PPR measurements, we use superconducting Nb films (1 cm^2) deposited on Yttrium stabilized Zirconia (YSZ) substrates fabricated with sputter deposition (MIT Lincoln lab) and $YBa_2Cu_3O_{7-\delta}$ films deposited on buffered sapphire substrate (STI Inc.). The Nb films used in this work have slightly lower surface resistances (~12 $\mu\Omega/\square$ at 4.2 K, scaled to 6.5GHz) and give higher Q values than the YBCO films. Although the surface resistance of YBCO films can be less than 9 $\mu\Omega/\square$ at 4.2 K scaled to 6.5GHz [3], the YBCO films in our measurement has higher R_s of about 27 $\mu\Omega/\square$ at the same temperature and frequency. However, because of Nb's low critical temperature (T_c) of 9.2 K, it can only be used for measurements up to ~7 K before the loss from the superconductor begins to dominate. On the other hand, the $YBa_2Cu_3O_{7-\delta}$ films can be used to measure a relatively large temperature range (up to ~80K).

For measurements of the surface resistance of the superconductor films, we used Teflon® FEP films (ε_r =2.1, DuPont Electronics, Wilmington, DE, 12.5μm thick). We also characterized the properties of high-performance microwave dielectric $Ba(Zn_{1/3}Ta_{2/3})O_3$ ceramics, ε_r =29, commercially produced by Trans Tech (Adamstown, MD) and made in our lab using conventional powder processing methods.[13]

The overall unloaded Q of the resonator can be modeled by the equation:

$$Q^{-1}=Q_d^{-1}+Q_r^{-1}+Q_c^{-1}=\tan\delta+\alpha s+(\beta R_s/s). \qquad \text{Equation 1}$$

In this equation, the first term is the loss tangent of dielectric spacer (dielectric loss), the second term is from near-field energy radiation from the surrounding of the resonator (radiation loss), and the third term is caused by surface resistance of superconductor films (conduction loss). The three have different dependences on the spacer thickness, *s*. Dielectric loss is independent of spacer thickness; the radiation loss is proportional to the spacer thickness, and the conduction loss is inversely proportional to it. α and β are the associated coefficients. $\beta=1//(\pi\mu_0)=3.9\times10^{-5}$m/Ω at 6.5 GHz [1], and α relates to the near field losses and is determined by the sample dimensions and ε_r.

To accurately measure dielectric loss using this technique, the dielectric loss, quantified by tan δ, must dominate over other two terms. For this, we need to determine the optimal thickness of the spacer, which corresponds to when the interlayer thickness is varied to maximize the Q. Our experiments on 1 cm^2 Si samples measured at low temperature indicate that the unloaded Q increases almost linearly at spacer thickness less than 100μm, because the superconductor surface resistance loss dominates in this range. At thicknesses larger than 100μm, the unloaded Q decreases because the dielectric loss and near-field radiation loss become more important. At around 175 μm, the unloaded Q reaches its maximum of ~165,000 at 6.5 GHz. By fitting the data to within experimental error, we find that we can resolve changes in the quality factor from dielectric loss to be about an order of magnitude higher than measured, we estimate the sensitivity of the tan δ measurement for a material with a ε_r of 11 to be 1×10^{-6} at 6.5 GHz. From this, we also conclude that the optimal thickness of the dielectric should be ~175 μm. Note that, α is inversely proportional to square root of ε_r so the near-field radiation loss will be lower for higher dielectric constant material, such as BZT, and thus the sensitivity would be expected to be proportionately better.

For the details on how to accurately measure the surface resistance of the superconductor, the reader is referred to Taber's work [1]. For details on the measurement of the dielectric loss tangent or the EPR spectra, the reader is referred to Liu's work [13].

For measurements at ~10 GHz, the technique has a sensitivity of better than 5 μΩ for superconductor surface resistance measurements and 5 x 10^{-7} for dielectric loss tangent measurements with a ε_r of 10 and even lower for higher ε_r materials. The use of the DR technique as a function of magnetic field allows the exploration of the effect of spin excitations, including EPR processes, on the loss tangent.[13]

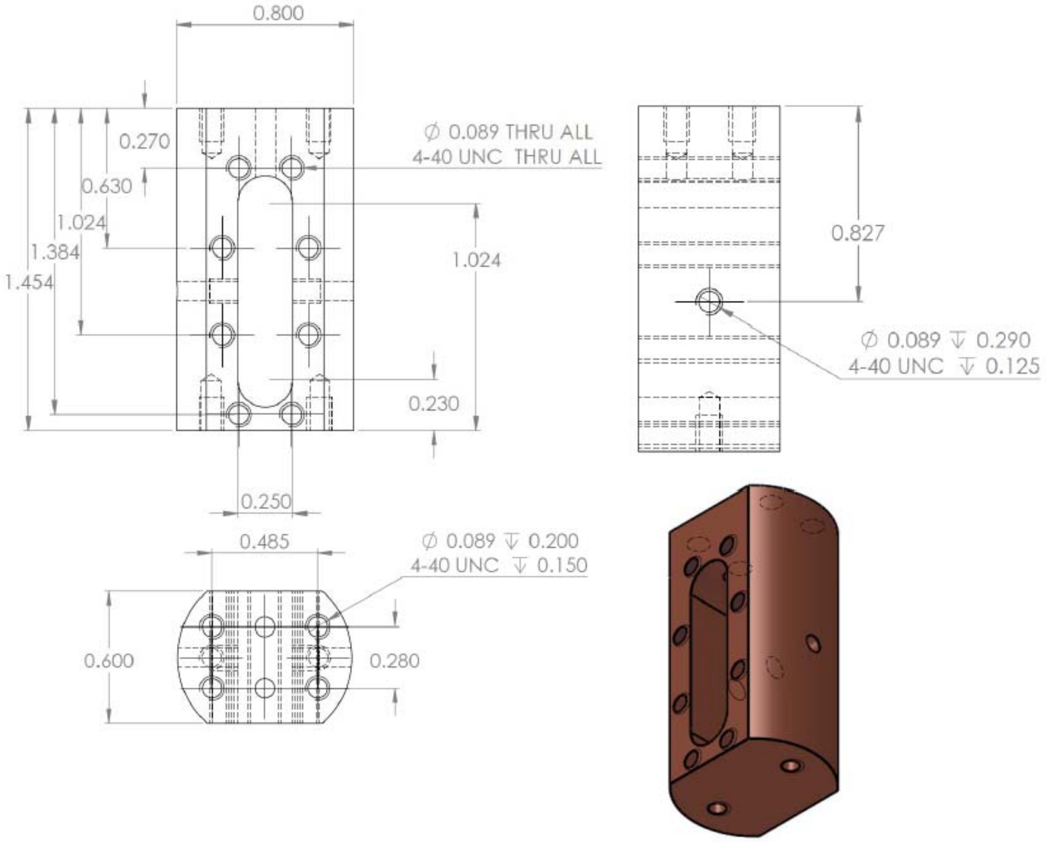
Section III: Conlusion

We use both parallel plate resonator (PPR) and dielectric resonator (DR) structures to accurately determine the surface resistance of superconductor films and the loss tangent and EPR spectra of small dielectric samples over a range of temperatures and magnetic fields.

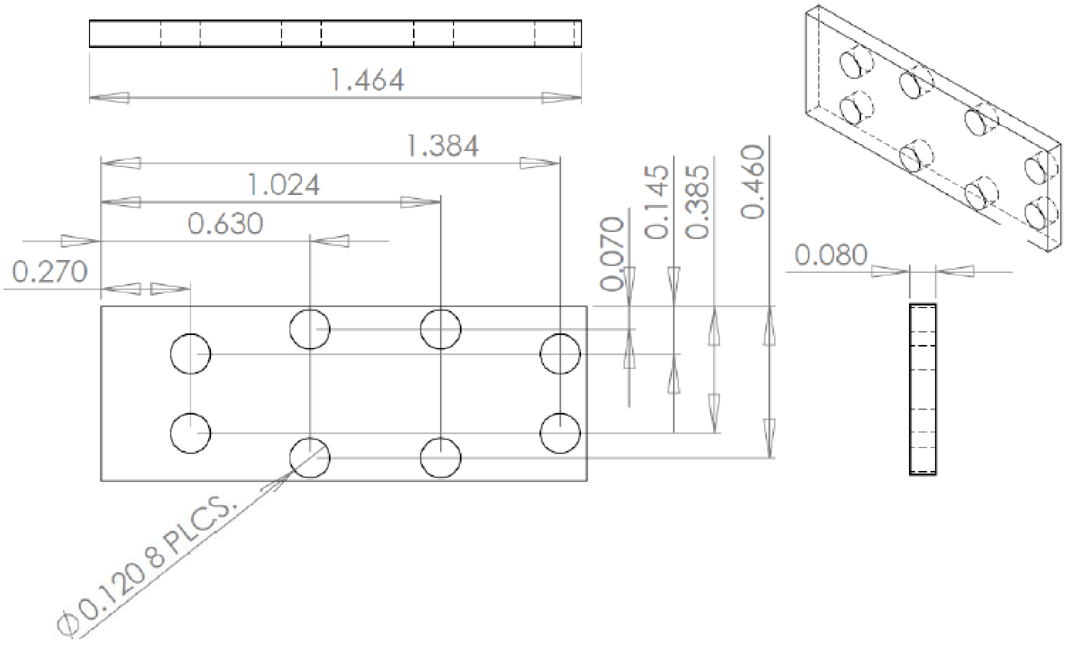
Section IV: Acknowledgment

We would like to acknowledge the Army Research Office for support of this work under contract W911NF-11-1-0508. We would also like to thank Brian Moeckly for supplying the $YBa_2Cu_3O_{7-\delta}$ thin film samples and Dan Oates and Paul Welander for supplying the Nb thin film samples

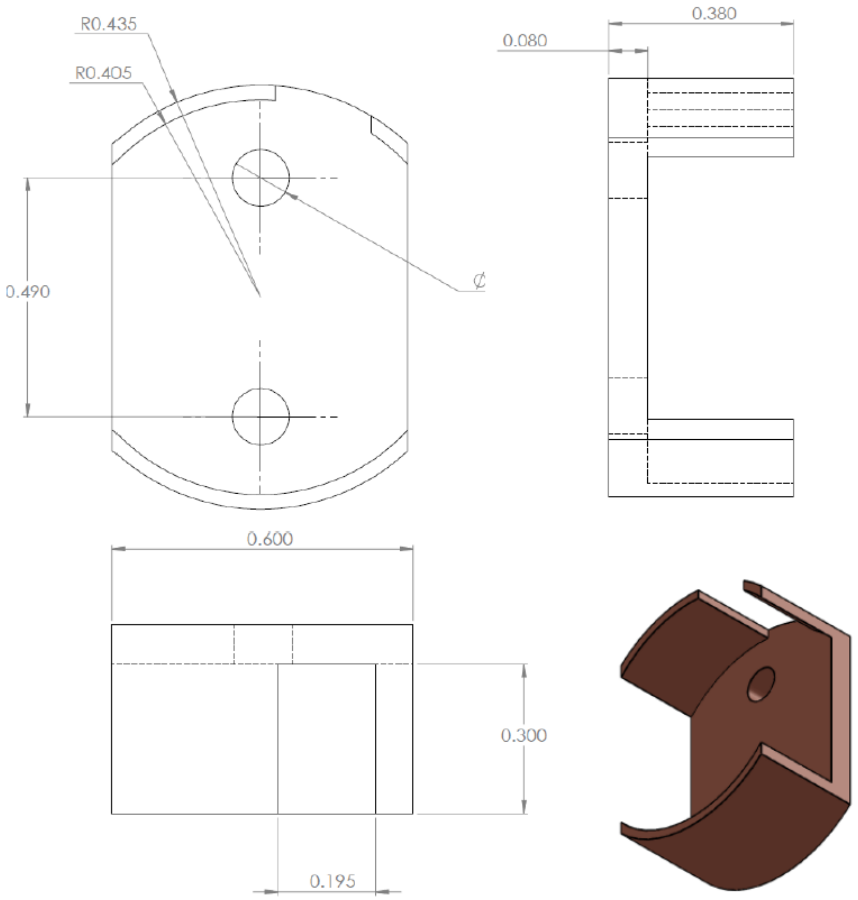
Appendix Shop Drawings (dimensions are in inches)



Appendix Figure 1: rf cavity:
Material: OFHC Copper with 500 microinch high purity gold



Appendix Figure 2: rf cavity cover plate
Material: OFHC Copper with 500 microinch high purity gold



Appendix Figure 3: Temperature sensor enclosure
Material: Copper

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