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# An Experimental Platform for Performing Measurements of the RF Magnetic Permeability and Electric Permittivity of Functional Materials at Cryogenic Temperatures

## A NISE funded
## Basic Research Project

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## ADMINISTRATIVE INFORMATION

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# 1. INTRODUCTION

The experimental apparatus described here was developed to support research into a class of electronics based on modifying superconducting circuitry through the integration of complex functional materials with exploitable frequency-dependent electric, magnetic, and structural properties. For instance, by inserting materials with physical properties that are responsive to incident radio-frequency (RF) radiation as the tunneling barrier in superconducting junctions, the inherently fixed physical parameters governing the superconducting tunneling properties can assume frequency-dependent behavior. The interfacing of multiferroic and superconducting materials can potentially be exploited wherein applied localized magnetic or electric fields or RF excitations on-chip would enable dynamic tuning of the characteristic performance of various types of superconducting circuit structures. This could potentially enable high-precision dynamic control of current–voltage linearity of a circuit or device.

All known superconductors require cryogenic temperatures (below ≈ -140 °C/133 K) for the materials to enter into the superconducting state. The defining characteristics of a superconductor are that the material can (1) transport an electrical current below a critical value without any measurable dissipation, (2) form an energy gap associated with the pairing energy of the superconducting electrons, and (3) behaves as a perfect diamagnet, expelling all applied magnetic field below a critical field strength, $H_c$. For Type-I superconductors, when the applied magnetic field is greater than $H_c$, superconductivity is quenched and the material returns to its normal state. In the class of superconductors referred to as Type-II, with applied magnetic field values above $H_c$ and below a larger critical field, $H_{c2}$, the material maintains its superconducting state by allowing the field to penetrate in thread-like structures referred to as vortices. The dynamical properties of vortices in these materials are relevant to the performance of subsequent circuitry and devices, particularly in a high-frequency RF environment. It is important to have an accurate characterization of the main parameters related to vortex motion, i.e., the viscous drag coefficient, $\eta$, the pinning constant, $k_p$, and the de-pinning frequency, $\omega_0$.

Multiferroic materials form a special class of materials exhibiting the coexistence of two or more ferroic orders (ferromagnetism, ferroelectricity, and ferroelasticity). While many multiferroic materials are useful at ambient and even very high temperatures, many of these compounds enter their ordered state at temperatures approaching or well into the cryogenic temperatures needed for the superconducting materials discussed above. It is desirable then to be able to characterize multiferroic materials as to their magnetic and electric RF properties over a wide range of temperature and frequencies, as well as the integrated film structures that will be based on new functional circuitry. Information extracted from electric permittivity and magnetic permeability measurements reveal varying complex functional properties of these materials in single crystal, bulk pollycrystalline, and thin film forms, all of which often evolve with temperature. Often, the properties of interest are determined by structural, magnetic, and/or electric polarization domain walls. This presents an opportunity to utilize the inherent interface of domain walls in multilayer films for microscopic sensor applications.

# 2. DESIGN AND OPERATION

The experimental platform described here uses off-the-shelf electronic equipment. With the appropriate vendor-supplied material measurement firmware, the Agilent E4991A RF impedance/material analyzer can measure the impedance parameters: $|Z|$, $|Y|$, $L_s$, $L_p$, $C_s$, $C_p$, $R_s$(R), $R_p$, X, G, B, D, Q, $\theta_z$, $\theta_y$, $|\Gamma|$, $\Gamma_x$, $\Gamma_y$, and $\theta_\gamma$; the electric permittivity parameters: $|\varepsilon_r|$, $\varepsilon_r'$, $\varepsilon_r''$, and tanδ; and the magnetic permeability parameters: $|\mu_r|$, $\mu_r'$, $\mu_r''$, and tanδ at ambient temperature. The temperature range of the measurement of the electric permittivity and the magnetic permeability is here extended down to ~ 4.2 K by using the custom-designed probe and cryogenic dewar setup described below and shown in Figures 1–10. The temperature of the sample is monitored by using two thermometers and a Lakeshore 332 temperature controller. An alternate temperature controller having the appropriate calibration files could be used if desired. A PC with LabVIEW® installed automatically collects the data at preselected temperature intervals. Other software platforms are compatible with the Agilent and Lakeshore hardware and could be used if desired. A schematic drawing of the experimental platform is shown in Figure 1.

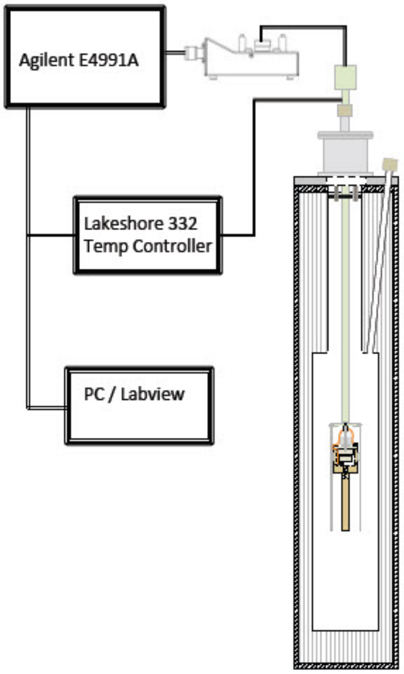

Figure 1. Schematic layout of the experimental platform hardware connections.

The magnetic permeability probe head can accommodate flat ring-shaped samples having dimensions of an outer diameter (OD) ≤ 8 mm, an inner diameter (ID) ≥ 3.1 mm, and $t \leq 3$ mm. The electric permittivity probe head ideally accepts disc-shaped samples with $d = 7.9$ mm and $t \leq 2$ mm. Other flat shapes having dimensions exceeding the capacitive plate diameter of 7.9 mm are acceptable as long as the sample fits within a 13.9-mm circular area.

It is important to minimize or eliminate the possible effects of the atmosphere that is present in the sample space of the probe heads. Since the apparatus is cooled to the temperature of liquid helium, any atmospheric gasses in the same space as the sample will freeze inside the sample space. This presents the possibility of damaging the sample and probe head, and also impacting the accuracy of the measurements. The sample may not completely fill the sample space of the magnetic permeability and electric permittivity probe heads, so it is recommended that the sample be loaded into the sample space by using a helium glove box, if possible. Alternatively, if the sample can be loaded in ambient atmosphere, then the sample/probe head can be placed into a high-vacuum chamber that can be back-filled with helium gas. After either method is used, and following RF calibration of the probe at the probe head connection (see Section 3), attach the prepared probe head to the probe and immediately begin insertion of the probe into the top of the liquid helium dewar so that helium gas evaporating out of the dewar flows around the probe.

Drawings and the schematic layout of the probe and test head connections are shown in Figures 2–7. The probe body is constructed from standard stock G-10 materials. The stainless steel flange is custom made using a “Large Flange” and standard ½-inch slide seal assembly. A rigid stainless steel Micro Coax RF transmission line (part # UT 085B-SS) with male and female SMA connectors runs from the top of the probe to the test head assembly at the bottom. APC-7 connectors are then attached to each end of the transmission line. The top APC-7 connector is connected the Agilent E4991A test head port via an APC-7 cable. The test head is connected to the E4991A via vendor-provided standard cables. The RF magnetic permeability and electric permittivity probe heads are connected to the bottom APC-7 connection on the probe. An oxygen-free, high-conductivity copper “cup” with a 6-inch tail is attached to the probe heads and suspended from the bottom of the G-10 body of the probe. Thermometers are positioned on the test heads to measure the sample temperature. Finally, a G-10 sleeve covers the probe head assembly. Drawings of the dewar and top plate assembly are shown in Figures 8–10.

As seen in Figures 4–6, holes in the copper cup and tail are positioned to allow the flow of helium gas. The G-10 sleeve encloses the sleeve so that a closed path forms for the gas to flow up through the tail and cup, around the sample test head, and up through the neck of the probe. This configuration helps to ensure a uniform temperature in the region of the probe head sample space and the attached thermometers. Currently, the probe cannot be directly heated, hence the temperature cannot be controlled using the Lakeshore 332. This feature is planned for future modifications. At this time, temperature of the sample is controlled by the position of the sample in the neck and body of the dewar. After the dewar is filled by about ½ to ¾ of the top of the belly with liquid helium, and the boil off of the helium becomes steady, the probe is lowered by hand into the top assembly and neck of the dewar. The cold evaporating helium gas flows up through the probe, cooling the probe head and sample. The rate of cooling of the sample can be reliably controlled to a rate of ~ 0.02 K/s by small incremental lowering of the probe through the slide seal. As the tail of the copper piece reaches the layer of gas a few inches above the liquid helium and then lowers into the liquid, the sample will cool at a faster rate, so smaller changes in the positioning of the probe are required. After reaching base temperature, the sample is then warmed up by raising the probe slowly into the top of the neck of the dewar. At temperatures above ~ 150 K the sample can be warmed up further in situ by slowly flowing warm helium gas into the dewar through the liquid helium transfer line port. Slowly flowing the warm gas will minimize acceleration of the burn off of the remaining liquid helium in the bottom of the dewar. A typical sample run in an already cold dewar uses about 6 to 8 liters of liquid helium. At temperatures above ~ 250 K, the probe can be removed from the dewar and allowed to warm up to ambient temperature naturally or with the use of a low-power heat gun/fan.

Prior to acquiring data during the cooling and warming the sample, the Agilent E4991A needs to be configured for the desired measurements following the documentation provided in the operation manual (see Section 3). After the E4991A is configured and the probe is attached and calibrated, a software program (Labview) is run to automatically collect the data as the sample is cooled. The operator determines the temperature intervals at which the data is collected. The software records the data from the Agilent E4991A along with the tempera0ture reading from the Lakeshore 332 and a time stamp.

## 3. CALIBRATION

The probe must be calibrated before performing measurements of samples if the probe, cables or E4991A were disconnected/reconnected after earlier calibration. It is recommended that the probe be re-calibrated before each measurement of a new sample if a long period of time has elapsed since the previous calibrated data run. To perform proper calibration, refer to the following documents, which are available at the Agilent website: [http://www.home.agilent.com/agilent/home.jspx?cc=US&lc=eng.](http://www.home.agilent.com/agilent/home.jspx?cc=US&lc=eng)

| | |
|---|---|
| e4991-90062.pdf | Programming Manual |
| e4991-90090.pdf | Operation Manual |
| e4991-90201.pdf | Installation and Quick Start Guide |

The calibration procedure is performed at room temperature, though it is in principle possible to calibrate at desired fixed temperatures with care by repeating each step at the desired temperature and saving and recalling the calibration file for each temperature selected.

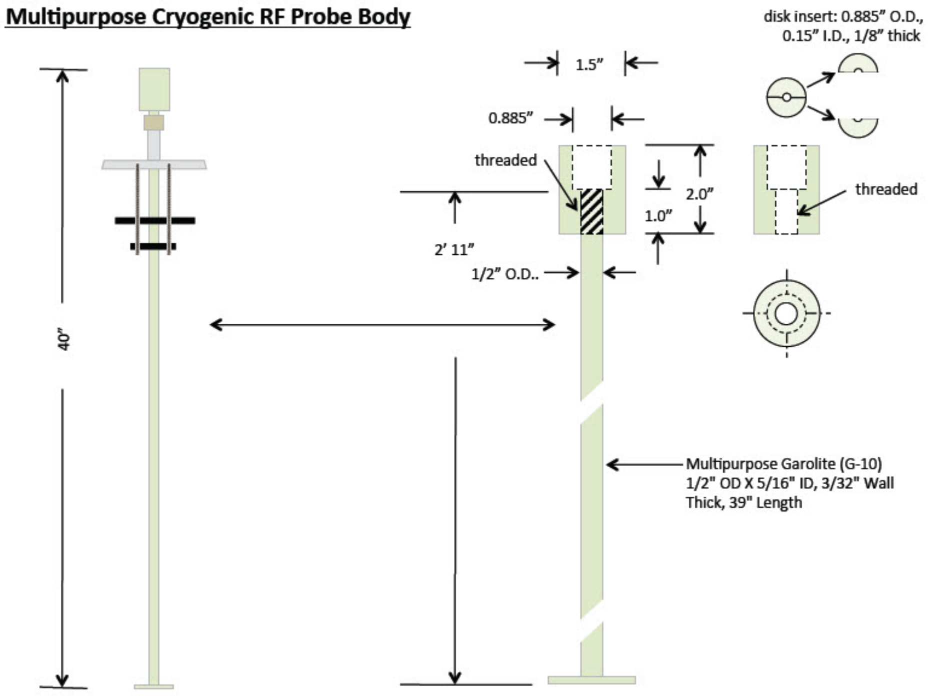

Figure 2. Dimensions and layout of the main body of the RF probe.

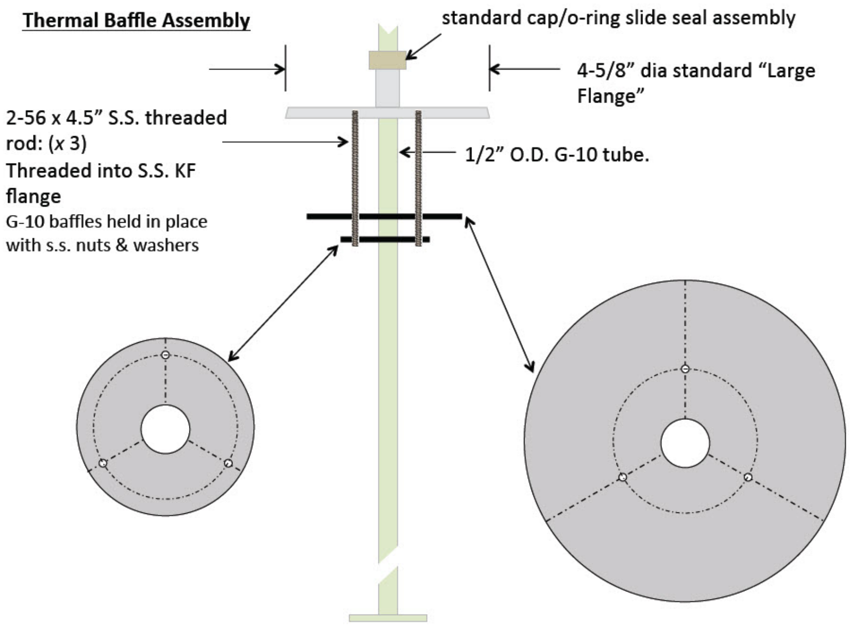

Figure 3. Details of the thermal baffle assembly, slide seal, and s.s. flange sections of the RF probe.

## 4. DIFFERENTIAL MEASUREMENT TECHNIQUE

Due to the difficulty and time required in performing an accurate RF calibration of the probe at all desired temperatures, an alternate method was established for accurate measurement of sample properties.

The probe is calibrated at room temperature using the procedure in the Installation and Quick Start Guide (vendor document e4991-90201), and using the vendor-supplied hardware (open, short, load, capacitance) provided in the Agilent 16195B (7-mm) Calibration Kit. Instead of performing the calibration at the plane of the Agilent E4991A test head, the calibration is made at the APC-7 connector at the bottom end of the RF probe (see Figure 4)—the location where the magnetic permeability or electric permittivity probe heads are attached.

For measurements of bulk or single crystal samples, an empty sample holder is attached following the calibration procedure and the empty holder is measured as a function of temperature over the same conditions as the sample is to be measured. This provides a reference of the changes in the RF transmission properties of the probe as it is cooled. Following this step, the sample is loaded into the same sample holder and the measurements are repeated. The first set of data obtained is then subtracted from the latter set (as a function of temperature) to obtain the RF properties of the sample alone. It is important that the rate at which the probe/sample are cooled is kept below ~ 0.02 K/s and temperature is as uniform as possible.

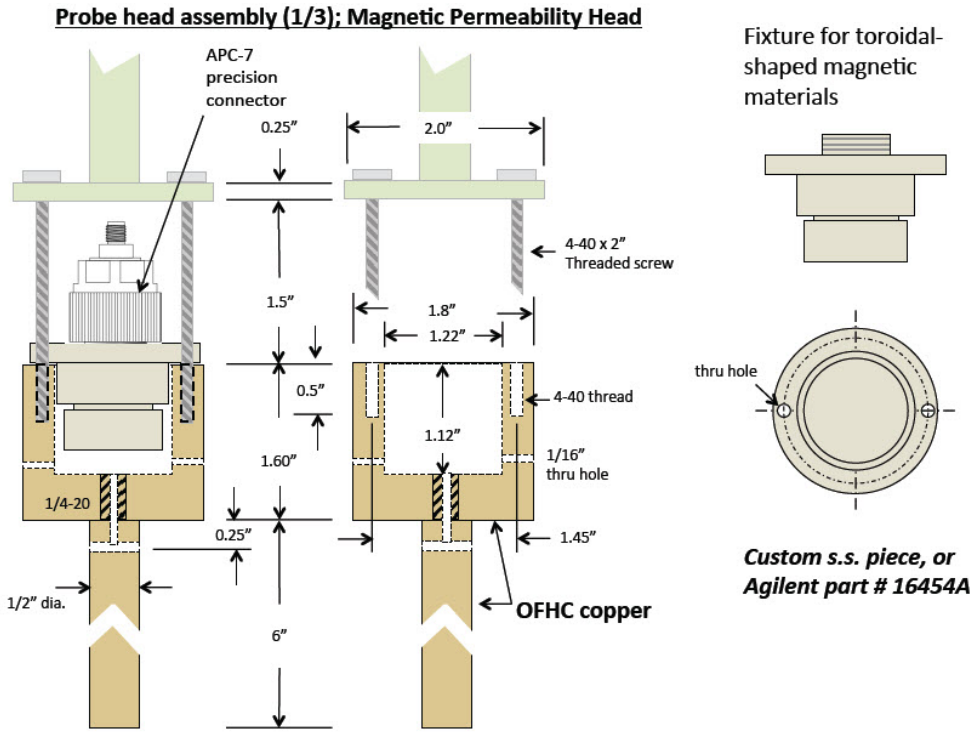

Figure 4. Further details of the RF probe dimensions. Also shown are the dimensions of the bottom copper piece and location of the magnetic permeability probe test head.

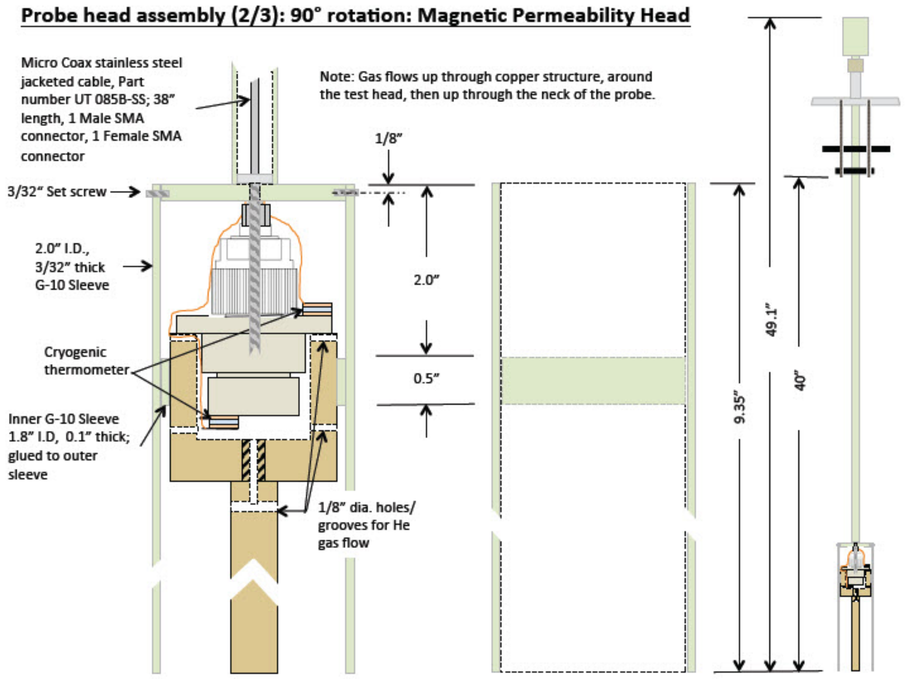

Figure 5. A 90º rotation of the probe assembly shown in Figure 4. Also shown are the locations of the thermometers and the installation of an outer thermal shield (G-10 sleeve).

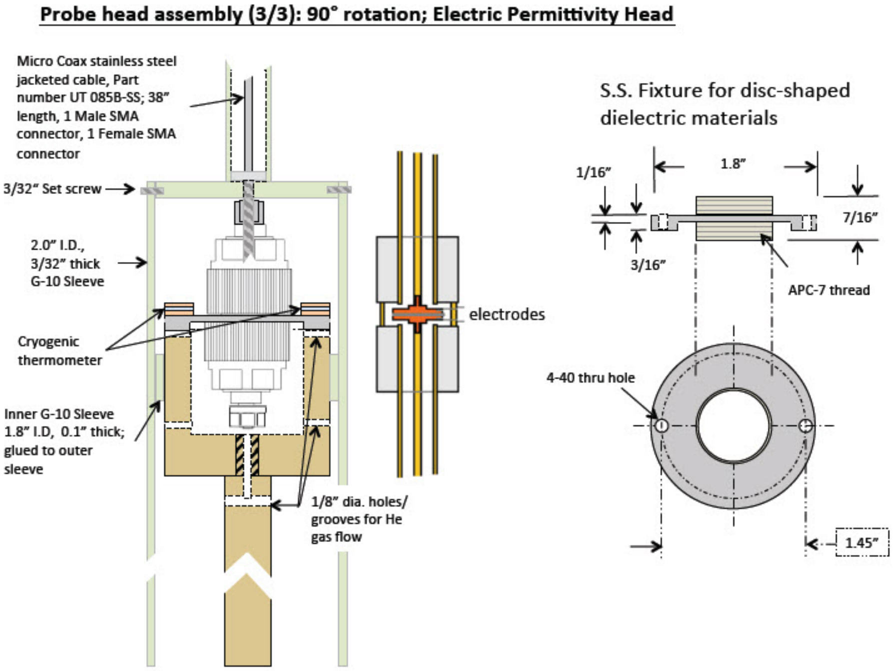

Figure 6. Same view of probe head assembly as in Figure 5 with the electric permittivity probe head installed. Dimensions of the s.s. ring fixture that form the cavity of the probe are shown on the right.

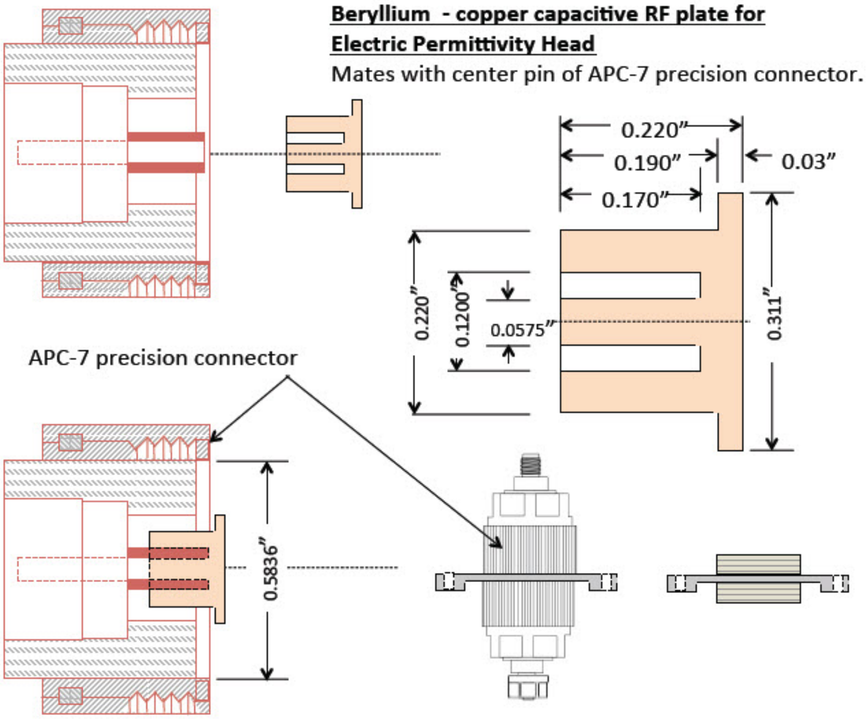

Figure 7. Drawing of the capacitive plate electric permittivity probe head. The custom capacitive plates fit onto the center conductor of the APC-7 connectors. The connectors fasten to the custom stainless steel sleeve forming the body of the probe head. The connectors tighten down, holding the sample is securely in place.

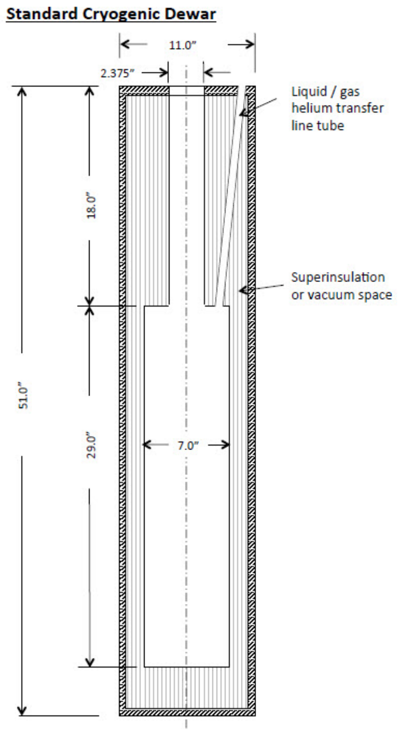

Figure 8. Dimensions of a standard commercially available cryogenic dewar.

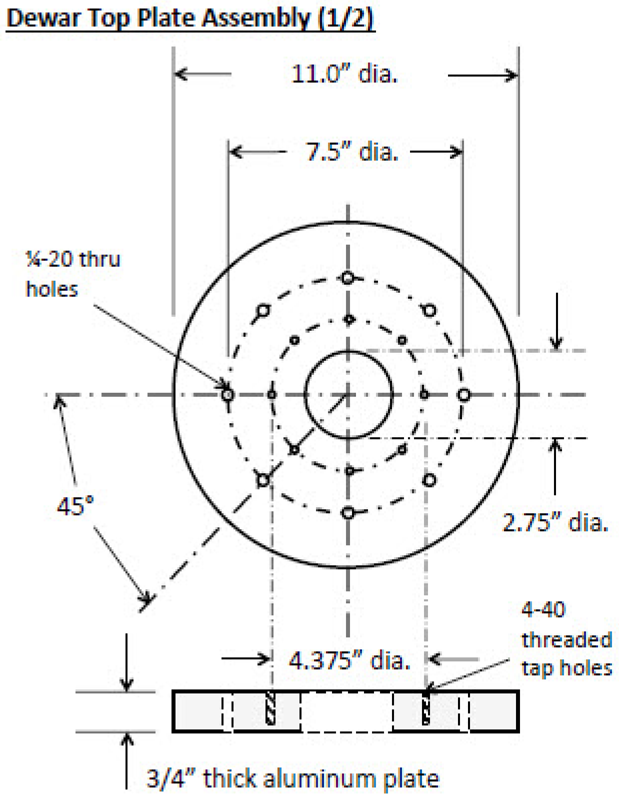

Figure 9. Drawing of the top plate to the dewar.

## Dewar Top Plate Assembly (2/2)

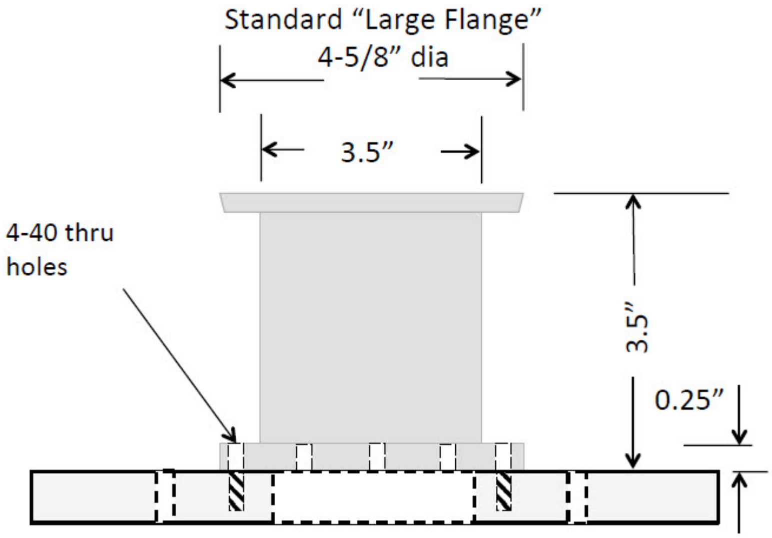

Figure 10. Top plate assembly with an s.s. spool with a mating flange connection to the top plate and the "Large Flange" of the probe slide seal assembly.

For measurements of bulk or single crystal samples, an empty sample holder is attached following the calibration procedure, and the empty holder is measured as a function of temperature over the same conditions as the sample is to be measured. This provides a reference of the changes in the RF transmission properties of the probe as it is cooled. Following this step, the sample is loaded into the same sample holder and the measurements are repeated. The first set of data obtained is then subtracted from the latter set (as a function of temperature) to obtain the RF properties of the sample alone. It is important that the rate at which the probe/sample are cooled is kept below ~ 0.02 K/s and the temperature is as uniform as possible.

For thin film samples deposited on custom ring or disc-shaped substrates (Figure 11), the reference data set is obtained by performing the first set of measurements on the pristine substrate prior to deposition of the film. After film deposition, the same substrate/film is then measured. It is important that the film is prepared as soon as possible following the calibration of the probe and collection of the reference data of the substrate. If the substrates to be used have adequately identical physical dimensions and/or are of a material having negligible RF magnetic/electric properties relative to the sample film, then a reference substrate can be used to obtain the first set of data and the already prepared film sample can be measured immediately afterwards.

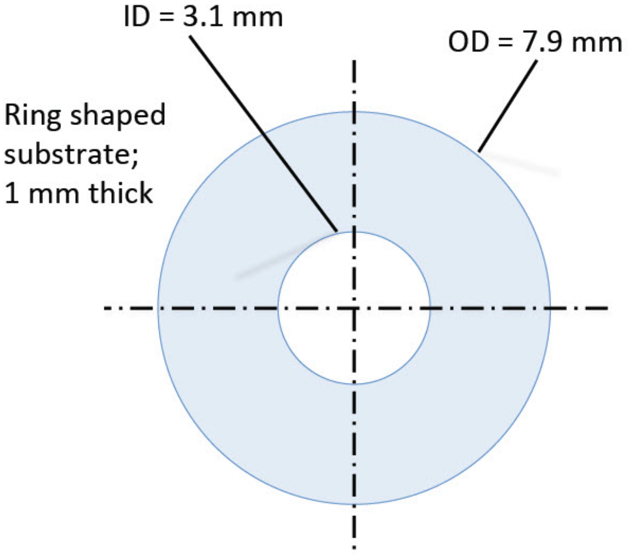

Figure 11. Dimension of single crystal substrates used in the measurement of the magnetic permeability of deposited thin films.

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We describe the design and procedures for use of a simple experimental platform that enables the measurement of the electric permittivity and magnetic permeability of samples over the frequency range of 1 MHz to 3 GHz and at temperatures between 300 to 4.2 K. The platform makes use of an economically designed experimental cryogenic probe capable of utilizing interchangeable probe heads for separate measurements of electric permittivity and magnetic permeability. The system is designed for general use by researchers carrying out research and development and science and technology efforts at SPAWAR Systems Center Pacific (SSC Pacific) that involve the use of materials in radio-frequency responsive applications.

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