

# A Simple Technique for Measuring the Transition Temperature at Microwave Frequencies

Jeffrey M. Pond, Laura H. Allen, and Edward J. Cukauskas  
Naval Research Laboratory, Washington, DC 20375-5000

**Abstract**—A technique is described which enables contactless measurements at microwave frequencies of the superconducting transition. The approach employs an electrically small microwave loop antenna to sense the change in the reflected microwave signal as flux is expelled, due to the Meissner effect, from the superconductor. Advantages of this technique include the ability to measure small areas of a superconducting thin film after photolithographic patterning into a device geometry. This approach is very sensitive in the frequency range from 0.05 GHz to 5 GHz and for some YBCO films a dependence of the transition temperature width on frequency has been observed. Such frequency-dependent signatures may provide valuable information regarding improvements in film deposition and device processing.

## I. INTRODUCTION

Measurement of the superconducting transition temperature,  $T_c$ , is of great interest for the development of superconducting materials and devices processed from those materials. Particularly useful to electronic processing is the ability to measure the transition temperature without patterning or contacting the superconductor. Contactless methods, such as the use of a spiral pancake coil [1] to measure changes in the self inductance have proven to be particularly useful.

Other contactless techniques for measuring the superconducting transition have been employed at radio and microwave frequencies. The use of coils in an LC resonant circuit at frequencies of a few MHz for the detection of the superconducting transition has been reported [2]-[4]. The coil size is comparable to the spiral pancake coil technique [1] making it inappropriate for small patterned samples. In addition, since it relies on resonant coils it can not measure the response over a broad frequency range. Microwave [5] and millimeter-wave [6] transmission and reflection measurements of superconducting films have been used to characterize the superconducting transition. These approaches also sample large areas of a superconducting film and are usually limited in frequency to a waveguide band or less. Microwave absorption in the presence of a modulated magnetic field [7] has been used to make very sensitive measurements of the superconducting transition, but only at a single frequency.

In this paper a high frequency extension of the pancake coil technique is presented which offers several distinct advantages. Similar to the pancake coil, this technique measures changes

in a coil's self inductance due to the Meissner effect in a near-by superconducting film. Rather than a multi-turn coil which is on the order of a 0.3 cm in diameter this technique uses a small microwave loop antenna with an mean diameter of less than 0.05 cm. To maintain sensitivity, inductance is traded off for frequency in order to retain a measurable change in reactance. Although the inductance is orders of magnitude smaller than the spiral pancake coil the frequency used in the measurement is orders of magnitude larger, ~1 GHz rather than ~10 KHz, resulting in a measurable inductive reactance. Since the measurement frequency is 0.05 GHz to 5.0 GHz and the loop size is comparable with the patterned dimensions of passive microwave circuit components, this technique may be particularly useful in the development of microwave applications of superconducting thin films.

## II. EXPERIMENTAL TECHNIQUE

The measurement equipment required consists of a microwave network analyzer. Although a scalar network analyzer can be used, a vector network analyzer has a higher sensitivity as discussed in the following section. The only additional piece of hardware required is the small loop antenna which can easily be fabricated from a piece of small diameter semi-rigid coaxial cable and an SMA-compatible connector. The loop antennas were formed by removing a couple millimeters of the outer conductor of Uniform Tubes UT-34 semi-rigid copper coaxial cable. A little over half of the exposed dielectric was then removed. The center conductor was bent backwards and soldered to the outer conductor forming a loop. An SMA compatible coaxial connector was then attached to the other end of the antenna coax for connection, via coaxial cables to the network analyzer.

A small clamp at the edge of a test fixture is used to position and hold the loop antenna during cryogenic cycling. The loop antenna is separated from the superconducting film by 25.4  $\mu\text{m}$ -thick Teflon sheet. This reduces the possibility of scratching the film due to differential contraction of the various components during the cryogenic cycle. The test fixture is mounted in a two-stage closed-cycle refrigerator. Measuring the reflection coefficient data during the cool down resulted in approximately ten temperature samples per degree which is sufficiently accurate to map out the transition.

## III. ANALYSIS

The measured microwave response is given by the microwave scattering parameter,  $S_{11}$ , which for a one port network is the reflection coefficient and is given by,

$$S_{11} = \frac{Z_L - Z_0}{Z_L + Z_0} \quad (1)$$

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The characteristic impedance,  $Z_0$ , of the coaxial cable system is  $50 \Omega$ . Since the loop is very small,  $Z_L/Z_0 \ll 1$ , and (1) is approximated by

$$S_{11} = -(1 - 2Z_L/Z_0) \quad (2)$$

Assuming the loop can be modeled as a series combination of an inductor,  $L$ , and a resistor,  $R$ , at an angular frequency of  $\omega$  the magnitude of  $S_{11}$  is given by

$$|S_{11}| = \sqrt{(1 - 0.04R)^2 + (0.04\omega L)^2} \quad (3)$$

while the phase angle of  $S_{11}$  is

$$\theta = \tan^{-1}\left(\frac{0.04\omega L}{-(1 - 0.04R)}\right) \quad (4)$$

Since the loop is very small compared to the wavelength and the losses are expected to be small it is reasonable to assume that  $0.04\omega L \ll 1$  and  $0.04R \ll 1$ . This leads to a simplification of (4) and (5) to the form

$$|S_{11}| = 1 - 0.04R + 0.0008(\omega L)^2 \quad (5)$$

and

$$\theta = \pi - 0.04\omega L(1 + 0.04R) \quad (6)$$

Thus,  $\theta$  is much more sensitive to a small change in the inductive reactance than is  $|S_{11}|$ . Since the Meissner effect will induce a change in the field patterns of the loop, the primary change in  $S_{11}$  when traversing the transition temperature region will be a change in  $\theta$ .

#### IV. DISCUSSION OF MEASURED DATA

In order to establish the viability and accuracy of this technique, the superconducting transition of a sputtered film of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO) was extensively studied. This film was deposited onto a (100)-oriented MgO substrate using inverted cylindrical magnetron sputtering. For the deposition, the substrate was silver-pasted onto a stainless steel substrate holder and heated to a temperature of  $800^\circ\text{C}$  (as measured by a thermocouple inserted into a well in the substrate holder) in an atmosphere of equal parts of argon and oxygen with a small amount of hydrogen present. The sputter rate was  $0.08 \text{ nm/s}$ , and the film was  $300 \text{ nm}$  thick. Greater detail of the sputtering process has been reported elsewhere [8, 9]. These deposition conditions yield very smooth, c-axis-oriented YBCO films that have resistivities at  $100 \text{ K}$  of  $150 \mu\Omega\text{-cm}$  and resistive ratios (resistance at  $295 \text{ K}$  to resistance at  $100 \text{ K}$ ) of 2.5 or higher. The transition temperature of this film was measured both inductively by monitoring the change in inductance (at  $10 \text{ KHz}$ ) of a 140 turn, 3mm diameter pancake coil [1] and resistively with a conventional 4-point technique. This film was then mounted in the fixture described in the previous section for the inductive measurement at microwave frequencies. By measuring at many different frequencies at the same time it is possible to determine the frequency at which the loop's response is greatest and also to examine the effect of frequency itself on the transition.

As can be seen in Figs. 1 and 2, both  $|S_{11}|$  and  $\theta$  show a decided signature at the transition temperature. There is adequate sensitivity from  $50 \text{ MHz}$  to  $5 \text{ GHz}$ , although the

signal at  $50 \text{ MHz}$  is close to the limits of measurement sensitivity as presently configured. For clarity representative data at  $0.05 \text{ GHz}$ ,  $0.10 \text{ GHz}$ ,  $0.20 \text{ GHz}$ ,  $0.50 \text{ GHz}$ ,  $1.0 \text{ GHz}$ , and  $2.0 \text{ GHz}$  are shown. As expected,  $\theta$  shows a small change in angle as the temperature of the film moves through the superconducting transition. For these data, compensation for the effects of the offset short calibration were accomplished by inserting an electrical length into the network analyzer's data processing algorithm. It should be remembered that a simple response calibration is not a complete correction for de-embedding a response from measured data. In addition, the electrical length correction for the offset short is only an approximation. These two factors limit the absolute precision of the ordinate reference. Since the calibration was done at  $120 \text{ K}$  it is to be expected that "gain" will appear to occur below the transition temperature since the losses are reduced. The relative scales of the vertical axis are correct, however.

To either side of the transition  $\theta$  is remarkably constant, except at the highest frequencies, which indicates that any phase changes due to thermal expansion or temperature dependent losses are much smaller than those resulting from a

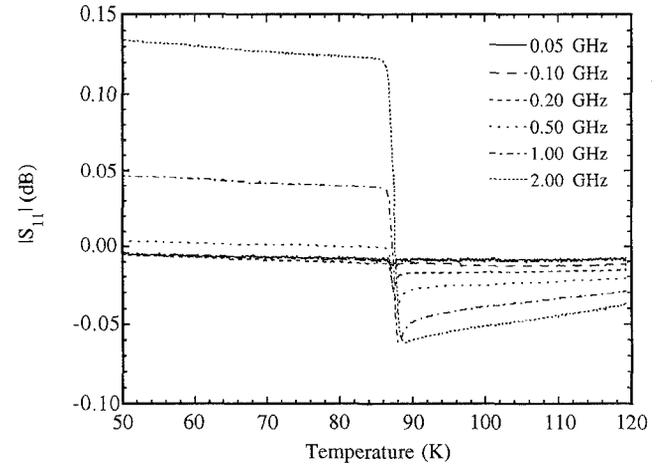


Fig. 1. Measured magnitude of the loop antenna reflection coefficient versus temperature at several microwave frequencies.

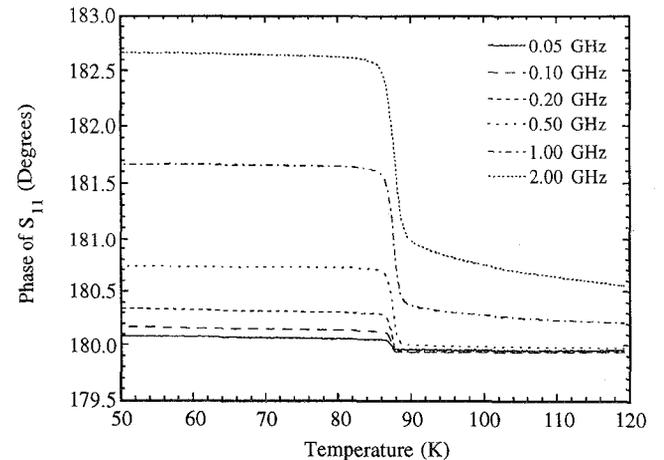


Fig. 2. Measured phase of the loop antenna reflection coefficient versus temperature at several microwave frequencies.

change in magnetic field pattern due to the Meissner effect. At the higher frequencies the temperature dependent losses in the normal state result in the slope observed.  $|S_{11}|$  exhibits a distinctive signature as the temperature is raised through the superconducting transition temperature. As discussed earlier, below the transition temperature it appears as if there is gain but this is merely a result of the calibration being done at 120 K. Above the transition temperature the majority of the energy is still reflected but, particularly at higher frequencies, there is a measurable attenuation in the magnitude of the reflected signal. The losses in the YBCO film while in the normal state are responsible for this increased attenuation. At many of the frequencies measured there appears to be a noticeable null in the  $S_{11}$  right at the transition temperature.

As will be discussed below, there are some notable differences in the shapes of the superconducting transitions at different frequencies. In order to guarantee that these features are not due to some other artifact of the measurement procedure several tests were performed. The first of these was to demonstrate that the thermal environment was isothermal to within the measurement accuracy. By stepping the temperature up and then back down again it is possible to show the effects of thermal time constants, if any, on the signature of the superconducting transition. By stepping the temperature and testing for stability of the temperature in the data acquisition algorithm the thermal history of the procedure was shown to have no effect on the shape of the transition region. Another aspect of this technique which could conceivably affect the shape of the superconducting transition would be the strength of the microwave magnetic field. In order to test this hypothesis, measurements made at different microwave power levels showed no field strength (power) dependence on the shape of the transition.

Since  $\theta$  is a more sensitive measure of the Meissner effect, it is appropriate to focus on Fig. 2. However, the shapes of the curves associated with  $|S_{11}|$  also possess interesting features. In order to compare the transitions at different frequencies it is useful to normalize the data in Figs. 1 and 2. The resultant normalized curves for some of the frequencies are shown in Figs. 3 and 4, respectively. For comparison purposes the normalized superconducting transitions measured with the spiral pancake coil and the four-point resistive method are plotted in Fig. 4. Note that at 0.20 GHz the onset of the transition agrees well with the onset from the 10 kHz coil measurement, about 87.5 K. As the frequency is increased the onset of the superconducting transition moves up in temperature until at approximately 2.0 GHz it appears to agree well with the value of the onset obtained from the four point resistive measurement, approximately 89 K. The other significant feature is that the width of the transition increases with frequency.

It should be remembered that the losses also affect  $\theta$ . Above the transition temperature there is a measurable slope, increasing with frequency, associated with  $\theta$ . There is no obvious reason to expect that the inductance should continue to change with temperature above the transition region. Therefore it must be the microwave losses associated with the film in its normal state that cause this additional signature in  $\theta$ . It might also be expected that below, but very near the transition temperature, the strongly frequency dependent mi-

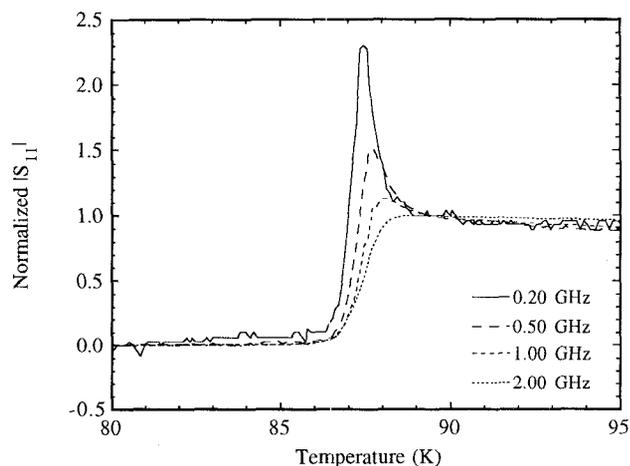


Fig. 3. Comparison of the normalized magnitude of the reflection coefficient at several microwave frequencies.

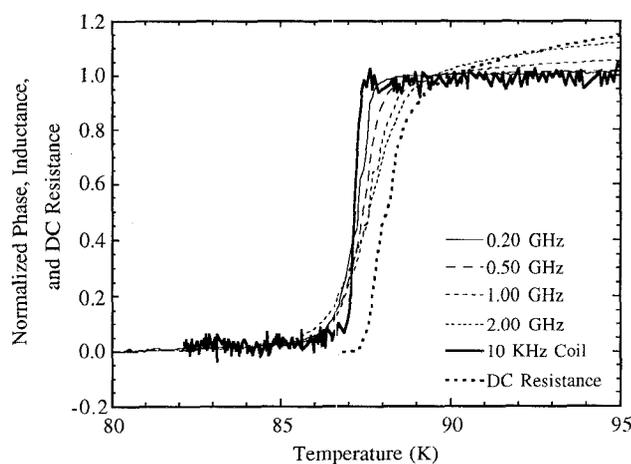


Fig. 4. Comparison of the normalized phase shift of the reflection coefficient at several microwave frequencies compared to the shape of the superconducting transition found using the a spiral pancake coil at 10 KHz and the four-point resistance measurement.

crowave losses of a superconductor could be noticeable at the higher frequencies and hence effect the shape of the  $\theta$ .

The transition mid-points and the transition widths are plotted versus frequency in Fig. 5. The transition widths are defined as the temperature differences between 90 % of the transition and 10 % of the transition. Due to possible thermometry differences the temperature at 10 KHz corresponding to 50 % of the transition can not be expected to lie on a natural extension of the curve obtained using the loop antenna results. It can be inferred that there is approximately a 0.1 K thermometry error between the cryogenic environment used at 10 KHz and that used for the loop antenna measurements. This is a very small error and local inhomogeneities in the transition temperature of the superconducting film could also account for apparent transition temperature variations of this magnitude.

The measured frequency dependence of the superconducting transition width is not well understood. The transition given by the four point resistive technique, of course, corresponds to the temperature at which some path becomes

superconducting. Whereas, the 10 KHz coil senses the transition of a relatively large area which must be entirely superconducting in order to expel flux and yield the characteristic change in coil inductance. The loop antenna senses a much smaller region of superconducting film. This could certainly account for the transitions lying between the transitions measured by the four point resistance technique and the 10 KHz coil. It may be that the capacitance associated with the grain boundaries provides increased coupling between grains at the higher microwave frequencies resulting in the beginning of measurable flux expulsion at higher temperatures. An alternative possibility is that from (6) it is known that the losses can also affect the phase angle. It should be remembered that the model of the loop, a simple series RL circuit is overly simplistic and obviously does not contain the correct frequency dependence. A more complete model would have to include separate inductances and resistances describing the superconducting film in both superconducting and normal state. These inductances and frequency dependent resistances are coupled to the self inductance of the loop antenna. In addition, there will be a capacitance between the loop antenna and the superconducting film as well as parasitic losses due to the surrounding environment. To date a model has not been identified which fully accounts for these effects. It is expected that measurements of a number of YBCO samples fabricated under different conditions may help provide further insight.

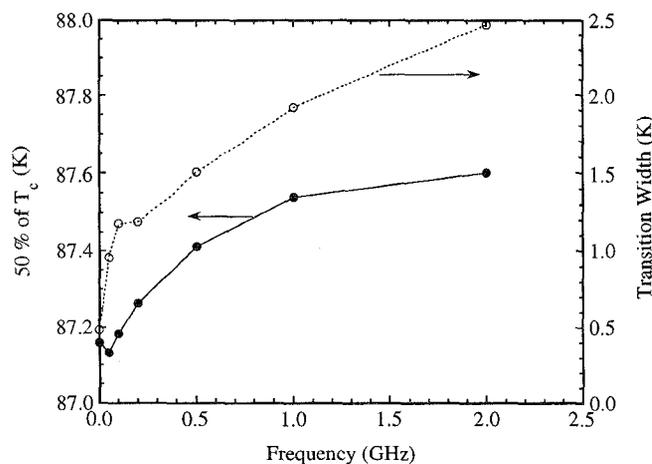


Fig. 5 Plot of the temperatures corresponding to 50 % (●) of the superconducting transition as a function of frequency. The width of the transition (○) is referenced to the right hand vertical axis scale and is defined as the difference between the 90 % temperature and the 10 % temperature.

#### IV. CONCLUSIONS

The variation on the inductive technique presented here has advantages over the conventional spiral pancake coil approach. The instrumentation required is trivial for any microwave laboratory which is equipped with either a vector

or scalar network analyzer. Fabrication of the loop antenna is simple and it is very rugged. Adequate sensitivity has been shown to exist over a broad frequency range. The small size of the loop could facilitate using this approach to map out local variations in the transition temperature over the surface of a thin film. This approach has been applied to monitor changes in the transition due to photolithographic patterning and other device processing [10].

Future efforts to refine this technique include the implementation of the loop in thin film form. With a smaller loop higher frequencies can be employed. Significant increase in sensitivity should result from the use of a thin film loop. At present the loop is constrained by the diameter of the coaxial cable to be separated from the superconducting film by approximately the radius of the coax cable. This is, of course, the same dimension as the width of the loop. A thin film implementation would allow for the separation to be much less than the loop width.

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