R & D STATUS REPORT

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CONTRACTOR: David Sarnoff Research Center

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SHORT TITLE OF WORK: High Performance YBCO Films

REPORTING PERIOD: Period 9/24/91 to 1/31/92

DESCRIPTION OF PROGRESS:

The following progress report describes HTS sample characterization techniques that have been developed during this period for both the high microwave frequency range (Section I) and for low frequencies below 1 GHz (Section II). For the microwave range, the resonator configuration consists of a cylindrical dielectric resonator probe in direct contact with an HTS sample and surrounded by a cylindrical metal (non-HTS) shield. The technique will be used for the nondestructive testing of HTS films on dielectric substrates with very large areas. Furthermore, it probes the quality of the HTS film over a small selective area (~3mm) with high resolution of the surface resistance. It has the ability to measure surface resistance at least an order of magnitude better than gold at millimeter wave frequencies. Our current set up is suitable for substrate diameters up to 5 cm limited by the size of the cryogenic cooler. For characterization at low frequencies below 1 GHz, we have designed and built self-resonant spirals patterned on HTS samples that require very little substrate area. A resonant frequency as low as 30 MHz with a spiral requiring only 1 cm^2

area is possible. Q values as high as 20,000 have been measured with these spirals patterned on HTS samples.

Due to a change in subcontractors no specific progress in thin film deposition on new low-ε substrates is reported for this period. We presently are in the final negotiations with Neocera, Inc. located at College Park in Maryland for the thin film deposition and optimization task of the program.

I. Superconductor Sample Characterization at Microwave Frequencies

A. Summary of Previous Characterization Techniques:

Different characterization techniques for microwave surface resistance were described by [1], and included the TE_{011} mode cavity, the disc resonator, the stripline resonator, the coaxial line resonator, the cavity perturbation technique, and the suspended meander line resonator technique. However, all these various techniques have some drawbacks as will be described in the following.

The end plate replacement measurement technique with a TE_{011} mode cavity has low sensitivity and requires relatively large sample areas, especially at low frequencies. The cavity is only partially superconducting and the Q is dominated by the loss of the metal side wall [2]; for example, the cavity Q will only improve by roughly 10% if one copper plate is replaced by an ideal superconducting plate. The loss dominance of the side walls reduce the measurement accuracy and sensitivity. Higher resolution can be achieved when the surrounding walls are made of an excellent superconducting material such as Niobium at 4K. But this measurement scheme is mechanically elaborate to allow measuring the material's performance at 77K [3].

The disc resonator method was first suggested by Sarnoff and used for bulk material evaluation [4,5]. It was modified by HP [6] for thin film evaluation as well. Two discs were used and were separated by a very thin dielectric material (25 to 50μm thick); hence, the overall resonator's Q is dominated by the conductor's loss. Orders of magnitude improvement in the measured Q can be obtained when copper discs are replaced by superconducting ones. However, this method requires two sample areas, suffers from air gaps, and evaluates the whole sample at once and not a selective area. Radiation and closely spaced higher order modes limit the resolution and accuracy of this measurement scheme.

Stripline [7] and suspended meander line [8,9,10] resonators require thin film patterning. Both require the ability to vary the coupling in order to measure the unloaded Q, thus complicating the feed arrangement. The stripline resonator may require superconducting ground planes for higher accuracy. The suspended substrate scheme, on the other hand, avoids this requirement by reducing the effect of the ground planes on the overall Q; hence, normal ground planes can be used. These printed resonator tests evaluate the thin film material in a configuration very close to their final use, where the measured Q includes the effect of definition and patterning. Unfortunately, these tests are very elaborate and destructive and not suitable for fast feedback.

We have investigated a noncontacting, nondestructive simple probing measurement technique using an open resonator, where a confocal resonator sustains a Gaussian beam at resonance. An arrangement of a plane mirror and a superdirective hemispherical antenna separated by half-the confocal length is a special case of the confocal resonator. The Gaussian beam's waist (minimum spot size) will be located at the plane mirror surface where a wafer HTS sample can be inserted; the reflection coefficient of the open resonator is used as an indication of the surface resistance of the sample under test. Depending on the size of the beam's waist, wafer uniformity can be tested. However, the practical minimum spot size is approximately one wavelength [11]. At 30 GHz this minimum spot size corresponds to a diameter of 2 cm, which is relatively large. Different research groups are currently developing open resonators at > 90 GHz to assure smaller spot size probing schemes [12].

B. Our New Measurement Technique

A dielectric resonator (DR) probe measurement scheme described in the following pages is simple, sensitive, and capable of measuring selected areas of large samples in a nondestructive manner. The probe operates at high microwave or mm-wave frequencies and has increasingly smaller dimensions at higher frequencies of operation. State of the art high-T_c superconductor deposition techniques are currently capable of large sample deposition > 5 cm; this capability is stimulating the need for uniformity evaluation where samples need to be measured at various selected wafer sites and both the relative and absolute measurements can be utilized.

The basic principle of the DR probe method is well known and has been widely used for dielectric constant and loss tangent measurements of small dielectric samples [13]. We have used it previously for the characterization of dielectric materials suitable for superconductor deposition at cryogenic temperatures [14]. Fiedziuszko et al [15], and Kobayashi et al [16] have used dielectric rods mounted between normal metal and superconducting plates to measure the surface resistance of high T_c films. A TE_{011} mode is used for measurements and can be easily identified, and is relatively insensitive to small gaps between a dielectric rod and the bottom ground plane where no axial currents across any possible joints exist. However, the reproducibility of these measurements and the dominant effect of the normal ground plane limited the sensitivity of this measurement technique. We have modified Kobayashi's test to separate the high ε_r dielectric resonator rod from the normal metal ground plane with a thin low ε_r support rod and include the dielectric resonator in a cylindrical metal cavity to establish a $TE_{01(1+d)/2}$ mode. Higher resolution and accuracy is obtainable with this mode compared to the TE_{011} mode used by Kobayashi. A drawing of the resonator structure is shown in Fig. 1.

The HTS sample evaluation fixture uses a 3 mm dia x 1.36 mm long dielectric resonator located in a Au-plated OFHC copper cavity. The cavity is approximately 8.5 mm in dia. and 3 mm deep. A low dielectric constant, low loss plunger is mounted in the center of the cavity. A beryllium copper leaf spring located below the cavity applies pressure to force the resonator against the film under test. The test fixture is depicted below in Fig. 2.

The sample under test is clamped against the fixture to cover the open end of the cavity. Coupling to the magnetic fields in the cavity is provided by two probes in recessed bores at the periphery of the cavity. Coupling is adjusted by advancing or retracting the penetration of the probes relative to the dielectric resonator.

The test fixture is mounted in a vacuum cryostat which uses a closed cycle refrigerator and associated temperature controller to provide operation from 13K - 300K. The cryostat is diagramed below in Fig. 3.

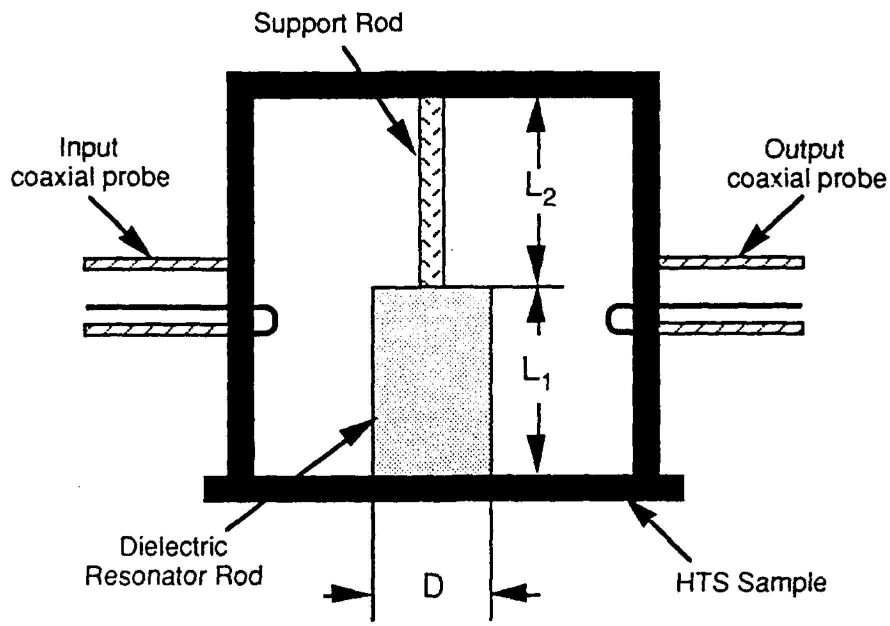

Fig. 1 Dielectric resonator probe structure.

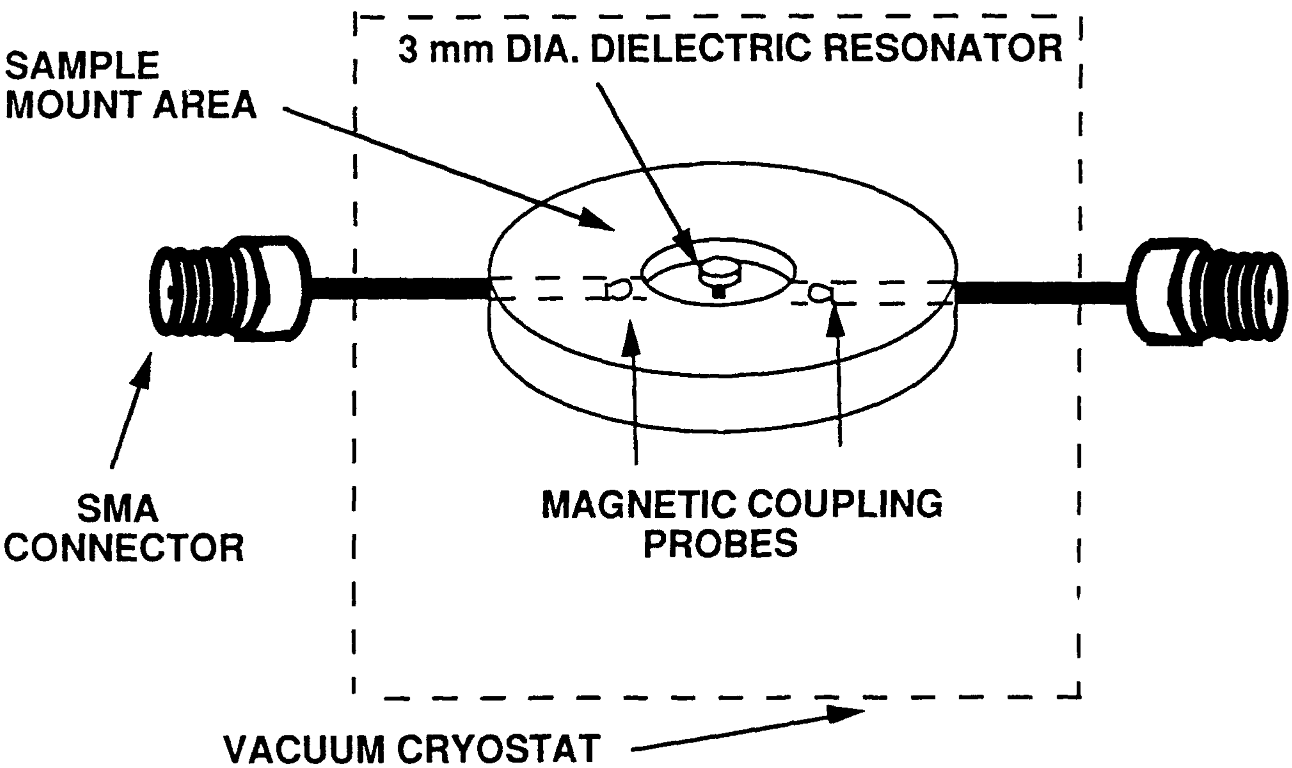

Fig. 2 HTS sample evaluation fixture.

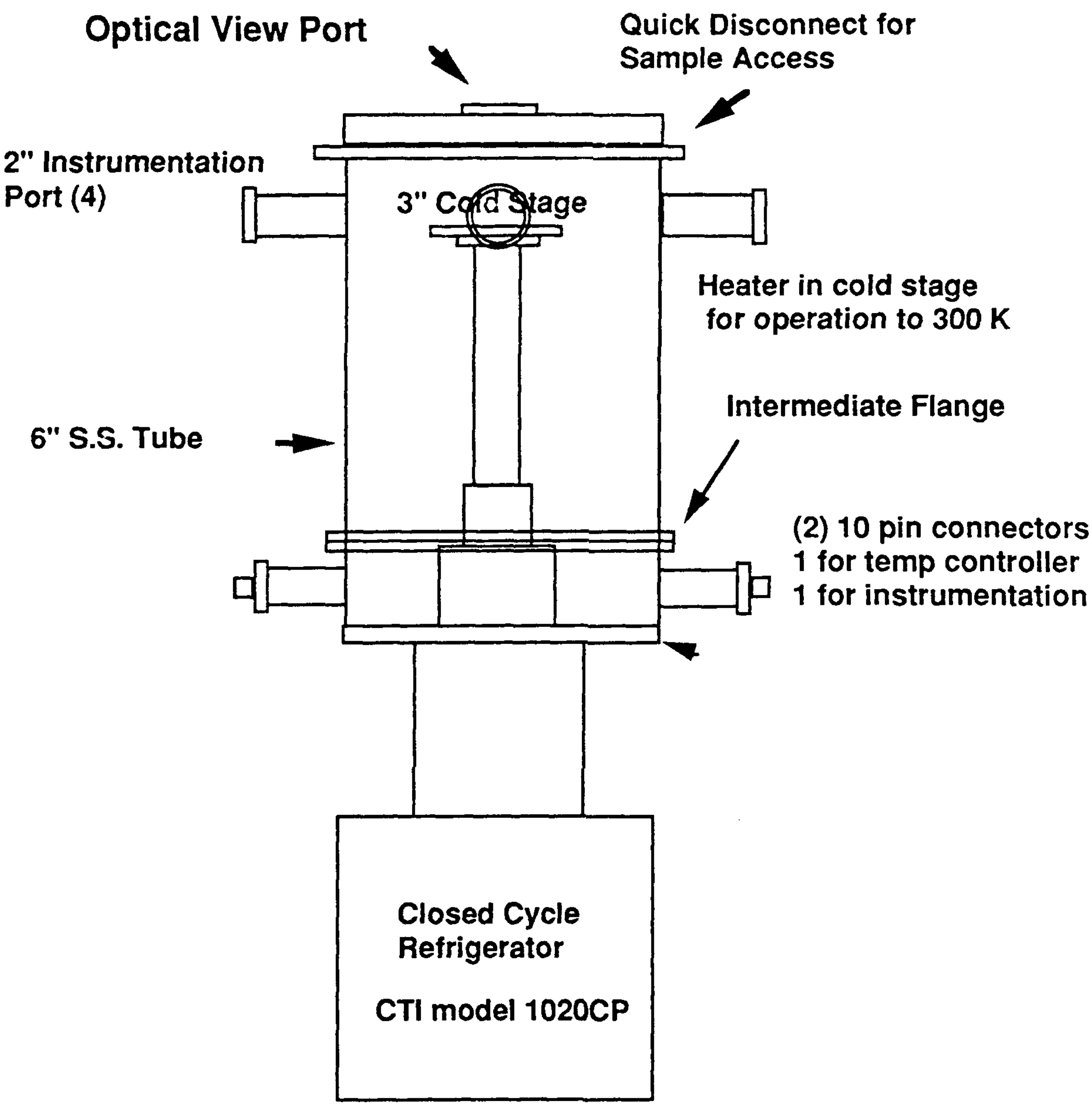

Fig. 3 Vacuum cryostat used for sample evaluation.

Testing of superconducting microwave circuits is routinely carried out using the Hewlett Packard 8510B Network analyzer and associated cryogenic apparatus. Temperature ramps and other low temperature measurements are performed with a closed cycle refrigerator system.

The test instruments are computer-controlled and all test data is logged to a disk for later analysis. The system is capable of monitoring a variety of test parameters while ramping temperature and tracking the sometimes temperature-dependant center frequency of the DUT. The dielectric resonator technique for evaluating HTS samples makes use of the test systems' ability to monitor and track the Q factor of the resonator while a temperature ramp is performed.

The resonant frequency of the $TE_{01(1+d)/2}$ is determined rigorously by the mode matching method, and analytically can be derived from the solution of the following characteristic equation given by [17]:

$$\det H (f_o, D, L_1, L_2, d, \varepsilon_r) = 0 \qquad (1)$$

As depicted in Fig. 1, L_1 is the length of the DR rod, D is its diameter, L_2 is the length of the post (distance between the top of the rod and the shield), d is the diameter of the cavity (d = 3D), ε_r is the relative dielectric constant of the rod. Computer programs were developed to solve the eigen value equations and to determine the resonant frequencies of the fundamental and higher order modes. Perturbation techniques were used to evaluate the Q of the resonant structure.

Dimensions of the test set-up were optimized to measure surface resistance values as low as one order of magnitude better than copper at 26 GHz. The sample bottom plate was assumed to have one order of magnitude better than copper and the top plate is made out of copper and is placed at a distance of L_2 from the top. The ceramic rod material was assumed to have ε_r=24.5, diameter D=3 mm and length L_1=1.36mm. The effect of the shield location "L_2" on the measured Q is shown in Fig. 4, where L_2= 2mm is shown to be adequate to optimize the performance of the resonator structure. Figure 4 shows the effect of the shield location L_2 on the predicted conductor's Q "Q_c" and the total Q "Q_{tot}". The calculations were done at room temperature and assumed a dielectric Q ("Q_d") of 10,000. For L_2=2mm, the predicted Q_{tot} is 3600. Increasing L_2 beyond 2mm is avoided as it will influence the propagation of higher order modes; the latter's close-proximity to the desired fundamental mode will degrade the accuracy and resolution of these measurements.

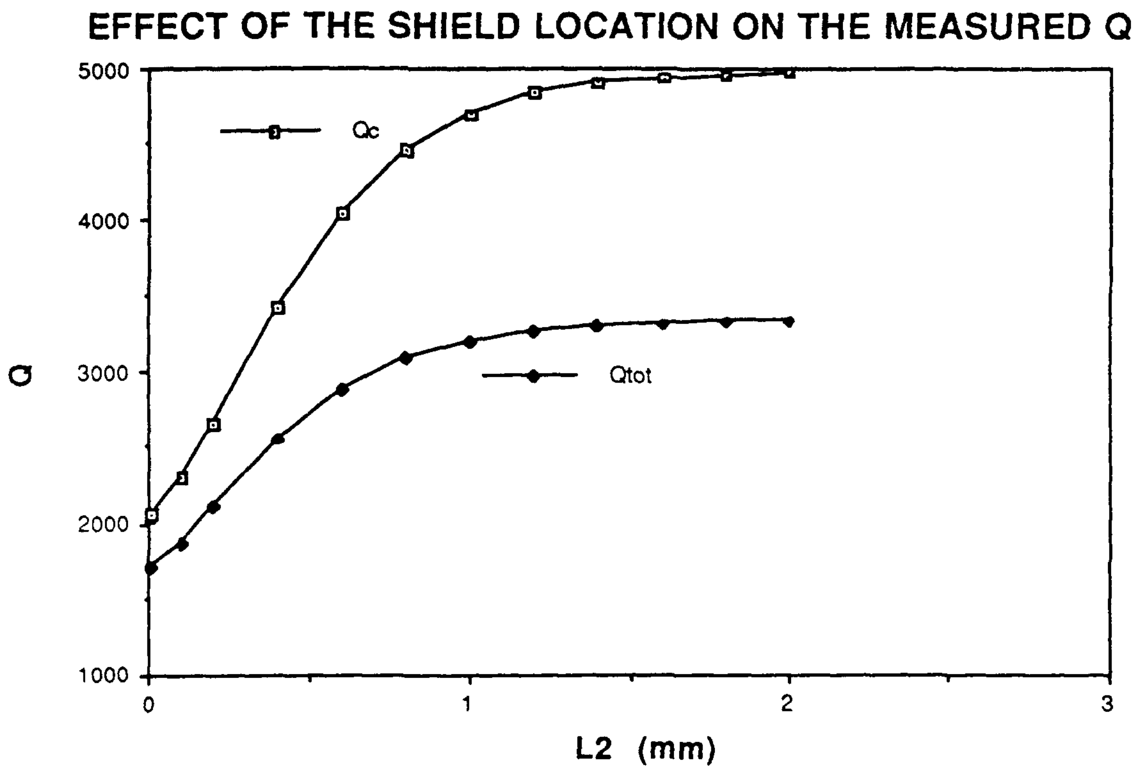

Fig. 4 Effect of shield location on conductor Q and total Q of dielectric probe resonator structue.

(Tan δ=0.0001; T=293K; relative resistivity is 1 for both the top and bottom plates).

CALIBRATION STEP:

For the calculation of the surface resistance of the bottom HTS sample, both the temperature-dependent surrounding metal conductivity and the loss tangent of the DR material are needed. The conductivity of the plates at room temperature and at 77K were calculated as shown by Kaur [14] using a modification of Courtney's technique [13] to be 4×10^7mhos/m at 290K and 15×10^7 mhos/m at 77K. The conductivity of gold plated brass was extrapolated and represented by a conductivity equation given by

$$\sigma = \frac{0.877\ \sigma_o}{1.+0.00339\ (T-293)} \qquad (2)$$

where $\sigma_0 = 4.562 \times 10^7$ mhos/m is the theoretical conductivity of gold at room temperature [15]. The 0.877 factor is calculated from the ratio of 4.562 to 4.0 and the constant 0.00339 is calculated by using the measured conductivity at 77K. Table 1 shows the conductivity of the plates versus temperature obtained using the above equation along with the theoretical conductivity for bulk gold [15]. Fig. 5 shows the graphs of both the conductivity of gold-plated brass and the theoretical conductivity of gold.

Table 1

Conductivity of Gold Plated Brass Versus Temperature

Temp. (K)	Calculated conductivity of gold plated brass (mhos/m)	Theoretical conductivity of gold (mhos/m)
293	4.000×10^7	4.562×10^7
120	9.675×10^7	•
110	10.539×10^7	•
100	11.572×10^7	•
90	12.830×10^7	•
77	14.942×10^7	23.084×10^7

We have used the published loss tangent data of BMT DR rods as a function of temperature shown in Fig. 6. The DR material has a Q of 10,000 at 293K and improves to over 100,000 at 10K. The sensitivity of the experiment is greatly enhanced by the significant Q_d improvement. At room temperature, the dielectric and conductor losses are comparable. Upon cooling, the DR material's Q improves and the dielectric losses are significantly reduced, which results in an improvement of measurement sensitivity.

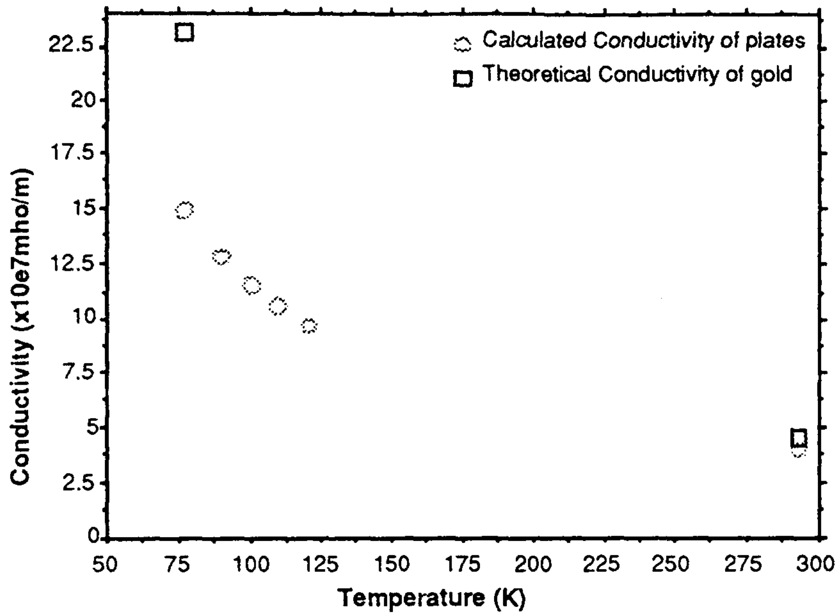

Fig. 5 Conductivity of gold plated brass versus temperature

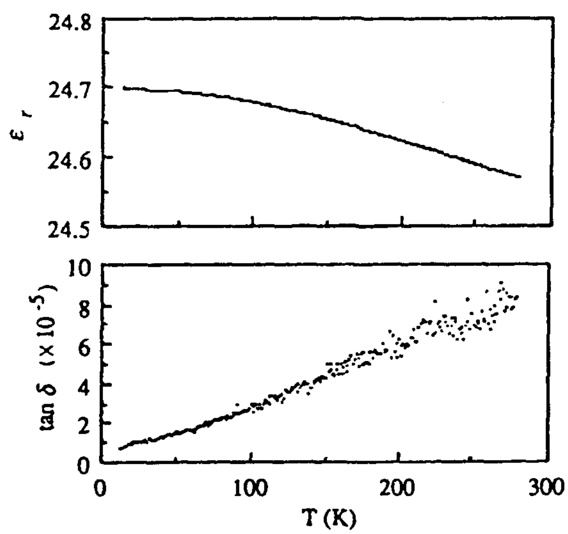

Fig. 6 Properties of BMT, DR rods vs. temperature.

We have measured the total unloaded Q of a gold plated OFHC plate as a function of temperature as shown in Fig. 7. Close agreement between predicted and measured results is due to the calibrated gold conductivity and dielectric loss data as a function of temperature. We, accordingly, can evaluate the relative resistivity of different samples compared to that of gold using Fig. 8.

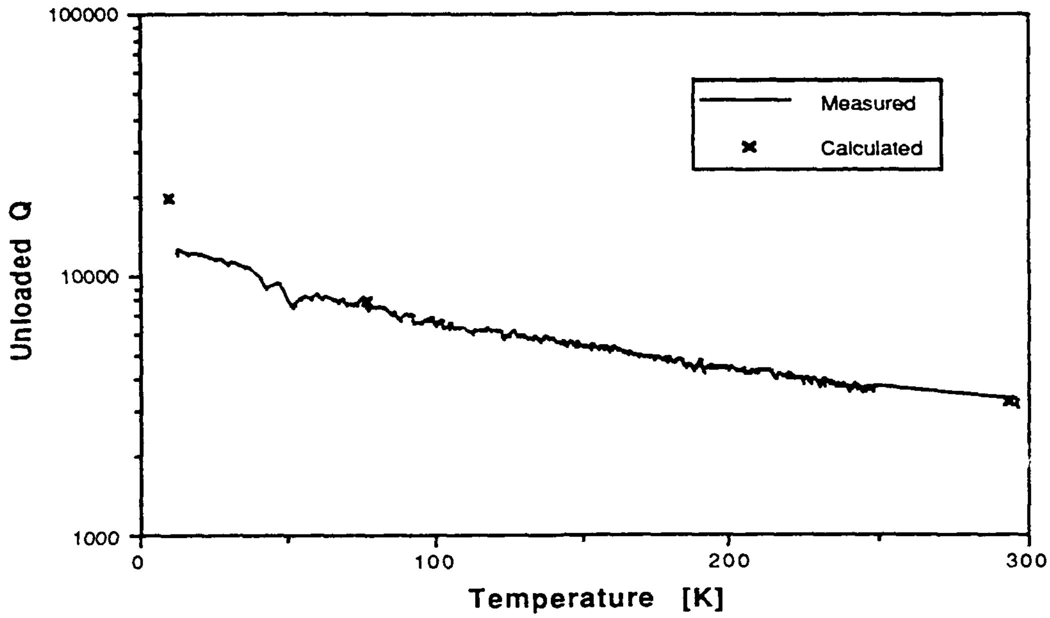

Fig.7 Total unloaded Q of resonator structure using a sample gold-plated OFHC plate.

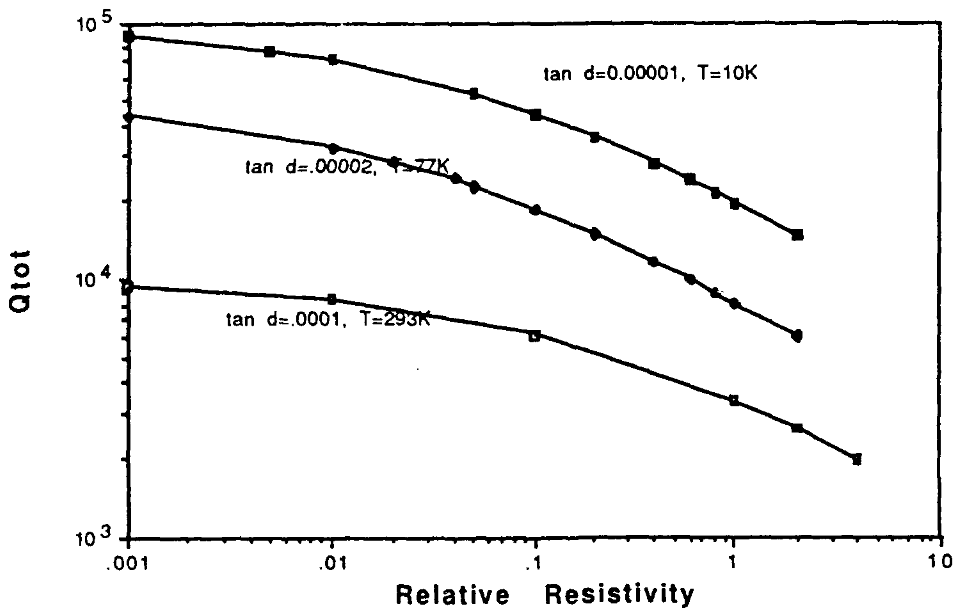

Fig. 8 Total Q of resonator structure versus relative resistivity (compared to gold) of HTS sample.

HIGH T_C MATERIALS EVALUATION

We have measured the Q as a function of temperature of a high Tc superconducting thin film deposited on a sapphire substrate. This sample was obtained from Neocera representing early deposition results on a concurrent NASA program. The measured Q is shown in Fig.9 as compared to that of gold. The material is almost an order of magnitude better than gold at 10 K, but shows poor surface resistance at 77K.

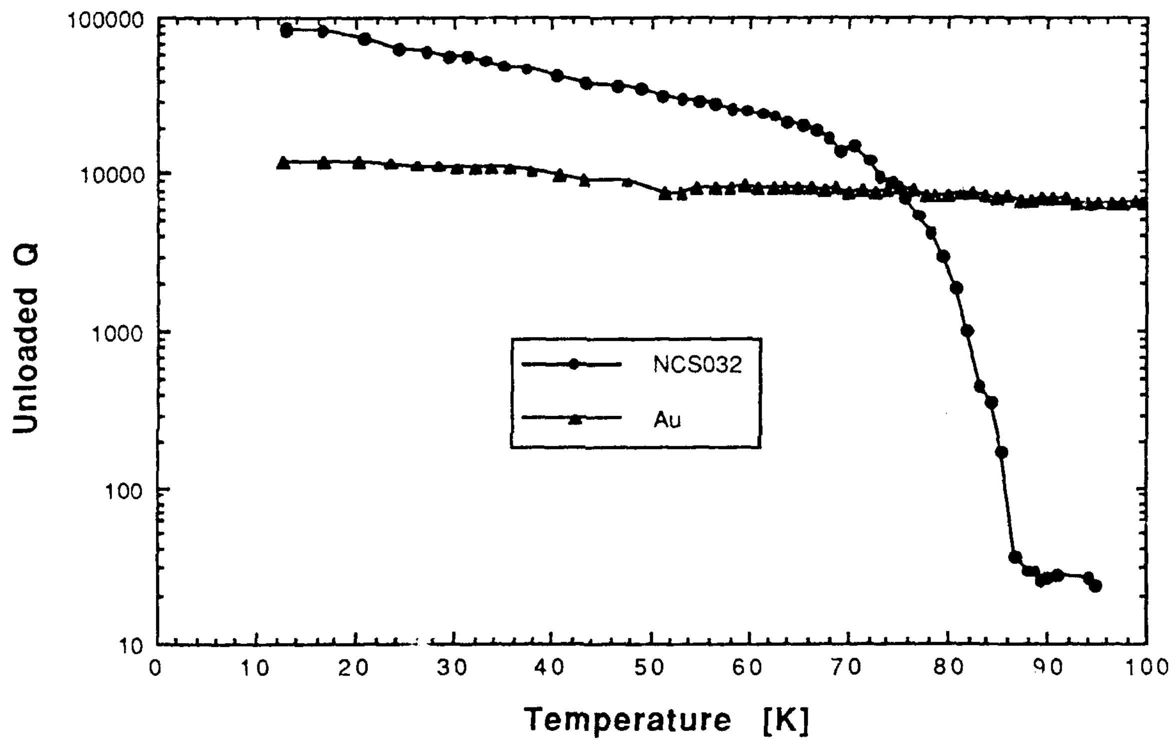

Fig.9 Measured Q of high T_c superconducting film on sapphire with a comparison to gold.

In summary, our measurement goals can be summarized in the following:

- simplicity
- small selective area measurements (~3mm)
- wafer probing over 5 cm diameter
- high surface resistance resolution
- nondestructive testing
- ability to accurately measure at least an order of magnitude improvement in surface resistance over gold at mm wave frequencies.

II. SELF RESONANT SPIRALS FOR HTS SAMPLE CHARACTERIZATION AT LOW FREQUENCIES

Meander lines have been widely used in the measurement of the surface resistance of HTS thin films. Determined by maximum size considerations, an area of 1 cm^2 is generally preferred. This provides a measurement range for very narrow HTS lines from approximately 1 to 12 GHz. For lower frequencies, into the ten's of MHz range, meander lines become much too bulky. We, therefore, started to explore self-resonant spirals as very small low frequency resonators.

A typical print-out for the resonances of a small, self-resonant spiral within an area of less than 1 cm^2 is shown in Fig. 10, together with its relevant dimensions. This geometry permits the fabrication of low frequency resonators in a minimum volume. Since the fields are closely bound to the substrate, provided the dielectric constant is relatively high, ground planes and shielding have little effect on the resonator losses. Such spirals are thus very well suited for measuring the surface resistance of HTS films at HF and UHF frequencies. Since very small high-Q resonators would also have important systems applications for lower frequency satellite communication and COMINT receivers, we decided to start with some initial resonator experiments in the interim time until the first HTS films become available from Bellcore, our original subcontractor for the fabrication of HTS thin films.

For reasons outlined in the section on changes in key personnel, we elected, after some initial delay, to have the process development for the HTS films performed by Neocera instead of Bellcore. The first resonant spirals were, therefore, patterned using available YBCO films on $LaAlO_3$ from Neocera. Fig. 11 shows the unloaded Q of the spirals as a function of frequency for two different films. The spirals consists of 50 μm wide lines with 50 μm spacings. With a total of 25 turns, the overall diameter is less that 6 mm, resulting in a fundamental resonant frequency of only 140 MHz.

For resonant frequencies above 900 MHz the frequency response is approximately as expected, with the Q decreasing towards higher frequencies. The measured Q's of 20,000 around 1 GHz agree well with those measured on the best meander lines. The unexpected result is the rapid decline in Q towards frequencies below 800 MHz. Since both spirals show the same effect, it is unlikely to be caused by a mechanical defect in

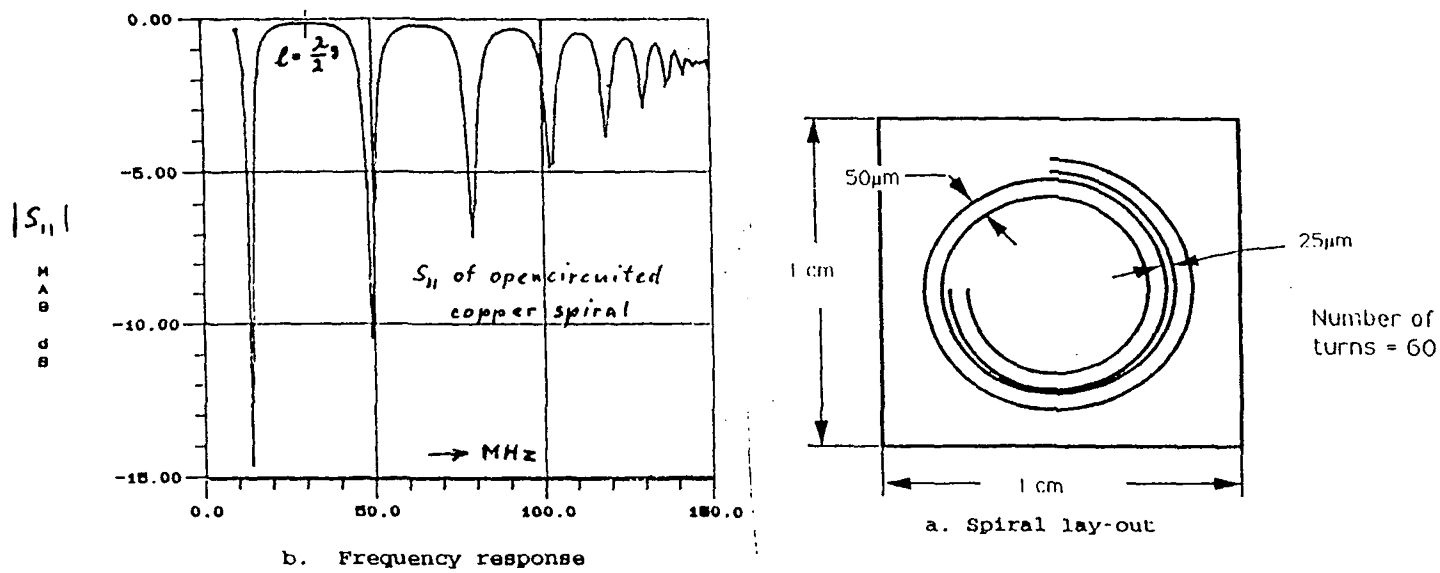

Fig. 10 Calculated response of self-resonant spiral.

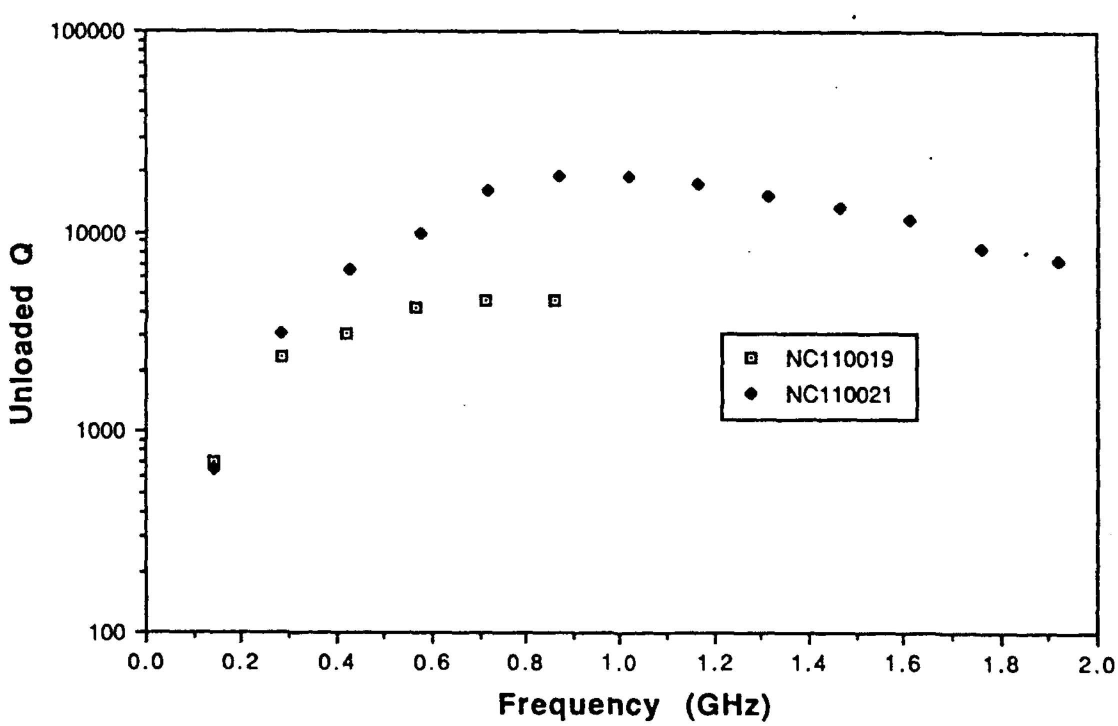

Fig. 11 Measured Q of two sample spirals on $LaAlO_3$.

the patterning. Careful probing around the spiral with lossy material did not indicate any losses due to radiation, which should be completely negligible at the lower frequencies as found from theoretical considerations.

Similar sharp drops in Q below 3 to 5 GHz had previously been observed in certain HTS meander lines. Fig. 12 is an example of such an earlier measurement. In both cases the drop-off does not seem to be power-related; i.e. a reduction in power level by 15 dB (limited by the measurement arrangement) does not significantly affect the Q. Our present preliminary thinking is that the drop-off below a certain frequency is materials-related, possibly having to do with poor grain boundary contacts. Other high-quality meander lines do no show this drop-off within the measurement range down to 1.4 GHz. The spirals, having much lower resonance frequencies, may thus show material defects that are not normally visible in regular microwave circuits.

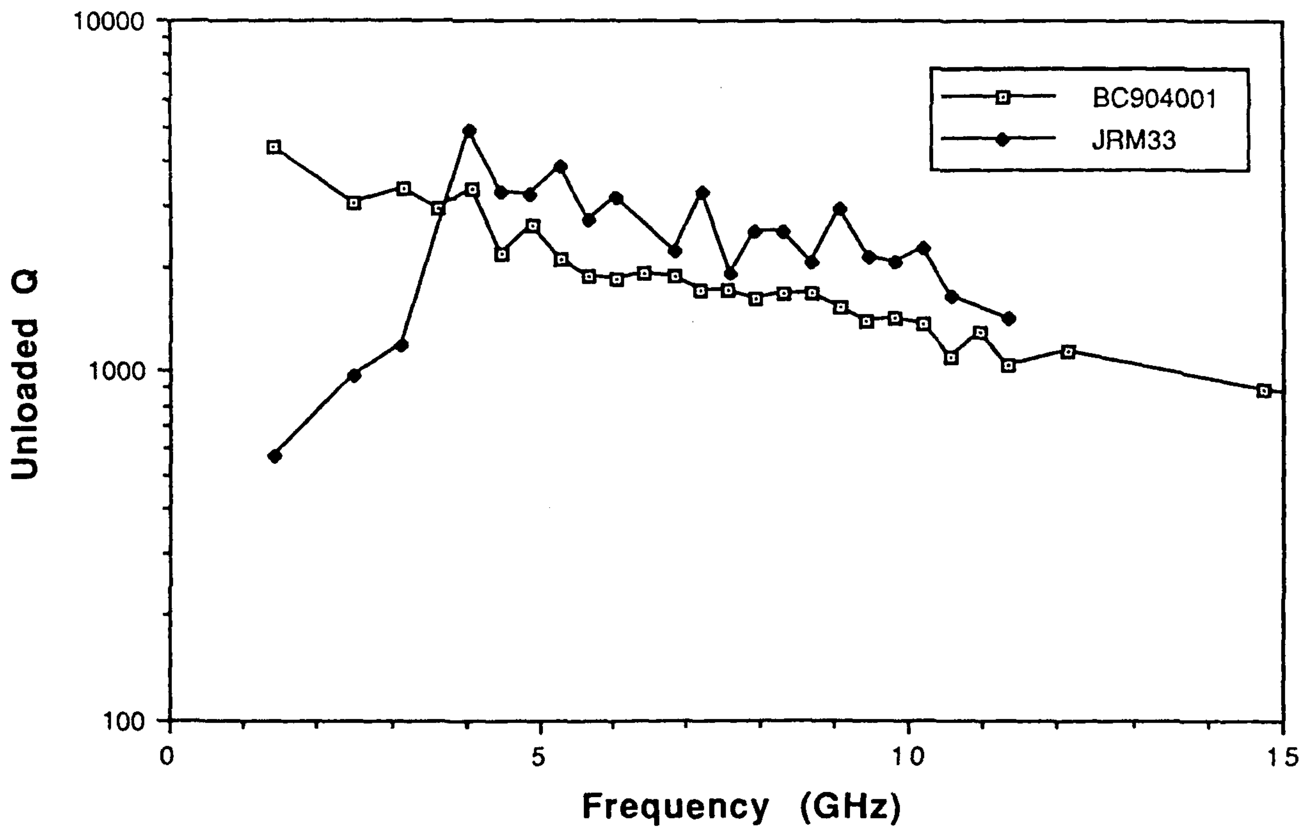

Fig. 12 Measured Q's of two meander lines (both on $LaAlO_3$).

The main conclusions from the study so far are:

- Resonant spirals can be patterned to have very low resonance frequencies using only a minimum of substrate area. (As shown in Fig. 10, a spiral in a 1 cm^2 area can have a resonance frequency as low as 30 MHz).

- The self-resonant spirals are good candidates for measuring Rs below 1 GHz. To obtain accurate surface resistance values will require the calibration with a copper spiral of equal dimensions and thick enough to avoid skin effect losses.

- Provided the low frequency drop-off can be eliminated, Q-values approaching 10^5 should be achievable at 100 MHz and below. This assumes that dielectric losses are not becoming dominant.

Although the present results are very preliminary, they point out the possibility of achieving very high-Q resonators in miniature form at HF through UHF frequencies that could lead to the eventual development of highly compact, high quality filter structures that can not be realized with any other technology known today.

For the next quarter we plan to refined the measurement set-up for spirals, make a calibration run on a copper spiral, explore the drop-off behavior and make some tests on spirals patterned on other substrates.

REFERENCES

[1] A. Fathy, D. Kalokitis, E. Belohoubek, "Microwave characteristics and characterization of high T_c superconductors," invited paper, Microwave Journal, 1988, pp. 75-94.
[2] H. Piel et al.,"Superconducting perovskites in microwave filds," Int. Conf. on High Tc Superconductors and Materials, Interlaken, Switzerland, Feb. 1988.
[3] H. Padamsee et al.,"Microwave superconductivity for particle accelerators: how the high Tc superconductors measure up," Conf. on superconductivity and applications, Inst. of Superconductivity, Buffalo, NY, Apr. 1988.
[4] E. Belohoubek, A. Fathy, D. Kalokitis," Microwave characteristics of bulk high T_c superconductors", 1988 IEEE MTT-S International Microwave Symposium Digest, New York, 1988, pp. 445-448.

[5] A. Fathy et al., "Microwave surface resistivity of bulk YBaCuO material", Physics Review B, October 1, 1988, pp. 7023-7025.
[6] R. C. Taber, "A parallel plate resonator technique for microwave loss measurements on superconductors," Rev. Sci, Instrum. 61(8), pp. 2200-2206, August 1990.
[7] D. E. Oates et al.," Superconducting stripline resonators and high Tc materials, 1989 IEEE MTT Sympo., Long Beach, Cal.,1989, pp. 627-630.
[8] A. Fathy et al " Microwave properties and modeling of High T_c superconducting thin film meander line, IEEE-MTT International Symp. 1990, Dallas, May, 1990, pp.859-862.
[9] E. Belohoubek et al., "Microwave measurements on patterned high temperature Superconducting Thin Film Circuits," Proceedings SPIE Symposium, Santa Clara, Oct. 12, 1989.
[10] D. Kalokitis, A. Fathy et al., " Measurements of microwave surface resistance of superconducting thin films, " IEEE J. of Electronic Materials, Jan. 1990, pp. 117-121.
[11] A. F. Harvey, "Microwave Engineering,"1963 Academic Press, chapter 13, pp. 586-588.
[12] J. S. Martens et al., " Confocal resonators for measuring the surface resistance of high temperature superconducting films," 1991 Apl Phys. Lett. 58, 3 June 1991, pp. 25432545.
[13] Courtney, W.E., "Analysis and evaluation of a method of measuring the complex permittivity and permeability of microwave insulators", IEEE Transactions on Microwave Theory and Techniques, vol. MTT-18, no. 18, August, 1970, pp.476-485.
[14] S. Kaur et al.," Cryogenic characterization of potential high temperature superconductor substrates at microwave frequencies," 1990, Electronic Material Conf., New Jersey.
[15] J. F. Fiedziuszko," Dielectric Resonator used as a probe for high tc superconductor measurements," 1989 IEEE, MTT-Symp., Long Beach Cal., 1989, pp. 555-5558.
[16] Y. Kogami, Y. Kobayashi, et al.,"Low Babdpaa filter using dielectric rod resonators oriented axially in a high Tc superconductor cylinder," 1990 IEEE, MTT-Symps., Boston, Ma, 1990, pp.1345, 1348.
[17].Dielectric Resonators, editors Darko Kajfez and Pierre Guillon, Artech House Inc., 1986, p. 105.
[18] Matula, R.A., "Electrical resistivity of copper, gold, palladium and silver", Journal of Physical and Chemical Reference Data, vol. 8, no. 4, 1979, pp.1147-1298

CHANGE IN KEY PERSONNEL:

Since over a year had passed between writing the original proposal and the actual contract award, research priorities at Bellcore, our originally selected subcontractor for this program, had changed sufficiently to make a reevaluation of the subcontract award necessary. In a series of discussions with representatives from Bellcore, Sarnoff realized that recent changes in the business objectives of Bellcore and associated personnel changes considerably reduced the confidence of success for this program.

In view of the fact that a number of the researchers listed in the original proposal are now part of Neocera, including Prof. T. Venkatesan who is the president of the new company, we believe that the program's success is best assured by delegating the materials development part of the DARPA program to Neocera. This decision is further enhanced by Neocera's other concurrent contracts with NASA, SDIO and Oakridge

National Laboratory which all will contribute to the technological aspects of this program. Neocera is a small business firm, originally established out of collaborative efforts between Bellcore and Rutgers University. The firm recently relocated to the TAP Center at the University of Maryland where Dr. Venkatesan is a full Professor. The Center of Superconductivity at the University of Maryland has an extensive effort aimed at the development of high temperature superconducting active and passive devices, and work ongoing there is expected to provide rich cross fertilization for the DARPA program.

The planned transfer of this subcontract to Neocera was discussed verbally with Dr. F. Patton and Dr. W. Smith and they are both in agreement that the proposed change will enhance the technical output of this program.

SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS: None

PROBLEMS ENCOUNTERED AND/OR ANTICIPATED: None

ACTION REQUIRED BY THE GOVERNMENT:
Approval for transfer of subcontractor

FISCAL STATUS:

1. Amount currently provided on contract:	$200K
2. Expenditures and commitments to date:	$64.9K
3. Funds required to complete work:	$1,585,085