R & D STATUS REPORT

DARPA ORDER NO: 8100 **PROGRAM CODE NO:** DO-C9

CONTRACTOR: David Sarnoff Research Center

CONTRACT NO.: N00014-91-C-0216 **CONTRACT AMOUNT:** $1,585,150

EFFECTIVE DATE OF CONTRACT: 26 August 1991

EXPIRATION DATE OF CONTRACT: 25 August 1994

PRINCIPAL INVESTIGATOR: Dr. Edgar J. Denlinger

TECHNICAL CONTRIBUTORS: Dr. Aly Fathy, David Kalokitis,
Valerie Pendrick,
Barry Thaler,
Dr. Erwin Belohoubek,
Dr. K. S. Harshavardhan (Neocera),
Dr. T. Venkatesan (Neocera)

TELEPHONE NO: (609) 734-2481

SHORT TITLE OF WORK: High Performance YBCO Films

REPORTING PERIOD: 2/25/94 to 5/25/94

1 The views and conclusions contained in this document are those of the authors and should not be interpreted as necessarily representing the official policies, either expressed or implied, of the Defense Advance Research Projects Agency of the U.S. Government.

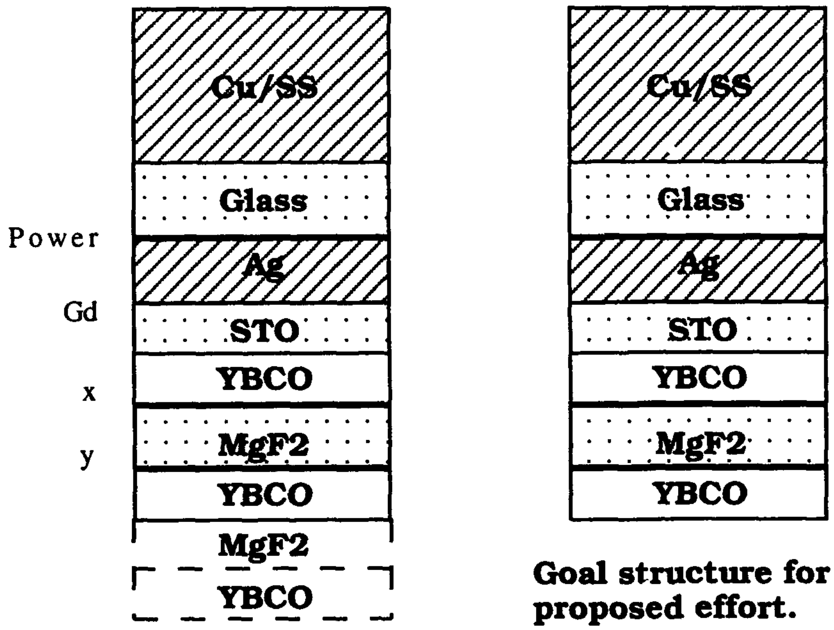
Table of Contents

SUMMARY

- A Bi_2O_3-based glass composition was found suitable for use under typical YBCO deposition conditions, which is in a vacuum under partial oxygen pressure at about 750°C.
- It was experimently determined that a Ag capping layer thickness of about 5 microns is needed for use with this glass. This Ag thickness will prevent any degradation in the silver layer's conducting properties due to Ag/glass interaction during the glass firing operation at 640°C.
- Lanthanum aluminate samples with YBCO topped with a thin layer of strontium titanate (STO) were prepared by Neocera and had excellent YBCO surface resistance properties as measured with a dielectric resonator test system. These samples will be used to test the compatibility of the YBCO with the silver capping layer deposited over the STO.

I. INTRODUCTION

An MCM technology based on a YBCO/bulk magnesium fluoride microstrip structure and attached to a glass/metal handle is being developed. Figure 1 is a cross-sectional view of the various layers in the desired MCM configuration. During this past quarter, emphasis has been placed on the following problems: (1) developing a silver capping layer with the optimum thickness to shield the YBCO layers from the very reactive glass /metal handle; (2) developing a glass composition that will adhere well to the metal handle and also withstand the harsh environment imposed during the 750°C partial oxygen-pressured vacuum deposition of YBCO. The following report shows some very promising results relating to both the capping layer and the glass.



Goal structure for the entire MCM effort.

Goal structure for this proposed effort.

Figure 1. MCM/YBCO Structure Using Ag Cap Layer. *The proposed structure (right) provides low circuit loss at high frequencies for microwave / millimeter wave circuitry and accelerates ARPA's effort toward its overall MCM program goals (left).*

II. PROCESSING DEVELOPMENT OF MCM STRUCTURE

A. Summary of Development Plan

As shown in the last quarterly report the diagram showing the breakdown of tasks for fabricating the MCM structure is given in Figure 2 with Tasks I and II running in parallel. Detailed descriptions of the tasks are given in the

Program Plan for Phase III dated November 30, 1993, that was submitted to ARPA.

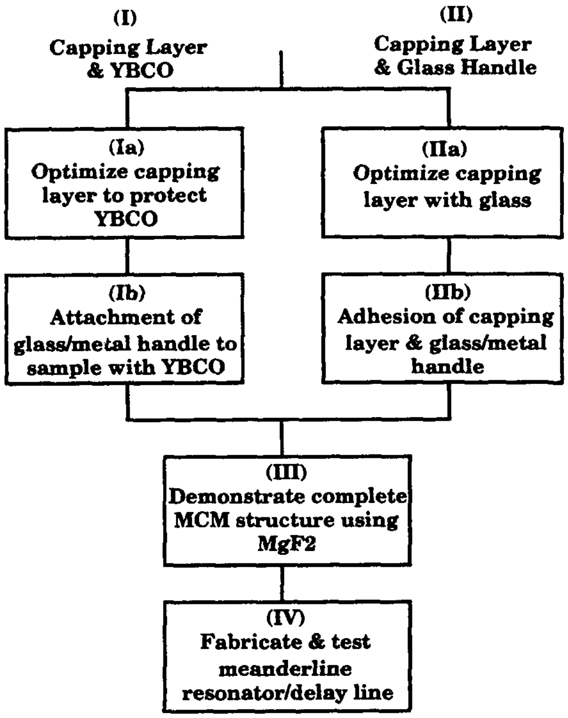


Figure 2. Breakdown of Program Tasks. *Our plan provides parallel paths for Tasks I and II reducing both risk and development time.*

B. MgF_2 Substrate Processing

The crystallographic quality of the MgF_2 substrates directly affects the structural quality of the subsequently grown buffer layers as well as the superconducting films. A figure of merit for surface quality is the minimum yield from ion channeling or "χ-min" using Rutherford Backscattering. This type of characterization requires two measurements. For the first measurement, a beam of energetic ions is aligned with a major symmetry direction and the ion channeling yield is noted. In the second measurement, the beam is not aligned. The ratio of these two numbers is the minimum yield. In addition to the surface finish produced by the polishing of the crystal, the quality of the crystal itself as well as the actual cutting of the crystal may influence the minimum yield. Earlier work at Neocera had shown that a value of χ-min equal to 5% or less was needed to grow high quality YBCO films on MgF_2. Since the planned process of our MCM structure requires the signal YBCO layer to be deposited on the thinned and polished side of the MgF_2 substrate, the crystallographic quality of the surface must be preserved.

Recent Rutherford Backscattering measurements on 1 cm x 1 cm, one-side polished samples (see Figure 3) obtained from Commercial Crystal Labs gave an RBS spectra shown in Figure 4 that corresponded to a minimum yield of 3%. Polished samples, 2.5 cm x 2.5 cm, obtained from a different source and repolished by Sarnoff gave a minimum yield of 26% as shown by the RBS spectra in Figure 5. In view of these excellent results from Commercial Crystal Labs, we will use MgF_2 single crystal substrates obtained from Commercial Crystal Labs for the final circuits. The first YBCO layer will be deposited on the polished side. However, the sample must still be thinned to less than 10μm and polished. Commercial Crystal Labs only offers MgF_2 substrates with one side polished. Sarnoff will concentrate on the thinning and polishing of the back side. Examination of the Commercial Crystal samples has shown that the samples were polished as a larger piece and then cut into small, 1 cm x 1 cm samples. This eliminates any rounding off the edges and allows areas that did not polish well to be discarded. Sarnoff will start with these 1 cm x 1 cm samples which are known to be of high quality and will polish the unpolished side. The surface finish will be checked by ion channeling.

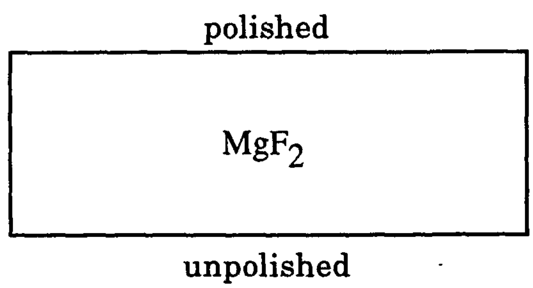


Figure 3.
MgF_2 As Received from Commercial Crystal Labs

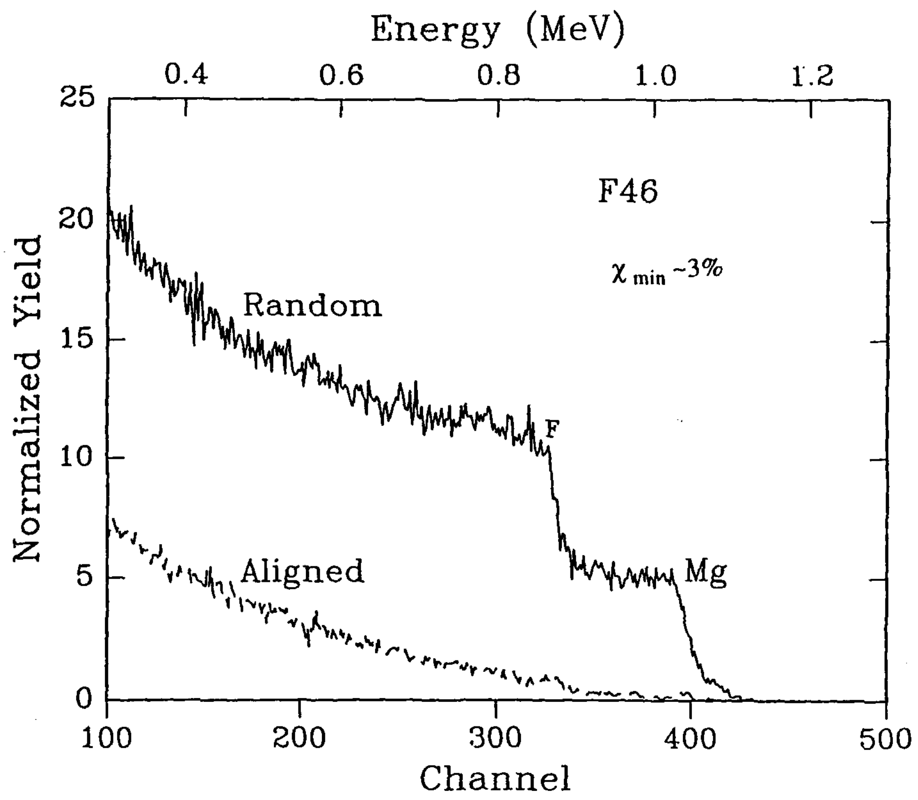


Figure 4. RBS spectra of MgF_2 substrate from a commercial vendor.

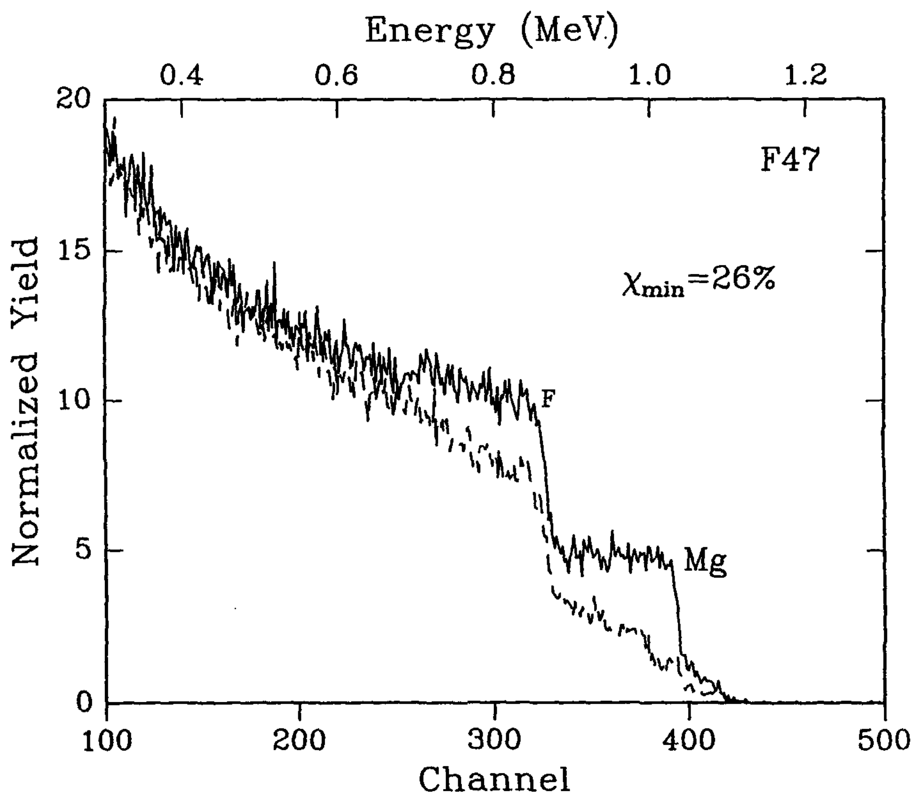


Figure 5. RBS spectra of thinned (10 mils thick) and polished MgF_2 substrate.

C. Glass/Metal Handle Processing

1. Glass Material Considerations

The glass/metal handle relies on a glass to attach the YBCO superconducting circuit to a metal plate. This places a substantial number of requirements on the glass. The attachment must withstand the polishing operations for thinning the MgF_2 crystal as well as the subsequent YBCO deposition at 750°C in a vacuum chamber. The glass/metal handle attachment process is performed in a furnace under ambient atmosphere conditions; therefore, the attachment temperature should be as low as possible so that the YBCO superconducting layer is minimally degraded by this process. Two low temperature glass systems are being evaluated for use in the glass/metal handle; a PbO-based glass and a Bi_2O_3-based glass (hereafter referred to as "50/50 glass"). Additional requirements on this glass include being thermal expansion matched to Cu/Stainless Steel #409/Cu metal base and having good adhesion to the Ni-plating of the metal base.

Both glasses under consideration are thermally matched to the metal base and exhibit adequate adhesion to both the metal base and the Ag capping layer that is applied over the YBCO film. The attachment temperature for PbO-glass is approximately 550°C, while the 50/50 glass requires 640°C for attachment. These glasses are presently being evaluated for their reheat stability in a vacuum chamber to simulate the subsequent YBCO deposition. Preliminary data indicates that the 50/50 glass can withstand a 750°C reheat under vacuum, while the PbO-based glass exhibits excessive flow in the 650 - 675°C range.

2. Optimization of the Ag Capping Layer with the Glass

Work continued on Task II(a) in Figure 2, which involved the determination of the proper thickness for the Ag capping layer. Last quarter we worked with only the PbO glass. Since then we have found that the new 50/50 glass seems to be better suited for the second YBCO deposition. Tables 1 and 2 summarize the dielectric resonator Qo results (measure of the conductive quality of the Ag) for both glasses. The PbO glass samples with 2µm and 5µm of Ag listed in Table 1 were tested last quarter but are shown again for completeness. For the PbO glass, the thickness of the Ag had to be increased above 5µm to prevent the lead glass from completely interacting with the Ag. This increased thickness also increases the stress in the sample. By using the 50/50 glass, 5µm of silver was adequate for preventing any glass/Ag interaction that would degrade the conductivity of the Ag layer.

Table 1.
Qo of Ag Samples Before and After Heating
LEAD GLASS

Ag thickness (μm)	without glass, no heat			with glass, after 550°C			with glass, after 700°C		
	Side	Qo	f (GHz)	Side	Qo	f (GHz)	Side	Qo	f (GHz)
2	front	3500	24.87	front	2742	24.68	front	0	0
2	back	5439	21.77	back	4530	21.76	back	0	0
5	front	3502	24.87	front	3863	24.71	front	1548	24.71
5	back	5446	21.77	back	5802	21.75	back	2316	21.69
8	front	3600	24.86	front	3937	24.74	front		
8	back	5495	21.76	back	6127	21.76	back		
10	front	3600	24.87	front	3898	24.70	front		
10	back	5760	21.75	back	6287	21.75	back		

Table 2.
Qo of 50/50 Glass Samples with Ag.

Sample	Ag thickness (μm)	without glass, no heat			with glass, after 640°C			with glass, after 750°C		
		Side	Qo	f (GHz)	Side	Qo	f (GHz)	Side	Qo	f (GHz)
1	2	front	3500	24.87	front	3601	24.79	front	0	0
1	2	back	5439	21.77	back	6088	21.76	back	0	0
2	5	front	3502	24.87	front	4283	24.79	front	4270	24.79
2	5	back	5446	21.77	back	5808	21.76	back	6100	21.75
3	5	front	-	-	front	4085	24.74	front		
3	5	back	-	-	back	6530	21.77	back		

3. Glass Reactions Under Conditions for YBCO Deposition

Annealing experiments were carried out to establish the structural and chemical stability of the glass/metal handle at temperatures and oxygen partial pressures at which YBCO film depositions will be carried out in our subsequent processing steps. Two types of samples were prepared, one with an Al_2O_3 substrate, the other with a Ni-plated Cu/SS/Cu handle. The Ni-plated Cu/SS/Cu handle will be used for the final circuit configuration. The Al_2O_3 substrate is a useful test structure. Figure 6 shows typical samples. The glass was deposited using one of two processes, screen printed or slurry. The screened printed deposition used a typical thick film ink vehicle system. This will be used for the final circuits. The slurry deposition is a pre-screened condition without any additives or binders. These samples were tested to provide a baseline in case the screen printed glass outgassed at high temperatures under vacuum.

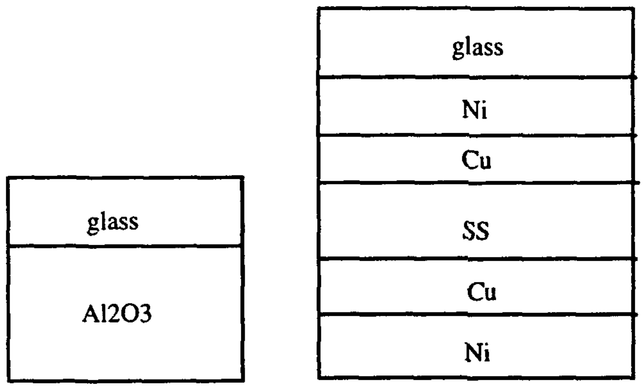


Figure 6.
Glass Test Samples

The annealing process is as follows. The film deposition chamber is evacuated to about 10^{-6} Torr after the glass/substrate sample is mounted on the heating stage. Oxygen is introduced into the chamber to maintain a constant pressure of 250 mTorr. The temperature is then gradually raised to a maximum of 750°C. The sample is maintained at these temperatures and pressures for about 20 minutes (approximate time for a typical YBCO film deposition). The chamber is then filled with 400 Torr of oxygen and the sample is cooled to room temperature. Table 3 lists the various glass samples and the results. Enough information was obtained from the Cu/SS/Cu tests that the Al_2O_3 samples did not require testing. The 50/50 white glass in the screened format performed very well. This is the glass that we will use for the final circuits. Although this glass did blister in its slurry format, this could be due to an insufficient drying of the sample causing solvents to be trapped in the glass powder. The PbO glass flowed which would make it difficult to use this glass in a pattern. The 50/50 white glass in a screened format will be repeated to verify the results.

Table 3.
Glass Samples for Vacuum Test

Sample #	Glass	Format	Temperature	Result
1	50/50 white	slurry	680°C	blistered
2	50/50 white	screened	750°C	OK
3	PbO	slurry		blistered
4	PbO	screened	700°C	glass flowed
5	50/50 pink	screened	650°C	glass peeled

D. Optimization of Capping Layer to Protect YBCO

Three STO/YBCO/$LaAlO_3$ samples were prepared by Neocera in the configuration shown in Figure 7 for further optimization of the silver capping layer. These samples are for Task I(a) of the Phase III proposal. The superconducting properties were first measured by the AC susceptibility method with the resulting data shown in Figures (8-10). Thin film Ag will next be deposited on the samples. The STO acts as an insulating layer between the YBCO and the Ag. All three samples were characterized using the dielectric resonator test set. Q_0 measurements were performed on the front and back of each sample to test the conductive quality of the YBCO. The samples were measured through the back (i.e. through the $LaAlO_3$ substrate) so that after the Ag is deposited on the top of the samples, the YBCO can be retested. The dielectric resonator test results, summarized in Table 4, show excellent Q_0 values and corresponding surface resistances of < 1 mΩ.

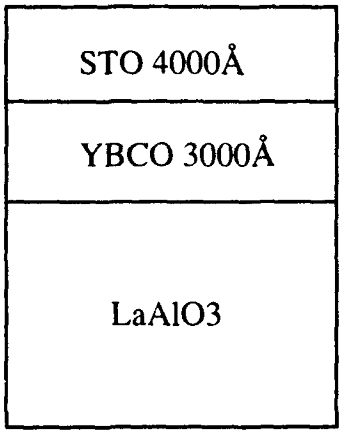


Figure 7.
YBCO samples with
STO Cap Layer.

Table 4.
Qo Data for STO/YBCO/$LaAlO_3$ Samples

Sample #	Qo (front)	f (front)	Qo (back)	f (back)
NGL1	30472	24.876 GHz	33546	22.651 GHz
NGL2	24422	24.875 GHz	28269	22.646 GHz
NGL3	28323	24.850 GHz	33349	22.645 GHz

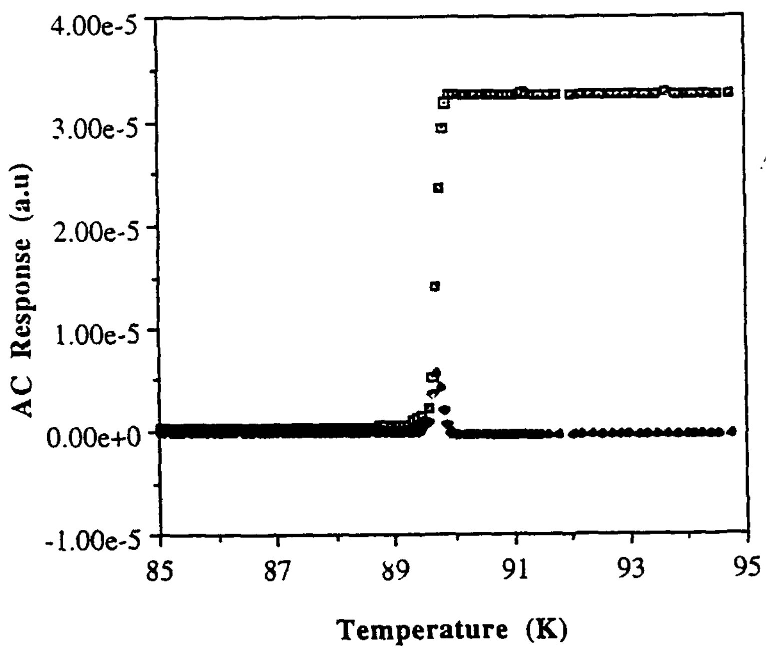


Figure 8. AC susceptibility data of sample NGL1 ($SrTiO_3$-YBCO/$LaAlO_3$).

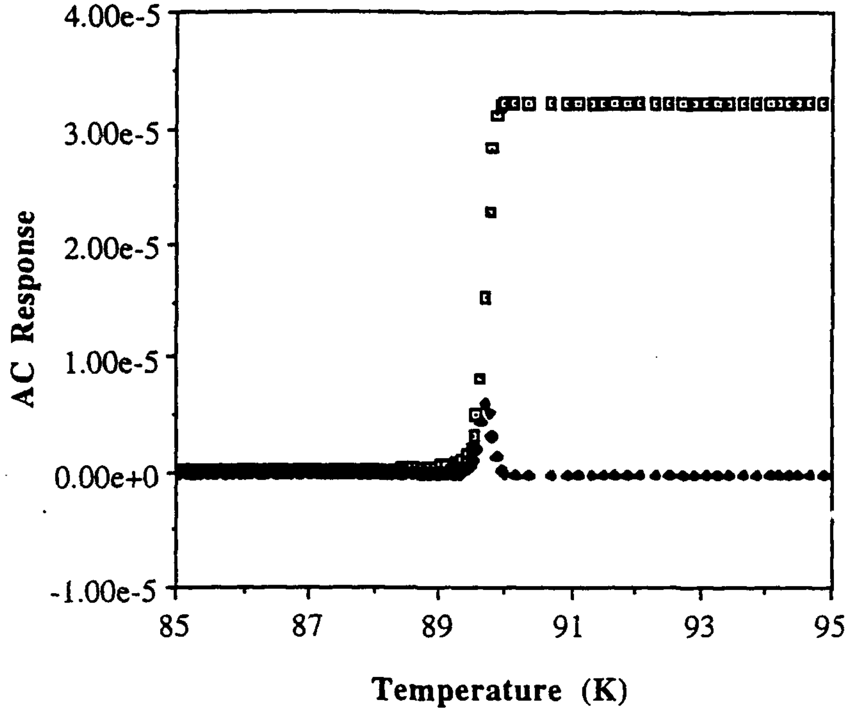


Figure 9. AC susceptibility data of sample NGL2 ($SrTiO_3$-YBCO/$LaAlO_3$).

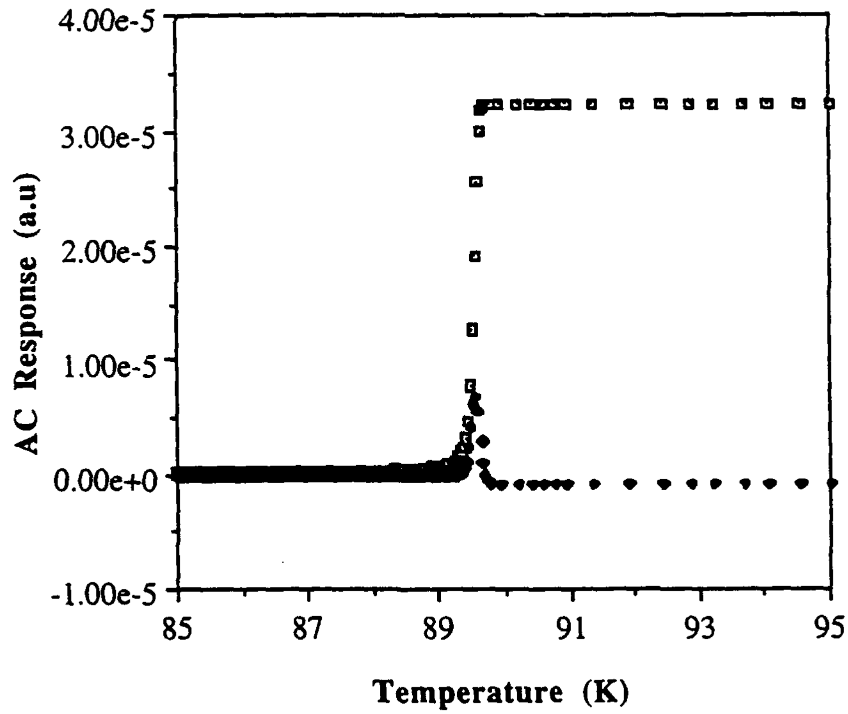


Figure 10. AC susceptibility data of sample NGL4 ($SrTiO_3$-YBCO/$LaAlO_3$).

III. **REFERENCES:** None

IV. **CHANGE IN KEY PERSONNEL:** None

V. **SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS:** None

VI. **ACTION REQUIRED BY THE GOVERNMENT:** None

VII. **FISCAL STATUS:**

1. Amount currently provided on contract:	$1275K
2. Expenditures and commitments to date:	$988K
3. Funds required to complete work:	$1585K