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Dependence of the critical temperature of laser-ablated $YBa_2Cu_3O_{7-\delta}$ thin films on $LaAlO_3$ substrate growth technique

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Abstract. Samples of $LaAlO_3$ made by flame fusion and the Czochralski method were subjected to the same temperature conditions that they have to undergo during the laser ablation deposition of $YBa_2Cu_3O_{7-\delta}$ thin films. After oxygen annealing at 750 °C, the $LaAlO_3$ substrate made by the two methods experienced surface roughening. The degree of roughening on the substrate made by the Czochralski method was three times greater than that on the substrate made by flame fusion. This excessive surface roughening may be the origin of the experimentally observed lowering of the critical temperature of a film deposited by laser ablation on a $LaAlO_3$ substrate made by the Czochralski method with respect to its counterpart deposited on a $LaAlO_3$ substrate made by flame fusion.

1. Introduction

High-temperature superconducting (HTS) thin films have been grown on $LaAlO_3$ (100) substrates because of its lattice match with $YBa_2Cu_3O_{7-\delta}$ and its possible suitability for the fabrication of microwave devices [1–3]. Since the surface quality of the substrate is very important in determining the quality of the HTS film to be deposited, it is necessary to know what kind of changes, if any, can take place on the substrate's surface during HTS film growth. For $LaGaO_3$ substrates, Miyazawa has shown that their surface roughened during the thermal cycling of growing $YBa_2Cu_3O_{7-\delta}$ thin films [4]. Since $LaAlO_3$ is isomorphic with $LaGaO_3$ and undergoes a second-order phase transition at 400 °C, one might expect that its surface would roughen as well. It is well known that for metal–substrate interfaces, the substrate's surface roughness increases the microwave losses [5]. Therefore, it is worthwhile to see if this same effect in the $YBa_2Cu_3O_{7-\delta}$–$LaAlO_3$ interface would cause any degradation of the superconducting properties of the film.

At present there are two different commercial methods of making single crystals of $LaAlO_3$. These are the Czochralski and the flame fusion methods. The method of substrate preparation could possibly determine the extent of surface roughening during thermal cycling of the substrate. Motivated by this possibility, substrates made by the two methods and obtained from different suppliers were subjected to the same thermal cycling conditions that they would have to undergo during the growth of $YBa_2Cu_3O_{7-\delta}$ films by laser ablation. In addition, to determine the effect of the surface roughening on the critical temperature of the superconducting films, films of $YBa_2Cu_3O_{7-\delta}$ were grown on each type of substrate, with identical growth conditions, by laser ablation.

2. Experimental procedures and results

The substrates measured were from two commercial vendors. Each 500 μm thick substrate was polished on both sides. The surface roughness of the substrate made by the Czochralski method (hereinafter called Czochralski substrate) and of the substrate made by the flame fusion method (hereinafter called flame fusion substrate) was compared before and after annealing. The roughness was measured by a profilometer and calculated by taking the root mean square deviation of the distribution of data points from a smooth curve fitted through the profilometer's data. For measurements before the annealing, a piecewise, continuous, second-order function was fitted to the data; for measurements after annealing, a straight line was fitted to the data. The substrates were annealed together at 750 °C for 1 h with a slow cool to room temperature in oxygen.

As can be seen from figure 1, both susbtrates roughened after annealing, although the Czochralski substrate roughened considerably more than the flame fusion substrate. For the flame fusion substrate, the average

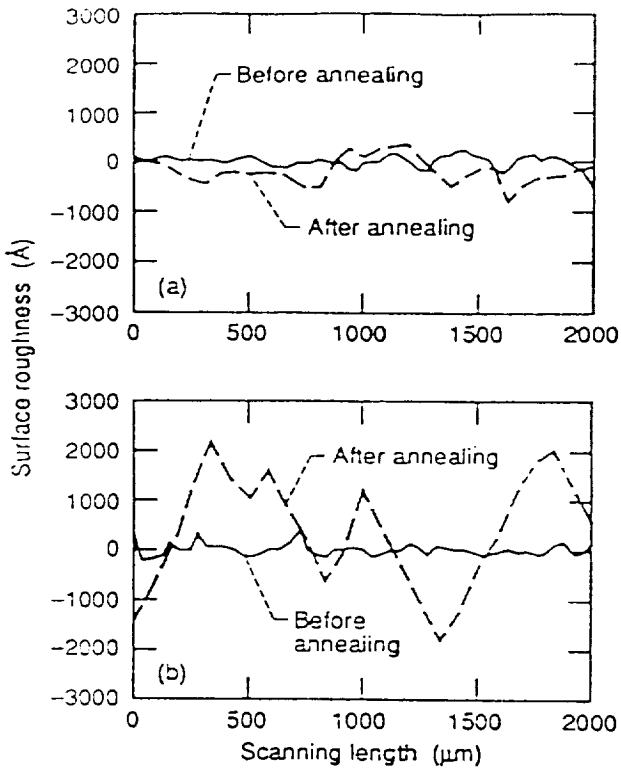

Figure 1. Surface roughness of $LaAlO_3$ as measured with a profilometer before and after annealing for (*a*) flame fusion substrate and (*b*) Czochralski substrate.

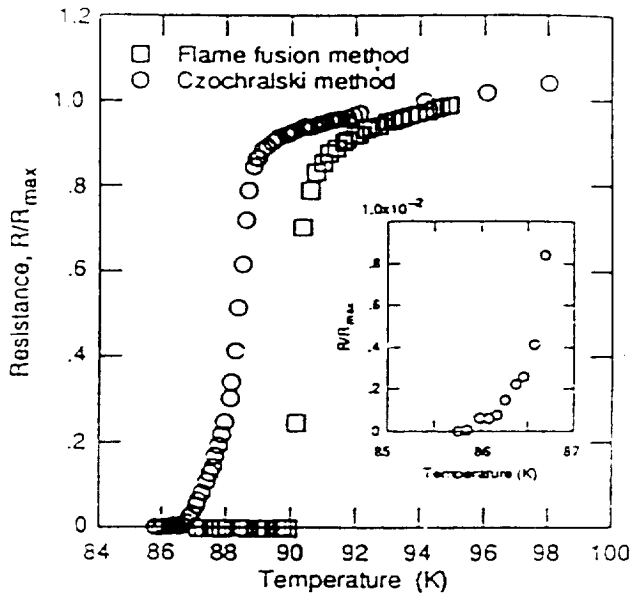

Figure 2. Plots of relative resistance against temperature for $YBa_2Cu_3O_{7-\delta}$ films on (100) $LaAlO_3$ made by flame fusion and by the Czochralski method.

surface roughness before the annealing was ~100 Å, and after annealing it was ~530 Å. On the other hand, for the Czochralski substrate the average surface roughness before the annealing was ~130 Å and after annealing an order of magnitude greater, ~1490 Å.

Thin films of $YBa_2Cu_3O_{7-\delta}$ (~0.6 μm thick) were grown on the two substrates by a laser ablation technique [6–8]. The growth conditions of films on the two substrates were the same (see table 1). The T_c of the film made on a flame fusion substrate was 89.8 K and exhibited a narrow transition region (~0.6 K); while the film made on the Czochralski substrate had a T_c of 85.9 K, a broad transition region (~4 K), and a tail, as can be seen in the inset of figure 2. The difference in the T_c, 4.1 K, of the two films is approximately eight times the run-to-run repeatability normally observed for $YBa_2Cu_3O_{7-\delta}$ films grown on $SrTiO_3$ or cubic zirconia.

Table 1. Deposition and *in situ* annealing parameters of high-temperature $YBa_2Cu_3O_{7-\delta}$ superconducting thin films grown by laser ablation.

Growth temperature (°C)	750
Oxygen pressure (Torr)	0.17
Laser energy density per pulse at target (J cm^{-2})	2
Pulses per second	4
In situ annealing oxygen pressure (atm)	1
In situ annealing time to cool down to 50 °C (h)	5

Since two roughening behaviours were observed in $LaAlO_3$ substrates made by the flame fusion method and the Czochralski method, we believe that the roughening is not due to the intrinsic properties of $LaAlO_3$. The relatively large post-anneal roughening observed in the Czochralski substrate could have been caused by relief of stress created during the growth process or during the sawing or polishing of the susbtrate. Since the stress could be manufacturing dependent, no conclusion about $LaAlO_3$ substrates made by the Czochralski method from other manufacturers can be drawn from this study.

3. Conclusions

We have shown that there is roughening of the surface of $LaAlO_3$ substrates when subjected to an annealing process similar to the one that the substrate undergoes during the deposition of $YBa_2Cu_3O_{7-\delta}$ thin films by laser ablation. The extent of the roughening was much greater in the sample made by the Czochralski method than in the sample made by flame fusion. The roughening of the surface of the flame fusion substrate had no noticeable effect on the T_c of the film deposited on it. However, we believe that the 4 K decrease in T_c for the film deposited on the Czochralski substrate may be a direct consequence of the enormous surface roughness developed by the substrate during the thermal cycling involved in the deposition process. Therefore, a careful testing of $LaAlO_3$ substrates must be performed, before the deposition of HTS films by laser ablation, to determine if their surface roughens as a result of the heating and cooling cycle involved in the deposition method.

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Electromigration failure in $YBa_2Cu_3O_{7-x}$ thin films

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Electromigration failure in highly oriented $YBa_2Cu_3O_{7-x}$ thin films below the superconducting transition temperature is reported here for the first time. The film on $SrTiO_3$ failed at 86 K, 2.3×10^5 A cm^{-2}; while that on $LaAlO_3$ failed at 84 K, 9.3×10^5 A cm^{-2} Scanning electron microscopy and energy dispersive x-ray analysis of the films after failure shows that Cu migrates preferentially away from the failure region towards the electrode.

The phenomenon of electromigration (EM) has been extensively studied in metals and alloys of Al, Au, and Cu because of their usage in semiconductor integrated circuits.[1] The study of EM in oxide materials had not been possible earlier because of their poor electrical conductivity. The recently discovered oxide superconductors, however, exhibit metallic conduction behavior in the normal state and when fabricated into stripes of thin film, were found to carry current densities $>10^6$ A cm^{-2} in the superconducting state.[2] It is also known that holes and not electrons are the major charge carriers,[3] and the oxygen atoms are highly mobile in these compounds.[4] Hence the study of EM in these materials is important both for basic understanding and practical applicability. Recently, EM at 200 and 350 °C in $YBa_2Cu_3O_{7-\delta}$ sintered rods has been reported.[5] In this letter, results obtained from EM below the superconducting transition temperature in $YBa_2Cu_3O_{7-x}$ thin films on $SrTiO_3$ and $LaAlO_3$ substrates are reported.

In the present work, thin films of $YBa_2Cu_3O_{7-x}$ were deposited onto $SrTiO_3$ and $LaAlO_3$ single-crystal substrates by pulsed excimer laser ablation.[6] The films, ≈0.25 μm, were patterned using conventional optical lithography and chemical etching into stripes of size 40 μm wide and 2.0 mm long between the voltage contact pads. Very low resistivity ohmic contacts to the film were obtained by a thorough cleaning of the film surface followed by an evaporation of 0.3 μm Ag onto the pads. The $T_c(R=0)$ for the film on $LaAlO_3$ is 88.6 K as compared to 90 K for the film on $SrTiO_3$. The transition width in both the films however, is <2 K and the critical current density at 77 K defined by 1.0 μV cm^{-1} is $>10^6$ A cm^{-2}.

Following the critical current measurements, the current voltage (I-V) characteristics of the two films were studied. During these studies the samples were biased with currents which resulted in the development of electric fields as high as 50 mV cm^{-1} along the stripe. However the time for which such high electric fields were sustained was very short (<60 s). The films were subjected to repeated temperature and current cycles during these observations. Since the temperature range of cycling was only between 30 and 90 K, thermal stress failure can be ruled out. The films on both the substrates failed after ≈40 h. The film on $SrTiO_3$ substrate failed at ≈86 K. The current density at this temperature was 2.3×10^5 A cm^{-2} corresponding to a field of 50 mV cm^{-1} between the two voltage contacts. The film on $LaAlO_3$ on the other hand failed at ≈84 K; 9.3×10^5 A cm^{-2} and 20 mV cm^{-1}. The surface morphology of the films after they failed was studied both by optical and scanning electron microscopy and is shown in Figs. 1 and 2. In both films, failure occurred both across the main stripe and at the junction between one of the large current pads and the main conductor stripe where there is a large change in the cross-sectional area of the film. The failure pattern at the pad stripe junction is similar to that observed in the mechanical failure of materials—initiation and propagation of a crack. The tendency of the crack-like feature to extend radially outward towards a current pad is reminiscent of the EM failure pattern in metals. Close observation of the scanning electron micrographs reveals a macroscopic material displacement at both the failed regions. A clear material buildup can be seen along the sides of the crack-like feature and also away from it. This is similar to that observed in electromigration failure of metals and alloy thin films—formation of hillocks and voids.[7] In the case of metals and alloys, voids or cracks are observed at the cathode and material buildup in the form of hillocks is found both at the anode and along the length of the conductor stripe. In the present case of $YBa_2Cu_3O_{7-x}$ a clear distinction between voids and hillocks as seen in metals is not observed.

The composition analysis of the film using energy dispersive x-ray analysis technique at different points along the length of the stripe shows several salient features. Far away from the failure region and towards the seemingly unaffected current pad, a contour mapping of the three cations, Y, Ba, and Cu, shows that they are uniformly distributed over the entire area. However, at the regions of failure, a clear segregation of the three elements is observed. A very noticeable fact is that there is a relative depletion of Cu at the failed region on the stripe as compared to that near the current pad.

In the EM studies of $YBa_2Cu_3O_{7-\delta}$ at 200 and 350 °C,[5] it was found that the material decomposes into a number of phases at the anode accompanied by an oxygen enrichment at the cathode. This has been attributed to the nature of charge carriers (holes) and also the ease of oxygen diffusion. It is well known from the crystallographic

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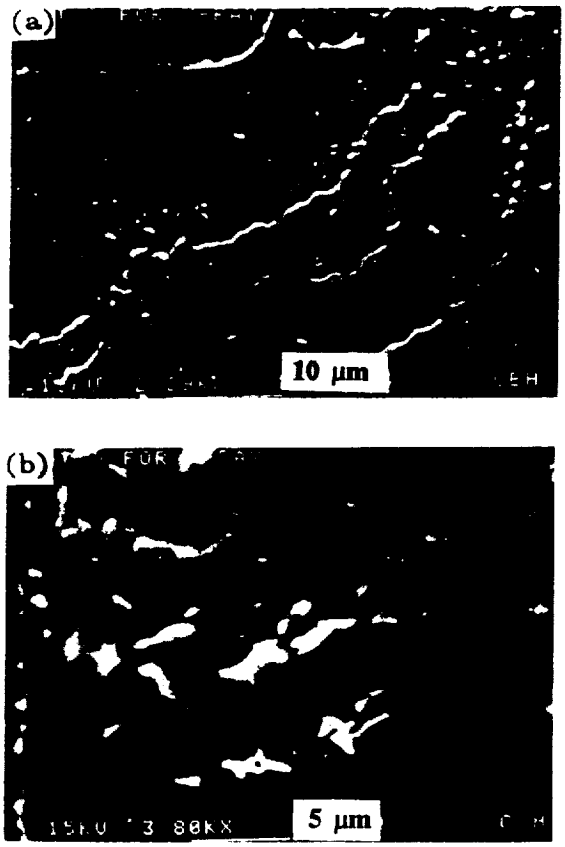

FIG. 1. Scanning electron microscope (SEM) micrographs of electromigration failure in the $YBa_2Cu_3O_{7-x}$ film on (100)$SrTiO_3$ substrate: (a) at the current pad-main conductor stripe junction, (b) across the main conductor stripe.

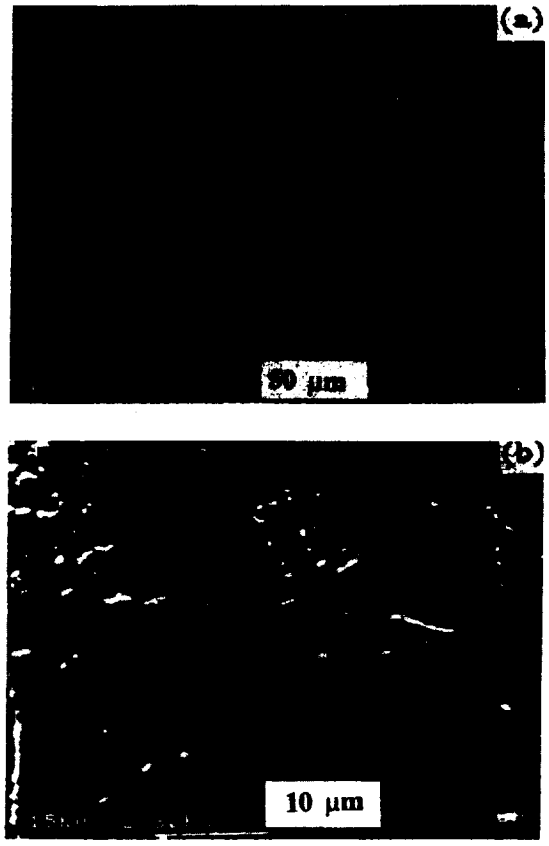

FIG. 2. SEM micrographs of electromigration failure in the $YBa_2Cu_3O_{7-x}$ film on (100)$LaAlO_3$ substrate: (a) at the current pad-main conductor stripe junction, (b) across the main conductor stripe.

studies[8] that the O atoms are primarily in the Cu and Ba planes. Hence migration of the anion (O) towards the cathode should lead to the migration of Cu and/or Ba. The relative depletion of Cu in the middle and an enrichment at the pad observed in the present work clearly indicates that the migrating cation is Cu. The diffusion studies of ^{63}Ni in $YBa_2Cu_3O_{7-\delta}$ thin films in the temperature range 500–650 °C yielded activation energies and diffusion coefficients similar to those of Cu self-diffusion,[9] further indicating that Cu is the migrating cation. The $YBa_2Cu_3O_{7-x}$ compound is known to be unstable below an oxygen concentration of 5.8 and decomposes into various phases.[10] The conductivity of the decomposed phases varies from metallic to insulating values. Decomposition of $YBa_2Cu_3O_{7-x}$ by EM into various low conductivity phases in the presence of the high current densities used in these experiments could lead to a thermal failure known as thermomigration. The rounding of the walls of the crack-like feature is evidence for possible heating.

Electromigration of atoms, in general, is due to diffusion of the atomic species in the presence of an external driving force. In the case of thin polycrystalline films, diffusion will be along the grain boundaries and the surface as the bulk diffusivity will be negligible at low temperatures. The driving force for electromigration in the free electron-gas description of a conductor is typically assumed to have two components: (i) direct force on the charge of the migrating ion due to the applied electric field (E), and (ii) force due to momentum transfer from the charge carriers to the migrating atom "wind force."

The average grain size in both the films is <0.25 μm, which is comparable to the film thickness, and the grains are preferentially aligned with the c axis perpendicular to the film plane. However the in-plane alignment of the grains is known to be random. These factors are known to enhance electromigration. The residual resistivity fraction, which contributes to the wind force at the failure temperature obtained by extrapolating the normal-state resistance, is found to be 0.14 and 0.19, respectively for the films on $SrTiO_3$ and $LaAlO_3$. These relatively high values, as compared to metals and alloys together with the microstructural features, lead to electromigration failure in the high-temperature superconducting materials.

In conclusion, electromigration-related failure in $YBa_2Cu_3O_{7-x}$ thin films has been reported here for the first time. The phenomenon of EM in oxide superconductors in general is complex due to the associated phase changes and decomposition as a function of oxygen con-

centration. The grain boundary diffusivity and its anisotropy, if any, are not currently known. Also, since the normal-state conduction in these materials is completely different, the driving force for EM cannot be estimated using the models applicable to metals and alloys. A detailed understanding of the charge distribution and screening effects is necessary to estimate the driving force. Detailed investigation of these factors is currently in progress.

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ELLIPSOMETRIC STUDY OF $YBa_2Cu_3O_{7-x}$ LASER ABLATED AND CO-EVAPORATED FILMS

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ABSTRACT

High temperature superconducting films of $YBa_2Cu_3O_{7-x}$ (YBCO) were grown on $SrTiO_3$, $LaAlO_3$, and YSZ substrates using two techniques: excimer laser ablation with in-situ annealing and co-evaporation of Y, Cu, and BaF_2 with ex-situ annealing. Film thicknesses were typically 5000 Å, with predominant c-axis alignment perpendicular to the substrate. Critical temperatures up to $T_c(R=0)=90K$ were achieved by both techniques. Ellipsometric measurements were taken in the range 1.6-4.3 eV using a variable angle spectroscopic ellipsometer. The complex dielectric function of the laser ablated films was reproducible from run to run, and was found to be within 10% of that previously reported for (001) oriented single crystals. A dielectric overlayer was observed in these films, with an index of refraction of approximately 1.55 and nearly zero absorption. For the laser ablated films the optical properties were essentially independent of substrate material. The magnitude of the dielectric function obtained for the co-evaporated films was much lower than the value reported for single crystals, and was sample dependent.

INTRODUCTION

Ellipsometry of thin films is a non-destructive technique used to characterize the properties and morphology of thin film materials. It is particularly suitable for thin films since it is non-destructive, highly accurate, and self-normalizing. Recently several investigators have used ellipsometry to study the dielectric function of sintered pellets [1-4] made from superconducting materials such as YBCO, $CaBi_2Sr_2Cu_2O_8$, and $MBa_2Cu_3O_7$, where M is a lanthanide, as well as single crystal YBCO [1,4]. In general, their results show that the oxygen content has a strong effect on the imaginary part (ϵ_2) of the dielectric function $\epsilon(E)$, but the absolute values depend on the sample. For instance, the measured values of the dielectric function of single crystal samples when compared show very different values in the literature [1,4]. Thus, there is no calibration spectrum that can be used for further ellipsometric studies of morphology and/or overlayers. This fact has essentially prevented any meaningful application of ellipsometry to high temperature superconductors in the last two years.

In this study we will present the application of ellipsometry to the study of sample surface, and overlayer nature and growth rate mainly on laser ablated YBCO superconducting films deposited on several substrates. First, we demonstrate the detection of the presence of a thin overlayer on laser ablated films, its refractive index, thickness, and time evolution. Second, we compare, using SEM and ellipsometry, the surface morphology of YBCO thin films made by two techniques: laser ablation with in-situ oxygen anneal and co-evaporation

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with ex-situ anneal. Thus, we show that ellipsometry is a valid technique for the evaluation of surface quality in thin HTS films. It is expected that the above study should lead to extensive application of ellipsometry to the investigation of the surface properties of high temperature superconductors.

EXPERIMENTAL

Samples were prepared by two methods: laser ablation and co-evaporation. The laser ablation technique [5] was based on an excimer laser working at 248 nm, energy density of 1.5 J/cm^2/pulse with 4 pulses per second. The target was a sintered 25 mm diameter YBCO pellet located 8 cm from the sample at 45° to the laser beam. The beam was rastered up and down 1 cm over the target using an external lens on a translator. The substrates used in this study, namely $SrTiO_3$, YSZ (yttria stabilized ZrO_2), and $LaAlO_3$, were mounted on a stainless steel plate with a diameter of 63 mm. The plate was heated from the backside by resistive heating, while a type K thermocouple, welded to the plate, was used for thermometry. After a preheat to 500 °C, oxygen was introduced into the chamber and the samples were heated to temperatures at or near 775 °C. During deposition an oxygen pressure of 170 mTorr was maintained. After deposition the oxygen pressure was raised to 1 atm and the temperature was lowered slowly (at a rate of 2 °C/min) to 450 °C, maintained there for 2 hours, then lowered slowly to 250 °C and finally cooled below 40 °C for removal. The co-evaporation method was essentially the same as the one described in reference [6]. Two electron guns were used to evaporate Y and Cu while thermal evaporation was used for BaF_2. The ex-situ anneal step started with wet oxygen for 1 hour at 875 °C, followed by a dry oxygen anneal, 1 hour step down to 550 °C, 1 hour at 550 °C, and finally oven cool down. The critical temperature, T_C, was measured resistively using a four point technique. In some cases the actual samples were tested, while other times samples made in the same run were measured. The ellipsometric technique was described elsewhere [7] and will not be repeated here. Analysis of the results was done mainly using a substrate and ambient model, while in some cases a substrate, film, and ambient model was applied. Rough surfaces were modeled by a combination of voids and substrate material using the effective medium approximation (EMA) [8].

RESULTS

Results reported here were obtained on samples prepared in separate runs on both the laser ablation and co-evaporation techniques. All samples show c-axis alignment, with only (001) peaks present in the X-ray diffraction pattern. Altogether 12 laser ablated and 5 co-evaporated films were measured. Data will be reported only for samples with a $T_C(R=0)$ higher than 82 K. This is essentially all of the films since a $T_C(R=0)$ higher than 85 K was characteristic of most of the films, especially in the case of the laser ablated films. The thickness of the films was typically 5000 Å, thus ensuring that it was much larger than the optical penetration depth. Several measurements at different angles of incidence, in the range 65°-75°, were performed for all samples. The measured ellipsometric parameters $\tan(\psi)$ and $\cos(\Delta)$ were used to calculate the dielectric function, ϵ, directly.The results gave essentially the same value of the dielectric function, ϵ, independent of the angle of incidence for the samples shown here. Results for ϵ for four representative samples made by laser ablation are compared with that of single crystal YBCO [4] in Fig. 1.

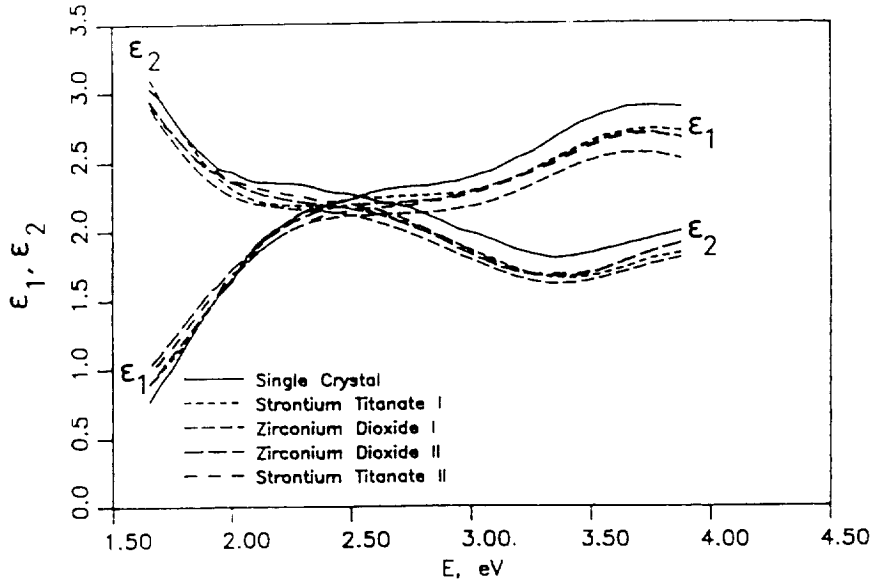

FIGURE 1. - DIELECTRIC FUNCTION OF FOUR LASER ABLATED FILMS AND A SINGLE CRYSTAL YBCO [4].

All laser ablated samples, irrespective of the substrate material, gave dielectric functions with very similar shapes. However, some of them gave absolute values lower than the majority. The lowest $\epsilon(E)$ values obtained for any laser ablated sample are shown in Fig. 2, labeled "before Q tip cleaning".

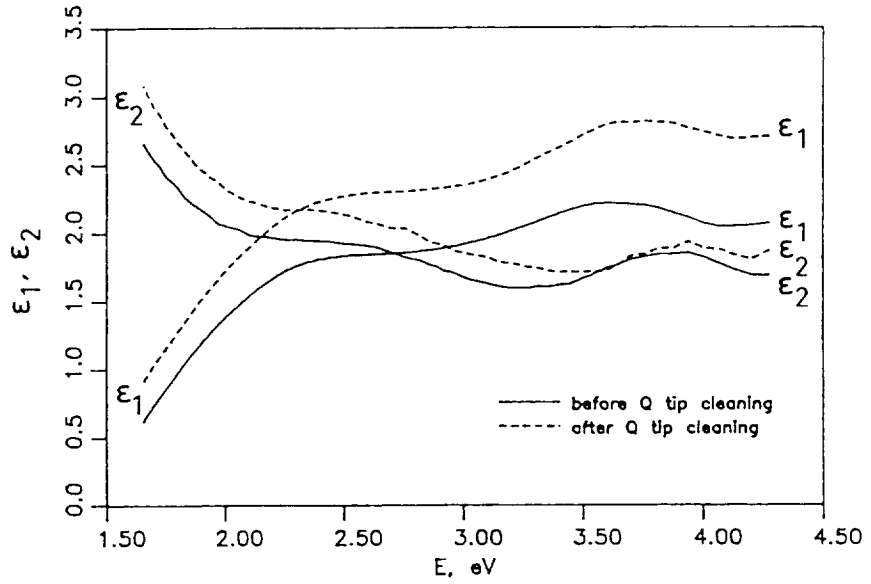

FIGURE 2. - DIELECTRIC FUNCTION OF A LASER ABLATED FILM BEFORE AND AFTER MECHANICAL CLEANING.

In the course of making electrical contacts to the laser ablated films, we found that the quality of the contacts is poor. A solution to this problem was mechanical cleaning of the surface. This fact is related to the result previously reported for YBCO, namely the interaction with air to produce $BaCO_3$ and $Ba(OH)_2$ [9-11]. The $Ba(OH)_2$ is formed only as an intermediate step to the $BaCO_3$ formation, with grain boundaries covered primarily by $BaCO_3$ [9-11]. Mechanical cleaning with a Q tip removed a small amount of white film from the samples with low values of the dielectric function. Fig. 2 displays ϵ before and after mechanical cleaning. Cleaning was done with the sample in the ellipsometer, with dry nitrogen flowing for the measurement. The difference between these two spectra was analyzed as follows: $\epsilon(E)$ after mechanical cleaning was assumed as the calibration $\epsilon(E)$, while the result for $\epsilon(E)$ before cleaning was analyzed as substrate-dielectric film-ambient. The refractive index of the 72 Å film was found to be 1.55. Moreover, it is not a function of energy, i.e., it does not have dispersion in the visible. This fact shows that the overlayer is an insulator. This value of the refractive index and the color are in good agreement with published data [12] for $BaCO_3$, not $Ba(OH)_2$. The $BaCO_3$ film growth rate in air, at temperatures around 20 °C, was studied by measuring the film thickness versus time. Measurements started immediately after the mechanical cleaning and lasted 480 hours. The final thickness was 35 Å, showing a rather slow growth rate, of the order of 1.5 Å/day, for this sample. We found that the quality of electrical contacts made by silver evaporation and annealing in oxygen depends on the thickness of the dielectric layer. In the worst cases, the silver contact would peel off the sample when a small force was applied. Results of $\epsilon(E)$ functions of cleaned samples show much less variation in absolute value versus the original measurements.

Dielectric functions of two co-evaporated samples are shown in Fig. 3. $\epsilon(E)$ in this case is sample dependent and bear small similarity to the single crystal result. SEM micrographs of the films show the presence of voids on otherwise dense films. In addition, the films show more roughness on the surface as compared to the laser ablated films which have <0.25 μm surface roughness as seen by SEM.

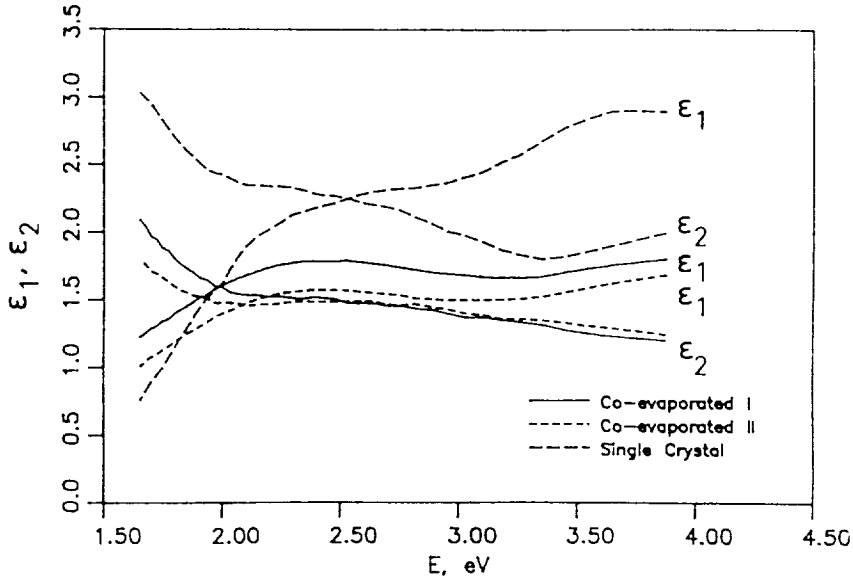

FIGURE 3. - DIELECTRIC FUNCTION OF TWO COEVAPORATED FILMS AND A SINGLE CRYSTAL YBCO [4].

We tried to describe the morphology by the EMA method [8], i.e. using a combination of voids and YBCO. The free parameter used was the void volume fraction, f. A test of the quality of this model is given in Fig. 4, where the best value of f (25.8%) was used to generate ψ and Δ, and compared with the experimental results. The quality of the fit is only moderate, showing that this model is marginally applicable. We obtained large void fractions typically around 20-25%.

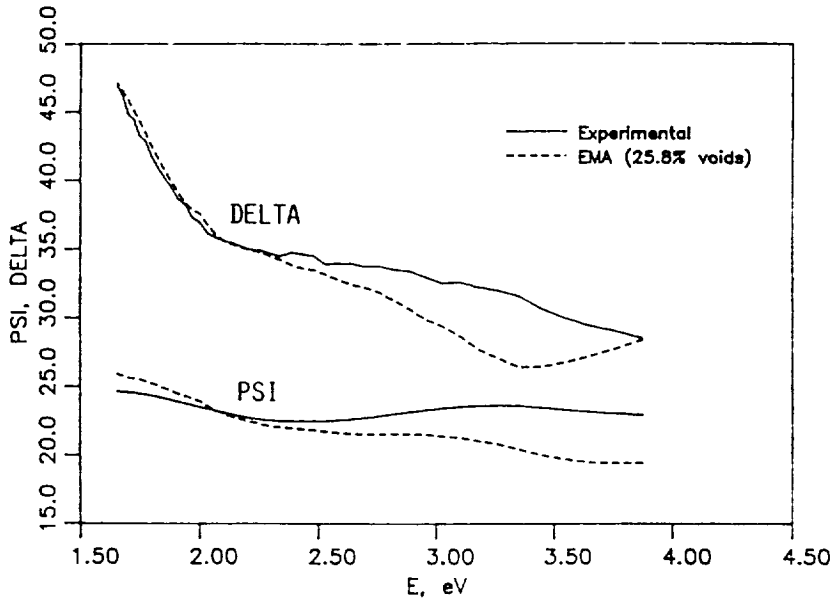

FIGURE 4. - CALCULATED AND EXPERIMENTAL PSI AND DELTA VERSUS ENERGY FOR A COEVAPORATED YBCO FILM.

DISCUSSION

Evaluation of a dielectric function for ellipsometric characterization purposes requires an absolutely perfect surface. As YBCO reacts with air, and the surface morphology of this new type of material is not completely under control, the $\epsilon(E)$ determination is not a simple matter. Our results show a scatter just below 10% among themselves and versus the single crystal result. The best analysis of the overlayers, which are below 10 Å in many cases, is reached when the $\epsilon(E)$ of the cleaned sample is used as the substrate $\epsilon(E)$, i.e. not by using a universal $\epsilon(E)$ function. In this way surface irregularities do not interfere with the overlayer analysis. The fact that our results are so similar to the single crystal result of reference [4] shows that the laser ablation technique gives smooth crystalline films and that the $\epsilon(E)$ given in [4] is for a good quality single crystal. The overlayer, probably $BaCO_3$, was shown to grow very slowly. However, we believe that the growth rate depends on the stoichiometry of the material on the top surface layer. Thus, the experimental growth rate measured here is only for one particular case, obtained for a high quality crystalline film.

The EMA analysis of the co-evaporated films was not completely successful. The reasons for this, in our opinion, are mainly the complex nature of the surface roughness, and, to a lesser extent, the imperfect stoichiometry on the surface of these films. We expect the void fraction to be a function of depth, with lower values of f as we move into the film. These facts, along with the

anisotropy of the material, result in a dielectric function that is probably different from our calibration function.

CONCLUSIONS

We obtained reproducible results for the dielectric function on fully c-axis aligned samples. In absolute value, this result is very similar to the function obtained for (001), platelet-like, free-grown, single crystals [4]. The differences are smaller than 10% at any value of the spectrum. We used our result as the calibration spectrum for subsequent studies, although the single crystal result of reference [4] is probably just as reliable. An overlayer of $BaCO_3$ was measured on top of these films and its growth rate was determined. Results of the overlayer properties analysis compare favorably with the literature. The comparison of the ellipsometry and SEM studies shows that smoothness and density of the film surface are the crucial parameters in the ellipsometric results on YBCO films. At present, ellipsometry has been shown to be useful to the study of laser ablated YBCO films. More work is needed to assess the application of this technique to the study of co-evaporated films.

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Millimeter-wave surface resistance of laser-ablated $YBa_2Cu_3O_{7-\delta}$ superconducting films

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We have measured the millimeter-wave surface resistance of $YBa_2Cu_3O_{7-\delta}$ superconducting films in a gold-plated copper host cavity at 58.6 GHz between 25 and 300 K. High quality laser-ablated films of 1.2 μm thickness were deposited on $SrTiO_3$ and $LaGaO_3$ substrates. Their transition temperatures (T_c's) were 90.0 and 88.9 K, with a surface resistance at 70 K of 82 and 116 mΩ, respectively. These values are better than the values for the gold-plated cavity at the same temperature and frequency.

The characterization of the new high transition temperature (T_c) superconductors at millimeter-wave frequencies is necessary in order to determine their suitability for practical microwave applications. A parameter of these new materials which directly provides this information is the surface resistance (R_s). The R_s of high quality $YBa_2Cu_3O_{7-\delta}$ superconducting films deposited on various microwave substrates has been measured at different frequencies and temperatures by several researchers.[1-4] To the best of our knowledge, however, no measurements have been reported at frequencies around 60 GHz. In this letter we report on the first measurements of the millimeter-wave surface resistance at 58.6 GHz of two predominantly c-axis oriented polycrystalline $YBa_2Cu_3O_{7-\delta}$ films deposited on $SrTiO_3$ and $LaGaO_3$ by pulsed laser ablation.

The pulsed laser ablation technique is similar to that used by other researchers.[5,6] The deposition was performed at a substrate temperature of 750 °C and an ambient oxygen pressure of 170 mTorr. The laser wavelength was 248 nm, the pulse length was 20–30 ns, and the pulse rate was 4 pulses per second. The distance between the target and the sample was kept at 7.5 cm, and the laser fluence on the target was maintained at 2.0 J/cm^2 per pulse. During the deposition process, the laser beam was scanned up and down 1 cm over the target using an external lens on a translator. At the end of the deposition process, the oxygen pressure was raised to 1 atm, and the temperature was lowered to 450 °C at a rate of 2 °C/min. The temperature was held at 450 °C for 2 h before it was lowered to 250 °C, also at a rate of 2 °C/min. The heater power was turned off, and the sample was allowed to cool down to 40 °C or less before it was removed from the chamber. A more detailed explanation of the deposition technique can be found in Ref. 7.

The films were analyzed by x-ray diffraction, dc resistance versus temperature measurements, and scanning electron microscopy (SEM). The dc resistance was measured using a standard four-probe technique. Zero dc resistance was attained at 90.0 and 88.9 K for the films on $SrTiO_3$ and $LaGaO_3$, respectively (see Fig. 1). A typical critical current density for films on $SrTiO_3$ at 77 K was 2×10^6 A/cm^2 when using the 1 μV/cm measurement criteria. The resistivity at 300 K for the films on $SrTiO_3$ and $LaGaO_3$ was 130 ± 30 and 220 ± 50 $\mu\Omega$ cm, respectively. The uncertainties arise from the irregular geometry of the samples and uncertainties in the film thicknesses. The x-ray diffraction pattern revealed that both films are predominantly c-axis oriented. Figure 2 shows SEM micrographs for the two films under study, which indicate that both films are polycrystalline.

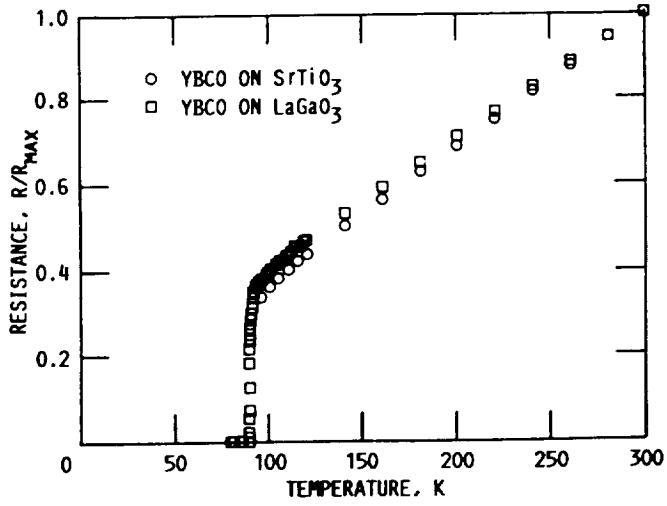

FIG. 1. dc resistance vs temperature measurement of laser-ablated $YBa_2Cu_3O_{7-\delta}$ superconducting films on $SrTiO_3$ and $LaGaO_3$ substrates.

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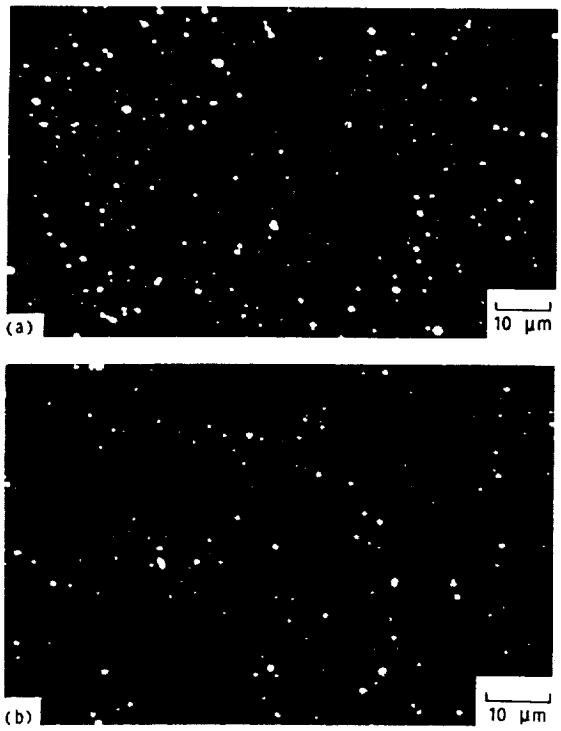

FIG. 2. Scanning electron micrographs of laser-ablated $YBa_2Cu_3O_{7-\delta}$ superconducting films on $SrTiO_3$ (a) and $LaGaO_3$ (b) substrates.

The surface resistance R_s is measured by looking at the change in the Q factor of a cylindrical, TE_{013} mode, gold-plated copper cavity operational at 58.6 GHz when one of its end walls is replaced by the superconducting film sample. Using an HP-8510 network analyzer and the Ginzton's impedance method,[8,9] the Q factor for the cavity is determined from the reflection coefficient. The Q factor is defined as

$$Q=\omega_0(\text{maximum energy stored})/\text{power loss}$$

$$=\frac{1}{2}\omega_0\epsilon\int_V |E|^2\,dV\Big/\frac{1}{2}R_s\int_S |H_t|^2\,dS, \qquad (1)$$

where R_s is the surface resistance of the cavity walls, S is the inner surface of the cavity, V is the volume of the cavity, and H_t is the tangential field. Integrating Eq. (1) we obtain that the Q for the bare cavity is given by

$$Q_1=\frac{(2\pi laf)^3\epsilon\mu_0^2[-J_0(q_{01})J_2(q_{01})]}{R_s[2l^3q_{01}^2J_0^2(q_{01})-4\pi^2a^3n^2J_0(q_{01})J_2(q_{01})]}, \qquad (2)$$

where l is the length of the cavity, a is the radius of the cavity, f is the frequency, J_0 and J_1 are normal Bessel functions, $q_{01}=3.8317$ is the first zero of J_1, and n is the longitudinal mode number. When one of the cavity's end walls is replaced with the sample, the Q factor becomes

$$Q_2=\frac{(2\pi laf)^3\epsilon\mu_0^2[-J_0(q_{01})J_2(q_{01})]}{R_{s1}[2l^3q_{01}^2J_0^2(q_{01})]-2(\pi n)^2a^3J_0(q_{01})J_2(q_{01})(R_{s1}+R_{s2})}, \qquad (3)$$

where R_{s2} is the surface resistance of the superconducting sample and R_{s1} is the surface resistance of the other cavity walls. Hence, the surface resistance R_{s1} of the metal is obtained from Eq. (2) by measuring the Q factor of the bare cavity (Q_1), and then R_{s2} is computed from Eq. (3) by measuring the Q factor with the sample in place (Q_2).

In Fig. 3 the measured $R_s(T)$ curves for the two films under consideration are shown. Also plotted is the experimental surface resistance of the gold-plated reference cavity for comparison. The R_s for the two films is comparable in the normal state, while the R_s of the film on $SrTiO_3$ decreases faster than that for the film on $LaGaO_3$ at temperatures just below T_c. Using the normal skin depth formula $R_s=(\omega\mu_0\rho/2)^{1/2}$, we obtained values for the resistivity, ρ, and skin depth, δ, at 300 K of approximately 118 $\mu\Omega$ cm and 2.3 μm for the film on $SrTiO_3$ and 158 $\mu\Omega$ cm and 2.7 μm for the film on $LaGaO_3$. Note that the strong "oscillatory" behavior of R_s as a function of temperature in the normal state, observed for films on $SrTiO_3$ by other researchers,[3] was not observed in our case. This is not unexpected due to the greater film thicknesses employed here.

In the superconducting state, the films on $SrTiO_3$ and $LaGaO_3$ exhibit a drop of R_s to effective values of 103 ± 15 and 144 ± 20 mΩ at 77 K, and 82 ± 15 and 116 ± 20 mΩ at 70 K, respectively. The surface resistance at 77 K for the film on $SrTiO_3$ is less than that of the gold-plated cavity, while for the film on $LaGaO_3$, R_s is the same as for the

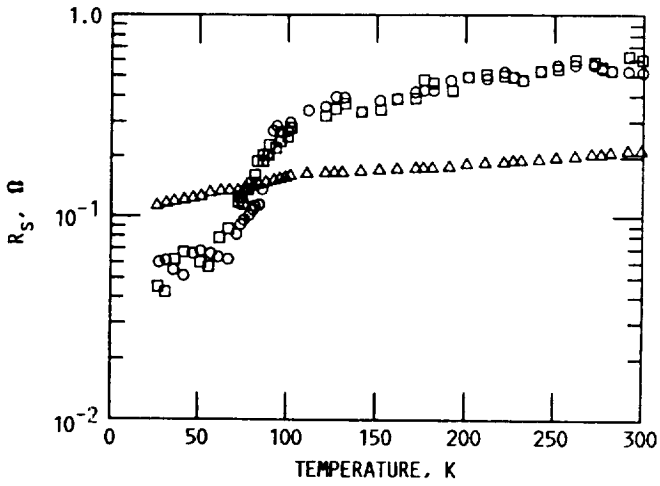

FIG. 3. Surface resistance (R_s) at 58.6 GHz vs temperature for 1.2 μm films of $YBa_2Cu_3O_{7-\delta}$ deposited by laser ablation onto $SrTiO_3$ (○) and $LaGaO_3$ (□) substrates, and for the gold-plated cavity (△).

gold-plated cavity. Since we are operating at a fixed frequency, we cannot study the frequency dependence of R_s directly from our measurements. Nevertheless, a comparison with R_s values reported by other researchers in similar types of films and at different frequencies, may be helpful to formulate a frequency dependence trend for R_s. Thus, using the results obtained by Klein *et al.*[10] for *c*-axis textured layered samples of $YBa_2Cu_3O_{7-\delta}$, and fitting their data to a quadratic frequency dependence for R_s give an R_s value of 102 mΩ at 58.6 GHz and 77 K. This value agrees very well with our experimentally obtained value of 103 mΩ at the same temperature. An analog approach was used for our film on $LaGaO_3$. Using the data reported by Cooke *et al.*[4] for a $YBa_2Cu_3O_{7-\delta}$ superconducting film deposited by magnetron sputtering on $LaGaO_3$, an interpolation of R_s between their reported values measured at 22, 86, and 148 GHz and at 70 K was performed. The resulting R_s value of 106 mΩ at 58.6 GHz compares favorably, within experimental error, with our value of 116 mΩ at the same frequency and temperature. This result indicates that our value fits well with the nearly quadratic dependence for R_s ($R_s \propto \omega^n$, $n = 2.06 \pm 0.14$), as reported in Ref. 4. The results obtained from this correlation are very consistent with the experimental behavior observed in low T_c superconductors and also with the predictions of the Bardeen–Cooper–Schrieffer (BCS) theory.

In conclusion, the surface resistance of preferentially *c*-axis oriented polycrystalline $YBa_2Cu_3O_{7-\delta}$ superconducting laser-ablated films has been measured at 58.6 GHz via a TE_{013} cavity wall replacement technique. The values of R_s obtained for the films on $SrTiO_3$ and $LaGaO_3$ at 77 K are less or equal to that of the gold-plated cavity. A comparison of the R_s values obtained for both films with values for similar films measured by other researchers at different frequencies shows that our values are consistent with the frequency dependence for R_s observed in classical superconductors and with the predictions of the BCS theory.

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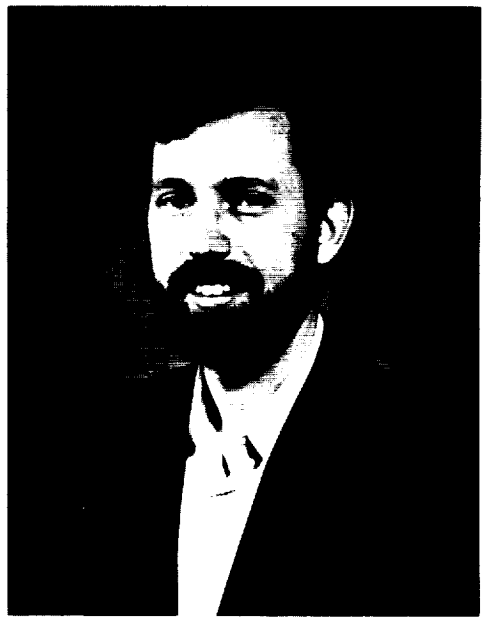
BIOGRAPHIES

Samuel A. Alterovitz earned his Ph.D. in Solid State Physics in 1971 from Tel Aviv University, Israel. After a two-year postdoctoral appointment at the University of Illinois, he joined the staff of the Physics Department at Tel Aviv University where he achieved the status of tenured associate professor. His work there focused on properties of superconducting materials, emphasizing critical currents and critical fields. In 1981, he accepted an appointment as senior engineering research scientist in the Electrical Engineering Department of the University of Nebraska, where he worked to develop the ellipsometric technique. In 1983, he transferred to NASA Lewis Research Center where he is now a senior research scientist in the Solid State Technology Branch. He played a key role in developing materials (e.g. InGaAs) for a new generation of electronic devices for high-speed, low-noise, high-efficiency space applications. Dr. Alterovitz also developed ellipsometry for novel and multilayer structures, specializing in insulators, superconductors, and semiconductor materials. Dr. Alterovitz is now working on materials for cryogenic electronics applications, including semiconductors, superconductors, and their combination. He is also working on furthering applications of the ellipsometric technique. Dr. Alterovitz has authored 103 papers in referred journals, 101 meeting presentations, and has edited two books. He is an active postdoctoral advisor for the National Research Council.

Christopher M. Chorey earned a Master's degree in Materials Science in 1987, and a Bachelor's degree in Electrical Engineering in 1984 from Case Western Reserve University. With the support of NASA Lewis Research Center, he preformed two years of additional graduate work at Case Western Reserve University in 1987 and 1988. His work focused on fabrication and testing of AlGaAs based, high-frequency, electro-optic modulators.

Alan M. Downey earned his B.E.E. from Cleveland State University in 1979, and his M.S.E.E. from the University of Toledo in 1983. He joined NASA Lewis Research Center in 1977 as a co-op student. In 1979 he joined the Space Communications Division. From 1979 to 1985, he was engaged in microwave measurements and solid state technology research, followed by a three-year hiatus in the Communications Projects Branch as Experiments Manager for the Applications Technology Satellites Program. Mr. Downey returned to the Solid State Technology Branch in July 1989. His current research interests include the measurement of novel HEMT structures at cryogenic temperatures, coplanar waveguide circuit design, and MMIC applications.



Edward J. Haugland earned his B.S. degree in Physics from the University of Minnesota, and M.S. and Ph.D. degrees in Solid State Physics from Case Western Reserve University. He joined NASA Lewis Research Center in 1980 as a member of the Solid State Technology branch. Since that time, he has been involved with experimental research on electrical properties of III-V semiconductor materials, heterostructures, and SiC. He was responsible for contracts concerning development of high-power IMPATT diodes and MMIC power amplifiers. Dr. Haugland is a member of the American Physical Society.

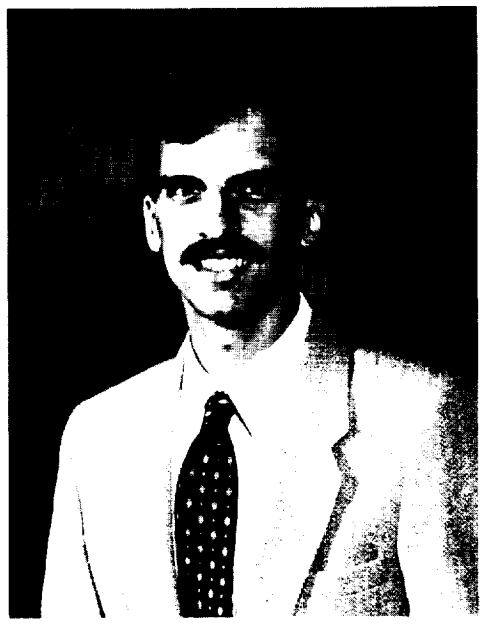
Thomas J. Kascak earned a B.S.E.E. degree from Case Western Reserve University in 1959 and joined Union Carbide Corporation where he worked with performance and reliability aspects of dry cell and alkaline batteries. Mr. Kascak received an M.S. degree in Physics in 1965 from John Carroll University, Cleveland, OH. From 1966 to the present, he has been employed at NASA Lewis Research Center. He has been involved in several work areas from research on thermonic heat to electric power conversion devices. In 1973, he transferred to the Space Electronics Division where he has been involved in various aspects of microwave and millimeter wave solid state devices and circuits. He was responsible for formulating and managing several NASA sponsored contractual efforts involving MMIC development programs. One of these efforts, a 20-GHz MMIC Transmit Module, resulted in the R&D Magazine IR-100 award. For the last few years, he has been responsible for the set-up of the division's in-house solid state facilities. Mr. Kascak has authored papers on thermionic direct energy conversion devices, 20- and 30-GHz MMIC devices, and MMIC-based phased array antennas. Mr. Kascak is a member of the American Vacuum Society.

Regis F. Leonard earned his Ph.D. in Physics from the Carnegie Institute of Technology in 1963 and came to NASA Lewis Research Center that same year. Since that time, he has completed 10 years of basic research concerning the physics of nuclear structures. For six years, Dr. Leonard worked towards the development of a unique Lewis facility that uses neutron radiation to treat cancer patients. For the last 11 years, he has helped develop technology in support of NASA's communications programs. This assignment includes work on the ACTS proof-of-concept technoloy program and, as head of the RF Systems Section, the development of an in-house communications system test capability for the ACTS POC hardware. For the last six years he has served as Chief of the Solid State Technology Branch. Dr. Leonard is responsible for NASA's MMIC technology development program; an active, in-house, solid state, research program; and a sizeable university grant program which supports basic research as applicable to solid state electronics.

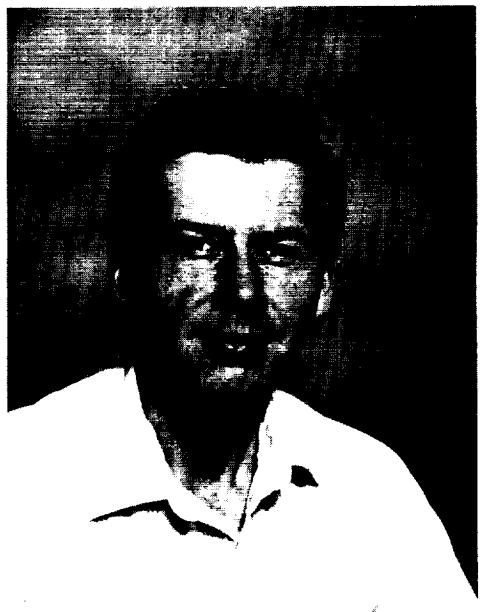
Rafael A. Mena earned his B.S. degree in Electrical Engineering in 1988 from the University of Texas at El Paso and an M.S. degree in Solid State Physics in 1990 from Arizona State University. While at Arizona State, he made theoretical calculations on the effect of a magnetic field on the optical properties of semiconductor alloys. While pursuing his degree, he participated in several co-op programs. In the summer of 1987, while with the Arizona Public Service, he conducted an economic study on the installation of underground power lines. The following summer, he was accepted into the NASA Lewis Summer internship program. During that time, he was involved in a research program to investigate the magnetic field penetration depth of superconducting materials. He returned to NASA Lewis in the summer of 1989 where he asssisted in the development of automation software for a secondary ion mass spectrometer. In 1990, he joined the Solid State Technology Branch at NASA Lewis Research Center as a full-time employee. His current interests lie in both the theoretical and experimental investigation of the electrical properties of novel semiconductor materials. Mr. Mena has authored a paper on his work conducted at Arizona State University.

Carlos R. Morrison, a native of Jamaica, earned his B.S. (Honorary) degree in Physics in 1986 from Hofstra University where he was elected to the Sigma Pi Sigma National Physics Honor Society, the Kapa Mu Epsilon National Mathematics Honor Society, and the Society of Physics Students. He received a Master's degree in Physics in 1989 from the Polytechnic Institute of New York. He joined NASA Lewis Research Center in September of 1989. He worked for a short time in Reliability and Quality Assurance, then transferred to the Solid State Technology Branch in January of 1990. Currently Mr. Morrison is involved in thin film deposition and ellipsometry.

George E. Ponchak earned his B.E.E. from Cleveland State University in 1983, and his M.S.E.E. from Case Western Reserve University in 1987. He joined the Space Electronics Division at NASA Lewis Research Center in July, 1983. Since joining NASA, he has been responsible for research of microwave and millimeter wave transmission lines and managing the development of monolithic microwave integrated circuits. Mr. Ponchak has coauthored 22 papers on these topics. In September of 1990, Mr. Ponchak attended the University of Michigan for one year to complete coursework towards a Ph.D. in Electrical Engineering.

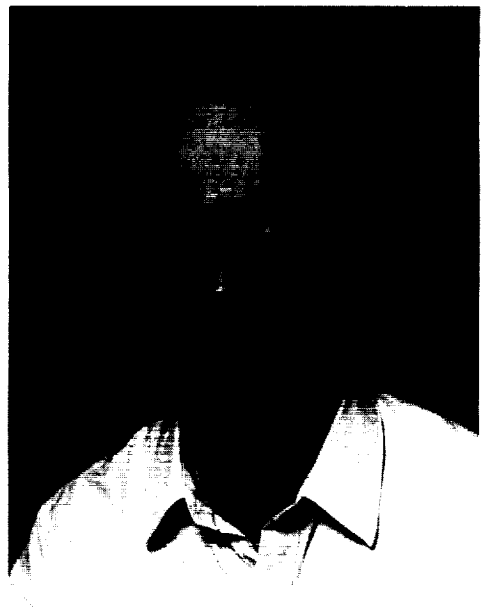


John J. Pouch earned his Ph.D. in Solid State Physics from Wayne State University, Detroit, MI, in 1981. His research activities at NASA Lewis Research Center include surface analysis of thin films for microelectronic applications, plasma and reactive ion etching, and plasma deposition.



Robert R. Romanofsky earned a Bachelor of Science Degree in Electrical Engineering from Pennsylvania State University, and a Master of Science Degree in Electrical Engineering from he University of Toledo. He has been employed in the Space Electronics Division of NASA Lewis Research Center since 1983. He spent one year at NASA Headquarters in Washington, D.C., as the acting program manager for superconductivity and RF communications. His work has involved microwave transmission line research and device characterization and modeling. Recently, he has been investigating microwave applications of high-temperature superconductivity.

Samuel E. Schacham earned his B.S. degree in Mathematics and Physics with honors in 1971, and an M.S. degree in Physics in 1973 from Bar Ilan University, Ramat-Gan, Israel. He performed research work on nonlinear optical effects in liquid crystals at the Weitzmann Institute and Bar Ilan University. He received a Ph.D. degree in Biomedical Engineering in 1978 from Northwestern University, Evanston, IL, working on applications of lasers to microendoscopy. From 1978 to 1981, he was the manager of the optic group at Fibronics Ltd., Haifa, Israel. From there he joined the Department of Electrical Engineering at the Technion in Haifa. In 1988 he was a visiting scientist at MCNC Research, Traingle Park, NC, working on optical interconnects as part of the packaging group. Presently, he is with NASA Lewis Research Center as a National Research Council Senior Research Associate. His present research interests are in the physical properties of quantum structures and narrow bandgap semiconductors. His list of publications includes 40 papers in international scientific journals and referred conferences.

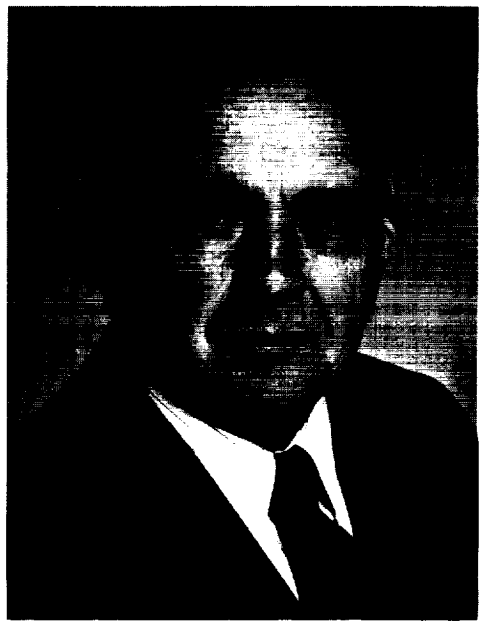


Ajit K. Sil earned his B.S. degree from Calcutta University, India. He emigrated to the United States and was employed by Oakwood Downriver Medical Center. He received his second B.S. degree in Electronic Engineering Technology in 1988 from Wayne State University, Detroit, MI. He joined the NASA Lewis Research Center in July of 1989 as a member of the Solid State Technology Branch where he is working with microwave integrated circuits and solid state devices. He is currently working toward his M.S.E.E. at Cleveland State University.

Rainee N. Simons earned a B.E. degree in Electronics and Communications from the Mysore University in 1972, and an M. Tech. degree in Electronics and Communications from the Indian Institute of Technology, Kharagpur, in 1974, and a Ph.D. in Electrical Engineering from the Indian Institute of Technology, New Delhi, in 1983, where he was a Senior Scientist Officer. From 1985 to 1987, Dr. Simons was a National Research Council Research Associate and investigated the direct optical control of GaAs microwave semiconductor devices and circuits. Since 1990, he has been with Sverdrup Technology, Inc., NASA Lewis Research Center Group. His research interest include GaAs microwave semiconductor devices, optical control, and superconductivity. Dr. Simons is the author of a book entitled "Optical Control of Microwave Devices" published by Artech House. He has received the Distinguished alumni award from his alma mater and is a senior member of IEEE.

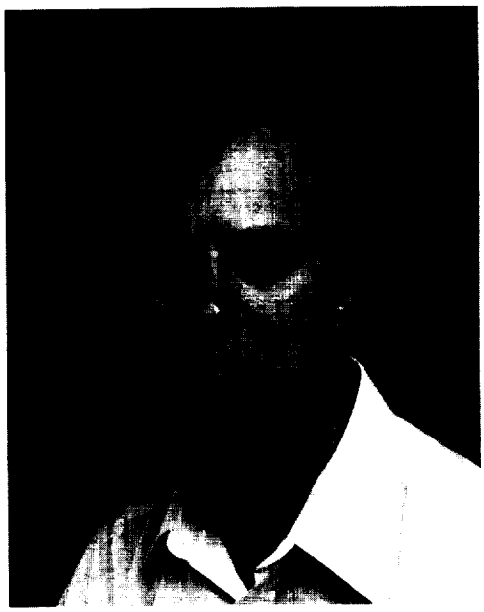
Mark A. Stan earned his B.S.E.E. degree in 1978 from the University of Akron. He was employed by the Allen-Bradley Company as a digital circuit design engineer until 1980. He then returned to graduate school at Case Western Reserve University to study the physics of melting in two-dimensional systems, completing work for his Master's degree in 1982 and a Ph.D. degree in 1988. In 1987 he began research work at NASA Lewis Research Center in the areas of characterization and growth of high-temperature superconductors. Currently Dr. Stan is a Resident Research Associate. He is a member of the American Physical Society (APS), the Materials Research Society (MRS), and the Institute of Electrical and Electronic Engineers (IEEE).

Stephan Stecura earned an M.S. degree in Physical Chemistry in 1957 from Western Reserve University, and a Ph.D. in Solid State Reaction Kinetics-Thermodynamics in 1965 from Georgetown University. From 1958 to 1965, at College Park Metallurgy Center, he studied the kinetics of crystallographic transformation and the high-temperature properties of oxides by high-temperature, x-ray diffraction techniques. He designed and built high-temperature, x-ray diffraction equipment and was invited to present the high-temperature, x-ray diffraction, arc-image furnace, capable of reaching 3000 degrees Celsius in air, at the International Crystallographic Society meeting. Since 1965, he has been with NASA Lewis Research Center. His work on heat pipes led him to determine the corrosion mechanism and the true solubilities of containment metals and alloys in alkali metals. He developed thermal barrier systems for the protection of alloy components at very high temperatures, up to 1600 degrees Celsius, on air-cooled components. Currently, he is studying the properties of superconducting materials and is trying to identify substrate materials for superconducting films. Dr. Stecura is recognized as an authority on the thermal barrier system technology that he developed. For his work in this field, he has received two IR-100 awards, one major Space Act award, and three major patents. He has written more than 30 original publications and is a member of the American Ceramic Society.



Susan R. Taub earned her B.S. degree in Electrical Engineering Technology in 1988, and a Master of Science degree in Electrical Engineering in 1990 from Temple University. In 1988 and 1989, she worked for AT&T Bell Laboratories, Reading, PA, developing PSPICE compatible models for power MOSFET's. She joined the Solid State Technology Branch of NASA Lewis Research Center in 1990, and is currently involved in the design and characterization of MMIC's and the investigation of HEMT performance at cryogenic temperatures. Ms. Taub is a member of IEEE.

Joseph D. Warner earned an M.S. in Physics from Carnegie Melon University in 1977. From 1977 to 1981, he performed research at C.M.U. on magnetic phase transition at low temperature. Since that time, he has been with NASA Lewis Research Center where he characterized various insulators on GaAs and was among the first to demonstrate growth of GaAs by laser-assisted OMCVD at temperatures below 500 degrees Celsius. Presently, he has set up a laser ablation experiment to grow high-temperature superconducting thin films. In 1989, he received a NASA Achievement Award for his part in establishing a high-temperature superconductor program at NASA Lewis. Mr. Warner has authored papers on magnetic phase transitions, electrical properties of insulation films on III-V compounds, laser assisted growth of GaAs and AlGaAs, and properties and growth of high-temperature superconductors. Mr. Warner is a member of the American Physical Society (APS), the American Vacuum Society (AVS), and the Materials Research Society (MRS). Currently, he is on a one-year assignment to the Jet Propulsion Laboratory where he participates in the development of Photonic intergrated circuits.



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13. ABSTRACT ***(Maximum 200 words)***

The digest is a collection of papers written by the members of the Solid State Technology Branch of NASA Lewis Research Center from June 1990-June 1991. The papers cover a range of topics relating to superconductivity, MMIC's, coplanar waveguide, and material characterization.

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