

NASA Technical Memorandum 102 436

Growth and Patterning of Laser Ablated Superconducting $\text{YBa}_2\text{Cu}_3\text{O}_7$ Films on LaAlO_3 Substrates

(NASA-TM-102436) GROWTH AND PATTERNING OF
LASER ABLATED SUPERCONDUCTING $\text{YBa}_2\text{Cu}_3\text{O}_7$
FILMS ON LaAlO_3 SUBSTRATES (NASA) 8 p
CSCL 20L

N90-22421

Unclass

G3/76 0279878

J.D. Warner, K.B. Bhasin, N.C. Varaljay, and D.Y. Bohman
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

and

C.M. Chorey
Sverdrup Technology, Inc.
NASA Lewis Research Center Group
Cleveland, Ohio

Prepared for the
36th National Symposium and Topical Conference
sponsored by the American Vacuum Society
Boston, Massachusetts, October 23-27, 1989



100

100

100

100

GROWTH AND PATTERNING OF LASER ABLATED SUPERCONDUCTING $\text{YBa}_2\text{Cu}_3\text{O}_7$,
FILMS ON LaAlO_3 SUBSTRATES

J.D. Warner, K.B. Bhasin, N.C. Varaljay, D.Y. Bohman,
NASA Lewis Research Center Cleveland, OH 44135

C.M. Chorey*
Sverdrup Technology, Inc., NASA Lewis Research Center Group,
Cleveland, OH 44135

ABSTRACT

A high quality superconducting film on a substrate with a low dielectric constant is desired for passive microwave circuit applications. In addition, it is essential that the patterning process does not effect the superconducting properties of the thin films to achieve the highest circuit operating temperatures. We have grown $\text{YBa}_2\text{Cu}_3\text{O}_7$ superconducting films on lanthanum aluminate substrates using a laser ablation technique with resulting maximum transition temperature (T_c) of 90 K. The films were grown on LaAlO_3 which was at 775 °C and in 170 mtorr of oxygen and slowly cooled to room temperature in 1 atm of oxygen. These films were then processed using photolithography and a negative photoresist with an etch solution of bromine and ethanol. Results are presented on the effect of the processing on T_c of the film.

I. INTRODUCTION

Laser-ablated, high-temperature superconducting (HTS) $\text{YBa}_2\text{Cu}_3\text{O}_7$ films have been made on many substrates including SrTiO_3 , MgO , LaGaO_3 , and ZrO_2 .¹⁻⁷ These substrates were used because they either had very small interaction with the HTS films during growth or annealing or because the substrates with HTS films had potential electronic applications. In this paper, we report on the growth and patterning of thin $\text{YBa}_2\text{Cu}_3\text{O}_7$ films on LaAlO_3 for microwave applications. LaAlO_3 was chosen as a substrate because of its relatively low dielectric constant of 22⁸ and because of its moderate loss tangent of 8×10^{-5} at 10 GHz⁹.

Lines varying in width from 10 to 20 μm were patterned using photolithography and wet etching techniques. To determine if the etching or lithography process had influenced the transition temperature of the films. A ring resonator circuit operating at 35 GHz was also fabricated, since the resonator allows the determination of loss and dispersive properties of microstrip transmission line. From the measurement of the quality factor "Q" of a resonator circuit one can determine the microwave losses of the HTS films as compared with those of gold on the same substrates.

*Work done under NASA contract #NAS3-25266; Regis Leonard, monitor.

II. Film Growth

The laser ablation technique used to grow the films on substrates of LaAlO_3 is similar to the techniques reported in the literature.¹⁻⁷ The details of the geometry of the laser ablation is shown in Fig. 1. The substrates (15 by 15 by 0.25 mm) with orientation (001) were mounted onto a stainless steel plate with a diameter of 63 mm. The plate was heated from the backside using a

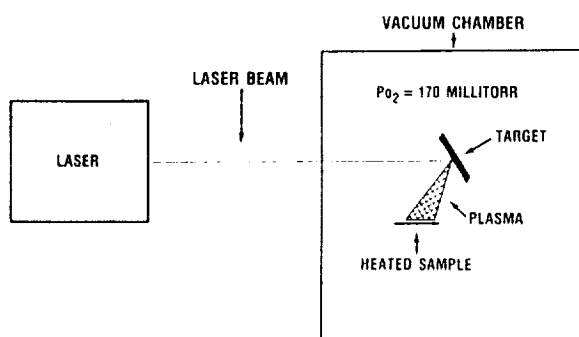


Figure 1. - Schematic of the laser ablation experiment.

resistive heater made from Kanthal A-1 wire (made by Kanthal, Inc.). The temperature was measured with a type K thermocouple which was welded to the plate. The thermocouple was 2 mm away from the sample. The sample chamber was evacuated to 3×10^{-7} torr, or lower, using a liquid nitrogen cold trapped diffusion pump before the sample was warmed up to 500 °C. A continuous

flow of oxygen (120 sccm) was then introduced into the chamber, and the sample heated to 775 °C. During deposition the chamber pressure was 170 mtorr; the laser wavelength was 248 nm; the energy density was 1.5 (J/cm²)/pulse; the pulse rate was 4 pps; and the distance between the target and the sample was 8 cm. The laser beam was rastered up and down 1 cm over the target using an external lens on a translator. The angle between the laser beam and the normal to the target was 45°. The target used was a sintered 25-mm-diameter pellet of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$. After deposition 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 hr before it was lowered to 250 °C at a rate of 2 °C/min. The heater power was then turned off, and the sample was allowed to cool to 40 °C or less before it was removed the chamber.

The thickness of the HTS films on LaAlO_3 was estimated by measuring the thickness of a film grown on quartz plate that was shadow masked. The quartz plate had been placed 1 mm below the bottom of the LaAlO_3 on the substrate holder such that the sweep of the plasma plumb was along the line connecting the centers of the quartz and the LaAlO_3 .

The best film had a T_c of 89.8 K immediately after deposition as determined by a standard four point resistance measurement. Its resistance versus temperature behavior is shown in Fig.2. From the intercept of the extrapolated resistance at 0 K and from the resistance above T_c , one can see that the film is c-axis aligned. This is confirmed by only having the (001) peaks in the x-ray diffraction data (Fig. 3). The surface morphology of the HTS on LaAlO_3 is shown in Fig. 4. The surface is very smooth with some small structure. We do not observe large numbers of HTS particulates due to the laser ablation process.

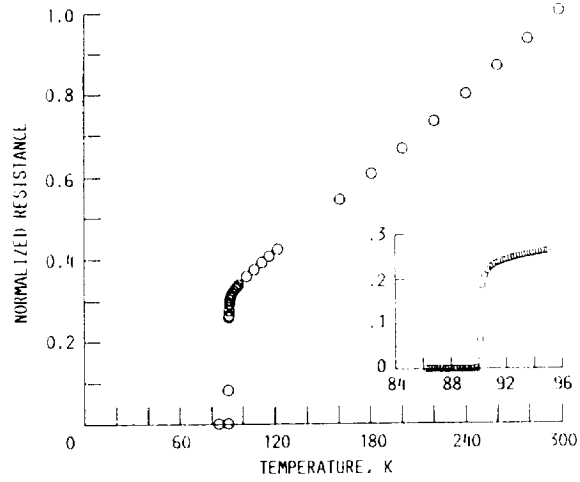


Figure 2 - Normalized resistance of laser ablated $\text{YBa}_2\text{Cu}_3\text{O}_x$ film on LaAlO_3

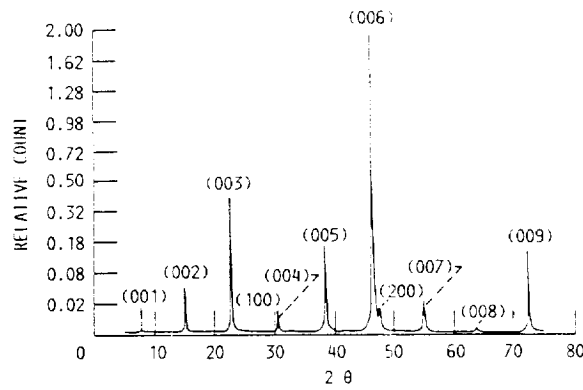


Figure 3 - X-ray diffraction pattern of laser ablated $\text{YBa}_2\text{Cu}_3\text{O}_x$ film on LaAlO_3



Figure 4 - Scanning electron micrograph of laser ablated $\text{YBa}_2\text{Cu}_3\text{O}_x$ Film on LaAlO_3

ORIGINAL PAGE IS
OF POOR QUALITY

PATTERNING

Films on LaAlO_3 and SrTiO_3 were patterned using photolithography and wet etching. The negative photoresist (752), its developer

Table I Effect of Photolithography Process on T_c of $\text{YBa}_2\text{Cu}_3\text{O}_x$ Films

Exposure to negative photoresist	None
Soft baking of photoresist at 90°C for 1 hr)	None
Exposure to photoresist developer and rinse	None
Exposure to different stripping solvents after patterning:	
Boiling acetone (58°C) for 10 min	None
Boiling ethanol (78°C) for 10 min	None
Boiling toluene (111°C) for 10 min	None
Losalin IV (76°C) for 5 min	None

^aOnly boiling ethanol and Losalin IV successfully removed exposed photoresist.

(802), and the associated rinse (n-butyl acetate) used were obtained from KTI. The photoresist strippers used were Losalin IV (from E.C. Merck), acetone, toluene, and ethanol. Each step of the process was checked to see if it had an effect on the T_c of the HTS films. The results of the different processing steps on T_c are shown in table I. The full process of patterning the HTS films was to spin on the negative

photoresist to a thickness of $2\ \mu\text{m}$, followed by a soft bake at 90°C for 1 hr, and then to expose the photoresist. After developing the photoresist, the film was etched for 500 s in 1 percent molar of bromine in ethanol. After etching, the films were rinsed in ethanol and the photoresist was removed with the Losalin IV photoresist stripper which was at 70°C . We did not observe any drop in the T_c

of the HTS films.

The films used to determine the effect of the various fabrication steps had transition temperatures between 77 and 85 K. Fig. 5 shows the effect of exposing the film directly to the photoresist stripper Losalin IV at 70°C . No change in T_c occurred, but the slope of the resistance versus temperature curve did change. Fig. 6 shows the T_c of the film on LaAlO_3 before patterning and after it was patterned into a ring resonator, that had operated at 33 GHz, and after it had

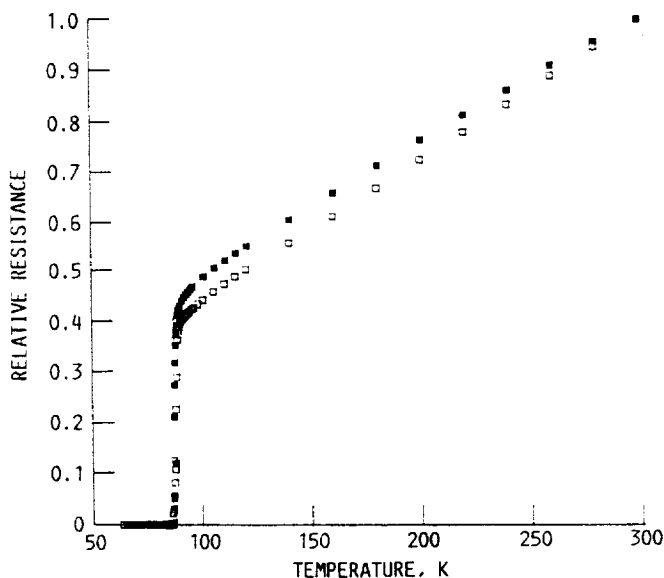


Figure 5. - Normalized resistance of a laser ablated $\text{YBa}_2\text{Cu}_3\text{O}_x$ film on SrTiO_3 . The film □ is before processing while the film ■ is after exposure to negative photoresist.

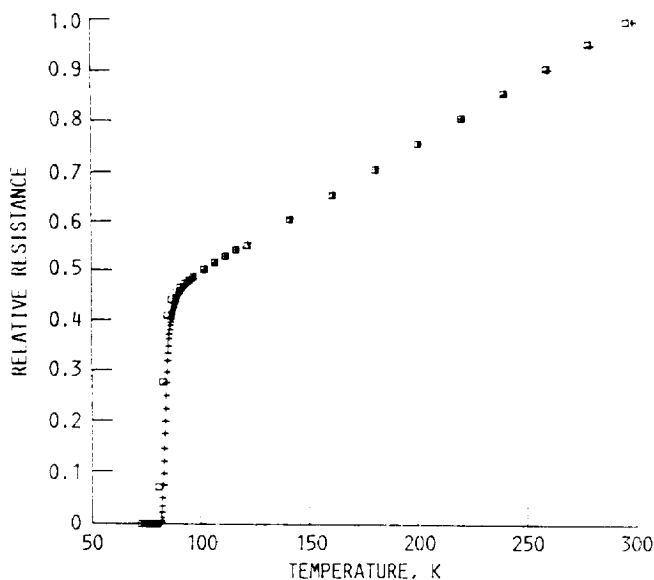


Figure 6. - Comparison of normalized resistance of a laser ablated $\text{YBa}_2\text{Cu}_3\text{O}_x$ film on LaAlO_3 before processing \square and after being fabricated into a 35-GHz ring resonator $+$.

silver contacts evaporated onto it and annealed at 500°C for 1 hr. There is no apparent difference in the T_c or the resistance versus temperature behavior between the film before and after processing.

To test the laser ablation technique's ability to produce uniform film thickness and the variation of T_c across the film, Hall bars with silver contacts were fabricated (Fig. 7). The width of the bar is $10\ \mu\text{m}$. The film thickness is not very uniform over the 5-by-10-mm area. The time needed to etch the film

until the substrate was exposed varied by a factor of 2 from one edge to the other edge of the substrate. However, the T_c did not vary from region to region (table II).

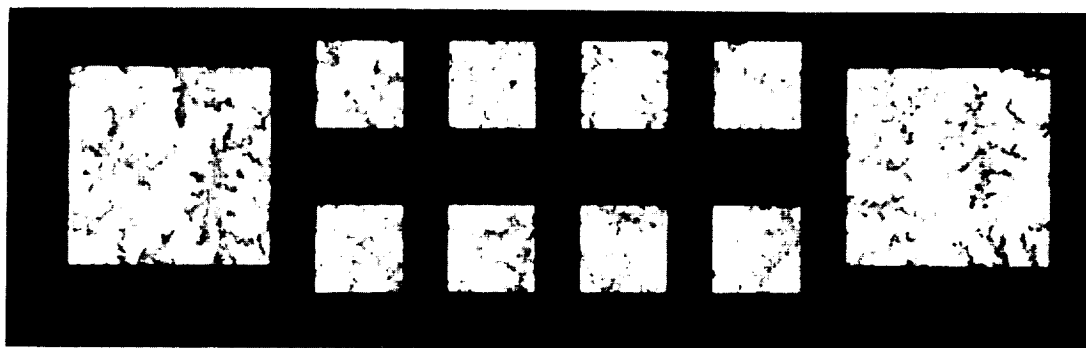


Figure 7. - Finished Hall bar of $\text{YBa}_2\text{Cu}_3\text{O}_x$ film on SrTiO_3 substrate.

Table II Variation of T_c on SrTiO_3 as Measured With Patterned Hall Bars

Before patterning	70.8 K
10- μm lines after patterning	71.0, 71.5, 72.5 K
20- μm lines after patterning	75.3, 75.5, 76.5 K

Conclusions

Laser-ablated, high-temperature superconducting films on LaAlO_3 and SrTiO_3 have been grown. The best films had a T_c of 90 K and have their c-axis aligned to the substrate. There is no variation of T_c across the films, but there is a variation of film thickness. These films have been patterned with negative photoresist and a bromine/ethanol etch. There is no detectable degradation of T_c by any step of the fabrication process even though the films were heated to 122° C in toluene.

This fabrication process should be able to be used to make most of the passive and one layer structures without any degradation of the transition temperature.

REFERENCES

1. J.D. Warner, J.E. Meola, and K.A. Jenkins, NASA TM-102350, 1989.
2. D. Dijkkamp, T. Venkatesan, X.D. Wu, S.A. Shaheen, N. Jisrawi, Y.H. Min-Lee, and W.L. Mclean, M. Croft, Appl. Phys. Lett. 51, 619 (1987).
3. X.D. Wu, D. Dijkkamp, S.B. Ogale, A. Inam, E.W. Chase, P.F. Miceli, C.C. Chang, J.M. Tarascon, and T. Venkatesan, Appl. Phys. Lett. 51, 861 (1987).
4. J. Narayan, N. Biunno, R. Singh, O.W. Holland, and O. Auciello, Appl. Phys. Lett. 51, 1845 (1987).
5. L. Lynds, B.R. Weinberger, G.G. Peterson, and H.A. Kransinski, Appl. Phys. Lett. 52, 320 (1988).
6. T. Venkatesan, C.C. Chang, D. Dijkkamp, S.B. Ogale, E.W. Chase, L.A. Farrow, D.M. Hwang, P.F. Miceli, S.A. Shwarz, J.M. Tarascon, X. D. Wu, and A. Inam, J. Appl. Phys. 63, 4591 (1988).
7. A.M. Desantolo, M.L. Mandich, S. Sunshine, B.A. Davidson, R.M. Fleming, P. Marsh, and T.Y. Kometani, Appl. Phys. Lett. 52, 1995 (1988)
8. F.A. Miranda, W.L. Gordon, V.O. Heinen, B.T. Ebihara, and K.B. Bhasin, NASA TM-102123, 1989.
9. R.W. Simon, C.E. Platt, A.E. Lee, G.S. Lee, and K. Daly, Appl. Phys. Lett., 53, 2677 (1988).

1. Report No. NASA TM-102 436		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Growth and Patterning of Laser Ablated Superconducting $\text{YBa}_2\text{Cu}_3\text{O}_7$ Films on LaAlO_3 Substrates				5. Report Date	
				6. Performing Organization Code	
7. Author(s) J.D. Warner, K.B. Bhasin, N.C. Varaljay, D.Y. Bohman, and C.M. Chorey				8. Performing Organization Report No. E-5205	
				10. Work Unit No. 307-51-00	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546-0001				14. Sponsoring Agency Code	
15. Supplementary Notes Prepared for the 36th National Symposium and Topical Conference, sponsored by the American Vacuum Society, Boston, Massachusetts, October 23-27, 1989. J.D. Warner, K.B. Bhasin, N.C. Varaljay, and D.Y. Bohman, NASA Lewis Research Center; C.M. Chorey, Sverdrup Technology, Inc., NASA Lewis Research Center Group, Cleveland, Ohio 44135.					
16. Abstract A high quality superconducting film on a substrate with a low dielectric constant is desired for passive microwave circuit applications. In addition, it is essential that the patterning process does not effect the superconducting properties of the thin films to achieve the highest circuit operating temperatures. We have grown $\text{YBa}_2\text{Cu}_3\text{O}_7$ superconducting films on lanthanum aluminate substrates using laser ablation with resulting maximum transition temperature (T_c) of 90 K. The films were grown on LaAlO_3 which was at 775 °C and in 170 mtorr of oxygen and slowly cooled to room temperature in 1 atm of oxygen. These films were then processed using photolithography and a negative photoresist with an etch solution of bromine and ethanol. Results are presented on the effect of the processing on T_c of the film and the microwave properties of the patterned films.					
17. Key Words (Suggested by Author(s)) Laser ablation; HTS; Superconductor; High temperature superconductor; LaAlO_3 ; Thin film; Patterning			18. Distribution Statement Unclassified - Unlimited Subject Category 76		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of pages 8	22. Price* A02

