Resolution limits of ion milling for fabricating $Y_1Ba_2Cu_3O_x$ nanostructures

F-O. Fong,[a)] S. C. Deshmukh,[b)] M. F. Davis,[a)] D. P. Stumbo,[a)] J. C. Wolfe,[a)] and D. J. Economou[b)]
Texas Center for Superconductivity, University of Houston, Houston, Texas 77204

(Received 8 July 1991; accepted for publication 25 November 1991)

A process is described for fabricating 0.23-μm-wide lines in $Y_1Ba_2Cu_3O_x$ thin films where patterns are defined in a commercial, negative tone, epoxy-based resist by masked ion beam lithography and transferred to the superconducting film by argon ion milling. Lines in 80-nm-thick films had the same zero-resistance temperature (89 K) as the starting films, and a critical current density of 0.7×10^6 A/cm^2 at 77 K, representing a threefold reduction from the starting value. A consistent interpretation of these results is that the line consists of a superconducting core 70 nm in width with the critical current density of the starting film and with 80-nm-wide nonsuperconducting sidewalls. The results were reproducible in lines which did not cross outgrowths in the superconducting film.

The fabrication of high-resolution superconducting structures is important to probing the nature of grain boundaries,[1,2] for the formation of planar proximity effect devices,[3] and for minimizing white noise in superconducting quantum interference devices (SQUIDs).[4] It is important, in each of these applications, that the patterning process faithfully preserve the superconducting properties of the starting film. The smallest lines yet reported which do not suffer serious small-size effects are the 0.5-μm-wide, ion milled, grain-boundary junctions of Ref. 4. Further linewidth reductions to 0.25 μm showed a J_c reduced by a factor of 25 relative to the 0.5-μm-wide lines. In this paper we again use the ion milling approach and describe a process which produces high quality 0.23-μm-wide lines. The data further suggests that the process will reach a fundamental limit near 0.16 μm.

A total of 14 samples were cut from a 3×2 cm, 80-nm-thick epitaxial thin film deposited on a $LaAlO_3$ substrate by laser ablation with substrate scanning.[5] The transition temperature of the unpatterned samples was 89 K, and the critical current density of a 10-μm-wide bridge, 100 μm in length, was 2×10^6 A/cm^2 at 77 K using Elkin's offset criterion.[6] The resistivity at 100 K was 100 $\mu\Omega$ cm.

Lithographic patterns were defined using masked ion beam lithography (MIBL).[7] Briefly, MIBL is a high-resolution proximity printing process where a stencil mask is illuminated by a beam of light ions. The transmitted ions transfer the mask pattern to resist. The MIBL tool was used in these experiments because of the high level of linewidth reproducibility. In addition, the possibility of defining very high aspect ratio resist structures will be important to future extensions of the process to thicker films. Mask patterns were defined in poly(methylmethacrylate) resist by electron beam lithography on 0.8-μm-thick silicon membranes. The resist pattern was transferred into the membrane using magnetically enhanced reactive ion etching in molecular bromine.[8] Figure 1 shows the mask window used to define a 0.23-μm-wide bridge, 1.0 μm in length.

The mask was used to expose 0.45-μm-thick Shipley[9] SAL 601 ER7™ resist with 100 keV H_2^+ ions. The exposure dose was 0.4 μC/cm^2 and the exposure time was 1 s. This light dose (5×10^{12} protons/cm^2) had no apparent effect on either the transition temperature or the critical current density of the films. The resist was baked in a convection oven at 80 °C for 30 min before exposure and at 115 °C for 1–15 min after exposure. The developer was a 1:3 mixture of methylisobutylketone and isopropanol. This solvent-based developer was used instead of the recommended aqueous developers (Shipley MF-319™ or MF-622™) to avoid water damage to the film. A hard bake at 150 °C for 30 min was used after development. The resist pattern was transferred into the film by argon ion milling at 500 V. It was found that a resist-to-film thickness ratio of 5:1 was required for etching 0.2-μm-wide lines, even though the resist-to-$Y_1Ba_2Cu_3O_x$ etch rate ratio was 2:1 for large areas. This is because resist faceting during the ion milling process increases the effective etch rate of these small resist lines. An O_2-microwave plasma was

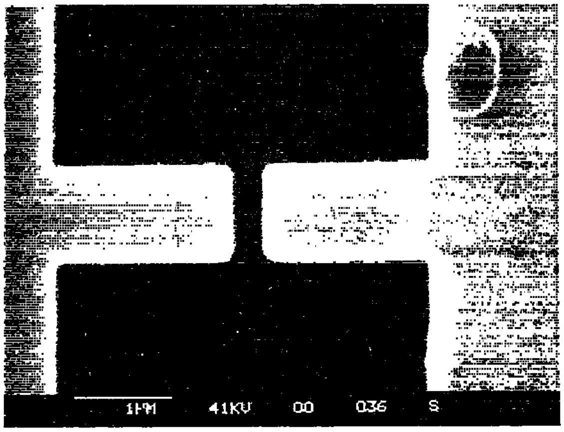

FIG. 1. Micrograph of a 0.8-μm-thick silicon stencil mask, for fabricating a 0.2-μm-wide line 1.0 μm in length.

[a)]Department of Electrical Engineering.
[b)]Department of Chemical Engineering.

 0021-8979/92/052461-03$04.00

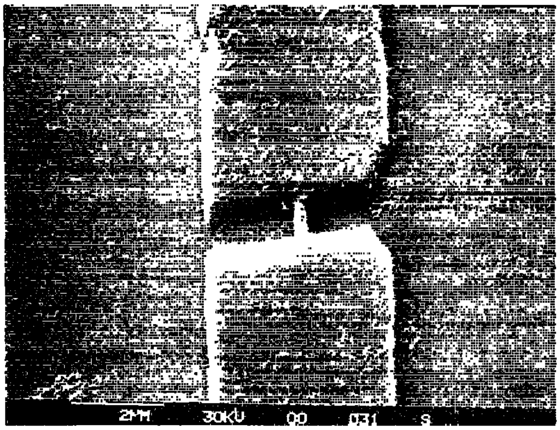

FIG. 2. Negative tone resist image formed by masked ion beam lithography using the mask in Fig. 1.

used to strip the resist. This process minimized ion bombardment of the film and appears to have no detrimental effect on these very thin films even for over etching by factors of 5–10. Figure 2 shows the resist structures used to define the 0.23-μm line and Fig. 3 shows the superconducting line after ion milling and resist stripping. Transition temperature and critical current density measurements were performed using a four-point, lock-in technique with a 1-kHz small signal, excitation current of 1.0 μA. A short bridge length of 1 μA. A short bridge length of 1 μm was used to minimize the possibility of crossing outgrowths on the ablated film. This short length caused two limitations on the measurements. First, because of long YBCO leads between the voltage contact pads and the bridge itself, the structure is relatively insensitive to the bridge resistance. In fact, the bridge resistivity could increase by a factor of 2 relative to that of the leads and represent only a 10% change in the measured resistance between the voltage probes. The second, and more fundamental, limitation is that the voltage sensitivity of the lock-in amplifier (10 nV) limits our minimum detectable electric field to 100 μV/cm.

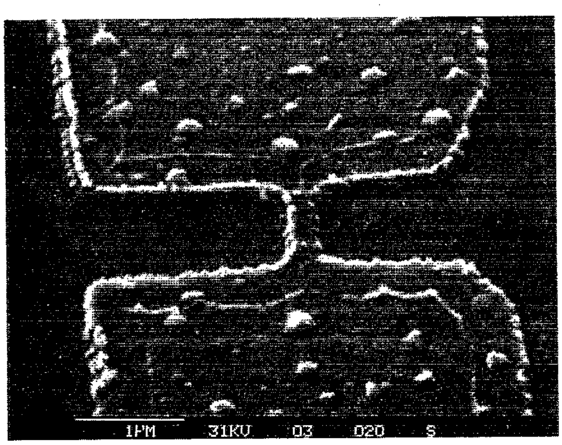

FIG. 3. Micrograph of a 80-nm-thick, 0.23-μm-wide, 1.0μm-long $Y_1Ba_2Cu_3O_x$ line formed by masked ion beam lithography and ion milling.

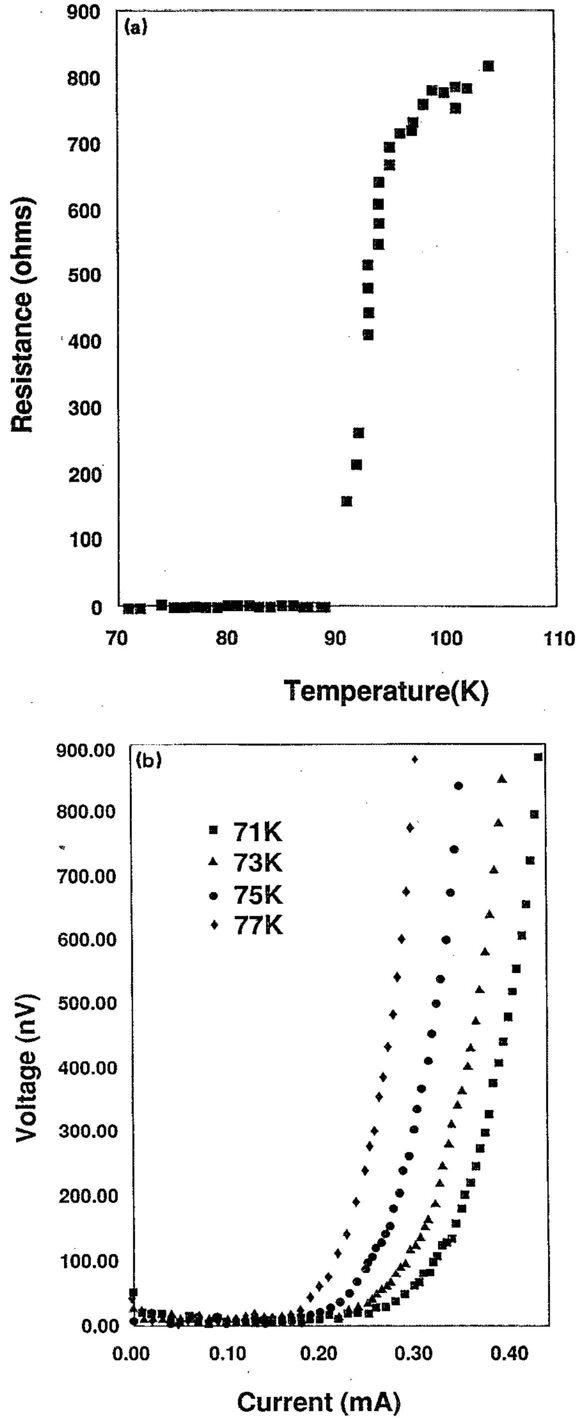

FIG. 4. Resistance vs temperature (a) and small signal voltage as a function of dc-bias current at several temperatures (b), for the line in Fig. 2.

This is a factor of 10 larger than the field used to define the critical current density by the offset criterion. In this paper, we define "critical current density" as the current corresponding to a small-signal voltage of 25 nV (corresponding to a field of 250 μV/cm). Thus, our value of J_c is somewhat higher than that which would be obtained using the offset criterion. Figures 4(a) and 4(b) show, respectively, the resistive transition and the dependence of small-signal voltage amplitude on bias current at several temperatures for a 0.23-μm-wide line. The zero resistance temperature 89 K and the critical current density is 0.7×10^6 A/cm^2 at 77 K. The normal state resistivity at 100 K is consistent with the value 100 $\mu\Omega$ cm measured on the 10-μm-wide line, but again, we cannot accurately determine this parameter because of the shortness of the line. A total of twelve such bridges were formed and, of these, eight failed as open circuits. The failures have been tentatively attributed to the random occurrence of "outgrowths," present in the starting film, in the pattern area. The remaining four had the same zero resistance temperatures within 0.5 K and the same J_c within 10%. These errors are at the limits of measurement accuracy.

In summary, we have demonstrated, for the first time, successful patterning of sub-0.5-μm-wide lines in 80-nm-thick YBCO films. The transition temperature of the structures was unchanged from that of the unpatterned film and the critical current density of an 0.23-μm-wide line decreased by a factor of 3 from that of a 10-μm-wide line. This suggests that the line consists of an undamaged core 70–80 nm wide with a 70–80-nm-wide nonsuperconducting region on each sidewall. An ion damaged region with this width is expected on the basis of the results of Pang *et al.* in ion assisted etching of GaAs.[10] This result suggests that the minimum physical linewidth which will have an undamaged superconducting core will be about 0.16 μm, a hypothesis which will be tested in a future systematic study of J_c versus linewidth.

This work has been supported by the State of Texas through the Texas Center for Superconductivity at the University of Houston and the Advanced Technology Program.

[1] P. Chaudhari, J. Mannhart, D. Dimos, C. C. Tsuei, J. Chi, M. M. Oprysko, and M. Scheuermann, Phys. Rev. Lett. **60**, 1653 (1988).
[2] S. E. Russek, D. K. Lathrop, B. H. Moeckly, R. A. Buhrman, D. H. Shin, and J. Silcox, Appl. Phys. Lett. **57**, 1156 (1990).
[3] P. M. Mankiewich, D. B. Schwartz, R. E. Howard, L. D. Jackel, B. L. Straughn, E. G. Burkhardt, and A. H. Dayem, Proc. Soc. Photo-Opt. Instrum. Eng. **37**, 848 (1988).
[4] M. Kawasaki, P. Chaudhari, T. H. Newman, and A. Gupta, Appl. Phys. Lett. **58**, 2555 (1991).
[5] M. F. Davis, J. Wosik, K. Forster, S. C. Deshmukh, H. R. Rampersad, S. Shah, P. Siemsen, J. C. Wolfe, and D. J. Economou, J. Appl. Phys. **69**, 7182 (1991).
[6] J. Ekin, Appl. Phys. Lett. **55**, 905 (1989).
[7] F-O Fong, D. P. Stumbo, S. Sen, G. Damm, D. W. Engler, J. C. Wolfe, J. N. Randall, P. Mauger, and A. Shimkunas, Microcircuit Engineer. **11**, 449 (1990).
[8] F-O Fong, S. Sen, D. P. Stumbo, J. C. Wolfe, and J. N. Randall, J. Vac. Sci. Technol. B **6**, 2112 (1988).
[9] Shipley Co., Newton, MA 02162.
[10] S. W. Pang, W. D. Goodhue, T. M. Lyszczarz, D. J. Ehrlich, R. B. Goodman, and G. D. Johnson, J. Vac. Sci. Technol. B **6**, 1916 (1988).