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# Lower-Submicron Patterning Process for BiSrCaCuO High-T<sub>c</sub> Superconducting Thin Films

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Ion bombardment enhanced etching (IBEE) has been successfully applied to the BiSrCaCuO high-Tc superconducting thin films by using "nonaqueous" Br<sub>2</sub> solutions. It was found that the etch rate of the damaged region formed by the ion irradiation with 200 keV Si<sup>++</sup> focused ion beam was enhanced with the Br<sub>2</sub> treatment. The critical dose was about  $1\times10^{14}$ ions/cm<sup>2</sup>. Bridge structures with a width of 0.2  $\mu$  m were fabricated with little degradation in Tc and Jc.

# 1. Introduction

Many studies have been made on microfabrication techniques for high-Tcsuperconducting thin films. High-resolution patterning processes which cause little degradation of the film properties must be developed for the electronic applications of these materials. Several authors have reported dry etching processes such as ion beam etching<sup>1)</sup> and maskless sputter etching with focused ion beams (FIB).2.3 Submicron features were obtained by using such processes.

Ion bombardment enhanced etching (IBEE) is an effective method for making fine structures in Si or GaAs crystal with high resolutions.4) Furthermore, the IBEE process generally leaves little damage in the fabricated patterns because the damaged region formed by the ion bombardment is selectively removed by the etching process. We have reported, for the first time, the IBEE applied process for the high-Tcsuperconducting thin films.<sup>5)</sup> In the report. we used alkaline solutions as the etchant and the region irradiated at the dose more than certain critical dose could be etched by the treatment. However, the critical dose of the IBEE process using the alkaline solutions was much higher than the dose which destroys the superconductivity of the film. As a result, part of the damaged region remained in the treated films and submicron superconductive features could not be obtained.

In this paper, we present a successful IBEE process for BiSrCaCuO (BSCCO) high-Tc superconducting thin films using bromine solutions. It was found that the bromine solutions dissolve the region irradiated at the dose of about three orders of magnitude lower than the critical dose of the IBEE process using the alkaline treatment. It is expected that the films are patterned with high resolution and that little damage is left in the fabricated patterns.

## 2. Experimental

Thin films of Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> (BSCCO) were prepared on (100) MgO. The epitaxial and in situ growth of the films was made by using a molecular-beam-epitaxy (MBE) technique with an atomic oxygen radical beam source<sup>6</sup>. The substrate temperature was 700 °C during deposition. No post annealing was carried out. The thickness of the films used in this study was 0.1 - 0.13  $\mu$  m. The BSCCO film exhibited a superconducting transition temperature Tc of 50 - 60 K. A 100 kV FIB apparatus was used in this study. Ions of Si<sup>++</sup> were used for the ion irradiation. A typical probe current of the 200 keV Si<sup>++</sup> FIB was 1 pA and a beam diameter was about 0.1  $\mu$  m. After the ion irradiation, the samples were immersed in the Br<sub>2</sub> solutions<sup>7.8)</sup> to the irradiated regions. The Br2 dissolve etching solutions was 0.1 % by volume Br<sub>2</sub> in absolute ethanol (ETOH). The treatment was typically carried out at 6  $^\circ\!C$  for several seconds in the solutions. The samples were rinsed with ETOH after the Br<sub>2</sub> treatment.

Bridge structures were fabricated to investigate the electrical properties of the patterned films. We made multibridge structures by delineating broken lines in the BSCCO films. We made 20 bridges in a sample and measured the current flowing along the bridges. Resistance versus temperature curves of the multibridge structures were measured by a standard four-probe technique. The critical current densities of the bridges were estimated from the measured critical current divided by the sectional area of the multibridges.

## 3. Results and Discussion

Figure 1 shows SEM micrographs taken before and after the Br<sub>2</sub> treatment of the BSCCO film which was irradiated with the Si\*\* FIB. The irradiation dose of Fig. 1(b) was  $1.4 \times 10^{14}$  ions/cm<sup>2</sup>. In Fig. 1(b), it is clearly seen that the damaged square region was selectively dissolved and no residue was seen on the exposed MgO substrate. This enhanced etching occurred at the region irradiated at the dose more than a critical dose of about 1×10<sup>14</sup> ions/cm<sup>2</sup>. The critical dose is about three orders of magnitude lower than that of the IBEE process using the alkaline solutions. The critical dose is roughly the same as that which destroys the superconductivity of the film.") Thus, it is expected that the nonsuperconductive region formed by the ion irradiation is etched away almost completely. Moreover, one should note that the "nonaqueous" Br2 treatment of this process causes less damage to the BSCCO films as well as the MgO substrate than the other etching processes treated with aqueous etchants.

A groove was formed by the IBEE process to see the resolution of the process. Figure 2 shows an SEM micrograph of a groove formed by the Si<sup>++</sup> FIB with a line dose of  $1.9 \times 10^{\circ}$ 

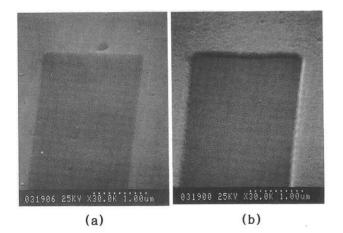


Fig.1 SEM micrographs of the BSCCO film. Square region was irradiated with the FIB. The photograph of (a) was taken before the  $Br_2$  treatment and (b) was taken after the  $Br_2$  treatment.

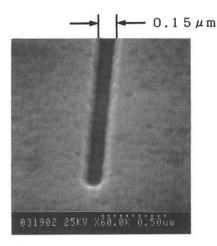


Fig.2 SEM micrograph of a groove formed in the BSCCO film.

ions/cm. It is seen that a narrow groove with a width of 0.15  $\,\mu$  m is fabricated.

Figure 3 shows etching characteristics of the irradiated BSCCO films. Initial thickness of the film was 130 nm. The etch rates of the film increased with increasing the dose. The regions irradiated at the dose more than 1×10<sup>14</sup> ions/cm<sup>2</sup> were etched entirely with 2-second treatment. An etch rate ratio of the region irradiated at the dose of 1×10<sup>14</sup> ions/cm<sup>2</sup> to the unirradiated is about 6. The critical region dose decreased with the treatment time. However, the remaining thickness at the unirradiated region decreased with the treatment time, and the surface morphology at the unirradiated region significantly degraded as well. Thus, the optimum etching condition is considered to be about 2-second treatment.

Bridge structures were fabricated by the IBEE process to examine the minimum width at which the superconducting current flows and to evaluate the remaining damaged layer. Figure 4 shows an SEM micrograph of the

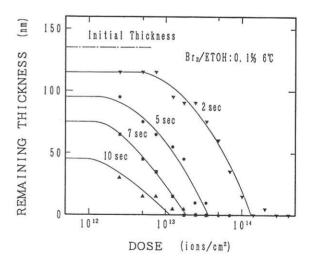


Fig.3 Etching characteristics of the irradiated BSCCO films.

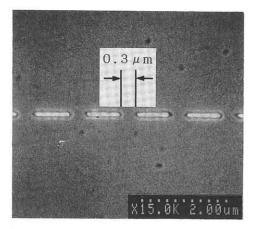


Fig.4 SEM micrograph of the multibridge with the width of 0.3  $\mu$  m

multibridge structure, where the bridges with a width of 0.3  $\mu$  m are fabricated. Resistance versus temperature curves of the virgin film and multibridges with widths of 0.7  $\mu$  m and 0.3  $\mu$  m are shown in Fig. 5. The method to measure the current flowing along the multibridge by the four-probe technique is also shown in the inset. It is confirmed that the electrical conductivity was isolated by the groove. The resistivity of the groove was estimated to be over 500 Ω cm. The bridges exhibited little change in Tc as compared with the virgin film. The multibridge with a width of 0.2  $\mu$  m were also fabricated. Figure 6 shows the temperature dependence of the critical current densities of the  $0.2-\mu$  m bridge. From the figure, it is seen that Jc of the  $0.2-\mu$  m bridge has not been significantly degraded by the IBEE process. Thus, the damaged layer is considered to be etched away almost completely by the Br<sub>2</sub> treatment, and the width of the damaged layer remaining around the grooves is estimated to be much less than 0.1  $\mu$  m.

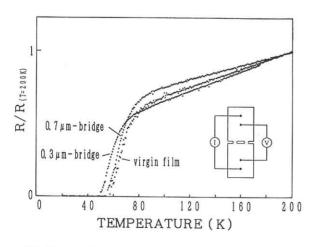


Fig.5 Resistance versus temperature curves of the multibridge and the virgin film.

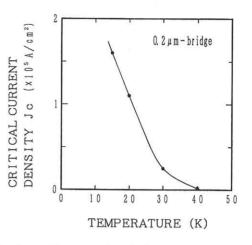


Fig.6 Temperature dependence of the critical current densities of the  $0.2-\mu$  m bridge

#### 4. Conclusions

The IBEE process has been successfully applied to the BSCCO high-Tc superconducting thin films by using "nonaqueous" Br<sub>2</sub> solutions. The etch rate of the damaged region which was formed by the ion irradiation was enhanced with Br<sub>2</sub> treatment. The critical dose of the IBEE process is roughly the same as that which destroys the superconductivity of the film. Bridge structures with a width of 0.2  $\mu$  m were successfully fabricated with little degradation in Tc and Jc. Therefore, it became clear that the IBEE process allows us to create lowersubmicron patterns in the BSCCO films with little degradation of the film properties. It is extremely difficult to make such narrow superconducting bridges by other etching techniques.

#### 5. References

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