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Patterning of $Bi_2Sr_2CaCu_2O_{8+x}$ and $SrTiO_3$ films using lift-off technique combined with MOD

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Abstract

Micrometer size patterns of $Bi_2Sr_2CaCu_2O_{8+x}$ (BSCCO) and $SrTiO_3$ (STO) films were successfully prepared on STO substrates using lift-off technique combined with metal-organic decomposition (MOD) method without any vacuum processes. The minimum size of the pattern obtained was 3 μm for BSCCO and 5 μm for STO. The BSCCO film after patterning showed an X-ray diffraction with a *c*-axis orientation just like the film without patterning. On the other hand, the patterned STO showed an epitaxial relationship with the STO substrate. The BSCCO film with an anti-dot pattern of 3 μm and 20 μm in width showed T_c of 50 K and 83 K, respectively.

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Keywords: $Bi_2Sr_2CaCu_2O_{8+x}$; $SrTiO_3$; MOD method; Lift-off; Photoresist; Anti-dot

1. Introduction

Recently integrated electronic devices consisting of high-T_c superconductor (HTS) are attracting interest, which requires establishment of micro-patterning techniques. Conventionally, HTS and insulator thin films have been prepared using pulsed laser deposition, sputtering, molecular beam epitaxy (MBE) techniques. However, these techniques require expensive equipments, such as vacuum systems and high power lasers. On the contrary, metal-organic decomposition (MOD) method, which does not require any expensive equipment, is promising as one of the useful preparation techniques for HTS or other oxide materials. In our previous studies, we used MOD method to prepare high quality films of HTS. For example, we have succeeded in preparing $Bi_2Sr_2CaCu_2O_{8+x}$ (BSCCO) thin films with a critical temperature T_c of 84 K and a critical current density as large as 10^6 A/cm^2 at 15 K [1].

On the other hand, conventional patterning processes for micrometer dimension, such as reactive ion etching [2], laser-beam etching [3], and electron-beam lithography [4] also require expensive equipments. Wet chemical etching [5] is not suited for a micrometer size patterning of HTS films, since this technique often gives considerable damages on HTS films.

Lift-off technique is also popular in micrometer-size fabrications. However, this technique cannot be combined with epitaxial growth techniques such as the pulse-laser deposition, the sputtering method, and the MBE method, since the resist pattern used in the lift-off process does not endure during the deposition process with a high substrate temperature above 200 °C. Although there is a report on the use of Nb instead of the polymer as a resist for the lift-off, a degradation of superconductivity is reported to occur due to the diffusion of the niobium oxide into BSCCO films [6].

On the contrary, in the MOD method, the polymer resist can be used in the lift-off patterning, since the patterning of the precursor is possible in this technique. In the present study, we employed the lift-off technique combined with the MOD process as a low-cost and simple method to prepare micrometer-size patterns of BSCCO and $SrTiO_3$ (STO) films. In this work, anti-dot patterns were prepared to study

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resolution of the patterning process and it was also useful for measuring electrical properties of patterned BSCCO structures.

2. Experimental

Schematic drawings of our process are shown in Fig. 1. Square shaped dot-patterns of 3–20 μm in edge were prepared on STO (001) substrates using a photolithography technique with a photo resist (MICROPOSITS1808:SHIPLEY corp.). Thickness of the photo resist layer was approximately 500 nm. In the case of a small size pattern less than 5 μm in size, the photo resist layer was soaked in monochlorobenzene for 10 min and was rinsed with water prior to photolithography to form overhangs suitable for the lift-off [7] as shown in Fig. 1(a) and (b). The MOD solution of BSCCO, (BSCCO:SKBSCCO-008, Kojun-do chemical Lab.-Symetrix) or STO (STO:STO-06, Kojundo Chemical Lab.) were spin coated on the resist patterns with the rotation speed of 1000 rpm for 5 s and 3000 rpm for 60 s as shown in Fig. 1(c). The spin coated films were dried in an oven at 120 °C for 40 min. After drying process, the photo resist was removed with acetone (Fig. 1(d)). The patterned precursor of BSCCO was annealed at 870 °C in O_2 for 2 h and then annealed at 885 °C in $O_2 + N_2$ (10:90) atmosphere for 2 h. The patterned precursor of STO was annealed at 800 °C and then annealed at 1000 °C in O_2 for 2 h. Thickness of patterned film was approximately 50 nm for both BSCCO and STO.

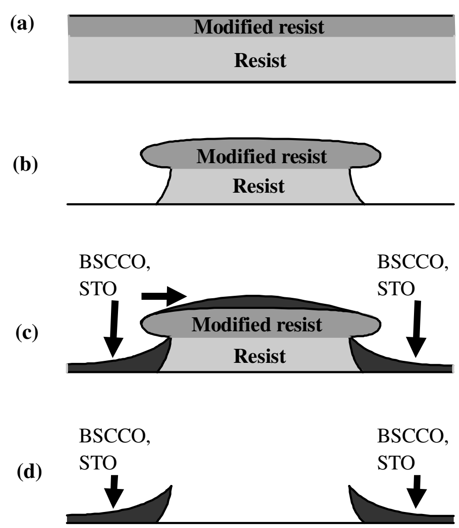

Fig. 1. Micro-patterning process used in this study: (a) soaking photoresist in monochlorobenzene, (b) photoresist with overhangs after development, (c) spin coat on photoresist pattern and (d) remove the photoresist with acetone.

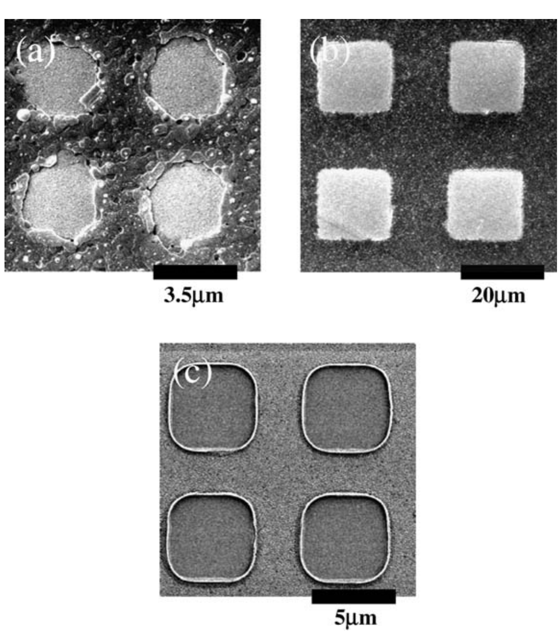

Fig. 2. SEM images of anti-dot patterned BSCCO with (a) 3 μm × 3 μm, (b) 20 μm × 20 μm, and (c) anti-dot patterned STO with 5 μm × 5 μm.

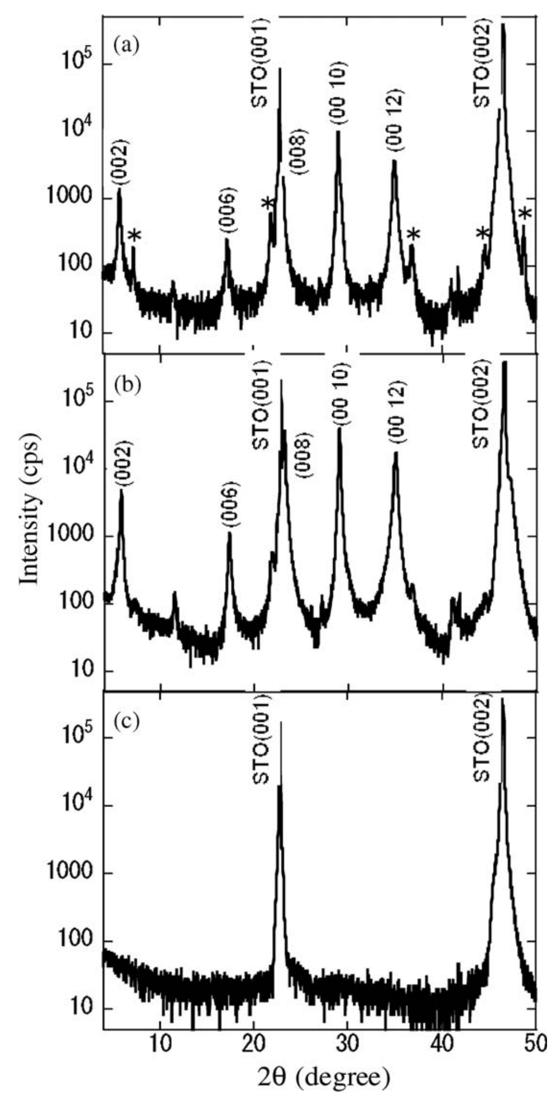

Fig. 3. XRD patterns of (a) anti-dot patterned BSCCO with (a) 3 μm × 3 μm, (b) 20 μm × 20 μm, and (c) anti-dot patterned STO with 5 μm × 5 μm. The asterisk marks indicate the 2201 phase.

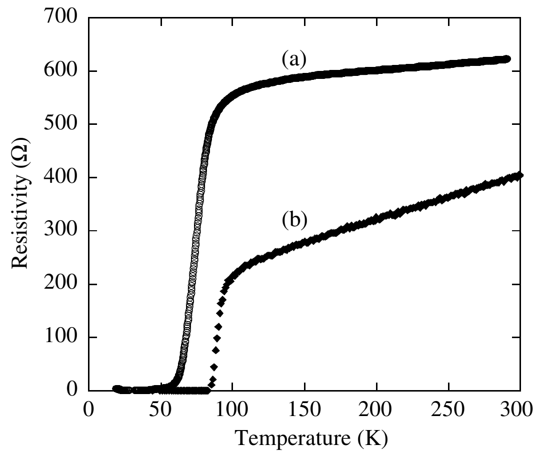

Fig. 4. *R*–*T* curves of (a) anti-dot patterned BSCCO with 3 μm × 3 μm, (b) anti-dot patterned BSCCO of 20 μm × 20 μm.

3. Results and discussion

Fig. 2 shows scanning electron microscope (SEM) images of the BSCCO anti-dot patterns of (a) 3 μm × 3 μm, (b) 20 μm × 20 μm squares and (c) the STO anti-dot pattern of 5 μm × 5 μm squares. Anti-dot patterns were prepared by the lift-off process using a photo mask of a dot pattern. In this way the BSCCO and STO patterns were successfully obtained. However, the BSCCO patterns of 3 μm in size failed to keep the original shape of the resist pattern, due to an occurrence of the lateral growth of BSCCO crystals of as small as 1 μm.

On the other hand, the BSCCO patterns of almost complete square shape with 20 μm and the STO patterns of almost complete square shape with 5 μm in edge were successfully prepared as shown in Fig. 2(b) and (c). Fig. 3 shows XRD patterns of the BSCCO anti-dot pattern of (a) 3 μm × 3 μm, (b) 20 μm × 20 μm, and (c) the STO anti-dot pattern of 5 μm × 5 μm. The patterned BSCCO films of (a) 3 μm × 3 μm and (b) 20 μm × 20 μm consisting of 2212 phase with a *c*-axis orientation were obtained, although small amount of 2201 phase was found. In the X-ray chart of patterned STO film in Fig. 3(b), only the peaks indexed to 001 are observed without any additional peaks. This result indicates that the patterned STO film is grown homoepitaxially on the STO substrate. Fig. 4 shows *R*–*T* curves of the BSCCO anti-dot patterns of (a) 3 μm × 3 μm and (b) 20 μm × 20 μm. The critical temperature T_c of the pattern of 20 μm × 20 μm is comparable to that of continuous BSCCO thin films, whereas the T_c of the pattern of 3 μm × 3 μm decreases to 50 K. We ascribe deterioration of the T_c to a mechanism of the crystallization of the patterned BSCCO. The diffusion length of the constituent atom is comparable to the pattern size. Therefore, in the case of 3 μm size, the chemical composition of the pattern becomes inhomogeneous, resulting in a segregation of additional phase such as 2201 phase as observed in Fig. 3(a). We also consider that the deterioration of the T_c is due to a contamination from the photo resist. In fact, 2201 phase with additional phases such as CuO, resulting lower the T_c of the sample, has been obtained, when the BSCCO precursor with a large quantity of residual photo resist was annealed.

4. Conclusions

Micrometer-size patterning of BSCCO and STO films was successfully demonstrated using the lift-off technique combined with the MOD method. The minimum size of the patterned films was 3 μm for BSCCO film and 5 μm for STO film. The critical temperature T_c of the BSCCO film with anti-dot patterns of 3 μm and 20 μm in width was 50 K and 83 K, respectively.

Acknowledgements

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