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ScienceDirect
Surface Science 600 (2006) 3673–3676
SURFACE SCIENCE
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The analysis of superconducting thin films modified by AFM lithography with a spectroscopic imaging technique

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Available online 21 April 2006

Abstract

A practical application of nanolithography using atomic force microscopy (AFM) was accomplished in fabricating superconducting flux flow transistors (SFFTs). It was found that it is essential to oxidize a superconducting thin film, grown on $LaAlO_3$ substrates by a thermal CVD process, by an applied bias voltage between a conducting AFM tip and the films, since I/V characteristics of the device were mainly controlled by the modified gate area in the SFFT. After AFM lithography, the critical current of an YBCO thin film was found to be degraded. Raman lines in the modified YBCO film were observed at 340, 502, and 632 cm^{-1} in Ar the laser system and 142, 225, and 585 cm^{-1} in the He–Ne laser system. Raman fluorescence images were also produced by mapping the Raman peaks. A strain image of the peak at 142 cm^{-1} was most clear, which means that a surface of the YBCO thin film was changed into the $YBa_2Cu_3O_6$ insulator. AFM nanolithography enables us to fabricate a channel between a source and a drain in SFFT in order to get I/V characteristics.

Keywords: Superconducting thin films; Raman spectroscopy; AFM lithography; Superconducting flux flow transistor (SFFT); Raman imaging

1. Introduction

Since the discovery of high temperature superconducting materials such as $YBa_2Cu_3O_x$, many electronic devices have been fabricated with the YBCO thin films. In order to visualize such superconducting electronic devices, it is necessary to develop suitable microfabrication techniques. Especially, etching and lithography for patterning are very important processes in the microfabrication techniques. The etchant such as HNO_3, and a mixture of Br and isopropanol are used for wet etching technique, while a focused ion beam and inductively coupled plasma (ICP) for a dry etching technique [1–6]. Recently, nanostructure fabrication processes using the scanning tunnelling microscope (STM) and the atomic force microscope (AFM) have advanced markedly, and are quite attractive for an application to electron and optical devices, because AFM can be used not only as a microscopic tool but also a fabricating tool. In spite of such merits of AFM lithography, its application to the electronic devices is not common since proper applications have not been found. We have fabricated a superconducting flux flow transistor (SFFT) by a lithograph technique and YBCO thin films. The basis of the SFFT is control of the Abrikosov vortex flowing along a channel composed of an array of weak links. In the fabrication of SFFT, one of the most important processes is to construct a reproducible weak link as a channel. Generally, the channel is constructed by etching a region between a source and a drain via chemical wet etching or dry etching to reduce the critical super current [7–10]. However, such conventional etching methods are not efficient in fabricating a weak link in nanometer size. We have been interested in the potential of nano lithography with AFM. In this report, we present an analysis of the superconducting thin film modified by AFM lithography with confocal micro-Raman spectroscopy which enables us to carry out

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doi:10.1016/j.susc.2006.02.060

microscopic and spectroscopic studies on a target area, simultaneously. Elucidation on the structural properties and composition of the modified surfaces is important for the development of cryoelectronic applications based on this material because the properties of the modified region affects the performance of the device. The analysis of $YBa_2Cu_3O_x$ using Raman spectroscopy has been presented in several publications as a non-destructive technique [11–14], while the analysis of the modified surface by the AFM lithography process has not been accomplished. Hence in order to observe the properties of the modified surface we used the imaging function of Raman spectroscopy.

2. Experiment and results

The YBCO superconducting film with 330 nm thickness was prepared by thermal CVD onto a $LaAlO_3$ substrate. The critical current and the critical temperature were 2 MA/cm^2 and 89.2 K, respectively. The YBCO film was lithographed by a 40×20 μm strip with AFM (Nanoscope IV, Veeco, USA). The applied bias voltage between the sample and an AFM tip was 11 V in the AFM lithograph. The AFM system was operated in the contact mode at ambient condition, a relative humidity of about 50%. Highly doped Si cantilevers with 30-nm-thick Ti coating were used as a conducting AFM tips (Mikromasch, Russia). After accomplishing the lithography, the sample was analyzed by Raman spectroscopy using 514 nm Ar and 633 nm He–Ne laser sources. Spectroscoping imaging was carried out with Nanofinder (Nanofinder 30, Tokyo Instruments, Japan).

Fig. 1 displays a topology image and a phase image of YBCO thin film. The roughness is approximately 45 nm. The protrusion is originated by a Cu-rich part in the picture so that YBCO thin films become rough.

Fig. 2 displays an AFM image of the modified surface (data scale, 200 nm) by AFM lithography with section analysis. The image (b) shows the position of the scan for a measurement of the morphology. The oxidization depends on the thickness of thin films, the applied bias voltage and the contact resistance between the sample and the sample holder [15]. In order to promote the oxidization, we kept a low contact resistance and high applied voltage. The modified region is very rough, but clearly distinguished from the matrix film. The thickness of the modified surface from the matrix was measured as an average value, and turned out to be about 148 nm. In comparison to other metal samples, YBCO thin films are well oxided. The increase in the thickness is mainly attributed to oxygen in the atmosphere.

Fig. 3 shows a variation of the critical current before and after AFM oxidization of the thin film. The critical current which is measured at the 1 μV was degraded from 90 to 58 mA, which means the properties of the YBCO thin film were changed by the AFM lithography.

We analyzed the modified surface by Raman spectroscopy using an Ar laser source of 514 nm wavelength.

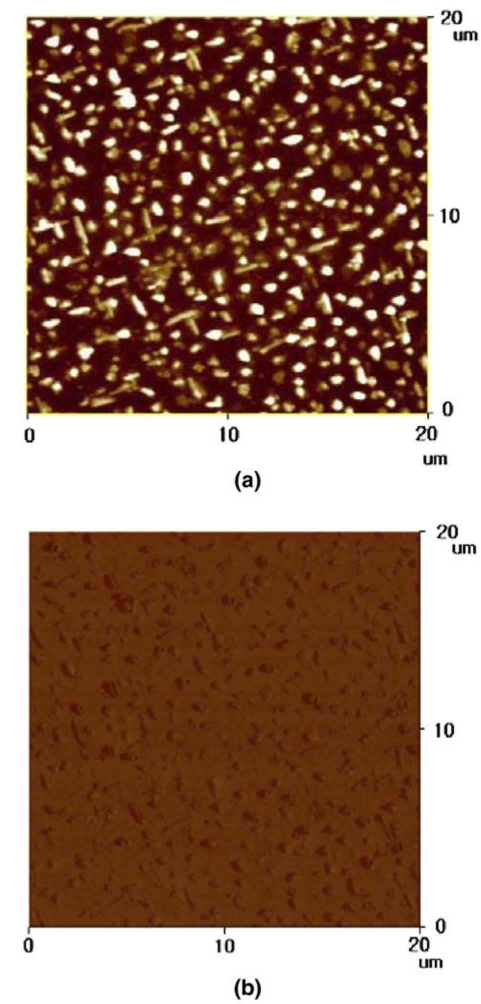

Fig. 1. Topology image and phase image of a YBCO thin film: (a) topology image, (b) phase image.

The Raman spectra of an original thin film and the modified surface were compared in Fig. 4. The main peaks were observed at 340, 502 and 632 cm^{-1}. The 502 cm^{-1} peak is very weak and 340 cm^{-1} peak is strong for the *c*-axis oriented orthorhombic YBCO thin film, while it is reversed for the *a*-axis oriented orthorhombic YBCO thin film [11]. Especially, we take note of the peak at 632 cm^{-1} which is related with the oxygen content in the composition of $YBa_2Cu_3O_{x-7}$. The intensity of the peak decreases, accompanied by small energy shift. It practically disappears in samples with $x = 6.4$, when the material becomes tetragonal and non-superconducting [14]. In our observation, the peak was split into two components, which means a modification of the YBCO thin film surface due to the strong electric field between the sample and AFM tip. The penetration depth of the laser for a YBCO thin film is longer than the modified depth of the thin film, so the 632 cm^{-1} peak is rather faint. The broad peak between 502 and 632 cm^{-1} is related with the related-BaCuO moiety [16]. It is considered that $YBa_2Cu_3O_x$ superconductor is dissociated into the related-BaCuO compound by AFM lithography.

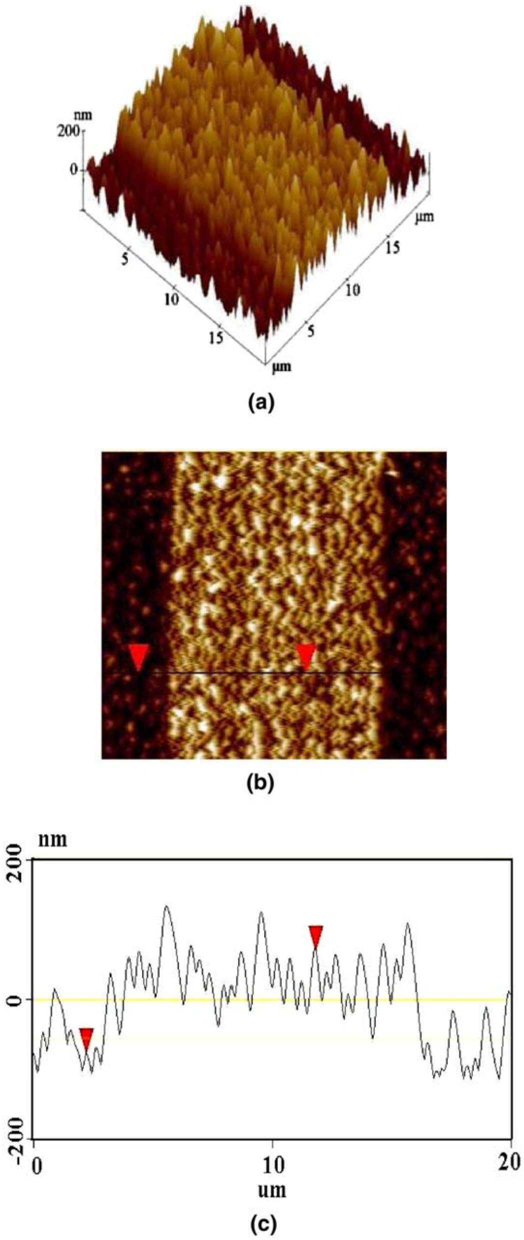

Fig. 2. The AFM image of modified surface by the AFM oxidization process at 11 V: (a) 3-D AFM topology image, (b) its 2-D image and (c) its section analysis.

Fig. 5 displays the Raman spectra of YBCO thin film lithographed at 11 V. The spectra were measured by a Nanofinder (in KBSI) using 633 nm laser. Peaks at 142, 225, and 585 cm^{-1} were observed from the sample. Although the peaks are different from the mentioned peaks in Fig. 4, they are known as Raman peaks of YBCO thin films. The 142 cm^{-1} peak corresponds to $YBa_2Cu_3O_6$, and those at and 225 and 585 cm^{-1} to inactive YBCO phonons that become active through loss of inversion symmetry or disorder. The peaks were captured to display the intensity image and the strain image as shown in Fig. 6. The images resulting from the difference of the intensity show identical images (Fig. 6a, c, and d), while the images

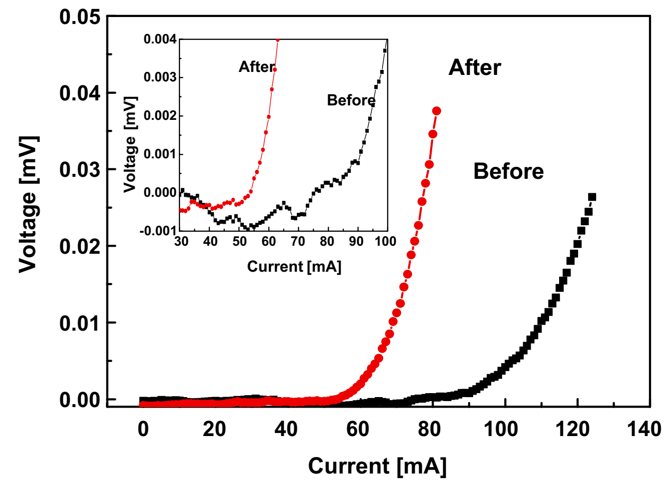

Fig. 3. Variation of the critical current after and before AFM lithography of a channel.

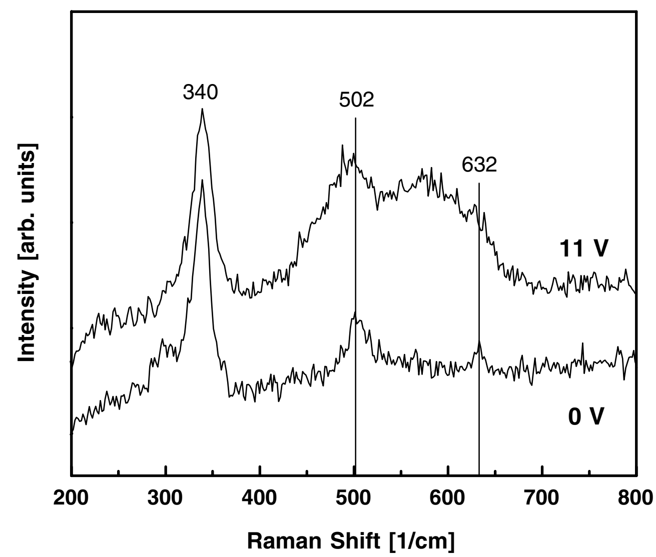

Fig. 4. Comparison of an anodized sample by AFM lithography and a raw sample.

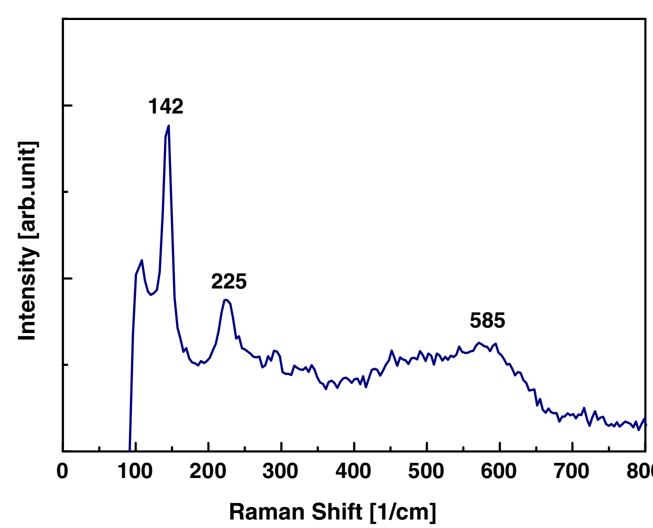

Fig. 5. A Raman spectrum of the YBCO thin film lithographed at 11 V.

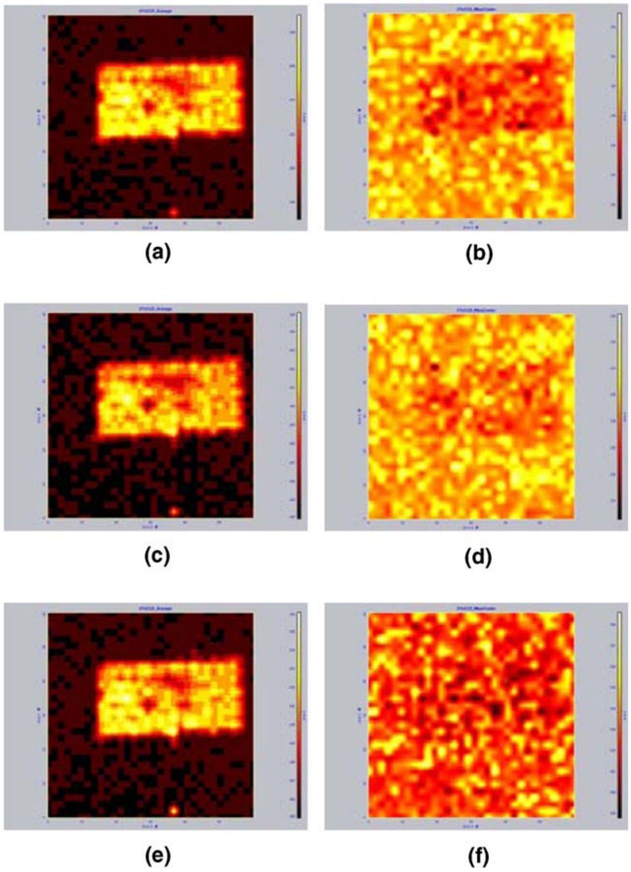

Fig. 6. Strain images and intensity images of the modified surface by AFM: (a) an intensity image, (b) a strain image for 142 cm^{-1}, (c) an intensity image, (d) a strain image for 225 cm^{-1}, (e) an intensity image and (f) a strain image for 585 cm^{-1}.

resulting from the difference of the strain are different. Especially, the strain image for the 142 cm^{-1} peak clearly shows the modified pattern. The 142 cm^{-1} peak is one of those Raman peaks observed in the insulator $YBa_2Cu_3O_6$, which means that the superconducting surface was modified into an insulator by the AFM oxidization process.

3. Summary

An YBCO superconducting thin film was modified by AFM lithography and was analyzed with Raman spectroscopy. After the lithographic process, the critical current was degraded from 90 to 58 mA and the related-BaCuO peak was observed. It is attributed to a modification of the composition in the YBCO thin film. The Raman spectra of the modified YBCO film were observed at 340, 502, and 632 cm^{-1} in Ar laser system and at 142, 225, and 585 cm^{-1} in the He–Ne laser system. Raman fluorescence images were also produced by mapping the Raman peaks. A strain image of 142 cm^{-1} peak was most clear, which means that a surface of the YBCO thin film was changed into the $YBa_2Cu_3O_6$ insulator. Accordingly, a channel which can control the I/V characteristics of SFFT can be fabricated at any place between the source and the drain.

Acknowledgements

This work was supported by the National R&D Project for NT-IT fusion strategy of Advanced Technologies and Nano Science and Technology (Contract No. M10214000110-02B1500-01800) of Ministry of Science and Technology, the Republic of Korea.

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