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Preparation of variable-thickness MgB_2 thin film bridges by AFM nanolithography

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Abstract

In this paper we focus our attention on preparation of superconducting MgB_2 thin films and variable-thickness MgB_2 thin film bridges using the Atomic Force Microscope nanolithographic technique. Microstructures and their following variable-thickness bridges were prepared on nonsuperconducting MgB thin films. Final structures were annealed in argon atmosphere at temperature 680 °C and exhibit transition to the superconducting state $T_{con} = 33$ K and zero critical temperature $T_{c0} = 30.5$ K. Critical current density j_c (4.2 K) measured on the bridge was higher than 10^6 A/cm^2.

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Keywords: MgB_2; Superconducting bridge; AFM nanolithography

1. Introduction

Since the discovery of superconductivity in MgB_2 considerable progress has been made in determining the physical properties of the material. Both for applications and basic studies, extensive effort to develop a viable weak link fabrication technology has been made worldwide. There have been a number of reports on fabrication of various types of MgB_2 weak links, including point-contact or break junctions [1], nanobridges, sandwich-type tunnel junctions [2], planar junctions by localized ion damage in thin films [3] and ramp-type junctions [4]. Sandwich-type tunnel junctions are very important from the viewpoint of the integration of superconducting circuits. Fabrication of sandwich-type all-MgB_2 Superconductor/Insulator/Superconductor (SIS) tunnel junctions (MgB_2/AlOx/MgB_2) using as-grown MgB_2 thin films formed by molecular-beam epitaxy has been reported [5].

Ion irradiation has the potential to be used as a means to modify superconducting properties as well as to create superconducting weak links. Kang et al. [6] reported successful creation of SNS type MgB_2 junctions by localized ion implantation. Fabrication of junctions without interfaces, i.e. weakened structures, by ion or electron irradiation is particularly attractive due to its controllability. Another group succeeded in fabrication of MgB2 superconductor–normal metal–superconductor (SNS) Josephson junctions using localized ion damage caused by FIB, and they created a single-layer thin film SQUID [7].

Nanolithography using scanning probe microscopes (SPM) offers powerful methods for patterning of surfaces with a resolution beyond the range of conventional lithographies based on resist exposures. Local probe techniques based on near field interactions show a greatly reduced proximity effect, which limits resolution in e-beam lithography [8].

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Resist-less AFM local oxidation of the surface of a material by a biased tip of an AFM is a versatile method for making nanoscale quantum devices. In order to fabricate the structure in a single step, the film thickness must be less than the typical depth of oxidized metal (10 nm), thus allowing direct writing of fully insulating regions. Such a process can be well controlled and is sufficiently reproducible to control the oxide line width to values defining either a complete electrical separation or, for a single line drawn at high speed and low voltage, a metal/insulator/metal tunnel barrier with low transparency [8,9].

Another new direct mechanical technique for fabrication of weak links on MgB_2 is AFM scratching. AFM enables direct machining of the sample surface by means of AFM cantilever tips. This can be achieved in two ways, called static plowing (scratching) and dynamic plowing (dynamic scratching). Irmer et al. [10] reported capability to prepare Josephson junctions by AFM nanoplowing. In the more common AFM scratching techniques, the tip is scanned under strong loading forces to remove the substrate or resist. This technique utilizes the principle of plowing in the same way as the traditional tool: the material is removed from the substrate in a well-defined way, leaving behind deep trenches with the characteristic shape of the plow used. The advantages of applying the nanoscratching for lithography are obviously the precision of alignment, the non-damaging definition process compared to electron- or ion-beam structuring techniques, and the absence of additional processing steps, such as etching the substrate [11,12].

In this paper, we report the preparation of superconducting MgB_2 variable-thickness bridge by AFM scratching lithography. Because MgB_2 thin film is very hard and it is not possible use this technique directly, the AFM scratching technique was applied on precursor MgB thin film microstrip. Final structure on MgB was annealed in argon at temperature 680 °C and became superconducting with onset critical temperature $T_{con} \leqslant 33$ K, zero critical temperature $T_{c0} \leqslant 30.5$ K and critical current density j_c (4.2 K) $> 10^6$ A/cm^2.

2. Experimental

Precursor MgB thin films were prepared by dc and rf magnetron sputtering of boron and magnesium from two independent magnetrons on unheared r-sapphire substrate. The preparation procedure of our MgB thin films is described in detail in [13].

Tescan – VEGA TS 5136 MM scanning electron microscope and Atomic Force Microscope type NT MDT SOLVER P47 were used for imaging the surface morphologies of the thin films and microstructures. For control of composition of MgB thin films the Auger electron spectroscopy was used. Resistance vs. temperature ($R(T)$) characteristics were measured by four-point method using computer-controlled current source. The bias current (typically 10 μA) was alternated in order to exclude parasitic thermo-voltage in potential contacts. Voltage vs. current ($V(I)$) characteristics were measured using standard dc four probe measurements in transport He Dewar container.

The microstrip, as a precursor for the preparation of variable-thickness bridge, was prepared on the MgB thin films by optical lithography. For direct optical lithography technique we used positive photoresist Microchemicals-AZ 6624. The resist coating was performed by spin-coating, which consists of dispensing the resist solution over the sample surface and rapid spinning of the sample until it becomes dry. Final spin speed was 4000 rpm for a duration of 20–30 s. The resist coating was followed by a soft baking for 25–30 min in an oven at a temperature ranging between 90 and 95 °C. Projection of the pattern image from a photo mask onto the sample surface was carried out using a Xe light source. Finally, the exposed photoresist was removed by developer Microchemicals-AZ 826 MIF. For patterning of the microstructures, the Ar^+ ion etching with energy of ions 500 eV or aqueous HF solution were used. The strips with thickness about 200 nm and width 5 μm were obtained after stripping of the photoresist. On such strips the AFM nanolithography was applied.

For nanolithography (scratching) we use a commercial atomic force microscope (NT MDT Solver P47). The maximum lateral scan range of this piezo tube is 50×50 μm^2, the maximum vertical range is 2 μm. For scratching we utilize special AFM tips with W_2C coating. For AFM dynamic scratching (nanolithography), it is necessary to know which force acts on the cantilever (amplitude setpoint). Here, the indentation force depends on many elements of uncertainty. When the AFM is operating in the tapping mode, the cantilever vibrates near its free resonance frequency (100–300 kHz). The distance between the cantilever and the sample depends on the setpoint amplitude. For simple imaging the setpoint amplitude is chosen to be 50% of the free oscillation amplitude and a feedback loop keeps the vibration amplitude constant. Scratching is achieved by increasing the free modulation amplitude and by reducing the setpoint amplitude by several percent of the free resonance amplitude. (The spring constant of the cantilevers used is 20–30 N/m.)

3. Results and discussion

The main idea was the preparation of the microstrips and variable-thickness bridge on the precursor MgB thin films, with subsequent annealing in order to obtain superconducting MgB_2 structures. We chose this type of technology because MgB_2 superconducting thin films are very hard. The surface of the MgB thin films was very smooth thus suitable for AFM nanotechnology. Using the optical lithography, three strips with area 15×200 μm^2, 10×200 μm^2, and 5×200 μm^2 were patterned on precursor MgB thin film, with subsequent etching by Ar^+ ion beam. Final MgB microstrips were obtained after removing the photoresist (see Fig. 1).

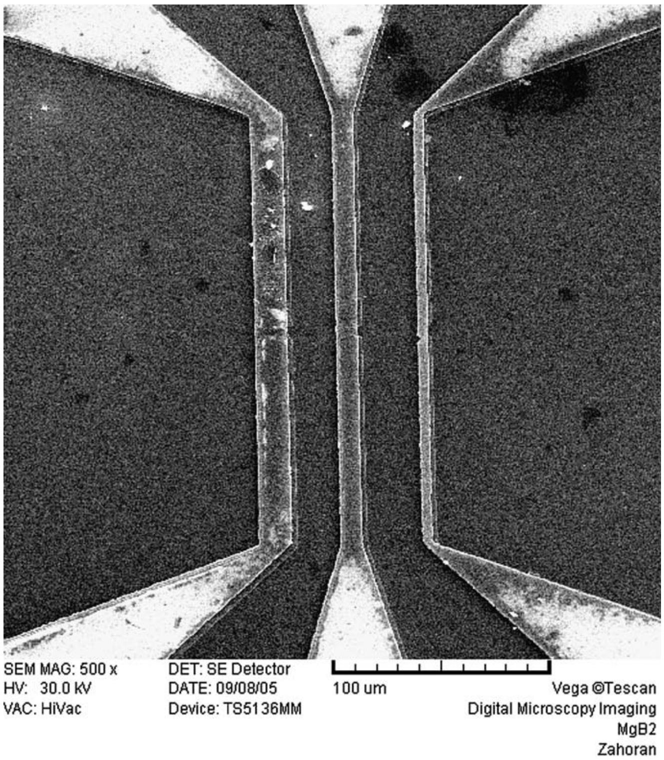

Fig. 1. The microstripes with width 15, 10 and 5 μm prepared on the MgB precursor thin film (scale bar from SEM is 100 μm). Scaling bar of SEM is 100 μm.

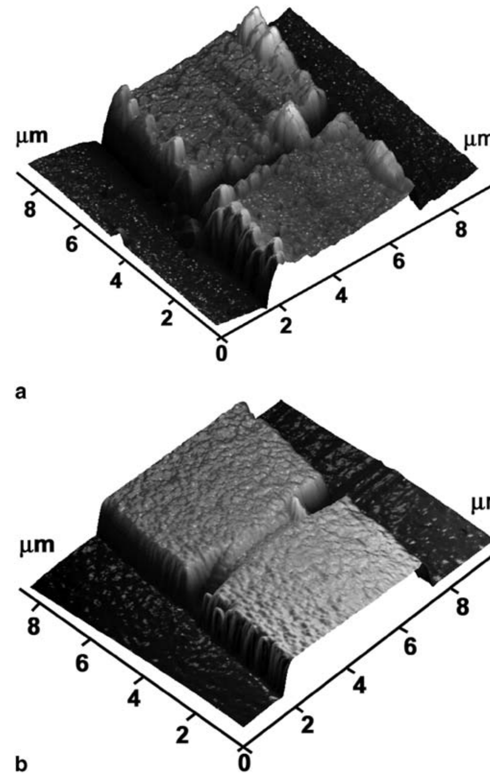

Fig. 2. AFM image of variable-thickness bridge prepared on unannealed MgB 5 μm width strip – as prepared (a) and after Ar^+ ion-beam etching (b).

The variable-thickness bridge was prepared on the 5 μm MgB strip using AFM nanolithography. The scratching of nanosized trenches through the central strips can be achieved in the contact mode, as well as in the tapping mode (dynamic scratching). Well-defined scratches were obtained, and protrusions along the sides of the grooves were observed exhibiting a surface deformation induced by mechanical treatment, and indicating the presence of stress. The width and the depth of the grooves depend on the applied parameters (loading force, number of cycles, scan velocity). Plowing across the central strips we produced small bridge with submicrometer dimension. Fig. 2 shows a variable-thickness bridge using AFM scratching after removing the photoresist. One can see some mountains on the strip border, probably due to existence of residual photoresist after ion-beam etching. The shape, size and the thickness were measured by AFM. The cross-section of the variable-thickness bridge from Fig. 2(a) is shown in Fig. 3(a) and (b). We applied additional Ar^+ ion-beam etching and removed about 100 nm of the prepared structures. The final thickness after the etching of the MgB strip was 100 nm and the thickness of the bridge was about 50 nm.

In next step the structures with variable-thickness bridge were annealed in argon atmosphere at 680 °C during 150 s. The four-point measurements show transition to the superconducting state with onset critical temperature $T_{con} = 33$ K and zero critical temperature $T_{c0} = 30.5$ K

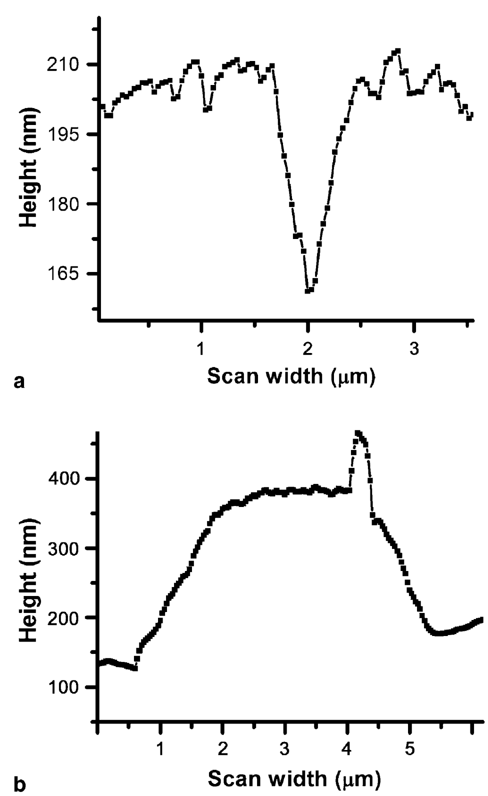

Fig. 3. Longitudinal (a) and cross (b) AFM scan across the bridge measured before Ar^+ etching.

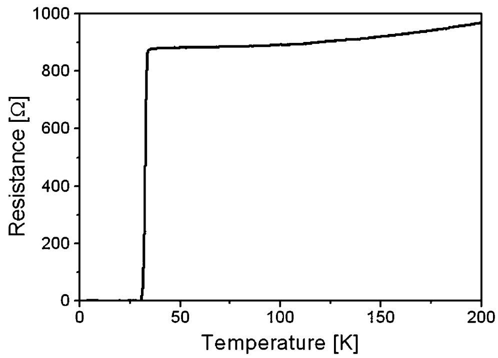

Fig. 4. $R(T)$ dependence of the 5 μm wide strip with variable-thickness bridge prepared by AFM nanolithography on MgB and consequently annealed at temperature 680 °C for 2.5 min.

(Fig. 4). On the strip with the variable-thickness bridge the $V(I)$ characteristics at various temperatures $T \leqslant T_{c0}$ (see Fig. 5 caption) were measured. The $V(I)$ characteristics exhibit critical current with a value higher than 5 mA at temperature $T = 4.2$ K which yields critical current density j_c (4.2 K) $\approx 2 \times 10^6$ A/cm^2. From the measured $V(I)$ characteristics at various temperatures the critical current density j_c vs. temperature T was reconstructed (Fig. 5(b)). The $V(I)$ characteristics exhibit hysteresis which should be caused by the intrinsic capacitance of the junction with a larger parameter β, typical for tunnel junctions. However, one can see quasi-linear dependence of j_c on T which is typical for SS'S bridges [14]. Also the very high critical current density j_c (4.2 K) $\approx 2 \times 10^6$ A/cm^2 suggests the existence of SS'S bridge, and the hysteresis could be explained by the creation of hot spots in the bridge and/or penetration of the vortices into the bridge.

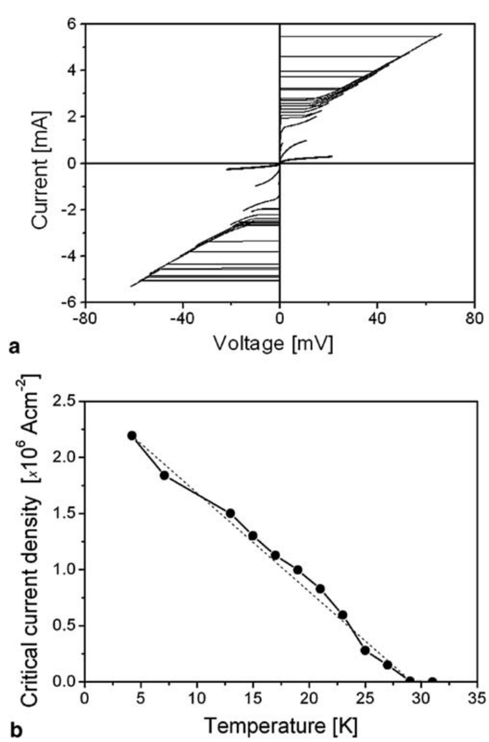

Fig. 5. V–I characteristics measured on variable-thickness bridge at temperatures 4.2, 7, 10.5, 13, 15, 17, 19, 21, 23, 25, 27, 29, and 31 K, respectively (a) and temperature dependence of the critical current I_c (b).

4. Conclusions

In conclusion, the superconducting MgB_2 variable-thickness bridge prepared by AFM lithography exhibits onset critical temperature $T_{con} \leqslant 33$ K, zero critical temperature $T_{c0} \leqslant 30.5$ K and critical current density j_c (4.2 K) $\approx 10^6$ A/cm^2. Our submicrometer structures do not exhibit Josephson junction behaviour, however, a way was shown how it is possible to do it. The main result of this paper is the presentation of a new method of preparation of superconducting MgB_2 submicrometer structures by AFM nanolithography. The main idea was the preparation of a variable-thickness bridge on a nonsuperconducting MgB thin film strip. It was shown that annealing of final nonsuperconducting MgB structures in argon atmosphere makes it superconducting with high critical temperature and critical current density. It is a promising result for the preparation of MgB_2 superconducting structures in nanometer scale, and Josephson weak links as well. Our work in this direction is in progress.

Acknowledgments

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