



MWA and scanning-probe study of argon-ion irradiation effects on superconducting properties of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ thin films

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ABSTRACT

$\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ films with thickness of 200 and 800 nm were irradiated with monovalent argon ions with an energy of 40 keV and a dose ranging between 10^{14} and 10^{17} ion/cm². The dose dependences of (i) the superconducting transition temperature, (ii) the critical current density value and (iii) the irreversibility line position on the magnetic field-temperature phase diagram were determined for two series of samples of different thickness. Atomic force microscopy images of the irradiated samples showed an appearance of defects in the form of surface holes. The obtained data were used to establish conditions for improving properties of thin-film superconducting materials. Firstly, the irradiation dose should be at least 10^{16} ion/cm² to form embedded gas bubbles and surface holes serving as artificial pinning centers. Secondly, the film thickness and the average depth of the defect formation should be of a comparable value and sufficiently exceed the thickness of the surface layer sputtered as a result of irradiation.

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1. Introduction

Preparation of superconducting materials with high critical current density requires development of methods for creation of artificial defects. The problem of inhibiting the magnetic vortex motion remains unsolved for superconductors of a nanometer size. A traditional method for formation of pinning centers by irradiation of a bulk superconductor with high-energy (about 1 GeV) ion beams [1] appears to be destructive for superconducting films with thickness of several hundred nanometers. As a result of such an impact the crystal structure of a thin-film superconductor becomes sufficiently degraded and its critical parameters are noticeably reduced [2,3]. Ion irradiation of significantly lower energy (tens and hundreds kiloelectron-volt) is not so destructive but it does not always lead to the formation of effective pinning centers (see, e.g., [4]). A search for optimal regimes of low-energy irradiation that would induce radiation defects, strongly pinning the vortex filaments, requires much scientific effort.

In the previous paper [5] we have demonstrated an efficiency of irradiation of high-temperature superconductor (HTSC) thin films with monovalent transition-metal ions with an energy of 40 keV. Iron-ion irradiation with doses ranging between 5×10^{11} and 3×10^{13} ion/cm² produces defects in the form of separate amorphous regions. The increased critical current density and the ex-

panded area of non-dissipative current flow with almost unchanged temperature of the superconducting transition indicate the efficiency of vortex pinning on the defects induced by irradiation with doses below 10^{12} ion/cm².

The goal of the present work is to create artificial defects of another type, in particular, in the form of embedded gas bubbles or blisters, and to study the efficiency of vortex pinning on such defects. To form blisters a target material should be irradiated with sufficiently high doses of inert-gas ions ($\sim 10^{17}$ ion/cm²) [6]. Attempts to modify HTSC materials with beams of inert-gas ions have been made previously by other research groups but, in most cases, they have been unsuccessful. There are reports in literature on the decreasing critical temperature after irradiation of cuprate HTSC with high doses of helium ions with an energy ranging between several hundred kiloelectron-volt and several tens megaelectron-volt [4,7]. We have taken these data into account when choosing ion energies and irradiation doses in order to improve the superconducting properties.

The present paper reports on process of the radiation defect formation revealed for the $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ thin films with thickness of 200 and 800 nm upon increasing the irradiation dose of 40 keV Ar^+ ions from 10^{14} to 10^{17} ion/cm². The irradiation effect on the surface structure and critical parameters of the samples has been estimated with the help of scanning-probe and radiospectroscopic measurements. The factors both contributing to and weakening the magnetic flux pinning on radiation-induced defects have been studied.

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2. Experimental

The $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ thin films were prepared by magnetron sputtering of the initial compound on a single-crystal substrate LaAlO_3 with subsequent annealing in the oxygen atmosphere at 820°C . All procedures of the film synthesis were carried out at the Institute of Physics, Gutenberg University (Mainz, Germany). There were two series of films with thickness of 200 and 800 nm, respectively. The samples with dimensions of about $1.5 \times 2 \text{ mm}^2$ were irradiated at room temperature with 40 keV monovalent argon ions in the ion-beam accelerator ILU-3 at the Zavoisky Physical-Technical Institute (Kazan, Russia). The ion current density was kept below $5 \mu\text{A}/\text{cm}^2$ to avoid the sample heating during irradiation. The argon irradiation dose was set sufficiently high in order to create blisters and varied in a wide range of $10^{14} \div 10^{17} \text{ ion}/\text{cm}^2$ for the 200 nm films and $10^{15} \div 10^{17} \text{ ion}/\text{cm}^2$ for the 800 nm films.

We have studied the irradiation effect on such superconducting parameters of the films as the critical temperature, critical current density j_c and irreversibility line (IL, which is a boundary between the regions with zero and non-zero critical current density). A special nature of the objects under study, which are the type-II superconductor films with a vortex system, has an important bearing on the research methods applied. These methods include measurements of AC-susceptibility and microwave absorption (MWA), and registration of magnetic flux profiles by a scanning Hall probe.

The MWA method is based on the dissipation of microwave power in superconducting samples by vibrating vortices. The MWA amplitude is proportional to the number of vortices in the sample and their gradient, i.e., to the critical current density (for details see [8]). In a system with pinning, when j_c is not equal to zero, MWA exhibits hysteresis upon changing the direction of the magnetic field sweep. The hysteresis vanishing at a certain value of magnetic field and temperature means the sample transition to the reversible area, where j_c is equal to zero. This effect has been used for determining points of the IL. MWA measurements were carried out on an X-band “Bruker BER-418S” spectrometer using a helium-flow cryostat for obtaining temperatures in the range from 20 K to T_c . A sample was placed in a cavity, with the film plane being perpendicular to the applied magnetic field, and cooled down to the temperature of measurements in a zero magnetic field. The MWA hysteresis loop was registered upon sweeping the magnetic field up to 8 kOe and down. IL points were determined as values of the magnetic field where the MWA hysteresis vanishes.

The critical current density was estimated from the full-penetration field using a theoretical model of Brandt [9]. To find the full-penetration field an evolution of the shape of the magnetic flux profile was monitored upon decreasing the external magnetic field. The magnetic flux profile was registered by a miniature scanning Hall sensor with dimensions of an active zone of $0.1 \times 0.05 \text{ mm}^2$.

The superconducting transition temperature T_c was determined from the temperature dependence of AC-susceptibility at a frequency of 20 MHz. The technique is based on measuring the phase shift of the detected signal which rises as the natural frequency of the oscillatory circuit with a superconducting sample in a coil changes below the critical temperature. The experimental setup is tuned in such a way that only the real (in-phase) part of the complex AC-susceptibility is registered.

Atomic force microscopy (AFM) was used to control the surface state before and after irradiation. The structural features and distribution of implanted defects were visualized by means of AFM microscope “Solver P-47”. The sample surface was studied in a tapping mode of the microscope operation.

3. Results and discussion

Critical parameters of superconducting samples were measured before and after irradiation with argon ions by a set of methods described above. A comparative analysis of the obtained data allowed us to draw conclusions on the efficiency of irradiation with various doses of 40 keV argon ions. As has been mentioned above, the experiments were carried out on the films with different thickness of 200 and 800 nm. Such an approach allows us to consider the influence of the film thickness, as well.

3.1. Defect formation and surface modification

The irradiation impact on the film surface structure was controlled by means of AFM. Fig. 1 shows two-dimensional AFM images of the surface of the 200 nm films irradiated with different doses of argon ions. The film irradiated with the lowest dose under study ($10^{14} \text{ ion}/\text{cm}^2$) has a rather smooth surface with an exception of several inclusions (white dots in Fig. 1a), which may be attributed to external contaminations (e.g., particles of dust), and accidental scratches by scanning probes (dark lines in Fig. 1a). One can see that irradiation with the dose of $10^{14} \text{ ion}/\text{cm}^2$ had no significant impact on the film surface. The dose increase by one order ($10^{15} \text{ ion}/\text{cm}^2$) leads to the formation of a few defects in the form of surface holes (black dots in Fig. 1b) with lateral dimensions of about $0.2 \mu\text{m}$. Besides the presence of separate holes, the surface layer seems to be flaking off as a result of irradiation. With further dose increase ($10^{16} \text{ ion}/\text{cm}^2$) the number of surface holes per area unit (black dots in Fig. 1c) grows. By rough estimates, the depth of these holes exceeds 15 nm. The maximum amount of artificial defects is observed for the highest irradiation dose under study ($10^{17} \text{ ion}/\text{cm}^2$, Fig. 1d). At this dose the film surface is rather deteriorated and surface holes overlap with each other. Quantitative estimates of the changes in the surface state as a function of dose are summarized in Table 1. The film roughness has been calculated as a mean-square deviation in height, while the hole concentration has been estimated by counting the defects in AFM images. As can be seen in Fig. 1 and Table 1, the surface becomes more developed with the increasing dose and the concentration of surface holes grows proportionally to the irradiation dose.

The nature of the surface holes formation can be explained as follows. Upon irradiation bombarding ions of argon penetrate into the target material at a certain depth and diffuse to form gas bubbles. Besides the blistering process, the film is subjected to sputtering and flaking. As the sputtering process reaches the depth of the gas bubble formation, the bubbles come to the surface, the inner argon gas evaporates and only the cavities remain. One may assume that the observed surface holes are the cavities of argon bubbles broken and emptied as a result of the film sputtering.

Besides tracking the surface evolution with increasing dose, AFM method was also used to control the film thickness. After irradiation with different doses of argon ions the samples were scratched and the height of the formed step was measured. These measurements revealed a tendency to the film thinning with increasing irradiation dose as a result of the film sputtering by argon ions. The film irradiated with the maximum dose of $10^{17} \text{ ion}/\text{cm}^2$ is three times thinner than the as-prepared film. The film thinning is a negative but unavoidable effect of irradiation.

3.2. Superconducting transition

Fig. 2 shows superconducting transitions for the 200 nm films determined from the temperature dependence of AC-susceptibility. An average critical temperature T_c of as-prepared samples is equal to $90 \pm 0.5 \text{ K}$. Dependences of the critical temperature and

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