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Effect of ion irradiation and stabilizing annealing on critical currents of superconducting NbN thin film

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The effect of composite ion irradiation on the critical currents of superconductive niobium nitride NbN thin-film is considered. The I–V curve of $20 \times 20\,\mu$mm samples with a thickness of 5.5 nm were obtained after irradiation with a composite ion beam (consisting of protons with 1% of oxygen) with an energy of 1 keV to various fluencies. The effect of irradiation and stabilizing annealing at 200°C for 1 hour on the critical currents of the superconducting transition is estimated.

Keywords: Niobium nitride, ion irradiation, superconductors, thin films.

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Classical superconducting electronic devices, which offer low energy consumption levels and potentially high clock speeds, appear to be promising alternatives to semiconductor processors. For example, advanced superconductor rapid single-flux-quantum (RSFQ) logic devices have switching times on the order of ~ 1 ps and switching energies of a single element $< 1 \cdot 10^{-19}$ J [1].

Hardware components for classical superconducting electronic non-RSFQ logic devices are being designed at the National Research Center „Kurchatov Institute"The key logic elements for implementation of classical computing have already been constructed. Their operation relies on transitions of sections of superconducting nanowires into the normal state under the influence of heating by a resistive section of a different nanowire positioned in a neighboring layer, which is separated by a thin spacer dielectric layer [2]. Superconducting nanowires with integrated resistances. which are formed using the technique of adjustment of the chemical composition and properties of thin-film materials under irradiation with a composite ion beam at an ion energy of 1 keV [3], are the key elements of such devices. The irradiation regimes used in this case differ substantially in both energy and type of ions from those applied in earlier studies carried out by other research groups. For example, a niobium wire $30\,\mu$m in width and 20 nm in thickness was irradiated in [4] with Fe ions with energies of 22, 45, and 65 keV to fluences of $10^{15}-10^{16}$ ion/cm^2 in order to examine the Josephson effect induced by the formation of weak bonds due to implantation of impurity magnetic atoms into a film. The authors of [5] have irradiated a Ta film 46 nm in thickness with Cu or Fe ions with an energy of 60 keV within the $10^{15}-3 \cdot 10^{16}$ ion/cm^2 fluence range to study the emerging proximity effect and compare the results with other methods used to induce this effect (anodic treatment and deposition of metallic coatings).

In the present study, thin-film niobium nitride (NbN), which is used in single-photon detectors [6], serves as the main superconducting material. This material was produced by room-temperature ion sputtering and was chosen for its stability, processibility, and a high superconducting transition temperature T_c (up to 10.5 K for a 5-nm-thick film [7,8]).

The irradiation technique for fabrication of integrated resistances is specific in that the electric parameters of resistors reach saturation within a certain time interval after irradiation. Stabilizing annealing at a temperature of 200°C was performed for 1 h to speed up this process. The annealing temperature was chosen on account of the technological features of application of the electron resist (polymethyl methacrylate) in lithography and was targeted at the fabrication of multilayer devices.

In addition to forming integrated resistances in the produced cryogenic logic devices, one needs to reduce controllably the critical transition current within designated sections of superconducting nanowires, since this provides an opportunity to simplify the circuit design of elements operating via transitions of superconducting sections into the normal state (and vice versa). Ion irradiation is also used for this purpose. Thus, the present study is focused on examining the effect of irradiation and subsequent annealing on critical currents of film transition from the superconducting state to the normal one (and vice versa).

Square-shaped samples $20 \times 20\,\mu$m in size were used to measure the properties of thin (5.5 nm) NbN films. Macroscopic contacts for measurements were formed by depositing platinum (with a nickel underlayer) on top of the samples.

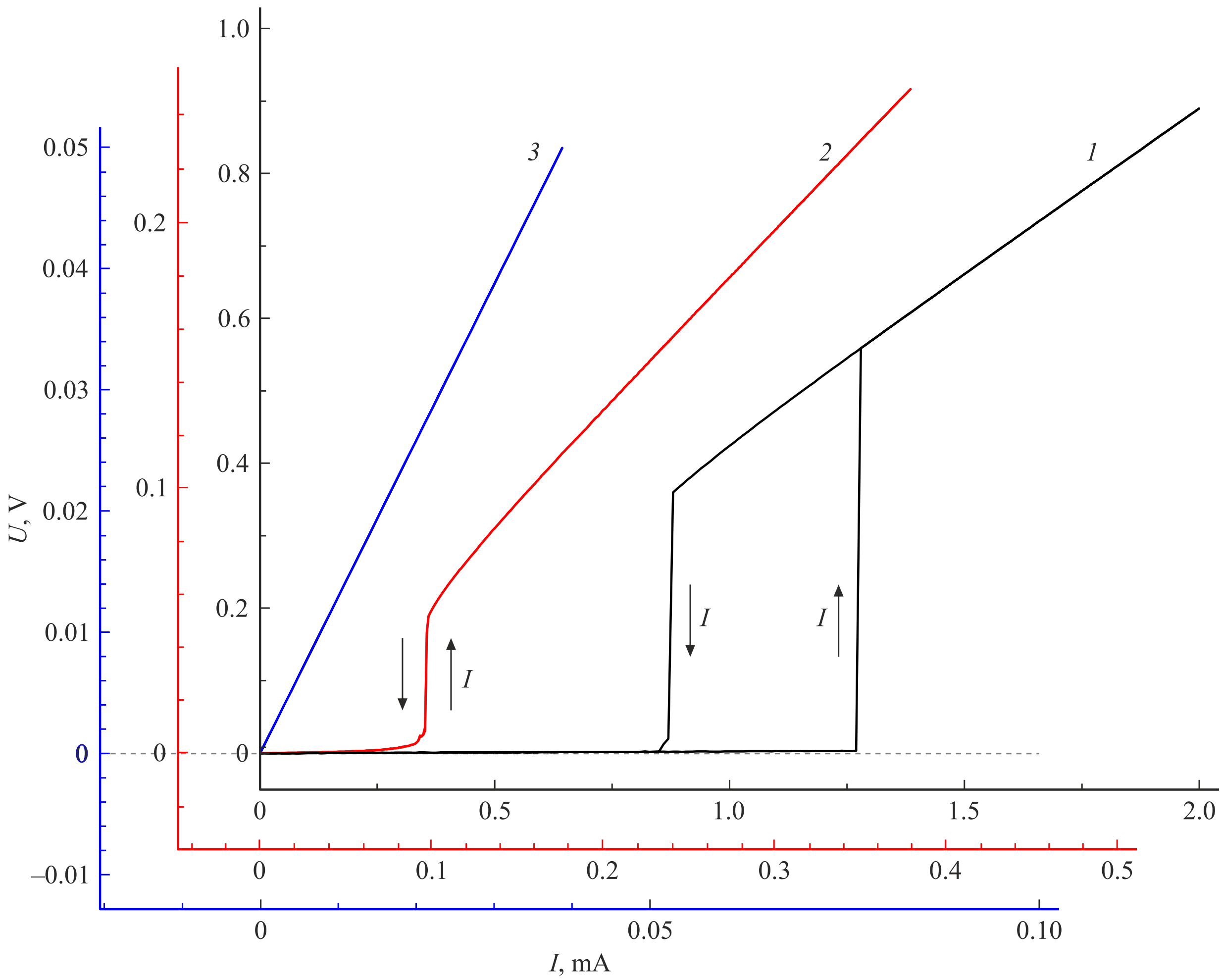

Figure 1. I–V curves of microbridges constructed from an NbN film with a thickness of 5.5 nm on a sapphire substrate. *1* — non-irradiated sample, *2* — sample irradiated with a composite proton–oxygen beam to fluence $\Phi = 1.06 \cdot 10^{20}\,\mathrm{m}^{-2}$, and *3* — sample irradiated to fluence $\Phi = 5.31 \cdot 10^{20}\,\mathrm{m}^{-2}$. A color version of the figure is provided in the online version of the paper.

Ion irradiation was performed using a setup with a high-frequency plasma source at ion current density $j = 0.849\,\mathrm{A/m^2}$. The ion energy was 1 keV (the beam consisted of protons with $\sim 1\%$ oxygen).

A current-voltage (I–V) curve was recorded at a temperature of 4.2 K for each sample after irradiation. A sample was secured with pressure brass contacts to the measurement stand and introduced into a vessel filled with liquid helium. An I–V curve was recorded with a KEITHLEY 4200-SCS oscilloscope. Following these measurements, the sample was subjected to stabilizing annealing, and the measurement procedure was repeated.

Figure 1 presents the I–V curves of the initial sample and two samples irradiated with a composite ion beam to different doses.

It can be seen (curve *1*) that the initial sample features a current hysteresis loop in transition from the superconducting state to the normal one and vice versa. As the fluence increases, the critical currents and the difference between forward and backward transition currents decrease, but hysteresis is retained at a fluence below $(0.9{-}1.0) \cdot 10^{20}\,\mathrm{m}^{-2}$. The hysteresis loop vanishes within the $(1.0{-}1.6) \cdot 10^{20}\,\mathrm{m}^{-2}$ fluence range; i.e., forward and backward transitions occur at the same current (curve *2* in Fig. 1). When the radiation fluence rises above $1.6 \cdot 10^{20}\,\mathrm{m}^{-2}$, the superconducting transition region expands significantly in the current domain (the I–V curve loses verticality in the transition region), and a further dose increase up to a fluence of $\sim 5 \cdot 10^{20}\,\mathrm{m}^{-2}$ renders the film metallic at 4.2 K, making its I–V curve linear (curve *3* in Fig. 1). Since a strongly diffuse transition makes it hard to determine the critical current accurately, radiation fluences ranging from 0 to $1.6 \cdot 10^{20}\,\mathrm{m}^{-2}$ were used in the present study.

Figure 2, *a* presents the variation of currents of forward and backward transitions with fluence after irradiation, while Fig. 2, *b* shows similar dependences measured after irradiation and stabilizing annealing (200°C).

The relative difference between currents before and after annealing was examined as a measure of the effect of

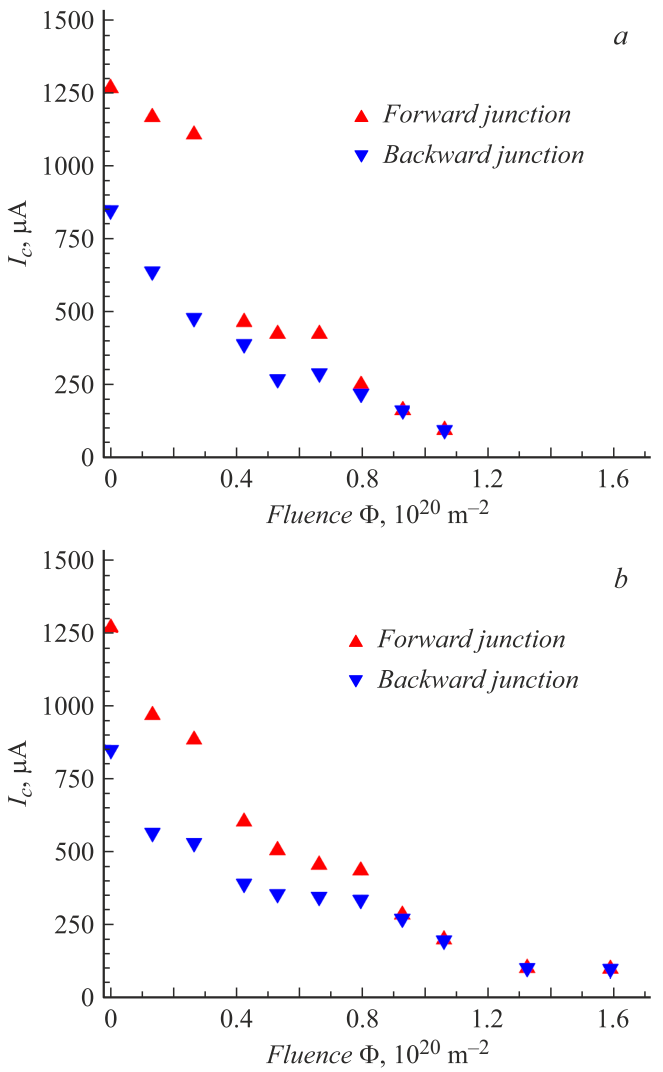

Figure 2. Dependences of critical currents of forward and backward transitions on the fluence of composite ion radiation. *a* — After irradiation, *b* — after irradiation and stabilizing annealing.

annealing on the critical current:

$$\delta_I = \frac{I_{ann} - I_{irr}}{I_{irr}} \cdot 100\%, \quad (1)$$

where I_{irr} is the critical current for an irradiated non-annealed sample and I_{ann} is the critical current for the same sample after annealing. If $\delta_I > 0$, annealing has restored partially the superconducting properties that deteriorated under irradiation. If $\delta_I < 0$, annealing has contributed to the deterioration of superconducting properties. Diagrams of δ_I variation with irradiation fluence for forward and backward currents were plotted based on the I–V curves (Fig. 3).

Error bars in Fig. 3 represent the spread of film thickness in different samples ($\Delta\delta_I = 8\%$) and the scatter of data measured for different samples under equal irradiation fluences, which is especially prominent in the low-fluence region ($\Phi < 0.53 \cdot 10^{20}\,\text{m}^{-2}$).

It follows from Fig. 3 that the superconducting properties of NbN films irradiated with high fluences ($\Phi > 0.53 \cdot 10^{20}\,\text{m}^{-2}$) were restored partially after annealing. Specifically, the critical currents of forward and backward transitions corresponding to a fluence of $0.53 \cdot 10^{20}\,\text{m}^{-2}$ rose by 18 and 32%, respectively, after annealing. At a fluence of $1.06 \cdot 10^{20}\,\text{m}^{-2}$, the critical currents increased by a factor of more than 2 ($\delta_I > 100\%$). Thus, the contribution of annealing to the partial restoration of superconducting properties becomes more pronounced as the irradiation fluence increases.

In the low-fluence region, annealing has almost no effect on the backward transition current (Fig. 3, *b*) and reduces slightly the critical current of the forward transition (Fig. 3, *a*). Although the points of relative variation of the forward transition current after annealing in the low-fluence region remain within the measurement accuracy from $\delta_I = 0$, the mean values are negative (Fig. 3, *a*).

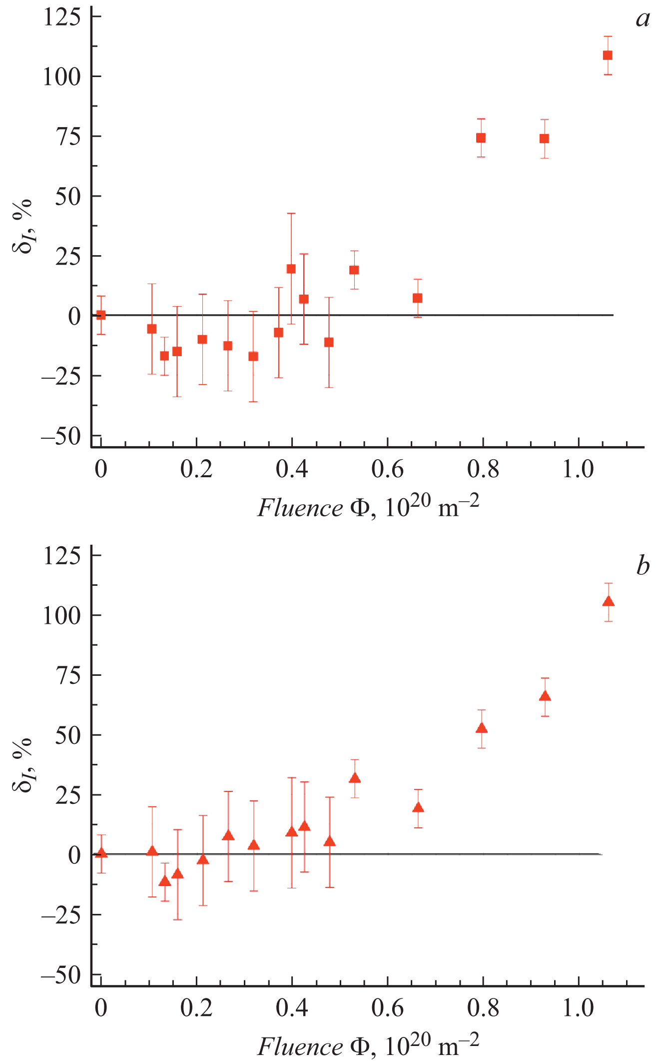

Figure 3. Relative differences between critical transition currents before and after annealing at different fluences of composite ion radiation. *a* — Forward transition, *b* — backward transition.

It has been demonstrated in our earlier studies that composite ion irradiation induces the process of selective substitution of nitrogen atoms by oxygen atoms in NbN. Approximately one half of nitrogen atoms get substituted at fluences of $\sim 10 \cdot 10^{20}\,\mathrm{m}^{-2}$, and crystalline phases close in their composition to NbNO form [9]; however, radiation defects, which may affect the critical currents of a superconductor if their density is sufficiently high, are also produced in the process. Annealing reduces the number of defects and facilitates additional substitution of atoms at sites that were left unperturbed in the course of irradiation. Therefore, post-irradiation annealing enhances the critical currents at fluences $\Phi > 0.53 \cdot 10^{20}\,\mathrm{m}^{-2}$.

Annealing performed after irradiation with low fluences $\Phi < 0.53 \cdot 10^{20}\,\mathrm{m}^{-2}$ exerts a less significant influence on the critical currents of the forward transition. The current was measured reliably to decrease after annealing in certain samples, but tests carried out for a large number of samples with varying thickness values do not presently reveal any statistically significant effect. Additional experiments are needed to construct a model of the influence of annealing in this fluence range (with especially detailed coverage in the region from 0 to $0.32 \cdot 10^{20}\,\mathrm{m}^{-2}$).

Thus, it was demonstrated that ion irradiation with subsequent stabilizing annealing may be used to reduce controllably the critical currents of a thin NbN film. The influences of annealing on the critical currents of a film irradiated to a fluence lower than $0.53 \cdot 10^{20}\,\mathrm{m}^{-2}$ and to a higher fluence were found to be opposite.

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Conflict of interest

The authors declare that they have no conflict of interest.

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