

Local insulator-to-superconductor transition in amorphous InO_x films modulated by e-beam irradiation

I. F. Llovo^{1,2,3,*} and J. Delahaye^{4,†}

¹*Galicia Supercomputing Center (CESGA), 15705 Santiago de Compostela, Spain*

²*QMatterPhotonics, Departamento de Física de Partículas,*

Universidade de Santiago de Compostela, 15782 Santiago de Compostela, Spain

³*iMATUS, Universidade de Santiago de Compostela, 15782 Santiago de Compostela, Spain*

⁴*Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, 38000 Grenoble, France*

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We present a novel method enabling precise post-fabrication modulation of the electrical resistance in micrometer-scale regions of amorphous indium oxide (a-InO_x) films. By subjecting initially insulating films to an electron beam at room temperature, we demonstrate that the exposed region of the films becomes superconducting. The resultant superconducting transition temperature (T_c) is adjustable up to 2.8 K by changing the electron dose and accelerating voltage. This technique offers a compelling alternative to traditional a-InO_x annealing methods for both fundamental investigations and practical applications. Moreover, it empowers independent adjustment of electrical properties across initially identical a-InO_x samples on the same substrate, facilitating the creation of superconducting microstructures with precise T_c control at the micrometer scale. The observed resistance modifications likely stem from photoreduction induced by X-ray and/or UV radiation emitted during electron beam interactions with the film and substrate.

I. INTRODUCTION

Disordered amorphous indium oxide (a-InO_x) films have long served as an experimental platform for studying the electrical properties of disordered systems. During the 1980s, many theoretical predictions related to the Mott-Anderson metal-insulator transition were verified in this system [1–3] and in the early 1990s, two new topics rose from disordered indium oxide films measurements. The first topic was the search for an electron glass [4–6], with the reports of slow and glassy relaxations of the conductance in field effect experiments on insulating films [7–9]. The second topic was the study of the superconducting-insulator transition (SIT) in amorphous films as a function of magnetic field [10–12] and disorder [13]. The nature of this transition, especially the insulating state terminating superconductivity, remain the subject of heated experimental and theoretical debates [14, 15]. The spectacular properties of this insulating state may indeed reflect the existence of a many body localized phase [16], a topical and striking issue in condensed matter physics [17–21].

From an applications perspective, a-InO_x films are also a promising system for the fabrication of high kinetic inductances. Such electrical circuit elements can be obtained from superconducting a-InO_x films approaching the SIT, in the form of long wires of a few micrometers wide [22]. They enable the creation of low-loss and small size microwave resonators, which are crucial for the development of quantum circuits [15, 22] as well as sensitive photon detectors [23]. Moreover, doped a-InO_x films have found extensive use in the production

of semiconducting microdevices such as thin-film transistors (TFT). Remarkably, oxygen-vacancies in a-InO_x exhibit electronic properties equivalent to those of tin-doped amorphous indium-tin oxide (a-ITO) [24]. Consequently, fields already benefiting from the commercial use of a-ITO (e.g., TFT and flat-panel display research) could potentially leverage the control of the electronic properties of a-InO_x .

Therefore, the prospects for fundamental studies and applications of a-InO_x rely on the ability to control and adjust the electrical properties of disordered indium oxide films, which exhibit significant variability across the SIT. Typically, these films are prepared by electron beam (e-beam) evaporation of pure In_2O_3 under a partial pressure of oxygen [3]. Microcrystalline films result from evaporation at substrate temperatures exceeding 150°C , while substrate temperatures below 40°C yield amorphous films [3]. Common methods for adjusting the electrical properties of the resulting films include adjusting the oxygen pressure, the evaporation rate or the film thickness during the evaporation process, and employing post-evaporation annealing [13, 25]. In the case of amorphous films, annealing steps restricted to $40\text{--}60^\circ\text{C}$ lead to irreversible reductions in electrical resistance without inducing crystallization [3, 13].

In this article, we introduce a novel technique for modulating the electrical properties of a-InO_x films in relation to the SIT. We demonstrate that upon exposure to the e-beam of a scanning electron microscope (SEM), an initially insulating film undergoes a local transformation into the superconducting phase across the SIT, featuring a superconducting transition temperature of up to 2.8 K. Moreover, we demonstrate the capability of controlled e-beam exposure to locally manipulate the SIT, achieving a lateral resolution of a few micrometers. Finally, we discuss the potential origins of these alterations and the

* ifllovo@cesga.es

† julien.delahaye@neel.cnrs.fr

opportunities afforded by this new technique.

II. METHODS

The insulating a-InO_x films were manufactured via e-beam evaporation. A resist mask was first made using standard e-beam lithography (EBL) on highly-doped Si substrates coated with 100 nm of thermally grown SiO₂. Subsequently, pure In₂O₃ was e-beam evaporated and deposited on the masked substrates at a rate of 2 Å/s under a partial O₂ pressure. The evaporation was stopped after depositing 30 nm on the substrate. A precise O₂ pressure of 3.0 – 3.1 × 10⁻⁵ mbar (with base pressure of the evaporator at 10⁻⁶ mbar) was selected through trial-and-error to ensure that the samples were in the regime of interest, close to the SIT transition, yet retaining insulating behavior down to temperatures of ≈ 1.7 K. Finally, the resist mask was removed using acetone (lift-off). Up to 20 samples were made for each batch, on a single 1 × 1 cm² substrate. Different geometries were tested, with channel width (W) × length (L) ranging 5 – 10 × 20 – 25 μm², except for certain samples designed as long wires (1 × 25 μm², as detailed below). No external heating or cooling of the substrate was applied throughout these fabrication steps (evaporation and lift-off).

Following sample deposition, the DC electrical resistance R of the samples was measured with a 4-contact method using a Keithley 6221 current source and a Keithley 2182A nanovoltmeter. The sheet resistance of the samples $R_{\square} = R \cdot L/W$ was then obtained from the actual geometric factor L/W of samples, calculated from optical microscopy images. In the R_{\square} range of our films (around 3 kΩ at room temperature), a small resistance drift was observed, well described by a $\log(t)$ decrease, where t is the time elapsed at room temperature after deposition. To mitigate the effects of this drift, the samples were left at room temperature for at least one day after lift-off, prior to low temperature characterization and subsequent e-beam treatment. After one day, the relative variations in resistance were less than 1% over the following day. The samples were then cooled and measured down to 1.6 – 1.7 K in a Variable Temperature Insert (VTI), in order to verify their insulating nature. For each sample, sufficiently low currents were selected to prevent self-heating while maintaining a high signal-to-noise ratio. A custom-made voltage preamp was occasionally employed to reduce the noise level of the most resistive samples.

Our deposition setup facilitated the achievement of high sample-to-sample repeatability (within the same batch deposited on a single 1 × 1 cm² substrate) of both the geometrical factors of the a-InO_x channel and their sheet resistance at both room temperature and 4.2 K, with the exception of the long wires, for which the geometrical factors were less accurately determined during development. However, the batch-to-batch variation of

R_{\square} was between 2.7 and 3.7 kΩ at 295 K; 12 and 22 kΩ at 4.2 K; and 40 kΩ and 200 kΩ at 1.7 K (with 10 different evaporation runs conducted), despite maintaining identical nominal evaporation parameters (oxygen pressure and evaporation rate). This highlights the challenge of finely controlling the electrical properties of the a-InO_x films solely through e-beam evaporation.

Following the initial characterization, the 1 × 1 cm² substrates were transferred into the chamber of a LEO1530 scanning electron microscope, where they underwent exposure to the e-beam using a standard lithography controller and software (RAITH Elphy Plus). This system offers flexibility in adjusting both the acceleration voltage of the electron beam and the electron dose per surface area received by the samples, while also enabling precise control over which part of the channel is exposed (as detailed below). On each substrate, one sample (designated as the reference sample) remained pristine throughout the experiments. This reference sample served to monitor any potential resistance drift at room temperature and to investigate possible spurious effects that could induce annealing. Subsequently, the samples were reinstalled in the VTI, and resistance-versus-temperature curves for both the e-beam-exposed and reference channels were measured and compared. No significant effects were observed on the pristine samples following the exposure of the samples of the same batch, except for the natural evolution of a-InO_x film properties due to the brief periods of time during which the samples remained at room temperature.

III. RESULTS

Our main finding is the discovery of a novel technique for local modulation of the electronic properties of a-InO_x films across the superconductor-insulator transition through prolonged exposure to an e-beam. This is highlighted in Fig. 1, in which the R_{\square} versus T curves demonstrate an evolution with increased dose, from insulating ($dR/dT < 0$ at low T) to metallic ($dR/dT > 0$) and even superconducting behavior ($R_{\square} = 0$) at sufficiently low temperatures. A saturation value of $T_c \simeq 2.85$ K was achieved, closely matching observations in superconducting a-InO_x films obtained by e-beam evaporation of pure In₂O₃ under low oxygen pressure [13], defining T_c from the linear extrapolation to $R = 0$ of the $R(T)$ curve at the point of maximum slope of the superconducting transition. The evolution towards the superconducting state and the increase of T_c are accompanied by a decrease of the normal state resistivity with the applied dose.

Given this result, we tested the possibility of drawing superconducting microstructures within an insulating a-InO_x matrix. We exposed lines measuring 1 μm wide within 5 – 10 μm wide channels (see Fig. 2). A zero resistance state is observed at sufficiently low temperatures, evidencing the presence of a continuous line of superconducting material in the channel. The effect of the acceler-

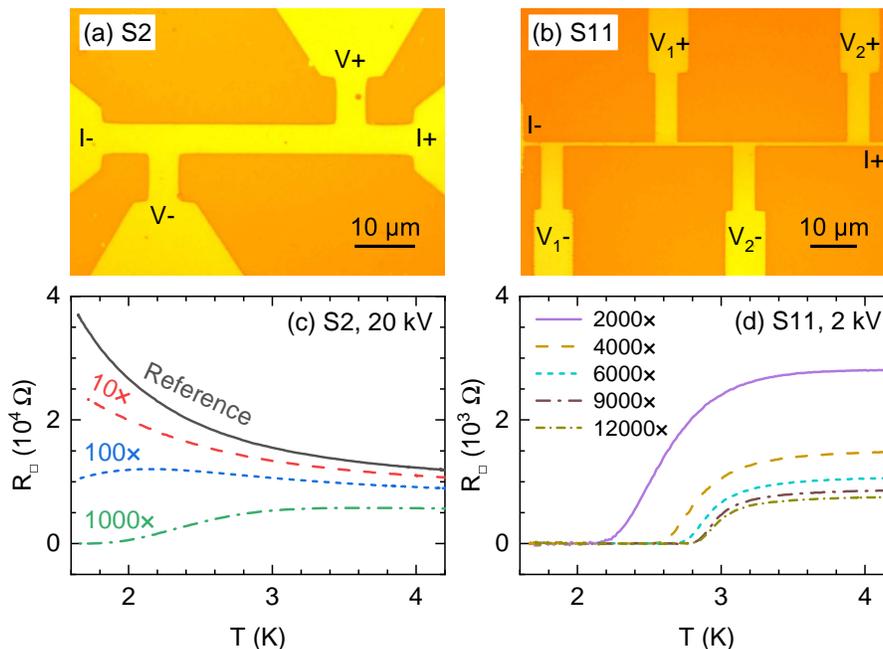


Figure 1. Effect of dose at different constant acceleration voltages. (a, b) Geometry of the channels used in substrates S2 and S11, respectively. (c, d) $R_{\square}(T)$ curves before (Reference) and after the e-beam treatment, with doses indicated as multiples of the standard dose for PMMA resist lithography, $250 \mu\text{C cm}^{-2}$. The samples from substrate S2 were exposed to a 20 kV e-beam, finding that progressively increasing the dose brings the sample from the insulating to the superconducting behavior. With a dose of 1000 \times , the sample became superconducting below $\simeq 1.9$ K. The samples from substrate S11 were exposed to a 2 kV e-beam at higher doses. As shown, all the channels of sample S11 became superconducting after the treatment, with a progressive increase of T_c with dose from 2.28 K up to 2.84 K, as well as a diminution of the normal state sheet resistance from 12 k Ω at 4.2 K (Reference unexposed sample, not shown in (d)) down to less than 1 k Ω for dose factors 9000 \times and 12000 \times . This correlation between normal state resistivity and T_c , observed for all treated samples, is also present in a-InO $_x$ samples grown by the usual O $_2$ -atmosphere method (see main text for details).

ation voltage on T_c is clearly discernible: for substrate 9 (S9) (left panel of Fig. 2), we observed a T_c increase from 2.0 K up to over 2.8 K as the acceleration voltage was decreased from 20 to 2 kV. This trend was consistently observed across different samples (see the right panel of Fig. 2). A rough estimate of the effective e-beam-treated line width can be obtained from a comparison of the R_{\square} values in the normal state ($T \simeq 4.2$ K) before and after the e-beam treatment. For a 1 μm wide line exposed in a 10 μm wide channel (corresponding to S9), R_{\square} decreased from 15 k Ω to 5 k Ω . In other samples where the whole channel was exposed, R_{\square} was approximately $\simeq 1$ k Ω for similar e-beam parameters (acceleration voltage of 2 kV and dose factor of 6000 \times). Assuming that the measured resistance results from a parallel conduction between a homogeneous line affected by the e-beam and the rest of the channel, which remains unaffected, we infer that the effective line width is approximately $\sim 1.4 \mu\text{m}$, significantly larger than the nominal width.

Other microstructures were drawn to estimate the spatial resolution of the e-beam treatment. For instance, a single line of width w much narrower than the total channel width was exposed while leaving a length s unexposed in the center of the wire, creating an insulating interruption or “gap”, as shown in Fig. 3 (a). Another ge-

ometry exposed two parallel wires offset by a distance s but overlapping by a distance o longitudinally, as shown in Fig. 3 (c). Additionally, the samples shaped as long wires allowed us to test the exposure of the entire channel width at high doses while leaving a small insulating gap s embedded a fully superconducting wire, as pictured in Fig. 3 (e). The results of these tests were unequivocal – we successfully created short insulating interruptions within the superconducting exposed sections, as evidenced by the $R_{\square}(T)$ curves shown in Fig. 3 (b,d,f). At lower temperatures than the clearly visible superconducting transition of the exposed parts, an insulating behavior ($dR/dT < 0$) was observed down to $s \gtrsim 4 \mu\text{m}$. For gaps below this value, the wires become either metallic ($dR/dT > 0$) or superconducting. This indicates that the resolution of our treatment is about $\sim 2 \mu\text{m}$, half the size of the gap, confirming the locality of the proposed treatment. A spatial resolution of this order of magnitude was observed for all acceleration voltages. Finally, for one of the batches (S6), we investigated the effect of reducing line widths by adopting the second geometry and employing $w = (100 \text{ nm}, 300 \text{ nm}, 1 \mu\text{m})$. In this case, we observed very similar T_c and resolution length scale for $w = 300 \text{ nm}$ and 1 μm , while a smaller T_c was

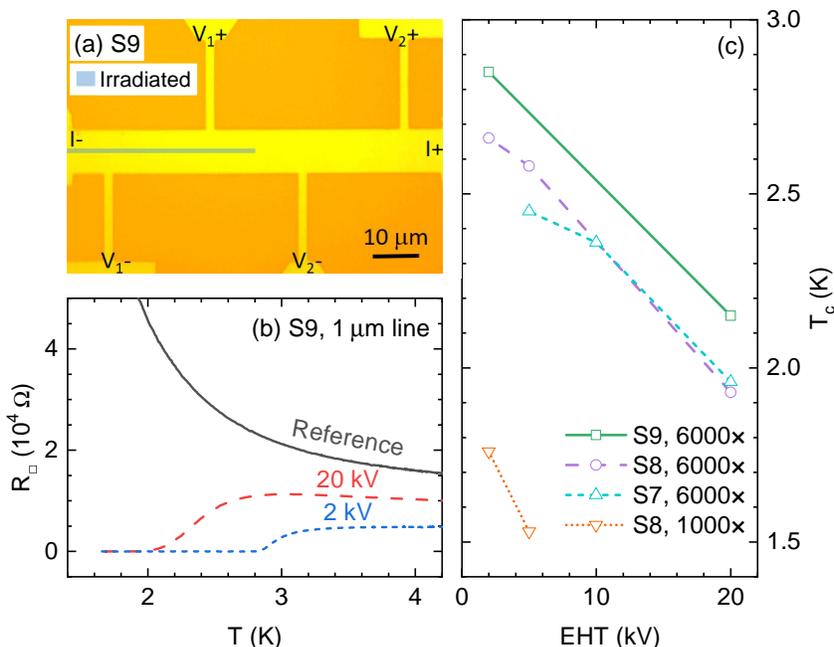


Figure 2. Results of the e-beam treatment with different acceleration voltages. (a) Diagram of the irradiated area, a 1 μm wide line in the middle of the a-InO $_x$ channel, represented as the shaded area on top of the optical microscope image. (b) $R_{\square}(T)$ curves measured for substrate S9 before (Reference, solid black line) and after the treatment. A clear T_c increase was observed when the acceleration voltage of the electrons was reduced from 20 kV (long-dashed red line, $T_c = 2.0$ K) to 2 kV (short-dashed blue line, $T_c = 2.8$ K). (c) Evolution of T_c with the acceleration voltage EHT for three different samples.

obtained for $w = 100$ nm (see Fig. 3 d).

IV. DISCUSSION

We will now discuss the mechanisms likely to explain the observed drop in electrical resistance after e-beam exposure. A standard process for tuning amorphous InO $_x$ film resistance across the SIT is annealing [12, 13]. Below $\simeq 100^\circ\text{C}$, annealing induces an irreversible decrease in resistance while preserving the amorphous structure of the films [3], and is accompanied by a densification of the film [27, 28] while the charge carrier density remains essentially unchanged [12, 13]. In films with low carrier density ($n \simeq 10^{20} \text{ cm}^{-3}$), which exhibit high resistance in their non-annealed state [29], the resistance drop at room temperature can be of several orders of magnitude [29]. However, in our amorphous films, which are less resistive and have higher charge carrier density (according to our e-beam evaporation parameters – evaporation rate of 2 \AA/s and $P_{\text{O}_2} = 3 \times 10^{-5} \text{ mbar}$, $n \sim 10^{22} \text{ cm}^{-3}$), annealing may decrease the room temperature resistance by at most a factor of 4 [28]. Remarkably, this decrease is commensurate with the decrease observed at room temperature between the resistance of an unexposed film, and the same film exposed to the highest doses (12000 \times). For example, in S11, R_{\square} decreases from 2.6 k Ω prior to e-beam exposure to 0.6 k Ω after (note that the relative changes at 4.2 K are larger: from 15 k Ω to 0.7 k Ω). Hence, the

question arises as to whether the SEM e-beam can induce such annealing. When the electron probe diameter is much smaller than the electron range λ in a solid, which seems reasonable in our case (see simulations below), an estimate of the local temperature increase ΔT is given by [30]

$$\Delta T = 3fUI/(2\pi\kappa\lambda). \quad (1)$$

In Eq. 1, f is the power conversion factor to heat, U the acceleration voltage of the electrons, I the current intensity of the e-beam, λ the electron range and κ the thermal conductivity of the material. Taking $f = 100\%$, $U = 20 \text{ kV}$, $I = 4 \text{ nA}$, $\lambda \simeq 1 \text{ \mu m}$ and $\kappa \simeq 100 \text{ W m}^{-1}\text{K}^{-1}$ (for Si++ substrate), we obtain $\Delta T \simeq 0.4^\circ\text{C}$. This value is significantly smaller than the ΔT required for annealing of amorphous InO $_x$, which is typically several tens of degrees. It is worth noting that much larger ΔT have been reported [31], when significantly higher currents (μA) and/or voltages (100 kV) are used. To evaluate the significance of an e-beam heating effect, we conducted experiments comparing resistance changes obtained under 20 kV but using two different currents and the same dose: 4.2 nA (120 μm diaphragm) and 1.0 nA (60 μm diaphragm). Minute R_{\square} and T_c changes were observed: R_{\square} increases from 2.25 k Ω to 2.28 k Ω at room temperature and T_c decreased from 2.65 K to 2.55 K.

The simulations of the electron trajectories at different acceleration voltages can provide insight into the potential mechanisms underlying the observed resistance

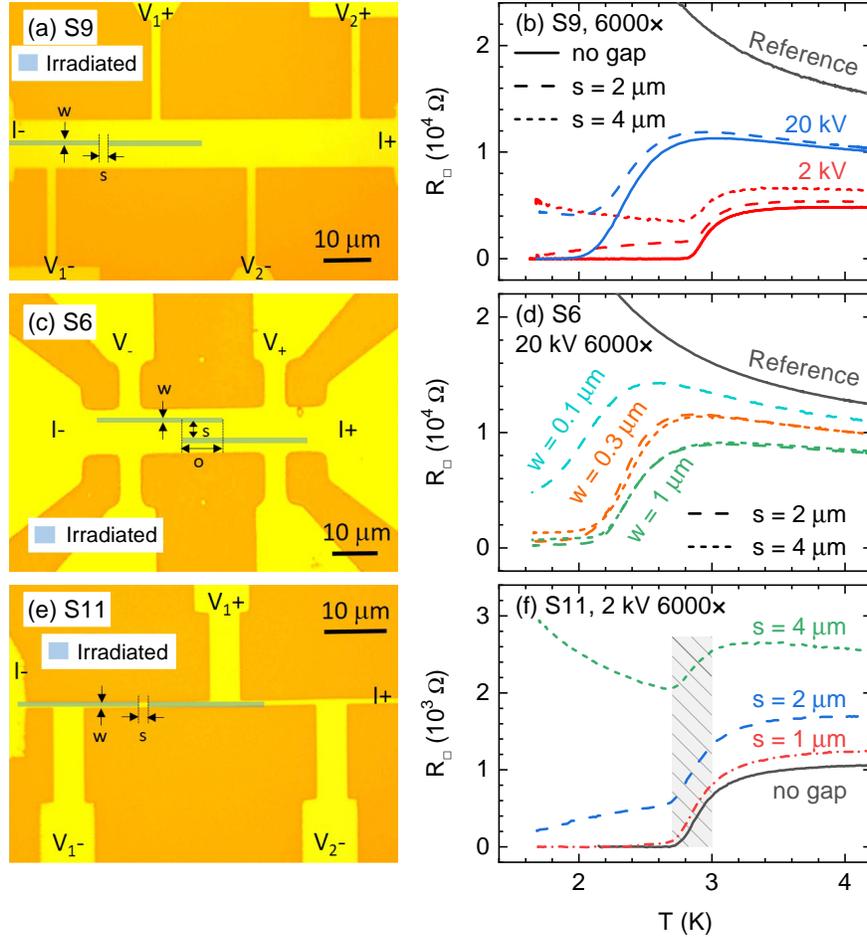


Figure 3. The spatial resolution of the e-beam treatment was tested using different acceleration voltages and geometries, and with a dose of 6000 \times , almost sufficient to saturate the T_c value (see Fig. 1 (c)). (a, b) 1 μm wide lines, embedded in an insulating matrix, and separated by an insulating gap of length s , were drawn on substrate S9 at both 2 kV and 20 kV. (c, d) On substrate S6, lines of different width w , overlapping 10 μm longitudinally and separated by a distance s transversally were drawn instead. (e, f) Substrate S11 was finally used to draw 1 μm wide fully superconducting channels separated by an insulating gap of length s . As shown, a superconducting behavior (i.e., $R = 0$) was not observed above 1.6 K for $s \geq 2 \mu\text{m}$, and the gap remained insulating for $s \geq 4 \mu\text{m}$. Nevertheless, the superconducting transition of the exposed part is still visible in all samples, evidencing the characteristic sudden resistance drop at a remarkably repeatable T_c within the same batch [see e.g. the shaded area in (f), with the exception of the 100 nm lines in (d)].

changes. To conduct these simulations, we used CASINO Monte-Carlo software [26] and considered a structure comprising 30 nm of In_2O_3 , 100 nm of SiO_2 , and 10 μm of Si. From the results, illustrated in Figure 4, we conclude that, at 20 kV, the electron range extends to a few micrometers within the Si substrate, whereas at 2 kV it remains confined within the 30 nm layer of a- InO_x . However, the part of the a- InO_x film directly affected by primary (and thus secondary) electrons is confined to a few tens of nanometers radially from the electron beam (assumed to have a diameter of 10 nm). Given that the region with reduced resistance due to the e-beam treatment extends approximately 1 μm beyond the exposed area, direct action of the e-beam electrons alone cannot explain these changes. Instead, our simulations

strongly suggest that they may be attributed to indirect energetic sources, such as X-rays or UV radiation generated in the “electron pear” region. This hypothesis is reinforced by the fact that significant resistance changes have been reported under UV exposure in amorphous InO_x films made by sputtering techniques [32–35]. These changes were attributed to a UV-photoreduction mechanism where the conducting photoreduced state was found to be unstable in oxidizing atmosphere, and subsequent ozone or O_2 plasma exposure of the films achieved the opposite effect, reverting the samples to the high resistance state. These photoreduction-oxidation cycles were associated with significant changes in the oxygen content of the topmost 10 nm layer of the film, and correlated with oxygen outdiffusion from the film [35].

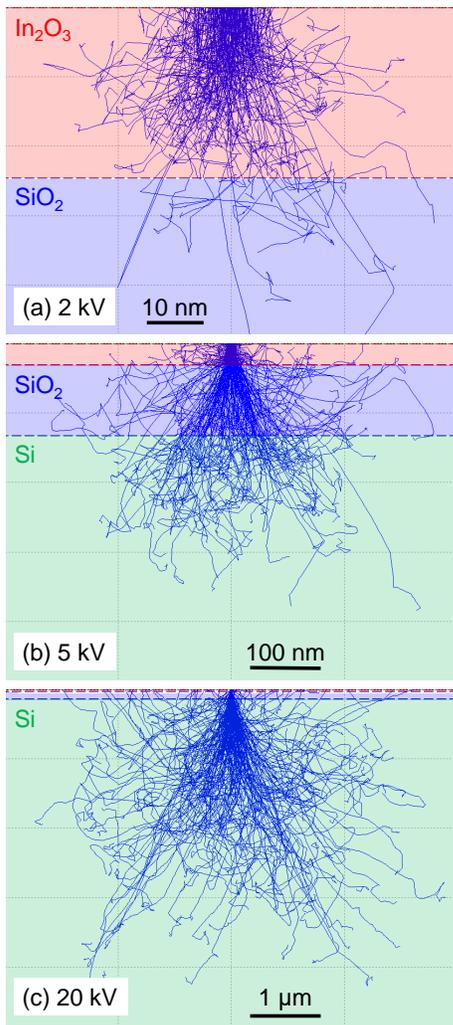


Figure 4. Simulations of the e-beam electron trajectories at different acceleration voltages: (a) 2 kV; (b) 5 kV; (c) 20 kV. The simulations were done using CASINO [26], and assuming 30 nm of In_2O_3 (red), 100 nm of SiO_2 (blue) and 10 μm of Si (green).

Additional tests were conducted to evaluate the stability of the low resistance state obtained after e-beam exposure. A superconducting channel (with T_c around 2 K) was exposed to ambient air at room temperature for 3.5 days. A small resistance increase of the exposed channel from 2.66 to 2.70 k Ω at 300 K was observed, while during the same time, the resistance of a non-exposed insulating channel decreases by a few %. Additionally, a small increase of T_c was noted, from 1.91 K to 1.96 K. These observations indicate that the exposed channel is not reverting to the non-exposed insulating state. Note that for samples kept at lower temperature (-30°C), no significant evolution was observed. This room temperature stability rules out a surface-limited mechanism driven by oxygen exchange between the film and the atmosphere. Mechanisms not involving oxygen outdiffusion which can affect the bulk of the films have been previously discussed

in a- InO_x films [32]. If a photon causes an electron transfer from O^{2-} to In^{3+} ion, the resulting O^- ion may form weak covalent bonds with a neighboring O^{2-} ion or stronger bonds with another O^- ion. This process increases the electron concentration, leading to higher charge carrier density and electrical conductance. Such photoreduction events could be induced by X-ray and/or UV radiation produced in the e-beam pear. The intensity of the X-ray/UV radiation in the InO_x film depends in turn on their penetration length and the distance at which they are produced. At an acceleration voltage of 20 kV, most of the X-ray/UV photons are produced deep within the substrate, far from the InO_x film, which may explain the observed inverse correlation of the achieved resistance decrease and T_c with the acceleration voltage for a given electron dose (see Fig. 2).

Another important question pertains to how the $R_{\square} - T$ curves of our e-beam-exposed superconducting films compare to those obtained through the conventional method of adjusting the oxygen pressure during film deposition. We prepared a few films with thicknesses ranging from 20 to 47 nm and oxygen pressures between 0 (no added oxygen in the evaporation chamber) and 6×10^{-5} mbar with a fixed evaporation rate of 2 $\text{\AA}/\text{s}$. In these films, the R_{\square} value for a given thickness is determined by the amount of oxygen vacancies present [3, 36]. The highest T_c achieved in 30 nm thick films was about 1.4 K, when no O_2 was introduced during e-beam evaporation. The $R_{\square} - T$ curve of this film appears to fall between those of exposed films with T_c of 1.2 and 1.9 K (see Fig. 5). Moreover, comparing an e-beam-exposed sample and a 40 nm thick film evaporated under 3×10^{-5} mbar of O_2 , both having similar $T_c \approx 1.2$ K, their $R_{\square} - T$ curves are almost identical. These results suggest that the effect of e-beam exposure is equivalent to a change in oxygen vacancies content, at least regarding the $R_{\square} - T$ dependency. Additionally, our e-beam exposure process enables achieving much higher T_c in very thin films compared with the standard evaporation process: a saturation value of 2.8 K for our 30 nm thick films, compared to 1.4 K for 30 nm unexposed films (although T_c as high as 3.5 K has been reported in thicker 60 nm films [25]).

V. CONCLUSION

A new technique to locally transform an insulating amorphous indium oxide film into superconducting by e-beam exposure has been proposed. The T_c values of the irradiated superconducting region can be finely tuned by adjusting the acceleration voltage and electron dose, achieving a maximum T_c value of approximately 2.8 K in 30 nm thick films. The resulting $R_{\square} - T$ curves closely resemble those of amorphous indium oxide films deposited under different oxygen pressures.

This innovative approach to fabricating superconducting amorphous indium oxide films opens avenues for fundamental studies of the superconducting-insulating tran-

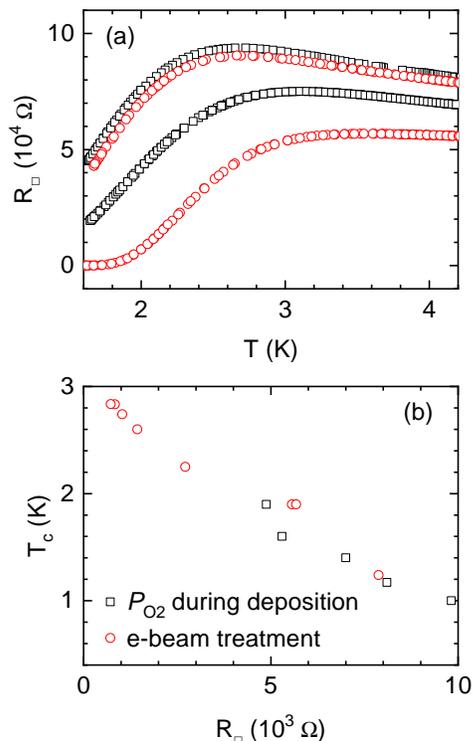


Figure 5. Comparison between samples manufactured using the standard method (changing P_{O_2} during deposition, black squares) and the technique proposed in this paper (red circles). (a) R_{\square} versus T curves between 1.7 K and 4.2 K for samples obtained using the standard method (upper curve: 40 nm thick, $P_{O_2} = 3 \times 10^{-5}$ mbar; lower curve: 30 nm, no O_2) and samples from this paper. (b) T_c versus R_{\square} at 4.2 K for samples obtained using the standard method (in ascending order of T_c : 20 nm, no O_2 ; 40 nm, $P_{O_2} = 3 \times 10^{-5}$ mbar; 30 nm, no O_2 ; 70 nm, $P_{O_2} = 3.8 \times 10^{-5}$ mbar; 47 nm, $P_{O_2} = 3.5 \times 10^{-5}$ mbar) and samples from this paper.

sition in this system. Typically, films made under different oxygen pressures lack precise control over R_{\square} and T_c , necessitating subsequent annealing steps. Our SEM e-beam exposure method enables fine control of electrical parameters, allowing the production of samples with varied R_{\square} and T_c values within a single SEM session and after a single evaporation step, while also ensuring same-batch sample-to-sample reproducibility.

Moreover, the resolution achieved is approximately $\sim 2 \mu\text{m}$, enabling the creation of superconducting lines just a few micrometers wide, with well-defined T_c or with micrometer-scale changes in T_c , within an insulating matrix of amorphous indium oxide. These characteristics hold promise for various applications, such as the development of high kinetic inductances or superconducting nanowire single-photon detectors.

The observed resistance changes may be linked to the photoreduction of the films by X-rays and/or UV radiation emitted during e-beam interactions with the film and its substrate. Further detailed structural and chemical investigations are required to elucidate this aspect.

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