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INVESTIGATION OF ENERGY RELAXATION IN DISORDERED METAL FILMS

PhD Dissertation Summary
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General work description

Physical mechanisms governing superconducting and electronic properties of ultrathin films have been studied extensively in order to understand the impact of disorder and quantum effects on electron transport in that sort of materials [1]. Despite its fundamental importance, these studies are also motivated by usability of thin disordered films in nanoscale superconducting devices, such as photon detectors [2—5] and others. To optimize the operation of these thin-film devices, it is essential to know parameters, which control the nonequilibrium response to radiation: for instance, electronic and phonon heat capacities, electron diffusivity, rates of inelastic electron-electron (e-e) and electron-phonon (e-ph) scattering processes. Numerous studies of electron transport in disordered metals reveal significant impact of disorder on mechanisms of inelastic scattering. For example, an enhancement of the e-e scattering rates is expected due to a strong elastic scattering of quasiparticles in thin disordered films or due to presence of a moderate density of magnetic impurities. It is also proposed that strong disorder can modify the e-ph scattering, and one can expect weakening or strengthening of the e-ph interaction, depending on the specific properties of disordered systems, or emergence of additional relaxation channels. In samples with reduced dimensions, relaxation processes also depend on sample size, which can lead to an even greater variety of effects in inelastic relaxation. Thus, understanding a role of disorder in inelastic scattering in thin-film devices can come mainly from an empirical study of a specific material.

Thin films of materials such as Nb, NbN, TiN are widely used in modern electronics. They are sensitive elements of such devices as SNSPD (Superconducting nanowire single-photon detectors), HEB (hot electron bolometer) mixers, microwave nanoinductors and resonators, quantum phase-slip devices, etc.. For some applications, it is advantageous to increase the level of disorder in the superconducting film, for example, this can increase the quantum efficiency of a single-photon detector. However, the effect of disorder on other device parameters should be taken into account.

The **aim** of the dissertation research is to obtain data on inelastic relaxation in thin films of Nb, NbN and TiN, to determine the dominant relaxation mechanisms, temperature dependences of these mechanisms, the influence of magnetic and non-magnetic disorders to improve the quality indicators of superconducting single-photon detectors.

To achieve this goal the following **objectives** were considered:

1. To investigate inelastic electron scattering in a series of NbN films with controlled voltage distribution characterized by the Joffe-Regel parameter $k_F l$ in the range from 6.3 to 1.6, in a set of TiN films with a good level of nonmagnetic testing, in two series of Nb films with and without a protective layer layer. For each series, resistance measurements are performed on temperature in the range from 1.7 K to 300 K, depending on the second critical magnetic field on temperature, depending on the resistance on the magnetic field at various temperatures from the critical temperature of the superconductor T_c to about $3T_c$, and also measurements of the Hall coefficient at a temperature of ~ 25 K.
2. Process experimental data in order to determine the main transport parameters of films, such as critical temperature, charge carrier concentration, diffusion coefficient, Debye temperature, mean free path, and others.
3. Analyze magnetoresistance data and obtain the dependence of the phase shift time of the electron wave function on temperature. By calculating the degree of influence of various scattering mechanisms, identify the main factor in each set of films and its temperature dependence.
4. To study the influence of magnetic and non-magnetic disorders on the electron scattering frequency.

Provisions presented for defense:

1. The phase breaking time of electrons at low temperatures in thin NbN films does not change with increasing disorder of the films; the main contribution to the process is made by inelastic electron-phonon scattering, the time of which is inversely proportional to the second power of temperature at $T < 10$ K and smoothly transitions to proportionality to the third power of temperature at $T > 10$ K.
2. Inelastic electron scattering in ultrapure ($k_F l \approx 150$) TiN films at temperatures in the range from the critical superconducting transition temperature T_c to 3-4 T_c is characterized by high (compared to

the rates of e-e, e-ph scattering and scattering on superconducting fluctuations) rates with a weak temperature dependence, which is associated with the formation of an oxide layer on the surface of the films, initiating inelastic electron scattering on surface magnetic moments with calculated characteristic times of 2-8 ps, which practically coincides with experimental data.

3. The critical temperature of thin Nb films without a protective layer decreases with decreasing thickness, which is determined by the presence of surface magnetic disorder with a magnetic moment density of $(8.6 \pm 0.8) \cdot 10^{12}$ cm^{-2}. The presence of magnetic disorder is also confirmed by the fact that in films without a protective layer the phase shift time - $\tau_\phi = 4.0$ ps at T ≈ 10 K for a 3 nm sample - is less than the time in films with a protective layer - $\tau_\phi = 9.0$ ps at T ≈ 10 K for a 3 nm sample, which is due to the contribution of scattering on magnetic moments.

Scientific novelty of the research:

1. The inelastic scattering in ultrathin superconducting films with controlled disorder change was studied for the first time. It was found that the inelastic electron scattering time at low temperatures in thin NbN films does not depend on the Joffe-Regel parameter $k_F l$.
2. Suppression of superconductivity in thin Nb films with decreasing thickness is found, which is explained by magnetic disorder on the film surface. The phase-shift time in Nb films without a protective layer is characterized by high rates compared to τ_ϕ of films covered by a silicon layer. These rates do not have an obvious dependence on temperature.
3. An original study of the surface magnetic disorder of TiN films in the absence of non-magnetic disorder was performed. A dominant effect of magnetic moment scattering on the overall phase-shift time at low temperatures was found.

Theoretical relevance of the work is to obtain information on the role of various processes of electron energy relaxation in superconducting films and the influence of disorder on them. The results obtained can stimulate theoretical work in this direction.

Practical relevance. Thin films of niobium nitride, niobium and titanium nitride, which are used in the development and manufacture of modern nanoelectronic devices, were investigated. The dependences of various relaxation times of these films on the conditions of their deposition and the presence of a protective layer were obtained. These data are necessary and were directly used to improve the quantum efficiency and time resolution of superconducting single-photon detectors manufactured by Skontel LLC.

The **reliability** of the obtained results is confirmed by their correspondence with the results of scientific works published after the publication of the author's works.

Approbation The main results of the work were summarized at the following conferences:

XXVII International symposium «Nanophysics and Nanoelectronics» (N. Novgorod, March 2023), report "Evidence of the disorder-independent electron-phonon scattering time in thin NbN films"

XXVIII International symposium «Nanophysics and Nanoelectronics» (N. Novgorod, March 2024), report "Detection of Surface Magnetic Disorder in Magnetoresistance of Epitaxial Titanium Nitride Films"

Superconductivity in nanostructures (Skolkovo, September 2023), report "Evidence of the disorder-independent electron-phonon scattering time in thin NbN films"

III International Conference «Condensed Matter Physics» (Chernogolovka, May-June 2023), report "SIGNATURE OF DEPHASING BY SURFACE MAGNETIC DISORDER IN MAGNETORESISTANCE IN EPITAXIAL TIN FILMS"

Saint-Petersburg OPEN 2022, report "ELECTRON PHASE-BREAKING TIME IN ULTRA-THIN NB FILMS".

The author's personal contribution to the research consists of participation in the discussion and formulation of tasks, conducting all experimental measurements at low temperatures presented in this work. The author processed all the obtained experimental results and took an active part in their interpretation. The indicated works were carried out by the author in the laboratory of quantum detectors of Moscow State Pedagogical University.

Publications on the topic of the dissertation:

A. I. Lomakin, E. M. Baeva, Triznova A. D., Titova N. A., P. I. Zolotov, Semenov A. V., Sunegin D. E., Lubenchenko A. V., A. I. Kolbatova, G. N. Goltsman "Evidence of the disorder-independent electron-phonon scattering time in thin NbN films"// Physical Review B: Condensed Matter and Materials Physics. 2023. Vol. 107. No. 5. Article 054205. DOI: 10.1103/PhysRevB.107.054205

A. I. Lomakin, E. M. Baeva, Titova N., P. I. Zolotov, A. I. Kolbatova, G. N. Goltsman "Electron phase-breaking time in ultra-thin Nb films"// St. Petersburg Polytechnical University Journal: Physics and Mathematics. 2022. Vol. 15. No. 3.3. P. 64-69. DOI: 10.18721/JPM.153.312

Samsonova A. S., Zolotov P. I., Baeva E., Lomakin A., Titova N. A., Kardakova A., Goltsman G. "Signatures of Surface Magnetic Disorder in Niobium Films"// IEEE Transactions on Applied Superconductivity. 2021. Vol. 31. No. 5. Article 7000205. DOI: 10.1109/TASC.2021.3065281

Contents of the work

The **introduction** substantiates the relevance of the research conducted within the framework of this dissertation, provides a review of the scientific literature on the problem under study, formulates the goal, sets the objectives of the work, and formulates the scientific novelty and practical significance of the presented work.

The **first chapter** is devoted to a review of the literature on the topic of the dissertation. It provides a physical interpretation of the corrections to conductivity used to describe resistance near the critical temperature and to model the process of changing the material resistance at these temperatures. In addition, a description of modern electronic devices is given, for the design of which the research presented in the dissertation will be useful.

Chapter two is devoted to the description of the studied samples, measurement schemes and initial processing of the results. Three materials were used in the study. High-quality Nb films were grown on a 400 μmthick substrate by magnetron sputtering. During the deposition process, the substrate is heated to 400°C. Films of different thicknesses, from 2.5 to 62 nm, were fabricated. The thinnest samples were coated in situ with a 1 nm thick Ti layer. Under atmospheric conditions, the Ti layer transforms into TiO_x oxide and prevents rapid oxidation of the ultrathin Nb film in air. High-quality epitaxial TiN films are grown on a c-sapphire ⟨111⟩ substrate at a temperature of 800°C by reactive magnetron sputtering on a direct current from a Ti target with a purity of 99.999%. The film thicknesses are from 4 to 20 nm. Structural characterization by X-ray diffraction and atomic force microscopy of nominally identical films revealed single-crystal order and atomically smooth surface of the studied samples. Chemical properties of TiN/sapphire epitaxial heterostructures were studied using X-ray photoelectron spectroscopy (XPS). Quantitative analysis of relative component concentrations revealed the presence of different phases in TiN films: TiO_2, TiO_x, $Ti(NO)_x$, TiN_x, TiN. Ultrathin NbN films were deposited on r-cut sapphire substrates using a magnetron sputtering system. All films had the same thickness $d = 2.5$ nm, but different disorder levels. The disorder level in five NbN films (s1–s5) is varied by changing the substrate temperature in each deposition process, T_{dep}: 500°C, 400°C, 300°C, 150°C, and 25°C (without additional heating), respectively. Films s1–s5 are also grown at a fixed nitrogen concentration of 22% and maintaining a constant working pressure of 3.6 mTorr. The most disordered samples are grown under the following conditions: $T_{dep} = 500°\mathrm{C}$; 27% nitrogen at 6.5 mTorr for s6 and 23% nitrogen at 6.8 mTorr for s7. To prevent unintentional oxidation of NbN in the atmosphere, the films are coated in situ with a 5 nm thick silicon passivation layer.

The main part of the studies mentioned in the work was carried out in a helium Dewar vessel STG-40 in a stainless steel case, or in a stainless steel pumping insert to achieve lower temperatures. At the end of the pumping insert there is a throttle for pumping out helium vapor ^{4}He, which allows reaching a temperature of 1.7 K. The samples were installed at the end of the measuring model. For the studies, all films were pre-structured into Hall bridges 1000 μm long and 500 μm wide. Resistance was measured using a four-point scheme. A solenoid made of a high-temperature superconductor and a DC power source were used to create a magnetic field. A current of up to 60 A was applied to the coil, which corresponds to a magnetic induction in the central part of the solenoid of up to 4 T. The maximum value of the magnetic field varied depending on the set of samples. For all samples, measurements of resistance versus temperature, the second critical magnetic field versus temperature, resistance versus magnetic field at several fixed temperature values (magnetoresistance) were performed, as well as the determination of the Hall coefficient for calculating the charge carrier concentration.

Also in this chapter are given calculation formulas for processing magnetoresistance results in order to obtain the electron phase shift time.

The **third chapter** is devoted to the study of a set of TiN samples. The high quality of the films, previously unavailable for low-temperature studies, allows us to focus on the analysis of magnetic surface disorder, neglecting the influence of non-magnetic disorder. The main result of processing the magnetoresistance measurements is a set of electron phase-kick times for temperatures from T_c to about $3T_c$ for all samples. The phase-kick mechanisms typical for thin superconducting films above T_c are caused by inelastic and magnetic scattering processes. Inelastic scattering is usually represented by electron-electron (e-e) scattering, scattering by superconducting fluctuations (e-fl), and e-ph scattering. The combined effect of inelastic scattering rates $\tau_{\mathrm{e-e}}^{-1}$, $\tau_{\mathrm{e-fl}}^{-1}$, $\tau_{\mathrm{e-ph}}^{-1}$, and magnetic scattering rates τ_{s}^{-1} can be described by the following expression [6]:

$$\tau_{\phi}^{-1} = \tau_{\mathrm{e-e}}^{-1} + \tau_{\mathrm{e-fl}}^{-1} + \tau_{\mathrm{e-ph}}^{-1} + 2\tau_{\mathrm{s}}^{-1}. \tag{1}$$

The previous studies of the disordered TiN films [7—9] show that the phase-breaking rates are in quantitative agreement with the inelastic e-e scattering rates expected for thin disordered films [10]: $\tau_{\mathrm{e-e}}^{-1} = \frac{\pi g k_{\mathrm{B}} T}{\hbar} \ln\left(\frac{1}{2\pi g}\right)$, where $g = e^2 R_{\mathrm{s}}/(2\pi^2\hbar)$. In the case of high-quality TiN samples with $R_{\mathrm{s}} \ll \hbar/e^2$, the estimate $\tau_{\mathrm{e-e}}^{-1}(T)$ corresponds to time values >60 ps at T <10 K (for MR1) and turns out to be negligible compared to the observed τ_{ϕ}^{-1}. The estimated value of $\tau_{\mathrm{e-fl}}^{-1}$ and the absence of characteristic T-behavior indicate its insignificant contribution to τ_{ϕ}^{-1} in TiN epitaxial films. The rate of e-ph scattering by acoustic phonons depends on T according to the power expression: $\tau_{\mathrm{e-ph}}^{-1} = 7\pi\zeta(3)\lambda_{\mathrm{3D}} k_{\mathrm{B}} T^3/(2\hbar\theta_{\mathrm{D}}^2)$. [11] With fitted values of the Debye temperature θ_{D} and the e-ph coupling constant $\lambda_{\mathrm{3D}} \approx 0.73$ [12] the estimated $\tau_{\mathrm{e-ph}}$ are in the range of 0.1 - 10 ns at temperatures of 10 - 3 K, respectively. This indicates a minor contribution of e-ph scattering to the electron dephasing in TiN films.

In turn The presence of surface magnetic defects is known to significantly increase τ_{ϕ}^{-1} and lead to the T-independent behavior of τ_{ϕ}^{-1} at low temperatures [13—16]. In this case, the spin-flip scattering time τ_{s} can be estimated using the Abrikosov-Gorkov (AG) theory as [17]:

$$\ln\left(\frac{T_{\mathrm{c}}^{0}}{T_{\mathrm{c}}}\right)=\Psi\left(\frac{1}{2}+\frac{\hbar}{2\pi k_B T_c \tau_s}\right)-\Psi\left(\frac{1}{2}\right), \tag{2}$$

where T_{c}^{0} is the critical temperature in the absence of magnetic disorder. To compare the experimental data for $\tau_\phi^{-1}(T)$ with the spin-flip scattering rate, we use the AG model (Eq. (2)) assuming that the critical temperature is controlled by the magnetic disorder. The estimated values of τ_{s} are 2.4 ps, 8.5 ps, 8.3 ps and 8.0 ps for TiN1, TiN2, TiN3 and TiN4, respectively.

Where magnetic disorder comes from in non-magnetic structures is always an intriguing question. The magnetic moments in TiN films can originate from the unpaired 3d electrons bound to $Ti^{+3} - O_V$ defect complexes [18], where O_V is the oxygen vacancy or the unpaired localized spins mediated by nitrogen vacancies [19]. The surface character of magnetic scattering indicates the importance of the film interfaces with either the substrate below [20] or the oxidized layer above [21]. It is also worth noting that a similar T-behavior of τ_ϕ^{-1} with a tendency to saturation at low T is observed for ultra-thin films of niobium (Nb) and copper (Cu) with native oxide on the film surface. Thus, we believe that the observed increase in the phase-breaking rate in thin epitaxial TiN films with decreasing film thickness may be due to the influence of surface magnetic disorder, which can be reduced by the deposition of a protective dielectric layer.

Chapter 4 describes the studies of high-quality niobium films. To characterize the electrical properties of the samples, we determine the resistivity ρ and the residual resistance ratio (RRR) as $\rho = R_s d$ и $RRR = (R_s^{300K} - R_s^{10K})/R_s^{10K}$, where R_s is the resistance per square, d is the film thickness.

As a function of the decreasing temperature, ($\rho \propto T$) decreases linearly at high temperatures, over a wide range between 50 K and 300 K, and then, at approximately T =20 K, it reaches a residual value, ρ_0. Upon further cooling, $\rho(T)$ drops down to zero resistance below the critical temperature. For thinner films the residual resistivity ρ_0 increases and the T_c decreases. The linear temperature dependence of ρ at high temperatures is typical for metals and usually due to electron-phonon scattering [22].

The phonon conductance $G_{ph} = (R_s^{300K} - R_s^{10K})^{-1}$, as expected, linearly decreases with the film thickness. However it approaches zero at some thickness, approximately $d_{dl} \approx 2.1 \pm 0.5$ nm. This thickness, called the “dead” layer, is presumably related to a native oxide on the surface of the Nb films, which mainly consists of non-conductive niobium oxide(Nb_2O_5) [23]. Taking into account the thickness of the dead layer, we characterize other electronic parameters of the Nb films such as the carrier density at low temperatures. Due to the revision of the film thickness, we observe that the carrier density does not significantly change and remains close to the value of $n_0 = 6{,}7 \cdot 10^{22}\text{см}^{-3}$, obtained for the 60-nm Nb film. This finding suggests that the electronic properties in Nb films are persistent to a change in the film thickness.

Next we looked at various possible reasons for the suppression of the T_c. The theoretical prediction of the weak disorder model [24], where impurities enhance Coulomb interactions, does not describe the observed suppression of T_c in Nb films. Thus, the influence of nonmagnetic disorder on T_c in these films can be neglected. Having considered the inverse proximity effect caused by the presence of a non-superconducting layer on the film surface or at the film-substrate interface as the mechanism for suppressing T_c in thin Nb films, we found that, in general, the model with a normal layer thickness $d_N = 0.8 \pm 0.2$ nm describes the experimental data. Despite the obvious agreement between theory and experiment, we question the conductivity of the capping layer. First of all, the surface oxide layer consists mainly of Nb_2O_5, which exhibits insulating properties [23]. We also eliminate the change in the charge density due to the observed trend with thickness decrease. Therefore, we conclude that the inverse proximity effect cannot be a primary mechanism of the T_c suppression in the studied Nb films.

Meanwhile, the niobium oxide is suspected to contain a small density of magnetic moments, which can be detrimental for superconductivity in thin Nb films. In this case, the observed suppression of T_c with decreasing thickness may be explained by the interaction of Cooper pairs with the localized spins of the magnetic moments (spin-flip interactions) [25]. The spin-flip scattering time τ_s and critical temperature T_c are related via the famous Abrikosov-Gorkov equation (Eq. (2)).

Experimental dependence of the spin-flip scattering time τ_s on the inverse thickness d^{-1} allows to determine the effective density of magnetic moments N_M, including the contributions of surface magnetic moments N_s and magnetic moments in bulk N_b. The analysis of $\tau_s^{-1}(d^{-1})$ dependence provides with $N_s = (9.5 \pm 1.9) \cdot 10^{-3}$ and $N_b = 0$, that corresponds to density of surface magnetic moments $N_s a^{-2} = (8.6 \pm 0.8) \cdot 10^{12}$ cm^{-2}. The estimated density of surface magnetic moments is in agreement with the previously reported results for Nb films ($\approx 5 \cdot 10^{13}$ cm^{-2}).

Next, we analyze the effect of the protective silicon layer on niobium films on the processes of inelastic electron scattering. For this purpose, we selected two pairs of samples with and without a protective layer with similar thicknesses from the available films. The magnetoresistance of the samples was measured in the temperature range from T_c to $3T_c$ and the $\tau_\phi(T)$ dependences were obtained. First of all, we observe the close power-law T-dependence of τ_ϕ for the passivated samples ($\tau_\phi(T) \sim T^{-2.5}$ for A1 and $\tau_\phi(T) \sim T^{-1.5}$), meanwhile τ_ϕ for the uncovered samples does not show a pronounced dependence on T. The observed results for $\tau_{e-ph}(T)$ in the passivated samples are also close to previous reported data for thin Nb films. In contrast, we observe the enhanced phase-breaking rate τ_ϕ^{-1} for the uncovered samples B1 and B2, which evidences an additional phase-breaking mechanism. This result, together with the observed decrease of τ_ϕ and the saturation in $\tau_\phi(T)$ - dependence, indicates that the electron dephasing in the uncovered samples may be caused by the magnetic disorder in the native oxide layer [26].

In **chapter 5** a description of measurements of disordered niobium nitride films is given. The magnetoresistance of the samples was measured in the temperature range from T_c to $3T_c$ and the $\tau_\phi(T)$ dependences were obtained. First of all, the data demonstrate the close resemblance of the results for NbN samples with different level of disorder. The data are characterized by close values of τ_ϕ^{-1}, as well as a similar power-law decrease in τ_ϕ^{-1} with lowering temperature. The exact expression for τ_ϕ^{-1} is represented by sum of scattering mechanisms due to superconducting fluctuationsτ_{e-fl}^{-1}, the e-e scattering rate τ_{e-e}^{-1}, the spin-flip scattering rate τ_s^{-1}, and the e-ph scattering rate τ_{e-ph}^{-1} (Eq. (1)).

Next, from the total dephasing rate τ_ϕ^{-1} by subtracting τ_{e-e}^{-1} and τ_{e-fl}^{-1}, the temperature dependences τ_{e-ph} were extracted (XPS analysis showed that passivating Si layer on top of NbN films prevented strong oxidation. Thus, we treat the effects of magnetic disorder as negligible in analysis of τ_ϕ^{-1} dependencies). We observe that the magnitude and the temperature dependencies of τ_{e-ph} for the studied NbN films do not depend on disorder, but demonstrate the nonmonotonic temperature dependence: it is proportional to T^{-3} above 10 K, and it modifies to T^{-2} at lower temperatures.

The **conclusion** presents the main results of the work.

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