MOSCOW INSTITUTE OF PHYSICS AND TECHNOLOGY (MIPT)

As a manuscript

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# EXPERIMENTAL STUDY OF THE QUANTUM PHENOMENA IN HYBRID SUPERCONDUCTING SYSTEMS BASED ON TOPOLOGICAL INSULATORS

PhD Dissertation Summary

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## The goal of this work

**Relevance.** In a search for novel quantum states and functionalities, the number of theoretical and experimental studies of superconducting hybrids involving topological insulators (TIs) is rapidly growing. A general hope is to induce superconducting correlations from a superconductor (S) into a TI, while preserving the specific electronic properties of these singular materials. The interest in topological superconductors arose largely from the wish to combine the inherent electron–hole symmetry of excitations in a superconductor with the helical nature of the electronic states in topological materials so as to form Majorana zero energy states. Several theoretical works suggested specific geometries for S/TI hybrids in which such a topological superconductivity could appear [1]. The coherent manipulation and braiding of such states is of interest for quantum computing, taking advantage of their non-abelian statistics. Unfortunately, unlike in theories, controllable induction of topological superconductivity into TIs remains challenging, limiting the exploration and further applications of the phenomenon. Technologically, success is hardly possible without solving two essential problems: (i) Control of the S/TI interface and (ii) Attending a ballistic regime in S/TI devices [2—4].

This work is devoted to the experimental solution of these problems. Synthesis of high-quality single crystals of a topological insulator is the basis for studying and utilizing their unique properties. The thin form of TI can be obtained by the molecular beam epitaxy (MBE) [5; 6], but it is expensive and less accessible. Mechanical exfoliation of thin wafers from bulk crystals, which is widely used to produce graphene from graphite [7], can also be used to obtain thin TI layers, but with very low output in combination with the irregular shape of obtained objects. In this work, the physical vapor deposition (PVD) method was used [8—10]. Synthesis and characterization of nanoscale monocrystals of various compositions ($Bi_2Se_3$, $Bi_2Te_3$, $Bi_2Te_2Se$, etc.) and various morphologies including nanorods, nanoplates, nanowires, etc. have been reported. However, the large concentration of bulk residual carriers has been a major problem for revealing the properties of topological surface states. Using the PVD method, it is possible to obtain single crystals with triangular and hexagonal morphologies with thicknesses down to 10 nm, which contributes to the reduction of the concentration of bulk carriers and easy regulation of the Fermi levels near the Dirac point by means of

a gate. The observation of singular quantum transfer of topological surface states means that high-quality single crystals of topological insulators have superiorities for investigating new physical properties and developing potential applications.

Recently, the number of works with the observation of Josephson supercurrent in hybrid structures superconductor - topological insulator - superconductor has increased [11; 12]. For topological superconductivity studies it is very important whether the transport is diffusive or ballistic. The well-known theoretical descriptions of the "Proximity Effect"in superconductor-normal metal interfaces are based on the semiclassical Uzadel equation. The equations developed for ordinary diffuse metals are most often extended to topological insulators. In articles [13; 14], the diffusive character of conductivity in the weak-coupling region is clearly expressed. For a further study of the Andreev states and the Majorana states with zero energy, a ballistic transport mode is required [15]. There are very few articles with the ballistic transport in S/TI/S structures [16; 17]. The first experiments with such structures were performed by the author of this dissertation and published in a recent paper. These initial experiments have raised a number of problems and questions listed in the first section of this form: demonstration of ballistic transport in the obtained hybrid structures; nontrivial responses of the critical current dependence on the magnetic flux; study of multiple Andreev reflections at the S/TI boundary; estimation of the number of ballistic channels involved in electron transport; demonstration of excess current at high voltages; disappearance of first Shapiro steps, etc. In light of this, the studies carried out within the framework of the present dissertation seem to be relevant, and the results - are a significant contribution to the further development of the field.

**Aim of work** of this thesis was to create and provide experimental studies on the superconducting properties of hybrid structures involving a superconductor/topological insulator/superconductor. These structures were fabricated using single crystals of $Bi_2(Te_xSe_{1-x})_3$. The research aimed to uncover and elucidate novel phenomena emerging in these systems at ultra-low temperatures.

To achieve this goal, it was necessary to solve the following **Tasks**:

1. Develop the setup for the synthesization of single crystals of the topological insulator $Bi_2(Texe_{1-x})_3$ using the PVD method. This setup should offer the capability to manipulate the position of the Fermi

level by adjusting the composition. Produce ultrathin single crystals of topological insulators with lateral dimensions ranging from 0.1 to 1 m, and thicknesses of up to 10 nm, with hexagonal and triangular shapes.

2. Investigate the morphology and composition of the synthesized topological insulator single crystals utilizing a range of analytical techniques including electron backscatter diffraction (EBSD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), and transmission electron microscopy (TEM).
3. Develop a fabrication process for hybrid structures comprising a superconductor, topological insulator, and superconductor, utilizing electron lithography and magnetron sputtering.
4. Explore the low-temperature Josephson transport properties of the fabricated structures, specifically at ultra-low temperatures down to 11 mK. Study the IV characteristic as a function of temperature, externally applied magnetic fields, high-frequency radiation, as well as various technological and geometrical parameters.
5. Conduct a comprehensive analysis of the experimental data acquired and perform calculations based on theoretical models.

**Main results submitted for defense:**

1. The setup for synthesis single crystals of topological insulators through physical vapor deposition (PVD) has been engineered. The system has a two-zone induction heating system. This setup provides precise control over the selenium content in bismuth $Bi_2(Te_xSe_{1-x})_3$, changing the position of the Fermi level.
2. Ultra-thin nanocrystals of $Bi_2(Te_xSe_{1-x})_3$ with a non-stoichiometric selenium content ($Bi_2Te_{2.3}Se_{0.7}$) were synthesized. These crystals exhibited high resistance, measuring R = 2.5 kOhm with a RRR value of 2, showing metallic behavior as temperature decreased. Interestingly, for the thinnest crystals, approximately 10 nm in thickness, a slight upturn in resistance at low temperatures was observed. This phenomenon suggests that the Fermi level may be located near the bottom of the conduction band.

3. The Josephson planar junction (JJ) contacts Nb-$Bi_2Te_{2.3}Se_{0.7}$-Nb with various geometries were performed. These junctions demonstrated a critical current at approximately 1 $A$, accompanied by a normal resistance of about 1 $k\Omega$. Theoretical analysis of the conductivity, along with an examination of the temperature dependence of the critical current, strongly suggests the ballistic nature of the supercurrent transport through the $Bi_2Te_{2.3}Se_{0.7}$ nanocrystals.
4. In the presence of an external high-frequency radiation, Josephson contacts of Nb-$Bi_2Te_{2.3}Se_{0.7}$-Nb exhibit the missing of the first Shapiro step as the frequency decreases from 2 GHz to 1 GHz. Theoretical calculations employing the tRSJ model suggest that this behavior may signify the presence of a $4\pi$ component contributing to the supercurrent, estimated to be at least 5%.

**Scientific novelty:**

1. An original unit employing external induction heaters for PVD synthesis were developed and patented.
2. PVD synthesis has enabled the creation of a series of ultrathin single crystals of the topological insulator Bi2($Tex Se_{1-x}$)$_3$, with controlling the Fermi level position by changing the Se concentration.
3. In a submicron planar Josephson S/TI/S hybrid junction based on Nb/Bi2Te2.3$Se_{0.7}$/Nb with high interface transparency, a ballistic transport mode has been demonstrated. The surface ballistic channels of TI crystals significantly contribute to the transport mode.
4. The voltage-current characteristics and critical current dependence in S/TI/S junctions were studied. Using theoretical models, the contributions of ballistic and diffusive transport were assessed.
5. IV and critical current in response to high-frequency radiation were studied. The experiments show the missing of the first Shapiro step across different power and frequency ranges. The theoretical model indicates a 5% contribution from the $4\pi$ component to the transport mode, providing an explanation for the observed step disappearance.

**Author's personal contribution.** All results presented in the thesis were obtained by the author personally or with his direct participation.

**PUBLICATIONS AND APPROBATION OF RESEARCH.** The numbering of publications in the lists of publications of the advanced level and the standard level corresponds to the numbering of the list of publications of the author in the dissertation text.

**First-tier publications.**

**[A1]** Stolyarov, V. S., **Yakovlev, D. S.**, Kozlov, S. N., Skryabina, O. V., Lvov, D. S., Gumarov, A. I., ... and Roditchev, D. (2020). Josephson current mediated by ballistic topological states in $Bi_2Te_{2.3}Se_{0.7}$ single nanocrystals. Communications Materials, 1(1), 38.

**[A2] Yakovlev, D. S.**, Lvov, D. S., Emelyanova, O. V., Dzhumaev, P. S., Shchetinin, I. V., Skryabina, O. V., ... Stolyarov, V. S. (2022). Physical Vapor Deposition Features of Ultrathin Nanocrystals of $Bi2(Tex Se_{1-x})_3$. The Journal of Physical Chemistry Letters, 13(39), 9221-9231.

**[A3]** Stolyarov, V. S., Roditchev, D., Kozlov, S. N., **Yakovlev, D. S.**, Skryabina, O. V., ... and Golubov, A. A. (2022). Resonant Oscillations of Josephson Current in Nb-$Bi_2Te_{2.3}Se_{0.7}$-Nb Junctions. Advanced Quantum Technologies, 5(3), 2100124.

**Other publications.**

**[A4]** Stolyarov, V., Kozlov, S., **Yakovlev, D.**, Bergeal, N., Feuillet-Palma, C., Lvov, D., ... and Roditchev, D. (2023). Anomalous microwave response in the dissipative regime of topological superconducting devices based on $Bi_2Te_{2.3}Se_{0.7}$ arXiv preprint arXiv:2309.10897.

**[A5] Yakovlev, D. S.**, Egorov, S. V., Skryabina, O. V., Kozlov, S. N., and Stolyarov, V. S. (2020) Josephson junction based on topological insulator/superconductor heterostructure for topological quantum computation. 3rd International School on Quantum Technologies, book of abstract.

**[A6] Yakovlev, D. S.**, Lvov, D. S., Gurtovoi, V. L. and Stolyarov, V. S. Phsyycal vapor deposition setup (2020) RO Patent 203,742 Moscow, R.O. Patent and Trademark Office.

**[A7] Yakovlev, D. S.** (2020) Ballistic transport through surface states topological insulator single crystals $Bi_2Te_{2.3}Se_{0.7}$. 63 conference MIPT, book of abstract.

**[A8] Yakovlev, D. S.**, Egorov, S. V., Skryabina, O. V. and Stolyarov, V. S. (2017) The synthesis of nanodimensional monocrystals of a topological

insulator and the investigation of hybrid superconductor structures on their basis. Superconducting hybrid nanostructures: physics and application conference, book of abstract.

**Talks at conferences.** The main results of the dissertation were reported at international conferences:

1. Conference: 26eme Congres General de la SFP «Gate Tunable Supercurrent in Nb-$Bi_2Te_{2.3}Se_{0.7}$-Nb topological Josephson junctions» Dmitry Yakovlev, Ivan Nazhestkin, Vasiliy Stoliarov, Conference: 26 eme Congres General de la SFP, Paris, France 3–7 July 2023

2. «Fractional ac Josephson effect in ultrasmall $Bi_2Te_{2.3}Se_{0.7}$ single crystal based junction» Dmitry Yakovlev, Sergei Kozlov, Vasily Stolyarov and Dimitri Rodichev, International Workshop on The physics of disordered superconductors and their application to quantum circuits, Les Houches, France, 4 – 9 Juner 2023

3. «Ballistic transport through surface states of topological insulator single crystals» $Bi_2Te_{2.3}Se_{0.7}$ D.S. Yakovlev, V.S. Stolyarov 63rd All-Russian Scientific Conference MIPT 23-29 November 2020;

4. Yakovlev, D. S., Egorov, S. V., Skryabina, O. V., Kozlov, S. N., and Stolyarov, V. S. «Josephson junction based on topological insulator/superconductor heterostructure for topological quantum computation», International School on Quantum Technologies, Russia. Sochi. 1-7 March 2020;

5. Yakovlev, Dmitry «Josephson supercurrent in single-crystalline nanoplate of $Bi_2Te_2Se$ topological insulator». Traditional the 22nd International conference on Low Temperature Physics and Superconductivity, Superconducting Quantum Circuits. Mandarfen, Austria, March 16-23, 2019;

6. «Coherent effects in junctions based on p-wave superconductor» Golubov A., Stolyarov V.S., Yakovlev D.S., Skryabina O.V., Gurtovoy V., Lvov D.S., Egorov S.V., Ryazanov V.V., Roditchev D., Vinokur V.M. Quantum Coherent Phenomena at Nanoscale 2019 workshop, Ischia, Naples (Italy);

7. «Study of hybrid superconductor structures of topological insulator» Yakovlev D.S., Skryabina O.V., Lvov D.S. in Proceedings of the International Youth Scientific Forum Lomonosov-2017 ;

8. «Josephson effect in hybrid structures superconductor - topological insulator - superconductor based on nanoscale single crystals $Bi_2Te_2Se$» Yakovlev D.S., Lvov D.S., Skryabina O.V., Egorov S.V., Golubov A.A., Stolyarov V.S., in

Proceedings of Jubilee XV Kurchatov Interdisciplinary Youth Scientific School, September 2017;

9. «The synthesis of nanodimensional monocrystals of a topological insulator and the investigation of hybrid superconductor structures on their basis» Yakovlev D.S., Skryabina O.V., Egorov and N S Stepakov, Stolyarov V.S. Conference and International School Superconducting hybrid nanostructures: physics and application, 2016;

10. Yakovlev D.S., Skryabina O.V., Egorov S.V., Stolyarov V.S. «Synthesis of nanoscale topological insulator single crystals and study of hybrid superconductor structures on their basis» XIV Kurchatov Interdisciplinary Youth Scientific School (2016).

## Contents

**Introduction** substantiates the relevance of the topic, formulates the goal of the work, the novelty and practical value of the results obtained are substantiated, the content of the dissertation is described in chapters.

**The first chapter** of the thesis comprises a comprehensive literature review. It delves into the foundational topics underpinning the research conducted in this thesis. This encompasses a concise theoretical exploration of topological insulator-superconductor hybrid systems, along with an overview of the current state of the topic.

**The chapter two** delves into a discussion of various aspects related to the technological and experimental methods employed in this study.

The chapter starts with detailing the setup for synthesizing nanoscale single crystals of topological insulators (TIs) through physical vapor deposition (PVD). The design and functionalities of the installation used for TI synthesis were outlined. Notably, the setup encompasses individual induction heating of both the sample and TI material sources, providing a high degree of control over the deposition process.

Following this, the fabrication process utilizing a range of technological methods is outlined. This includes a comprehensive overview of electron lithography steps, encompassing resist deposition, electron beam illumination, and magnetron sputtering of superconducting contacts.

The measurement setups used throughout the study were also detailed. These include an Advanced Research System (ARS) cryostat, which enables measurements at temperatures down to 4.2 K, an Oxford Heliox VL transport insert that facilitates measurements down to 300 mK, and a Bluefors LD250 dissolution refrigerator that extends measurements down to 20 mK. Each of these setups is accompanied by a description of its respective measurement scheme.

**In the chapter three** structural and electronic properties of ultrathin nanocrystals of chalcogenide $Bi_2(Te_xSe_{1-x})_3$ were studied. Nanocrystals were formed from the parent compound $Bi_2Te_2Se$ on as-grown and thermally oxidized Si(100) substrates using Ar-assisted physical vapor deposition, resulting in well-faceted single crystals several quintuple layers thick and a few hundreds nanometers large. The chemical composition and structure of the nanocrystals were analyzed by energy-dispersive X-ray spectroscopy, X-ray photoelectron spectroscopy, electron backscattering, and X-ray diffraction. The chapter focuses

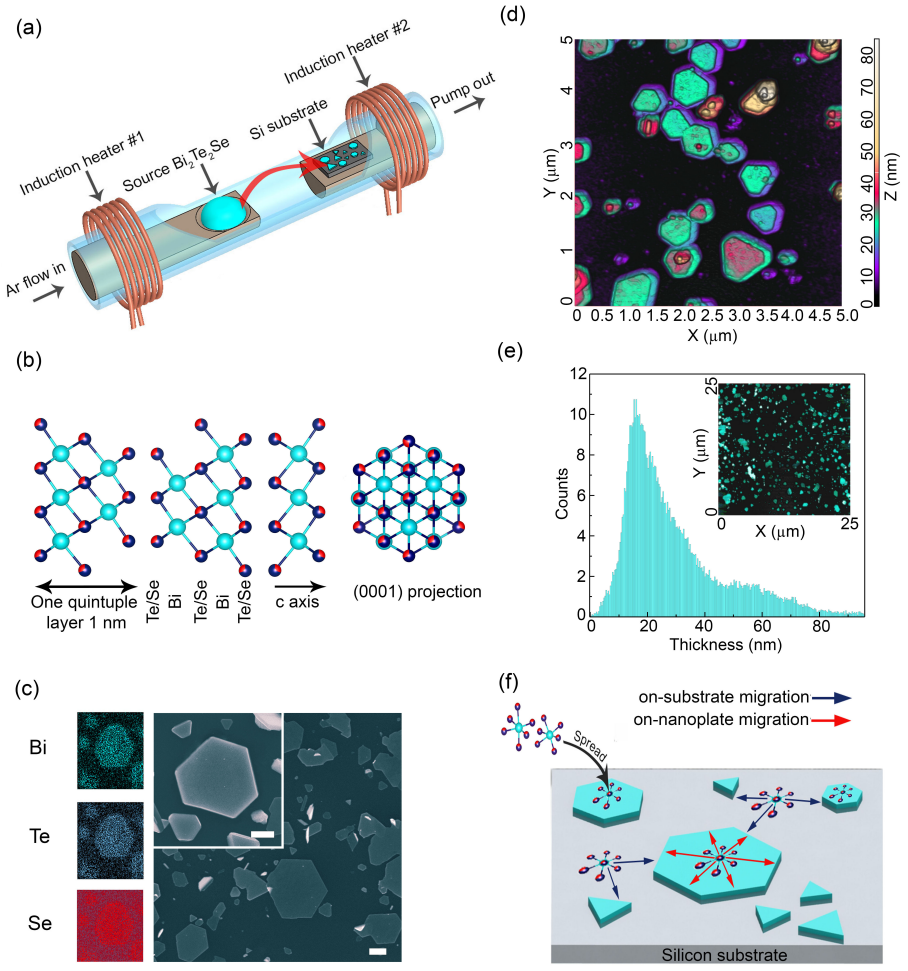


Рис. 1 — **PVD growth of $Bi_2(Te_xSe_{1-x})_3$ nano-crystals.**

on the effect of the growth conditions on the morphology, structural, and electronic properties of nanocrystals.

Morphology of the synthesized nanocrystals strongly depends on several factors: (i) the substrate material, (ii) the substrate temperature $T_2$ and (iii) the source – substrate distance (Fig. 1).

The junctions through the nanocrystals (J1–J3) clearly demonstrated a metallic tendency ($dR/dT > 0$) from 300 down to 40 K. The resistance of these samples decreases almost linearly as a result of electron-phonon scattering of the bulk carriers. Below 40 K, the samples demonstrate different behaviour: the

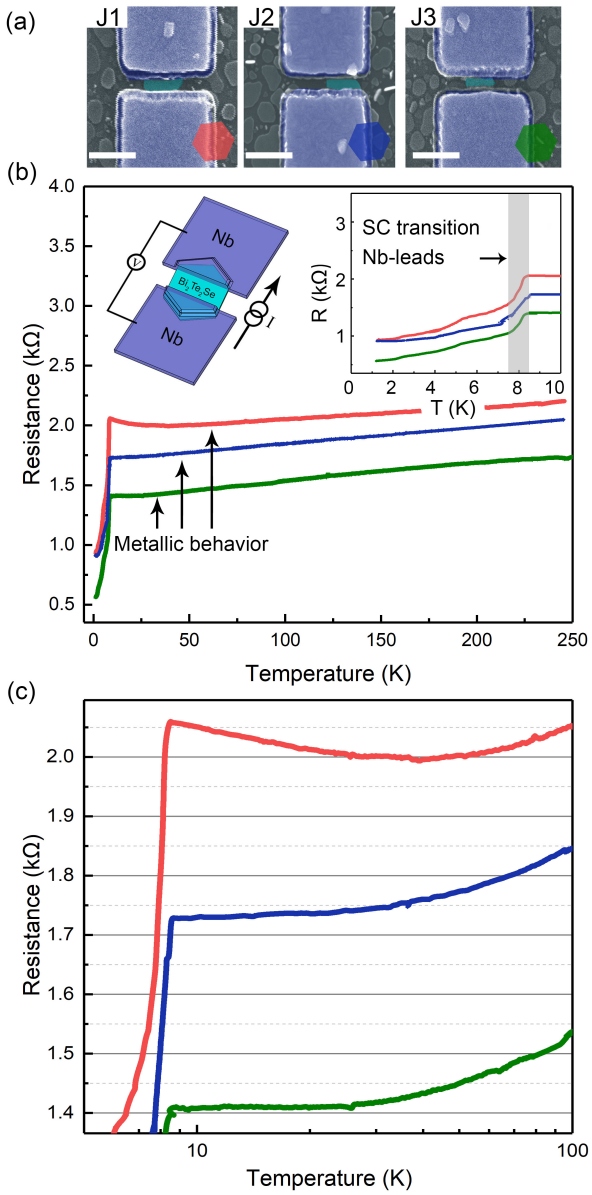


Рис. 2 — **SEM images of Nb - $Bi_2Te_{2.43}Se_{0.57}$ - Nb junctions and their R(T) characteristics.**

resistance of J1 has a slight 50 Ω kink before the superconducting transition, while the resistance of the other two samples J2 and J3 continues to drop monotonically. A more clear comparison of the resistance is shown in the "semi-ln"plot (Figure 2(c)). This distinction can be explained by manifestations of surface state resistance against the background of freezing bulk carriers in topological insulators, which is more apparent in thinner samples.

**The chapter four** shows superconducting proximity devices using low-dimensional semiconducting elements enable a ballistic regime in the proximity transport. The use of topological insulators in such devices is considered promising, owing to the peculiar transport properties these materials offer, as well as the hope of inducing topological superconductivity and Majorana phenomena via proximity effects. There is demonstration of the fabrication and superconducting properties of proximity Josephson devices integrating nanocrystals single of $Bi_2Te_{2.3}Se_{0.7}$ with a thickness of a few unit cells. Single junctions display typical characteristics of planar Josephson devices; junctions integrating two nanocrystals behave as nanodimensional superconducting quantum interference devices. A peculiar temperature and magnetic field evolution of the Josephson current along with the observed excess current effect point towards the ballistic proximity regime of topological channels.

The temperature dependence of the critical current $I_c(T)$ in zero-field for three single junctions SJ1, SJ2 and SJ3 is presented in Fig. 3. The first observation is a clear relation between the geometry of the devices and their $I_c(T)$ characteristics. The highest $I_c$ and $T_c$ are realized in SJ3 involving the largest nano-crystal and the strongest Nb/TI overlaps; the lowest values are observed in the smallest SJ1. Another remarkable effect is an almost linear rise of the critical current when the temperature is lowered. This is hardly compatible with the diffusive regime, which usually leads to a saturation of $I_c(T)$ at low temperatures. Black dashed lines in Fig. 3 are fits assuming a diffusive regime.

In the **fifth chapter** described the influence of external high-frequency radiation on the Josephson contacts of $Nb/Bi_2Te_{2.3}Se_{0.7}/Nb$. This chapter reveals the missing of the first Shapiro step as the frequency decreases from 2 GHz to 1 GHz. It is postulated that these transitions may signify the presence of Majorana bound states, with the absence of the first Shapiro step in the IV characteristics providing potential evidence of Majorana zero modes.

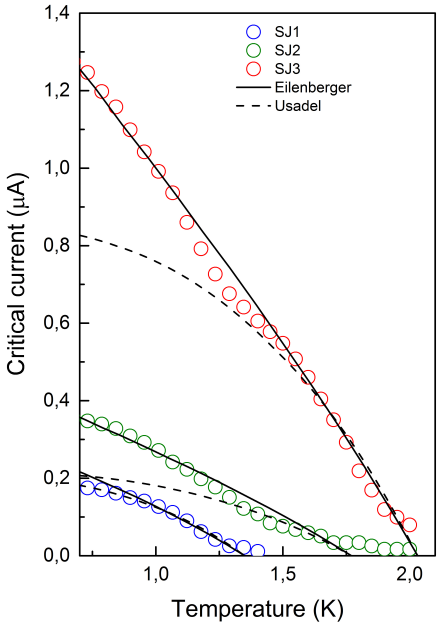


Рис. 3 — **Evolution of the critical current with temperature.** Blue, green and red open circles represent the experimental data points corresponding, respectively, to SJ1, SJ2 and SJ3. Black dashed lines: fits considering a diffusive transport. The curves fail in reproducing a steep rise of $I_c(T)$ at low temperatures. Black solid lines: fits within the ballistic regime. See further explanations in the text.

Interestingly, these transitions exhibit a ballistic nature and display Shapiro steps when subjected to microwave radiation. However, within a specific range of frequencies and powers, the first step remains elusive. Theoretical calculations utilizing the tRSJ model suggest that this behavior might imply a contribution of the $\pi$-component to the supercurrent, potentially reaching a level of at least 5%.

The text elucidates the theoretical underpinnings behind the emergence of Andreev bound states and Majorana zero modes in superconductor/normal metal/superconductor transitions. It further expounds that Majorana zero modes give rise to a $4\pi$-periodic component in the current phase dependence.

The acquired results were systematically compared with previous experiments and theories. Notably, the phenomenon of the first step's absence has been documented in diverse systems, encompassing spin-orbit-coupled nanowires and three-dimensional topological insulators. Various rationales for this absence are examined, encompassing the topological essence of the proximized material and the ballistic mode of electron transit through the transition. The

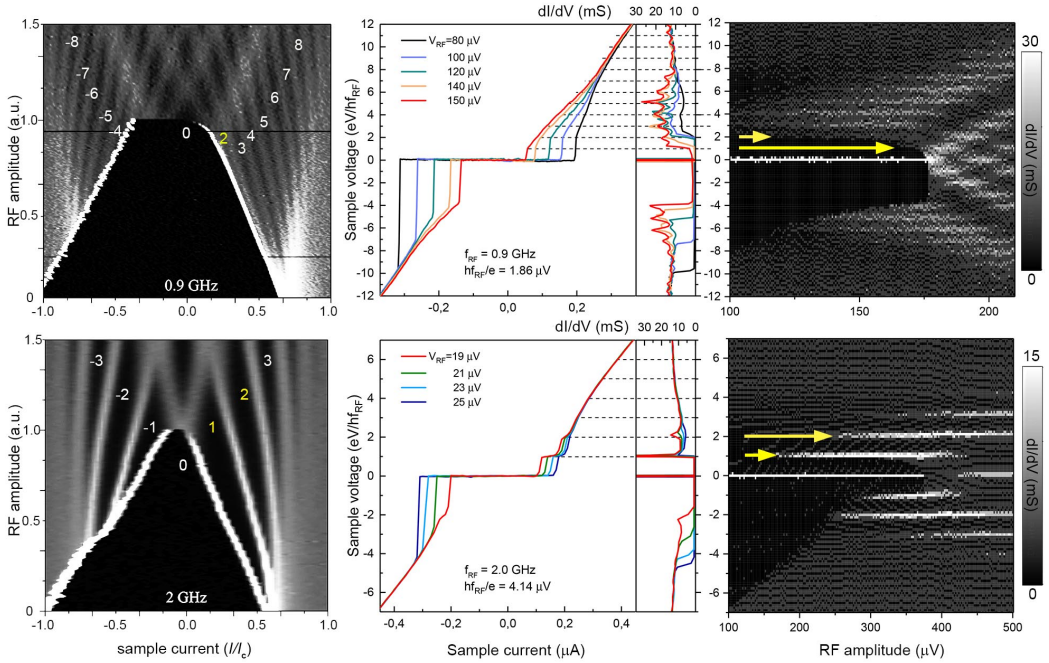


Рис. 4 — Shapiro maps dV/dI for different RF drive frequencies.

"quasiparticle poisoning"effect's role in damping the $4\pi$ component and its impact on the Shapiro first step is also scrutinized. Additionally, the distinction between topological and trivial effects, grounded on the presence of the "quasiparticle poisoning"effect, is contemplated.

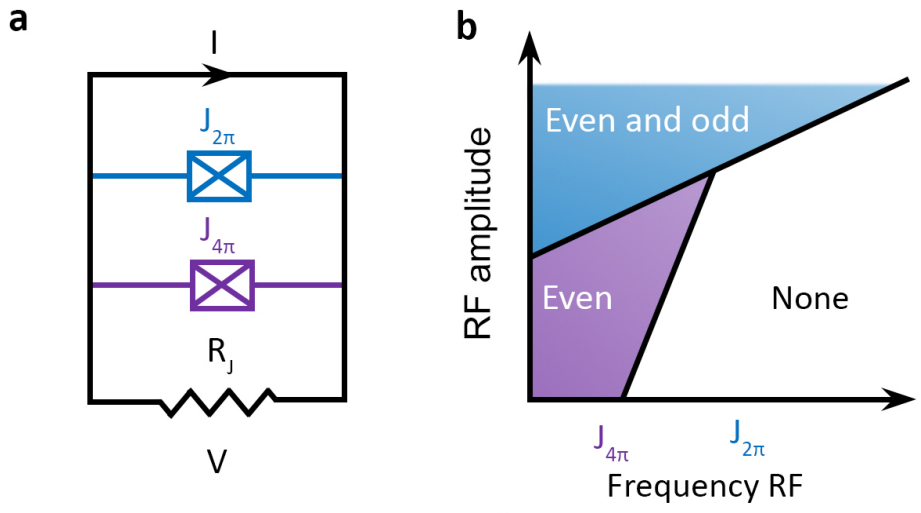


Рис. 5 — **a** Scheme of the thermal RSJ model with two channels. One of the two parallel Josephson junctions ($J_1$) represents the trivial supercurrent, and the other one ($J_2$) represents the topological channel parallel to the shunt resistance $R_n$. **b** Phase diagram of odd and even Shapiro steps in two-channel thermal RSJ model.

The chapter showcases experimental data through 3D representations of Shapiro steps, plots of differential conductivity, and IV characteristics. It delves into the behavior of these steps, examining their responsiveness to changes in RF radiation amplitude and frequency. Detailed scrutiny of critical current and return current branches were carried out, highlighting distinctions between low and high frequencies (cf. Fig 4).

Furthermore, the chapter introduces a two-channel thermal transport model (tRSJ). This model demonstrates its capacity to replicate observed behavior, including the notable absence of the initial stage, by factoring in the interplay between the $2\pi$- and $4\pi$-periodic components within the current phase relationship. Numerical results obtained from the model align closely with the experimental data presented in Fig 5.

In sum, this chapter shows experimental findings regarding the missing of the first Shapiro step in superconducting Josephson transitions of Nb/$Bi_2Te_{2.3}Se_{0.7}$/Nb. The data suggest that this phenomenon can manifest itself even in ballistic transitions. Additionally, a theoretical model is proposed that elucidates the observations and provides support for the explanation behind the absence of the initial stage, particularly when considering the 5% contribution of the $4\pi$-component.

**In the conclusions** the main results of the work and its conclusions were formulated.

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