

== ORDER, DISORDER, AND PHASE TRANSITION IN CONDENSED MEDIA ==

OBSERVATION OF REENTRANT DEPENDENCE OF THE CRITICAL CURRENT IN Nb-PdFe-Nb JOSEPHSON JUNCTIONS ON PdFe-BARRIER THICKNESS AND TEMPERATURE

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Received June 05, 2024

Revised August 02, 2024

Accepted August 03, 2024

Abstract. The results of studying multilayer superconductor-ferromagnet-superconductor (SFS) Josephson junctions based on superconducting niobium and weak ferromagnetic alloy $\text{Pd}_{0.99}\text{Fe}_{0.01}$ are presented. A minimum in the dependence of critical current density on F-layer thickness is discovered, which is one of the signs of π -state realization with a negative sign of current-phase relation. The presence of $0-\pi$ -transition, i.e., transition between π -state and 0 -state with increasing F-layer thickness, is also confirmed by the observation of reentrant temperature dependence of critical current at ferromagnet thickness of about 43 nm. Modeling predicts the second order number of the observed $0-\pi$ -transition.

The article is presented as part of the proceedings of the 39th Low Temperature Physics Conference (LT-2024), Chernogolovka, June 2023

DOI: 10.31857/S004445102412e058

1. INTRODUCTION

Josephson junctions with ferromagnetic barrier (superconductor-ferromagnet-superconductor, SFS) are very important for the development of superconducting electronics. One of the most important properties is the negative (actually, shifted by half a period) current-phase relation [1], which allows shifting the operating point of logic elements to the region of zero bias signals, thus reducing the size and power consumption of logic elements [2, 3]. Using SFS junctions, it is possible to implement Josephson elements with π -periodic current-phase relation [4, 5], which have been recently used for developing an adiabatic family of digital superconducting electronics [6]. Equally important is the creation of Josephson magnetic memory devices [7–9], the absence of which hinders the development of a full-fledged superconducting computer [10].

The origin of the negative current-phase relation is the spin antagonism between ferromagnetism and superconductivity, leading to spatial oscillations of the superconducting order parameter [11–13], induced in the F-layer due to the proximity effect [14, 15]. With increasing thickness of the F-layer, the amplitude of the current-phase relation periodically decreases to zero, and then begins to increase with the opposite sign ($0-\pi$ -transition, or transition between 0 - and π -states). Josephson junctions with a negative sign of the current-phase relation are called π -junctions (also π -state), since positive and negative current-phase relations are shifted relative to each other by π . Experimental observation of this effect is carried out by fabricating a series of samples with different thicknesses d_F of the F-layer and subsequent measurement of the critical current density dependence on thickness, $j_c(d_F)$ [16, 17]. The temperature dependence of the

oscillation period of the induced order parameter, $2\pi\xi_{F2}(T)$, allows verification of the $0-\pi$ -transition through measurement of the reentrant temperature dependence of the critical current [18, 19].

The demonstration of oscillations of the superconducting order parameter in the F-layer can be conveniently conducted using dilute ferromagnetic alloys with low Curie temperatures T_C and exchange interaction energy E_{ex} , since the oscillation period is inversely proportional to $\sqrt{E_{ex}}$ for the case of a diffusive Josephson barrier [15]. With a small value of E_{ex} the oscillation period becomes sufficiently large, which allows for experimental investigation with adequate detail. The presence of other "unpaired factors" (i.e., processes causing the destruction of Cooper pairs in the Josephson barrier) further increases the oscillation period, although it leads to a faster decrease in critical current with increasing d_F [16]. The most popular alloys are $\text{Cu}_{1-x}\text{Ni}_x$, $x \approx 0.5$ (see, for example, works [16, 19, 20]), whose components are widely available in nature, inexpensive, and well-suited for mechanical processing. Their opposites are alloys based on palladium or platinum, which become ferromagnetic at extremely low concentrations of magnetic atoms [21]. Therefore, the value of E_{ex} in them can be tuned over wide ranges by changing the concentration of, for example, iron, nickel, or cobalt (see review [22]).

At the Laboratory of Superconductivity of ISSP RAS, research on layered structures based on the alloy $\text{Pd}_{0.99}\text{Fe}_{0.01}$ has been conducted for a long time with the aim of developing superconducting memory elements. The Curie temperature for thin-film layers of this composition increases from 2 to 42 K when the F-layer thickness changes from 12 to 180 nm [7]. Despite the low content of magnetic atoms, layered structures $\text{PdFe}-\text{Nb}-\text{PdFe}$ demonstrate spin valve effect with a voltage range up to 700 μV , which corresponds to a characteristic frequency of 340 GHz when using this element in rapid single quantum logic devices [23]. Multilayer $\text{Nb}-\text{PdFe}-\text{Nb}$ Josephson junctions can also be used as memory elements due to the in-plane anisotropy of PdFe thin film layers [7]. The use of multilayer Josephson IsF barriers (insulator (I)-superconductor (s)-ferromagnet (F) tunnel layer) allows the implementation of Josephson elements that are frequency-compatible with rapid single-flux quantum logic (RSFQ) elements [8]. However,

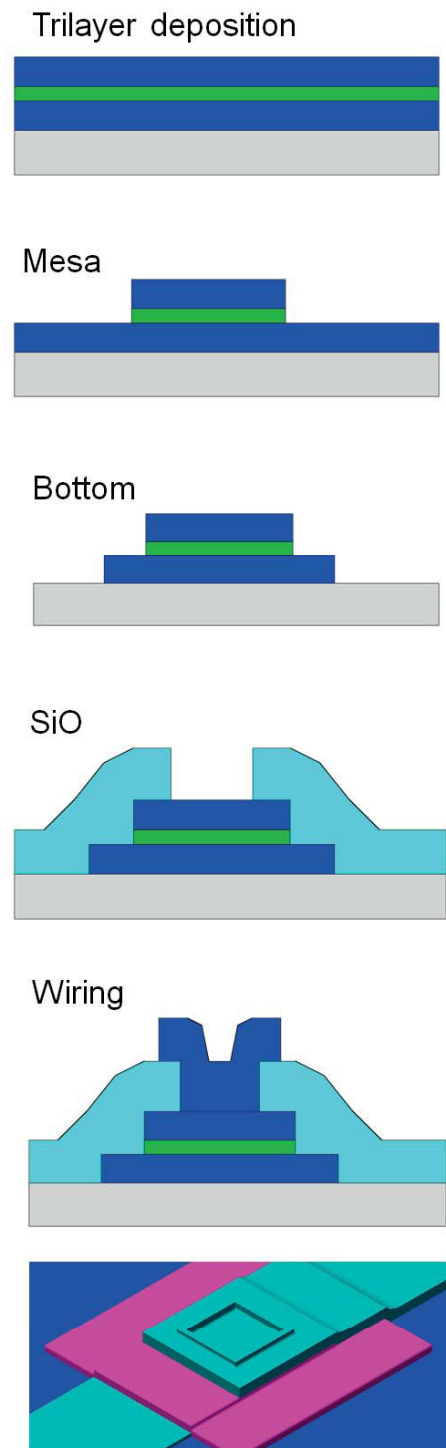


Fig. 1. Schematic representation of the manufacturing stages of the SFS Josephson junction $\text{Nb}-\text{Pd}_{0.99}\text{Fe}_{0.01}-\text{Nb}$

the question of the possibility of implementing the π -state in Josephson junctions based on PdFe has not been previously considered. This work demonstrates the reentrant nature of the critical current dependencies of junctions $\text{PdFe}-\text{Nb}-\text{PdFe}$

on the F-layer thickness and temperature, which is an indicator of the change in sign of the current-phase relation of the sample (transition to π -state).

2. EXPERIMENTAL METHODS

We studied multilayer Josephson junctions fabricated in a four-stage technological process (Fig. 1). In the first stage, Nb–Pd_{0.99}Fe_{0.01}–Nb trilayer was deposited using magnetron deposition of niobium and RF-sputtering of Pd_{0.99}Fe_{0.01}. Niobium deposition was carried out at an argon pressure of 8 μ bar, discharge current of 2 A, and power of 0.9 kW with a rate of about 7 nm/s. The deposition of Pd_{0.99}Fe_{0.01} was performed at an argon pressure of 21 μ bar and bias voltage of 1 kV. The iron content in the deposited PdFe layers was 1.1–1.5 at.% according to X-ray photoelectron spectroscopy data and approximately 1.25 at.% according to measurements using a scanning electron microscope at the Shared Research Center of ISSP RAS. The thickness of niobium layers was 150 nm, and the thickness of the PdFe layer varied within 30–50 nm. In the second stage, the top electrode and the Josephson junction barrier (mesa) were formed using plasma-chemical etching of niobium (see parameters in [24]) with a photoresist mask, as well as ion-plasma etching of the PdFe layer at an argon pressure of 12 mbar, bias voltage of 1 kV with a rate of about 1 nm/s. The mesa had a square shape with a side size of 10 μ m. In the third stage, thermal deposition of a 350 nm thick SiO insulation layer was performed, followed by lift-off photolithography. The window size in the insulation layer, open for access to the top electrode of the Josephson junction, was about 4 μ m. In the final stage, superconducting wiring was deposited followed by liftoff photolithography. At this stage, contact pads and their connection to the sample were formed.

Measurements were performed in a liquid helium ⁴He cryostat using a SQUID-based picovoltmeter. The design of the cryostat and cryogenic inserts allowed measurements at temperature $T < 4.2$ K by pumping helium vapor, as well as operation at $T > 4.2$ K, involving sample placement in a vacuum volume, temperature control using a resistive heater, and its monitoring using a calibrated thermometer. Shielding from the Earth's magnetic field and parasitic external signals during sample cooling and investigation was provided by a system of magnetic

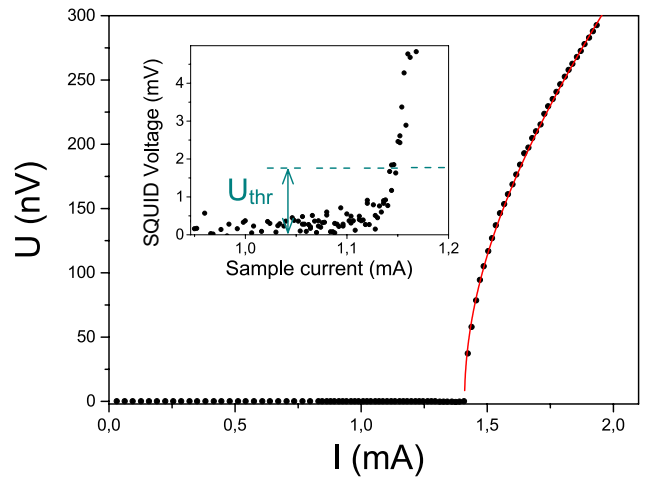


Fig. 2. Typical current-voltage characteristic of the studied samples. Black dots represent experimental data, red line shows the approximation by Josephson hyperbola $U = R_N \sqrt{I^2 - I_c^2}$. Experiment temperature 4.2 K, sample resistance $R_N = 220 \mu\Omega$. The inset illustrates the method of automatic determination of critical current when exceeding the threshold voltage U_{thr} during measurement of dependence $I_c(H)$

shields, including an outer shield made of 81HMA permalloy protecting the entire cryostat; an inner three-layer shield made of cryopermalloy protecting the internal cryostat equipment; a superconducting niobium shield directly protecting the sample holder. The magnetic field was applied parallel to the layer plane along one of the sides of the SFS mesa using a superconducting solenoid placed inside the niobium shield.

Current-voltage characteristics were measured using a 4-point scheme with a JeSEFF SQUID picovoltmeter. Current was supplied by remotely controlled Keithley 224 sources via a filter system to reduce the introduced noise. The Keithley 182 digital voltmeter was used to measure voltage on the SQUID amplifier. The gain factor was 10^6 – 10^8 and was determined by observing Shapiro steps on the control sample. The field dependence of critical current was measured using the so-called threshold method: for each value of applied magnetic field, the current-voltage characteristic was measured, and the critical current value was recorded when exceeding a preset "threshold voltage." The threshold voltage was selected empirically (see inset in Fig. 2): this value should significantly exceed the noise level of measuring equipment to exclude erroneous readings; but it should be small enough to ensure critical current measurement with a relative error not exceeding 5%. When studying

temperature dependencies of critical current density j_c a series of measurements of critical current field dependencies $I_c(H)$, was conducted for each selected temperature, since simple current-voltage characteristics measurements were insufficient (see discussion below). Temperature was stabilized using Shavrin's membrane manostat at $T < 4.2$ K, and at $T > 4.2$ K using a heater with temperature feedback. Temperature stabilization accuracy was not worse than 0.05 K during one measurement.

3. EXPERIMENT AND DISCUSSION

The main objective of the experiment is to measure the dependence of critical current density j_c on ferromagnet thickness d_F and temperature. However, the measurement of j_c itself involves certain difficulties when using PdFe alloys as a Josephson barrier, since the latter possess in-plane magnetic anisotropy and can create magnetic flux through the SFS sandwich. This property is the basis for the Josephson magnetometry method [7] (absolute fluxometry [25]), as well as the application of such junctions as Josephson magnetic memory [7–9]. The problem is that during transport experiments, the critical current I_c is measured, which represents an integral function of magnetic induction $\mathbf{B}(x, y)$ in the Josephson barrier of the form

$$I_c = \max_{\phi_0} \int_S j_c(d_F, T) \sin \left(\frac{2\pi\Phi(x, y)}{\Phi_0} + \phi_0 \right) dx dy,$$

where Φ_0 is the magnetic flux quantum, and the flux $\Phi(x, y)$ is expressed as

$$\Phi(x, y) = \int_0^x B_y(x, y) d_m dx - \int_0^y B_x(x, y) d_m dy,$$

where $d_m = 2\lambda + d_F$ is magnetic length, λ is London length, ϕ_0 is the phase difference at the origin of coordinates. The spatial distribution of magnetic induction $\mathbf{B}(x, y)$ in the Josephson barrier (including the spatial distribution of magnetization $\mathbf{M}(x, y)$) is not known in advance and may depend on mesa dimensions and magnetic history of the sample. In this sense, more convenient materials are alloys $\text{Cu}_{1-x}\text{Ni}_x$, which possess fine-domain magnetic structure with out-of-plane magnetic anisotropy [26]. When using such alloys, domain magnetizations and scattering fields are well averaged and do not

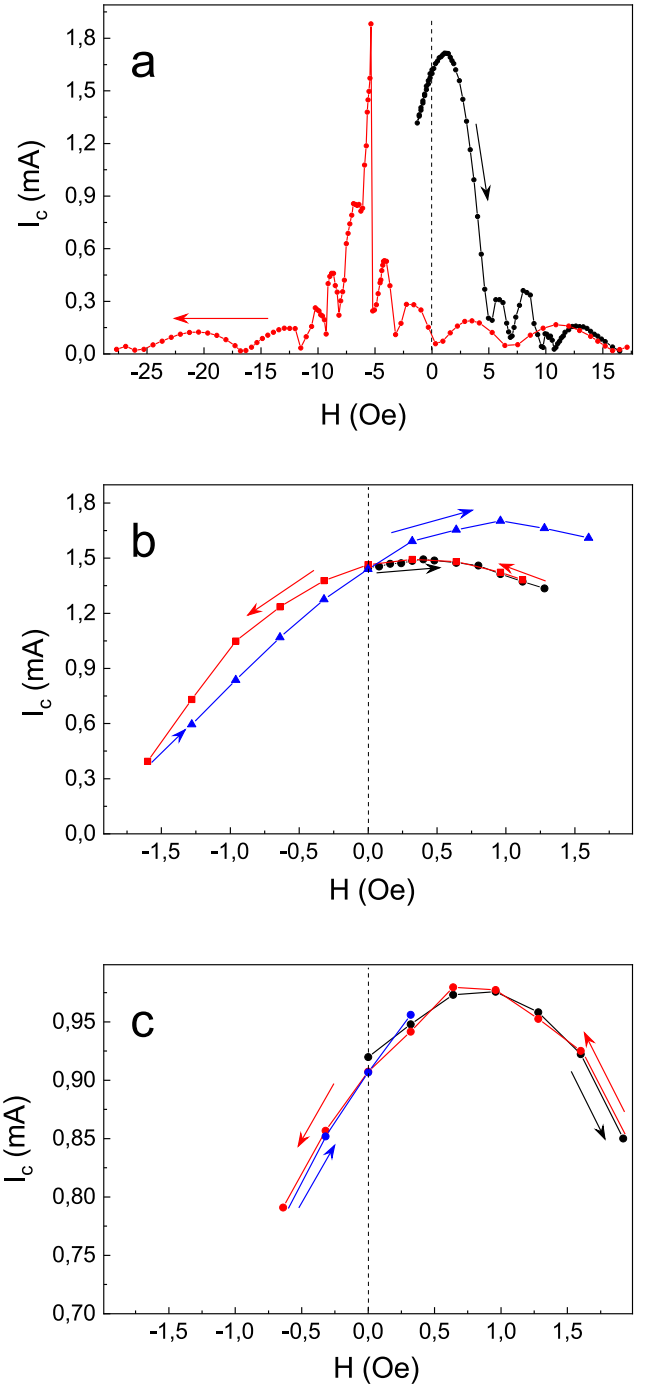


Fig. 3. Examples of critical current I_c dependencies on applied magnetic field H , used to determine the critical current density j_c using the method of "large" (a) and "small" (b, c) fields (see discussion in text). In all panels, black dots show field dependencies of critical current obtained after cooling the sample in zero magnetic field; red dots show "continuations" of black curves obtained after changing the sweep direction of field H . Blue dots in panels b, c show "continuations" of red curves obtained after repeated change in sweep direction. Experimental temperature 4.2 K (a, b) and 1.22 K (c)

create significant magnetic flux through the SFS sandwich. The latter is confirmed by the observation of Fraunhofer field dependence of critical current, independent of magnetic history [16,18]. Josephson junctions based on PdNi alloy possess similar properties [17]. This makes it possible to determine the value j_c as the ratio of critical current at $H = 0$ (determined from current-voltage characteristic) to the mesa area. Using PdFe alloys requires a more complex research procedure.

Each experiment began with cooling the sample in zero magnetic field followed by measuring the field dependence of the critical current within one magnetic flux quantum Φ_0 . The magnetic field measurement limits varied within up to 3 Oe depending on the experiment temperature or F-layer thickness (see Figs. 3b, c). With such magnetic field variation, a insignificant magnetic hysteresis was observed, which did not interfere with determining the maximum value I_c . The critical current density is defined as the ratio of the maximum critical current value to the junction area. Magnetic field was varied in positive and negative directions, which allowed demagnetizing the Josephson barrier before the next measurement. It was preliminarily verified that during sample cooling, the magnetic structure of the sample is disordered: the shape of the field dependence of the critical current is close to Fraunhofer, and the maximum occurs near $H = 0$ (see black curve in Fig. 3a).

An alternative method is measuring the dependence of $I_c(H)$ within wide limits, clearly exceeding the saturation field of the F-layer (see red curve in Fig. 3a). Due to the high labor intensity and duration of the conducted experiments, this method was used only for some samples to provide additional confirmation of the observed effects. The duration of the experiment significantly increased in cases of irreproducible changes in the field characteristic shape during measurement, which is typically interpreted as the entry of an Abrikosov vortex into one or several junction electrodes [27–32]. In such cases, the sample was warmed up and the experiment was started anew. Both methods used provide similar results (see Fig. 5), indicating sufficiently reliable determination of critical current density in the conducted experiments.

The formation of Josephson vortices (more precisely, the observation of "long Josephson

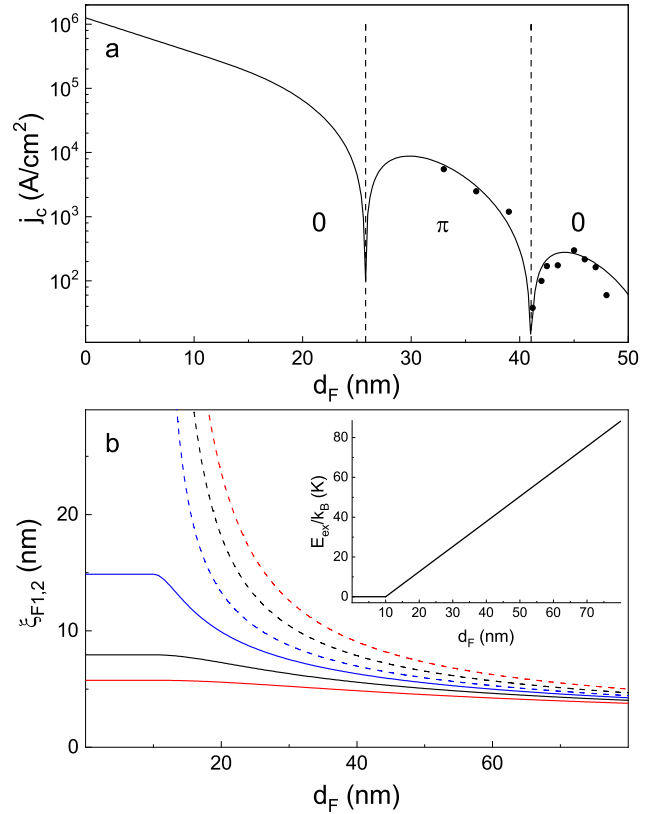


Fig. 4. *a* — Dependence of the critical current density of samples on the $\text{Pd}_{0.99}\text{Fe}_{0.01}$ layer thickness (black dots). The black line shows approximation according to equations (3) and (4). Symbols "0" and " π " mark the regions of 0- and π -states for the calculated curve. Experiment temperature 4.2 K. *b* — Dependencies calculated according to equation (3) for $\xi_{F1}(d_F)$ (solid lines) and $\xi_{F2}(d_F)$ (dashed lines) for three temperatures: 1.2 K (blue), 4.2 K (black), 8.0 K (red). The inset shows a graph corresponding to equation (2)

junction" effects [33]) is unlikely for most of the studied samples. Indeed, a Josephson junction can be considered "long" if the mesa size L is sufficiently large compared to the characteristic Josephson length $\lambda_J = (\Phi_0/2\pi\mu_0 j_c d_m)^{1/2}$, where μ_0 is the vacuum magnetic permeability and $\lambda = 80$ nm for superconducting electrodes [34]. At least, the condition $L/2\lambda_J > 1$, must be satisfied, which for a square junction reduces to a limitation on the minimum critical current:

$$I_c > \frac{2\Phi_0}{\pi\mu_0 d_m} = I_0. \quad (1)$$

Here $I_0 = 5$ mA for the chosen sample design. Since condition (1) is not satisfied for most of the studied junctions at all experimental temperatures, they can be considered "short".

Figure 4a shows the dependence $j_c(d_F)$ in the thickness range of 30–50 nm. In general, the critical current density decreases exponentially with increasing d_F , however, near the thickness of 43 nm, a reentrant behavior of the experimental curve is observed, which is one of the signs of transition between 0- and π -states. The temperature dependencies $j_c(T)$ for samples with 42 nm $< d_{\text{PdFe}} < 45$ nm are also reentrant (see Fig. 5), although the critical current for other samples increases monotonically with decreasing temperature. This effect is related to the temperature dependence of the oscillation period of the superconducting order parameter and is an additional indication of the change in the current-phase relation sign, as mentioned above (see also [5, 16, 18, 35]). The reentrant temperature dependence of the critical current is observed when using both methods of measuring the critical current density described above (compare black and red experimental points in Fig. 5).

Currently, it is impossible to definitively indicate the direction of the 0– π -transition, i.e., the sign of the current-phase relation at thicknesses less than 42 nm and greater than 45 nm. For this, it is necessary to expand the studied thickness range to detect another 0– π -transition and determine the oscillation period of the superconducting order parameter [16]. However, this involves certain technological and fundamental difficulties. In particular, to study thicknesses less than 35 nm, it is necessary to reduce the contact dimensions to 2–5 μm to limit the magnitude of measured critical currents during exponential growth j_c . This is especially important since a large bias current can affect the magnetic state of the barrier (through the created magnetic field) and the measured critical current [8]. Manufacturing small-sized samples, in turn, requires the use of advanced lithography methods and subsequent processing of deposited layers. The exponential decrease in critical current density when studying the range of $d_F > 45$ nm requires increasing the mesa dimensions above 10 μm , however, this disrupts the magnetic homogeneity of the sample [7]. A promising method is direct measurement of the current-phase relation of samples in the vicinity of the 0– π -transition [35, 5].

Some assumptions about the direction of the 0– π -transition can be made using numerical

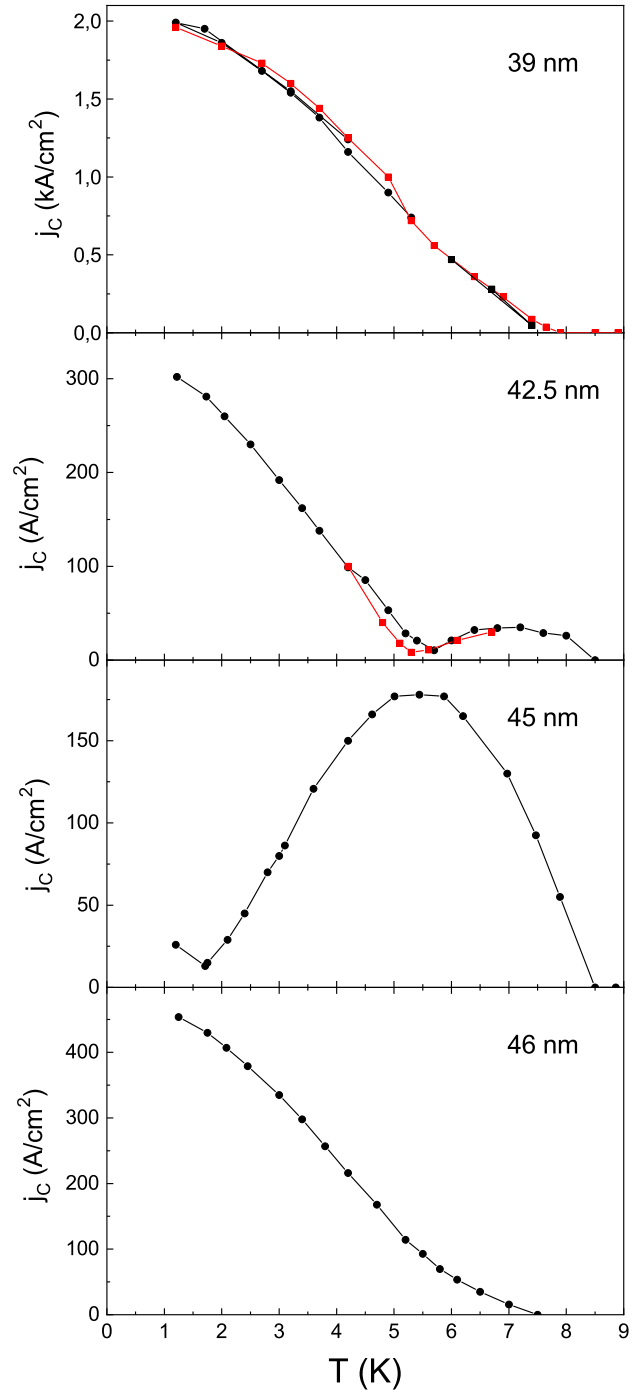


Fig. 5. Temperature dependence of the critical current density of samples for different thicknesses of the $\text{Pd}_{0.99}\text{Fe}_{0.01}$ layer. Black points are determined in small fields (see Figs. 3 b,c). Red dots correspond to large fields (see red curve in Fig. 3a)

calculations based on the data presented in work [36]. In it the dependence of the critical temperature of FS-bilayers $\text{Pd}_{0.99}\text{Fe}_{0.01}$ -Nb was investigated when changing d_F in the range up to 75 nm. When approximating the experimental curve, it

was shown that the effective exchange interaction energy increases with increasing d_F according to a linear law,

$$E_{ex}(d_F)/k_B = \alpha(d_F - d_0), d_F > d_0, \quad (2)$$

with growth coefficient $\alpha = 1.26$ K/nm and threshold thickness $d_0 = 10$ nm (see inset to Fig. 4 b). The coherence lengths $\xi_{F1,2}$ in the F-layer can be calculated [18] as

$$\xi_{F1,2} = \xi_F^* \sqrt{\frac{2\pi k_B T_c}{\sqrt{(\pi k_B T)^2 + E_{ex}^2} \pm \pi k_B T}}, \quad (3)$$

where $T = 4.2$ K is the experimental temperature and $\xi_F^* = \sqrt{\hbar D_F / 2\pi k_B T_c} = 5.6$ nm is the characteristic length in $\text{Pd}_{0.99}\text{Fe}_{0.01}$, acting as a fitting parameter. The resulting dependencies $\xi_{F1,2}(d_F)$ are shown in Fig. 4b for three temperatures corresponding (approximately) to the middle and edges of the experimentally accessible temperature range. When substituting the obtained values into the expression for $j_c(d_F)$ [16],

$$j_c = j_0 \exp\left(-\frac{d_{F1}}{\xi_{F1}}\right) \left[\cos \frac{d_F}{\xi_{F2}} + \frac{\xi_{F1}}{\xi_{F2}} \sin \frac{d_F}{\xi_{F2}} \right], \quad (4)$$

we get the black curve in Fig. 4a. It can be seen that the theory predicts the second order number of the detected $0-\pi$ -transition, while the first one should be observed at $d_F \approx 26$ nm. Assumptions about the first or third order number of the $0-\pi$ -transition at $d_F = 42$ nm either give unrealistic values of fitting parameters or disagree with experimental data. Verification of this prediction will be the subject of our further research.

4. CONCLUSIONS

Thus, in this work, we conducted a study of multilayer Josephson superconductor-ferromagnet-superconductor contacts based on superconducting niobium and weakly ferromagnetic alloy $\text{Pd}_{0.99}\text{Fe}_{0.01}$. A reentrant dependence of the critical current density on the $\text{Pd}_{0.99}\text{Fe}_{0.01}$ layer thickness was discovered at thicknesses around 43 nm, which is one of the signs of the implementation of π -state with a negative sign of the current-phase relation. This effect is confirmed by the observation of the reentrant temperature dependence of the critical current at these thicknesses. Numerical estimates predict

the second order number of the $0-\pi$ -transition, i.e., transition from π - to 0 -state with increasing ferromagnet thickness.

ACKNOWLEDGMENTS

The authors express their gratitude to V. N. Shilov, N. S. Stepanov, and D. S. Sobanin for their assistance in sample preparation and conducting experiments.

FUNDING

The work was carried out within the state assignment of the Osipyan Institute of Solid State Physics of the Russian Academy of Sciences.

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