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Andreev reflection study of the new magnetic superconductor Mo_3Sb_7: evidence for the gap anisotropy

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We report the point-contact (PC) Andreev-reflection experiments on the new paramagnetic superconductor Mo_3Sb_7 for which we have observed strong distinctions in the PC spectra measured for different contact axis orientations. Analysis of the PC spectra in the framework of the Blonder–Tinkham–Klapwijk theory has shown that the gap parameter Δ is strongly anisotropic varying over a very wide range depending on the contact orientation when the maximum Δ_{max} can exceed at least 40 times the minimum Δ_{min}. For these and other reasons we suggest that Mo_3Sb_7 is not a trivial BCS (s-wave) superconductor but rather has a $(s+g)$-wave or another unconventional pairing symmetry.

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The examination of the Mo–Sb system indicates that only one intermediate phase is formed. This is the compound Mo_3Sb_7 which crystallizes with the $D8f$ (Ir_3Ge_7) type structure, space group $Im3m$. Mo_3Sb_7 has a cubic cell, but Sb occupies two nonequivalent positions: Sb_1 in 12(d) and Sb_{11} in 16(f) [1]. It means that there are two Sb sublattices, and the Fermi surface, electronic structure and quasiparticle interactions may be quite complicated. This provides expectation of some interesting and unusual properties of Mo_3Sb_7.

Recently, Bukowski et al. [2] synthesized such a Mo based compound, Mo_3Sb_7, and carried out the resistivity and magnetic susceptibility measurements according to which this material became superconducting with the onset of transition at $T_c^{on} \simeq 2.2$ K. So far the Mo based superconductors have been only presented by the well known Chevrel phase molybdenum chalcogenides $M_xMo_6X_8$ (M is metal, X = S or Se, $1 \le x \le 4$) with a complicated magnetic structure. In these compounds, the superconductivity coexists easily with the antiferromagnetic order or even with the ferromagnetic one in a narrow temperature interval (see, for example, [3]). However, the Mo_3Sb_7 compound demonstrates no magnetic transitions and keeps the paramagnetic behavior in a wide temperature range [2].

In the present work, we investigated the Andreev-reflection point contact (PC) spectra ($dV/dI(V)$) of Mo_3Sb_7 with the aim of finding the superconducting gap parameter Δ and its temperature dependence $\Delta(T)$. (Ag was used as a counterelectrode.) It appears that the $\Delta(0)$ value measured in different directions of the Mo_3Sb_7 sample varies within a wide range 0–0.32 meV (the corresponding characteristic ratio $2\Delta(0)/kT_c^{on} \simeq 0$–3.2), whereas the BCS isotropic value $\Delta^{BCS}(0)$ should be about 0.35 meV. The temperature dependence of the

maximum Δ value was found to be very close to that followed from the BCS theory. For contacts with smaller Δ, the deviation of $\Delta(T)$ from the BCS law is enhanced with Δ decreasing. The very low values of Δ obtained for some PC axis orientations imply the existence of gap function nodes (or lines of nodes) in some regions of the Fermi surface. The PC spectra for such contacts prove to be much narrower than one could expect for conventional superconductors with the *s*-wave pairing type. Therefore, we suggest an unconventional type of the Cooper pairing in Mo_3Sb_7.

The Mo_3Sb_7 crystals were prepared via the peritectical reaction between the liquid Sb and solid Mo in the Ar atmosphere. The crystal structure and the chemical composition were examined by the x-ray and electron diffraction methods. The PC measurement was made on small (1–2 mm) shiny crystalline pieces of irregular shape and appreciable roughness. Some of them were cleft to avoid the surface contamination of the contact area. Either the native (without any chemical or mechanical treatments) or cleft surfaces were probed with a sharpened Ag electrode etched chemically just before placing into a He cryostat. A standard modulation technique operated at 437 Hz was used to register the $dV/dI(V)$ characteristics. The electrode holding and driving mechanism controlled outside of the cryostat provided multiple probing of different S-electrode points per one measurement cycle. Evidently, to avoid the heating effects in the point contacts (i.e., to be in the spectroscopic regime), the size of contact a should be less than the diffusive mean free path of electrons $l_\varepsilon = (l_1 l_2)^{1/2}$ (l_1 and l_2 are the elastic and inelastic scattering lengths, respectively). In this case, the electron flow through the interface may be ballistic (i.e., without any scattering) if $a < l_1$ or diffusive if $l_1 < a < l_\varepsilon$ [4,5].

A fairly high and stable (up to voltage biases $V \geq 10$ mV) Andreev-reflection current was observed in the point contacts with normal state resistances R_N within approximately 1–10 Ω. Therefore, the contacts with $R_N \simeq 1\,\Omega$ occurred to be in a spectroscopic regime. Generally, the upper limit of a can be estimated from the Maxwell formula $a = \rho_0/2R_N$ [6] valid for the diffusive regime (see, e.g., Refs. 4 and 5). However, assuming the residual resistivity value $\rho_0 = 95$ $\mu\Omega\cdot$cm found for Mo_3Sb_7 in [2], one would get for the 1 Ω contact a equal to about 500 nm, which undoubtedly cannot be consistent with the spectroscopic regime. We suggest that this contradiction is probably associated with a poor uniformity of the sample measured. The dc resistivity measurements from which the residual resistivity mentioned was obtained mirror the averaged over volume properties of samples, which may contain the regions with a possible deterioration of the structure, stiochiometry, etc. Indeed, the magnetic susceptibility measurements performed on the same sample, which was used in the dc resistivity measurements, showed that the diamagnetic response was smeared over a wide temperature range (no less than 0.5 K). This indicates that the distribution of the superconducting properties over the sample volume is not homogenous. The same is true for the normal state properties.

Therefore, we suggest that ρ_0 cited above is not the inherent characteristic of our samples and strongly varies over a sample volume. For example, $\rho_0 = 0.5$ $\mu\Omega\cdot$cm was measured for the isostructural compound Ru_3Sn_7 [2], which is closely reminiscent of that for a very good metal such as Ag. With using the latter ρ_0 value, a would be about 200 times smaller than that presented above. For investigation, we selected the point contacts with a detectable Andreev-reflection current and T_c close to 2.25 K found in [2] for Mo_3Sb_7. Our PC experiments unambiguously show that the ρ_0 value for separate Mo_3Sb_7 regions, where reliable and reproducible PC spectra could be registered, should be much less than 95 $\mu\Omega\cdot$cm.

When probing different directions of the Mo_3Sb_7 electrode we have revealed that the gap parameter Δ varies in a wide range from about zero to a value slightly exceeding of 0.3 meV in spite of the same $T_c^{\rm on}$ registered in the corresponding contacts. In the following, we will consider three distinctive types of PC spectra representing the contacts with maximum and minimum Δ values as well as an intermediate case.

Figure 1 presents a typical temperature set of the PC spectra $dV/dI(V)$ for one of the contacts for which $\Delta(0)$ is found to be close to the maximum measured value of 0.32 meV. Considering the spectra in a wider voltage range than that presented in this figure, one can infer that the Andreev-reflection current is not reduced up to $V \sim 10$ mV, suggesting that these contacts are spectroscopic in character. The small humps seen in the 2.2 K- and 2.25 K-curves are indications of weak depression of the superconductivity in the PC area due to the current injection. As evident from Fig. 1, the superconductivity in this contact arises slightly below 2.3 K, which can be taken as the onset critical temperature $T_c^{\rm on}$. In the other contacts, $T_c^{\rm on}$ could be somewhat lowered down to about 2.23 K but always remained in the range noted. The procedure of finding the gap parameter Δ was based on the Blonder–Tinkham–Klapwijk (BTK) theory [7] which describes adequately the processes of Andreev reflection in N–S point contacts.

We have fitted the experimental curves to the modified BTK formulas [8] which take into account the quasiparticle life-time limitation [9] that results in a

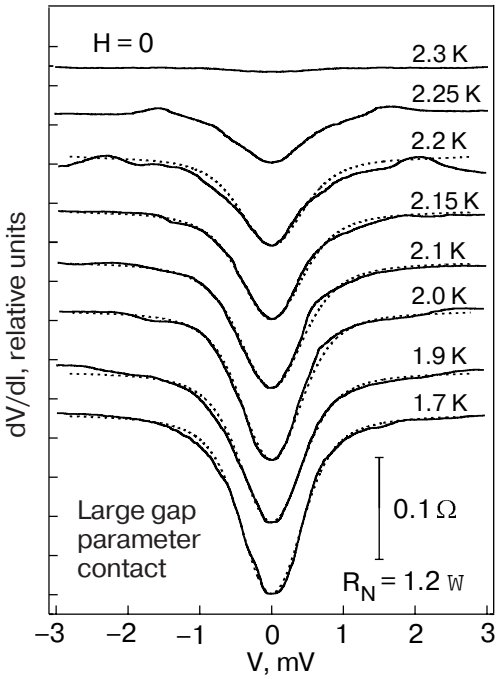

Fig. 1. The point-contact spectra $dV/dI(V)$ (solid lines), measured at temperatures indicated in each curve, of the Ag–Mo_3Sb_7 contact characterized by a relatively large gap parameter value $\Delta(0) \simeq 0.32$ meV. The BTK fits are shown by the dashed lines. For clarity, the curves are shifted vertically.

broadening of the experimental curves. The latter phenomenon is characterized by the broadening parameter Γ. It should be noted that Γ may also reflect some possible inhomogeneous distribution of Δ over the contact area. To ensure the proper fitting, the formulas incorporate the barrier strength parameter Z as well. This parameter describes both a possible scattering at the electrode boundary due to the presence of oxides, defects, etc. and a decrease in the interface transparency caused by the discrepancy of the Fermi parameters which generally determine the residual resistivity ρ_0. The fit of the experimental data to the theory as regards the three fitting parameters Δ, Γ, and Z was performed only for the curve recorded at the lowest temperature. For fitting the higher temperature curves, Z was kept unchangeable because the barrier scattering may not depend significantly on temperature in a narrow temperature range (< 2.3 K). The fitted curves are shown in Fig. 1 by the dashed lines.

Noticeably, none of the spectra measured exhibited a double minimum gap structure typical of N–S contacts based on conventional *s*-wave superconductors in the case of nonzero Z and the lack of any smearing effects, i.e., when $\Gamma = 0$ [7]. For the spectra presented in Fig. 1, Z was found to be about 0.2. One of the probable reasons of this situation is that the residual resistivity ρ_0 in the Mo_3Sb_7 electrode is rather low what dictates the comparatively small Z. As it follows from [8], the double minima cannot be observed for Z exceeding significantly 0.2 if the ratio $\Gamma/\Delta \neq 0$. Evidently, the suppression of the double minimum structure would be intensified essentially if the effects of temperature and modulation smearing of the PC spectra were taken into account what has not been done in [8]. Another possible reason may be resulted from the specific features of the Andreev reflection in the case where the S electrode in the N–S contact is a nontrivial superconductor. Recently, we observed a similar shape of the PC spectra in contacts based on $PrAg_6In_6$ where the *d*-wave pairing was suggested relying on the observation of a pseudogap structure [10].

The temperature dependence of the gap parameter Δ calculated via BTK fitting for the contact presented in Fig. 1 is shown in Fig. 2 by the solid squares. For this contact $\Delta(0) \simeq 0.32$ meV, if the BCS temperature dependence of the gap parameter (solid line in Fig. 2), is used which results in the characteristic ratio $2\Delta(0)/kT_c^{on} \simeq 3.2$. This is quite close to 3.52 expected from the BCS theory for weak-coupled superconductors. A small discrepancy between experimental and theoretical $\Delta(T)$ dependences could be somewhat less if the temperature corresponding to the middle point of a superconducting resistivity transition would be used instead of T_c^{on}. As it follows from the BTK fit for the spectra presented in Fig. 1, the broadening parameter Γ value changes with increasing temperature in the moderate interval from 0.1 meV at 1.5 K to 0.25 meV in the vicinity of T_c. This interval seems to be quite reasonable taking into account the magnetic character of Mo_3Sb_7 (Pauli paramagnet) and some possible violations in the crystal structure which may occur in the contact area.

The PC spectra measured at different temperatures for the contact with an intermediate Δ value ($\Delta(0) \simeq$ $\simeq 0.15$ meV) are shown in Fig. 3 by the solid lines. The temperature dependence of Δ found as a result of

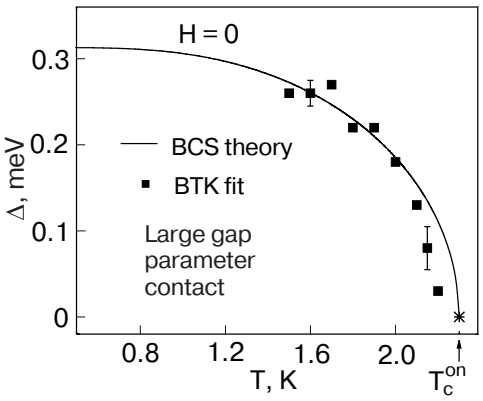

Fig. 2. The temperature dependence of the gap parameter $\Delta(T)$ (solid squares) as compared to the BCS theoretical curve (solid line) for the contact presented in Fig. 1.

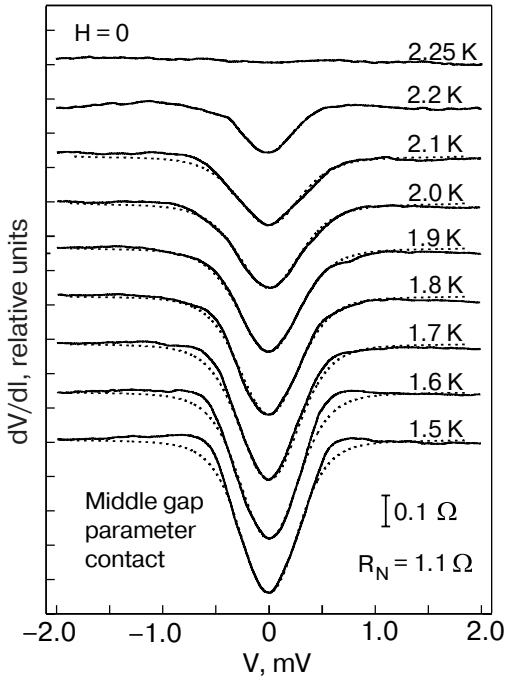

Fig. 3. The temperature set of the PC spectra (solid lines) for the Ag–Mo_3Sb_7 contact with a moderate gap parameter value $\Delta(0) \simeq 0.15$ meV. The dashed lines present the BTK fits.

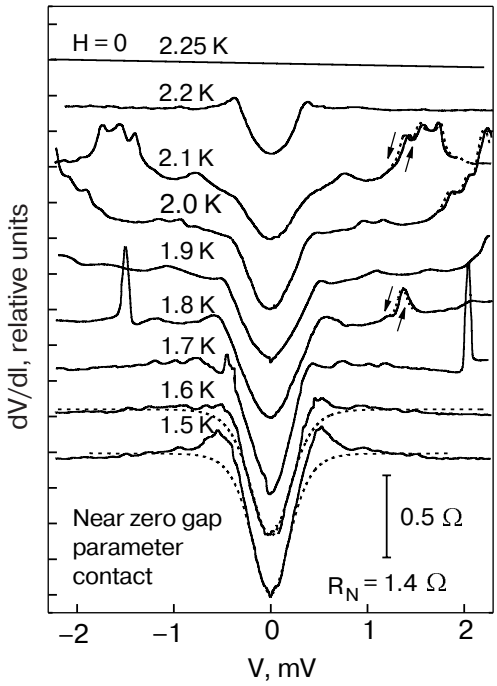

Fig. 5. The PC spectra (solid lines) of the Ag–Mo_3Sb_7 contact is characterized by the extremely small broadening and very small gap parameter value $\Delta(0) \simeq 0.008$ meV. Due to an unusual fast narrowing of the zero bias minimum with temperature increasing, the proper BTK fits could be done only for the 1.5 K- and 1.6 K-curves (dashed lines).

the BTK fitting of the spectra (the dashed lines in Fig. 3) demonstrates the significant deviation from the BCS theory (Fig. 4) that was not the case for the contact with the maximum $\Delta(0) \simeq 0.32$ meV (Figs. 1 and 2). The best BTK fitting of these spectra was realized with the same barrier parameter $Z = 0.2$ as for the spectra presented in Fig. 1. Surprisingly, the PC spectra in Fig. 3 turn out to be much less smeared than those presented in Fig. 1 for the contact with the maximum Δ. Really, for the spectra in Fig. 3 the broadening parameter Γ was found to vary from 0.02 meV to 0.04 meV with rising temperature which is approximately by a factor of 5 less than that for the previous spectra. This points to the fact that the ratio Γ/Δ increases when Δ decreases.

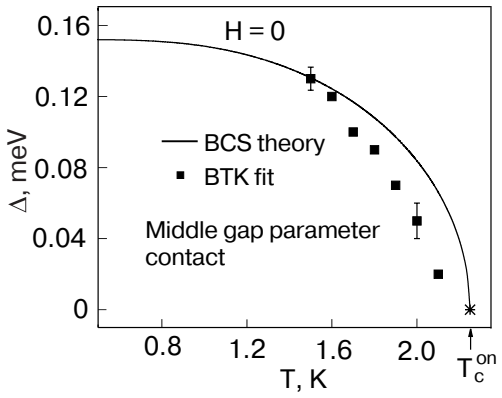

Fig. 4. The $\Delta(T)$ dependence (solid squares) and that expected from the BCS theory (solid line) for the contact presented in Fig. 3.

The last, third type of the PC spectra measured at different temperatures is presented in Fig. 5. As was derived from the BTK fitting, these spectra are characterized by the close to zero broadening parameter Γ and extremely small gap parameter Δ. The $dV/dI(V)$ minima near $V = 0$ appears as those produced by the destruction of superconductivity due to exceeding the critical current in the contact area. However, the lack of the hysteretic behavior and the smooth character of the minima structure indicate that this is not the case. In these spectra (Fig. 5), the local suppression of superconductivity manifests itself as spikes arising first in the 1.7 K-curve near $V = 2.0$ mV and shifting to smaller voltage biases with increasing temperature. Noteworthy, the most spectra of the third type do not exhibit such spikes. In Fig. 5, the spectra with spikes are chosen with the aim of proving the spectroscopic character of the $dV/dI(V)$ minima observed near $V = 0$ for the contacts of the third type. The obvious current destruction effects were observed only for the contacts with $R_N < 0.1\ \Omega$ as the hysteretic behavior of $dV/dI(V)$ in the vicinity of $V = 0$.

Performing the BTK fits of the PC spectra in Fig. 5, we set $Z = 0.2$ which was found for the contacts of the first and second types whereas for Γ a quite close to zero value was required. The reliable fits were made only for the 1.5 K- and 1.6 K-curves (the dashed lines in Fig. 5) that allowed us to obtain $\Delta(0) \simeq 0.008$ meV. For the 1.7 K-curve, we managed to establish in an acceptable way the upper limit of Δ only, because the inherent temperature broadening of the gap function according to the BCS theory appeared to be noticeably larger than that observed in our measurements for the contacts of the third type. This conclusion can be verified by comparing the theoretical BTK curves calculated for several temperatures (1.0, 1.7, and 2.1 K) with the experimental PC spectrum measured at $T = 2.1$ K (Fig. 6). As seen from Fig. 6, the theoretical 2.1 K-curve (the dashed line) cannot describe adequately the experiment (the solid line) even if we set $\Gamma = 0$. A more or less reasonable agreement with the experimental curve can be achieved if we put $T = 1.7$ K in the BTK formulas (the dotted line). The theoretical 1.0 K-curve (the dash-dotted line) demonstrates the rate of decreasing the BTK spectrum width with decreasing temperature. The results of the Δ calculations on the basis of the BTK fitting of the experimental 1.5 K-, 1.6 K-, and 1.7 K-curves (Fig. 5) are presented in Fig. 7 by the solid squares. A deficiency of Δ data did not allow the expected BCS dependence $\Delta^{BCS}(T)$ to be depicted correctly. In order to plot somehow this dependence in Fig. 7 we brought it into coincidence with the 1.5 K-data deduced from the BTK fit. In spite of the very arbitrary way of plotting the BCS curve, the large discrepancy between experiment and theory for the third type of contacts is evident.

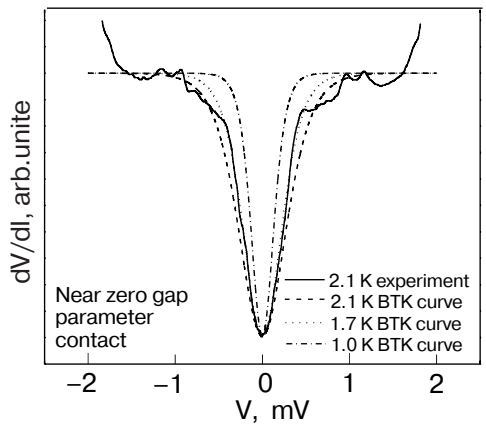

Fig. 6. The 2.1 K spectrum taken from Fig. 5 (solid line) together with the BTK curve (dashed line) calculated for negligibly small $\Delta = 0.0002$ meV and $\Gamma = 0$. The BTK curve together with other hypothetical 1.0 K- and 1.7 K-BTK curves calculated with the same Δ and Γ demonstrate the expected temperature change of the PC minimum width for conventional BCS (*s*-wave) superconductors.

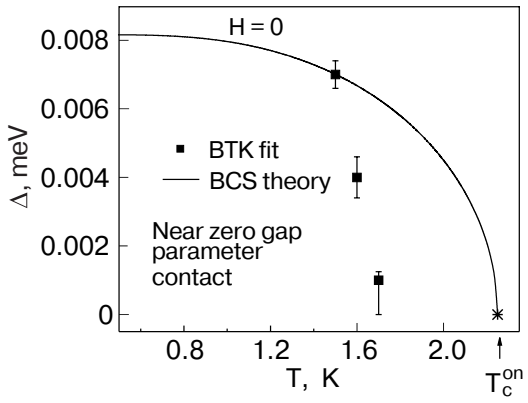

Fig. 7. The gap parameter values found via the BTK fitting for the 1.5 K-, 1.6 K-, and 1.7 K-PC spectra (Fig. 5) plotted against temperature for the contact with a very small Δ (solid squares). The BCS curve (solid line) is fixed to the 1.5 K experimental data.

The data presented above undoubtedly evidence that the gap function varies over crystallographic directions of Mo_3Sb_7 and hence has a non-*s*-wave pairing symmetry but rather some unconventional one (for example, the $s + g$ symmetry). The strong variations of Δ (not less than 40 times) with changing the contact orientation regardless of T_c keeping unchanged (with an accuracy of a few 0.01 K) prove this suggestion. Indeed, if the changes of Δ were resulted from the deteriorated areas on the S-electrode surface, the strong correlation between Δ and T_c should be observed. Such the correlation was really observed in [11] where tunneling into the normal side of the sandwich Ag/Pb was studied. Because both films were deposited at a pressure of $\sim 5 \cdot 10^{-7}$ Torr within 20–30 s, only a very thin absorbed layer could be formed at the Ag/Pb interface that ensured the good metallic contact between films and, therefore, very high transparency of the interface. Meanwhile, in the case of arising significant potential barrier separated N and S layers, T_c measured at the N side of bilayer persists at the level typical for the S side in spite of a strong reduction of Δ as it was found in another tunnel experiments [12]. In this work, the electron transmission probability for the bilayer occurred to be much smaller than unity due to the poor vacuum in the setup used for the deposition of films.

We do not rule out the possible presence of some deteriorated layer with the depressed superconducting parameters on the Mo_3Sb_7 surface though its thickness should not be significant if to take into attention the results of sample examinations. However, an appearance of any barrier between this layer and the unspoilt base was not possible. So, even in the case of the presence of relatively thick deteriorated layer, the genuine Δ feature in PC spectra relevant to the native material should be seen at some reduced voltage [13].

This is due to the fact that incoming from the N electrode quasiparticles ranged within the $\Delta - \Delta_{ind}$ (Δ_{ind} is gap induced in the deteriorated layer) energy interval can attain the native region of Mo_3Sb_7 and undergo there the Andreev reflection. In this case, an appearance in PC spectra of another feature associated with Δ_{ind} cannot distort significantly the information about a true Δ value, especially if the quasinormal layer is thin enough [13] that, in principle, could happen in our experiments.

We believe that point contacts of the first type (like those presented in Figs. 1 and 2) were probably oriented along one of the gap function maxima in the k space and are featured by the maximum gap parameter value $\Delta(0) \simeq 0.32$ meV derived from the classical BTK fitting. For these contacts, the temperature dependence of Δ is in close agreement with that expected from the BCS theory (Fig. 2). Qualitatively, this agreement may be explained by the fact that the portions of the k space with maximum gap function amplitudes should have some analogy to those for s-wave type superconductors. Under this suggestion, the axis of the third type contacts (Figs. 5–7) should have been approximately oriented along a nodal direction where the superconducting gap is absent. It is worth of note that even for the total coincidence of both orientations, Δ will not have a zero value due to a not very high angle selectivity of the PC spectroscopy method which detects electrical characteristics spread over some space angle although the main contribution should come from the nodal direction with the zero gap. A very fast decrease of Δ with increasing temperature (Fig. 7) may be associated with the occurrence of gapless states in the vicinity of the nodal directions. The extremely small width of the third type PC spectra (Fig. 5) indicates that in these contacts the nodal area was really probed. Assuming that the gap function in Mo_3Sb_7 is anisotropic, we may associate the PC spectra of the second type (Fig. 3) with the contacts whose axis was located in between the nodal direction and the direction corresponding to the gap function maximum. Accordingly, the temperature dependence of $\Delta(T)$ (Fig. 4) shows a moderate deviation from the BCS theory.

Recently, a strong anisotropy of the superconducting energy gap with the ratio $\Delta_{max}/\Delta_{min} \sim 4.5$ was observed in the single crystals YNi_2B_2C by means of the directional PC spectroscopy [14]. Meanwhile, studying the thermal conductivity in this material showed that the ratio indicated could be > 100 [15]. The data obtained provide evidence for the $s + g$ symmetry of the gap function [16] in YNi_2B_2C. In our work, the ratio $\Delta_{max}/\Delta_{min} \simeq 40$ was found for Mo_3Sb_7. It was also revealed that another ratio, Γ/Δ, increased with decreasing Δ in a similar way as for YNi_2B_2C [14]. Furthermore, the same tendency of changing the sign of curvature for the $\Delta(T)$ dependence as a temperature goes to T_c was observed (compare Fig. 3 from [14] and Figs. 2 and 4 from the present work). The common characteristic features in the behavior of Δ for both the well studied superconductor YNi_2B_2C and the novel superconductor Mo_3Sb_7 imply that a probability of the unconventional gap function with the $s + g$ like symmetry in the latter superconductor is quite high. It should be mentioned that in our experiments the large Δ values were observed more often than the minimum ones. This is additional evidence for the $s + g$ like gap symmetry because in this case $\Delta(k)$ is flat in the vicinity of poles and hence can be measured much frequently. Meanwhile, it was much more difficult to get the contacts oriented along the nodal directions (i.e., corresponding to the minimum Δ values) due to the sharp fall of $\Delta(k)$ near the nodes.

In conclusion, we have investigated the point contacts based on the new paramagnetic superconductor Mo_3Sb_7 and found strong evidence for the significant gap parameter Δ anisotropy in this material. The maximum Δ_{max} found for a particular direction of the contacts was at least 40 times larger than the minimum Δ_{min} found for another direction of the contacts. These and other facts enable us to suggest that Mo_3Sb_7 is not a conventional s-wave superconductor, but rather has the $s + g$ unconventional pairing symmetry. The PC spectra with Δ_{max} are associated with the contacts directed along the gap function maxima, whereas Δ_{min} can be ascribed to the contacts with a nodal orientation where the superconducting gap is absent.

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