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Andreev reflection study of a new magnetic superconductor Mo_3Sb_7 in magnetic field

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Andreev reflection in contacts based on a magnetic superconductor Mo_3Sb_7 in magnetic field has been investigated by the point-contact method. It is found that the behavior of the order parameter in the magnetic field $\Delta(H)$ is essentially dependent on the value of Δ, which varies widely ($\Delta(0,0) \approx 0.01-0.31$ meV) for different contacts at practically invariant T_c. At high Δ the dependence $\Delta_{max}(H)$ deviates slightly from the theoretical prediction applicable under the point-contact conditions. As the order parameter decreases, the deviation starts to increase and becomes very large when Δ approaches its minimum. Proceeding from the results obtained in this study and from the temperature measurements on Mo_3Sb_7 performed formerly, we are inclined as before to assign the compound to the class of superconductors with anisotropic gap function. So, in no way can it be considered as conventional BCS-type superconductor. The upper critical field $H_{c2} \simeq 16.5$ kOe found here is close to that obtained from magnetization measurements in another study. According to the estimation performed, the pair-breaking effect of the Pauli paramagnetism is rather weak in Mo_3Sb_7.

PACS: 74.70.Ad Metals; alloys and binary compounds (including A15, MgB_2, etc.).

Keywords: magnetic superconductor, point-contact method, Andreev reflection.

Introduction

Recently the family of superconducting Mo-based compounds has been enlarge by a new member, Mo_3Sb_7. This compound was synthezied by Bukowski et al. who also measured its resistive and magnetic properties [1]. According to their results, the superconducting transition in Mo_3Sb_7 starts at $T \simeq 2.2$ K. Mo_3Sb_7 has a cubic $D8f$ (Ir_3Ge_7)-type crystal lattice (space group $Im3m$) in which Sb can hold two nonequivalent positions in the cubic cell: Sb_1 at the site 12(d) and Sb_{11} at the site 16(f) [2]. Mo_3Sb_7 is the only intermetallic compound in the Mo–Sb system. This largely determines the single-phase character of the samples despite their somewhat exotic preparation by the peritetic reaction of the structural components. Before the advent of Mo_3Sb_7, the superconducting Mo-based compounds were widely represented by the Chevrel phases $M_xMo_6X_8$ (M — metal, X = S or Se, $1 \le x \le 4$) having a complicated magnetic structure. In these compounds superconductivity coexists tolerantly with antiferromagnetism in a wide temperature range or even with ferromagnetic order, though in a very narrow temperature interval [3]. The new superconductor Mo_3Sb_7 is free of any magnetic transitions and remains paramagnetic in the investigated interval from room temperature to the temperature of the superconducting transition [1].

We thoroughly investigated the basic superconducting characteristics (order parameter Δ and upper critical field H_{c2}) of a new magnetic superconductor Mo_3Sb_7 using the point-contact method. This is the most efficient technique for superconductors with complicated crystal structure enables one to obtain spectroscopic information from

small-volume areas of different crystallographic orientation. These results were partly published in Refs. 4, 5. The point-contact spectra of the Andreev reflection in metallic (no dielectric barriers) Ag–Mo_3Sb_7 contacts, measured at different temperatures, are presented in Ref. 4. They are seen to vary considerably from contact to contact. The matching of the experimental data and the modified Blonder–Tinkham–Klapwijk (BTK) theory, which most adequately accounts for the Andreev reflection in N–S contacts, shows that the order parameter Δ is strongly anisotropic and varies widely depending on the contact orientation: $\Delta_{\max}$ can be up to 40 times in excess of $\Delta_{\min}$, in spite of practically unchanged T_c. The dependence $\Delta_{\max}(T)$ agrees closely with the BCS theory, while $\Delta_{\min}(T)$ diverges from it drastically. These results suggest that Mo_3Sb_7 is not a conventional BCS superconductor with the s symmetry of the Cooper pairing. Most likely, it has an anisotropic gap function.

Here we present the experimental dependences of the order parameter on the magnetic field $\Delta(H)$ and of the upper critical field on temperature $H_{c2}(T)$ that were measured on the magnetic superconductor Mo_3Sb_7. The preliminary data on $H_{c2}(T)$ are partly available in Ref. 5. The data analysis shows that the dependence $\Delta(H)$ for contacts with rather large Δ is comparable with that following from the Ginzburg–Landau theory within some reasonable assumptions. However, there is a considerable discrepancy between theory and experiment for contacts with low Δ. In both cases the behavior of $\Delta(H)$ suggests the existence of a narrow gap-free region near H_{c2}. H_{c2} was obtained from the dependences $dV/dI(H)_{V=0}$ measured at different temperatures. It varies within 7.8–9.2 kOe at 1.6 K for different contacts and does not correlate with Δ. This means that H_{c2} is independent of the crystallographic orientation. The order parameter has such dependence, which was demonstrated convincingly in Ref. 5. The dependence $H_{c2}(T)$ matched to the empirical law $H_{c2}(T) \sim 1-(T/T_c)^2$ leads us to conclude that Mo_3Sb_7 is a weak Pauli paramagnet.

Experiment

The Mo_3Sb_7 crystals were prepared in the process of the peritectical reaction between liquid Sb and solid Mo in an inert Ar atmosphere at $T = 1000$ °C. The crystalline structure and the chemical composition were examined using x-ray and electron-diffraction methods, which showed a perfect quality of the samples and no evidence of impurities and foreign phases. The point contacts were made in liquid He by touching gently a fresh cleaved facet of the Mo_3Sb_7 polycrystalline sample (prepared immediatelly before placing it into the cryostat) with a chemically etched sharpened Ag wire. Note that the spectroscopic characteristics of the cleaved surface remained practically unchanged even after a prolonged exposure to air. Moreover, in many cases the results measured on contacts formed on a natural surface of the sample and on a freshly cleaved facet were closely similar. In both cases the experiments were made on irregular, rather rough pieces 2–3 mm in section. Owing to the large (~ 20–50 μm) crystallites in the Mo_3Sb_7 polycrystals, there was a high probability that the Ag needle would touch single-crystalline regions of the S electrode, though their crystallographic directions with respect to the contact axis were unknown. In this way we performed directional spectroscopy of the Andreev reflection in Mo_3Sb_7 and could watch different (varying more than fortyfold) order parameters in different contacts at practically invariant T_c. Thus, we have concluded that the pairing in Mo_3Sb_7 is not related to the s-type symmetry [5].

The design of the electrode holder allowed the relative movement of the electrodes under an off-cryostat control. It was thus possible to touch repeatedly different points on the S-electrode surface during one measurement cycle. The point-contact spectra $dV/dI(V)$ and $dV/dI(H)$ were measured using the standard modulation technique at 487 Hz frequency. The magnetic field was produced with a superconducting solenoid and was always perpendicular to the contact axis. To maintain the spectroscopic regime, the size of the contact must be smaller than the mean scattering path of charge carriers [6,7]. Unfortunately, because of the lack of information about the electronic parameters of this new superconductor, we could not estimate adequately the size of the contact and compare it with the scattering length. Nevertheless, the high and steady Andreev reflection currents observed up to biases over an order of magnitude exceeding the typical gap-related voltages in the point contacts with normal-state resistance R_N = 1–10 Ω count in favor of a stable spectroscopic condition in them. Contacts with higher resistance are less suitable for investigation as they show worse stability against vibrations during several hours of a complete measurement cycle.

Results and discussion

The measurements of point-contact spectra at different points of the S electrode show that the order parameter Δ can change over an order of magnitude from contact to contact. The critical temperature found from the onset of the superconducting transition T_c^{on} remained within 2.2–2.3 K. The highest Δ reduced to zero temperature in accordance with the BCS theory was $\Delta_{\max}(0) \simeq 0.31$ meV, which provides the characteristic ratio $2\Delta_{\max}(0)/kT_c \simeq 3.2$. This is a little lower than Δ expected in the isotropic BCS model $\Delta^{BCS}(0) \simeq 0.35$ meV. The discrepancy may be due to the effects of pair breaking in this magnetic superconductor. The typical set of spectra ($dV/dI(V)$ characteristics) measured on the same point contact with high Δ in different magnetic fields is shown in Fig. 1 (solid lines).

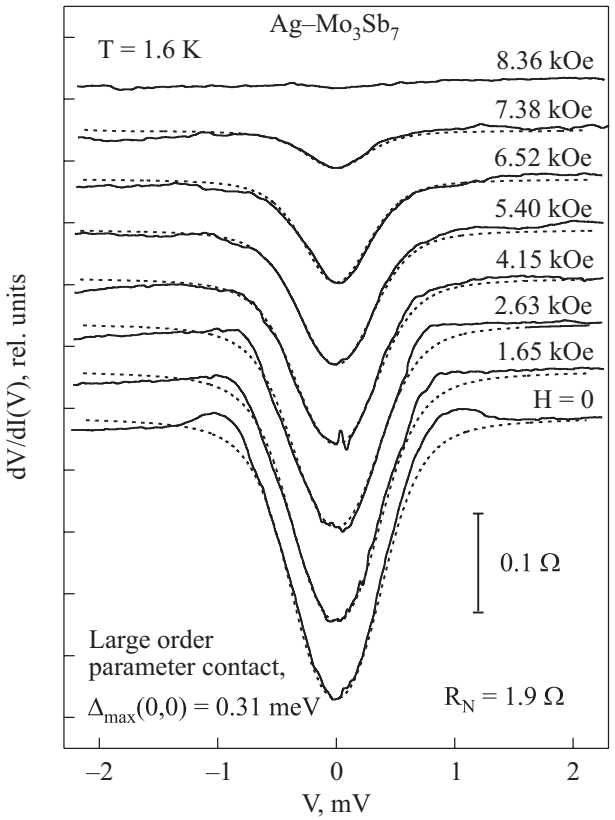

Fig. 1. Point-contact (PC) spectra $dV/dI(V)$ (solid lines) measured in different magnetic fields, indicated on each curve, of an Ag–Mo_3Sb_7 contact characterized by a relatively large order parameter value $\Delta(0) \simeq 0.31$ meV. The BTK fits are shown by the dashed lines (Z = 0.15, $\Gamma \approx 0.02$ meV). For clarity, the curves are shifted vertically.

According to the BTK theory [8] modelling electron scattering at the N–S boundary, the minima in spectra near $V = 0$ in magnetic fields smaller than 8.36 kOe are due to the processes of Andreev reflection in the point-contact area (Fig. 1). The magnetic field in which an appreciable dip first appears in spectrum was taken as the upper critical field H_{c2} ($H_{c2} \approx 8.3$ kOe for this contact). Δ was found by fitting the formulas of the modified BTK theory [9] to the experimental $dV/dI(V)$ curves. This theory allows for the spectral line broadening near $V = 0$ through the so-called smearing parameter Γ which was first introduced by Dynes et al. [10] to account for the finite lifetime of quasiparticle excitation at the Fermi level. It is important that in actual practice the parameter Γ can also include other factors, primarily, the inhomogeneous distribution of Δ over the contact area and the effects of pair breaking caused by spin-flip scattering of electrons. Besides, the BTK theory contains another important parameter Z describing the elastic electron scattering directly at the N–S boundary. This scattering is caused by defects of the surface layers of both the electrodes and by the discrepancy between their Fermi momenta. Noticeably, the original BTK theory is strictly applicable only for s-wave superconductors, whereas in a case of d-wave symmetry the extended BTK should be used [11]. Meanwhile, the spectra calculated in either of these two models for $Z < 0.5$ do not have strong distinctions. Therefore, the results of fitting experimental spectra with small Z in both cases should be close to each other, as was shown in [12]. This fact justifies the use of s-wave BTK model in our work.

It should be noted that in each magnetic-field set of $dV/dI(V)$ curves (similar to those in Fig. 1), only the zero-field curve was fitted by three parameters Δ, Γ, and Z. For the rest of the curves the fitting was performed with Z invariant. The fitted curves are shown in Fig. 1 (dashed curves). In general, the experimental and fitted curves agree quite well, except for those measured in low fields near $V = 1$ meV, where smeared maxima sometimes occurred. Such maxima, typical for low-ohmic contacts, are probably caused by the delay of the charge disbalance relaxation in the above-gap energy region. Meanwhile, calculation of the spectra in BTK theory was done without considering this fact. The order parameters Δ obtained in different magnetic fields using this procedure are shown in Fig. 2 (solid squares).

However, it is hardly possible to correlate our point contact with the objects analyzed by known theories which consider the order parameter as a function of the magnetic field. It seems that $\Delta(H)$ measured in point-contact experiments can be compared with the calculations for type-II superconductors (Mo_3Sb_7 belongs to this class [1]), $\Delta(H) \sim (1 - H/H_{c2})^{1/2}$ (e.g., see [13,14]).

However, the calculated dependence (not shown in Fig. 2) runs much lower than the experimental one. The reason for this significant discrepancy is that the point-contact method of measuring the order parameter of type-II superconductors in the magnetic field cannot yield results similar to those obtained by other methods, e.g., heat capacity measurement in which the measured

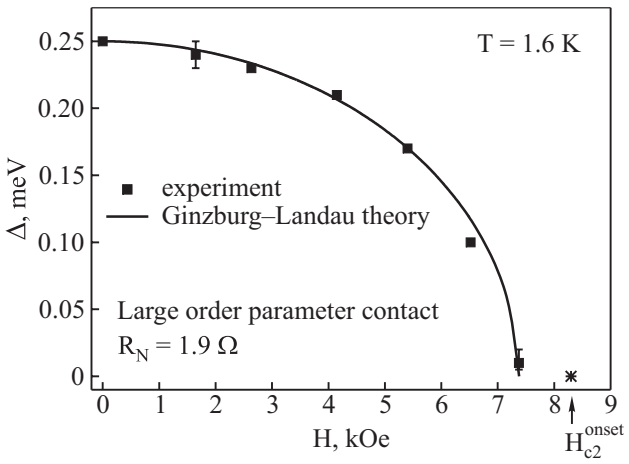

Fig. 2. Magnetic field dependence of the gap parameter $\Delta(H)$ (solid squares) as compared to the Ginzburg–Landau theory (dashed line) for the contact presented in Fig. 1.

values are averaged over a whole bulk volume. Meanwhile, the point-contact spectra account for only the properties of a thin surface layer of the sample. The thickness of this layer is close to the contact diameter (usually within 2–20 nm), which is much smaller than the London penetration depth Λ_L. In the applied magnetic field the Abrikosov vortices that penetrate into the *S* electrode are arranged so that the normal vortex core can never get into the contact area (except for fields close to H_{c2}) and cannot contribute to the point-contact spectrum. (Note that in our experiments the contact axis is perpendicular to the magnetic field). It is therefore natural that our Δ values exceed those obtained by other bulk-measurement techniques.

The close agreement of the experimental curve $\Delta(H)$ with the law $\Delta(H) \sim (1-(H/H_{c2})^2)^{1/2}$ (Fig. 2) calculated by the phenomenological Ginzburg–Landau (GL) theory [15] for a thin film in the parallel field may be not altogether casual, because such a film does not contain normal regions either. Thus, the regimes realized in a point contact and a thin film in a parallel field are in some sense similar. The Ginzburg–Landau theory was later confirmed by Gorkov's basic microscopic relations of the theory of superconductivity [16]. It appeared that the equations of this theory held not only near T_c, as was expected earlier, but could work with quite good accuracy in a much wider temperature interval. Later on the fact was strengthened by Maki [17]. The comparison with other theoretical dependences $\Delta(H)$ shows that our experimental results are less close to microscopic calculations [17–19] than to the dependence following directly from the Ginzburg–Landau theory (Fig. 2).

Note that in Fig. 2 the theoretical Ginzburg–Landau dependence was fitted to the whole set of experimental points, expect for the 8.3 kOe point. The critical field was estimated at the point of intersection of the theoretical curve and the abscissa. The obtained value $H^*_{c2} \approx 7.4$ kOe is significantly lower than $H_{c2} \approx 8.3$ kOe, which was derived from the complete set of $dV/dI(V)$ curves taken in different magnetic field and which accounts for the onset of the superconducting transition. One of the reasons for this discrepancy may be the smearing of the superconducting transition in magnetic field, which can be rather large for materials with a complicated crystalline structure. Hence, the field value in the middle of the transition would to some extent approach 7.4 kOe. Unfortunately, it is impossible to find this mid-point straightforwardly from the point-contact spectra. There is another reason for the uncertainty in the critical field value. In many contacts the gap minima registered near $V = 0$ do not vanish completely after sharp decrease of their amplitudes in the magnetic field, i.e., near the expected critical value of the field. In many cases the low-intensity minima persist up to $H \sim 2H_{c2}$. This effect is similar to that observed in many high-T_c superconductors and is attributed to the so-called pseudogap that appears in the spectrum of quasiparticle excitation above T_c. (The effect is not discussed in this work.) The rather large amplitude of the zero-bias minimum at $H \simeq 7.4$ kOe (Fig. 1) may be a result of summing up the main, gap-like, and pseudogap minima.

As noted above, many contacts have extremely low Δ (down to ~ 0.01 meV), while the critical temperature remains practically invariant to within several hundredths of a Kelvin. The typical set of spectra taken on one of the point contacts with rather low Δ ($\simeq 0.04$ meV) is shown in Fig. 3 (solid lines). The fitting of these spectra to the modified BTK theory [9] gives quite acceptable results (Fig. 3, dashed lines). Nevertheless, the obtained dependence $\Delta(H)$ differs appreciably of the one following from the Ginzburg–Landau theory (Fig. 4). The fitting procedure proves to be less successful when applied to contacts with $\Delta < 0.04$ meV, though it can be quite satisfactory for curves measured in zero or comparatively low magnetic fields (< 4 kOe). However, it is not applicable entirely to curves measured in high fields because of the strange narrowing of the gap minima. In such fields the calculated

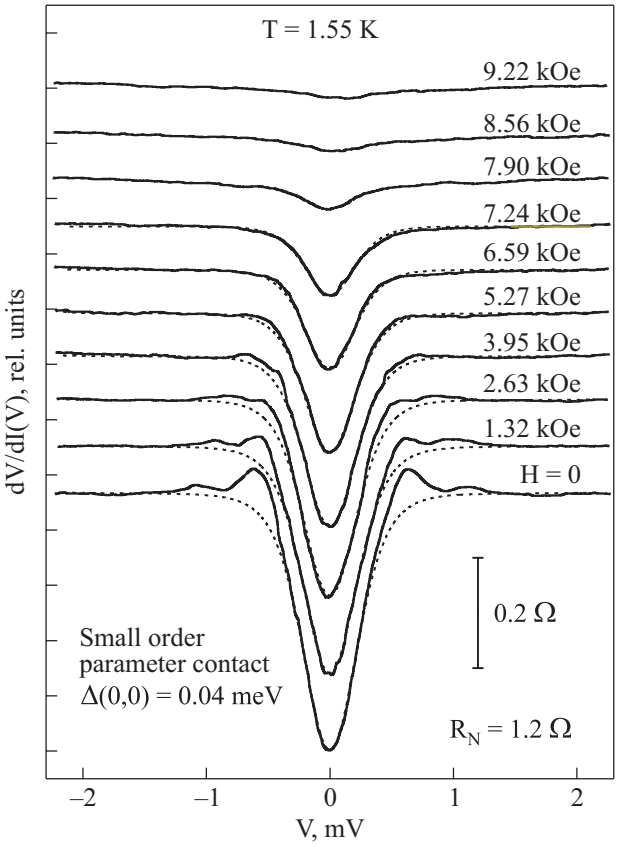

Fig. 3. Magnetic field set of the PC spectra (solid lines) for Ag–Mo_3Sb_7 contact with a small order parameter value $\Delta(0) \simeq 0.04$ meV ($Z = 0.15$, $\Gamma \approx 0$). The dashed lines present the BTK fits.

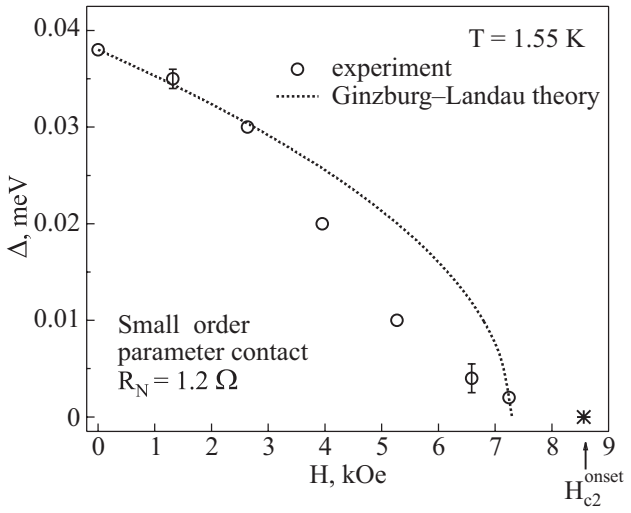

Fig. 4. The experimental $\Delta(H)$ dependence (open circles) in comparison with the Ginzburg–Landau law attached to the first low-field experiment points (dashed lines) for the contact presented in Fig. 3.

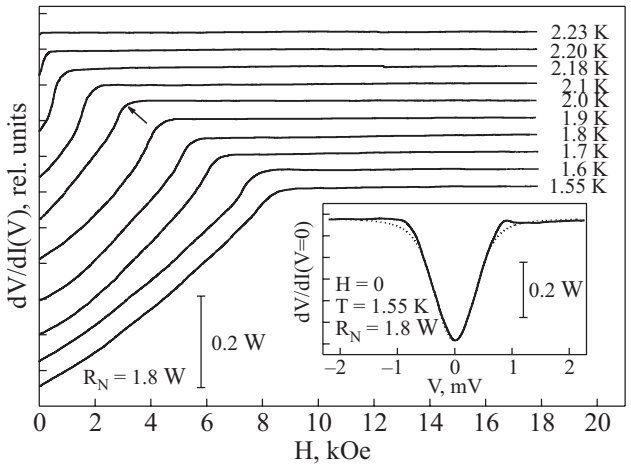

Fig. 5. Zero-bias differential resistance vs magnetic field dependences measured at different temperatures for Ag–Mo_3Sb_7 contact with $R_N \simeq 1.8\,\Omega$ (for clarity the curves are shifted vertically). H_{c2} values were taken at the points of the maximum curvature, as indicated on the 2.0 K-curve by an arrow. The inset shows the PC spectrum $dV/dI(V)$ of this contact measured in a zero magnetic field at 1.55 K (solid line), and BTK fitted curve.

curves are more broadened than the experimental ones no matter how much the fitting parameters are varied.

A similar situation occured while measuring the temperature dependence of the order parameter of Mo_3Sb_7 in [4], where it was assumed that this superconductor has anisotropic symmetry of pairing, e.g., of the *d*- or *sg*-type. So, the highest Δ is observed in the contacts oriented along the crystallographic directions where the gap function has a maximum. In the other so-called nodal directions with zero or close-to-zero gap function the spectra reveal the many times lower Δ. As before [4], we believe that the behavior of Δ for the lobe directions (i.e., corresponding to the maxima of gap function) with a change of temperature or magnetic field is roughly similar to that observed in conventional *s*-type superconductors with an isotropic gap function. Indeed, the experimental dependences $\Delta(T)$ (Fig. 2, [4]) and $\Delta(H)$ (Fig. 2, this study) measured on contacts with nearly maximum Δ deviate only slightly from the corresponding theoretical curves suitable for conventional BCS superconductors.

The dependence of the magnetic field on temperature (*H–T* phase diagram) and the upper critical field in Mo_3Sb_7 were found using the magnetic field dependences of the differential zero-bias resistance of the contacts $dV/dI(H)_{(V=0)}$ measured at different temperatures. Figure 5 shows the typical set of such curves for one of the point contacts with $\Delta(0,0) \approx 0.22$ meV and its zero-field spectrum. The curves demonstrate suppression of the Andreev current in the magnetic field. As this magnetic field increases, the superconducting electrode changes to the *N* state and the curves become saturated. Despite some smearing of the *S–N* transition near saturation region, it is possible to estimate H_{c2} quite accurately for each particular temperature. H_{c2} values were estimated at the points of the largest curvature (e.g., see Fig. 5, the arrow at one of the curves). It should be noted that the *N–S* transition is less smeared in our experiment than in magnetization measurement on the same material [1]. The critical field H_{c2} estimated at ~ 1.6 K for over 20 contacts is within 7.9–8.9 kOe.

No correlation was found between H_{c2} and Δ. This is not surprising because in the general case H_{c2} is highly anisotropic only in layered superconductors, e.g., chalcogenides and copper oxide compounds whose critical fields are three- or fourfold different along two main crystallographic directions. Mo_3Sb_7 has a cubic crystal lattice, which itself cannot lead to anisotropy of H_{c2}. Nor is the anisotropic character of the gap function $\Delta(k)$ in Mo_3Sb_7 a sufficient factor to cause anisotropy of H_{c2}. This is evident from the analysis of published data on the superconducting properties of borocarbide superconductors with an anisotropic $\Delta(k)$ function. Indeed, the anisotropy of critical field is quite moderate in nonmagnetic $LuNi_2B_2C$ with the ratio $\Delta_{min}/\Delta_{max} < 0.1$ [20] and in weakly paramagnetic YNi_2B_2C ($\Delta_{min}/\Delta_{max} < 0.01$) [21]. For example, in $LuNi_2B_2C$ the difference between the H_{c2} values measured along the main crystallographic directions was no more than 15%. In YNi_2B_2C no difference was registered within the measurement accuracy [22]. It is known that H_{c2} is highly anisotropic in magnetic superconductors with large magnetic moments oriented along certain crystallographic directions. Mo_3Sb_7 does not belong to this group because it exhibits only a weak paramagnetism (see below) and hence its magnetic

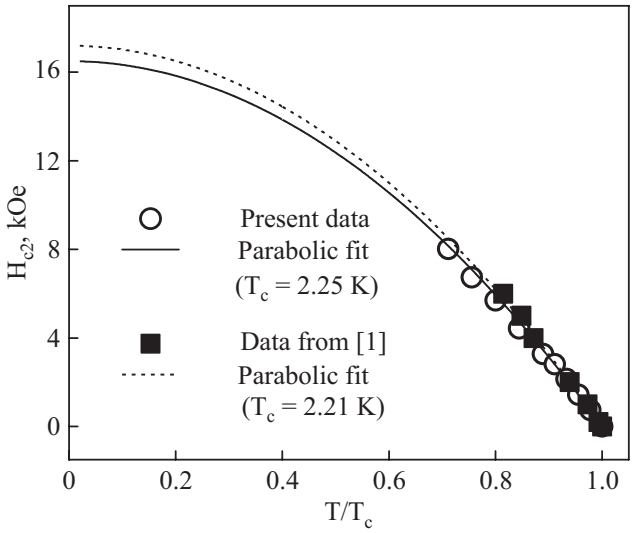

Fig. 6. $H-T$ phase diagram for Mo_3Sb_7 constructed on the basis of data derived from Fig. 5 (open circles) and fitted to the quadratic law (solid line). The solid squares and dashed line present the H_{c2} data and quadratic fit to them, respectively, reproduced from [1].

subsystem has no directionality. Thus, variations of the critical field observed in this material can hardly be evidenced of its possible anisotropy. Most likely, they are a consequence of inhomogeneous distribution of the critical parameters in the contact area.

Figure 6 illustrates the averaged H_{c2} values for seven contacts with quite small scatter of critical fields (8.2–8.5 kOe) (light circles). These data were obtained from plots similar to those in Fig. 5. In addition, Fig. 6 carries the results of magnetization measurement (solid square) [1]. The data of both sets exhibit a linear temperature dependence near T_c, which follows from the Werthamer–Helfand–Honenberg (WHH) microscopic theory [23] for conventional type-II superconductors. Earlier, this behavior was predicted in the phenomenological GL theory which holds a high accuracy in a narrow temperature interval near T_c. The results of Fig. 6 were fitted to the empiric low $H_{c2}(T) = H_{c2}(0)[1-(T/T_c)^2]$ (solid and dashed lines for our results and the data of Ref. 1, respectively). The law is a good approximation (several percent error) to the accurate microscopic equation with no temperature limitation according to the WHH theory. The obtained field $H_{c2}(0) \simeq 16.5$ kOe is quite close to 17.2 kOe in Ref. 1.

We also estimated the slope of $H_{c2}(T)$ with respect to the temperature axis ($-dH_{c2}/dT$) near T_c. Its value (~12.9 kOe/K) is comparable with the corresponding data for many intermetallic A-15 type superconductors. Knowing this value, we can estimate the pair-breaking effect of the Pauli paramagnetism in the material investigated. For this purpose we use a formula from Ref. 23 (a version of that in Ref. 24) that relates the dimensionless pair-breaking parameter α and the slope (in Oe/K units) $\alpha \simeq (5.28 \cdot 10^{-5})(-dH_{c2}/dT)_{T=T_c}$. We thus arrive at $\alpha \simeq 0.7$, which is typical for systems with moderate intensity of spin-flip scattering.

Conclusion

The investigation of the Andreev reflection in the magnetic superconductor Mo_3Sb_7 has shown that the behavior of the order parameter Δ in the magnetic field is essentially dependent on its own value. In our experiments Δ varied widely, $\Delta(0,0) \approx 0.01-0.31$ meV, in contacts with different orientations at practically invariant T_c. No correlation was found between Δ and H_{c2}. When Δ is high, its field dependence $\Delta(H)$ deviates slightly from the known theoretical relation that apply to conventional superconductors under point-contact conditions. However, the deviation starts to increase with decreasing Δ and becomes very large in the region $\Delta_{\min}$. Comparing this fact with the similar behavior of $\Delta(T)$ in [4] we persist in the belief that the gap function in Mo_3Sb_7 is anisotropic. In the context of this assumption $\Delta_{\max}$ can be registered in contacts oriented along the crystallographic axes close to the maxima in the gap function, which may have the isotropic *s*-symmetry behavior in a limited interval of angles. This assumption is helpful in explaining the nearly standard dependences $\Delta(H)$ and $\Delta(T)$. The lower $\Delta_{\max}(0) \simeq 0.31$ meV as against the BCS prediction $\Delta_{BCS}(0) \simeq 0.35$ meV can be attributed to the magnetic nature of this material. Hence, the pair-breaking effect of spin-flip scattering should not be neglected. The upper critical fields H_{c2} values measured at different temperatures and averaged over seven contacts were compared with the quadratic dependence $H_{c2}(T)$ for type-II superconductors. On the basis of experimental *dH/dT* value and the corresponding theoretical equation [23,24] we were able to estimate the efficiency of the pair-breaking effect caused by the Pauli paramagnetism. The effect has turned out to be quite moderate.

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