Critical currents, flux creep, and current-voltage characteristics of single crystals of the organic superconductor $\varkappa$-$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$

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We investigated, at normal pressure, single crystals of the organic superconductor $\varkappa$-$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$ ($x = 0.5$) with the highest superconducting-transition temperature (~ 12.5 K) of all organic superconductors. We obtained the temperature and field dependences of the critical current density, determined the anisotropy of the critical current, investigated the relaxation of the magnetic moment, and determined the dependence of the activation energy on the field. We calculated the current-voltage characteristics from the time dependences of the magnetic moment at a fixed temperature. We have found that the current-voltage characteristics can be described by either an exponential or a power-law $E(j)$ dependence.

INTRODUCTION

Substantial advances were made in the last three years towards raising the superconducting transition of organic superconductors. This success is due to synthesis of the $\varkappa$ phase of the cation-radical salt $\varkappa$-$(BEDT\text{-}TTF)_2X$ with different anions X: $Cu(NCS)_2$, $Cu[N(CN)_2]Br$, $Cu[N(CN)_2]Cl$.[1–3] Whereas the first two compounds are superconductors at normal pressure, the latter, having the maximum $T_c \approx 12.8$ K for this class, becomes superconducting only under pressure ($P \sim 0.3$ kbar).[4] Single crystals of the organic superconductor $\varkappa$-$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$ with $T_c \approx 12.5$ K at normal pressure were synthesized for the first time ever in our country. The magnetic properties of single crystals of this superconductor have not been investigated to this day, and we have therefore attempted to find the most important characteristics of this substance—the first critical field H_{c1}, the field and temperature dependences of the critical current j_c, and the average activation energy for two crystallite orientations relative to the magnetic field **B**—and to compare the results with data for other organic superconductors of this family.

EXPERIMENTAL RESULTS. SAMPLE PRODUCTION AND EXPERIMENTAL PROCEDURE

Single crystals of $\varkappa$-$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$ with characteristic dimensions $0.8 \times 1.3 \times 0.2$ mm^3 were obtained by the standard technology (see, e.g., Ref. 3). The measurements were made with a SQUID magnetometer, and also with a SQUID magnetometer MPMS manufactured by Quantum Design.[6] While having the same sensitivity and the same temperature interval, these magnetometers have substantially different field intervals, types of magnetic-flux transformers, rates of field application in relaxation-process measurement, and signal-reduction principles. The SQUID magnetometer used by us[5] had the conventional design of our domestic ones; it was therefore of interest to compare it with the QD magnetometer widely used world-wide to investigate magnetic properties of superconductors not yet available in our country. The magnetic field in our magnetometer is produced by a combination of a superconducting magnet with an Nb–Ti tube (short-circuited second solenoid in other installations). The field is therefore quite uniform and stable in time. It should be noted that these features require a solenoid of sufficient length to maintain the field inhomogeneity within 0.1% in the region of the receiving loops of the flux transformer, requiring inevitably larger dimensions of the apparatus. The magnetometer uses only one superconducting magnet, and the possible problems with inhomogeneity and instability of the field are avoided by incorporating a second-order gradient meter in the design of the flux transformer (FT). This flux-transformer configuration, however, has the following shortcomings. To optimize the FT it is necessary to satisfy the relation $l = D$, where l is the distance between the loops and D is the

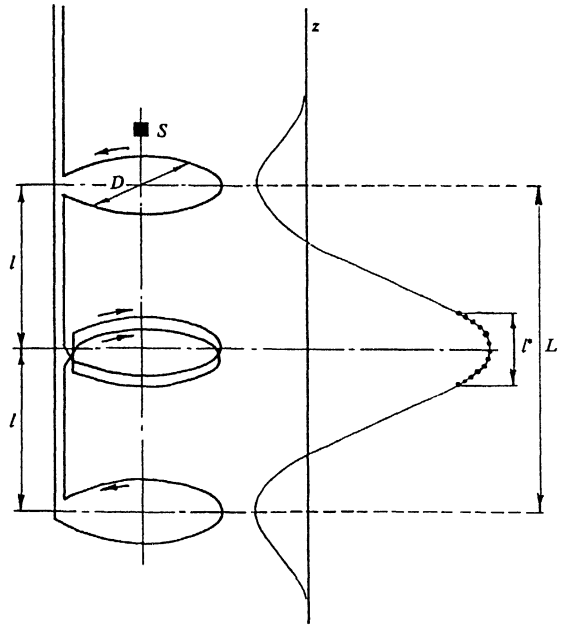

FIG. 1. Second order gradient meter of the flux transformer of the SQUID magnetometer. The solid line shows the calculated dependence of the SQUID-magnetometer response on the displacement of the sample along the z axis of the gradient meter. The points show experimental values, S marks the position of the sample.

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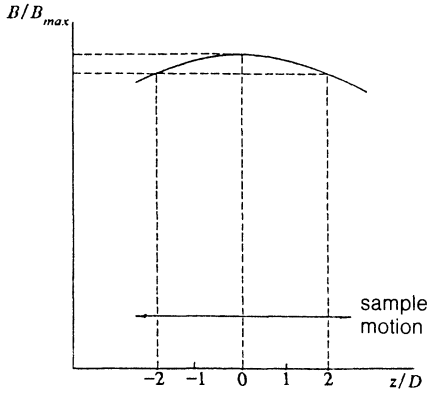

FIG. 2. Field distribution along the gradient meter in the SQUID magnetometer.[6,8]

FT-loop diameter.[7] This leads inevitably to a longer sample scan length L (Fig. 1). The MPMS unit has $l = 13$ cm, and the field inhomogeneity at the end of the FT reaches 5%. In this case displacement of the sample along the FT axis is equivalent to motion along the hysteresis loop (the field increases initially as $z \to 0$, and decreases after passing through the middle of the FT) (Fig. 2). If the field **B** is perpendicular to the sample plane, this can lead to complete reversal of the crystal magnetization and reversal of the signal sign.[9,10] This shortcoming was eliminated in the following manner: the sample moved not over the entire length L between the receiving loops of the FT (the full-scan regime), but only on a path $l^* = 3$ cm near the center of the FT. About 32 experimental points of the SQUID response to the displacement of the sample were measured, and the total response was calculated from them in the point-dipole approximation.[6] This procedure is correct enough at a ratio of the characteristic sample dimensions $d/D \leqslant 0.1$ (Ref. 7) and if the form of the signal is "regular" (Ref. 10). The last condition is difficult to meet when thin films are measured in the zero-field cooling (ZFC) regime and in strong fields (field cooling—FC). Thus, each of the facilities used by us had both advantages and shortcomings, and these determined the investigation range in which they were used.

The method of securing and reorienting organic-superconductor single crystals in an ampul container was described earlier (see, e.g., Ref. 11).

DEPENDENCES OF MAGNETIC MOMENT ON TEMPERATURE AND FIELD

The temperature dependences of the magnetic moment of single-crystal $\varkappa$-(BEDT-TTF)$_2$ Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$ were investigated in two orientations, $\mathbf{B} \perp bc$ and $\mathbf{B} \| bc$ (where bc is the basal conducting plane of the crystal) (Figs. 3a, b). The temperature of the superconducting transition, revealed by the substantial increase of the magnetic moment P_m, was $T_c \sim 11$ K in a field $B = 1$–2 mT for both orientations. In the normal state these single crystals are weak diamagnets,[12] and since their $P_m(T)$ dependences near T_c have not been determined to date, the value of T_c depends strongly on the sensitivity of the apparatus ($\Delta P_m \sim 10^{-10}$ A·m^2–10^{-11} A·m^2 in our case). Notice must be taken of a small, substantially smaller percentage of the Meissner phase than for the orientation $\mathbf{B} \perp bc$, $\sim 3\%$ for the orientation $\mathbf{B} \| bc$ in an external field $B \sim 12$ mT (the demagnetizing factor for this orientation is $N \sim 0.1$). The presence of a $P_m(T)$ section independent of T at $2\ \mathrm{K} \leqslant T \leqslant 3.5\ \mathrm{K}$ on the $P_m^{\mathrm{ZFC}}(T)$ dependence for $\mathbf{B} \| bc$ indicates the presence of complete diamagnetic screening of the crystal and that the field $B = 1.2$ mT is the first critical one for this orientation at $T \sim 3$ K. This value is substantially higher than the H_{c1} obtained for $\varkappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ single crystals in the same orientation.[13] The dependence of H_{c1} on T for the orientation $\mathbf{B} \perp bc$ is shown in Fig. 4. The values of H_{c1} were obtained from the deviations of the field dependences of $P_m(B)$ from lilnearity. Since the form of $P_m(B)$ near B_{c1} is likewise unknown, we obtain only a qualitative estimate of H_{c1}. In the investigated temperature interval we can approximate $H_{c1}(T)$ by linear relations, just as in Ref. 14. The

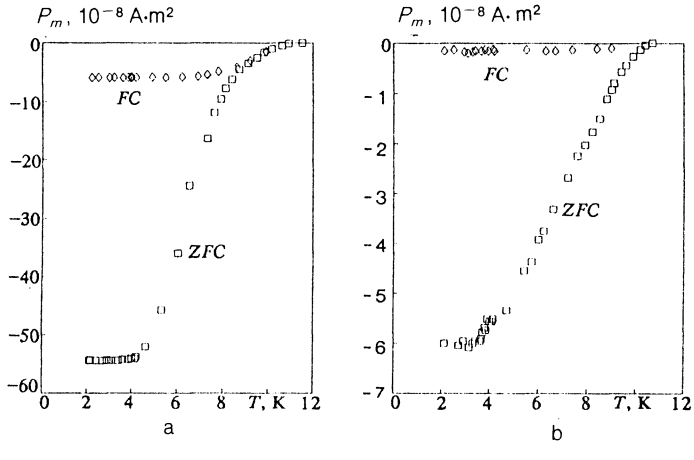

FIG. 3. Temperature dependences of the magnetic moment of the single crystal $\varkappa$-(BEDT-TTF)$_2$Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$: a) $\mathbf{B} \perp bc$, $B = 2.1$ mT, □—ZFC, ◇—FC; b) $\mathbf{B} \| bc$, $B = 1.2$ mT, □—ZFC, ◇—FC.

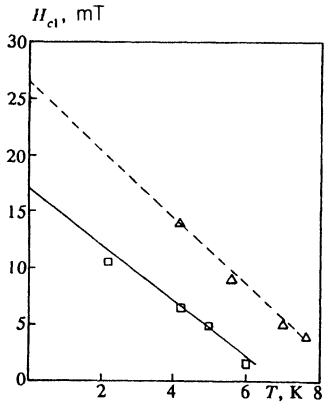

FIG. 4. Temperature dependences of the first critical field of the single crystals $\varkappa$-$(ET)_2X$[X = $Cu(NCS)_2$(Δ), $Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$(□)] at an orientation $\mathbf{B}\perp bc$.

difference between the values of H_{c1} of the single crystals $\varkappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ and $\varkappa$-(BEDT-TTF)$_2$ Cu[N(CN)$_2$] $Cl_{0.5}Br_{0.5}$ for the orientation $\mathbf{B}\perp bc$ can be related, apparently to within an error in the determination of the demagnetizing factor $N\sim 0.8$, since the investigated crystals had no regular form. The calculated value of H_{c1}

$$H_{c1} = \frac{\Phi_0}{4\pi\lambda_i\lambda_k}\left[\ln\frac{(\lambda_i\lambda_k)^{1/2}}{(\xi_i\xi_k)^{1/2}}\right],$$

where $\lambda_{i,k}$ are the penetration depths in a direction perpendicular the applied field B, and $\xi_{i,k}$ are the components of the anisotropic coherence length, with allowance for the parameters ξ and λ which are typical of the system $\varkappa$-$(ET)_2X$ (Refs. 15–19) are of the same order as the experimental value of H_{c1}. The field dependences of P_m (B) for both orientations past the ZFC (residual field $\sim 10^{-5}$ T) are shown in Fig. 5. If the P_m (B) dependence for the orientation $\mathbf{B}\perp bc$ has a form typical of soft superconductors of the second kind, for the $\mathbf{B}\|bc$ orientation, past the section of the abrupt decrease of ΔP_m ($|B| \leqslant 40$ mT), the hysteresis loop width ΔP is practically independent of the field B. This can apparently be attributed to the fact that pinning on a large number of small centers is significant only in weak fields, while in strong fields an important role is probably played for this orientation by the pinning proper between the conducting planes of the crystal.

TEMPERATURE AND FIELD DEPENDENCES OF CRITICAL CURRENT DENSITY

On the basis of the irreversible parts of the P_m (B) hysteresis curves within the framework of the anisotropic Bean model (see, e.g., Ref. 20), neglecting the anisotropy in the basal plane and assuming j_c in this plane to be independent of the **B** direction,[21] we obtained the temperature and field dependences of j_c (Fig. 6). The temperature dependences of $j_c(T)$ can be written in the form $j_c(T,0) = j_c(0,0)\exp(-T/T_0)$ with $T_0 = 1$–2 K for both orientations. Note that the critical current densities for single crystals of the family $\varkappa$-(BEDT-TTF)$_2$X, where X = $Cu(NCS)_2$, $Cu[N(CN)_2]Br$, and $Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$ (Refs. 12 and 22) for the orientation $\mathbf{B}\perp bc$ are practically the same. The anisotropy j_c

$$K = j_c(B \perp bc)/j_c(B \parallel bc) \approx 17$$

for $T = 4.2$ K and decreases as $T \to T_c$.

It should be noted that the determination of the value of K of strongly anisotropic materials [$K(T\to 0)\sim 10^2$, Ref. 13] from magnetic measurements encounters substantial difficulties. One of the main reasons is the possible error in the single-crystal orientation when the field is applied along the conducting layers. In this case, an error by an angle $\alpha \sim 2°$ can conceal completely the true anisotropy, since the measured longitudinal magnetization of the sample acquires a contribution $\sim P_m \sin\alpha$ from the magnetization P_m due to the currents in the basal plane. In our case both errors were eliminated because the sample was mounted on a side wall of an extended ($l = 200$ mm, $d = 5$ mm) quartz ampul container. In addition, one can doubt the correctness of the statement that j_c in the basal plane is independent of the angle between the field and the plane, since the mechanism

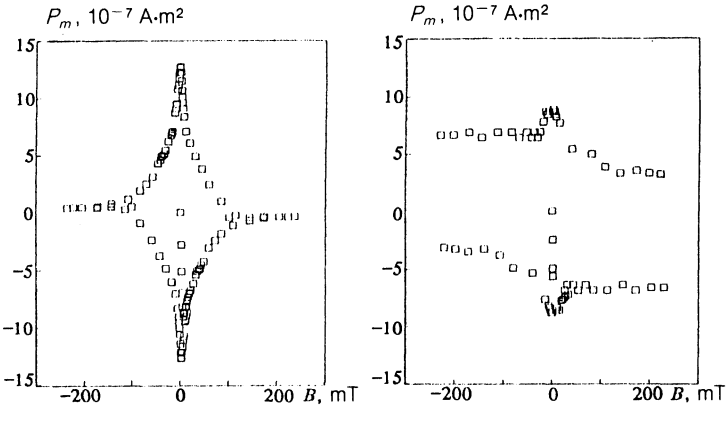

FIG. 5. Field dependences of the magnetic moment of the single crystal $\varkappa$-$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$, $T = 4.2$ K: a) $\mathbf{B}\perp bc$, b) $\mathbf{B}\|bc$.

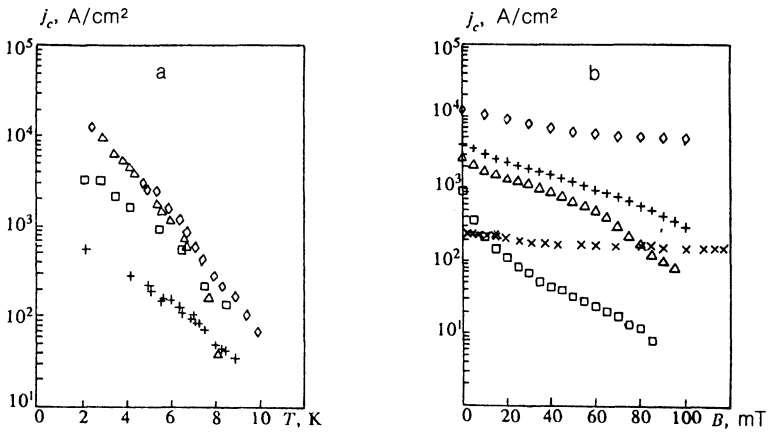

FIG. 6. a) Temperature dependences of the critical current density of single crystals as $B \to 0$: ◇—$\varkappa$-$(ET)_2$ $Cu[N(CN)_2]$ $Cl_{0.5}Br_{0.5}$, $\mathbf{B} \perp bc$; +—$\varkappa$-$(ET)_2$ $Cu[N(CN)_2]$ $Cl_{0.5}Br_{0.5}$, $\mathbf{B} \| bc$; □—$\varkappa$-$(ET)_2$ $Cu[N(CN)_2]Br$, $\mathbf{B} \perp bc$; Δ—$\varkappa$-$(ET)_2$ $Cu(NCS)_2$, $\mathbf{B} \perp bc$. b) Field dependences of the critical current density of the single crystal $\varkappa$-(BEDT-TTF)$_2$Cu$[N(CN)_2]Cl_{0.5}Br_{0.5}$: ◇—$T = 2.5$ K, $\mathbf{B} \perp bc$; +—$T = 4.2$ K, $\mathbf{B} \perp bc$; ×—$T = 4.2$ K, $\mathbf{B} \| bc$; Δ—$T = 5$ K, $\mathbf{B} \perp bc$; □—$T = 6$ K, $\mathbf{B} \perp bc$.

that pins the vortices located between the conducting layers and the vortices "piercing" these layers are apparently different.

The field dependences of the critical current density of $\mathbf{B} \perp bc$ of the single crystal $\varkappa$-$(ET)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$ (Fig. 6b) is apparently intermediate between the purely exponential dependences of the single crystals $\varkappa$-$(ET)_2Cu(NCS)_2$ (Fig. 7a)[22] and the more complicated $j_c(B)$ dependences of $\varkappa$-$(ET)_2Cu[N(CN)_2]Br$ single crystals (Fig. 7b).[12] The anisotropy of the critical currents K decreases rapidly with increasing B and $K \to 1$ for $T = 4.2$ K and $B = 120$ mT, just as in the case of $\varkappa$-$(ET)_2Cu(NCS)_2$ single crystals.[22]

TIME DEPENDENCES OF MAGNETIC MOMENT

The time dependences of the magnetic moment of the investigated crystals were obtained in the ZFC by successive introduction of the field B. The $P_m(t)$ dependences for single-crystal $\varkappa$-$(ET)_2Cu[N(CN)_2]Cl_{0.5}Br_{0.5}$ at $\mathbf{B} \perp bc$ and $T = 4.2$ K are shown in Fig. 8. Just as for other crystals of the family $\varkappa$-$(ET)_2X$ (Refs. 13, 23–25) these dependences are well described by the logarithm of t in the time interval 100 s—4000 s and in the entire range of fields used. The relaxation rate $S = d(\ln P_m^*)/(\ln t)$ (Ref. 26) in the complete-penetration field (Ref. 20)

$$B^* = \frac{\sqrt{2}\mu_0 j_c t}{\pi} \ln\left(2\alpha \frac{L}{t}\right),$$

$P_m^* = P_m - P_m^{\text{rev}}$ (P_m^{rev} is the reversible contribution to the magnetization), t is the slab thickness, L is the side length and $d \approx 1.105$) reaches a plateau which is continued by further S growth when B is increased (Fig. 9). Similar data were obtained earlier for the single crystals $\varkappa$-$(ET)_2Cu(NCS)_2$ (Ref. 27) and $\varkappa$-$(ET)_2Cu[N(CN)_2]Br$ (Ref. 23). The equality of B^* for both systems is due to the parctically identical j_c (Fig. 6a) and characteristic dimensions of the samples.

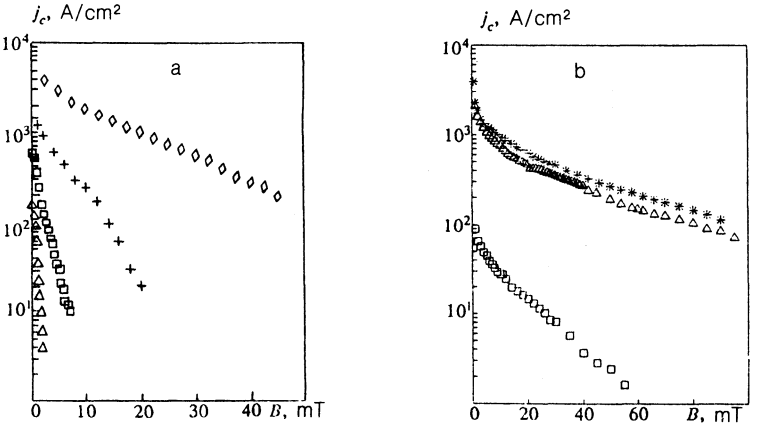

FIG. 7. a) Field dependences of the critical current density of the single crystal $\varkappa$-(BEDT-TTF)$_2$Cu$(NCS)_2$, $\mathbf{B} \perp bc$: ◇—$T = 4.2$ K, +—$T = 5.6$ K; □—$T = 6.5$ K; Δ—$T = 7.7$ K. b) Field dependences of the critical current density of the single crystal $\varkappa$-(BEDT-TTF)$_2$Cu$[N(CN)_2]$Br: *—$T = 2.1$ K; Δ—$T = 4.2$ K; □—$T = 6.5$ K.

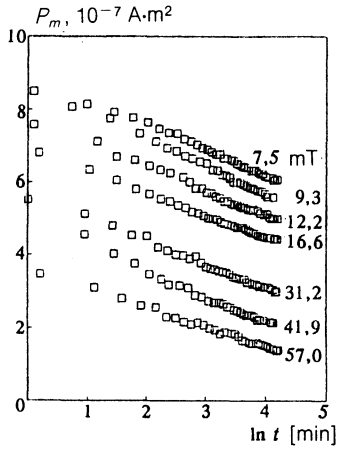

FIG. 8. Time dependences of the magnetic moment of the single crystal $\varkappa$-(BEDT-TTF)$_2$Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$, $T = 4.2$ K, $\mathbf{B} \perp bc$.

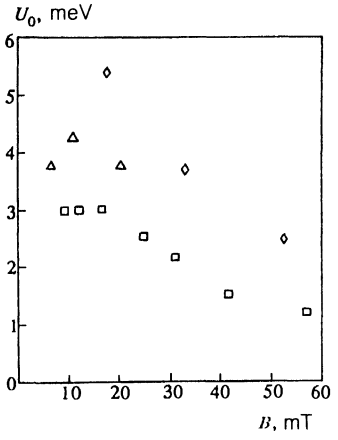

FIG. 10. Field dependences of the average activation energy U_0 at $T = 4.2$ K: □—$\varkappa$-(ET)$_2$Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$, $\mathbf{B} \perp bc$; Δ—$\varkappa$-(ET)$_2$Cu(NCS)$_2$, $\mathbf{B} \perp bc$; ◇—$\varkappa$-(ET)$_2$Cu(NCS)$_2$, $\mathbf{B} \| bc$.

From the field dependences of $S(B) = k_B T/U_0$ at $B > B^*$ we obtained the field dependences of the average activation energy $U_0(B)$ (Fig. 10). The values of U_0 for single crystals of $\varkappa$-(ET)$_2$X with different anions X were practically equal, and there was apparently no anisotropy of U_0, although the anisotropy of the currents of K for these temperatures of K is 10–10^2 (Refs. 13, 22, 27). Note that the relaxation rate in the theory of collective creep[28] is independent of the angle between the magnetic field and the conducting layers, as is in fact observed in our case. The U_0 dependence can be taken in fields $B > B^*$ to be $\sim B^{-1}$.

The magnetic-moment $P_m(t)$ dependences can be recalculated into current-voltage characteristics[29,30] on the basis of the following assumptions. We consider for simplicity the case of a thin ring of radius $R \gg \Delta R$, and then the variation of the flux through the ring is given by Faraday's law

$$-\frac{d\Phi}{dt} = 2\pi RE. \tag{1}$$

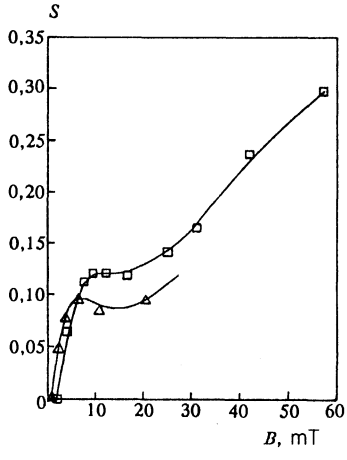

FIG. 9. Field dependences of the relaxation rate $S = d(\ln P_m^*)/d(\ln t)$, $T = 4.2$ K, $\mathbf{B} \perp bc$: □—$\varkappa$-(ET)$_2$Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$; Δ—$\varkappa$-(ET)$_2$Cu(NCS)$_2$.

For a thin ring we have

$$\frac{d\Phi}{dt} = \mu_0 \pi R^2 \frac{dH_e}{dt} - LS\frac{dj}{dt}, \tag{2}$$

where L is the inductance, S is the ring cross section perpendicular to the current. The magnetic moment of the current-carrying ring is

$$P_m = \pi R^2 I = \pi R^2 Sj. \tag{3}$$

Substituting (2) and (3) in (1) we get

$$E = -\frac{\mu_0 R}{2}\frac{dH_e}{dt} - \frac{L}{2\pi^2 R^3}\frac{dP_m}{dt}. \tag{4}$$

If the $j_c(B)$ dependence is weak, one can neglect the first term for relaxation measured in a constant external field and then

$$E(j) = -\frac{L}{2\pi^2 R^3}\frac{dP_m}{dt} \propto \frac{dP_m}{dt}, \quad j \propto P_m,$$

so that j and E can be obtained from $P_m(t)$.

A rigorous calculation for a slab sample yields the relation

$$E = \frac{3\mu_0 I}{2\pi^2 R^2}\frac{dP_m}{dt}, \tag{5}$$

where R is the characteristic dimension and $I = 3.328$ (Ref. 29). The results for $T = 4.2$ K and $\mathbf{B} \perp bc$ are shown in Fig. 11. Evidently, these relations are well described by the power-law relations $E/E_0 \sim (j/j_0)^n$ for fields $B < 31$ mT and the transition to an exponential dependence takes place, as predicted by the Anderson model.[31]

As shown in Ref. 29, for sufficiently long times $t \gg t_0$ the relaxation rate is

$$S = \frac{d(\ln P_m)}{d(\ln t)} = \frac{d(\ln j)}{d(\ln E)} = \frac{1}{n},$$

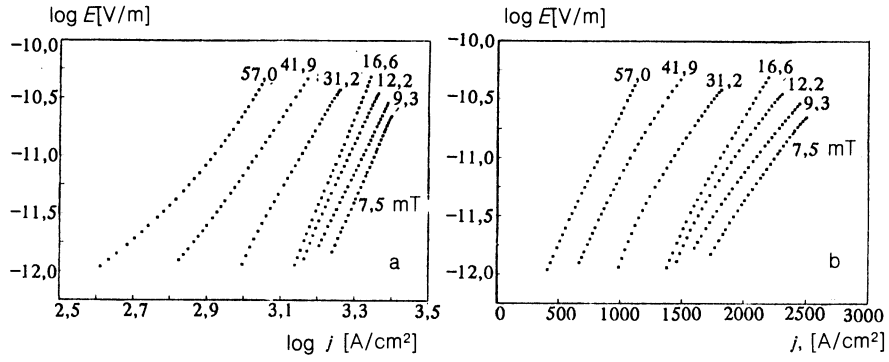

FIG. 11. Current-voltage characteristics of the crystal $\varkappa$-(BEDT-TTF)$_2$ Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$ in various fields, $T = 4.2$ K, $\mathbf{B} \perp bc$.

It determines thus the angles between $j(E)$ and the axes $\log j$ and $\log E$ and yields the reciprocal of the exponent n in the power-law dependence of $E(j)$, as is well confirmed by our experiments (see Figs. 12 and 9).

Note that the calculated current-voltage characteristics are located in a region where E is lower by at least three orders than the sensitivity threshold E_c of the resistance measurements (see, e.g., Refs. 32 and 33).

PRINCIPAL RESULTS AND CONCLUSIONS

1. It was shown that many properties of a single crystal of the new organic superconductor $\varkappa$-(BEDT-TTF)$_2$Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$, such as the first critical field H_{c1}, the temperature and field dependences of the critical-current density, and the average activation energy U_0 are common to single crystals of the family $\varkappa$-(BEDT-TTF)$_2$X, where X = Cu(NCS)$_2$ or Cu[N(CN)$_2$]Br, and their values were determined.

2. It was established that the current-voltage characteristics of the single crystal $\varkappa$-(ET)$_2$Cu[N(CN)$_2$]Cl$_{0.5}$Br$_{0.5}$, obtained from the time dependences of the magnetic moment P_m are transformed when the field B is increased from power-law to exponential. The exponent n and its field dependence are determined. It is shown by experiment that if the time t is long enough then $n = 1/S$, where S the relative rate of the logarithmic relaxation.

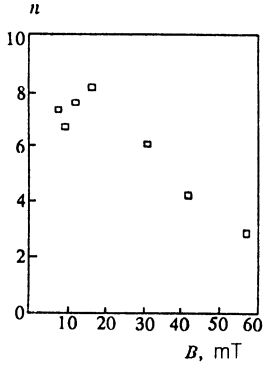

FIG. 12. Dependence of $n = d(\ln E)/d(\ln j)$ on B for the single crystal $\varkappa$-(BEDT-TTF)$_2$ Cu[N(CN)$_2$] Cl$_{0.5}$Br$_{0.5}$.

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