Partially ordered vortex lattices in the high-field low-temperature mixed state of quasi-two-dimensional organic superconductors

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We report the results of high-field, low-temperature μSR measurements of the quasi-two-dimensional organic superconductors κ-$(ET)_2Cu(NCS)_2$, and κ-$(ET)_2Cu[N(CN)_2]Br$. The μSR lineshapes for these compounds indicate the existence of partially ordered vortex lattice phases in the high-magnetic-field regime, up to 2.5 T for the former compound and 4 T for the latter compound. The observed sharp loss of order is found to be consistent with a vortex-lattice melting transition that is predicted by numerical simulations of weakly coupled layers of pancake vortices. It is argued that the robustness of the partially ordered vortex lattice phases could be due to strong flux-line pinning by a dilute ensemble of defects.

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I. INTRODUCTION

In quasi-two-dimensional (2D), extreme type-II superconductors, such as the organic charge-transfer salts $(ET)_2X$ [1,2], and the high-transition-temperature (T_c) compound $Bi_2Sr_2CaCu_2O_8$ (BSCCO) [3], the existence and extent of highly ordered three-dimensional (3D) vortex lattice states are known to be strongly dependent on the coupling between the underlying 2D superconducting (SC) layers. In such compounds with weakly coupled SC layers (via very small Josephson tunneling currents) subjected to a magnetic field applied perpendicular to the layers [4], the vortex system consists of stacked layers of pancake-like vortices [5]. For applied magnetic fields not far below the upper critical field (H_{c2}), the relatively strongly coupled intralayer pancake vortices can form well-ordered 2D lattices, whereas the pancake vortices in adjacent layers couple very weakly by the electromagnetic dipole interaction. Consequently, while at low temperatures straight magnetic flux lines threading weakly coupled pancake vortices may essentially form a 3D ordered lattice, these flux lines can be easily distorted by thermal fluctuations at elevated temperatures. This leads to a loss of 3D order, which occurs at a dimensional crossover field (H_{2D}) far below H_{c2}. Further disordering of the flux lattice, by strong thermal fluctuations within the individual layers of vortices, can occur at much higher magnetic field, causing a complete loss of order (vortex-lattice melting) at an intermediate field $H_{2D} \ll H < H_{c2}$ [6–8]. These order-disorder transitions have been extensively studied in BSCCO, via, for example, small-angle neutron scattering (SANS) [9], muon spin rotation (μSR) [10,11], and irreversibility-line [12] and magnetization [13] measurements. Similar vortex-lattice transitions have also been investigated in the low-T_c, quasi-2D organic superconductors (see Ref. [2] for a review), with applications of the μSR technique focusing on the charge transfer salts κ-$(ET)_2Cu(NCS)_2$ [14,15] and κ-$(ET)_2Cu[N(CN)_2]Br$ [16].

Theoretical estimates of the 3D-2D crossover field H_{2D}, based on a continuum elastic description of the vortex lattice [17,18], yield characteristic values in the range of $H_{2D} \sim \Phi_0/\lambda^2$ (where Φ_0 is the magnetic flux quantum, and λ is the in-plane magnetic penetration depth), which is about three orders of magnitude smaller than H_{c2}. These estimates have been confirmed in a number of μSR experiments performed on the relevant compounds [10,11,15]. However, in some cases the loss of order deduced from changes in the μSR lineshape with increasing magnetic field was incomplete [19], indicating the existence of partially ordered 3D flux-line networks at magnetic fields well above the theoretically predicted values of H_{2D}. The existence of such partially ordered 3D vortex phases at magnetic fields $H \gg H_{2D}$ has been demonstrated by transport [20] and SANS measurements [21] of BSCCO single crystals.

In the present paper we provide clear μSR evidence for the existence of a partially ordered vortex lattice phase in the quasi-2D organic superconductors κ-$(ET)_2Cu[N(CN)_2]Br$ (κ-Br) and κ-$(ET)_2Cu(NCS)_2$(κ-CuNCS) at magnetic fields much higher than the values of H_{2D} predicted by the continuum elastic model. We also show that the observed sharp changes of the μSR lineshape, generally associated with the loss of flux-line order, is consistent with melting of pancake vortex lattices within the layers, rather than a 3D-2D crossover. We suggest that, in the low-temperature, high-magnetic-field region of the phase diagram investigated, small concentrations of defects that pin flux lines perpendicular to the SC layers effectively resist the breakup of the entire 3D vortex lattice by thermal fluctuations.

II. EXPERIMENTAL

Transverse-field (TF) μSR measurements with applied magnetic fields in the range $1.0 < H < 4.5$ T were carried out on the M15 muon beam line at TRIUMF by using a spectrometer consisting of a top-loading Oxford Instruments dilution refrigerator (DR). The magnetic field was applied perpendicular to the highly conducting layers, i.e., parallel

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to the b axis or to the a^* axis of each sample, which were mosaics consisting of tens of single crystals of κ-Br or κ-CuNCS. The use of a mosaic was necessary, due to the small size of the individual single crystals—typically less than 1 mm wide and 100 μm thick. The crystals were mounted on a pure Ag sample holder by using Apiezon grease. The single crystals were grown by standard electrochemical crystallization according to the literature methods [22,23]. The superconducting transition temperature T_c determined by thermodynamic methods is 9.3 ± 0.1 K and 11.5 ± 0.1 K for κ-CuNCS and κ-Br, respectively.

A fast Fourier transform (FFT) of the TF-μSR signal closely resembles the internal magnetic-field distribution $P(B)$ [24]. The measurements reported here were typically done by first cooling the sample from room temperature to $T = 20$ mK in a magnetic field of $H = 1$ T and subsequently measuring the field dependence of the TF-μSR signal. For each value of the applied field, measurements were also performed at $T = 10$ K, which is well above the irreversibility lines for both compounds [25,26], and hence at a temperature where the vortex structure has no influence on the μSR lineshape. The measurements at $T = 10$ K (henceforth referred to as the reference signal) provide a visualization of the broadening of the TF-μSR lineshape by the sample nuclear dipole moments, the field inhomogeneity of the external magnet, and the background from muons stopping in the sample holder and other places outside the sample.

III. RESULTS

Figure 1 shows typical FFTs of the TF-μSR signals for κ-CuNCS at $T = 20$ mK, and the corresponding probability field distribution, $P(B)$, obtained by deconvoluting each FFT in the frequency domain with respect to the reference signal. Figure 2 shows results of similar measurements for the sister compound κ-Br. The most striking feature of the deconvoluted TF-μSR lineshapes shown in Figs. 1 and 2, is their clear asymmetry, which is characterized by a positive skewness parameter $\alpha = \langle \Delta B^3 \rangle^{1/3} / \langle \Delta B^2 \rangle^{1/2}$, where $\Delta B = B - \langle B \rangle$ and $\langle B \rangle$ is the average internal field [24,27]. This is clearly observed in a broad field range, up to a $H = 2.5$ T for κ-CuNCS, and up to $H = 4$ T for κ-Br—above which α changes sign rather abruptly. As shown in Fig. 3, at $T = 20$ mK positive values of the skewness parameter in the range $0.5 < \alpha < 1.0$ reflect the striking existence of partially ordered 3D vortex-lattice states at magnetic fields $H \gg H_{2D}$ in both materials, which are comprised of very weakly coupled 2D conducting layers [28]. At higher temperatures, the field range of the ordered phase shrinks considerably, as shown in Fig. 3 for κ-Br at $T = 1$ K, where the onset of a negative α is observed above $H = 2$ T. Note that previous TF- μSR lineshape measurements on κ-CuNCS performed at $T = 1.8$ K [14,15] showed positive values of the skewness parameter $\beta = \langle \Delta B \rangle / \langle \Delta B^2 \rangle^{1/2}$, which drops to zero in fields of 10 to 20 mT, consistent with the continuum elastic theory [17,18]. As we discuss in the next section, the rapid precooling of the samples that occurred when they were loaded into the dilution refrigerator may have resulted in intrinsic structural disorder. Pinning associated with this disorder may be responsible for the observed robustness

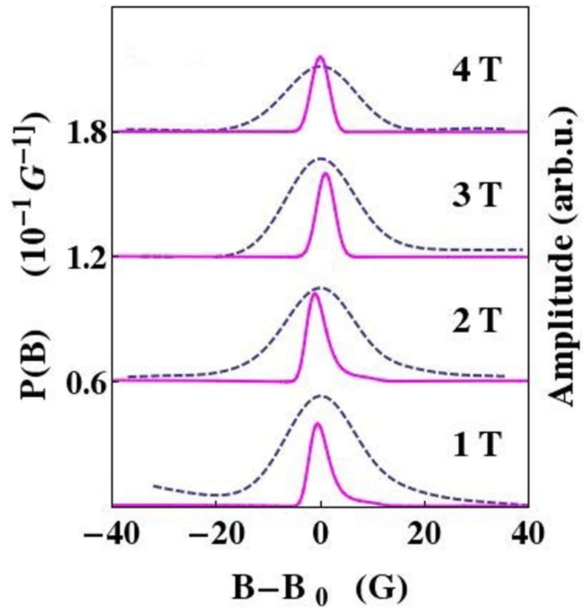

FIG. 1. (Color online) Probability magnetic field distribution, $P(B)$, for κ-$(ET)_2Cu(NCS)_2$ (magenta solid curves and left-hand scale) at $T = 20$ mK and different external magnetic fields, obtained by deconvoluting the FFTs (blue dashed curves and right-hand scale) of the TF-μSR signals. An order-disorder transition is observable between $H = 2$ T and $H = 3$ T. These measurements were performed after field cooling to $T = 20$ mK at $H = 1$ T. The curves are offset vertically for visual clarity. The reference signals used in the deconvolution process (not shown) were measured on the same sample at $T = 10$ K, well above the irreversibility line.

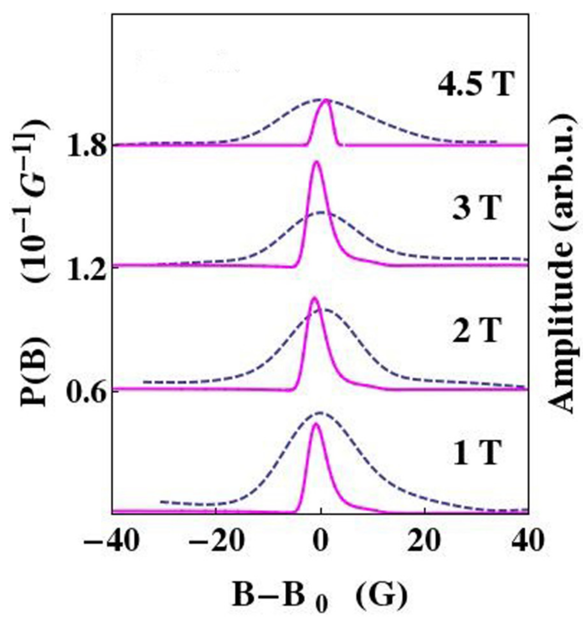

FIG. 2. (Color online) Probability magnetic field distribution, $P(B)$, for κ-$(ET)_2Cu[N(CN)_2]Br$ (magenta solid curves and left-hand scale) at $T = 20$ mK and different external magnetic fields, obtained by deconvoluting the FFTs (blue dashed curves and right-hand scale) of the μSR signals. A disordering transition is observed between $H = 3$ T and $H = 4.5$ T. These measurements were performed after field cooling to $T = 20$ mK at $H = 1$ T. As in Fig. 1, the reference signals recorded at $T = 10$ K are not shown.

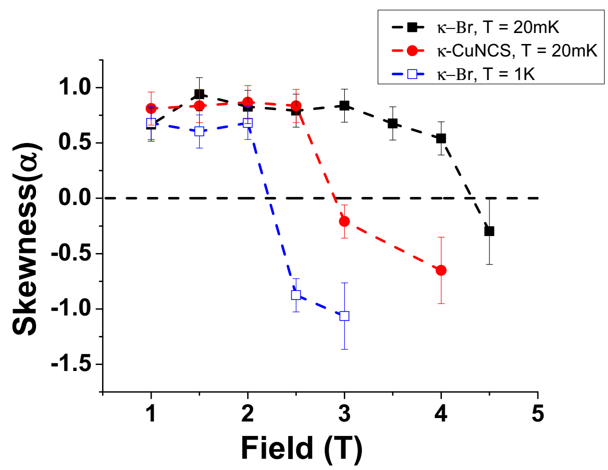

FIG. 3. (Color online) Field dependence of the skewness parameter (α) for κ-$(ET)_2Cu(NCS)_2$ at $T = 20$ mK (red solid circles), and for κ-$(ET)_2$ $Cu[N(CN)_2]Br$ at $T = 20$ mK (black solid squares) and $T = 1$ K (blue open squares). The lines connecting the data points are guides to the eye.

of the vortex lattice against thermal fluctuations up to high magnetic fields.

IV. DISCUSSION

For the first time, the high-field, low-temperature region of the vortex state of the quasi-2D organic superconductors, κ-CuNCS and κ -Br, have been investigated by μSR. At the lowest temperature considered ($T = 20$ mK), an order-disorder crossover field, as determined from the sign change of the skewness parameter α, is found for both materials—occurring near $H = 2.5$ T and 4 T for κ-CuNCS and κ-Br, respectively. These values are of the order of the respective irreversibility fields extracted from low-temperature magnetization measurements [25,26], and much larger than the 3D-2D crossover fields for electromagnetically coupled layers of pancake vortices that have been reported for these materials (e.g., $H_{2D} \sim 10^{-2}$ T for κ-CuNCS [14]). Furthermore, the observation of positive values of α in κ-Br for magnetic fields up to $H = 2$ T at the higher temperature of $T = 1$ K reflects a robustness of the 3D vortex lattice to thermal fluctuations of pancake vortices in the individual SC layers. Analytical calculations based on a 2D Ginzburg–Landau theory [7,8], which agree well with numerical Monte Carlo simulations [6], show, for material parameters characteristic of κ-CuNCS [see Fig. 3(b) in Ref. [29]], that melting of an ordered vortex lattice at $T = 0.1T_c \approx 1$ K, in each individual 2D SC layer, occurs around $H = 0.4H_{c2} \approx 2$ T. Moreover, the irreversibility field [25], which may be associated with a critical field for depinning of vortices, follows closely the 2D vortex-lattice melting field [also shown in Fig. 3(b) of Ref. [29]]. Numerical simulations of the skewness parameter α, performed for weakly coupled layers of pancake vortices [30], show that a 3D-2D crossover reduces α to about 30% of its (positive) low-field value, whereas melting of the constituent 2D vortex lattices causes the sign of α to abruptly change from positive to negative. Since the values of α shown in Fig. 3 below the order-disorder transition fields are larger than that predicted for a disordered multilayer of 2D vortex lattices (i.e., $\alpha \simeq 0.3$) [30], it is conceivable that no 3D-2D crossover takes place in the low-field region. Instead, it appears that the only order-disorder transition that occurs in the entire field range investigated is melting of a partially ordered 3D vortex lattice. This conclusion is consistent with the single first-order vortex lattice melting transition predicted in a Monte Carlo study of the Lawrence–Doniach model for a layered superconductor [31].

It should be noted that the values of the skewness parameter extracted from our measurements in the high-field regimes, i.e., $0.5 < \alpha < 1.0$, are quite similar to those predicted recently for glassy vortex phases in BSCCO [32], where interlayer vortex correlations are substantially reduced with respect to the fully ordered 3D vortex-lattice state. The transition from the low-field 3D ordered vortex-lattice state to such high-field vortex-glass phases, revealed by the μSR measurements in Ref. [32], was found to depend on the doping level of the crystals. In the optimally doped crystal the transition occurred through an intermediate phase consisting of a pinned-vortex liquid, characterized by a negative value of α, whereas in the over-doped crystal a direct transition between the two vortex solid phases took place. In both cases no indication of a 3D-2D crossover, similar to that proposed for κ -CuNCS in Ref. [14], was observed.

The apparent absence of a low-field 3D-2D crossover in our measurements suggests that the observed 3D partially ordered vortex lattice is probably stabilized by strong pinning due to a small concentration of defects. This scenario is supported by the proximity of the irreversibility (depinning) transition to the melting transition, previously observed for κ-CuNCS [29]. It should be noted that the negative values of α, attributed here to the presence of vortex liquid phases in both materials, arise from some nontrivial intralayer three-body correlations [32], which were also shown to yield negative α values for glassy phases in much less anisotropic superconductors, such as $La_{1.9}Sr_{0.1}CuO_{4-\delta}$ [33].

The primary source of flux-line pinning may be a consequence of the high rate at which the samples were precooled when loaded into the dilution refrigerator. During the precooling stage, the sample is cooled from room temperature to 4.2 K in about 15 to 20 minutes, passing through the glass-like structural transition at $T_g \sim 75$ K, which—for the κ-phase ET salts with polymeric anions $Cu(NCS)_2$ and $Cu[N(CN)_2]Br$—is related to a certain degree of disorder in the orientational degrees of freedom of the ET molecules' ethylene end groups (EEGs) (see Ref. [2] for an overview). The EEG orientations are thermally disordered at room temperature and, for kinetic reasons, become frozen in a nonequilibrium configuration depending on the cooling rate at T_g [34]. The precooling rate in our experiments was about 20 K/min, which corresponds to a random lattice potential associated with structural disorder in 3% to 4% of the sample [35]. Clustering of metastable EEG configurations in this volume fraction of the sample and into domain sizes exceeding the in-plane coherence length [36] is a likely source of the strong flux-line pinning that accommodates the partially ordered vortex lattice.

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