

## Observation of a transverse Meissner effect in $\text{Cu}_x\text{TiSe}_2$ single crystals

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We report on local magnetization measurements showing the presence of an unexpectedly strong transverse Meissner effect in the superconducting  $\text{Cu}_x\text{TiSe}_2$  single crystals. We show that for tilted magnetic fields ( $H_a$ ) vortices remain aligned with the  $ab$  planes up to field  $H_k \sim H_p / \sin \theta_H$  (where  $\theta_H$  is the angle between the applied field and the  $ab$  plane and  $H_p$  the first penetration field) and that for  $H_a > H_k$ , the field dependence of the vortex direction  $\theta_B(H_a)$  can be well described assuming that vortices remain partially locked in the planes forming a staircase structure of average direction  $\theta_B \neq \theta_H$ . These results indicate the existence of a strong modulation of the vortex core energy along the  $c$  axis but its origin remains unclear.

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Layered type-II superconductors have attracted considerable attention over the past decades since their anisotropic structure gives rise to a large variety of novel vortex phases (for a review, see [1]). Indeed, the structure of the vortex matter is determined by the minimum of the total Gibbs energy which is sensitive to various microscopic parameters such as the mass anisotropy  $\Gamma = \sqrt{m_c/m_{ab}}$ , the Ginzburg-Landau parameter  $\kappa = \lambda_{ab}/\xi_{ab}$  ( $\lambda_{ab}$  and  $\xi_{ab}$  being the in-plane London penetration depth and coherence length, respectively), and/or the nature of the defects present in the sample. In the case of large  $\Gamma$  ( $> 7$ ) and  $\kappa$  ( $> 10$ ) values, the angular dependence of the Gibbs energy [2] has, for instance, two degenerated minima and two distinct crossed vortex lattices can then coexist in the sample at the same time [3,4]. Furthermore, when  $\xi_c$  becomes on the order of the interlayer distance  $d$ , the modulation of the superconducting order parameter from plane to plane leads to the existence of a lock-in phase for which vortices penetrate into the sample with their normal cores locked between the layers [5,6] up to a field  $H_L \sim [H_p / \sin \theta_H][d/\xi_c]$  (where  $\theta_H$  is the angle between the applied field and the plane and  $H_p$  the first penetration field at angle  $\theta_H$ ). Experimental evidence for this *intrinsic* lock-in effect have been obtained in both high- $T_c$  cuprates [7] and organic materials [8].

On the other hand, vortices can also be trapped by correlated pinning sites such as columnar defects [9] or twin planes [10] giving rise to an *extrinsic* lock-in effect (so-called *transverse* Meissner effect). This effect can then be observed for field orientations close to the correlated defects and the magnetic induction perpendicular to the defects remains null up to  $H_L \sim [H_p / \sin \theta_H]\sqrt{\varepsilon_P/\varepsilon_I}$  [11] where  $\theta_H$  is now the angle between the magnetic field and the defect orientation,  $\varepsilon_I$  the line tension, and  $\varepsilon_P$  the amount of core energy suppressed by the artificial structure. Note that in both an intrinsic and an extrinsic case,  $H_L$  scales as  $1/\sin \theta_H$  and such a scaling is hence a strong signature for the existence of a lock-in effect. Finally note that, in the Bean model, the *irreversible* magnetic moment can

also remain locked along the normal direction of thin plates up to a “critical” angle  $\theta_c \approx \arctan(w/l)$  due to geometrical effects [12] where  $w$  and  $l$  are the width and thickness of the slab, respectively.

We present here a detailed study of the field distribution in Cu-doped  $\text{TiSe}_2$  [13] single crystals. The main result of our study is the clear evidence of an unexpected and large transverse Meissner effect for fields oriented close to the  $ab$  planes. We show that vortices remain trapped along the layers up to an applied field  $H_k(\theta_H) \sim H_p / \sin \theta_H$  ( $\theta_H$  being the angle between the field and the plane) which can be attributed neither to the *intrinsic* lock-in effect as  $\xi_c \approx 20$  nm [14] is much larger than  $d \approx 0.6$  nm, nor to the existence of crossed lattices as the anisotropy is too small ( $\Gamma \sim 1.7$ ). Our study hence strongly indicates the presence of correlated “defects” parallel to the  $ab$  planes leading to a strong modulation of the vortex core energy along the  $c$  direction. The nature of those defects still has to be clarified.

$\text{Cu}_x\text{TiSe}_2$  belongs to the group of transition-metal dichalcogenides and shares some similarities with other unconventional materials such as their layered structure and the presence of an electronic instability competing with the superconducting state. Indeed,  $\text{TiSe}_2$  hosts an excitonic charge density wave [15] which is progressively destroyed by copper intercalation between the  $\text{TiSe}_2$  layers [13] giving rise to a superconducting dome in the phase diagram emerging above a doping content  $x \sim 0.04$  with a maximum critical temperature  $T_c \sim 4.1$  K reached around  $x \sim 0.08$  where the quantum critical point is supposed to be. Three single crystals with different dopings from the underdoped, optimally doped to (slightly) overdoped regions of the phase diagram ( $T_c = 2.8, 4.1,$  and  $3.8$  K, i.e.,  $x \sim 0.06, 0.08,$  and  $0.085$  for samples 1, 2, and 3, respectively) have been investigated. The response of the samples to an applied magnetic field has been measured by an array of ten miniature GaAs-based quantum well Hall sensors with dimensions  $10 \times 10 \mu\text{m}^2$  and a pitch of  $30 \mu\text{m}$ . The sample

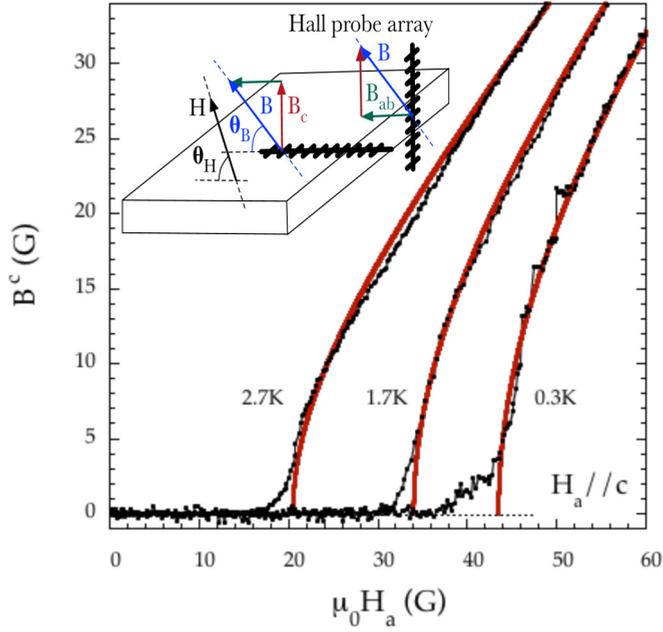


FIG. 1. Field dependence of the magnetic induction  $B^c$  for  $H_a \parallel c$  in a  $\text{Cu}_x\text{TiSe}_2$  single crystal at the indicated temperatures (sample 3). Solid (red) lines are fits to the data in the presence of geometrical barriers (see [20] and text for details). A small upturn in  $B(H_a)$  visible close to  $H_p$  (corresponding to 5–10 vortices) can be attributed to the presence of low but nonzero bulk pinning. Inset: Scheme of the experimental setup to measure different components of  $B$  by Hall probes.

(of typical dimensions  $\sim 500 \times 500 \times 100 \mu\text{m}^3$ ) was placed on top of the probes and cooled down in zero magnetic field to the lowest temperature of 0.3 K. The Hall voltage has been converted into local magnetic induction  $B$  using the respective probe sensitivity. The field dependence for both components of the induction,  $B^c = B \sin \theta_B$  and  $B^{ab} = B \cos \theta_B$ , were obtained by placing the probes either perpendicularly or parallel to the sample  $ab$  planes (see sketch in the inset of Fig. 1) for a given temperature and  $\theta_H$  value. The  $B^c/B^{ab}$  ratio between those two components then led to the field dependence of the vortex orientation  $\theta_B(H_a)$ . As discussed below for an applied field oriented close to the  $ab$  planes,  $\theta_B$  clearly differs from  $\theta_H$  for low magnetic fields but tends towards  $\theta_H$  with increasing fields.

The main panel of Fig. 1 displays the field dependence of  $B^c$  for increasing  $H_a$  oriented parallel to the  $c$  axis ( $\theta_H = 90^\circ$ ). For small magnetic field  $B^c$  remains zero as the sample screens out the field from the Hall sensors in the Meissner state. When  $H_a$  reaches the first penetration field  $H_p$ , vortices start to penetrate into the sample and the signal becomes nonzero. This sharp increase in  $B^c$  (common to all three samples) clearly indicates that pinning by point defects is extremely small in this system [16,17]. As shown in Fig. 1 (solid red lines),  $B^c(H_a)$  can be well described by a  $\mu_0 H_a \sqrt{1 - (H_p/H_a)^2}$  law as proposed by Zeldov *et al.* [18] in the case of a penetration process dominated by geometrical barriers [19,20]. The small upturn in the signal visible close to  $H_p$  (corresponding to 5–10 vortices) indicates the presence of very low [17] but nonzero bulk (point) pinning which does not change the conclusions

drawn below. The main influence of the geometrical barriers is further confirmed by the domelike shape of the field profiles reported in [16] for  $H_a > H_p$ , clearly showing that the vortices first accumulate in the center of the sample.  $H_p$  was thus detected by probes located close to the center of the sample but for the discussion below it is important to note that geometrical barriers do not play any role for fields exceeding  $\sim 2H_p$  [18,21] and the lock-in effect discussed below cannot be related to the field penetration process.

The  $B^c(H_a)$  dependence measured for various field orientations  $\theta_H$  is displayed in Fig. 2(a). As discussed above, for large field angles  $\theta_H$ ,  $B^c$  sharply increases when vortices penetrate into the sample at  $H_a = H_p$ . On the other hand, for fields oriented close to the  $ab$  plane, a second characteristic field can be clearly recognized in the  $B^c(H_a)$  curves. As shown for  $\theta_H < 23^\circ$ ,  $B^c$  remains close to zero well above the first penetration field and a sharp kink appears in  $B^c$  when  $H_a$  reaches a characteristic field, called here  $H_k$ . The reduced  $B^c$  value observed for  $H_p < H_a < H_k$  is strongly suggesting that vortices penetrating into the sample remain trapped along the  $ab$  planes and leave the sample through the sides perpendicular to the  $ab$  planes, being hence not detected by the Hall probes. The nonzero  $B^c$  signal in this field range probably originates from the small but nonzero distance between the probe and the sample surface. When  $H_a = H_k$ , vortices unlock and the rapid change in their orientation leads to the sharp increase in  $B^c$  (see discussion below). Note that the angular dependence of  $H_p$  depends on the geometrical factors and the mass anisotropy with  $H_p^{ab}/H_p^c \sim (\sqrt{w/l})(H_{c1}^{ab}/H_{c1}^c) \sim \sqrt{w/l}\Gamma^2$  [18,21]. For sample 2,  $H_p^{ab}/H_p^c \sim 1$  (geometrical factors compensating the anisotropy) rising up to  $\sim 2$  in samples 1 and 3. However, since  $H_k$  largely exceeds this value for small angles, the observed increase in  $B_c$  at  $H_k$  cannot be mistaken for vortex penetration.

Figure 2(b) displays the curves from Fig. 2(a) plotted as a function of  $H_a \sin \theta_H$ . As clearly shown, all the upturns in  $B^c$  at  $H_k \sin \theta_H$  collapse onto the same value as expected for the lock-in effect. Note that the critical lock-in angle scales as  $\sin \theta_H \propto 1/H_a$  and can hence not be attributed to geometrical effects [12]. Moreover  $H_k \sin \theta_H \sim H_p^c$  emphasizing that the lock-in field is close to its maximum value and the lock-in effect is particularly strong in our system. This scaling has been observed in all measured samples (i.e., whatever the doping content) and at temperatures up to  $T/T_c \sim 0.7\text{--}0.8$  (see Fig. 3). For higher temperatures  $H_k$  could not be distinguished from  $H_p$  anymore.

To analyze the field dependence of the vortex orientation into more details both  $B^c$  and  $B^{ab}$  components of the magnetic induction were needed. The inset of Fig. 2(a) displays the field dependence of  $B^{ab}/\cos \theta_H = B[\cos \theta_B/\cos \theta_H]$  and  $B^c/\sin \theta_H = B[\sin \theta_B/\sin \theta_H]$  for  $\theta_H = 25^\circ$ . If vortices were aligned with the magnetic field ( $\theta_H = \theta_B$ ) the two signals would be equal (to  $B$ ), which is obviously not the case. Indeed, in contrast to the rapid increase in  $B^{ab}/\cos \theta_H$  for  $H_a > H_p$ , the field detected perpendicular to the planes  $B^c/\sin \theta_H$  remains small up to  $H_k$ . This clearly shows that for  $H_a > H_p$  vortices present in the sample do not intersect the  $ab$  plane and remain locked in this plane up to  $H_k$ . The field dependence of the vortex orientation ( $\tan \theta_B/\tan \theta_H$ ) for  $H_a > H_k$  is obtained by dividing the two components of the magnetic induction and is displayed in Fig. 4(b).

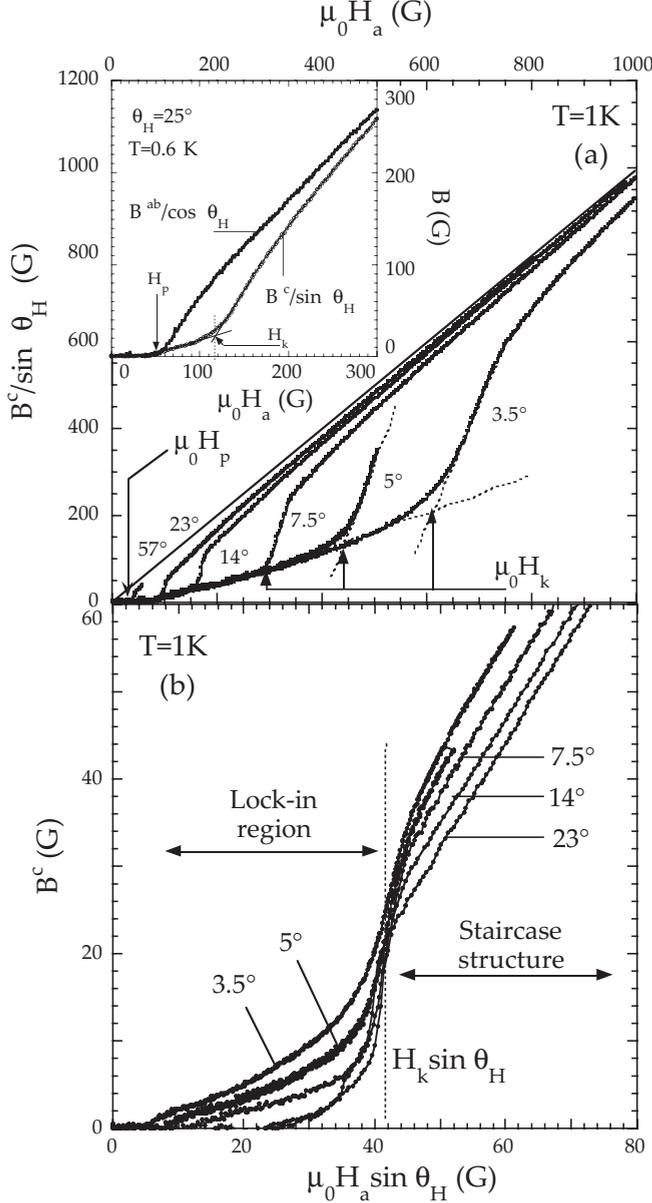


FIG. 2. Upper panel (a): Magnetic field dependence of  $B^c/\sin\theta_H = B[\sin\theta_B/\sin\theta_H]$  in  $\text{Cu}_x\text{TiSe}_2$  (sample 3) for the indicated field orientations  $\theta_H$  ( $\theta_B$  being the orientation of the induction inside of the sample; see text for details). As shown for large  $\theta_H$  values,  $B^c$  rapidly increases for  $H_a > H_p$  (see also Fig. 1 for  $\theta_H = 90^\circ$ ), whereas for smaller  $\theta_H$  values,  $B^c$  remains small ( $\theta_B \approx 0$ ) up to  $H_k$ , which clearly indicates the presence of a transverse Meissner effect. The nonzero  $B^c$  value (for  $H_p < H_a < H_k$ ) probably originates in the small but nonzero distance between the probe and the sample surface. As shown,  $B^c$  (and hence  $\theta_B$ ) rapidly increases for  $H_a > H_k$  indicating a rapid change in the orientation of the flux lines. Inset: Magnetic field dependence of the  $B^c/\sin\theta_H$  and  $B^{ab}/\cos\theta_H$  components of the induction for  $\theta_H = 25^\circ$  at  $T = 0.6$  K (sample 2). Lower panel (b): Curves from (a) scaled by  $\sin\theta_H$  showing that  $H_k \sin\theta_H \sim 40$  G (at 1 K), being close to the  $H_p^c$  value of the sample (see also Fig. 3).

To theoretically describe this field dependence, we started with the standard London model. In the absence of disorder,

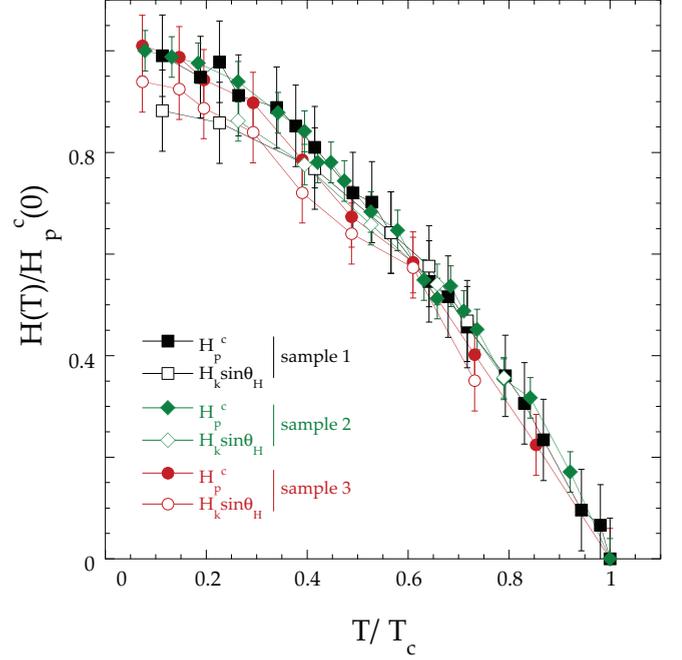


FIG. 3. Temperature dependence of the first penetration field  $H_p^c$  (solid symbols) for  $H_a \parallel c$  in sample 1 (black squares), 2 [(green) diamonds], and 3 (red circles) for  $H_a \parallel c$  (see Fig. 1) and  $H_k \sin\theta_H$  (open symbols) where  $H_k$  is the lock-in field observed for tilted magnetic fields (see Fig. 2 and text for details). As shown, in all samples,  $H_k \sin\theta_H \sim H_p$ , tracking its temperature dependence on a large part of the diagram.

the vortex structure results from the minimization of the total Gibbs energy density [2]:

$$G = \frac{B^2}{2\mu_0} + \frac{\varepsilon_l B}{\Phi_0} f(\theta_B, \Gamma, \kappa) - BH \cos(\theta_B - \theta_H) \quad (1)$$

for  $H \sim H_{c1}$  ( $H_a \sim H_p$ ), where  $\Gamma$  is the anisotropy,  $\kappa$  the Ginzburg-Landau parameter,  $\varepsilon_l$  the line tension:  $\Phi_0^2 \ln \kappa / 4\pi\mu_0 \lambda_{ab}^2$ , and  $f(\theta, \Gamma, \kappa)$  the angular dependence of the core energy [2]. Given the reduced  $\kappa$  and  $\Gamma$  values of our system, the observed lock-in effect cannot be related to the possible coexistence of crossed lattices [2] and it is hence necessary to add some extra (pinning) contribution to the Gibbs energy in order to account for the observed behavior. As suggested by Blatter *et al.* [11] the formation of a staircase structure in the presence of correlated defects can be described by assuming that this pinning energy can be written as  $\mathcal{E}(\theta_B, r(\theta_B)) = r(\varepsilon_l/\Gamma - \varepsilon_p) + s\varepsilon_l f(\theta_B) - t\varepsilon_l f(\theta_B)$  [see sketch in the inset of Fig. 4(b) for the definition of  $r$ ,  $s$ , and  $t$ ]. The “optimal” energy gain  $[\mathcal{E}_{opt}(\theta_B, r_{opt})]$  is then obtained by minimizing  $\mathcal{E}$  with respect to  $r$  and the corresponding energy density  $E = B\mathcal{E}_{opt}/\Phi_0 t$  is obtained by accounting for a small but finite density of vortices equal to  $1/a_0^2 t$  (with  $a_0^2 = \Phi_0/B$ ).

We have extended the model discussed in [11] in order to include the anisotropy factor  $\Gamma$  and large  $\theta_B$  values. Numerical solutions for selected  $\Gamma$  and  $\varepsilon_R = \Gamma\varepsilon_p/\varepsilon_l$  values are displayed in the insets of Fig. 4(a) together with the analytical solution obtained for isotropic systems (dotted line) for which  $E(\theta_B) = \varepsilon_l(B/\Phi_0)[(1 - \varepsilon_R)\cos\theta_B +$

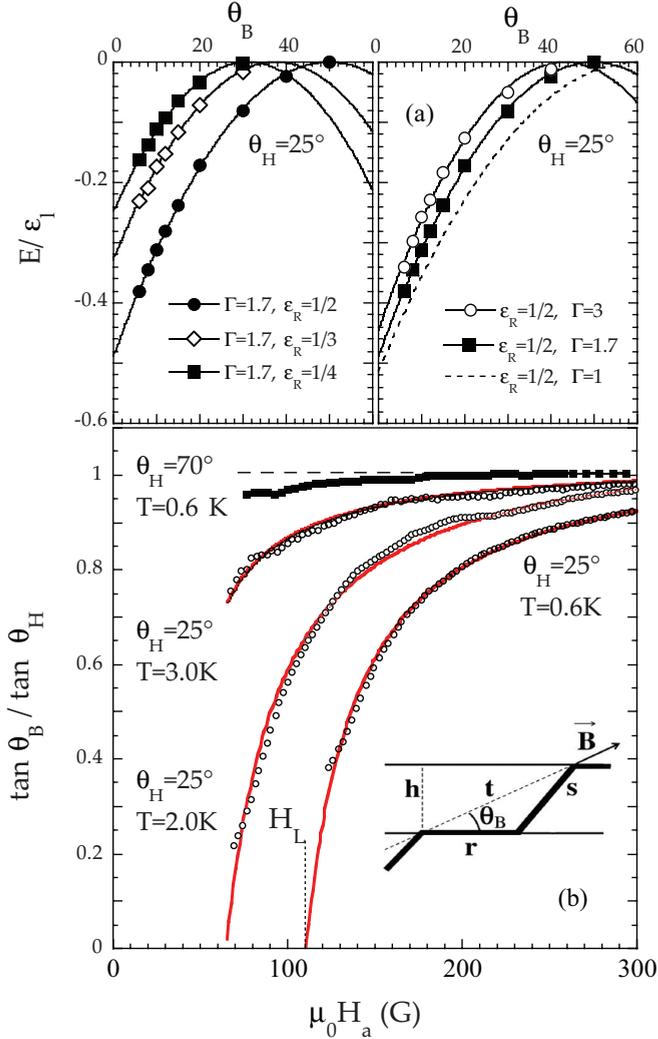


FIG. 4. Upper panel (a): Angular dependence of the pinning energy for the staircase vortex structure sketched in the inset of (b) and for the indicated  $\varepsilon_R = \Gamma \varepsilon_p / \varepsilon_l$  and  $\Gamma$  values (see text for details). The dotted line (right panel) corresponds to the analytical dependence obtained in the isotropic case ( $\Gamma = 1$ ) and the solid lines are parabolic fits to the data (see text for details). Lower panel (b): Field dependence of the orientation of the induction ( $\theta_B$ ) for  $\theta_H = 25^\circ$  (open circles) and  $\theta_H = 70^\circ$  (closed squares) at the indicated  $T$  values. Data for  $\theta_H = 25^\circ$  and  $T = 0.6$  K has been deduced from the measurements displayed in the inset of Fig. 2(a). The solid (red) lines are fits to the data using Eq. (2). As shown, for  $\theta_H = 25^\circ$ , a very reasonable agreement is obtained up to 3.0 K ( $0.7T_c$ ), whereas for large  $\theta_H$  values ( $\theta_H > \theta_t$ ),  $\theta_B \approx \theta_H$  even at low temperature.

$\sqrt{2\varepsilon_R - \varepsilon_R^2} \sin \theta_B - 1$ ]. As shown,  $E$  can be very well described by a simple parabolic approximation for all investigated  $\varepsilon_R$  and  $\Gamma$  values (solid lines):  $E(\theta_B, \Gamma, \varepsilon_R) \approx -\frac{1}{2} \varepsilon_l(\Gamma, \varepsilon_R)(\theta_B - \theta_t(\Gamma, \varepsilon_R))^2$  hence generalizing the result previously obtained for small angles in [11]. As shown,  $\varepsilon_l$  is almost independent of  $\varepsilon_R$  (increasing slightly with  $\Gamma$ ), whereas the critical trapping angle  $\theta_t$  increases for increasing  $\varepsilon_R$  values [with  $\tan \theta_t = \sqrt{2\varepsilon_R - \varepsilon_R^2} / (1 - \varepsilon_R)$  in the isotropic case]. In this parabolic approximation, the minimization of the total

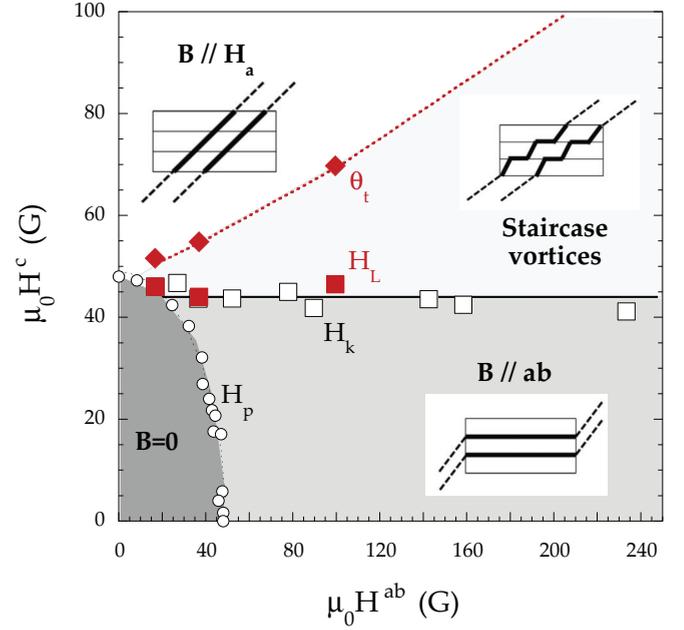


FIG. 5. Phase diagram of the vortex structure in  $\text{Cu}_x\text{TiSe}_2$  as a function of the field orientation (sample 2).  $H_k$  and  $H_p$  values have been obtained directly from the  $B^c(H_a)$  curves for various field orientation [see Fig. 4(a) for  $\theta_H = 25^\circ$ ] while  $H_L$  and  $\theta_t$  are the parameters deduced from the fits to the data using Eq. (2) [solid red lines in Fig. 4(b)]. Similar phase diagrams were obtained for all three studied samples with different copper concentration.

Gibbs energy density with respect to  $\theta_B$  then leads to >

$$\frac{\tan \theta_B}{\tan \theta_H} \approx \begin{cases} 0, & H < H_L \\ \frac{1 - \frac{H_L}{H}}{1 - \frac{H_L \tan \theta_H}{H \tan \theta_t}}, & H_L < H, \theta_H < \theta_t \\ 1, & \theta_t < \theta_H, \end{cases} \quad (2)$$

where the lock-in field  $H_L \approx [H_{c1}^c / \sin \theta_H] \mathcal{F}(\Gamma, \varepsilon_R)$  [with  $\mathcal{F} = (2\varepsilon_R - \varepsilon_R^2)^{1/2}$  for  $\Gamma = 1$ ]. This equation describes three phases of vortex structure: at first the lock-in effect ( $\theta_B = 0$ ) for  $H < H_L$ , then the staircase structure for  $H > H_L$ , and finally  $\theta_B = \theta_H$  when vortices align with the applied field.

As shown in Fig. 4(b) (solid red lines), very reasonable fits to the data can be obtained using Eq. (2) supporting the existence of a staircase structure for  $H_a > H_k$ , on a large temperature range (see also Fig. 3). As expected, the  $H_L$  values deduced from Eq. (2) are indeed equal to the  $H_k$  values directly deduced from the sharp increase in  $B^c$  observed in Fig. 2(a). Note that since  $\Gamma = 1.7$ , the small but nonzero angular dependence of the second term in Eq. (1) has been neglected. However, we have numerically checked that this angular dependence only induces small shifts in the  $\theta_B$  values (typically by  $1^\circ$ – $2^\circ$ ). Similarly, we did not take into account the field dependence of the demagnetization effect simply assuming that the real magnetic field  $H = \alpha(\theta_H)H_a$  but again, this only induces minor quantitative changes in the fitting parameters as  $\alpha$  is close to 1 for  $\theta_H = 25^\circ$ .

In summary, Fig. 5 displays the vortex phase diagram of  $\text{Cu}_x\text{TiSe}_2$  crystals deduced from our vectorial magnetization measurements. Very similar phase diagrams were obtained for

all three samples. We have shown that vortices remain locked along the  $ab$  planes, up to a lock-in field  $H_L$ . For  $H_a > H_L$  the field dependence of the orientation of the vortices can be well described assuming that the vortex matter forms a staircase structure.  $H_L = [H_p^c / \sin \theta_H] \mathcal{F}(\Gamma, \varepsilon_R) \approx H_p^c / \sin \theta_H$  indicating that the suppression of the core energy is very strong ( $\varepsilon_R \sim 1$ ), largely exceeding the value expected for twin planes ( $\varepsilon_R \sim 10^{-3}$ ) [11]. It has been suggested [22] that spatial modulations of the superconducting order parameter could exist in systems with competing—and coupled—order parameters in the presence of defects which could locally alter the competing phase but, as the effect has been observed in all three samples (from underdoped to slightly overdoped),

it cannot be directly related to the charge properties of the system. The nature of the defects leading to such a strong modulation of the core energy hence still has to be clarified.

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