

Why pinning by surface irregularities can explain the peak effect in transport properties and neutron diffraction results in NbSe₂ and Bi-2212 crystals?

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Abstract. The existence of a peak effect in transport properties (a maximum of the critical current as function of magnetic field) is a well-known but still intriguing feature of Type II superconductors such as NbSe₂ and Bi-2212. Using a model of pinning by surface irregularities in anisotropic superconductors, we have developed a calculation of the critical current which allows estimating quantitatively the critical current in both the high critical current phase and the low critical current phase. The only adjustable parameter of this model is the angle of the vortices at the surface. The agreement between the measurements and the model is really very impressive. In this framework, the anomalous dynamical properties close to the peak effect is due to coexistence of two different vortex states with different critical currents. Recent neutron diffraction data in NbSe₂ crystals in the presence of transport current support this point of view.

Keywords. Vortex pinning; neutron scattering; critical current.

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1. Introduction

The understanding of the values of the critical current in a Type II superconductor is still a challenging problem. The competition between elastic properties and disorder induced by pinning leads theoretically to different vortex matter states. In this respect, the peak effect observed in few Type II superconductors, i.e. a sudden increase of the critical current close to the superconducting-normal transition, has

been for a long time considered as a proof of a disorder transition in the vortex lattice. Bulk pinning centers can become more effective on a less rigid lattice at high field and that can lead to a peak effect. Larkin–Ovchinnikov collective pinning model [1] provides a more precise theoretical approach to the link between the loss of long range order and the high critical current. Numerous experiments have also shown that the link between vortex lattice order and critical current, is far from being direct, in contradiction to the previous assumptions. Thorel’s neutron scattering experiments have first shown that the flux line lattice (FLL) quality can be modified without changing the critical current [2]. A possible explanation, already proposed by different authors [3], is that the exact order of the FLL in the bulk is not the most important parameter governing transport properties.

In addition to the peak effect, very peculiar transport properties are observed in the same region of the phase diagram, in particular hysteretic $V(I)$ curves [4]. A model has recently emerged, supported by different experiments made in 2H-NbSe₂ [5]. The key ingredients are a supercooling of a high critical current into a low critical current state, and an annealing effect over surface barrier. To explain the high critical current phase, it is usual to involve a strongly disordered FLL, amorphous or liquid like, created after a genuine phase transition through the peak effect. Nevertheless, very little is known about the genuine bulk structure of these phases. There are even contradictory and puzzling results. Indeed, recent decoration experiments have shown that no disordered state can be specially evidenced in the peak effect region of pure or Fe-doped NbSe₂ samples [6]. The high critical current FLL state remains unexplained.

In the present paper, we will review our recent and less recent results from different methods to test the role of the surface pinning in the critical current values. In the first part, we will discuss the model of pinning by surface irregularities to calculate the critical current without any adjustable parameter (the surface roughness is measured in this case). Then we will discuss the case of the peak effect and conclude on Bi-2212 system in which the very large anisotropy makes it difficult to calculate the elasticity.

2. Surface treatments in Nb films

The sample used is a film of niobium (thickness = 3000 Å) deposited at 780°C on a sapphire substrate by the ion beam technique. The film has a resistivity of about 0.5 $\mu\Omega\cdot\text{cm}$ at the critical temperature, $T_c = 9.15$ K, and exhibits a surface rms roughness of 5 nm, measured by atomic force microscopy (Nanoscope III, Digital Instruments). Microbridges of $W = 10\ \mu\text{m} \times L = 30\ \mu\text{m}$ have been patterned using a scanning electron microscope, this irradiation step being followed by a reactive ion etching process. The critical currents have been measured by means of the standard four-probe technique, at 4.2 K in the whole range of field values covering the mixed state. The critical current values I_c were determined with a voltage criterion of 10 nV. We have therefore measured the microbridge roughness using AFM in the tapping mode over the whole surface of the microbridge. The next step was to use a focused ion beam etching (FIBE) to etch its surface and then to modify the surface structure. Following a simple analysis of the surface roughness

described elsewhere [7], we have extracted a roughness α of about 2.2° for the rough surface, to be compared to its value of 0.6° before processing. In figure 1, we have presented the critical current measured as a function of magnetic field B at 4.2 K. In order to analyze the data, we have also presented in the same figure the reversible magnetization ε . In the Mathieu–Simon (MS) theory [3] of surface pinning, there is a direct relation between these two quantities in the case of an isotropic Type II superconductor for a single surface:

$$I_c/W = \varepsilon \sin(\alpha). \quad (1)$$

It is out of the scope of this paper to describe in detail all the subtleties of the MS theory that can be found elsewhere [3,8–10], but let us summarize in a few words the idea of this model. The two fundamental equations of this model are the Maxwell equation and the current conservation at the sample surface:

$$\mathbf{J}_s + \text{curl } \varepsilon = 0, \quad (2)$$

$$\varepsilon \mathbf{n} = 0, \quad (3)$$

(\mathbf{J}_s is the superconducting current and \mathbf{n} is the vector perpendicular to the perfect flat surface). A very important point of the model is that ε is a function not of the magnetic field B , but of the local field ω , which is given by $\omega = B - (1/\mu_0) \text{curl } \mathbf{J}_s$. In the ideal case where the vortices are perpendicular to the ideal flat surface, $\mathbf{J}_s = 0$. In the case of a real surface, the vortices can join the surface with a maximum angle α and a non-dissipative current may flow close to the surface ($\mathbf{J}_s = -\varepsilon(\omega) \sin(\alpha)$ in the isotropic case). Above the critical current, the vortices start to move with a velocity, which is proportional to $I - I_c$ which is the dissipative current flowing in the sample thickness. This perfectly explains the shape of the I – V curves ($V = R(I - I_c)$ where $I_c = \mathbf{J}_s W$). In the case of an anisotropic sample with an anisotropy γ , the angle α is the angle between ε and the vector \mathbf{n} , but the vortex lines ω are inclined by a larger angle θ such as $\tan \theta = \gamma^2 \tan \alpha$. For example, if $\alpha = 1^\circ$, $\theta = 9^\circ$ for $\gamma = 3$. Following the calculations in ref. [8], one finds

$$I_{c0} = w(B_{c2}^* - B)/(2\mu_0 1.16\gamma^2 \kappa^2), \quad (4)$$

where

$$B_{c2}^* = B_{c2}(1 + \gamma^2 \tan^2 \alpha)^{-1/2}. \quad (5)$$

B_{c2} is the second critical field, and κ is the ratio of the two characteristic lengths, λ and ξ .

In the inset of the top panel of figure 1, we have reported the value of α calculated from eq. (1). One can see that α is roughly constant and equal to what is observed by AFM. The damaged surface presents a roughness which is increased from 0.8° to 1.6° . One can see that it gives very reasonable values, with a rather small magnetic field dependence. This type of analysis gives a simple explanation for the high critical current density observed in this kind of clean thin films, compared to the moderate one observed in bulk crystals. It also gives evidence that the interaction between the surface corrugation and the vortex elasticity is a key point for understanding the vortex lattice pinning and dynamics.

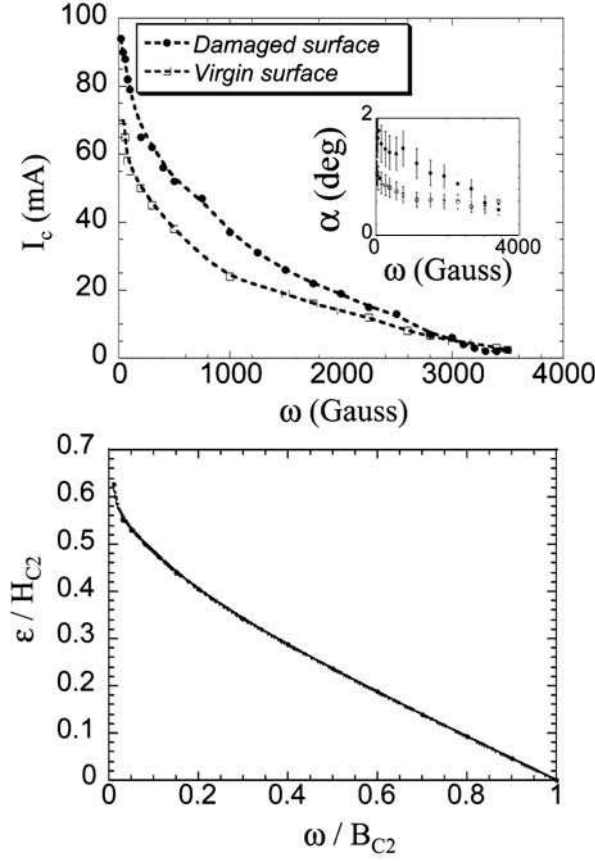


Figure 1. The critical current of the virgin niobium microbridge as a function of the magnetic field at 4.2 K, for two different surface states. In the inset, the calculated value of the angle α for the two surface roughnesses is shown. The calculation of α was performed using the value of ε shown in the lower panel of the figure.

3. Surface treatments in NbSe₂ crystals

The fact that this model works quite well in a thin film is indeed impressive, but the same type of results can be observed in NbSe₂ crystals. Large single crystals of H-NbSe₂ (size = $8 \times 6 \times 0.5$ mm³, $T_c(0) = 7.5$ K measured by specific heat) were used. The magnetic field was applied parallel to the c -axis of the crystal. In figure 2, we have presented some typical results in these crystals. The data can be fitted easily by the MS model with a single adjustable parameter irregularities α of 0.9° for the pristine sample. One can note that in the Abrikosov regime, ε decreases linearly with ω . By sandblasting the surface, one increases α from 0.9° to 2.4° . By cleaving the sample after sandblasting, it decreases back to 1.1° . In each case, the critical current follows the model described in eqs (4) and (5). One should

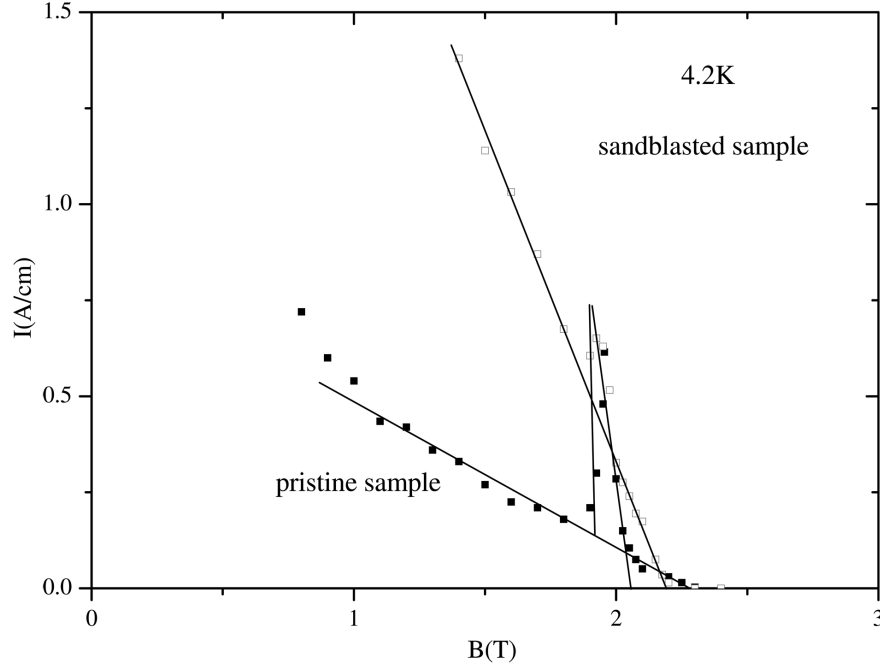


Figure 2. The critical current as a function of the magnetic field at 4.2 K for the same NbSe₂ sample before and after sandblasting, showing the influence of the surface roughness on the peak effect.

note here that B_{c2}^* decreases as α increases as can be seen in figure 2. The other three parameters of the fit (B_{c2} , γ and κ) are obtained by independent magnetic measurements. These values of surface roughness are in good agreement with AFM observation. One should also note that the critical current does not depend on the thickness, so cleaving the sample does not decrease the critical current. One can also see in figure 2 that a very important effect is not explained by the model, i.e. the peak effect close to B_{c2} .

In the peak effect, the critical current I_c is larger than the calculated one I_{c0} . One can assume that the current also flows in the surface and calculate the associated critical angle α . It gives 8° whatever be the surface state. An important observation can be made on the data which supports this assumption. The magnetic field ω close to the surface is increased by the curvature of the flux lines ($\omega_{\text{surface}} = \omega_{\text{applied}} / \cos(\theta)$). For this reason, the critical current vanishes first for $\alpha = 1^\circ$ and then for $\alpha = 8^\circ$ as one can see in eq. (4). This is clearly observed in the data here, and also in previous publications.

In fact, the nature of this critical current is different from the previous one since the shape of the I - V curve is very different: even when the critical current has a high value, the curve at high currents extrapolates to the small critical current value. In addition, spatially resolved measurements exhibit two different phases (the high critical current close to the edge, and the small one in the sample center). One can see the S-shape of the I - V curve as a continuous progression of percentage of the low

critical current phase as one increases the current in the sample. $V = xR(I - I_{c0})$, where x is the phase fraction. In the following, we have summarized the process of dissipation above I_c : due to edge effects, the magnetic field in the sample center is slightly larger than that close to the edges, so dissipation will appear there first. As soon as dissipation appears, quite a large dissipation appears close to the surface, leading to a damped oscillation of the vortex head which will reduce the stability of the vortex pinning at large angle. This effect will spread out the current into the sample bulk. The presence of induced vortex loops perpendicular to the main magnetic field will act as a zip and depins the large critical current area. In fact, the situation is very similar to solid friction in which the critical force necessary to start the motion is always larger than the frictional force in movement. The analogy here can be seen as described in the following: immobile, the vortex lattice may subtend large surface angles. When it starts to move, dissipation in the surface region creates additional ‘heating’ (or breathing of the vortices) which reduces the stability of the large curvatures. Once the movement starts, it is impossible to recover the stability.

However, two questions remain: The ‘magic’ angle 8° is probably related to the surface properties of NbSe₂ crystals. It is difficult to be more precise in the framework of this model but one can imagine that the step edges are surface defects which are not modified by sandblasting and can be at the origin of the magic angle 8° . When the magnetic field decreases, the critical current increases and the surface dissipation $I_c V$ increases. The density of the perpendicular vortex loops also increases. It is clear that a limit should appear for stability of the high critical angle phase. This interpretation of the data is supported by the fact that it is possible for example, by field-cooling the sample, to stabilize metastable high critical current phases even below $B/B_{c2} = 0.75$. The low magnetic field limit of the high critical current phase appears to be crucially related to the experimental conditions and due to metastable states.

The present data suggest that the critical current in the peak effect is due to surface pinning. The metastable equilibrium of the vortices can be reached by special preparation of the vortex state. This unstable state can be destabilized in the sample center by a very large current and propagated to the whole sample by the presence of perpendicular vortex loops. Sandblasting does not modify this high critical current state and so the irregularities responsible for this pinning should be at a different scale such as the step edges of the cleaved surface.

4. Transport data in Bi-2212 crystals

Let us now study the case of a very anisotropic sample such a Bi-2212. The samples used in this study are slightly Pb-doped single crystals of the Bi-2212 family ($\text{Bi}_{1.8}\text{Pb}_{0.2}\text{Sr}_2\text{CaCu}_2\text{O}_{8-\delta}$). They were grown by the self-flux technique. The cleaved single crystal was laser-tailored in the form of a microbridge with a controlled pattern of ($W = 200 \text{ mm} * L = 400 \text{ mm}$). The crystal was annealed under a controlled pure oxygen gas flow and was kept in the slightly overdoped regime ($T_c = 79.5 \text{ K}$). Low resistance electrical contacts were made by bonding gold wires with silver epoxy. The DC transport measurements were performed using a standard four probe [11].

We have performed $V(I)$ curves at low temperature ($T = 5$ K) in order to minimize thermal fluctuations. Let us first discuss the results for high magnetic field values. The $V(I)$ curves present the usual linear form as soon as I is slightly higher than the critical current. There is no evidence of an ohmic regime at low applied current and the depinning is rather stiff. Furthermore, while comparing the effect of field-cooling (FC) and zero-field-cooling (ZFC), or FC under different cooling rates, we measure the same dissipation in the time-scale of our experiment. In particular, no aging effect is observed on the critical current, which is not in agreement with a glassy nature of the vortex lattice governing transport properties.

When the magnetic field is decreased, we observe a different behavior in a restricted region of the phase diagram. When the vortex lattice is prepared after FC, the $V(I)$ curves exhibit an S-shape with a high threshold current, but only for the first ramp of current. After this, the same lower I_c is obtained (figure 3). This has been previously observed in the pulse current experiments, and this ‘high threshold current state’ has been evidenced as a metastable state with a very long relaxation time [11]. Our measurement using a DC current evidences that the observation of this state is not due to the kind of stimulation used. Concerning the metastable $V(I)$ curves, the peak effect in the critical current, the coexistence of two VL states and the same kind of behavior is currently observed in NbSe₂. The strong difference is that the peak effect and the associated metastable effects appear close to B_{c2} in NbSe₂ but here it is restricted to a very low field value. As the temperature applied in both experiments is similar, it is likely that the explanation of this field value difference has to be found in the large difference in the electronic anisotropy. For field lower than about 0.05 T, we do not observe any hysteresis within the $V(I)$ curves. The case of very anisotropic samples is especially interesting, because it is predicted that for not too low magnetic field values and for realistic accessible surface roughness, the surface critical current becomes independent of the surface quality and depends solely on parameters of the condensate. For clarity, we restrict the comparison to the high field values in order to use the Abrikosov limiting expressions. One expects [8]

$$I_c/W = B_{c2}/(2\mu_0\beta\kappa)(1 - B/B_{c2}^{2/3})^{3/2}. \quad (6)$$

One can see in figure 4 that the agreement with the experimental data is really very good. The exact understanding of the peak effect is slightly more delicate. One can estimate what should be the B_{c2}^* value using eq. (5). If γ is very large (typically 60 here), one can develop eq. (5) neglecting 1 compared to $\gamma^2 \tan^2 \alpha$ into

$$B_{c2}^* = B_{c2}/(\gamma \tan \alpha) \quad (7)$$

which is typically 2000 G here, in very good agreement with the experimental value of figure 4. The difficulty to give an exact value of J_c is that $\varepsilon(B)$ is not known in Bi-2212 where κ is of the order of 100, too large for any simple approximation of ε if B/B_{c2} is typically 10^{-6} . This is something which should be calculated exactly.

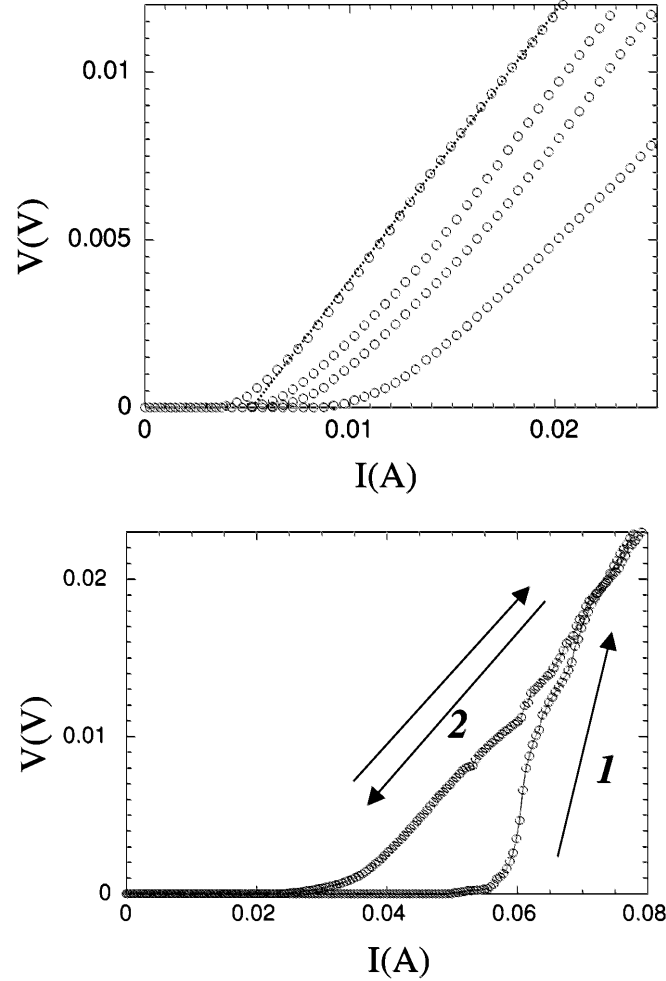


Figure 3. Typical I - V curves of a Bi-2212 microbridge under different magnetic fields. Top curve: above the peak effect from 1 T to 4 T; bottom curve: at 300 G.

5. Small angle neutron scattering in Pb-In and NbSe₂ crystals

Small angle neutron scattering thus appears as a unique technique, since the order of the FLL can be tested. It is also possible to measure *in-situ* $V(I)$ curves together with the FLL diffraction and hence to investigate the relationship between the current distribution and the FLL structure in the sample. One constraint is with respect to the Maxwell equation which relates the bending of the field lines and the current density [12,13]. The aim of these types of experiments is to use SANS in order to compare different FLL states with respect to their dynamical properties. We will focus on the case of FLL states close to the peak effect. The small angle

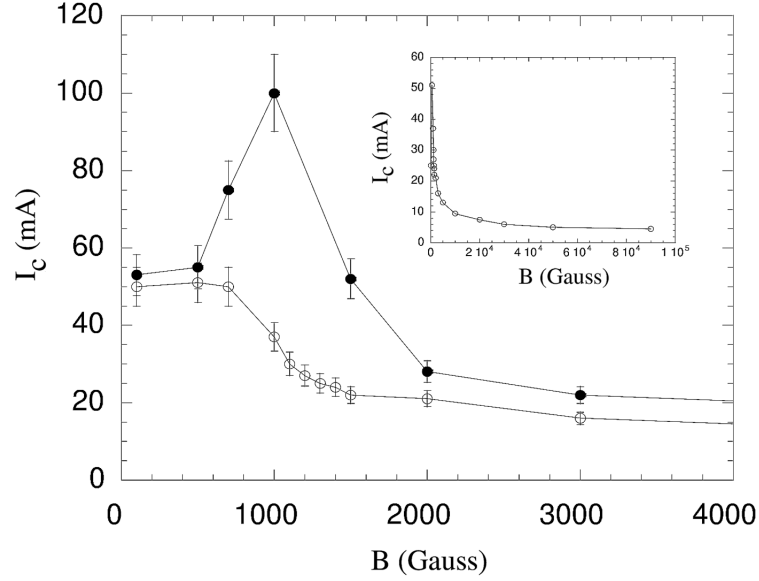


Figure 4. The critical current as a function of the magnetic field at 4.2 K for the Bi-2212 microbridge. The two different curves correspond respectively to the small and the high critical currents observed in the bottom part of figure 3. In the inset, the data are presented for higher magnetic field, showing the coherence with the proposed model.

neutron scattering (SANS) experiments were performed in the Laboratoire Leon Brillouin (Saclay, France). Large single crystals of Fe-doped H-NbSe₂ (200 ppm of Fe, size $8 \times 6 \times 0.5 \text{ mm}^3$, $T_c = 5.5 \text{ K}$) and of polycrystalline Pb-In (10.5% of In by weight, size $30 \times 5.5 \times 0.5 \text{ mm}^3$, $T_c = 7 \text{ K}$) were used. The magnetic field was applied parallel to the c -axis of the crystal and to the incident neutron beam. The scattered neutrons of wavelength 10 \AA were detected by a 2D detector located at a distance of 6.875 m. Superconducting leads were attached using indium solder pressed between copper slabs: they gave us the possibility of passing a high enough transport current for this experiment. At the working temperature of 2 K (in condensed superfluid He), we can pass about 8 A current without overheating. In the following, ω will refer to a rotation around the vertical axis, and Φ to a rotation around the horizontal axis.

We have performed the same kind of experiments in crystals of NbSe₂ in order to compare the different states of FLL responsible for the anomalous transport properties. We first tried to observe the simplest case of the ZFC FLL. This is supposed to reflect the ordered and equilibrium state, because it corresponds to the state with a low critical current. The diffraction patterns exhibit an ordered hexagonal lattice. We also performed ω rocking curves. Small widths are obtained by analyzing the peaks with Lorentzian fits (figure 5). We obtained, for the ZFC FLL (without external current applied) $\Delta\omega = 0.232^\circ$. This is close to and a little higher than the experimental resolution given by the angular divergence of the beam. If we increase the transport current, but stay below the critical current

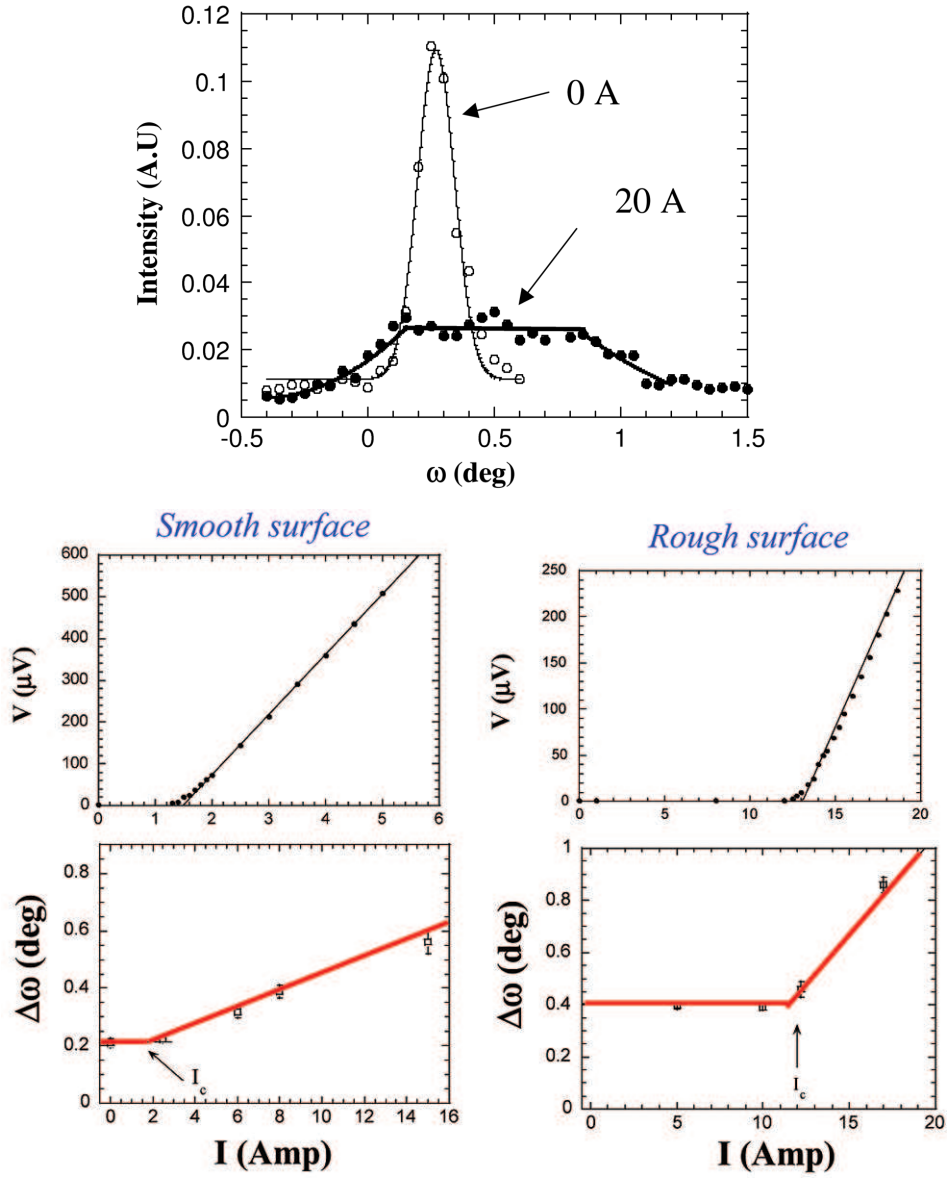


Figure 5. The rocking curves at different currents at 4.2 K in a Pb-In sample. In the bottom part, the I - V curves are presented, compared to the broadening of the rocking curve, for two different surface roughness of the same sample.

value, absolutely no change is observed. When the applied current is higher than the critical current of 2.5 A, a slight increase of the rocking curve width $\Delta\omega$ is observed. It is clear that the points are separated by values just slightly higher

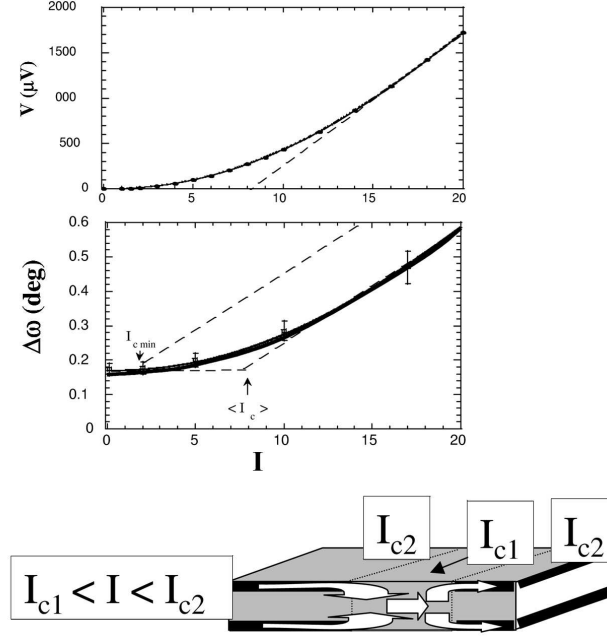


Figure 6. Comparison of the I - V curve and the broadening of the rocking curve in a sample in which the critical current is not homogeneous. The solid line in the bottom part of the figure is the calculation obtained assuming the critical current inhomogeneity measured in the upper part of the figure showing the I - V curve. A schematic drawing of the current flow is also shown.

than the experimental resolution (given mainly by the mechanical precision of the angle during rotation). One can nevertheless estimate that no bulk current is present for $I < I_c$ and that a bulk current that is of value $(I - I_c)$ is observed for $I > I_c$. This result, added to the $V(I)$ curve that exhibits the usual form $R_{ff}(I - I_c)$ and not as $R_{ff}I$ indicates that (under critical) superficial current due to surface pinning and critical current dissipative in the bulk offers a natural explanation for this behavior. Concerning SANS coupled with transport experiments in NbSe_2 , it is quite natural to cite Yaron *et al* experiments [14], whose purpose was to measure the longitudinal correlation length characteristic of the FLL order. Yaron *et al* observed a narrowing of the rocking curve that they attributed to an improvement of the FLL order. On the contrary, we observe here, what was previously observed in other superconductors, that the rocking curve broadens as the part of the current over the critical current penetrates the bulk. As this is a simple consequence of the Maxwell equations, it is not clear to us why such an effect was not observed by Yaron *et al*. A possible interpretation is that the reported rocking curves are recorded in a direction perpendicular to those reported here. In such a case and as observed in Nb-Ta samples, a very small narrowing can be observed but it can reasonably be attributed to a smoothing of FLL Bragg plane spacing (due to the homogeneous bending in the perpendicular direction) rather than to a change in the correlation length.

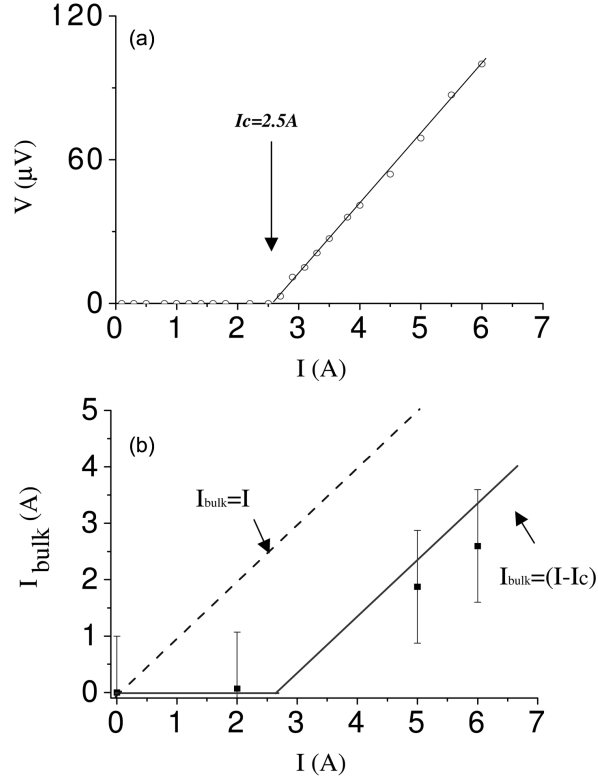


Figure 7. (a) Comparison of the I - V curve and the broadening of the rocking curve in a NbSe_2 sample at 2.5 K and 3000 G. The solid line in the bottom part (b) is obtained by calculation assuming that the critical current is flowing at the sample surface. The dotted line corresponds to the assumption of the current flowing homogeneously in the sample bulk.

The FLL in NbSe_2 , created by ZFC, appears quite similar to the FLL in the conventional Type II superconductor with moderate critical current. In fact, more differences are expected after FC, because in this case the $V(I)$ curve looks very peculiar. The samples we used for the SANS experiments are larger than those usually employed for transport properties. Thus it is important to stress that we have measured $V(I)$ curves (figure 6) very similar to what was already studied by others in detail. They exhibit a hysteretic behavior, with an S shape for the first run after FC and a linear and reversible behavior for all ramps of current thereafter. It is worth noting that such $V(I)$ curves classically observed in NbSe_2 are a particular case of vortex dynamics. For a large number of Type II superconductors, these effects are not observed.

If the ZFC FLL observed in Fe-doped NbSe_2 was close to what we call a conventional FLL, obtaining information on the FLL structure after FC was a real difficulty. For the same Bragg conditions as for the ZFC state, we do not see any scattered intensity. The first guess was that the FLL was so strongly disordered

that the Bragg peaks were considerably broadened and thus almost invisible. But this reasoning is not right, as evidenced in figure 7 where the corresponding rocking curve is shown. Comparing with the results of figure 8, it is easy to see that the Bragg conditions for FLL have changed and that the rocking curve exhibits a double peak, which is quite unusual. The sum of the integrated intensity contained in these two peaks compared well, within error bars, to the integrated intensity of the Bragg peak of the ZFC FLL, and the widths of the peaks are comparable too. Consequently, we cannot attribute these strange Bragg peaks to a FLL disorder in its proper sense. It would better correspond to two very similar families of FLL that are ordered, but slightly tilted from the magnetic field direction by few 0.1 degrees. We can eliminate a rotation coming from Doppler shift because the FLL frame is not moving. Another possibility is that we are now observing two FLL possessing two different Bragg plane spacings because of different magnetic densities. But this assumption would imply a field gradient of more than half the magnetic field present in the sample. This hardly looks compatible with the strong interaction between the flux lines that limit the compressibility of the vortex array. Finally, it is quite reasonable to think that the two peaks we observe are the signatures of the ‘two phases’ observed by Marchevski *et al* using scanning hall AC probe [5]. Their experiments show that two states possessing different critical currents are coexisting in the region of the peak effect. Our SANS experiment offers complementary information. The fact that the two peaks are very similar is not in favor of the two states characterized by different bulk underlying disorder. The shift between these two peaks indicates that the two FLL are slightly tilted by static and small in-plane field components. It looks clear that these field components are due to a peculiar and non-symmetric distribution of screening current. From the point of view of previous authors, we adopt that the border of the sample is a region of high superficial current density. Both Bragg peaks cover roughly the same surface and we can speculate that the width of the sample is divided into two parts of roughly the same dimension, i.e. 3 mm each. We know that the low critical current is 2.5 A and that it corresponds to a superficial value of 2 A/cm. At the same time, we have measured a ratio of about 7 when the peak effect is at its maximum. With the reasonable assumption that it corresponds to a state where the high critical current state invades most of the sample, we can deduce that the high critical value is about 14 A/cm. Using the Ampere theorem and making a superposition with the top and bottom surfaces, we find that the two sheets transporting currents generate bulk components of magnetic field which are 2.5 and 18 G respectively. This is not too far from the measured values (9 and 24 G), considering the highly schematic picture used here. In this picture, the FC state is made up of a large number of loops of current, that are as many in number as there exist non-dissipative paths for a transport current. Increasing the transport current induces a preferential direction and one loop should be turned off. This implies that one of the two tilted FLL disappears. If the transport current is increased further up to the high critical current value, the second loop disappears. All the flux lines are now along the main magnetic field and the Bragg angle returns close to its normal position. Finally, the surfaces cannot transport more non-dissipative current, so the excess penetrates the bulk and the flow becomes resistive. The $V(I)$ curve returns to a classical behavior in the linear form.

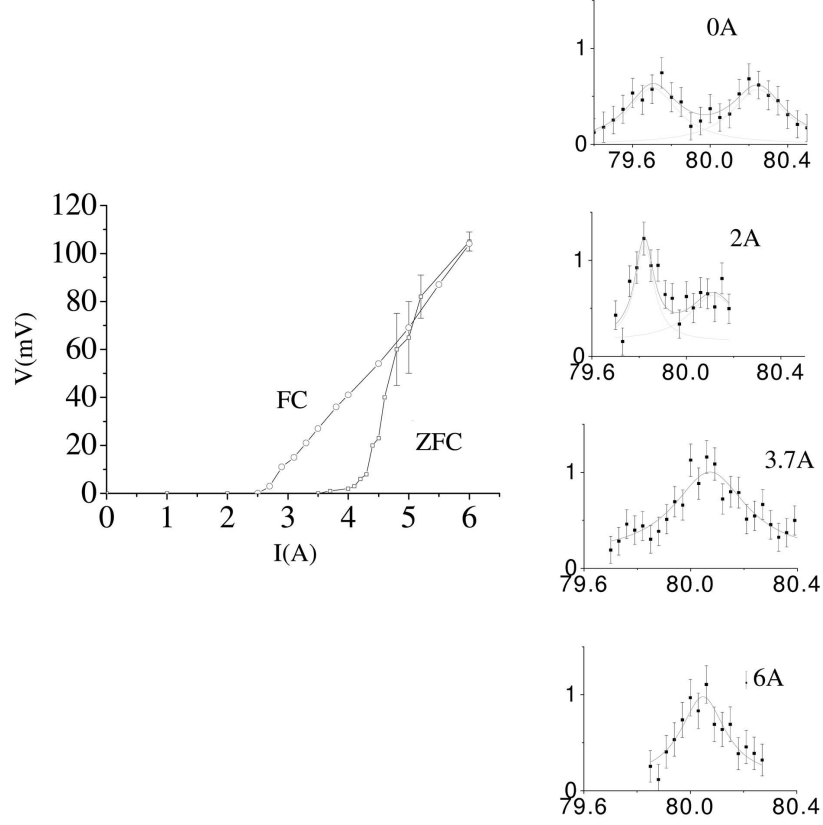


Figure 8. The rocking curves obtained in the field-cooled (FC) procedure with different currents are shown. On the left side of the figure, the FC and ZFC I - V curves are shown.

6. Conclusion

Using a model of pinning by surface irregularities in anisotropic superconductors, we have developed a calculation of the critical current which allows estimating quantitatively the critical current in both the high critical current phase and the low critical current phase. The only adjustable parameter of this model is the surface roughness which can be measured by AFM and modified by FIBE processes. The agreement between the measurements and the model is really very impressive. In this framework, the peak effect is due to coexistence of two different vortex phases with different critical currents. A detailed discussion of the vortex geometry is necessary to understand the details of the peak effect. Neutron diffraction data in NbSe₂ crystals in the presence of transport current support this point of view.

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