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Observation of periodic vortex pinning induced by Bitter decoration

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In this paper we report on a type of vortex pinning, Bitter pinning. In this technique, a vortex lattice in a type II superconductor, $NbSe_2$, is decorated with magnetic particles which form clumps on the surface of the sample where the vortices exit. Because of the van der Waals forces, these magnetic dots stay well fixed after the sample is returned to room temperature. We present data showing that these patterns can act as pinning sites for vortices in subsequent experiments. This method allows one, in a simple and effective way, to produce periodic, quasiperiodic, or disordered pinning structures for static and dynamic vortex experiments. [S0163-1829(99)51342-8]

The discovery of the high-T_c superconductors created a resurgence of interest in vortices in type II superconductors. In those materials, the interplay between pinning, elastic, and thermal energies produces a complex and interesting phase diagram[1] with lattice, glass, and liquid vortex phases all evident in the magnetic-field/temperature plane. As the static phase diagram has become more well understood, attention has shifted to the study of dynamic states of the vortex lattice. This is part of a more general trend in condensed-matter physics in which the statics and dynamics of flowing structures are being extensively studied.[2]

In addition to the intrinsic interest in vortex lattices because of their relevance to technologically important issues such as critical currents, flux-line lattices (FLL) are ideal model systems to address those general basic questions.[2,3] The ability to change the lattice constant over a wide range, the availability of a large number of imaging, transport, and thermodynamic probes, and the wide range of systems and regimes available to experiments, have contributed to making this one of the most important and well-studied areas of dynamic flow and an area in which serious confrontation between theory and experiment continues to rapidly advance our understanding.

Engineered pinning centers in type II superconductors have a long and venerable history. Previous work on periodic structures includes approaches such as focused Ga ion-beam irradiation of Nb films,[4] magnetic dots placed under Nb films,[5] and lithographically defined patterns in the superconducting film itself,[6,7] to name but a few.[8] However, most of these approaches are quite limited in their range of applicability because they require sophisticated processing which is not always compatible with the systems of interest. In this paper we report on a simple technique we call Bitter pinning which we believe will significantly open up the phase space of samples and systems in which experiments can be performed with engineered pinning centers. The technique is based on the Bitter decoration technique which has been shown[9] to work over a wide range of materials and experimental regimes. In the technique, one induces a vortex lattice to form in the sample, decorates it with magnetic particles which stay quite firmly attached to the surface, and then uses this array of magnetic particles as pinning centers for subsequent experiments. In this paper we will present experimental evidence that this simple idea can actually be made to work in practice.

Our samples are high quality $NbSe_2$ single crystals with typical dimensions of $1\times1\times0.05\,\mathrm{mm}^3$ with the *c*-axis parallel to the thin dimension. Typical critical current densities are $\sim$1000 amps/cm^2 and are field independent[10] in the range of fields used in these experiments. This indicates that the samples are in the single vortex pinning regime. For our samples, $T_c=7.3\,\mathrm{K}$, the coherence length $\xi\sim77\,\text{Å}$, and the penetration depth $\lambda\sim690\,\text{Å}$.

The magnetic decoration technique has been extensively described elsewhere.[11] The experiments described here were performed at 4.2 K and after decoration the samples were warmed up to room temperature and the images taken with scanning electron microscopy (SEM). The resolution of our image processing is typically 620 pixels/μm^2. The iron dots which the technique deposits on the vortex locations are approximately a cone with a base of radius of $\sim$0.2 μm and a height four times lower. For the experiments in which the first decoration was used as a pinning structure for a subsequent decoration, part of the sample was masked in the first experiment to allow a detailed, quantitative comparison to be made.

Shown in Fig. 1(a) is an example of data from a typical field cooled (FC) experiment with an applied field $H=36.1\,\mathrm{Oe}$. The image is a Delaunay triangulation where nearest-neighbor vortices are connected with solid lines and the nearest neighborhoods of non-six-fold-coordinated vortices are shaded to show the topological defects. The inset shows the Fourier transform (FT) averaged from 15 different regions of the sample. The ring of intensity makes it clear that both the positional and orientational order in this FLL are short ranged.

Shown in Fig. 1(b) is a typical image of a second FC decoration at $H=36.1\,\mathrm{Oe}$, after a first FC decoration at the same field and a warming to above T_c between runs. In some regions of the sample the two structures coincide well (re-

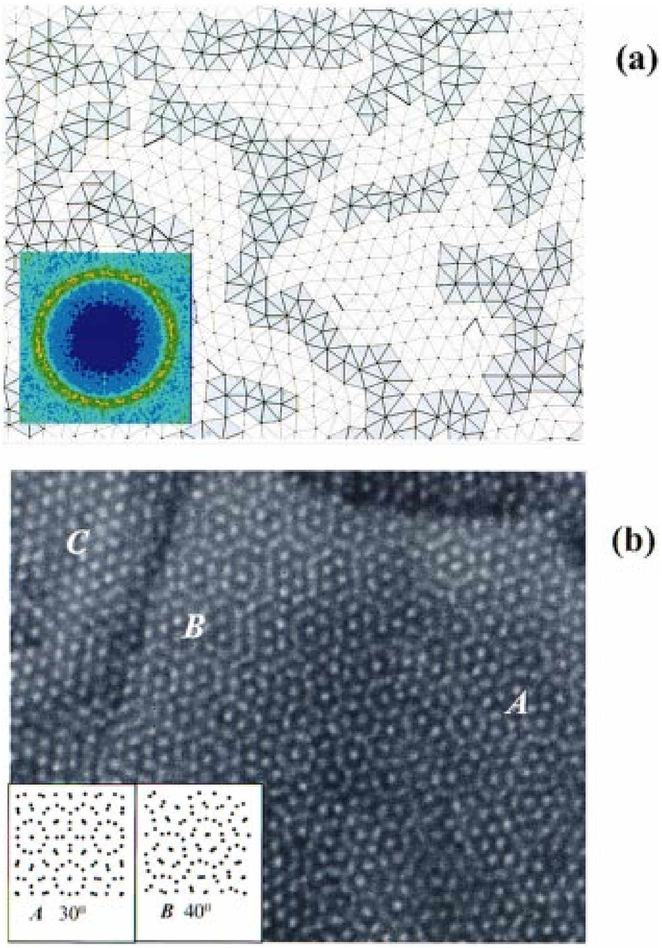

FIG. 1. (Color) (a) The Delaunay triangulation of a vortex structure for a first FC decoration at $H=36.1$ Oe is shown. The polycrystalline nature of the structure is made evident by the Fourier transform shown in the inset. (b) The figure shows the vortex pattern after a second FC decoration at $H=36.1$ Oe on top of the magnetic structure of (a). In the inset can be observed the moiré patterns obtained by rotation of two perfect lattices of 30° and 40°, corresponding to the regions *A* and *B* in the figure. In the region *C* both structures coincide.

gion *C*) and in others (regions *A* and *B*) what is seen is a moiré pattern indicative of only a partial alignment between the two lattices from the two experiments. In the moiré pattern regions, some dots are brighter than others indicative of spots where there were vortices in both decorations. In the inset we show examples of moiré patterns corresponding to rotations of 30° and 40° of two perfect lattices which mimic the data in regions *A* and *B*, respectively. In addition to the analysis of many similar images, we have also measured the critical current in decorated samples and find little effect due to the surface pinning centers. We conclude that the bulk pinning due to defects dominates the surface pinning due to the magnetic dots. Given that the sample is 400 times thicker than the dots, this is plausible. To see an unambiguous effect of surface pinning required a different approach which we describe below.

Previous experiments have demonstrated three different techniques to suppress the effectiveness of bulk pinning in this system. In the first, a current in excess of the critical current was applied to the sample and then rapidly removed.

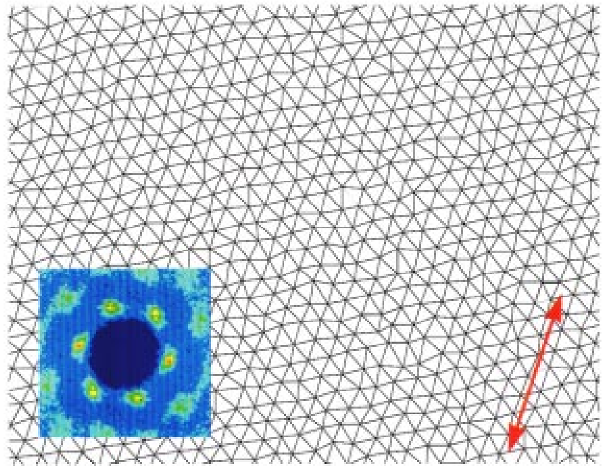

FIG. 2. (Color) The Delaunay triangulation of a vortex structure for a FCR decoration at $H=36.1$ Oe and a rotation at 60° is shown. The arrow indicates the direction of the field rotation axis. The inset shows the Fourier transform, where the six peaks are indicating the crystalline nature of the structure.

The lattices quickly quenched from a flowing state were found[10,12,13] to be quite well ordered due to a dynamical memory effect. In the second, somewhat related technique, a sample was field cooled to below T_c and then the field was reduced to zero. After a short while vortices in a quasistatic, critical state region near the edge of the sample were also found[3] to be quite well ordered. The third technique to be used here is a double rotation of the magnetic field. In these experiments[14] a sample is FC, the field is rapidly rotated to a given angle α and then back to the original (perpendicular) orientation and then the decoration is made. In these FCR experiments, the rotation is accomplished by rapidly switching on and off a perpendicular component and so both the magnitude and direction of the field changes. The advantage of this technique is that without electrical leads, the vortex lattice can be made much more well ordered than what would be given by bulk pinning alone.

In Fig. 2 we show the order induced in a FCR experiment of $\alpha=60°$ for $H=36.1$ Oe. In the inset is shown the FT. The six sharp peaks in the FT are clear evidence that the FLL after this history is much more well ordered, and prove the effectiveness of the double rotation technique for minimizing the influence of bulk pinning. In the image in Fig. 2, the arrow indicates the orientation of the FLL. As previously seen,[14] this is always found to be parallel to the axis of field rotation.

The decoration pattern shown in Fig. 2 was used as a pinning pattern for a second FCR experiment of $\alpha=60°$ shown in Fig. 3. As an experimental check, during the first experiment, part of the sample was masked and this was removed for the second decoration as a control. The data clearly show that the two structures coincide, in contrast to what was shown in Fig. 1(b). This indicates that after a quick, double rotation the magnetic dots on the surface can clearly pin the vortex structure. In both experiments, care was taken to keep the field rotation direction the same with respect to the crystal.

Clear evidence of the effectiveness of the dynamic memory effect in inducing magnetic pinning in the dynami-

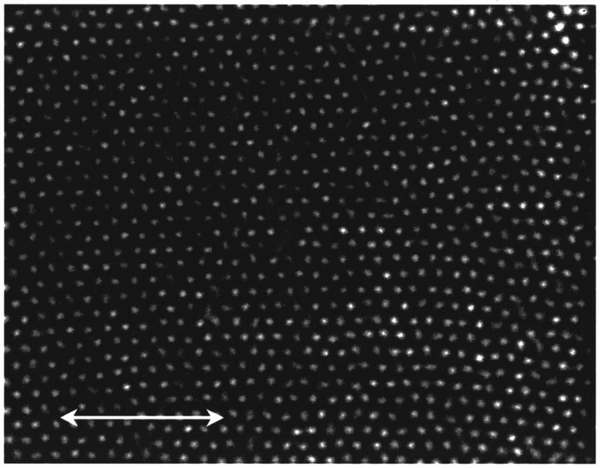

FIG. 3. Vortex pattern after a second FCR decoration at $H = 36.1$ Oe and 60° on top of the magnetic structure of Fig. 3. The arrow indicates the direction of the field rotation axis.

cally ordered vortex structure is shown in Fig. 4. In Fig. 4(a) it is shown the FT of the second 36.1 Oe FC decoration (with no field rotation) whose real-space structure is shown in Fig. 1(b). The FT is averaged from 15 different pictures of the vortex images. The ring makes evident the apparent disordered vortex structure due to the random rotation of the second vortex lattice with respect to the magnetic pinning structure induced in the first decoration. The radius of the ring corresponds to a field greater than the 36.1 Oe applied field used in the first and second decorations. The fictitious larger magnetic field is due to the extra magnetic dots of the second decoration induced by vortices that were not pinned by the magnetic structure.

In Fig. 4(b) we show the FT taken from the image of a second FCR decoration for $\alpha = 60°$ on top of the magnetic structure of Fig. 2, whose real-space structure is shown in Fig. 3. The six peaks clearly demonstrate that the second decoration reproduces the ordered structure created in the first rotated decoration. The FT in Fig. 4(c) is taken from the image of the vortex lattice in the second decoration but on the region of the sample that was masked during the first decoration. Both pictures show the same degree of order of the vortex structure.

The results provide conclusive evidence that the extended magnetic dot lattices are capable of pinning three-dimensional kinetically ordered vortex structures. Thus, the data are consistent with Ref. 15 that shows that long-range lattice order can be induced by rapid vortex displacements on top of disordered pinning potentials.

Extensions of this work will be to use the Bitter pins as nucleation sites for a chemical treatment of the surface to increase the strength of the pinning centers. Other possibilities include covering the surface of the sample with a strong pinning material allowing the Bitter pins to become antipinning sites for vortices, and the study of lattice distortions and defects induced by the lack of commensurability between lattices. We believe that with proper refinement, these structures will allow us to do a series of experiments on static and dynamic vortex lattices which will be difficult or impossible to do in any other way.

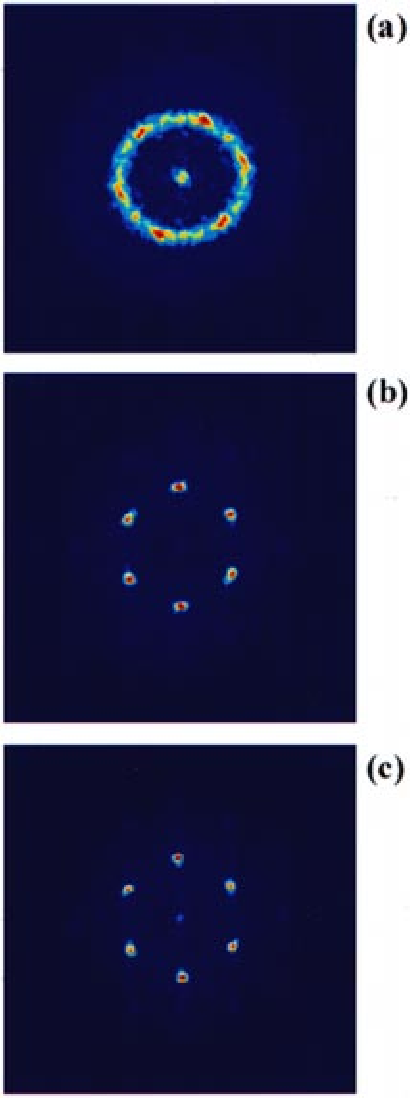

FIG. 4. (Color) (a) Fourier transform of the second 36.1 Oe FC decoration (with no field rotation) as that shown in Fig. 1(b). (b) The Fourier transform taken from the image of a second FCR decoration for 60° on top of the magnetic structure of Fig. 2 (see Fig. 3). (c) The Fourier transform of the vortex lattice in the second decoration as that in (b) but on the region of the sample that was masked during the first decoration (see text).

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