Imaging the structure of the domain wall between symmetries interconnected by a discontinuous transition

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Abstract

We have been able to observe with single particle resolution the interface between two structural symmetries that cannot be interconnected by a continuous transition. By means of an engineered 2D potential that pins the extremity of vortex strings an square symmetry was imposed at the surface of a 3D vortex solid. Using the Bitter decoration technique and on account of the continuous vortex symmetry we visualize how the induced structure transforms along the vortex direction before changing into the expected hexagonal structure at a finite distance from the surface.

First order phase transitions are often associated with a discontinuous change of topological symmetry. The analysis and detection at microscopic scale of mechanisms that allow the nucleation and propagation of new symmetries is a formidable task of difficult experimental resolution. An ensemble of superconducting vortices, having a continuous symmetry along the average vortex orientation and discrete structural symmetry in planes perpendicular to it, is a unique toy model to investigate first and second order phase transitions.

The vortex liquid in superconducting $Bi_2Sr_2CaCu_2O_8$ crystals transforms through a first order thermodynamic transition into a solid (Bragg Glass) with hexagonal quasi-long range order. As a result of the almost ideal behavior of the vortex structure large vortex single crystals are usually obtained after the liquid-solid transition. The topology of the solid state with individual vortex resolution can be detected by magnetic decoration, observing the clumps generated by small Fe particles deposited at the vortex positions.

The scope of this work is to develop a strategy to investigate how a change of structural symmetry induced at the surface of a solid of elastic strings propagates along its length. This is achieved imposing the vortex liquid to solidify in a three dimensional square lattice at one surface of a $Bi_2Sr_2CaCu_2O_8$ crystal while the natural hexagonal vortex structure is observed at the opposite surface. In this way, we were able to study the vortex structure within the interface between two single crystals of vortices with symmetries that cannot be interconnected by a continuous phase transition.

By means of electron beam lithography Fe dots of 300 nm in diameter and 60 nm height were deposited on the top surface of $Bi_2Sr_2CaCu_2O_8$ single crystals of approximate dimensions $0.5 \times 0.5 \times 0.03 \text{mm}^3$. The dots are of similar dimensions as the clumps generated in magnetic decorations that act as effective surface pinning centers (Bitter pinning).
this way periodic Fe patterns of square symmetry with 50x50 dots were created. Large surface regions of the sample were left free of Fe dots to detect the hexagonal pattern and to compare the induction field $B$ inside and out of the Fe patterned region. The vortex structure was nucleated by cooling the sample in the presence of a magnetic field. The Fe pattern was chosen to make the area of its unit cell, $a_{sq}^2$, equal to that of the corresponding hexagonal vortex lattice induced by the applied field (one flux quantum per unit cell). The vortex lattice was visualized by magnetic decoration. The Fe structure from the magnetic decoration, as well as that of the periodic pattern, was observed at room temperature with a scanning electron microscope. When the Bitter and the periodic pattern coincide, the Fe clumps from the decoration were recognized by their irregular appearance as compared to the circular shape of the dots of the periodic pattern. The number of vortices per unit area in the patterned area was found to be the same as that in the non-patterned region (the same $B$).

Figure 1 shows the vortex decoration on the Fe-patterned surface of a sample with $a_{sq} = 0.835\, \mu m$, hereafter called top surface. The vortex structure corresponds to $B = 30.2\, \text{Gauss}$ and the decoration was made at 4.1 K. The dark region marks the area where the square 2D pinning was generated. The square topology of the vortex structure induced by the periodic pattern is evident. The natural hexagonal symmetry of the solid is seen to be recovered outside the Fe patterned region in no more than two lattice parameters. The one to one site correspondence of Fe clumps from the decoration with the square pattern reveals that the 2D periodic pinning imposes the vortex liquid to solidify in a single crystal with square symmetry at the top surface.

To verify whether the sample is thick enough to allow the recovery of the hexagonal vortex symmetry, a magnetic decoration was made on the opposite surface of the Fe patterned one, hereafter called bottom surface. Figure 2 shows the decoration at the bottom surface of a 4.5 $\mu m$ thick sample, beneath the square Fe patterned region. The Delaunay triangulation of the vortex lattice is depicted. The picture shows that the hexagonal vortex lattice has one of its principal directions parallel to one of the square pinning potential axis. This shows that the presence of the square potential at the top surface breaks the rotational degeneracy of the hexagonal lattice. The hexagons at the bottom of the sample make evident the coexistence of square and hexagonal symmetries along the vortex strings.

The results in Fig. 1 and 2 suggest the presence of a domain wall between structures with equivalent energy: The square structure at one side of the wall, where the surface pinning potential compensates the increase of interaction energy corresponding to the square lattice, and the natural hexagonal symmetry of the vortex crystal at the other side.

To explore the change of the vortex topology along the sample thickness, decorations of freshly cleaved bottom surfaces of the sample were performed. The cleaving method has the advantage of preparing excellent surfaces for decoration but does not allow us to choose a precise thickness. Repeating the cleaving process we were able to change the thickness of the original sample of 12.5 $\mu m$. Decoration of the bottom surface of samples with thickness, $d$, of 12.5, 5.75, 4.5, 3.5, 2.5 and 0.5 $\mu m$ verified that the hexagonal symmetry was recovered at a distance between 3.5 $\mu m$ and 4.5 $\mu m$ from the top surface.

The thinnest sample, with $d = 0.5\, \mu m$, shows that the vortex structure has the square symmetry in the whole region beneath the Fe pattern, as depicted in Fig. 3. This result indicates that the engineered 2D pinning potential can be used to modify the melting or...
wetting conditions for the vortex system in a finite thickness close to the surface of the sample.

The magnetic decoration of vortices at the bottom surface of samples with different thicknesses allowed to locate the domain wall between the square and hexagonal symmetries and to investigate its internal microscopic structure. Since the interface is detected close to the top surface, starting somewhere between 0.5 \(\mu m\) and 2.5 \(\mu m\) from the top surface and extends to a distance between 3.5 \(\mu m\) and 4.5 \(\mu m\) from it, the domain wall has a thickness of the order of the average vortex near neighbors distance (a \(\approx 1 \mu m\)).

Decoration of vortex structures at the bottom of 2.5 \(\mu m\) and 3.5 \(\mu m\) thick samples have shown a distribution of three distorted hexagonal structures coexisting with regions containing a small number of vortices (15 in average) with fourfold symmetry which lattice parameter is equal to that of the square Fe pattern on the top surface. As an example, Fig. 4 shows the decoration of one pattern obtained at the bottom of 3.5 \(\mu m\) sample. Statistics made on hundreds of vortex unit cells below the square Fe pattern indicate that from the total number of vortices in the domain wall 83% belong to hexagonal deformed structures, while the rest are distributed in regions of square symmetry of few lattice parameters.

The structure at the bottom of 2.5 \(\mu m\) thick sample shows the same relative distribution (within 3%) of distorted hexagonal and square symmetry regions. It is clear that at these two different thicknesses (one close to the beginning and the other to the end of the domain wall) the interface embraces the coexistence of deformed hexagons and square vortex structures. Similar configurations were found by recent numerical simulations of vortex interfaces between square and hexagonal symmetries. A fraction of 85% of the deformed hexagons are distributed between two possible degenerate states rotated in 90 degrees. Each state is characterized by one lattice parameter equal in magnitude and parallel to one of the square Fe pattern, see Fig. 4. The results from four decorations made at the bottom of 3.5 \(\mu m\) and 2.5 \(\mu m\) thick samples indicate that, despite the proportion of square and deformed hexagons is maintained, the relative abundance between the two hexagonal degenerate states differs widely from one experiment to another. The comparison among vortex structures at 3.5 and 2.5 \(\mu m\) underneath the same square Fe pattern shows that the specific location of square and distorted hexagonal vortex regions varies from one experiment to the other, see Fig. 4. This strongly supports that the proportion of deformed hexagons and square symmetries is an intrinsic characteristic of the interface between the square and hexagonal single crystals nucleated along the vortex string.

Another characteristic of the interface is that the area of both, the hexagonal distorted as well as the square unit cell at the bottom of the 3.5 and 2.5 \(\mu m\) samples coincides within 2% with that of the square and hexagonal crystals at each side of the domain wall. This strongly supports that the transformation from the square to the hexagonal symmetry is made under the condition of constant unit cell area (uniform \(B\)) across the interface. The uniaxial compression of the hexagons results from a distortion induced to match one of the hexagonal lattice vector with one of the square pattern on the top surface. The transformation from the described mixture of phases within the interface region to the uniform hexagonal symmetry takes place within one micrometer (one lattice parameter). We have not observed this transformation because the cleaving technique does not permit to choose a thickness of the sample with the necessary precision.

In conclusion, the combination of electron-beam lithography and magnetic decoration
techniques have allowed to demonstrate that the vortex liquid can be solidified in two coexisting crystals with different symmetries along the vortex length. The square symmetry induced by the engineered 2D Fe pattern at one end of the vortex strings propagates up to a distance of the order of a micrometer and converts into a hexagonal lattice through a domain wall. The square symmetry is seen to transform across the domain wall creating large regions of distorted hexagonal symmetry coexisting with others of square symmetry of few lattice parameters size. The continuous nature of the vortex strings is the unique property that allows us to visualize the transformation of the structure along the vortex direction in a transition width of the order of the average separation between vortices. The experimental data provide valuable information to stimulate and verify the results of theoretical modeling of interfaces.

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FIG. 1. Vortex decoration of the top surface of a sample with commensurate square magnetic pattern for a field of 30.2 Gauss at 4.1 K. The Fe dots of the periodic structure are 260 nm in diameter and 60 nm in height. The dark area indicates the region where a periodic 2D square pattern was generated. The Fe dots positions are depicted with white circles. The scale bar corresponds to 5 µm.
FIG. 2. Vortex decoration of the bottom surface of a 4.5 $\mu$m thick sample beneath the square Fe pattern indicated by the dark region with brighter spots. The insert depicts the corresponding Delaunay triangulation of the framed region, obtained joining the positions of next-near-neighbors vortices with straight lines. The picture shows that the hexagonal vortex lattice has one of its principal directions parallel to one of the square pinning potential axis (indicated by the arrows). The scale bar corresponds to 5 $\mu$m.
FIG. 3. Vortex decoration of the bottom surface of the thinnest sample investigated, $d = 0.5 \mu$m. It shows a complete square symmetry of the whole vortex structure beneath the periodic $2D$ square pattern in the top surface (dark area). The scale bar corresponds to $5 \mu$m.
FIG. 4. Image of the vortex topology in the interface. Decoration of the bottom surface of the 3.5 µm thick sample. Arrows indicate the principal directions of the square pattern on the top surface. Three distorted hexagonal symmetries coexisting with fourfold symmetry regions are shown. Black clumps on white: Domains of vortices in square symmetry matching the square Fe pattern at the top surface. White clumps on light and dark gray: Vortices in distorted hexagons with unit cell area equal to the square pattern unit area ($a_1 = a_{sq}$ parallel to one of the principal directions of the pattern and $a_2 = 1.11a_{sq}$). White clumps on gray: Vortices in distorted hexagons with unit cell area equal to the square pattern unit area, rotated 15 degrees with respect to the horizontal principal direction of the square pattern. The scale bar corresponds to 5 µm.
FIG. 5. Comparison of vortex interface structures at 3.5 and 2.5 μm underneath the same square Fe pattern. The picture is that of the 3.5 μm sample where the black clumps areas depict the square symmetry regions and the superimposed white frames indicate the location of the square symmetry regions in the 2.5 μm thick sample. The scale bar corresponds to 5 μm.