

Observation of a Commensurate Array of Flux Chains in Tilted Flux Lattices in Bi-Sr-Ca-Cu-O Single Crystals

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We report the observation of a novel flux-lattice structure, a commensurate array of flux-line chains. Our experiments consist of the magnetic decoration of the flux lattices in single crystals of Bi-Sr-Ca-Cu-O where the magnetic field is applied at an angle with respect to the conducting planes. For a narrow range of angles, the equilibrium structure is one with uniformly spaced chains with a higher line density of vortices than the background lattice. Our observations are in qualitative agreement with theories which suggest that in strongly anisotropic materials, the vortices develop an attractive interaction in tilted magnetic fields.

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Flux lattices in the high- T_c superconductors have proven to be a rich and interesting area for research. Strong anisotropies and the importance of thermal fluctuations have combined to produce new structures and regimes not previously seen in flux lattices in conventional superconductors. Oval vortices,¹ hexatics,² liquids,³ and intrinsic pinning⁴ have all been seen in static flux-lattice structures. Flux lattices have become important model systems to study the influence of disorder on positional and orientational correlations,⁵ as well as its effect on the character of phase transitions in the lattices.⁶

In this Letter we report the observation of a novel flux-lattice structure which arises when the magnetic field is tilted with respect to the highly anisotropic conducting planes in single crystals of $\text{Bi}_{2.1}\text{Sr}_{1.9}\text{Ca}_{0.9}\text{Cu}_2\text{O}_{8+\delta}$ (BSCCO). For a narrow range of tilt angles, some of the flux lines form an array of flux chains uniformly spaced along the sample. Our experiments provide supporting evidence for recent theories⁷ which suggest that in strongly anisotropic materials, the normally purely repulsive interaction between flux lines develops a bound state at a finite separation.

Our data consist of Bitter patterns^{8,9} of magnetic flux lattices in high-quality single crystals of BSCCO. This technique uses very small ferromagnetic particles to sense the field distribution at the surface of a material. The "smoke" is made by evaporating a magnetic material in a background of inert (helium) gas. A freshly cleaved sample is first field cooled to trap the flux. Magnetic Fe particles of size ~ 50 Å are then formed by evaporation; they thermalize in the gas, drift to the sample surface, and preferentially decorate regions with strong magnetic fields. The surface van der Waals forces hold the particles immobile. The sample is then warmed to room temperature and the lattice is examined using a scanning electron microscope.

The crystals, which were grown using a directional

solidification process,¹⁰ are typically in the form of thin sheets with basal-plane dimensions of up to several cm^2 and thicknesses up to 100 μm . The lattice constants, $a_0 = 5.413(2)$ Å, $b_0 = 5.411(3)$ Å, and $c_0 = 30.91(1)$ Å, were determined using a four-circle x-ray diffractometer. As is typical in these materials, an incommensurate

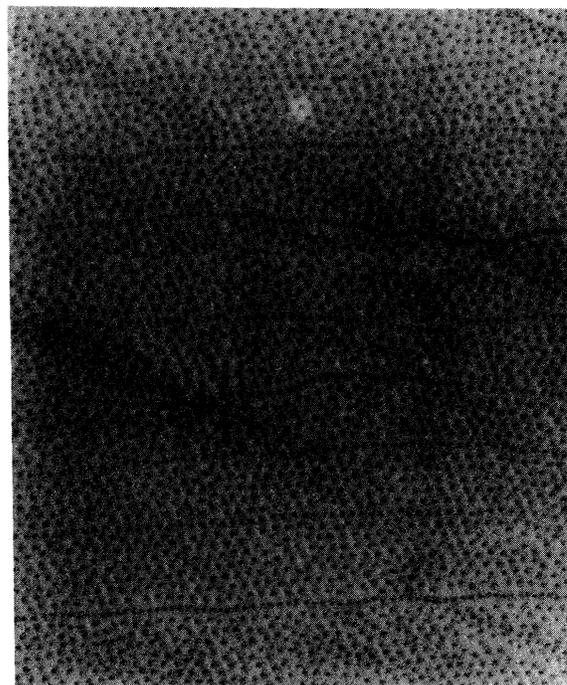


FIG. 1. A region of the decorated crystal taken at an angle of 70° with an applied field of 35 G. The dark regions are the vortices, with an average spacing of 1.4 μm . The chains run approximately perpendicular to the rotation axis, and define the orientation of the vortex lattice between chains. (The field of view is 75 μm by 60 μm .)

periodicity of $4.7(1)b_0$ was observed along the b axis. The crystals, as extracted from the melt, demonstrated a large, sharp (< 5 K 10%-90% transition width) Meissner transition with an onset at 88.5 K, indicative of a homogeneous, bulk superconductor. The crystals used for this experiment were not annealed. Torque magnetometry¹¹ has shown that this system is three dimensional with a large anisotropy parameter, $\Gamma = (m_c/m_a)^{1/2} = 55$.

Figure 1 shows a micrograph of a flux lattice taken with an applied field B_0 of 35 G at an angle $\theta = 70^\circ$. The dark spots indicate the vortices, separated by $1.4 \mu\text{m}$ on average. The geometry is defined in the inset of Fig. 2. The obvious chain structures seen in the photo are always found to run "up and down hill" in the sample, independent of the orientation of the a, b axes. These chains have been seen for angles between 60° and 85° at fields between 23 and 97 G. In the photo, the chains are separated by approximately $10 \mu\text{m}$. This structure is the main result of this paper, and its phenomenology will now be discussed in detail.

Figure 2 shows the data for decorated flux lattices at angles between 0° and 90° . The quantity \bar{B} is the measured average density of flux lines in a picture times the flux quantum. What is plotted is this quantity normalized by the applied field B_0 versus the tilt angle. The simplest picture which emerges is that the field component parallel to the c axis of the sample, given simply by $B_0 \cos \theta$, forms the lattice that we see. The solid line shown in the figure is this dependence, which fits the data quite well.

Up to angles where we begin to see chains, we see no distortion or elongation beyond 4%-5% of the hexagonal

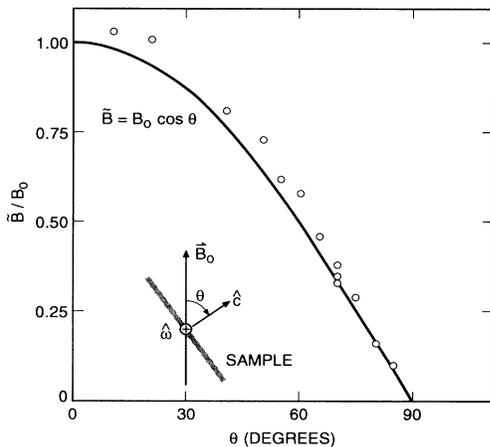


FIG. 2. The field $\bar{B} = n\Phi_0$ defined from the vortex density n divided by the applied field B_0 vs the tilt angle θ . At low angles, both the density $\bar{B} = B_0 \cos \theta$ and correlation functions (not shown) are equivalent to what would be generated by the normal component of B_0 . Inset: The sample geometry defining the tilt axis ω .

flux lattice. This already points to an important role of the anisotropy in this system. For an isotropic superconductor, ignoring surface effects, one would expect to see a distorted vortex lattice with a ratio of lattice parameters proportional to $\cos \theta$. This is due to the projection of a hexagonal vortex lattice perpendicular to the applied field into the plane of the picture. More detailed comparisons with the distorted vortex lattices predicted¹² for tilted fields will be published elsewhere.⁵ In addition, over the range of fields we have studied, we see no effect of the field component parallel to the surface on either the positional or orientational correlation lengths. Also, the parallel component of field does not seem to break the degeneracy of the orientational order with respect to the underlying crystal lattice or tilt direction. However, when the chains form they *do* break the degeneracy of the orientational order. The chains always run up and down hill independent of the a and b directions in the crystal. As can be seen from the photo, this locks in the orientational order in the hexagonal lattice *between* the chains and orients the lattice with one of the lattice vectors always parallel to the chains.

Figure 3 shows a plot of the perpendicular distance between chain vortices d as a function of the lattice parameter of the applied field $a_0 = 1.075(\phi_0/B_0)^{1/2}$ for all fields and angles θ . As shown in the inset of Fig. 3, D is the mean measured distance between the vortices in the $a-b$ plane and $d = D \cos \theta$ is the distance between vortices in a plane perpendicular to the applied field. As the externally applied field is increased, the line density of the vor-

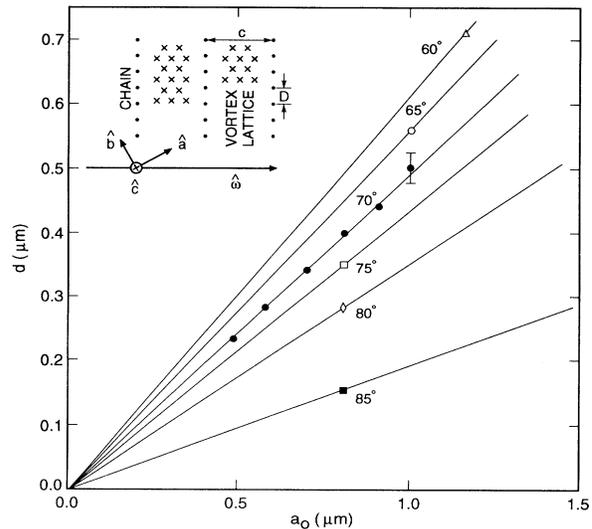


FIG. 3. Inset: The geometry of the chains in the crystal $a-b$ plane. The spacing along the chain is D and between chains is c . The \hat{a} direction is arbitrary. The data at all angles and applied fields are plotted against $a_0 = 4.808/B_0^{1/2}$, the vortex lattice constant for a hexagonal lattice created by the applied field.

tices in the chains also increases. Also, as the angle increases, the line density of the vortices in the chains decreases. It is also clear that the interaction *in* the chains is relatively hard as the vortex spacing is seen to be constant over the length of the chain and constant from one chain to the next. However, the photos suggest that the interaction *between* chains is relatively soft as the distance between them is found to vary quite a bit.

The data for our decorations are tabulated in Table I. The last column gives the quantity D/\tilde{a}_0 , where $\tilde{a}_0 = a_0 \times (\cos\theta)^{-1/2}$ is the lattice parameter due to \tilde{B} . Note that the values cluster around $(\frac{3}{4})^{1/2}$. The inset of Fig. 4 shows a Delaunay triangulation² for a chain formed at an angle of 70° at a field of 97 G where the background lattice is ordered.² The decreased separation of the vortices in the chains over the background lattice implies the existence of topological lattice defects. Our images show dislocations strung along the chains, ordered into pairs with opposite Burgers vectors. The dislocation pairs are roughly evenly spaced apart a distance c (equal to the average interchain distance) along the chain and can be viewed as an array of interstitials. In the triangulation, a dislocation is marked by a fivefold-coordinated vortex separated by one lattice spacing from a sevenfold-coordinated vortex. The pairs consist of two dislocations with equal and opposite Burgers vectors. The cancellation of the strain field of a dislocation pair with equal and opposite Burgers vectors at large distances allows the lattice between the chains to be undisturbed.

Figure 4 shows a plot of the distance between chains c (see inset of Fig. 3) versus a_0 . The data clearly show

TABLE I. Composite data from our decorations at various tilt angles.

Angle (deg)	Field B (G)	Field \tilde{B} (G)	D (μm)	c (μm)	d/c	D/\tilde{a}_0
10	35	36.0				
20	35	35.4				
40	35	28.2				
50	35	25.6				
55	35	21.6				
60	17		1.42	17.6	0.040	0.861
60	35	20.1				
65	23		1.32	16.0	0.035	0.856
65	35	16.1				
70	23		1.46	11.4	0.044	0.852
70	28		1.29	11.1	0.040	0.830
70	35	13.4	1.14	9.5	0.042	0.796
70	47	16.5	1.00	8.7	0.039	0.834
70	69	22.8	0.84	6.8	0.042	0.848
70	97	36.4	0.69	5.2	0.045	0.848
75	35	10.1	1.35	7.5	0.047	0.819
80	35	5.8	1.56	7.9	0.034	0.775
80	47		1.89	7.1	0.046	1.09
85	35	3.7	1.80	8.3	0.019	0.634

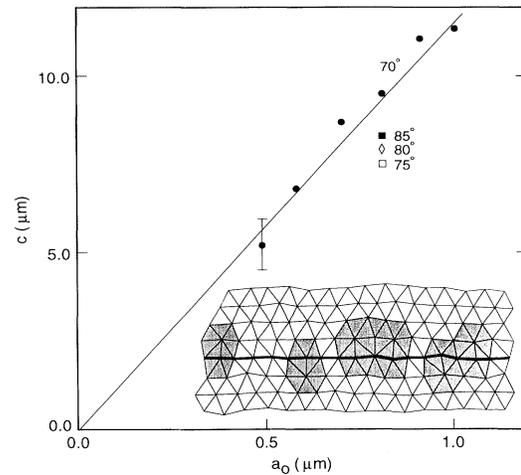


FIG. 4. The spacing between chains c as defined in Fig. 3 vs a_0 for $\theta=70^\circ$. Data for three other angles are also shown. Inset: A Delaunay triangulation for a region near a chain (thick line) taken at 70° and 97 G. To accommodate the increased vortex density along the chain, pairs of dislocations (shaded regions) are formed.

that the distance between chains depends strongly on the magnitude of the applied field and weakly on the angle θ . The linear dependence suggests that c/a_0 is a constant or that the number of vortices between chains is a constant for a fixed angle, independent of the applied field, and depends only weakly on the angle, if at all. The chain structure is then incommensurate with respect to the underlying crystal lattice but commensurate with respect to the flux lattice. This last point is evident in the triangulation shown in Fig. 4. The distance between chains is, on average, c . The line density of vortices in the chain is such that one accumulates *one* extra vortex along the chain for every distance c traveled. Thus the structure that we see is the result of one and only one extra vortex line being added for every chain-superlattice unit cell.

It is clear that the chains orient the flux lattice and their formation destroys the rotational degeneracy which exists in this system, because there are no line defects to orient the flux lattice such as are found in twinned Y-Ba-Cu-O.⁴ This suggests that tilted fields could be used to orient flux lattices, which might be important for technological applications as well as in experiments such as neutron scattering in which one does not wish to obtain a powder pattern for the scattering.

Experimentally, it is clear that the chains are formed by at least a weakening of the repulsive interaction between the vortices in the direction orthogonal to the tilt axis ω due to a tilt of the current paths with respect to the vortex axis resulting from the strong anisotropies in the system. The chains are roughly evenly spaced along the sample due to a repulsive interaction between them. The distance between them is presumably determined by a trade-off in the energy gained by forming a chain and

the cost in energy due to the repulsive interactions between them.

The bound state which we suppose is the underlying driving force for the emergence of this structure has been discussed in several recent theoretical papers.⁷ The general idea is that because of the strong anisotropy, the screening currents must run in the conducting planes and not in planes perpendicular to the axis of the vortex as for an isotropic superconductor.

There have been several calculations⁷ for this situation within the effective-mass approximation. Kogan, Nakagawa, and Thiemann have calculated the potential for a single vortex. A shallow attractive region develops in a direction perpendicular to the rotation axis. Parallel to the rotation axis the interaction is purely repulsive. Buzdin and Simonov have considered a similar situation, but examined the total energy for a chain of vortices near H_{c1} . In both calculations, the equilibrium line density along the chain had no field dependence, in contradiction to the results presented here. In addition, neither calculation gave any indication of a normal vortex lattice between the chains as is seen in our experiments. However, we believe that these calculations have captured the essential physics and need only to be extended to finite vortex density to fully explain our data.

In conclusion, we have observed a novel flux-lattice structure which consists of uniformly spaced chains of vortex lines embedded in a background of a hexagonal flux lattice. These chains may arise from the formation of a bound state between vortices in strongly anisotropic materials in the presence of a tilted magnetic field. The chains appear to repel each other and the distance between them seems to be determined by a competition between the energy gained by forming a chain and that lost by not having them infinitely far apart. These chains provide a nondestructive way of orienting the flux lattice in a given direction without requiring the presence of line defects in the crystal.

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¹G. J. Dolan, F. Holtzberg, C. Field, and T. R. Dinger, *Phys. Rev. Lett.* **62**, 2184 (1989).

²C. A. Murray, P. L. Gammel, D. J. Bishop, D. B. Mitzi, and A. Kapitulnik, *Phys. Rev. Lett.* **64**, 2312 (1990).

³R. N. Kleiman, P. L. Gammel, L. F. Schneemeyer, J. V. Waszczak, and D. J. Bishop, *Phys. Rev. Lett.* **62**, 2331 (1989).

⁴G. J. Dolan, G. V. Chandrasekar, T. R. Dinger, C. Field, and F. Holtzberg, *Phys. Rev. Lett.* **62**, 827 (1989).

⁵E. M. Chudnovsky, *Phys. Rev. B* **40**, 11357 (1989); (to be published); A. Houghton, R. A. Pelcovits, and A. Sudbo (to be published); C. A. Bolle, P. L. Gammel, D. Grier, C. A. Murray, D. J. Bishop, D. B. Mitzi, and A. Kapitulnik (to be published).

⁶M. P. A. Fisher, *Phys. Rev. Lett.* **62**, 1415 (1989); D. S. Fisher, M. P. A. Fisher, and D. A. Huse, *Phys. Rev. B* **43**, 130 (1991).

⁷A. I. Buzdin and A. Yu Simonov, *JETP Lett.* **51**, 191 (1990); V. G. Kogan, N. Nakagawa, and S. L. Thiemann, *Phys. Rev. B* **42**, 2631 (1990).

⁸H. Trauble and U. Essmann, *J. Appl. Phys.* **25**, 273 (1968); N. V. Sarma, *Philos. Mag.* **17**, 1233 (1968).

⁹P. L. Gammel, D. J. Bishop, G. J. Dolan, J. R. Kwo, C. A. Murray, L. F. Schneemeyer, and J. V. Waszczak, *Phys. Rev. Lett.* **59**, 2592 (1987).

¹⁰D. B. Mitzi, L. W. Lombardo, A. Kapitulnik, S. S. Laderman, and R. D. Jacowitz, *Phys. Rev. B* **41**, 6564 (1990).

¹¹D. E. Farrell, S. Bonham, J. Foster, Y. C. Chang, P. Z. Jiang, K. G. Vandervoort, D. J. Lam, and V. G. Kogan, *Phys. Rev. Lett.* **63**, 782 (1989).

¹²L. J. Campbell, M. M. Doria, and V. G. Kogan, *Phys. Rev. B* **38**, 2439 (1988); (private communication).

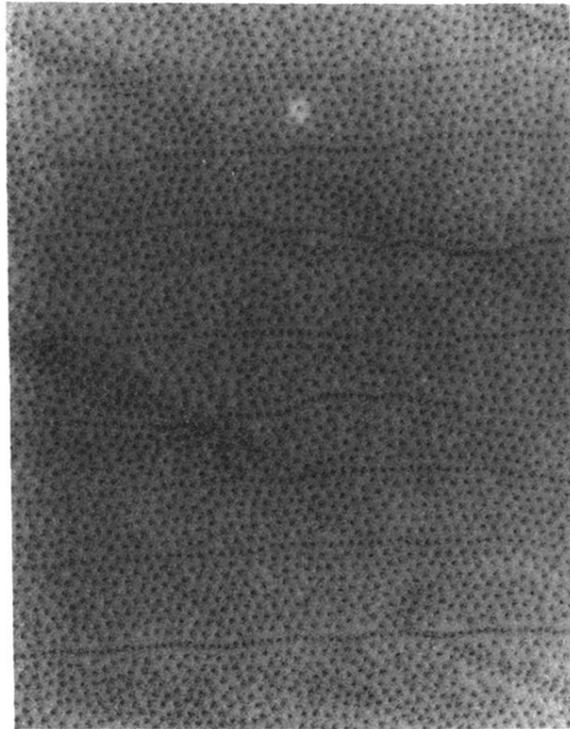


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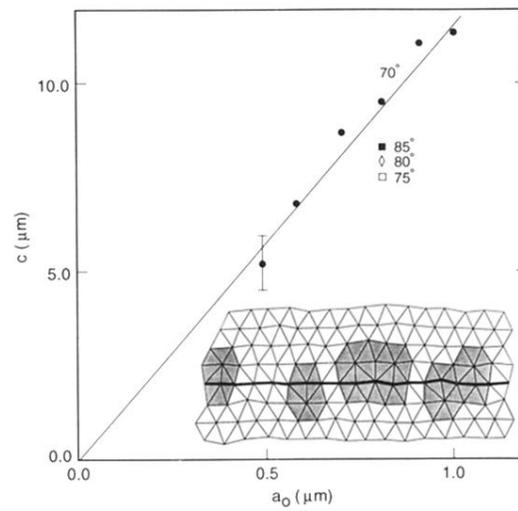


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