PHYSICAL REVIEW B

## Mechanical measurements of two-dimensional flux lattices: Observation of two-stage melting

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Using a high-Q silicon mechanical oscillator we have studied flux lattice melting in twodimensional amorphous composite  $In/InO_x$  superconducting films. In addition to the previously observed solid and liquid phases, the flux lattice exhibits a third stable phase, intermediate between the solid and liquid, which exists at high sheet resistances, large magnetic fields, and low temperatures. For a range of sheet resistances, this phase coexists at a tricritical point with the liquid and solid phases. In the limit of high sheet resistance, only this new phase is stable, preempting the solid phase. We argue that this intermediate phase is unlikely to be a hexatic phase, and is more likely to be an amorphous solid phase.

When the applied magnetic field is between the lower critical field,  $H_{c1}$ , and the upper critical field,  $H_{c2}$ , a type-II superconductor will be permeated by an array of flux lines, each with one quantum of flux hc/2e. As predicted by Abrikosov<sup>1</sup> and subsequently verified by numerous experiments,<sup>2</sup> in three dimensions, these lines form a triangular lattice with long-range positional order and a well-defined shear modulus. However, in thin superconducting films it is possible to study a flux-line lattice which is two dimensional. In this case, the interactions between vortices are logarithmic and a Kosterlitz-Thouless-type melting should occur.<sup>3,4</sup>

For two-dimensional systems Halperin and Nelson<sup>5</sup> and Young<sup>6</sup> argued that melting should occur in two stages. As the temperature is increased, at the first transition, the dislocation pairs unbind and destroy the quasi-long-range positional order of the two-dimensional solid. At this transition the orientational order should remain essentially unchanged. At the second transition, the lattice disclinations unbind, and destroy the orientational order to result in a high-temperature liquid phase with exponential decay of both the orientational and positional correlations. The intermediate, or hexatic phase is expected to be characteristic of all two-dimensional melting. This hexatic phase has been identified and studied in a variety of two-dimensional systems, including freely suspended liquid-crystal films<sup>7</sup> and colloidal systems.<sup>8</sup>

In this Rapid Communication we report on mechanical measurements of flux lattice melting in amorphous composite  $In/InO_x$  superconducting films. The films are sufficiently thin so that the flux lattice is two-dimensional in character. We will present data which indicates that in addition to the previously observed solid and liquid phases for the two-dimensional flux lattice there also exists an intermediate phase in which there is only very short-range positional order for the flux lines. This phase is stabilized by increasing the disorder which may be accomplished by increasing either the magnetic field or the sheet resistance of the films. Although this new phase occurs between the solid and liquid phases, and coexists with them at a tricritical point, we argue that it is unlikely to be a hexatic phase, although its detailed identification remains an open question.

The experimental apparatus is shown in Fig. 1. It is similar to that used in Ref. 9. The experiment consists of a high-Q silicon oscillator<sup>10</sup> onto which a thin ( $\sim$ 100 Å)  $In/InO_x$  film is grown by reactive ion beam sputter deposition in a low-pressure oxygen environment. The films used in this experiment were smooth with an amorphous composite microstructure.<sup>11</sup> Transport measurements on samples deposited simultaneously onto silicon from the same wafer as the oscillator served to characterize the films. The resistive transitions in zero field were always sharp ( $\leq 0.1$  K), indicating uniform films. The superconducting transition in zero field has been shown<sup>12</sup> to be the result of a Kosterlitz-Thouless phase transition describing the thermally induced dissociation of bound vortexantivortex pairs. Homogeneity of the films over long length scales is a prerequisite for this observation since unbinding of the largest pairs dominates the transition.

The oscillators were operated self-resonantly in the simplest beam-bending mode as shown in Fig. 1. The oscillators typically had a Q of  $2 \times 10^5$ , a resonant frequency of 3 kHz and a frequency stability of  $1:10^{-8}$ . By measuring the resonant frequency and dissipation of the oscillator, the bulk modulus of the vortex lattice and dissipation can be determined.

The oscillator's response to the presence of a vortex lattice is as follows. At low temperatures, the vortex lattice will be pinned so that the vortices will move with the film.



FIG. 1. A schematic diagram of the mechanical oscillator.

In the cantilever mode used here the average vortex spacing will oscillate as  $\Delta a_0/a_0 \sim Ax \delta e^{i\omega t}$ , where A is the amplitude of the oscillation, x the distance from the suspension point, and  $\delta$  the thickness of the oscillator. This density modulation gives a coupling to longitudinal sound in the vortex array at the frequency of oscillation. In two dimensions the elastic modulus for longitudinal sound is  $B + \mu$  where  $B \propto H^2$  is the bulk modulus and  $\mu \propto H$  is the shear modulus. As a result of the long-range interaction between vortices,  $B \gg \mu$  and the contribution due to shear can be ignored.

This coupling to longitudinal sound will depend on the relaxation rate of the flux lattice. Since the Hall angle is known to be small<sup>13</sup> the response is purely dissipative. At low temperatures,  $T \ll T_M$ , there is a well-formed flux lattice which remains pinned to the substrate. In this regime, the relaxation rate  $\tau$  for compression of the flux lattice is very long and  $\omega \tau \gg 1$ . Hence, the vortices move with the underlying film. The vortex lattice will then contribute to the elastic response of the oscillator. For  $T \gg T_M$ , thermal fluctuations will dominate the motion of the vortices. In this regime the vortex fluid will relax rapidly ( $\omega \tau \ll 1$ ). The vortices are decoupled from the film and there will be no contribution to the oscillator's response from the elastic properties of the flux lattice. Near  $T_M$  this rate will be strongly temperature dependent and a crossover between the two regimes will occur with a dissipation peak and a softening of the oscillator's response at  $\omega \tau \sim 1$ . We define this dissipation peak as the melting temperature as in Ref. 9. The subject of this paper is a second dissipation peak which occurs at higher fields and generally lower temperatures than our previous study. We feel that this indicates a transition into either a hexatic phase or, which we argue is more likely, an amorphous glass phase of the two-dimensional flux lattice.

Shown in Fig. 2 is the frequency shift and dissipation for a film with  $R_{\Box}$ =343  $\Omega/\Box$  at 80 kG. The feature labeled  $T_M$  is the melting temperature where there is a large (off scale) dissipation peak and a frequency shift of ~1700 ppm. The second feature, which we believe is the transition to an amorphous glass phase, is labeled  $T_G$ . It is accompanied by a small dissipation peak and a frequency shift of ~5 ppm. Shown in Fig. 3 is the phase diagram in both field and temperature for the melting and glass



FIG. 2. The frequency shift and dissipation are shown as a function of temperature at a field of 80 kG for a film with a sheet resistance of 343  $\Omega/\Box$ .



FIG. 3. The phase diagram is shown as a function of field and temperature for all three phases of the flux lattice for the same film as in Fig. 2.

transitions. These data are for the same film as shown in Fig. 2. Note that the three phases meet at what appears to be a tricritical point.

Shown in Fig. 4 is the phase diagram for a film with a sheet resistance of 904  $\Omega/\Box$ . As was shown in Ref. 3, when the sheet resistance is increased, the melting temperature is reduced according to the formula

$$\frac{T_c}{T_M} = \left[ 1 + 3.8 \frac{R_{\Box}}{A_1 R_c} \right] \tag{1}$$

with  $R_c = 4.12 \text{ k}\Omega/\Box$  and  $A_1$  predicted to lie in the range  $0.4 < A_1 < 0.75$ . Experimentally,<sup>9</sup> it was found that  $A_1 \approx 0.5$  for In/InO<sub>x</sub> films. For the sample shown in Fig. 4 the solid phase has disappeared, preempted by the new phase. As the sheet resistance is increased, the  $T_G$  line moves down in field. In Fig. 4,  $T_G$  for the  $R_{\Box} = 343 \Omega/\Box$ 



FIG. 4. The phase diagram is shown for a film with  $R_{\Box}=904$   $\Omega/\Box$ . Note the absence of a solid phase. The dash-dotted line is  $T_G$  for the film with  $R_{\Box}=343 \ \Omega/\Box$ . Note that the  $T_G$  line moves down as the disorder is increased.

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film shown in Fig. 2 is shown as the dash-dotted line.

Clearly this third phase cannot arise from gross sample inhomogeneities as these films are known to be very uniform, with their properties completely determined by  $R_{\Box}$ . In addition, the slope of the  $T_G$  line is opposite to that of  $T_M$ . Since earlier measurements<sup>9</sup> have shown the effect of increasing disorder on melting is a reduction of  $T_M$ , with little change in the field-dependent slope,  $T_G$  could not represent a portion of the sample with a different  $R_{\Box}$ . This new phase is clearly favored by increasing disorder. These measurements are all below the collective pinning peak due to flux flow as independently measured in these samples. This means that in this regime, as the field is increased the effectiveness of pinning increases and the flux lattice is increasingly disordered. The data in Figs. 3 and 4 show that this increase in disorder pushes the transition temperature higher. Also, as the sheet resistance is increased and the solid phase is inhibited, the new phase becomes favored over a larger part of the phase diagram.

A qualitative analysis of the magnitudes of the frequency shift shows that this phase is not a solid with a long mechanical relaxation time. The bulk modulus of the solid phase may be obtained from the total interaction energy of the vortex array

$$\frac{\Phi_0^2}{8\pi^2\Lambda(T)}\int n(r)\ln(r-r')n(r')d^2r\,d^2r'\,.$$
 (2)

The vortex density is  $n(r) - B/\Phi_0$ . The integral is cut off by either the size of the system, or  $\Lambda(T)$ , the effective two-dimensional penetration depth. In the solid phase, this gives the observed  $H^2$  dependence of the frequency shift and also the approximate magnitude (1700 ppm at 80 kG). However, the transition to the glass phase shows only a 5 ppm shift suggesting much shorter mechanical relaxation rate and a weaker coupling to the film.

The observed magnitudes of the oscillator response are consistent with either hexatic phase or an amorphous glass phase. We feel that the hexatic phase is an unlikely explanation of our observations based on the following three reasons. First, theoretical analysis<sup>3</sup> and simulations<sup>14</sup> have suggested that the hexatic phase is not particularly sensitive to disorder. In particular, it should be no more sensitive than the melting temperature itself. However, there is no evidence that increasing disorder stabilizes the hexatic phase and causes the liquid to hexatic transition to occur at *higher* temperatures.

The second reason is that the mechanical response of a hexatic phase has been extensively studied theoretically.<sup>15</sup> The hexatic phase should have a mechanical response identical to that of a liquid. This would make the hexatic-liquid transition unobservable in an experiment such as ours. It should be noted, however, that in mechanical measurements of freely suspended films<sup>16</sup> the hexatic-liquid transition *is* seen and is characterized by a small (100 times smaller than at the solid-hexatic transition) dissipation peak. In our experiment the feature at  $T_G$  is approximately 300 times smaller than that at  $T_M$ .

The third reason is, as is shown in Ref. 15, that as the density is increased, the hexatic phase should become less favored. This is in contrast to the observed intermediate phase, which is observed at high fields (high flux line density).

Because of these reasons we feel that this newly observed phase is more likely to be an amorphous glass stabilized by the disorder.<sup>17</sup> It is an open question as to whether  $T_G$  is a true phase transition as has been suggested for flux lattices in the high- $T_c$  superconductors<sup>18</sup> or merely a crossover point in the dynamical response of the vortex liquid. Clearly more theoretical and experimental work is required to address these issues.

In conclusion, we have measured melting in twodimensional flux lattices. We have found the first experimental evidence in this system for a new phase which occurs intermediate between the liquid and solid phases with a tricritical point where all three phases can coexist. This phase is stabilized by increasing disorder in the flux lattice. We argue that it is unlikely to be a hexatic phase, and is probably an amorphous glass. The details of the identification are as yet incomplete.

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