

## Vortex phase transformations probed by the local ac response of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ single crystals with various doping

Yoichi Ando

*Central Research Institute of Electric Power Industry, Komae, Tokyo 201-8511, Japan*

K. Nakamura

*Central Research Institute of Electric Power Industry, Komae, Tokyo 201-8511, Japan  
and Department of Energy Science, Tokyo Institute of Technology, Nagatsuta, Yokohama 226-8502, Japan*

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The linear ac response of the vortex system is measured locally in  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$  crystals at various doping, using a miniature two-coil mutual-inductance technique. It was found that a steplike change in the local ac response takes place exactly at the first-order transition (FOT) temperature  $T_{FOT}(H)$  determined by a global dc magnetization measurement. The  $T_{FOT}(H)$  line in the  $H$ - $T$  phase diagram becomes steeper with increasing doping. In the higher-field region where the FOT is not observed, the local ac response still shows a broadened but distinct feature, which can be interpreted to mark the growth of a short-range order in the vortex system. [S0163-1829(99)50518-3]

The vortex phase diagram of the highly anisotropic high- $T_c$  superconductor  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$  (BSCCO) has been intensively studied in the past few years. In superconductors, strong supercurrents flow near the surface, which produce nonuniform magnetic-field distribution in the sample. Such nonuniformity broadens the thermodynamic phase transitions and thereby hinders the study of the phase diagram. Also, the surface (or edge) currents produce a geometrical barrier<sup>1</sup> in flat samples at low fields, which gives rise to a hysteretic behavior.<sup>2</sup> In higher fields, the Bean-Livingston surface barrier is known to be strong in BSCCO and this makes the global properties of the vortex system complicated.<sup>3</sup> A useful way to get rid of the effects of the surface and the magnetic-field nonuniformity is to measure the electromagnetic properties locally. There have been a number of efforts along this line,<sup>2-7</sup> and the true nature of the vortex phases of BSCCO is beginning to be fully understood. For example, local magnetization measurements using microscopic Hall probes have found, quite conclusively, the presence of a first-order transition (FOT) of the vortex system.<sup>4</sup> With the improvement of instrumentation and crystal quality, it has become clear that the first-order transition can also be determined as a step in the global dc magnetization measured with a superconducting quantum interference device (SQUID) magnetometer.<sup>8-10</sup>

The miniature two-coil mutual-inductance technique<sup>11</sup> has been used for the study of the vortex phase diagram of BSCCO.<sup>6,7</sup> With this technique, a small ac perturbation field is applied near the center of the crystal and therefore the surface barrier, which hinders vortex entry and exit at the edge, has minimal effect on the measured response. Because of this advantage, a sharp distinct change in the local ac response has been observed<sup>6,7</sup> and such a feature has been associated with a decoupling transition<sup>12-14</sup> of the vortex lines. It is naturally expected that the “decoupling line” thus determined is identical to the FOT measured by dc magnetization measurements, although there has been no direct comparison between the two phenomenon measured on an identical sample. Since it is known that the first-order transition

in the dc magnetization has a critical point and thus disappears above a certain field,<sup>4</sup> it is intriguing how the “decoupling” signal of the miniature two-coil technique transforms at higher fields, above the critical point. In fact, the nature of the vortex matter in the field range above the critical point is still controversial;<sup>15-17</sup> since the ac technique can probe the growth of the correlation lengths of the vortex system,<sup>18</sup> it is expected that the local ac measurement using the two-coil technique gives a new insight into the vortex phase transformations.

In this paper, we present the results of our miniature two-coil measurements and the global dc magnetization measurements on the same crystals. It is found that these two techniques detect the anomaly at the same temperature  $T_{FOT}(H)$ , directly demonstrating that the two phenomena are of the same origin. We measured crystals with three different dopings and confirmed that the result is reproducible among systems with different anisotropy. In higher fields where the FOT is not observed by the global dc magnetization measurement, a distinct feature is still observable in the local ac response and the position of such feature is weakly frequency dependent. We discuss that the frequency-dependent feature above the critical point is likely to originate from the growth of a short-range order in the vortex system.

The single crystals of BSCCO are grown with a floating-zone method and are carefully annealed and quenched to obtain uniform oxygen content inside the sample. We obtained three different dopings by annealing the crystals at different temperatures in air; annealing at 800 °C for 72 h gives an optimally doped sample with  $T_c=91$  K (sample A), 650 °C for 100 h gives a lightly overdoped sample with  $T_c=88$  K (sample B), and 400 °C for 10 d gives an overdoped sample with  $T_c=80$  K (sample C). All the samples have the transition width of less than 1.5 K. A tactful quenching at the end of the anneal is essential for obtaining such a narrow transition width.  $T_c$  is defined by the onset temperature of the Meissner signal in the dc magnetization

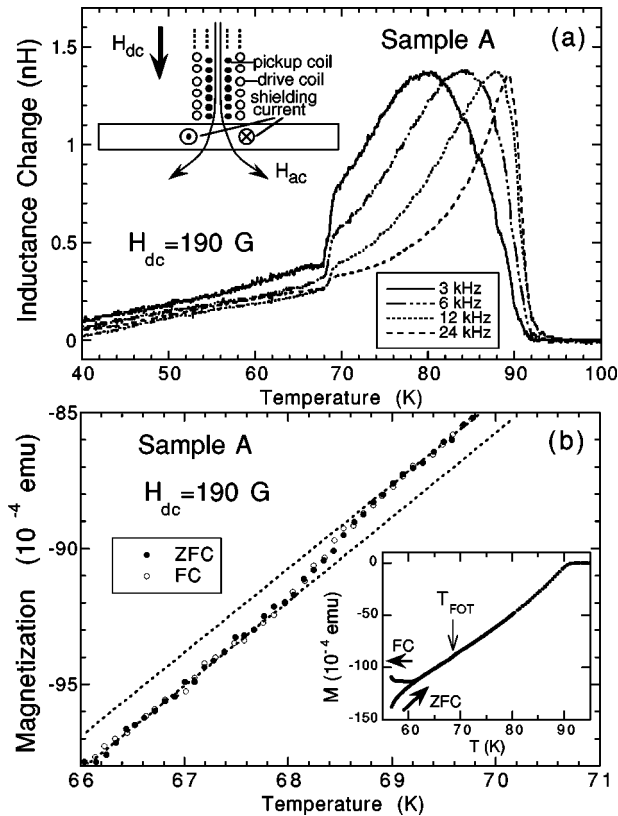


FIG. 1. (a)  $T$  dependence of the in-phase signal from the two-coil measurement on sample A in 190 G, taken at 3, 6, 12, and 24 kHz. Inset shows a schematic of our two-coil measurement. (b)  $T$  dependence of the dc magnetization of sample A in 190 G. Both the zero-field-cooled (ZFC) data and the field-cooled (FC) data are shown. The dotted lines are guides to the eyes. Inset show the dc magnetization in a wider temperature range.

measurement. The crystals are cut into platelets with lateral sizes larger than  $3 \times 3$  mm<sup>2</sup> and the thickness of the samples are typically 0.02 mm. We used a very small (0.6 mm diameter) coaxial set of pickup and drive coils for our two-coil mutual-inductance measurements (see the inset to Fig. 1). The details of our technique have been described elsewhere.<sup>6,18</sup> The amplitude of the drive current  $I_d$  was 7.5, 7.5, and 1.0 mA for the measurements of samples A, B, and C, respectively. The linearity of the measured voltage with respect to  $I_d$  was always confirmed. These  $I_d$  produce the ac magnetic field of about 0.01–0.1 G at the sample. We emphasize that our two-coil geometry mainly induces and detects shielding currents flowing near the *center* of the sample, while usual ac-susceptibility measurements are most sensitive to shielding currents flowing near the *edge* of the sample. All the two-coil measurements are done in the field-cooled procedure. The global dc magnetization measurements are done with a Quantum Design SQUID magnetometer equipped with a slow temperature-sweep operation mode.

Figure 1(a) shows the temperature dependence of the in-phase signals of our two-coil measurement on sample A in 190 G, taken at various frequencies from 3 kHz to 24 kHz. To compare the signals from different frequencies, the data are plotted in the unit of inductance change. It is apparent that there is a frequency-independent steplike change at a temperature  $T_d$ , which is 68.5 K here. The temperature de-

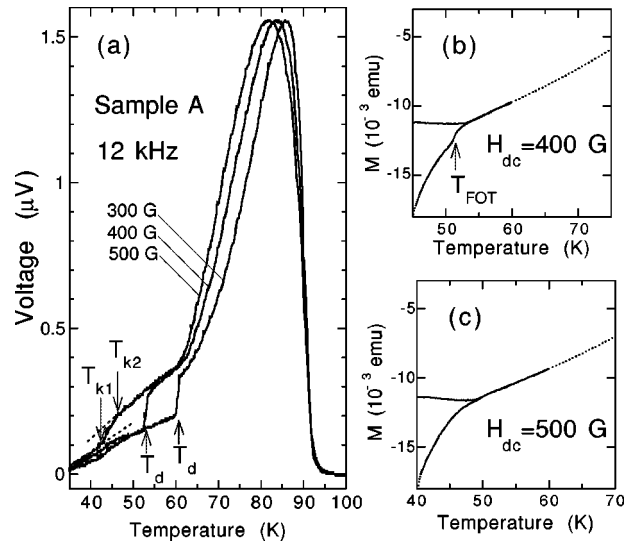


FIG. 2. (a)  $T$  dependence of the in-phase signal of the two-coil measurement on sample A in 300, 400, and 500 G. The dotted lines near  $T_{k1}$  and  $T_{k2}$  are guides to the eyes. (b) and (c):  $T$  dependence of the dc magnetization in 400 and 500 G, respectively.

pendence of the global dc magnetization in the same field is shown in Fig. 1(b), which shows that the FOT is taking place at exactly the same temperature as the steplike change in the two-coil signal.

According to the linear ac-response theory of the vortex system, the ac response is governed by the ac penetration depth  $\lambda_{ac}$ .<sup>19,20</sup>  $\lambda_{ac}$  in our configuration is related to the in-plane resistivity  $\rho_{ab}$  in the manner  $\rho_{ab} = \text{Re}(i\omega\mu_0\lambda_{ac}^2)$ .<sup>18</sup> It has been reported that the apparent resistivity measured in the mixed state of BSCCO is largely dominated by the surface current.<sup>3</sup> Recent measurement of the bulk and surface contributions to the resistivity found<sup>21</sup> that the bulk contribution shows a sharp change at the FOT, while the surface contribution is governed by the surface barrier and shows a broader change. Since our measurement is not sensitive to the edge current, it is expected that  $\lambda_{ac}$  of our measurement reflects mainly the bulk resistivity. Therefore, the steplike change in the local ac response is most likely to originate from the reported sharp change in the bulk resistivity.<sup>21</sup> We note that there has been a confusion about the origin of the steplike change in the local ac response measured by the miniature two-coil technique and it was discussed that the source of the sudden change may be related to a change in the  $c$ -axis resistivity.<sup>6,7</sup>

Figure 2(a) shows the  $T$  dependence of the in-phase signals of our two-coil measurement on sample A in three different magnetic fields. We observed that the sharp steplike change in the two-coil signal becomes broadened when the magnetic field exceeds a certain limit  $H_{lim}$ ; in the case of sample A, the steplike change is observed in up to 400 G, but becomes broadened at 500 G. It was found that this  $H_{lim}$  corresponds to the magnetic-field value at the critical point of the FOT; namely, the FOT in the dc magnetization measurement also disappears in fields above  $H_{lim}$ . Figures 2(b) and 2(c) show that the FOT is observed in the dc magnetization at 400 G but is not detectable at 500 G. This is also a clear evidence that the origin of the steplike change in the two-coil signal is the FOT.

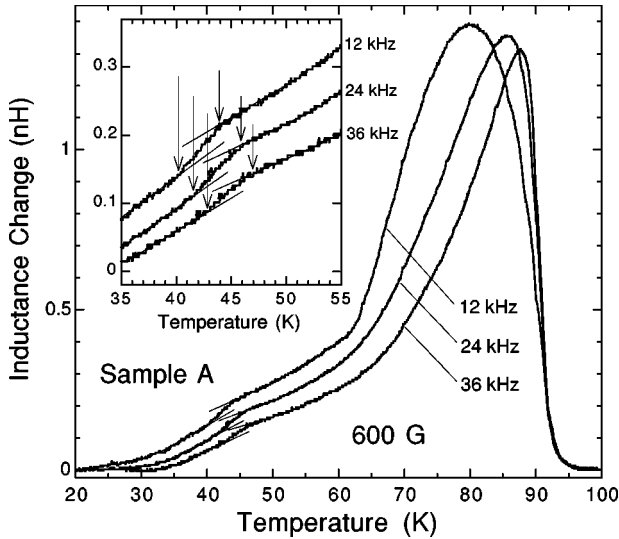


FIG. 3.  $T$  dependence of the in-phase signal of the two-coil measurement on sample A in 600 G taken at 12, 24, and 36 kHz. Inset shows a magnified view of the data near  $T_{k1}$  and  $T_{k2}$ , which are marked by arrows. The thin solid lines are guides to the eyes.

In Fig. 2(a), the 500 G data do not show a steplike change, but clear changes in the slope at two separate temperatures,  $T_{k1}$  and  $T_{k2}$ , are discernible. The signal changes much more rapidly between  $T_{k1}$  and  $T_{k2}$  compared to the temperatures outside of this region, so the data show that the steplike change at  $T_d$  is broadened to the temperature region of  $T_{k1} < T < T_{k2}$ . Figure 3 shows the in-phase signals of sample A in 600 G, which is above  $H_{lim}$ , taken at various frequencies. Apparently,  $T_{k1}$  and  $T_{k2}$  inferred from the 600 G data change with frequency, although the change is small. This indicates that  $T_{k1}$  and  $T_{k2}$  do not mark a true phase transition but mark a crossover.

Figures 4(a) and 4(b) show the in- and out-of-phase signals of samples B and C, respectively, in two selected magnetic fields below and above  $H_{lim}$ . Also in these two samples, the  $T$  dependence of the two-coil signals show a steplike change in magnetic fields below  $H_{lim}$ , while the change is broadened in  $H > H_{lim}$ . Figure 5 shows the  $T_d(H)$  lines for the three samples determined by our two-coil measurements. Clearly, the  $T_d(H)$  line tends to be steeper for more overdoped samples. The  $T_{FOT}$  data obtained from the dc magnetization are also plotted in Fig 5; apparently,  $T_d(H)$  and  $T_{FOT}(H)$  agree very well in all three samples. The inset to Fig. 5 shows the  $T_d(H)$  lines together with the  $T_{k1}(H)$  and  $T_{k2}(H)$  lines at higher fields (determined with 12 kHz), plotted versus normalized temperature  $T/T_c$ . The  $T_{k1}(H)$  and  $T_{k2}(H)$  lines are much steeper compared to the  $T_d(H)$  line.

After the existence of the first-order transition of the vortex system in BSCCO had been established,<sup>4</sup> much effort was devoted to the clarification of the details of the phase diagram. There has been accumulating evidence that the FOT line is a sublimation line, at which a solid of vortex lines transforms into a gas of pancake vortices.<sup>22,23</sup> In the  $H$ - $T$  phase diagram, there are two lines other than the FOT line, called the ‘‘depinning line’’ and the ‘‘second-peak line.’’<sup>24</sup> The three lines merge at the critical point; the depinning line separates the low- and high-temperature regions at

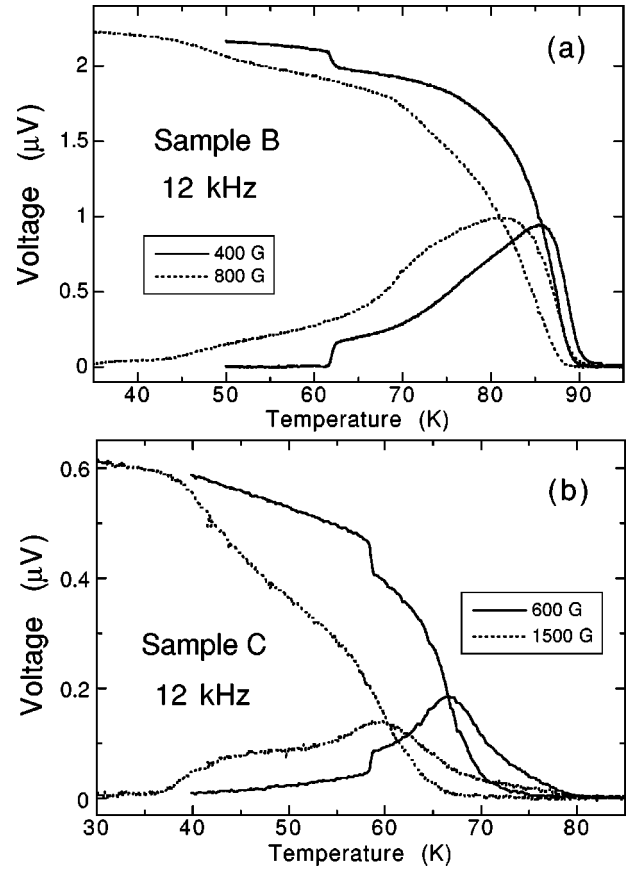


FIG. 4. (a) and (b): In- and out-of-phase signals of samples B and C, respectively, in two selected magnetic fields below and above  $H_{lim}$ .

fields above  $H_{lim}$  and the second-peak line separates the high- and low-field regions at low temperatures. Apparently, our  $T_{k1}(H)$  and  $T_{k2}(H)$  lines are very similar to the depinning line; thus, an examination of the  $T_{k1}(H)$  and  $T_{k2}(H)$  lines is expected to give an insight into the nature of the depinning line.

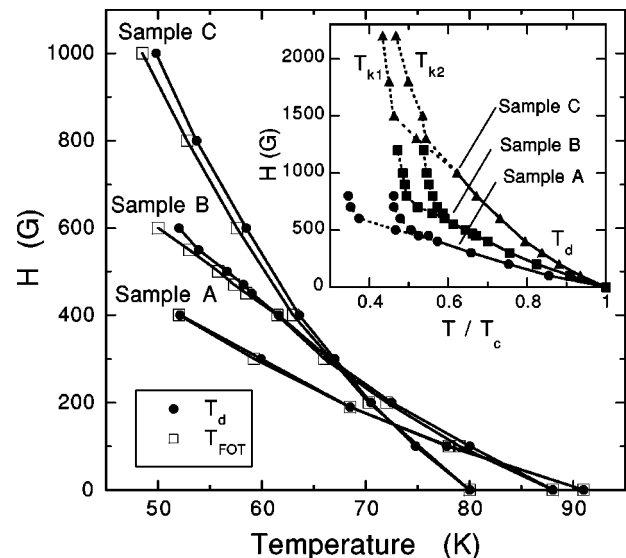


FIG. 5.  $T_d(H)$  lines and the  $T_{FOT}(H)$  lines for the three samples. Inset:  $T_d(H)$ ,  $T_{k1}(H)$ , and  $T_{k2}(H)$  lines (determined at 12 kHz), plotted vs  $T/T_c$ .

Since the steplike change at  $T_d(H)$  marks an abrupt onset of the long-range correlation in the vortex system, the broadened change between  $T_{k1}(H)$  and  $T_{k2}(H)$  is expected to indicate an increase of a (short-range) correlation in the vortex system. In general, a probe with higher frequency looks at physics at shorter length scale;<sup>25</sup> in the case of our local ac response,  $\lambda_{ac}(\omega)$  is smaller for larger  $\omega$ . With decreasing temperature, it is expected that the local ac response shows a qualitative change when the  $c$ -axis correlation length  $L_c$  of the vortex system starts to grow, and another qualitative change at a lower temperature is also expected when  $L_c$  becomes comparable to  $\lambda_{ac}(\omega)$ . This is one possible scenario for what is happening at  $T_{k2}$  and  $T_{k1}$ . The facts that  $T_{k1}$  and  $T_{k2}$  are dependent on frequency and that a higher frequency gives a higher apparent  $T_{k1}$  are consistent with the above scenario.

Recently, Fuchs *et al.* used the change in the surface-barrier height for the determination of the vortex phase transformations<sup>15</sup> (note here that the surface barrier is different from the geometrical barrier which is only effective at low fields near  $H_{c1}$ ), and the presence of a new transition line,  $T_x$  line, at temperatures higher than the depinning line (and above the FOT line) was suggested. Since it is almost clear that the vortex phase above the FOT line is a gas of pancake vortices at temperatures higher than this new  $T_x$  line,<sup>15</sup> the existence of the  $T_x$  line implies that the depinning line separates a highly disordered entangled vortex solid (low-temperature side) from either (a) disentangled liquid of lines with hexatic order, or (b) some kind of solid which consists of an aligned stack of ordered two-dimensional pancake layers.<sup>15</sup> Our data suggest that the latter possibility (b) is more likely, because the growth of the short-range correlation between  $T_{k1}$  and  $T_{k2}$  has a natural meaning of a growth of the alignment of the pancake layers in the latter

picture. Note that we did not observe any feature which can be associated with the  $T_x$  line; this is reasonable because the  $T_x$  line only manifests itself in a change in the surface barrier, which has little effect on our measurement.

Finally, let us briefly discuss the magnetic-field dependence of  $T_d$ . As has been reported,<sup>6,7</sup> the  $T_d(H)$  line measured with the two-coil technique can be well fitted with the formula for the decoupling line.<sup>12-14</sup> This is actually a matter of course, because our  $T_d(H)$  line is identical to the  $T_{FOT}$  line and the FOT is most likely to be a sublimation transition, which is essentially a decoupling transition. Fittings of our data to the decoupling formula<sup>12,13</sup>  $H \approx H_0(T_c - T_d)/T_d$  give the anisotropy ratio  $\gamma$  of  $\sim 100$ ,  $\sim 85$ , and  $\sim 77$  for samples A, B, and C, respectively (the prefactor is given by  $H_0 \approx \alpha_D \gamma^2 \phi_0^3 / [4\pi\lambda(0)]^2 T_c d$ , where  $\alpha_D \approx 0.1$  is a constant,  $d = 15 \text{ \AA}$  is the spacing between the bilayers, and  $\lambda(0) \approx 2000 \text{ \AA}$  is the penetration depth).

In summary, we measured the local ac response of three BSCCO crystals (optimally doped, lightly overdoped, and overdoped samples) using a miniature two-coil technique and compared the result with a global dc magnetization measurement. The origin of the step-like change in the two-coil measurement is identified to be the first-order transition (FOT), where the *bulk* resistivity (which is free from the edge contribution) is reported to show a sharp change.<sup>21</sup> The sudden steplike change in the two-coil signal starts to be broadened at fields above  $H_{lim}$ , where the FOT is no longer observed. This broadened change takes place between  $T_{k1}$  and  $T_{k2}$  and these two temperatures are still well defined, although they are frequency dependent. We discussed that the observation of the feature at  $T_{k1}$  and  $T_{k2}$  is likely to indicate the growth of a short-range correlation of the vortex matter, which gives a clue in identifying the nature of the depinning line.

<sup>1</sup>E. Zeldov *et al.*, Phys. Rev. Lett. **73**, 1428 (1994); Th. Schuster *et al.*, *ibid.* **73**, 1424 (1994).

<sup>2</sup>D. Majer, E. Zeldov, and M. Konczykowski, Phys. Rev. Lett. **75**, 1166 (1995).

<sup>3</sup>D.T. Fuchs *et al.*, Nature (London) **391**, 373 (1998).

<sup>4</sup>E. Zeldov *et al.*, Nature (London) **375**, 373 (1995).

<sup>5</sup>T. Tamegai *et al.*, Phys. Rev. B **45**, 8201 (1992).

<sup>6</sup>Y. Ando *et al.*, Phys. Rev. B **52**, 3765 (1995); **59**, 6563(E) (1999).

<sup>7</sup>R.A. Doyle *et al.*, Phys. Rev. Lett. **75**, 4520 (1995).

<sup>8</sup>H. Pastoriza *et al.*, Phys. Rev. Lett. **72**, 2951 (1994).

<sup>9</sup>T. Hanaguri *et al.*, Physica C **256**, 111 (1996).

<sup>10</sup>S. Watauchi *et al.*, Physica C **259**, 373 (1996).

<sup>11</sup>B. Jeanneret *et al.*, Appl. Phys. Lett. **55**, 2336 (1989).

<sup>12</sup>L.I. Glazman and A.E. Koshelev, Phys. Rev. B **43**, 2835 (1991).

<sup>13</sup>L.L. Daemen *et al.*, Phys. Rev. B **47**, 11 291 (1993).

<sup>14</sup>R. Ikeda, J. Phys. Soc. Jpn. **64**, 1683 (1995).

<sup>15</sup>D.T. Fuchs *et al.*, Phys. Rev. Lett. **80**, 4971 (1998).

<sup>16</sup>E.M. Forgan *et al.*, Czech. J. Phys. **46**, 1571 (1996).

<sup>17</sup>B. Horovitz and T.R. Goldin, Phys. Rev. Lett. **80**, 1734 (1998).

<sup>18</sup>Y. Ando *et al.*, Phys. Rev. B **50**, 9680 (1994).

<sup>19</sup>E.H. Brandt, Phys. Rev. Lett. **67**, 2219 (1991).

<sup>20</sup>J.R. Clem and M.W. Coffey, Phys. Rev. B **46**, 14 662 (1992).

<sup>21</sup>D.T. Fuchs *et al.*, Phys. Rev. Lett. **81**, 3944 (1998).

<sup>22</sup>Y. Matsuda *et al.*, Phys. Rev. Lett. **78**, 1972 (1997).

<sup>23</sup>D.T. Fuchs *et al.*, Phys. Rev. B **55**, R6156 (1997).

<sup>24</sup>B. Khaykovich *et al.*, Phys. Rev. Lett. **76**, 2555 (1996).

<sup>25</sup>D.S. Fisher, M.P.A. Fisher, and D. Huse, Phys. Rev. B **43**, 130 (1991).