## Specific heat of the random-field Ising system $Fe_{0.46}Zn_{0.54}F_2$

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Heat pulse specific-heat measurements are compared with data from optical linear birefringence techniques in the dilute antiferromagnet  $Fe_{0.46}Zn_{0.54}F_2$  for applied magnetic fields  $H \leq 1.5$  T. The optical technique is shown to reproduce the dynamically rounded symmetric specific-heat peak for  $H \neq 0$ . Hysteretic effects resulting from cooling the sample in the field are observed in a similar manner for both techniques. The relationship to previous specific-heat measurements in other random-field systems is discussed.

The static and dynamic properties of the threedimensional (d=3) random-field Ising model (RFIM) are being characterized as a result of numerous experimental and theoretical investigations. It is now well established<sup>1-3</sup> that a phase transition exists for the d=3RFIM and appears to be second order.<sup>4,5</sup> Experimental attention is now directed toward measurements of the static and dynamic critical behavior. We present measurements of the specific heat obtained from pulsed heat techniques and address the relation of this more conventional technique to the optical ones first used to characterize the magnetic specific-heat critical behavior of the RFIM.

The prime experimental realization of the RFIM is found in the dilute anisotropic antiferromagnet with short-range interactions and a magnetic field (*H*) applied along the easy axis.<sup>6,7</sup> Of the systems studied,  $Fe_xZn_{1-x}F_2$  is the best understood and characterized, although experiments have been carried out on systems such as  $Mn_xZn_{1-x}F_2$  and  $Fe_xMg_{1-x}Cl_2$  as well. These latter systems will be discussed after the results of the present experiment.

The antiferromagnet FeF<sub>2</sub> has a body-centered tetragonal (bct) structure and is isomorphous with the diamagnet  $ZnF_2$ . The magnetic interactions in  $FeF_2$  consist of a dominant second-nearest-neighbor antiferromagnetic ex-change  $J_2 = 2.62$  K, each Fe<sup>2+</sup> ion with eight such neighbors, and a strong, predominantly single-ion anisotropy D = 9.29 K.<sup>8</sup> Fe<sub>x</sub>Zn<sub>1-x</sub>F<sub>2</sub> crystals can be grown with extremely high crystalline quality. With considerable effort, crystals can be produced with relatively small gradients in the concentration.<sup>9</sup> The gradients, if large, may drastically effect critical behavior determinations.<sup>10</sup> For the sample used in the present measurements the gradient has been well characterized and gradient effects will not be observed for reduced temperatures  $|t| > 4 \times 10^{-4}$ , where  $t = (T - T_c)/T_c$ . This is the same extremely homogeneous sample used in a variety of recent experiments. It is approximately 1 cm in diameter and 2 mm thick and has a mass of 0.60 g.

The critical behavior of the magnetic specific heat in the RFIM has been extensively investigated in the dilute antiferromagnet  $Fe_x Zn_{1-x}F_2$  using the optical techniques of linear birefringence<sup>11</sup> and, more recently, Faraday rotation (FR).<sup>12,13</sup> Strong evidence for the existence of the RFIM phase transition in three dimensions was first observed using the birefringence technique.<sup>1,14</sup> That evidence is in conflict with interpretations derived from early neutron scattering studies,<sup>15</sup> in which the transition was claimed to be destroyed for three dimensions. It is now realized from a variety of experiments, including neutron scattering,<sup>4</sup> that dynamical effects and metastable domain structure are intrinsic to the observations of the RFIM transitions in the dilute Ising magnets, even on laboratory time scales.<sup>12</sup> It was the unjustified neglect of these effects that led, in part, to the incorrect conclusion that the d = 3 transition was destroyed. Similar criticism applies to later claims by several of the same authors that the same transition is first order.<sup>16</sup> If, on the other hand, the dynamical effects are properly accounted for, the new critical behavior for the d = 3 transition may be studied.

The interpretation of the temperature derivative of the optical linear birefringence,  $d(\Delta n)/dT$ , as being proportional to the magnetic specific heat,  $^{11,17-20}C_m/R$ , has allowed for the evaluation of the specific-heat critical behavior of the random-field Ising model. With this interpretation,  $d(\Delta n)/dT$  shows that the d = 3 RFIM exhibits a symmetric, nearly logarithmic divergence, albeit with severe dynamical rounding.<sup>12-14</sup> Initially the proportionality was assumed to hold in the dilute samples since it had been shown to do so in the pure systems FeF<sub>2</sub> and  $MnF_2$  to a high degree of accuracy throughout the critical region. <sup>11,17-20</sup> This still held open, to some extent, the question of whether an applied magnetic field could in some way effect the proportionality. This could conceivably happen, for example, if the Zeeman contributions to the energy behaved differently from the spin-spin coupling contributions.<sup>21</sup> The birefringence technique, being insensitive to the Zeeman energy would, in this case, be misleading. This issue was essentially settled by Faraday rotation experiments on  $Fe_x Zn_{1-x}F_2$  by Kleemann, King, and Jaccarino.<sup>12</sup> The Faraday rotation is proportional to the uniform magnetization, and, hence, the Zeeman energy. The temperature derivative of the uniform magnetization shows the same symmetric logarithmic behavior as that of  $d(\Delta n)/dT$ . Since the spinspin interactions and the uniform magnetization show similar critical behavior, the magnetic specific heat, which is a combination of the two, must also.

One might also be concerned that the optical techniques might be affected by magnetostrictive strains.<sup>22</sup> A comparison with data obtained using heat pulse specificheat techniques on the same crystal could settle this issue. If the same qualitative behavior, namely the peak becoming nearly symmetric and dynamically rounded, is observed with both techniques upon the application of a magnetic field, it can be concluded that the optical techniques do correctly represent the specific-heat critical behavior.

If the optical techniques do show the specific-heat critical behavior, the only difference expected when using  $d(\Delta n)/dT$ , FR, or direct specific-heat measurements is in the dependence of the amplitude of the random-field logarithmic behavior on the applied field.<sup>12</sup> The amplitudes have been shown to scale as  $H^y$ , where y = 0.56 and 0.13 for the FR and  $C_m/R$  measurements, respectively. The amplitude for  $d(\Delta n)/dT$  scales as  $H^y$  with y = 0.13 in the limit of small field, where the Zeeman contribution becomes relatively small. Hence, one would expect the peak amplitude for  $C_m/R$  and  $d(\Delta n)/dT$  to have a very similar field dependence for the small fields, H < 2T, used in this experiment. The prediction y = 0.13 appears to be consistent with birefringence observations.<sup>14</sup>

We chose to perform the specific-heat measurements using a variation of the classical heat pulse technique in which the rise in temperature of the sample is related to a well-determined heat pulse given to the sample.<sup>23</sup> Techniques which are based upon oscillating the heat input (ac specific heat) to the sample or upon continuous heating or cooling of the sample seem not to yield critical behavior characterizations which are as accurate as those obtained using pulse techniques. For example, Ikeda et al.,<sup>24</sup> using an ac technique, obtained non-Ising critical parameters for  $MnF_2$ , whereas Nordblad et al.,<sup>25</sup> observed Ising-like critical behavior for the same system. Moreover, in systems such as the random-field Ising systems, which display severe dynamical effects, techniques that rely upon the rapid response of the sample may yield very misleading results.

With the pulse technique the sample is often very well isolated thermally from its enclosure by hanging it from, for example, a silk thread. The small drift in temperature is then observed and subtracted from the temperature change following the heat pulse. This method had to be modified in our case since a strong magnetic field must be applied along the c axis to generate the random fields. To hold the sample in place in the strong field, we used three pairs of No 46 copper wires spanning about 0.6 cm from the sample to the walls of the sample container. Two of the twisted pairs were used for a four-wire measurement of the carbon thermometer resistance. The third pair was used for the sample heater. The sample temperature was stabilized not by thermal isolation but rather by carefully controlling the thermal shield temperature so that the heat generated in the sample thermometer (approximately 2  $\mu$ W) was precisely equal to the heat conducted to the shield along the wires and to a much smaller degree by

radiation. This was accomplished by using a matched carbon resistance thermometer on the shield in the same orientation and in close proximity to the sample thermometer. The difference between the sample and shield temperature was maintained such that there was a drift of less than 20  $\mu$ K per minute in the sample temperature. This stability is demonstrated in Fig. 1. Heat pulses were applied at a constant power of 400  $\mu$ W, determined using a four-wire measurement, for 5- to 30-s durations. The heat pulse was accurate to at least 0.1%. When a heat pulse is given to the sample, both the sample and shield temperatures rise together to a new equilibrium temperature. The absolute shield temperature is determined using a third carbon thermometer, which has been calibrated. From the change in shield temperature and the sample-shield temperature difference, the actual sample temperature rise is calculated. The error in the measured specific heat introduced by the tracking error of the temperature controller is much less than 1% of the total specific heat. As the sample temperature rises with successive hot pulses, the sample-shield temperature difference must be adjusted appropriately to maintain sample temperature stability after the heat pulse has ended. The adjustment was typically 20  $\mu$ K per 0.01 K change in T. This latter procedure assumes that the sample attains thermal equilibrium on at least the time scale of the sample holder, a point to which we will return.

For the random-field Ising model systems in three dimensions, metastable domain formation results in hysteresis near the phase boundary.<sup>26</sup> It was necessary, therefore, to investigate the behavior of the specific heat upon cooling the sample through the critical region with  $H \neq 0$  [field cooled (FC)]. This was accomplished by adjusting the sample-shield temperature difference,  $\Delta T$ , so that the sample temperature decreased. When the appropriate temperature was reached,  $\Delta T$  was again adjusted to stabilize the sample temperature. The heat pulse



FIG. 1. The drift of the sample container temperature with time. The output voltage of the sample container bridge is shown as a function of time, while the temperature difference between the sample and container is maintained with a second bridge and a temperature controller. The voltage change corresponding to a  $60-\mu K$  temperature change is indicated by offsetting the first bridge, inducing the voltage jumps shown at one-minute intervals in the figure. When the temperature difference is accurately adjusted, the drift in the sample container temperature varies on the order of 20  $\mu K$  per minute or less.

was then applied in the same manner as in the zero-fieldcooled (ZFC) procedure, producing a small rise in the sample temperature which then yielded the specific heat. Although this FC procedure proved to be slightly less accurate than the ZFC one, the FC peak is rounded and less resolution is needed to accurately characterize its shape.

The total specific heat measured for H = 1.5 T, including contributions from the sample thermometer, heater, and varnish, is shown in Fig. 2. The phonon contributions and the combined contributions from all nonmagnetic sources are indicated. The nonmagnetic contributions clearly constitute a significant part of the total specific heat.

Figure 3 shows the results for the magnetic specific heat after ZFC in fields of H = 0, 0.5, 1.0, ad 1.5 T. The specific heat of the wire, thermometer, and GE7031 varnish was independently measured and has been subtracted. The nonmagnetic phonon contributions have been subtracted as well. The phonon subtraction was done by invoking the corresponding states arguments used by Stout and Catalano<sup>27</sup> to determine the phonon contributions for  $FeF_2$  using the data directly obtained for  $ZnF_2$ . We used the approximation that at these low temperatures the phonon contributions to the specific heat of  $Fe_x Zn_{1-x}F_2$  can be obtained from that determined for FeF<sub>2</sub> and ZnF<sub>2</sub> simply by taking into account the squareroot dependence of the specific heat on the mass and using the appropriately weighted average. The assumptions are that the long-wavelength phonons are insensitive to the short-range chemical randomness, and that the elastic bonds are largely insensitive to whatever constituents are nearest neighbors.

For comparison we also show in Fig. 4 the data obtained by Ferreira *et al.*,<sup>28</sup> using the optical linear birefringence technique. In the latter case no adjustment for the phonon contribution is made since  $d(\Delta n)/dT$  is very insensitive to it, which is one of the great advantages of the technique. We have normalized the  $d(\Delta n)/dT$ versus *T* data using the same proportionality found between  $C_m/R$  and  $d(\Delta n)/dT$  for the pure FeF<sub>2</sub> case. This



FIG. 2. The total specific heat, including contributions from the spin system, the lattice, the thermometer, the wires, and the varnish, vs T with an applied field of 1.5 T.



FIG. 3. The magnetic part of the specific heat  $(C_m/R)$  vs T for H=0, 0.5, 1.0, and 1.5 T obtained upon cooling the sample in zero field, raising the field, and warming through the transition (ZFC).

is partly justified by the constancy of the proportionality<sup>29</sup> found for the system  $Mn_xZn_{1-x}F_2$ . Unfortunately, the large single-ion anisotropy in the  $Fe_xZn_{1-x}F_2$  system prevents an accurate determination of the proportionality directly from energy or from entropy<sup>11</sup> considerations as was done for  $MnF_2$ .

The similarity in the shape and magnitude between  $d(\Delta n)/dT$  versus T and  $C_m/R$  versus T is quite encouraging. The asymmetric cusp at  $T_N$  expected<sup>30-33</sup> for the random-exchange Ising model (REIM) is apparent for both sets of data for H=0. As H increases, the peak shapes become symmetric in both cases. The only difference between the two sets of data is in the noncritical background. This is most probably a result of systematic errors in the magnetic specific-heat determina-



FIG. 4.  $d(\Delta n)/dT$  vs T, obtained using the ZFC procedure by Ferreira *et al.*, normalized using the measured proportionality between  $C_m/R$  and  $d(\Delta n)/dT$  for FeF<sub>2</sub>. No correction for the very small nonmagnetic contributions to  $d(\Delta n)/dT$  has been made.

It is important to verify that the FC nonequilibrium behavior of  $d(\Delta n)/dT$  versus T actually reflects the behavior of  $C_m/R$  versus T. We have verified this by using the FC technique described to measure the specific heat of  $Fe_{0.46}Zn_{0.54}F_2$  while cooling in an applied field. The accuracy of this procedure is not as high as that of the ZFC procedure used to obtain the data shown in Fig. 2. We were, nevertheless, able to verify that the peaks are rounded by the applied field, and the data, shown in Fig. 5, compare well with those obtained optically, as shown in Fig. 6. Unfortunately, the measurements in the latter case were made at H = 0.5, 1.0, and 1.9 T, whereas we could only reach a field of H = 1.5 T in our present apparatus. The hysteresis in the specific heat is readily evident in both cases and is of comparable magnitude and shape. We therefore conclude that the hysteresis observed optically is indeed a reliable representation of the nonequilibrium properties of  $C_m/R$  versus T.

As mentioned earlier, the pulse technique is used here with the assumption that the crystal comes into thermal equilibrium within a few seconds of the heat pulse. The drift in temperature after the pulse is eliminated by slightly adjusting the temperature shift  $\Delta T$  between the sample and shield. In Fig. 7 we show  $\Delta T$  versus T determined in this manner for H=0, 1.0, and 1.5 T. For  $H=0, \Delta T$  varies nearly linearly with T and no indication of the transition occurring at T = 36 K is observed. This is expected since  $\Delta T$  should depend only upon the heat generated in the thermometer and the heat conduction via the wires and radiation. The  $H \neq 0$  cases, on the other hand, show marked deviations from linearity in  $\Delta T$ versus T near the transition temperatures. Although these deviations from linearity represent very small changes in  $\Delta T$ , and hence affect the measurements of



FIG. 5.  $C_m/R$  vs T with H = 1.0 and 1.5 T for ZFC and for the procedure in which the sample is cooled through the transition with the field applied (FC). Metastable domains are formed upon FC, and the peak appears rounded.



FIG. 6.  $d(\Delta n)/dT$  vs T for ZFC and FC with H = 1.0 and 1.9 T. The hysteretic behavior in this optical technique corresponds very well with that observed in the  $C_m/R$  measurements.

 $C_m/R$  versus T very little, they do reflect the strong dynamical effects of the RFIM as observed in dilute anti-ferromagnets.

It has been shown with various techniques that extreme critical slowing down takes place close to  $T_c$ . The dynamical effects are manifested as a rounding of the nearly logarithmic divergence in  $C_m/R$  as observed optically at very small |t|. In ac susceptibility measurements<sup>34</sup> it is observed as a frequency-dependent rounding of the uniform susceptibility peak near  $T_c$ . In neutronscattering experiments,<sup>4</sup> nearly logarithmic time dependences are observed in the scattering intensity from order-parameter fluctuations close to  $T_c$  near the antiferromagnetic (100) Bragg point. In the present measurements we find that the crystal takes a long time to equilibrate. The procedure used to stabilize the sample temperature by adjusting  $\Delta T$  after each heat pulse overcompensates in that the natural time dependence of the



FIG. 7. The difference in temperature,  $\Delta T$ , between the sample and its enclosure vs  $T - T_c$  for H = 0, 1.0, and 1.5 T.  $\Delta T$  is set so that the sample temperature drift is minimized. The expected linear behavior is observed for H = 0. For  $H \neq 0$  non-linearity is caused by the critical dynamics near  $T_c$ .

crystal's temperature is masked by making  $\Delta T$  slightly too small. We may, of course, estimate the error introduced in this manner. At  $T_c$ , for H = 1.5 T, the error in  $\Delta T$  is at most 0.0016 K. For measurements of order 100 s, it is estimated that the error in  $C_p/R$  resulting from the small error in  $\Delta T$  is of the order of only 0.1% of the value measured, which is less than errors from other sources.

An early experiment by Wong et al.,<sup>35</sup> on  $Fe_{0.7}Mg_{0.3}Cl_2$  yielded a very different interpretation of the behavior of the RFIM specific heat from that given here. The  $Fe_xMg_{1-x}Cl_2$  system is, like the  $Fe_xZn_{1-x}F_2$  one, a very anisotropic antiferromagnet. However,  $Fe_xMg_{1-x}Cl_2$  differs in that the spins are strongly correlated ferromagnetically within the planes with weaker antiferromagnetic interplaner correlations. Although its d=3 ordering transition should asymptotically be described by the d=3 Ising universality class, one might be concerned since well above the expected percolation limit spin-glass-like behavior is observed. Nevertheless, recent  $H \neq 0$  FR measurements for x=0.7 by Leitão and Kleemann<sup>36</sup> reveal critical behavior, time dependence, and hysteresis very similar to FR results for  $Fe_xZn_{1-x}F_2$ .

The experiments by Wong *et al.*<sup>35</sup> on  $Fe_xMg_{1-x}Cl_2$ were made by lowering and raising the temperature of the crystal at an extremely rapid rate. The entire critical region was scanned in about 1 min and the specific heat was inferred from the rate of temperature change of the sample. Considering the very slow dynamics associated with the RFIM transition, <sup>37,38</sup> one must be concerned with the validity of this procedure. Two points were made based upon the data obtained. The first is that the crossover exponent was determined to be  $\phi = 1.25 \pm 0.10$ . That value of the exponent is disputed by Leitão and Kleemann who, based upon optical studies, give the value  $\phi = 1.41 \pm 0.05$ , a value in excellent agreement with results from the  $Fe_x Zn_{1-x}F_2$  system. In the optical system  $T_c$  was chosen by examining the detailed shapes of the peaks; whereas for the specific-heat experiments,  $T_c$  was simply chosen to be at the maximum of the peak. Concentration gradients have been shown to introduce systematic errors into the determination of  $C_p/R$  when the latter procedure is used. 10

The second point made by Wong *et al.*<sup>35</sup> which warrants comment concerns the interpretation of the peak shapes. It is argued that the H=0 peak is symmetric with the steeper side at higher *T*. This is consistent with the observations of all other investigators. However, for  $H\neq0$  it is argued that the peak is again asymmetric, but now with the steeper side on the low-*T* side. This is contrary to the results of the shapes observed in other experiments on  $Fe_xMg_{1-x}Cl_2$  and  $Fe_xZn_{1-x}F_2$ , in which a symmetric, dynamically rounded peak is observed. <sup>1,12-14,36</sup> Perhaps if the transition was more slowly approached and the large noncritical and rapidly increasing background was subtracted from the data, the peaks would also be observed to be symmetric in the specific-heat measurements. Based upon the work of Leitão and Kleemann, <sup>36</sup> there is no reason to suspect any difference between the RFIM specific-heat critical behavior of  $Fe_2Mg_{1-x}Cl_2$  and that of  $Fe_xZn_{1-x}F_2$ .

The ac specific-heat technique has been used to study random-field effects in the weakly anisotropic  $Mn_x Zn_{1-x}F_2$  system by Ikeda and Kikuta.<sup>39,40</sup> This system has also more recently been examined using the optical linear birefringence technique by Ramos et al.<sup>41</sup> This antiferromagnet is structurally similar to the  $Fe_x Zn_{1-x}F_2$  one, and the exchange fields are nearly equal.<sup>42</sup> The main differences between the two systems are the nature and strength of the anisotropy. The anisotropy in  $Fe_xZn_{1-x}F_2$  is single ion, whereas in  $Mn_xZn_{1-x}F_2$  it is dipolar and an order of magnitude smaller.<sup>43</sup> Pure  $MnF_2$  only shows<sup>20</sup> asymptotic Ising behavior for  $|t| < 10^{-3}$ . With dilution the local dipolar fields should not be precisely along the c axis. Nevertheless, the  $Mn_xZn_{1-x}F_2$  system is interesting since, despite its weak anisotropy, random-field Ising effects very similar to those seen in  $Fe_x Zn_{1-x}F_2$  are observed. The asymmetric zero field cusp and the symmetric peak at  $H \neq 0$ are observed in the  $Mn_xZn_{1-x}F_2$  system, and the crossover exponent has been shown to be  $1.43\pm0.03$ , in excellent agreement with the  $Fe_xZn_{1-x}F_2$  results. It is observed with ac specific-heat techniques that the peaks become very rounded in a way that might at first appear to be different from  $Fe_x Zn_{1-x}F_2$ . However, it was shown in the optical experiments that the concentration gradients and extreme slowing down of critical fluctuations can round the peak substantially. The dynamic rounding in particular should be much more pronounced in the case of the ac specific-heat measurements since the time scale for measurements is 0.024 s as opposed to the 100-s time scale of the optical measurements. Hence, despite conlcusions based on previous specific-heat measurements, all of the dilute antiferromagnets,  $Fe_x Zn_{1-x}F_2$ ,  $Fe_xMg_{1-x}Cl_2$ , and  $Mn_xZn_{1-x}F_2$ , show similar randomfield behavior with an applied magnetic field.

We have seen that the optical linear birefringence does accurately reflect the critical behavior and hysteretic behavior in dilute anisotropic antiferromagnets, with or without a magnetic field applied. Hence, one may feel confident using the optical technique to establish the critical exponents and amplitude ratios for the specific heat in anisotropic systems with quenched disorder. This is extremely convenient since the effects of concentration gradients can be greatly reduced using the birefringence technique. Furthermore, the systematic errors involving the subtraction of the nonmagnetic specific-heat contributions are not encountered in the optical measurements.

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