Letter

Phase shift in AC magnetocaloric effect measurements as an indicator of the order of magnetic phase transitions

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It is shown that the phase shift between an applied weak alternating magnetic field and the magnetocaloric response signal of the magnetic material is drastically sensitive to the order of phase transition. Namely, at the second-order phase transition, the phase shift does not depend on the magnetic field magnitude, while in the first-order phase transition this one depends significantly on the field strength. We have shown that this effect follows from the general critical dynamics theory.

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According to the Ehrenfest's classification, the first derivative of the free energy with respect to some thermodynamic variable exhibits a discontinuity across the first-order phase transitions (FOPT). On the other hand, the second-order phase transitions (SOPT) are continuous in the first derivative and exhibit discontinuity in a second derivative of the free energy [1–4]. The abstract derivative of the Gibbs thermodynamic potential corresponds to an experimentally measured macroscopic parameter of the material. Accordingly, the order of phase transition can be determined by studying temperature or pressure dependences of the appropriate parameters.

The type of phase transition can also be determined by the behavior of the magnetocaloric effect (MCE). Materials with FOPT are characterized by temperature hysteresis of the magnetocaloric effect, one-sided temperature boundaries of the effect independent of the magnetic field (temperature effect limited from below or above), a nonmonotonic dependence of the effect on the magnetic field, high-asymmetry temperature dependence of MCE with respect to transition point, etc. [5–9]. However, determining the phase transition order using MCE data is not always objective, since many of these criteria are not strictly established. Moreover, this approach often requires the use of strong magnetic fields. There is one more (and more stringent) quantitative parameter that allows us to distinguish between first and second order transitions. This is the exponent n in the field dependence of the change in magnetic entropy $\Delta S_M \sim H^n$, which has a maximum $n > 2$ only for thermomagnetic FOPT [10].

Currently, MCE is studied using various methods [5]. The most widely used method for indirectly estimating is evaluating the isothermal entropy change from magnetization or heat capacity data [11,12]. A direct technique for studying MCE is to measure the adiabatic temperature change of a material under a fast change in the applied external magnetic field [5]. Currently, MCE is also being studied using theoretical methods [13]. When used in magnetic cooling technology, the magnetocaloric material will be exposed to a cyclic (alternating) magnetic field; accordingly, several techniques for measuring the MCE in alternating magnetic fields have been developed to carry out relevant studies [14–17]. The essence of the method proposed elsewhere [17] is that the magnetocaloric material is exposed to an alternating magnetic field; in general, such a field can be represented as

$$H = H_0 \sin(\omega t), \tag{1}$$

where H_0 is the amplitude value of the magnetic field and ω is the cyclic frequency of the magnetic field. The temperature response of a material to the applied alternating magnetic field in general can be presented in the form

$$\Delta T_{ad} = \pm \Delta T_0 |\sin(\omega t - \phi)|, \tag{2}$$

where ΔT_0 is the amplitude value of the temperature change and ϕ is the phase shift between the magnetic field and the sample response. Phase shift occurs due to relaxation phenomena occurring during phase transitions. Different materials have different field dependence of the MCE that results in the different temperature response. It follows from this that the temperature response can be functionally different from the magnetic field. For MCE measurements this does not matter; the main condition is that this response occurs at the same frequency, which is the same as the disturbance frequency, and are in a certain way related in phase. The sign of ΔT will be positive in the case of direct and negative in the case of inverse MCE. The modulus of the function $\sin(\omega t - \phi)$ means that we have ΔT of the certain sign, regardless of the direction of the magnetic field (in the absence of anisotropy). In the conventional direct MCE measurement, the measured parameter is only the adiabatic temperature change ΔT_{ad} and, when measured in an alternating field, we have the values of two parameters—ΔT_{ad} and the phase shift ϕ. A phase shift can provide valuable information about the behavior of

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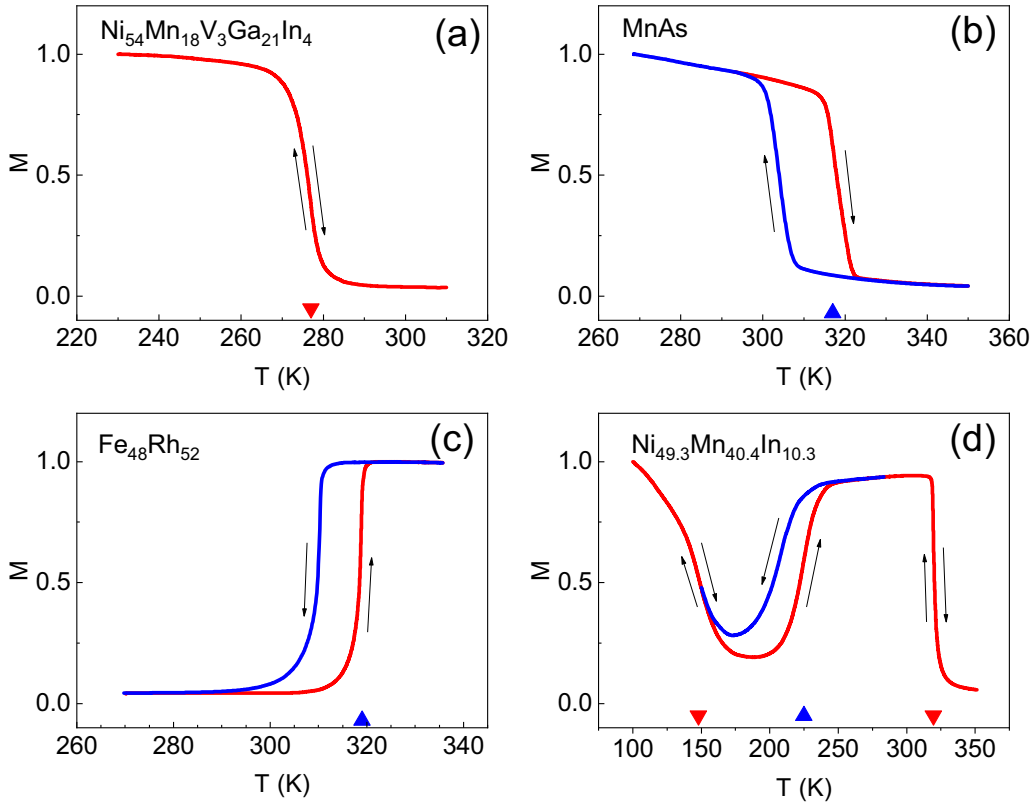

FIG. 1. Normalized temperature dependences of magnetization in a magnetic field of 200 Oe. Blue up triangles mark temperatures of FOPT (at heating run); red down triangles mark SOPT temperatures.

a magnetic system in alternating magnetic fields, including some characteristics of magnetic phase transitions. For this purpose, we studied the magnetocaloric properties of several magnetocaloric materials with phase transitions of different natures, in alternating magnetic fields of low frequency and amplitude.

The measurements were carried out in alternating magnetic fields according to the technique described in [17]. The source of the alternating magnetic field was an electromagnet, through the coil of which an alternating current was passed using a current source with external analog control. To obtain alternating current, the external control input of the current source was supplied with voltage from the built-in AC generator of the lock-in. Due to the inductance of the electromagnet coil, a temperature independent constant phase shift occurs between the magnetic field on the coil and the original control voltage. An additional constant phase shift may also appear between the AC voltage generator and current source. For our studies, such constant phase shifts do not matter, since the initial constant phase shift can always be set equal to zero using the phase shifter of the lock-in.

We studied the magnetocaloric properties of several samples of different classes of magnetic materials: $Ni_{54}Mn_{18}V_3Ga_{21}In_4$, $Ni_{49.3}Mn_{40.4}In_{10.3}$ (Heusler alloys), $Fe_{48}Rh_{52}$, and MnAs. The choice of these materials is conditioned so that the most possible magnetic phase transitions can be observed in these materials. The magnetocaloric properties of these materials in moderate and high magnetic fields were previously studied elsewhere [6,18,19].

Figure 1 shows the normalized temperature dependences of the magnetization of all samples in a magnetic field of 200 Oe. In the $Ni_{54}Mn_{18}V_3Ga_{21}In_4$ Heusler alloy, a second order ferromagnetic-paramagnetic phase transition occurs at the Curie temperature $T_C = 277$ K. In the MnAs compound, a first-order ferromagnetic-paramagnetic phase transition is observed, with the Curie temperature $T_C = 317$ K in the heating run and 304 K in the cooling run. In the $Fe_{48}Rh_{52}$ alloy, a first-order phase transition is observed from a low-temperature antiferromagnetic phase to a high-temperature ferromagnetic one, with Néel temperatures $T_N = 319$ K in the heating run and 310 K in the cooling run. In the $Ni_{49.3}Mn_{40.4}In_{10.3}$ Heusler alloy a series of phase transitions is observed: high-temperature second order ferromagnetic austenite–paramagnetic austenite phase transition with a Curie temperature of 319.5 K and low-temperature magnetostructural martensite–austenite phase transition, with Néel temperatures $T_N = 224$ K in the heating run and 210 K in the cooling one. Further cooling in the martensitic phase transforms the sample into a ferromagnetic state with the Curie temperature $T_C = 149$ K. All magnetostructural phase transitions are accompanied by a sharp change in lattice parameters.

Figure 2 shows the temperature dependences of the adiabatic temperature change ΔT_{ad} of all samples in alternating magnetic fields 200–3000 Oe. For clarity, the figures show the MCE curves only in heating runs. The results obtained reveal direct MCE in the case of FM-PM transitions and an inverse one in the region of the Néel temperature and in the region of

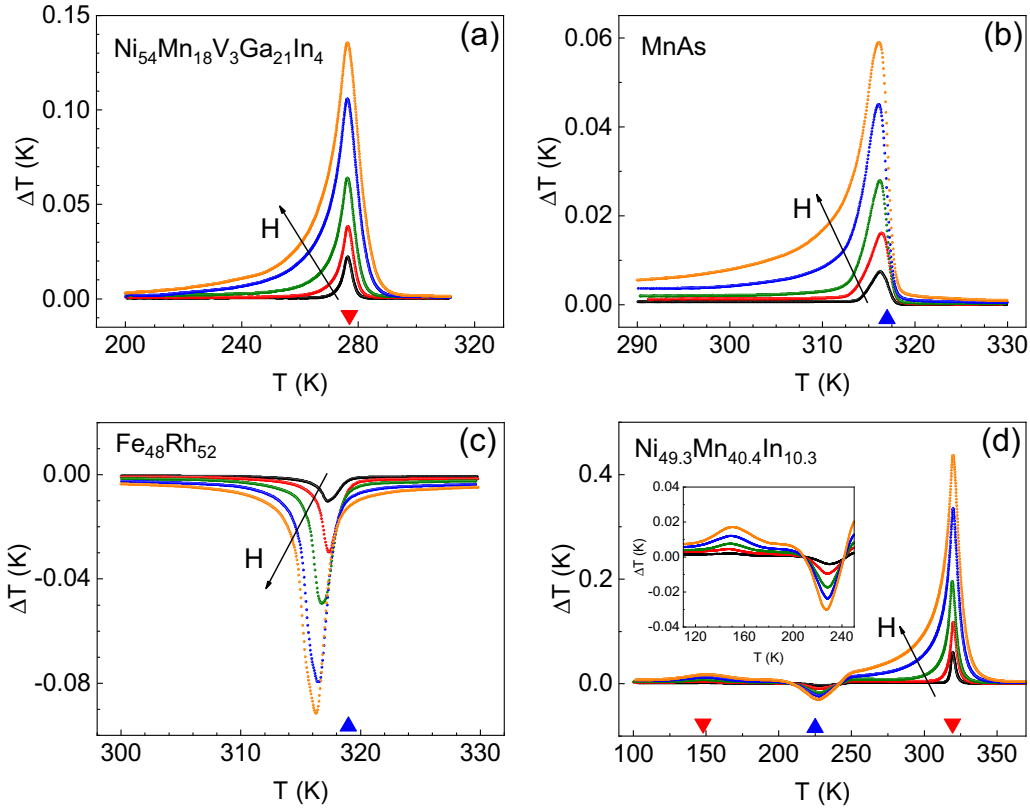

FIG. 2. Temperature dependences of the adiabatic temperature change ΔT_{ad} in alternating magnetic fields with an amplitude from 200, 500, 1000, 2000, and 3000 Oe. Blue up triangles mark temperatures of FOPT; red down triangles mark temperatures of SOPT (according to magnetization).

the magnetostructural phase transition martensite–austenite. All materials studied reveal significant and even giant MCE values under moderate and high fields, but under weak field change the MCE values are small; this is especially typical in the region of the first-order phase transitions. This is a consequence of the fact that these fields are insufficient to induce magnetostructural transitions; accordingly, there is no lattice contribution to the overall MCE.

The temperature dependences of the phase shift are shown in Fig. 3. When measuring the phase shift, the following procedure was carried out. Initially, ΔT_{ad} and the phase shift were measured in a magnetic field of 200 Oe. After that, using the phase shifter of the SR830 lock-in, the phase was set to be zero at the phase transition point. In the case of the $Ni_{49.3}Mn_{40.4}In_{10.3}$ Heusler alloy, where several phase transitions are observed, the zero phase was set at the point of the ferromagnetic-paramagnetic phase transition (319.5 K). Then, with this phase set, measurements of ΔT_{ad} and phase shift were carried out in magnetic fields of 200, 500, 1000, 2000, and 3000 Oe.

The following peculiarities of the temperature dependences of the phase shift are observed in Fig. 3. First, in a narrow region near the SOPT point, the phase shift weakly depends on the magnetic field. With distance from the Curie point, the phase shift changes and strongly depends on the magnetic field. As well, the phase shift in the region of the FOPTs strongly depends on the magnetic field. The points of maximum phase shifts at first-order phase transitions shift in temperature with field, which is not observed at the point of second-order phase transitions. The temperature of the maximum rate of change of magnetization in a weak magnetic field (200 Oe) was taken as the transition temperature.

Note that the some nonzero phase shift occurs for the 150 K SOPT [Fig. 3(d)]. This transition, although it belongs to a second-order phase transition, is significantly subject to a magnetic prehistory. Also, due to the temperature width of the transition, it can be assumed that fluctuations of the order parameter at the transition are weakly developed. These two reasons can be associated with the behavior of the phase shift, which is somewhat different from that expected near the second-order phase transition. Therefore, in further discussions we will not pay attention to the behavior of the phase shift in the region of this transition.

At around 225 K in Fig. 3(d), a significant change in the phase shift at 3.0 kOe compared to 2.0 kOe is evident. This effect is related to the following. In materials with a first-order PT, with increasing magnetic field, the MCE begins to increase sharply as it approaches the critical field. The critical field in this case is the field that causes a reversible magnetostructural phase transition and the critical field depends on temperature. The critical field depends on the temperature hysteresis width and this field must be sufficient to shift the transition temperature in the heating run below the transition temperature in the cooling run in zero field. Anomalies of various physical parameters, including MCE and phase shift in magnetostructural phase transition

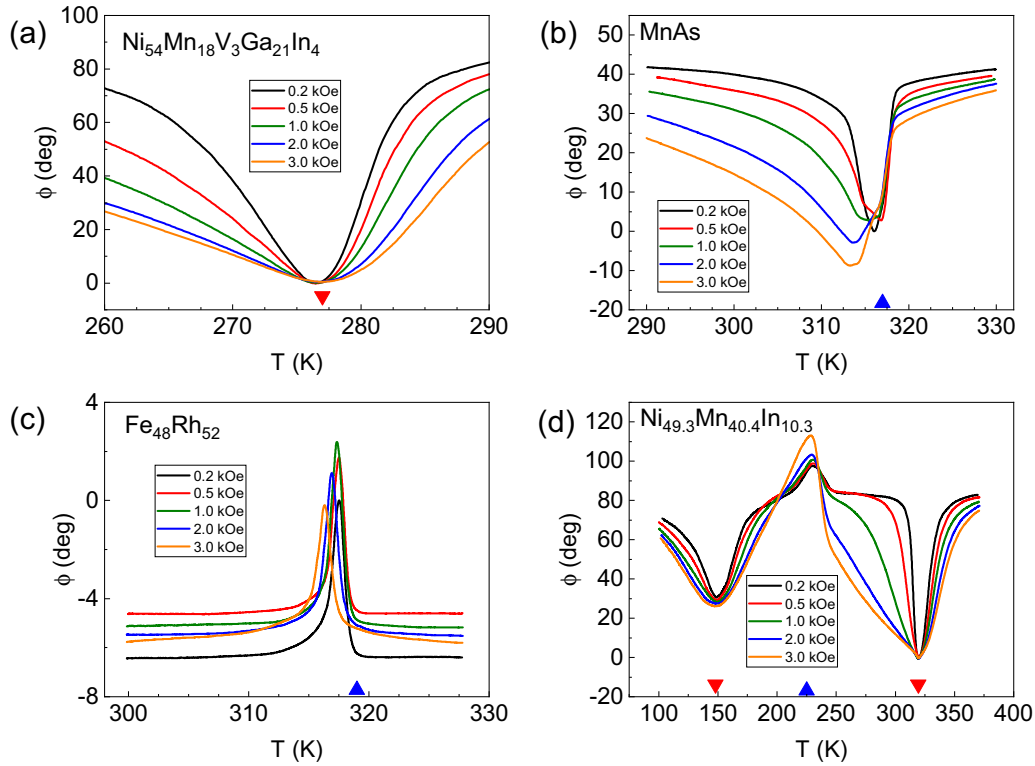

FIG. 3. Temperature dependences of the phase shift in alternating magnetic fields with an amplitude from 200, 500, 1000, 2000, and 3000 Oe. Blue up triangles mark temperatures of FOPT; red down triangles mark temperatures of SOPT (according to magnetization).

materials, will depend nonlinearly on the field. The nature of the martensite–austenite phase transition, which in this composition is observed at temperatures of about 225 K, has been well studied in the literature [20–22].

Measurements taken at other frequencies show a shift in the entire phase curve with a change in the frequency of the magnetic field (see Fig. 4). It is obvious that the shift is primarily due to a change in frequency; at different frequencies, different phases will correspond to the same lag in the response of the system to the external disturbance. In this case, there will be additional phase shifts at different temperatures and, only in the region where the phase does not depend on frequency, the shift will be linear with the change in frequency. As can be seen from Fig. 4, this is exactly the picture observed: at different temperatures this shift is different, since a phase shift due to the different response of the system at different temperatures is added to the regular one. Only in the region of a second-order phase transition is the phase shift independent of frequency.

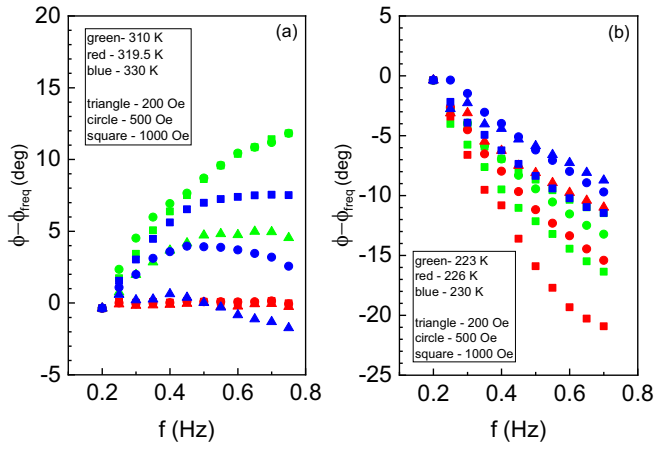

FIG. 4. Dependence of the phase shift on frequency in the $Ni_{49.3}Mn_{40.4}In_{10.3}$ alloy: (a) near a second-order magnetic phase transition; (b) near a first-order phase transition under different magnetic fields. The phase shift is represented as the difference between the phase at various frequencies, magnetic fields, and temperatures and the phase at a second-order phase transition point in a magnetic field change of 200 Oe and a frequency of 0.2 Hz.

Let us now give a qualitative interpretation of the experimental data presented above. The main effect is that the phase shift does not depend on the applied magnetic field at the SOPT. From the point of view of dynamic theory, this means that this shift does not depend on the rate of the field change. Indeed, the sweep rate of the magnetic field changes when the field amplitude changes at a frequency remain constant. In other words, we are dealing with a situation when the response of the system (in our case, this is an adiabatic temperature change) does not lag behind the disturbance. As we demonstrate below, an absence of frequency dispersion at the SOPT point can be described within the framework of the general theory of critical dynamics and the linear response.

We use the simplest fluctuation-dissipative model of phase transition. The main idea of the our qualitative interpretation is based on the fact that fluctuations of the order parameter are static (they do not depend on time) at the SOPT. On the other hand, such fluctuations strongly depend on time, for example, they quickly decay at the FOPT. This conclusion follows from

the theory of critical dynamics [23,24]. Let us introduce the order parameter in the form of a scalar field

$$\Phi(r,t) = \Phi_0 + \Delta\Phi(r,t), \tag{3}$$

where Φ_0 is the regular part of the order parameter, depending only on temperature, and $\Delta\Phi(r,t)$ is the fluctuation, which in general is a function of space and time. The rate of change $\Phi(r,t)$ is proportional to the thermodynamic force for small deviations from equilibrium [23]

$$\partial_t \Phi(r,t) \equiv \partial_t \Delta\Phi(r,t) = -\Gamma \frac{\delta \mathcal{F}}{\delta \Phi^*}, \tag{4}$$

where we introduce free energy $\mathcal{F}\{\Phi\}$ as a functional of the fluctuations field and Γ is a kinetic coefficient that is finite at the phase transition point [23]. From this expression it follows that at the SOPT point we have $\partial_t \Delta\Phi(r,t) = 0$, since $\delta\mathcal{F}/\delta\Phi^* = 0$. Thus the fluctuations of the order parameter are static at the SOPT. Based on this property, we study the frequency dispersion of the thermodynamic quantity, i.e., the phase difference between the disturbance (in the case of MCE this is an alternating magnetic field) and the response.

In general, the response $x(t)$ is related to the perturbation $f(t)$ by the relation (according to linear response theory)

$$x(t) = \int \alpha(t-\tau) f(\tau) d\tau, \tag{5}$$

where $\alpha(t-\tau)$ is the response function (susceptibility). In the case of MCE, the quantity $x(t)$ is the magnetization and the perturbation is the magnetic field. The Fourier transform gives $\overline{x}(\omega) = \overline{\alpha}(\omega)\overline{f}(\omega)$. In the general case $\overline{\alpha}(\omega) = \overline{\alpha}'(\omega) + i\overline{\alpha}''(\omega)$. The presence of a frequency dependence in the real part of the susceptibility $\overline{\alpha}'(\omega)$ means the presence of frequency dispersion in the system, due to which the response function lags compared to the disturbance signal.

The fluctuation-dissipation theorem (FDT) relates the imaginary part of susceptibility with the so-called spectral density of fluctuations $\overline{S}_x(\omega)$ of the thermodynamic quantity $x(t)$

$$\overline{S}_x(\omega) = \hbar\overline{\alpha}''(\omega)\coth\left(\frac{\hbar\omega}{kT}\right). \tag{6}$$

In the low-frequency limit $\hbar\omega \ll kT$ we have

$$\overline{\alpha}''(\omega) \approx \frac{\omega}{2kT}\overline{S}_x(\omega). \tag{7}$$

Now let us take advantage of the fact that at the SOPT point the fluctuations are static. This means that the value of S_x at the transition point does not depend on time: $S_x = \text{const}$ [near SOPT the time evolution of this function is $S_x \sim \exp(-t/t_\xi)$ [24], where $t_\xi = \xi^\kappa$ and ξ is the fluctuation size and κ is some positive number], which gives for their Fourier transform (spectral density)

$$\overline{S}_x(\omega) = \text{const} 2\pi\delta(\omega). \tag{8}$$

Thus, for a SOPT, we obtain

$$\overline{\alpha}''(\omega) \approx \frac{\pi\omega}{kT}\text{const}\delta(\omega). \tag{9}$$

Let us find the real part of the susceptibility using the Kramers-Kronig relations $\overline{\alpha}'(\omega) = \frac{1}{\pi}P\int \frac{\overline{\alpha}''(z)dz}{z-\omega}$. This integral is nonzero only at $\omega = 0$. Thus

$$\overline{\alpha}'(\omega) = \frac{\text{const}}{kT}\delta_{\omega,0}. \tag{10}$$

Thus, at the SOPT point, there is no frequency dispersion. At this point there should be no lag in the phase of the response from the disturbance, i.e., $\phi = 0$. This can be understood from general considerations. Le Chatelier's principle states that any equilibrium system resists changes in its state caused by external forces. The response of the system is defined by the thermodynamic force, which is a variation of the free energy with respect to the order parameter. At the point of the second-order phase transition, this force is zero ($\delta\mathcal{F}/\delta\Phi = 0$) and the system does not resist the external perturbations. As a result, it responds without any delay. At the FOPTs the fluctuations have a finite (very short) lifetime, i.e., not static. This means that the $\overline{\alpha}'(\omega)$ will be a function of frequency. Thus we can conclude that the FOPT point is characterized by the presence of susceptibility dispersion, leading to a phase shift. This shift is measured by a lock-in (see the experimental part of the Letter). If the lock-in input receives an MCE signal in the form $u_i = \Delta T_0 \sin(\omega t + \phi)$, then the output signal turns out to be proportional to the amplitude and also contains a phase factor: $u_f = \Delta T_0 \cos\phi$. From this we get $\varphi = \arccos(\frac{u_f}{\Delta T_0})$. Because the temperature change ΔT_0 during MCE is a nonlinear function of the magnetic field (this can be understood from Maxwell's relations, for instance), then the ϕ will be a function of the magnetic field amplitude. Let us demonstrate this for a simple case when the fluctuation correlator has the form $S_x = \exp(-\gamma|t|)$, where γ is the inverse fluctuation lifetime (for second-order transitions $\gamma = 0$). In this case

$$\overline{\alpha}'(\omega) = \frac{1}{kT}\left[\frac{\gamma^2}{\omega^2+\gamma^2}(1-\delta_{\omega,0}) + \delta_{\omega,0}\right]. \tag{11}$$

Applying the inverse Fourier transform, we obtain

$$\alpha'(t) = \frac{1}{kT}(\pi\gamma\, e^{-\gamma|t|} + \delta_{\gamma,0}). \tag{12}$$

So, we have obtained an expression for the real part of the susceptibility for the case of fluctuations with a finite lifetime. This function describes the time dispersion in the system, i.e., delay of the response signal from the perturbation. Let us show this directly. Let the perturbation be given in the form $f(t) = \cos\omega t$. Then for the response we obtain

$$x(t) \sim \gamma\,\text{Re}\frac{e^{i\omega t} - e^{-\gamma t}}{i\omega + \gamma} + \delta_{\gamma,0}\sin\omega t. \tag{13}$$

The quantity γ is large for the FOPT. Therefore, we can neglect the term $e^{-\gamma t}$. Then we have (at $\gamma \neq 0$)

$$x(t) = \frac{\pi\gamma}{kT}\frac{\gamma\,\cos\omega t + \omega\,\sin\omega t}{\omega^2+\gamma^2}. \tag{14}$$

If we set $\gamma = A\sin\phi$ and $\omega = A\cos\phi$, then

$$x(t) = \frac{\pi\gamma}{kT\sqrt{\omega^2+\gamma^2}}\sin(\omega t + \phi). \tag{15}$$

Thus the finite lifetime of fluctuations leads to a phase shift $\phi = \arctan(\gamma/\omega)$ of the response from the perturbation. From this it can be seen that the phase shift is zero at $\gamma = 0$ (SOPT). In the case of MCE, the measured quantity is the

adiabatic change in temperature ΔT, which is determined by the magnetization M. In this case we have $x = M$ and the perturbation f is the magnetic field H. To estimate the change in temperature, we can use Maxwell's relations, which give (see, for example, Ref. [13])

$$\Delta T = -\int_0^{H_0} \frac{T}{\rho c_p}\left(\frac{\partial M}{\partial T}\right)_H dH. \tag{16}$$

Using Eq. (15) for the magnetization with $f = H\cos\omega t$, we obtain

$$\Delta T = \Delta T_0 \sin(\omega t + \phi), \tag{17}$$

where $\Delta T_0 \approx \frac{1}{2kT}\frac{1}{\rho c_p}\frac{\pi\gamma H_0^2}{\sqrt{\omega^2+\gamma^2}}$. Thus the qualitative picture presented by us of the time dynamics is completely consistent with the experimental data.

From the results obtained, it can be stated that in the immediate vicinity of magnetic phase transitions there is no dependence of the phase shift between the magnetic field and the temperature response of the sample on the magnetic field and the rate of change of the magnetic field. Thus this parameter can be used as an indicator of the order of the phase transition. The absence of a dependence of the phase shift on the field indicates a second-order phase transition and the dependence on the field of the phase shift and the temperature shift of the point of maximum phase shift indicate a first-order phase transition.

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