Melting artificial spin ice

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Abstract. Artificial spin ice arrays of micromagnetic islands are a means of engineering additional energy scales and frustration into magnetic materials. Here we demonstrate a magnetic phase transition in an artificial square spin ice and use the symmetry of the lattice to verify the presence of excitations far below the ordering temperature. We do this by measuring the temperature-dependent magnetization in different principal directions and comparing it with simulations of idealized statistical mechanical models. Our results confirm a dynamical pre-melting of the artificial spin ice structure at a temperature well below the intrinsic ordering temperature of the island material. We thus create a spin ice array that has the real thermal dynamics of artificial spins over an extended temperature range.

Geometric frustration is observed in many physical systems. A textbook example is the frustration of proton interactions in water ice, giving rise to proton disorder, as revealed by the pioneering experimental work of Giauque and Stout \cite{1} and the theoretical interpretation by Pauling \cite{2}. Frustration in antiferromagnets analogous to the ice model was predicted

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theoretically by Anderson in 1956 [3]. Only much later was ice-type disorder observed in magnets, and most surprisingly, this was in ferromagnetic materials that were thus named ‘spin ice’ [4–6]. The spin ice phenomenon relies on dipole–dipole interactions and the concept was later generalized to include arrays of magnetic islands with a spin ice-type geometry, the artificial spin ices [7–13]. Beyond the ice-type systems, the property of frustration is responsible for the occurrence of thermodynamically metastable phases in a variety of systems, including structural (amorphous materials) [14], magnetic (spin glasses) [15] and polymeric systems [16]. In general, the absence of unique ground states [8] and the presence of kinetic constraints [17] make the study of excitations extremely important. These, in combination with the degree of frustration, are the driving forces for disorder in such systems [18]. As an example, an order–disorder transition has been proposed for colloid spin ice model systems with adjustable interactions [19] and dynamical studies have already proven the importance of distortions [20].

The aim of this work is to realize a thermally induced transition in an artificial spin ice array. In previous work on artificial magnetic spin ice, dynamics have been induced by changes in an applied magnetic field rather than by thermal excitations [21–24]. The use of field-cycling protocols has enabled the realization of quasi-degenerate spin states reminiscent of real spin ice [7, 11], as well as ordered states [12]. All these results are obtained at temperatures far below the ordering temperature of the patterned structures. Recently, Morgan et al [13] observed ground states in artificial spin ice for the first time. In their system, thermalization occurs during the growth of the magnetic material, providing a limited time window for real thermal dynamics. These states match the lowest-energy state of a dipolar array which carries no magnetic moment and is made up of the type I vertices shown in figure 1. Here we propose an experimental system where thermal dynamics can be introduced by varying the temperature of the array.

We used δ-doped Pd(Fe) to enable the exploration of such a thermally driven order–disorder transition of the artificial spin ice structure. The Curie temperature of δ-doped Pd(Fe) films can be adjusted by the variation of the iron (Fe) layer thickness in the palladium (Pd) [25, 26]. A 1.2 monolayer thick Fe layer was embedded between 10 monolayers of Pd, which resulted in a Curie temperature of $T_C = 230$ K. The film was patterned by electron beam lithography and Ar ion milling into an artificial square spin ice array, of $750 \times 250$ nm$^2$ islands with a period of 1000 nm, extending over a $1.5 \times 1.5$ mm$^2$ area (figure 1). The total magnetic moment for one island was determined to be $M_0 = 1.1 \times 10^{-16}$ A m$^2$ at 5 K. As a reference, we determined the temperature-dependent magnetization of a reference patch, patterned on the same substrate as the spin ice arrays. The patterning process did not influence the magnetization of the reference region, which was found to be identical to an unprocessed twin sample.

Magnetic hysteresis loops were recorded using the magneto-optical Kerr effect (MOKE) at temperatures from 5 to 300 K [26] (see the inset of figure 2), from which we extract the zero-field remanent magnetization at each temperature. For the patterned array, the field was applied in two directions: (i) parallel to the [10] and (ii) parallel to the [11] directions; see figure 1. The zero-field states obtained from this protocol are magnetic. The normalized remanent magnetization, $M_{\text{rem}}$, of the magnetic island array is compared to the continuous film in figure 2. The three datasets follow a similar trend up to 100 K, but at higher temperatures the magnetic island array data cross over to a regime in which the magnetization is strongly reduced, approaching zero at about 200 K, while that of the continuous film falls to zero at $T_C = 230$ K. The existence of a finite remanent magnetization strongly suggests that such a field cycling effectively removes vertices of type I, at least at lower temperatures. Our islands contain $n \sim 10^7$ microscopic moments and hence couple strongly to an external field, making type I
vertices highly unfavourable in even small applied fields. Hence, we expect the low-energy states to be made up of the type II vertices shown in figure 1.

In order to discuss these magnetic structures in detail and describe the disordering process as the temperature is raised, we distinguish between the microscopic magnetic moments within the islands (micro-spins, \( \vec{\sigma} \)) and the island macro-spins, composed of all \( n \) moments within an island (\( \vec{S} = \sum_{i=1}^{n} \vec{\sigma}_i / |\sum_{i=1}^{n} \vec{\sigma}_i| \)). The coupling between the micro-spins is due to the exchange interaction, with a coupling constant \( J \) related to the \( T_C \) of the magnetic material, while the coupling between the islands is magnetostatic. These interactions are long-ranged but the characteristic energy scale can be defined through the interaction that between macro-spins, \( \vec{S} \), on neighbouring islands. The most relevant interaction is between the nearest neighbours, coupling spins along rows or columns of the array. We define this coupling as \( K = K_0 m^2 \), where \( K_0 \) is of order \( n^2 \) and \( m^2 = (\frac{1}{n} \sum_{i=1}^{n} \vec{\sigma}_i)^2 \).

Introducing the thermally averaged quantity \( m^2 \) implies that thermal fluctuations of the macro- and micro-degrees of freedom are decoupled, allowing the two sets to order at different temperatures. In this case, the temperature dependence of the effective moment of the islands can be captured by using \( m^2 = (1 - T / T_C)^{2\beta} \), with \( T_C = 230 \text{ K} \) and \( \beta = 1/3 \), which matches the...
Figure 2. Normalized remanent magnetization versus temperature curves after a magnetic field has been applied parallel to the [10] and [11] directions, compared with the remanent magnetization of the continuous film used for patterning the arrays. The collapse of the array magnetization at a temperature well below the Curie temperature of the material ($T_C = 230$ K) is consistent with the appearance of thermally induced dynamics of the macro-spins that constitute the array. The inset shows representative normalized magnetic hysteresis loops for a magnetic field applied parallel to the [10] and [11] directions, at a temperature of 12 K.

temperature variation of the remanent magnetization of the continuous film shown in figure 2 to a good enough approximation for our purposes. This decoupling assumption could break down for smaller units, in which case the two ordering processes could be renormalized into a single transition, but it should be valid for the islands studied here, consisting of $\sim 10^7$ micro-spins. Ordering of the macro-spins due to magnetostatic interactions should therefore occur at a temperature $T_M \leq T_C$, as even in the limit $K_0 \gg J$, the term $m^2$ ensures that $K < J$ at $T_C$. Thus, we expect disordering of the macro-spins at $T_M$ and a range of temperatures $T_M < T < T_C$, in which the islands are decoupled.

The macro-spin $\vec{S}$ is confined to lie along the long axis of the island through the magnetic shape anisotropy $K_S$, which minimizes the collective dipolar energy of an island for the moment in one of the two configurations with Ising symmetry. The energy barrier for reversing the direction of $\vec{S}$ can be approximated by $E_r(T) = K_S V$, with $K_S(T) = \mu_0 D (M_0 m)^2 / 2$, and is temperature-dependent through $m^2$. Here $\mu_0$ is the permeability of free space, $D$ is the demagnetization factor of the island, $V$ its volume and $k_B$ is Boltzmann’s constant. The active magnetic material is assumed to have a thickness of 1 nm \[27\] when estimating $D$. Setting $E_t = E_0 m^2 T$ and using our approximate expression for $m^2$, we find that $E_0/k_B \approx 1400$ K and $E_t/(k_B T) = 1$ at 214 K. Note that the barrier height can be modified by the dipolar fields from neighbouring islands \[13, 28\], so that, in principle, it depends on the local environment as well as $E_t$, but we have not taken these effects into account here. For inter-island magnetic fluctuations to occur, the energy barrier to be crossed for an Ising-like spin flip must be thermally accessible. The macro-spin reversal time scale can be assumed to follow a Néel–Brown law $\tau = \tau_0 e^{E_t/(k_B T)}$,
so that thermal fluctuations for macro-spins should become relevant in a temperature regime below $T_C$.

The possible vertex configurations for artificial square spin ice are summarized in figure 1(d). States constructed from type I and II vertices form the topologically constrained manifold that makes spin ice materials so interesting. In three-dimensional (3D) spin ice, states of the manifold are quasi-degenerate, leading to a Coulomb liquid phase. Although the dynamics of the Coulomb liquid are slow, the system is not completely frozen as excitations create pairs of topological defects that are deconfined and hence free to propagate through the system. Explicitly the deconfinement is a result of the separation of energy scales between that for the band width of the constrained manifold and that for breaking the constraints, and it allows for significant dynamics well below the latter temperature scale [29]. Square ice does not have the same narrow band of low-energy states, as the long-range part of the dipolar interaction is less well screened than in the 3D counterpart [30]. In this case, topological defects out of a constrained state should be confined by local fields, at least for finite-size systems [31], so that one can expect the macro-spins to be essentially frozen into a single constrained state at lower temperature. Hence, our protocol for preparing a remnant magnetized state should stabilize topologically constrained states made up of the ensemble of states shown in figure 1(d) only, up to the temperature at which thermal fluctuations of the macro-spins force the system into the paramagnetic regime. This is consistent with the fact that a transition to a type I ordered state is not observed. Rather, thermal excitations take the system directly from the frozen constrained state into the paramagnetic region of the macro-spin phase diagram. We propose therefore that the simplest model that captures the essential physics observed in this multi-scale problem is a 16-vertex model, in which the type I states are raised in energy, leaving a four-vertex (type II) manifold of ground states. This phenomenology is tested in the rest of this paper.

Within this scenario the application of a saturation field along the [10] direction would result in a partially lifted degeneracy at lower temperature, in favour of the set of states shown in figure 1(b). Applying the field along the [11] direction, followed by a return to remanence, would lead to the selection of the fully ordered state illustrated in figure 1(c). To capture the temperature dependence of the excitations, we can therefore compare the ratio of the remanent to the saturation magnetization, $M_{\text{rem}}/M_{\text{sat}}$, for these two directions. The order parameter of the islands, $m^2$, cancels out, leaving the ordering behaviour of the macro-spins $\vec{S}$ only. The resulting ratio should therefore be 1/2 for the [10] direction and $1/\sqrt{2}$ for the [11] direction. As seen in figure 3, the measured ratio falls on the prediction up to 125 K, illustrating the collective behaviour of the macro-spins, with the single magnetic domain structure illustrated in figures 1(b) and (c). Remarkably, the four-vertex macro-spin ground states remain in place up to temperatures where thermal fluctuations of the micro-spins have reduced the squared order parameter of the islands, $m^2$, to less than one-half of its zero temperature value. In the case of the [11] field, the data fall below the $1/\sqrt{2}$ value at 125 K, whereas that for the [10] field stays on the line up to a slightly higher temperature. The magnetization, for the field applied in both directions, finally collapses as the ordering temperature of the material, $T_C = 230$ K, is approached. The fall of the magnetization ratio falls below the upper limit set by the manifold of constrained states, which indicates that thermal excitations of the macro-spins do occur. These excitations will lead to vertex configurations other than type II, including type I, as the constraints are lost and the array becomes paramagnetic. Our simple model offers an incorrect Boltzmann weight for the type I vertex, but it should maintain the correct qualitative behaviour in the paramagnetic region.

New Journal of Physics 14 (2012) 035009 (http://www.njp.org/)
Figure 3. The ratio of the remanent to the saturation magnetization for the array. At lower temperatures the ratios are 0.5 and 0.707 for the [10] and [11] directions. This is consistent with an ordering of type II A + B and type III A + B vertices as defined in figure 1(d). Above 125 K, the macro-spin order diminishes (‘pre-melts’) which is well below the Curie temperature of the material ($T_C = 230$ K). The solid black lines are numerical simulations for the 16-vertex model, described in the text, that account for the thermally induced dynamics of the macro-spins.

The model is implemented by Monte Carlo simulations on a system with $N = 45000$ macro-spins. A 16-vertex ice model of $N$ Ising spins $\mathbf{S}$ was used, arranged on a square lattice. To mimic the experimental conditions the energy of type I vertices was raised above those of type II, and for simplicity, we set them equal in energy to the type IV vertices, giving a single energy scale, $\epsilon_{III} - \epsilon_{II} = K$, $\epsilon_{IV} - \epsilon_{III} = K$. This model is equivalent to a grill of interpenetrating Ising spin chains coupled uniquely through the first neighbours along the chains (see figure 1(d)). The ground state is thus an $\Omega = 2^{2L}$ degenerate manifold of type II states with ferromagnetic ordering along chains (rows) of length $L = \sqrt{N/2}$ but with disorder among the chains. The parameter $K$ defines the effective coupling of macro-spins within a chain and its temperature dependence is obtained by scaling with $m^2$. As expected, there is no phase transition to an ordered state at finite temperature and in zero field the system remains disordered to $T = 0$. However, the model does have remarkable finite-size effects related to the zero-temperature critical point of the 1D Ising model [32]5. This effect is illustrated in figure 4, which shows the temperature evolution of the magnetic moment for macro-spins on a single row (or column) of spins, $\tilde{M}_{\text{sim}} = |\frac{1}{L} \sum_{i=1}^{L} \mathbf{S}_i|$ for different values of $K_0/T_C$. In each case, the data are compared with $\tilde{M}_{\text{ana}}$, calculated for a 1D Ising chain of $L$ macro-spins (see footnote 5), with a coupling constant $K = K_0 m^2$, which is exact to leading order in $1/L$. The Monte Carlo

5 The finite-size Ising chain is often discussed in passing as a prelude to the results in the thermodynamic limit. For two recent papers that focus on the model in its own right, see [33]. The analytical expression is $\langle \tilde{M}_{\text{ana}}(T) \rangle = \left( \frac{1}{2} \exp \left( \frac{2K}{T} \right) \frac{1 - \tanh \left( \frac{K}{T} \right)}{1 + \tanh \left( \frac{K}{T} \right)} \right)^{1/2}$. New Journal of Physics 14 (2012) 035009 (http://www.njp.org/).
Figure 4. Illustration of the thermal collapse of chain-like correlations between macro-spins. The array is approximated by an assembly of finite-length Ising chains that order below a temperature, $T_M$. The points are numerical data from Monte Carlo simulations and the lines the analytical solution for the magnetization of an Ising spin chain of length $L = 150$. The extent of this ‘finite-size decoupling’ regime can be varied by changing the coupling strength. Inset: the relation between the spin coupling constant and the pre-melting temperature $T_M$, extracted from the Monte Carlo simulations. The finite-size decoupling regime occurs for any value of the coupling $K_0$. Results are excellently reproduced by the analytic expression for all values of $K_0/T_C$, with small differences coming from corrections to scaling [34]. The chain moment approaches saturation below a crossover temperature, which we can interpret as $T_M$ when modelling the array. It scales logarithmically to zero with $L$; thus for any reasonable system size, there is a finite region of temperatures for which each chain of the vertex model is ordered.

This finite-size chain ordering is at the origin of the observed pre-melting as it ensures that the field hysteresis protocol, leading to $M_{\text{rem}}$, will order the chains giving a nonzero total moment for the vertex model below this crossover temperature scale. For a field along the [10] direction, the chains parallel to the field will be ordered but those perpendicular will be free, whereas for the field along [11] all chains should be ordered, as in the experiment. This field-induced ordering is confirmed in figure 3, where we compare calculated and experimental remanents to saturation magnetization ratio values. The numerical data were taken from simulations using two fitted parameters: $K_0 \approx 530$ K, corresponding to $K_0/T_C = 2.3$ (see figure 4), and a remanent applied field $B_0 = 1.8 \times 10^{-5}$ T. The simulation and experimental data are in good agreement up to about 200 K, above which the experimental data fall more rapidly towards zero. This could arise due to the development of magnetic fluctuations perpendicular to the Ising spin axis or thermal macro-spin flips becoming more favourable. These are not taken into account numerically.
The best fit value of $K_0$ compares favourably with calculated values: the direct interaction of the island magnetic poles (treated as point charges) yields $K_0 \approx 530$ K. In addition, this value of $K_0$ yields a crossover temperature, $T_M \sim 160$ K, which is compatible with the experimental data. The general agreement between experiment and simulation and the closeness of the fitted parameters to expected values therefore illustrates that the 16-vertex model proposed here does indeed provide a useful qualitative description of the experimental data.

In both experiment and simulation, the crossover from the ordered spin ice regime to the pre-melting regime for the [11] field direction begins at a temperature lower than that for the [10] direction. Pre-melting begins as vertex excitations disobeying the ice rules occur, breaking up the correlated chains of macro-spins. In such an excitation, a vertex of type II becomes a vertex of type III through the flipping of the direction of a macro-spin. In the case of remanent magnetization along the [10] direction, there are vertex excitations that preserve the magnetic moment, for example type II $A + B \rightarrow$ type III $A + B$, as illustrated in figure 1(d). However, for $M_{em}$ along [11] all excitations are moment reducing. This suggests that the magnetization ratio will remain near the upper bound in the first stages of pre-melting for ordering along the [10] direction, while the ratio will be reduced from this bound as soon as defects appear, for ordering along the [11] direction. These defects carry accumulations of effective magnetic charge and, in the case of 3D spin ice, have been successfully described using the language of emergent magnetic monopoles [29, 35, 36]. In this case, the term ‘monopole’ should be used with great caution, as long-ranged interactions are less well screened for fourfold symmetry in a plane than in spin ice. Here we have demonstrated the possibility of measuring the order-to-disorder transition in artificial magnetic spin ice. The possibility of observing emergent monopoles is therefore conceivable, following the general approach that we describe in the design of spin ice arrays.

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New Journal of Physics 14 (2012) 035009 (http://www.njp.org/)
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