

Magnetic texture control in ion-implanted metamaterials

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We study experimentally the impact of the additive fabrication method on the magnetic properties of Fe⁺-implanted Pd square artificial spin ice lattices. Our findings show that the lattices exhibit a higher ordering temperature than their continuous film counterparts. This behavior is attributed to the additive fabrication process, which induces an inhomogeneous Fe concentration within the lattice building blocks. Moreover, the implantation process creates a magnetic depth profile, enabling temperature-dependent tunability of the magnetic thickness. These additional internal degrees of freedom broaden the design possibilities for magnetic metamaterials, allowing precise fine tuning of their static and dynamic properties to achieve complex and customizable behaviors.

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I. INTRODUCTION

Complex systems are characterized by interactions among their components that lead to the spontaneous emergence of collective properties. In condensed matter physics, magnetic metamaterials have become an exemplary platform for investigating such complexity. These systems consist of arrays of ferromagnetic elements of sub- μm size that interact magnetostatically [1,2], with these interactions giving rise to emergent phenomena, including collective magnetic order [3–8] and dynamics [9–15]. The building blocks of magnetic metamaterials, mesoscopic ferromagnetic elements, are often approximated as pointlike magnetic dipoles or artificial magnetic atoms. While these analogies are useful, they fall short in capturing phenomena such as thermal fluctuations and phase transitions. Recent studies have provided a more nuanced analysis of the intrinsic magnetic properties and dynamics of these elements [16–20]. In a notable example, the initially simulated internal degrees of freedom, leading to excess entropy and modification of dynamics as reported by Gliga *et al.* [16] were later experimentally verified, with their temperature-dependent behavior characterized in detail by Skovdal *et al.* [21]. These systems bridge theoretical models of complexity with experimental realizations, enabling studies of the interplay between competing interactions [22–24],

frustration, and exotic magnetic phases [25,26]. In addition to their fundamental significance, the complexity in magnetic metamaterials has fueled technological advancements in fields such as spintronics [27], data storage [28], and unconventional computation [29,30].

In this study, we investigate magnetic metamaterials as a platform for gaining deeper insights into complexity in physics. Instead of removing material to form the array, we employ electron-beam lithography and ion implantation to directly embed magnetic functionality into a host medium, thereby defining the metamaterial structure [31]. This approach produces ferromagnetic elements that inherently exhibit compositional and magnetic inhomogeneities. Our investigation examines the impact of these inhomogeneities on the ordering temperature of extended arrays and the internal magnetic textures of individual elements. The study focuses on square artificial spin ice (ASI) lattices composed of elongated, stadium-shaped nanoelements—referred to as mesospins. While ASIs have been widely studied for emergent order [3,5] and thermally induced dynamics [9,21,32] in mesospins made from isotropic and homogeneous magnetic materials, our work provides a direct comparison with systems exhibiting intrinsic mesospin inhomogeneities. This comparison not only enhances our understanding of their fundamental behavior but also underscores their potential for practical applications [16], as it enables the deliberate design of both the static and dynamic properties of magnetic metamaterials.

II. MATERIALS AND METHODS

A. Sample fabrication

Two samples were studied, having different Fe concentrations. Both were fabricated by implanting 30 keV ⁵⁶Fe⁺

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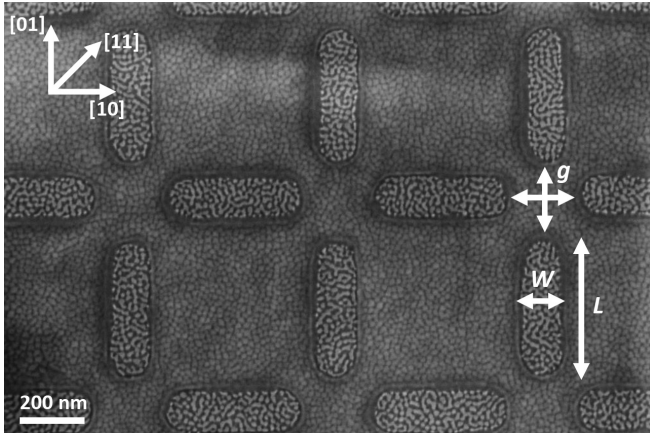


FIG. 1. Scanning electron microscopy (SEM) image of implanted square artificial spin ice lattice (ASI) with nominal fluence 2.7×10^{16} ions/cm².

into a Pd film through a patterned Cr mask. The Pd film, deposited using DC magnetron sputtering on a MgO substrate, consisted of a 60 nm Pd layer with a 5 nm V adhesion layer and a 6 nm Cr capping layer. This additive fabrication process yields flat $\text{Fe}_x\text{Pd}_{100-x}$ (where x stands for at.%) ferromagnetic structures, where Fe has a concentration-depth profile. A detailed description about the fabrication process is provided in Vantaraki *et al.* [31]. One sample was implanted with a nominal fluence of 2.7×10^{16} ions/cm², while the other was implanted with 3×10^{16} ions/cm². Higher fluences yield higher Fe concentration [33]. In both samples, the stadium-shaped elements have a length of $L = 470$ nm and width $W = 170$ nm, while the edge-to-edge gap between the elements is $g = 170$ nm (see Fig. 1).

B. Monte Carlo simulations

The Fe distribution within the implanted samples was investigated by performing stopping and range of ions in matter (SRIM) simulations [34]. To imitate the experimental conditions, the simulations were performed for 30 keV Fe^+ ions implanted in a 600 Å Pd film through a 60 Å Cr capping layer. The angle of ion incidence was set to zero degrees (normal to the surface).

C. Hysteresis protocols

The temperature-dependent hysteretic behavior of implanted ASIs was studied using a longitudinal magneto-optical Kerr effect (L-MOKE) setup and a vibrating sample magnetometry (VSM) system. In the L-MOKE setup, a p -polarized incident laser beam with a wavelength of 659 nm was modulated by a Faraday cell and subsequently reflected by the sample. The reflected beam was passed through an analyzer (with extinction ratio of 10^5) and then measured using a biased Si photodiode. The detector was connected with a pre-amplifier and a lock-in amplifier. For a single hysteresis loop measurement, the external magnetic field was cycled from $+H_{\text{max}}$ to $-H_{\text{max}}$ and then back to $+H_{\text{max}}$, where $H_{\text{max}} = 50$ mT. To increase the signal-to-noise ratio, 50 hysteresis loops were recorded at each temperature, and the final

hysteresis loop was obtained by averaging these measurements. The external magnetic field was applied as a sinusoidal waveform $H = H_0 \sin(2\pi ft)$, with $f = 0.1$ Hz. The field was applied along the [11] and [01] directions of the ASI lattice, as indicated in Fig. 1. The 659 nm laser beam achieves a penetration depth of approximately 12 nm in the Pd film [35], detecting the magnetization response from the surface of the sample. The cryostat in the L-MOKE system operates up to 400 K.

In addition to MOKE measurements, magnetic hysteresis loops were recorded by vibrating sample magnetometry (VSM) with a 2 T Lakeshore 8600 magnetometer, equipped with a single-stage variable temperature unit that can reach a temperature of 950 K. This technique records the magnetic response from the entire sample volume.

D. Magnetic imaging

The magnetic state of the implanted mesospins was directly imaged using photoemission electron microscopy, employing x-ray magnetic circular dichroism (PEEM-XMCD). These experiments were conducted at the CIRCE (bl24) beamline of the ALBA synchrotron [36]. The images were acquired at room temperature with an x-ray photon energy tuned to the Fe L_3 edge. The lattices were imaged at the remanent state after applying a high enough field to saturate the array [37].

E. Micromagnetic simulations

The magnetic texture of the inhomogeneous mesospins was explored by performing micromagnetic simulations using MuMax³ [38]. The simulations were performed with stadium-shaped elements of dimensions 470×170 nm² arranged on a square lattice. The edge-to-edge gap between the elements was $g = 170$ nm. To mimic the experimental conditions, the elements were considered to be composed of Fe with a thickness of 3.18 nm. This thickness corresponds to a continuous Fe layer implanted with a nominal fluence of 2.7×10^{16} ions/cm² [35]. It is important to note that this is a rough approximation, as the fabrication process yields $\text{Fe}_x\text{Pd}_{100-x}$ (where x stands for at.%) elements. Consequently, the values for saturation magnetization and exchange stiffness constant in simulations deviate from those of the actual material. The elements were discretized into grid cells with cell size smaller than the exchange length and equal to 2.5 nm.

III. RESULTS AND DISCUSSION

We begin by examining the distribution of the implanted Fe on the Pd film. To do so, we perform Monte Carlo simulations using the SRIM software. The simulations provide information about the Fe^+ distribution both in depth and laterally from a single implantation point, assuming an amorphous Pd target. As shown in Figs. 2(a) and 2(b), the simulation results show that implanted Fe exhibits a concentration depth profile [Fig. 2(a)] and a concentration lateral profile [Fig. 2(b)]. Both depth and lateral profiles follow approximately a Gaussian distribution. In the concentration depth profile, the peak of the distribution corresponds to the average depth at which the ions come to rest, while in the concentration lateral profile, the peak indicates the surface spot that receives the highest

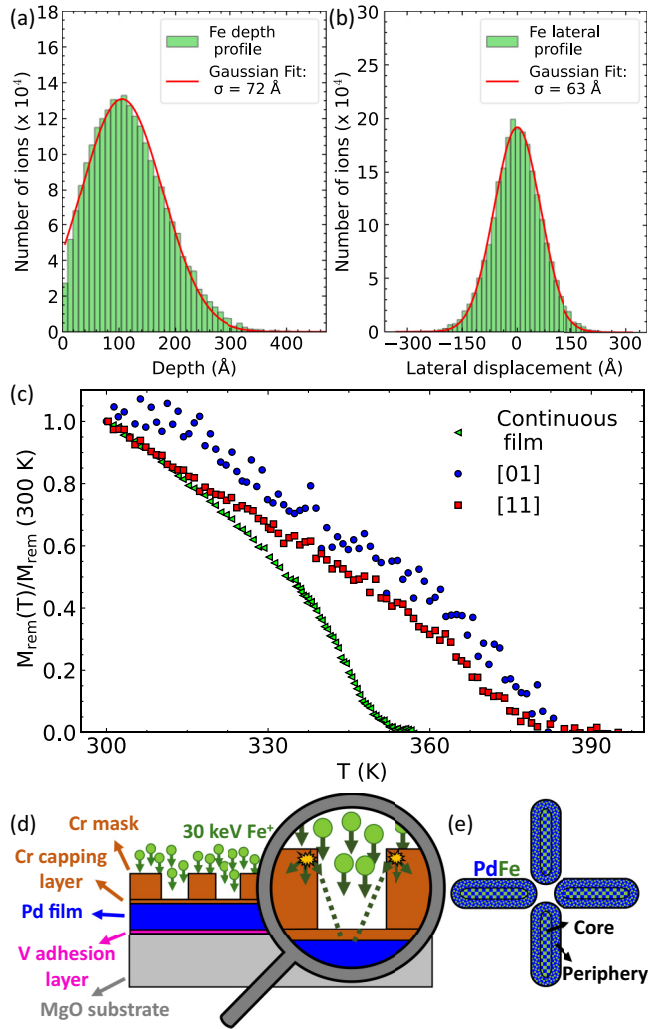


FIG. 2. (a), (b) The simulated (a) depth and (b) lateral profile of Fe as a consequence of the ions straggling along the beam and perpendicular to it. (c) Normalized remanent magnetization M_{rem} as a function of temperature T for the ASI and its reference continuous film implanted with 30 keV Fe of nominal fluence 2.7×10^{16} ions/cm² measured with L-MOKE. (d) Schematic representation of the side view of the patterned film and the scattering events during Fe implantation. Because of the presence of the mask, the implanted mesospins have higher Fe concentration compared to the reference continuous film. (e) The Fe concentration gradually decreases toward the edges of the elements, resulting in a higher concentration at their cores compared to the peripheries.

ion fluence. For both profiles, the standard deviation σ of the Gaussian distribution represents the extent of the ion spread around the peak, indicating the depth variation ($\sigma = 72$ Å) in the depth profile and the lateral spread or straggling ($\sigma = 63$ Å) in the lateral profile. The concentration depth profile has been investigated previously in continuous films [31,33]. In contrast, the lateral implantation profile is often neglected in continuous films, as the lateral motion of ions creates a broader distribution due to the system's extension. However, the concentration lateral profile can no longer be neglected in the lattices due to the clear boundary between the implanted and unimplanted regions [39].

The concentration profiles are difficult to be probed in lattices with nanometer elements; however, the amount of implanted Fe can be investigated using the ordering temperature as the probe. The ordering temperature is defined as the temperature at which the spontaneous magnetization vanishes. To a good approximation, this temperature coincides with the temperature where the remanent magnetization disappears [40]. As such, we examine the temperature-dependent behavior of the remanent magnetization M_{rem} from the MOKE measurements for an implanted ASI array and its reference continuous film that was prepared and implanted next to the lattice, with a nominal fluence of 2.7×10^{16} ions/cm². As shown in Fig. 2(c), the remanent magnetization vanishes at higher temperatures for the implanted ASI compared to the reference implanted film, reflecting a higher ordering temperature. This trend is opposite to the one previously observed in conventional ASI lattices [9]. It is worth noting that the collapse of the magnetic order occurs at a lower temperature for the continuous film compared to the lattice even when considering the saturation magnetization [35]. This observation highlights a higher Fe concentration for the patterned film. As shown schematically in Fig. 2(d), the additional Fe concentration in ASI is attributed to Fe ions that impinge the mask near the edges. Due to multiple scattering events, some ions manage to scatter back out of the mask and then become embedded in the Pd film at an angle. As a consequence, the elements are enriched with additional Fe dopants, which are not implanted in the continuous reference film, because of the presence of the mask. Based on the increase in critical temperature, we estimate that the ASI lattice has approximately 1–3% higher Fe concentration compared to the continuous film. This estimation was derived from the relationship between the Fe concentration and the ordering temperature found in Refs. [33,41,42]. Nevertheless, the additional dopants cannot prevent the lateral spread of ions at the edges of the elements. As a consequence, the Fe concentration gradually decreases toward the edges of the elements [43]. This effect results in a higher Fe concentration in the core area of the elements compared to their periphery [Fig. 2(e)]. Similar mask proximity effects have been reported for CMOS devices [44–46].

To directly illustrate whether the inhomogeneities influence the magnetic state of mesospins, we image the ASI array in the remanent state by performing XMCD-PEEM experiments, after applying an external in-plane field to saturate the array. The field was applied along the diagonal of the elements, corresponding to the [11] direction [Fig. 3(a), left], and parallel to the long axis of half of the elements, corresponding to the [01] direction [Fig. 3(b), left]. Representative XMCD-PEEM images are shown in Figs. 3(c) and 3(d), when the field was applied along the [11] and [01] directions, respectively. In both images, the elements are displayed in a uniform white or black contrast. The white and black colors indicate a magnetization component parallel and antiparallel to the x-ray beam, respectively. Consequently, we conclude that the ASI array has obtained a ferromagnetic (or type II) remanent state when the field is applied along the [11] direction. Similarly, applying a field along the [01] direction and then returning to the remanent state results in a full ferromagnetic order for the elements parallel to the [01] direction. For the mesospins

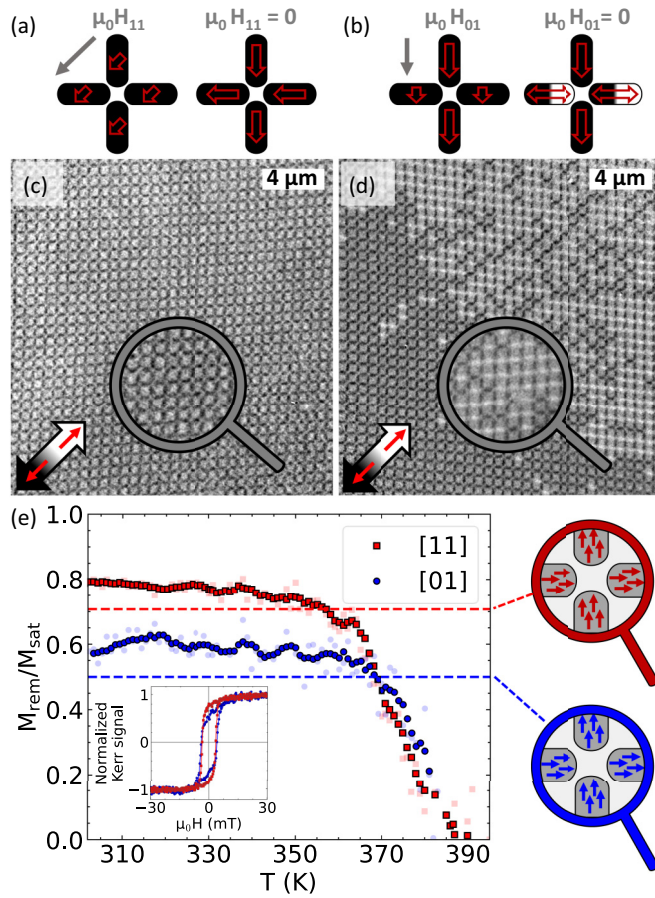


FIG. 3. (a), (b) Schematic representation of the magnetization orientation for an ASI array at the saturation (left) and remanence state (right) for a field applied along the (a) [11] and (b) [01] directions. (c), (d) PEEM-XMCD images for implanted ASI lattices of nominal fluence 2.7×10^{16} ions/cm². The images were captured at the remanence state after the field was applied along the (c) [11] and (d) [01] direction. The PEEM-XMCD images are distorted due to electron microscope optics artifacts. (e) The ratio between the remanence and saturation magnetization, $M_{\text{rem}}/M_{\text{sat}}$, extracted from the L-MOKE measurements for the implanted ASI when the field was applied along the [11] and [01] direction. The saturation magnetization was determined from the hysteresis loops by averaging the values between 30 mT and 50 mT. Both the raw data (transparent points) and the smoothed data (nontransparent points), obtained using a five-point running average, are presented. The inset shows representative hysteresis loops for the ASI lattice. The dotted lines indicate the $M_{\text{rem}}/M_{\text{sat}}$ values for collinear magnetic texture for the [11] (red) and [01] (blue) directions.

parallel to the [10] direction, a mixture of ferromagnetic and antiferromagnetic configurations is observed. However, the ferromagnetic order is more dominant than the antiferromagnetic one, as it creates vertices of lower energy ($E_{\text{type II}} < E_{\text{type III}}$) [35]. These observed remanent states confirm that shape anisotropy determines the magnetization direction, as expected [Figs. 3(a) and 3(b)], while they indicate a dominant collinear magnetization component within the elements. However, the resolution of the XMCD-PEEM technique does not allow the detection of magnetization components not aligned

with the x-ray beam, thereby preventing the investigation of noncollinearities in the magnetization texture.

To investigate the presence of magnetization bending, we examine the remanent magnetization normalized by the saturation magnetization, $M_{\text{rem}}/M_{\text{sat}}$, obtained from the L-MOKE measurements. This quantity is an indirect way of determining if the magnetization texture within the elements is fully collinear [21]. For the observed remanent states of an ASI lattice with fully collinear magnetization, $M_{\text{rem}}/M_{\text{sat}}$ has been previously shown to take characteristic values: 0.707 for the [11] direction and 0.5 for the [01] direction [9]. However, as shown in Fig. 3(e), the implanted ASI exhibits higher $M_{\text{rem}}/M_{\text{sat}}$ values at temperatures below 350 K, approximately 0.8 for the [11] direction and 0.6 for the [01] direction. These values reflect the presence of a noncollinear magnetization component. It is important to mention that such noncollinearities in the mesospin texture have also been reported for conventional δ -doped Pd(Fe) ASI lattices at low temperatures [21]. However, the inhomogeneities inherent in implanted patterns seem to amplify the effect further.

Consequently, we perform micromagnetic simulations to investigate the noncollinearities in implanted mesospins. In the simulations, we model a vertex using periodic boundary conditions (see Fig. S3), where each element consists of a core and a periphery area with distinct magnetic properties to imitate the experimental conditions [35]. The simulations confirm the presence of a noncollinear magnetization component at the mesospin edges for any periphery lateral width [35]. Subsequently, we calculate the ratio $M_{\text{rem}}/M_{\text{sat}}$ as a function of the angle θ between the collinear and noncollinear components, by assuming constant periphery width and noncollinearities at the whole mesospin edges (semicircles). For the calculation of the ratio $M_{\text{rem}}/M_{\text{sat}}$, the system was not allowed to relax. The results are presented in Fig. 4. For collinear magnetic texture ($\theta = 0$), the ratio $M_{\text{rem}}/M_{\text{sat}}$ is equal to 0.707 for the [11] direction and 0.5 for the [01] direction, as expected. By introducing noncollinearities ($\theta > 0$), $M_{\text{rem}}/M_{\text{sat}}$ increases.

To explore if the aforementioned effects are fluence dependent, we chose to investigate the same ASI lattice implanted with a nominal fluence of 3×10^{16} ions/cm². The pattern was investigated in the [11] direction and measured with the L-MOKE system at the temperature range of 200–400 K and the VSM system at the temperature range of 200–700 K. The remanent magnetization as a function of the temperature for the ASI lattice and its reference continuous film extracted from the VSM measurements is presented in Fig. 5(a). The array magnetization collapses at higher temperatures compared to the film magnetization, verifying that the ordering temperature is higher for the array. The temperature-dependent ratio $M_{\text{rem}}/M_{\text{sat}}$ from both L-MOKE and VSM measurements is shown in Fig. 5(b). The MOKE measurements show that $M_{\text{rem}}/M_{\text{sat}}$ has a plateau with a height of approximately 0.8, hinting toward noncollinearities in mesospin magnetic texture. However, the corresponding VSM measurements indicate lower values. As MOKE detects the surface magnetization, whereas VSM quantifies the volume magnetization, and the Fe concentration depth-profile decays from the sample surface towards the substrate [Fig. 5(c)], the difference in $M_{\text{rem}}/M_{\text{sat}}$ indicates that the magnetic moments are more rigid close to the sample surface but fluctuate deeper. This

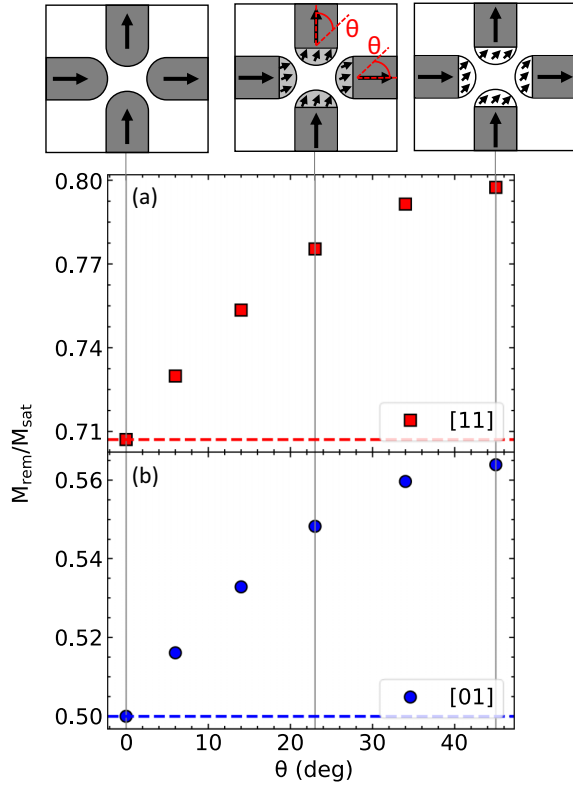


FIG. 4. The simulated ratio $M_{\text{rem}}/M_{\text{sat}}$ as a function of the angle θ between the collinear and noncollinear magnetization components if a field had been applied along the (a) [11] and (b) [01] directions. The dotted lines indicate the $M_{\text{rem}}/M_{\text{sat}}$ values for collinear magnetic texture.

observation highlights a tunable magnetic thickness with temperature, for the implanted building blocks.

In conclusion, we have demonstrated that the magnetic order in Fe-implanted artificial spin ice (ASI) lattices collapses at higher temperatures than in their reference continuous films, with the mesospins exhibiting magnetic inhomogeneities both vertically and laterally. The magnetic inhomogeneities along the depth enable for a tunable magnetic thickness with temperature for implanted building blocks, introducing an additional degree of freedom. This tunability may allow the mesospins to adjust their magnetic dimensionality, transitioning from 2D behavior at higher temperatures to 3D behavior at lower temperatures. In addition to that, the lateral magnetic inhomogeneities can allow modifications to the gap between the elements and their shape, enabling modifications to the dipolar coupling between building blocks after fabrication. These findings open new pathways for tuning the static and dynamic properties of magnetic materials by leveraging fabrication processes that go beyond traditional subtractive methods such as electron-beam lithography and milling. Such advances may significantly impact future applications of magnetic metamaterials. It was recently demonstrated that the ability to design and control magnetic textures in systems such as ASI enables the realization of tailored magnonic regimes [47]. These emerging textures play a crucial role in defining the accessible ground states and governing transitions between magnetization states,

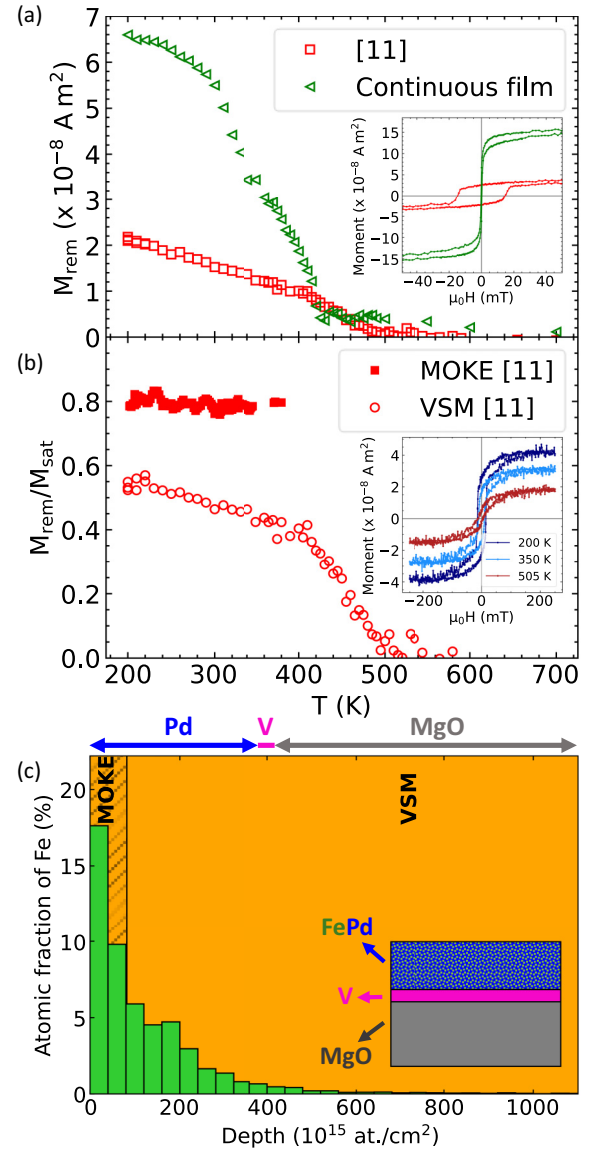


FIG. 5. (a) Remanent magnetization of the ASI lattice and its reference continuous film, implanted with 30 keV Fe and nominal fluence $3 \times 10^{16} \text{ ions/cm}^2$, extracted from the VSM measurements. For both samples, the remanent magnetization of the magnet and the substrate has been corrected. The ASI exhibits lower magnetic moment due to the smaller coverage. (b) The ratio $M_{\text{rem}}/M_{\text{sat}}$ for the implanted ASI lattice with nominal fluence $3 \times 10^{16} \text{ ions/cm}^2$ as a function of temperature extracted from the L-MOKE and VSM measurements. The saturation magnetization was determined from the hysteresis loops by averaging the values between 30 mT and 50 mT for the MOKE measurements and between 150 mT and 250 mT for the VSM measurements. The inset shows hysteresis loops measured by the VSM. (c) The Fe concentration depth profile for the continuous film implanted with 30 keV Fe and nominal fluence $3 \times 10^{16} \text{ ions/cm}^2$ measured with time-of-flight elastic recoil detection analysis (ToF-ERDA). The background shading indicates the penetration depth of the MOKE and VSM measurement systems.

thereby shaping the system's overall dynamics [19]. The engineering of energy landscapes through fabrication techniques like the ion implantation presented here—harnessing the internal degrees of freedom in mesospins—is expected to

be important in the advancement of reconfigurable magnetic metamaterials [47], where dynamic behavior simultaneously functions as the readout. Furthermore, these enhanced internal dynamics, combined with the ability to tune mesospin couplings, open promising avenues for the development of reconfigurable reservoir computing networks based on magnetic metamaterials [48–50]. The thermal behavior described here is also critical for practical implementations of Ising machines using magnetic metamaterials [51]. The design and precise control of their stochastic dynamics are essential prerequisites for these systems to be effectively employed in solving optimization problems [52]. As previously suggested [51], the readout can be achieved using spin-transfer torque magnetic tunnel junctions. An alternative approach involves performing the readout in reciprocal space. By illuminating the array with photons of suitable wavelengths, a diffraction pattern is generated from the reflected light. The positions of the diffraction peaks reveal the periodicity in real space, thereby providing access to the energy-optimized state solution.

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DATA AVAILABILITY

The data that support the findings of this article are openly available [53].

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