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Ergodicity transitions in spin spiral domains in amorphous FeGe thin films

A. Singh,^{1,2,*} E. Hollingworth,^{1,3} S. A. Morley,⁴ A. Us Saleheen,⁴ R. Tumbleson,^{1,5} D. Raftrey,^{1,5} M. R. McCarter,⁴ P. Fischer,^{1,5} F. Hellman,^{1,3} S. D. Kevan,^{4,†} and S. Roy^{1,4,5,‡}

¹*Materials Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA*

²*Materials Sciences Division, Argonne National Laboratory, Lemont, IL 60527, USA*

³*Department of Physics, University of California, Berkeley, California 94720, USA*

⁴*Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA*

⁵*Department of Physics, University of California, Santa Cruz, California 95064, USA*

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Magnetic domains and other forms of quasi-static spontaneous disorder drive non-ergodic behaviors that can lead to unusual material properties. We examine these behaviors near a spin-spiral stripe melting transition in an amorphous FeGe thin film using coherent resonant soft X-ray scattering. We find that the system is ergodic very close to the transition, as reflected by a nearly Gaussian distribution of fluctuations. At slightly lower temperature the distribution is skewed, implying non-ergodicity. The distributions can be resolved into two distinct components reflecting two different kinds of fluctuating spin-structures; one at a shorter and one at a longer length scale. Our results delineate a new approach to evaluate the statistics of fluctuations in many classes of nanoscale heterogeneous materials.

Microscopic domains in ferromagnetic materials generally cause hysteresis that render these materials non-equilibrium on a macroscopic scale. This is manifested, for example, by multivalued magnetization (M) at a given temperature (T) and applied magnetic field (H) and, as H is varied, intermittent motion among a multitude of metastable domain patterns having similar total energy [1]. These domain patterns are often nearly static, so the system cannot sample a large fraction of phase space on the time scale of an experiment, and the system is formally non-ergodic. By contrast, at a spatial scale much smaller than a typical domain, magnetic materials are approximately at equilibrium; e.g., $M(T, H)$ is a well-defined, single-valued order parameter, reflecting local ergodic behavior. This interplay between length scale and ergodicity relates to many systems that exhibit different kinds of spontaneous heterogeneity, from magnetic, spin, and quantum materials to enzymes [2–5]. In the well-known ergodicity breakdown example of Many Body Localization (MBL) phenomena, local functions retain memory of the initial state by reaching a non thermal stationary state that can also lead to longer quantum coherence [6–8]. The interplay between ergodic and non-ergodic behaviors and its evolution plays an important role in the function of many systems in condensed matter systems.

In this paper, we present measurements of the ergodicity of spin spiral stripe domains in amorphous $\text{Fe}_x\text{Ge}_{1-x}$ (a-FeGe) thin films. Several competing interactions in a-FeGe support anomalous magnetotransport, a weakly perpendicular spin spiral phase, and several disordered real-space topological structures [9–11]. The interactions

can be tuned by varying the magnetic field, temperature, composition, and film thickness. a-FeGe exhibits a metal-insulator transition at an iron fraction $x \sim 0.4$, just below the concentration used in this study. This implies that electronic properties and correlations are sensitive functions of composition, reminiscent of many quantum materials.

The spin spiral stripe phase in a-FeGe results from competition between a randomized Dzyaloshinskii-Moriya (DM) and scalar exchange interactions. These stripe domains exhibit a resonant soft x-ray scattering signature that broadens and nearly disappears over a narrow temperature range [11]. The results presented below suggest this is best represented as a melting transition, and we will discuss it as such. Accompanying this melting transition we find and report here an ergodicity transition in which a large fraction of the domains transform from statistically non-ergodic, where the distribution of fluctuation amplitudes exhibits substantial kurtosis and skew, to approximately ergodic, where this distribution becomes essentially Gaussian. Our results exhibit behaviors qualitatively similar to melting transitions often observed in small molecule and polymer glasses, and therefore suggest that spontaneous charge, spin, or orbital disorder in many magnetic and quantum materials might exhibit glassy behaviors. Our approach provides a new and comprehensive way to evaluate the spatio-temporal statistics of domain-scale fluctuations in complex electronic materials, and moreover sheds light on the impact of ergodicity breaking associated with spontaneous heterogeneity.

We studied an 80 nm thick amorphous $\text{Fe}_{0.53}\text{Ge}_{0.47}$ film deposited onto an X-ray transmissive 500 μm square silicon nitride membrane using DC magnetron co-sputtering at rates of approximately 0.1 nm/s, with the substrate at room temperature at an Argon pressure of 1.8 mTorr and a base pressure of $< 5 \times 10^{-8}$ Torr. The films were capped with a 5 nm layer of aluminum

* Corresponding author: arnabsingh@lbl.gov

† Corresponding author: sdkevan@lbl.gov

‡ Corresponding author: sroy@lbl.gov

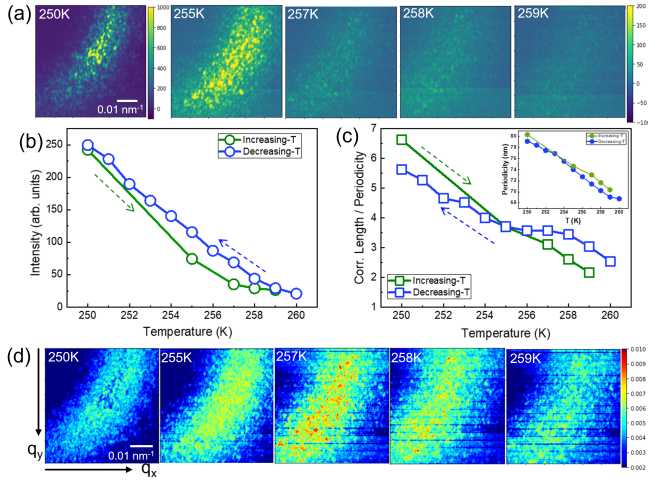


FIG. 1. **Static and dynamic properties of $a\text{-Fe}_x\text{Ge}_{1-x}$** (a). Mean intensity maps, $I_{\text{mean}}(t')$, of the 1st order magnetic diffraction peak due to the stripe lattice for the heating cycle. Mean intensity is derived from the mean intensity image of 2000 images taken at 0.9s/frame (τ) for a total duration, $t = 30$ minutes. Temperature dependent evolution of the (b) peak intensity and (c) the correlation length/periodicity and (inset) periodicity of the helical stripe domains during heating and cooling. (d) Fluctuation distribution maps of the speckle pattern for the portion of the resonant soft x-ray scattering pattern near the wave vector of the spin strip (Fig. 1 (a)).

to prevent oxidation. We measured static and temporal magnetic correlations of this film using coherent resonant X-ray magnetic scattering in a transmission geometry at the COSMIC scattering beam line (7.0.1.1) at the Advanced Light Source, Lawrence Berkeley National Laboratory. The incident beam was tuned to the Fe L3 edge at $E = 707$ eV to provide maximum magnetic scattering contrast. A $7\ \mu\text{m}$ diameter pinhole was inserted at the beam path upstream of the sample to establish transverse coherence of the incident X-ray beam. Data was recorded using a fast charge coupled device camera placed at 0.284 m downstream of the sample. We took data both while increasing and decreasing the temperature through the transition (T_{st}). We note here that T_{st} is lower than ferromagnetic transition temperature T_c which is around 400K for Fe concentration = 0.53. At every measured temperature, we stabilized the temperature to within 10 mK of the target temperature.

We first discuss the zero-field magnetic/spin phase diagram for an $\text{Fe}_{0.53}\text{Ge}_{0.47}$ film, specifically, near the spin spiral melting transition noted above. The phase diagram was reported previously [9, 11] and schematically shown in the Supplementary Materials (Fig. S2). In Fig. 1(a) we show the resonant soft x-ray scattering patterns corresponding to the spin spiral periodicity as the temperature is increased towards T_{st} . The full peak profiles extracted from these images are presented in the Supplementary Materials (see Fig. S1). Consistent with previous findings [11], Fig. 1(a, b, c) show, respectively,

that the stripe diffraction pattern becomes diffuse and disordered, its intensity decreases to nearly zero, stripe periodicity and correlation length decrease, as temperature increases from 250 - 260K. Most interestingly, the ratio of the spin-spiral stripe correlation length to its periodicity, which measures the number of periods over which the stripes are coherent, decreases from 7 at 250K to 2 at 260K. This suggests a melting transition between a marginally ordered phase at lower temperature, consistent with meandering stripes as observed in previous Lorentz transmission electron microscopy measurements [10], to a liquid-like phase at higher temperature composed of short-range-ordered spin structures, the structure of which remains to be determined. This conclusion suggests the stripes might show interesting dynamical properties as one approaches the melting transition from the ordered state and vice-versa.

The scattering patterns in Fig. 1(a) are speckled due to the transverse coherence of the X-ray beam. The envelope of the observed scattering peak, which was the subject of the previous paragraph, allows us to measure the average properties and spatial correlations of the spin spiral domains. The seemingly random speckle reflects the square modulus of scattering from the illuminated domain structure, and this will fluctuate in intensity and position due to fluctuations in size, periodicity or number density of the domains. An increasingly popular technique to measure these fluctuations is x-ray photon correlation spectroscopy (XPCS), in which the temporal autocorrelation in each pixel is calculated to determine the time scale on which the scattered signal in that pixel fluctuates. The minimum time resolution is set by the frame rate of the movie, which is 0.9 s, i.e., we measure

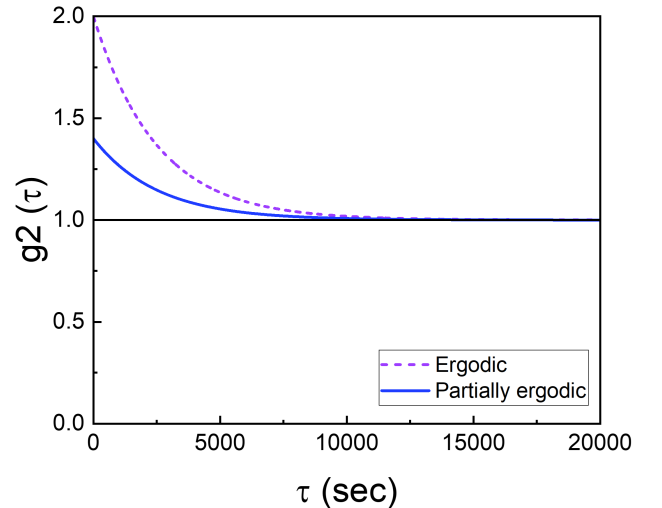


FIG. 2. **Ergodic and non-ergodic behaviour.** Schematic representation of the time-average g_2 plot for an ergodic system (purple dashed curve) and a partially ergodic system (blue solid curve) as a function of the delay time (τ) for a typical well-behaved system. [12]

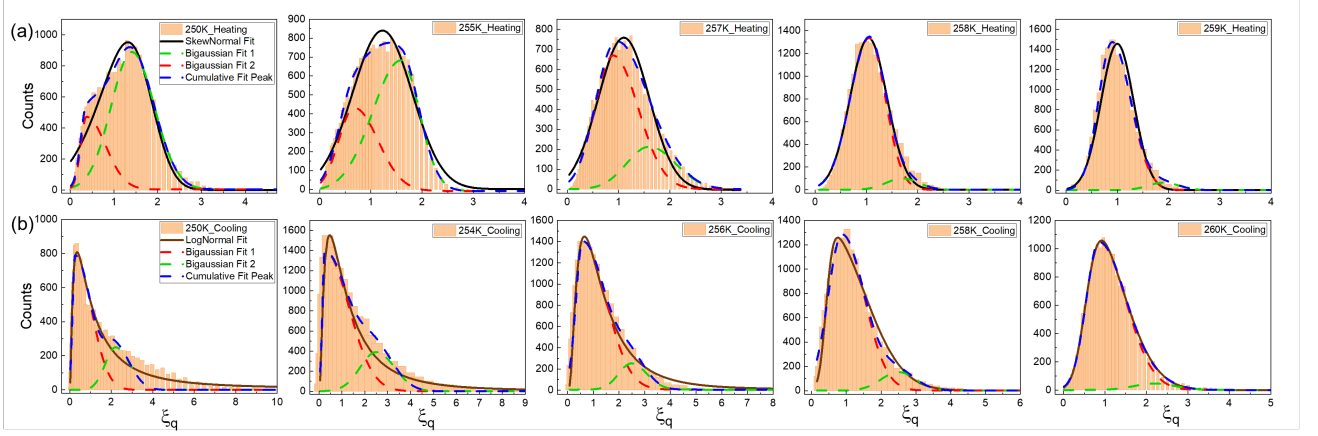


FIG. 3. **Distribution of fluctuation amplitude and appearance of twin peaks.** Histograms of the ξ_q with temperature from 250K - 260K during the (a) heating and (b) cooling cycles. The solid curve denotes the fitting to the distribution using either a skew-normal function (black curve) or a log-normal function (brown curve). The red and green dashed curves shows the fitted distribution of the two peaks present in the distribution using bigaussian functions with the blue dashed curve representing the cumulative sum of both the peaks.

fairly slow domain fluctuations. This normalized auto-correlation function is normally called $g_2(q, \tau)$, where q is the scattering wave vector and τ is the autocorrelation delay time:

$$g_2(q, \tau) = \frac{\langle I(q, \tau)I(q, 0) \rangle_t}{\langle I(q) \rangle_t^2} \quad (1)$$

Here, t is the total duration of the measurement. It is instructive to understand how this function provides information about possible non-ergodicity in the system under study. This was discussed long ago in the context of “frozen-in disorder” in polymer gels, which [13], like nearly static domain walls in magnetic systems discussed in the introduction, can lead to non-ergodicity. The role of non-ergodicity is often not explicitly considered in most XPCS experiments, but it is the key feature of our results and analysis. The ergodicity of a system breaks when the time average of a particular motion differs from the ensemble average; that is, there can be more than one type of fluctuating state in the sample with different time-dependence or the system is far from attaining its equilibrium state [14, 15]. In this way, non-ergodicity refers to a system in which slow dynamics of domains keeps the system from achieving equilibrium on the length and time scales probed.

Fig. 2 shows schematically the generic behavior of $g_2(\tau)$ for ergodic and partially ergodic systems. In an ideal experiment, an ergodic system (dashed line) will have $g_2(0) = 2$, and $g_2(\infty) = 1$. A non-ergodic system (solid line) will suppress g_2 at shorter time scales on which the system cannot fully equilibrate. For our work we extract two important quantities at each (q_x, q_y) pixel. 1]. the fluctuation amplitude, which is the $g_2(q, 0)$ value and, 2]. The ergodicity parameter ξ_q . To define the above two parameters that are consistent with the liter-

ature, and that represent all the distributions at different temperatures during heating or cooling on the same scale, it is crucial to normalize experimental factors, e.g., incomplete spatial and temporal coherence of the photon beam and finite spatial and temporal bandwidth of the detector. All of these experimental factors reduce $g_2(0)$ and would otherwise appear to reflect non-ergodicity.

To achieve this normalization, we have adopted a two-step approach. In the first step, we calculate the normalized variance of each pixel (or speckle) along the time axis:

$$\varepsilon_q = \frac{\langle I(q)^2 \rangle_t - \langle I(q) \rangle_t^2}{\langle I(q) \rangle_t^2} \quad (2)$$

This is equal to $g_2(q, \tau \rightarrow 0) - 1$. Eq. (2) creates a 2D variance map over the entire time series, with each pixel normalized by the time-average intensity square $(\langle I(q) \rangle_t^2)$.

In the second step, we further divide this time-normalized 2D variance map by its mean-spatial (q) average $\langle \varepsilon_q \rangle$ to create a spatio-temporal averaged distribution of the fluctuation amplitude ξ_q . given by:

$$\xi_q = \frac{\varepsilon_q}{\langle \varepsilon_q \rangle_q} \quad (3)$$

Here $\langle \varepsilon_q \rangle_q$ is the average value of the ε_q over all the pixels (q) in the 2D variance map and thus is a single-value. We note that the above step is equivalent to division by square of visibility, which will be similarly impacted by finite coherence and resolution. We define ξ_q as the ergodicity parameter and its histogram (see Fig. 3) provide an estimate of skew and kurtosis. We note that $1 - \langle \xi_q \rangle$ is defined as ergodicity breaking parameter [15]. A system

that is perfectly ergodic on the length scale being probed over a sufficiently long measurement time (t) will make a symmetric Gaussian distribution of fluctuations with the peak centered at $\xi_q = 1$. For a non-ergodic system, the distribution will be asymmetric with finite skew [12, 14].

For the fluctuation distribution maps shown in Fig. 1 (d), we have used a rectangular area ROI of size 150×400 pixels, which includes a significant background so to get a better visibility between static and dynamic parts. In order to bring all the fluctuation distribution maps taken at different temperatures within the same scale range, we have plotted the distribution of $\sqrt{\xi_q}/I_{max}$, with I_{max} being the maximum intensity value in the ROI at a given temperature [11].

In what follows, we will present and discuss the distributions of ξ_q from a ROI consisting only of the diffracted signal (see SM for more details), near the spin-stripe melting transition. We show histograms of ξ_q in Fig. 3, with panel (a) for increasing temperature, and panel (b) for decreasing temperature. We see that the distribution is symmetric at or near the melting transition. The distribution becomes increasingly asymmetric as the temperature decreases. This observation of the transition from symmetric to non-symmetric fluctuation amplitude distribution would imply that the system is ergodic near transition temperature, meaning that the fluctuation are Brownian in character [5]. At lower temperature, we find non-ergodic behavior.

We were unable to achieve good fits of the lower temperature histograms with any common distribution function. We did find that to a reasonable extent the lower temperature distributions during heating could be fitted with a skew-normal distribution and during cooling by a log-normal distribution. However, a cursory look at several histograms suggests that there are two dominant

components in the distributions. We were able to achieve much better fits for the histograms with the cumulative distributions of two independent bi-Gaussians, as shown in the red and green curves, with blue being the sum of these two. A bi-Gaussian distribution is an asymmetric distribution with different widths on either side of the peak. We found that at any temperature the red curve shows more asymmetry than the green curve for both the cooling and heating cycles.

An efficient way to characterize the distributions is in terms of the skew and kurtosis, the third and fourth central moments of a histogram. These values obtained from the fits of the skew-normal and log-normal distribution are plotted as a function of temperature during heating and cooling, respectively, across the transition temperature in Fig. 4(a, b). Skew measures the magnitude of asymmetry of the distribution, and kurtosis measures the strength of the tails relative to a normal distribution that has zero skew and kurtosis. During both increasing and decreasing temperature scans the distributions approach a Gaussian (normal) distribution as T approaches within 1-2 K of the transition temperature, where the distribution of ξ_q is closely centered at 1.

At temperatures a few degrees below the transition, the skew and kurtosis deviate increasingly from the Gaussian values and, moreover, the shape of the histograms becomes very different for increasing vs. decreasing temperature (see both Fig. 3 and Fig. 4(a, b)). This results in a significant difference in the value of skew and kurtosis between the heating and cooling cycles. Although we do not understand this difference in detail, it is reasonable for the distribution of fluctuations to be different for heating and cooling. In cooling, nucleation must play a key role in the formation of spin-spiral stripes. By contrast nucleation is less relevant when approaching the transition from lower temperatures where the phase is already highly heterogeneous.

We also plotted the area of the two composite curves in the bigaussian, as a function of temperature for the heating and cooling cycles (normalized by the total area under the histogram) (Fig. 4(c, d)). For the heating case, at $T < 256$ K the green curve (in Fig. 3) dominates, but as the temperature increases there is a crossover and the red curve dominates. For the cooling cycle (Fig. 4(d)), we did not observe a crossover within the studied temperature range. The crossover might be happening at temperatures below 250 K. One way to understand this result is in terms of the degree of stripe ordering which is reflected in the correlation length. As shown in Fig. 1(c), the strength of the ordering of the stripe at 250 K at the start of heating is greater than when $T=250$ K is approached during cooling, indicating that the magnetic topology at a certain temperature after a temperature cycle may not be the same.

We believe that the observed crossing of the two bi-gaussian peaks in the distribution provides important clues for how to understand the role of heterogeneity in transitions like this one. Recall that at low temperatures

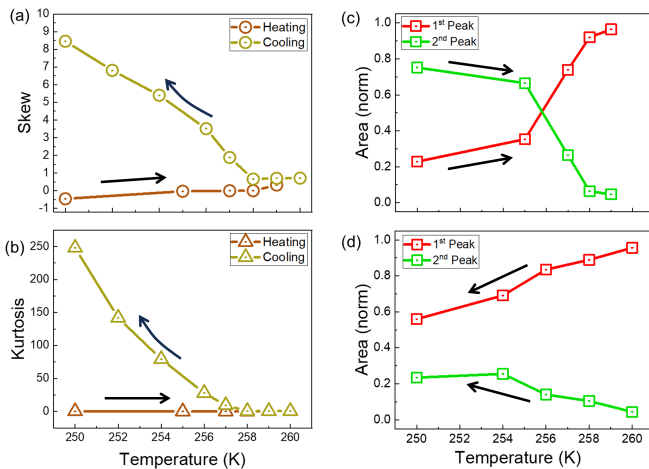


FIG. 4. **Skew, kurtosis and area of the two fluctuation distribution peaks.** Variation of (a) skew and (b) kurtosis with temperature during both heating and cooling cycles. Area (normalized by the total area of the histogram) of the two peaks (See Fig. 3) for (c) heating and (d) cooling cycles.

during heating, parts of the system are frozen into an intrinsically heterogeneous spin stripe phase leading to an increasingly non-ergodic state. We suggest that the motion in this low temperature regime is mostly local motion of the domain walls (see SM). As temperature increases domains starts to break into smaller domains and near transition all domains fluctuate due to increased thermal energy.

Irrespective of heating and cooling cycle, we find that the red Gaussian has higher counts at higher temperatures, and tends to dominate away from the center of the diffraction peak. The green Gaussian, on the other hand, tends to dominate closer to the center of the peak. (see SM Fig. S6 for details). This suggests that the red Gaussian probes spatially smaller and less correlated structures, while the green Gaussian probes the imperfect order of the spin stripe domains. This interpretation is consistent with the increasing stripe meandering as the temperature increases, as suggested by the increasing width of the diffraction peak in Fig. 1(c). Proceeding closer to the actual transition, these two motions become indistinguishable, and the histograms ultimately collapse into a Gaussian peak as the system becomes ergodic within a degree or two of the transition.

Some of these findings share similarities to the classical glass transition, where two kinds of motion are delineated [16, 17]. The so-called beta transition occurs at lower temperature and is thought to be related to local fluctuations in a glass, and the alpha transition occurs at higher temperature and is taken to reflect large-scale motion, e.g., of polymer chains, that makes the system essentially ergodic. Many classical glasses are in soft mat-

ter which are characterized by many coupled modes at low energy; consequently, these materials have low bulk moduli. In terms of bulk moduli, our a-FeGe films are hard matter, but the manifold of spin degrees of freedom, taken separately, has many coupled low energy degrees of freedom, and the observation of glassy behavior might not be surprising.

Finally, returning to the motivation for this work, we note that observational non-ergodicity suggests some fraction of a system does not explore the phase space very well - is “quasi-static”, while the rest does. Non-ergodicity might be an important criteria for the design of thermal machines whose efficiency is not restricted by classical thermodynamics [18]. Partial ergodicity is suggested to be important [19, 20], for example, in enzymes, which often involve a nearly static ‘scaffold’ that creates a reaction center that performs biocatalysis with high selectivity and precision [5]. Thus, both the ergodic and non-ergodic parts are important to achieve function. It is likely that understanding both parts will be necessary to realize the evident potential of spin and quantum materials [21].

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