Spin freezing in the spin-liquid compound $FeAl_2O_4$

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Spin freezing in the A-site spinel $FeAl_2O_4$, which is a spin-liquid candidate, is studied using remnant magnetization and nonlinear magnetic susceptibility and isofield cooling and heating protocols. The remnant magnetization behavior of $FeAl_2O_4$ differs significantly from that of a canonical spin glass, which is also supported by analysis of the nonlinear magnetic susceptibility term $\chi_3(T)$. Through the power-law analysis of $\chi_3(T)$, a spin-freezing temperature $T_g = 11.4 \pm 0.9$ K and critical exponent $\gamma = 1.48 \pm 0.59$ are obtained. A Cole-Cole analysis of magnetic susceptibility shows the presence of broad spin relaxation times in $FeAl_2O_4$, however, the irreversible dc susceptibility plot discourages an interpretation based on conventional spin-glass features. The magnetization measured using the cooling-and-heating-in-unequal-fields protocol brings more insight into the magnetic nature of this frustrated magnet and reveals unconventional glassy behavior. Combining our results, we arrive at the conclusion that the present sample of $FeAl_2O_4$ consists of a majority spin-liquid phase with "glassy" regions embedded.

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I. INTRODUCTION

AB_2X_4 (A = Mn, Fe, Co; B = Al, Sc, Rh; X = O, S) compounds where a magnetic atom occupies the tetrahedrally coordinated A site are known as A-site spinels and are frustrated magnets where the frustration effects arise from competing nearest-neighbor (NN) and next-nearest-neighbor (NNN) exchange interactions [1–4]. The spinel structure is composed of a diamond lattice formed by the A-site atoms and the pyrochlore network of the B site. The ideal diamond lattice with only NN interactions is not geometrically frustrated, in contrast with the pyrochlore lattice, which is inherently geometrically frustrated. Additional NNN interactions are necessary to create frustration in the diamond lattice. In A-site spinels, frustration in the diamond lattice can manifest as a "spiral" spin liquid [5], spin liquid [2,6], orbital liquid [4], orbital glass [3], or a spin-orbital singlet state with a quantum critical point [7], and hence these materials are of immense interest to condensed matter physicists. Specifically, the theoretical work by Bergmann *et al.* [5], which invokes the mechanism of "order by disorder" [8] as the degeneracy-breaking mechanism, predicts the emergence of a "spiral" spin liquid in the A-site spinels which is characterized by a manifold of degenerate ground states in a system devoid of defects. In a subsequent work, the influence of quenched random impurities such as a random bond, a vacancy, or an interstitial spin on the "spiral" spin-liquid properties was undertaken [9]. It was found that quenched disorder can act as a degeneracy-breaking mechanism. A "Swiss-cheese model" was introduced which explained, to some extent, the contrasting findings of long-range ordered and "glassy" magnetic ground states reported for the spinel $CoAl_2O_4$ [1,10]. The fact that the magnetic properties of A-site spinels are governed by two factors—frustration and site disorder—has been reiterated through experimental studies on Al-based systems [11–13]. A magnetic phase diagram for $CoAl_2O_4$ tuned by the defect content (η, the inversion parameter) has been proposed, where spin-liquid and spin-glass phases compete as a function of η [13].

The effect of weak disorder on geometrically frustrated lattices has been treated theoretically in the parlance of fully frustrated Ising [15] and Heisenberg systems [16,17]. Though these theoretical studies have pointed towards the realization of a spin-glass state at low temperatures due to the weak disorder effect, the question as to whether additional random interactions on the top of frustration would lead to a "true" spin-glass phase at low temperatures remains relevant [18]. Our previous studies on the A-site spinels $FeAl_2O_4$ and $MnAl_2O_4$ using magnetometry and polarized neutron diffraction have revealed the presence of significant spin correlations arising purely from frustration effects [12]. $MnAl_2O_4$ was observed to possess an ordered magnetic state below $\approx$45 K whereas $FeAl_2O_4$ exhibited only short-range order down to 4 K. In the present paper, we study the spin freezing in $FeAl_2O_4$ through ac and dc nonlinear susceptibility and thermoremanent (TRM) and isothermoremanent (IRM) magnetization, along with other magnetization protocols, with the aim of elucidating the magnetometric "signature" of a spin liquid.

II. REMANENT MAGNETIZATION

The magnetic measurements reported in this paper were all performed on polycrystalline pellets (the details of the sample preparation are discussed in our previous publication [12]) using a commercial superconducting quantum interference device (SQUID) magnetometer (Quantum Design, Inc.). In order to measure TRM, the sample was field cooled down to 4 K from 300 K; the magnetic field was switched off and the remnant magnetization was recorded instantly. Similarly, for IRM, the sample was cooled in zero field down to 4 K from 300 K, and the magnetic field was switched on and instantly switched off to measure the remnant magnetization [14]. The measurements were repeated for different values of applied magnetic field. In Fig. 1 the field dependences of TRM/IRM of

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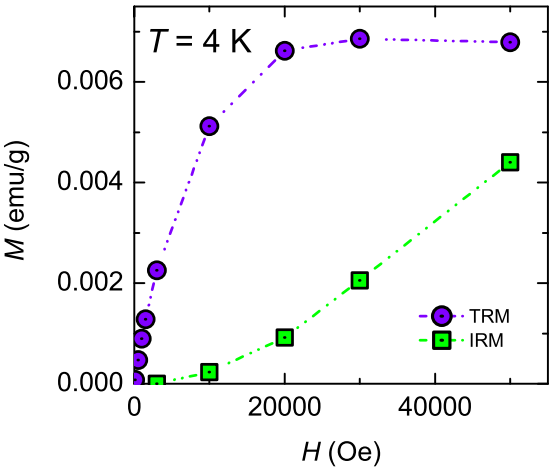

FIG. 1. (Color online) The thermoremanent (TRM) and the isothermoremanent (IRM) curves for $FeAl_2O_4$ at 4 K as a function of applied magnetic field. The TRM and IRM responses of $FeAl_2O_4$ are characteristically different from thos of a canonical spin glass or a superparamagnet (see Ref. [14] for a comparison of TRM and IRM curves of different magnetic systems).

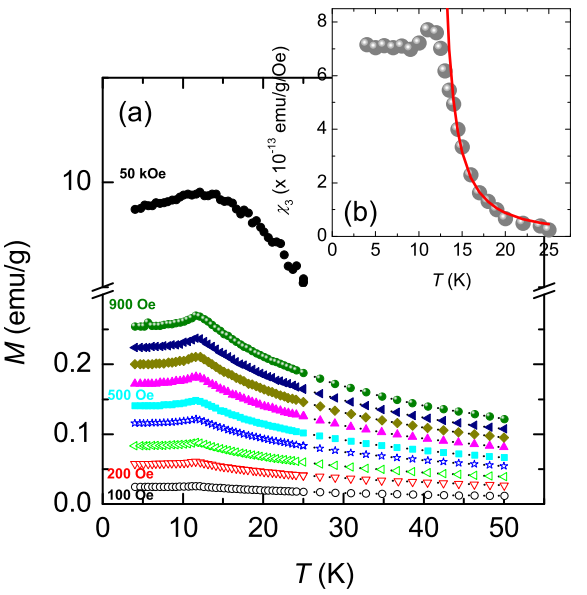

FIG. 2. (Color online) (a) The temperature dependence of field-cooled dc magnetization plots of $FeAl_2O_4$ at various applied fields in the range 100 Oe (black open circles, bottom curve) to 50 kOe (black solid circles, top curve). M vs H plots at different temperature values are extracted from these data. (b) Third harmonic of nonlinear susceptibility χ_3 along with a power-law fit (solid line) using $\chi_3 = \chi_3^0(T/T_g - 1)^{-\gamma}$.

$FeAl_2O_4$ are presented. The remnant magnetization curves are evidently distinguished from those of canonical spin glasses but resemble those of a diluted antiferromagnet in field (DAFF) [14], albeit with some notable differences. In the case of DAFF, the IRM response is expected to be identically zero for all fields and the TRM is normally inversely proportional to the domain size. In $FeAl_2O_4$, however, the IRM response is not zero, but reaches up to a value which is half that of the TRM response at high fields. The field stability observed in the case of $FeAl_2O_4$ is a notable feature, especially when the remnant magnetization response does not show any similarity to canonical spin glasses [14].

III. NONLINEAR SUSCEPTIBILITY

In order to understand more about the spin-freezing process in $FeAl_2O_4$, we performed dc magnetic measurements to extract the nonlinear susceptibility terms. The nonlinear terms of dc susceptibility are sensitive to the spin-freezing order parameter. In order to estimate the nonlinear contributions, the magnetization data were obtained under field-cooled conditions in the range 3–50 K for different values of applied magnetic fields in the range 100 Oe to 50 kOe (see Fig. 2). Prior to each measurement, the sample was heated up to 150 K and then field cooled to the lowest temperature in order to measure the magnetization as a function of temperature. The nonlinear susceptibilities are extracted from the magnetization data by writing

$$M/H(T) = \chi_1(T) - \chi_3(T)H^2 + O(H^4) \quad (1)$$

$$= \chi_1(T) - a_3(T)\chi_1^3(T)H^2 + O(H^4), \quad (2)$$

$$\chi_{\rm nl}(T,H) = 1 - M(T,H)/\chi_1 H, \quad (3)$$

where $\chi_1(T)$ is the linear susceptibility at temperature T, $\chi_3(T)$ is the third harmonic of nonlinear susceptibility, the coefficient $a_3 = \chi_3/(\chi_1)^3$, and $\chi_{\rm nl}$ is the net nonlinear susceptibility [19].

The nonlinear terms of susceptibility were extracted from the data following a polynomial fit to the magnetization using $M = \chi_1 H - \chi_3 H^3 + \chi_5 H^5$. The χ_1 and χ_3 terms extracted from the fit (see the inset of Fig. 2) show a peak at $T_a \approx 13$ K (T_a is designated as the temperature at which a peak is observed in the magnetic response in Fig. 2). In order to test the static criticality wherein the χ_3 term should diverge, a fit was administered using the power law $\chi_3 = \chi_3^0(T/T_g - 1)^{-\gamma}$. Here, T_g is the glass-transition temperature and γ is a critical exponent. The fit yielded $T_g = 11.4 \pm 0.9$ K, $\gamma = 1.48 \pm 0.59$. The γ value is smaller than that observed generally in spin glasses [20] but much greater than that for a mean-field system. The net nonlinear susceptibility was obtained as $\chi_{\rm nl} = (1 - M/\chi_1 H)$. A plot of $\chi_{\rm nl}$ vs H^2 is presented in Fig. 3. From the power-law dependence $\chi_{\rm nl}(T = T_g, H) \propto H^{2/\delta}$, an estimate of the critical exponent δ can be obtained as the slope of the plot of the natural logarithm of $\chi_{\rm nl}$ and H. This led to an estimate of $\delta = 3.57 \pm 03$ in the present case. In the low-field limiting case, $\chi_{\rm nl} = H^{2/\delta} f(\tau^{(\gamma+\beta)/2}/H)$ (where $\tau = (T/T_g - 1)$) can be written as $\chi_{\rm nl} = H^{2/\delta}.\ \tau^{-2\gamma/(\gamma+\beta)}$, then $\delta = 1 + \gamma/\beta$. In Fig. 3(b), a scaling plot with $\delta = 3.57$ demonstrates the data collapse onto a universal plot for temperatures below 25 K.

From the neutron diffraction studies using polarized neutrons [12], it is clear that short-range spin-spin correlations assume importance in this material. The short-range magnetic order in frustrated magnets reflects as a statistical distribution of relaxation times where each cluster acts as an independent unit [21]. This distribution of relaxation times can be experimentally extracted through the analysis of complex ac magnetic susceptibilities. For this purpose, the real and imaginary parts of the complex ac magnetic susceptibilities were recorded for $FeAl_2O_4$ using a commercial physical

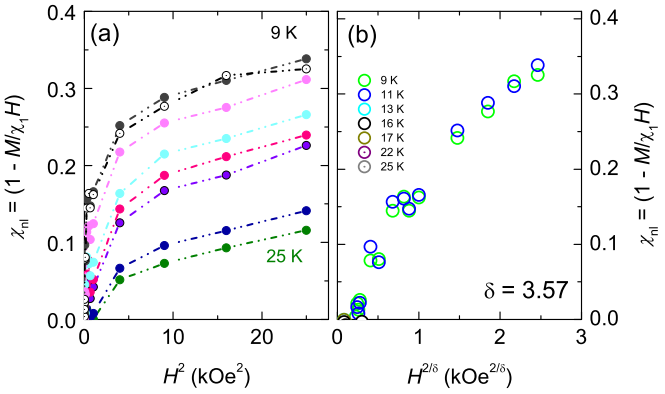

FIG. 3. (Color online) (a) The nonlinear susceptibilities χ_{nl} as a function of H^2 at different selected temperatures above and below T_a (which is identified as the peak observed in dc magnetization curves in Fig. 2). (b) The scaling plot of the data presented in (a) with $\delta = 3.57$ clearly brings out the data collapse conforming to the scaling exponent δ (see text).

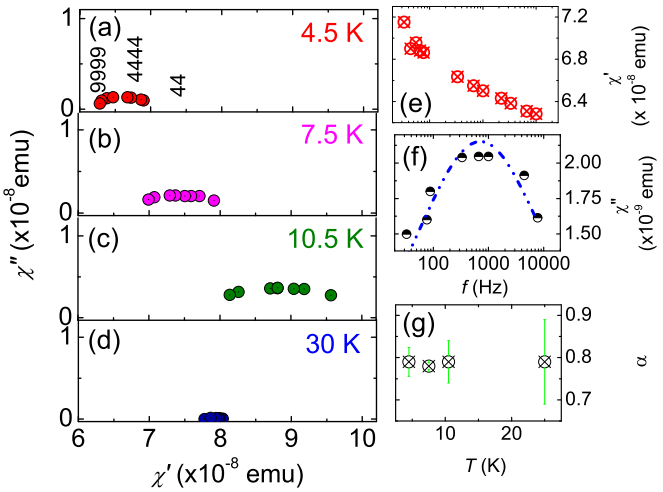

FIG. 4. (Color online) (a)–(d) The Cole-Cole plot (χ''-χ') at different temperatures below 30 K supports the claim of a broad distribution of relaxation times in $FeAl_2O_4$. The numbers in (a) indicate the applied frequency in Hz. The x axis is offset from zero for clarity. (e) Dependence of χ' on frequency. (f) The imaginary part of the magnetic susceptibility χ'' as a function of frequency along with the theoretical curve according to Eq. (5). (g) The parameter α has a magnitude around 0.8, which indicates a broad relaxation.

property measurement system (Quantum Design, Inc.). The ac susceptibility data $\chi(f,T)$ did not present a clear shift in the peak value with frequency (presented in Ref. [12]), as is normally observed in the case of canonical spin glasses [22]. However, in the presence of applied dc magnetic field, the peak in $\chi(f,T)$ was observed to broaden. At this point it is interesting to note the case of another frustrated magnet, $Dy_2Ti_2O_7$, which is a spin ice, where the application of dc magnetic field raised the spin-freezing temperature [23]. The spin dynamics in $Dy_2Ti_2O_7$ spin ice in the limit of very low disorder was peculiar, with a very narrow range of relaxation times, and was claimed to represent a new form of spin freezing in a frustrated magnet.

IV. AC SUSCEPTIBILITY

The spin dynamics and the relaxation time distribution in frustrated or glassy magnets can be analyzed using the Cole-Cole formalism, which is the magnetic analog of the Debye model developed for frequency dispersion of the dielectric response [24]. The relaxation of a magnetic system can be formulated as

$$\chi = \chi_s + \{(\chi_0 - \chi_s)/[1 + (i\omega\tau_c)^{1-\alpha}]\}, \quad (4)$$

where χ_0 and χ_s are the isothermal ($\omega = 0$) and adiabatic ($\omega \to \infty$) susceptibilities, respectively, and τ_c is the median relaxation time around which the distribution of relaxation times occurs. The parameter α describes the "flatness" of the distribution times; $\alpha = 1$ corresponds to an infinitely wide distribution whereas $\alpha = 0$ gives the familiar Debye relaxation with a single relaxation time. The ideal case of a single relaxation time can be contrasted from the case where a distribution of relaxation times is present [25]. Equation (4) can be decomposed to obtain the relation

$$\chi''(\omega) = \chi^0 \left(1 - \frac{\sin[(1/2)\beta\pi]}{\cosh[\beta \ln(\omega\tau_c)] + \cos[(1/2)\beta\pi]}\right), \quad (5)$$

where $\omega = 2\pi f$, $\chi^0 = \frac{\chi_0 - \chi_s}{2}$, and $\beta = (1 - \alpha)$. The Cole-Cole plots obtained for $FeAl_2O_4$ are presented in Figs. 4(a)–4(d) for the temperatures 4.5, 7.5, 10.5, and 30 K showing a "flat" spin relaxation compared to the case of $Dy_2Ti_2O_7$ where a semicircular arc was obtained [23]. A flat Cole-Cole plot for $FeAl_2O_4$ indicates broad relaxation times are present in the system. Figure 4(e) shows the frequency variation of the real part of susceptibility. The imaginary part of susceptibility $\chi''(f)$ as a function of frequency was fitted to Eq. (5) at various temperatures, as shown in Fig. 4(f). The χ'' does not follow a single relaxation time as per the Casimir–du Pré relation $\chi''(f) = f\tau[(\chi_0 - \chi_s)/(1 + f^2\tau^2)]$ used for spin ice [23]. The parameter α extracted from the fit [Fig. 4(g)] displays a value around 0.79 ± 0.05 at low temperatures, thereby indicating the presence of strong relaxation, compared to the spin ice $Dy_2Ti_2O_7$, which had $\alpha \approx 0.5$. Alternatively, the value of $\beta \approx 0.2$ also implies a deviation from the mean-field value.

The importance of quenched disorder on the spin-liquid properties of A-site spinels has been theoretically treated in recent literature [9]. A systematic dependence of the ground state spin-liquid properties of the spinel $CoAl_2O_4$ on the content of disorder η has been experimentally investigated recently [13]. The proposed $T - \eta$ phase diagram shows that the spin-liquid phase is stable only for a low content of disorder. With $\eta > 0.08$, though a high degeneracy of ground states exists in the spin-liquid state, a spin-glass ground state is selected by the system. The magnetic ordering in the spin-glass phase in the presence of an applied field can be studied by plotting the irreversible susceptibility $\Delta M/H = (M_{FC} - M_{ZFC})/H$, as presented in Fig. 5. The disordered spin-glass compositions of $CoAl_2O_4$ showed the occurrence of a "weak" and a "strong" irreversibility as the slope changes in $\Delta M/H$ [13]. Such two-step irreversibilities are commonly observed for canonical spin glasses, for example, CuMn [26] and $Y_2Mo_2O_7$ [27]. Any indication of such two-step irreversibilities are absent in Fig. 5.

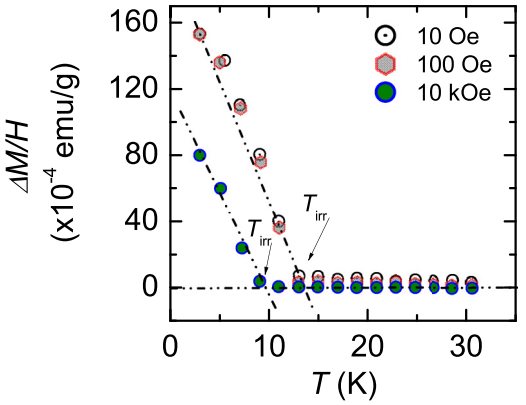

FIG. 5. (Color online) The "irreversible" dc susceptibility $\Delta M/H$ of $FeAl_2O_4$ where $\Delta M = (M_{FC} - M_{ZFC})$. The irreversibility temperature is shown as T_{irr} and is indicated by arrows. The dashed-dotted lines are guides to the eye.

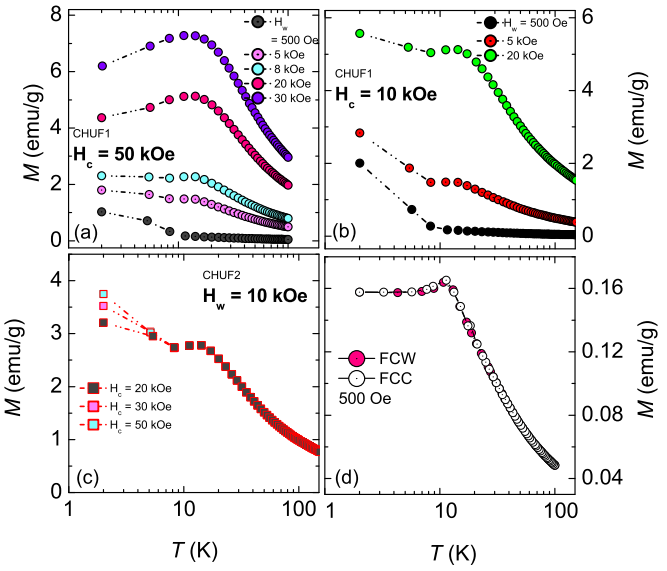

FIG. 6. (Color online) (a), (b) Magnetization as a function of temperature using the protocol of CHUF1: The sample was cooled in a constant field of (a) $H_c = 50$ kOe or (b) $H_c = 10$ kOe and the magnetization is measured while warming in various applied fields H_w, as marked in the figure. (c) The magnetic response under the protocol of CHUF2: The sample was cooled in different applied fields H_c while the magnetization was measured in a constant field $H_w = 10$ kOe. (d) A field-cooled magnetization measurement in cooling and warming cycles does not give evidence for any thermal hysteresis.

A previous work by MacDougall *et al.* [10] on single crystals of $CoAl_2O_4$ put forward the idea of kinetic freezing of domain walls inhibiting long-range magnetic order. This concept was highlighted as prevailing in a broader class of frustrated magnets and would then offer an explanation of the reports of "anomalous" glassy behavior. When a first-order magnetic phase transition is kinetically inhibited, it can give rise to metastable states which are then referred to as "magnetic glasses" [28]. The kinetics of the phase transition can, for example, get arrested due to the coexistence of a metastable and a transformed stable state at low temperatures. Several studies [29–31] investigating first-order magnetic phase transitions have used the "cooling-and-heating-in-unequal-fields" (CHUF) protocols to confirm metastable states at low temperatures or to distinguish the equilibrium phase from a glasslike phase. In order to gain more insight into the spin freezing in $FeAl_2O_4$, we have performed CHUF measurements. The results are presented in Fig. 6.

V. CHUF MEASUREMENTS

We have performed two protocols under the CHUF measurements. In the first set (CHUF1), the sample is field cooled from 300 to 2 K at $H_c = 50$ kOe (also repeated for $H_c = 10$ kOe). At 2 K, the field is put to zero and various applied fields of $H_w = 500$ Oe, 5 kOe, 8 kOe, 20 kOe, and 30 kOe were used to measure magnetization while warming the sample. In the second set (CHUF2), various applied fields (20, 30, and 50 kOe) were used to field cool the sample to 2 K, but the magnetization was measured in the warming cycle using a constant field of 10 kOe. The results of the first set of experiments using $H_c = 50$ kOe (CHUF1) are presented in Fig. 6(a) for $H_c = 50$ kOe and in Fig. 6(b) for $H_c = 10$ kOe. A notable difference in the magnetization profile using the CHUF protocol is the increase in magnetization with an increasing value of H_w. It is also interesting to note that the higher the value of H_c used to cool through T_g, the more responsive is the magnetization roughly below T_g. An anomaly close to T_a becomes more prominent and broadened as H_w reaches 20 kOe. This could imply that some part of the kinetically arrested "glassy" phase is transformed into an equilibrium phase. The results of CHUF2 are presented in Fig. 6(c). It can be noted that, in this protocol, the magnetization remains the same, irrespective of the cooling field until T_a is reached. Below T_a, when $H_c > H_w$, there is an enhancement of magnetization. However, the temperature range measured below T_a is inadequate to conclude with authority about a general trend. The result of a field-cooled magnetization measurement at 500 Oe performed in warming and cooling cycles presents no significant thermal hysteresis [see Fig. 6(d)].

The purpose of our work was to search for magnetometric "signatures" of the spin-liquid phase in the frustrated diamond lattice antiferromagnet exemplified by the A-site spinels. Frustration effects induced by the NNN exchange competition lead to a degenerate ground state of spin spirals in this system. The degeneracy of the ground state may be lifted by the "order by disorder" mechanism or by quenched random disorder. While the spiral spin-liquid phase persists even in the presence of finite weak disorder, a spin-glass or glasslike feature can develop due to the effect of disorder. Our study confirms that $FeAl_2O_4$ consists of a majority phase that is predominantly short-range ordered, such as a spin liquid and a minority phase thats remains "glassy." It is shown that the "glassy" phase transforms under the influence of suitable applied fields, clearly evidenced in the CHUF experiments.

VI. CONCLUSIONS

We conclude that the TRM/IRM "landscapes" of $FeAl_2O_4$ are quite different from those of a canonical spin glass or a superparamagnet, though some similarity to DAFF systems can be assumed. Nonlinear susceptibility and a critical

analysis of higher harmonic terms lead to exponents very similar to those obtained for comparable systems of frustrated magnet classes of materials. However, a scaling relation advised for spin glasses according to the common prevailing understanding of this class of ordering phenomena is not met. Studying the dynamics of spin relaxation using the Cole-Cole formalism, it is found that a broad relaxation is present in $FeAl_2O_4$, as signified by a high value of α. The "irreversible" part of dc susceptibility indicates no sign of a conventional spin-glass freezing. We argue that $FeAl_2O_4$ resides near the spin-liquid–spin-glass boundary in the T-η phase diagram proposed for A-site spinels [13]. This picture is reinforced by the results of the magnetization measurements using CHUF protocols. Our study could be extended to other spin-liquid systems where, using a combination of TRM/IRM responses, Cole-Cole analysis, and the CHUF protocols, one could obtain "maps" of spin-liquid states in frustrated magnets.

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