Identification of a Griffiths singularity in a geometrically frustrated antiferromagnet

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We report the observation of a Griffiths phase in the geometrically frustrated antiferromagnet $DyBaCo_4O_{7+\delta}$. Its onset is identified using measurements of the thermoremanent magnetization, which appears to be superior to conventional in-field measurement protocols for the characterization of the Griffiths phase. Within this phase, the temporal relaxation of magnetization exhibits a functional form which is expected for Heisenberg systems, reflecting the nature of spin interactions in this class of materials. Interestingly, the effective Co^{2+}/Co^{3+} ratio tailored by varying the oxygen nonstoichiometry δ is only seen to influence the antiferromagnetic ordering temperature (T_N), leaving the Griffiths temperature (T_G) invariant.

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I. INTRODUCTION

A Griffiths phase (GP) pertains to the formation of arbitrarily large magnetically ordered regions *within* the global paramagnetic phase, at temperatures exceeding that of long range magnetic ordering. First postulated in the context of random site dilution in Ising ferromagnets [1], this phase is characterized by the magnetization remaining nonanalytical at temperatures above the ferromagnetic Curie temperature (T_C), where the system is neither a pure paramagnet, nor does it it exhibit long range ordering. Analyticity is restored only above the Griffiths temperature T_G, which refers to the Curie temperature of the pristine (undiluted) system. This model was further generalized to account for random magnetic spin systems, where T_G now refers to the highest ordering temperature allowed by the bond probability distribution of that system [2]. The physics of this regime between T_C and T_G is now known to be extremely rich, and has even been extended to quantum phase transitions where these effects are thought to manifest themselves in the form of power laws in thermodynamical observables [3]. This area of research saw renewed interest when it was suggested that the phenomenon of colossal magnetoresistance observed in mixed valent manganites could be understood in the context of a Griffiths singularity [4]. A variety of factors like doping, Jahn-Teller distortion [5], size variance of the A site ions in the ABO_3 structure [6], finite size effects [7], and magnetic site dilution [8] were seen to stabilize a GP in these materials. This phenomena appears to be ubiquitous to a number of strongly correlated electron systems, and materials as diverse as transition metal oxides [5–9], chemically substituted f-electron systems [10], and magnetocaloric intermetallics [11,12] are now reported to harbor such a state.

The probability of a magnetically ordered rare region existing within a global paramagnetic phase would fall exponentially as the volume of this region. Thus the experimental verification of the GP primarily relies on our ability to detect the contribution which this rare region makes to measurable thermodynamic quantities. The most popular route has been to evaluate the inverse of the measured zero field cooled (ZFC) dc magnetic susceptibility $\chi^{-1} = (T - T_C)^{1-\lambda}$, where the nonuniversal exponent λ exhibits positive values less than unity just above the magnetic ordering temperature [4]. Within the GP, this measured magnetic susceptibility is the sum of the true paramagnetic (PM) susceptibility (χ_{PM}) and the susceptibility of the magnetically ordered rare regions (χ_{RR}). When the rare regions are ferromagnetic (FM), $\chi_{RR} > \chi_{PM}$, and a sharp downturn in $\chi^{-1}(T)$ is observed as one approaches T_C from the high temperature side. On the contrary, if these rare regions are antiferromagnetic (AFM), this condition of $\chi_{RR} > \chi_{PM}$ may not be satisfied. Hence, it is not surprising that experimental reports of GPs have been scarce in systems exhibiting a PM to AFM phase transition. The few examples which exist are systems which either have mixed interactions. or have a FM ground state in close proximity to the AFM one. For instance, in the inherently AFM half-doped manganites, a GP was reported to be stabilized by the presence of short range FM superexchange interactions driven by structural distortions [13]. In the spin chain compound Ca_3CoMnO_6 with $\uparrow\uparrow\downarrow\downarrow$ type of magnetic ordering, competing AFM and FM interactions are known to result in a GP [14]. In the magnetocaloric intermetallics of the form $R_5Si_xGe_{1-x}$(with R = Gd, Tb, Dy, or Ho) with competing intralayer and interlayer interactions, local disorder is thought to promote the stabilization of FM short range correlations above the ordering temperature [11]. Very recently, a geometrically frustrated intermetallic system was reported to exhibit a GP in the vicinity of its PM-AFM transition [15]. However, this phase was also reported to originate from *ferromagnetic* clusters arising as a consequence of the inherent nonstoichiometry of the compound. The only notable exception is the site diluted antiferromagnet $Fe_{1-x}Zn_xF_2$, where the presence of a GP was proposed using a linear regression analysis of ac susceptibility data [16].

II. METHODS

Polycrystalline specimens of $DyBaCo_4O_{7+\delta}$ were synthesized by the standard solid state reaction technique using high purity Dy_2O_3, $CaCO_3$, $BaCO_3$, and Co_3O_4 as the ingredients. Stoichiometric amounts of these starting materials were thoroughly mixed and treated at temperatures from 900 to 1150 °C with intermediate grindings. The oxygen nonstoichiometry was varied by quenching the specimens from 1150 °C in different media, and the δ values were determined by idiometric titration. X-ray powder diffraction patterns were

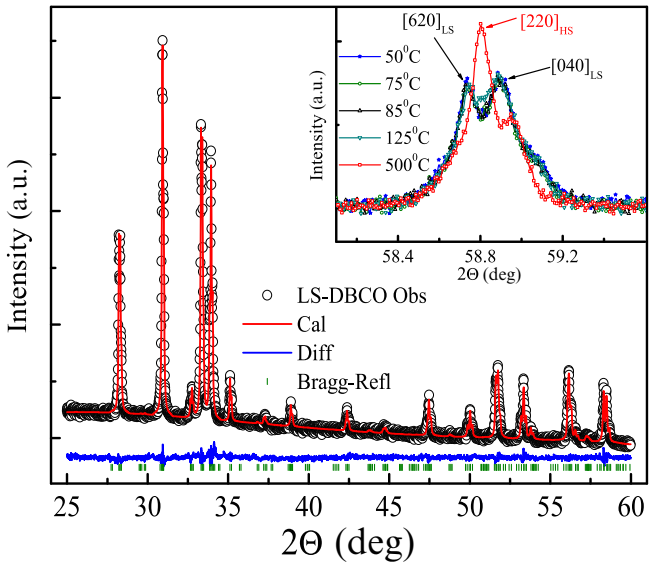

FIG. 1. A Rietveld fit to the room temperature x-ray diffraction data of the low symmetry $DyBaCo_4O_{7.07}$ (LS-DBCO) specimen. The inset depicts the structural transition from a low symmetry orthorhombic $Pna2_1$ phase to a high symmetry trigonal $P31c$ one at temperatures in excess of 125 °C. The subscripts LS and HS refer to the peaks corresponding to the orthorhombic and trigonal structures, respectively.

measured using a Bruker D8 Advance diffractometer with Cu K_α source under the continuous scanning mode, and data was analyzed by the Rietveld method using the Fullprof refinement program [17]. Magnetization measurements were performed using a Quantum Design (MPMS-XL) SQUID magnetometer.

III. RESULTS AND DISCUSSIONS

Antiferromagnetic $DyBaCo_4O_{7+\delta}$, the system under investigation here, belongs to the class of geometrically frustrated antiferromagnets called the *swedenborgites*. This class of systems (of the form $ABaCo_4O_7$, where typically A = Y, or a trivalent rare earth) have attracted attention due to the fact that the magnetic species (Co in this case) is exclusively tetrahedrally coordinated [18]. The structure comprises of alternating triangular and kagome slabs stacked along the crystallographic c axis, and the frustration mandated by the lattice is typically relieved by a temperature driven structural phase transition which drives the system from a high symmetry (trigonal $P31c$ or hexagonal $P6_3mc$) phase to a low symmetry orthorhombic (typically $Pbn2_1$ or $Pna2_1$) phase [19]. We observe that with subtle variation of the synthesis conditions, polycrystalline specimens of $DyBaCo_4O_{7+\delta}$ can be *preferentially* synthesized in either the high symmetry (HS) trigonal $P31c$ or the low symmetry (LS) orthorhombic $Pna2_1$ phase, hitherto referred to as HS- or LS-DBCO, respectively. Figure 1 depicts the x-ray diffraction pattern of the LS-DBCO specimen, and structural parameters as obtained from Reitveld refinement of room temperature x-ray diffraction data is given in Table I. The corresponding details for the HS-DBCO specimen have been reported earlier [20]. The LS-DBCO specimen is seen to exhibit a structural transition from a low symmetry $Pna2_1 \rightarrow$ high symmetry $P31c$ transition at temperatures in excess of 125 °C as shown in the inset of Fig. 1. The influence of the oxygen nonstoichiometry δ on the crystal structure of the swedenborgites is known [21,22], and our HS- and LS-DBCO specimens correspond to δ values of 0.13 and 0.07, respectively.

TABLE I. Structural parameters as obtained from the Rietveld refinement of $DyBaCo_4O_{7.07}$ (LS-DBCO) at T = 300 K; space group: $Pna2_1$, unit cell parameters: a = 10.970(71) Å, b = 6.311(70) Å, c = 10.232(03) Å. Crystal system: orthorhombic, $\alpha = \beta = \gamma = 90^\circ$.

Atom	Wyckoff	x/a	y/b	z/c
Dy	4a	0.66(914)	0.99(700)	0.87(282)
Ba	4a	0.65(968)	0.00000	0.49(975)
Co1	4a	0.98(894)	0.00000	0.94(338)
Co2	4a	0.16(721)	0.00(600)	0.68(519)
Co3	4a	0.08(570)	0.75(109)	0.18(377)
Co4	4a	0.91(407)	0.74(963)	0.69(829)
O1	4a	0.99(800)	0.00(100)	0.25(100)
O2	4a	0.16(140)	0.04(900)	0.49(700)
O3	4a	0.09(930)	0.76(900)	0.99(900)
O4	4a	0.94(010)	0.73(200)	0.49(600)
O5	4a	0.49(300)	0.99(800)	0.24(700)
O6	4a	0.25(600)	0.22(500)	0.78(200)
O7	4a	0.75(200)	0.22(000)	0.22(100)

As shown in Fig. 2, this transition from the high symmetry to low symmetry phase is characterized by a distortion of the perfect kagome layer to one which comprises of three corrugated CoO_4 tetrahedra. With Dy^{3+} having a spin only moment of 10.6 μ_β, the magnetism in $DyBaCo_4O_{7+\delta}$ is plagued by the large paramagnetic contribution of Dy^{3+} ions. However, the onset of magnetic order within the Co sublattice in both the specimens can be clearly identified from conventional ZFC magnetization measurements (Fig. 2), and the antiferromagnetic transition temperature (T_N) for the HS-DBCO and LS-DBCO specimens are seen to be 73 and 63 K, respectively. The lower panel shows temperature dependent measurements of the in-phase component of ac susceptibility $\chi^R{}_{ac}(T)$ for both the HS- as well as the LS-DBCO specimens, where the absence of frequency dependence rules out the presence of a glasslike phase in either of these systems.

Measurements of the dc magnetic susceptibility as a function of temperature at different applied fields reveal telltale signatures of a GP in the LS-DBCO specimen. This is shown in the main panel of Fig. 3 where the inverse of the magnetic susceptibility (χ^{-1}) exhibits a pronounced field dependent downturn on approaching the magnetic phase transition from the high temperature paramagnetic region. As described earlier, this downturn is expected when the susceptibility of the magnetically ordered rare regions is larger than that of the paramagnetic matrix, and application of moderate fields of the order of 1 kOe is sufficient to negate this condition in LS-DBCO. The onset of the GP can be deduced to be from T_G = 76 K, which is very close to the T_N of the high symmetry specimen. Interestingly, a similar measurement protocol does not indicate the presence of a GP in HS-DBCO, as is evident from the inset of Fig. 3.

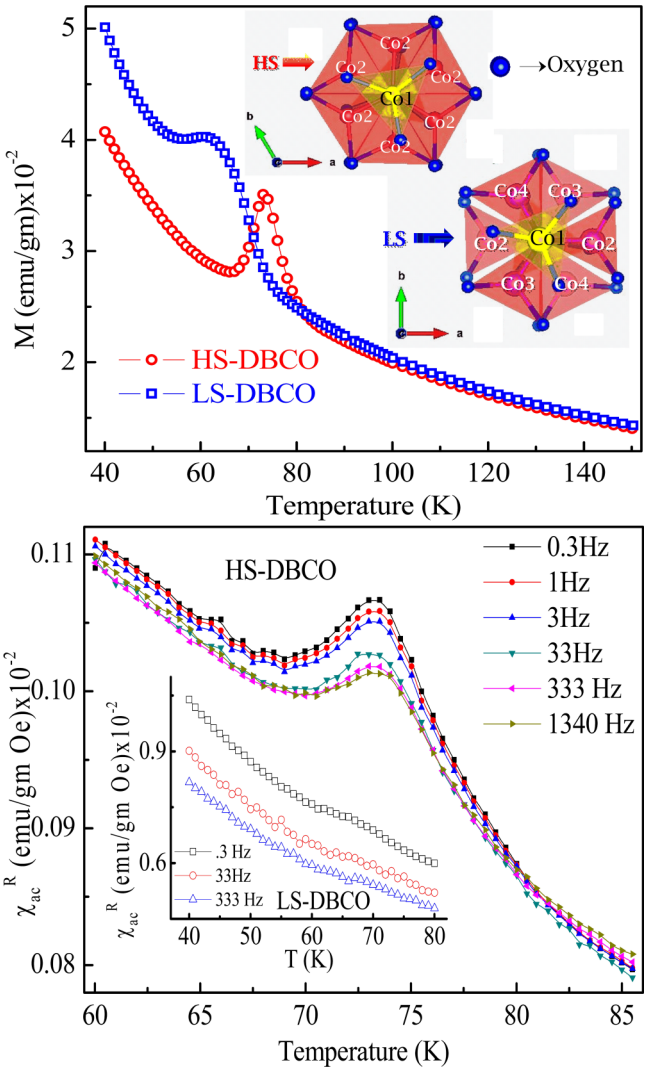

FIG. 2. The zero field cooled dc magnetization of the high symmetry and low symmetry $DyBaCo_4O_{7+\delta}$ specimens (top). The distortion within the kagome layer which distinguishes between the two structures is also depicted. The bottom panel exhibits frequency dependent ac susceptibility measurements on both these specimens, ruling out the possibility of a glasslike state.

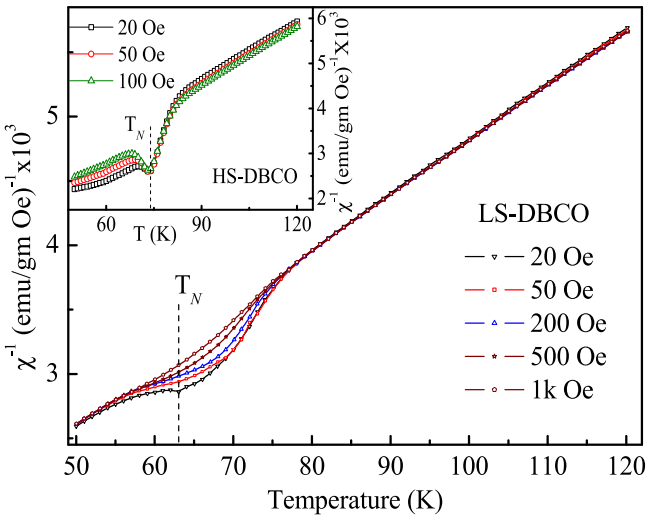

FIG. 3. The inverse of the dc magnetic susceptibility as measured at different applied fields indicating the presence of a Griffiths phase in the low symmetry $DyBaCo_4O_{7+\delta}$. The inset shows the absence of such a phase in the high symmetry specimen.

It is to be noted that for well after Griffiths theoretical work, the possibility of experimentally observing a Griffiths singularity was thought to be remote. Though slow spin dynamics within the paramagnetic phase of the hole doped manganites was inferred from muon spin rotation [23] and noise measurements [24], these spatiotemporal inhomogeneities were not directly connected to the possibility of a GP in these materials. A paradigm shift in experimental investigations of the GP in magnetic systems was the use of magnetic susceptibility measurements to infer on the possibility of a GP in the $La_{0.7}Ca_{0.3}MnO_3$ system [4]. Though subsequent work have used techniques like electron spin resonance [5], specific heat [25], and nonlinear magnetic susceptibility measurements [8] to infer on the existence of a GP, in-field dc magnetic susceptibility measurements continue to be the technique of choice. As described earlier, though this appears to be sufficient in the case of ferromagnetic clusters, its efficacy in the investigation of a GP in a PM-AFM transition is suspect, since the susceptibility of the paramagnetic matrix (χ_{PM}) is likely to be of the same order (or even exceed) the susceptibility of the magnetically ordered rare regions (χ_{RR}).

To circumvent this problem, we re-investigate the GP in the LS-DBCO specimen using the technique of thermoremanent magnetization (TRM). This measurement protocol has been used earlier in the investigation of spin glasses [26], and involves cooling the specimen from well above its magnetic transition in the presence of an applied magnetic field, which is then switched off to zero at $T < T_C$. The magnetization is measured on warming up in this *zero field* condition, and the thermoremanent magnetization $M_{TRM}(T)$ typically exhibits a sharp upturn at the transition temperature. This technique has also found limited utility in the investigation of dilute antiferromagnets, and in the investigation of phenomenalike piezomagnetism [27–29]. Being a zero field measurement, it is likely to be advantageous in investigating the GP since the contribution of the paramagnetic susceptibility to the magnetization is likely to be suppressed in comparison with conventional in-field magnetic measurements. The upper panel of Fig. 4 exhibits $M_{TRM}(T)$ as measured in the LS-DBCO at different cooling fields. A possible signature of the GP is seen in the form of an upturn of susceptibility at temperatures well above the T_N of 63 K. The T_G as inferred from this $M_{TRM}(T)$ measurement is 101 K—well in excess of that inferred from more traditional zero field cooled measurements depicted in Fig. 3, suggesting that in-field magnetic measurements tends to underestimate the true T_G.

The presence of magnetically ordered rare regions within the GP is expected to drastically influence the dynamics of the magnetic susceptibility. In the high temperature paramagnetic limit, the susceptibility would be expected to fall exponentially as a function of time. However, in the Griffiths phase, the time required to reverse the effective spin of large magnetically ordered clusters would be larger, thus resulting in a nonexponential decay of the spin-autocorrelation function [30]. For

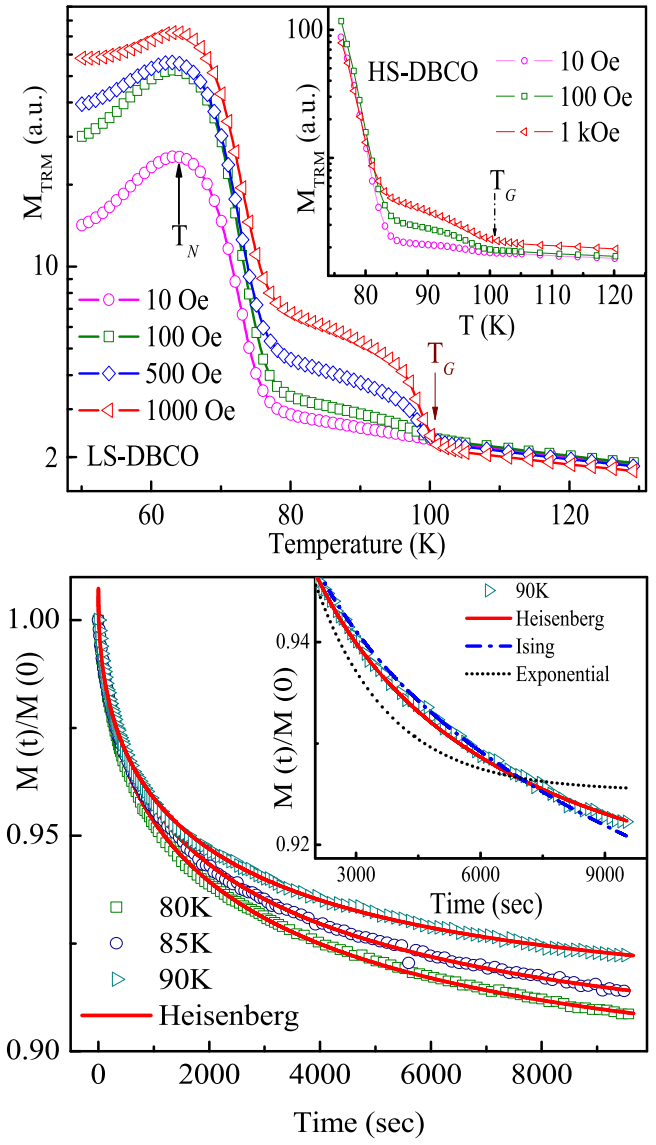

FIG. 4. Measurements of the thermoremanent magnetization (M_{TRM}) as measured for the low symmetry $DyBaCo_4O_{7+\delta}$ specimen at different cooling fields, showing the onset of the Griffiths phase (T_G), and the antiferromagnetic transition temperature (T_N). The inset depicts the onset of the Griffiths phase (T_G) in the high symmetry $DyBaCo_4O_{7+\delta}$ specimen. The lower panel shows the time decay of the isothermal remanent magnetization at $T_N < T < T_G$ for the LS-DBCO specimen, fitted with a $\exp(-Bt^{0.5})$ form expected for Heisenberg spin systems. The inset also shows the relative lack of fitting with an exponential decay and with the form expected for an Ising system. The reduced-χ^2 values for the fits to the Heisenberg, Ising, and exponential decay functions are 7×10^{-7}, 1.3×10^{-6} and 1×10^{-5}, respectively.

randomly diluted ferromagnets, this decay has been calculated to be of the form $\exp[-A(\ln t^{d/d-1})]$ and $\exp(-Bt^{0.5})$ for Ising and Heisenberg spin systems, respectively [31]. This slowing down would be expected to manifest itself in the form of a power law in measurements of the magnetization decay measured as a function of time. The lower panel of Fig. 4 shows measurements of the isothermal remanent magnetization (IRM) for the LS-DBCO specimen within the

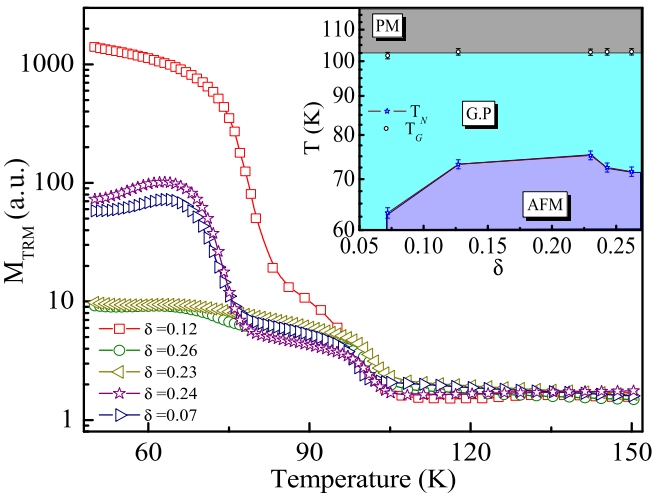

FIG. 5. Measurements of the thermoremanent magnetization (M_{TRM}) as measured for different $DyBaCo_4O_{7+\delta}$ specimens at a cooling field of 500 Oe. The inset shows the relative invariance of the onset of the Griffiths phase for specimens with different oxygen nonstoichiometry δ.

GP regime. These measurements were performed by cooling the system from 300 K at an applied magnetic field of 500 Oe, and measuring the decay of the magnetization as a function of time on switching off the magnetic field. Our measurements indicate that the observed decay follows a functional form expected for systems where the interaction between spins are Heisenberg-like. The lack of agreement with the functional form expected for Ising-like interactions, and an exponential decay is also shown in the inset. Our observation is in broad agreement with prior data, where the nature of spin correlations in the cobalt based swedenborgites was inferred to be Heisenberg-like from neutron scattering experiments [32].

Considering the utility of TRM measurements in identifying the presence of a GP, we re-investigate the HS-DBCO specimen using this measurement protocol. Interestingly, our $M_{\mathrm{TRM}}(T)$ measurements indicate that a GP is *also* present in the HS-DBCO as is shown in Fig. 4, though it could not be detected using conventional ZFC measurements. Moreover, this T_G is also seen to be invariant of the room temperature crystallographic symmetry, and both specimens exhibit similar values of $T_G \approx 101$ K. To further corroborate this observation, we have also synthesized a number of specimens with varying levels of oxygen nonstoichiometry δ with slight modifications of the synthesis conditions. As can be seen from Fig. 5, though the onset of long range magnetic order varies across the different specimens, the onset of the GP is invariant, with all specimens exhibiting a $T_G \approx 101$ K. Thus T_G appears to be relatively insensitive to changes in the Co^{2+}/Co^{3+} ratio, as well as to subtle variations in the crystallographic symmetry. Corroborating evidence in the form of an independent measurement technique would be imperative, to reconfirm the accuracy of T_G deduced from these TRM measurements.

Systems stabilizing in geometrically frustrated motifs are known to have an extended region in phase space, where short range correlations are prevalent. In the swedenborgites, the presence of trigonal bipyramid clusters satisfying the $\sum S_i = 0$ condition have been inferred from prior neutron

scattering experiments. Monte Carlo simulations in the closely related $YBaCo_4O_7$ system have indicated that the nature of spin correlations can be understood by modeling the parameter J_{out}/J_{in}, where J_{out} and J_{in} refer to the exchange interaction strength between the kagome and the triangular layers, and within the kagome layer, respectively [33]. The magnetic diffuse scattering observed above the ordering temperature in $YBaCo_4O_7$ could be modeled by using $J_{out}/J_{in} \approx 1$, which corresponded to quasi-1D magnetic correlations along the c axis. Other members of the swedenborgite family would correspond to different values of J_{out}/J_{in}, with this ratio being determined by factors such as the ionic radii of the A site cation, and the preferential occupation of the kagome and triangular sublattices by Co^{2+} and Co^{3+} ions. The specific experimental signatures evident in our bulk magnetic measurements indicate that the *onset* of these short range correlations could be possibly looked upon as a Griffiths singularity. The fact that T_G is invariant of both the room temperature structure and the oxygen nonstoichiometry δ indicate that these factors do not appear to drastically modulate the effective exchange interactions.

IV. SUMMARY

In summary, we report on the identification of a GP in a geometrically frustrated antiferromagnet $DyBaCo_4O_{7+\delta}$ using bulk dc magnetic measurements. Conventional in-field measurements tend to underestimate the true T_G, and measurements using the TRM protocol outlined here is seen to offer an effective means of characterizing such transitions. The temporal evolution of magnetization is seen to follow a functional form expected for Heisenberg spin systems. Measurements on specimens with varying oxygen stoichiometry δ indicate that the onset of this GP (T_G) is relatively insensitive to variables such as the Co^{2+}/Co^{3+} ratio. The measurement protocol described here would find utility in the detection of GPs in a number of strongly correlated materials. We also expect this work to spur further experimental and theoretical investigations into the presence of Griffith singularities in geometrically frustrated magnets.

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