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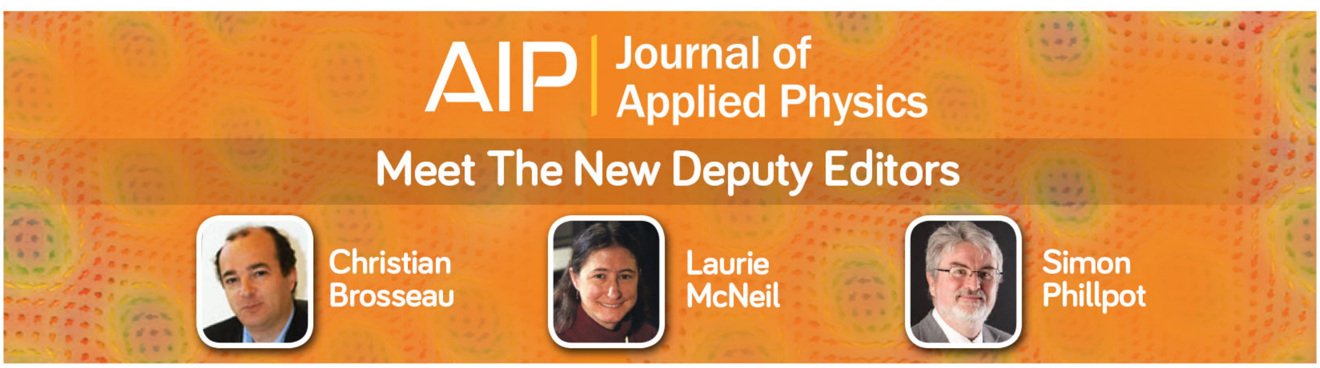

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Evidence for spin glass state of $NdCo_{1-x}Ni_xO_3$ ($x = 0.3-0.5$)

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Low-temperature magnetic properties of single phase $NdCo_{1-x}Ni_xO_3$($x = 0.3-0.5$) have been studied using *ac* and *dc* magnetic susceptibility measurements. Nickel substituted samples have been found to exhibit a different magnetic state at low temperature as compared to pristine $NdCoO_3$. The temperature dependent *dc* magnetization *M* (*T*) revealed the presence of a sharp cusp occurring at characteristic temperatures T_P, for x = 0.3, 0.4, 0.5. Below T_P, clear effect of magnetic field can be seen in *M* (*T*) curves and T_P decreases with increasing magnetic field as well as Ni substitution content. The isothermal magnetization measurements at low temperatures shows small unsaturated hysteresis loop at lowest temperature (10 K). The ac susceptibility results show a clear frequency dependent feature. These results are analyzed to distinguish superparamagnetic and spin glass behavior by using Néel-Arrhennius, Vogel-Fulcher law, and power law fitting. This analysis ruled out the superparamagnet like state and suggests the presence of significant inter-cluster interactions, giving rise to spin-glass like cooperative freezing.
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I. INTRODUCTION

Magnetic oxides with perovskite crystal structure have proven to be a fertile research area for physicists, solid-state chemists, and material scientists, due to the fascinating array of superconducting, magnetic, and electronic properties they exhibit. Perovskite related cobalt oxides have attracted intense interest because of the existence of unique property of spin-state transition[1–5] and the peculiar magnetic ground state of substituted cobaltites.[6–11] The doped cobaltite perovskite oxides, such as $La_{1-x}Sr_xCoO_3$ (LSCO), $Nd_{1-x}Sr_xCoO_3$, have been studied extensively because of the unusual phase competition between ferromagnetism (FM) and spin-glass (SG) or cluster-glass (CG) ground states.[9–11] The $LaCo_{1-x}Ni_xO_3$ system also shows some properties similar to $La_{1-x}Sr_xCoO_3$. The glassy ferromagnetism and giant magnetoresistance (GMR)[12–15] around the metal-insulator transition have been reported in this system, but there are a few studies on this system with other rare earth ion, viz. Nd, Gd, Pr.[16–18] Recently, we have reported on the crystal structure and magnetic properties of polycrystalline $NdCo_{1-x}Ni_xO_3$ samples,[18] prepared by solid state reaction method. These samples are single phase and show an orthorhombic *Pbnm* structure. At low temperature, we have found composition dependent crossover from antiferromagnetic (AFM) to ferromagnetic (FM) interactions. Low temperature FM component in substituted samples has been attributed to the stabilization of Co^{+3} ions in intermediate-spin (IS) state. The FM and AFM interactions are observed to coexist as confirmed by M-H hysteresis. The temperature dependence of the ac magnetic susceptibility and zero field cooled (ZFC) magnetization shows a characteristic maximum which is the signature of blocking/freezing process of the superparamagnetic/spin glass systems.[19–23] So to further understand the nature of this peak, here, we present more detailed data and discussions concerning the low temperature magnetic behavior in the same set of $NdCo_{1-x}Ni_xO_3$ samples.

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II. EXPERIMENTAL DETAILS

A series of single phase $NdCo_{1-x}Ni_xO_3$ ($0 \leq x \leq 0.5$) samples were prepared by conventional solid state reaction method, as described in Ref. 18. For all measurements, the same batch of samples is used as characterized in our previous work.[18] The *dc* magnetization measurements including magnetic hysteresis loop and temperature dependent magnetization with zero field cooling (ZFC), field cooled cooling (FCC), processes were performed at different magnetic fields using physical properties measurement system (PPMS) of Quantum design. The ac-susceptibility (χ') was measured in an ac field of 1 Oe at frequencies of 1.3, 13, 133, and 1333 Hz, using SQUID (Superconducting Quantum Interference Device) magnetometer (Quantum Design).

III. RESULTS AND DISCUSSION

A. DC susceptibility

The *dc* magnetization data for $NdCo_{1-x}Ni_xO_3$ were collected in ZFC and FC modes, at three different magnetic fields, 0.5, 1 and 5 kOe (see Fig. 1). From Fig. 1, it can be

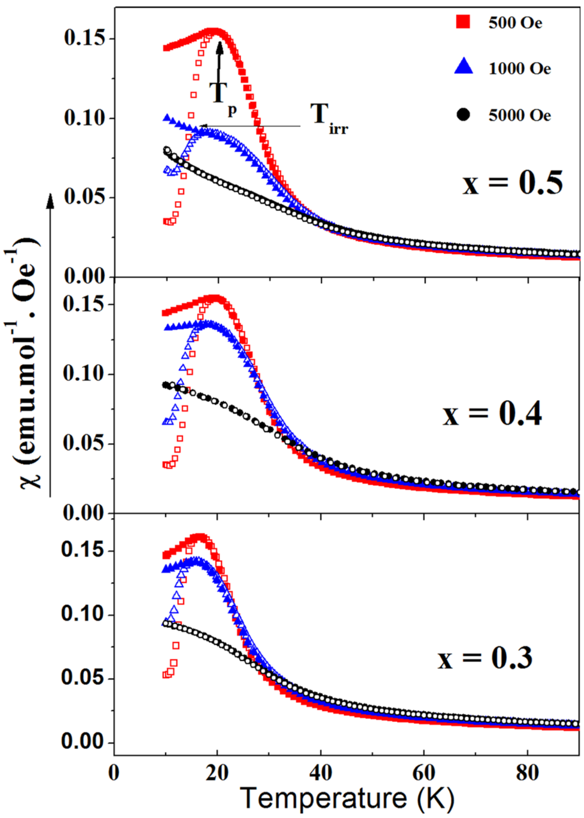

FIG. 1. Temperature dependence of magnetization at different magnetic fields for $NdCo_{1-x}Ni_xO_3$ ($0.3 \le x \le 0.5$). The solid symbols represent the data in ZFC mode and open symbols in FC mode.

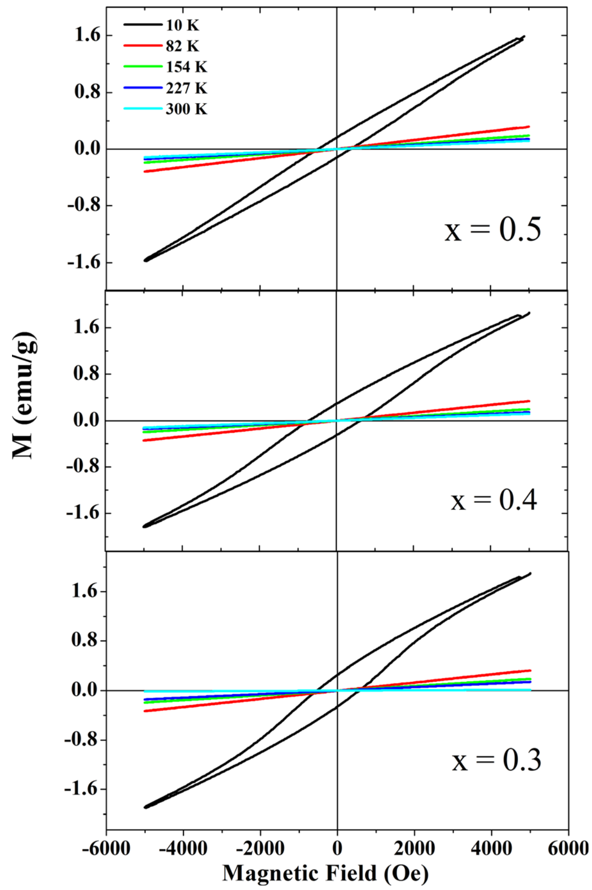

FIG. 2. Isothermal Magnetization hysteresis loops (M-H) of $NdCo_{1-x}Ni_xO_3$ ($0.3 \le x \le 0.5$) at different temperatures.

seen that χ(T) shows strong field dependence. At 500 Oe, one can clearly see broad peak/maxima around a characteristic temperature (T_p) which is called T_p. A clear bifurcation of ZFC and FC curves can be seen at T_{irr} (thermomagnetic irreversibility temperature). Furthermore, with increasing magnetic field, T_p and T_{irr} shifted to lower temperatures and ZFC peak becomes broader and the bifurcation between ZFC and FC curves also decreased. At 5 kOe magnetic field, this peak is completely smeared out and the bifurcation of ZFC and FC curves also disappears, i.e., showing no thermomagnetic irreversibility. This is due to the fact that at a sufficient high field, all the moments can align themselves along the field direction and hence the glassy state disappears. All these features are the characteristics of glassy magnetic state. These different features observed in $NdCo_{1-x}Ni_xO_3$(x = 0.3, 0.4, 0.5) indicate the presence of a spin/cluster-glass or a superparamagnetic state at low temperature.[24–29] At low fields and below T_p the FC magnetization does not show complete temperature independence. This observation is not in agreement with that of canonical spin-glasses and suggests the possibly of cluster-glass or super-paramagnetic state. A similar observation has already been made on the other relatively well studied half doped disordered cobaltites.[10,30,31]

Further evidence of such type of magnetic state can be found by studying the field dependence of magnetization. Isothermal magnetization measurements performed at different temperatures are shown in Fig. 2. It can be seen that above 10 K, magnetization is almost linear with respect to the field. On the other hand at temperature below T_p, i.e., at 10 K, a clear hysteresis loop appears at lower fields and a linear behaviour is seen at high fields. This suggests the presence of a glassy state at low fields, which is due to the competing FM/AFM interactions. Since at high fields the frozen moments tend to orient with the applied field, the glassy state disappears. It is to be noted that the curves at 10 K show non zero remnant magnetization, but there is no saturation even at higher magnetic field. It suggests the presence of a non-saturating (AFM) component. The existence of such non-ferromagnetic component along with the ferromagnetic component is in agreement with the cluster model of other disordered cobaltites, where a number of studies have shown the presence of non-ferromagnetic Co^{+3} matrix with antiferromagnetic interactions that coexist along with ferromagnetic-clusters.[10,30,31]

In order to further investigate the glassy magnetic behavior at low temperature, we have performed *ac* susceptibility measurements at different frequencies. These measurements are used to probe the dynamics of the system at the time scales which are decided by the measuring frequency range.

B. *AC* susceptibility

Fig. 3 shows the temperature dependence of the real, χ'(T) and imaginary, χ''(T) parts of *ac* magnetic susceptibility of the samples at different frequencies in the range of 1.3 Hz–1333 Hz at an *ac* magnetic field of 1 Oe. In Fig. 3,

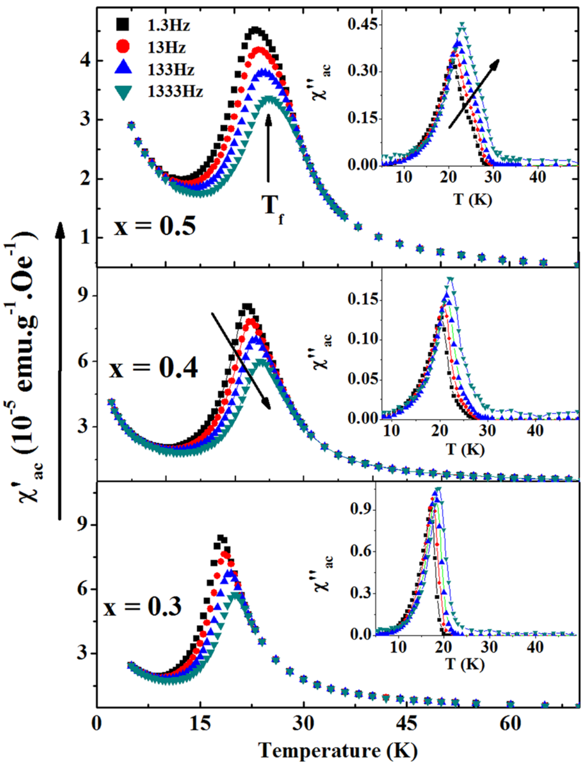

FIG. 3. Temperature dependence of the in-phase and out-of-phase (insets) components of the *ac* susceptibility for $NdCo_{1-x}Ni_xO_3$ ($0.3 \leq x \leq 0.5$), measured at 1.3, 13, 133, and 1333 Hz.

$\chi'(T)$ and $\chi''(T)$ show a peak at a temperature, T_f *(*freezing temperature) which is frequency (*f*) dependent and gets shifted to higher temperature with increasing *f*. This is also a common feature of spin-glass, and super-paramagnetic systems; and the *f* dependence of T_f clearly rules out the possibility of any long range magnetic ordering at low temperature. All these aforementioned properties (*dc* and *ac* magnetization) thus did not completely distinguish the spin-glass and super-paramagnetic nature of the transition at low temperature. So to understand the nature of this peak, we have further analyzed the *ac* susceptibility data with various models.

The peak appearing at T_f, in each $\chi'(T)$ versus temperature plot, brings up to the situation where the measurement time (t) is equal to the relaxation time (τ) of the system. Thus, useful information on the relaxation dynamics can be obtained from the dependence of T_f on the measurement time "t." It is clear from Fig. 3 that with increasing *f* or, equivalently, decreasing time $t = 1/f$, T_f increases. An useful model independent empirical parameter ϕ, can be used to distinguish between the blocking and freezing processes. This represents the relative shift of T_f per decade of *f* on a logarithmic scale[32] and is given by,

$$\phi = \frac{\Delta T_f}{T_f \Delta \log_{10}(f)}.$$

Here, T_f represents the mean value of blocking temperatures corresponding to measuring frequencies and ΔT_f is the difference between T_f measured at frequencies separated by $\Delta(\log_{10} f)$ frequency. The values of ϕ obtained for $NdCo_{1-x}Ni_xO_3$ ($x = 0.3$, 0.4, 0.5) are 0.025, 0.026, 0.027, respectively. Our obtained values are close to those for typical spin/cluster-glasses,[24,33–35] but an order of magnitude smaller than that observed in non-interacting super-paramagnets (0.1–0.3).[21,24,36–39] Therefore, the observed peak in $\chi'(T)$ seems to be associated with interacting magnetic clusters leading the system to be a typical spin-glass (SG) just below the T_f. The possible interpretations of the spin-glass freezing assume the existence of ferromagnetic homogeneous/non-homogeneous clusters embedded in AFM matrix with non-equilibrium freezing.[40] Further, we fit (see Fig. 4) the data to Neel-Arrhenius law given by,

$$\tau = \tau_0 \exp\left(\frac{E_a}{K_B T_f}\right).$$

This yields unrealistically high values for the characteristic fluctuation time scale ($\tau_0 \sim 10^{-31}$, 10^{-38}, 10^{-40} s) corresponding to attempt frequency and for the barrier height ($E_a/K_B = 1240$, 1844, 2079 K for $x = 0.3$, 0.4, 0.5 respectively). This failure of the Arrhenius model also rules out the possibility of superparamagnetism as well as of non-interacting dynamics in the present system and it hints the presence of cooperative dynamics due to inter cluster interactions.

Now by considering the possibility of dynamic blocking of the interacting spin clusters, we have also analyzed the spin dynamics by using the empirical Vogel-Fulcher law.[24,36] For magnetically interacting particles constituting spin-glass system Vogel–Fulcher law was used by Shtrikman and Tholence *et al.*[41,42] According to this,

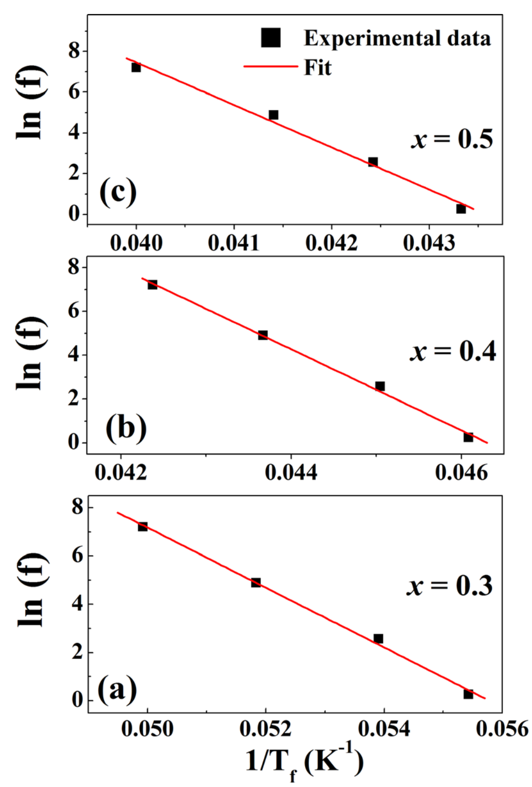

FIG. 4. Plot of ln(f) versusv1/T_f, where T_f is estimated from ac susceptibility data, solid line shows best fit using Arrhenius law for $NdCo_{1-x}Ni_xO_3$ ($0.3 \leq x \leq 0.5$).

$$\tau = \tau_0 \exp\left(\frac{E_a}{K_B(T_f - T_0)}\right),$$

where T_0 is a phenomenological parameter having value between 0 K and T_f. It is often related to intercluster interaction strength. The fitting of above equation to the data, shown in Fig. 5, gives $\tau_0 \sim 10^{-12}$, 10^{-11}, 10^{-8} s, $E_a/K_B = 166$, 203, 56 K and $T_0 = 12$, 15, 20 K for x = 0.3, 0.4, 0.5, respectively.

According to the criterion introduced by Tholence, $T^* = (T_f - T_0)/T_f$ should be very small for spin glass behavior. Our values of T^* are 0.35, 0.33, 0.16 for $x = 0.3$, 0.4, 0.5, respectively. Again these results are an order of magnitude higher than those reported for canonical spin glasses or ideal spin glasses,[24] but still too low for our system to be superparamagnetic ($T^* \sim 1$).[21] These results are intermediate between those of spin glasses and non-superparamagnets and are comparable to the ones obtained for systems displaying evidence of a progressive freezing of clusters.[21,39,43] Also, the ratio of activation energy and T_0 (E_a/k_BT_0) gives a measure of the strength of interactions between the dynamic entities freezing at T_f and hence the level of magnetic clustering where the size of the clusters is assumed to be directly related to the coupling between them.[44] The values of this ratio obtained for our samples are 14, 13, and 2.8, respectively, which also lie well above than those for canonical spin glass systems ($E_a/k_BT_0 = 2$–3),[44] except for x = 0.5. Further the τ_0 value obtained from the Vogel-Fulcher fit is order of magnitude larger than the spin-flip time of atomic magnetic moments ($\sim 10^{-13}$ s). This further suggests that the fluctuating entities are spin-clusters with a significant inter-cluster interaction among them. Such strong inter-cluster interactions can give rise to spin-glass like cooperative freezing. In this case, the frequency dependence of peak in χ'(T) is expected to follow power law divergence of the standard critical slowing down system given by dynamic scaling theory[24,36]

$$\tau = \tau_0 (T_f/T_g - 1)^{-z\nu},$$

where τ is the dynamical fluctuation time scale corresponding to measurement frequency at the peak temperature of χ'(T), τ_0 is the microscopic flipping time for fluctuating entities, T_g is the spin-glass transition temperature in the limit of zero frequency, and z and ν are the critical exponents. In the vicinity of spin-glass transition, the spin cluster correlation length ξ diverges as $\xi \propto (T/T_g - 1)^{-\nu}$ and the dynamic scaling hypothesis relates τ to ξ as $\tau \sim \xi^z$. As shown in Fig. 6, we have obtained a good scaling relation with $\tau_0 \approx 10^{-10}$, 10^{-11}, 10^{-12} s, $T_g = 15$, 19, 21 K, and $z\nu = 13.5$, 12.8, 10.6 for x = 0.3, 0.4, 0.5, respectively. The value of exponent $z\nu$ is comparable to that observed in case of spin-glasses (4–12) and τ_0 is order of magnitude smaller than the values reported

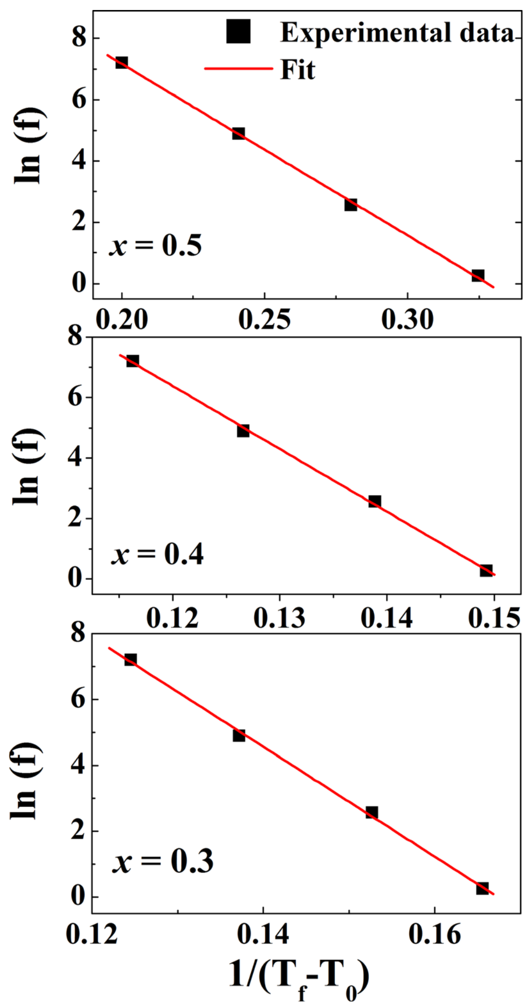

FIG. 5. Plot of ln(f) versus $1/(T_f - T_0)$, solid line shows best fit using Vogel-Fulcher law for $NdCo_{1-x}Ni_xO_3$ ($0.3 \le x \le 0.5$).

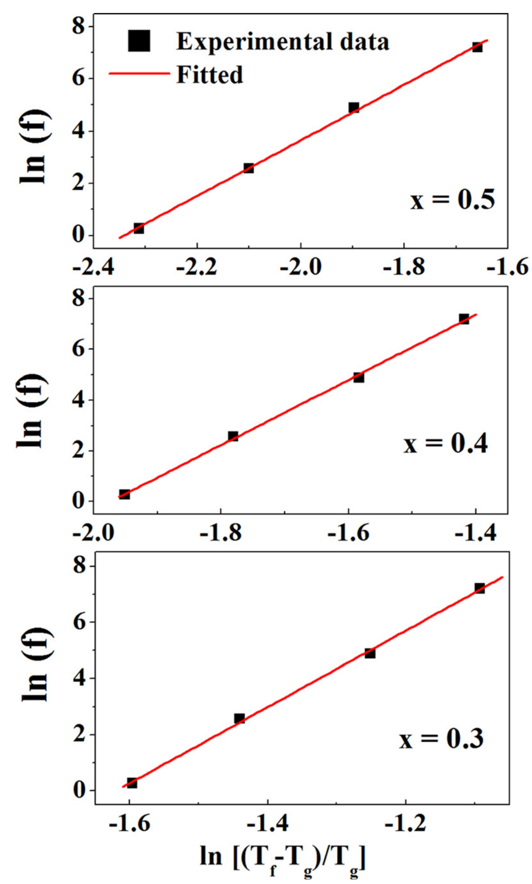

FIG. 6. ln(f) vs ln $(T_f - T_g)/T_g$) for $NdCo_{1-x}Ni_xO_3$ ($0.3 \le x \le 0.5$), demonstrating the agreement with power law. The solid line is a best fit to the data.

for cluster-glass (10^{-9}–10^{-6} s) and is consistent with those for spin-glass (10^{-11}–10^{-13} s). The value of τ_0 is even larger than the spin-flip time of a single atom ($\sim 10^{-13}$ s), which indicates that the spin dynamics in the system exhibit the critical slowing down on approaching T_g (given above) as expected from the dynamic scaling. So, it can be inferred that the inter-cluster interactions present in the system are significant and strong enough to cause a spin-glass like transition. Thus, the glassy nature might be arising from the competing magnetic interactions due to the random distribution of magnetic ions (Ni/Co) at the similar crystallographic sites. The super-exchange between Ni^{3+}-O-Ni^{3+} and Ni^{3+}-O-Co^{3+} gives AFM interactions and antiferro-orbitally ordered (IS) Co^{3+}-O-(IS) Co^{3+} ions gives FM interactions as explained and reported in our previous work.[18] Such a spin glass and/or cluster glass effect, induced by disorder is well known in doped manganites.[45,46] Therefore, our investigations on low temperature magnetic behavior of $NdCo_{1-x}Ni_xO_3$ ($x = 0.3 - 0.5$) suggest that the random and competing magnetic interactions lead to freezing of local magnetic moment of the spin-clusters in random orientations which finally leads to a spin glass.

IV. CONCLUSION

We have presented a detailed study of the low-temperature (T < 100 K) magnetic properties of $NdCo_{1-x}Ni_xO_3$ samples. At T < 50 K, competing inter cluster (FM/AFM) interactions set in, which are associated with the sudden increase in the low-field dc susceptibility as well as with the broad peaks observed in χ'(T) and χ'' (T) at $H_{ac} = 1$ Oe. Below ∼20 K, freezing of the clusters occurs due to enhanced inter cluster frustration, which is evident from the low-temperature frequency-dependence of χ'(T) peak. The clusters formed are randomly distributed and lead to disorder as seen by non-saturation of the M(H) curves. Further, the analysis of frequency dependent peak of ac susceptibility by Néel-Arrhennius, Vogel-Fulcher and scaling law, ruled out the super paramagnet like state and suggests that spin-glass like features are possibly due to significant inter-cluster interactions.

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