Magnetic correlations in subsystems of the misfit [Ca$_2$CoO$_3$]$_{0.62}$[CoO$_2$] cobaltate

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[Ca$_2$CoO$_3$]$_{0.62}$[CoO$_2$], a two dimensional misfit metallic compound, is famous for its rich phases accessed by temperature, i.e., high temperature spin-state transition, metal-insulator transition (MIT) at intermediate temperature (~100 K) and low temperature spin density wave (SDW). It enters into short range SDW phase below $T_{MIT}$ and long range SDW completes at 27 K [Phys. Rev. B 67, 104410 (2003)]. Information on independent role of misfit layers (rocksalt/Ca$_2$CoO$_3$ & triangular/CoO$_2$) in these phases is scarce. By combining a set of complementary macroscopic (DC magnetization and resistivity) and microscopic (neutron diffraction and X-ray absorption fine structure spectroscopy) measurements on pure (CCO) and Tb substituted in the rocksalt layer of CCO (CCO1), magnetic correlations in both subsystems of this misfit compound are unraveled. CCO is found to exhibit glassiness, as well as exchange bias (EB) effects, while CCO1 does not exhibit glassiness, albeit it shows weaker EB effect. Our results show that the magneto-crystalline anisotropy associated with the rocksalt layer acts as a source of pinning, which is responsible for EB effect as well as for localization of spins in triangular (CoO$_2$) layer, giving rise to glassiness in CCO. By combining local structure investigations from extended X-ray absorption fine structure (EXAFS) spectroscopy and neutron diffraction results on CCO, we also confirm that the SDW arises in the CoO$_2$ layer.

Magnetism in misfit cobaltates is a debated topic of investigation although interesting[4]. The misfit structure makes the physics of these systems complex. For example a famous misfit structure, sodium cobaltate (Na$_x$CoO$_2$), offers superconductivity in hydrated form and thermoelectricity with the metallic conductivity[23]. Moreover, the existence of cobalt ion (having spin state variant[13]) in such misfit cobaltates makes the task daunting for the magnetic structure prediction. Besides, another ingredient of complexity is geometric frustration due to triangular lattice CoO$_2$, having edge shared Co ions octahedra in D$_{3d}$ symmetry[5]. In Na$_x$CoO$_2$, sodium content decides the valency of Co ions in the triangular lattice (CoO$_2$) and it shows rich phases with different concentrations of Na e.g. the extreme member, Na$_x$CoO$_2$ (x = 1) is a non-magnetic insulator[14] and for x ~ 0.62, the compound shows the boundary in between the anti-ferromagnetic (AFM) and ferromagnetic (FM) correlations dominant compositions[1]. In the crystal structure of Na$_x$CoO$_2$, the CoO$_2$ layers are separated by the layers of Na atoms and even with the two dimensional structure it has been found that for such structures interlayer and intralayer magnetic interaction have comparable strength[1].

Famous for its thermoelectricity the Ca$_3$Co$_4$O$_9$, more precisely [Ca$_2$CoO$_3$]$_{0.62}$[CoO$_2$] (hereinafter abbreviated as CCO), has two subsystems as intergrowth of one on the other aperiodically. According to the chemical formula it is comparable with x ~ 0.6 composition of Na$_x$CoO$_2$. One can roughly compare the magnetism of the CoO$_2$ layer in both structures, however, in CCO the role of the [Ca$_2$CoO$_3$] layer (having stack of CaO-CoO-CaO with rocksalt structure) is significant, therefore the overall magnetic behavior is unique. The CCO exhibits ferrimagnetic ordering $T_{Ferri}$ ~ 19 K, long range spin density wave (SDW) ordering at $T_{SDW}$ ~ 27 K and short range SDW at ~ 100 K. Many researchers have tried to alter its properties by doping. For example, it is reported that Sr doping at the Ca site weakens the ferrimagnetism and shows AFM correlation[3]. The electron doping at the Co site of rocksalt layer by the trivalent ion doping at Ca site (Y$^{3+}$ & Bi$^{3+}$) diminishes the ferrimagnetism and affects the $T_{SDW}$, which highlights the role of Co valency in rocksalt layer[5]. It is also reported that the SDW in the CoO$_2$ subsystem has oscillating moments in the c direction and motion in the ab plane and, by comparing the results with the doped CCO, it is suggested that SDW can be tuned by doping in the rocksalt layer at the Ca site[3].

Theoretical calculation under generalized gradient approximation (GGA) scheme showed[12] FM and AFM orderings in rocksalt layer. Density functional theory with correlation (U) (DFT+U) framework provides a small moments in CoO$_2$ layer while the main contribution comes from the rocksalt layer[12]. In contrary to GGA and DFT+U, the results of cluster quantum chemical method show[12] that FM and AFM correlations are fluctuating and comparison with magnetism of Na$_x$CoO$_2$ (0.6 ≤ x ≤ 0.7) is valid.

Here we report on the drastic alteration in magnetic properties of the CCO by electron doping at the Co site.
in the rocksalt layer by Tb doping at the Ca site. Doping concentration of Tb is decided on the basis of earlier studies of CCO\textsuperscript{13}. We use the exchange bias, present in both CCO and Tb substituted CCO, as a tool to discern the role of different magnetic lattices. Competition between rocksalt layer $c$ axis magnetism and triangular layer itinerant magnetism has been found as cause of ferrimagnetism. Magnetocrystalline anisotropy associated with the rocksalt layer has been identified as cause of pinning for exchange bias. The Tb doping has been found to change the effect of rocksalt on the SDW in CoO$_2$ via ferrimagnetic interlayer coupling. In CCO, magnetic reflections with incommensurate wave vectors has been observed for a narrow temperature range ($\sim 15$-20 K) which then transfers to commensurate magnetic structure at temperatures below $\sim 15$K. Concomitant with this, an anomaly in the spin phonon coupling in the CoO$_2$ layer (observed via EXAFS) is also observed. Long range incommensurate SDW (ISDW) AFM localizes on further lowering the temperature and results into a glassy phase. Pure (CCO) and Tb doped, Ca$_{2.9}$Tb$_{0.1}$Co$_4$O$_9$ (CCO1) have been synthesized using solid state route, as reported elsewhere\textsuperscript{13}. Phase purity of the samples have been confirmed using X-ray diffraction\textsuperscript{13}. X-ray photoemission spectroscopy (XPS) has been performed using Omicron energy analyzer (EA-125) with Al K\textalpha\ (1486.6 eV) X-ray source. Magnetization measurements were done using a 7T Quantum Design magnetometer (MPMS-3). Isotherms, virgin and full loop $M$ ($H$) have been recorded at various temperatures across the mentioned transitions i.e. $T_{SDW}$, $T_{Ferrit}$ in FC and ZFC modes. Magnetization as a function of temperature $M$ ($T$) at different applied magnetic fields were recorded in FC and ZFC protocols. ZFC relaxation measurements have been done at 5 and 30 K by cooling the sample in zero field down to the desired temperature and, after 100 seconds delay, magnetization have been recorded at 50 Oe for up to 8000 sec. Neutron diffraction patterns have been collected at General Materials Diffractometer (GEM), ISIS facility, UK, in the temperature range 6-110 K. JANA 2006 was used for fitting the neutron diffraction patterns. Extended X-ray absorption fine structure spectroscopy (EXAFS) measurements have been performed at beamline P65 at PETRA III, DESY, Germany. The EXAFS measurements were done in fluorescence and transmission mode at Co K edge (7.7 keV). The sample amount was calculated for 1 absorption length and homogeneously mixed with boron nitride and pressed in a pellet shape. A liquid helium flow cryostat has been used for low temperature EXAFS measurements. Athena has been utilized for data processing. In Artemis, the FEFF and IFEFFIT codes were used to calculate theoretical scattering paths and to fit the experimental spectra, respectively.
First we will discuss the results of CCO. Fig. 1 (a) displays the change in magnetic entropy ($\Delta S_M$) calculated for different applied fields using Maxwell’s equation, $\Delta S_M = \int_a^b \left( \frac{dM}{dT} \right) dH$. A transition is found at $\sim 10$ K which we denote as the $T_{Ferri}$. This reflects the first derivative of M (T) while in literature $T_{Ferri}$ was calculated by the upturn of M(T). Fig. 1 (b) shows Arrott plot (5 to 101 K), representing absence of spontaneous magnetization (no intercept at y-axis). FC magnetic susceptibility (see Fig. 1 (c)) with the bifurcation in FC and ZFC in low field (see inset) indicates presence of magnetic glassiness of some type or presence of magnetocrystalline anisotropy or both together $^{16}$. ZFC relaxation measurements (see Fig. 1 (d)) show the time dependence of magnetization at 5 K but not at 30 K. To confirm the glassiness $^{17}$ we have fitted the ZFC relaxation curve with the stretched exponential function $M(t) = M_o - M_r \exp\left( \left( \frac{t}{t_r} \right)^\beta \right)$ where the value of $\beta$ tells the distribution of barrier and comes $\sim 0.37$ which is close to the value for canonical spin glass $^{17} (\sim 0.42)$. No anomaly has been observed related to the SDW transitions in the magnetization data that shows the insensitivity of DC magnetization to SDW ordering. From the inset Fig. 1 (c) it is observed that the bifurcation exists below $\sim 20$ K for magnetic fields up to $\sim 40$ kOe. For now, we designate the bifurcation as related to the glassiness.

![FIG. 3. For CCO1, (a) Temperature dependent $\Delta S_M$ for different fields. Inset shows the 3D contour plot ($\Delta S_M$-H-T), (b) Arrott plot between 5 to 101 K showing absence of spontaneous magnetization, (c) field cooled susceptibility as a function of temperature, measured at various fields. Inset shows the 3D plot (\chi-H-T), showing the absence of bifurcation between FC and ZFC. (d) ZFC relaxation curves measured at 5 and 30 K under the magnetic field of 50 Oe.](image)

Interestingly, we have observed the exchange bias (EB) in CCO at 5 K, +70 kOe FC with magnitude $H_{EB} \sim 1.7$ kOe and coercivity $H_c \sim 5$ kOe, calculated using $H_{EB} = (H_{c1} + H_{c2})/2$ and $H_c = (|H_{c1}| + |H_{c2}|)/2$, respectively. Here $H_{c1}$ and $H_{c2}$ are the coercive fields in negative and positive field side, respectively. The magnitude is considerably large, however one has to authenticate the existence of it. Fig. 2 (a) shows the M (H) hysteresis measured in ZFC, +70 kOe FC and -70 kOe FC at 5 K. The loop shifted to negative and positive directions for cooling in positive and negative fields, respectively. This is according to conventional EB system $^{18}$. The cooling field dependence (see Fig. 2 (b)) at 5 K and temperature dependence at +70 kOe has been observed (see Fig. 2 (c)). These trends also match with the conventional EB cases $^{19,20}$ (see Fig. 2 (d & e)).

For conventional EB systems with AFM and FM layers with the strong interfacial coupling the $H_{EB}$ is defined as $^{20} H_{EB} = -J S_{AFM} S_{FM} \mu_{FM} M_{FM}$ where $J$ represents the coupling strength across the interface, $S_{FM/AFM}$ is the interface magnetization of FM/AFM phase and $\mu_{FM}$ & $M_{FM}$ are the thickness and bulk magnetization of FM layer. From this relation, it is clear that $H_{EB}$ will increase with the increase in interfacial FM, which increases with the cooling field (H$_{CF}$) due to spin alignment in field direction. Although, the more enhancement in the
H_{CF} results in the increase in the cluster size (decrease of S_{FM}) and enhances the bulk magnetization, M_{FM} therefore reducing the H_{EB}. Moreover, for the phase separated systems, with FM clusters in the SG matrix, the above situation is also observed, but the effect of magnetic field on the glassy phase has to be considered, which usually diminishes with the applied field.

The important and unusual observations in the present case are the suppression of EB for temperature \( \gtrsim 15 \) K (see Fig. 2(d)), the non saturation behavior of H_{EB} up to 70 kOe (see Fig. 2(e)) and the suppression of bifurcation in field above \( \sim 40 \) kOe (see Fig. 1(c)). These open the question about the origin of EB, because if the glassiness is considered as the origin of pinning then it should vanish for field above \( \sim 40 \) kOe, which is not the case here.

Before making any comment on the origin of EB we will discuss the results of CCO1 (Tb doped CCO). For the doping at the Ca site, the chemical formula can be written as [Ca_{1.959}Tb_{0.041}Co_{0.50}O_{2.02}]Co_{0.22}. Tb^{3+} is a magnetic ion with the total spin moment \( S = 3 \) (4f^8) with the theoretical paramagnetic moment \( \sim 9.72 \) \( \mu_{B} \).

Fig. 3(a) shows the temperature dependent \( \Delta S_{M} \) at various fields and (b) shows the Arrott plot, while (c) and (d) displays the \( \chi \) and isothermal ZFC relaxation, respectively. The magnitude of moment is larger for CCO1 because of the paramagnetic contribution from the Tb. No clear transition of any type is observed in the \( \Delta S_{M} \), which means that the Tb destabilizes the ferrimagnetism. Arrott plot is similar to that for CCO, i.e. \( M_{s} = 0 \). Moreover, there is no bifurcation and the related relaxation is also not present. These observations indicate that there is no glassiness in the CCO1 and it should not behave like CCO, i.e. it should not possess EB. We have carried out the same set of magnetization measurements as done for CCO, shown in Fig. 3 (a-e). Counterintuitive, this system, CCO1, also exhibits the EB albeit with lower strength (H_{EB}) and with lower coercivity (H_c). In H_{EB} plot as a function of cooling field H_{CF}, no saturation or decreasing trend has been observed up to 70 kOe, similar to CCO. Recalling literature, for CCO it has been observed that magnetization measured along the c-direction is higher (with hysteresis in M(H)) than that measured in the ab plane (very small hysteresis). This means Tb^{3+} doping decreases the c axis magnetic component in host CCO.

In order to know what is happening at microscopic scale, photoemission spectroscopy measurements were carried out on both the samples, CCO and CCO1. Fig. 5 (a & b) display the Co 2p and O 1s XPS results for CCO and (c & d) show the corresponding for CCO1. The estimated fraction of Co^{3+} in CCO is \( \sim 68 \) % while for CCO1 it is \( \sim 69.5 \) %, calculated from the peak ratio. This clearly indicates that the Tb substitution increases the Co^{3+} in the system. Similar observation has been made by other groups for high concentration of Tb doping.

The observations from CCO1 indicate that the origin of bifurcation in CCO may come from the magnetocrystalline anisotropy and it should follow the equation suggested by Joy et al.\(^{[12,13]}\) given as \( M_{FC} = \frac{H_{app} - H_{app}^{'}}{\chi_{FC}} \approx M_{app} = \frac{H_{app}}{\chi_{ZFC}} \). Fig. 6 (c-k) shows that bifurcation appears because of the glassy phase and not because of the magnetocrystalline anisotropy, which get suppressed for higher fields (\( > 5 \) kOe). This explains the reason for large hysteresis in CCO, i.e. glassy phase, while CCO1 does not exhibit it. However, the existence of EB in both samples indicates that the origin of EB is not the glassiness. Therefore, we propose that the Tb doping directly affects the magnetocrystalline anisotropy, which indirectly suppresses the glassiness. For, ingredient of EB\(^{13}\), we have AFM SDW in the CoO$_2$ layer\(^{23}\) which is common in both the samples.

The SDW generally appears as an AFM ordering in low dimensional metallic systems. This is simple fact by which one can distinguish the AFM (localized insulating) and SDW (metallic systems). SDW is itinerant but can show the similar behavior as showed by localized helical or cycloidal ordered systems. These orderings can be described by the orientation of spins S in all directions (S_x, S_y and S_z) having same magnitude |S|, while for the SDW the direction remains the same (S_x or S_y or S_z) but the magnitude have an oscillatory behavior\(^{23}\). The rocksalt layer possesses FM because of mixed valency (Co^{3+} and Co^{4+}). Further the low spin state (LSS) Co^{4+} (S = 1/2, \( \widehat{L} = 1 \)) can offer the magnetocrystalline anisotropy through spin-orbit coupling (SOC\(^{23}\)). As a result of Tb doping at the Ca site, the amount of Co^{4+} decreases and hence the anisotropy too.
FIG. 6. Curie-Weiss fits to the temperature dependent inverse susceptibility ($\chi^{-1}(T)$) data of (a) CCO and (b) CCO1. (c-k) show the comparison between $\chi_{ZFC}$ (filled symbols) and calculated $\chi_{FC}'$ (empty circles) for the CCO (see text). Yellow background plots belong to CCO.

TABLE I. Various parameters obtained from resistivity and magnetization measurements for CCO and CCO1.

| Parameter | CCO   | CCO1  |
|-----------|-------|-------|
| $\Delta$ (K) | 14.63 K | 23.55 K |
| $\theta_P$ (K) | -73 | -22 |
| $\mu$ ($\mu_B$/Co) | 0.14 | 0.131 |
| $T_{MIT}$ (K) | 90 | 120 |

This also explains the low temperature shifting of $T_{Ferri}$, as a result of the decrease in the $J$ value associated with the FM interaction ($J \propto k_B T$). The schematic shown in Fig. 7 represents the above mentioned hypothesis that CoO$_2$ exhibits SDW with spins arranged in the wave pattern (red arrow), the green arrows represent their effective moment. In the rocksalt layer the spins (blue arrows) are arranged in FM manner, which is coupled with the crystal c axis via SOC. The effective moments, as a sum of these two contributions result as ferrimagnetic spin (yellow arrows) ordering. The EB is the result of the coupling between these two layers. The reason for the glassiness will be discussed in the following.

Fig. 8 shows the profile matched neutron diffraction pattern measured at 17 K. The crystal structure of CCO can be indexed using superspace group $C2/m(0q0)00$ which is marked as phase 1 having unit cell parameters as $a = 4.83$ Å, $b = 4.56$ Å, $c = 10.83$ Å, $\beta = 98.13^\circ$ and $q = (0, 1.612, 0)$. Phases 2 and 3 represent the individual subsystems ([Co$_2$CoO$_3$] & [CoO$_2$]), respectively, each of them modulated with the magnetic propagation vector, $q_{mag} = (0.481, 0.377, 0.0015)$, obtained using the k-search software. Similar three components modulation was observed for the well known SDW material (TMTSF)$_2$ PF$_6$. The phase 2 have lattice parameters as $a = 4.83$ Å, $b_1 = 4.56$ Å, $c = 10.83$ Å and $\beta = 98.13^\circ$ with $q_1 = q_{mag} = (0.481, 0.377, 0.015)$, and phase 3 have $a = 4.83$ Å, $b_2 = 4.56/q = 2.819$ Å, $c = 10.83$ Å and $\beta$
σ = 98.13° with \( q_2 = (0.481, 0.377^* q, 0.015) \). The magnetic modulation is quite complex, as from C2/m symmetry only the P1 space group is allowed (found using MAXMAGN program\(^{20}\)). From the propagation vector it is clear that the moments are propagating in the \( ab \) plane. Using the magnetization data and reported data on single crystal\(^{22}\), the scenario should be similar to the schematics shown in Fig. 7. Interestingly, from the contour plot it is observed that the intensity of magnetic peaks are considerable at \( \sim 16 \) K and disappears at lower temperatures (see inset (b)).

On closer look at nuclear peak intensities, it is observed that the intensity from the magnetic modulation is quite complex, as from C2/m symmetry only the P1 space group is allowed (found using MAXMAGN program\(^{20}\)). From the propagation vector it is clear that the moments are propagating in the \( ab \) plane. Using the magnetization data and reported data on single crystal\(^{22}\), the scenario should be similar to the schematics shown in Fig. 7. Interestingly, from the contour plot it is observed that the intensity of magnetic peaks are considerable at \( \sim 16 \) K and disappears at lower temperatures (see inset (b)).

On closer look at nuclear peak intensities, it is observed that the intensity from the magnetic peaks shift to the nuclear peak (see inset (a)). This indicates the change of the incommensurate magnetic structure to \( q_{mag} = 0 \). A very similar feature was observed\(^{23}\) for CeRu\(_2\)Al\(_2\)B, where incommensurate SDW AFM changes to FM within a very short temperature window (\( \sim 1.4 \) K).

It has been reported\(^{31}\) that the Co-Co correlation in the \( CoO_2 \) results in an anomaly in the mean square relative displacement (MSRD) related to this pair, i.e. \( \sigma^2_{Co-Co} \). We have fitted the EXAFS data using a standard protocol\(^{22}\) by assuming the first shell parameters as temperature independent, as observed previously\(^{31}\), and only iterate the second shell parameters. We observe an anomaly in the \( \sigma^2_{Co-Co} \) at temperature \( \sim 15 \) K, as shown in Fig. 8 (a), which matches with the magnetization upturn. This result supports the spin-phonon coupling in the \( CoO_2 \) layer. This type of observation has been made earlier also by temperature dependent Raman scattering\(^{23}\). This type of strong spin-phonon coupling observation and the AFM ordering via neutron diffraction confirms that the AFM SDW is originating from the \( CoO_2 \) only. Moreover, below \( 15 \) K, the spins in \( CoO_2 \) tend to localize to their atomic sites (\( q = 0 \)) in arbitrary directions, which is the reason of glassiness. Similar pinning of SDW and resultant glassiness has been observed\(^{24}\) also for \( Ni_{0.71}CoO_2 \). Based on our observation, we arrive at a plausible explanation that the internal field generated by the rocksalt layer in the \( c \) direction is the responsible for the \( CoO_2 \) spins localizing, which is weaker in the CCO1 hence \( T_f \) shifts to zero.

To further investigate the effects of Tb substitution it will be informative to look at the transport results. Fig. 9 (b) comprises temperature dependent resistivity of both the samples, showing the shift of \( T_{MIT} \) towards higher temperature with the Tb doping. The magnitude of resistivity of CCO1 is found larger compared to CCO. We have fitted the curves with the activated behavior (not shown here) using the relation \( \rho = \rho_o \exp (\Delta/k_B T) \), as suggested in earlier report\(^{33}\) and found a significant enhancement in the band gap, \( \Delta \) (see Table I). This shows that the doping in the rocksalt significantly affects the overall band structure and hence also to the SDW gap. The \( T_{MIT} \) in general is directly related with the correlation via the relation \( k_B T_{MIT} = 1.14 \rho_o e^{\Delta / k_B T} \), where \( \lambda_e = U n(E_F) \) is the electron-electron coupling constant\(^{15}\). Using the parameters from Table I and assuming the same density of states, \( n(E_F) \), for CCO and CCO1, one can see the relatively large \( U \) in case of CCO1. It is to be noted that large \( U \) links to more localization and less AFM exchange (\( J \propto \frac{1}{e^{\Delta / k_B T}} \)) between spins. Interestingly, this scenario is in accordance to the low value of \( \theta_R \) obtained for CCO1. However, the above mentioned argument is based on the assumption that both the samples (CCO & CCO1) have same nesting vector/magnetic vector (\( q \)) (i.e. same DOS), while in reality the position of the Fermi level controls the \( q \).

In conclusion, we have studied the pure and Tb substituted CCO by means of DC magnetization, neutron diffraction, XPS, EXAFS and resistivity measurements. Exchange bias has been observed in both the samples. Glassiness has been found as the origin of larger hysteresis in CCO than in CCO1. Interlayer coupling between triangular (\( CoO_2 \)) and rocksalt (\( Ca_2CoO_3 \)) has been attributed as the reason behind ferrimagnetism. Magnetocrystalline anisotropy in rocksalt layer acts as pinning for EB. Neutron diffraction and EXAFS results combinedly hints that incommensurate SDW is present in triangular layer, which tends to localize at \( T_{Ferri} \) and gives rise to glassiness. Weaker AFM correlation observed in CCO1 is substantiated by increased correlation effects as manifested from electrical transport data, highlighting the intricate relation between magnetism and electron correlations in these samples.

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