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Anomalous magnetic behavior and complex magnetic structure of proximate LaCrO_3 – LaFeO_3 system

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Abstract:

We investigated complex magnetic properties of multifunctional LaCrO_3 - LaFeO_3 system. The magnetic measurements substantiate the presence of competing complex magnetic ordering against temperature, showing paramagnetic to ferrimagnetic transition at ~ 300 K, followed by antiferromagnetic (AFM) transition near ~ 250 K superimposed on ferrimagnetic phase. The onset of weak ferrimagnetic ordering is attributed to the competing complex interaction between two AFM LaCrO_3 - LaFeO_3 sublattices. The low-temperature AFM ordering is also substantiated by temperature-dependent Raman measurements, where the intensity ratio of 700 cm^{-1} Raman active mode showed a clear enhancement with lowering the temperature. The non-saturating nature of magnetic moments in LaCrO_3 - LaFeO_3 suggests the predominating AFM ordering in conjunction with ferrimagnetic ordering between $250\text{ K} - 300\text{ K}$ up to 5 T magnetic field. A complex magnetic structure of LaCrO_3 - LaFeO_3 is constructed, emphasizing the metastable magnetic phase near room temperature and low-temperature antiferromagnetic state.

Keywords: Magnetism; proximity effect; ternary magnetic oxides, Raman Spectra, transition temperature; ferrimagnetism; antiferromagnetism.

Introduction:

The multifunctional materials, especially complex oxide materials, are not only attracting attention due to their potential but also providing a rich understanding of the fundamentals, which allows designing novel materials with desired functional properties. LaTMO₃ (TM = Transition Metal) is one such family of complex oxide materials, having a perovskite structure with TM magnetic ions. Direct or indirect cross-coupling among spin, orbital, lattice and charge degrees of freedom provide an avenue for potential application and fundamental study in these materials. The canted spin structure of TM ions also exhibits competing magnetic interaction and thus, giving rise to the intricate magnetic structures. LaCrO₃ and LaFeO₃ oxide systems exhibit antiferromagnetic transitions at ~ 290 K and 740 K respectively [1-3, 4, 5] and more interestingly, also exhibit weak ferromagnetisms near room temperature [6-9]. The search of more than one ferroic ordering in oxide systems is always attracting attention for their potential in a new class of electronic devices such as four state memories, voltage-controlled magnetic switches and sensors, electric field controlled spintronic devices. The recent studies provide evidences about the room temperature magnetoelectric coupling in LaCrO₃ [6] and probably the ferroelectric ordering in LaFeO₃ perovskite systems [5]. In spite of magnetic and magnetodielectric properties, these oxide systems exhibit enhanced oxygen ionic conductivity and their electronic conductivity can be tailored by manipulating the suitable dopant at different cation sites. For example, the calcium doping at La site in LaCrO₃ makes it highly conducting and is a potential candidate for high-temperature solid oxide fuel cell electrode material. The distorted TMO₆ octahedra in LaTMO₃ systems is the driving cause for the complex physical properties such as variation of transition temperature, strong electron-phonon coupling, weak ferromagnetism, electrical conductivity by manipulating the exchange and hopping strengths [10]–[12]. The order and amplitude of such changes in physical properties are associated with the degree of distortion.

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3 The divalent doped lanthanum manganite system shows charge ordering, which is closely
4 related to antiferromagnetic phase, while charge delocalization, i.e. metallic state, coincides
5 with ferromagnetism. The screened potential energy becomes large in certain TM oxide
6 materials, due to the various external factors (doping, temperature, pressure, etc), causing
7 electron localization and thus, inhibiting the electrical conduction. This is known as Mott –
8 transition in such transition metal-based perovskite systems [13], [14]. In addition, the hole-
9 doped La_2CuO_4 antiferromagnetic becomes a high-temperature superconductor because of
10 the strong electron-electron correlation. These systems provide a wide avenue to understand
11 the underlying physics and related mechanism governing such functional properties and their
12 possible tunability. The double perovskite materials with and without transition metals are
13 gaining attention for designing multifunctional material systems [15] [16]–[18]. Way back in
14 the 1960's, Goodenough-Kanamuri predicted that double perovskite $\text{La}_2\text{CrFeO}_6$ should have
15 a ferromagnetic ground state with T_c close to room temperature [19]–[21]. Since then a lot
16 more efforts are made experimentally and theoretically with varying conclusions. Some
17 concluded ferromagnetic ground state [20] while others ferromagnetic [21]. This prompted
18 us to explore $\text{La}_2\text{CrFeO}_6$ double perovskite while synthesizing LaCrO_3 - LaFeO_3 system. This
19 system is usually showing intermediate properties of both perovskites, however, some unique
20 magnetic and optical phonon properties are also noticed due to close proximity of these two
21 predominantly antiferromagnetic compounds. It is a fundamental challenge in materials'
22 design to control and understand the change in materials behavior in close proximity to other
23 materials under varying external conditions. In the present work, we investigated the effect of
24 spin ordering on the magnetic and electronic properties of closely proximated LaCrO_3 -
25 LaFeO_3 system.

Experimental Details:

Samples were synthesized by solid state reaction route from oxide precursors, La_2O_3 , Cr_2O_3 and Fe_2O_3 (from Alpha Aesar with 99.9 % purity). To remove moisture La_2O_3 was pre-heated at 800 °C and grounded thoroughly in a stoichiometric ratio; 2:1:1:: La_2O_3 : Cr_2O_3 : Fe_2O_3 to homogenize the pre-synthesized materials. This homogeneous material was heated at 950 °C for 48 hours with intermediate grinding to ensure the homogeneity of a solid solution. For structural and phase identification, powder X-ray diffraction (XRD) data of the samples were recorded using a PANalytical X'Pert Pro X-ray diffractometer with $\text{Cu K}\alpha$ (1.5406 Å) radiation in the range of 20°-90° in steps of 0.017°. DC magnetic measurements were performed using a vibrating sample magnetometer as an attachment in Physical Property Measurement System (Model 6000, Quantum Design, USA) in the temperature range of 100–350 K. Magnetization measurements were performed as a function of temperature in zero-field cooled (ZFC) and field cooled (FC) modes. Various magnetization isotherms were recorded at different temperatures up to an applied magnetic field of 50 kOe in the vicinity of magnetic transitions. Raman spectra were recorded for LaCrO_3 , LaFeO_3 and proximate LaCrO_3 - LaFeO_3 at room temperature with the help of 532 nm green laser source (Nd: YAG) using confocal Raman microscope (WITec Alpha300 R) in backscattering geometry. However temperature-dependent Raman spectra were also recorded for LaCrO_3 - LaFeO_3 system down to 100 K in order to understand the spin-phonon coupling if any, following the magnetic behavior. For this a Jobin-Yvon Horiba Triax 550 spectrometer with a liquid-nitrogen cooled CCD detector while 514.5 nm argon ion laser source is used for excitation. The scattered light collected with a microscope objective that focused on the entrance slit of the spectrometer with a 1200 line/mm diffraction grating.

Results and discussion:

The phase identification of synthesized materials was confirmed using X-ray diffraction and the respective diffractograms are shown in Fig. 1 in conjunction with LaCrO_3 and LaFeO_3 perovskite structure to understand the phase evolution of LaCrO_3 - LaFeO_3 system. The XRD patterns, Fig. 1 (lower and middle panel), confirm the phase purity of pristine LaCrO_3 and LaFeO_3 bulk materials and results are consistent with the reported literature [6], [7], [22]. Observed peak positions are in agreement ICDD card No 330701 and representative (h k l) planes are marked for LaCrO_3 system. LaCrO_3 and LaFeO_3 both systems crystallize in a distorted orthorhombic perovskite system with almost similar lattice parameters [2, 22]. It is important to note that for LaCrO_3 , the highest intensity peak (121) is observed at 32.686° and for LaFeO_3 at 32.299° whereas for LaCrO_3 - LaFeO_3 composite the observed peaks at 32.329° and 32.686° , correspond to LaFeO_3 and LaCrO_3 systems, respectively. The difference observed in the peak shift is not very significant and thus, inconclusive in studied resolution of X-ray diffraction measurements. The structure consists of corner shared tilted TMO_6 (TM = Cr, Fe) octahedra. The structural and magnetic details as reported earlier suggest that the tilted octahedral may induce non-collinearity in the spin structure, giving rise to the weak ferromagnetism in these systems [2], [6]. The XRD diffractogram, Fig. 1 (upper panel), can be visualized as the superimposed XRD spectrum of these pristine materials. These closely spaced doublets confirm the formation of mixed-phase LaCrO_3 - LaFeO_3 system without any additional impurities.

The room temperature Raman spectrographs of these systems are shown in Fig. 2 and analyzed to understand the microscopic phase evaluation for LaCrO_3 - LaFeO_3 system. The factor group analysis of orthorhombic (Pnma space group) suggests that there are 24 ($\Gamma = 7A_g + 5B_{1g} + 7B_{2g} + 5B_{3g}$) Raman active modes in this distorted perovskite LaCrO_3 and detail of mode assignments are given in references [6], [23], [24]. The identical crystallographic

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3 structure of LaCrO_3 and LaFeO_3 , space group Pnma , gives rise to the similar vibrational
4 modes for both systems with a small deviation for different atomic masses of Cr and Fe
5 atoms. Thus, it was difficult to separate out the vibrational contribution of one from other, as
6 evident in XRD graphs, Fig. 1, as well as from Raman spectra, Fig 2. The temperature-
7 dependent Raman spectra are shown in Fig 2, where some of the Raman active modes in
8 proximate LaCrO_3 - LaFeO_3 system show peculiar temperature dependence as compared to
9 pristine systems. After careful analysis of first and second-order optical phonon $\text{B}_{2g}(1)$
10 modes, it is observed that intensity ratio (I_1/I_2) of these modes shows a sharp increase near the
11 second magnetic transition, as shown in Fig 2(b). This increase in the intensity of first-order
12 $\text{B}_{2g}(1)$ mode as compared to second-order mode near magnetic anomaly indicates a spin-
13 dependence of optical phonon mode in Raman scattering that can be a manifestation of
14 electron transfer with lattice vibrations and/or anisotropic exchange interactions.
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33 LaCrO_3 and LaFeO_3 materials are known antiferromagnetic with Neel temperature 290 K and
34 710 K, respectively [6], [9], [25]. The tilted Cr/Fe- O_6 octahedra lead to the canted TM
35 electron spins and thus, causing weak ferromagnetism in these systems. In conjunction with
36 the observed weak ferromagnetism in these systems, the near room temperature
37 magnetodielectric coupling has also been reported in both systems. Considering the complex
38 magnetic interactions in pristine systems, DC magnetization as a function of temperature
39 from 350 K to 100 K has been recorded under zero-field cooled (ZFC) and field cooled (FC)
40 condition at 1000 Oe for LaCrO_3 - LaFeO_3 system. The measured temperature-dependent
41 magnetic moment is shown in Fig. 3. The observed sudden rise in magnetization near 290 K
42 in this sample is due to the antiferromagnetic ordering of LaCrO_3 , superimposed with weak
43 ferromagnetism because of spin canting. The proximate presence of LaFeO_3 tries to reorient
44 the magnetic spin structure of LaCrO_3 sublattice along the weak ferromagnetic structure of
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3 LaFeO₃, causing relatively larger ferromagnetic component below 290 K. This ferromagnetic
4 state of LaCrO₃-LaFeO₃ system preserves down to 250 K, after that the antiferromagnetic
5 ordering of LaCrO₃ starts dominating. This LaCrO₃ antiferromagnetic dominance, leads to
6 another antiferromagnetic transition for LaCrO₃-LaFeO₃ system. Thus, two magnetic
7 transitions are observed clearly at 290 K (weak ferromagnetic) and 250 K (antiferromagnetic)
8 in LaCrO₃-LaFeO₃ system. Further, to understand the dynamic nature of these magnetic
9 transitions, we carried out temperature-dependent AC magnetic measurements at 100, 300,
10 1000, 3000 and 10000 Hz frequencies in the temperature the same range and plots are shown
11 in Fig. 3. The first magnetic transition around 290 K is coinciding to that of LaCrO₃ long-
12 range antiferromagnetic Neel temperature, superimposed with the weak ferromagnetic
13 ordering of both pristine LaCrO₃ and LaFeO₃ systems [4]. The onset of an additional
14 magnetic transition at ~ 250 K may be the consequence of competing spin interaction
15 between close proximity of magnetic Cr and Fe sublattices. To our surprise even transition at
16 250 K is also frequency independent, suggesting a long-range spin ordering in LaCrO₃-
17 LaFeO₃ system. The observed frequency independence magnetic transitions also rule out the
18 possibility of any clustering/spin glassy impurities in the synthesized LaCrO₃-LaFeO₃
19 system. The simultaneous presence of two AFM transitions in this system may be the
20 consequence of the proximity of Fe and Cr magnetic ions and complex magnetic interaction
21 between them. The XRD results confirm the synthesis of LaCrO₃-LaFeO₃ mixed-phase
22 system and observed complex magnetic properties suggest the presence of competing
23 magnetic interaction between different Cr and Fe ion sites in LaCrO₃-LaFeO₃ system. The
24 magnetization isotherms are measured near these transition temperatures to probe the nature
25 of magnetic ordering in conjunction with the measured temperature-dependent magnetization
26 measurements. The measured magnetic isotherms are shown in Fig. 4 for temperatures 200,
27 250, 280 and 315 K. The weak ferromagnetic component at 315 K is lower than that of 280
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3 K, suggesting that at a higher temperature, only LaFeO₃ weak ferromagnetic component is
4 contributing in this system. However, at lower temperatures, the contribution of LaCrO₃
5 weak ferromagnetic component is also added, as can be observed in Fig. 4. In contrast to the
6 observed weak ferromagnetism, the magnetization curves are not saturating up to 50 kOe
7 magnetic field suggesting the dominance of antiferromagnetic state in LaCrO₃ – LaFeO₃
8 system. The magnified magnetization curves are shown in Fig. 4 (lower panel), showing
9 nearly temperature-insensitive weak remnance $\sim 3 \times 10^{-3}$ emu/g. However, the respective
10 coercive field is much larger ~ 1 kOe for LaCrO₃ – LaFeO₃ system, suggesting the robust
11 metastable ferromagnetic state.
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24 The magnetic phase diagram against temperature is constructed for LaCrO₃-LaFeO₃ system
25 and is shown in Fig. 5. The high-temperature phase (>740 K) is paramagnetic, changing into
26 the antiferromagnetic phase dominated by LaFeO₃ in the proximate LaCrO₃-LaFeO₃ system.
27 This antiferromagnetic phase is superimposed with the weak ferromagnetic phase because of
28 the canted iron spins in FeO₆ octahedron in LaFeO₃ sublattice, as marked in Fig. 5. Further
29 reducing the temperature below 290 K, this changes into a metastable ferromagnetic phase at
30 the onset of LaCrO₃ antiferromagnetic transition. This phase persists until 250 K, where the
31 system shows another antiferromagnetic phase with weak ferromagnetism simultaneously.
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45 **Conclusion:**

46 We studied the complex magnetic behavior of proximate LaCrO₃-LaFeO₃ system with
47 different magnetic phases and intertwining of optical phonons with magnetic ordering. These
48 studies may lead to the materials engineering to design complex magnetic structured
49 materials with competing magnetic phases at or above room temperatures in mixed-phase
50 systems. The observed spin-lattice coupling from temperature-dependent Raman spectra
51 shows a possibility of inducing magnetodielectric coupling in such mixed-phase systems.
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Further investigations are required to understand the microscopic origin of observed complex magnetic structure and spin-lattice coupling proximate LaCrO_3 - LaFeO_3 system for possible tunability of spin-lattice functional properties.

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10 **Figure Captions:**

- 11
12 1. X-ray diffraction of bulk LaCrO₃-LaFeO₃ material with LaCrO₃ (Bottom panel) and
13 LaFeO₃ (Middle panel).
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15 2. (a) Raman Spectra of LaCrO₃, LaFeO₃ and LaCrO₃-LaFeO₃ at room temperature
16 recorded using 532 nm laser source. (b) Temperature-dependent Raman spectra of
17 LaCrO₃-LaFeO₃ using 514 nm laser source. (c) Intensity ratio of first order and
18 second order Raman peak as a function of temperature suggesting suppression of
19 second order peak upon magnetic ordering.
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21 3. Temperature-dependent DC (Zero-Field Cooled and Field Cooled) and AC (at
22 different frequencies) magnetic susceptibility plots for LaCrO₃-LaFeO₃.
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24 4. Representative isothermal magnetization loops close to magnetic transitions.
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Figures:

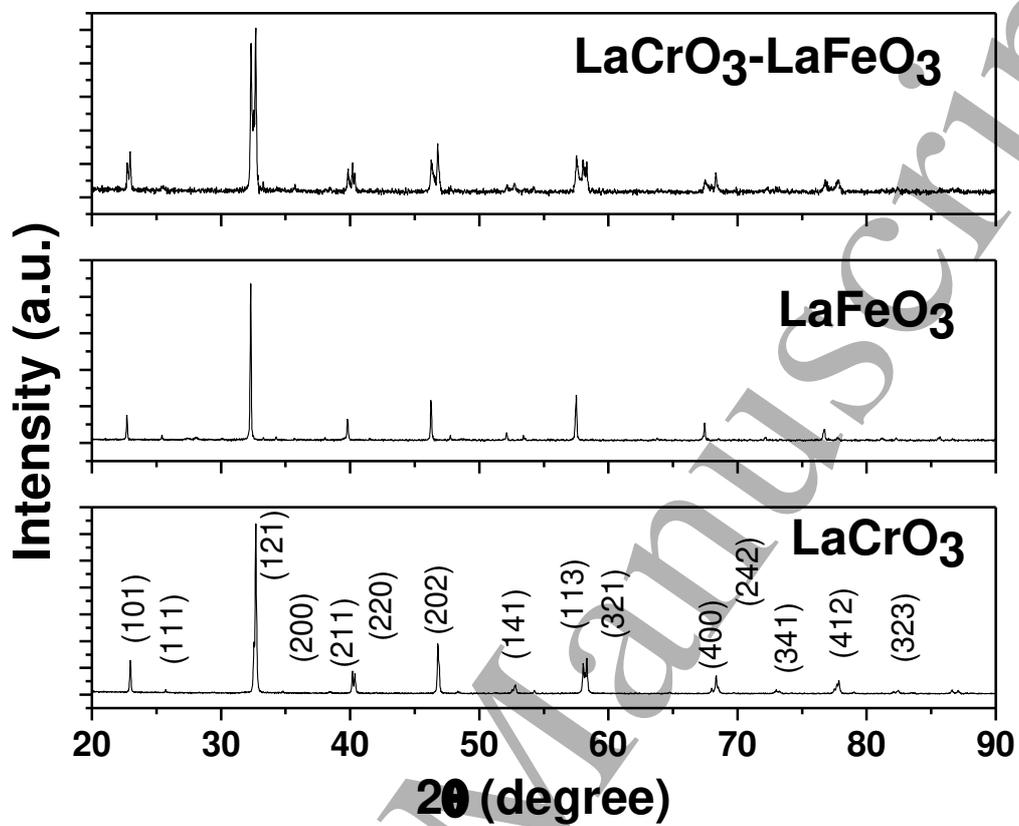
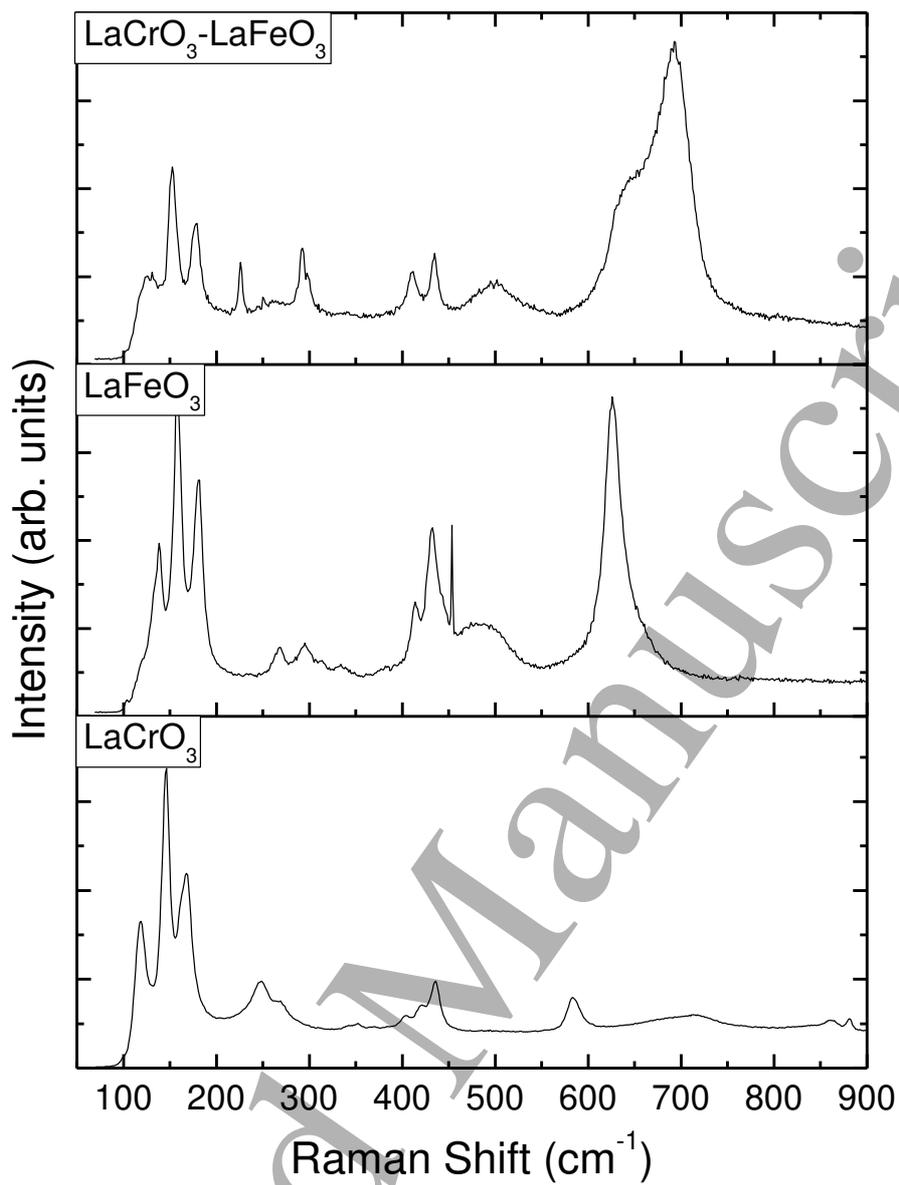


Figure 1.

**Figure 2(a)**

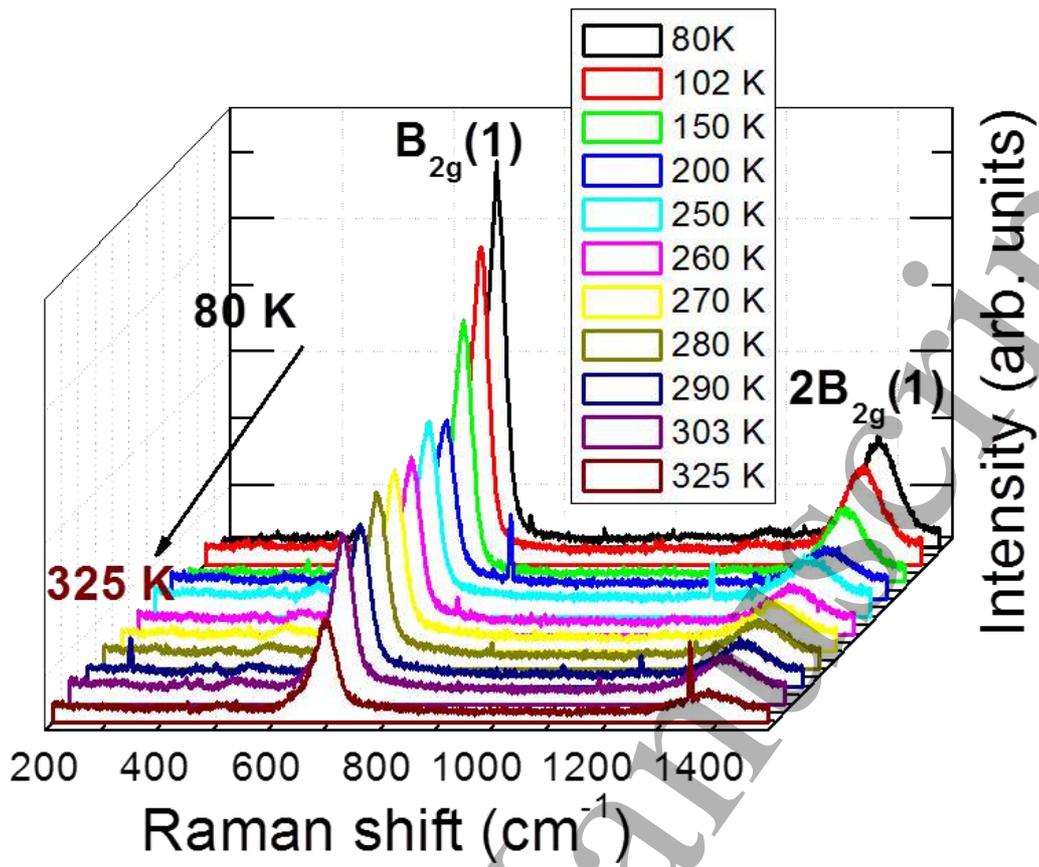


Figure 2 b

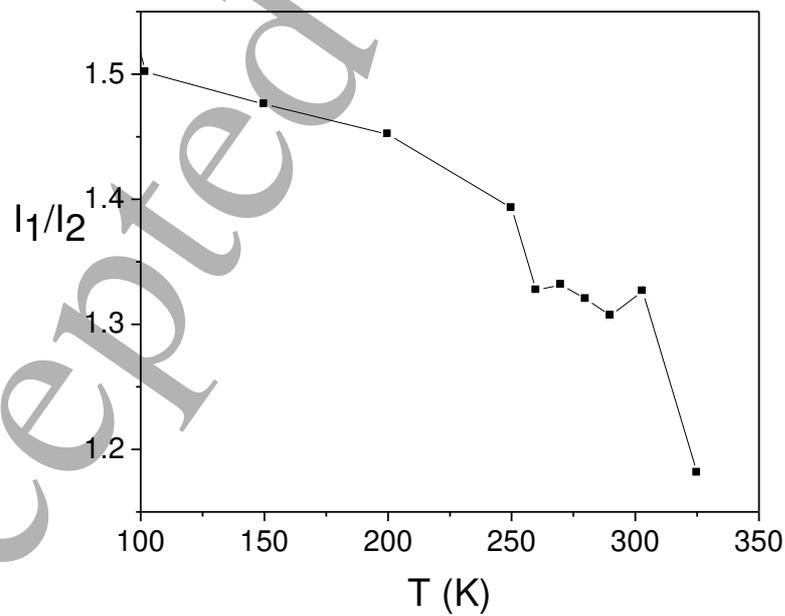
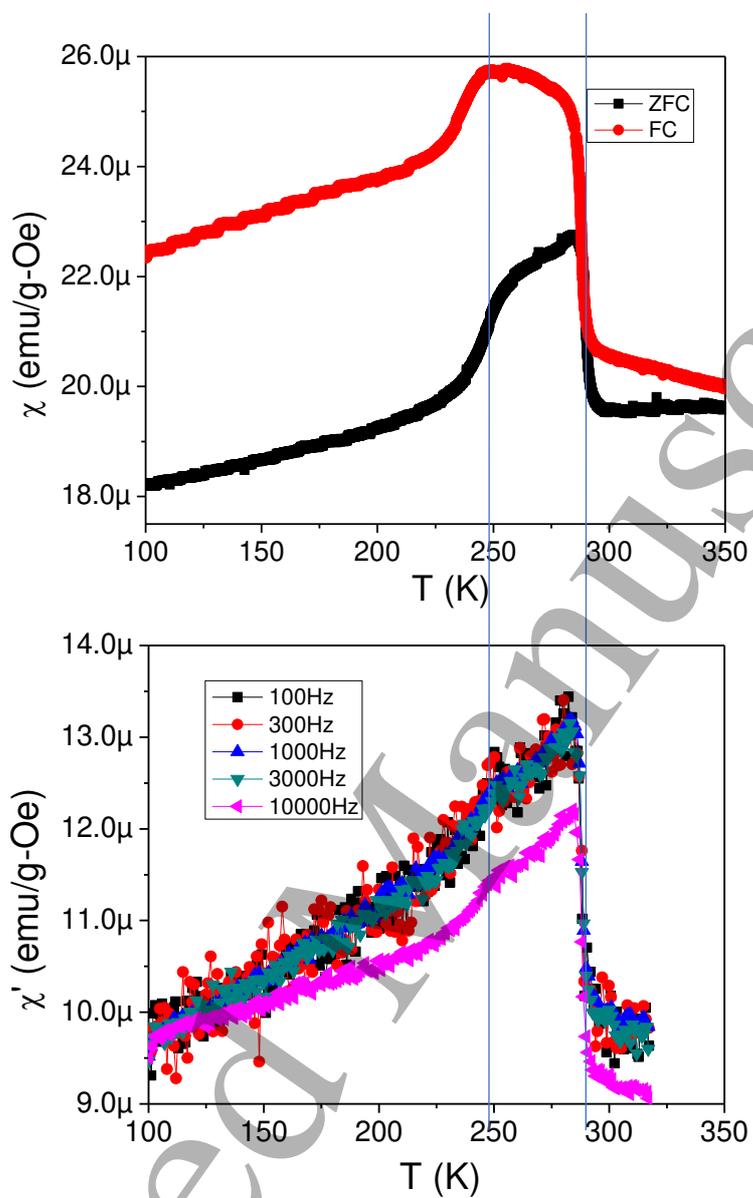


Figure 2 (c)

**Figure 3**

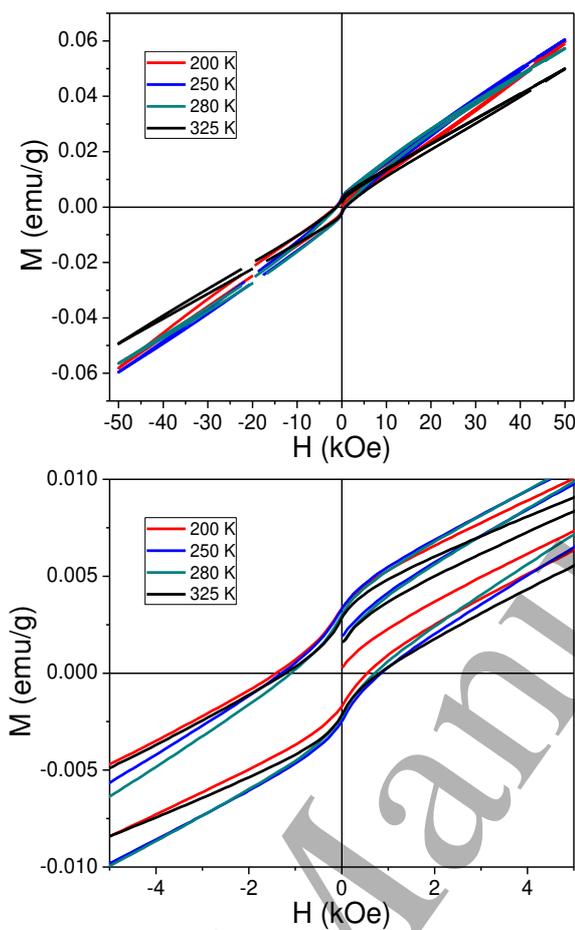


Figure 4

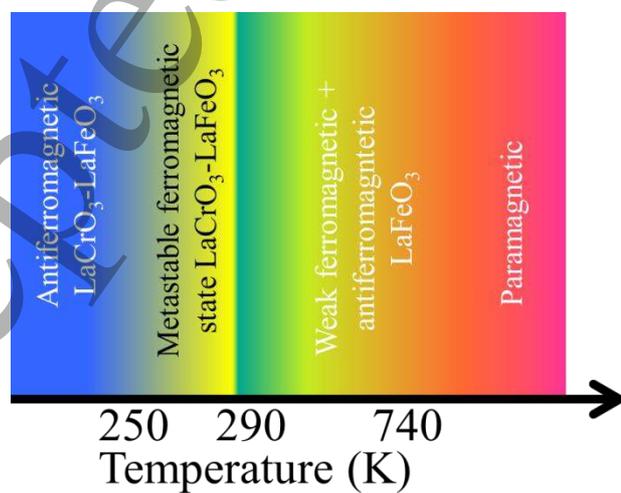


Figure 5