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# Exchange Bias and Magnetization Reversal in Ni( $Cr_{1-x}Fe_x$ )<sub>2</sub>O<sub>4</sub> (x = 0 - 0.20)

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**Abstract:** Exchange bias and magnetization reversal in single phase samples of Ni(Cr<sub>1-x</sub>Fe<sub>x</sub>)<sub>2</sub>O<sub>4</sub> (x = 0 - 0.20) were studied through magnetic measurements. Substitution of Fe for Cr changes the crystal structure at room temperature from tetragonal (space group: I4<sub>1</sub>/amd) to cubic (space group: Fd $\overline{3}$ m) form. Temperature variation of magnetization measurements show that these samples undergo ferrimagnetic transitions and the transition temperature ( $T_C$ ) increases from 73 K for x = 0.0 to 314 K for x = 0.20. An interesting magnetization reversal phenomenon was observed for x = 0.06 sample with a magnetic compensation temperature of 49 K. *M-H* loop measurements at different temperature show the signature of presence of strong antiferromagnetic interactions especially at low temperature (T < 50 K). Tunable exchange bias behavior with a maximum exchange bias field of 5670 Oe is observed for x = 0.06 sample and it is explained by considering the exchange anisotropy between the ferrimagnetic and the antiferromagnetic components. The exchange bias field and the vertical shift in magnetization decrease exponentially with increase in temperature.

Keywords: Ferrimagnetic; Antiferromagnetic; Exchange bias field; Magnetization Reversal

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#### Introduction:

The exchange bias (EB) is a phenomenon associated with the exchange anisotropy at the interface between an antiferromagnetic (AFM) and a ferromagnetic (FM) / ferrimagnetic (FIM) phase and this effect is seen as a shift in the magnetic hysteresis loop along the field axis [1]. Study of EB effect [2] has drawn a considerable interest due to its potential applications in magnetic read heads [3], thermally assisted magnetic random access memory devices [4], spintronics devices, etc. [5]. It has been studied in wide variety of magnetic materials such as oxidized FM particles, bilayers and multilayers of FM/AFM, FM/FIM, FM/spin glass, FIM/AFM, etc. and also in magnetic nanoparticles, charge ordered manganites, phase separated cobaltites, orthochromites, some spinel compounds, binary and intermetallic alloys, etc. [1, 6-15]. Recently tunable EB and its temperature dependence have been studied in several chromites such as nanoparticles of  $CoCr_2O_4$  and  $NiCr_2O_4$  [16, 17], Co and Cu doped  $CoCr_2O_4$  [14, 18]and in  $NiCr_2O_4/NiO$  composite [19].

Magnetization reversal (MR) is a phenomenon where the sign of magnetization can be changed without reversing the applied field direction. It was first predicted by Néel on spinel ferrites and is attributed to the different temperature dependences of antiferromagnetically coupled sublattice magnetization. MR has been studied in several spinel compounds such as  $Co_2VO_4$ , NiFe<sub>2-x</sub>V<sub>x</sub>O\_4, Fe<sub>2</sub>MoO<sub>4</sub>, CoCr<sub>2-x</sub>Fe<sub>x</sub>O<sub>4</sub> and FeCr<sub>2-x</sub>Al<sub>x</sub>S<sub>4</sub> [20] and was successfully explained on the basis of Néel's theory. The polycrystalline rare earth iron garnets Tb<sub>3-x</sub>Bi<sub>x</sub>Fe<sub>5</sub>O<sub>12</sub> and Ho<sub>2</sub>BiFe<sub>5</sub>O<sub>12</sub> are also reported to show negative magnetization [20]. Recently MR has been observed in many other systems like Sr<sub>2</sub>RRuO<sub>6</sub> (R = Y, Yb), orthochromites, orthoferrites, orthovanadates, molecular magnets, intermetallic alloys and perovskite manganites [20]. The mechanism of MR is found to differ depending on the nature of systems.

 $NiCr_2O_4$  is a normal spinel oxide having interesting structural, magnetic and magnetoelectric properties [21-23]. It is known to crystallize in cubic structure with space group  $Fd\overline{3}m$  above room temperature (T > 320 K). The Jahn-Teller distortion lifts the orbital degeneracy of  $t_{2g}$ orbitals of Ni<sup>2+</sup> ( $e_g^4 t_{2g}^4$ ) in tetrahedral site and it gives rise to structural transition into tetragonal (space group I4<sub>1</sub>/ amd) at around 320 K [24, 25]. NiCr<sub>2</sub>O<sub>4</sub> is known to undergoes FIM transition with  $T_C = 75$  K and a small saturation magnetization ( $M_s$ ) value of 0.3  $\mu_B$  / f.u. [21]. Klemme et al., [24] have found an additional transition at 29 K from heat capacity measurements and it has been identified as the transition due to AFM coupling of transverse components of magnetic moments. A new magnetic structure model having both longitudinal and transverse components of magnetic moment by grouping two A site ions as a single group and four B site ions as two different group was proposed by Tomiyasu and Kagomiya [26]. Due to such complicated magnetic structure NiCr<sub>2</sub>O<sub>4</sub> exhibits many interesting properties including EB. A few authors have already observed EB behavior in some composite and nanoparticles of NiCr<sub>2</sub>O<sub>4</sub> based compounds as discussed above [15, 16, 27, 28]. However, the temperature and composition dependence of EB field and the MR phenomenon have not been studied in details. In the present work, we have studied the structural and magnetic properties of Ni(Cr<sub>1-x</sub>Fe<sub>x</sub>)<sub>2</sub>O<sub>4</sub> (x = 0 to 0.2) to understand their MR and EB behaviors.

#### 2. Experimental details:

Ni(Cr<sub>1-x</sub>Fe<sub>x</sub>)<sub>2</sub>O<sub>4</sub> (x = 0 – 0.20) were prepared by using sol–gel method. Stoichiometric ratio of Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O, Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O and Cr(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O of 99 % purity were weighed and dissolved in distilled water and they were converted into citrates by adding Citric acid. The uniform solution added with ethylene glycol was heated slowly such that gel is obtained and the further

heating of gel yielded a fine precursor powder. The precursor was grinded and calcined at 600 °C, 800 °C and 1000 °C for 12 hours each. Final sintering was done in pellet form at 1100 °C for 24 hours. X-ray powder diffraction at room temperature was recorded by using Rigaku make TTRAX III X-ray diffractometer using Cu-K $\alpha$  radiation. Magnetization measurements were performed by using a Lakeshore make Vibrating sample magnetometer (VSM) of model no. 7410 and Quantum Design made 9 Tesla PPMS-VSM.

#### 3. Results and Discussions:

#### 3.1 Structural and magnetic properties

The Rietveld analysis of X-ray diffraction (XRD) patterns of Ni(Cr<sub>1-x</sub>Fe<sub>x</sub>)<sub>2</sub>O<sub>4</sub> (x = 0 – 0.20) using Fullprof software reveals that these compounds are in single phase form with tetragonal structure (I4<sub>1</sub>/amd space group) for x = 0 and in cubic structure for Fe doped samples (Fd $\overline{3}$ m space group). Typical XRD patterns along with Rietveld refinement for x = 0 and x = 0.20 samples are shown in Fig.1. For x = 0, the lattice parameters are a = b = 5.828 Å and c = 8.415 Å which are in agreement with that reported earlier [27]. Thus Fe doping drives the system into cubic structure at room temperature. It may be due to the occupation of some of the doped Fe<sup>3+</sup> ions at A site rather than occupying only the B site and as a result some of Ni<sup>2+</sup> ions are expected to occupy B site. Such substitution would reduce Jahn-Teller distortion and thus stabilize the cubic structure. The lattice parameter of Fe doped samples gradually decreases from a = 8.316 Å for x= 0.02 to a = 8.310 Å for x = 0.20. This decreasing trend of lattice parameter with increasing Fe concentration is also consistent with earlier report [29].



**Fig.1.** XRD patterns along with Rietveld refinement for x = 0 and 0.20. Open circles and continuous line represent the experimental data and refined fit, respectively.

Temperature variation of dc magnetization (M-T) was measured under zero field cooled (ZFC) and field cooled (FC) conditions for an applied field of H = 200 Oe. Fig.2 shows the M-T plots for x = 0, 0.06, 0.10 and 0.20 samples. The *M*-*T* plot under ZFC condition for each sample shows a peak highlighting the FIM transition. The x = 0 sample exhibits FIM transition ( $T_C$ ) at 73 K and it is found to increase quite sharply with increase in Fe concentration. Even for 2 at % of Fe substitution (not shown),  $T_C$  is found to increase drastically to 101 K and it shifts beyond room temperature for x = 0.2 (T<sub>C</sub> = 314 K). It highlights that substituted Fe ions are strengthening the superexchange interactions in the system. Strong irreversibility is observed for the samples at  $T < T_C$  due to the presence of competing AFM interactions. The ZFC magnetization value at 30 K is found to decrease slightly with increase in Fe concentration up to x = 0.06 and for further increase in Fe concentration it increases. According to the magnetic structure of NiCr<sub>2</sub>O<sub>4</sub> given by Tomiyasu et al. [26], the resultant B site moment is antiparallel to the A site moment. Again the B site is further divided into two groups namely B1-B3 and B2-B4 sublattices and the moment due to one of them (B1-B3) aligns parallel to that of the A site while that of B2-B4 group aligns antiparallel to A site moment. Thus the total magnetization per

formula unit can be written as  $M = |\uparrow \mu_{A1-A2} + \uparrow \mu_{B1-B3} - \downarrow \mu_{B2-B4}|$ , where  $\uparrow \mu_{A1-A2}, \uparrow \mu_{B1-B3}$  and  $\downarrow \mu_{B2-B4}$  are the longitudinal components of the magnetic moments of A1-A2, B1-B3 and B2-B4 sublattices respectively along the field direction. The decrease in magnitude of magnetization with increase in Fe concentration can be attributed to some of the Fe ions occupying the B2-B4 site. The FC *M*–*T* plot of *x* = 0.06 sample shows the sign reversal of magnetization with compensation temperature  $T_{comp} = 49$  K for H = 200 Oe.



**Fig.2:** Temperature dependence of ZFC and FC magnetization for samples with (a) x = 0.0, (b) x = 0.06, (c) x = 0.10 and (d) x = 0.20.

In order to understand the magnetization behavior of x = 0.06 sample exhibiting magnetization reversal; we have carried out *M*–*T* measurements at different applied fields ranging from 200 to 4000 Oe (not shown). The  $T_{comp}$  value is found to decrease from 49 K for H = 200 Oe to 38 K for H = 4000 Oe. The occurrence of negative magnetization can be explained by considering different temperature dependences of different sublattices of Ni(Cr<sub>1-x</sub>Fe<sub>x</sub>)<sub>2</sub>O<sub>4</sub>. At  $T_{comp} < T < T_C$ , for small applied field the A sublattice moment (Ni and Fe moments) is aligned along the field

direction and the resultant magnetic moment of two B sublattices align antiparallel to A site moment and in this arrangement A site moment dominates over net B site moment, i.e.,  $\mu_A > \mu_B$ . As the temperature is reduced, the B site moment continues to grow such that it compensates A site moment at  $T = T_{comp}$  and for further decrease in temperature negative magnetization is observed. The above argument can be substantiated from the reported saturation magnetization value (0.3  $\mu_B$  / f.u.) of the parent compound NiCr<sub>2</sub>O<sub>4</sub>, where  $\mu_A$  dominates over  $\mu_B$  down to low temperature, thereby without any magnetization reversal [21]. Upon Fe substitution, especially for x = 0.06,  $\mu_B$  is expected to dominate over  $\mu_A$  for  $T < T_{comp}$  and it results in magnetization reversal. However for  $x \ge 0.10$ , due to possible substitution of relatively larger concentration of Fe ions at A site compared to that of B site, no magnetization reversal is observed.

*M*–*H* loops recorded at 45 K are shown in Fig.3 for all the above samples. The initial *M*–*H* curve of x = 0 sample shows a slow increase of magnetization up to a threshold magnetic field of  $H_t = 2500$  Oe and for  $H > H_t$ , it increases rather sharply. This phenomenon can be attributed to the presence of domain wall pinning or the competing AFM interaction. The observed large coercivity for x = 0 and 0.02 samples can be attributed to the presence of competing AFM in addition to the existing long range FIM interaction. On the other hand for the intermediate compositions, i.e. x = 0.04 to 0.10, almost linear loop is observed due to the dominant AFM interaction which completely overtakes the FIM [16, 30]. For these samples, FIM  $T_c$  is quite large (> 128 K) and far away from 45 K. However in the vicinity of FIM  $T_c$ , these samples also exhibit considerable coercive field compared to that of x = 0 sample. For further increase in Fe concentration, i.e. for x = 0.20, large coercivity is observed due to stronger FIM interaction through considerable concentration of Fe<sup>3+</sup> – O<sup>2-</sup> – Fe<sup>3+</sup> networks [12]. The saturation magnetization,  $M_s$  values were determined after subtracting the linear part at higher field and its

value for x = 0 sample is 0.138  $\mu_B$ / f.u. and is comparable to the reported value of 0.135  $\mu_B$ / f.u. [27]. The maximum  $M_s$  value obtained for Fe substituted samples are found to vary from 0.047  $\mu_B$ / f.u. for x = 0.02 to 0.008  $\mu_B$ / f.u. for x = 0.06. These values are determined from *M*-*H* loops measured in the vicinity of  $T_C$  due to dominant AFM interaction at low temperature. For x = 0.20,  $M_s$  rise to 0.108  $\mu_B$ / f.u.



**Fig.3:** *M*-*H* loops at 45 K for x = 0.0 to x = 0.20 under ZFC condition.

#### 3.2 Exchange bias behavior

In the present work we have also tried to explore the EB effect in x = 0.02, 0.06, 0.10 and 0.20 samples. For this purpose M-H loops of each sample at different temperatures were recorded after cooling the samples in a field of 3000 Oe from above  $T_C$ . The value of EB field  $H_{EB}$  and the shift in magnetization  $M_{EB}$  were determined using the relations,  $H_{EB} = (H_1 + H_2)/2$  and  $M_{EB} =$ 

 $(M_1 + M_2)/2$ , where  $H_1$  and  $H_2$  are two magnetic fields and  $M_1$  and  $M_2$  are two magnetization values corresponding to M = 0 and H = 0 respectively, during the descending and the ascending branches of M-H loop [31]. For x = 0.02 sample the loops are found to shift towards negative Haxis with decrease in temperature and both  $H_{EB}$  and  $M_{EB}$  show an exponential rise in magnitude with decrease in temperature. However for T < 70 K, only minor loops could be observed due to the limitation of maximum applied field in the present set up and hence the measurement of  $H_{EB}$ and  $M_{EB}$  were limited down to 70 K. The EB behavior of x = 0.06 sample exhibits a distinct behavior, where for  $T < T_{comp}$  the loops are found to shift towards positive field axis while at higher temperatures ( $T > T_{comp}$ ), they shift towards negative field axis as shown in Fig.4. The maximum EB field value is ~ 5670 Oe. For x = 0.10, the M-H loops are found to shift towards negative field direction down to 20 K and no EB behavior is observed for x = 0.20 sample.



**Fig.4:** Field cooled *M*–*H* loops of x = 0.06 sample at different temperatures close to  $T_{comp}$  in expanded scales.

The temperature dependences of  $H_{EB}$  and  $M_{EB}$  for x = 0.02 sample are shown in Fig.5 (a) and (d). These data were fitted to the exponential functions  $H_{EB} = H_{EB}(0) \exp(-T/T_1)$ and  $M_{EB} = M_{EB}(0) \exp(-T/T_2)$  where,  $H_{EB}(0)$  and  $M_{EB}(0)$  are the values of  $H_{EB}$  and  $M_{EB}$  at T = 0 K and,  $T_1$  and  $T_2$  are constants. The fitted data are shown as red solid line. Similar trend is reported in other type of compounds such as in  $La_{0.25}Ca_{0.75}MnO_3$  nanoparticles [31], disordered  $R_{0.5}Sr_{0.5}MnO_3$  (R = Y,  $Y_{0.5}Sm_{0.5}$ , and  $Y_{0.5}La_{0.5}$ ) [9], amorphous/crystalline NiFe<sub>2</sub>O<sub>4</sub> [32], La<sub>1</sub>.  $_{x}Ca_{x}MnO_{3}$ ferromagnetic/AFM multilayers [33] La<sub>2/3</sub>Ca<sub>1/3</sub>MnO<sub>3</sub>/La<sub>1/3</sub>Ca<sub>2/3</sub>MnO<sub>3</sub> and superlattices [34], etc. The temperature dependences of  $H_{EB}$  and  $M_{EB}$  for x = 0.06 exhibit a sign reversal in the vicinity of  $T_{comp}$  as shown in Fig.5 (b) and (e). For x = 0.10 sample, the temperature dependence of  $H_{EB}$  and  $M_{EB}$  exhibits similar behavior as that of x = 0.06 except that they do not undergo any sign reversal (Fig.5 (c) and (f)).



**Fig.5.** Temperature variation of  $H_{EB}$  for (a) x = 0.02, (b) x = 0.06 and (c) x = 0.10 samples.  $M_{EB}$  versus temperature plots for these samples are shown in (d) to (f) respectively.

Presence of EB effect in x = 0.02 sample can be understood in terms of exchange anisotropy between the FIM and the AFM components. From the earlier report [26], it is known that the magnetic structure of NiCr<sub>2</sub>O<sub>4</sub> comprises of both FIM and AFM components at low temperature and in the present samples such behavior is observed in *M*-*H* loop. So when these samples are field cooled, the exchange anisotropy between the FIM and the rigid AFM phase gives rise to EB at low temperature. Although the long range AFM ordering is expected to appear at low temperature there is a possibility of such ordering taking place in a short range scale at higher temperatures. The EB behavior in x = 0.06 sample can be explained using the above argument. However, the change of domination of magnetic moment due to one FIM sublattice over the other one with decrease in temperature gives rise to sign reversal in  $H_{EB}$  and  $M_{EB}$ . This can be farther explained by considering the longitudinal component of magnetic moment of A sublattice  $(\mu_A)$  and B sublattice  $(\mu_B)$  as follows. In the temperature range  $T_{comp} < T < T_C$ ,  $\mu_A (\mu_{Ni} + \mu_{Fe})$ aligned along the field direction dominates over the antiparallel  $\mu_B (\mu_{Cr1} - \mu_{Cr2} - \mu_{Ni} - \mu_{Fe})$  as shown in Fig.6. Under this condition due to dominant  $\mu_A$  along the positive field direction it is relatively easy to align the net magnetization along the positive field direction. However, during the descending branch of M-H loop large negative field is required for such alignment. So effectively *M*–*H* loops shift towards the negative field axis. On the other hand, for  $T < T_{comp}$ ,  $\mu_B$ dominates over  $\mu_A$  and as per the above argument here the *M*-*H* loops shift along the positive field direction. The temperature dependence of  $H_{EB}$  and  $M_{EB}$  for x = 0.10 exhibit similar trend as that of x = 0.06, i.e. tendency towards positive  $H_{EB}$  for  $T < T_{comp}$ , however it remains negative down to low temperature due to the possible domination of  $\mu_A$  in the entire temperature range. Such tunable EB behavior has been reported in different materials such as in Sr<sub>2</sub>YbRuO<sub>6</sub> [35], NdCr<sub>1-x</sub>Fe<sub>x</sub>O<sub>3</sub> (x = 0.05–0.2) [36], La<sub>0.2</sub>Ce<sub>0.8</sub>CrO<sub>3</sub> nanoparticles [37], Sm<sub>0.975</sub>Gd<sub>0.025</sub>Cu<sub>4</sub>Pd [38],

Co(Cr<sub>0.95</sub>Fe<sub>0.05</sub>)<sub>2</sub>O<sub>4</sub> [39], Nd<sub>0.75</sub>Ho<sub>0.25</sub>Al<sub>2</sub> [13] etc. However, the mechanism of EB is different in different samples depending on their nature of magnetic and the micro- structures. The explanation of EB behavior in the present sample resembles the result in Co(Cr<sub>0.95</sub>Fe<sub>0.05</sub>)<sub>2</sub>O<sub>4</sub> and Nd<sub>0.75</sub>Ho<sub>0.25</sub>Al<sub>2</sub>. But some flipping / reorientation of the spins of different ions in different sublattices is considered below  $T_{comp}$  in case of Co(Cr<sub>0.95</sub>Fe<sub>0.05</sub>)<sub>2</sub>O<sub>4</sub>. In Nd<sub>0.75</sub>Ho<sub>0.25</sub>Al<sub>2</sub>, it is explained in terms of the different kinds of exchange coupling between the moments corresponding to conduction electron polarization and two dissimilar rare-earth ions. In the present sample no such spin reorientation or presence of conduction electrons are observed. Moreover, here the EB field is observed in a wide range of temperature. Since we are dealing with bulk system so there is no possibility of having core-shell structure which is used to explain EB in La<sub>0.2</sub>Ce<sub>0.8</sub>CrO<sub>3</sub> nanoparticles. In NdCr<sub>1.x</sub>Fe<sub>x</sub>O<sub>3</sub> (x = 0.05-0.2) the EB is observed due to the opposite alignment of the paramagnetic moment of rare earth ions to the canted ferromagnetic component of Cr<sup>3+</sup> ions which is also quite different from the present sample.



**Fig.6:** Schematic block diagram of the magnetic configuration with respect to applied field to explain the EB effect.

#### 4. Conclusions:

Ni(Cr<sub>1-x</sub>Fe<sub>x</sub>)<sub>2</sub>O<sub>4</sub> (x = 0, 0.02, 0.04, 0.06, 0.01 and 0.20) samples have been prepared in single phase form using sol-gel route. Due to the substitution of Fe, a structural transition from tetragonal cell with I4<sub>1</sub>/amd space group to cubic cell with Fd3m space group is observed at room temperature. The lattice parameter of Fe doped samples is found to decrease from a =8.316 Å for x = 0.02 to a = 8.310 Å for x = 0.20. Magnetization measurements show the presence of ferrimagnetic transition and the transition temperature is found to increase from 73 K for x = 0to 314 K for x = 0.20 and it signifies the strong superexchange interaction. The exchange bias behavior is observed for x = 0.02, 0.06 and 0.10 samples with a maximum exchange bias field of 5670 Oe. Tunable positive and negative exchange bias behavior is observed in x = 0.06 sample. Exchange bias behavior is explained in terms of anisotropic exchange interaction between the ferrimagnetic and the antiferromagnetic components of magnetic moment. Magnetization reversal along with the observed reversal in the exchange bias field around  $T_{comp} = 49$  K in x =0.06 sample is explained by considering the AFM interaction between the two sublattice moments present in the sample.

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# Highlights

- Magnetization reversal is observed for the first time in Fe doped Nickel Chromites.
- Tunable exchange bias field is reported.