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journal homepage: www.elsevier.com/locate/jmmmExchange bias and magnetization reversal in $\text{Ni}(\text{Cr}_{1-x}\text{Fe}_x)_2\text{O}_4$
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ABSTRACT

Exchange bias and magnetization reversal in single phase samples of $\text{Ni}(\text{Cr}_{1-x}\text{Fe}_x)_2\text{O}_4$ ($x=0-0.20$) were studied through magnetic measurements. Substitution of Fe for Cr changes the crystal structure at room temperature from tetragonal (space group: $I4_1/amd$) to cubic (space group: $Fd\bar{3}m$) 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.

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1. Introduction

The exchange bias (EB) is a phenomenon associated with the exchange anisotropy at the interface between an anti-ferromagnetic (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 $\text{NiCr}_2\text{O}_4/\text{NiO}$ 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 anti-ferromagnetically coupled sublattice magnetization. MR has been studied in several spinel compounds such as Co_2VO_4 , $\text{NiFe}_{2-x}\text{V}_x\text{O}_4$, Fe_2MoO_4 , $\text{CoCr}_{2-x}\text{Fe}_x\text{O}_4$ and $\text{FeCr}_{2-x}\text{Al}_x\text{S}_4$ [20] and was successfully explained on the basis of Néel's theory. The polycrystalline rare earth iron garnets $\text{Tb}_{3-x}\text{Bi}_x\text{Fe}_5\text{O}_{12}$ and $\text{Ho}_2\text{BiFe}_5\text{O}_{12}$ are also reported to show negative magnetization [20]. Recently MR has been observed in many other systems like Sr_2RRuO_6 ($R=Y, Yb$), 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 $Fd\bar{3}m$ above room temperature ($T > 320$ K). The Jahn–Teller distortion lifts the orbital degeneracy of t_{2g} orbitals of Ni^{2+} ($e_g^4 t_{2g}^4$) in tetrahedral site and it gives rise to structural transition into tetragonal (space group $I4_1/amd$) at around 320 K [24,25]. NiCr_2O_4 is known to undergo FIM transition with $T_C=75$ K and a small saturation magnetization (M_s) value of $0.3 \mu_B/\text{f.u.}$ [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

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group and four B site ions as two different group was proposed by Tomiyasu and Kagomiya [26]. Due to such complicated magnetic structure NiCr_2O_4 exhibits many interesting properties including EB. A few authors have already observed EB behavior in some composite and nanoparticles of NiCr_2O_4 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 $\text{Ni}(\text{Cr}_{1-x}\text{Fe}_x)_2\text{O}_4$ ($x=0-0.20$) to understand their MR and EB behaviors.

2. Experimental details

$\text{Ni}(\text{Cr}_{1-x}\text{Fe}_x)_2\text{O}_4$ ($x=0-0.20$) were prepared by using sol-gel method. Stoichiometric ratio of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ 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 h each. Final sintering was done in pellet form at 1100 °C for 24 h. X-ray powder diffraction at room temperature was recorded by using Rigaku make TTRAX III X-ray diffractometer using $\text{Cu-K}\alpha$ radiation. Magnetization measurements were performed by using a Lakeshore make Vibrating sample magnetometer (VSM) of model no. 7410 and Quantum Design made 9 T PPMS-VSM.

3. Results and discussions

3.1. Structural and magnetic properties

The Rietveld analysis of X-ray diffraction (XRD) patterns of $\text{Ni}(\text{Cr}_{1-x}\text{Fe}_x)_2\text{O}_4$ ($x=0-0.20$) using Fullprof software reveals that these compounds are in single phase form with tetragonal structure ($I4_1/amd$ space group) for $x=0$ and cubic structure for Fe doped samples ($Fd3m$ 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^{3+} ions at A site rather than occupying only the B site and as a

result some of Ni^{2+} 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].

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 $x=0.20$ ($T_C=314$ 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_2O_4 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.

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 $\text{Ni}(\text{Cr}_{1-x}\text{Fe}_x)_2\text{O}_4$. At $T_{comp} < T < T_C$, for small applied field the A sublattice moment (Ni and Fe moments) is aligned along the field

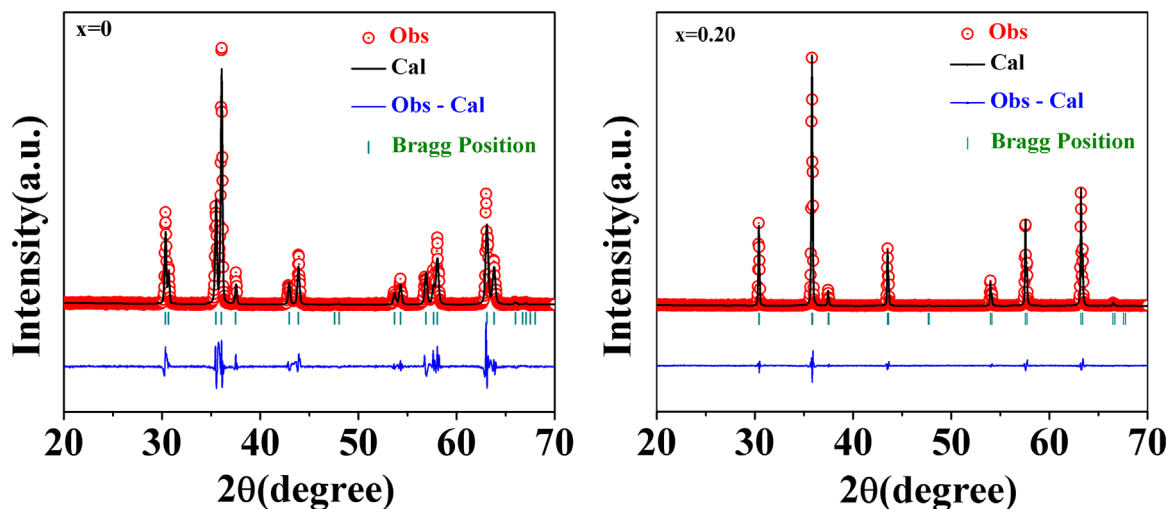


Fig. 1. XRD patterns along with Rietveld refinement for $x=0$ and 0.20 . Open circles and continuous line represent the experimental data and refined fit, respectively.

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