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WASP-Waisted Hysteresis Loops in case of High Riched Cobalt Substituted Magnesium Ferrites

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Abstract: The ferrite samples $Co_xMg_{1-x}Fe_2O_4$ (x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) were synthesized by ceramic technique. Normal hysteresis loops were observed in case of samples having poor cobalt content (i. e. x = 0.0, 0.2, 0.4, 0.6) while wasp - waisted hysteresis loops were observed in case of samples having high riched cobalt content (i. e. x = 0.8 and 1.0) . As regards wasp - waisted hysteresis loops observed in case of Co - riched samples , cobalt ions play an important role. Firstly , wasp - waisted hysteresis loops may be due to impurity phase of cobalt as reflected from XRD patterns .Secondly, cobalt ions (Co^{2+} and Co^{3+}) on the octahedral site induce anisotropy in the domains and domain walls . Thus stabilizing a Co - riched ferrite structure and inhibiting domain wall motion. This is one of the cause of formation of wasp- waisted hysteresis loop Thirdly the wasp - waisted hysteresis loop with x = 0.8 is attributed due to presence of mixture of SD and MD grains while wasp - waisted hysteresis loop with x = 1.0 is attributed mixture of SD and SP grains .
Keywords: Wasp - waisted hysteresis loop, grain size, Multi – Domain (MD) , Single - Domain (SD), Super – Paramagnetic domain (SP).

I. INTRODUCTION

Hysteresis loops that are constricted in the middle section but are wider above and below the middle section are called as wasp - waisted hysteresis loops . Such a wasp - waisted hysteresis loops are reported by Robert et. al. [1] in case of mixed magnetic assemblages , rare earth permanent magnets , basaltic lava flows , demagnetized paleozonic carbonate rocks etc and explained on the basis of structural and magnetization characterizations . The constricted hysteresis loop in Co - ferrite is observed [2] and explained on the basis of spinel structure and aging process . Recently wasp - waisted behavior in the magnetic hysteresis curves of nano - powdered Co – ferrite prepared by ball milled process is observed[3] and explained on the basis of domain behavior of the material.

There is a little information available in the literature on the wasp - waisted hysteresis behavior of high riched Co- substituted Mg - ferrite. Thus in this research work, typical wasp - waisted hysteresis loops of the $Co_xMg_{1-x}Fe_2O_4$ (x = 0.8 and x = 1.0)ferrites are presented.

II. EXPERIMENTAL

The ferrite samples $Co_xMg_{1-x}Fe_2O_4$ (with x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) were synthesized by the standard ceramic technique. High purity ferric oxide (Fe_2O_3), Cobalt oxide (CoO) and magnesium oxide (MgO) were taken in calculated molar proportions and mixed thoroughly in the acetone medium in agate mortar. The pre-sintering was carried out at 800^{O}C for 10 hours and samples were cooled at the rate of 80^{O}C per hour.

To prepare pellets, approximately 1gm of pre-sintered powder was taken and subjected to a pressure of about 5 tons per square inch for two minutes by keeping it in a die of 1 cm diameter. The pellets were again sintered at 1250^{O}C for 40 hours and cooled at the rate of 80^{O}C per hour.

For characterization of the samples, technique of X-ray diffraction (XRD) performed on a diffractometer (Philips pw 1820) using Fe- K_α radiation (λ=1.936 A^0) was used . For the hysteresis loops of the samples , a high field hysteresis loop tracer (TIFR make) was used.

III. RESULTS AND DISCUSSION

Hysteresis loops of ferrite samples $Co_xMg_{1-x}Fe_2O_4$ (with x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) are given in Fig. 1. From Fig.1 , it is seen that normal shape of hysteresis loops are observed for the samples with x = 0.0, 0.2, 0.4 and 0.6 . However for Co – rich samples (x = 0.8 and 1.0) , abnormal or constricted or wasp - waisted hysteresis loops are seen. As regards the wasp - waisted hysteresis loops in case of Co -riched ferrites , Co^{2+} and Co^{3+} ions play an important role.

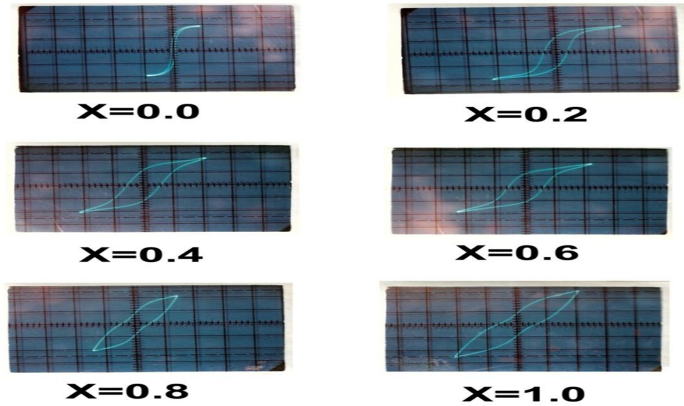

Fig.1: Hysteresis loops of ferrite samples $Co_xMg_{1-x} Fe_2O_4$ (with x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0)

The XRD patterns of the ferrite samples $Co_xMg_{1-x} Fe_2O_4$ (with x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) are shown in Fig. 2.

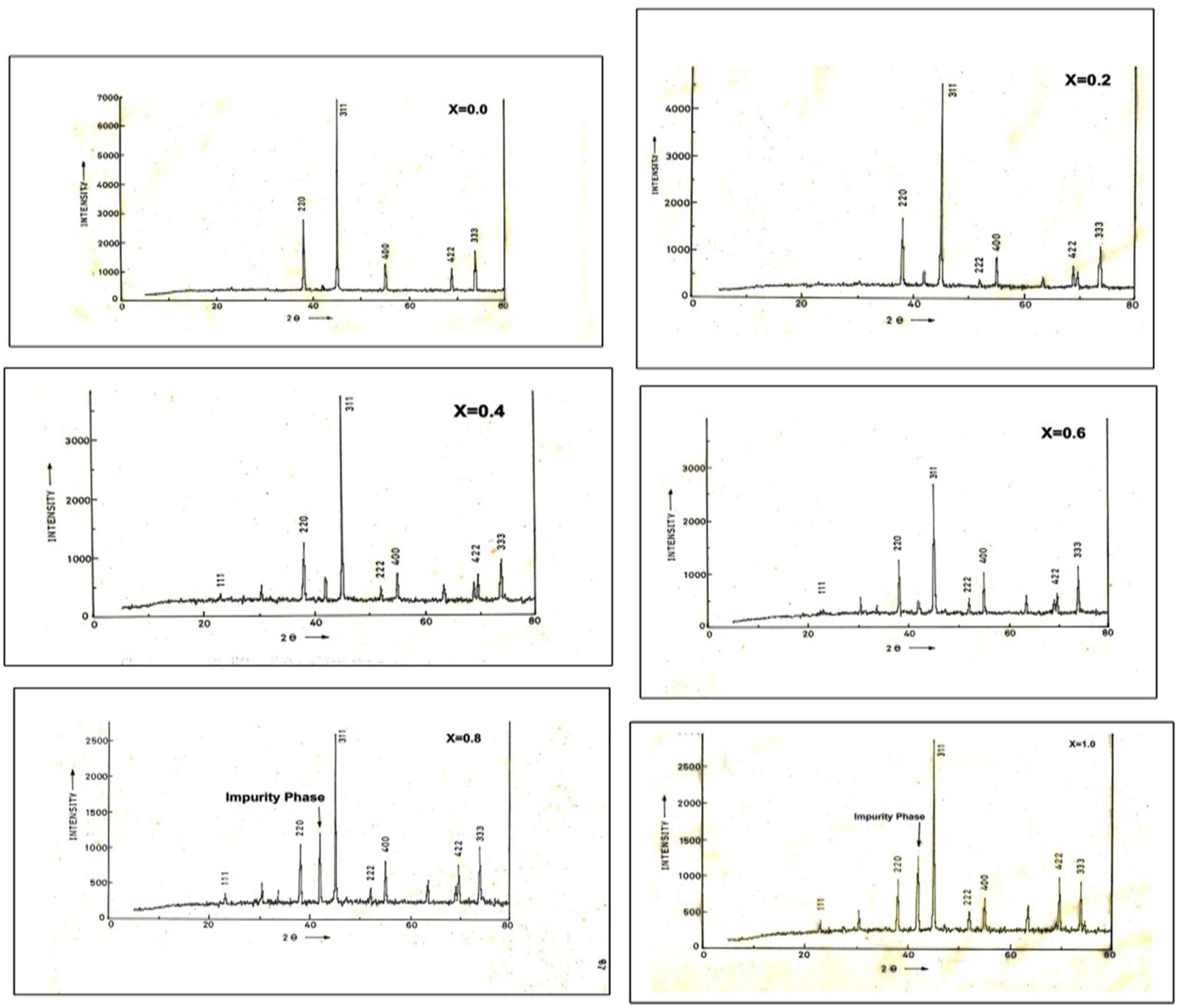

Fig.2: XRD patterns of the ferrite samples $Co_xMg_{1-x} Fe_2O_4$ (with x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0)

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Analysis of XRD patterns confirm the formation of spinel structure. The diffraction maxima have been indexed in the light of crystal structure of the natural spinel $MgAl_2O_4$. The diffraction patterns for the series show that there is no impurity phase present in the samples except for a trace in case of Co – riched ferrites (x = 0.8 and 1.0). The wasp - waisted hysteresis loops may be due to impurity phase of cobalt as reflected from the XRD patterns.
The distribution of cations (Mg^{2+}, Co^{2+} , Co^{3+}, Fe^{2+} and Fe^{3+}) over tetrahedral site (A – site) and octahedral site (B – site) in spinel structure is already reported [4] and is given in Table I

Table I : Cation distribution in $Co_xMg_{1-x}Fe_2O_4$ ferrite system.

COMPOSITION	CATION DISTRIBUTION	
	A - site	B - site
$MgFe_2O_4$	$Fe_{0.90}Mg_{0.10}$	$Fe_{1.10}Mg_{0.90}$
$Co_{0.2}Mg_{0.8}Fe_2O_4$	$Fe_{0.94}Mg_{0.04}Co_{0.02}$	$Fe_{1.06}Mg_{0.76}Co_{0.18}$
$Co_{0.4}Mg_{0.6}Fe_2O_4$	$Fe_{0.95}Mg_{0.01}Co_{0.04}$	$Fe_{1.05}Mg_{0.59}Co_{0.36}$
$Co_{0.6}Mg_{0.4}Fe_2O_4$	$Fe_{0.95}Mg_{0.02}Co_{0.03}$	$Fe_{1.05}Mg_{0.30}Co_{0.57}$
$Co_{0.8}Mg_{0.2}Fe_2O_4$	$Fe_{0.95}Mg_{0.04}Co_{0.01}$	$Fe_{1.05}Mg_{0.16}Co_{0.79}$
$CoFe_2O_4$	$Fe_{0.85}Co_{0.15}$	$Fe_{1.15}Co_{0.85}$

For an ideal inverse spinel ferrites , divalent metal ions occupy octahedral site (B – site) while trivalent metal ions occupy equally between tetrahedral site (A – site) and octahedral site (B – site).From table I it is seen that Mg^{2+} ions as well as Co^{2+} ions exist in both sites but they have strong preference (≈ 85% to 95 %) for octahedral site (B- site) while fe^{3+} ions are nearly equally distributed over A- and B- sites. Thus all the samples of $Co_xMg_{1-x}Fe_2O_4$ ferrite series tend to exhibit inverse spinel structure. From cation distribution (Table I), it is also seen that Mg^{2+} ions and Co^{2+} ions substitute each other in the spinel lattice during the formation of solid solution of $Co_xMg_{1-x}Fe_2O_4$ ferrites . This is not surprising because the ionic radii of Mg^{2+} (0.75 A^0) and Co^{2+} (0.78 A^0) are very close to each other.
In the cobalt containing ferrites , the anisotropic effect of cobalt ions out weights all the possible contribution to the induced anisotropy. The ionic processes of Co – diffusion were studied by number of investigators [2] ,[3] and concluded that for low cobalt content in ferrites , Co^{2+} ions and $Co^{2+} \leftrightarrow Co^{2+}$ pair on octahedral sub - lattice is responsible for induced anisotropy . However , for higher cobalt content and if Co^{2+} and Co^{3+} ions are present on octahedral site then direct electron exchange $Co^{2+} \rightarrow Co^{3+}$ is possible as in Co – riched spinels [5]. Thus the presence of cobalt ions on octahedral site of spinel ferrites favors the conduction mechanism

$$Co^{2+} + Fe^{3+} \leftrightarrow Co^{3+} + Fe^{2+}$$

The cobalt ferrite is characterized by high values of magneto crystalline anisotropy. This property is due to the triplet orbital states of Co^{2+} ($3d^7$) ions on the octahedral site of crystal lattice . Compared with other 3d ions , such ions have a large unquenched orbital momentum and therefore a large spin – orbital interaction. The diffusion of electrons between Co^{2+} and Co^{3+} ions gives rise to an induced anisotropy in the domains and domain walls . Thus stabilizing a Co – rich ferrite structure and inhibiting domain wall motion . This is one of the cause of formation of wasp - waisted hysteresis loops in case of samples having x = 0.8 and x = 1.0 .
Generally demagnetized state of the ferrites is characterized by a region of spontaneous magnetization called as magnetic grains . If the magnetic grains contain domain walls then thses are called as multi - domain (MD) . The sizes of these domains are of the order of 10 µm. If the magnetic grains do not contain domain walls due to their small sizes (≈ 100 A^0) then these are called as single domain (SD). If the thermal energy of the SD grains is comparable to or greater than the magnetic energy then the magnetization of the grains undergo fluctuations and the grains are in super- paramagnetic (SP) state [6].
According to Bean [7] , for the magnetic material containing MD particles , one observes very small values of coercive field (Hc) whereas for SD particles, these are considerably larger. For Mg -ferrite , the value of the coercive field is very low (Hc = 50 gauss) while that of Co – ferrite , it is very large (Hc = 850 gauss) as already reported [4] . Hence Mg – ferrite contains MD particles or grains whereas Co – ferrite contains SD particles or grains. Domain behavior in $CoxMg1_{-x}Fe_2O_4$ ferrites is already reported [8] and concluded that Mg ferrite contains MD particles . Ferrites with x = 0.2 and 0.4 (Mg – rich ferrites) contain mixed MD and SD

particles but with predominance of MD states. Ferrites with x = 0.6 and 0.8 (Co – rich ferrites) contain mixed SD and MD particles but with predominance of SD states .

According to Robert [1] , wasp - waisted hysteresis loop of magnetic material can be attributed to mixture of either SD and SP particles or SD and MD particles. Thus in present work (Co – riched Mg – ferrites) , the wasp - waisted hysteresis loop of ferrite with x = 0.8 is attributed to the mixture of SD and MD grains in it while wasp - waisted hysteresis loop of ferrite with x = 1.0 is attributed to the mixture of SD and SP grains in it.

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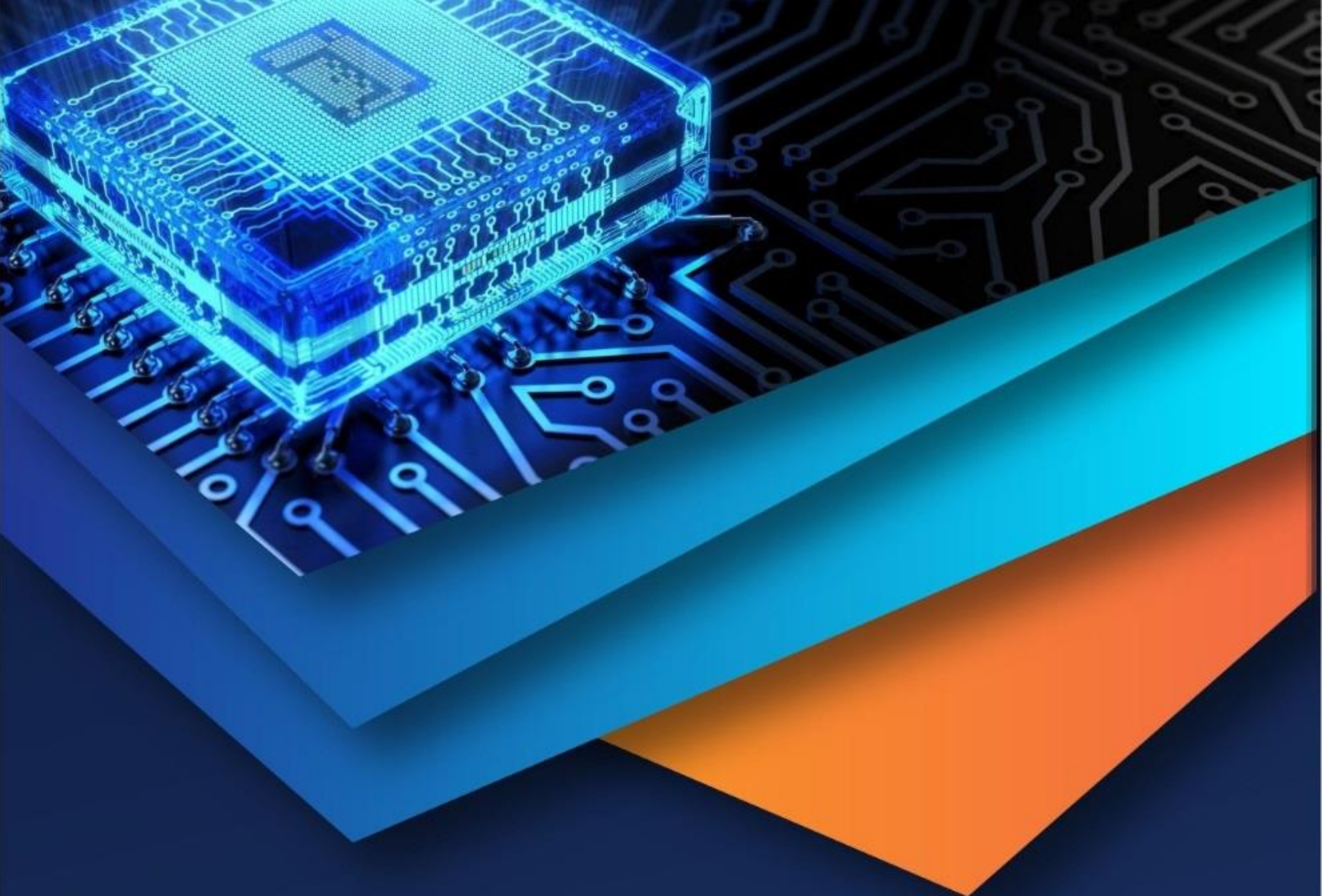

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