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Magnetic properties of Fe–Nd Silica Glass Ceramics

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Abstract. Soda lime silica glass ceramics containing iron and neodymium have been synthesized. The XRD pattern revealed that the glass samples devitrified into multiple phases. Fe₂O₃ as an initial component converted into Fe₃O₄ in the sample during the synthesis, and was the main contributor to the magnetic property of the sample. The inclusion of Nd was found to enhance the magnetization of the sample at 5K. The coercivity of the sample increased with decrease in temperature from room to 5K.

INTRODUCTION

The magnetic properties of oxide glass and ceramic systems have attracted much interest. One of the main reasons for this is that they form good insulating spin glasses [1-6]. This phase of spin glass is mostly observed at low temperatures. The oxide glass system containing Mn²⁺, Fe³⁺ and Co²⁺ and have been reported to show negative Weiss temperatures, and paramagnetic to spin glass transitions at low temperatures²⁻⁶. On the other hand, there are also a few reports on glasses with rare earth *4f* ions, which are not reported to show any magnetic transition even at 2K[7-9].

Akamatsu et al. (2011) have reported a glass system with both iron and rare earth ions (Sm, Gd and Tb). The detailed study on these samples show that there is a link between the interaction of rare earth ions and iron ions in the magnetic property of the sample. However, these authors maintain that the contribution by rare earth ions in such systems is expected to be small.

Although there have been studies on the glass or glass ceramic samples with either only transition metal ions or rare earth ions, and a few reports on glass samples with both the rare earth and transition metal ions together, not much has been reported on the devitrified glass sample containing both the irons ions and the rare earth ions together.

The magnetic property of a glass ceramic sample with iron co-doped with neodymium ions is examined in this study. The magnetic properties of this sample are also compared with those of a ceramic having only iron ions.

EXPERIMENTAL

Compositions

The starting materials for glass preparation were SiO₂ (99.9%, Sigma Aldrich) and oxides of sodium, calcium, iron and neodymium (99.8%, Thomas Baker). The carbonates of sodium and calcium (Na₂CO₃ and CaCO₃) were used in the initial charge to obtain the oxides Na₂O and CaO. Details of the compositions of both the samples are tabulated in Table 1. The sample without Nd is denoted as S1 while that with Nd is referred to as S2.

Preparation

Table 1: Compositions in mole% of the glass ceramics under study.

Sample	SiO ₂	Na ₂ O	CaO	Fe ₂ O ₃	Nd ₂ O ₃
S1	0.40	0.20	0.10	0.30	0.00
S2	0.40	0.20	0.10	0.25	0.05

The samples were heated in alumina crucibles in batches of 10 g. All the samples were finely ground by mortar and pestle to ensure thorough mixing and homogenization. The crucibles were heated to 1450 C in a 1600 C carbolite furnace and quenched by pouring each melt onto a steel plate. The samples were then transferred to a pre-heated (430°C) muffle furnace and cooled slowly to room temperature to obtain the desired ceramics.

Characterization

X-ray powder diffraction (Rigaku, CuK α) (XRD) pattern was used to determine the possible crystalline phases present in the glass ceramics. The 2 θ range was 10° – 70°, with the 2 θ step = 0.02°. The diffraction pattern was then matched with the JCPDS files.

The magnetic properties of the samples were analyzed using the data collected by commercial Quantum Design, USA made 9 Tesla PPMS based Vibrating Sample magnetometer in the temperature range of 5K to 300K, in magnetic field up to 9T.

RESULTS AND DISCUSSION

The Powder X-Ray Diffraction and Structural Analysis

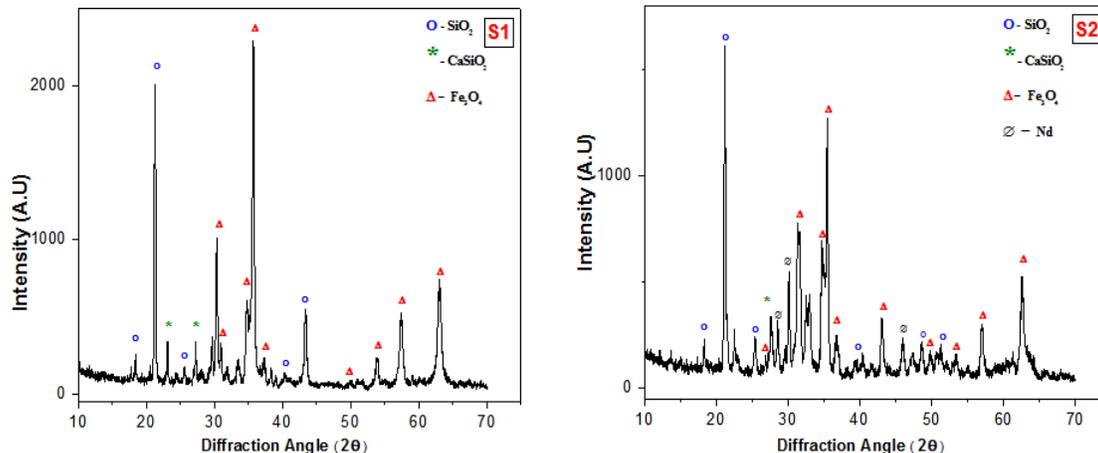


Figure 1: X – Ray diffraction patterns of the samples S1 and S2

The measured XRD patterns for the two samples are shown in Figure 1. The XRD pattern clearly indicates that the samples have devitrified into multiple phases. The crystalline phases matched those expected for Fe₃O₄ (JCPDS file no. 19-629) and SiO₂ (JCPDS file no. 46-1045). There are also few reports of the other phases reported in the past [5-9]. The Fe₂O₃ in the samples has been shown to convert to Fe₃O₄. This phase, i.e. Fe₃O₄ along with Nd is responsible for the magnetic property of the ceramics samples discussed below.

Magnetization studies by Vibrating Sample Magnetometer

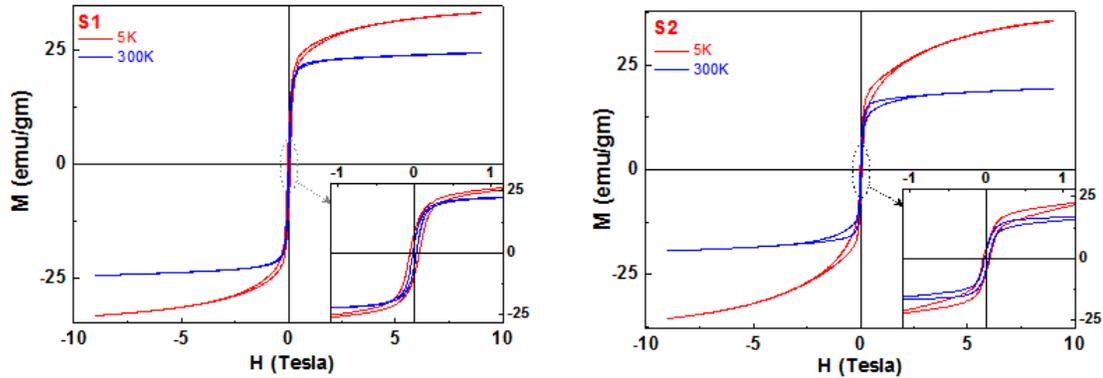


Figure 2: M vs H loop for S1 and S2 at 300K and 5K.

Figure 2 depicts the room temperature and low temperature magnetic hysteresis (M– H) loops for both samples. In S1 the coercive field increases from 270Oe to 650 Oe as T decrease from 300K to 5K, while the saturation magnetization increases from 24.75em/g to 33.40 emu/g in the same temperature range.

In case of S2 the coercive field increases from 300 Oe to 430 Oe with the decrease in temperature, while the saturation magnetization increases from 19.38 emu/g to 35.64 emu/g with decrease in temperature to 5K. The lower saturation magnetization at room temperature in case of S2 is due to the low iron content. A summary of the measured magnetization parameters is shown in Table 2. It may be noted that in S2, the magnetization at 5K is higher than would be expected had the sample contained only Fe ions. The extra observed magnetization is likely to be due to the presence of Nd clusters in the devitrified glass. The possibility of these clusters being superparamagnetic [O’Horo et al.] exists and needs further investigation.

Table 2: Magnetic parameters of the glass ceramics.

Sample	M_s (emu/g)		H_c (Oe)		Calculated Magnetic moment/f.u. (μ_B)	
	RT	5K	RT	5K	RT	5K
S1	24.75	33.40	270	650	2.59	3.51
S2	19.38	35.64	300	430	6.46	11.88

CONCLUSION

Based on the magnetic characterizations, it may be said that the ceramic samples formed are of soft magnetic type. Doping Nd together with Fe ions resulted in an enhanced magnetic property of the samples at 5K. However, the Nd doped ceramic shows low magnetization at room temperature due to the lower concentration of iron ions. The enhanced magnetization at low temperatures when Nd is present is likely to be due to clustering of Nd ions in these devitrified samples.

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