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Synthesis and Magnetic Properties of the Ceramic Nano Ferrite $MnFe_{2-x}Sm_xO_4$

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ABSTRACT

Ceramic nano-ferrite series of the formula $MnFe_2$-xSmxO_4 (x=0, 0.5, and 0.1) is prepared, using solution combustion process. XRD-analysis showed poorly resolved patterns of as-burnt powders which are progressively mended by progressive raising of heat treatment temperature.TEM particle-size analysis indicated that the dimensions of the as-burnt powder is of the nano-order. Moreover, thermal analysis of these powders exhibited a continuous decline in the TG-curve in the temperature range of up to 1000C. On the other hand no explicit reaction was detectable in the behavior of the DTA-curve of the as-burnt ferrite powder for the same range. Room temperature magnetization measurements of green compacted or sintered powders of various compositions, confirmed the superparamagnetic nature of the nano-ferrites. However, modifications are observed in hysteresis loops when various ferrite powders are subjected to the above mentioned heat tretment skeems. This may be attributed to variations in atomic order (inversion of the spinel structure) , also the partial replacement of Fe-ions by rare earth Sm-ions may induce local strains, impurities, or lattice defects which may also cause such modifications .On the other hand, values of magnetic parameters like permeability and remanent magnetization are explicitly altered upon Fe-ion replacement or heat treatments.

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INTRODUCTION

It is known that physical properties of materials undergo change when the size of its particles is drastically reduced (quantum-size effect)(1). Under such circumstances the material surface-to-volume would play a dominant role in influencing its properties. Structurally, small particles differ from the bulk of a solid in having large fraction of its atoms residing at the surface. There, atoms would experience quite different environment where coordination numbers reduced as compared with interior atoms. However, coordination numbers of individual surface atomic sites would differ depending on their local atomic arrangement geometry and bonding. Hence, it should be quite possible to alter or modify the structure and physical properties of a material by reducing its particle sizes to the level of nano-scale. This has been made realistic by the use of new methods of synthesis specially performed at low temperatures (R.T), like ball milling or sol-gel (1-6). Particles of magnetic materials having sufficiently small sizes are known to exhibit some very special behavior which may be explained the superparamagnetic phenomenon (1, 2). Accordingly, Gobin et al. (2) investigated the magnetic properties of nano ferrites and confirmed that chemical compositions, particle sizes, particle-matrix interactions, and the degree of lattice defectiveness, are promising factors in allowing for the control in such materials design. Also Gatelyte et al. (3) have investigated Mn-ferrites which were prepared by copricipitation, while Chinnasami et al. (4) investigated the influence of site of Fe-ion site occupation on the mgnetism observed in the spinel structure. A. Lakshman et al. (5), on the other hand investigated the effect of In-ions and Cr-ions in the Mg-Mn ferrite which focuses on the compositional effects on these properties. Xu Feng et al. (6) thought of investigating the effect of doping Lanthenum rare earth ions in the Ni-ZN-Cr ferrites and monitor the resulting magnetic properties. In the present work it is thought to be of interest to investigate an Mn-ferrite which may be prepared by a novel method proposed by Nilar Lwin and Ahmad Fauzi (and co workers) (7), doped by partial replacement of Fe-ion with Sm-ion (rare earth) .By measuring structural, thermal , and magnetic properties.

EXPERIMENTAL TECHNIQUES

Nano crystalline powders of the ceramic ferrite series MnFe2-xSmxO4 (x=0, 0.05, 0.1) were prepared by following the steps outlined in the process flow diagram listed in figure (1). Appropriate amounts of hydrated nitrates of Fe, Mn, and Sm (analytical purity) were separately dissolved in and resulting aqueous solutions were mixed with citric acid in a molar ratio of 1:1. On the other hand PH-values of the dark brown mixture was adjusted by slow additions of NH_4OH (25%) drops. The mixture was allowed to condense by evaporating at

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80Cfor some time, until becoming sticky gel. At this stage, the temperature of the hot plate was raised immediately, and at a solution temperature of about 130C the mixture ignited in a short combustion, liberating large amounts of CO_2, H_2O, and N_2 gases. The dark brown powder left behind was ground mechanically for prolonged period of time and then investigated.

Some of the as-burnt powders were separately calcined for 5 hours at high temperatures (500°, 600°, and 800°C) and analyzed by XRD for crystallinity

Transmission electron microscope (TEM), was used for particle size analysis of all ground powders, with special efforts made to ensure appropriate particle dispersion allowing single particle measurements.

Magnetization measurements for various powders were made using a vibrating sample magnetometer (VSM) at room temperature to establish their respective hysteresis loops. Calcined powders of different compositions compacted into thin disks, were used for the measurements in their green state. Moreover, powders of the same compositions above were compacted and sintered at 1000°C and 1250°C for 5 hours were also investigated .Also, the as-burnt ferrite powder was thermally analyzed by a NETZESH 409E STA for temperatures of up to 1000° C to establish their TG and DTA behaviors.

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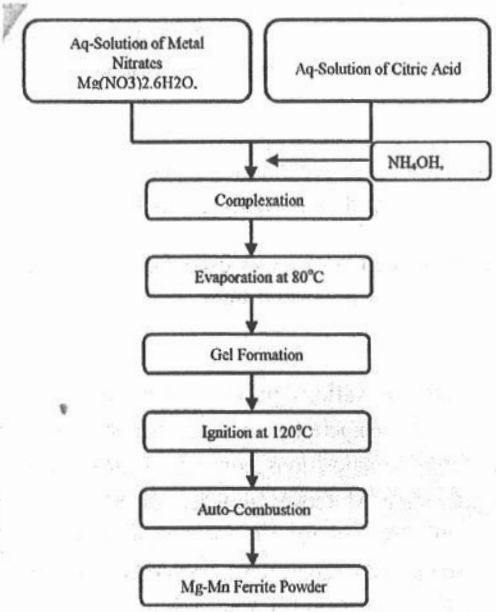

Figure 1: Showing the process flow diagram of the solution combustion process.

RESULTS AND DISCUSSION

All ferrite powders prepared for the present work are phase analyzed by standard XRD and the resulting diffractograms are shown in fig. (2) .

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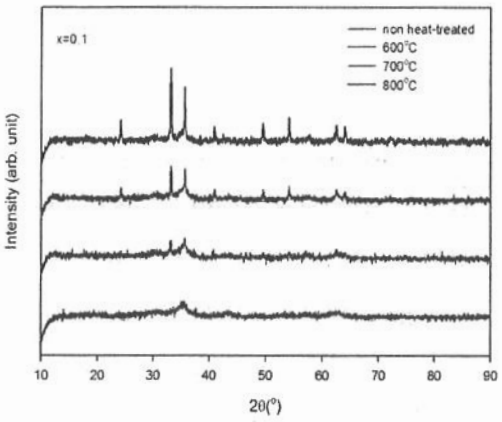

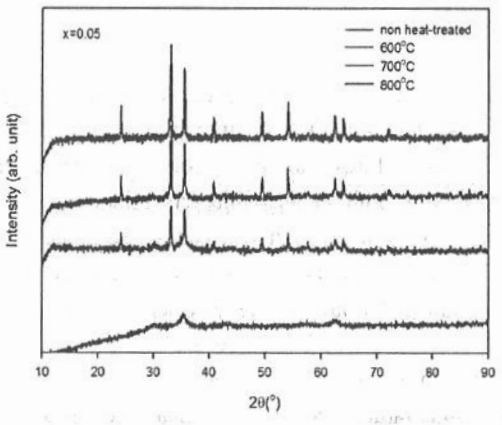

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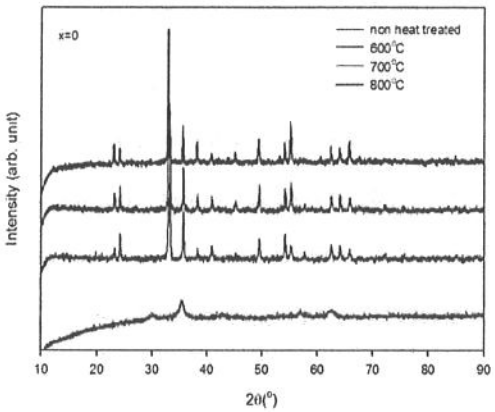

Figure 2: XRD diffraction patterns of the series $MnFe_{2-x}Sm_xO_4$

The diffraction patterns of the as-burnt powders exhibited rather poor resolution, characterized by very small peaks with huge line broadening. Such line broadenings are known to result from small crystallites; moreover prolonged mechanical grinding of powders may subject the particles to severe high energy impacts. Resulting into particle fragmentation and mechanical rewelding of crystalline grains, which would induce considerable amounts of structural and microstructural defects. This is expected to reflect heavily upon the physical properties of the nano crystalline product. However when these initially prepared powders are calcined for 5 hours at 500°, 600°, and 700° separately, the diffractograms became more resolved with the peaks becoming sharper and their intensities progressively enhancing with increasing calcination temperature. Suggesting that the heat treatment process has progressively mended the already deformed crystalline lattice by annealing the remnant strains and restoring atomic order within.

TEM particle size analysis indicated that the powders contained particles in the range of (6-8)nm in diameter, as shown in the micrograph exhibited in fig.(3) where special efforts were needed to achieve reasonable particle dispersion.

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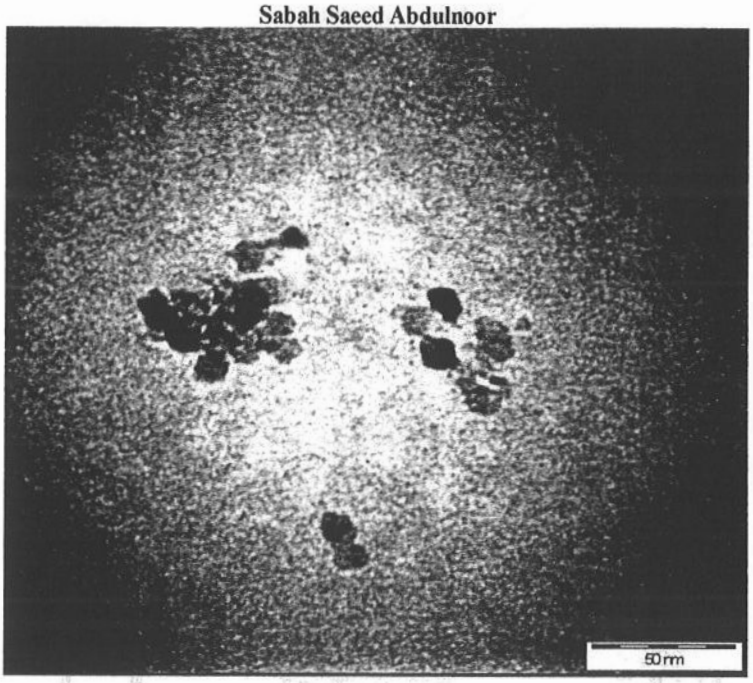

Figure 3: TEM microphotograph of $MnFe_{2-x}Sm_xO_4$ nano particles.

The above mentioned powders were cold pressed into thin pellets and taken for magnetic analysis in their green state. Moreover, a second set of pelletized as-burnt powders were sintered for 5 hours at 1100°C and 1250° C separately, magnetically analyzed by a vibrating sample magnetometer (VSM) and the resulting set of hysteresis loops are exhibited in fig. (4).

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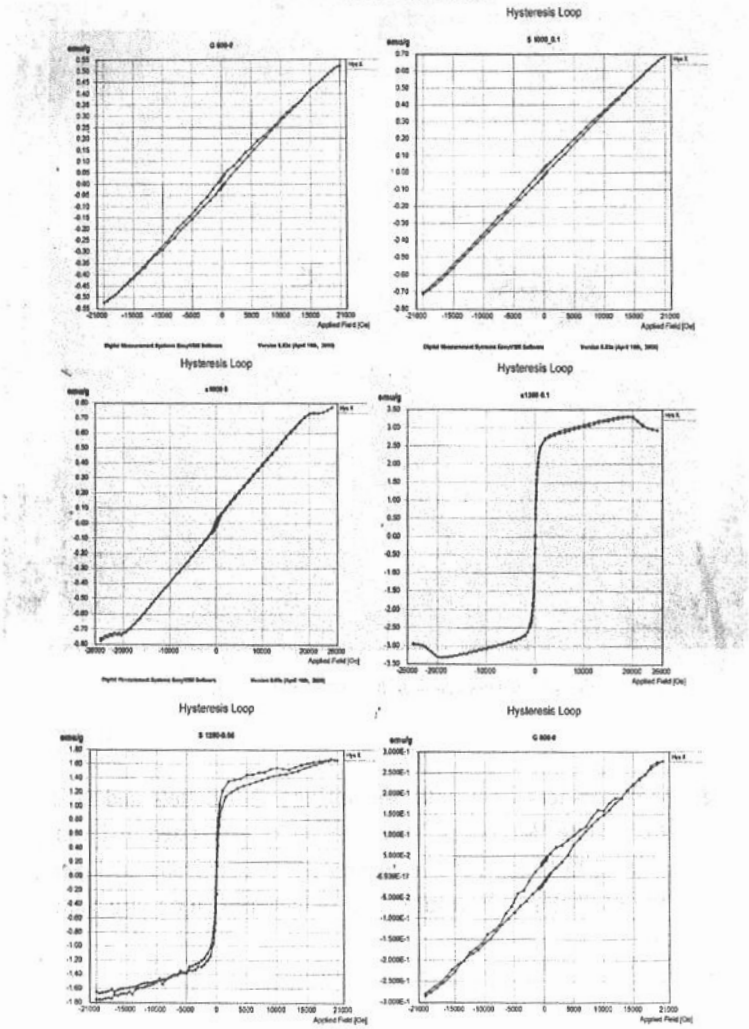

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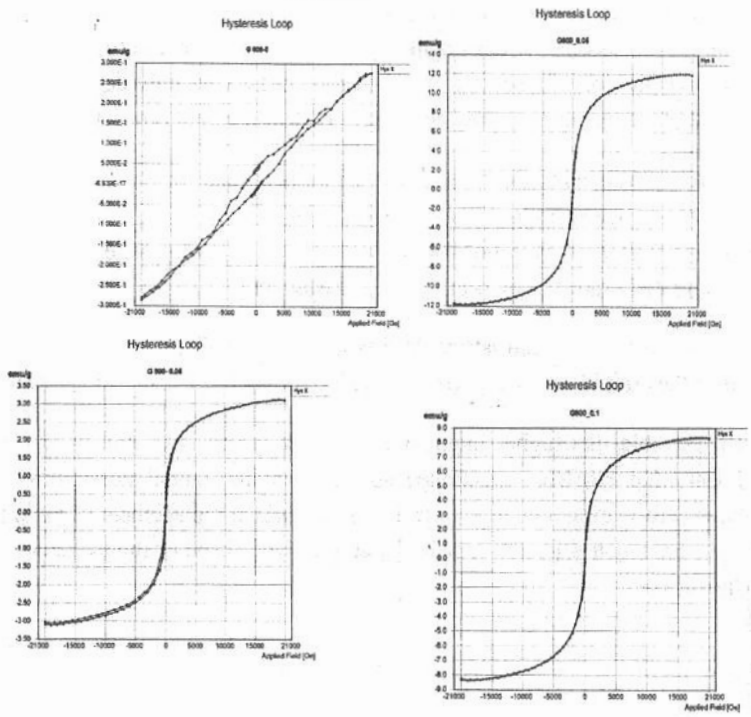

Figure 4: Hysteresis loops of various ferrite powders of $MnFe_{2-x}Sm_xO_4$ with different x-values and various heat treatment schemes.

The hysteresis loop is in fact a state of magnetization of a solid material registered as a function of the strength and direction of the magnetizing field. The exhibited loops suggest that the nano ferrite powders are basically superparamagnetic materials ,which means that the each nano particle is a single magnetic domain individually characterized by spontaneous magnetization(ferromagnetic).Yet the material has no net magnetic moment because the particles of the powder (i,e the magnetic domains) are loose and have no fixed positions. So it will behave like a paramagnet as a whole. The loop in fig.4-x may be considered as an shaped-loop of a superparamagnet, where the remnant magnetization is almost zero, and so is the coercive field. However, the modifications exhibited by the other loops are mainly due to a number of factors.

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- Transition metal-ferrites usually favour spinel structures,while rare earth ferrites might favour other structures like garnet or when temperatures are increased to allow for precipitation of a second phase. Second phases are only detectable by XRD if they exist in percentages ranging between 5-10% of the total powder volume.

- Atomic order is related to the whether the spinel is normal or inverse, and the difference between both is the site occupancy of divalent ions and trivalent ions of the tetrahydral or the octahydral sites, where such inversion might be initiated gradually by heat treatment.

- Local strains, impurities, and lattice defects may also modify Hysteresis loops since they might alter the energy needed to move the Bloch wall.

- It is also possible that higher intensity of internal strains may enhans the coercive field. From what has been mentioned above as circumstantial possibilities for the loop-shape deformation ,one may suggest that the partial replacement of Fe-ion by the Sm-ion is very much expected to modify the observed shape of the hysteresis loop of the ferrite powde.

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