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## Exchange bias effect in nickel zinc ferrite–mesoporous silica nanocomposites

Shilpi Banerjee<sup>a,b</sup>, Partha Hajra<sup>a</sup>, Mykanth Reddy Mada<sup>c</sup>, Asim Bhaumik<sup>b</sup>,  
Sri Bandyopadhyay<sup>c</sup>, Dipankar Chakravorty<sup>a,\*</sup><sup>a</sup> *MLS Professor's Unit, Indian Association for the Cultivation of Science, Kolkata 700032, India*<sup>b</sup> *Department of Materials Science, Indian Association for the Cultivation of Science, Kolkata 700032, India*<sup>c</sup> *School of Materials Science and Engineering, University of New South Wales, Kensington, Sydney-2052, Australia*

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## ABSTRACT

Nickel zinc ferrite–mesoporous silica nanocomposite (NZF–MS) was synthesized using impregnation method. The microstructure was investigated by transmission electron microscopy. A magnetic exchange bias effect was exhibited by the nanocomposites. This was ascribed to the presence of a ferromagnetic core and antiferromagnetic shell structure. Electron microscopic studies confirmed the presence of a core–shell structure with NZF forming the core. The zero–field cooled magnetization data as a function of temperature indicated the presence of an antiferromagnetic phase which is believed to be formed by the diffusion of  $\text{Fe}^{3+}$  or  $\text{Ni}^{2+}$  ions into the silica glass.

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## 1. Introduction

Magnetic nanocomposite materials [1–3] have attracted a lot of attention in recent years. These exhibit the exchange coupling (exchange-bias) at the interface between ferromagnetic and antiferromagnetic phases [4–7]. Exchange bias effect [8] has potential technological applications in spin electronic devices, high density magnetic data storage, magnetic random access memory, etc. Many of the studies on exchange bias have focused on core–shell nanoparticles, bilayers, FM nanoparticle embedded within an antiferromagnetic phase. Magnetic nanocomposites have been fabricated using sol–gel, hydrothermal–solvothermal, coprecipitation, microemulsion method, etc. [5,9,10]. But there is a tendency of agglomeration and coarsening during the thermal treatment which affects their unique properties. To prevent this nanoparticles are embedded within a suitable matrix [11,12]. Magnetic nanomaterials grown within pores of mesoporous materials show different physical properties. These have been found to have technological applications particularly in the fabrication of nanoscale devices [13–15]. Recently the study of highly ordered silica based mesoporous materials have received considerable attention due to their applications in many fields like catalysis, absorbents, optically active materials, sensor devices, etc. [16–20]. There were many types of ordered mesoporous silica template developed viz., SBA-15, MCM-41, KIT-5, KIT-6, etc. Among which KIT-6 (Kentucky Institute of Technology-6) is a three-dimensional cubic highly ordered mesoporous silica template with high pore volume.

KIT-6 can be used as templates in which magnetic compounds can be conveniently confined in a quasi-two-dimensional structure because of its particular topology with cubic symmetry and very small average pore width [21].

In this paper we describe the growth of nickel zinc ferrite inside the pores of an ordered mesoporous silica template. Magnetic properties showed exchange bias effect. Nickel zinc ferrite has a spinel structure in which cations are situated at tetrahedral and octahedral sites. The magnetic behavior has been explained on the basis of a ferromagnetic core–antiferromagnetic shell structure. The details are reported in this paper.

## 2. Experimental

Mesoporous silica template KIT-6 powder was synthesized according to the method reported earlier [22]. 1 g of P123 (poly(ethylene glycol)–poly(propylene glycol)–poly(ethylene glycol)) as a template was dissolved in 36 g of distilled water and 1.96 g of conc. HCl (35%) with stirring for 1 h at 308 K. P123 is a triblock copolymer with chemical formula  $\text{HO}(\text{CH}_2\text{CH}_2\text{O})_{20}(\text{CH}_2\text{CH}(\text{CH}_3)\text{O})_{70}(\text{CH}_2\text{CH}_2\text{O})_{20}\text{H}$  and molecular weight around 5800. 1 g of butanol was added and the solution stirred for 1 h. Then 2.15 g of TEOS (tetraethyl orthosilicate) was added. The mixture was stirred for 24 h at 308 K. The solution was transferred to a Teflon-lined stainless steel autoclave and treated hydrothermally at 393 K for 24 h. The product was filtered and washed with distilled water and dried at 333 K for 1 day. The resulting white powder was calcined at 823 K for 5 h to remove the surfactant. The nanocomposite was prepared by impregnation procedure. At first iron nitrate, nickel nitrate and zinc nitrate salts were dissolved in

\* Corresponding author.

E-mail address: [mlsdc@iacs.res.in](mailto:mlsdc@iacs.res.in) (D. Chakravorty).

ethanol to make a solution. Then 0.5 g KIT-6 silica powder was immersed into ethanolic solution of nitrate salts and stirred for 1 day. The incorporated KIT-6 was collected through filtration and washed with ethanol and distilled water. Then it was dried at room temperature for 1 week. Then the dried powder was heated at 873 K for 4 h. The nickel zinc ferrite (NZF) impregnated MS nanocomposite will be referred to as NZF-MS. The preparation of NZF-MS nanocomposite is shown schematically in Fig. 1.

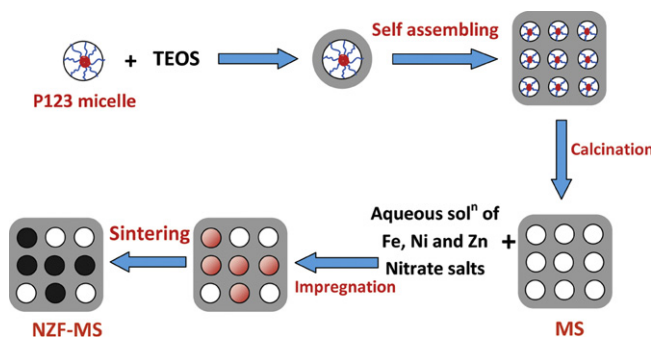


Fig. 1. Schematic representation of preparation of the NZF-MS nanocomposite.

The microstructures of the synthesized MS and NZF-MS were studied by a JEOL Transmission Electron Microscope. Magnetic properties were investigated by an MPMS Superconducting Quantum Interference Device Magnetometer (supplied by M/S Quantum Design U.S.A.). The temperature range of measurement was 2–300 K.

### 3. Results and discussion

Fig. 2(a) shows the transmission electron micrograph of ordered mesoporous silica template (MS). From this it is seen that the diameter of the pore channels has a value around 5 nm. Fig. 2(b) shows the microstructure of the NZF-MS from which it is found that some of the channels of MS are filled with NZF. This was confirmed by the electron diffraction pattern shown in Fig. 2(c) which was obtained from Fig. 2(b). The interplanar spacings were calculated from Fig. 2(c) and these values were in good agreement with JCPDS data with File no. 08-0234 of NZF which are summarized in Table 1. In Fig. 2(d) the high resolution electron micrograph corresponding to Fig. 2(b) is shown. The interplanar spacings corresponding to (311) plane of NZF is marked in the figure.

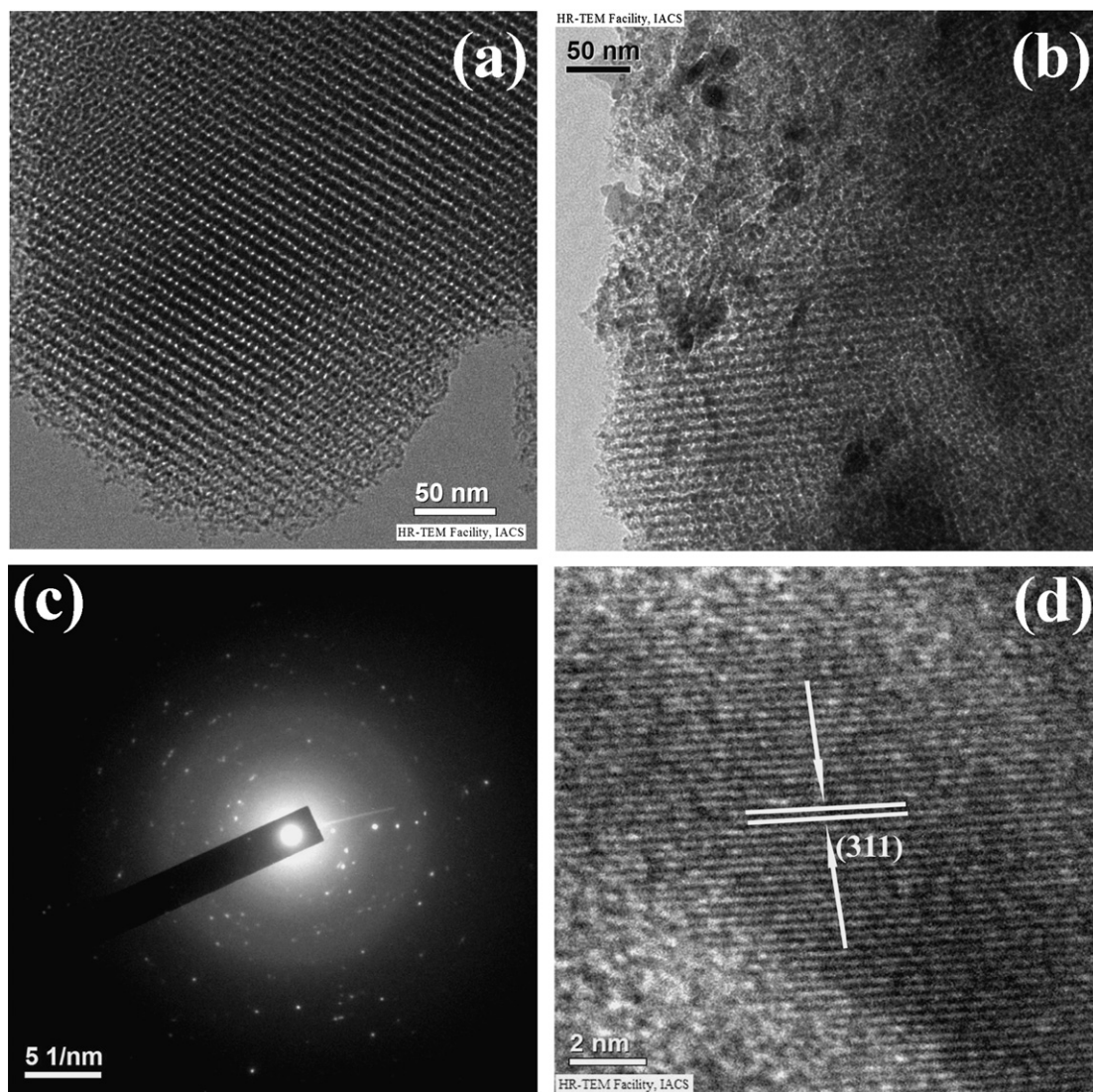


Fig. 2. (a) Transmission electron micrograph of the KIT-6 mesoporous silica template, (b) transmission electron micrograph of the nanocomposite, (c) electron diffraction pattern of (b) and (d) HRTEM image of the nanocomposite.

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