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MAGNETIC AND ELECTRICAL BEHAVIOUR OF Ni_3Fe/SiO_2 NANOCOMPOSITE

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Abstract

The present paper presents a facile and low-cost liquid-solid interaction chemical method to prepare pure Ni and its alloy with Fe nanoparticles by reducing $NiCl_2.6H_2O$ and $FeCl_2.4H_2O$ with aluminum as a reducing agent at room temperature. The structural phase, composition, magnetic properties and morphology of the nanoparticles were monitored by x-ray diffraction (XRD), energy dispersive x-ray analysis (EDX), vibrating sample magnetometer (VSM) and scanning electron microscopy (SEM). The as-prepared Ni nanoparticles are well-defined cubic crystalline structure. In Ni_xFe_{1-x} alloy shows the fcc phase for x=75 at.%, mixture of bcc and fcc phase for x = 0.50 at.%. The magnetization of Ni-Fe alloy nanoparticles increases from 74 to 175 emu/g with Fe percentage.

Keywords: Ni-Fe alloy, Magnetic nanocomposite, Chemical synthesis;

1 INTRODUCTION

Surface coating of nanoparticles with various materials to form core-shell morphologies results in the formation of composite materials that have been used in varied applications such as catalysis, optoelectronics.[1-9] The core-shell structures have also functioned as precursors in the

preparation of hollow structures by the complete removal of core materials through chemical etching or combustion [9,10] and partial elimination of the core has resulted in novel nanostructures inside the shell.[11,12] Surface coating of nanoparticles with silica is one important method adopted in this category. In the case of magnetic materials which are usually metallic in nature, silica offers protection against oxidation by forming a passive layer around the nanoparticle and being a dielectric it also yields nanocomposites for specific applications in the high frequency regime. The silica coating of metal or metal oxide has attracted considerable attention in the past decade, due to the many new and unusual physical, chemical properties arising thereby. [7]

Surface coatings allow manipulation of the interaction potential and offer new possibilities for the shape control of a particle. Silica coating improves the chemical stability and electrical resistivity of the material. The core-Shell concept has been shown to inhibit the oxidation process. Accordingly, the silica coating of magnetic nanoparticles is one of several promising tools to ensure the specific biocompatibility and leads to low toxicity in materials. Recently M. Ammar et. al [13] had made use of the classical Stöber method [14] to encapsulate FeNi nanoparticles within a silica shell. Magnetic-metal nanoparticles encapsulated in a dielectric inorganic material have potential for practical applications in electromagnetic devices, biology and fundamental studies to improve the local physical investigation of magnetic nanostructures.

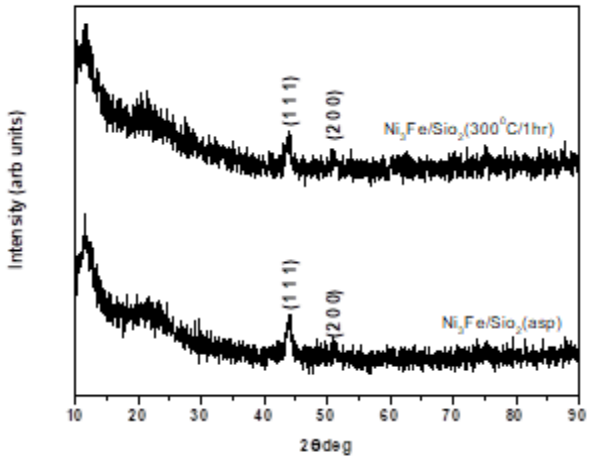

Fig. 1. X-ray diffraction patterns for the as-prepared and annealed samples of silica coated Ni3Fe

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In view of the importance of the stable well ordered Ni_3Fe composition among the various alloys studied in this work, an attempt was made to chemically coat the nanoparticles of this composition with silica and investigate particularly their suitability for high frequency magnetic applications. The structural, microstructural and magnetic properties of the coated nanoparticles were examined as described in earlier chapters. In addition, the dielectric behaviour of this metal-insulator nanocomposite was analyzed by Impedance Spectroscopy. The electrical properties such as dielectric constant, a.c. conductivity and dielectric loss factor were thus determined. The protocols used for impedance measurements are provided in this chapter whereas other characterizations were carried out as described in Chapter 3. The details of the silica coating procedure and results of the investigations on the silica coated Ni_3Fe nanoparticles follow.

2. SYNTHESIS OF SILICA COATED Ni_3Fe NANOPARTICLE

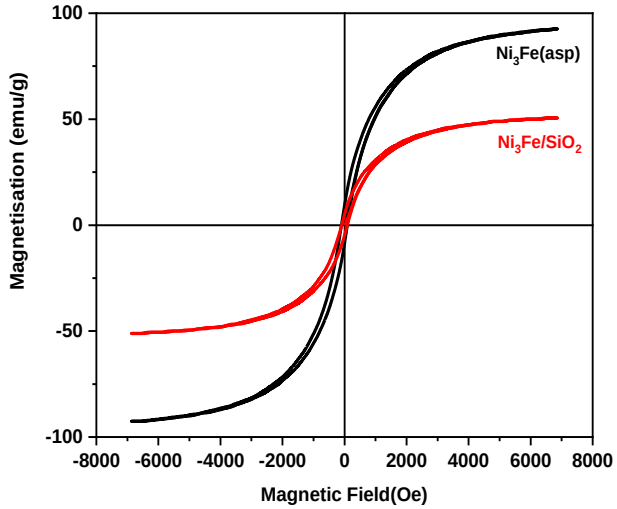

Fig. 2. Hysteresis loops for as-prepared Ni_3Fe and Ni_3Fe/SiO_2 nanocrystalline powder

Firstly, Ni_3Fe (core) nanoparticles were prepared as mentioned in Ref 3. The silica shell for this core was synthesized according to the Stöber method [14]. In order to prepare the Ni_3Fe/SiO_2 core-shell nanocomposite, certain quantity of Ni_3Fe nanoparticles were redispersed in 25 ml of ethanol containing 5 vol% of ammonia (28% NH_3 in H_2O) solution and immediately 25 ml of 10 vol% TEOS in ethanol was added slowly under vigorous stirring for 24 h and the product was then aged for 48 h. The resulting Ni_3Fe/SiO_2 nanocomposite powder was washed

with acetone. Portions of this nanocomposite were also annealed in air at temperatures of 100ºC and 300º C for an hour respectively. Nanocrystalline Ni_3Fe/SiO_2 powder was made into pellets of 6 mm diameter and 0.5 mm thickness with an applied pressure of 70 MPa. The impedance measurements were made for the compacted pellets, using a Solartron 1260 impedance/gain phase analyzer in the frequency range 1Hz –10 MHz from room temperature to250º C. The samples were characterized by XRD, VSM, SEM and impedance spectroscopy (IS)

Fig. 1 shows the XRD patterns of as-prepared and 300º C annealed sample of Ni_3Fe/SiO_2. A characteristic hump in the low angle range confirms the presence of amorphous cristobalite phase [15] of SiO_2 . No evidence for crystalline SiO_2 was seen in the XRD pattern suggesting that the SiO_2 phase in the Ni_3Fe/SiO_2 nanoparticles is in an amorphous state. From the (111) diffraction peak, the size of Ni_3Fe cluster in the Ni_3Fe/SiO_2 composite was estimated to be 8 nm and 9 nm respectively for the as-prepared and annealed samples. The encapsulation of the particle by silica has apparently prevented any appreciable grain growth.

4. ANALYSIS OF MAGNETIC PROPERTIES

Fig. 2. displays the hysteresis loop obtained for the silica coated Ni3Fe powder from VSM measurements comparing it with that reported for the uncoated powder in previous work. From a

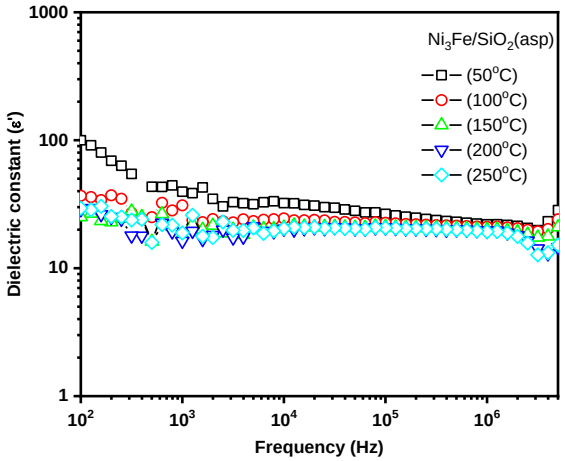

Fig. 6.. Dielectric constant vs. frequency graphs for as-prepared Ni_3Fe/SiO_2 at different temperatures.

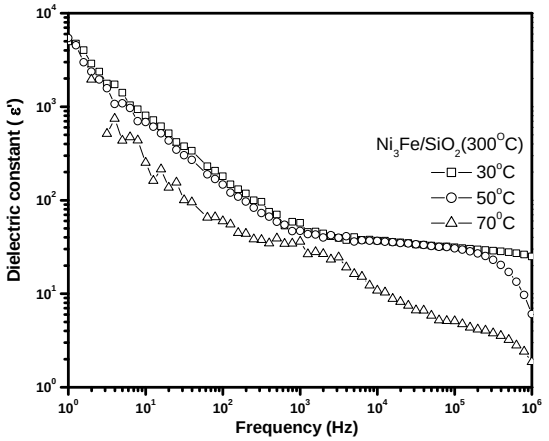

Fig. 7. Dielectric constant vs. frequency graphs for 300ºC annealed Ni_3Fe/SiO_2 at different temperatures

comparison of the saturation magnetization values for coated and uncoated particles in Fig. 2, the Ni_3Fe to silica ratio was estimated to be 59:41, assuming that the magnetization in the coated sample is purely contributed by the available weight % of the ferromagnetic phase. The negligible change in particle size justifies this further. A small decrease in the coercivity value is probably due to morphological changes associated with the coating.

6. IMPEDANCE SPECTROSCOPY ANALYSIS

The complex impedance plots of the as-prepared and 300ºC annealed Ni_3Fe/SiO_2 nanoparticles measured at room temperature in the frequency range of 1 Hz – 10 MHz are shown in Fig. 3.4 and Fig. 3.5 respectively. In impedance spectra at room temperature for both the as-prepared and annealed sample shows the grain and grain boundary effects. Since, due to some technical difficulties a proper fitting of the data could not be completed, only qualitative inferences are drawn. For the as-prepared sample the graph shows a single semicircular arc at high frequency region and in the case of the annealed sample another semicircular arc of small radius has developed. The single semi-circular arc at high frequency region can be attributed to the grain interior while in the annealed sample the grain boundary effect is seen in the second arc. The smaller resistance offered by the boundary is probably because of discrete boundary phases. The two semi-circular arcs can be attributed to different relaxation processes in the grains and grain boundaries. The impedance plots were fitted with complex non-linear least square fit (CNLLS) to obtain the resistance of the sample. The resistance of the as-prepared and annealed samples were found to be 1.29×10^7 Ω and 3.93×10^6 Ω respectively. With the geometry of the sample, this resistance values has been converted into resistivity using the relation,

$\rho = RA/l$

Here R is the resistance of the sample, A is the area of cross-section and l is the thickness of the pellet. The resistivity of the Ni_3Fe/SiO_2 as-prepared samples were calculated to be of the order of 6×10^4 Ω-cm Annealed samples show even greater values of the order of 21×10^4 Ω-cm, this is

mainly due to the densification of silica on Ni_3Fe nanocomposite by removal of the organic impurities.[16] .

From the impedance quantities Z' and Z'' at each frequency , the real and imaginary parts of the frequency dependent dielectric constant can be calculated using

$$\varepsilon'(\omega) = Z' / (\omega C_o | Z|^2) \qquad \varepsilon''(\omega) = Z'' / (\omega C_o | Z|^2) \text{ ---------- (3.1)}$$

The effects of frequency ω on the dielectric constant ε' for the as-prepared and annealed samples are illustrated in fig 6 and 7. For the as-prepared sample ε' remains constant at all frequencies measured at different temperatures.

REFERENCES

1. E. Thirumal, D. Prabhu, K. Chattopadhyay, and V. Ravichandran," Physica Status Solidi (a), vol. 207, no. 11, pp. 2505–2510, 2010

2.E. Thirumal, D. Prabhu, K. Chattopadhyay, V. Ravichandran, J. Alloys. Compd. 502 (2010) 169-175.

3. K. Sakiyama, K. Koga, T. Seto, M. Hirasawa, and T. Orii, J. Phys. Chem. B 108 (2004) 523.

4. D.Yu, J. Colloids . Surf. A: Physicochem. Eng. Aspects 237 (2004) 87.

5. L. M. Liz-Marzan, M.Giersig, and P. Mulvaney, Langmuir 12 (1996) 4329.

6. C. Wang, and A. Harrison, J. Colloid Interface Sci. 217 (1999) 203.

7. B. Dong-Sik, H. Kyong-Sop, and J. H. Adair, J. Mater. Chem. 12 (2002) 3117.

8 .V. V. Hardikar, and E. Matijevic, J. Colloid Interface Sci. 221 (2000)133.

9 . L. Josephson, C. H. Tsung, A. Moore, and R. Weissleder, Bioconjugate 10 (1999) 186.

10. D. Gerion, F. Pinaud, S. C. Williams, W. J. Parek, D. Zanchet, S.Weiss, and A. P. Alivisatos, J. Phys. Chem. B 105 (2001) 8861.

11. J . Lee, S. Hong,Y. Lee, and J. Choi, Adv. Mater.15 (2003) 1655.

12.S .M. Marinakos, M. F. Anderson, J.A. Ryan, L. D. Martin,and D. L. Feldheim, J. Phys. Chem. B 105 (2001) 8872.

13. B. Rodriguez, V. Salguerino, F. Garcia, and M. Liz-Marzan, Nano Lett. 2 (2002) 471.

14 . Y. G. Sun, B.T. Mayers, and Y. N. Xia, Nano lett.2 (2002) 481.

15. M. Ammar, F. Mazaleyrat, J. P. Bonnet, P. Audebert, A .Brosseau, G. Wang and Y. Champion, Nanotechnology 18 (2007) 285606.

16. W. Stober, A. Fink, E. Bhon, J. Colloid Interface Sci. 26 (1968) 62.

17. L. Zhiyu ,H. Chenghui and S. Jiani , J. Mater. Sci .41 (2006) 3473

.

18 . K. M. Chang, S. W. Wang, T. H. Yeh, C. H. Li, and J .J. Luo, J. Electrochem. Soc. 144 (1997) 1754.