Synthesis and Characterization of Nickel Magnetic Monodisperse Nanoparticles

Kash Dev Sharma¹, Girish mishra²

1. Rajshree Institute of Management and Technology, Bareilly

2. Gangasheel Mahavidyalaya Faijulapur, Nawabganj, Bareilly

Abstract

The nickel nanostructures have been synthesized by thermal decomposition of nickel-oleate precursor in presence of 1-octadecene and oleic acid with controlled synthesis temperature. The X-ray diffraction pattern of Ni nanostructures synthesized from nickel oleate at 638 K showed pure (faced center cubic) fcc-Ni phase. The field emission scanning electron microscopy (FESEM) image of Ni nanostructures showed nearly spherical morphology. The average particle size calculated by considering 100 particles is found to be around 28 nm which is close to the crystallite size calculated using Scherrer formula. These changes in nano-materials compared to its bulk lead to anomalous physical and chemical properties. The transition metal nickel exhibits definite magnetic and catalytic properties ^[1]. Nickel nanocrystals are stable in air due to their better anticorrosion ability compared to other metallic nanostructures such as cobalt and iron nanostructures. As the particle size of nickel nanostructures, nickel has attracted much attention because of their numerous practical applications like conducting materials, magnetic materials, and catalysts for hydrogen generation ^[2] and growth of carbon nanotubes ^[3]. Thus, it is of great significance to synthesize high quality nickel nano-materials using convenient and low cost methods ^[4].

Introduction

Nano-materials are of great interest not only in fundamental research but also for wide range of technological applications. The size, shape, and morphology of nanostructures play vital role to control optical, electrical, magnetic, and catalytic properties ^[5]. The study of transitions maetals and metal oxides nano-particles has significantly active research area in the last decade. The Ni nanocrystals have different properties that of the bulk and individual atoms. Nickel is typically crystallizes in two phases, (i) fcc-phase, and (ii) hcp-phase. Nickel in bulk form shows rock-salt structure. Bulk nickel exhibited ferromagnetic (Curie temerrature $T_C = 358$ °C) and electroconductive behavior ^[1]. In comparison to bulk counterparts, Nickel nanostructures exhibit magnetic parameter such as Curie temperature (T_C), saturation magnetization (M_S), and coercivity (H_C) vary with particle size in nonlinear fashion.

Synthesis of Nickel nanoparticles : The Nickel nanoparticles have been synthesized using thermolysis method. For the synthesis of nickel nanostructures, 2.2 g of nickel oleate complex, 21.6 mL of 1-octadecene, and 1.3 mL of oleic acid were properly mixed in five neck round

bottom flask and stirred by mechanical rotor for 2 hours under flow of N_2 gas at room temperature Then, the reaction mixture solution was heated with a constant heating rate of 3 K/min to the temperature 638 K and kept at this temperature for 80 min under the flow of N_2 gas with continuous mechanical stirring. The resulting solution was cooled down to room temperature and then ethanol and hexane were added to the solution. Finally, the nickel nanocrystals were separated by centrifugation and then dried.

Results: Fig. 1.1 shows the θ - 2θ X-ray diffraction (XRD) pattern of nickel nanostructures synthesized from thermal decomposition of nickel oleate precursor at 638 K temperature using mechanical stirrer. The XRD pattern Ni nanostructures synthesized at temperature 638 K shows five sharp at 44.78°, 52.16°, 76.72°, 93.23° and 98.76° which can be indexed as the (111), (200), (220), (311) and (222), respectively. The XRD pattern Ni nanostructures synthesized at temperature 638 K reveals the formation of pure face centered cubic (fcc) phase of Ni. The x-ray diffraction pattern does not show any impurity peaks, indicating the Ni nanostructures obtained by thermolysis method consist of pure fcc phase of Ni. The average lattice parameter calculated from the X-ray diffraction pattern of pure fcc-Ni phase is found to be a = 0.350 nm which is close to the bulk lattice parameter 0.353 nm ^[5]. The average crystallite size calculated using Scherrer formula is found to be around 21 nm.

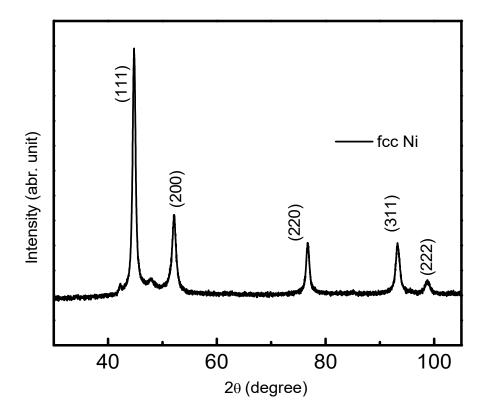


Fig. 1.1 θ -2 θ X-ray diffraction pattern of Ninanostructures synthesized using thermal decomposition method with mechanical stirring.

The field emission scanning electron microscopy (FESEM) image of Ni nanostructures synthesized by thermal decomposition of nickel oleate at 638 K temperature using mechanical stirrer is shown in Fig. 1.2. The FESEM image of Ni nanostructures indicates that the nanoparticles of fcc-Ni are weakly agglomerated. The morphology of Ni nanostructures is nearly spherical and the average particle size calculated by considering 100 particles is found to be around 28 nm.

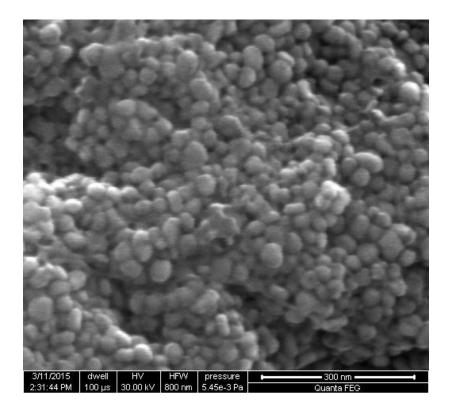


Fig. 1.2 shows the FESEM image of Ni nanostructures synthesized at temperature 638 K.

The M(H) of these fcc-Ni nanostructures show the hysteric field dependent magnetization. The saturation magnetization (Ms) was extracted by extrapolating the linear part of the hysteresis loop toµ₀H = 0. The saturation magnetization of fcc-Ni is ~35.2 emu/g, at 20 K which is relatively lower than the bulk value (~ 55 emu/g). The decrease in M_S for fcc Ni nanostructures compared with bulk might be due to the decrease of particle size ^[6] and the presence of surfactant on magnetic nano-particles surface ^[7, 8]. The M_S (32.5 emu/g) measured at 300 K is closer to the value observed at 20 K. The remanent magnetization (*M_r*), and coercivity (*H_C*) values are 11.2 emu/g, and 0.0284 T, respectively. While the remanent magnetization (*M_r*), and

coercivity (H_C) of these nanostructures are 2.48 emu/g, and 0.0039 T, respectively. The M_r and H_C of these nanostructures observed at 20 K are found to be lower than that of the 300 K.

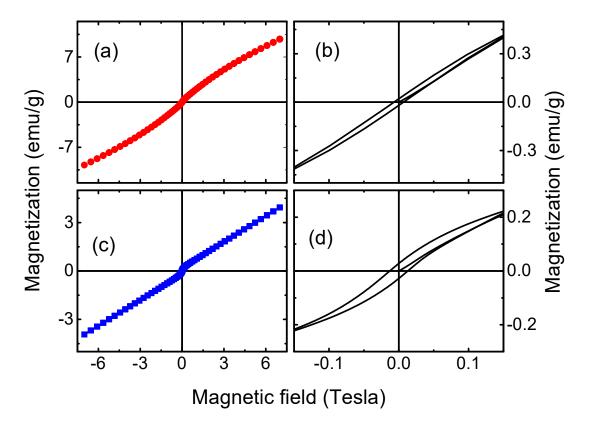


Fig. 1.3 Field-dependent magnetization of CoO nanostructures measured at 20 K (a) and 300 K (b). The low field magnetization data are shown in panel (c) 20 K and (d) 300 K.

Conclusion: The X-ray diffraction pattern of Ni nanostructures showed the fcc-Ni phase. FESEM image of the Ni, nanostructures showed nearly spherical morphology. The average size of the spherical particles calculated for Ni is around 28 nm. The field-dependent magnetization of Ni nanostructures at 20 K exhibits hysteretic behavior with saturation magnetization (M_S), remanent magnetization (M_r), and coercivity (H_c) values as 35.2 emu/g, 11.2 emu/g, and 284.4 Oe, respectively. At room temperature there is a mild reduction in MS, but a significant reduction in H_c .

References:

[1] Xuemin He, Wei Zhong1, Chak-Tong Au and Youwei Du (2013) Size dependence of the magnetic properties of Ni nanoparticles prepared by thermal decomposition method, *Nanoscale Research Letters* 8, 446.

[2] Ö. Metin, V. Mazumder, S. Özkar, and S. Sun (2010) Monodisperse nickel nanoparticles and their catalysis in hydrolytic dehydrogenation of ammonia borane, *J. Am. Chem. Soc.* 132, 1468.

[3] E. F. Kukovitsky, S. G. L'vov, N. A. Sainov, V. A. Shustov, and L. A. Chernozatonskii (2002) Correlation between metal catalyst particle size and carbon nanotube growth. *Chem. Phys. Lett.* 355, 497 (2002).

[4] N. Bao, L. Shen, W. An, P. Padhan, C. Heath Turner, and A. Gupta (2009) Formation mechanism and shape control of monodisperse magnetic CoFe2O4 nanocrystals, *Chem. Mater Chem. Mater*. 21, 3458–3468.

[5] HaiTao Zhang, Jun Ding, GanMoog Chow, Min Ran, and JiaBao Yi (2009) Engineering magnetic properties of Ni nanoparticles by non-magnetic cores, *Chem. Mater.* 21, 5222–5228.

[6] E. V. Gopalan, K. A. Malini, G. Santhoshkumar, T. N. Narayanan, P. A. Joy, I. A. Al-Omari, D. S. Kumar, Y. Yoshida, and M. R. Anantharaman (2010) Template-assisted synthesis and characterization of passivated nickel, nanoparticles. *Nanoscale Res. Lett.* 5: 889-897.

[7] M. Respaud, J. M. Broto, H. Rakoto, and A. R. Fert (1998) Surface effects on the magnetic properties of ultrafine cobalt particles. *Phys. Rev.* B 57, 2925.

[8] M. V. Limaye, S. B. Singh, S. K. Date, D. Kothari, V. R. Reddy, A. Gupta, V. Sathe, R. J. Choudhary, and S. K. Kulkarni (2009) High coercivity of oleic acid capped CoFe2O4 nanoparticles at room temperature. *J. Phys. Chem.* B 113, 9070.