



A COMPREHENSIVE STRUCTURAL AND MAGNETIC STUDY OF NI NANOPARTICLES PREPARED BY THE CHEMICAL REDUCTION

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ABSTRACT

Nickel nanoparticles around 12nm were prepared by chemical reduction of Nickel Chloride dissolved in Ethylene glycol, in which hydrazine act as a reducing agent and NaOH as stabilizer. The powder XRD study confirms that the average size of the particle is 12nm. The Ni particles show the characteristic of super paramagnet with saturation magnetization (M_s) and the remanent magnetization (M_r) at 5K is 2.55 emu/g and 1.11 emu/g, respectively.

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INTRODUCTION

In the past two decades, considerable attention has been devoted to the synthesis of metal nanoparticles because of their unusual properties and potential applications in optical, electronic, catalytic and magnetic materials [1-3]. Until now, only a few works on the preparation of Nickel nanoparticles have been reported and they usually were performed in Organic media to avoid the formation of nickel oxide or hydroxide [4]. Szu-Han Wu *et al.* [5] and Yang long Hou *et al.* [6] prepared Nickel nanoparticles by chemical reduction method and characterized Nickel to be pure crystalline with a FCC structure. Xiao-Min Ni *et al.* [7] had prepared the Nickel nanorods in Water-in-oil micro emulsion technique with a FCC structure. Kumar *et al.* [10] investigated that Nickel nanoparticles show a typical behaviour for their field-dependent magnetization. Kumar *et al.* [8] investigated that Nickel nanoparticles show a typical behaviour for their field-dependent magnetization. Yingwen Duan *et al.* [9] have synthesized Ni nanoparticles by spontaneous autocatalytic reduction and found that the specific saturation magnetization decreases as the grain size decreases. Our objective in this paper is to synthesize nickel nanoparticles by chemical reduction method using nickel chloride and hydrazine in an aqueous solution.

MATERIALS AND METHODS

Ethylene glycol was chosen as a solvent and NiCl_2 was dissolved in it. Then, an appropriate amount of hydrazine was added. Hydrazine act as a reducing agent & NaOH, which acts as a catalyst of 10-72 μml , were added in sequence and stirred well. The resultant product was taken and kept in the water bath above the room temperature and heated. No significant change takes place. When the temperature raised to above 90° C, there was a change in the resultant product. That is, the bluish green color precipitate slowly changed in to black

color, indicating the formation of Nickel nanoparticles. Then precipitate is taken in the silica crucible and heating the precipitate in the water bath at the temperature of ~96° C to get the dried black Ni nanopowders. The XRD pattern of the sample was collected with a Rigaku D/Max-2000 diffractometer equipped with a Cu K α radiation source ($\lambda \sim 0.15418 \text{ nm}$).

RESULTS AND DISCUSSION

Structural studies

Fig.1 shows a typical XRD pattern of the Nickel sample. From the Fig three characteristic peaks for Nickel [$2\theta = 44.6^\circ, 51.9^\circ, 76.4^\circ$] corresponding to Miller indices (111), (200), (220) were observed. This revealed that the resultant particles were pure face-centered cubic (FCC) Nickel. No obvious peaks of Nickel oxides and hydroxides were detected, possibly attributed to the observed phenomenon that Nitrogen gas was produced and bubbled up continuously during the reaction. It could be suggested that the above said gas produced might auto create an inert atmosphere; hence the input of extra Nitrogen gas was not necessary for the synthesis of Nickel nanoparticles [4,5].

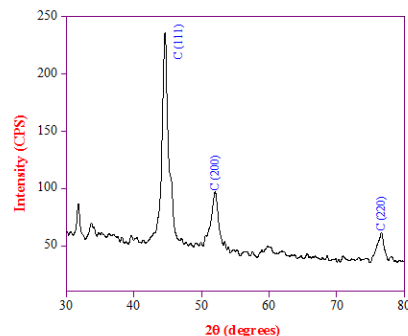


Fig. 1. XRD Spectra of Nickel Nanoparticles

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In Fig 1, the intensity ratio of the peaks [111]: [200]: [220] were calculated to be [100, 26, 16] which exactly coinciding with the JCPDS [no: 040850] values. This confirms the presence of Nickel particles in the sample and it is in good agreement with the earlier reports [7, 10-12]. However from the Fig 1 intensity ratios are not coinciding with the JCPDS values. This observed variation may be due to the change in the synthesis parameter (i.e., due to the heat at $\sim 96^\circ\text{C}$). The lattice constant and average particle sizes of the sample calculated from the full-width at medium height of the peaks in their XRD patterns using Scherrer formula (1) were summarized in the Table 1.

Table 1. XRD spectrum of Nickel nanoparticles

Sample	2 θ Degree	FWHM (β) Degree	h k l	Experimental	Standard cubic (JCPDS-040850)	Particle Size (D) (nm)	Lattice Constant a (\AA)
				d (\AA)	d (\AA)		
Ni	44.6	0.9444	111	2.03003	2.0340	9.503	3.5161
	51.9	1.2800	200	1.76139	1.7620	7.213	2.4909
	76.5	1.2300	220	1.24417	1.2460	8.593	3.5190

Magnetic Studies

The magnetic measurements were carried on the Nickel nanoparticles at a temperature 5K and 300K. Fig 2. shows the field dependence of the Magnetization M at 300K. Within the experimental error, hysteresis was not observed at 300K, which clearly explains the absence of Coercive force. Usually, the magnetic particles having zero coercive force (H_c) exhibits super paramagnetic state rather than ferromagnetic state [13]. This is due to the quantum size confinement effect, in Nickel nanoparticles. A gradual increase of magnetization (M) is observed below and above 2K Oe respectively. The saturation Magnetization (M_s) has been calculated from the Fig.2 as $M_s=0.9236$ emu/g. This is lower than the constant saturation value 55 emu/g of bulk Nickel [14].

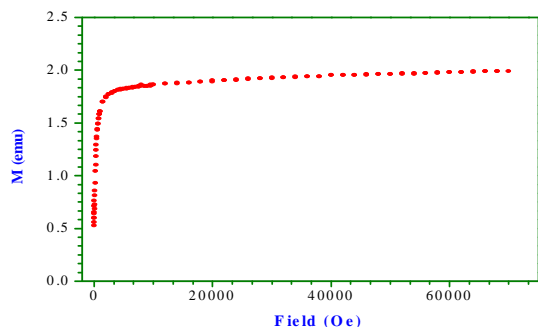


Fig.2. Field dependence of the Magnetization at 300 K

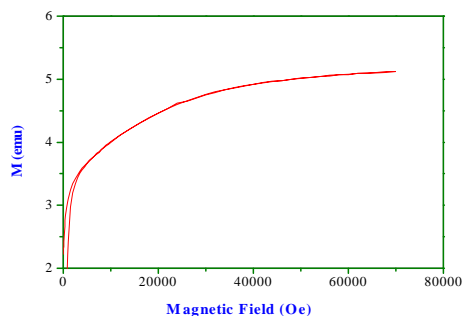


Fig.3. Field dependence of the Magnetization at 5K

Since the magnetization of bulk Nickel shows saturation exceeding 0.2K Oe [15], the gradual increase around 1 K Oe is described to the

super paramagnetic behavior. The decrease in M_s might be due to the decrease in particle size and accompanied increase in the surface area. The Fig.3 displays the field dependence of the magnetization at 5K. Existence of hysteresis is clearly observed below 2.5K Oe. A striking feature of the hysteresis curve is that the magnetization does not saturate even at 70K Oe. It is due to the existence of paramagnetic components. This is because the particles do not have adequate thermal energy to attain complete thermal equilibrium with the applied field during the measurement time and hence exhibited a hysteretic feature. In Fig 3 the saturation magnetization (M_s) and the remanent magnetization (M_r) for the Nickel nanoparticles at 5K are

2.55 emu/g and 1.11 emu/g, respectively. It was observed as in Figs 2 and 3 that the magnetization strongly increased on decreasing temperature. This conforms the super paramagnetic behavior of the Nickel nanoparticles. The magnetic molecules on the surface lack coordination and the spins are likewise disordered. This Phenomenon is more significant for nanoparticles due to their large surface-to-volume ratio and may be another factor that leads to the decrease in M_s [16]. The lower value of saturation magnetization (M_s) compared to bulk nickel can be explained in terms of the core-shell morphology of the nanoparticles. If this surface layer were absent, the magnetization of the particles would saturate with increase in applied field, up to a particular magnetic field the core magnetic moments align with the applied magnetic field. At some stage the resource of the "core mode" of the magnetization response is exhausted and the "core magnetization" of the system is saturated in a usual Langevin – like way. Beyond this stage any increase in the magnetic field on the particles has an effect only on the surface layer of the particles and thus the increase in the magnetization of the particles slow down. The specific state of the surface plays a virtual absence of saturation magnetization that keeps the hysteresis loops unclosed even in very strong fields. As discussed above, a protective layer from ethylene glycol might be formed on the particle surface. The protective layer also could cause the decrease in saturation magnetization (M_s). Accordingly, it is reasonable that the magnetization of nanoparticles is usually smaller than that of corresponding bulk materials [5].

Conclusions

The Nickel nanoparticles have been synthesized by chemical reduction method. As per the XRD analysis the structure of the Nickel nanoparticles is found to be FCC crystalline in nature, with the average particle size calculated as 12 nm. The values of saturation magnetization of Nickel nanoparticles were found as 0.9236 emu/g and 2.55 emu/g and these of the remanent magnetization as 0.298 and 1.11 emu/g at 300 K and 5K, respectively. These quantities exhibit an increasing trend with decrease in temperature as expected. The size reduction in Nickel nanoparticles leads to the change in ferromagnetic to super paramagnetic state.

REFERENCES

1. Fendler, J.H. 1987. Atomic and molecular clusters in membrane mimetic chemistry, *Chem. Rev.* 87: 877-99.
2. Schmid, G. 1992. Large clusters and colloids. Metals in the embryonic state, *Chem. Rev.* 92: 1709-727.
3. Lewis, L.N. 1993. Chemical Catalysis by Colloids and Clusters, *Chem. Rev.* 93: 2693-730.
4. Chen, D. and Wu, S. 2000. Synthesis of Nickel Nanoparticles in Water-in- oil Microemulsions, *Chem. Mater.* 12:1354-60.

5. Wu, S.H. and Chen, D.H. 2003. Synthesis and characterization of nickel nanoparticles by hydrazine reduction in ethylene glycol, *J. coll. Int. sci.* 259:282-86.
6. Yang long Hou and Song Gao, 2003. Monodisperse nickel nanoparticles prepared from a monosurfactant system and their magnetic properties, *J. Mater. Chem.* 13: 1510-512.
7. Ni, X.M., Su, X.B., Yang, Z.P. and Zheng, H.G. 2003. The preparation of nickel nanorods in water-in-oil microemulsion *J. Cryst. Growth*, 252:612-17.
8. Kumar, D., Pennycook, S. J., Lupini, A., Duscher, G., Tiwari, A. and Narayan 2002. Synthesis and atomic-level characterization of Ni nanoparticles in Al₂O₃ matrix, *J. Appl. Phys. Lett.* 81:4204-206.
9. Yingwen Duan and Jiangong Li, 2004. Structure study of nickel nanoparticles, *Materials Chemistry and Physics*, 87:452-54.
10. Hou, Y., Kondoh, H., Ohta and Gao, S.2005. Size-controlled synthesis of nickel nanoparticles, *Applied surface Science*, 241: 218-22.
11. Zhang, D. E., Ni, X. M., Zheng, H.G., Li, Y., Zhang, X. J. and Yang, Z. P.2005. Synthesis of needle-like nickel nanoparticles in water-in-oil microemulsion, *Materials Letters*, 59: 2011-2014.
12. Meining Wu, Yong Chun Zhu, Huagui Zheng and Yitai Qian,2002. A metal-reduction route to synthesis of nickel nanotubules, *Inorganic Chemistry Communications*, 5: 971-74.
13. Erb, U., El-Sherik A. M., Palumbo, G. and Aust, K.T.1993. Synthesis, structure and properties of electroplated nanocrystalline materials, *Nanostruct. Mater.* 2:383-390.
14. Danan, H., Herr, A. and Meyer, J. P. 1968. New Determinations of the Saturation Magnetization of Nickel and Iron, *J Appl Phys*, 39:669-70.
15. Kittel, C. 1996. Introduction to Solid State Physics, 7th ed. Wiley and Sons, Newyork, 15: 448.
16. Dong-Hwang Chen and Chih-Hsuan Hsieh, 2002. Synthesis of nickel nanoparticles in aqueous cationic surfactant solutions, *J. Mater. Chem*, 12:2412 -415.
