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Research Paper

Large Scale Synthesis of Ni nanoparticles by Novel Chemical Method

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ABSTRACT

Ultrafine particles of ferromagnetic nanocrystalline Ni was synthesized by a novel chemical method in aqueous medium. Compositional analysis by Energy dispersive X–ray analysis showed that the as-prepared Ni powders are pure. Crystalline size of as-prepared Ni Powders was controlled by precursor concentration. X-ray diffraction studies indicated fcc crystal structure. Saturation magnetization increases as a function of crystalline size. Surface morphologies and magnetic properties were discussed.

1 Introduction

Ultrafine nano-metallic powders have important potential for wide variety of applications counting catalysts[1], magnetic recording media[2], magnetic fluids[3], magnetic sensors[4] and composite materials. Recently, nanocrystalline ferromagnetic materials such as NiCo, FeNi and CoFe have been found to be promising candidates for high frequency applications[5]. Various techniques exist for the preparation of metal powders. Finely isolated metal particles can be attained by reducing metal salts in aqueous or non-aqueous solvents using alkali metals (lithium, sodium, or potassium). However in the literature there is no report on the synthesis of metal powders using aluminium (alkaline metal) as a reducing agent. Here we have prepared for the first time, the nanocrystalline metal powders by reducing metal salts in aqueous medium using Al metal powder. In this project work we have processed this technique to produce

nanocrystalline powders from a Ni halide solution in water at room temperature. The aim of this research is to create the fine Ni powders and observe the properties of obtained nanoparticles to compare with other methods. In this chapter we describe the observed results of pure nanocrystalline nickel powders synthesized by Al reduction chemical method.

2 Sample Preparation

Chemical production is a smart manufacture technique due to the lack of focused and costly equipment required, the ready availability to simply increase the quantity of materials produced. Rieke and co-workers[6] have developed a general approach for preparing highly reactive metal powders by reducing metal salts in hydrocarbon solvents using alkali metals (Ref.7). Recently D.L. Leslie-Pelecky and co-workers have used high-temperature chemical synthesis to produce cobalt nanocrystalline by reducing cobalt iodide salts in undecane and pentadecan solvent reacting with lithium[8]. Their limited solubility of anhydrous cobalt iodide in decane prohibits synthesis at room temperature. In our earlier reports on Fe and FeCo shows best alternative method for large scale synthesis of metal alloy nanoparticles[9-10]. In this work, we have carried out room temperature reaction of nickel chloride hexahydrate and aluminum powders in aqueous medium to produce nanostructure nickel.

Nanocrystalline nickel is produced using reduction of the form

$$3NiCl_2 + 2Al \rightarrow 3Ni + 2AlCl_3$$

All manipulations were carried out under air atmosphere in the closed glow box. After completion of the above exothermic reaction, remaining solution consisted of Ni, Al and $AlCl_3$, due to reduction of $NiCl_2$ by Al via the above exothermic reaction. The bi-products such as $AlCl_3$ and excess of Al powder can be easily washable by water and alkaline solution of sodium hydroxide using magnetic decantation.

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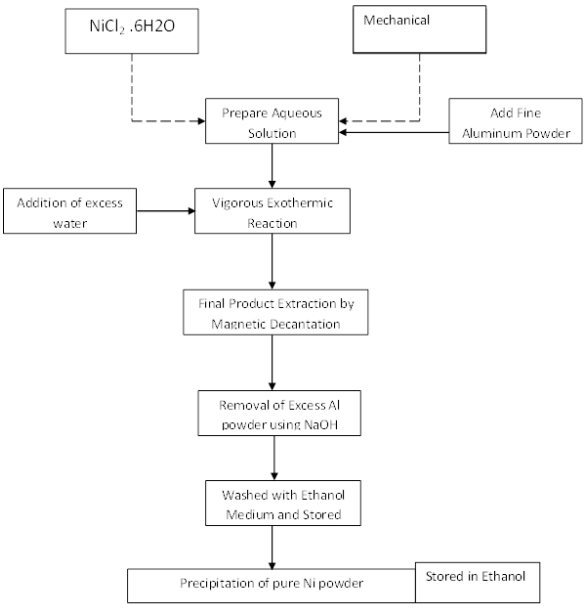

Fig. 1. Schematic diagram of the novel chemical process employed in the synthesis of nanocrystalline Nickel-Iron alloy powder.

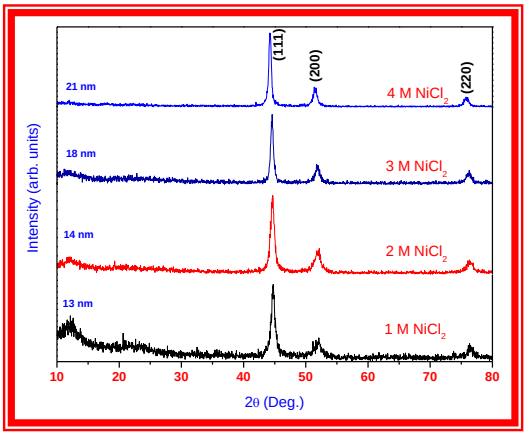

Fig2: XRD patterns for nanocrystalline pure nickel prepared using various Ni salt concentrations. The peak indices and grain size from line broadening are shown

3. Characterization of Pure Nanocrystalline Nickel

3.1 X-ray diffraction

Fig. 1 shows the XRD pattern of as-synthesized nickel powders with 1, 2 and 3 M concentration of nickel chloride hexahydrate. It can be seen that all the sample shows crystallinitiy with peak positions corresponding to the Miller planes (111), (200) and (220). These miller planer are correspond to face centered cubic crystalline structure**.** However there are no peaks corresponding to NiO or $Ni(OH)_2$ which indicates the high purity of the final products successfully synthesized by our novel chemical method with experimental conditions. The effects of nickel chloride concentration on crystalline size of nickel are shown in Table 1. The mean crystalline size of the nickel increases with increasing nickel chloride concentration at a constant concentration of aluminum powder. Average crystalline (D) size was calculated using Sherrer's formulae

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

and lattice constant could be calculated by the formulae

$(1/d_{hkl}) = (h^2 + k^2 + l^2)/a^2$.

These values are listed in Table1.

Table 1 Variation of grain size and lattice parameter as function of Ni^{2+} ion concentration

It can be seen that there is very small change in the lattice parameter with change in crystalline size.

Conc. of $NiCl_2$	**Grain size**	**Lattice parameter**
1 M	13 nm	0.351 nm
2 M	14 nm	0.351 nm
3 M	18 nm	0.352 nm

3.2 Compositional Analysis

Elemental analyzes of the as-prepared samples were analyzed using energy dispersive X-ray analysis (EDX). Fig 3.2 shows the obtained EDX spectra of nickel powder synthesized from 0.2 M concentration of nickel chloride. Results from EDX spectra shows that the sample containing 97at% Ni and 3at% Al. The residual aluminum powder may attributed to improper washing and formation of $Al(OH)_3$ layer on the nickel particles.

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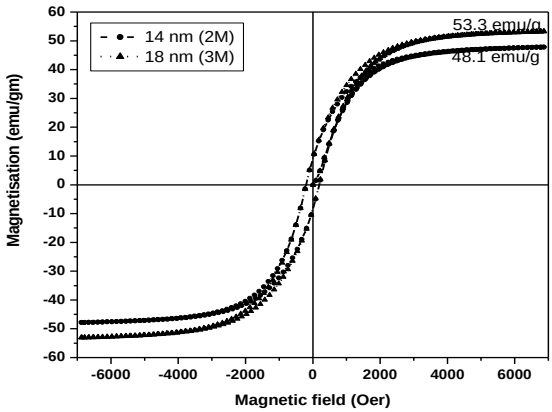

Fig.5 : Hysteresis plots for the nano-Ni samples compared by their crystalline grain sizes.

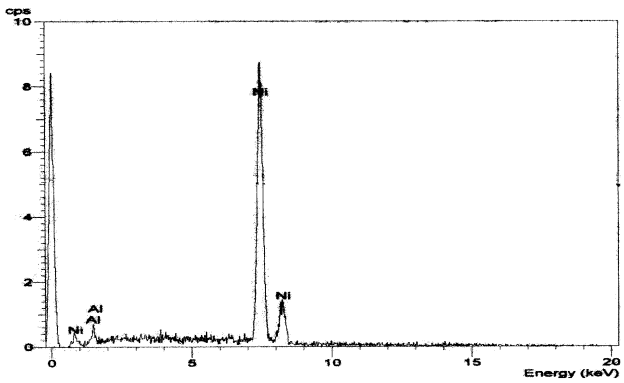

Fig.3. Energy dispersive X-ray analysis of Ni nanoparticle.

3.3 Microstructure Studies

Direct observation of the powder was made by scanning electron microscopy to reveal the shape of the particles. Fig. 4 shows the SEM microstructure of as-synthesized nickel powders

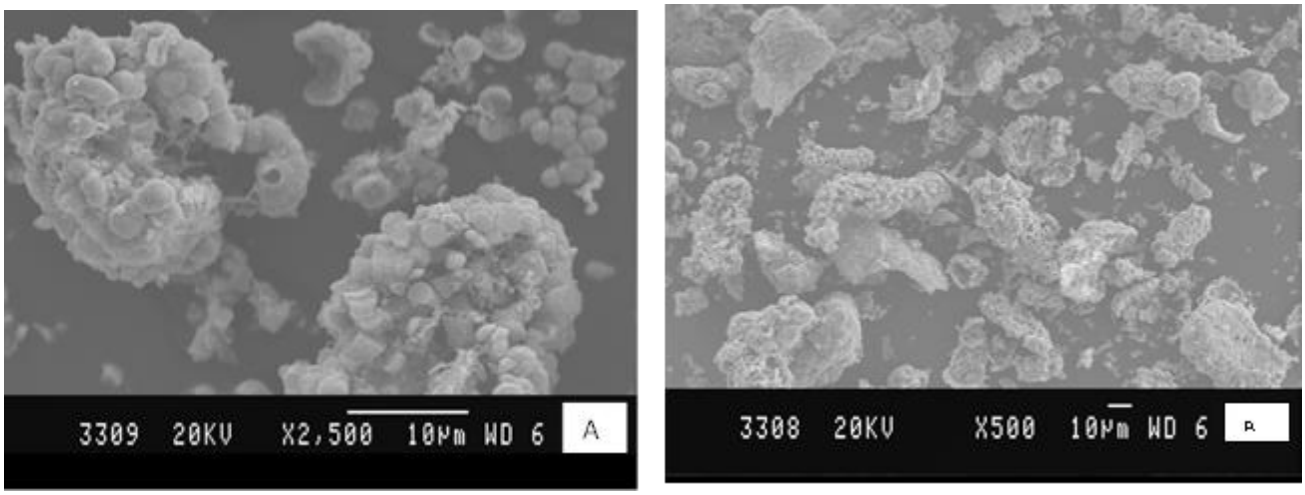

Fig.3.3: (A) Low- and (B) high-magnification SEM images of Ni nanocrystalline synthesized with the 0.2 M concentrations of nickel chloride

for 0.2 M concentration of Ni^{2+} solution. The larger particle size confirms that the agglomeration of particles, which can be attributed by magnetic interaction between nanocrystalline nickel, since the grain sizes observed from the XRD pattern are much smaller.

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3.4 Magnetization studies

The hysteresis loops were recorded for asprepared Ni samples at room temperature (300 K), it was found that all the samples exhibits ferromagnetic behaviour with change in saturation magnetization. Fig 5 shows **t**he hysteresis loop (at room temperature) of the as-obtained nickel powders of 14 nm and 18 nm crystalline.

The saturation magnetization values (M_s) of 0.2 M and 0.3 M samples are 48.1 and 53.3 emu/g respectively. This is very close to the bulk value. The small difference in saturation magnetization value might be attributed to the variation in the grain size and accompanied by increase in surface area followed by more oxidation with decrease in grain size. However, observed remnant magnetization (*Ms*) and coercivity (*Hc*) values for both the samples are 8.7 emu/g and 196 Oe respectively.

The *Ms*, *Mr* and *Hc* values of bulk nickel at 300 K about 55 emu/g, 2.7 emu/g and 100 Oe, respectively[8] (R). The increased *Mr* and *Hc* value might be attributed to difference in microstructure.

4. Conclusions

We have achieved alkali metal (Al) reduction of Ni cations in aqueous medium to produce small magnetic particles. The average grain sizes were dependent on the precursor concentration. The observed saturation magnetization value of pure nickel powders were very close to the bulk value. No extra inert atmosphere was required. The effect of precursor concentration on the structure and magnetic properties of Ni samples were studied. The saturation magnetization increases linearly with crystalline size.

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