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Research Paper

Large Scale Synthesis of Nanocrystalline $Ni_{1-x}Co_x$ Alloy by Direct Chemical Method

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Abstract

The present paper presents a facile and low-cost liquid-solid interaction chemical method to prepare pure Ni-Co alloy nanoparticles by reducing $NiCl_2.6H_2O$ and $CoCl_2.4H_2O$ with aluminum as a reducing agent at room temperature. This simple and direct chemical method gives particles of Nanocrystalline NiCo alloy powder in aqueous medium.. Compositional analysis showed that the precursor ratio was almost preserved in the Ni-Co alloy powders. X-ray diffraction indicated fcc structure for Ni-Co solid solutions.. Saturation magnetization increases as a function of Co content in Ni-Co alloy and the alloy with high Co content saturates only at a high field. In this work we report promising chemical method for preparation Ni-Co alloy for large scale production with low cost.

Keywords: Ni-Co alloy, Magnetic nanoparticles, Chemical synthesis;

1 INTRODUCTION

Nickel and Cobalt nanocrystals show size dependent structural, magnetic, electronic and catalytic properties. Critical size for magnetic cobalt particle to become single domain is proposed to be below 20 nm [1]. Size effect related to free energy changes may determine crystal structure. Magnetic nanoparticles prepared by high pressure sputtering show size induced phase

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Vol.11, Iss.9, Dec 2022

transition from hcp to fcc below 30 nm [2]. This phase transition from hcp to fcc as a function of size is attributed to lower surface energy of fcc-Co phase [2]. Molecular beam deflection measurements show enhanced magnetic moment per atom in small cobalt clusters compared to bulk cobalt [3]. This result is confirmed by cobalt nanoparticles of 1.8 nm prepared using microemulsion technique [3]. The variation of superparamagnetic blocking temperature (T_B) and anisotropy constant as a function of particle size is studied well in literatures [3, 4]. This paper discusses about the synthesis and characterization of Co nanoparticles and the effect of capping ligands on their structural and magnetic properties. Ferromagnetic nanocrystalline materials such as NiCo, FeNi and CoFe have been found to be capable materials for high frequency applications [5-6]. The Ni-Co binary alloy is an excellent system with good chemical stability and high magnetic anisotropy. It forms a complete solid solution with fcc structure over a wide composition range despite some phase transitions in bulk state This offers a possibility to investigate the composition dependence of magnetic properties in a simple manner. Though chemical reduction of metal salts using alkali metals [7-8] and its hydrides is a well-known route to produce nanocrystalline metal powders, no reports exist on the synthesis of Ni-Co alloy powders using aluminum (alkali earth metal) as a reducing agent. Here we report for the first time in the literature, successful synthesis of Ni-Co alloy nanocrystals by reduction with aluminum.

2 EXPERIMENTAL PROCEDURE

Commercially available AR/LR grade of NiCl2 .$6H_2O$ (98%), $CoCl_2.6H_2O$ (99%), NaOH (99%) and Al metallic powder (300 mesh, 98%) were obtained and used without further purification. The Co-Ni alloy particles were obtained by co-reducing the nickel and cobalt metal precursors in the desired molar ratios with fixed aluminum powder (2 g). The total metal ion concentration was held constant at 1 M for all preparations. In order, to obtain alloy particles of the compositions $Ni_{90}Co_{10}$ 4.266 g of $NiCl_2.6H_2O$and 0.474 g of $CoCl_2.6H_2O$ were dissolved in 10 ml of water and stirred well. Then aluminum powder was mixed well with above solution and stirred until complete the reaction. Within a few minutes vigorous exothermic reaction was observed, during which excess of water was slowly added to complete the reaction. After completion of chemical reaction, the precipitate was repeatedly washed with distilled water and separated by magnetic decantation. The precipitate was again washed with 0.5 M sodium

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Vol.11, Iss.9, Dec 2022

hydroxide solution followed by water and ethanol to remove any unreacted aluminum powder. A similar procedure was followed to synthesis Ni-Co alloys with various composition by co-reducing the nickel and cobalt salts in the desired molar ratios.

3 Elemental Analysis

Elemental analyzes of the as-prepared alloy samples were analyzed using energy dispersive X-ray analysis (EDX). It was found that precursor molar concentration was maintained in the as-prepared alloy composition. Since the lattice constant of Ni and Co having small difference, the ratio of the X-ray intensities from these elements approximates the alloy composition. Since the grain size is less than 20 nm, the X-ray intensities need not be corrected for absorption and fluorescence effects.

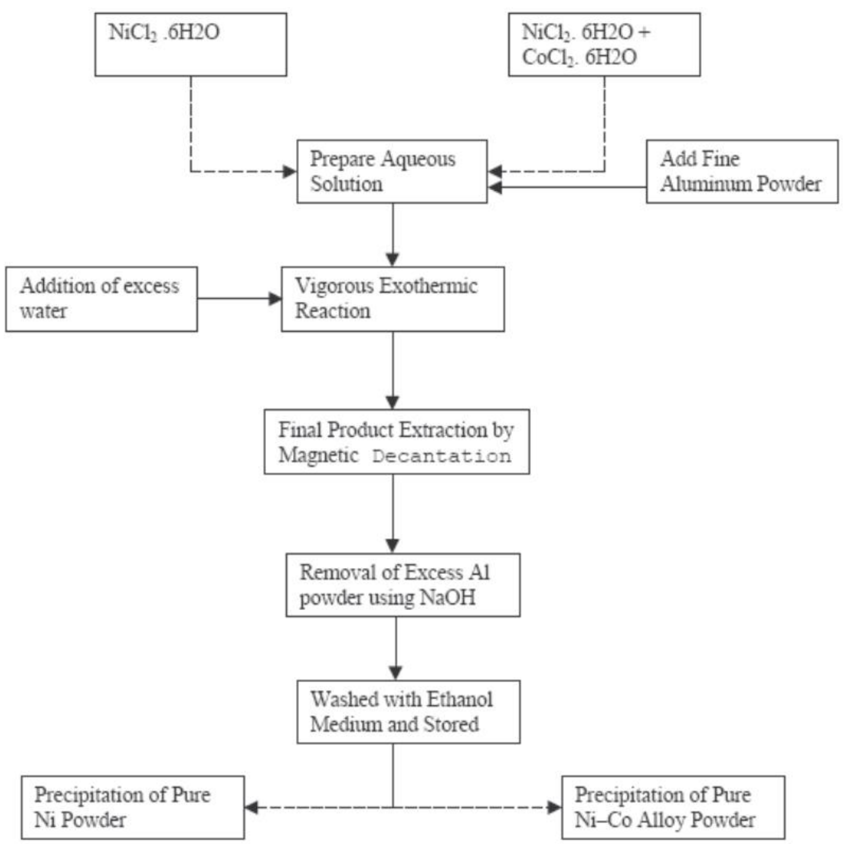

Fig. 1 schematic flow chart diagram for synthesis of Ni-Co alloy nanoparticles

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Vol.11, Iss.9, Dec 2022

4. X-ray diffraction pattern of Ni-Co powder

Co-Ni solid solutions, such as $Ni_{0.9}Co_{0.1}$, $Ni_{0.5}Co_{0.5}$ and $Ni_{0.3}Co_{0.7}$ alloys have been prepared by ion exchange chemical process. The crystal structures of as-synthesized samples were studied by X-ray diffraction pattern. Fig. 1 shows the XRD pattern of as-prepared NiCo alloy powders with various cobalt percentages. The as prepared sample has f.c.c. structure upto 70at% of cobalt. This suggests that formation of Co-Ni alloy proceeds upto 70at% cobalt. However, pure cobalt

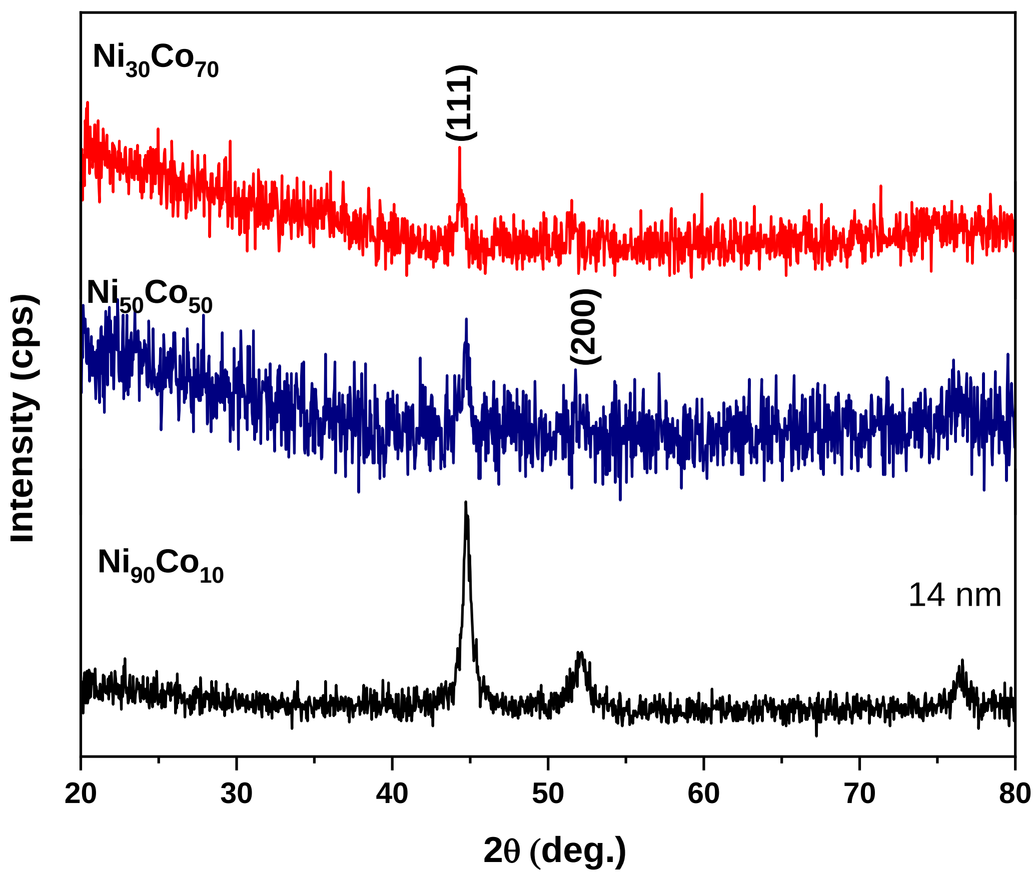

Fig .1 Shows the XRD pattern of as-prepared Ni-Co powder samples with 10, 50 and 70 at% of cobalt content

powder produced by this method shows the major h.c.p. phase (7). On the other hand, owing to the fact that the Co and Ni f.c.c. phases have very close lattice parameters, other characterization techniques besides XRD must be used to confirm the alloy formation. To study the effect of annealing on as-prepared samples, $Ni_{30}Co_{70}$ samples was annealed at 400 ˚C for an hour in the flow of nitrogen gas. The X-ray diffraction spectrum of as-prepared sample confirms the poor crystallinity with increasing cobalt content. Figure 2 shows the XRD pattern of annealed Ni-Co

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sample ($Ni_{30}Co_{70}$). We now describe the effect of annealing on the structure of Cobalt rich powder. The as-prepared nanocrystals were annealed at 400 ˚C in furnace for an hour (in nitrogen atmosphere) forming a well-defined fcc crystalline peaks. The calculated mean grain size indicated in the spectrum shows the efficient grain growth of $Ni_{30}Co_{70}$ sample.

5. **Magnetic properties**

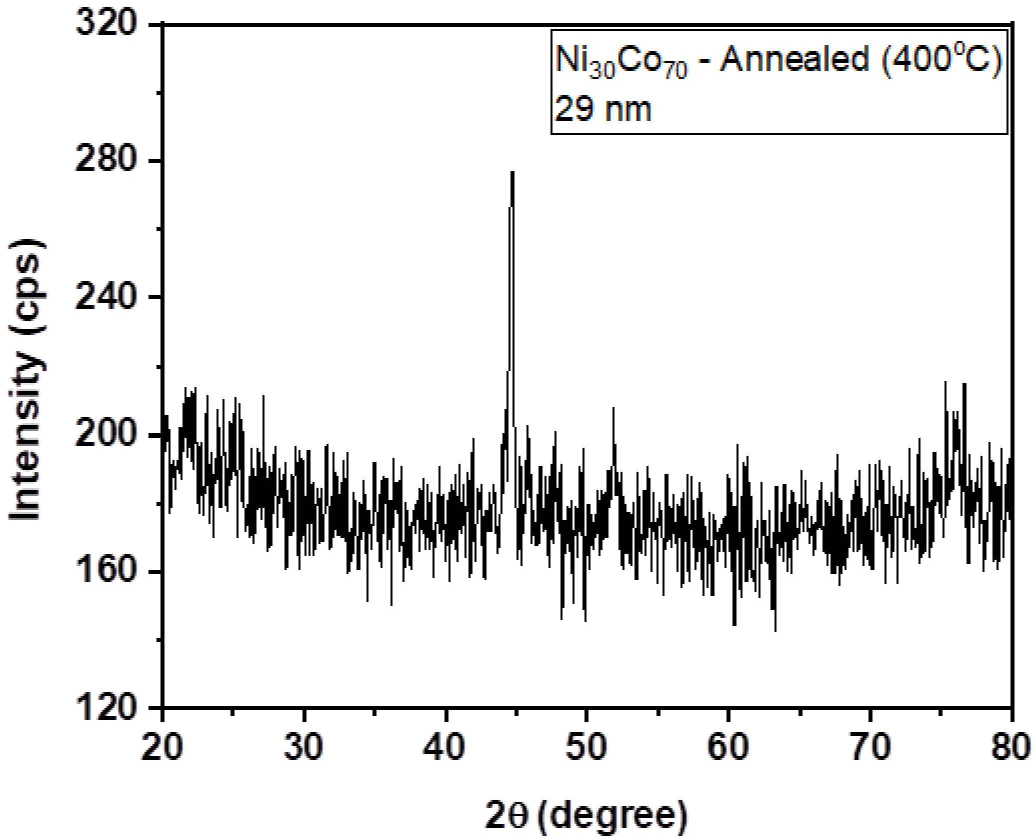

Fig. 2 Shows the XRD pattern of annealed at 400°C for an hour Ni-Co powder samples with 70 at% of cobalt content

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Vol.11, Iss.9, Dec 2022

Fig 3 shows the typical *M-H* magnetic hysteresis curves for $Ni_{1-x}Co_x$ alloy powders measured at room temperature. It can be seen that the entire sample exhibits ferromagnetic

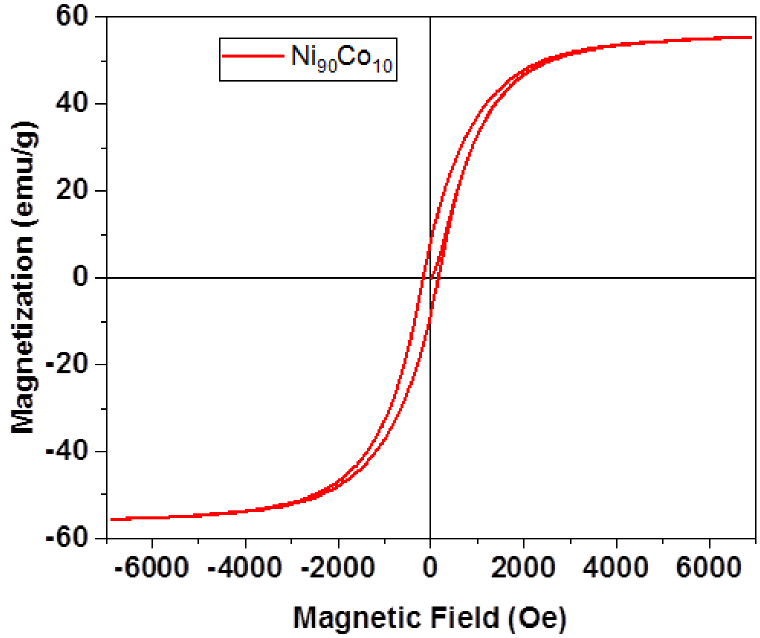

Fig.3: Typical Hysteresis loops of as-prepared $Ni_{90}Co_{10}$ alloy particles measured at room temperature

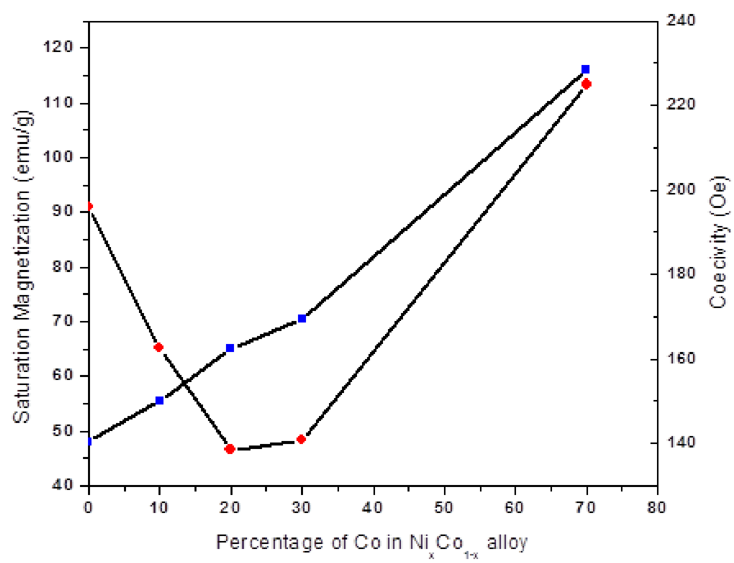

Fig. 4: Saturation Magnetization and Coercivity as a function of Co content in Nickel

behavior. It was found that increasing saturation magnetization as a function of cobalt content. It was noted that Ni rich compositions tend to magnetize at low field (2.5 kOe), while $Ni_{30}Co_{70}$

alloy display no tendency for saturation. In order to estimate the changes in the magnetic moment with composition, the magnetization at 7 kOe were examined in figure 4. It was found that the saturation magnetization increases progressively with Coat% and shows maximum magnetization (116 emu/g) value for 70at% cobalt.

Also it was found that the M-H magnetic hysteresis curves for annealed $Ni_{1-x}Co_x$ alloy powders measured at room temperature, shows ferromagnetic nature. It was noted that there is an increase in the magnetic moment for $Ni_{0.9}Co_{0.1}$, $Ni_{0.5}Co_{0.5}$, and $Ni_{0.7}C_{0.3}$ samples, however a drastic increase in the magnetic moment (116 emu/g) was observed in $Ni_{0.3}Co_{0.7}$ sample which might be due to the high content of cobalt.

Conclusions

We have achieved alkali metal (Al) reduction of Ni and $Ni_{1-x}Co_x$ 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 cobalt content on the structure and magnetic properties of Ni-Co alloys were studied. It was found that the fcc phase was stable upto 70at% of cobalt content. The saturation magnetization increases linearly with cobalt content upto 116 emu/g.

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