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Research Paper

Structural and Magnetic Properties of Nanocrystalline Ni-Fe Ferromagnetic Alloy

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Abstract

The simple and cost effective liquid-solid interaction chemical method is developed to prepare nanocrystalline Ni_xFe_{100-x} (x = 73) alloy powder. $NiCl_2.6H_2O$ and $FeCl_3$ were directly reduced using aluminum at room temperature in aqueous medium. The X-ray diffraction (XRD) measurements were carried out on Ni73Fe27 alloy powder. Ni73Fe27 alloy shows the FCC phase. The average crystalline size of Ni73Fe27 alloy powder was found that 8 nm from Sherrer's method. The magnetization of Ni73Fe27 alloy nanoparticles was 88.6 emu/g. The alloy formation and Curie temperature of Ni73Fe27 alloy particles also will be studied and reported in future communication.

1 INTRODUCTION

Since the specific alloy compositions corresponding to Ni73Fe27 and NiFe are known to be important in the structural and so magnetic properties, attempts were made to study them further by annealing the materials. The Ni73Fe27 composition (as also in many other Ni3M compositions where M is a metal such as Al, Mn etc) is particularly known to be favourable for highly ordered structure of L12 type. The NiFe composition on the other hand tends to assume a phase different from that of the Ni rich compositions.

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So, studies on annealed materials of these compositions were undertaken. Here, we demonstrated first time a different synthesis approach of chemically reducing metal salts by aluminium to obtain highly crystalline Ni and Ni-Fe alloy powders. This process having considerable advantages: (i) room temperature synthesis, (ii) inert atmosphere is not required, (iii) direct synthesis of alloy powder from metal salts (iv) a simple and cost effective method and (v) Aluminium is nontoxic metal(1-7).

The as-prepared powders were characterized for structural and magnetic properties as a function of annealing temperature.

The as-prepared samples of the above mentioned compositions were typically annealed at 300 ^{o} C and 500^{o} C for one hour under flowing nitrogen gas for these investigations.

2 Experimental Methods

Commercially available AR/LR grade of NiCl2 .6H_2O (98%), $FeCl_3$ (99%), NaOH (99%) and Al metallic powder (300 mesh, 98%) were obtained and used without further purification.

The Ni73Fe27 alloy particles were obtained by co-reducing the nickel and iron 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. Typically, to obtain alloy particles of the compositions Ni73Fe27 0.75 M of $NiCl_2.6H_2O$and 0.25 M of $FeCl_3$ 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 hydroxide solution followed by water and ethanol to remove any unreacted aluminum powder. A similar procedure were reported in previous work to synthesis Fe and Fe-Co alloys with various composition by co-reducing the iron and cobalt salts in the desired molar ratios.

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3 X-RAY DIFFRACTION ANALYSIS

The sample for XRD measurement in this case was prepared by using pure silicon as an internal standard to enable accurate theta determination. Fig. 1. Shows the XRD patterns obtained for the annealed Ni73Fe27 samples along with that of the as-synthesized powder. The pattern for the as-prepared sample shows a peak corresponding to the Miller (111) plane of the fcc (or sc) phase of Ni73Fe27 matching very well with the JCPDS PDF # 881715 and with reported literature [1]. No impurity peaks are observed indicating the reasonably good purity of the final products. The patterns for the annealed samples show improved crystallinity and grain growth in these samples, as is usually the case with annealed samples. Favourably, this has facilitated detection of other peaks too. Thus, three characteristic peaks for Ni73Fe27 corresponding to (111), (200) and (220) planes are also observed. The absence of peaks of any pure Fe phases indicates that no Fe segregation has happened of that scale during the synthesis and the alloys have formed reasonably well.

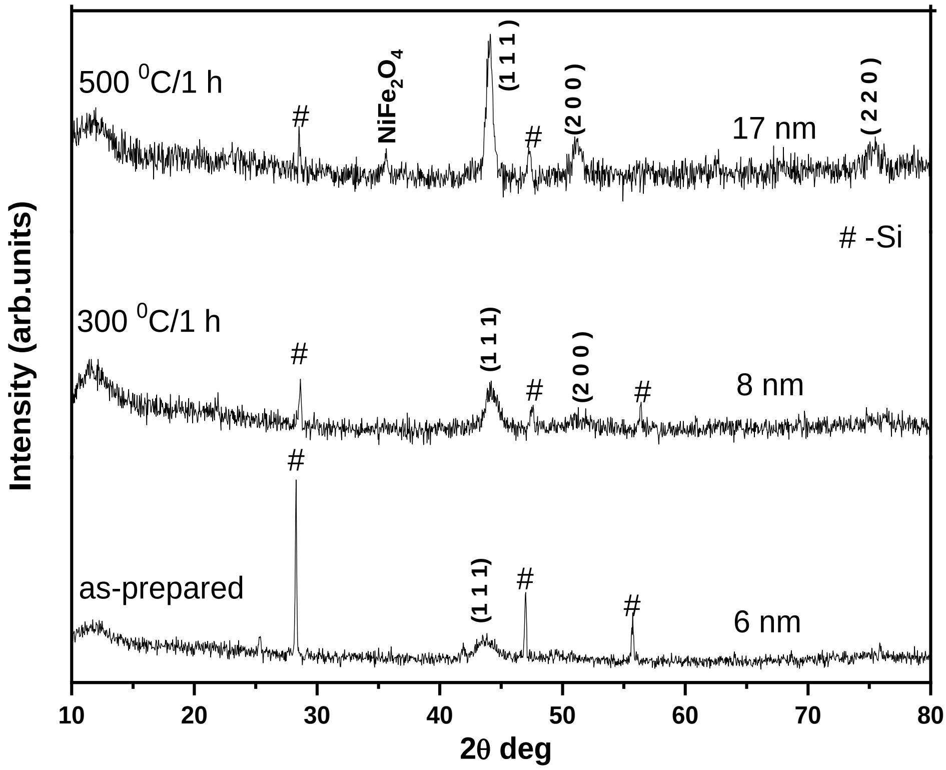

Fig.1. X-ray diffraction patterns of as-prepared and annealed Ni73Fe27 nanocrystalline powder. Si was used as an internal

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No oxide peaks are noticed in the XRD pattern for 300 °C annealed sample. . This revealed that the resultant particles are pure Ni73Fe27 phase of cubic structure. However, the 500 °C annealed sample shows some oxidation leading to the formation of the fcc $NiFe_2O_4$ phase, whose (311) peak (100%) is detected in the pattern. Though the annealing was done in nitrogen such oxide formation is still noticed in some cases probably from diffused surface oxygen at high temperatures.

. The average crystallite sizes estimated from the broadening of (111) line using Scherrer's formula are marked in Fig.1. A large grain growth of more than 100% is noticed particularly on annealing at 500° C.

4. ANALYSIS OF MAGNETIC PROPERTIES

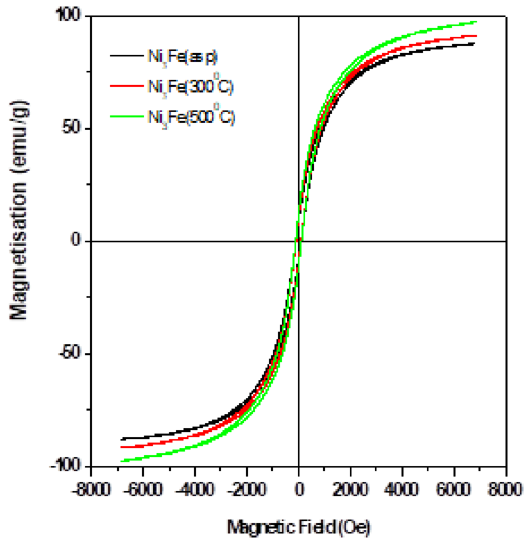

Fig. 2. Hysteresis loops obtained for the as-prepared and annealed Ni73Fe27

Fig. 2 shows the typical soft-magnetic hysteresis loops for the annealed Ni73Fe27 powders along with the hysteresis for the as-prepared sample for comparison. The saturation magnetization (M_s) and the coercivity (H_c) for the as-prepared sample at 88.26 emu/g and 95.3 Oe respecively, are typical of a soft ferromagnetic alloy. Table 1 lists these values for all samples illustrating the effect of heat treatment on the magnetic parameters. The M_s value obtained for the as-prepared sample is much less than that of the reported value of 110 emu/g and H_c is little less than 100 Oe reported for the

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Ni73Fe27 alloy [2]. The decrease in M_s might be an effect of reduced size and /or increased specific grain boundary area. On annealing the grain growth contributes to the increase in M_s for the same reason. However, due to the partial oxidation at 500° C the magnetization is still less than the reported bulk value. In the case of H_c the inhomegeneity in grain size associated with particle coarsening due to annealing could have contributed to an increased value according to the random anisotropy model [3].

Table 1. Magnetization data for Ni73Fe27 samples measured at room temperature

S.No	Sample	M_s(emu/g)	H_c(Oe)	M_r(emu/g)
1	As-prepared	88.26	95	8.4
2	300 °C	91.92	92	9.5
3	500 °C	97.95	109	11.4

Conclusions

An ultrafine magnetic particle of Ni73Fe27 was successfully synthesized by a novel chemical reduction of the precursor salts by an alkali earth metal (Al) in aqueous medium. It was found that as-prepared particles were in crystalline nature with size of nanometer range. Further annealing of alloy particles leads to better grain growth.

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