Structural and Magnetic Characterization of Nanocrystalline Ni_3Mn Alloy Prepared by Chemical Technique

E. Viswanathan and E. Thirumal

Research Journal of Agricultural Sciences
An International Journal

P- ISSN: 0976-1675
E- ISSN: 2249-4538

Volume: 13
Issue: 05

Res. Jr. of Agril. Sci. (2022) 13: 1433–1434

CARAS

Res. Jr. of Agril. Sci. (Sep-Oct 2022)
13(5): 1433–1434

ISSN: 0976-1675 (P)
ISSN: 2249-4538 (E)

www.rjas.org

Short Communication

Structural and Magnetic Characterization of Nanocrystalline Ni_3Mn Alloy Prepared by Chemical Technique

E. Viswanathan*[1] and E. Thirumal[2]

Received: 08 Jul 2022 | Revised accepted: 28 Aug 2022 | Published online: 19 Sep 2022

***Key words*:** Uttarmand reservoir, Zooplankton, Food web, Water quality

Ni_3Mn alloy is ferromagnetic and its magnetic properties were strongly influenced by degree of crystallinity [1]. Chemical synthesis of Ni-Mn alloy is great challenge because of large difference in reduction potential. Also, the reports on chemical synthesise of Mn based alloys were very limited in literature. In this paper, we present room temperature borohydride reduction technique to prepare Ni-Mn alloy the results from various characterization techniques, employed to study the structural, magnetic, elemental analysis and surface properties of the samples, prepared by sodium borohydride reduction technique are discussed.

Highly concentrated 10 ml aqueous solutions (2 M) of $NiCl_26H_2O$ and $MnCl_2.H_2O$ with predetermined molar ratio were prepared. 10 ml of 2 M solution of $NaBH_4$ was slowly added with above solution during mechanical stirring. Exothermic reaction took place and black precipitation was observed, this indicates the reduction of the salts. Precipitated particles are washed repeatedly using distilled water. Particles are stored in ethanol medium. The material was later dried in air before taking up further studies. Flow chart was created for the steps involved in the synthesis process (not shown). As-prepared samples were annealed at 673 K for 3 hours. Similar procedure was followed to synthesis pure Ni powder from $NiCl_2.H_2O$. The as-prepared samples and annealed samples were characterized using X-ray diffractometer (XRD), vibrating sample magnetometer (VSM-EG&G Princeton Applied Research-4500) and scanning electron microscopy (JEOL JSM-840A EDX attached with, SEM).

The sodium borohydride reduction technique has been widely used to synthesise metal and metal alloy nanoparticles. This method was recently applied to synthesis Ni naoparticles by Roy *et al.* [1], the reaction mechanism could be as follows:

$$NiCl_2 + NaBH_4 \rightarrow Ni(BH_4)_2 + NaCl;$$
$$Ni(BH_4)_2 + H_2O \rightarrow Ni + 2B(OH)_3 + 7H_2;$$

The excess $B(OH)_3$ remains dissolved in the aqueous solution and does not impart to the final product after the washing process in fresh water.

Two different molar ratios of 1:1 and 3:1 for the Ni and Mn sources were used in the synthesize experiments. The final products obtained from these batches were subjected to EDX analysis to find out the atomic content present in them. From the EDX spectrum, it was found that the observed Mn content is about half of that in the precursor composition. Thus, in the case of 3:1 molar ratio sample, only 12 at.% of Mn was present and 24 at.% Mn in the case of 1:1 sample. The reduced at.% of Mn in all the samples may be attributed to partial reduction Mn^{2+} to Mn because the reduction potential of Ni is -0.257V and for Mn is -1.18V. The samples obtained from 3:1 and 1:1 source ratios were accordingly labeled as $Ni_{88}Mn_{12}$ and $Ni_{76}Mn_{24}$.

(Fig 1) shows the XRD pattern of as-prepared Ni and $Ni_{76}Mn_{24}$ which exhibits a broad peak centered at 44.25° while the (111) peak for fcc-Ni occurs at 44.49[JCPDS#.652865]. This small difference is suggestive of the alloy formation with Mn. The noisy pattern with a single broad hump suggests amorphous character for the material in the as-prepared state. The as-prepared samples were annealed in nitrogen atmosphere at 673 K for 3 hours. (Fig 1) also shows the XRD pattern of annealed $Ni_{76}Mn_{24}$ sample. The annealed $Ni_{76}Mn_{24}$ exhibits improved crystalline structure with FCC phase, the peak position and d-spacing of $Ni_{76}Mn_{24}$ are as follows:

As the annealing temperature is increased, crystallization is induced, hence grain growth occurs. This is evident from the increase in the intensity and decrease in FWHM of the peaks in XRD pattern of the annealed sample. The average particle size of pure as prepared $Ni_{76}Mn_{24}$ and annealed $Ni_{76}Mn_{24}$ were obtained using Scherer's relation is 4 nm and 22 nm. The weak peak at 43.2° belongs to NiO due to partial oxidation of Ni during annealing. The lattice parameter values 'a' of the samples were found to be 3.53 Å for pure Ni, 3.54Å for as-prepared $Ni_{76}Mn_{24}$ and 3.53Å for annealed $Ni_{76}Mn_{24}$.

From the XRD pattern of $Ni_{88}Mn_{12}$ alloy annealed at 673 K for 3 hours also exhibits both fcc NiO and fcc Ni peaks. The NiO peaks are strong enough to suggest that considerable oxidation of Ni has taken place in this sample. It is interesting to note that even though both the samples were subjected to

* **E. Viswanathan**
✉ vijiyuvavelan@gmail.com

[1-2] Faculty of Arts and Science, Department of Physics, Bharath Institute of Higher Education and Research, Chennai - 600 073, Tamil Nadu, India

similar annealing procedure, only a minor oxidation is noticed for $Ni_{76}Mn_{24}$ sample. This could be because of the better ordering in this sample. The peak data obtained from the pattern XRD were tabulated. The lattice constant and grain size values are 3.53 Å and 37 nm [2].

The room temperature hysteresis behaviour of annealed $Ni_{76}Mn_{24}$ alloy was obtained using Vibrating Sample Magnetometer (VSM) with a maximum applied field of 7 kOe. The instrument was calibrated using pure bulk nickel for which a magnetic moment of 55 emu/g was obtained. (Fig 2) represent the M-H curve for the annealed $Ni_{76}Mn_{24}$ sample. A clear soft ferromagnetic behavior is observed and the saturation magnetization, remanence magnetization and coercivity values are 18 emu/g, 5.6 emu/g and 101 Oe respectively. Okazaki *et al* [3] have reported increasing saturation magnetization value as a function of annealing time at 673K for Ni_3Mn alloy. They have observed a room temperature Ms value of ~80 emu/g for 1000 h annealed and ~8 emu/g for 5 h in the two ends of their spectrum of samples for an annealing temperature of 673 K. They attribute the trend to the fact that the net magnetic moment of the Ni_3Mn alloy is strongly dependent on the crystal structure and ordering ($L1_0$) in the sample. In our case the value of 18 emu/g for the room temperature M_s for the $Ni_{76}Mn_{24}$ alloy indicates that our sample is more ordered than that reported by them even though our annealing duration was only 3 h. In order to understand the thermomagnetic behavior and identify the Curie transition temperature, thermomagnetic studies on annealed $Ni_{76}Mn_{24}$ alloy were carried out in VSM, with a constant applied field of 50 Oe. The higher Curie temperature for our samples may be because of the higher degree ordering in our samples. For example, Okazaki et al report a Curie temperature of ~ 630 K for a sample with an order parameter of 0.5 but obtained at annealing time of 116 h. It should be noted that their samples were prepared by induction melting [4-5].

Scanning electron microscope plays a vital role to study the surface property of the samples. The amorphous NiMn has the sponge like surface and the particles were aggregated strongly, which is shown in the magnified picture.

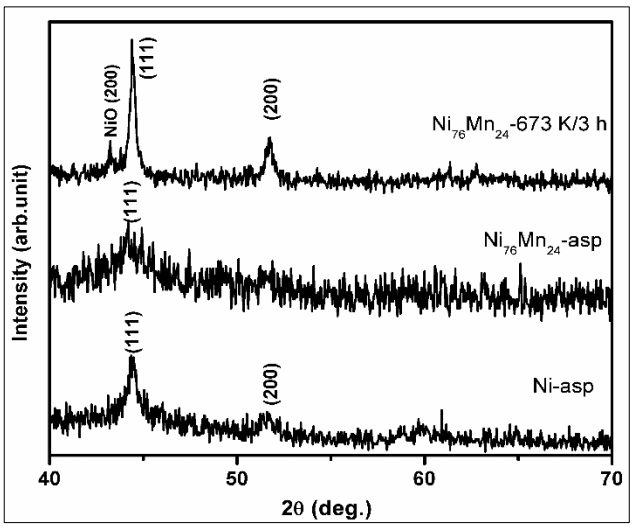

Fig 1 XRD pattern of as-prepared Ni, $Ni_{76}Mn_{24}$ and alloy annealed at 673 K for 3 h

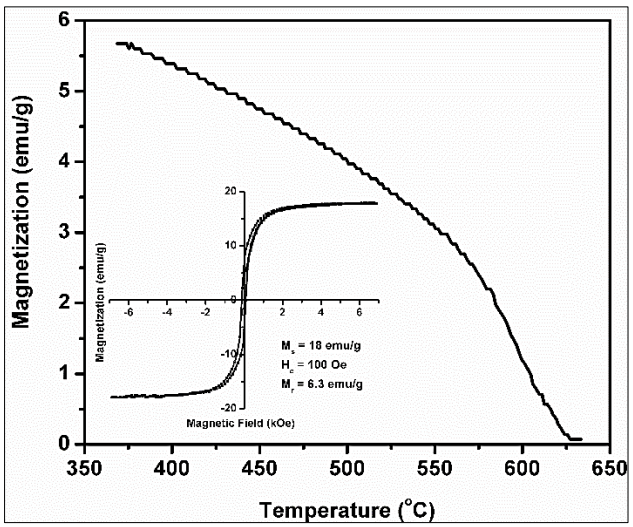

Fig 2 M-H curve for the annealed $Ni_{76}Mn_{24}$ sample

SUMMARY

Nanocrystalline Ni_xMn_{100-x} alloy samples were synthesized by borohydride reduction technique. Structural studies reveal that as-prepared Ni and Ni-Mn particles are poorly crystalline, annealing leads to fcc formation with highly crystalline state. It was found that increase in Manganese content improve the thermal stability of alloy powders. Magnetic properties of $Ni_{76}Mn_{24}$ alloy particles were studied for as-prepared and annealed samples. The saturation magnetization and Curie transition of annealed $N_{76}Mn_{24}$ alloy are 20emu/g and 627 K respectively.

LITERATURE CITED

1. Roy A, Srinivas V, Ram S, De Toro JA, Reverio JM. 2004. Effect of interstitial oxygen on the crystal structure and magnetic properties of Ni nanoparticles. *Jr. Applied Physics* 96(11): 6782-6788.
2. Yu A, Babanov V P, Pilyugian T, Miyanga AM, Patselov EG, Chenyshev AV, Ryazhkin, Ogasavara T. 2007. Spectroscopy of homogeneous deuterated hydrocarbon films redeposited from a T-10 tokamak plasma discharge. *Journal of Surface Investigation, X- ray, Synchrotron and Neutron Techniques* 1(3): 13-17.
3. Okazaki T, Miyanaga T, Sakissaka Y, Sugimoto S, Yamada K, Honda. 2004. GMR and soft magnetic properties of Ni-Mn alloys with dispersed ferromagnetic nano particles. *Rev. Adv. Mater. Sci*. 6: 150-161.
4. Kemeni T, Kaptas D, Kiss LF, Balogh J, Vincze I, Szabo S, Beke DL. 2000. Structure and magnetic properties of nanocrystalline soft ferromagnets. *Hyperf. Interact*. 130: 181-219.
5. Han J, Kwon S, Sohn S, Schroers J, Choi-Yim H. 2020. Optimum soft magnetic properties of the FeSiBNbCu alloy achieved by heat Treatment and tailoring B/Si ratio. *Metals* 10: 1297.