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Structural and Magnetic Studies of Nickel- Boron alloy

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Abstract- **Ni-based amorphous alloys exhibit excellent catalytic activity. Ni-B and Ni-P are the well-known amorphous alloys. In this work, Ni–B alloy has been synthesized by the chemical reduction method. The aim of the work is to elucidate the Structure, magnetic behaviour and morphology of Ni–B alloy. Nickel and boron were reduced from nickel sulfate and boric acid by using sodium borohydride. Polyvinyl pyrrolidone (PVP) is used as capping agent. Structure of alloys was discussed based on results of X-ray diffraction measurements. The ferromagnetic behavior of the material is observed from Vibrating Sample Magnetometer (VSM). FESEM analysis was done to study the morphology of the sample.**

Keywords- chemical reduction method; amorphous alloy; ferromagnetic material

I. INTRODUCTION

Recently, the transition metal, metallic alloy has gained much attention as promising novel catalytic materials[1]. It also has better selectivity and stronger sulfur resistance in many hydrogenation reactions. Because of the increasing technological interest in these materials, new preparation methods have been developed and then improved or modified. Recent attention has focused on the preparation of amorphous alloy powders, which are preferable to ribbons and films, for forming bulk amorphous sample. The rapid quenching method and chemical reduction by borohydride or hypophosphite are two of the most widely used techniques in producing amorphous alloys [2, 3].

Since Ni is a catalyst in a lot of chemical processes, numerous groups have been devoted to preparing Ni-based ultrafine amorphous alloys. Among these alloys, the Ni–P and Ni–B amorphous alloy catalysts are most thoroughly studied. Both the Ni–P and Ni–B amorphous catalysts have been obtained by chemical reduction. It is accepted that the promoting effect of alloying B or P is attributed to the modification of the catalyst structure, resulting in the short-range ordering and long-range ordering structural characteristics, the enrichment of the active Ni sites on the catalyst surface, the homogeneous dispersion of the active sites[4]. In 1953 Shlesinger et al. reported that the reduction of a nickel salt with sodium borohydride in aqueous solution yields a finely divided black material as a precipitate that contains both nickel and boron. Oka- moto et al. prepared Ni–B catalysts with a surface area of 38.2 m2/g using nickel acetate and sodium borohydride in 95% ethanol. Under similar conditions, Schreifels et al. reported a Ni_2B amorphous alloy with a surface area of 58.0 m2/g. In this present work, Ni-B alloy has been prepared using conventional chemical reduction method to study their structural information, morphological nature and magnetic behavior as the atomic percentage of boron varies[5-7].

II. MATERIALS AND METHODS

A. Materials

Nickel chloride from Alfa Aesar, Boric acid from Sd fine and Sodium borohydrate from Alfa Aesar. All experiments were carried out using double distilled water.

B. Method

In the preparation of Ni-B alloy, Nickel chloride is dissolved in double distilled water and Boric acid was added to the solution. Then Polyvinyl pyrrolidone (PVP) (0.5 g) was dissolved in 25 ml of water, This PVP solution was added to the metal solution for particle size stabilization. This solution was kept under stirring for half an hour for trough mixing of metal ions. Finally, the reducing again $NaBH_4$ (0.5 M) is added to the solution drop wise with the help of burette, when the pH of the solution reaches 14 the Ni-B alloy starts to form precipitates. Then the Ni-B alloy powder was washed with distilled water using centrifuge and dried in room temperature.

The Ni-B alloy was prepared for three different atomic percent of Boron (i) 75:25 (NB1), (ii) 50:50 (NB2), (iii) 25:75 (NB3). A portion of the as prepared Ni-B alloy was annealed to 550 °C for one hour under the vacuum of 10^{-4} pa. The annealed samples are named as ANB1, ANB2 and ANB3 according to their previous naming.

Both the as prepared and annealed Ni-B alloy samples are subjected to various characterizations. The X-Ray diffraction (XRD) analysis of the prepared sample was done using a GE X-ray diffraction system-XRD 3003 TT with CuKα1 radiation of wavelength 1.5406 Å under standard θ-θ geometry. High Resolution Scanning Electron Microscopy (HRSEM) was carried out using FEI Quanta FEG 200 instrument. The Vibrating Sample Magnetometer (VSM) measurement was used with the help of the Lakeshore VSM 7410 instrument.

III. RESULTS AND DISCUSSION

A. Structural Investigation

The structural information of the Ni-B alloys was calculated from XRD. The XRD pattern of the as prepared Ni-B alloy is shown in figure 1. This clearly shows, there is no observation of any definite diffraction peak in the scan angle which confirms the formation of amorphous nature of the as prepared alloy. The XRD pattern of annealed samples is shown in figure 2. The diffraction pattern for ANB1 and ANB2 sample shows the diffraction peak at Bragg's angle 44°, 52° and 76° corresponds to nickel phase, this has been confirmed with reference to standard JCPDS file no. 65-2865. This gives the both ANB1 and ANB2 in FCC structure with space group Fm-3m.

As annealed pattern clearly shows, as the atomic percentage of boron increases, there is an increase in amorphous nature in the annealed samples. The sample ANB3, which has 75 percent of boron has higher amorphous nature as compared to other two samples. But it also have minimal intense Bragg's diffraction peak at 23°, 32° and 45° confirms the presence of NiB phase, this has been confirmed with standard JCPDS reference number 06-0567 with orthorhombic crystal structure.

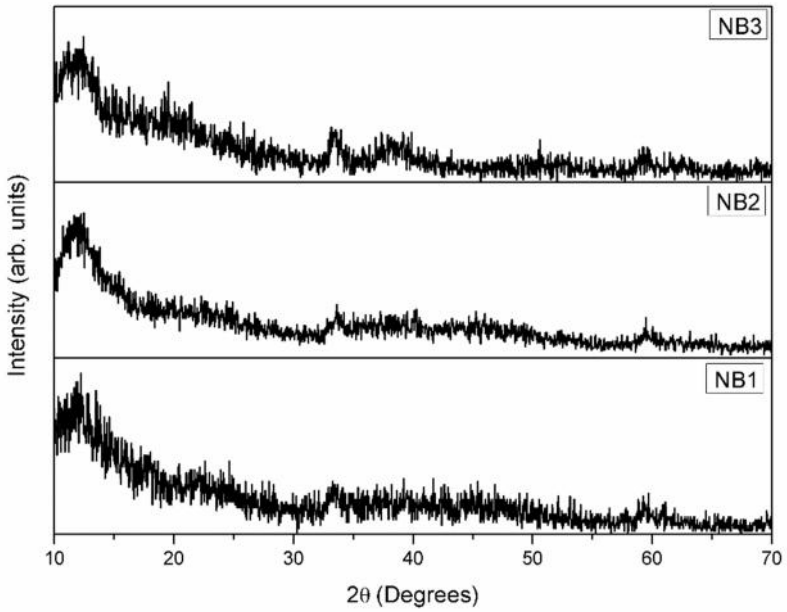

Figure 1: XRD pattern of as prepared Ni-B alloy

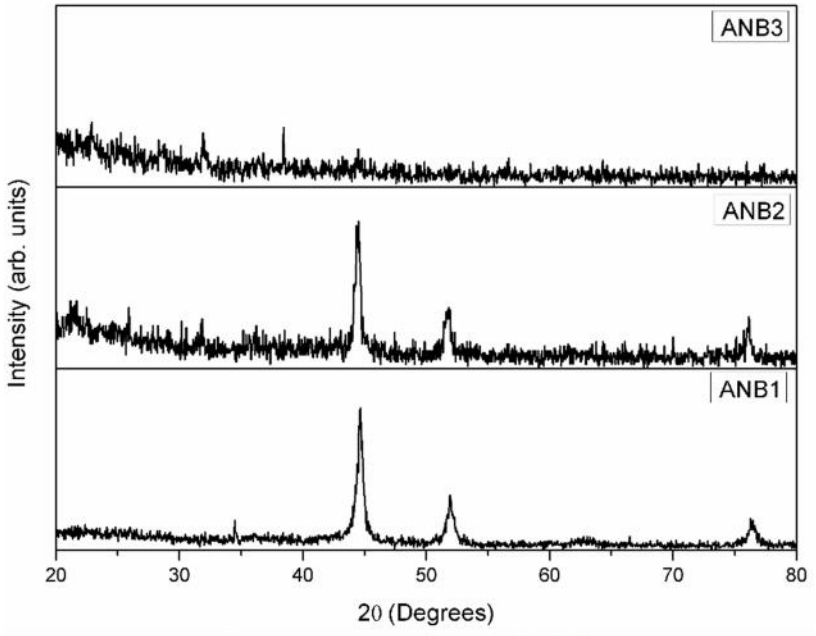

Figure 2: XRD pattern of annealed Ni-B alloy

B. Morphological study

The morphology of the annealed Ni-B alloy samples is observed using HRSEM analysis. The HRSEM image of annealed Ni-B alloy samples is shown in figure 3. The HRSEM image of Ni-B alloy shows there is no definite shape of the particles for all the three Ni-B alloys. There is an agglomeration of flat sheet throughout the sample, these flat sheet forms folded flakes like nature. The amorphous nature of boron causes these indefinite morphology natures of the Ni-B alloy.

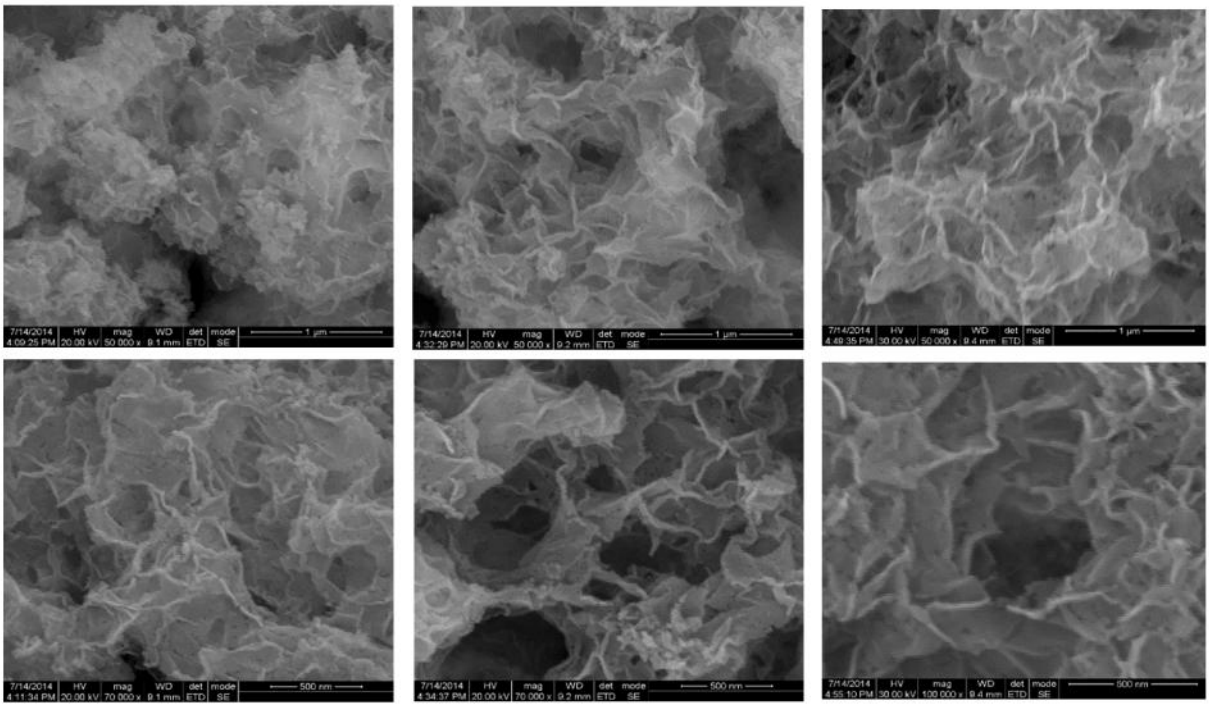

Figure 3: HRSEM images of annealed Ni-B alloy

C. Magnetic Measurement

Magnetic behaviour of the Ni-B alloy was studied using VSM. The magnetization curve of the as prepared Ni-B alloy is shown in figure 4. The magnetization curve of as prepared Ni-B alloys shows weak ferro magnetic naure for NB1 and NB2 sample and para magnetic nature for NB3 sample. This behaviour is from the role of boron in the particular composition. In the case of NB1 and NB2, the atomic percent of boron is low and equal to the nickel percent so there is presence of weak ferromagnetic along with the paramagnetic nature. This is due to, nickel contribute to ferro magnetic and boron contribute to diamagnetic nature which combines to form weak ferro magnetic nature.

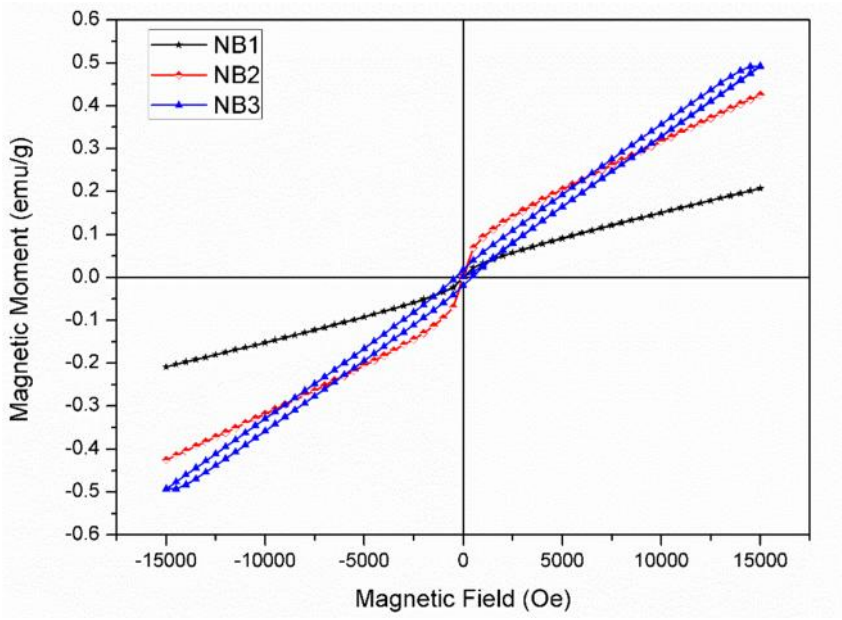

Figure 4: Magnetization curve of as prepared Ni-B alloy

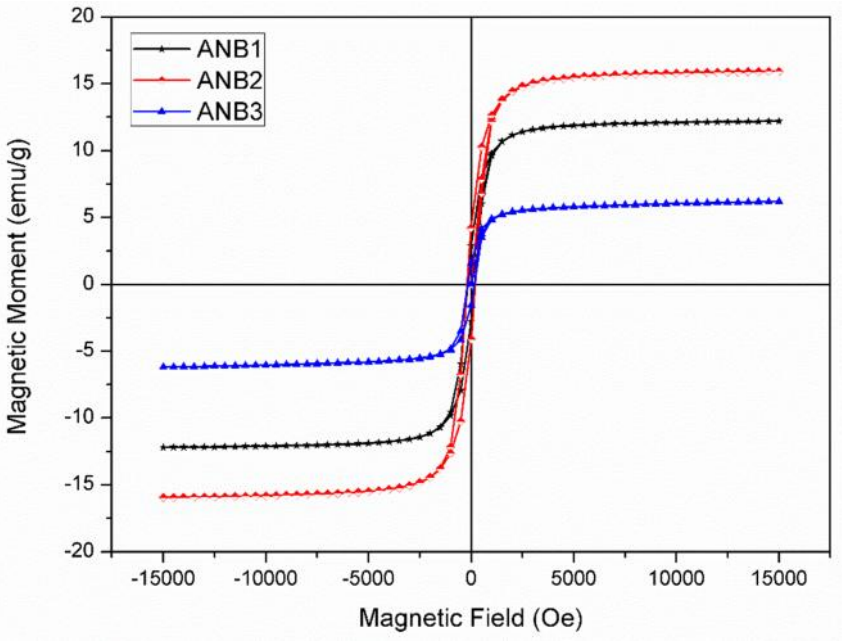

Figure 5: Magnetization curve of annealed Ni-B alloy

The magnetization curve of annealed Ni-B alloy samples is shown in figure 5. The all the three annealed Ni-B alloy samples show soft ferromagnetic nature. The saturation magnetization values of ANB1, ANB2 and ANB3 samples are 12.18 emu/g, 15.9 emu/g and 6.15 emu/g respectively. The coercivity of the all the three samples is same and the value is 180 Oe. The retentivity values of ANB1, ANB2 and ANB3 samples are 2.8 emu/g, 4.2 emu/g and
1.6 emu/g respectively. It is clear that for the ANB2 sample the saturation magnetization value is high when comparing other two samples. The coercivity of the all the samples is same while the retentivity and saturation magnetization varies this explains the role of boron on the Ni-B alloy. As the atomic percentage of boron varies the saturation magnetization and retentivity altered at the maximum of 15.9 emu/g is reached for 50 atomic percentages.

The maximum saturation magnetization (Ms) value is 15.9 emu/g calculated from the loop, which is less compared to the value 55 emu/g of bulk Ni. It is mainly due to the new structure evolved in alloying of the two metals. If there had been no atomic level mixing and the entire amount of Ni in the deposits was present as separate islands, then the magnetization values would have been much higher.

IV. CONCLUSION

The Ni-B alloy has been successfully synthesized using sodium borohydrate assisted chemical reduction method. The strutual information shows the amorphous nature of as prepared samples while the annealed samples show the crystalline nature with the formation of NiB phase. The indefinite particle shape of the Ni-B alloy was observed from the FESEM analysis. The variation of saturation mgnetizationa and retentivity of the annealed sample depends on the atomic percentage of boron and at the maximum of 15.9 emu/g was observed for ANB2 sample. In the case of as prepared sample the magnetic ordering also determined by the atomic percentage of boron, which shows soft ferro for lower atomic percentage and paramagnetic ordering for the higher atomic percentage of boron.

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