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Comparision of microstrucural and morphological studies of 40%Ni-Fe and 75%Ni-Fe nanopowder prepared by mechanical alloying

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ABSTRACT

Mechanical alloying through high energy ball milling was used to produce Ni-Fe alloy powders starting from elemental Ni and Fe powders of average particle size 80 and 25µm respectively. High Energy Planetary ball milling at room temperature wasperformed for various time durations ranging between 2 to 100 h. Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Particle Size Analyzer were used for characterization. In 40%Ni-Fe the grain size first decreases with milling till 8 h and then increases reaching the next highest value at 16 h. After 16h the grain size continuously decreases reaching the lowest value of 7.3 nm at the end of 100h of milling. In 75%Ni-Fe alloy the grain size decreases continuously with milling h reaching the value of 34 nm at 100 h of milling. It is observed from 40%Ni-Fe that the particle size decreases continuously with milling time as the fracturing forces dominate over the cold welding entire period. At the end of milling the particle size does not change appreciably due to the equilibrium between the fracturing and cold welding forces. The particle size reaches the lowest value of 0.967 µm at the end of 100 h of milling. In 75%Ni-Fe alloy during 2 to 8 h of milling the particle size remain constant at 13.5 µm. During 32 to 64 h of milling, it is observed that the particle size remains constant at 7.5 µm. This may be due to equilibrium between the fracturing and cold welding forces in these regions of milling. The particle size decreases after 64 h and reaches the lowest value of 3 µm at 100 h of milling.

Key words: Mechanical alloying, particle size, morphological study, microstructure.

INTRODUCTION

Ni-Fe alloys of varying compositions are extensively used in electromagnets due to their low hysterics loss, high permeability and high saturation flux density Ashokkumar (2008). Their mechanical properties can be improved using several techniques such as recrystallization Goren-Muginstein and Rosen (1997) solid state sintering Ryu (2000), cyclic heat treatment Noh (1993 and 1994) and formation of matrix pools Hong(2002), surface carburization Jung(1999) , formation of irregular particle shape Song (1997), and mechanical alloying Ryu (2003). During the last few years major advances have been made in the study of behavior of small particles due to their enhanced properties. The mechanical alloying process developed by Benjamin (1970), is an advanced fabrication process which can produce very fine and homogenous powder. Mechanical milling has been suggested as a process to develop alloys with improved mechanical properties and Magnetic properties. In the present work morphological study and microstructure analysis of Ni-Fe powders produced using mechanical alloying was carried out.

EXPERIMENTAL

Study

Elemental powders of Nickel and Iron with the purity of 99.8 and 99.5, particle size 80 and 25µm and grain size 280 and 160nm respectively were used as starting materials. Powder mixture of 40% Ni-Fe and 75% Ni-Fe were selected as the starting materials for the study. High Energy Planetary ball milling was performed with a laboratory planetary mill (RETSCH) with ball to powder mass ratio of 10:1 at a speed of 300rpm using stainless steel jars and balls. The jars were sealed under atmospheric pressure and temperature. Toluene was used as a process control agent. Small amounts of the powder were taken out from the mill at 2, 8,16,32,64 and 100hrs for morphological study. Phases and micro structure were analyzed by X-ray diffraction (XRD) using SIETRONICS XRD SCAN Diffraction meter. Powder morphological images were taken under varying milling hours by Scanning Electron Microscopy. The average grain size was calculated from XRD images using Scherrer's equation Carmen(1992). A Mastersizer Particle Size Analyzer was used to measure the particle size.

RESULTS AND DISCUSSION

XRD Pattern

The XRD spectra of pure Fe and Ni are shown in Figure 1 those of 40% Ni-Fe powder and 75% Ni-Fe powder at various milling hours are shown in Figure 2 and Figure 3 respectively. Comparing Figures 1, 2 and 3 it can be seen that the X-ray diffraction peaks widen as the milling time increased. This could be explained as due to the refinement of grain size and increases of micro strain during mechanical alloying. It is observed that the value of maximum counts of the diffraction peaks keep on decreasing with increasing milling hours due to the fracturing of the particles. It can be seen that at 8 h of milling of 40% Ni-Fe alloy exhibits XRD pattern reflecting amorphous structure. This shows that the amorphization is fully achieved by ball milling alone after 8h of milling with out performing additional heat treatments such as annealing for milled power products.

The average grain sizes were calculated from the super imposed XRD (111) peak of Ni and (110) peak of Fe using Scherrer's equation Islam (2006).The variation of the grain size with milling hours is listed in Table 1 and graphically shown in Figure 4. It can be seen in 40%Ni-Fe alloy that the grain size first decreases with milling till 8 h and then increases reaching the next highest value at 16 h. After 16 h the grain size continuously decreases reaching the lowest value of 7.3 nm at 100 h. In case of 75% Ni-Fe alloy the grain size decreases rapidly at the beginning of the milling and after 64 h of milling the change in grain size is not appreciable i.e. 35nm to 34nm only.

Morphological study

Figs. 5 and 6 show SEM images of pure Iron and Nickel respectively. Iron exhibits sub angular particles of various sizes (Figure 5) while Nickel shows large lumps of spherical particles of uniform size (Fig. 6). The average particle size of Fe is 24 µm while that of Ni is 79 µm.

The morphology of 40% Ni-Fe powders at different milling times was investigated using SEM analysis. Figs. 7 to 10 show the morphological development of the particles from 2 to 32 h of milling. It is observed from Figure 7 that after two hours of milling the compressive forces due to the collision of the milling balls flatten the particles as the material is usually soft at this stage. Figure 8 shows layered

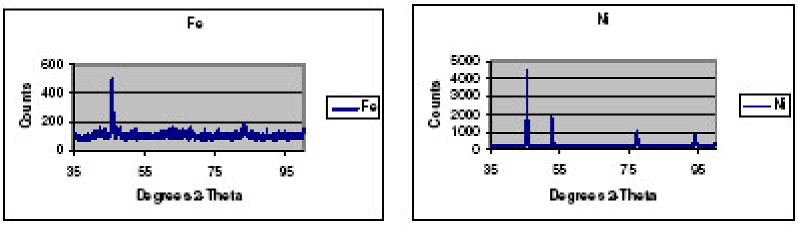

Fig. 1: XRD diffraction pattern of pure Ni and Fe powder

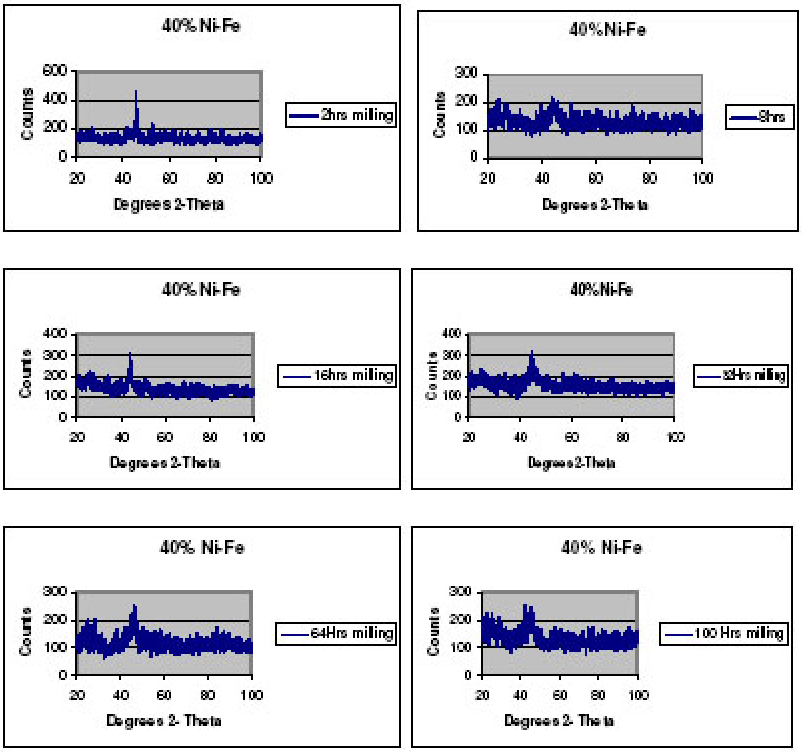

Fig. 2: XRD diffraction patterns of 40%Ni-Fe powder at various milling hours

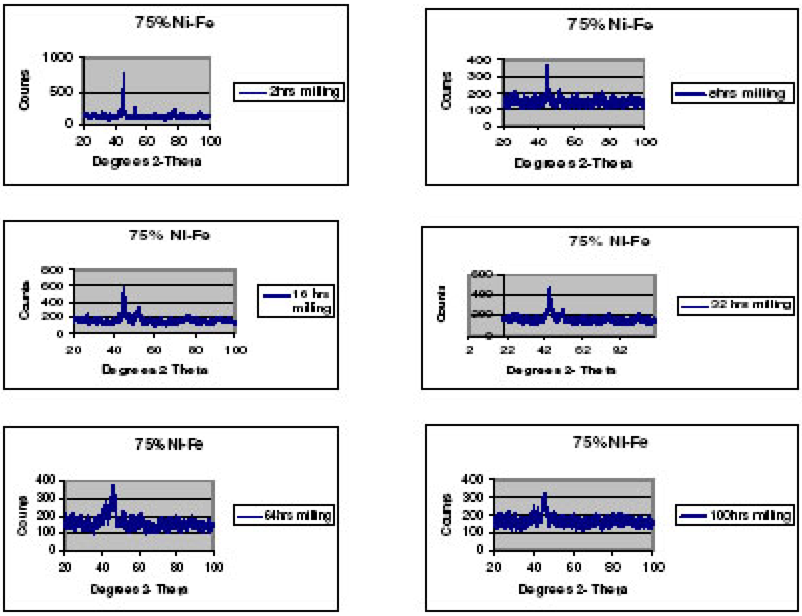

Fig. 3: XRD diffraction pattern of 75%Ni-Fe Powder at various milling hours 3.2 Grain size

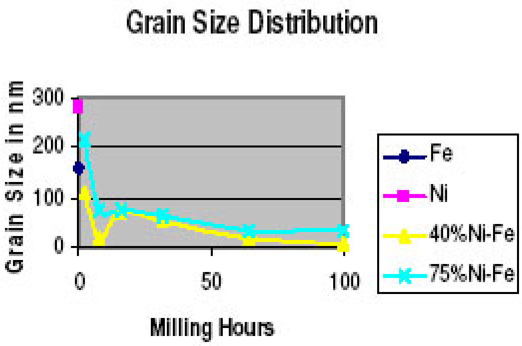

Fig. 4: Variation of the average grain size with milling time

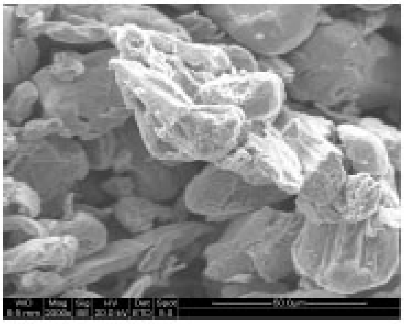

Fig. 5: SEM image of pure Iron

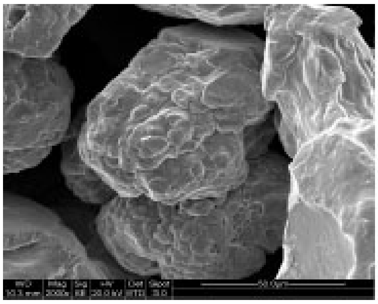

Fig. 6: SEM image of pure Nickel

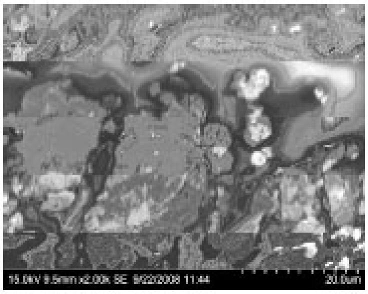

Fig. 7: 40%Ni-Fe (2hrs milling)

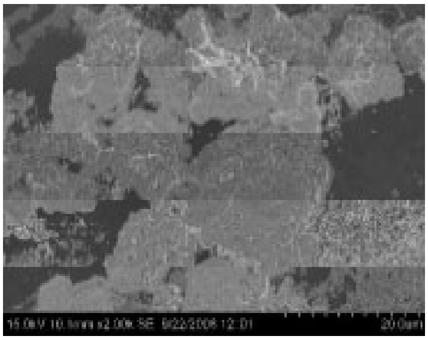

Fig. 8: 40%Ni-Fe (8hrs milling)

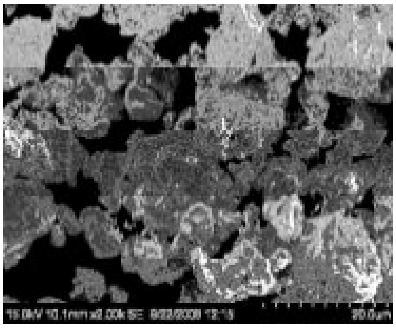

Fig. 9: 40%Ni-Fe (16hrs milling)

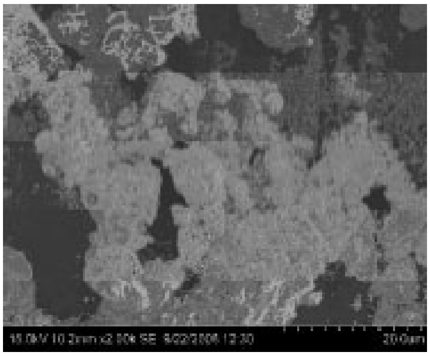

Fig. 10: 40%Ni-Fe (32hrs milling)

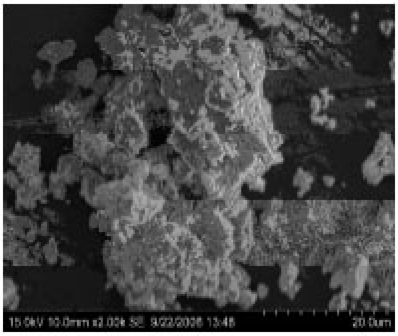

Fig. 11: 40%Ni-Fe (64hrs milling)

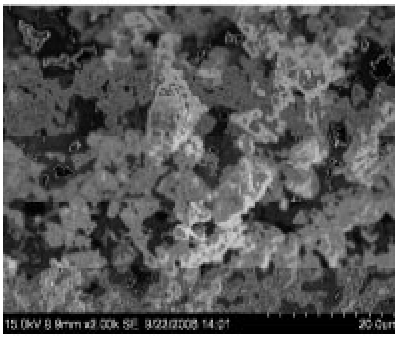

Fig. 12: 40%Ni-Fe (100hrs milling)

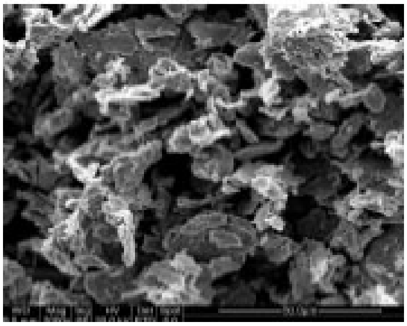

Fig. 13: 75%Ni-Fe (2hrs milling)

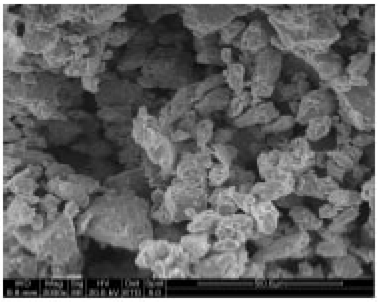

Fig. 14: 75%Ni-Fe (8hrs milling)

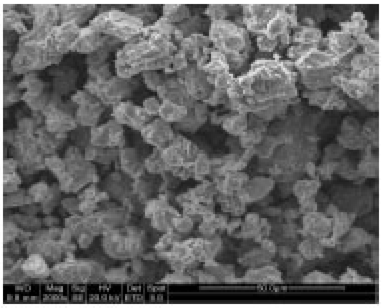

Fig. 15: 75%Ni-Fe (16hrs milling)

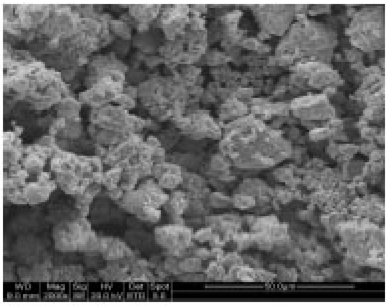

Fig. 16: 75%Ni-Fe (32hrs milling)

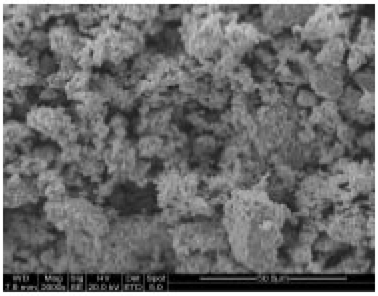

Fig. 17: 75%Ni-Fe (64hrs milling)

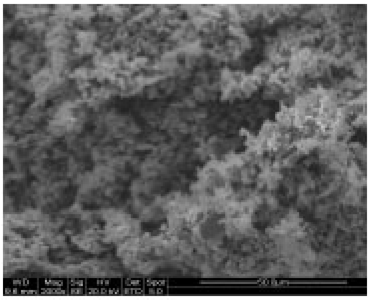

Fig. 18: 75%Ni-Fe (100hrs milling)

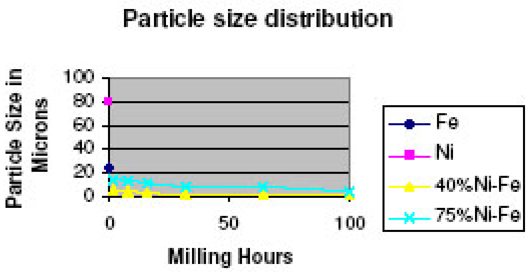

Fig. 19: Particle size at various milling hours

structures with distinct separation between the particles at eight hours of milling. It can be seen from Figure 9 that after 8 h of milling the mixture consists of both fine and large size particles.

Further milling up to 32h has resulted fracturing of only the largest particles and hence the particle size keeps on decreasing. Figures 10 to12 show that the particle size decreases slowly till the particle size is the lowest at 100 h of milling. (Figures 13 to 18) show the morphological development of the particles after 2, 8, 16, 32, 64 and 100 h of milling. It is observed from Figure.6 that after 2 h of milling the compressive forces due to the collision of the milling balls flatten the particles as the material is usually soft at this stage. (Fig. 14) shows layered structures with distinct separation between the particles at 8 h of milling. On 2 h of milling the mixture consists of both fine and large size particles. Further milling up to 8 h has resulted in fracturing of only the large particles and hence after 8 h of milling uniform particle size of about 13.2µm is obtained. (Figs. 15 to 16) show microstructure changes during 16 to 32 h of milling. The average particle size decreases as the fracturing forces dominate in this region. (Figs. 16 and 17) indicate that the average particle size variation between 32 and 64 h of milling is very small ie. 8 to 7.5µm only. This may be due to the equilibrium between the fracturing and cold welding forces during this period. Beyond 64 h of milling the particle size decreases as the fracturing force dominates in this region and the smallest size of 3µm results at 100 h of milling which is shown in Fig. 18.

Table 1: Average grain size with milling time

Milling Hours	40% Ni-Fe nm	75% Ni-Fe nm
0hr Fe	160.28	160.28
0hr Ni	280.22	280.22
2	109.78	214.66
8	18.213	77.43
16	73.028	74.54
32	54.72	65.02
64	18.515	35.24
100	7.2718	33.86

Table 2: Particle size

Milling Hours	Average particle size in µm 40% Ni-Fe	Average particle size in µm 75% Ni-Fe
0 hr Fe	24.22	24.22
0 hr Ni	79.29	79.29
2hrs	4.8	13.98
8hrs	3.846	13.19
16hrs	2.0	11.83
32hrs	1.72	7.81
64hrs	1.3	7.48
100hrs	0.967	3.22

The variation of average particle size with milling time is listed in Table II and shown in Figure19. In 40% Ni-Fe alloy for Ni powder the particle size decreases from 80 µm to 4.8 µm after 2 h while for Fe the decrease is only from 24 µm to 4.8 µm at 2 h. In 75% Ni-Fe alloy for Ni powder the particle size decreases from 80 to 13.98 µm at 2 h while for Fe the decrease is only from 24 to 13.92 µm at 2 h. The decrease is much higher for Ni due to its less ductility compared to Fe. In 40% Ni-Fe alloy the particle size decreases continuously with milling time as the fracturing forces dominate over the cold welding forces over the entire period. However between 32 h to 100 h, the particle size does not change appreciably due to the equilibrium between the fracturing and cold welding forces. The particle size reaches the lowest value of 0.967µm at the end of 100 h of milling. In 75% Ni-Fe alloy during 2 to 8 h of milling in the particle size remains constant at 13.5 µm. During 32 to 64 h of milling, it is observed that the particle size remains constant at 7.5 µm. This may be due to equilibrium between the fracturing and cold welding forces in these regions of milling. The particle size decreases after 64 h and reaches the lowest value of 3 µm at 100 h of milling. Comparing those two alloys, it could be observed that the percentage of nickel increases the rate of reduction of grain and particle size decreases because of Ni is little harder than Fe. It can be observed from Table 1 and 2 during mechanical alloying while the grain size changes the particle size almost remain constant or the particle size changes the grain size almost remain constant. This may be due to the energy consumption either changes in grain size or particle size.

CONCLUSION

Mechanical alloying of 40%Ni-Fe and 75%Ni-Fe have been investigated using X-ray Diffraction, Scanning Electron Microscope and Particle Size Analyzer. The main conclusions drawn from the study are:

- From the XRD spectra for 40%Ni-Fe alloy at various milling hours, it can be seen that at 8 h of milling the alloy exhibits XRD pattern reflecting amorphous structure. This shows that amorphization is fully achieved by ball milling alone after 8h of milling with out performing additional treatments such as annealing for milled power products.
- The grain size in 40%Ni-Fe alloy is observed to decrease with milling till 8 h and then increases and reaching the next highest value at 16h. After 16 h the grain size continuously decreases reaching the lowest value of 7.3 nm at 100 h. The grain size in 75%Ni-Fe alloy is decreases rapidly at the beginning of the milling and after 64 h of milling the change in grain size is not appreciable i.e. 35nm to 34nm only.
- In 40%Ni-Fe alloy the particle size decreases continuously with milling time. Between 32 h to 100 the change is not appreciable. The particle size reaches the lowest value of 0.967µm at the end of 100 h of milling. In 75%Ni-Fe the particle size generally decreases with milling time. Equilibrium between fracturing and cold welding forces is found to be established between 2 and 8 h and also between 32 and 64 h of milling as shown by the nearly constant particle size in these regions.
- When the percentage of nickel increases the rate of reduction of grain and particle size decreases. In mechanical alloying while the grain size changes the particle size almost remain constant or the particle size changes the grain size almost remain constant. This may be due to the energy consumption either changes in grain size or particle size while mechanical alloying.

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