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INVESTIGATION OF STRUCTURAL AND MAGNETIC PROPERTIES OF NI-FE-MO NANOCRYSTALLINE ALLOY SYNTHESIZED BY MECHANICAL ALLOYING

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ABSTRACT

Nanocrystalline $Ni_{78.5}Fe_{16.5}Mo_5$ has been synthesized by mechanical alloying. Appropriate amount of fine metal powders were mixed thoroughly and milled in a planetary ball mill in a dry medium under Argon (Ar) atmosphere. As milled powders were experienced a low temperature annealing under Ar atmosphere. Formation of alloy and the effect of low temperature annealing were studied by X-ray Diffraction (XRD) analysis. Nelson-Riley function was used in order to determine Lattice parameters. Theoretical density was calculated from the XRD peak. Mean crystallite size and strain calculations were carried out using Williamson-Hall plot. Particle size of the alloyed sample was studied by Scanning Electron Microscopy (SEM). Energy Dispersive X-ray (EDX) analysis was carried out at different points to investigate the alloy composition and its homogeneity. Magnetic properties of the samples were determined using a Vibrating Sample Magnetometer (VSM).

Keywords: *Supermalloy, Mechanical Alloying, Nanocrystalline, Magnetization, Coercivity.*

1. INTRODUCTION

Ni-rich materials are famous for their remarkable soft magnetic properties which draw intensive attention of researcher's all over the world over last half a century. Ternary alloy of Ni-Fe-Mo with the composition of 79%Ni-16%Fe-5%Mo is known as supermalloy (Boothby and Bozorth, 1947). This small amount of Mo addition on the permalloy (80%Ni-20%Fe) (Arnold and Elmen, 1923) shows superior magnetic properties over permalloy and leads to use in various electronic devices. In the coarse grain material, coercivity increases with the decrease of grain size (E. du Tremolet de Lacheisserie *et al.*, 2005). But low coercivity is found again when the grain size decreases to nanometer range. This phenomenon is explained by random anisotropy model (RAM) (Alben *et al*., 1978; Herzer, 1990 and 1997). Mechanical Alloying (MA) is one of the effective routes to produce nanocrystalline material (Murty, 1993). MA is a process where alloy forms by welding of different elemental powders and then fractured, re-welding and fractured again and so on (Suryanarayana, 2001). Although nanostructured grains formed by MA, mechanically alloyed materials do not follow RAM due to the high level defect employed during MA. In recent years some attempts have been taken to produce nanocrystalline Ni-rich materials by MA (Chicinas *et al.*, 2004; Denisa *et al.*, 2007; Isnard *et al.*, 2005; Neamtu *et al.*, 2011; Shen *et al.*, 2004a and 2004b). We investigated the structural and magnetic properties of $Ni_{78.5}Fe_{16.5}Mo_5$synthesized by MA.

2. EXPERIMENTAL

Metallic powders of Ni (99.8% pure), Fe (>99% pure) and Mo (>99% pure) was mixed thoroughly and milled in a planetary ball mill for 20hrs with stainless steel balls and vial. Milling program was set as 5mins of milling then 2mins of interval to avoid excessive heat during milling. Equally weighted balls of 5mm and 10mm were taken to mill with a speed of 300rpm. Ball to powder ratio was 20:1. To avoid oxidation of powders, milling was carried out under Ar atmosphere. The as milled samples were then annealed at 600K for 4hrs to release stress. Structural characterization was performed by XRD analysis with Cu-Kα radiation (λ=1.5406Å). Nelson-Riley function given by, $F(\theta) = \frac{1}{2}\left[\frac{cos^2\theta}{sin\theta} + \frac{cos^2\theta}{\theta}\right]$ was used, to calculate lattice parameters. Crystallite size and micro-strain was calculated from Williamson-Hall plot using XRD patterns. XRD peak broadening consists of three individual effects, broadening due to size, strain and instrumental and is given by,

$$\beta_{observed}=\beta_{size}+\beta_{strain}+\beta_{instrumental} \quad (1)$$

The peak broadening of the XRD patterns of primary sample (PS) (samples milled for 0hrs) can be considered as $\beta_{instrumental}$ and can be deducted from (1) which gives us,

$$\beta_r = \beta_{observed}-\beta_{instrumental} =\beta_{size}+\beta_{strain} \quad (2)$$

If the peak is Gaussian then β_r can be given by,

$$\beta_r= \sqrt{(\beta_o^2-\beta_i^2)} \quad (3)$$

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From the Schereerformula we have,

$$\beta_{size} = \frac{k\lambda}{D\cos\theta} \quad (4)$$

and, $\beta_{strain} = 4\eta\sin\theta$ (5)

Using these we get,

$$\beta_r = \frac{k\lambda}{D\cos\theta} + 4\eta\tan\theta$$

or, $\beta_r\cos\theta = \frac{k\lambda}{D} + 4\eta\sin\theta$ (6)

Now, plotting $\beta_r\cos\theta$ against $4\sin\theta$, intercept gives the crystallite size and slop gives the micro-strain. Particle size of the alloyed powders was determined by FESEM analysis using linear intercept technique. Composition and homogeneity of the alloyed powders were analyzed by EDX analysis at 4 different points. Magnetic properties were investigated by VSM analysis at room temperature.

3. RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns of the samples for different conditions. The vertical bars at the top of the figure shows the individual peaks of Ni, Fe and Mo, and the bars at the bottom shows the peak positions NiFeMo alloy. From figure it is clear that the after 20hrs of MA, all the individual peaks of Ni, Fe and Mo disappeared and combinedly forms 5 peaks of alloy. After annealed at 600K for 4hrs these peaks slightly shifted to the higher angle, elaborately shown in Figure 2 with an exact intensity, which was due to the effect of releasing stress and increased degree of crystallinity. A similar behavior was observed in (Chicinas *et al.*, 2003; Pop *et al.*, 2003).

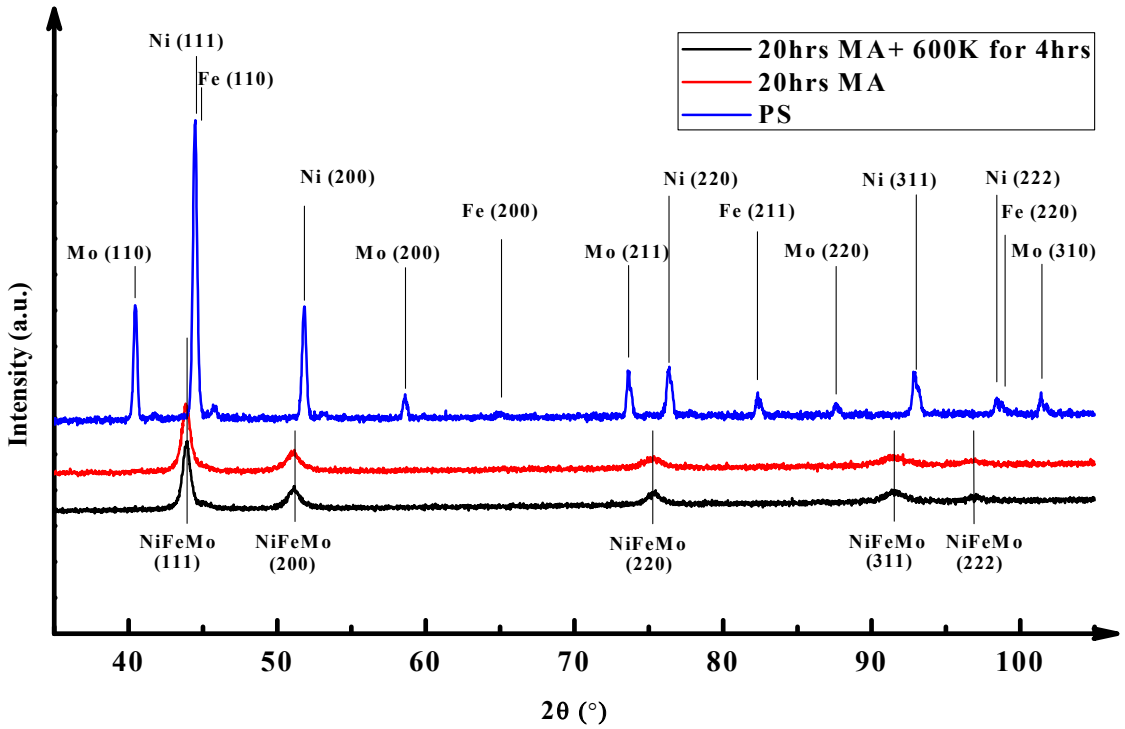


Figure 1: XRD patterns of PS, 20hrs milled and 20hrs milled plus annealed samples.

Figure 3 shows the lattice parameters of as milled and annealed samples calculated by using Nelson-Riley function and found to be decreased after heat treatment, which is another evidence of increased crystallinity. However the theoretical density is found to be increased after annealing in accordance with the fact of decreasing defect. Numerical values of lattice parameters and theoretical density is shown in Table 1. Average crystallite size and micro-strain were measured from the XRD pattern of 20hrs MA+600K for 4hrs and found 29nm and 0.43% respectively as shown in Figure 4.

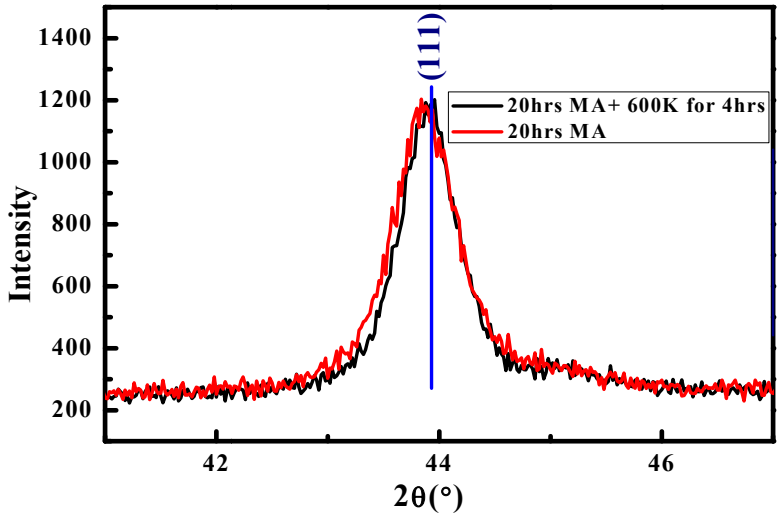


Figure 2:XRD patterns with an exact intensity of 20hrs as milled and milled plus annealed samples.

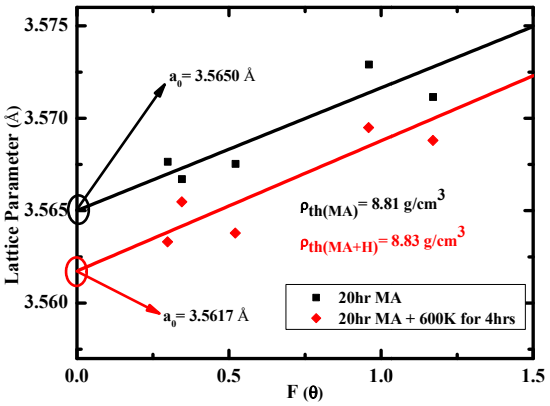


Figure 3: Lattice parameters of as milled and annealed samples.

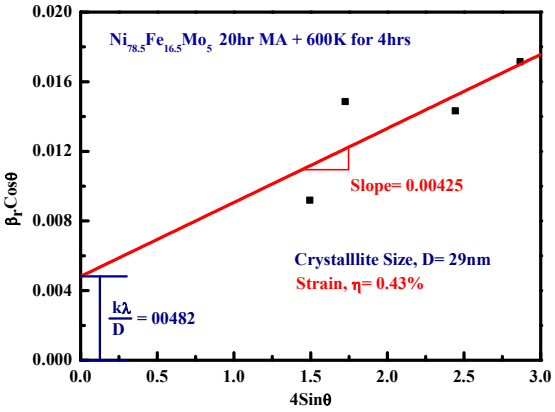
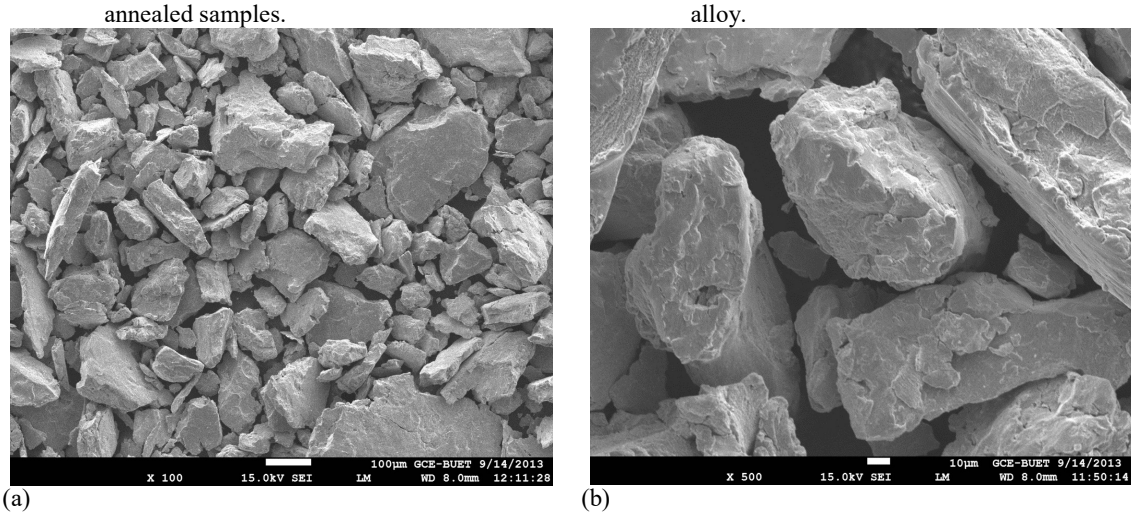


Figure 4: Crystallite size and micro-strain of the alloy.



(a) (b)

Figure 5: FESEM image of 20hrs milled samples (a) 100×, (b) 500× magnified.

Table 1: Numerical values of a, ρ, M_s, M_r and $H_c$for as milled and annealed sample.

	Lattice parameters, a (Å)	Theoretical density,ρ ($g/cm^{3)}$	Saturation magnetization, M_s (emu/g)	Remanence magnetization, M_r (emu/g)	Coercivity, H_c (Oe)
20hrs MA	3.5650	8.81	59.54	0.47	3.38
20hrs MA + 600K for 4hrs	3.5617	8.83	62.62	0.30	2.26

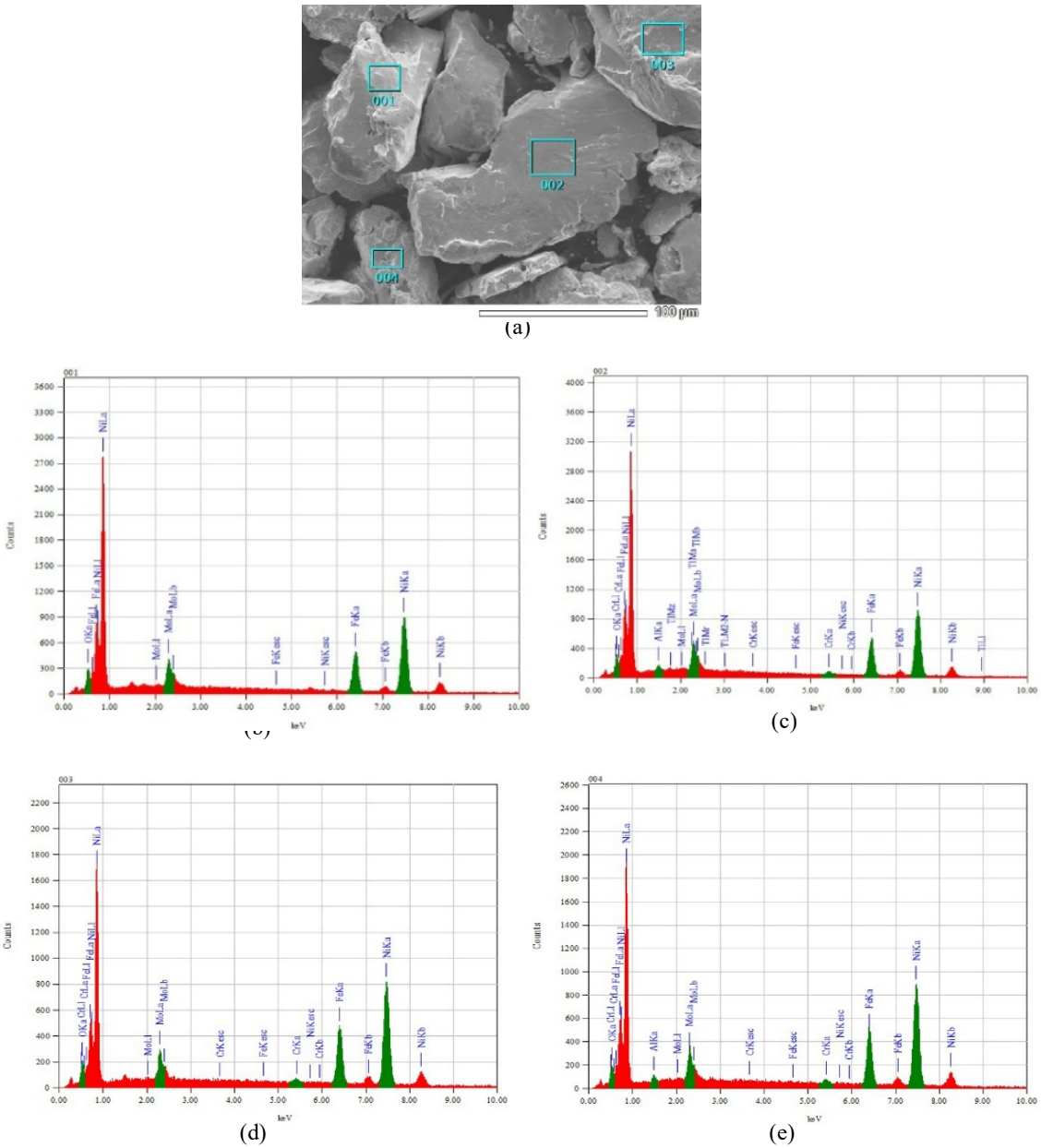


Figure 6: EDX-analysis for different points, (a) position of points, (b) counts for point 1,(c) point 2, (d) point 3 and (e) point 4.

Particles formed after milling of 20hrs found to be varied in size, shown in Figure 5. Average particle size of the as milled powders was measured from the 100X magnified image shown in Figure 5 (a) by using linear intercept technique and found to be 59µm after 20hrs of milling. Figure 6 shows the EDX-analysis for 4 different points. Numerical values of these analyses are shown in Table 2. From the data it is clear that the sample has got some impurity during milling which mostly comes from the milling medium, as we used stainless steel balls and vial.

It is also observed that the percentage of elements varies point to point, in a little amount, which indicates the fair enough homogeneity for the powders synthesized by MA. The percentage of Ni decreased from 78.5 to around 71% and Fe increased from 16.5 to around 22%, which indicates the impurity comes in the form of Fe mostly from the milling medium.

Figure 7 shows the magnetic properties of the samples determined from the M-H loop analysis. According to Figure 7 it is found that saturation magnetization increased after annealing whereas coercivity and remanence magnetization follows the opposite trends. It can be explained by the fact that annealing completed the alloying process, released stress, as well as minimizes defects involved during milling. A similar phenomenon was reported in (Gheisari *et al.*, 2011). Numerical values of saturation magnetization, remanence magnetization and coercivity is shown in Table 1.

Table 2: **N**umerical values of EDX-analysis of the powders for different points

PS- $Ni_{78.5}Fe_{16.5}Mo_5$	Point 1	Point 2	Point 3	Point 4
Ni	71.75	69.43	71.92	71.05
Fe	20.39	20.26	21.10	21.17
Mo	6.99	7.27	5.49	5.66
O	0.87	0.70	0.70	0.52
Cr	0.0	0.99	0.78	1.08
Ti	0.0	0.76	0.0	0.0
Al	0.0	0.60	0.0	0.52

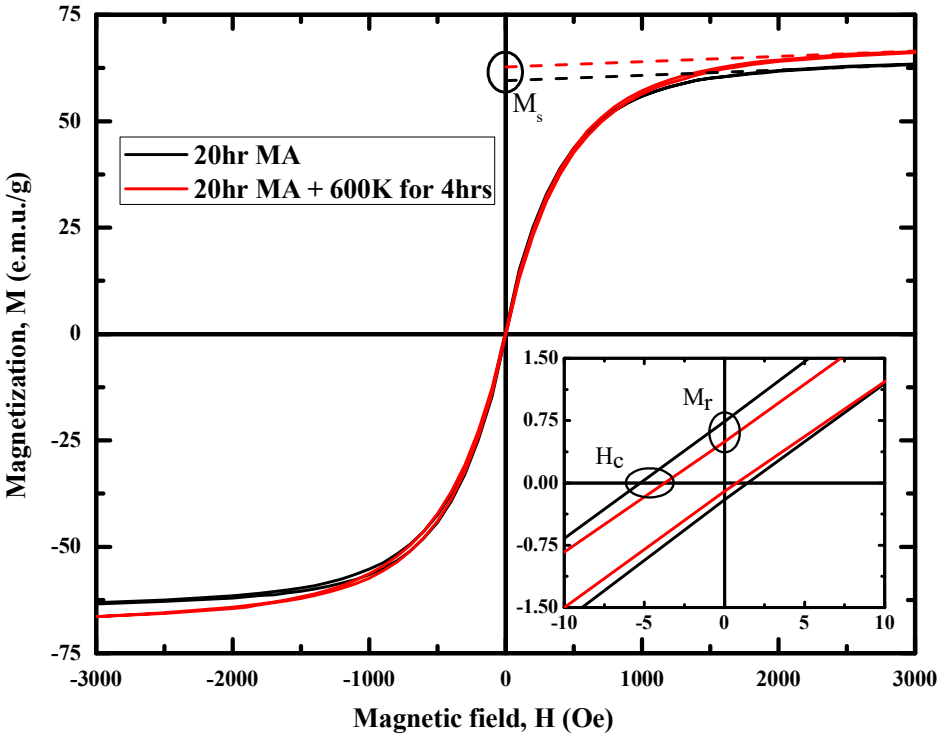


Figure 7: M-H loop of the as milled and annealed samples.

4. CONCLUSION

An alloy of NiFeMo has been found after 20hrs of milling with a face centered cubic structure. Heat treatment released stress and increased crystallinity. Lattice parameters decreased and theoretical density increased with annealing. An average crystallite size of the 20hrs milled and annealed samples were found to be 29nm with a micro-strain of 0.43%. Average particle size of the milled samples was calculated 59µm. Homogeneity was observed in the samples with some impurities. Saturation magnetization increased with heat treatment, whereas coercivity and remanence magnetization follows the opposite trends.

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