

Effect of Annealing on Magnetic and Structural Properties of FeNi Thin Films

A. Sharma^{1,*}, J. Tripathi², Yogesh Kumar¹, G.S. Chandrawat^{1,2}, R. Bisen^{1,2}, S. Tripathi³

¹ Department of Physics, Manipal University Jaipur, Jaipur, India

² Department of Physics, ISLE, IPS Academy, Indore, India

³ Atomic and Molecular Physics Division, Bhabha Atomic Research Centre, Mumbai, India

(Received 15 February 2020; revised manuscript received 10 April 2020; published online 25 April 2020)

In this work, we have deposited FeNi (60 nm) film on Si (111) substrate using electron beam evaporation technique. XRD measurements carried out on thin film with annealing the sample from 100 to 400 °C show increase in intensity of the FeNi (111) diffraction peak. The particle size increases from 19 nm (as deposited sample) to 27 nm (400 °C) with annealing. The magnetization measurements carried out using MOKE technique show enhancement in coercivity with annealing. The overall results are attributed in terms of annealing induced structural change which modify the magnetic properties.

Keywords: XRD, MOKE, FeNi, Structural, Magnetic.

DOI: [10.21272/jnep.12\(2\).02040](https://doi.org/10.21272/jnep.12(2).02040)

PACS numbers: 75.70.i, 68.37.Ps, 68.37.Rt

1. INTRODUCTION

Magnetic thin films have tremendous applications in different devices which are used to store data, read and write data such as storage devices, magnetic sensors, read and write heads [1-3]. The never ending demand to increase areal density requires a controlled over microstructure of underlying layers. In these devices, a soft magnetic layer is required which serves as a reference layer. For example, in recording heads a soft magnetic layer with low coercivity, high magnetic moment with higher electrical resistance required. However the performance of this reference layer is sensitive to various microstructure parameters such as film thickness, grain size deposition parameters and any variation in this may affect overall performance of the device [4-5]. Therefore, a systematic study is required to understand the variation in these parameters on the magnetic properties of thin films. In this respect FeNi system has attracted considerable attention of the researchers due to some unique properties exhibited by this system. FeNi system is widely studied owing to its excellent soft magnetic properties such as low coercivity and high permeability, high electrical resistance, high saturation magnetization, low magnetostriction, high corrosion resistance and one can also control magnetic anisotropy in this system [6-8]. Due to these features, this system is extensively studied by the researchers.

The recording media industry required low thermal fluctuation and higher uniaxial anisotropy of the material. However, some existing systems such as CoPt, FePt and FePd show higher uniaxial magneto crystalline anisotropy but here the main constraint in the presence of noble metal which makes this system expensive. The FeNi system provides advantage that it does not contain any noble metal and anisotropy can be easily tuned. All these properties makes this system a potential candidates in fabricating next generation storage devices [9-11]. This system has been studied extremely in term of thin films, bilayers, multilayers,

alloy, and nanoparticles [12-16].

In FeNi atomic alloy system by varying deposition and annealing conditions, a tetragonal L1₀ structure is formed which exhibits extraordinary magnetic properties equivalent to observe in rare earth based compounds [17]. A number of studies are reported in the literature focuses on synthesis and characterization of L1₀ ordered FeNi thin films [18-19].

In the literature researchers have done study of microstructural investigations of FeNi system such as variation in doping, annealing, deposition condition and film thickness in this system and various interesting observations observed like change in structure from crystalline to amorphous, change in anisotropy [biosensor], in plane magnetic softness with annealing [2]. In this system, variation of the working pressure is an effective tool for the tuning of magnetic properties and also dynamic anisotropy decreases with increase in film thickness [20].

However, despite several studies exist on this system. Very few studies deal with the annealing induced changes in structure and its impact on the magnetic properties. Keeping this point in mind, FeNi thin film was fabricated by electron beam evaporation in molecular beam epitaxy (MBE) chamber. The structural and magnetic properties of FeNi films were investigated by varying the annealing temperature, and strong annealing temperature dependence was observed.

2. EXPERIMENTAL

2.1 Thin Film Deposition

Prior to FeNi deposition, Si (111) substrate was thoroughly cleaned following a number of steps such as etching of native oxide by Hf, ultra-sonication and finally blowing with dry N₂. The target material FeNi was commercial product with purity higher than 99.99 %. FeNi (60 nm) film was deposited on cleaned Si substrate at room temperature by electron beam evaporation in a molecular beam epitaxy chamber (rate

* anupamcsr@gmail.com

~ 0.1 Å/s) under ultra-high vacuum (~ 5 × 10⁻¹¹ Torr) condition. The sample was annealed in situ from 100 °C to 400 °C in step of 1 hour in a separate vacuum chamber under UHV conditions.

2.2 Characterization Techniques

The magnetic properties of these thin film structures were measured by magneto optical Kerr effect (MOKE) technique in longitudinal configuration with He-Ne laser of wavelength 632.8 nm. X-ray diffraction measurements were carried out with CuKα (λ = 0.154 nm) radiation on Bruker D8-discover machine (40 KeV, 30 mA). The XPS measurements were done on VSW scientific instrument (UK) with twin anode facility was used (Al K_α source, resolution 0.9 eV). Binding energy (B.E.) correction was done using the reference of graphitic C-1s peak, while Au sample served as an external reference for resolution and B.E. reference. All the measurements were carried out at room temperature.

3. RESULTS AND DISCUSSION

3.1 X-ray Diffraction (XRD)

Figure 1 shows the XRD pattern of as deposited and annealed thin films. All the samples annealed from 100 to 400 °C showed FeNi (111) fcc fundamental peaks at 2θ position 44.46°. No other impurity peak is found in the all the samples. It is clear from the figure that the deposited samples grow in polycrystalline manner. In addition to main FeNi, a small peak due to underlying substrate is also visible.

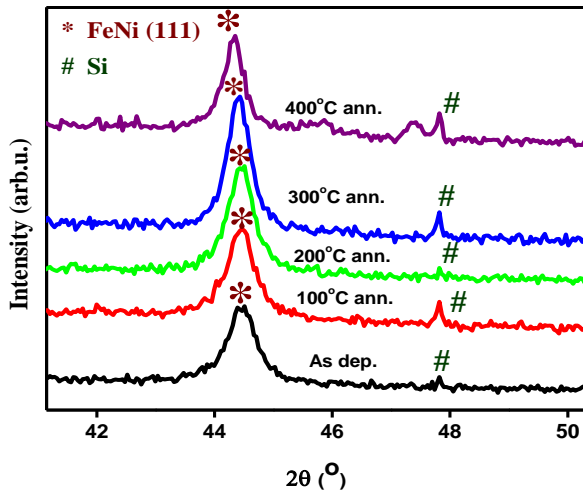


Fig. 1 – XRD patterns of as prepared and annealed thin films

With increase in annealing temperature, main peak shifted towards the left side due to stain relaxation .As it is well known that in as deposited film stress is present and with annealing stress releases. In all the samples, particle size calculated with the well-known standard Scherrer relation [21]

$$L = \frac{0.9\lambda}{\beta \cos \theta}$$

where, λ is the incident X-ray wavelength, β = full

width at half maxima of the peak and θ is the Bragg angle. The calculated particle size is plotted as a function of annealing temperature in Fig. 2. The particle size increases from 19 nm to 27 nm with increasing the annealing temperature from 25 to 400 °C. This attributed to the fact that the tendency for agglomeration of the particles increases with increasing the annealing temperature [15]. Increase in particle size with annealing is reported in the literature [16].

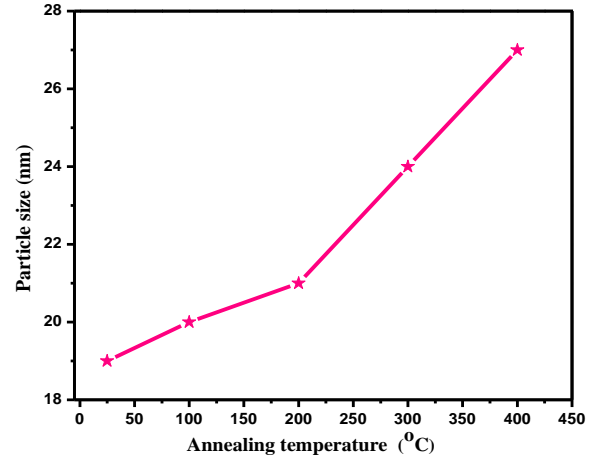


Fig. 2 – Variation in Particle size with annealing temperature

3.2 Magnetic Measurements

To record the magnetic properties of all the samples, MOKE technique has been employed. All the magnetic measurements have been carried out keeping applied magnetic field parallel to sample surface and loops recorded till the saturation magnetization is achieved (see Fig. 3). Some of the recorded loops show some noise which may be due to the roughness present in the sample or it may be due to electronic noise.

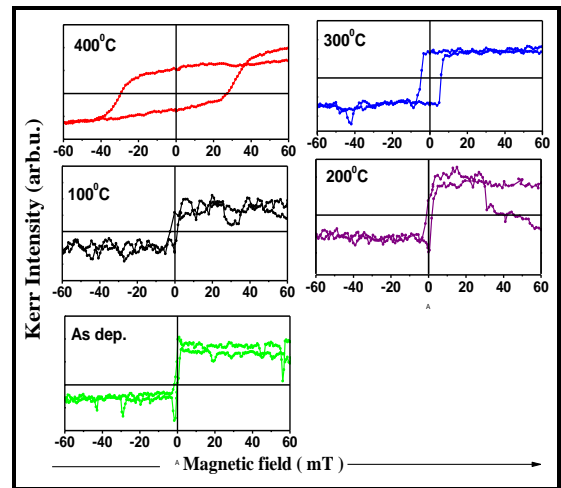


Fig. 3 – MOKE hysteresis loops of as prepared and annealed FeNi thin films

The coercivity and other parameters extracted from the hysteresis loop is tabulated in Table 1. The M-H loop of as deposited sample show very low coercivity of 1.2 mT which shows the soft magnetic nature of FeNi film. Low coercivity in as deposited FeNi films are also reported in the literature [22]. However, coercivity in-

increases to 2 mT in 100 °C annealed sample and decreases to 1.6 mT and increases thereafter. The initial increase in coercivity may be due to release of stress present in the sample. However, the increase in coercivity beyond 200 °C due to increases in intermixing between Fe and Ni atoms which leads to disorderness. The squareness ratio (M_r/M_s) also change with annealing which may be due to different reversal mechanism occurs in the sample.

Table 1 – Calculated magnetic properties with different annealing temperatures

Sr. No.	Annealing temperature (°C)	H_c (mT)	M_r	M_s	M_r/M_s
1.	As-prepared	1.2	0.306	0.260	1.17
2.	100 °C	2	0.028	0.025	1.12
3.	200 °C	1.6	0.039	0.040	0.97
4.	300 °C	5.4	0.320	0.340	0.941
5.	400 °C	28	0.291	0.230	1.26

3.3 XPS Measurements

In order to check the quality of as deposited films and phase formation, XPS measurements have been carried out on as deposited sample. The measurements done on as deposited sample shows presence of contamination such as elemental carbon and oxygen due to exposure of sample to atmosphere. However, spectra recorded after 20 min sputtering shows (Fig. 4) presence of Au-4f- $7/2$ peak at 84 eV and 4f- $5/2$ peak at 87 eV matches with the standard values reported for elemental Au and absence of any impurities [23].

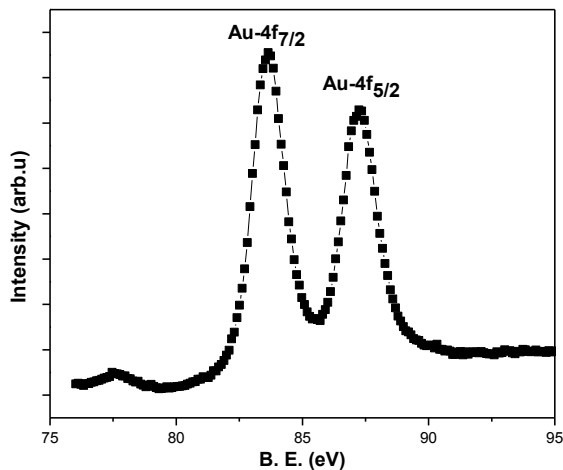


Fig. 4 – Au core level spectra recorded after 20 min sputtering

Figure 5 shows the Fe-2p core level spectra recorded after 20 min sputtering. It is clear from the spectra that these two peaks could not be resolved well and appear as one. The $2p_{3/2}$ peak observe at 713 eV B.E. position whereas peak position of $2p_{1/2}$ photoemission was 712 eV. The observed positions of these two peaks matches with the positions reported for FeNi alloy film [24].

In contrast to Fe-2p peaks, well resolved peaks of Ni-2p $3/2$ and $1/2$ were observed and same shown in Fig. 6.

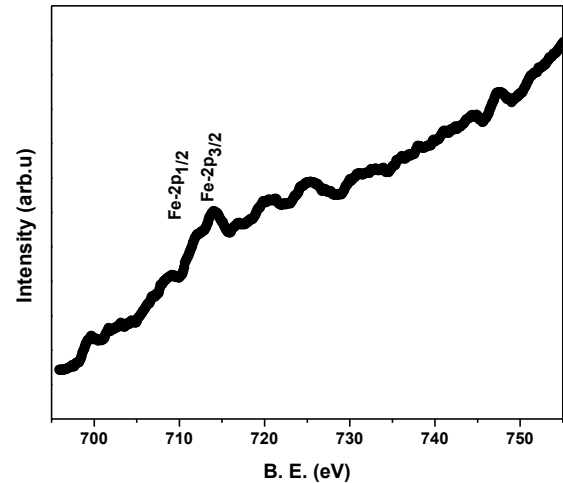


Fig. 5 – Fe-2p core level peaks recorded after 20 min sputtering

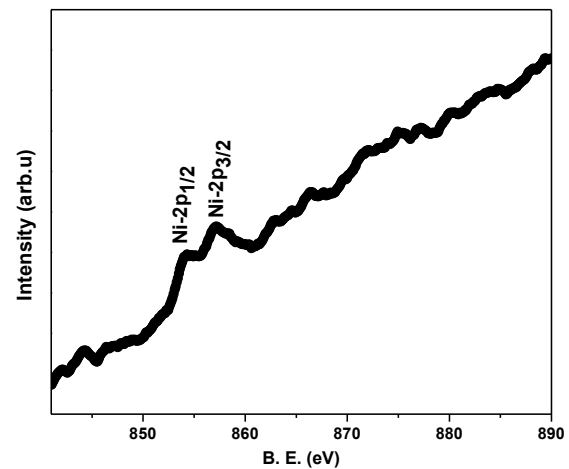


Fig. 6 – Ni-2p core level peaks recorded after 20 min sputtering

Both Fe-2p and Ni-2p peaks shows asymmetry towards higher binding energy side which suggest metallic nature of FeNi alloy film. Ni-2p $3/2$ and $1/2$ peaks were observed at 857 eV and 854 eV binding energy positions. The observed values of these peaks matches well with the reported values [25]

4. CONCLUSION

FeNi film with the thickness of 60 nm was prepared by electron beam evaporation in molecular beam epitaxy (MBE) chamber on Si (111) substrates at room temperature. FeNi film in-situ annealed in vacuum at different annealing temperatures. The structural and magnetic properties of FeNi film were investigated. XRD shows as annealing temperature increases, crystallinity also increases. With increase in annealing, FeNi (111) peak shifts due to release of stress and strain present in the film. MOKE results show an increase in coercivity with annealing. In addition, magnetization curves also show change in loop shape with annealing. XPS measurements shows good quality of deposited thin film and formation of FeNi phase. The observed results are attributed to annealing induced intermixing which modifies the structural and magnetic properties.

ACKNOWLEDGEMENTS

The authors A. Sharma and S. Tripathi thank Prof. A. Vantomme and the group, KUL, Belgium for providing deposition, characterization facilities and financial sup-

port during the research work. The authors are also thankful to Dr. T. Sripathi, UGC-DAE CSR, Indore for his help in XPS measurement.

REFERENCES

1. A.V. Svalov, I.R. Aseguinolaza, A. Garcia-Arribas, I. Orue, J.M. Barandiaran, J. Alonso, M.L. Fernández-Gubieda, G.V. Kurlyandskaya, *IEEE T. Magn.* **46** No 2, 333 (2010).
2. Zhenjun Xia, Jun He, Xiulong Ou, Shuli He, Dongliang Zhao, Guanghua Yu, *Appl. Phys. A* **122**, 860 (2016).
3. Huiyu Chen, Chunju Xu, Guizhe Zhao, Yaqing Liu, *Mater. Lett.* **91**, 75 (2013).
4. Marco Coisson, Gabriele Barrera, Federica Celegato, Paola Tiberto, Franco Vinai, *J. Phys.: Conf. Ser.* **365** No 1, 012003 (2012).
5. San-Sheng Wang, Yu Zhang, Zhu-Li Zhang, Wen Jiang, Fang Li, Zi-Yu Chen, *J. Magn. Magn. Mater.* **444**, 291 (2017).
6. Oliver Gutfleisch, Matthew A. Willard, Ekkes Brück, Christina H. Chen, S.G. Sankar, J. Ping Liu, *Adv. Mater.* **23** No 7, 821 (2011).
7. G.V. Kurlyandskaya, A.A. Chlenova, E. Fernández, K.J. Lodewijk, *J. Magn. Magn. Mater.* **383**, 220 (2015).
8. Huiyu Chen, Chunju Xu, Guizhe Zhao, Yaqing Liu, *Mater. Lett.* **91**, 75 (2013).
9. D. Weller, A. Moser, *IEEE T. Magn.* **35**, 4423 (1999).
10. S.N. Piramanayagam, *J. Appl. Phys.* **102**, 011301 (2007).
11. T.Y. Tashiro, M. Mizuguchi, T. Kojima, T. Koganezawa, Masato Kotsugi, T. Ohtsuki, K. Takanashi, *J. Appl. Phys.* **117**, 17E309 (2015).
12. G.V. Kurlyandskaya, J.L. Muñoz, J.M. Barandiarán, A. García-Arribas, A.V. Svalov, V.O. Vas'kovskiy, *J. Magn. Magn. Mater.* **242–245**, 291 (2002).
13. D. de Cos, V.N. Lepalovskij, G.V. Kurlyandskaya, A. García-Arribas, J.M. Barandiarán, *J. Magn. Magn. Mater.* **320**, 954 (2008).
14. C. Devi, R. Ashokkumar, E. Ranjith Kumar, *J. Inorg. Organometal. Polymer. Mater.* **28** No 5, 1787 (2018).
15. M. Nirouei, A. Jafari, K. Boustani, *J. Superconduct. Novel Magn.* **27**, No 12, 2803 (2014).
16. Zhichao Xu, Chuangui Jin, Ailin Xia, Junyan Zhang, Guohui Zhu, *J. Magn. Magn. Mater.* **336**, 14 (2013).
17. Laura H. Lewis, A. Mubarak, E. Poirier, Nina Bordeaux, P. Manchanda, A. Kashyap, Ralph Skomski, *J. Phys.: Condens. Matter* **26** No 6, 064213 (2014).
18. Masahiro Saito, Hisaaki Ito, Yuta Suzuki, Masaki Mizuguchi, Tomoyuki Koganezawa, Toshio Miyamachi, Fumio Komori, Koki Takanashi, Masato Kotsugi, *Appl. Phys. Lett.* **114** No 7, 072404 (2019).
19. Eric Poirier, E. Frederick, Pinkerton, Robert Kubic, Raja K. Mishra, Nina Bordeaux, Arif Mubarak, H. Laura, Lewis, I. Joseph, *J. Appl. Phys.* **117** No 17, 17E318 (2015).
20. Derang Cao, Lining Pan, Xiaohong Cheng, Zhenkun Wang, Hongmei Feng, Zengtai Zhu, Jie Xu, *J. Phys. D: Appl. Phys.* **51** No 2, 025001 (2017).
21. S.G. Deshmukh, A.K. Panchal, V. Kheraj, *J. Mater. Sci.: Mater. Electron.* **28**, 11926 (2017).
22. N. Villar Alzola, G.V. Kurlyandskaya, A. Larrañaga, A.V. Svalov, *IEEE T. Magn.* **48** No 4, 1605 (2012).
23. H. Piao, McIntyre, *Surf. Sci.* **421**, L171 (1999).
24. N. Okude, M. Furuya, *J. Surf. Anal.* **5**, 270 (1999).
25. K.J. Kim, D.W. Moon, C.J. Park, D. Simons, G. Gillen, H. Jin, H.J. Kang, *Surf. Interface Anal.* **39**, 665 (2007).