Development of iron thin films by electron beam physical vapour deposition (EBPVD): A Review

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Abstract. This review paper study about the possibilities of results obtained from conducted experiment of iron (Fe) thin films by electron beam physical vapor deposition (EBPVD). Previous studies showed that by exposing the substrate of the thin film to pre-heat environment, the changes of morphology and adatom mobility is expected. Furthermore, annealed influence in the thin film also will give better-quality thin films as the surface are expected to be smoother and flat. Three different annealed temperature is conducted on the samples, which are 400°C, 800°C and 1200°C. Structural changes such as transition from alpha phase to beta phase, is possible due to the presence of high temperature.

1.0 INTRODUCTION

Iron (Fe) is a readily well known Earth’s material that has been widely used in many sectors and industries. The crystal structure of iron itself which is capable to extinguish in many forms by depending their phase present, makes iron of the interesting earth elements to study. Bulk Fe exists in alpha, beta, gamma and delta phase. In alpha phase for example, the crystal structure is in body-centered cubic (bcc), however, when subjected to temperature or other growth parameter, the phase can change to beta with possess a face centered cubic (fcc) structure. These different crystal structures gives different mechanical properties of the iron performance in materials and/or samples which is very crucial in determining specification of the performance when it comes to heavy industries.

For the last decade, the focus on developing iron-based materials is to study their behavior under magnetization. The presence of phase difference in iron makes it interesting, as it is capable to change from ferromagnetism to anti-magnetism. To date there are more than 20 known iron based oxides, iron based nitrides, iron based hydroxides and iron based oxides-hydroxides. However, as the demand for technology increases, researches have diverted their attention on bulk Fe to thin films Fe. Thin films are of interest as the growth of thin films give similar crystal structure as their parent material, but it has better performance in term of mechanical and physical properties. As the demand from industries such as micromechanical system (MEMS), semiconductor, hard coatings and many, thin films mechanical properties have become indispensable where these thin films are used for not only structure materials as well as electrical materials. Therefore, it is crucial to understand the characteristics of thin films in order to produce high performance thin films [1].

Metal based thin films and alloys are commonly produced by techniques like sputtering, molecular beam epitaxy and many other methods. As for iron thin films, our main aim is to develop an ultra-fine film as the changes in physical properties exhibit smaller grain size which effect the ferromagnetic properties of the material via electron beam physical vapor deposition (EBPVD). Fe based materials are one of the most promising candidates for high-density recording media to its high saturation magnetization and coercivity. Our main aim for

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this paper is to get the idea, understanding of metal-based thin films generally, and Fe specifically in term of their properties such as roughness, morphologies and structural changes [2,3,4].

2.0 EXPERIMENTAL PROCEDURE

2.1 Thin film deposition

All Fe thin films were deposited by electron beam physical deposition (EBPVD) KES-200. The thin films were grown on silicon substrate with (100) plane. This machine carried out the growth at a base pressure of <10^{-5} mbar and the target to substrate distance is around 50 cm. All depositions were carried out at about 80°C to 100°C with the current of 7.17 A. The deposition rate is about 2.16 nm/s with the average thickness for thin films of 220±10 nm. For the first batch of thin films, the substrate was pre-heated up to 300°C before the deposition takes place while the second batch is following a normal procedure. The deposition rate is not affected by the condition of the substrate (preheat or not). Instead, the deposition rates depend solely on the ion bombardment of the atoms on the substrates.

2.2 Different temperature on the thin films

Thin films with two different condition (pre-heated substrate and normal substrate) are subjected to another annealing condition, which is from 400°C to 1200°C. Higher temperature also used in order to study the difference in term of the surface roughness of the thin films. Not only the thin films are subjected to different deposition exposure (due to substrate temperature effect), the thin films are also believed to have changes its phases and morphology when it is subjected to temperature.

3.0 RESULTS AND DISCUSSION

3.1 Effects of pre-heating substrate temperature on thin films

For the morphology, we believed the effect of temperature after the thin films deposition would be affected for all the thin film samples. The surface morphology of thin films at different substrate heat treatment temperature changes depending on the temperature given. This is in agreement with Jugyoon et al. [2] which the results are shown in Fig. 1. Four thin films that were subjected to different substrate temperature which were room temperature, 150°C, 200°C and 300°C. Based on this research, the influence of pre heating the substrate before deposition can be seen to have an impact on thin films surface. For substrate without any heating influence, fine grain morphology is observed. However, line-fibers arrangement can be seen on the substrate temperature of 200°C and higher.

A similar trend can also be seen in (atomic force microscopy) AFM images of zinc oxide (ZnO) thin films (Fig. 2) conducted by Li et al. [4], where the substrate was preheated to three different temperature, which were 100°C, 200°C and 300°C respectively. The substrate in the room temperature shows a coarse surface with larger grain size. However, as the preheating temperature increased, the grains becomes denser and resulting in smaller grain size, which leads to smoother surface.

Therefore, for our project, similar microstructure on Fe thin films are predicted. As regards to thin film thicknesses, the decrease in thin film is expected when preheating the substrate. This is because the evaporation rate of initial product increased with higher substrate temperature. When this occur, mass transport towards the hot substrates will be reduced hence leading the thinner thin film thickness [1]. However, this is contradict with our preliminary experiment that shows thicker samples for the unheated substrates.

The increased in surface roughness is a results of volatilization of the particles. Enough energy is obtained from the sputtered particles that leads to the formation of better compact in thin films. This is only possible to achieve with the influence of high temperature. Moreover, higher temperature of substrate gives perfect crystals with bigger crystal size due to the enhancement of adatom mobility during the growth process. Thence, giving higher surface roughness with temperature increment. However, issues like inhomogeneity [4] should also be taken into account when preheating the substrate. If the surface of the substrate is not fully exposed to the temperature, it is not possible to obtain a homogeneous thin film. This is because; only area that was exposed to temperature will have a better atoms interaction between the metal target and substrates[5, 6].
Fig. 1. Adapted from J.E.S Kim et al.[2] where substrate of the Cu-Pc thin films deposited at (a) room temperature; (b) 150°C, (c) 200°C and (d) 300°C.

Fig. 2. Adapted from Li et al. AFM images of ZnO thin films deposited at various substrate temperature of (a) room temperature, (b) 100°C, (c) 200°C and (d) 300°C [4].
3.2 Effect of annealing temperature on thin films

In addition, after the thin film deposition, these films are then subjected to different annealing temperature for 1 hour. This method is similar to this project, which used similar temperature given to the pre-heating substrate range of 150°C, 200°C and 300°C, which is presented in Fig. 3. The heat treatment given to thin film is expected to result in the formation of metastable and stable phases.

In our case of Fe thin films, the grains and grain boundary is expected to change as higher temperature is given to the films. This is because the diffusion of atoms along the grain boundaries may be happening thus leading to denser grains and flatter surface. This is also supported by Gmter and Myer [7] which stated that with the increase of substrate temperature, mobility in increased that leads to better crystallinity of the film and in agreement that the grain size is also affected where with the higher temperature the shape of particles gave more surface areas to the microstructure compared to lower temperature.

Fig. 3. Adapted from J.E.S Kim et al.[2], where the Cu-Pc thin films are annealed at (a) room temperature; (b) 150°C, (c) 200°C and (d) 300°C.

One of the main issue in Fe thin films is poor surface/interface of the materials which leads to depleted performance in their magnetic and electronic transport properties [8,9]. The higher temperature given to the thin films also contributed to better quality of thin film crystallinity. This is because, increasing temperature will leads to not only larger particle size but also increase in grain size, thus reducing grain boundary. Lower grain boundary will give an insight of better carrier mobility which will greatly influenced the sample electrical properties such as resistivity [10-12].

The phase of the thin films is expected to change once the sample is subjected to different temperature. For Fe thin films, phases like alpha or beta might be present after the heat treatment. Similar results were showed by Chen et al. [13] in their study of effect of thermal annealing of zinc oxide thin films. Based on Fig. 4, the x-ray diffraction (XRD) diffractogram, peak shifts are present as the temperature increased up to 950°C. As the temperature increased, there were two new peaks exist especially at the highest temperature (950°C). These additional peaks and peak shifts in the diffractogram suggested that the sample has undergone structural changes, which will attribute to their mechanical properties.
4.0 CONCLUSION

In summary, the formation of Fe thin film on silicon is expected to experience some mechanical changes as their structural and morphologies changed. There are two reasons contributing to this; (i) the pre-heating of silicon substrate prior to deposition by EBPVD and (ii) the annealed temperature given to both samples with pre-heated substrate and unheated substrate. Results such as better quality of thin film and structural changes can be obtained by exposing these films to higher temperature. The outcomes can be observed by the crystallize size and roughness of the sample which is obtained by XRD and AFM.

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