CHAPTER 6

CoFe/MgO THIN FILMS SUBJECT TO DEPOSITION FIELD, CONVENTIONAL FIELD COOLING AND E-BEAM BASED FIELD COOLING – A COMPARISON

In the following final set of experiments of this dissertation, a comparative study on the effects of using a deposition field, field cooling and electron beam annealing on the morphological and magnetic properties of CoFe/MgO thin film system is discussed. This study is done on an application point of view. In the case of magnetic tunnel junctions as well as spin injection systems, the CoFe/MgO as well as the CoFeB/MgO layer acts as the main spin injector. The manipulation of spin states within such metallic and other semiconductor based spin injectors has the potential to produce memory, sensors and circuit components that could provide increased speed, non-volatility, density and decreased power consumption as compared to existing technologies based on charge [82-84].

In recent years there has been much research focus on the creation of populations of spin-polarized charge carriers inside semiconductors, since this is the primary step towards the development of semiconductor based spintronic devices. Preliminary work using diluted magnetic semiconductors at low temperatures demonstrated highly efficient spin injection into semiconductors [85-87]. However, the low magnetic ordering temperatures of these materials make them less attractive than injectors using traditional ferromagnetic metals such as Fe, Co, Ni and their alloys, which have Curie temperatures well above room temperature.

There is a high level of conductivity mismatch between such traditional ferromagnetic metals and semiconductors, which may limit spin injection efficiency [88]. A major contributor to this issue is the irregularities introduced on the surface as well as interfaces during deposition as well as annealing. Moreover conventional
annealing (which usually takes significant amount of time) promotes higher intermixing of the substrate atoms and those in the deposited films. Annealing for a prolonged time also promoted increased oxidation rates. Hence there is a growing demand for flash annealing techniques. A potentials flash annealing technique is E-beam Rapid Thermal Annealing (ERTA) [89, 90].

ERTA, as an annealing technique is exceptionally rapid and uncomplicated. The growth of the nanostructures occurs in annealing times as short as 10-60 seconds. The extremely high uniformity coupled with high throughput and compatibility with conventional processing techniques makes this a promising industrial technique for flash annealing [90]. A comparative study on the morphology and the magnetic properties of CoFe/MgO thin films subjected to conventional annealing, deposition field and e-beam annealing is hence done in this study. CoFe nanostructures were grown on MgO (001) substrates using an RF sputter deposition system. Four samples were synthesized.

1. As-deposited (without annealing and any deposition field) which will be hereafter called as sample A.
2. Sample grown in the presence of a deposition field of 0.2 T normal to the plane of the sample (sample B).
3. Sample subjected to field cooling in which conventional resistive heater based annealing technique is used alongside a 0.2T magnetic field normal to the plane of the sample (sample C).
4. Sample subjected to field cooling in which ERTA is used alongside a 0.2T magnetic field normal to the plane of the sample (sample D).

In the case of sample D, the samples were manually overturned and subjected to ERTA in the presence of an external magnetic field. The annealing was done for time duration of 10 seconds. The expected temperature generated during ERTA is ~700°C.
The samples were initially characterized using AFM in order to determine the effect of magnetic field on surface topography, morphology and roughness. Figure 6.1-a, b show the AFM image and roughness histograms of as-deposited sample. It can be seen from this figure that CoFe grows in the Frank-van der Merwe growth fashion. The RMS roughness of this film was found to be 8.89 nm. It can be seen from the image that distinct layers are formed on the initially grown film. This is indicated inside red circles. There is a localized Frank van der Merwe growth in this case.

![AFM image and roughness histogram of sample A.](image)

**Figure 6.1:** AFM image (a) and the roughness histogram (b) of sample A.

When we compare the AFM image and the roughness histogram of sample A with that of sample B (figure 6.2 a, b), it can be seen that in the latter case, the growth fashion has changed to Volmer Weber type. This can be attributed to the influence of the 0.2T deposition field. As explained in chapter 5, the adatoms acquired a window of mobility before they settle down on the substrate surface within which they are influenced by the deposition field. It can also be seen that there are random agglomerations in this sample. This can be attributed to sputtering
irregularities. It can also be seen that the RMS roughness of sample B is higher than that of sample A which is mainly due to the Volmer Weber island formation.

Figure 6.2: AFM image (a) and the roughness histogram (b) of sample B.

Figure 6.3: AFM image (a) and the roughness histogram (b) of sample C.
When the as deposited sample was subjected to field cooling using resistive heating technique (sample C- figure 6.3-a) a striking difference noticed (when compared to sample B) was that, spherical nanostructures of CoFe were formed and were uniformly arranged over the MgO substrate. It is to be noted that the annealing provided additional mobility for Co and Fe atoms. This along with the influence of the magnetic lines of force passing through the sample promoted uniform Volmer Weber arrangement of the nanostructures. It can also be seen that the average roughness of sample C (24.52) is less than that of sample B. This can be predominantly attributed to the uniform arrangements of the nanostructures in sample C.

![AFM image and roughness histogram](image)

**Figure 6.4:** AFM image (a) and the roughness histogram (b) of sample D.

The sample subjected to ERTA (sample D: figure 6.4-a) had an entirely different morphology when compared to samples B and C. In this case the film had a smooth surface. The low lattice mismatch CoFe/MgO when compared to CoFe/Si as well as rapid annealing is expected to be the main cause behind these phenomena.
Figure 6.5-a1, a2, b1, b2, c1, c2, d1 and d2 shows the normal to the plane and in-plane MOKE hysteresis loop of samples A, B, C and D respectively. The corresponding saturation magnetization and coercivity values are summarized in table 1. It can be seen from the table that the saturation magnetization value of the as deposited sample is lesser than those of samples B, C and D. This can be attributed to the low grain size in the as deposited sample. The increased saturation magnetization of sample B when compared to sample A can be attributed to the formation of bigger grains due to the effect of the deposition field.

**Figure 6.5-a, b:** Normal-to the plane (a1, b1) and in-plane (a2, b2) hysteresis loops of samples A and B respectively.
Figure 6.5-c, d: Normal-to the plane (c1, d1) and in-plane (c2, d2) hysteresis loops of samples C and D.

In the case of samples C and D, the saturation magnetization values are comparable to that of sample B. It is expected that due to the formation of surface oxides the saturation should reduce upon annealing. Even then, the values are similar. It may be postulated that this similarity could be due to the higher crystalline ordering upon conventional annealing as well as ERTA. The increase in grain size upon annealing can contribute to an increase in saturation magnetization.
<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Sample name</th>
<th>Normal-to configuration</th>
<th>In-plane configuration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Coercivity ($H_c$) (Oe)</td>
<td>Saturation Magnetization ($M_s$) (arbitrary units)</td>
</tr>
<tr>
<td>1.</td>
<td>A</td>
<td>0.05</td>
<td>0.5</td>
</tr>
<tr>
<td>2.</td>
<td>B</td>
<td>0.06</td>
<td>1.45</td>
</tr>
<tr>
<td>3.</td>
<td>C</td>
<td>0.05</td>
<td>2.1</td>
</tr>
<tr>
<td>4.</td>
<td>D</td>
<td>0.055</td>
<td>1.95</td>
</tr>
</tbody>
</table>

**Table 6.1:** Comparison of saturation magnetization and coercivity values of samples A, B, C and D.

Another important observation is that in the case of sample A, the easy axis of magnetization is along the plane of the sample, whereas, in the case of samples B, C and D the easy axis of magnetization is along the sample normal. It is typical in the case of as-deposited samples with low thickness values to have their easy axis of magnetization in-plane. When there is deposition field, the nanostructures grow predominantly in the direction of this field. Hence, Oblong spheroid shaped (ellipsoid) structures are observed in sample B (figure 6.2-a) with the major axis along the sample normal. The orientation of the easy axis of magnetization along the sample normal is expected to be governed by the influence of the shape anisotropy of the nanostructures, where the easy axis is expected to be along the major axes of
the oblong spheroid shaped structures. Also in the case of sample C this principle is followed.

In the case of sample D, it can be seen that, as in the case of sample B and C, the easy axis is along the sample normal. Here, the film surface is observed to be extremely smooth (RMS roughness 0.17) when compared to samples A, B and C. Even though a competing shape anisotropy is expected in this sample (with easy axis along the sample plane), the ERTA has played a major role in confining the magnetization along the sample normal. This observation has major impacts in the magnetic recording industry where a low surface roughness along with easy axis oriented along the sample normal are preferred for magnetic tunnel junctions with perpendicular anisotropy (PMTJs) as well as spin injectors.

When we look at the hard axis magnetization loops of samples A-D, it can be seen that there are 2 major steps in the hysteresis loop before they reach saturation. This can be due to the presence of two different magnetic materials in the sample (Co and Fe). It is also to be noted that these steps are not visible in the easy axis loops of all the samples. It may be said that when the samples are magnetized along the easy axis, there is a strong exchange coupling between Co and Fe. Hence, they act at a single magnet in this case. Along the hard axis, due to the competing anisotropy factor, the exchange coupling between Co and Fe atoms are weakened which leads to two distinct saturation magnetization values (two distinct steps in the MOKE loop).