Highly Ordered FePt $L1_0$ Thin Films With Small Grains on RuAl Seed Layers

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We present an experimental approach for obtaining highly ordered $L1_0$ FePt-oxide thin film media with small grains by using a RuAl layer as a grain size defining underlayer. In most previous studies, the FePt grain size was controlled by tuning the oxide volume fraction of the film. By introducing the RuAl grain size defining layer, 6 nm of FePt grains can be obtained at 520°C with 9% SiO_2 in the film. A 5 nm thin barrier layer was introduced between FePt and RuAl to prevent the inter-diffusion between RuAl and FePt. The influence of different barrier layers was studied. With a thin Ag sacrificial layer inserted between the barrier layer and the FePt magnetic layer, a smaller grain size can be achieved, the ordering temperature was lowered, and the (001) texture of FePt was enhanced.

Index Terms—**High anisotropy media, $L1_0$, magnetic recording, perpendicular media.**

I. Introduction

DUE to its high anisotropy field and good environmental stability, FePt ($L1_0$) is a very promising media for achieving ultra-high magnetic recording densities. However, there are several challenges associated with the development of FePt as a perpendicular media. One of the major challenges is to obtain smaller grain size films while maintaining a high order parameter to improve the signal to noise ratio characteristics and to optimize magnetic transition parameters. In most previously studies, the magnetic grain isolation was achieved by means of either O_2 reactive sputtering and/or oxide additions [1], [2]. It has been reported that RuAl with the B2 structure has a small crystallite size [3]. RuAl has a lattice parameter of ~0.3 nm, which means along the face diagonal, the distance between the two atoms is 0.42 nm, which is nearly identical to lattice parameter of MgO. The c-axis normal configuration should be obtained by growing FePt onto (001) RuAl underlayer. This way, the epitaxial and small grain growth from the RuAl underlayer may result in small and uniform grains in the FePt layer and possibly reduce the volume fraction of oxide in the film and the center to center grain size of FePt grains.

II. Experimental Results and Discussion

In this work, FePt media with RuAl grain size defining layers were deposited on Si or glass substrates by RF sputtering. The base pressure was 5×10^{-7} Torr and the argon pressure varied between 5–50 mTorr. The FePt/Oxide layers were fabricated by sputtering from a $Fe_{55}Pt_{45}SiO_2$ composite target onto a heated substrate. The volume fraction of SiO_2 sputtered in the film is about 9%. The substrate temperature was varied from room temperature to 520°C during sputtering. X-ray diffraction and transmission electron microscopy were used to study the texture and microstructure of the films. SEM was used to measure the composition of deposited films with absorption and thickness taken into account. A physical property measurement system (PPMS) was used to investigate the magnetic properties.

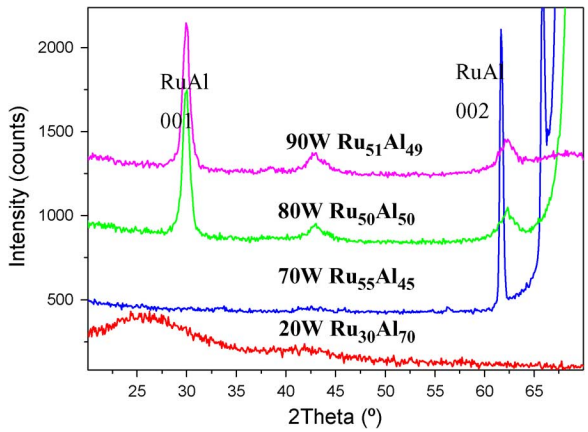

Fig. 1. X-ray diffraction pattern of RuAl(20 nm) deposited at 300°C on Si substrate with different sputtering rate.

A. RuAl Grain Size Defining Layer

The influence of sputtering rate on RuAl texture and structure was studied. It was found that the composition of RuAl changes with increasing of sputtering rate, mainly because of the long distance between the target and substrate in the our sputtering system. Fig. 1 shows the XRD Patterns and compositions of RuAl deposited at 300°C. When $Ru_{50}Al_{50}$ was reached, the RuAl B2 structure with (001) texture was obtained with some (110) variants in the film.

In order to eliminate the RuAl(110) in-plane variants, MgO or Cr with (002) texture was sputtered as an epitaxial underlayer beneath RuAl. Cr(002) texture can be obtained by sputtering Cr at 280°C with 5 mTorr low Ar pressure. By introducing

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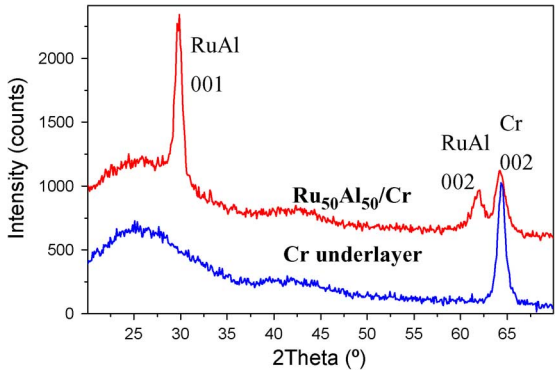

Fig. 2. X-ray diffraction pattern of RuAl deposited at 300°C, 80 W with Cr underlayer.

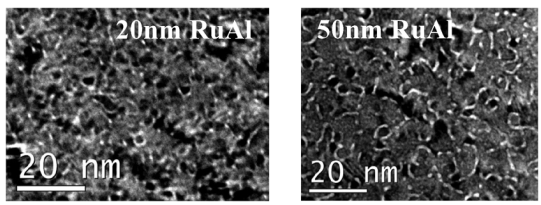

Fig. 3. TEM plan-view images of RuAl layers with different thickness deposited on MgO underlayer.

Cr or MgO underlayer, RuAl(110) variants was eliminated and the texture of RuAl was improved. As shown in Fig. 2, RuAl deposited on Cr (002) underlayer has good (001) texture; no RuAl(110) was observed. Rocking curves indicate that the full width at half maximum for RuAl (001) deposited on MgO and Cr are $\sim 6°$.

As a grain size defining layer, the grain size of RuAl layer is very important. As it can be seen in Fig. 3, the thickness of RuAl layer profoundly affects its grain size. 4 nm RuAl grains can be obtained with 20 nm of RuAl layer deposited on MgO underlayer; whereas a 50 nm of RuAl layer deposited on MgO underlayer has grain size of $\sim$9 nm.

B. Barrier Layer between FePt and RuAl

It was found that when FePt was deposited directly on the RuAl seed layer at elevated temperature, a significant degradation of the FePt properties was induced due to the inter-diffusion of FePt and RuAl layers. A thin barrier layer is needed in between FePt and RuAl layers to prevent the inter-diffusion. After a systematic experimental investigation, a few materials have been found to be effective barriers.

1) MgO Barrier Layer: As shown in Fig. 4, for the MgO/RuAl(300°C)/FePt(550°C) film stack, without barrier layers between FePt and RuAl, only RuAl and MgO peaks were observed, 8 nm of FePt deposited on RuAl layer was diffused into RuAl layer and can not be detected by X-ray diffraction analysis. With a 5 nm MgO barrier layer inserted between the FePt and RuAl layers, the FePt with $L1_0$ structure and perpendicular texture was obtained, showing that the inter-diffusion between FePt and RuAl was effectively prevented.

The TEM cross section images further confirmed the effectiveness of MgO as a barrier layer. As shown in Fig. 5, the

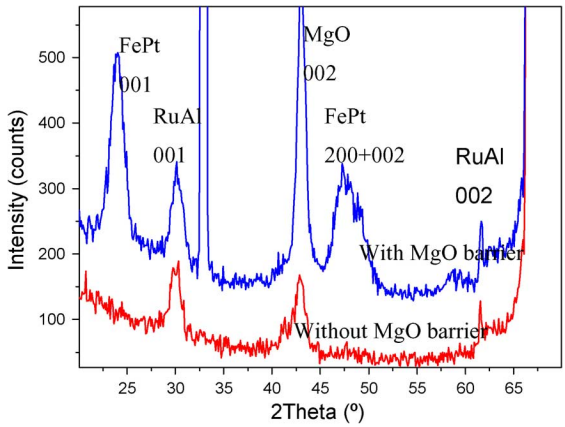

Fig. 4. XRD pattern of FePt films deposited at 550°C on RuAl(300°C)/MgO(20°C) film stack with (top) and without (bottom) 5 nm MgO barrier layer.

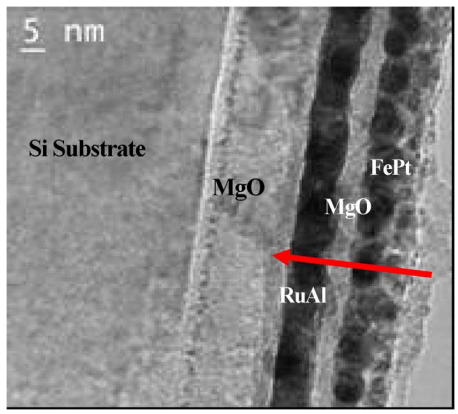

Fig. 5. TEM cross section images of FePt + SiO_2/MgO/RuAl/MgO film stack on Si substrate.

MgO/RuAl/MgO/FePt multilayer structure can be clearly observed. With $\sim$4 nm RuAl grains as a grain size defining layer deposited underneath, polycrystalline MgO barrier layer with small grain size was obtained, followed by the FePt magnetic layer on the top with $\sim$6 nm columnar grains. EDAX composition analysis of the RuAl/MgO/FePt multilayer (along the arrow shown in Fig. 5) cross section demonstrates no significant inter-diffusion of FePt and RuAl layers, as shown in Fig. 6, despite the MgO barrier layer being deposited at elevated temperature (300°C).

The microstructure of the FePt layer was greatly improved by inserting MgO barrier layer, as shown in Fig. 7(b) and (c). A TEM image of FePt films deposited directly on MgO underlayer without RuAl grain size defining layer is shown in Fig. 7(a). With 9% volume fraction of SiO_2 in the film, the FePt grain size is $\sim$ 30 nm. With the same amount of SiO_2 in the film, $\sim$ 6 nm FePt grains were obtained by introducing a 7 nm RuAl grain size defining layer and a 5 nm MgO barrier layer. 6 nm FePt grain size is significantly smaller than the FePt grain size without RuAl grain size defining layer but still is slightly larger than the grain size in RuAl layer. Thicker MgO barrier layers yield even larger FePt grains. Although Mgo is a good barrier to inter-diffusion between the FePt and RuAl layers, it does not copy the grain size of the RuAl. To copy the exact grain size

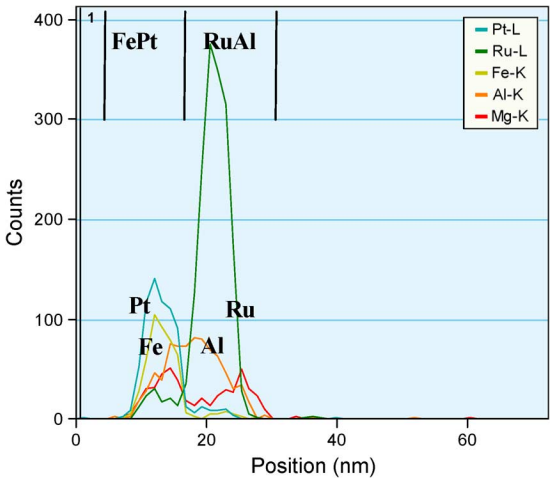

Fig. 6. EDAX line profile of Fe, Pt, Ru, Al, Mg, and Si element along the film stack interface.

from RuAl grain size defining layer, a new metallic like barrier layer will be needed.

2) Pt Barrier Layer: Pt has the FCC structure and a lattice parameter of 0.392 nm, Pt with (002) texture would be a good metallic barrier layer candidate because it not only prevents the inter-diffusion, but also reduces the lattice mismatch between FePt and RuAl. Furthermore, it is a good thermal conductor, which is desirable for heat assisted magnetic recording (HAMR).

However, it is very difficult to obtain Pt with (002) texture. We have tried a wide range of temperature, pressure, sputtering rate, and thickness, and have not been able to obtain Pt (even if deposited on an epitaxial underlayer) with (002) texture.

Pt FCC with (111) texture acting as a barrier layer was studied by X-ray, TEM, and EDAX. As shown in Fig. 8, with the Pt(111) texture underneath, 6 nm of FePt deposited on 7 nm of Pt(111) barrier layer has (111) texture as well. From Fig. 9, the HAADF cross section image shows a clear Si substrate/MgO/RuAl/Pt/FePt multilayer structure, however, some FePt grains are touching the Pt barrier layer; and the inter-connection between RuAl and Pt layer needs to be further investigated.

An EDAX line profile was measured along the cross-section interface of MgO/RuAl/Pt/FePt multilayer structure indicated by an arrow in Fig. 9. As it shown in Fig. 10, the FePt and RuAl layers were well separated by the Pt layer in between. However, Pt appears in the RuAl layer due to the Pt and RuAl inter-diffusion. An amount of Ru also appears in the Pt barrier layer.

3) TiN Barrier Layer: TiN as an extremely hard ceramic material, is often used as a coating to improve the substrate's surface properties because of its excellent corrosion, erosion resistance, and high hardness [4]. TiN thin films also have been used as a "metal barrier" in semiconductor industry to block the inter-diffusion between the metal and silicon and be conductive enough to allow a good electrical connection [4], [5]. Typical stoichiometric TiN has the NaCl crystal structure, with a lattice parameter around 0.425 nm, which is very close to that of MgO. The c-axis normal configuration should be obtained by growing

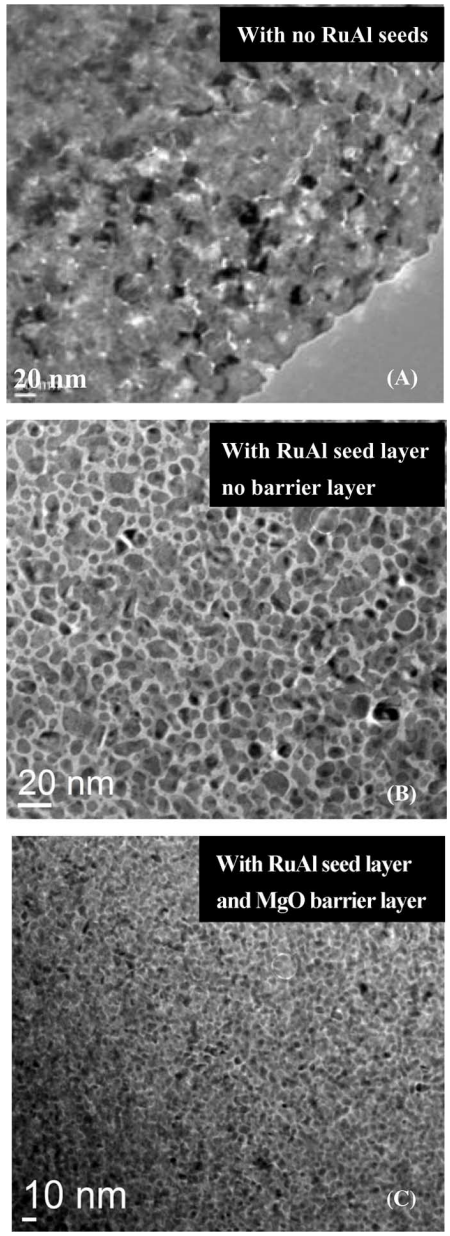

Fig. 7. TEM plan view images of: (a) FePt films with 9% SiO2 deposited on MgO underlayer without RuAl seeds, (b) FePt films with 9% SiO2 in the film deposited on RuAl grain size defining layer without 5 nm MgO barrier layer, (c) FePt films with 9% SiO2 in the film deposited on RuAl grain size defining layer with 5 nm MgO barrier layer in between FePt and RuAl.

FePt onto (002) TiN underlayer. The melting point of TiN is 3563 K, which means it is less likely to sinter into larger grains at FePt order temperature. Due to its lattice matching with FePt and its relative inertness, the "metallic like" TiN seems to be a very good candidate for the barrier layer between RuAl and FePt.

In order to obtain TiN with the NaCl structure, TiN thin films were deposited on a Si substrate from a TiN target with different sputtering powers at different temperatures and Ar pressures. It was found that the TiN composition changes with the sputtering rate. However, TiN with the NaCl structure is stable over a broad range of composition. Fig. 11 shows the X-ray diffraction patterns of 20–25 nm TiN thin films deposited on 1-in Si wafers.

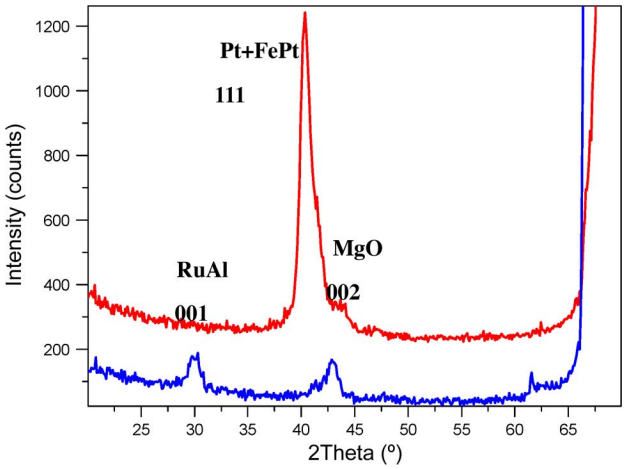

Fig. 8. XRD pattern of FePt films deposited at 550°C on $\mathrm{RuAl(300°C)/MgO(20°C)}$ film stack with (top) and without (bottom) 7 nm Pt barrier layer.

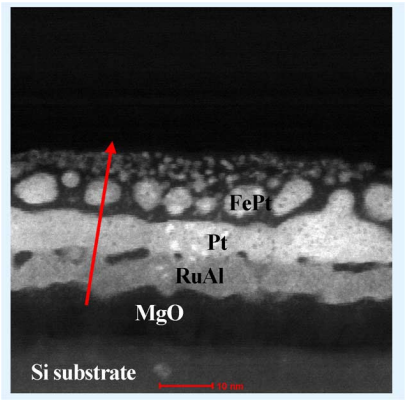

Fig. 9. TEM cross-section image(left) and corresponding HAADF image of $\mathrm{FePt + SiO_2/Pt/RuAl/MgO}$ film stack deposited on Si substrate.

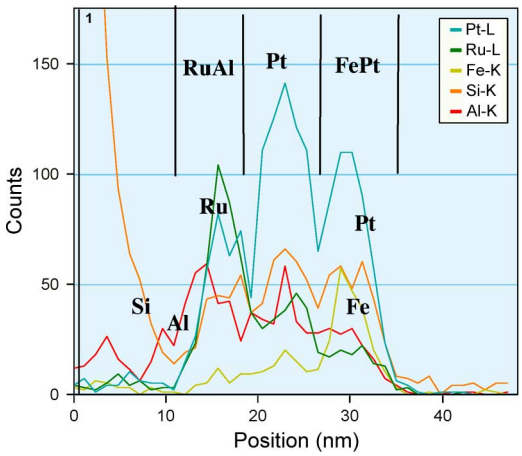

Fig. 10. EDAX line profile of Fe, Pt, Ru, Al, and Si element along the $\mathrm{FePt + SiO_2/Pt/RuAl/MgO}$ film stack interface.

The TiN peak intensities increase with the increasing power due to the change of composition. As can be seen, the TiN thin films

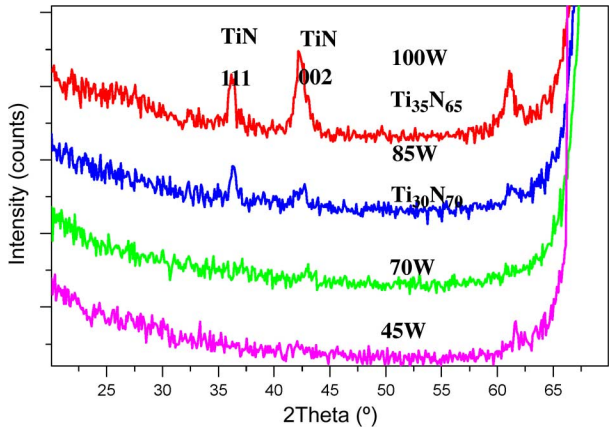

Fig. 11. XRD patterns of TiN thin films sputtered at different power at 450°C.

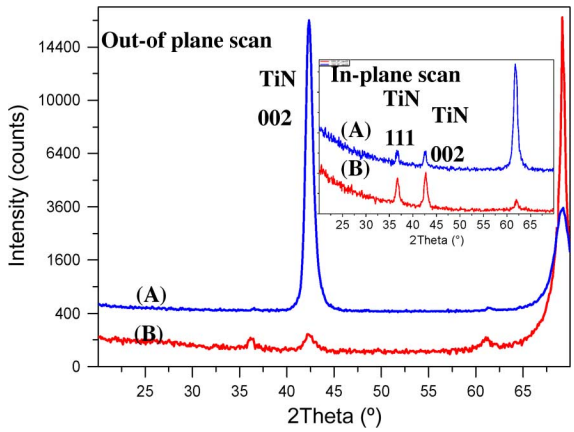

Fig. 12. XRD scans for TiN films sputtered on Si wafer with (A) or without (B) HF cleaning, In-plane scans inserted on the upper right corner.

deposited directly on Si substrates has both the (111) and (002) peaks, indicating a nearly random texture of TiN thin films.

TiN with (002) texture can be obtained by introducing a MgO underlayer with (002) texture or by HF cleaning the Si substrate.

As shown in Fig. 12, by HF etch cleaning the Si wafer, the naturally formed SiO_2 on the surface of Si wafer is cleaned off. TiN deposited on HF cleaned Si substrate at elevated temperature has a strong (002) texture. However, from in-plan XRD scan, the TiN (111) peak still exists, indicating some TiN (111) variants in the film.

By introducing MgO underlayer with (002) texture, TiN(111) variants can be completely eliminated. As shown in Fig. 13, with 17 nm of MgO deposited on Si substrate at room temperature as an underlayer, the TiN deposited on MgO(002) at elevated temperature has excellent (002) texture, no (111) variant is observed from both in-plane and out-of-plane XRD scans. Further experiments found that RuAl with (002) texture can also act as a TiN (002) texture encourager.

After obtaining TiN with the NaCl structure, 5 nm of thin TiN with (002) texture was inserted between FePt and RuAl at 450°C as a barrier layer to block the inter-diffusion. RuAl (002) texture was encouraged by MgO underlayer with (002) texture. The MgO under layer was grown at room temperature. Further

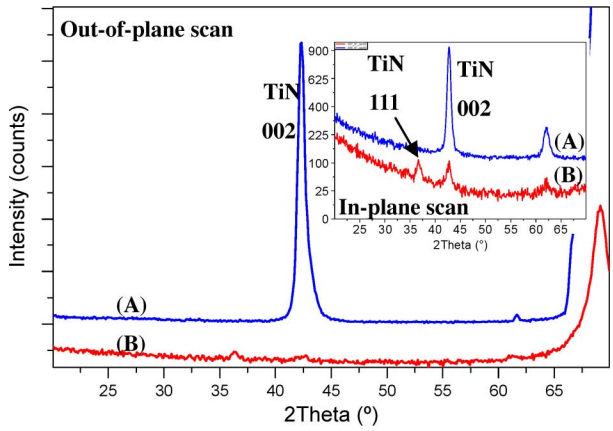

Fig. 13. XRD scans for TiN films sputtered on Si wafer with (A) or without (B) MgO underlayer, In-plane scans inserted on the upper right corner.

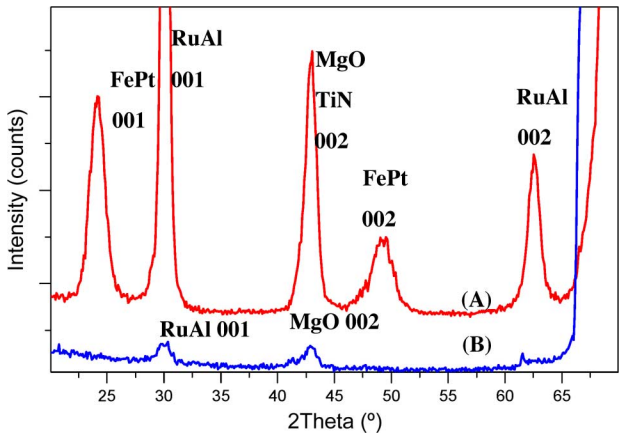

Fig. 14. XRD pattern of FePt films deposited at 550°C on $\mathrm{RuAl(300°C)/MgO(20°C)}$ film stack with (A) and without (B) 5 nm TiN barrier layer.

details involving the growth and epitaxy of MgO on Si are described in [1]. As shown in Fig. 14, TiN as a barrier layer yields excellent perpendicular texture of FePt, no in-plane variant is observed. Deposited at 550°C, the FePt films have an integrated intensity ratio of (001) to (002) close to 3, indicating the order parameter of FePt film is ∼0.84. Both FePt and RuAl peaks have high intensities; the inter-diffusion between FePt and RuAl is completely blocked. Later TEM and EDAX studies further confirmed this information.

As shown in Fig. 15, the high resolution TEM image reveals the lattice orientation of RuAl, TiN, and FePt layer and demonstrates the excellent epitaxial growth of RuAl (001)\\TiN(002)\\FePt(001) layers. It also can be clearly seen that TiN has copied the grain size from RuAl grain size defining layer; however, the FePt magnetic layer did not pick up the grain size from TiN barrier layer. TiN layer shows an average grain size of ∼3–4 nm, while FePt layer has an average grain size of ∼6–7 nm. Further effort is needed to force the FePt layer to follow the grain size from the layer beneath, therefore to produce smaller and more uniform FePt magnetic grains.

Fig. 16 shows the measured hysteresis curve for the film stack shown in Fig. 15 with RuAl seed layer and TiN barrier layer. The hysteresis curve shows a coercivity greater than 10 kOe.

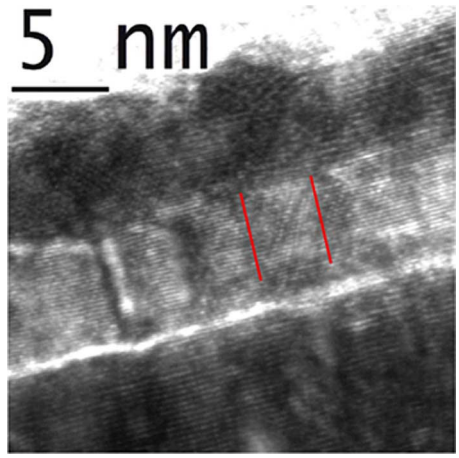

Fig. 15. TEM cross-section image of $\mathrm{FePtSiO_2/TiN/RuAl/MgO}$ film stack on Si substrate, the lattice orientation shows excellent epitaxial growth.

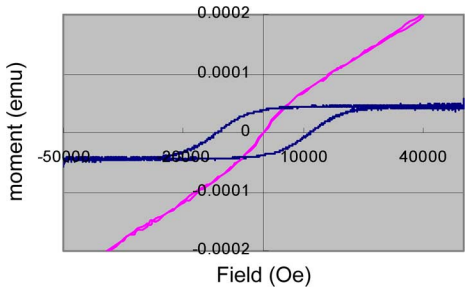

Fig. 16. out-of-plane hysteresis loop for FePt+9%SiO_2 thin films on RuAl seed layer with (loop) or without (line) TiN barrier layer.

The relatively low coercivity compared to high order parameter might be contributed to the small volume fraction of SiO_2 in the magnetic film; the FePt grains are not fully magnetically decoupled.

TiN deposited on HF cleaned Si substrates can also act as an underlayer to replace MgO underlayer in the film structure. However, the epitaxial growth of TiN on single crystal Si substrate yields large grain size of TiN and influences the microstructure of the whole film structure negatively. A Cr seed layer is needed in film structure in order to replace MgO underlayer.

From the experimental results and discussion above, acting as barrier layers, TiN is the most effective compare to MgO and Pt. TiN is also an excellent perpendicular texture "encourager" and grain size "copier."

C. Grain Size Defining Layer Plus Ag Sacrificial Layer Scheme

The Ag sacrificial layer scheme [6], which is a thin layer of Ag deposited in between the barrier layer and FePt magnetic layer, was used to lower the ordering temperature and further improve the order parameter.

After the deposition of MgO\RuAl\barrier film stack, 1.5 to 3 nm of Ag buffer layers with (002) texture were deposited on top of barrier layer before the deposition of FePt magnetic layer. Compared with the film structure has no Ag buffer layer, the intensity ratio of FePt(001) to FePt(002) increases to some extent with each of the three different type of barrier layers. Ag was also deposited directly on top of heated MgO\RuAl\barrier

film stack at elevated temperature without cooling down to room temperature, the texture and order parameter of FePt film did not SiO any degradation.

III. Conclusion

RuAl grain size defining layer was used to reduce the volume fraction of oxide in the film and the center to center grain size of FePt grains. By introducing the RuAl grain size defining layer, 6 nm of FePt grains can be obtained at 550°C with 9% SiO_2 in the film. A 5 nm thin barrier layer was introduced between FePt and RuAl to prevent the inter-diffusion between RuAl and FePt. The influence of different barrier layers was studied. The "metallic like" TiN barrier layer shows great potential due to due to its lattice matching with FePt and its relative inertness. With a thin Ag sacrificial layer inserted between barrier layer and FePt magnetic layer, the ordering temperature can be lowered and the order parameter of FePt is improved.

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