Abstract

Barium hexaferrite (BaFe$_{12}$O$_{19}$ or BaM) films were grown on c-plane sapphire (0001) substrates by pulsed laser deposition (PLD) to evaluate the effects of the laser fluence on their composition, structure, and magnetic properties. Continuum's Surelite pulsed 266nm Nd:YAG laser was employed, and the laser fluence varied systematically between 1 and 5.7 [J/cm$^2$]. Deviations from the stoichiometric transfer between the BaM targets and deposited thin films occurred as the laser fluence changed. The Fe to Ba ratio in the films increased with the laser fluence. The films deposited at the laser fluences below 4 J/cm$^2$ showed undesirable 3-dimensional islands on the surface. Moreover, insufficient laser energy resulted in the deposition of some secondary phases, for example, barium monoferrite (BaFe$_2$O$_4$) and Magnetite (Fe$_3$O$_4$). On the other hand, laser fluences above 5 J/cm$^2$ promoted resputtering and degraded the film quality, structure, and magnetic properties. BaM films deposited at 4.8 J/cm$^2$ - the optimal laser fluence - showed excellent c-axis orientation perpendicular to the film plane with the anisotropy field of 16 kOe and saturation magnetization of 4.39 kOe. These results clearly demonstrate a strong influence of the laser parameters on the PLD-grown hexaferrite films and pave the path for the high-yield production using PLD systems.

Introduction

Bulk hexagonal ferrites are being employed successfully in nonreciprocal microwave devices, such as phase shifters,isolators and circulators, because of their high permeability, non-reciprocal electromagnetic properties, and moderate to low high frequency losses [1]. Compared to the bulk counterparts, ferrite films offer a number of advantages, most importantly a possibility of integration with Microwave/Millimeter-Wave Monolithic Integrated Circuits, and for these reasons attract a lot of attention [2]. Over the past 25 years, several research groups have demonstrated the processing of hexagonal ferrite films with suitable RF properties. Several techniques such as sputtering [3], sol-gel [4], screen printing [5] and liquid phase epitaxy [6] have been investigated. Compared to said techniques, pulsed laser deposition has high deposition rate and is a low-cost method with major advantage that could transfer stoichiometry materials between the target and films if the deposition conditions are optimized. Notwithstanding these advances, this technology remains somewhat immature. Currently, there is a strong push to develop consistent and reliable processing protocols for such films, on large substrates, and demonstrate a practical MMIC prototype.

This goal requires a comprehensive understanding of the influence of the processing parameters
on the RF materials performance. While the influence of the process gas (oxygen) pressure, substrate surface properties, and substrate temperature have been investigated previously [7, 8, 9, and 10], the role of the laser beam parameters has not been explored in details for the ferrite materials. Several studies on the influence of the laser parameters on the structure and composition of alloys and simple metallic oxides have been published previously [11, 12, and 13]. Presented here is a comprehensive study of the impact of such laser parameters on the epitaxial growth of hexaferrite films.

As mentioned earlier, a Nd:YAG laser was deployed, and the laser fluence, beam diameter, and angular distribution varied in a systematic way. The resulting films were characterized using a multitude of techniques, including the VSM, XRD, SEM, EDX, and FMR. It was concluded that the laser parameters have a dominant influence on the film microstructure, composition, and magnetic properties. Other conditions, such as the oxygen pressure, are largely meaningful only for invariable laser settings.

**Pulsed Laser Deposition of Barium Hexaferrites**

PLD inherently allows to deposit a wide variety of materials, including oxides, nitrides, and carbides. Comparing to alternative approaches, PLD offers a number of advantages. Under optimal conditions the stoichiometry of the deposited films may be very close to the target stoichiometry, and the deposition rate can be as high as 100nm/min.

![Figure 1. A schematic illustration of the PLD process.](image)

PLD process can be arbitrary subdivided into 3 stages: (1) ablation of the solid target by the laser, (2) expansion of the plasma plume in the PLD chamber, and (3) plasma condensation on the substrate. A schematic illustration of the PLD process is shown in Figure 1.
The interaction of the laser beam and target primarily results in three sputtering mechanisms: (1) thermal sputtering, (2) electronic sputtering, and (3) hydrodynamic sputtering. The energy of the laser pulses gets immediately absorbed by the target and transformed into thermal, electronic excitation, and hydrodynamic energies. The local temperature on the target surface exposed to the laser pulse may reach up to 5000 K in a few nanoseconds. These high temperatures and heating rates are intended to result in the congruent evaporation of all chemical elements in the target. The resulting plume consists of ions, atoms, and also splashed particulates. There are three mechanisms that result in particulate content in the PLD plume: (1) exfoliation sputtering, (2) surface boiling, and (3) shock-wave recoil pressure expulsion.

The second stage of the PLD process is the plume expansion. The ions and atoms leave the target with energies as high as 100 eV, but eventually thermalize mainly due to collisions with the molecules of the background gas. Therefore, the energy of a given particle depends on its weight and the length of its path. The resulting element-dependent angular distribution of the energies leads to nonuniform deposition and limits the feasible substrate size. Excessive energies of the arriving particles may also cause element-dependent preferential re-sputtering on the film surface.

It’s particularly challenging to grow complex oxide films such as BaM by PLD. The desired outcome can be achieved only under a narrow range of process parameters due to the necessity to transfer several chemical elements with significantly different atomic masses stoichiometrically, and also the complexity of the phase diagram of the Ba-Fe-O system (Figure 2).

![Phase diagram of the Ba-Fe-O system](image)

**Figure 2. Phase diagram of the Ba-Fe-O system.**

**Experimental Section**

All hexaferrite films for these experiments were grown using the Neocera Pioneer 120 PLD...
deposition system. A Surrelite solid state Nd:Yag laser by Continuum was used for the deposition. The laser emitted ten 10ns pulses per second at 1066nm. However, the 266 nm harmonic was used for the deposition itself. Before roughly focusing on the target, the beam passed through a series of lenses, mirrors, and a UV transparent chamber entrance window. A 25mm BaM target was prepared according to the traditional powder metallurgy method, and had a high density of 5.0 g/cm³. The films were grown on single crystalline polished sapphire (0001) substrates supplied by MTI Corporation. The substrates were subsequently cleaned in acetone, alcohol, and DI-water, and then mounted on the heater using silver paint. The substrate to target distance was set at 70mm. The laser beam remained fixed, and the target was continuously rotating and rastering in order to achieve a homogeneous ablation. The PLD chamber was pumped down to 10⁻⁶ Torr base pressure, and then the substrate was heated to 920 °C. Then the chamber was filled with flowing oxygen to maintain the pressure at 200mTorr during the film deposition process. Said parameters have been optimized in previous studies and remained unchanged herein. The laser power in this experiment was intentionally controlled by the voltage output of the power supply. Figure 3 gives the relation between the applied voltage and the laser energy measured directly in the chamber. As noted earlier, the power density at the target was intentionally varied between 1 J/cm² and 5.7 J/cm². A sufficient preablation of the target was achieved by firing the laser for 10 minutes at 10Hz (1000 pulses total). The total deposition time remained fixed at 1 hour (36000 laser pulses) for all of the films discussed herein. After the deposition, the samples were slowly cooled down to the room temperature, and then the chamber was filled with nitrogen. Post-annealing was not employed in this study as it could obscure some features of the as-deposited BaM films.

A number of techniques was employed to analyze the resulting films. The composition and structure of the films were determined by Ultima III advanced X-ray diffraction (XRD) system and Energy-dispersive X-ray spectroscopy (EDX) system. High-resolution sample surface topography images and the film thickness measurement were obtained using a FEI SCIOS scanning electron microscope (SEM) system. The magnetic properties presented here were based on the Microsense Vibrating Sample Magnetometer (VSM) system measurements.

Figure 3. The laser power and fluence as a function of the power supply voltage.
Result and discussion

The composition and crystal orientation of the epitaxial films were determined by the XRD using a Cu-Kα radiation source. All peaks were identified using the standard XRD pattern for the BaM powder. A comparison of the typical XRD patterns for the films grown at 2.5 J/cm², 4 J/cm², and 4.8 J/cm² laser fluences are shown in Figure 4. Significant intensities of the c-axis BaM peaks (0 0 n) reveal the c-textured BaM growth on the substrates at a relatively high laser fluence of 4.8 J/cm². All films deposited at fluences exceeding 4 J/cm² showed the c-axis growth of BaM on sapphire substrates. However, non-stoichiometric secondary phases and randomly orientated BaM were observed in the XRD patterns of the films deposited at lower laser fluences (Figure 4). The secondary phases were determined to be the following: barium monoferrite (BaFe₂O₄), metastable hexagonal ferrite (Ba₂Fe₆O₁₁) and magnetite (Fe₃O₄). These common undesirable phases have been also detected in a number of previous studies [14, 15, 16, and 17]. Unlike the samples deposited at the optimal laser power density (4.8 J/cm²), said films contained randomly oriented BaM grains.

Energy-dispersive X-ray spectroscopy (EDX) was utilized to characterize the chemical composition of the PLD-grown BaM films. Nonstoichiometric material transfer in the pulsed laser deposition system was clearly observed. It was determined that the Fe to Ba ratio in the films increases with the laser fluence. Figure 5 shows the Fe/Ba ratio of the films grown under various deposition conditions. Previous studies reported that a congruent ablation happened above some certain laser fluence threshold [18 and 19]. In this study, the Fe/Ba ratio increased continuously and there was no obvious critical point. It can be attributed to the much lower energy (as compared to excimer lasers) of the solid state laser. Several mechanisms may be considered in order to explain the non-stoichiometric transfer observed during the PLD depositions. 1. Preferential ablation of certain chemical elements from the surface of the target. 2. Element-dependent thermalization/scattering of the plasma plume. 3. Preferential respattering of certain elements on the surface of the film. As noted earlier, the Fe/Ba ratio dramatically increased with the laser fluence in this study.

Let’s first consider the preferential ablation of certain chemical elements from the surface of the target. This mechanism should not play a major role here as the lowest laser fluence in this study was sufficiently high to achieve congruent ablation.

The element-dependent thermalization of the plasma plume, on the other hand, must be the primary mechanism that determines the Fe/Ba ratio in the PLD-grown films. Given that the atomic mass of Ba exceeds the one of Fe by more than a factor of two, Fe atoms and ions lose their energy and scatter much faster due to collisions with the background oxygen gas. Therefore if the laser fluence is insufficient, fewer Fe atoms reach the substrate, resulting in Ba-rich films.

As the energy of the ions and atoms reaching the substrate increases with the laser fluence, the preferential re-sputtering on the film surface becomes more likely [20,21]. Let’s consider a
sputtering process with two independent target ((1) Ba and (2) Fe). The ratio between the two elements can be described by the following equation

\[
\frac{Y_1}{Y_2} = \frac{C_1}{C_2} \left( \frac{M_2}{M_1} \right)^{2m} \left( \frac{U_2}{U_1} \right)^{1-2m},
\]

where \(C\) is the concentration, \(M\) is the mass and \(U\) is the cohesive energy, and \(m\) is the mass effect coefficient (roughly 0.05~0.1 [22,23]). The ratio \(Y_1/Y_2\) then gives the potential of re-sputtering for these two elements. Considering that the cohesive energies for barium and Iron are 1.9 eV and 4.28 eV respectively, the equation predicts preferential re-sputtering of barium and Fe-rich films at higher laser fluences. Therefore, the laser output should be controlled precisely in order to keep the Fe/Ba ratio in agreement with the stoichiometry of BaFe\(_{12}\)O\(_{19}\).

Figure 4. XRD Spectra of BaM films on sapphire substrates deposited at the following laser fluences: (a) 2.5 J/cm\(^2\), (b) 4 J/cm\(^2\), (c) 4.8 J/cm\(^2\).
High resolution surface topography and cross-section images of the samples shown in Figures 6 were obtained using a FEI SCIOS SEM. The figures demonstrate the evolution of the microstructures and morphologies of the films as functions of the laser fluence. The samples deposited at higher laser fluences had relatively smooth surfaces. Rough surfaces were typical for the samples deposited at lower power conditions. The films grown at relatively low laser fluences of 2.5J/cm² and 4J/cm² had small grains, velar grain boundaries, and rough surfaces. This indicates that the three-dimensional (3D) island growth mode dominated the process. As depicted in Figure 6 (d), the smoothest film surfaces were achieved at 4.8J/cm². This implies that the films mostly experienced the two-dimensional growth mode of complete monolayers. Generally, the growth mode is primarily determined by the dynamics of the surface energies and the plasma energy. Given that the process gas pressure (oxygen) and the substrate temperature were fixed in this study, only the plume plasma energy varied as a function of the laser fluence. Figure 6 (a) shows the surface topography of an area close to the edge of the film deposited at 4.8J/cm². Uniform hexagonal grains were exposed clearly, and the crystal (grain) size was measured to be around 0.4~0.5 μm, while the edges of the hexagonal grains were almost perfectly aligned. When the laser fluence was too high (5.7J/cm², Figure 6 (e)), the film surface once again became rougher and magnetic coercivity of the film increased. The grain outgrowth induces lattice distortions, grain cracks, and defects, and eventually affects magnetic properties of the films. Some elongated grain edges can be seen on the surfaces of the highly textured films deposited at laser fluences in the range between 4.8J/cm² and 5.7J/cm². Similar BaM film surfaces have been
also reported in earlier studies [24,25]. These edges belong to c-axis oriented grains on the surface. Said grains lay in the film plane and indicate that the film growth underneath also preferentially happened along the crystallographic C plane. Despite some imperfections on the surfaces of the films deposited at higher fluences, no cracks were observed in these films.

Focused ion beam milling (Ga+ primary ion beam) was employed to measure the thickness and observe the cross-sections of the BaM films (Figure 6 (f)). It was determined that the deposition rate strongly correlated with the laser fluence, given that the spot size remained unchanged. The rate grew from 0.06 μm/hour at 2.5J/cm² to 0.3 μm/hour at 4J/cm² and to 0.4 μm/hour at 4.8J/cm².
Figure 6. SEM images of BaM films. Surface morphology of a film deposited at (a) 4.8 J/cm$^2$, (b) 2.5 J/cm$^2$, (c) 4 J/cm$^2$, (d) 4.8 J/cm$^2$, and (e) 5.7 J/cm$^2$. (f) SEM cross-section image of a BaM film deposited at 4.8 J/cm$^2$ laser fluence.
The plume energies exert a remarkable influence on the PLD film growth. The following experiment was designed to evaluate the spatial distribution of the film parameters on the substrate surface. A 10mm square sapphire substrate was intentionally mounted with an offset with respect to the plume. The resulting film on this substrate had two distinct areas with different appearances (Figure 7). The side of the film that was closer to the center of the plume looked like a high quality BaM film grown at the optimal laser fluence – the fluence used in this experiment. The outer side of the film looked similar to the films grown at insufficient laser fluences. Both EDX and SEM were used to characterize these two areas. The Fe/Ba ratio gradually decreased towards the center of the plume, from 15.8 to 12.7. This observation revealed a non-stoichiometric angular distribution of chemical elements in the plume, a phenomenon that has been also observed in other studies [18, 26]. The Fe-rich area closer to the outer edge of the plume is consistent with the fact that the lighter Fe ions and atoms should have a broader angular distribution than heavy Ba atoms. Figure 7 shows that the surface morphologies of the two distinct areas of the film differ dramatically. The area closer to the center of the plume appears to have a high density and high degree of crystalline orientation – similar to what was observed in other high quality BaM films. In contrast, the outer edge has a granular structure and noticeable porosity.

Finally, the static and dynamic magnetic properties of the PLD films were measured using the VSM and FMR. In plane and out of plane hysteresis loops of the films deposited at different laser fluences are shown in Figure 8. The applied dc magnetic field varied form -20 kOe to +20 kOe in all of these measurements. In agreement with the XRD measurements, the static magnetic properties of the film deposited at a low fluence of 2.5J/cm² are nearly isotropic (Figure 8 (a)). Non-stoichiometric deposition at lower fluences results in secondary phases that must disrupt the
epitaxial growth of BaM. Figure 8 (b) clearly shows a significant difference between the in- and out-of-plane hysteresis loops and indicates a significant degree of the crystallographic alignment in the film grown at 4.8 J/cm². The saturation magnetization of said film equaled 4.39 kG. This value is very close to the theoretical maximum saturation magnetization for bulk BaM - 4.46 kG. The magnetic anisotropy field was evaluated using the singular point detection (SPD) method and equaled 16 kOe - again, just slightly lower than the 17 kOe value that has been previously reported for a highly textured bulk BaM sample. These lower values may be attributed to the substrate-induced strain and surface imperfections. It’s worth noting that the saturation magnetization and magnetic anisotropy can be further increased by post-annealing.

Compared to the film deposited at 4.8 J/cm², the film deposited at 5.7 J/cm² once again demonstrated slightly different static magnetic properties (Figure 8 (c)). The coercivity increased from 920 Oe to 1500 Oe and saturation magnetization decreased from 4.36 kG to 4.02 kG. The result is in agreement with other measurements for this sample. This film was Fe-rich (Fe/Ba ≈ 14) and therefore contained secondary phases. There was also a number of defects on the surface.
Figure 8. In plane and out of plane VSM loops of BaM films on (0001) sapphire substrates deposited at (a) 2.5 J/cm$^2$, (b) 4.8 J/cm$^2$, and (c) 5.7 J/cm$^2$. 
Dynamic magnetic properties of the films were measured using a coplanar waveguide reflection resonator [27,28]. The actual FMR measurement setup and FMR data are shown in Figure 9.

Figure 9. The FMR measurement setup and results.

In agreement with all other characterization results, the best (the lowest) FMR linewidth of 490 Oe was measured for the film deposited at 4.8 J/cm². As with the coercivity, this value can be further decreased by employing post annealing in oxygen. This step is intended to reduce the strain, eliminate some defects, and increase the resistivity of most oxide films. The films deposited at sub-optimal laser fluences demonstrated much broader linewidths. Additionally, the FMR center frequency was measured as a function of the applied dc magnetic field. The relationship between these values provided an alternative way to evaluate the anisotropy and saturation magnetization using the following equation: 

\[ f_{FMR} = \gamma (H_{dc} + H_a - (4\pi M_S \cdot N)) \] [29].

**Conclusion**

In summary, this study demonstrated that high quality epitaxial hexaferrite films can be grown by pulsed laser deposition only under a narrow range of process parameters. The laser fluence has a significant influence on the stoichiometry, phase, crystallographic texture, and magnetic properties of the deposited films. Relatively low laser fluences are not able to transfer hexaferrites stoichiometrically between the target and substrate. On the other hand, excessive laser fluences increase the likelihood of re-sputtering on the film surface. The Fe/Ba ratio in the films significantly increases with the laser fluence, but must remain fixed at the correct value in order to produce pure phase BaM. Therefore, nonoptimal laser fluences result in secondary phases, poor epitaxy, and defects. Eventually, nonoptimal laser fluences adversely affect static and dynamic magnetic properties of the films. Finally, the optimal laser fluence can only be determined for a fixed set of other PLD process parameters.

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