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14. ABSTRACT

Due to the nature of both grants FA9550-10-1-0199, and FA 9550-10-1-0198 as one is support to the other; the instrumentation grant DURIP FA9550-10-1-0199 will be reported as part of the research grant FA 9550-10-1-0198 and whenever specific details related to one , then it will be discussed under the specific grant and will be referred to the grant number. This will decrease any redundancy. Both projects were built on the successful funding of the PLD chamber; grant number FA9550-08-1-0363. The goal of this proposed project is to continue building the pulse laser deposition system by purchasing the Reflected High Energy Deflection (RHEED), Atomic Force Microscope (AFM), Target manipulator, and the Substrate heater which are essential integrated parts of the PLD system. These instrumentation enhanced and strengthen the research enterprise and research-related education capabilities and interdisciplinary activities among the Physics, Engineering, Chemistry, and Mathematics departments at Dillard University. It supported to train of five African American and prepares them for entering graduate schools in the area of Non materials and technology of thin film fabrication.

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Final Report

OPTICAL CHARACTERIZATION OF PULS LASER DEPOSITION OF THIN FILM OF HARD MATERIALS USING RHEED AND AFM TECHNIQUES

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1. Summery

Due to the nature of both grants FA9550-10-1-0199, and FA 9550-10-1-0198 as one is support to the other; the instrumentation grant DURIP FA9550-10-1-0199 will be reported as part of the research grant FA 9550-10-1-0198 and whenever specific details related to one , then it will be discussed under the specific grant and will be referred to the grant number. This will decrease any redundancy. Both projects were built on the successful funding of the PLD chamber; grant number FA9550-08-1-0363. The goal of this proposed project is to continue building the pulse laser deposition system by purchasing the Reflected High Energy Deflection (RHEED) , Atomic Force Microscope (AFM), Target manipulator, and the Substrate heater which are essential integrated parts of the PLD system. These instrumentation enhanced and strengthen the research enterprise and research-related education capabilities and interdisciplinary activities among the Physics, Engineering, Chemistry, and Mathematics departments at Dillard University. It supported to train of five African American and prepares them for entering graduate schools in the area of Non materials and technology of thin film fabrication.

We have been studying the parameters which effects the laser ablation of hard materials (Si, Ga, Gi, BaTiO3:Fe, and BaFeO3:Mn until now) to gain more insight understanding to the surface processes that occur during the thin film growth by the PLD of hard materials. One of the most important developments in this research is our innovative modification of the ablation process in fabricating a new sample holder which can hold two different targets with X-Y knobs for each and use two laser beams (same wavelength and power or different based on the ablation energy requirement of each target). This enables the interaction/overlap of the two target plumes and enables the doping the thin film in situ with transition metal ions, as we fabricated both thin films of $BaFeO_3$:Mn, and $BaTiO_3$:Fe.

One of the main interests in surface diffusion times, is the time it takes for a deposited atom to reach its nucleation site in hard materials, explore the temperature effect on the film growth and using RHEED and AFM to investigate the diffusion processes, during growth of complex oxides and hard materials will be studied using time resolved RHEED and AFM since, the PLD is a deposition technique in which deposition and film growth are intrinsically separated in time.

Dillard University opened the New Science and Professional building which is a "LEED certified building" and costs over $52M to a commitment to science, research and education. It took about one semester to move the new equipment to the new state-of-the-art Nano Materials Laser ablation laboratory which was designed as a place for expansion to accommodate more research equipment like X-Ray Machine, TEM and other needed equipment to expand this research enterprise program which was initiated by the support and the trust of Dr. Howard Schlossberg the program officer and Mr. Ed Lee the program Director at the AFOSR and we have delivered in a short period of time and in a process of establishing a Master Degree program in the area of Lasers, surface coating using thin film materials of hard materials. This will train and support more minority students for years and years to come.

Introduction:

Laser ablation provides a mechanism whereby non-volatile material can be ejected into the gas phase. As a novel way of making coatings and interesting thin films, PLD has been in use for a number of years and has found application in areas ranging from the growth of high temperature superconductors to diamond thin-film generation. PLD is initiated by laser ablation, which is essentially evaporation of a material by a high-powered laser. Subsequently, ejected material is deposited at a desired substrate. With regard to hard coatings such as SiC or TiC, PLD is being used to coat micro-electromechanical systems (MEMS) devices, which are becoming increasing important in Air Force related activities. Once put into space, it is clear that wear on components constitutes a serious maintenance issue, and minimizing wear is desirable. In terms of applying a hard coating, the advantages of PLD are numerous. The most important ones include ease of deposition, possibility of obtaining crystalline films with good adhesion and high optical response at low temperatures (even at room temperature), and the ability to reproduce the stoichiometry of the target in the film, including that of multi-component targets.

The new innovative target holder will enable to produce more a strong thin film fabricated as an alloy of two or more different ablated targets which can be revolutionary way for double pulse laser deposition /ablation of Nano-alloy- composite thin films for coating of MEMES in harsh environment to minimize the wear and tear.

The goals of the proposed experiments are as follows:

Experiment1: Study the effect of the magnetic field orientation of the quality of the film growth:

In order to unravel the exact role of the magnetic field in the deposition, three basic field configurations need to be investigated. A brief outline is presented below.

(i) Orthogonal Weak Field.

The field is orthogonal to the direction of movement of ions and parallel to the surfaces of substrate and target. The ions describe just one quarter circle in the field, reaching the substrate around the shield. This method is dependent on the ion mass and the field strength.

(ii) Longitudinal Strong Field

Field lines come out of the target and lead in a curve to the surface of the substrate. As the field is strong, the ions spiral around the lines and find the substrate independent of their ion mass and the specific field strength. (of course, the field must be low enough that lightly charged particles still miss the substrate).

(iii) Inhomogeneous Field by Permanent Magnet

This is the configuration empirically derived at this point. The field has components of both configurations (a) and (b), but it will certainly attract leading electrons to the grounded magnetic poles, creating a momentary positive space charge. The field gradients provide

the appropriate field strength for each ion type to be deflected to the substrate. The effects are not well understood and need further investigation.

Chapter 1

Preparation of $BaTiO_3$ thin Films by Double-pulse-lasers Deposition

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ABSTRACT

Epitaxial $BaTiO_3$ films were deposited by using innovative double-pulse--lasers Deposition (DPLD) technique. The modified DPLD enables the ablation of batio3 thin film to be doped with Fe and Mn in situ during the ablation process. Different substrates were used like MgO, $SrTiO_3$, and Si to decrease the misfit with $BaTiO_3$. The films deposited at different substrate-temperature in the range of 200◦C-800◦C. The thin film surface morphology was investigated using both Atomic Force Microscope (AFM) and Reflection high Energy Electron Diffraction (RHEED). The substrate temperature and the oxygen gas pressure affected the morphology of the thin film and in turn the optical response of the thin film. The AFM reviled a new harmonic grating structure of the thin film due to the DPLD ablation. The thin film shows an anomalous response to the HeNe laser as a self-modulation and grating formation pattern were observed.

1. ***INTRODUCTION***:

Since the work of Dijkkamp et al [1]., the PLD technique has been intensively used for many kinds of oxides, nitrides, carbides, and also for preparing metallic systems and intensely used to fabricate thin films of optical materials, including: $BaTiO_3$ ZnO as a piezo-electric, piezo-optical, transparent conductor ,TiO2 rare-earth-doped phosphate glasses [2-10], but the thin film has many issues which still need an answer to enhance the process and make it more attractive to be used. The technique was used by Smith and Turner [2] in 1965 for the preparation of semiconductors and dielectric thin films and was established due to the work of Dijkkamp and coworkers [1] on high temperature superconductors in 1987. Their work showed the stoichiometry transfer between target and deposited film, high deposition rates of about 0.1 nm per pulse and the occurrence of droplets on the substrate surface [3]. In the search of enhancing the technique, this paper will add another piece to the of technique to make the PLD more attractive especially for doubly doped thin film and make the thin film more attractive to be used nonlinear optical field. In this paper we have developed and built an innovative target holder to facilitate deposition/ablation in situ of two materials at the same time using two laser beams. The PLD research is in the field of hard coatings, such as boron nitride [15], carbon nitride [16], and diamond-like carbon [17]. In these cases, the energetic nature of the depositing vapor appears to be responsible for enabling the growth of the desired hard phases, which are not thermodynamically stable under the growth conditions of conventional techniques [18,19]. As we demonstrated on this paper, the method can be used to fabricate doubly doped transition metal Fe/Mn ions $BaTiO_3$ thin film or any doped thin film.

1. EXPERIMENTAL

1.1 Pulsed laser ablation/deposition chamber

The PLAD system is consists of two 6 inches cubes connected by a bellows assembly. The ablation chamber is fixed while the deposition cube is mounted on a rail assembly that is controlled by computer. This allows the selection of the targets during the ablation process, and also, the selection of dopant materials. This setup allows for a 0.15 m travel range in the distance between the cubes that can be varied quite quickly. On either side of the cube are view ports with 4 in. quartz windows allowing for the introduction of the laser ablation beam. The windows are also convenient for the visual inspection and alignment of the ablation targets.). A Reflected High Energy Election Diffraction system (RHEED aligned axially with respect to the ablation targets, for in situ monitoring of the film growth. All six samples rotate simultaneously at the same speed. For ion extraction, a grid assembly has a 0.625 in. hole right in front of the 0.5 in. sample stub. By adjusting the conditions such as ablation laser power and fluence, the neutral-to-ion ratio in the ablation plume can be controlled.

The new to this design is the double target holder. Due to the needs to have thin film growth and in situ doped with other materials like Fe, Mn and Cr, we have designed a new target holder which allows two laser beams to ablate two different materials at the same time as shown in Figure 1. This is called Double Laser pulse deposition /Ablation technique (DLPD or DLPA technique). The target as shown in Figure 1, split into two mechanical stages 0.5" each. Both stages can be set independently to different angle which allows the pulses of the two target to cross each other's and interact chemically to produce a different substance, or to allow any dopant to settle into the thin film lattice. This revolutionary target holder (patent in progress) will allow the PLD to go beyond the constrains of the process in doping the thin film in situ with transition metal ions .

2. Results and discussions:

2.1 *Double targets holder and plumes interaction:*

As shown in Figure 2, the two plumes are formed using the Nd:YAG laser with wavelength 532nm. Two laser beams were applied in situ to two targets and the energy and fluence of the lasers can be adjusted to accommodate the type of target or experiment in hand. The target holder can be adjusted by four knobs, two (X and Y) each for each target. This will adjust the two plumes to interact as shown in Figure 3.

2. 2 *Optical surface Morphology:*

The surface roughness and the homogeneity of the deposited thin film surfaces of both $BaTiO_3$:Fe and $BaTiO_3$:Mn analyzed using Atomic Force Microscope as shown in Figure 4-16 and RHEED as shown in Figure 17. The Figure shows that the DPLD thin film is relatively with a periodic surface structure as grating formation. The preliminary explanation of such phenomenon is due to the double plumes interaction. The time of the ablation of the doped materials controlled to have 1% or less doped materials in the thin film. The substrate temperature has a major effect of the

structure of the thin film as shown in Figures 4-16. The more higher temperature the more periodicity on the thin film as shown in Figure 11 . The roughness of the surface was measured at 50-70 nm for films of 190-270 nm thick. RHEED patterns of the $BaTiO_3$ thin film in 20 Pa oxygen at room temperature and at 760 C^o are shown in Figure 10. The RHEED reviled that the DPLD thin film is homogenous during the ablation process as shown in Figure 10.

3. *Nonlinear optical response for $BaTiO_3$:Fe:*

The thin film was tested by applying the HeNe laser with wavelength 625 nm on the far field through beam expander and collimator where the beam is project on a screen 4.5m from the thin film. The different films were fabricated at different temperatures which in turn produced grating space ranges from 2 to 5 nm. The diffraction pattern was similar to double slit experiment and the interference pattern can be easily observed as shown in Figure 18. For other thin films where the grating period increased, double interference pattern is observed as shown in Figure 19-22. In addition, when the period of the oscillation increased to 20 nm a self-modulation pattern is observed as shown in Figure 19. As a preliminary interpretation of this phenomenon, this can be explained by the amount of the plumes overlapped with each other's. Three patterns can be obtained from the ablation. One with homogenous doping of Fe/Mn into the $BaTiO_3$ which will be "C" as shown in Figure 3. Another two patterns will be formed one with less Fe/Mn and more $BaTiO_3$ and the third with More Fe/Mn and Less $BaTiO_3$. These thin films are different in the doping percentage which range from 0.5% to 1.5% in the same thin film. On the other hand, the temperature of the substrate and the amount of Oxygen in the chamber play an integral part of the formation of these grating and the nonlinear optical response of the thin film. As shown by Cermoe et. al., and confirmed by our experiment as shown in Figure 23, that the intrinsic stress increased by increasing the temperature of the substrate.

4. CONCLUSIONS

A new DPLD target holder design was fabricated which enabled us to ablate different targets using two laser beams .The techniques fabricated a thin film which has anomalous structure as observed by AFM. Also, we demonstrated that the interaction of two different plumes made it possible to have three different thin films with different optical response .This stress produces fringes structure when two plumes interact with one another. So, thermal stress and intrinsic stresses play a major role in formation of the grating structure in $BaTiO_3$.

Given preliminary nature of experimental results of double ablation and the new observed periodic structure of the thin film and the new optical response of the film, many questions were raised to catalog the conditions of the experiments and investigate the effect of the Oxygen environment in the chamber and the temperature of the substrate. Possible mechanisms that contribute to this effect can be due to temperature-induced bending of thin film through thermal stress may cause angular splitting and resulted in grating formation. Reflection grating recording will be used to investigate some of the experimental results.

ACKNOWLEDGMENTS

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his support and guidance through this project. This fund made it possible to establish the PLD laser deposition laboratory and the continuation of this work will support many minority students and prepare them for graduate schools in addition to those nine students who already trained on using the instrumentation .

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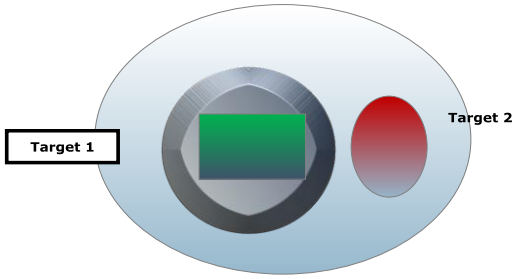


Figure 1: Schematic diagram of double target holder. Target 1 is off axes of Target 2 and can be controlled by two knobs to move the platform in two dimensions to adjust the plumes of the two targets.

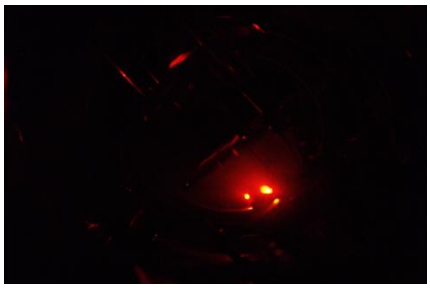


Figure 2: Picture of the double plume as seen from the top of the chamber.

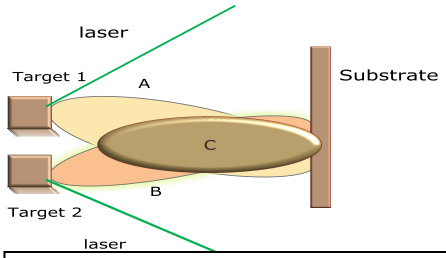


Figure 3: Schematic diagram of the overlap of the 2 plumes.

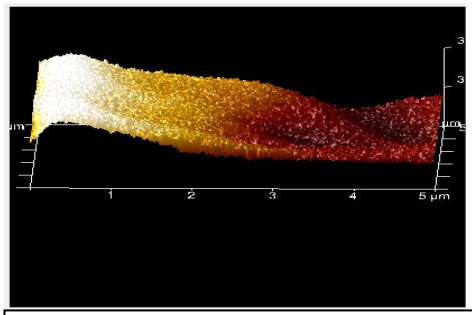


Figure 4: AFM of BaTiO3 thin film, substrate Temperature =100ºC.

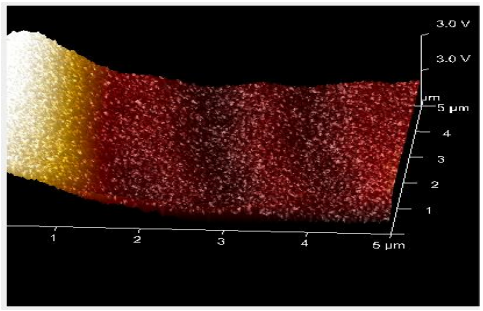


Figure 5: AFM of BaTiO3 thin film, substrate Temperature =200ºC.

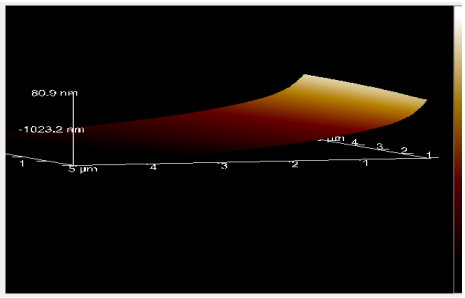


Figure 6: AFM of BaTiO3 thin film, substrate Temperature =400ºC.

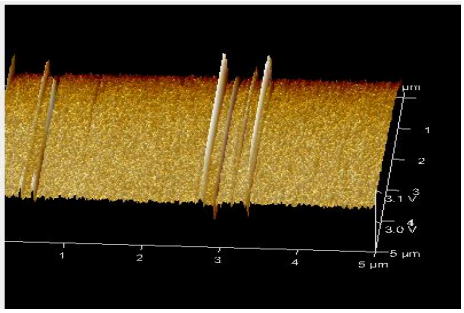


Figure 7: AFM of BaTiO3 thin film, substrate Temperature =600ºC.

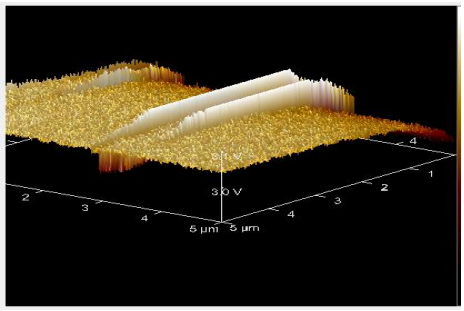


Figure 8: AFM of BaTiO3:Fe:Mn, substrate Temperature =200ºC (D= 55mm).

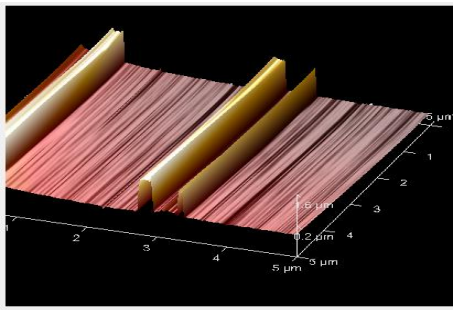


Figure 9: AFM of BaTiO3:Fe:Mn, substrate Temperature =400ºC (D= 55mm).

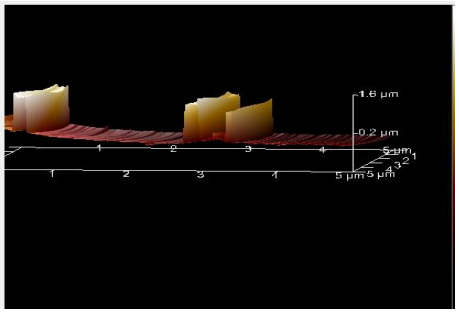


Figure 10: AFM BaTiO3:Fe:Mn, substrate Temperature =600ºC (D=55mm).

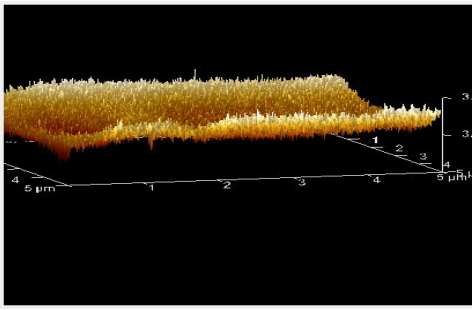


Figure 11: AFM of BaTiO3:Fe:Mn, substrate Temperature =800ºC (D = 55 mm).

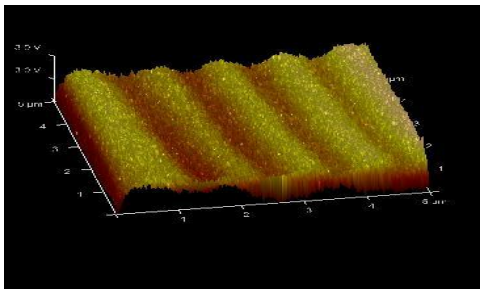


Figure 12: AFM of BTO:Fe with less B, substrate Temperature =800ºC (D=50 mm)

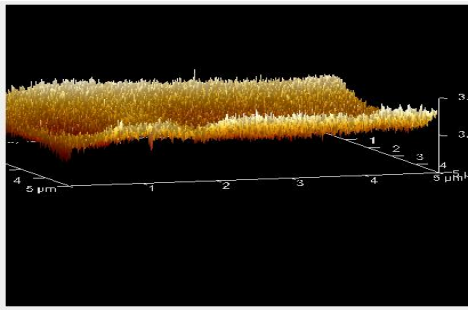


Figure 13: AFM of BaTiO3 thin film, substrate Temperature=100ºC.

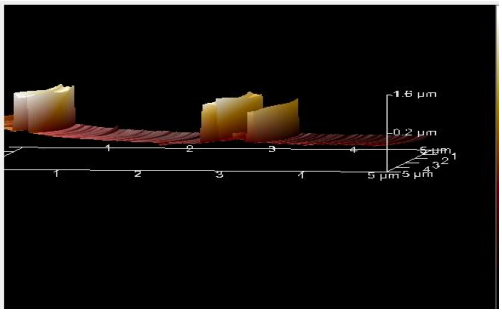


Figure 14: AFM of BaTiO3 thin film, substrate Temperature=100ºC.

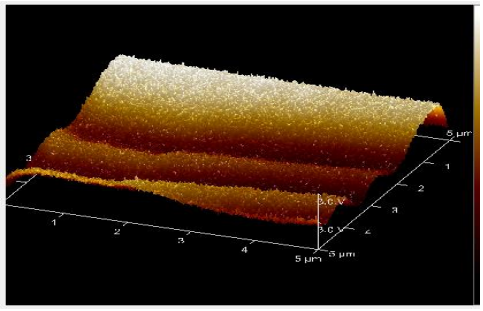


Figure 15: AFM BaFeO3:Mn, substrate Temperature =600ºC (D = 45mm).

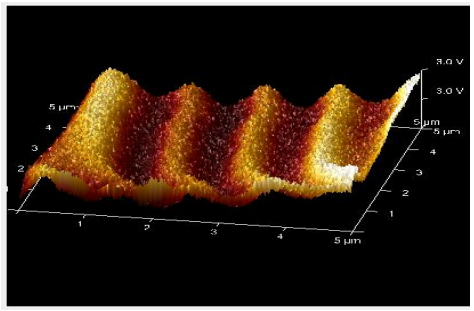


Figure 16: AFM BaFeO3:Mn, substrate Temperature =400ºC (D = 45mm).

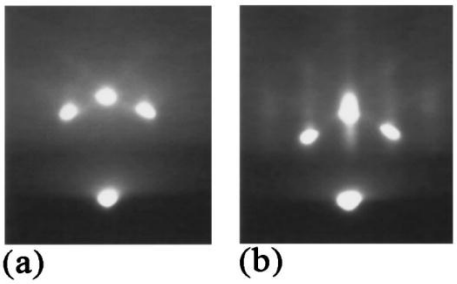


Figure 17: RHEED patterns of the BaTiO3 thin film in 15 Pa oxygen **A.** at room temperature and (**B**) a t600ºC.

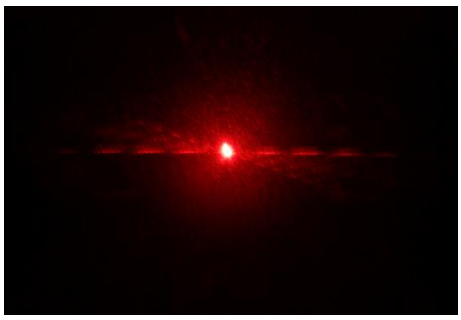


Figure 18: BATiO3:Fe film (A&B)

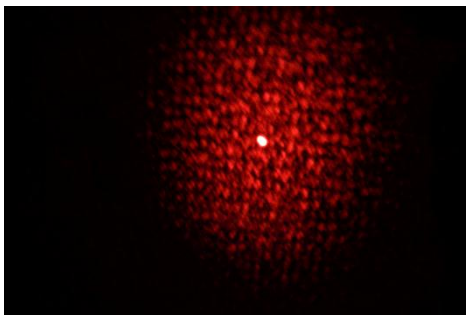


Figure 19: BaTiO3:Mn thin film (A with less B)

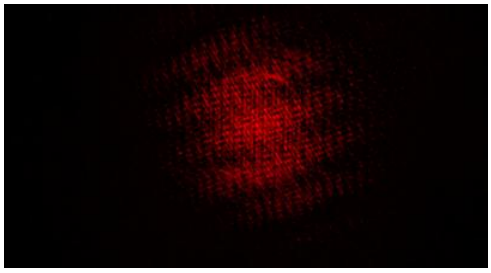


Figure 20: BaTiO3 :Fe(thin film A)

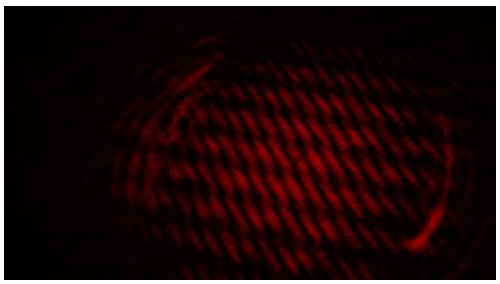


Figure 21: BTO:Fe T=300ºC.

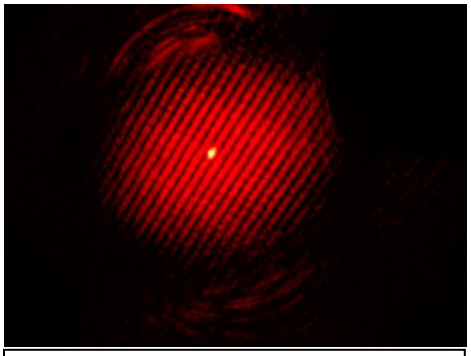


Figure 22: BaTiO3:Fe (Thin film B), T=600ºC.

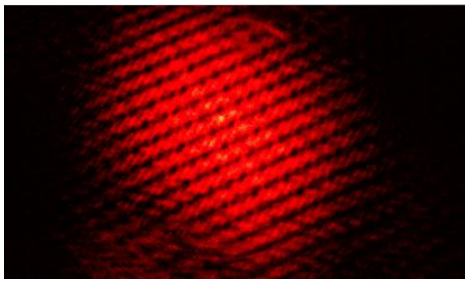
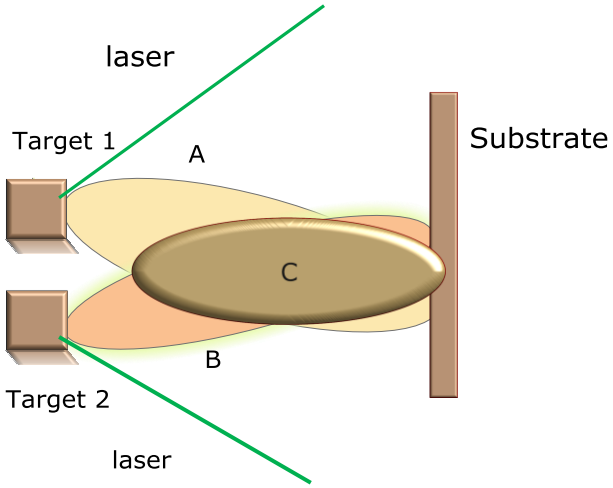
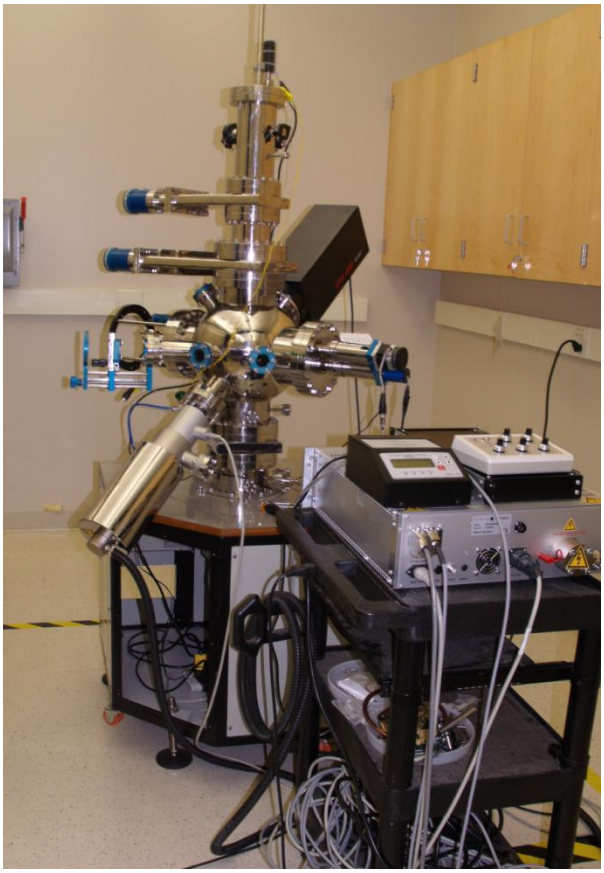
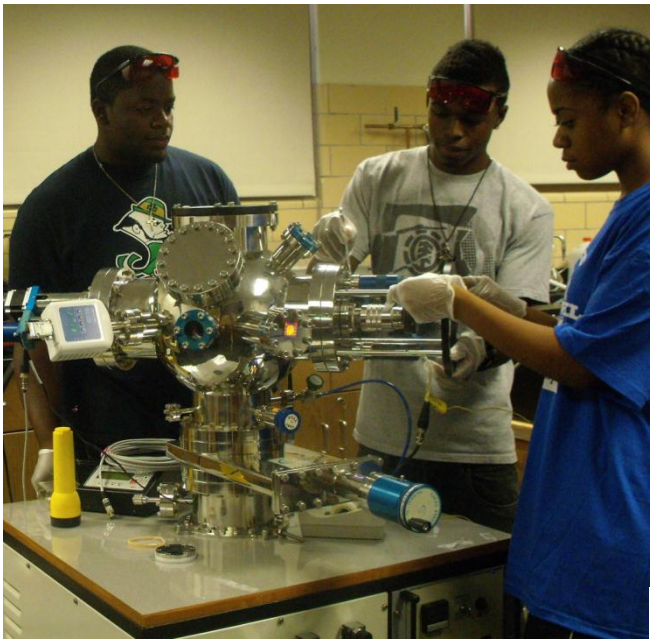
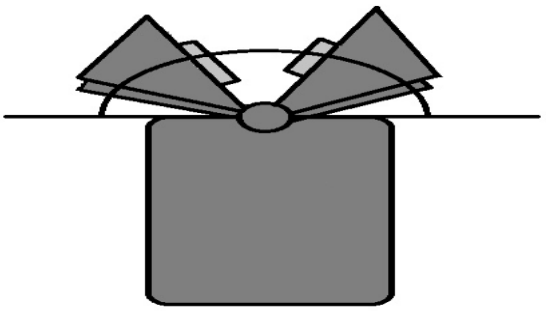


Figure 24: BaTiO3:Mn thin film B , T=450ºC.


laser
Target 1
A
Substrate
C
B
Target 2
laser







Target holder. Split into two pices where each can old different target for double ablation to facilitate chemical interaction of two beams.

Chapter 2

Pulsed laser deposition of epitaxial BaFeO3 thin films

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ABSTRACT

Epitaxial thin films of Barium Ferrite (BaFeO3) have been fabricated by the pulsed laser deposition technique on a Si substrate. The magnetic parameters were measured using vibrating sample magnetometer. The ferromagnetic resonance (FMR) measurements of the thin film were found to be close to the parameters associated with bulk materials. The Atomic Force Microscope was used to determine the surface morphology at different temperatures. The derivative FMR line width was measured to be 70 Oe at 60 GHz and 50 Oe at 90 GHz. The relationship among the Coercivity, crystalline orientation, and grain shape and size is presented.

1. INTRODUCTION

Barium ferrite powder was selected in this study because of its suitable coercive force (H_C) and large remnant magnetization (I_r), which are required for magnetic recording materials. Moreover, the hexagonal plate-like shape of the powder particles is known to be suitable for perpendicular magnetic recording since the easy magnetization axis is perpendicular to its hexagonal plane. It is believed that perpendicular-recording techniques will improve the recording density at least by a factor of ten, compared to the currently employed longitudinal recording. In addition, many papers have emphasized the importance of the use of this material in magnetic recording media [1-6].

In a ferromagnetic solid, the strongly coupled atomic magnetic moments tend to be aligned parallel to each other. As a result, a spontaneous magnetization appears in such a material. That is, the solid can have a bulk magnetic moment even in the absence of a magnetic field. Above a Curie temperature T_C, however, the spontaneous magnetization vanishes and the solid is paramagnetic and its susceptibility is given approximately by the Curie-Weiss law.[7] The work of Kittel [8] and Van Vleck, [9] who generalized the theory of Landau and Lifshitz, [10] contributed

greatly to the understanding of the FMR phenomena, and since then many papers related to this subject have been published.

In the presently conducted investigation, FMR absorption was studied in the case of six fine-powder samples of barium ferrite. Then the selected $BaFeO_3$ materials were used to produce a doped PLD thin film BaFeO3:Mn. The observation was carried out for each powder sample by employing an x-band spectrometer, which is equipped with a variable temperature accessory. The FMR absorption spectra and its first derivative curves were detected, and the resonance magnetic field was estimated.

The resonance field was then interpreted in terms of particle shape and magnetocrystalline anisotropy. In this analysis, the effect of particle shape was estimated with the use of the saturation magnetization M_S, which was measured employing a vibrating sample magnetometer (VSM). This analysis enables one to deduce the first anisotropic constant K_1 representing the magnetocrystalline anisotropic effect. It was found that the estimated values of K_1 differed greatly for the different samples and was affected by the preparation condition of the powder. The $BaFeO_3$:Mn thin film was further studied using the EPR technique. Paramagnetic resonance may serve as an effective way of investigating defects in crystals[11-13]. Such a possibility is connected with the fact that in paramagnetic crystals dislocation type defects alter the internal crystalline fields and thereby cause a displacement of the paramagnetic resonance line and a charge in the spin lattice interaction.

2.1 Theoretical Aspects of the Magnetic Resonance in Ferromagnetics

The resonance absorption of external electromagnetic radiation in ferromagnetics has been studied by RF spectroscopy. To determine g-factor of the atomic system, it is convenient experimentally to fix ω_0 and to find the resonance magnetic field H_{res} corresponding to the given frequency ($H_{res}= \omega_{res}/\gamma$). In ferromagnetic materials, there exist strong internal anisotropic magnetic fields, which are caused by the magnetic interaction between the electrons, which contribute to the spontaneous moment. The effective anisotropic field H_{eff} depends on the crystal symmetry, the shape of the sample, the magnetization M and its direction, the direction of the external magnetic field H_0 with respect to the crystallographic axes and to the surface of the sample.[14] In addition, the domain structure, which comes from the inhomogeneities in magnetization, is due to the finite size of the sample. This domain structure may influence the resonance conditions, but because ferromagnetic resonance is generally investigated in a state of magnetic saturation, then the external field destroys the domain structure, and the entire sample becomes a single domain.

2.2 The Effective Internal Field in a Ferromagnetic Sample

Pauli's equation yields the following vector equation for the time dependence of the average magnetization per unit volume for a system of uncoupled particles:

$$M = dm/dt = - ([M,H])$$

This means the length of the vector M remains constant in time. By considering the internal effective field H_{eff}, whose effect is equivalent to that of the external field H_0, then equation (1) becomes:

$$M = - ([M, H_{eff}])$$

In the thermodynamic equilibrium state, the direction of the magnetization vector M of a ferromagnetic material coincides with the direction of the internal effective field H_M, whose magnitude is

$$H_M = -\partial F/\partial M$$

where F is the free energy per unit volume. For a nonequilibrium state, the orientation of the vector M varies due to the influence of the nonvanishing terms H_θ and H_N, gives the general resonance frequency as [11]:

$$\omega_{res} = \gamma H_{eff} = \gamma/M \sin\theta_0 \{F_{\theta\theta}F_{\phi\phi} - F_{\theta\phi}^2\}$$

Now, an expression for the free energy density part is needed to determine ω_{res}, which depends on the orientation of the magnetization vector. If we assume that the sample be ellipsoidal with its principal axes along the coordinate axes, the free energy density of the sample is:

$$F = - M_s H_0 + ½ (N_x M^2_{sx} + N_y M^2_{sy} + N_z M^2_{sz})$$

where M_S is the saturation magnetization, and N_x, N_y, N_z are the demagnetization shape factor related by

$$N_x + N_y + N_z = 4\pi$$

Now, taking the x-axis of the Cartesian system as the polar axis, then

$$F = -M_s H_0 \sin\theta \sin\phi + ½ M^2_s (N_y \sin^2\theta \cos^2\phi + N_z \sin^2\theta \sin^2\phi + N_x \cos^2\theta)$$

Now, considering the magnetizing field H_0, two cases has to be considered; first, when H_0 is parallel to plane, and second, when it is perpendicular to the plane. For the first case, $\phi_0 = \pi/2$; $\sin\theta_0 = H_0/ M_s (N_z-N_x)$, then

$$M_s (N_z - N_y) \square H_0 \square M_s (N_z - N_Y)$$

For the second case, $\phi_0=\pi/2$ and $\theta_0=\pi/2$, then

$$H_0 \; f \; N_s (N_z - N_x)$$

To minimize the free energy in the direction of the major axes, let us assume that the major axis of the ellipsoid coincides with the z-axis, then $N_z < N_x$. Taking the second derivative of the free energy Equation (7) with respect to the angles in the equilibrium position, then

$$\omega_{res}/\gamma = \{N_y - N_x/ N_z - N_z [M^2_s (N_z - N_x)^2 - H^2_0] \}^{1/2} \approx M_s \{(N_y - N_x)(N_z - N_x) \}^{1/2}$$

for H parallel to plane. From this, we can see the dependence of the resonance frequency on the sample shape and the saturation magnetization M. Now, to study the shape-anisotropy factor ΔN which is a function of the ratio of the longitudinal to transverse axes, $n = l_\psi / l_\zeta$, and let the sample be an ellipsoid of revolution about the z-axis then, Equation (10) for $n < 1$ and $\Delta N < 1$, becomes

$$\omega_{res}/\gamma = \{(H_0 + (N_z - N_x)M_s) (H_0 + (N_y - N_z) M_s\}^{1/2}$$

From that, it is clear that to determine the shape anisotropy factor ΔN, we need only the value of n.

2. EXPERIMENTAL

2.1 Pulsed laser ablation/deposition chamber

The PLAD system is consists of two 6 inches cubes connected by a bellows assembly. The ablation chamber is fixed while the deposition cube is mounted on a rail assembly that is controlled by computer. This allows the selection of the targets during the ablation process, and also, the selection of dopant materials. This setup allows for a 0.15 m travel range in the distance between the cubes that can be varied quite quickly. On either side of the cube are view ports with 4 in. quartz windows allowing for the introduction of the laser ablation beam. The windows are also convenient for the visual inspection and alignment of the ablation targets.). A Reflected High Energy Election Diffraction system (RHEED aligned axially with respect to the ablation targets, for in situ monitoring of the film growth. All six samples rotate simultaneously at the same speed. For ion extraction, a grid assembly has a 0.625 in. hole right in front of the 0.5 in. sample stub. By adjusting the conditions such as ablation laser power and fluence, the neutral-to-ion ratio in the ablation plume can be controlled. The new to this design is the double target holder. We have designed a new target holder which allows two laser beams to ablate two different materials at the same time. This is called Double Laser pulse deposition /Ablation technique (DLPD or DLPA technique). Both stages can be set independently to different angle which allows the pulses of the two target to cross each other's and interact chemically to produce a different substance, or to allow any dopant to settle into the thin film lattice. This revolutionary target holder (patent in progress) will allow the PLD to go beyond the constrains of the process in doping the thin film in situ with transition metal ions .

The FMR spectrometer operates with a klystron microwave oscillator at a frequency of approximately 9.8 GHz. A sample is placed in a microwave cavity, and the energy absorbed by

the sample is detected. In addition, the vibrating sample magnetometer (VSM) which is used in this study is Digital Measurement System (DMS) model 880. The specimen is forced to vibrate in a direction perpendicular to an external magnetic field. The ac signal induced by the dipole field of the specimen in a pair of secondary coils placed on both sides of the specimen is amplified and compared with a signal produced by a standard magnet M, giving rise to an output signal which is exactly proportional to the magnetic moment of the specimen. By using the VSM, measurements have been made for the six samples over the temperature range -50°C to 500°C. The coercivity (H_C), the remnant magnetization (I_r), the squareness (SQ), the hystresis loops at room temperature and the saturation magnetization (I_S) for the samples were measured. The standard characteristic of these samples is listed on Table 1.

4. RESULTS AND ANALYSIS

4.1 Experimental Results

Study was carried out for the barium ferrite samples and both FMR absorption curves and their first derivative curves were detected over a temperature range of –195 ^{o}C to 500 ^{o}C. Figures 1 and 2 shows the peak intensity of the FMR absorption curves and the peak intensity of the first derivative curves at room temperature. This experiment was repeated for each sample at different temperatures. From these curves, it is evident that the shape of the absorption under a given condition was greatly different from one sample to another. In addition, it is evident that the signal broadening increases with increasing temperature . The figures show that the intensity approaches zero and gives the approximate Curie temperature T_C for these samples . The approximate values of T_C for the six samples are listed on Table 3.

By using the VSM the complete hysteresis loops at room temperature were measured, and plot in Figure 4. From the hysteresis loops the values of the coercive force (H_C), the saturation magnetization (I_r) and the saturation magnetization intensity (I_S) were determined (Table 3). Also the factor I_S, H_C, I_r and SQ for these samples were measured and plotted versus temperature.

4.2 Analysis of the experimental results

Optical surface Morphology:

The surface roughness and the homogeneity of the deposited thin film surfaces of both $BaFeO_3$ and $BaFeO_3$:Mn analyzed using Atomic Force Microscope as shown in Figure 5-17. The Figure shows that the DPLD thin film is relatively with a periodic surface structure as grating formation. The preliminary explanation of such phenomenon is due to the double plumes interaction. The time of the ablation of the doped materials controlled to have 0.5 % or less doped materials in the thin film. The substrate temperature has a major effect of the structure of the thin film as shown in Figures 4-16. The more higher temperature the more periodicity on the thin film as shown in Figure 11 . The roughness of the surface was measured at 30-50 nm for films of 190-270 nm thick.

An analytical model can be found to explain the ferromagnetic resonance absorptions for barium ferrite samples at high temperature. This model assumes the fine particles of barium ferrite powder, which have hexagonal shape, to be as a rotational ellipsoid. In addition, it assumes that the particle is completely isolated. Let n = D/L, as D is the diameter and L is the thickness. When n equals infinity for an ellipsoidal sample, then the magnetization field, which is parallel to the plane, is

$$H_{01} = -2\pi M_s + ((2\pi M_s)^2 + (\omega_{res}/\gamma)^2)^{1/2}$$

and for the other extreme case, when n = 0, the magnetization field perpendicular to the plane is

$$H_{02} = \omega_{res}/\gamma + 4\pi M_s$$

If M_s equals zero, then $H_0 = \omega_{res}/\gamma$

The powdered samples used represent randomly oriented particles. At a high temperature when M_s is very small, H_{01} will be stronger than H_{02} .

If the angle between the magnetization field and the perpendicular to the plane-axis is θ, then for θequals zero, which means the magnetization field is perpendicular to the plane, one can represent this case by plotting y ($y = H_0/(\omega_{res}/\gamma)$)versus x, as:

$$y = 1 - 2x ,$$

. and $x = (k_1/M_S - ½)N M_S) / (T_{res} /()$

For the other case when $\theta = \pi/2$ which means the magnetization field is parallel to the plane, one can find for y from to be:

$$y = (4x^2 - 1)^{½} , \quad 2x \geq y$$

and $y = x + (x^2 + 1)^{½} , \quad y \geq 2x$

A complete graph is plotted for this model in Figure 5.

Following this procedure, the first anisotropy term could be estimated from the FMR absorption spectroscopy. Plots of K_1 versus temperature are shown on Figures 7 and 8 for two samples.

The calculation is repeated for the case of the flat disk as n = 0, to compare with the case of barium ferrite where n is greater than zero. Two points for K_1 are found at corresponding temperatures. The first point T_1 is where K_1 changes sign (from + to -) and the second point T_2 is where K_1 approaches zero at high temperature. These temperatures are listed on Table 3. From VSM results, the temperature where H_C, I_S and I_r approximately equals zero can be determined. For sample 1, because the value of n was not known at the time of the calculation, only the calculation was done for the case of n = 0 and K_1 = 0 (Figure 4). The EPR spectrum for the:$BaFeO_3$:Mn thin film was measured at room temperature as well (Figure 6).

5. DISCUSSION AND CONCLUSIONS

5.1 Reliability of the Estimated Values For K_1.

The fine particles of barium ferrite powder are hexagonally shaped and so, the thin film. In this model , the shape is approximately replaced by a rotational ellipsoid. Thus, there is an uncertainty in determination of n. It is noted that n can be defined accurately only for a rotational ellipsoid for both powder and thin film. The assumption that the sample particle is completely isolated is not quite true.

From Figures 21 and 22, one can see that the sign of K_1 is different between the high and low temperature ranges. The figure shows K1 for the the thin film compared to powder. The temperature where the sign of K_1 changes is referred to as transition temperature T_t, . From Figures 3 and 4, one finds that the Curie temperature T_C is significantly different for different samples. For a given sample, T_C depends on the assumed value of n but for the high temperature range, K_1 becomes very small.
Moreover, the temperature where K_1 practically becomes zero is found to be independent of the assumed values of n.
Furthermore, this temperature is approximately equal to T_C, which was estimated from the relation between the FMR absorption curve intensity and temperature (see Table 3).

The positive sign for K_1 implies that the spontaneous magnetization or the easy magnetization axis is parallel to the hexagonal axis, or perpendicular to the hexagonal plane. The negative sign implies that the spontaneous magnetization is perpendicular to the hexagonal axis or parallel to the hexagonal plane. Obtained results of K_1 may have a potentially serious impact on selection of suitable barium ferrite powders for perpendicular recording. The powder with a large positive value of K_1 and a high value of T_t should be suitable for recording. The powder with a positive K_1 but a low value of T_t may be used but would be a rather poor material since the recorded signals would disappear at a relatively low temperature. However, the one with a negative K_1 cannot be used for perpendicular recording, since the spontaneous magnetization is parallel to the hexagonal plane. According to this criterion, sample 2 is the best since K_1 is relatively large and T_t is 100 ^oC; samples 5 and 6 may be used, but their T_t is below 60 ^oC, and consequently any recorded signals are possibly unstable; samples 1 and 3 cannot be used because K_1 is negative. This extensive study gave us an indication for the selection of the powder, which was used for the thin film doped by Mn. The preliminary results shows that the nonlinearity of the thin film $BaFeO_3$:Mn was enhanced. In conclusion, this investigation has established the importance of FMR spectroscopy in characterization of barium ferrite powders for perpendicular recording and

it shows that the PLD thin film of $BaFeO_3$:Mn characteristics very close to powder at rom temperature. The more the temperature of the substrate increased, the diverse these characteristics became . In addition, these investigations demonstrated the potential of PLD of $BaFeO_3$:Mn doped thin film using of Barium Ferrite powder ballet in nonlinear optical crystals.

ACKNOWLEDGMENTS

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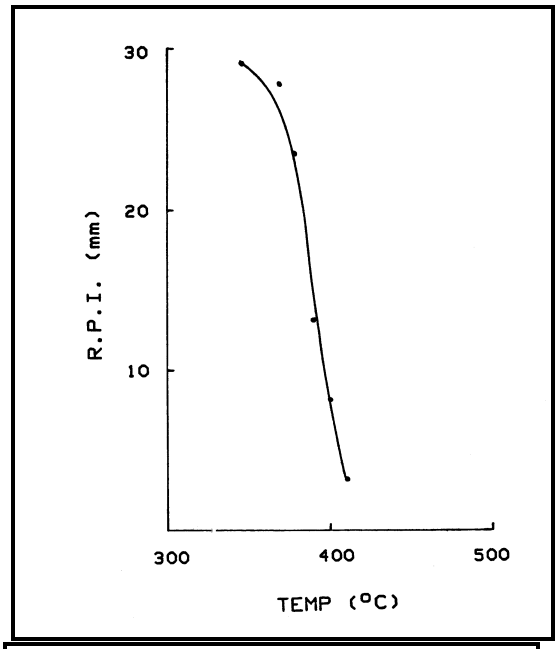


Figure 1: Relative peak intensity of the FMR absorption versus temperature for sample 1.

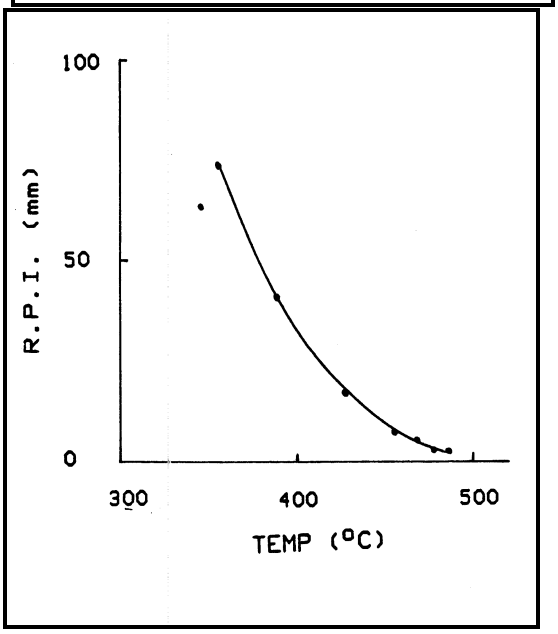


Figure 2: Relative peak intensity of the first derivative curve for sample 1.

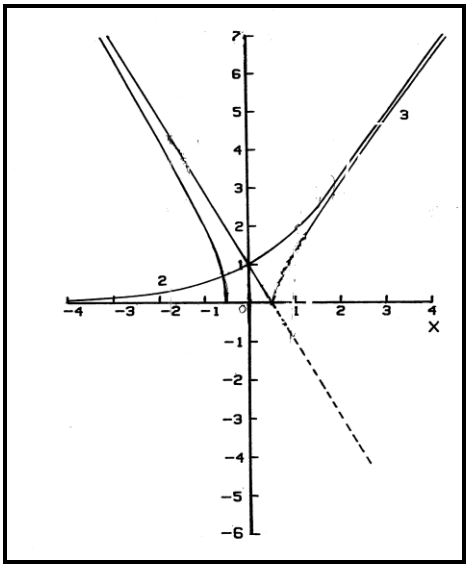


Figure 3: Plot of the analytical mode for FMR Field. Curve 1 for $y = 1 - 2x$
Curve 2 for $y = x + (x^2 + 1)^{1/2}$, $y \geq 2$
Curve 3 for $y = (4 + x^2)^{1/2}$, $y \leq 2$

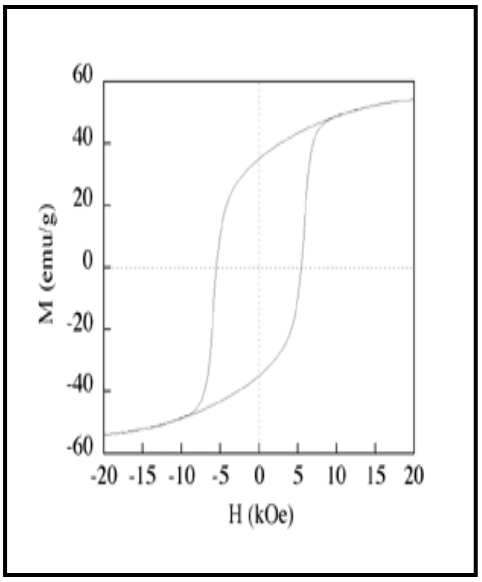


Figure 4: Hysteresis loop for sample 1 at room temperature.

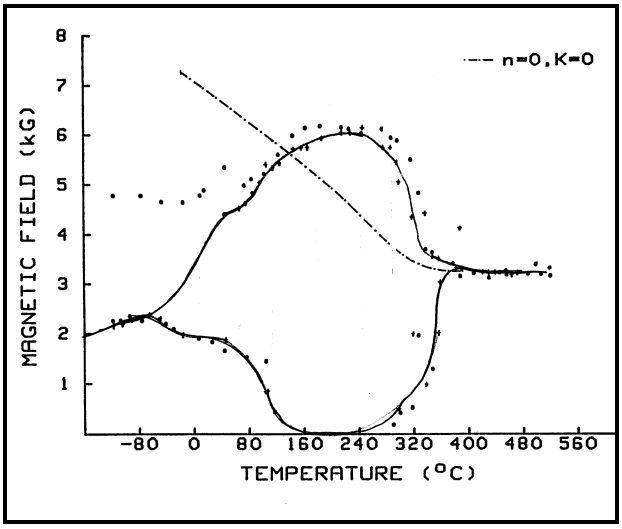


Figure 5: Sample 1, the relative peak of FMR and first derivative

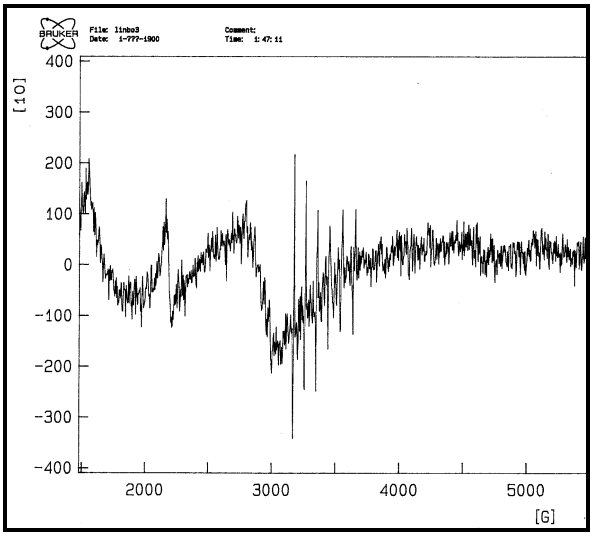


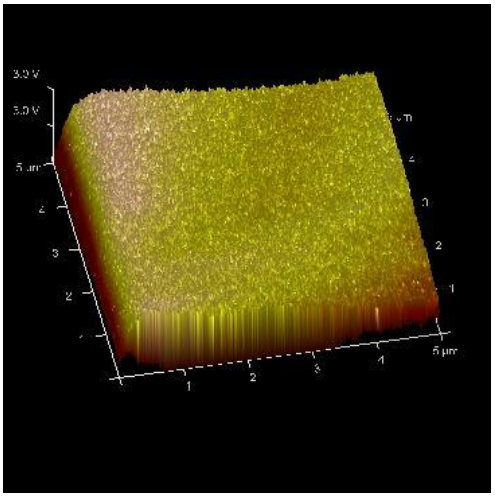
Figure 6 : EPR spectrum for Mn: $BaFeO_3$ thin film

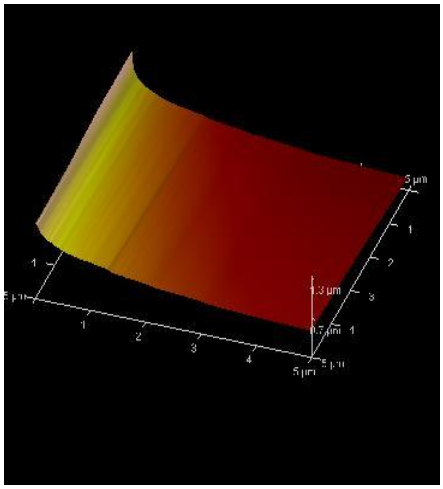
Sample No	H_C	O_S	BET	D	D/L
1	740	59.3	62.8	0.07	7
2	710	52.1	57.8	0.10	20

Table 1. Characteristics of the two barium ferrite samples. H_c: Coercivity (Oe), O_S: Saturation Magnetization (emu/g), BET: Specific surface Area (m^2/g), D: Average diameter of particles (:m),D/L: Average ratio of diameter to thickness.

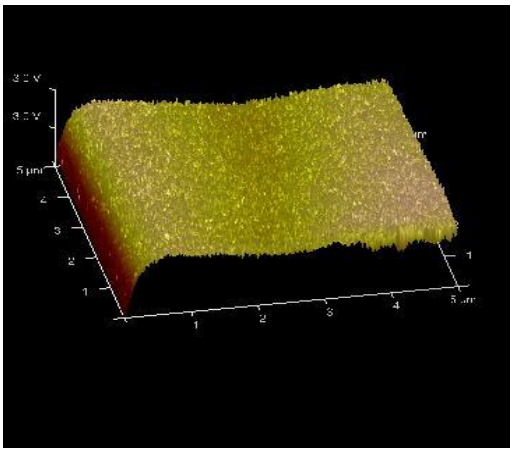
Sample No.	H_c (Or)	I_s (emu)	I_r (emu)	SQ
1	682.2	0.1043	0.0515	0.494
2	636.3	0.1577	0.0875	0.555

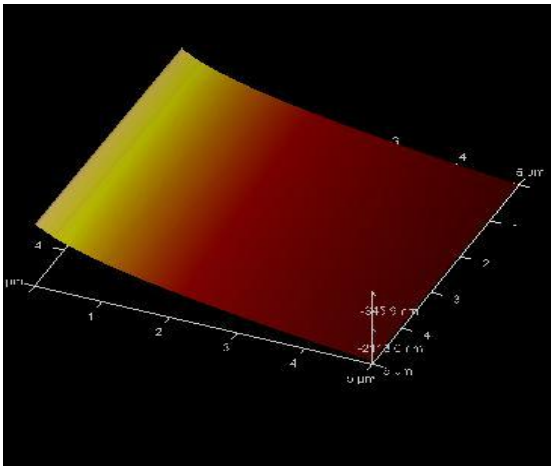
Table . H_c, I_s, I_r, and
SQ for the two powder samples at room temperature



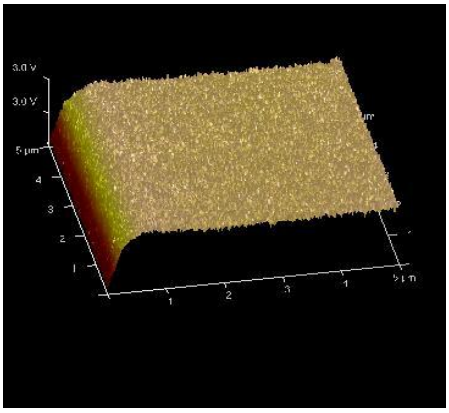


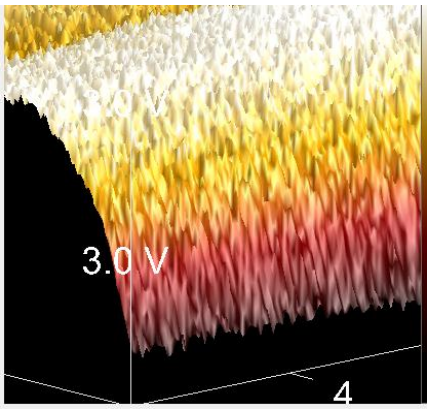
Figures 7&8: BaFeO3:Mn, P(O 30 mTorr) T=650C





Figures 9&10: BaFeO3:Mn P(O 10mTorr), T=750C





Figures 11&12: BaFeO:Mn P(O 20 mTorr), T=550C

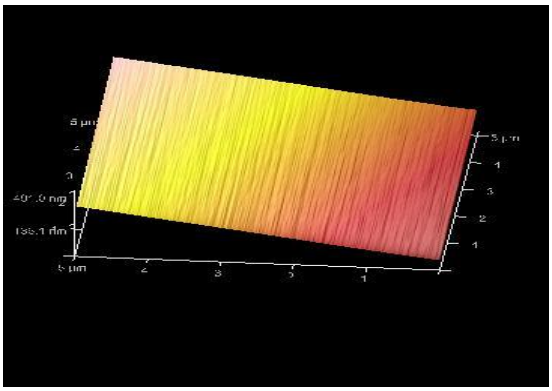


Figure 13: BaFeO:Mn P(O 20 mTorr), T=550C

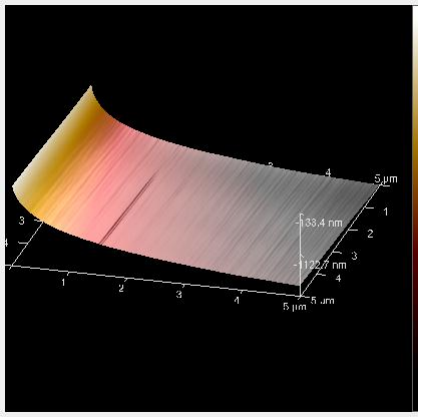


Figure 14: BaTiO3:Mn P(O 5mTorr), T=400C

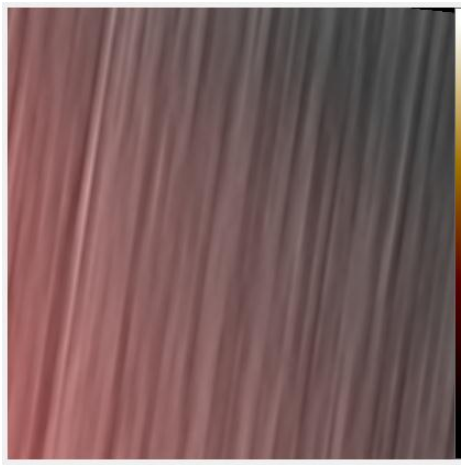


Figure 15: BaTiO3:Mn P(O 5mTorr), T=400C

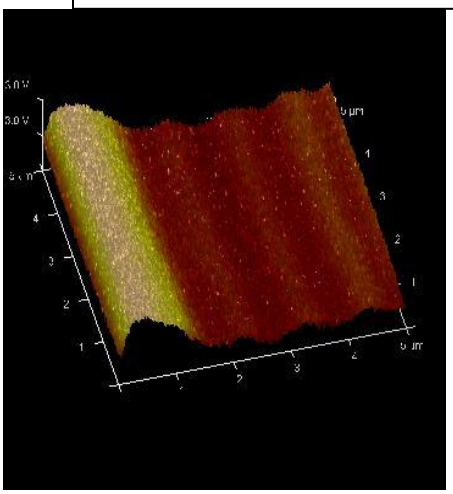


Figure 16: BFO:Cr

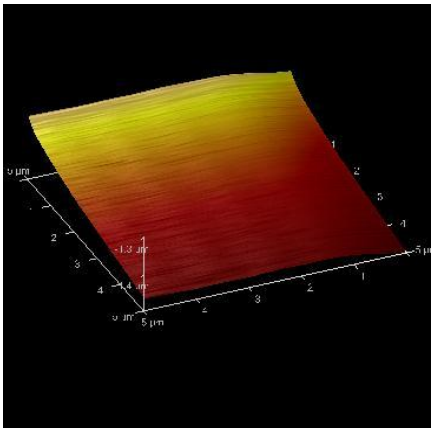


Figure 17: BFO:Cr

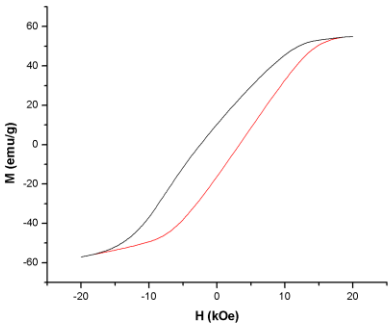


Figure 18: Hysteresis loop for sample 1 at room temperature

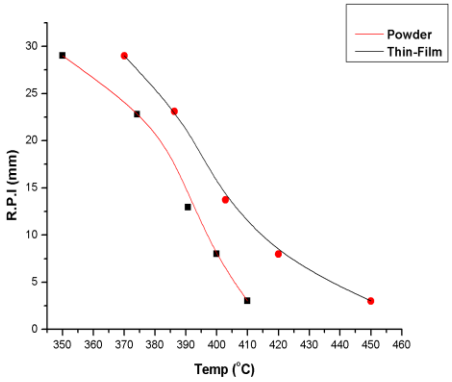


Figure 19: Relative peak intensity of the FMR absorption versus temperature for sample 1.

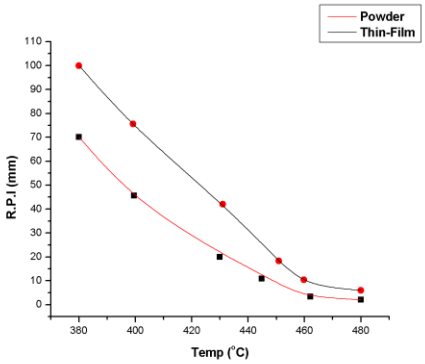


Figure 20: Relative peak intensity of the first derivative curve for sample 1.

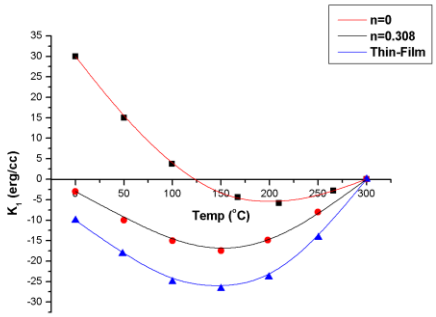


Figure 21 : K_1 for sample 2 versus temperature.

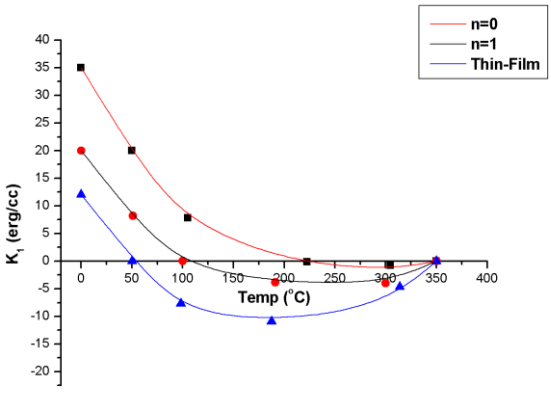


Figure 22: K_1 for sample 1 versus temperature.

Chapter 3

Outcomes and recommendations for future work

3.1 Conference attendance and presentation of the research results:

1. **Training five African American in the area of PLD of hard materials and attended four conference and presented three posters and two papers: Scientific Conferences /Meetings attended with students:**
2. ABRCMS-Annual Biomedical Research Conference for Minority Students Conference 2010 in- Charlotte, North Carolina
   Dr. A. Darwish and Dr. Bernard Singleton-Faculty

   Students
   A. Curvelle Lewins- Junior
   B. Simeon Wilson
   C. Joshua McCollum
   D. Ebone Pierce

3. ERN Conference 2011-Emerging Researchers National Conference in Washington, D.C.
   Dr. Bernard Singleton-Faculty, Dr. Hong Dai, Dr. A. Darwish

   Students
   A. Simeon Wilson
   B. Benson Dabney

4. Fattah Conference 2011 on Higher Education in Philadelphia, PA

   Students

A. Alexander Lopez	D. Ateriya Addley	G. Jon Goodwin J. Jasmine Hardman
B. Alexandri Broadnax	E. Chardai Gray	H. Charne Thomas
C. Tanesia Patterson	F. Valencia Wise	I. Shamala Criswell

5. 68th Annual Beta Kappa Chi and National Institute of Science Honor Societies joint Meeting in Atlanta, GA.
   Dr. Bernard SIngleton

   Students
   A. Tanesia Patterson
   B. Valencia Wise
   C. Michaela Bradley
   D. Shamala Criswell
   E. Ebone Pierce
6. SPIE conference in San Diego Aug 2011 Dr. Darwish and Simeon Wilson, two presentations and two papers published.

7. National Society of Black Physicists and joined Hispanic meeting in Austin , Texas Dr. Darwish, Dr. Hong Dai, Dr. Singleton, Simeon Wilson, Ebene' Pierce, Joshua McCollum, Denby. Three posters and one oral presentation.

3.2 Milestone accomplishments:

1. **Establishing a research program in lasers and Nano Alloy-composite thin film fabrication**
2. **Investigate the effect of laser wavelength and the Laser energy on the ablation process (Needs to be completed we just received the OPO system in Aug 2011 and the spectra-physics completed the setup by Oct 10, 2011). We are ready to continue the investigating the effect of the laser wavelength and energy on the ablation of the hard materials to produce a stronger, high temperature tolerance thin film.**
3. **Investigate the ablation/ coating of hard materials using the innovative double pulse laser Deposition/ablation (DPLD) (was communicated in two publications during the SPIE Aug meeting in San Diego 2011) already published.**
4. **Establishing a new research curriculum though out the STEM disciplines (Two courses were developed between Physics and Chemistry.**
5. **Seventeen students and five faculty were supported to travel and presented their research findings in PLD /DPLD research in five conferences ERN, NSBP, NSBE, SPIE annual Symposium 2011, HBCU-UP research conference, ABRECRAMB. All travel were supported by different projects of Dr. Darwish as matching for the AFOSR two funding.**
6. **Publishing two papers , presented five posters and three abstracts from this funded project**
7. **In a process of establishing a Master degree program in material science, thin film fabrication of materials and its optical characterizations in general.**
8. **Continue to expand the research enterprise facility and continue to apply for more funding to acquire the necessary equipment to continue the proposed research activities**
9. **Most significant results a. Modified the pulse laser deposition technique by designing a new double substrate holder which enable the access to an innovative double ablation technique which employed two laser beams to ablate two different targets in situ, like a crystal BaTiO3 doped with a transition metal ions.**
10. **This new technique produced a pattern of fringes and periodic grating which changed the nonlinear optical characteristics of the ablated thin film for the first time to be observed in PLD thin films**
11. **More investigations needs to be carried on to complete the reset of the proposed research work namely the manifestation of the hard materials thin films (Painting of thin film) and the effect of the laser wavelength and laser energy of the PLD process and the characteristic of the thin films.**
12. **Using the Optical Parametric Oscillator to identify different wavelengths which will be useful in the study. This study needs more fund and time to be completed since we just received the OPO in August 2011.**

More time is needed to carry out more investigation on the effect of the magnetic field on the ablation of materials.

3.3 Recommendation to continue the future work:

We completed one task which is the ablation of $BaFeO_3$:Mn and we need more time and fund to continue the other proposed modifications and experiments namely:

1. Establishing the ion/neutral ratios for various plume components as a function of laser wavelength and laser fluence (We just received the OPO in August 2011 and we will start the experiment in Oct 2011).
2. Investigate the role of the magnetic field strength on film growth (we already published part of this investigation $BaFeO_3$:Mn thin film in Aug 2011.
3. Investigate the role of pulse duration: ns, ps, fs (we will propose purchasing ps and fs laser once we conclude the investigation using the funded ns laser .
4. Analyze the films produced under such a matrix of conditions (Two papers already published and reported in this final report the finding.

Conference attendances:

Scientific Conferences /Meetings attended with students:

8. ABRCMS-Annual Biomedical Research Conference for Minority Students Conference 2010 in- Charlotte, North Carolina
   Dr. Bernard Singleton-Faculty

Students

   E. Curvelle Lewins- Junior
   F. Simeon Wilson
   G. Joshua McCollum
   H. Ebone Pierce

9. ERN Conference 2011-Emerging Researchers National Conference in Washington, D.C.
   Dr. Bernard Singleton-Faculty, Dr. Hong Dai, Dr. A. Darwish

Students

   C. Simeon Wilson
   D. Benson Dabney

10. Fattah Conference 2011 on Higher Education in Philadelphia, PA
    Dr. Ruby Broadway- Faculty

Students

D. Alexander Lopez	D. Ateriya Addley	G. Jon Goodwin	J. Jasmine Hardman
E. Alexandri Broadnax	E. Chardai Gray	H. Charne Thomas	
F. Tanesia Patterson	F. Valencia Wise	I. Shamala Criswell	

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Students

   F. Tanesia Patterson; Valencia Wise; Michaela Bradley; Shamala Criswell; ;Ebone Pierce

5. SPIE conference in San Diego Aug 2011 Dr. Darwish and Simeon Wilson, two presentations and two papers published.
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Participants

1. Simieon Wilson Junior physics and Engineering student

2. Ebene' Pierce Senior Physics and Engineering Student
3. Jashoua McClum Junior physics student
4. Dr. Hadi Alhkaby Professor in Mathmatics Department
5. Dr. Brent Koplitz Chemistry Professor, Chair of the Chmistry Department, Tulane University
6. Dr. Allan Burkett, Professor of Chemistry, Chemistry Department, Dillard University

Published papers

1. Darwish, A. M., Wilson, S., Alkahby, H., Koplitz, B., "Preparation of BaTiO3 thin films by double-pulse-lasers deposition" Photonic Fiber and Crystal Devices: Advances in Materials and Innovations in Device Applications V SPIE Vol. 8120 ,2011.

2. Darwish, A., Wilson, S., Koplitz, B., "Pulsed laser deposition of epitaxial BaFeO3 thin films" Photonic Fiber and Crystal Devices: Advances in Materials and Innovations in Device Applications V, SPIE Vol. 8120, 2011.