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Physico-Chemical Characterization of Multilayer YIG Thin Film Deposited by RF Sputtering

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Abstract

Yttrium iron garnet (YIG) film made as a magneto-optical medium suffers from the problem of crack formation, caused by the heating process. YIG thin film is deposited by radio frequency RF magnetron sputtering; the obtained layer is amorphous and it needs annealing to be crystallized. After heat-treatment at 740 °C of the sample realized on quartz substrate, we observe cracks on the entire film surface. This is due to the large difference between the thermal expansion coefficient ($5.5 \times 10^{-7} \text{ K}^{-1}$ for quartz and $10 \times 10^{-6} \text{ K}^{-1}$ for YIG). In this paper we present a new fabrication method to reduce this problem, we make a multilayer to obtain at the end a uniformly unique layer with excellent crystalline structure. Such films have the possibility to reach a thickness of 500 nm. YIG films have been studied by Rutherford Backscattering Spectrometry (RBS), optic Ellipsometry and the Scan Electron Microscope. The RBS spectra were collected in channelling geometry with incident particles energy 2 MeV and 3.5 MeV. The thickness and the stoichiometric value of the thin films have been evaluated. Simulation of all spectra indicates a constant composition. Ellipsometry method is well adapted to model the thin film structure layers, and to measure the thickness of the film and the complex index of refraction. The theoretical ellipsometric value of the index of refraction is (2.22) while the experimental value is ranging from 2.2 to 2.3 for a wavelength of 1550nm.

1 Introduction Some recent works on preparation of magnetic thin films are useful for magneto-optical applications [1,2]. The YIG film made the objective of a great number of studies for reasons related to its magnetic and magneto-optical properties in the fields of optics and microwave frequencies. The YIG is a ferrimagnetic garnet crystal with the composition of $\text{Y}_3\text{Fe}_5\text{O}_{12}$, with a substantial Faraday rotation in large parts of the optical and microwave spectrum [3]. YIG thin films are grown using various techniques: the conventional technology which is liquid phase epitaxy (LPE)[5], pulsed laser deposition (PLD)[6] and radio frequency (RF) magnetron sputtering [2-4]. In this work, we have used the RF-magnetron sputtering for its several advantages: dry process during deposition, possibility of high purity starting material, ability to sputter dielectric materials, commonly held industrial process and high compatibility with semiconductors technology. For this purpose, the radio frequency sputtering grown films can be used as high quality optical waveguides exhibiting the Faraday effect. Since the quartz substrate is cheaper than the GGG substrate, so we are interested to make a thin film of YIG on quartz substrate. The difficulty after heat-treatment at 740 °C to crystallize the film, is the cracks on the entire surface that are observed [7,8]. The stress during heat treatment is due to the large difference between thermal expansion coefficient of quartz and the film. For this reason we have decided to make a multilayer YIG thin film on a quartz substrate for optical and magneto-optical applications. We studied the deposition conditions on the physicochemical, magnetic and magneto-optical properties of the thin film of YIG, in order to determine the optimal parameters of deposition for obtaining a good quality film. The film must have some characteristics like smooth surface, correct stoichiometric values and suitable refractive index as necessary for magneto-optical applications. The essential aim of the new method of the present work is to carry out films of YIG on quartz substrate having a good surface quality with the minimum of cracks after a heat treatment.

2 Experimental

2.1 Film preparation In order to optimize the surface of the thin films deposited by RF magnetron sputtering, the following conditions should be set up such as argon pressure and flow rate, RF power and the sputtering time. YIG thin film was deposited by using RF magnetron non reactive sputtering system onto a quartz substrate [3]. The working pressure is 2×10^{-2} mbar and an argon gas of 99.999% is introduced into the sputtering chamber to create the plasma. The magnetron input power is 100 W. The rate of YIG film deposition is 0.3 $\mu\text{m/h}$. A thermal treatment promoted crystallization of the film; the samples are heated in vacuum. It is best not to exceed 300 degrees per hour in order to avoid thermal stress cracks in the thin film starting from room temperature. The film is heated at a rate of 4 °C/min till reaching 740 °C, and is maintained at this temperature for 2 hours.

We repeat this process many times (deposition and thermal treatment) until we obtained the required film thickness.

2.2 RBS Measurements A 5SDH pelletron tandem accelerator of 1.7 MV was used to perform Rutherford Backscattering Spectrometry measurements on the samples under normal incident beam and in a random direction to avoid channeling. The RBS is based on collisions between atomic nuclei. By this technique, it is possible to determine the atomic mass and elemental concentrations versus depth below the surface. YIG thin film is bombarded with a beam of high energy particles ($^4\text{He}^+$ of energy 2 MeV) [9]. The scattering angle θ is 165° referring to the beam direction, where the solid angle of the detector is 5.45×10^{-3} sr. The energy measured for the particle backscattering depends on the mass of the target atoms. RBS measurements was done and showed the influence of the multilayer film growth on the surface state of the film.

2.3 Ellipsometry Measurements The characterization of the optical constants and thickness of YIG thin films is a major part of our research, and ellipsometry is the primary method to determine these quantities. The instrument used is a variable angle spectroscopic and it is sensitive to several material characteristics, such as: layer thickness, optical constants (refractive index). The spectroscopic ellipsometer features high accuracy determination of the ellipsometric angles across their wide spectral range allowing superior precision and sensitivity for the characterization of transparent substrates thin films with low index contrast. For the purposes of our research we are most interested in determining film thickness and optical constants. The concentration of the effects of the Fresnel interference is essential in thin films design [10] since it can lead to large changes in the reflection and transmission of both thin film and substrate. The obtained data are fitted according to optical model permit us to determine the complex index of refraction and the film thickness.

2.4 SEM Measurements Scanning electron microscope (SEM) was performed with a JEM 100 C instrument equipped with a high resolution scanning device 9ASID-4D0 and a side-entry goniometer. An accelerating voltage of 40 KV was used images were recorded at a Kodak PXP-120 film. The resolution of the taken images is magnified from 100 times to 3000 times.

3 Results YIG thin films are deposited on quartz substrate by using a RF sputtering system. The control of the surface condition and the thickness of the films were measured using the (SEM) and a mechanical profilometer (table1), where the final thickness is 500 nm. The film crystallizes at a temperature 740°C , correct X-ray diffraction patterns are observed as shown in figure 1; the diffraction peaks observed being indexed to the JCPDS card 43-0507 and they corresponds to YIG.

Table 1: the layer with its corresponding thickness

Number of layers	Final thickness (in nm)
First layer	150
Second layer	365
Third layer	500

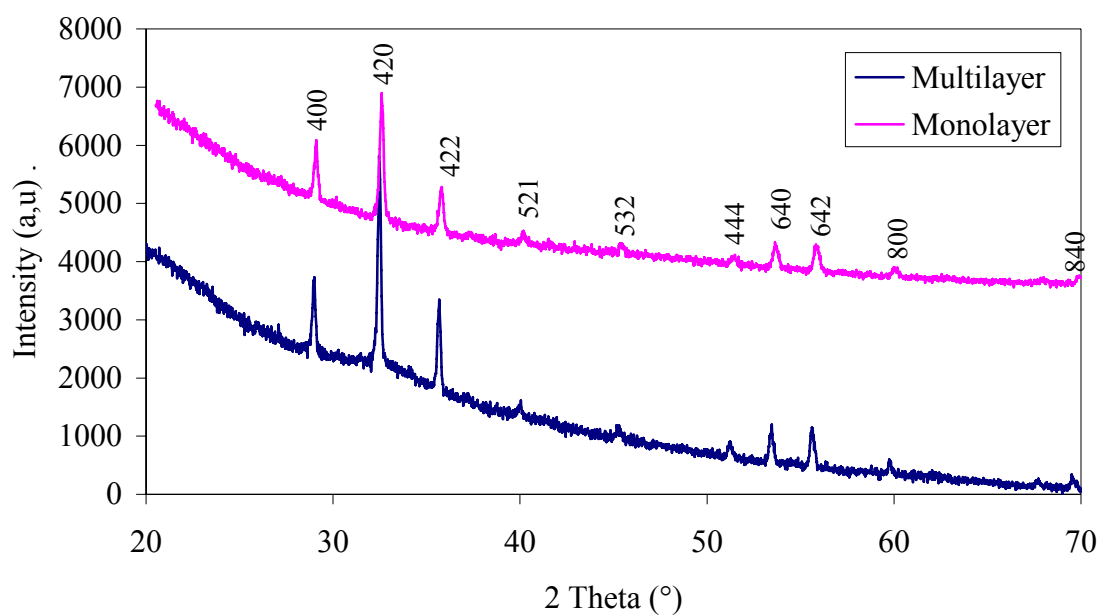
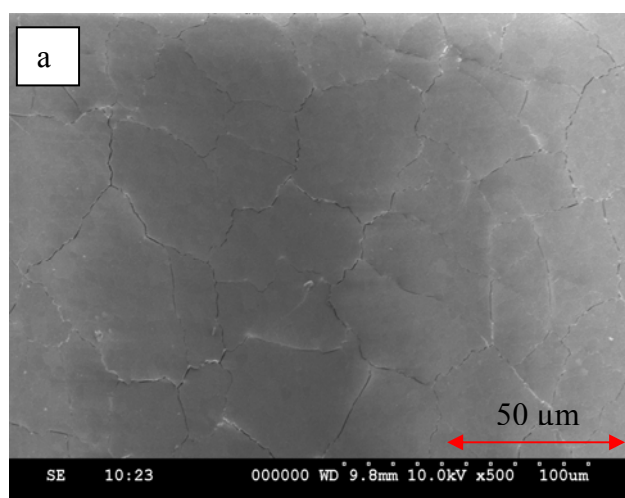


Fig 1: X-ray diffraction pattern of YIG multilayer and monolayer films.



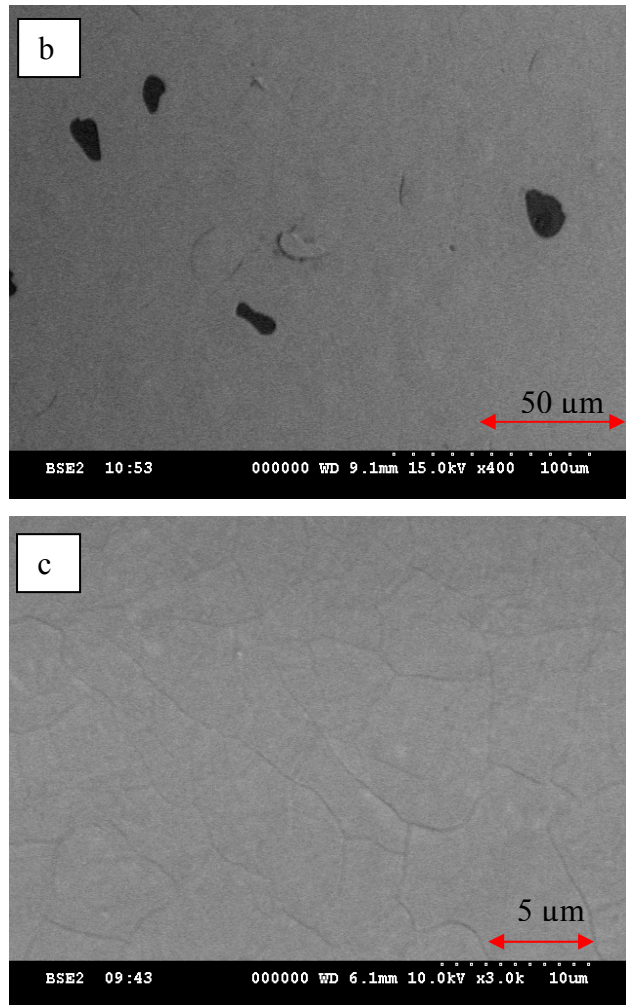


Fig 2: Pictures of SEM, a: deposition in one stage 500x magnifications, b: film deposited in many layers 400x magnification, c: film deposited in many layer 3000x magnification.

Figure 2.a presents the SEM photograph of YIG film on quartz substrate shows a formation of cracks on the surface due to the difference of thermal expansion between the YIG film and the substrate. In order to reduce the crake's formation a thin film is made by a multilayer annealed in each layer at 740 °C shown in figure 2.b. No crakes appear on the surface of the film for the same SEM resolution. The comparison between the SEM photos of the global method with those of the new method “multilayer” for the same magnification (500x) does not reveal the presence of cracks. Only at high magnification resolution (3000x), a cracks appear on the surface in figure 2.c. The thickness seems to be a limit, because crakes were observed for thickness higher than 500 nm, so the stress on the surface of the film can be reduced by the film thickness. The chemical stoichiometry of the film was analyzed by (RBS). The spectra recorded with the incident beam of 2 MeV energy of $^4\text{He}^+$ and backscattering ions were analyzed for an angle $\theta = 165^\circ$. Figure 3 represents the fitting of the RBS spectra, the four sharp discontinuities observed correspond to $^4\text{He}^+$ ions backscattered from O, Si, Fe and Y atoms.

These elements are present at the surface and in the film; also it is possible to simulate the RBS spectra using the SIMNRA program to obtain the stoichiometric value. Simulations and fits of the spectra are done where the simulated spectrum overlies the experimental data. The obtained stoichiometry of YIG thin film annealed at 740 °C is $\text{Y}_{2.68}\text{Fe}_{5.178}\text{O}_{12.14}$. The difference for Y atoms of the experimental value with respect to the theory is only 0.32. The experimental value for Fe atoms exceeds the prediction by about 0.178. For the O atoms, the excess is somewhat higher and it is 0.14. Such differences between experimental results and predictions for Fe and O are not large. In the case of Y atoms the difference is larger. They may be due to the dependence of the mass of the atoms and the thickness of the film.

Figure 4 represents the ellipsometry fitting data to determine the complex refractive index. A parameterized model was used to describe the YIG optical constants over the wide spectral range: from the far UV (190 nm) to near-IR (2100 nm). The ellipsometry determine the refractive index (n) and extinction coefficient (k) with a high degree of certainty for YIG thin film. The ellipsometry results show that the complex index of refraction is between 2.2 and 2.3 at a wavelength 1550 nm (the theoretical value is 2.2) and it is independent on the thickness of the film.

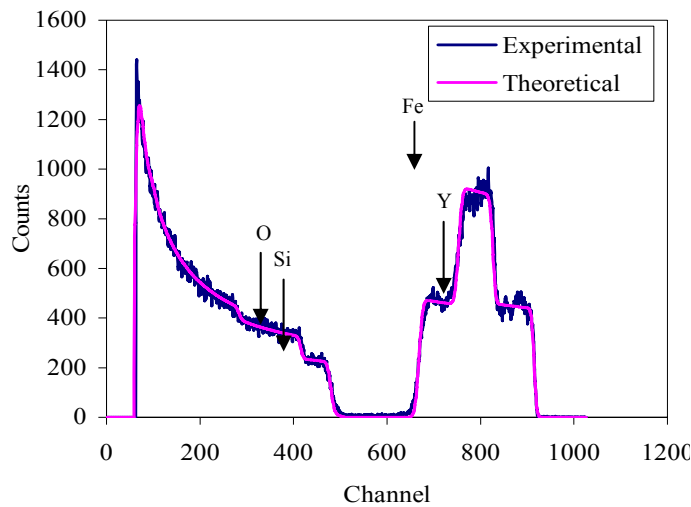


Fig 3: Fitting of RBS spectra for YIG thin film.

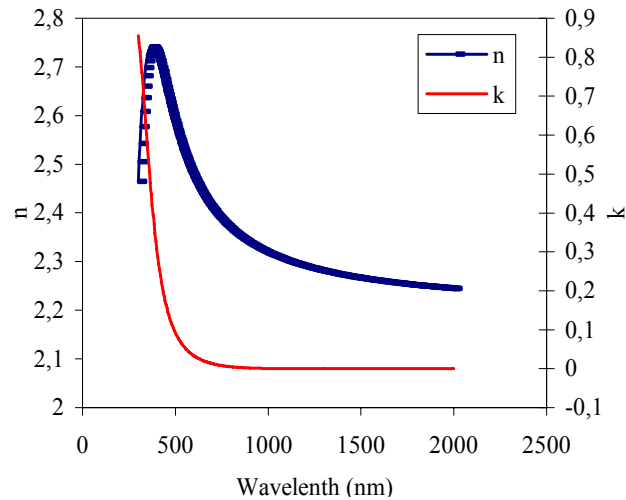


Fig 4: Fitting of Ellipsometry spectra between n, k and the wavelength.

4 Conclusion To improve the quality of the YIG thin film, we make many layers on the same substrate to obtain a uniformly unique layer with good crystalline structure. The film thickness is 500 nm and with minimum of cracks appears on the surface of the film. The XRD patterns of YIG films annealed at 740 °C for 2 hours shows a correct diffraction peaks according to the JCPDS card 43-0507. The obtained film has a near stoichiometry and the results: $\text{Y}_{2.68}\text{Fe}_{5.178}\text{O}_{12.14}$ show a composition near the theoretical values. Also in this paper we determine the index of refraction for YIG film which is between 2.2 and 2.3 at a wavelength 1550 nm. A YIG film on a quartz substrate can form a high quality optical waveguide.

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