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# Annealing effect on the microstructural and magnetic properties of $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films prepared by sol-gel method

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**ABSTRACT**

The effect of annealing on the microstructural and magnetic properties of $Er_{2.5}Y_{0.5}Fe_5O_{12}$ sol-gel films were studied at different temperatures using X-ray diffraction (XRD), field emission scanning electron microscopic (FESEM) and vibration sample magnetometer (VSM).The results show that the XRD pattern of the samples have a single phase garnet structure with reduction in the lattice parameter of Er:YIG films due to the smaller ionic radii of $Er^{3+}$ compare to $Y^{3+}$. The sizes of the particles are in the range of 50 to 90 nm. The saturation magnetization (Ms) increase and then decreases as the temperature increased.

| Annealing temperature | YIG | Er films | magnetic properties | sol-gel |


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## 1. INTRODUCTION

The study of yttrium iron garnet (YIG) films are becoming more important because of the properties that can be applied in microwave and magneto-optical devices [1-3]. For this, the magnetic and structure properties of the rare earth elements substituting YIG are studied in order to achieve various magnetizations and structures. The crystal lattice of YIG is cubic crystal, with the iron atoms occupying two different kinds of sites in the lattice. For this reason the formula is sometimes written as $\{Y_3^{3+}\}$ $[Fe_2^{3+}]$ $(Fe_3^{3+})$ $O_2^{-12}$. The magnetic properties of the crystal are mainly determined by the iron atoms. The iron atoms in the two kinds of sites interact antiferromagnetically with each other, giving a net magnetic moment equal to that of one atom. The Yttrium is magnetically polarized by the field from the iron atoms, but it has little influence on the strength of the magnetic interaction. The $Y^{3+}$ in YIG has no magnetic moment, so the net magnetic moment in YIG is due to the unequal distribution of $Fe^{3+}$ ions in two different sublattices.

A number of researches have been done in substituting $Bi^{3+}$, $Ce^{3+}$ for $Y^{3+}$ and $Co^{3+}$, $Co^{2+}$ for $Fe^{3+}$ in YIG [4-16]. However few works have been carried out to study Er-YIG powder nanoparticles [17-26]. When YIG bulk is prepared by ceramic methods, $YFeO_3$ and $Fe_2O_3$ are produced as intermediates and these phases remain as impurities unless heated to high temperatures [27, 28], so the sol–gel methods have attracted the attention due to the lower synthesis temperature and finer and more homogeneous particles produced. This paper focus on preparation and characterization erbium substituted YIG thin films with formula $Er_{2.5}Y_{0.5}Fe_5O_{12}$ at different annealing temperatures prepared by sol-gel method, followed by spin coating technique.

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## 2. EXPERIMENTAL

The Er:YIG precursor sol was prepared by sol-gel method using reagent grade nitrates purchased from Aldrich, Milwaukee, WI, USA. Yttrium nitrate hexahydrate [$Y(NO_3)_3.6H_2O$, 99.95% purity], iron (III) nitrate nanohydrate [$Fe(NO_3)_3.9H_2O$, 98+% purity], erbium nitrate penthahydrate [$Er(NO_3)_3.5H_2O$, 99.9 %, metals basis] as raw materials, 2-methoxyethanol and acetic acid were used as solvents. $Fe(NO_3)_3.9H_2O$ and $Y(NO_3)_3.6H_2O$ were dissolved in the 2-methaxythanol and refluxed at 80 $^oC$ for 3 hours. The $Er(NO_3)_3.5H_2O$ dissolved in acetic acid was added gradually into the Fe–Y solution. Then the refluxing process was carried out for 3 hours. A small quantity of diethylamine was added to the mixture solution while the pH value was adjusted in the range of 2-3. After cooling down to room temperature, the solution was stirred for 3 days.

The gel was transformed into thin film form (onto quartz substrates) using the spin coating technique. The rate of the spinning process was 3500 rpm and it was done for

30 second. After the spinning process, the films were heated at 90 $^{o}$C for 2 hours to remove the residual solvents. Then the heat treatment was carried out: initial heating at 350 $^{o}$C for 15 min at a heating rate of 3$^{o}$C/min to burn-off the organic materials, followed by annealing process at 800, 900, and 1000 $^{o}$C for 2 hours at a heating rate of 4$^{o}$C/min to crystallize them.

The X-ray diffractometer (XRD) (20$^{o}$-80$^{o}$) scan were made to investigate the structural properties of the films. Magnetic measurements were carried out at room temperature using a vibrating sample magnetometer (VSM). The magnetic hysteresis loops were corrected for paramagnetic substrate contribution. The field emission scanning electron microscopic (FE-SEM) were carried out at the magnification of 250,000X to examine the surface quality of the films.

## 3. RESULTS & DISCUSSION

### 3.1 Phase characterization

Figure 1 shows the XRD spectrum of pure YIG films and $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films, treated at different temperatures for 2 hours. All of the films have only a single phase of garnet structure and complete crystallizing at 800 ℃. The slight shift of the peaks positions towards higher 2θ angles as the annealing temperature increased shows that Er atoms have been incorporated into the garnet structure [29]. The values of average lattice constants of the pure YIG films and $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films calculated from the XRD pattern are shown in figure 2.

### 3.2 Microstructure characterization

The microstructures of all of the $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films were studied using field emission scanning electron microscopic (FESEM) at the magnification of 250,000X as shown in figure (3a-c). The particle size obtained for these samples increases with the annealing temperature as shown in (Fig 5b). The images show that the particles stuck to each other and have highly agglomerated form, due to their high surface energy [30]. It is impossible to obtain accurate values of crystallite sizes due to aggregation of the particles.

### 3.3 Magnetic characterization

The magnetic properties of $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films annealed at different temperatures were measured using vibrating samples magnetometer (VSM) with a maximum applied field of 12 kOe at room temperature. Figure 4 shows the in-plane magnetization properties and hysteresis loop for all of the films. The hysteresis curves indicate that the Er-YIG films which annealed at different temperatures are soft magnetic materials.

The effect of annealing temperature on the magnetic properties of $Er_xY_{3-x}Fe_5O_{12}$ (x=0.0 and 2.5) films is investigated. The variation of saturation magnetization (Ms) with annealing temperature indicates that the Ms is increased from 12.70 to 37.40 (emu/cc) when the temperature increased from 800 to 900 °C then the Ms decreased to 17.05 (emu/cc) when the temperature increased to 1000 °C with the variation of films thicknesses (525,316 and 433 nm). These variations in the thicknesses of the films could be the reason for the (Ms) results (Fig.5a). The variation of coercivity (Hc) with different annealing temperature is shown in (Fig 5c) where the coercivity increased with the annealing temperature.

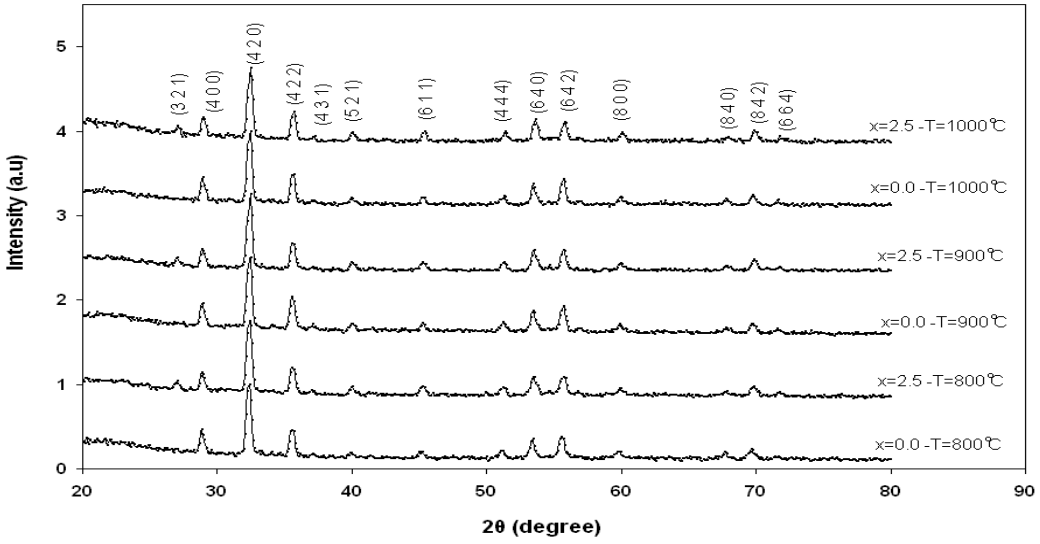


**Figure 1**: XRD pattern for $Er_xY_{3-x}Fe_5O_{12}$ films (x=0.0, 2.5), at different annealing temperatures.

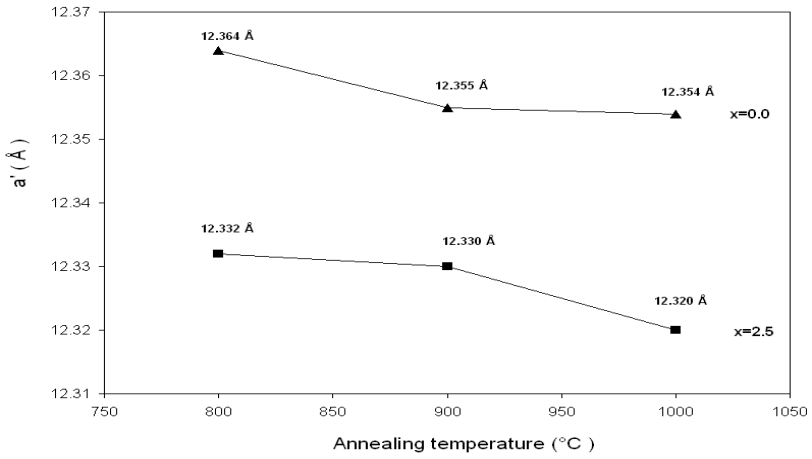


**Figure 2**: Variations of average lattice constant as a function of different annealing temperatures in $Er_xY_{3-x}Fe_5O_{12}$ films (x=0.0, 2.5).

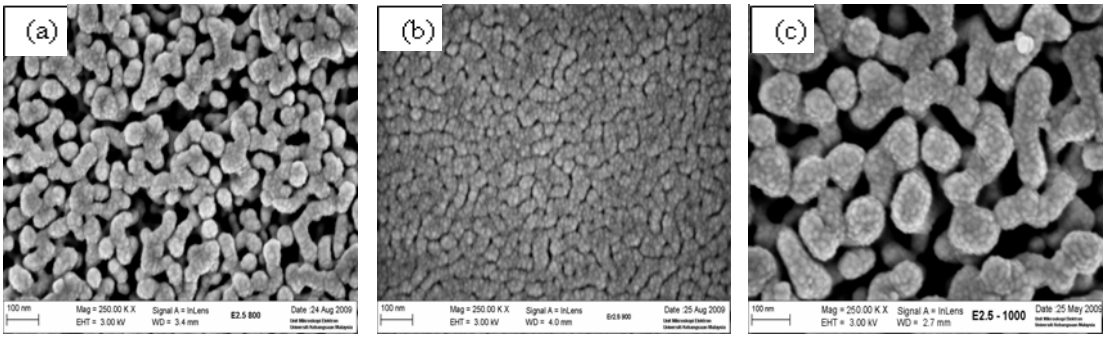


**Figure 3**: FE-SEM micrograph of $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films, at (a) 800℃ (b) 900℃ and (c) 1000℃.

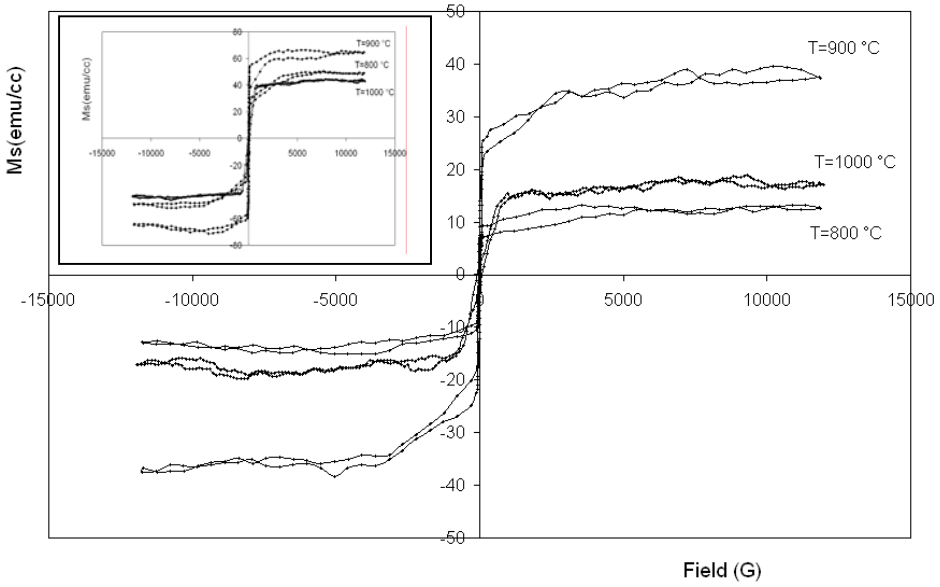


**Figure 4**: The hysteresis loops of the $Er_{2.5}Y_{0.5}Fe_5O_{12}$ films at different annealing temperatures; the insert shows hysteresis loops of the $Y_3Fe_5O_{12}$ films.

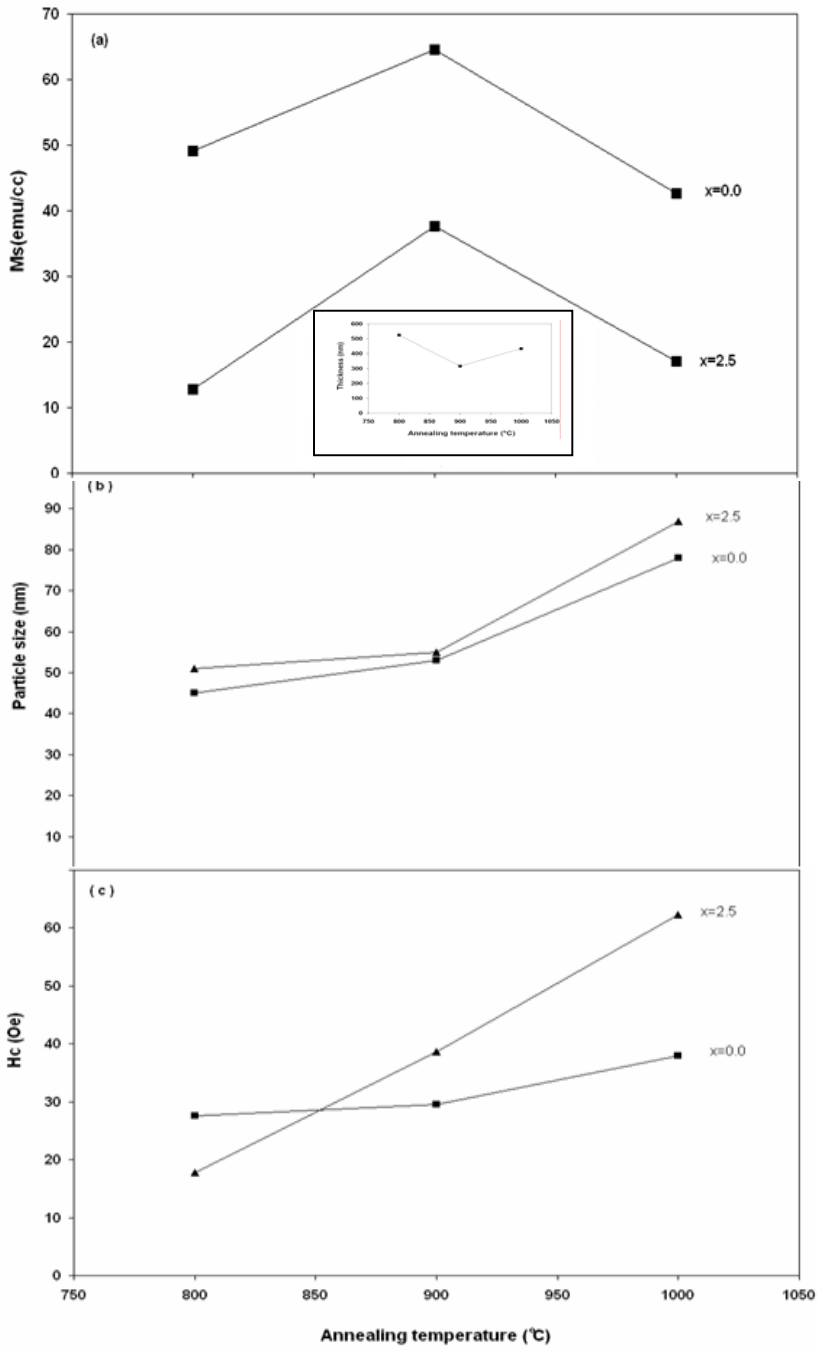


**Figure 5**: The variation of (a) saturation magnetization (insert films thicknesses with annealing temperature), (b) particle size, and (c) coercivity with different annealing temperature in $Er_xY_{3-x}Fe_5O_{12}$ films (x=0.0, 2.5).

## 4. CONCLUSION

The microstructural and magnetic properties of $Er_{2.5}Y_{0.5}Fe_5O_{12}$ sol-gel films have been investigated at different annealing temperatures. The single phase garnet structure was obtained and the crystallization begins at 800 °C. The samples particles sizes increased with the annealing temperature. The crystalline films are soft magnetic materials and the saturation magnetization increased then decreased with the variations of the annealing temperature. The coercivity also increased with the annealing temperature.

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