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Analyzing the Erbium-Doped Yttrium Iron Garnet's Structure Using the Standard Ceramic Approach

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ABSTRACT

Polycrystalline Erbium Doped Yttrium Iron Garnet nanoparticles (ErxY3−XFe5O12) where x=0.6 were discussed in this article and their typical ceramic synthesis method. Looking at what others have found for x=0.1 and x=0.3 may show you the correlation between Erbium concentration and sample properties. Blending by grinding and calcination at 1100°C for one hour yields the stoichiometric combination (ErxY3→XFe5O12), where x=0.6. The sample is ground into pellets and then sintered for two hours at 1400°C to ensure its purity. To examine the sample's finer points, scientists employed scanning electron microscopy (SEM) and X-Ray Diffraction (XRD). With the greatest peak in the (420) plane, XRD was able to produce the required phase for the sample. All peaks are sharp and well-defined with the exception of a single little YFe2O3 peak that has been deformed due to the strong bonding between iron and yttrium. The (420) plane's structural characteristics were studied. The lattice constant and d-spacing are found to decrease as the erbium concentration rises. The fact that Yttrium and Erbium have almost identical ionic radii also explains the lack of variation in crystallite size. All three samples have large, almost identical grains as a result of the high sintering temperature.

Keywords: Polycrystalline Erbium, Nanoparticles, Yttrium, Structural Characteristics

1. Introduction

In Latin, "ferrum" means "iron," and the word ferrite is an adjective that meaning "iron." Magnets with electrical and magnetic properties are known as ferrites. Iron oxide and other metal oxides are the main components of ferrites. People have known about the importance of ferrite material for a long time. Lodestones, which contain iron trioxide (Fe3O4), were invented by the Chinese and used in compasses for navigation in the early 12th century [1]. Garnets, ferrites with a spine, and ferrites with a hexagonal structure are the three most common varieties. The chemical formula for spinal ferrites is MOFe2O3 or MFe2O4, where M is the symbol for divalent cations such as Mn, Fe, Ni, Zn, Mg, and Cd. In the vertebral lattice, oxygen atoms form a tight pack structure. With M being (Pb, Ba, or Sr), the standard formula for a hexagonal structure is MFe12O19. Their primary use was as long-term magnetic components [2]. Magnetic rare earth garnets are often calculated using the formula M3Fe5O12, where M might be Yttrium or another rare earth ion. By exchanging one garnet for another, one may change the total magnetic moments of the stones. Substituted rare earth garnet is used in microwave systems and magnetic memories (bubble memory). Hexaferrites have a hexagonal structure, in contrast to the cubical spinel and garnet crystals. [3].

The microstructural features of ferrites were covered in this article. Bertaut and Forrat found a ferromagnetic insulator called yttrium iron garnet (Y3Fe5O12) in 1956[4]. Kittel called it the "fruit fly of magnetism" over 50 years ago [5]. In addition to its unique chemical make-up, yttrium iron garnet is characterized by its intricate crystal structure, which exhibits approximately cubic symmetry. A density of 5.17 g/cm3 was recorded for YIG [6]. A mere 310-5 is its magnetization damping, which is very low [7]. Its Curie temperature is at 560K, and its band gap is 2.85 eV, which is quite substantial. At 12.376 ±0.004°A, the YIG unit cell has a lattice constant. The yttrium iron garnet most probable space group is Oh10-Ia3d [8]. Eight $Y_3Fe_5O_{12}$ units, totaling 160 ions, make up the YIG unit cell, which is cubic in shape (Figure 1.1). Every unit of the YIG formula has three tetrahedral sites (d site), two octahedral sites (a site), and twenty-four Y^{3+}, forty Fe^{3+}, and ninety-six O^{2-} ions in three different configurations [9]. The Y^{3+} ions create an eight-cornered twelve-sided polyhedron by filling dodecahedral sites (c sites) and surrounding them with eight O^{2-} ions. The tetrahedral symmetry is achieved because the 24 Fe^{3+} ions are positioned in tetrahedral sites (d sites) and are encircled by four O^{2-} ions. The presence of six O^{2-} ions around the sixteen Fe^{3+} ions causes them to form octahedral symmetry, as seen in figure 1.1 [10].

Permeability is influenced by the microstructural feature known as grain size. According to research, the permeability increases with larger grains and fewer grain borders. Magnetic materials made of metals are a common example of when this principle holds. An 80% Ni-Fe alloy called Permalloy is one example of a high permeability metallic substance that supposedly does not care about grain size [12]. Variations in permeability are explained by the lengths between pores, according to newly published studies. Samples with consistently grown grains showed lower permeabilities than those with huge grains and porosity caused by excessive grain growth, even if the lengths between the pores were the same [13]. Another finding from the experiments is that a duplex structure might be the result of inadequate binder burn-off. The study's findings revealed the duplex structure's formation with a decline in magnetic properties, such as permeability and losses. [14].

Because of their high resistance, cheap cost, ease of synthesis, and higher magnetism qualities, ferrites are considered to be better magnetic materials than pure metals [15]. It is possible to construct sensors that detect electric currents, magnetic fields, and alternating and direct currents. The lead zirconate-titanate ($PbZr_{0.52}Ti_{0.48}O_3$) and nickel ferrite ($Ni_{1-x}Zn_xFe_2O_4$ with $x = 1-0.5$) sensors have shown remarkable performance. [16].

2. Methods and Materials

2.1 Chemical Used:

The PanReac application requires erbium oxide (Er_2O_3) powder with a purity level of 99.99%. Sigma Aldrich's iron oxide (Fe_2O_3) powder, which has a purity level of 99.99%. For the manufacture of Erbium substituted Yttrium Iron Garnet $Er_xY_{3-X}Fe_5O_{12}$, where x=0.6, Yttrium oxide Y_2O_3 powder of 99.99% purity was used, sourced from Sigma Aldrich.

Erbium substituted Yttrium iron garnet may be prepared using a number of different methods. The most common way to make polycrystalline solids from solid starting materials is via the solid state reaction pathway. Solids do not react with each other on ordinary time scales while they are at room temperature. The reaction can't happen quickly enough until they're heated to much higher temperatures, usually between 1000 and 1500 degrees Celsius. [17].

2.2 Working:

The steps below outline the technique for making Erbium-substituted Yttrium Iron

Garnet (Er: YIG).:For $Er_{0.6}Y_{2.4}Fe_5O_{12}$

Initial material	**Molecular weight(g)**	**Weight for chemical reaction**	**Weight for initial compound of 10g**
Y2O3	224.608g	269.9704g	3.5738g
Er2O3	381.601g	115.2466g	1.2164g
Fe2O3	158.588g	398.8176g	5.2182g
Total		784.0346g	10.0084g

Table 2.1: Figure for 10g composition of Er $_{0.6}Y_{2.4}Fe_5O_{12}$

A 10g Er-YIG sample containing 0.6 ppm of Erbium was subjected to the stoichiometric analysis. To make sure the powder components were mixed evenly, the sample was ground for two hours. To get an equivalent combination of Er ions in the end product, a homogeneous mixture is necessary. The powdered mixture is then heated in a furnace under regulated conditions after being mixed. After starting at ambient temperature, the heating profile calls for a slow ascent to 800°C (5°C/min), a 4-hour holding period at 800°C, and finally, a 5°C/min ascent to 1100°C. A furnace is used to cool the pellets to room temperature after sintering.

Crystal structure study and determining the substitution position of Erbium atoms in the YIG lattice of Erbium-substituted Yttrium Iron Garnet (Er: YIG) may be accomplished using XRD, one of many structural analysis methods. But the XRD parameters and settings may significantly impact the reliability of the results [18]. The correlation between the X-ray wavelength, diffraction angle, and lattice spacing of the YIG crystal is shown by Bragg's law. A number of structural parameters, including grain size, crystallinity, diffraction angle, and X-ray wavelength, may be explained by Bragg's equation [19], which also accounts for lattice spacing and phase preference in structure.

SEM is a tool for studying the material's surface morphology and microstructure. Yttrium iron garnet (YIG) samples doped with erbium have an Er: The scanning electron microscopy images showed several microstructural features, such as grain boundaries, pores, and fractures [20]. The grain size distribution was shown to be rather uniform, with an average grain size of around 2µm. It seemed like the material had a high degree of crystallinity since the grain boundaries were well defined. The discovery of pores and fractures suggests that the material may have developed specific faults during the sintering process. Photos taken with a scanning electron microscope (SEM) showed not just the material's microstructure but also its chemical make-up.[21, 22].

3. Results and Discussions

The XRD is used to find out the sample's phase. Constructive interference, the result of incident X-rays interacting with the sample, meets Bragg's law. In crystalline samples, Bragg's equation relates the wavelength of incoming X-rays to the diffraction angle and the distance between atomic planes. The connection was used to compute the cubic YIG lattice parameter a for all samples.

$$\{a \underset{2}{=} \sqrt{\frac{\lambda^2}{4\sin\theta}}\,(h^2 + k^2 + l^2)\} \qquad \text{Eq (3.1)}$$

Wherein λ (1.5405°A) is wavelength, θ is Bragg's angle in radian for given peaks, and [h k l] are Miller indices of respective peaks.

3.1X-ray Analysis:

The D8 Diffractometer is an XRD machine that observes the sample's XRD data. A range of $200 \leq 2\theta \leq 800$ was used to step scan and get the XRD data. Origin Pro Software is used to extract the data files (2016). Figure.3.1 displays the diffraction peaks for (Er0.6Y2.4Fe5O12) nanoparticles sintered at 1400°C. Table 3.1 displays the determined d-spacing and data list derived from Bragg's law. At planes of (420), the maximum intensity was noted at 2θ = 32.20. With the exception of a minor peak at 2θ = 33.14 in the provided sample of #YFeO3 (Yttrium Iron Pervoskite), the peaks are well defined and crisp. This is because of the strong bonding between the iron and yttrium atoms.

Position (2Θ)	Height(Intensity)	FWHM(β) (Radians)	d-spacing (^{0}A)
28.92	142.1	0.00377	3.084489
32.39	328.9	0.003211	2.76561
35.58	207.1	0.003810	2.5247
53.43	223.8	0.004255	1.71653
55.61	187.8	0.004557	1.652459
69.60	153.1	0.003584	1.344451

Table 3.1: Peak list of Er0.6Y2.4Fe5O12 where x = 0.6

Utilizing the Scherrer formula and the properties of the (420) XRD peaks, we can get the average crystallite size D.

$$D = \frac{C\lambda}{\beta \cos\theta}$$

λ= **0.15405nm** is the wavelength incident x-ray.

β is relative value of full width half maximum (FWHM) of diffraction peak (420).

θ Is Bragg's angle at 2θ scale. Angles are taken in radian.

The molecular weight, d-spacing, crystallite size, and lattice parameter of Er0.6Y2.4Fe5O12 in the (420) plane are shown in table 3.2.

3.2 Table Physical properties of Er0.6Y2.4Fe5O12 at (420) plane

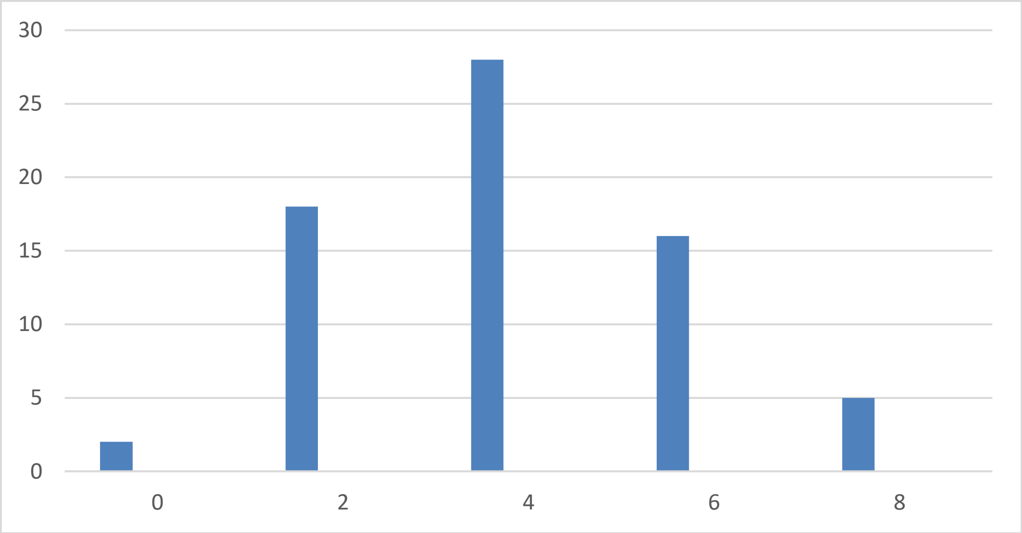
X	Position (2Θ)	Molecular Weight(g)	d-spacing (^{0}A)	Crystallite size(nm)	Lattice Constant(^{0}A)
0.6	32.41	784.8926	2.75436	45.96794	12.21546

3.2 SEM analysis

Using a scanning electron microscope (SEM), the sample's grain size was identified. Below is the SEM micrograph of Er0.6Y2.4Fe5O12. The grains in the micrograph are homogeneous in character and well-defined. The grain of Erbium (0.6) YIG is clearly visible in this 10μm magnified micrograph. Table 3.3 displays the results of measuring the grain size using Image J software and displaying the count rate with a histogram.

Length (μm)	Mean	Minimum	Median	Maximum
	3.88462	0.889	3.599	8.241

Table 3.3: Variation of Grain size (μm) of $Er_{0.6}Y_{2.4}Fe_5O_{12}$



Length

Fig-3.2 histogram of $Er_xY_{3-x}Fe_5O_{12}$

Evaluation of ErxY3-xFe5O12 at x=0.1,0.3, and 0.6 and Comparison of Results. The outcomes were evaluated by comparing the performance of Erbium replaced Yttrium Iron Garnet (ErxY3-xFe5O12) at a concentration of x = 0.6 to that of colleagues who utilized the same sample but varied the concentration, for example, by taking x = 0.1 or 0.3.

Table 3.4: Physical properties of $Er_xY_{3-x}Fe_5O_{12}$ at (420) plane

X	**Position (2Ө)**	**Molecular Weight(g)**	**d-spacing (^{0}A)**	**Crystallite size (nm)**	**Lattice Constant (^{0}A)**
0.1	**32.44**	**745.79621**	**2.768411**	**45.95410**	**12.27741**
0.3	**32.26**	**761.4147**	**2.762115**	**48.69962**	**12.24591**
0.6	**32.84**	**784.9652**	**2.74155**	**45.95271**	**12.21056**

For the (420) plane, the lattice parameters are determined from the diffraction pattern of ErxY3-xFe5O12 with x = 0.1, 0.3, and 0.6 using the following equation:

$$a = \sqrt{\frac{\lambda^2}{4\sin^2\theta}\{(h^2 + k^2 + l^2)\}} \qquad \text{Eq (3.2)}$$

At a single sintering temperature of 1400°C, the lattice constant decreases as the Erbium concentration increases. This is because the dodecahedral sites of the YIG unit cell are filled by somewhat bigger Yttrium ions, which replace the smaller Erbium ions with ionic radiuses of 1.04°A and 1.03°A, respectively.

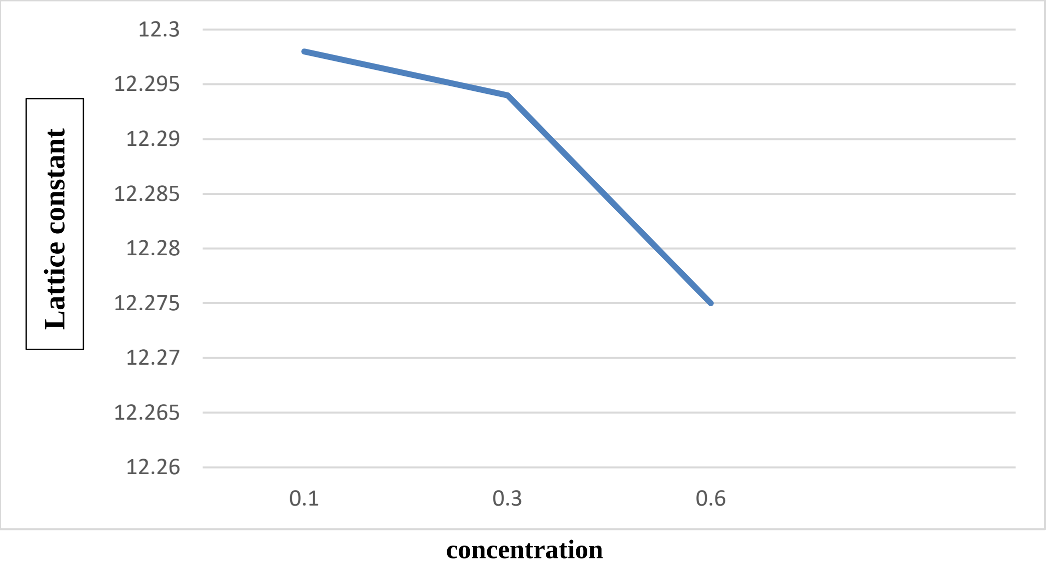


Figure 3.4 variation of lattice parameter of $Er_xY_{3-x}Fe_5O_{12}$ with Erbium (X).

The reduction in d-spacing may be attributed to the lattice constants decreasing, as seen in table 3.4 and Figure 3.5. Adding to this, the replacement of Erbium causes the XRD peaks to move to higher angles (2þ), which in turn causes a drop in d-spacing, as determined by Bragg's law.

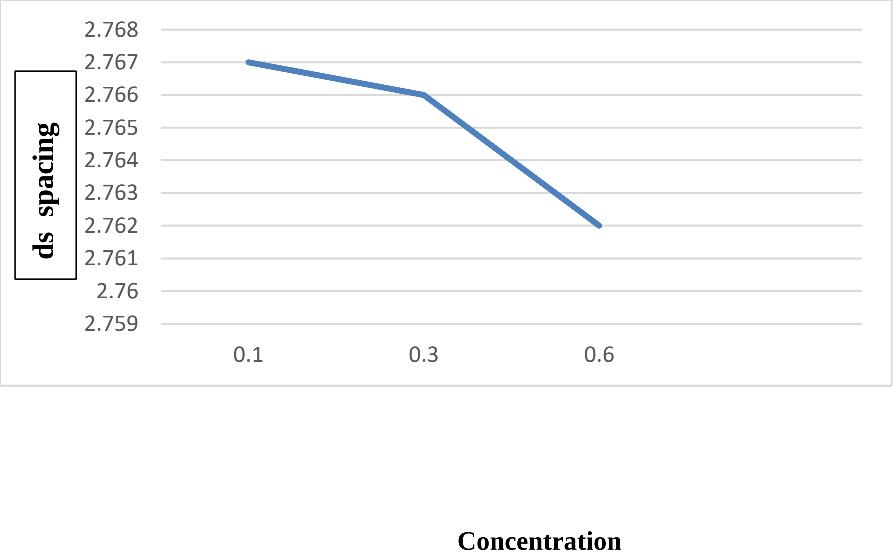


Figure 3.5 Variation of d-spacing of $Er_xY_{3-x}Fe_5O_{12}$ with Erbium (x)

The almost equal effective ionic radii of the Erbium ion (0.89°A) and the Yttrium ion (0.90°A) are likely responsible for the nearly same crystallite sizes at the same sintering temperature of 1400°C. The parameters in table 3.4 are used to compute the size of the crystallite for the (420) peak Scherrer formula. In figure 3.6, you can see the differences.

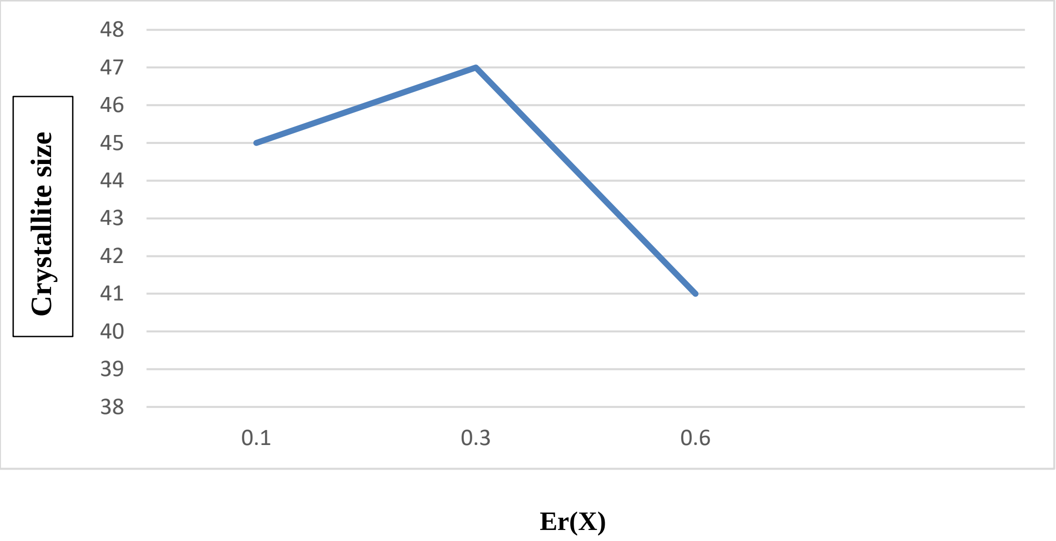


Figure 3.6 Variation of Crystallite size (nm) of $Er_xY_{3-x}Fe_5O_{12}$ with Erbium (x)

In Figure 3.7, we can see that the average grain sizes for all of the samples are somewhat smaller than before. This might be because to structural deformation, namely the shortening of the Fe-O bond as a result of erbium substitution at the same sintering temperature of 1400°C. Due of the very high sintering temperature of 1400°C, the grain sizes on average are somewhat big.

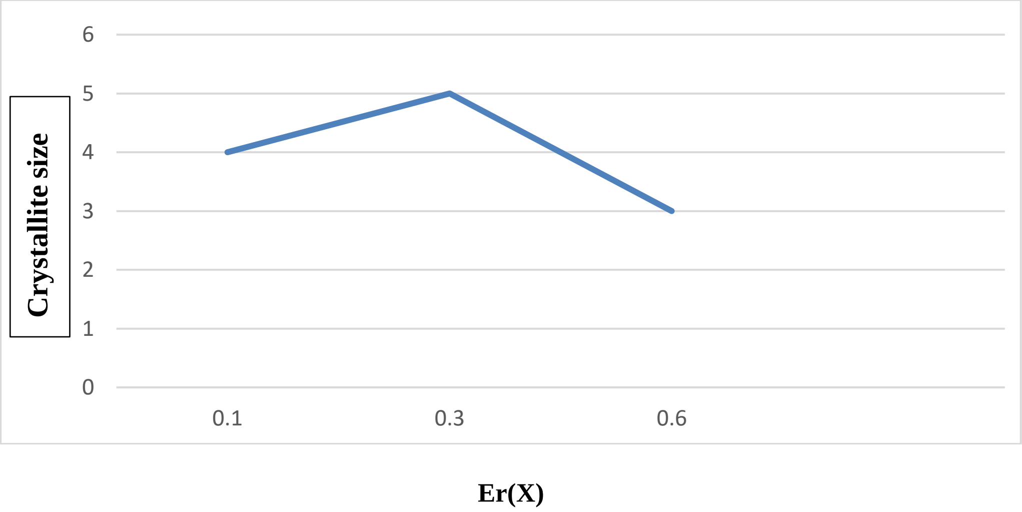


Figure 3.7: Variation of Grain size (µm) of $Er_xY_{3-x}Fe_5O_{12}$ with Erbium (x)

1. Summary and conclusions.

This article details the synthesis and physical properties of nanoparticles doped with erbium and yttrium iron garnet, denoted as ErxY3−XFe5O12, where x=0.6.It was made utilizing powdered oxides (Y2O3, Er2O3, Fe2O3) as a precursor in the conventional ceramic technique of synthesis. Cubic YIG phases have formed, as seen by the XRD pattern. It was found that the (420) plane has the highest peak.

Because of the strong interaction between iron and yttrium, all samples show a tiny distortion peak of YFeO3 (Yttrium Iron Perovskites). We learned from our XRD and SEM analyses that,

A change in the lattice constant was caused by the fluctuation in the concentration of erbium in ErxY3-x Fe5O12. The lattice constant is reduced as the concentration of erbium rises because d-spacing is decreased. The substitution of lower ionic radii (0.89°A) of Erbium ions for larger Yttrium ions at the same sintering temperature of 1400°C causes the lattice constant to drop as the Er concentration increases. By increasing the length of the Fe-O bond and, by extension, the d-spacing, the substitution of Yttrium into a cubic unit cell raises the lattice constant. As the Erbium concentration increases, the XRD peaks shift to higher angles (2F). The relationship between angle and d-spacing is inverse, as stated by Bragg's law. The drop in d-spacing is caused by the XRD peaks shifting to a higher angle.

As the erbium concentration increases from 0.1 to 0.6, the size of the crystallites varies non-linearly. At 1400°C, it varies between 45.94 and 48.65 nanometers. The almost identical ionic radii of Y and Er account for the little discrepancy in crystallite size.Grain sizes for Er content were found to vary within the range of (4.20951µm - 3.85462µm), according to the SEM result, which

reveals small non-linear variations. Due to the approximately similar ionic radii of Yttrium 'Y' and substituted Erbium 'Er', it is not possible to discern the huge average grain sizes.

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