Chapter IV
Results and Discussion of Europium Substituted Yttrium Iron Garnet

4.1 Introduction

Yttrium Iron Garnet (YIG) and substituted YIG have been investigated extensively because of their technical importance and the ease with which properties can be tailor made to suit various applications [1-4]. Number of ions can be substituted in YIG which occupy the lattice site determined primarily by their ionic radii [3]. It is found that certain ions occupy strictly a particular lattice sites while some others can occupy both tetrahedral (d) and octahedral [a] sites present in YIG structure [5, 6].

Substituted rare earth iron garnets show wide variety of interesting magnetic properties [1,5]. Theoretical models, one based on Neel’s model [7], another suggested by de-Geenes [8] based on Yafet-Kittel models [9], and one
suggested by Gilleo [10] have been used to explain the magnetic behaviour of YIG.

Yttrium iron garnet (YIG) is a cubic crystal (space group $Ia_3d-O_h^s$) characterized by the following structural-chemical formula

$$\left[\text{Y}^{3+}\right]^6\cdot\left[\text{Fe}^{3+}\right]^6\cdot\left[\text{Fe}^{3+}\right]^6\cdot\left[\text{O}^{2-}\right]$$

The trivalent iron cations are distributed over two different sets of lattice sites; Octahedral (a) and tetrahedral (d) sub-lattice in which each cation is tetrahedrally surrounded by oxygen anions and on Octahedral (a) sub-lattice where the co-ordination is octahedral. The yttrium ions are in dodecahedra (C) sites characterized by a dodecahedral oxygen environment.

A large number of cations can be substituted at yttrium sites or at iron ($\text{Fe}^{3+}$) sites enabling a wide variation in the properties of garnets. The magnetic properties of gallium and aluminum [10, 11] substituted yttrium iron garnet have been reported in the literature [11]. Similarly, the structural, electrical and magnetic properties of non-
magnetic Si and Ge substituted YIG, have also
been reported in the literature [12,13].

In the literature, studies on the
properties of pure yttrium iron garnet [14],
and pure europium iron garnet [15] have been
reported. But, to our knowledge no reports are
available in the literature on the structural,
electrical, magnetic properties of europium
substituted yttrium iron garnet.

Since yttrium is non-magnetic whereas
europium is a magnetic having magnetic moment
$0.74 \mu_B$. Therefore, it will be interesting to
see the effect of substitution of magnetic Eu$^{3+}$
ions in the lattice of pure yttrium iron garnet
at yttrium site.

In the present work, studies on the
synthesis and structural, electrical and
magnetic properties of Eu$^{3+}$ substituted yttrium
iron garnet have been carried out and the
results obtained on $Y_{3-x}Eu_xFe_5O_{12}$ ($x = 0.0$ to $0.5$)
garnet system are presented in this chapter.

The structural characterization of all the
samples of the series $Y_{3-x}Eu_xFe_5O_{12}$ ($x=0.0$ to $0.5$)
were done by X-ray diffraction technique. The magnetic properties like saturation magnetization ($\sigma_s$) and magneton number ($n_B$), Curie temperature ($T_C$) were investigated by means of magnetization measurements and a.c. susceptibility technique. The d.c. electrical resistivity of all the samples was investigated as a function of composition and temperature using two-probe technique.

4.2 Experimental procedure

Samples of $Y_{3-x}Eu_xFe_5O_{12}$ ($x = 0.0$ to $0.5$) were prepared by using high purity $Y_2O_3$, $Eu_2O_3$, $Fe_2O_3$ and using ceramic technique.

The oxides were mixed thoroughly in stoichiometric proportions to yield the desired composition and wet ground. The mixture was dried and presintered at $1050^\circ C$ for 24 hours in air and cooled to room temperature. The powder was reground and pelletized using hydraulic press. The cylindrical pellets were finally sintered at $1350^\circ C$ for 24 hours and slowly
cooled to room temperature at the rate of 2°C per minute to obtain garnet phase.

The powder X-ray diffractograms (XRD pattern) for all the samples were recorded at room temperature using Philips X-ray diffractometer (Model PW-3710). XRD patterns were obtained in the 2θ range of 20° to 80° and using Cr-Kα radiation.

The magnetization measurements were carried out using high field hysteresis loop technique [16] at 300K. Using hysteresis loop technique the saturation magnetization (σ_s) and magneton number (n_B) were obtained. The Curie temperature of all the samples were determined using a.c. susceptibility measurements. The measurements of a.c. susceptibility were carried out in the temperature range 300-800K using double coil set up [17].

The d.c. electrical resistivity measurements were carried out in the temperature range 300-800K using two-probe technique on the disc shaped pellets of 10 mm diameter and 3 mm thickness. For good ohmic
contact a silver paste was applied on both the surface of the pellets. The measurements were recorded in a regular interval of 20K. The temperature of the samples was sensed by Cr-Al thermocouple with an accuracy of ± 1°C.

4.3 Results and discussion

4.3.1 X-ray diffraction

X-ray powdered diffraction patterns of the series $Y_{3-x}Eu_xFe_5O_{12}$ with $x = 0.0$ to 0.5 are shown in Fig. 4.1 and Fig. 4.2. XRD patterns indicated that the materials formed in a single phase with a cubic garnet structure. All the peaks of the XRD patterns were indexed. The inter-planer spacing (d) for the recorded peak was calculated according to Bragg’s law for all the composition under investigation. The values of inter-planer spacing ‘d’ for different (hkl) are listed in Table 4.1.

The XRD data was used to evaluate unit cell dimensions. The lattice constant ‘a’ was determined using the following relation,

$$a = d\sqrt{N} \quad (4.1)$$
where,

\[ d \text{ is inter-planer spacing} \]

\[ N = (h^2+k^2+l^2), \quad (hk1) \text{ are Miller indices} \]

The values of lattice constant ‘\(a\)’ determined from XRD data with an accuracy of \(\pm 0.002\text{Å}\) are given in Table 4.2. The variation of lattice constant with composition has been studied. Fig.4.3 depicts the variation of lattice constant ‘\(a\)’ with Eu substitution. It is observed from Fig.4.3 that lattice constant increases with Eu substitution. The lattice constant increases linearly and obeys Vegard’s law [18]. The increasing behaviour of lattice constant with Eu substitution can be understood from the knowledge of ionic radii of the constituent ions. In the present series of garnet \(Y_{3-x}\text{Eu}_x\text{Fe}_5\text{O}_{12}\), yttrium ions of smaller ionic radii (0.89 Å) are replaced by \(\text{Eu}^{3+}\) of larger ionic radii (0.95 Å) and therefore lattice constant of present garnet system increases with Eu substitution. Similar behaviour of lattice constant with composition ‘\(x\)’ was observed in \(Y_{3-x}\text{Fe}_5\text{O}_{12}\) [19].
The XRD line width and particle size are connected through the Scherrer equation [20]

\[ t = \frac{0.9\lambda}{B \cos \theta_i} \] (4.2)

where,

- \( t \) is particle diameter,
- \( \lambda \) is the wavelength and
- \( B \) is a measure of the broadening of diffraction line due to size effect.

The values of particle size obtained from above relation are summarized in Table 4.2. It is clear from Table 4.2 that all the samples have almost same particle size.

The X-ray density of all the samples of the series \( Y_{3-x}Eu_xFe_5O_{12} \) have been calculated from the molecular weight and volume of the unit cell using the formula,

\[ d_s = \frac{8M}{Na} \text{ gm} \cdot \text{cm}^3 \] (4.3)

where,

- \( M \) is the molecular weight,
- \( N \) is the Avogadro's number and
- \( a \) is the lattice constant.
The values of X-ray density are listed in Table 4.3. It is observed from Table 4.3 that X-ray density increases with increase in Eu substitution.

The variation of X-ray density with composition 'x' is shown in Fig.4.4. It is clear from Fig.4.4 that X-ray density increases with increase in Eu substitution.

In the present series $Y_{3-x}\text{Eu}_x\text{Fe}_5\text{O}_{12}$, the lattice constant increases with Eu substitution. Therefore, naturally the X-ray density should decrease. However, in the present study X-ray density increases instead of decreasing. This is because of the fact that the increase in mass overtakes the increase in volume of the unit cell. The values of the molecular weight of all the samples of the series $Y_{3-x}\text{Eu}_x\text{Fe}_5\text{O}_{12}$ are given in the Table 4.3. The bulk density of all the samples of the series $Y_{3-x}\text{Eu}_x\text{Fe}_5\text{O}_{12}$ was obtained using the values of mass and dimensions of the pellets. The values of bulk density are presented in Table 4.3.
From Table 4.3 it is clear that the bulk density is of the order of 95% to that of X-ray density.

The percentage porosity (P) of all the samples was calculated using the relation

\[ P = \left( 1 - \frac{d}{d_x} \right) \times 100 \% \]  

(4.4)

where,

- d is bulk density
- \( d_x \) is X-ray density

The values of porosity of all the samples are listed in Table 4.3. The porosity values indicate that the samples are dense and compact.

### 4.3.2 Cation distribution

The study of the distribution of cations among the available sites in ferrites is very much important in understanding the structural and magnetic behaviour of ferrite. The cation distribution can be obtained from X-ray diffraction [21], Mossbauer spectroscopy [22],
neutron diffraction [23] and magnetization method [24].

In garnet cations are distributed at three sub-lattices namely dodecahedral (c), octahedral (a) and tetrahedral (d). The total magnetic moments on the 'a' and 'd' ions are aligned anti-parallel and moments on the 'c' ions are anti-parallel to those on the 'd' ion. Thus for the formula 

\[(3M_{2}O_{3})^{c}(2Fe_{2}O_{3})^{a}(3Fe_{2}O_{3})^{d}\]

the arrangement is \(6Fe^{d}, 4Fe^{a}, 6M^{c}\). The net moment \(m\) (in Bohr magnetons per unit formula) is

\[M = 6m_{c} - (6m_{d} - 4m_{a}) = 6m_{c} - 10\mu_{B}\] (4.5)

Assuming a moment of \(5\mu_{B}\) per Fe ion. In terms of the unit formula \(M_{3}Fe_{2}Fe_{3}O_{12}\) eq. (4.5) becomes

\[M = 3m_{c} - 5\mu_{B}\] (4.6)

In general, the cation distribution of yttrium iron garnet is expressed as,

\[\{Y_{3}\}^{a} [Fe_{2}]^{a} (Fe_{3})^{d} O_{12}\] (4.7)

Comparing the site preference energy of the constituent ions and assuming that \(Eu^{3+}\) has tendency to occupy dodecahedral C-sites, the
cation distribution of the present garnet system $Y_{3-x}Eu_xFe_3O_{12}$ can be written as,

$$\{Y_{3-x}Eu_x\}^c [Fe_2]^a (Fe_3)^d O_{12} \quad (4.8)$$

The average ionic radius of dodecahedral (c), octahedral (a) and tetrahedral (d) can be estimated using the relation [25]

$$r_d = x(Fe^{3+}) \cdot r(Fe^{3+})$$  \hspace{1cm} (4.9)

$$r_a = \frac{1}{2} \left[ x(Fe^{3+}) \cdot r(Fe^{3+}) + x(Fe^{2+}) \cdot r(Fe^{2+}) \right]$$  \hspace{1cm} (4.10)

$$r_c = x(Y^{3+}) \cdot r(Y^{3+}) + x(Fe^{3+}) \cdot r(Fe^{2+})$$  \hspace{1cm} (4.11)

where, x and r represents concentration and ionic radius of cations on the respective sites. Using above equations (4.9, 4.10 and 4.11), considering the ionic radii of yttrium ($Y^{3+}$) as (0.89 Å), Europium $Eu^{3+}$ (0.95 Å) and ferric ($Fe^{3+}$) as (0.64 Å) and the cation distribution formula given by eq. 4.8, the radius of dodecahedral (c), octahedral (a) and tetrahedral (d) were calculated and the values were summarized in Table 4.4.

The theoretical lattice constant of all the samples were also estimated using the formula [25],

$$a_{th} = b_1 + b_2 r_c + b_3 r_d + b_4 r_a + b_5 r_c r_d + b_6 r_c r_a$$  \hspace{1cm} (4.12)
where, $b_1 - b_6$ are exchange interaction in agreement with earlier report [26].

The values of theoretically obtained lattice constant are given in the Table 4.2. It is observed from Table 4.2 that the theoretical and experimental values of lattice constant are in good agreement, which indirectly suggest that assumed cation distribution is correct. The values of theoretical and experimental lattice constant are in good agreement.

### 4.3.3 Magnetization measurements

The values of saturation magnetization $\sigma_s$ and magneton number $n_B$ (the saturation magnetizations per formula unit in Bohr magneton) at room temperature were obtained from hysteresis loop technique [16] for all the samples of the series $Y_{3-x}Eu_xFe_5O_{12}$.

The values of saturation magnetization $\sigma_s$ and magneton number $n_B$ are presented in Table 4.5. From the field dependence of magnetization and observed magnetic moment (Table 4.5), it can be seen that both
saturation magnetization and magneton number decreases with increase in 'x'. The variation of magneton number with Eu substitution is represented in Fig. 4.5. It is evident from Fig. 4.5 that the decrease in 'n_B' is sharp up to \( x = 0.3 \). For \( x > 0.3 \), the decrease in 'n_B' is rather slow. This indicates that ferrimagnetic behaviour decreases with increasing Eu substitution. In the present series of \( Y_{3-x}Eu_xFe_5O_{12} \), the magnetic Eu\(^{3+}\) ion occupies dodecahedral (c) site. Though, the magnetic moment of dodecahedral (c) site increases because of magnetic Eu substitution (0.74 \( \mu_B \)), but the net magnetic moment of the garnet system \( Y_{3-x}Eu_xFe_5O_{12} \) decreases. Hence, we observe decrease in magnetic moment of \( Y_{3-x}Eu_xFe_5O_{12} \) with Eu substitution. The value of magnetic moment of pure yttrium iron garnet (YIG) i.e. for \( x=0.0 \) in the present series, calculated in this manner agrees fairly well with the reported values [27]. According to Neel’s model, the magnetic moment per formula unit in \( \mu_B \), \( n_B^N \) at 0K is expressed as [28]
\[ n^u = M_a(x) - (M_d - M_c) \]

where, \( M_d \), \( M_a \) and \( M_c \) are the \( d \), \( a \) and \( c \) sub-lattice magnetic moment in \( \mu_B \) respectively.

Using the cation distribution (eq. 4.8) and taking ionic magnetic moments of yttrium \((Y^{3+})\), europium \((Eu^{3+})\) and ferric \((Fe^{3+})\) as \( 0 \mu_B \), \( 0.74 \mu_B \) and \( 5 \mu_B \) respectively, we have calculated the Neel’s magnetic moment of all the samples of \( Y_{3-x}Eu_xFe_5O_{12} \). The calculated moment values of all the samples are given in Table 4.5. It is clear from Table 4.5 that calculated magneton number decreases with increasing in Eu substitution. The variation of calculated magnetic moment is shown in Fig. 4.5. Since the calculated Neel’s moment are valid at 0K, we have calculated the ratio of \( n_a(x)/n_a(0) \) for \( x = 0.0 \) to 0.5 and the values are presented in Table 4.5.

Fig. 4.6 depicts the variation of normalized calculated and observed magneton number with Eu substitution.
4.3.4 A.C. susceptibility measurement

The thermal variation of a.c. susceptibility of all the sample have been studied for all the samples in the temperature range 300-800K using double coil set up.

The plots of a.c. susceptibility ($\chi_T/\chi_{RT}$) (RT is room temperature) against temperature (T) for all the samples are shown in Fig.4.7 and Fig.4.8. The plots exhibit normal ferrimagnetic behaviour, which decreases with the addition of Eu substitution. These plots are used to determine Curie temperature of the samples. The Curie temperature obtained from a.c. susceptibility data is given in Table 4.5 for all the sample.

The dependence of Curie temperature with composition ‘x’ is depicted in Fig. 4.9. It is observed from Fig. 4.9 that $T_c$ decrease slowly with the increase in Eu substitution.

The decrease in Curie temperature is attributed to decrease in magnetic linkages.
4.3.5 D.C. electrical resistivity

The d.c. electrical resistivity measurements of all the samples were carried out using two-probe technique in the temperature range 300-800K for all the samples of $Y_{3-x}Eu_xFe_5O_{12}$. The variation of electrical resistivity $\rho$ as a function of temperature for all the samples are shown in Fig. 4.10, Fig. 4.11 and Fig. 4.12.

The nature of these plot suggest that the curve is divided in two parts corresponding to ferrimagnetic and paramagnetic region. The plot obeys the exponential relation,

$$\rho = \rho_0 \exp \left( \frac{E_g}{kT} \right)$$  \hspace{1cm} (4.13)

where,

- $E_g$ is activation energy,
- $\rho_0$ is the temperature dependent factor and
- $k$ is Boltzman constant.

All the plots exhibit a kink near a temperature, which may corresponds to the Curie temperature of the samples.
Using the above equation and the plots of $\log \rho$ versus reciprocal of temperature, the activation energy of all the samples has been calculated and the values were presented in Table 4.6. The variation of activation energy with Eu substitution as shown in Fig. 4.13. From Fig. 4.13 it is observed that activation energy is decreases with increase in Eu substitution.

The analogous behaviour of $\log \rho$ versus $1000/T$ was observed in $R_{3-x}Ca_{2-x}Fe_{5-x}Me_xO_{12}$ (where, $R = Y, Sm, Eu, Gd$ and $Me = Sb$ and Nb) garnet system.

### 4.4 Conclusions

All the samples of the series $Y_{3-x}Eu_xFe_5O_{12}$ synthesized by ceramic technique possesses single-phase cubic garnet structure, which is evidenced by X-ray diffraction data. The lattice parameter of pure YIG is in good agreement with the reported data [27]. The lattice constant increases with Eu substitution. The cation distribution of the
present garnet system suggest that Eu$^{3+}$ occupies dodecahedral (C) site and is in agreement with that of other garnet system [29].

The magnetization results suggest that magneton number decreases with Eu substitution. The observed and calculated magneton number shows discrepancy in their values.

The plots of $\chi_T/\chi_{RT}$ (RT = room temperature) exhibit normal ferrimagnetic behaviour which reduces with Eu substitution. The Curie temperature decreases with increase in Eu substitution. The Curie temperature of pure YIG ($x = 0.0$) is in good agreement with those reported by others.

The resistivity of all the samples decreases with increase in temperature. The activation energy obtained from log $\rho$ versus $1000/T$ plots is in the reported range.
REFERENCES


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Table 4.1

Miller indices (hkl) and inter-planer spacing (d) for Y$_{3-x}$Eu$_x$Fe$_5$O$_{12}$ system (x = 0.0 to 0.5)

<table>
<thead>
<tr>
<th>Plane (hkl)</th>
<th>Inter-planer spacing 'd' (Å)</th>
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<tr>
<td></td>
<td>x=0.0</td>
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<tr>
<td>(400)</td>
<td>4.009</td>
</tr>
<tr>
<td>(422)</td>
<td>4.905</td>
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<tr>
<td>(440)</td>
<td>5.699</td>
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<tr>
<td>(640)</td>
<td>7.228</td>
</tr>
<tr>
<td>(642)</td>
<td>7.500</td>
</tr>
</tbody>
</table>
Table 4.2

Lattice constant (a), particle size (t) and theoretical lattice constant (a_{th}) for Y_{3-x}Eu_xFe_5O_{12} system (x = 0.0 to 0.5)

<table>
<thead>
<tr>
<th>Composition 'x'</th>
<th>Lattice constant 'a' (Å)</th>
<th>Particle size 't' (Å)</th>
</tr>
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<tr>
<td></td>
<td>Obs.</td>
<td>The.</td>
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<tr>
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</tr>
<tr>
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<td>12.329</td>
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<tr>
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<td>0.4</td>
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<td>12.336</td>
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<tr>
<td>0.5</td>
<td>12.349</td>
<td>12.339</td>
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</table>
Table 4.3

Molecular weight (M), X-ray density ($d_x$), Bulk density ($d$) and Porosity for $Y_{3-x}Eu_xFe_5O_{12}$ system ($x = 0.0$ to $0.5$)

<table>
<thead>
<tr>
<th>Composition 'x'</th>
<th>Mol. wt. M (gm)</th>
<th>X-ray density 'd_x' (g/cm³)</th>
<th>Bulk density 'd' (g/cm³)</th>
<th>Porosity 'P' %</th>
</tr>
</thead>
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<tr>
<td>0.0</td>
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<td>5.339</td>
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<td>769</td>
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<td>5.382</td>
<td>0.80</td>
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Table 4.4

Ionic radii of dodecahedral ($r_c$), octahedral ($r_o$), tetrahedral ($r_d$) sites and average radius for $Y_{3-x}Eu_xFe_5O_{12}$ system ($x = 0.0$ to $0.5$)

<table>
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<tr>
<th>Composition X</th>
<th>$r_c$ (Å)</th>
<th>$r_o$ (Å)</th>
<th>$r_d$ (Å)</th>
<th>Average radius (Å)</th>
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<td>0.640</td>
<td>0.640</td>
<td>0.725</td>
</tr>
<tr>
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<td>0.896</td>
<td>0.640</td>
<td>0.640</td>
<td>0.725</td>
</tr>
<tr>
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<td>0.640</td>
<td>0.726</td>
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<td>0.640</td>
<td>0.640</td>
<td>0.727</td>
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Table 4.5

Saturation magnetization ($\sigma_s$), magneton number $n_B$ ($\mu_B$) and Curie temperature for $Y_{3-x}Eu_xFe_5O_{12}$ system ($x = 0.0$ to $0.5$)

<table>
<thead>
<tr>
<th>Com. 'x'</th>
<th>$\sigma_s$ (emu/g)</th>
<th>Magneton number $n_B$ ($\mu_B$)</th>
<th>Magneton number $n_B$ ($\mu_B$) normalized</th>
<th>Curie Temp. $T_c$ K</th>
</tr>
</thead>
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<tr>
<td>0.0</td>
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<td>1.000</td>
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<td>4.778</td>
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</tr>
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<td>0.2</td>
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<td>4.556</td>
<td>0.657</td>
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<tr>
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<td>18.65</td>
<td>2.527</td>
<td>4.334</td>
<td>0.568</td>
</tr>
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<td>16.58</td>
<td>2.265</td>
<td>4.112</td>
<td>0.509</td>
</tr>
<tr>
<td>0.5</td>
<td>15.54</td>
<td>2.141</td>
<td>3.900</td>
<td>0.481</td>
</tr>
</tbody>
</table>
Table 4.6

Activation energy \( (E_g) \) for \( Y_{3-x}Eu_xFe_5O_{12} \) system

\( (x = 0.0 \text{ to } 0.5) \)

<table>
<thead>
<tr>
<th>Composition ( 'x' )</th>
<th>Activation energy ( E_g ) (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( E_f )</td>
</tr>
<tr>
<td>0.0</td>
<td>0.23</td>
</tr>
<tr>
<td>0.1</td>
<td>0.21</td>
</tr>
<tr>
<td>0.2</td>
<td>0.29</td>
</tr>
<tr>
<td>0.3</td>
<td>0.19</td>
</tr>
<tr>
<td>0.4</td>
<td>0.21</td>
</tr>
<tr>
<td>0.5</td>
<td>0.21</td>
</tr>
</tbody>
</table>