CHAPTER 5

Results and Analysis

5.1 ZnS nano particles synthesis by chemical precipitation method

Five samples were prepared to investigate the TL properties of ZnS:Mn nanophoshor each sample has a mass of 30 mg. The first three samples have Mn concentration of 1M,2M and 3M respectively. Sample four does not contain Mn and sample five is a commercial ZnS powder.

The Annealing process takes place in a furnace of model Vulcan 3-400HTA. The furnace was programmed so that the samples were heated gradually to $500^{\circ}C$, $300^{\circ}C$ and $100^{\circ}C$. Each temperature was kept constant for 30 minutes. The heating rate was $40^{\circ}C/\min$. Then the samples were exposed to a gamma ray by using Gamma cell 220 which uses ^{60}Co as a source of gamma (γ) rays. The samples were exposed to a dose equal to 18 Gy, 36 Gy, 54 Gy, 72 Gy and 90 Gy then the thermoluminescence properties were studied by using Harshaw 3500 reader.

Fig. 5.1 and Fig 5.2 shows the glow curve of commercial ZnS powder and ZnS nanophosphors after they are irradiated by gamma ray with a dose equal to 54 Gy. By making comparison between the two graphs, it can be seen that the glow curve of ZnS nanophosphors shows a good clear peak compared with the glow curve of commercial ZnS powder which shows no fine clear peaks. That's means decreasing the particle size of ZnS gives a good emission of TL intensity.

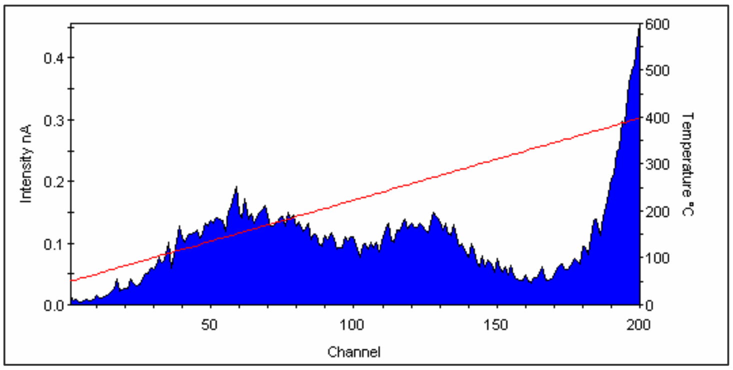

Figure .5.1 TL glow curve of commercial ZnS powder

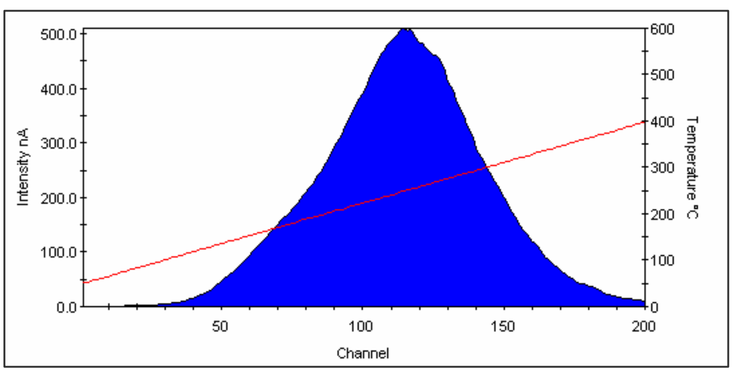

Figure 5.2 TL glow curve of ZnS nano particles

5.2 calculating the absorbed dose

The exposure time can be calculated according to the required absorbed dose by using the following equation:

$$exp\,ouser \quad time(\sec ond) = \frac{absorbed \quad dose(Gy)}{dose \quad rate(Gy/\sec ond)}$$

The previous equation can be rewritten as following

$absorbed \quad dose(Gy) = dose \quad rate(Gy/\sec ond) \times exp\,ouser \quad time(\sec ond)$ so, by this equation the required absorbed dose can be obtained.

The exposure rate of ^{60}Co is not constant so, the radiation dose rate will decrease every month as shown in the table (5.1). The exposure rate can be calculated for any time (t) by using the equation

$D_t = D_\circ e^{-\lambda t}$ where $\lambda = 4.1681 \times 10^{-9} s^{-1}$, $D_\circ = 0.525 Gy/\sec$

2005	
Time (month)	Dose Rate (Gy / sec)
January	0.15957
February	0.15789
March	0.15618
April	0.15450
May	0.15284
June	0.15120
July	0.14958
August	0.14797
September	0.14638
October	0.14481
November	0.14325
December	0.14171

2006	
Time (month)	Dose Rate (Gy / sec)
January	0.14019
February	0.13868
March	0.13719
April	0.13572
May	0.13426
June	0.13282
July	0.13139
August	0.12998
September	0.12858
October	0.12720
November	0.12583
December	0.12448

2007	
Time (month)	Dose Rate (Gy / sec)
January	0.12314
February	0.12182
March	0.12051
April	0.11922
May	0.11794
June	0.11667
July	0.11542
August	0.11418
September	0.11295
October	0.11174
November	0.11054
December	0.10935

2008	
Time (month)	Dose Rate (Gy / sec)
January	0.10817
February	0.10701
March	0.10586
April	0.10472
May	0.10359
June	0.10248
July	0.10138
August	0.10029
September	0.09921
October	0.09814
November	0.09709
December	0.09605

2009	
Time (month)	Dose Rate (Gy / sec)
January	0.09502
February	0.09400
March	0.09299
April	0.09199
May	0.09100
June	0.09002
July	0.08905
August	0.08809
September	0.08714
October	0.08620
November	0.08527
December	0.08435

2010	
Time (month)	Dose Rate (Gy / sec)
January	0.08344
February	0.08254
March	0.08165
April	0.08077
May	0.07990
June	0.07904
July	0.07819
August	0.07735
September	0.07652
October	0.07570
November	0.07419
December	0.07339

Table 5.1. ^{60}Co dose rate

5.3 The thermoluminescence properties of manganese doped ZnS nanophoshor

5.3.1 ZnS commercial powder

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	TL intensity (nC)
195	0.09199	18	4.9
391	0.09199	36	5.2
585	0.09199	54	7.3
780	0.09199	72	8.1
975	0.09199	90	11.0

Table 5.2 exposure time, dose rate , absorb dose and TL intensity of ZnS commercial powder

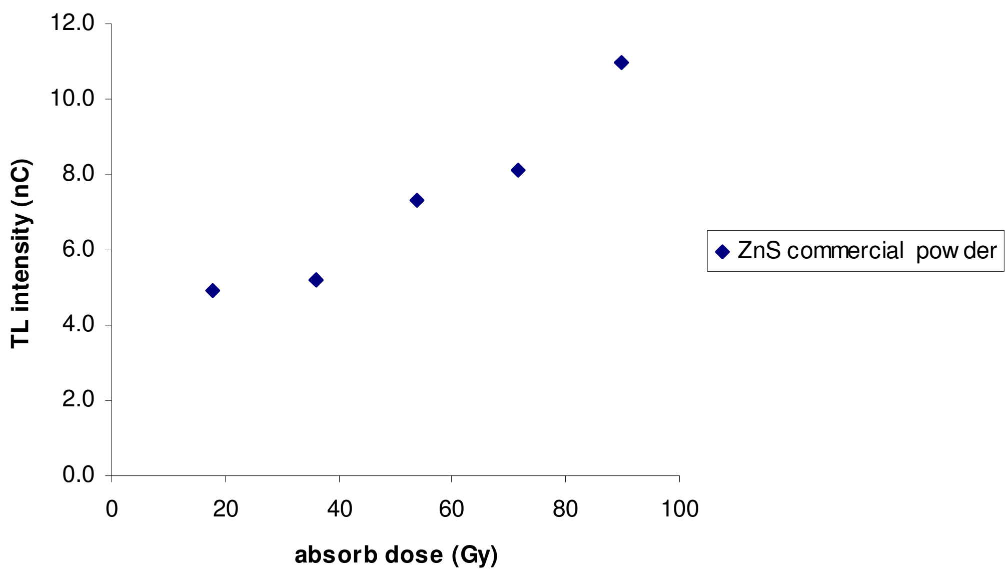

Figure 5.3 TL intensity vs absorb dose of ZnS commercial powder

5.3.2 ZnS nanophoshor without manganese

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	TL intensity (μC)
195	0.09199	18	1.3
391	0.09199	36	3.2
585	0.09199	54	5.8
780	0.09199	72	7.2
975	0.09199	90	11.3

Table 5.3 exposure time, dose rate , absorb dose and TL intensity of ZnS nanophoshor without manganese

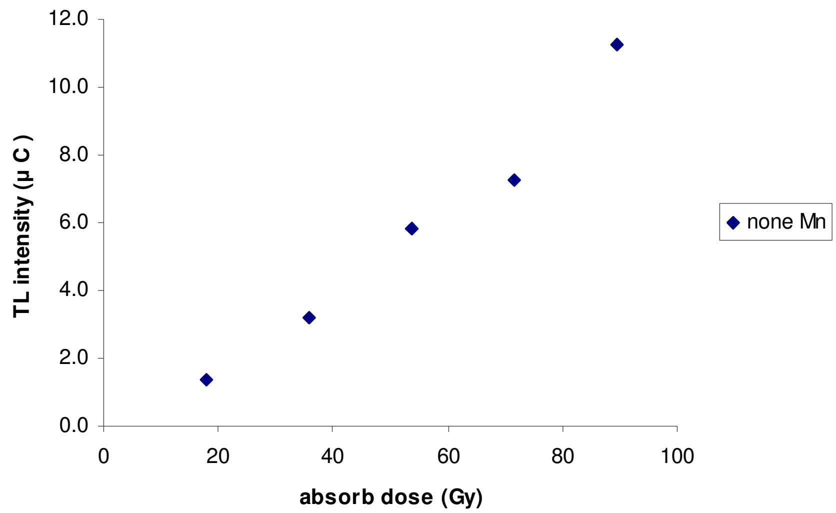

Figure 5.4 TL intensity vs absorb dose of ZnS nanophoshor without manganese

5.3.3. ZnS nanophoshor doped with 1mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	TL intensity (μC)
195	0.09199	18	2.0
391	0.09199	36	4.9
585	0.09199	54	9.9
780	0.09199	72	13.2
975	0.09199	90	19.7

Table 5.4 exposure time, dose rate , absorb dose and TL intensity of ZnS nanophoshor doped with 1mol of manganese sulfate

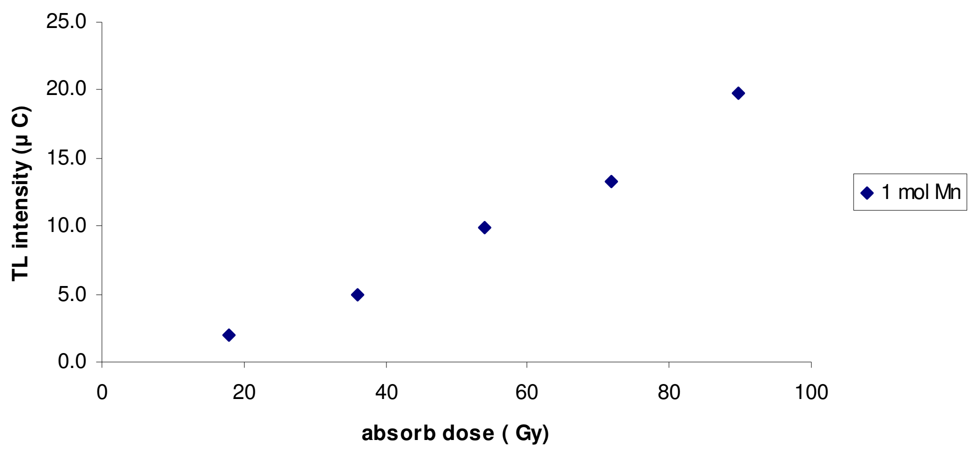

Figure 5.3 TL intensity vs absorb dose of ZnS nanophoshor doped with 1mol of manganese sulfate

5.3.4 ZnS nanophoshor doped with 2mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	TL intensity (μC)
195	0.09199	18	2.3
391	0.09199	36	6.1
585	0.09199	54	11.7
780	0.09199	72	15.7
975	0.09199	90	20.7

Table 5.5 exposure time, dose rate , absorb dose and TL intensity of ZnS nanophoshor doped with 2mol of manganese sulfate

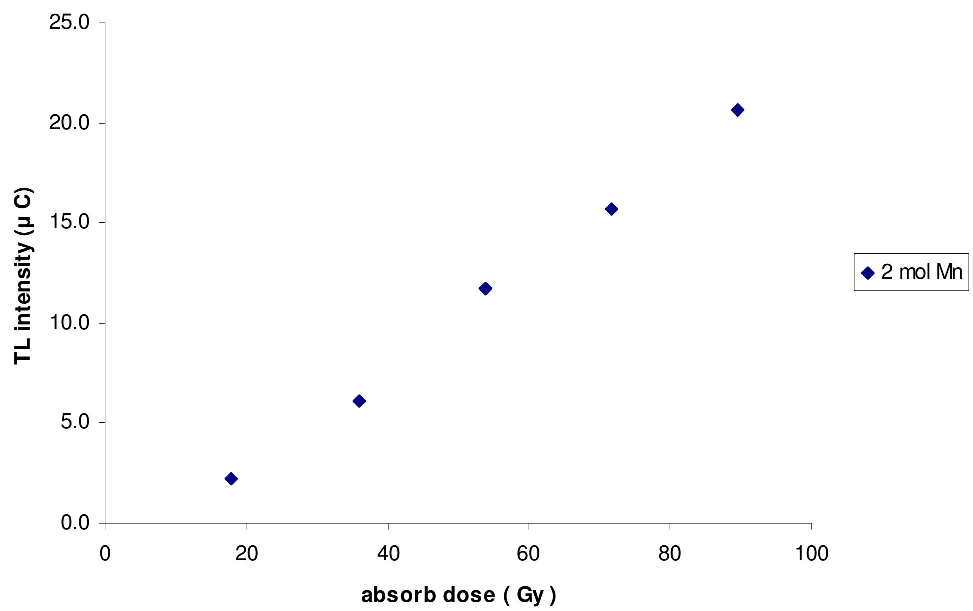

Figure 5.6 TL intensity vs absorb dose of ZnS nanophoshor doped with 2mol of manganese sulfate

5.3.5 ZnS nanophoshor doped with 3mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	TL intensity (µC)
195	0.09199	18	3.2
391	0.09199	36	6.3
585	0.09199	54	10.5
780	0.09199	72	12.4
975	0.09199	90	16.1

Table 5.6 exposure time, dose rate , absorb dose and TL intensity of ZnS nanophoshor doped with 3mol of manganese sulfate

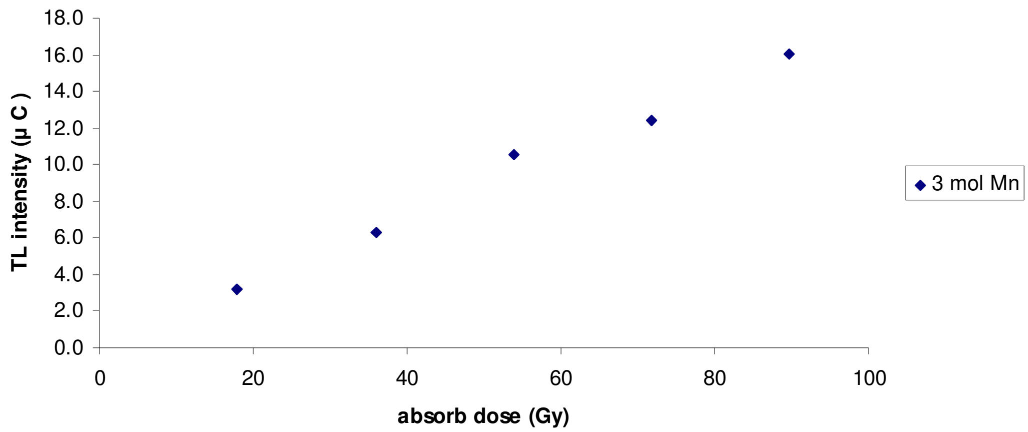

Figure 5.7 TL intensity vs absorb dose of ZnS nanophoshor doped with 3mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	TL intensity (µC) 1 mol Mn	2 mol Mn	3 mol Mn	none Mn
195	0.09199	18	2.0	2.3	3.2	1.3
391	0.09199	36	4.9	6.1	6.3	3.2
585	0.09199	54	9.9	11.7	10.5	5.8
780	0.09199	72	13.2	15.7	12.4	7.2
975	0.09199	90	19.7	20.7	16.1	11.3

Table 5.7 exposure time, dose rate , absorb dose and TL intensity of ZnS doped with different Mn concentrations

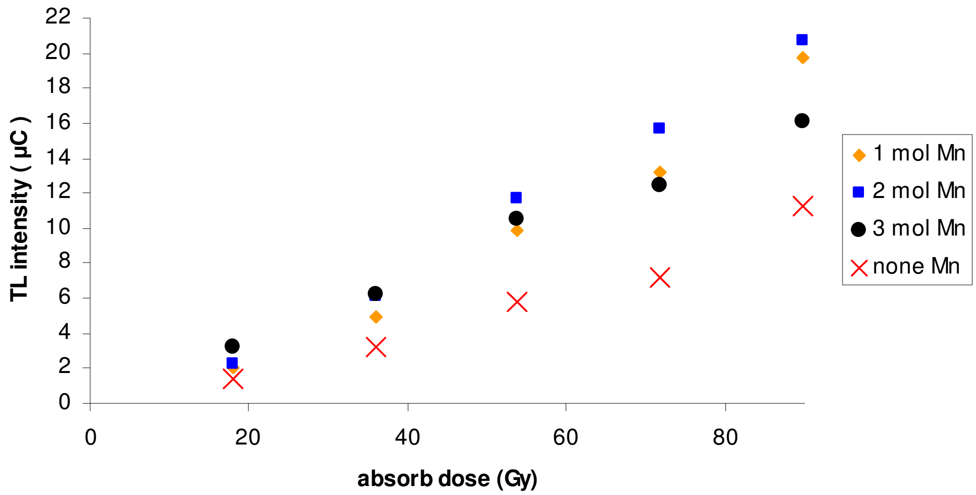

Figure 5.8 TL intensity vs absorb dose of ZnS doped with different Mn concentrations

As it is obvious in (fig.5) the TL response against absorbed dose shows linearity and increasing Mn concentration enhances the TL response. The amount of Mn concentration of 2 mole gives the best emission of TL but as the concentration is increased, the TL response is decreased. That is because ZnS becomes more conductivity as the Mn concentration increase. Semiconductors and insulators are good thermoluminescence materials unlike conductive materials which are poor thermoluminescence materials so; adding Mn in ZnS should be in a certain amount which increases TL properties of ZnS without changing it to conductive material.

5.4 Fading properties of manganese doped ZnS nanophoshor

5.4.1 Fading of ZnS commercial powder

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	fading	TL intensity (nC)
			No fading	9.4
			1 hour	9.1
			2 hour	7.9
			3 hour	8.3
591	0.091	53.8	4 hour	7.1
			1 day	5.2
			2 day	5.1
			3 day	4.4
			4 day	3.9

Table 5.8 exposure time, dose rate , absorb dose , fading and TL intensity of ZnS commercial powder

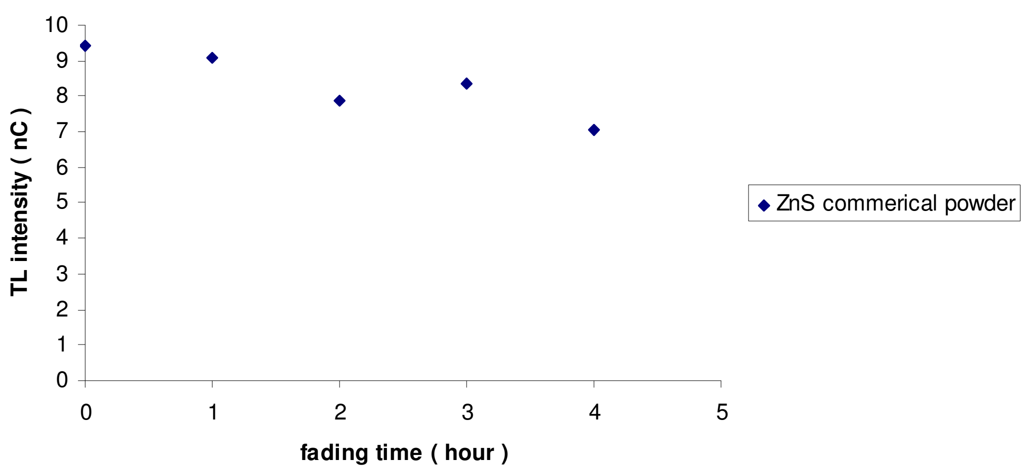

Figure 5.9 TL intensity vs fading time (hour) of ZnS commercial powder

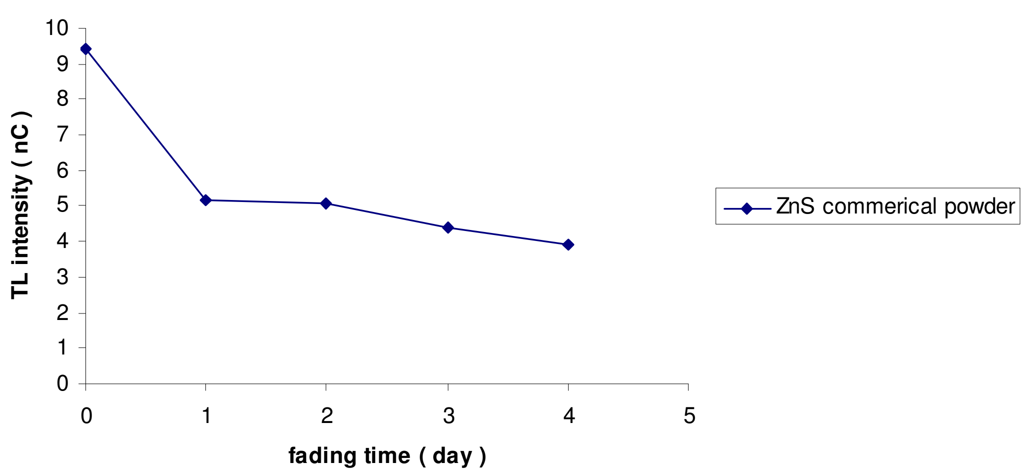

Figure 5.10 TL intensity vs fading time (day) of ZnS commercial powder

5.4.2 Fading of ZnS nanophoshor without manganese

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	fading	TL intensity (μC)
			No fading	6.12
			1 hour	4.7
			2 hour	4.2
			3 hour	3.7
591	0.091	53.8	4 hour	3.1
			1 day	3.6
			2 day	2.8
			3 day	2.8
			4 day	2.2

Table 5.9 exposure time, dose rate , absorb dose , fading and TL intensity of ZnS nanophoshor without manganese

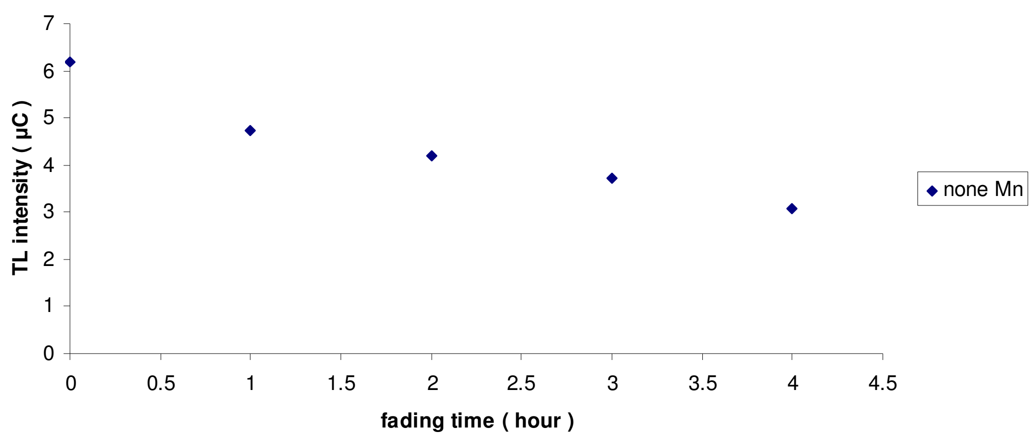

Figure 5.11 TL intensity vs fading time (hour) of ZnS nanophoshor without manganese

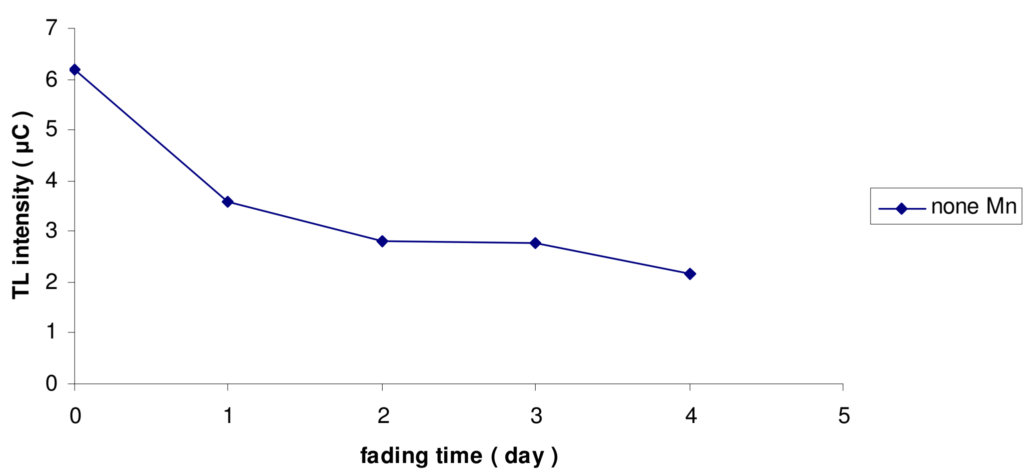

Figure 5.12 TL intensity vs fading time (day) of ZnS nanophoshor without manganese

5.4.3. Fading of ZnS nanophoshor doped with 1mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	fading	TL intensity (μC)
			No fading	12.9
			1 hour	11.3
			2 hour	10.6
			3 hour	8.2
591	0.091	53.8	4 hour	5.2
			1 day	7.7
			2 day	7.6
			3 day	7.0
			4 day	3.5

Table 5.10 exposure time, dose rate , absorb dose , fading and TL intensity of ZnS nanophoshor doped with 1mol of manganese sulfate

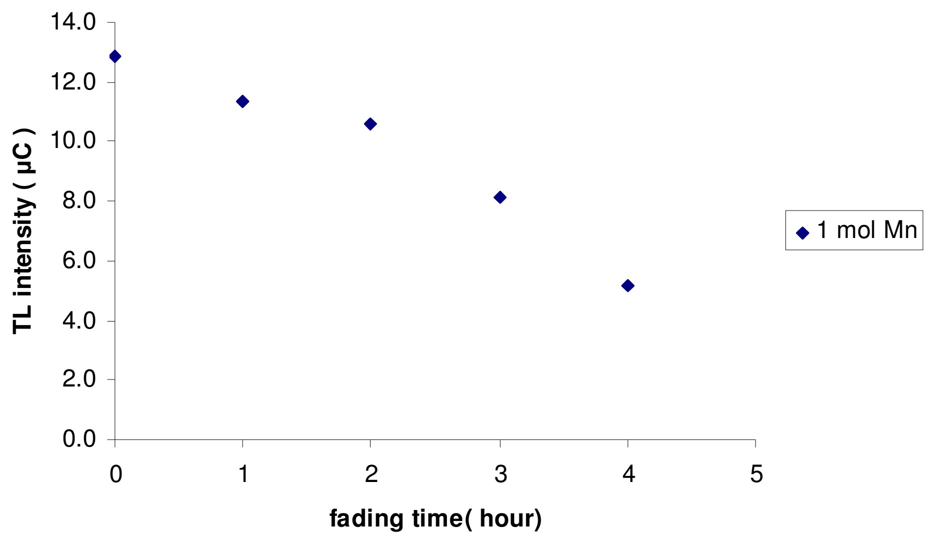

Figure 5.13 TL intensity vs fading time (hour) of ZnS nanophoshor doped with 1mol of manganese sulfate

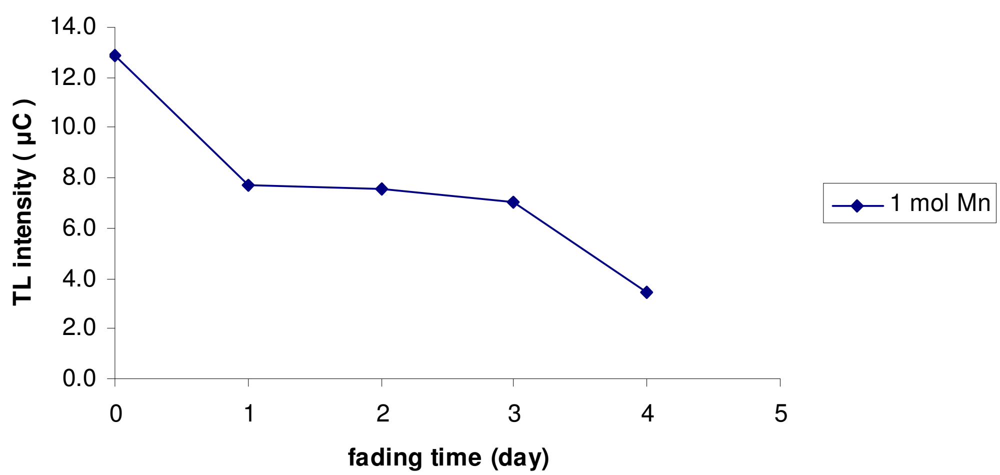

Figure 5.14 TL intensity vs fading time (day) of ZnS nanophoshor doped with 1mol of manganese sulfate

5.4.4. Fading of ZnS nanophoshor doped with 2mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	fading	TL intensity (μC)
591	0.091	53.8	No fading	12.8
			1 hour	11.3
			2 hour	10.8
			3 hour	8.9
			4 hour	5.3
			1 day	7.7
			2 day	7.3
			3 day	6.7
			4 day	3.7

Table 5.11 exposure time, dose rate , absorb dose , fading and TL intensity of ZnS nanophoshor doped with 2mol of manganese sulfate

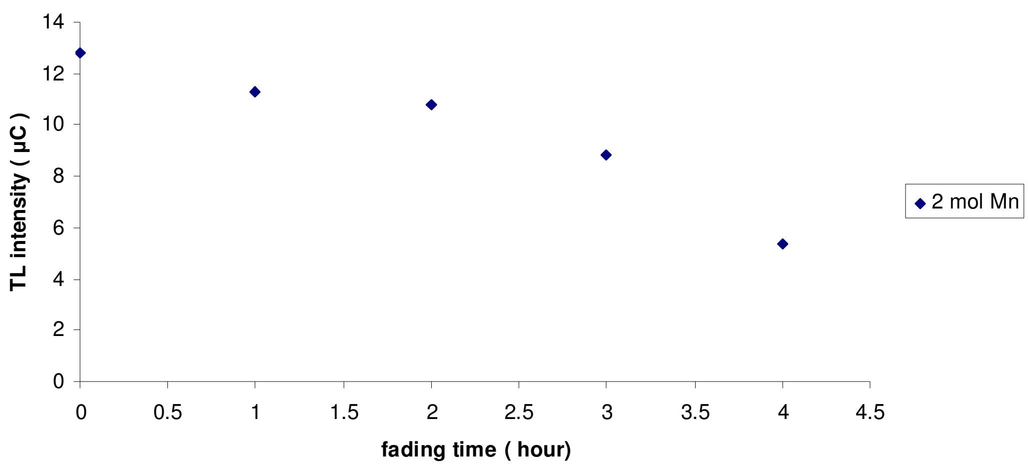

Figure 5.15 TL intensity vs fading time (hour) of ZnS nanophoshor doped with 2mol of manganese sulfate

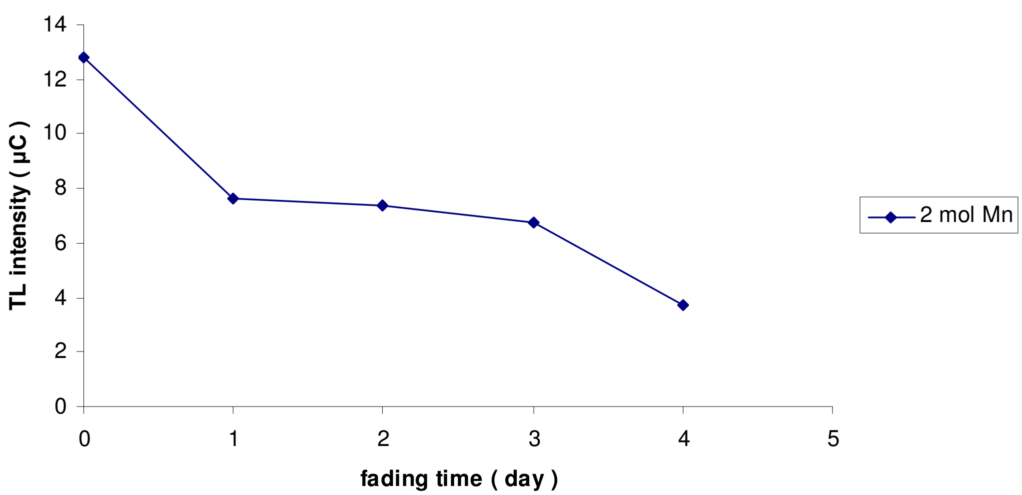

Figure 5.16 TL intensity vs fading time (day) of ZnS nanophoshor doped with 2mol of manganese sulfate

5.4.5. Fading of ZnS nanophoshor doped with 3mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	fading	TL intensity (μC)
			No fading	9.1
			1 hour	7.2
			2 hour	6.8
			3 hour	5.7
591	0.091	53.8	4 hour	2.8
			1 day	7.2
			2 day	6.8
			3 day	5.7
			4 day	2.8

Table 5.12 exposure time, dose rate , absorb dose , fading and TL intensity of ZnS nanophoshor doped with 3mol of manganese sulfate

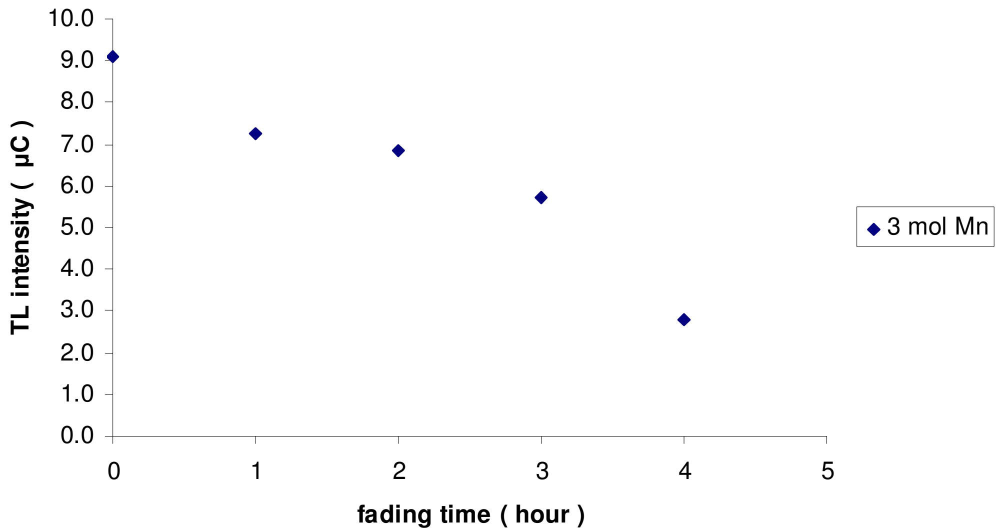

Figure 5.17 TL intensity vs fading time (hour) of ZnS nanophoshor doped with 3mol of manganese sulfate

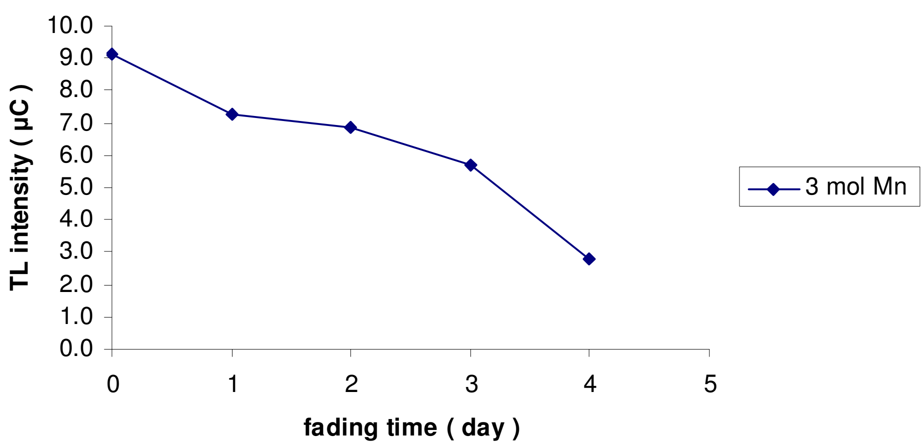

Figure 5.18 TL intensity vs fading time (day) of ZnS nanophoshor doped with 3mol of manganese sulfate

exposure time(sec)	dose rate(Gy/sec)	absorb dose(Gy)	fading	TL intensity (µC) 1 mol Mn	2 mol Mn	3 mol Mn	none Mn	TL intensity (nC) ZnS powder
591	0.091	53.8	No fading	12.9	12.8	9.1	6.2	9.4
			1 hour	11.3	11.3	7.2	4.7	9.1
			2 hour	10.6	10.8	6.8	4.2	7.9
			3 hour	8.2	8.9	5.7	3.7	8.3
			4 hour	5.2	5.3	2.8	3.1	7.1
			1 day	7.7	7.7	5.0	3.6	5.2
			2 day	7.6	7.4	5.2	2.8	5.1
			3 day	7.0	6.7	4.9	2.8	4.4
			4 day	3.5	3.7	1.9	2.2	3.9

Table 5.13 exposure time, dose rate , absorb dose , fading and TL intensity of ZnS doped with different Mn concentrations

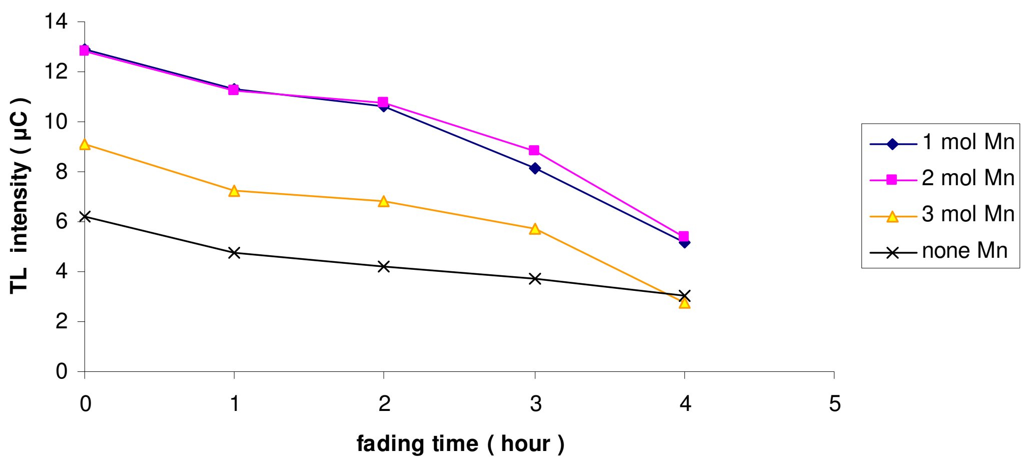

Figure 5.19 TL intensity vs fading time (hour) of ZnS nanophoshor doped with different Mn concentrations

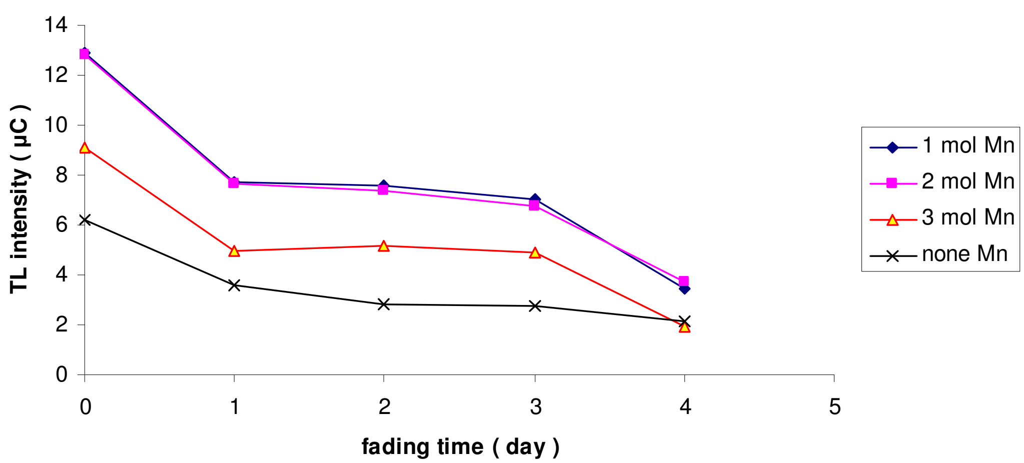

Figure 5.20 TL intensity vs fading time (day) of ZnS nanophoshor doped with different Mn concentrations

The thermal fading of glow peaks of ZnS:Mn were also measured for various durations at room temperature. FIGURE.6 shows the TL response of ZnS doped with different concentrations of Mn during four days. From the graph it can be seen that the ZnS doped with 1mole and 2 mole of Mn have low fading rate compared with the ZnS doped with 3 mole of Mn. For ZnS with 1mole and 2 mole of Mn the TL response loses approximately 40% of its original intensity after 1 day and this quantity is kept nearly constant for the next 2 days. After 4 days TL response loses 71% of its initial intensity.

5.5 ZnS nano particles synthesis by vapor transport deposition method

The silicon wafer covered with ZnS:Mn synthesis by vapor transport deposition method was Annealing and then exposed to a dose equal to 55 Gy, after that the thermoluminescence properties were studed by using Harshaw 3500 reader. The glow curve show a peak of 8nA intensity (fig 5.21)

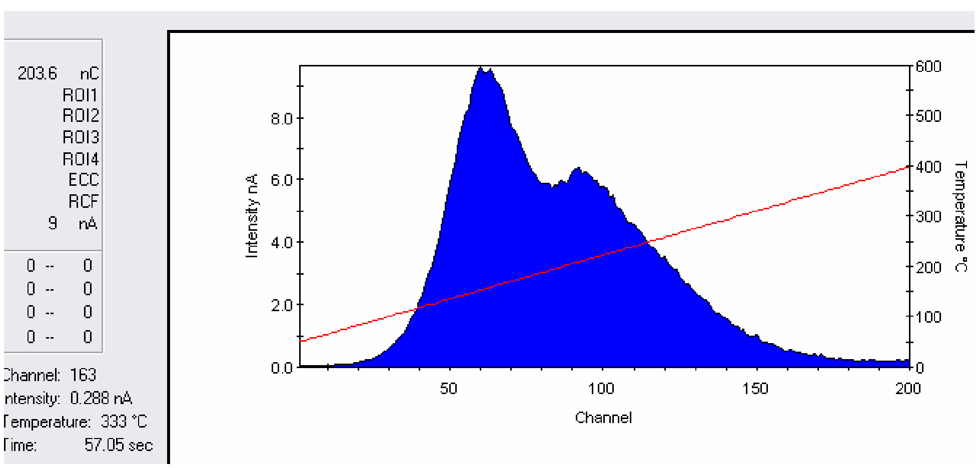

Figure 5.21 glow curve of ZnS:Mn nano particles made by vapor transport deposition method

30 mg of ZnS:Mn powder remains in the quartz tube was also Annealing and then exposed to a dose equal to 55 Gy and (fig 5.22) show the glow curve of this sample.

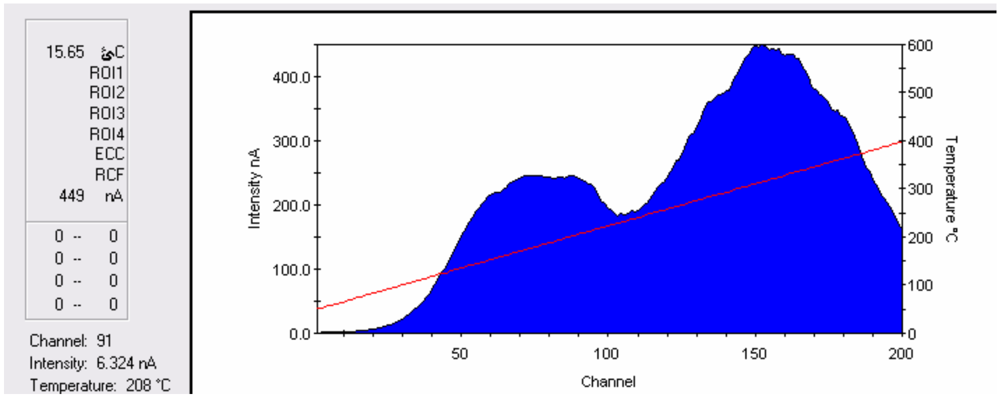

Figure 5.22 glow curve of ZnS:Mn powder remains in quartz tube

This powder gives a very high intensity equal to 400nA as it is shown in the previous figure.

The luminescence process in semiconductor nanoparticles is very complex phenomena and it was found that the luminescence of semiconductor nanoparticles is caused from the deep traps of surface states whose energy levels lie within the band gap of semiconductor. In nanophosphors, as the particle size is decreased the ions at the surface quickly increase. The excited electrons and holes from the surface ions are easily trapped at the surface states. So, by heating the sample the trapped carriers at the surface states are released and they recombine with each other and give out luminescence which is known as thermoluminescence. Decreasing the size of the particles makes the surface to volume ratio to be increased; which make the number of surface ions and surface states increase, so the number of trapped charged particles at surface state increases, as a result TL efficiency is increased. Therefore, the thermoluminescence enhancement of Mn-doped ZnS nanophosphor may be related with the surface states. [A. Necmeddin Yazici , 2007]

5.6 Zns nano particles as a thermoluminesence dosimeter.

Before the Zns nano particles are used as a thermoluminesence dosimeter, a calibration has to be done. There are many steps need to be followed to make the calibration. The first step is annealing. Then the element correction factor (Element Correction Coefficient, ECC) has to be measured with repetition process and linearity. After the previous methods are reached, calibration procedure will be done so that the ZnS powder can be used as a radiation dosimeter to detect the amount of absorbed dose after exposure.

The first step in the experiment is to selected 12 ZnS:Mn nano powder TLD (doped with 2mol of manganese sulfate). Those TLDs were divided into 4 groups, each group contains 3 TLD. Group (A_1, A_2, A_3), group (B_1, B_2, B_3), group (C_1, C_2, C_3), and group (D_1, D_2, D_3). The ZnS nano powders were put in small alumina containers. Each container contains 30 mg of ZnS nano powder.

5.6.1 Annealing process

The ZnS powder was annealed in the furnace at $500^{\circ}C$ and this temperature is reached with a constant heating rate then the samples are cooled gradually with constant rate to room temperature. After the annealing process is completed, the samples are stored in a black box to avoid any background radiation or any other source of light before they used as a radiation dosimeter.

Annealing process is very critical because it is used to return the dosimeters to their original condition especially when they had been irradiated at a very high dose. By annealing process the detection sensitivity of all used phosphors will be the same because the process will eliminate the thermal history for each dosimeter. The annealing process is very imported for the dosimeters that will be used for many time. After finished any TL reading, annealing process have to be done.

The second step in the experiment is annealing the four TLD groups by. The furnace which is used in this experiment is of model Vulcan 3-400HTA. The advantage of this furnace is the ability to be programmed to control the increasing and decreasing temperature in constant heating rate, also it can hold the required temperature for certain period of time. These properties are very important and critical in annealing process of TL dosimeters. The annealing cycle which has chosen is the one which consists of a high temperature anneal at $500^{\circ}C$ for 30 min followed by a low temperature anneal at $100^{\circ}C$ for 30 min. The furnace was programmed so that the samples were heated gradually to $500^{\circ}C$, $300^{\circ}C$ and $100^{\circ}C$. Each temperature was kept constant for 30 minute. The heating rate was equal to $40^{\circ}C/\min$.

5.6.2 Element Correction Coefficient, ECC

The TL efficiency is not exactly the same for all the TL dosimeters as a result of manufactured circumstances. To avoid this problem an Element Correction Coefficient, ECC is done. TL efficiency (TLE) is defined as emitted TL light intensity per unit of absorbed dose. All TL dosimeters do not give the same respond although they are constructed from the same material. Also, Element Correction Coefficient for all the dosimeters is used because they do not have similar masses correspond to each other.

After that the samples were exposed to a gamma ray by using Gamma cell 220 which use ^{60}Co as a source of gamma (γ) rays. The exposure time of the groups $(A_1, A_2, A_3), (B_1, B_2, B_3), (C_1, C_2, C_3)$ and (D_1, D_2, D_3) were 197sec, 395 sec , 592 sec and 789 sec respectively. Then the thermoluminescence properties were studied by using Harshaw 3500 reader (fig 5.23).