CHAPTER - 4

RESULTS AND DISCUSSION

4.1 Thermoluminescence study of synthetic quartz at room temperature

Synthetic Quartz (SQ), which is being widely used in wide spectrum of applications thus, it is significant to understand the systematic changes occurring due to different physical conditions such as radiation, grain size, pre-thermal treatment, duration of pre-thermal treatment etc to which the specimen is subjected. An attempt is also made to propose new applications.

The present work is aimed at understanding the effect of different protocols on Optically Stimulated Luminescence (OSL) characteristics and thereby to propose an appropriate protocol for some of the applications on the basis of the observed results. Systematic efforts are made to correlate the understanding of physical processes in the material proposed for use in dating and dosimetry.

Prior to systematic OSL study, it is essential to know the characteristics such as location, nature, emission wavelength and stability of color centers associated with the specimens. Perhaps, the most important property for the characterization of quartz sample is the thermoluminescence (TL) glow curve due to its hyper structure sensitive phenomenon. This property explains the changes in term of trapped charge carriers and hence the stability of the TL signal with/without various pre-thermal treatment. Therefore, TL glow curves are recorded for different grain sizes, pre-heat treated and identical beta exposed specimens.

As already mentioned earlier, enough attention and correlation were attempted for the 110°C and 230°C TL peak in quartz. But the present work also induced 375°C TL peak besides above-mentioned two peaks. The earlier work on this trap was not sufficient to conclude about its slower bleachability, though very important for many applications. Special efforts are made to initiate this work to collect more information regarding this trap in the present work. Thus the investigations are aimed to give more

information. The results of the present work are presented in **Graph-1** to **Graph-5**, and are briefly discussed for the following obtained data:

Table-4A: A Protocol for TL study of synthetic quartz at room temperature

Grain Size (mesh)	Annealing Temperature (°C)	Annealing Duration (Hrs)	Beta Dose (Gy)
120-140mesh	600°C - 1000°C	1hr	5.04Gy
230-270mesh	600°C - 1000°C	1hr	5.04Gy
230-270mesh	600°C - 1000°C	2hrs	5.04Gy
230-270mesh	600°C - 1000°C	3hrs	5.04Gy
270-325mesh	600°C - 1000°C	1hr	5.04Gy

It is clear from the literature that 'virgin (without beta exposure)' synthetic quartz does not exhibit any TL glow curve.[1] Hence it was pointed out that for TL emission the material has to be pre-exposed to the radiation. It clearly indicates that TL emission of the specimen is purely radiation induced.

Further, systematic TL observations as mentioned in the **Table-4A** for the described protocols reveal that prominent three TL peaks are exhibited at 108°C-140°C, 210°C-229°C and 350°C-372°C. Other barely discernible peaks appear at ~160°C and ~340°C.

The changes in the glow peak positions for an individual peak are due to change in physical protocol. No evidence of further peaks was found when heating continued upto 600°C (the practical limit of an instrument). The detailed observations and their revelations considering each protocol in turn are mentioned as below.

In order to examine, the thermal sensitivity of the centers responsible for changes in the TL glow curve with the change in the pre-heat treatment, TL was measured for 120-140mesh materials pre-heat treated at 600°C and

1000°C for 1hrs pre-heat duration and in another part thermally untreated material was also studied. Such types of material were identically exposed to 5.04Gy beta dose.

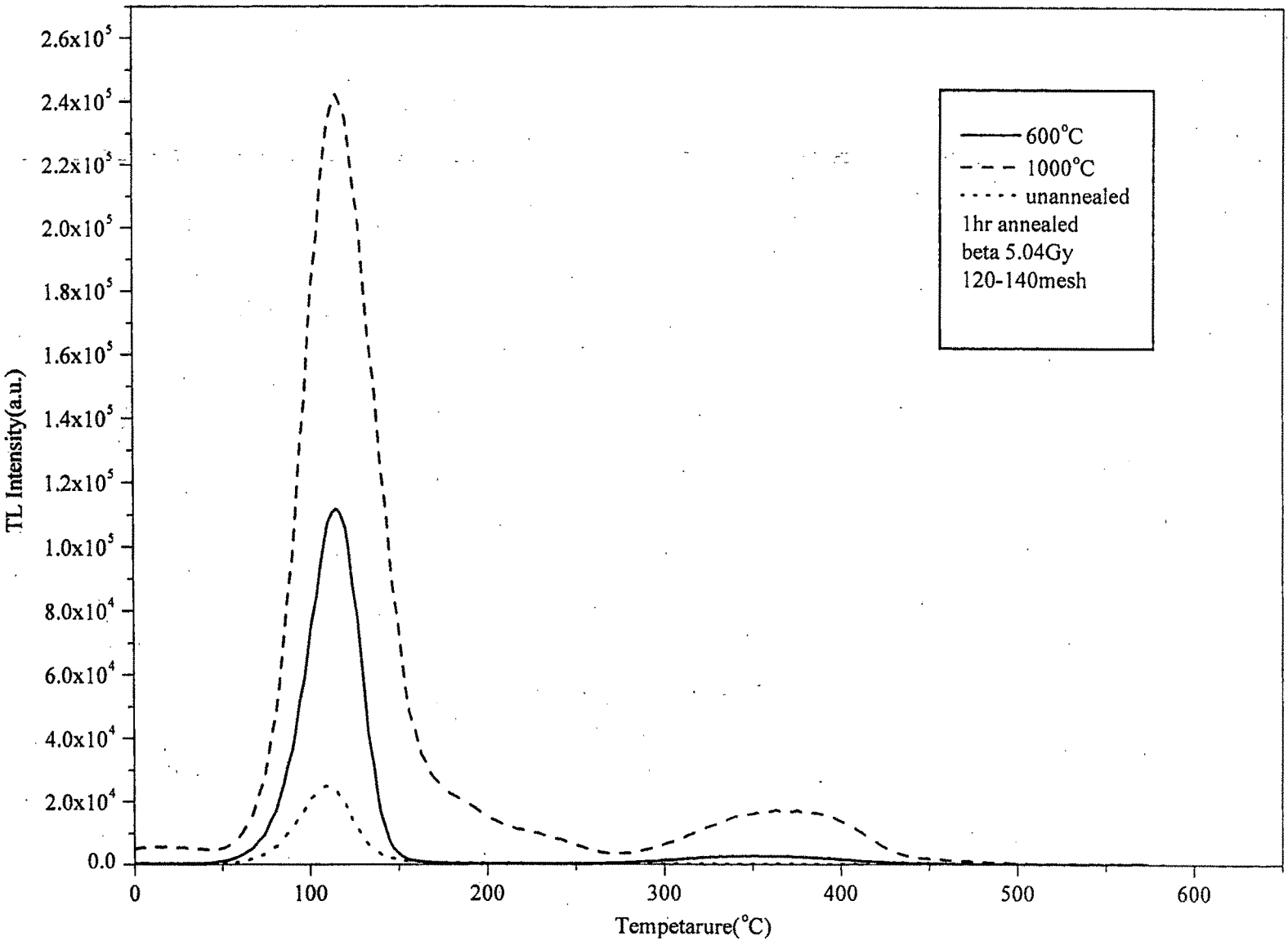
From **Graph-1**, it is found that an untreated material exhibits a strong TL peak at ~110°C(Low Temperature (LT)) peak along with two humps at 220°C and 350°C respectively. Also it is significant to note that High Temperature (HT) peak shifts toward higher temperature side with rise in temperature of annealing. TL intensity also grows with the rise in temperature of pre-thermal treatment **(Table-4B)**.

From data it is noteworthy to remark that the LT peak grows two times when the specimens were pre-heat treated from 600°C to 1000°C. However, for the same protocol the growth is seven times for the HT peak i.e. 2596.06a.u. to 18017.8a.u. In another observation it is found that the area under the TL glow curve also increases from 4.62625×10^{6} to 1.59377×10^{7} with the increase in temperature of annealing.

Comparison is also made for the observed TL results specimens with yet another grain size of 230-270Mesh. The identical physical treatment was given to the specimen as in previous study see in **Graph-2** i.e. ascending the temperature of annealing while keeping identical dose and duration of pre-heat treatment. The comparisons of TL patterns in both cases reveal that the LT peak shifts toward higher temperature side from 113°C to 122°C but HT peak shifts toward lower temperature side from 351.83°C to 218.77°C with the rise in temperature of annealing.

TL intensity also grows with the rise in temperature of annealing but the response to the temperature of annealing in finer grain (230-270mesh) is quite interesting as compared to coarse grain (120-140mesh).

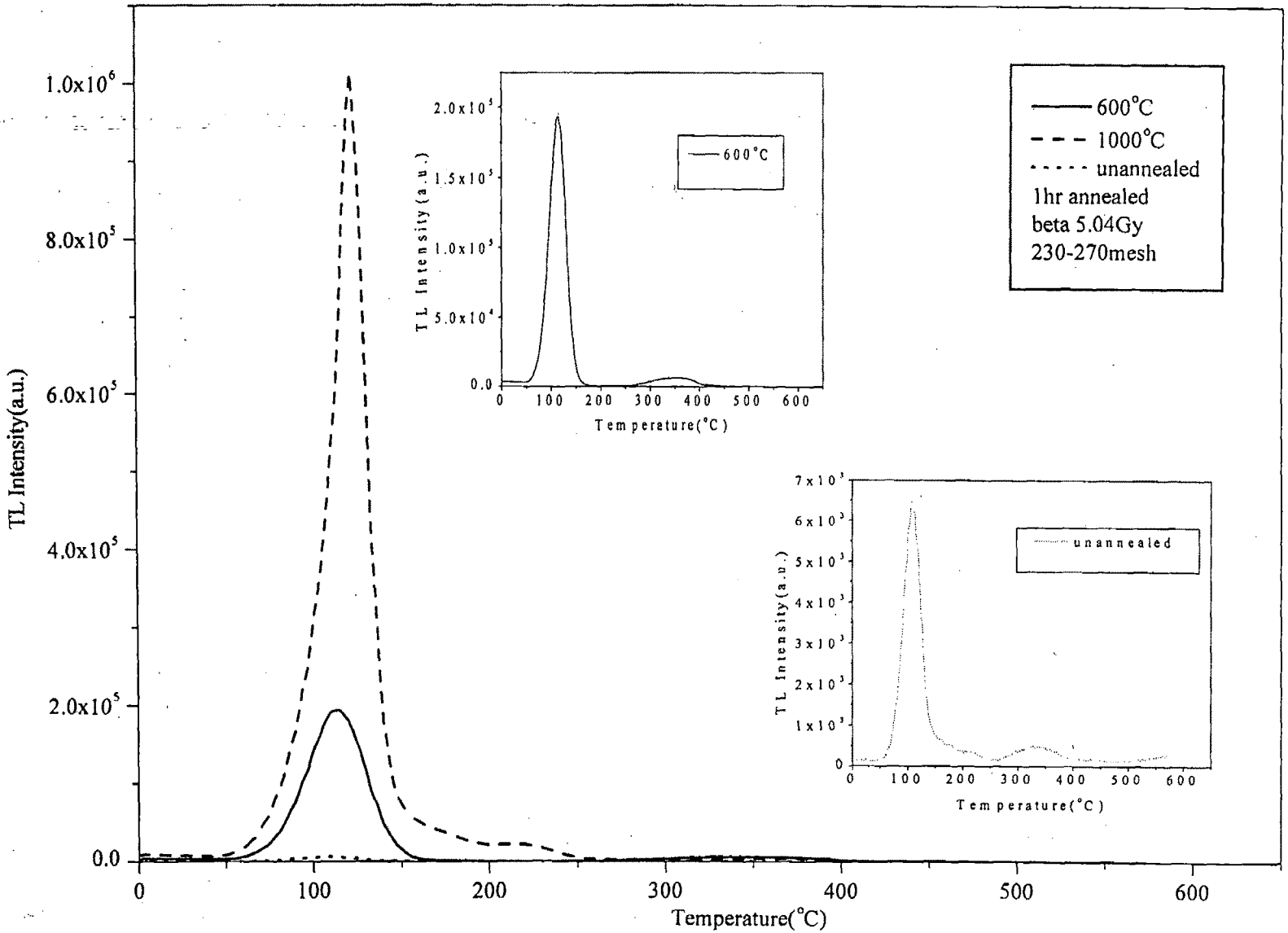
For the finer grains, LT peak grows about five times i.e. 19335 to 1.0131×10^{6}a.u., while the HT peak grows three times from 7567.94 to



Graph-1 TL glow curve measured at room temperature for 1hr annealed sample for a given 5.04Gy beta dose at different annealed and unannealed temperature for 120-140mesh.

Table-4B: A data table for TL peak position, intensity and area under the TL glow curve for synthetic quartz at different protocol.

Grain (mesh)	Annealing Temperature (°C)	Annealing Duration (Hrs)	Beta Dose (Gy)	Tm_1 (°C)	Tm_2 (°C)	Tm_3 (°C)	Intensity-1 (a.u.)	Intensity-2 (a.u.)	Intensity-3 (a.u.)	Area under TL glow curve
120-140	Unannealed	-	5.04	110	220	350	25018	559	197	1.08234×10^6
	600	1	5.04	115	357	-	111691	2596	-	4.62625×10^6
	1000	1	5.04	115	372	-	242319	18018	-	1.59377×10^7
230-270	Unannealed	-	5.04	108	221	332	6487	308	505	393304
	600	1	5.04	113	352	-	1.93335×10^5	7568	-	9.1749×10^6
	1000	1	5.04	122	219	-	1.0131×10^6	19213	-	3.30438×10^7
	600	2	5.04	163	-	-	485129	-	-	1.21739×10^7
	1000	2	5.04	110	219	360	320118	35473	23102	2.21835×10^7
	600	3	5.04	136	-	-	440081	-	-	1.4634×10^7
	1000	3	5.04	140	226	372	1.21735×10^6	49016	2624	4.0963×10^7
270-325	Unannealed	-	5.04	108	344	-	12366	1741	-	737604
	600	1	5.04	154	360	-	500357	14667	-	1.18656×10^7
	1000	1	5.04	113	221	-	7205591	13027	-	3.43277×10^7



Graph-2 TL glow curve measured at room temperature of 1hr annealed sample for a given 5.04Gy beta dose at different annealed and un-annealed temperatures for 230-270mesh.

19212.74a.u., with the identical change in temperature of pre-heat treatment, but the LT peak gets subdued for finest (270-325mesh) specimen. Here also the area under the glow curve increases from $9.1749X10^6$ to 3.30438×10^7 with the temperature of annealing but response of the finer grain specimen is more.

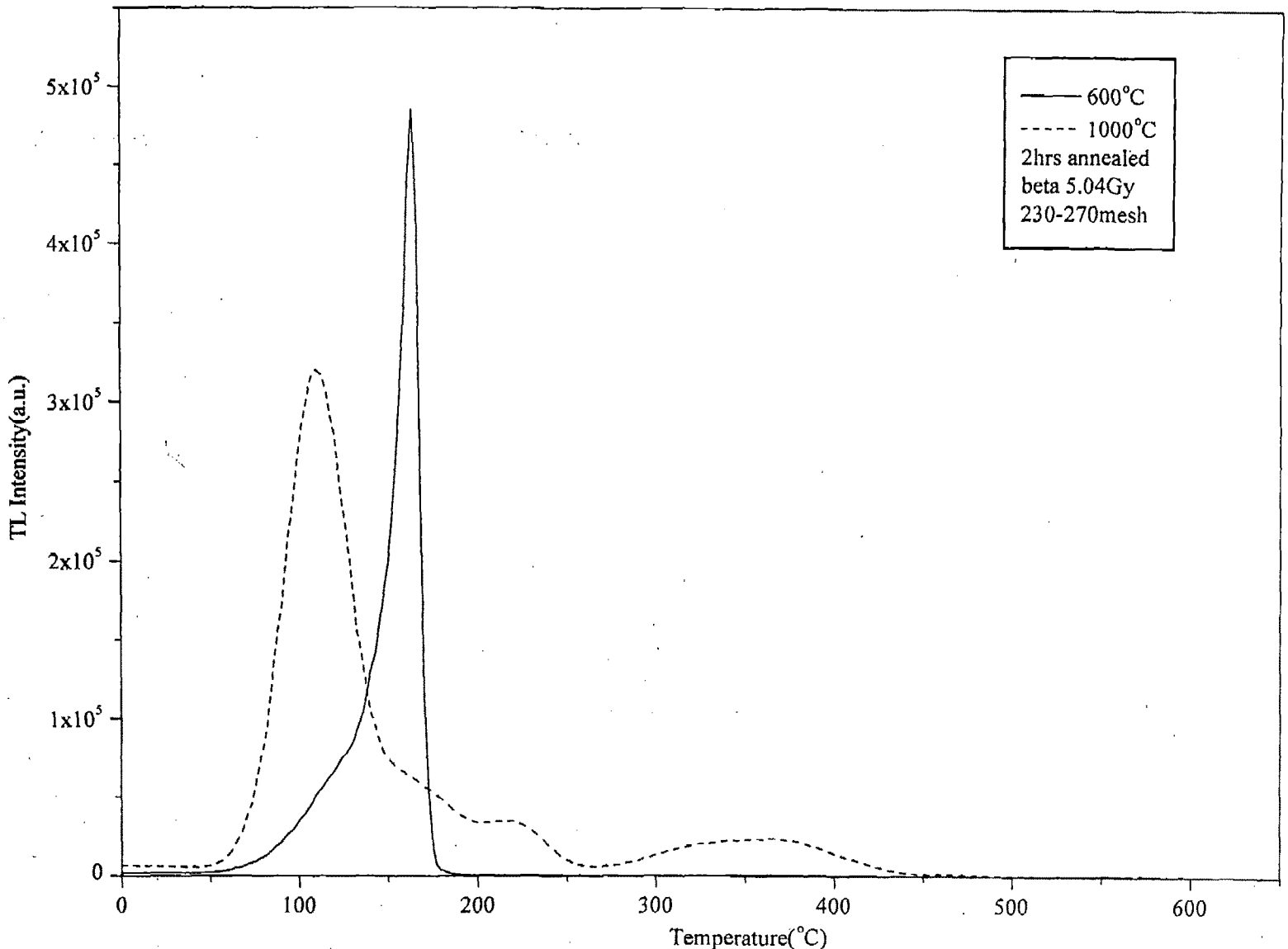
Effect of pre-thermal treatment was also studied for 2hrs duration, which is displayed as **Graph-3**. It is observed that the LT peak shifts towards lower temperature side to 110°C and along with it two separate glow peaks at 218.77°C and 359.5°C appear. It is observed that the LT peak intensity though reduced to 320118a.u. from 485129a.u., still, however, the area under the LT glow curve is systematically increased.

Also, specimen pre-heat treated from 600°C to 1000°C for 3hrs duration was also investigated for the TL characteristics **(Graph-4)**. It is noted that the LT peak remains almost stable, with the occurrence of two separate peaks at 226.25°C and 372.13°C but LT peak grows substantially from 440081a.u. to 1.21735×10^6a.u. Area under the curve also increased.

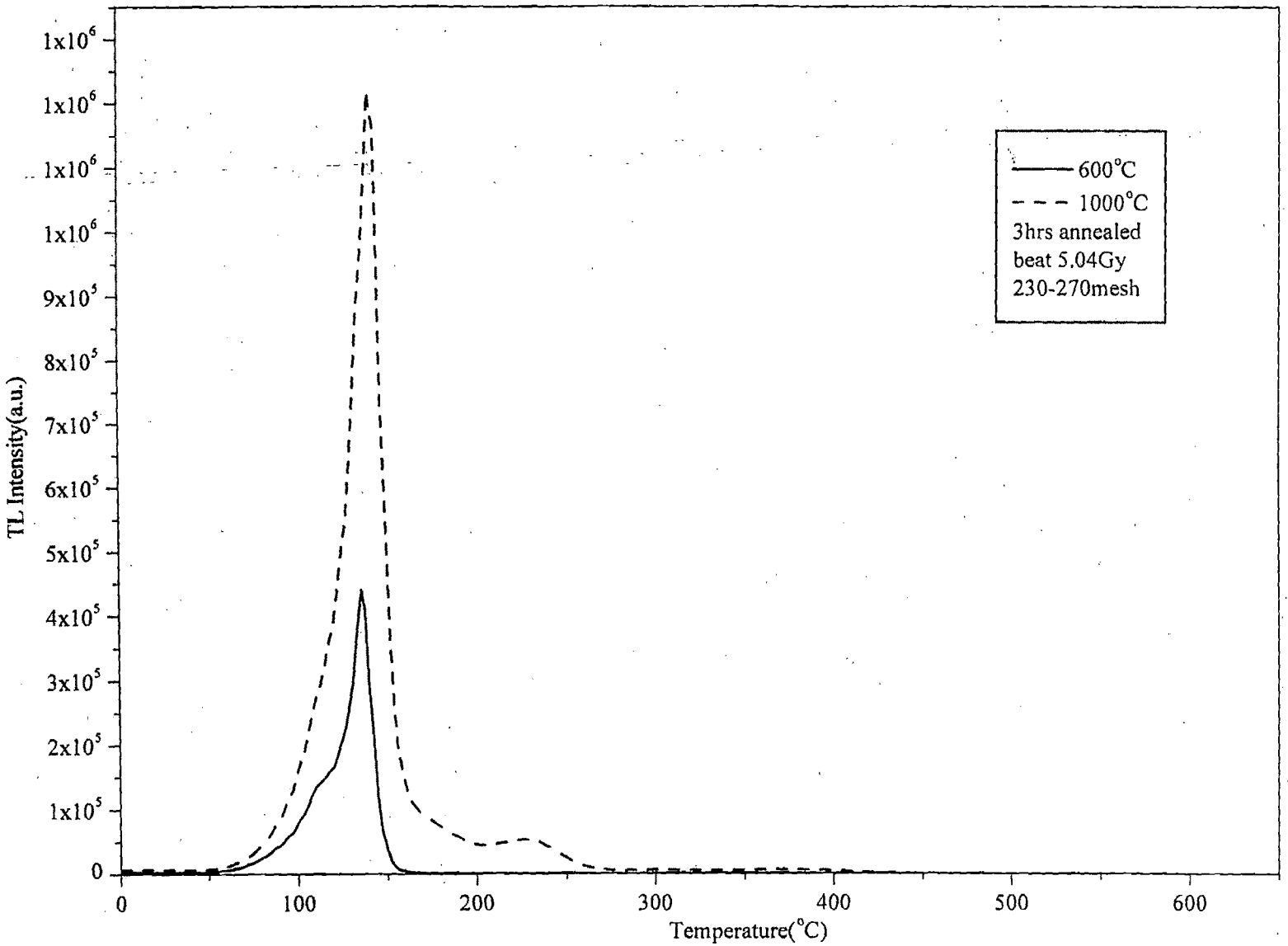
The finest grain (270-325mesh) specimen was also studied under identical protocol (see **Graph-5**). In this case, LT and HT peaks are shifted toward lower temperature side to 113°C from 154°C and 221.45°C from 359.84°C respectively with the increase in annealing temperature from 600°C to 1000°C. Here, TL intensity of LT peak grows with annealing temperature but not significantly whereas HT peak intensity is reduced a bit. Area under the TL curve gives noticeable changes from $1.18656X10^7$ to 3.43277×10^7 with increasing annealing treatment.

The explanation for the growth of TL glow curve or changes in the properties of TL pattern with changes in protocols.

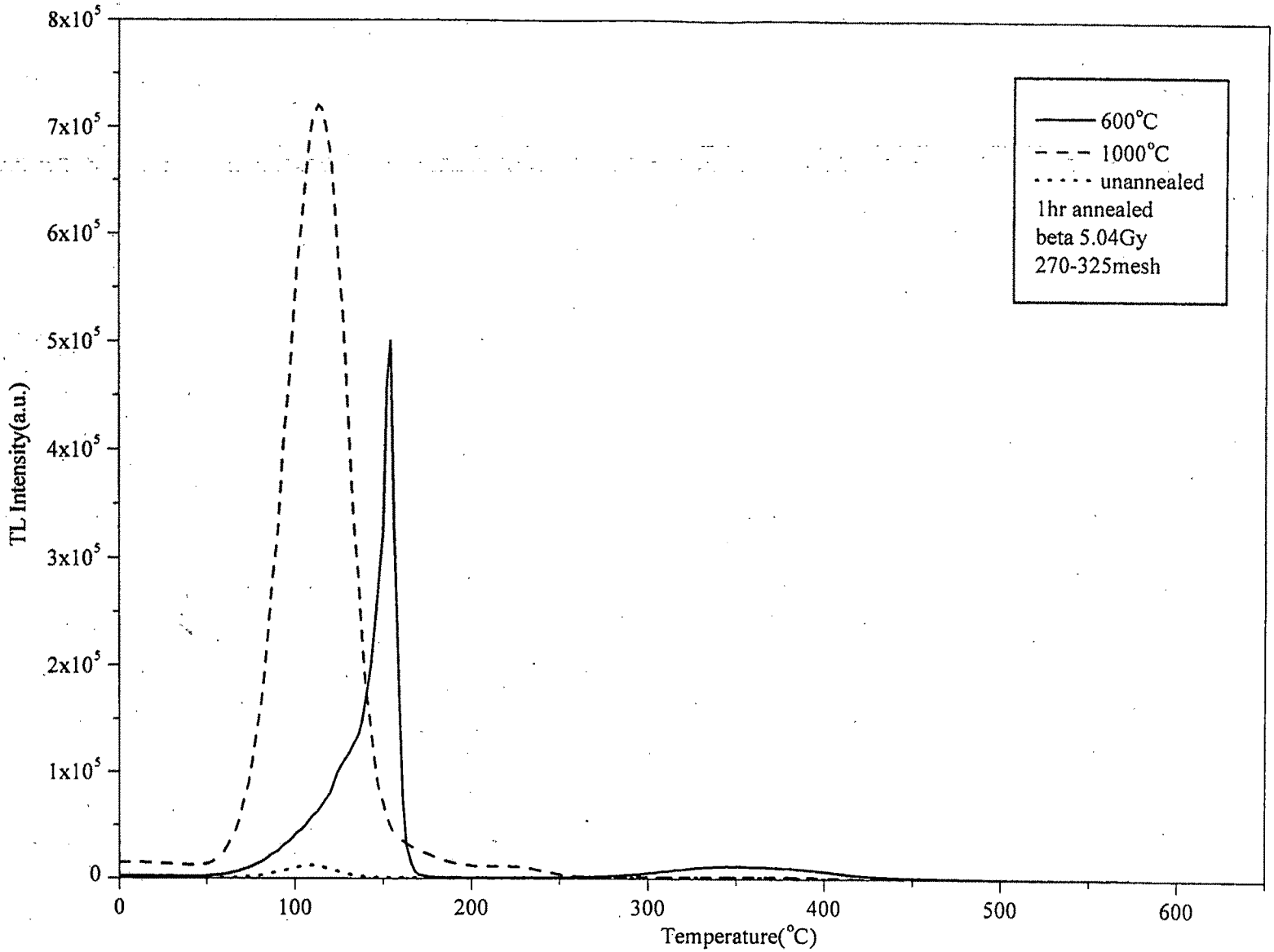
First, the information about TL process in quartz material must be given. Several workers offered explanation, about TL mechanism in quartz.



Graph-3 TL glow curve measured at room temperature of 2hrs annealed sample for a given 5.04Gy beta dose at different annealed temperatures for 230-270mesh.



Graph-4 TL glow curve measured at room temperature for 3hrs annealed sample for a given 5.04Gy beta dose at different annealed temperatures for 230-270mesh.



Graph-5 TL glow curve measured at room temperature for 1hr annealed sample for a given 5.04Gy beta dose at different annealed and un-annealed temperatures for 270-325mesh.

Among them, Spooner explained the TL process on the basis of atomic level theory for quartz but he explained this mechanism by using O'Brien model in which the Al/alkali center was involved. This center is created when trivalent Al^{+3} substitutes for tetravalent Si^{+4} in the SiO_2 lattice and therefore one adjacent oxygen is left unbounded. The resultant charge imbalance attracts a charge compensating monovalent ion, which becomes localized in the adjacent c-axis channel. Suitable compensating species are protons (H^+) and the lighter alkali metal ions (Li^+, Na^+ and possibly K^+). The range of available charge compensators and physical configurations gives rise to several types of Al/alkali centers.

On irradiation, the non-bonding oxygen traps a hole, and now more than enough (superfluous) alkali ions are free to diffuse away down the c-axis channel. The Al/alkali center thus becomes an Al/hole center- $(Al^{+3})^o$, believed to be the dominant luminescence center in most form of quartz. The free electron can become trapped at a variety of electron traps, giving rise to the complex structure of quartz glow curves. Substitutional Ge^{+4}, because of a high electron affinity, is a likely candidate as an important electron trap. Other possibilities are protons, alkali ion clusters, substitutional Ti^{+4} and self-trapping at Si^{+4}. The destination of the diffusing alkali ion is unknown; through it may become localized at the electron trap. Subsequent heating, untraps the electron which then recombines at the $(Al^{+3})^o$ center, emitting photon at 470nm. Sometime later the alkali ion returns so restoring the original Al/alkali center.[2]

Due to special interest of TL of quartz in dating and dosimetry, several workers have recorded the TL glow curves. Fleming explained the role of TL peak as per their required applications. He suggested that, quartz shows a number of TL peaks when irradiated grains are heated from room temperature to 500°C. He suggested the preferred peak for dating was

observed at 375°C. A peak occurring on lower shoulder of this peak, around 325°C, was found to show an unreliable dose response and was termed 'maline' or rapidly bleach peak (RBP). Another peak that attracted attention for dating pottery was only found in laboratory-irradiated sample. It occurred at about 110°C and became the basis of "pre-dose dating method". Franklin et al., reported the RBP (Rapidly Bleach Peak) at 150°C-180°C and 200°C-220°C. According to them the peaks at 110°C, 180°C, 220°C and 325°C are attributed to the same luminescence centers and its emission peak shifts to higher wavelengths, as the temperature of sample was increased.[3] The results of the present study also shows the same peak positions.

The 110°C TL peak is always found in all quartz and quartz-containing specimens. The important information obtained from several workers about the peak is that its position varies between temperature region of 90°C to 130°C due to sample type and treatment (annealing, dose, electrolytic sweeping etc). Yang gives a variation in peak position from 89°C-150°C in as received material. He labeled it as 110°C TL peak[6]. The pre-thermal treatment; mechanical and other treatments have been suggested to be responsible for these changes in peak position. Several workers explained the changes in TL pattern due to defects and electron traps of intrinsic origin. They have established that the major contribution to the 110°C peak is from the O_2^- center. Further, it has been indicated that, intrinsic ion (M^+) specifically Na^+ and Li^+, seem to participate in the O_2^- formation dynamics. In fact, both in natural & synthetic quartz, M^+ ions are always present in association with Al^{+3} ions (Al^{+3}-M^+ center) substitutional for Si^{+4}. This association can be broken by irradiation, which may give rise to the possible mechanism for TL in synthetic quartz.[4] In short in virgin synthetic quartz; the alkali ions (M^+) serve as charge compensators for the ever-present substitutional Al^{+3} impurities. In such a condition, Aluminum

Al^{+3} and M^{+} impurities are associated with each other in the form of $[AlO_4/M^{+}]^{o}$ centers. Zimmermann established that the increase of luminescence center is most likely factor to enhance the TL output rather than the activation of pre-existing traps. Therefore, it is suggested that the enhancement in 110°C TL peak in the present specimen is due to above proposed luminescence center[4].

The above mentioned mechanism for production of low temperature TL peak was explained by Martini et al.,[5] They explained,

Aluminum in unswept quartz is preliminary in the form of $Al\text{-}M^{+}$ centers.

Aluminum in swept-out quartz is preliminary in the form of $Al\text{-}OH^{-}$ centers.

$Al\text{-}M^{+}$ centers are converted into $Al\text{-}OH^{-}$ and $[AlO_4]^{o}$ centers by room temperature irradiation: the ratio of the resulting $Al\text{-}OH^{-}$ and $[AlO_4]^{o}$ depends on the types of electron traps in the sample.

In unswept quartz, $Al\text{-}OH^{-}$ centers are converted into $Al\text{-}M^{+}$ centers by annealing above 300°C, thus restoring the initial situation.

In swept-out material $Al\text{-}OH^{-}$ centers are not affected by annealing up to 500°C.

Subramaniam et al., revealed an increase of $Al\text{-}OH^{-}$ centers after room temperature irradiation followed by annealing in range 50-100°C. This type of treatment is very similar to a pre-dose cycle so that in the swept sample the presence of H at Al sites corresponds to an enhancement of the low temperature of TL peak.[6]

Yang and Mckeever explained the increase in sensitivity of 110°C TL peak after ionizing radiation at room temperature, followed by heating to 500°C. Such type of concept was used in dosimetric application. Zimmermann explained an increased luminescence efficiency following pre-dose and post-annealing treatment. Similar observations are observed in the present study. He verifies such notation by ESR technique, which is able to

provide information regarding to point defects, involved in the TL process at 110°C. He indicates that the electron traps were $(GeO_4)^-$ centers. The recombination centers, however, appeared to come from two origin-one being holes trapped at $(AlO_4)^o$ centers and the other being holes trapped at defects which were unidentified at that time. Additional ESR data, which indicate that the unknown hole traps are in fact $(H_3O_4)^o$ centers. Additionally, pre-dose sensitization is apparently activated over the same temperature range as that within which hydrogen impurities are being released within the lattice.[6]

Earlier worker had established that there must be two different recombination centers, which are involved in the production of LT (Low Temperature) peak. One was determined to be due to $(AlO_4)^o$ centers. The second was speculated to be a hole trap, which of course may be thermally unstable at 110°C and related to hydrogen. Nuttall and Weil investigated and described the $(H_3O_4)^o$ center. This hole center is similar to $(AlO_4)^o$ center except that three protons occupy the Si^{+4} vacancy instead of Al^{+3} ion.[6]

The enhancement in the sensitivity of 110°C TL peak after beta or gamma irradiation followed by thermal activation at 350°C-900°C is attributed to Zimmermann model which interprets about such sensitization, it is being due to filling of a hole 'reservoir' by the radiation, followed by the transfer of the holes into the recombination centers by the thermal activation. However, Chen modified this model and showed that the essential conditions for it to be consistent with results is the presence of a competing radiation less centers.

Yang and Mckeever identified the electron trap with $(GeO_4)^-$ centers and the hole centers with $(AlO_4)^o$ yielding ~470nm light. They also suggested from the TL-ESR measurements that, 380nm emission is related to $(H_3O_4)^o$ hole center.

Itoh et.al observed that a ~470nm emission is due to the recombination of self-trapped excitons. Many authors, reported that the main emission in the 100°C TL peak namely, the 380nm, was the one associated with the increase in sensitivity due to irradiation followed by thermal treatment.[7]

For higher temperature TL (>250°C) peaks of quartz, Jani et.al concluded that the TL in this region is the result of the recombination of electrons with the holes at Al^{+3}-h^{+} centers. The process was also supported by other detailed measurements on this system. Thus, it is reasonable to expect that the TL at 100°C should emit at the same wavelength as that at the higher temperatures. However Jani et al., report that the higher temperature TL emits at ~470nm, whereas Zimmermann reports that at 100°C TL peak emits predominantly at 380nm with a weaker emission band ~470nm.[9]

R.Chen suggested a model for high temperature TL glow curve in quartz, which appears in the UV at a temperature of 375°C, it was measured for emission in the UV as well as in the green part of the spectrum. Also he suggested a kinetic order between first and second order. For simple interactive model that possesses two separate recombination centers along with one-electron traps. It is necessary to attribute a distribution of activation energies to the electron trap, to allow for a significant re-trapping probability, and to also invoke the presence of a thermally disconnected electron trap.

The 375°C electron trap complex in quartz offer another example demonstrating that the simplest model fit to an experimental glow curve, such as the use of the general order theory equation, even if very accurate, need not yield physically correct parameter for the TL process involved.[8]

In present instant a large number of experimental results could be fairly well accounted for using a simple interactive kinetic model suggested by Chen. This model possesses recombination centers by transport through

the conduction band. The two centers emit light in different spectral region, one in the UV (~380nm) and another in the green region (~470nm).[8] Thus, although Al^{+3}-h^{+} centers may indeed by taking part in the TL process at 100°C, as recombination sites, they are not the dominant centers. Another defect, the identity of which is as yet unknown must be acting as the main recombination site. This defect must be acting as a hole trap during irradiation.[9]

In an isothermal annealing experiments, Mckeever et al., suggested that the population of Ge^{+4}-e^{-} center decreases over the temperature region due to increase in the Li^{+}-Ge^{+4}-e^{-} center concentration (because of diffusion and trapping of Li^{+}). An isothermal storage at room temperature clearly indicates that the Ge^{+4}-e^{-} center concentration is still decreasing even after the Li^{+}-Ge^{+4}-e^{-} center concentration has stabilized. It was also noted during the isothermal measurements that the rate of loss of Ge^{+4}-e^{-} centers as measured by ESR approximately matched the rate of loss of TL during isothermal storage implying a strong correlation between the two processes. Therefore TL at 100°C may be related to the concentration of Ge^{+4}-e^{-} center, but that loss of Ge^{+4}-e^{-} centers during isothermal storage (and during heating to 150°C) occur via two processes. One is the trapping of Li^{+} to form stable Li^{+}-Ge^{+4}-e^{-} centers and other is loss of electron from Ge^{+4}-e- sites. The electrons are then free to recombine with holes at both the Al^{+3}-h^{+} (to yield at ~470nm) and at unknown centers (to yield TL at ~380nm). The reaction paths, which are observed to be taking, place during the temperature regime of interest, are summarized below, where U^{+}-h^{+} is unidentified, hole trap.[9]

Ge^{+4}-e^{-}→+Li^{+}→Ge^{+}-e^{-}

↓

← e^{-} →

↓ ↓

U-h^{+} Al^{+3}-h^{+} → +O^{-2}+H^{o}→Al^{+3}-OH^{-} centers

↓ ↓

TL at 380nm TL at 470nm

Thus it is clear that the pre-thermal treatment influences defect structure in the material. It is believed that the internal structure of this material is highly sensitive to the thermal treatment. Annealing the material at various elevated temperature creates effect on internal structure of material. It is reported that the stability of quartz (also known as α-quartz or low-quartz) is up to 573°C under normal pressure. The low-quartz can convert itself to a high-quartz (β-quartz) on heating beyond 573°C. The α-β transition is a displacive phase change involving small adjustment of atomic position without any rearrangement of the bonds. As a results phase change is very fast and is completely reversible, although crystal twinning occurs as the transition goes from α to β and back to α.

Beyond 873°C quartz transforms into another phase i.e. beta to tridymite (~870°C). Thus material annealed up to 1000°C, undergoes α to β and β to tridymite phase transformation. This seems to be responsible for the significant changes in its TL signatures of the material.[10 & 38]

The effect of pre-heat duration on TL of synthetic quartz material has also been undertaken in the present work. In this protocol the grains were held at a given temperature for different duration. It is found that there is not only an increase in TL intensity but also shifting of glow peak position towards lower temperature side. This observation was attributed to a

competing electron traps (CETs) present in the virgin material, associated with proposed Al-hydrogen substitutional center to form E_1' center as a result of thermal pre-treatment followed by beta irradiation at room temperature. It is believed that annealing the specimen for higher duration increases the possibilities of generating E_1' centers, which results in the enhancement in the TL intensity with the increase in annealing duration.

Further, it is also suggested that the activation of pre-existing traps seem to take place as a result of higher duration of pre-thermal treatment. This in turn increased the concentration of a luminescence center. On account of large number, these centers come very close to each other. There exists internal field among themselves. The luminescence centers are not as free for emission as they were in isolated internal field free region. Hence, it is suggested that the luminescence centers under influence of internal field gives rise to shifting of TL glow curve towards lower temperature side.[1]

Moreover, the observed changes in TL characteristics of quartz with either pre-thermal treatment or dose is believed to be due to the production of E_1' centers. This center gives significant contribution to TL results. Martini[5] mentioned that the E_1' center is an oxygen vacancy having an unpaired electrons. Nailson noted that an enhancement in optical absorption of the E_1' center is produced by irradiation followed by 300°C heating. Whereas, Chen[11] suggested that competing electron traps present in virgin synthetic quartz, which are active at trapping electrons in the heating state of the TL read out. The associations of competing electron traps with the proposed Al-hydrogen substitutional center generates a complex referred as E_1' center. It is suggested that the thermal pre-treatment below 300°C supports the competitors to be associated with the proposed center (Al-H), which encourage radiative e-h recombination at the proposed luminescence center site, rather than competing electron trap. This may be the reason for

the enhancement in the TL intensity with increase in annealing treatment up to 300°C.

Jani et al., observed that room temperature irradiation alone doesn't enhance the E_1' center. They suggested two important features of E_1' center in quartz. Efficient production of E_1' center were possible only in unswept quartz. They also observed that E_1' centers could not be formed unless the sample has been irradiated above -73°C.[12] Rink and Odom[13] have discussed a model for E_1' center and its precursor. The precursor of E_1' center, an oxygen vacancy containing two electrons in a singlet state (Si=O), released an electron during the post irradiation heating and these electrons could get trapped at the competing deep traps, rendering the competing deep traps ineffective during the TL readout. The E_1' center becomes unstable and decays in the temperature range 400°C-500°C. This decay would involve the release of the E_1' center electron and results in bared oxygen vacancy. The released electron also has the probability of getting trapped at the competing deep traps. As a consequence, in the sensitized sample probability of recombination center will increase. This will give rise to an increased TL emission.[12]

We observed the complete TL characteristics of synthetic quartz crystal under the influence of different physical conditions at room temperature for different grains sizes also. It is clearly indicated that the finer grain size of 230-270mesh gives impressive results with significant changes in TL pattern particularly TL intensity (growth rate of TL) and area under glow curve compare to coarse grain (120-140mesh) and finest grain (270-325mesh). Also these grains (230-270mesh) are highly sensitive to annealing temperature as well as duration of pre-heat treatment.

The present work includes the treatment of material by mechanical actions such as grinding (milling), crushing, use of machinery for sawing

and cutting, which may result in the production of defect structure. However, the shape of glow curves at temperatures above 300°C doesn't reveal the same. This may be due to either to the chemical changes (impurities introduced into the powders) during the grinding process or to the surface area effects. Also, from literature survey, the surface alterations following a prolonged grinding, which results in significant changes in the physical and chemical characteristics of material. Further, A.H.Ranjbar et al., the TL output increases with decreasing particle size, but then declines for particle sizes below certain grain. Although below a certain size TL intensity decrease, which is attributed to a decrease in the available trapping centers, it is again thought that the former increase of TL intensity is due to an increase in the surface area per unit mass for the lower particle size of the powders.[14] Current work also shows identical pattern of results. Therefore, finer grain of 230-270mesh has been used for further study.

4.2 A Selection of 470nm stimulation wavelength for the present OSL study.

Mckeever[10] and Prescott et al.,[15] have studied spectral studies of quartz. But the detailed observations of M.R.Kribetschek et al.,[16] revealed that the quartz has three main TL emission bands at (i) 360-440nm(near UV to violet) (ii) 460-500nm(blue) (iii) 600-650nm(orange) region.

(i) 360-440nm

The TL emission band in this wavelength region is associated with $(H_3O_4)^{\circ}$ hole center on a Si vacancy as subjected by Nuttall and Weil[17]. Yang et al.,[6] supported this statement by observing TL emission spectra at 380nm wavelength related to 110°C peak in quartz. There are further TL peaks at higher temperature and their emission wavelengths depend on

corresponding temperature. Scholefield et al., explained this concept to support the above statement.[18] Franklin et al.,[19] categorically showed that

95°C- 110°C TL peak is emitting at 376nm.

150°C-180°C TL peak is emitting at 392nm.

200°C-220°C TL peak is emitting at 410nm.

305°C-325°C TL peak is emitting at 430nm.

For the later peak, thermal quenching of the luminescence has been found (Wintle) and the observations of Franklin et al., showed this effect to be the characteristics of all these peaks. Scholefield et al., have described the 325°C peak as a broad peak in energy with a symmetry factor in the temperature shape indicating first orders kinetics, as noted by Wintle.

OSL studies (Smith et al.,) have shown link between OSL and the optically induced decay of the 305-325°C TL peak (natural signal); both bleach rapidly, and also without UV light bleachable TL peak emits at about 420nm(Prescott and Fox, Scholefield et al.,). Franklin et.al reported that the TL emission listed above form a "family" of rapidly bleaching TL peaks which all feed the same luminescence center using the conduction band for electron transport.

(ii) 460-500nm

$(AlO_4)^0$ on a Si site seems to be responsible for 460-500nm emissions but other defect may also be associated with this emission. Hornyank et al.,[20] observed, this forms TL peak at 375°C, which is relatively broad, both in temperature and wavelength and it is asymmetric with extension towards lower temperature and higher energy.

(iii) 600-650nm

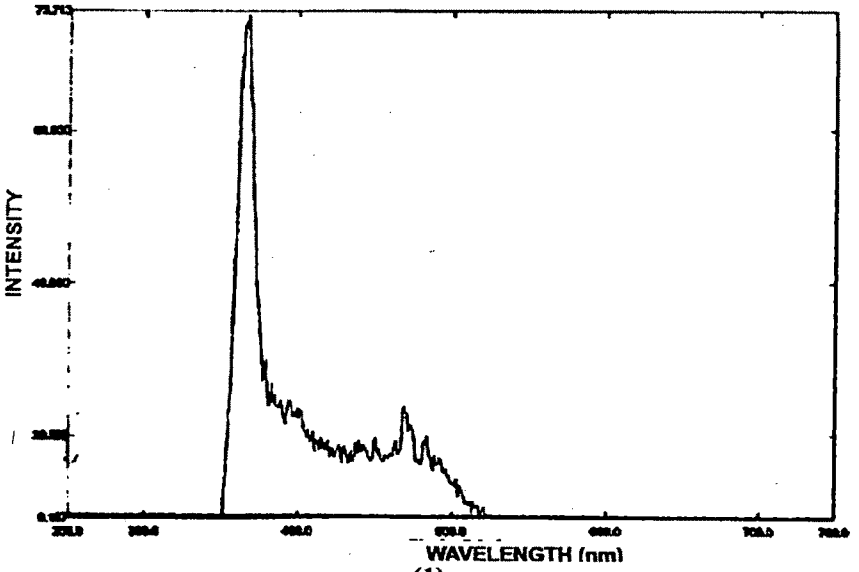
It is the less intense orange TL emission band (mostly reported as red TL) for which there is no clear link between the emission and particular type of defects.

The Photoluminescence (PL) studies of laboratory grown synthetic quartz gives the emission band in the bluish green (~470nm) region (see PL spectra-1, 2 and 3). As already mentioned, this emission corresponds to 375°C TL peak which is attributed to electron trap complex in quartz.[20] Moreover, it is worthwhile to recall that Jani et al., proposed the high temperature TL peak is emitted at about 470nm, but for ~110°C TL peak Zimmerman argued that it was predominantly emitted at 380nm, with a weaker emission at 470nm also[21]. Thus it is reasonable to believe that the same electron trap complex may be responsible for both the TL peaks. Since high temperature TL peak is of interest from the point of view of dating and dosimetry application, the present work mainly concerns with this TL emission peak {it is already mentioned that RBP i.e. 325°C and SBP i.e. 375°C are highly involved in such applications}.

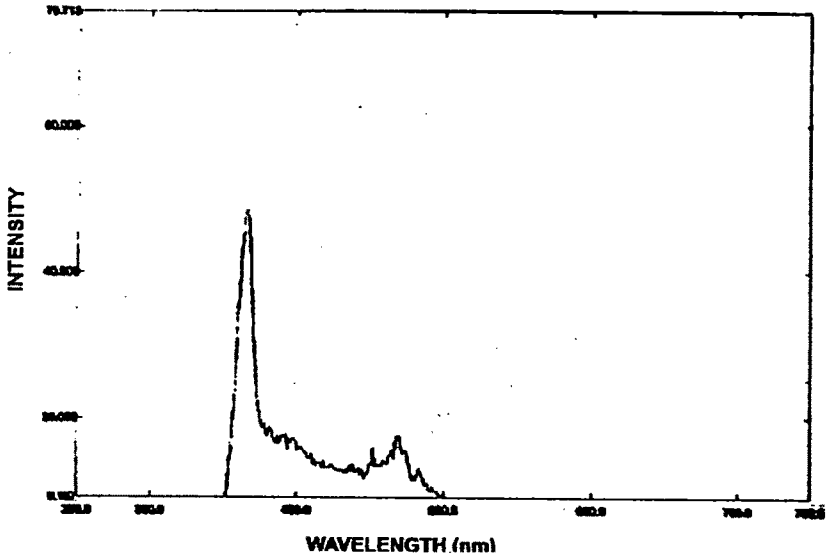
Thus the selections of 470nm light was made for the purpose of optical stimulation of irradiated material for different protocols.

Various sources like ion-argon laser and green LEDs may be used for optical stimulation of quartz. R.B.Galloway et al., [21] recently developed blue LEDs, which was tested with the aim of the using this device for visible light stimulation of quartz for dating and retrospective accident dosimetry. He showed the several practical advantages of using LEDs over other light sources such as filtered broadband lamps and laser. They are inexpensive, heat dissipation is negligible, there is no radiation loss, they are easy to operate and the power delivery can be controlled electronically, and hence can be programmed also.

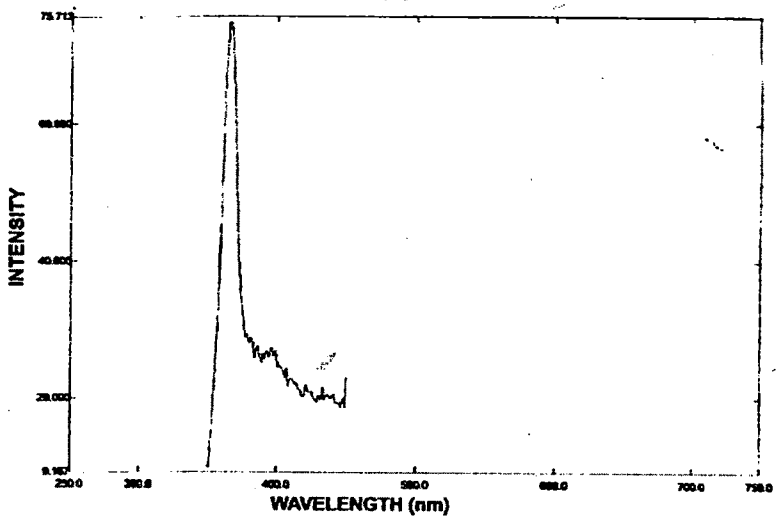
A blue LED OSL configuration has been described and compared with a broadband green/blue light source. It was found that for similar power densities, the high energy light provided by blue LEDs gives a very much greater quartz stimulation efficiency than that of green light, which is



(1)



(2)



(3)

Photoluminescence spectra of synthetic quartz at room temperature by (1) 220 nm (2) 260 nm (3) 250 nm excitation wavelength

consistent with the known exponential relationship between OSL and photon energy. A blue LED system delivering 6mWcm^{-2} to the sample stimulates quartz with a similar efficiency to that of broadband blue/green halogen source delivering 28mWcm^{-2}. Rapid switch on/off time allow pulses as brief as 5ms to be delivered using LEDs. This, combined with software-controlled power, greatly improves the flexibility of the illumination source compared with the broadband system.

Pre-heat experiments on quartz strongly suggest that the same sets of OSL traps in quartz are affected, whether using blue LED or broadband blue/green light. It is concluded that there is no evidence for enhanced stimulation of deep OSL trap by the higher energy blue LED light source. It is for the first time that the clearly distinguished OSL peaks have been identified as being associated with 325°C and 110°C TL traps. It is concluded that this new approach has great potential for further understanding of luminescence processes. It is very likely that there will be significant further improvements in high-energy visible light LED technology in the near future. However, existing designs are already very suitable as stimulation sources in OSL, and have great practical and experimental advantages over existing sources.

4.3 OSL measured at room temperature.

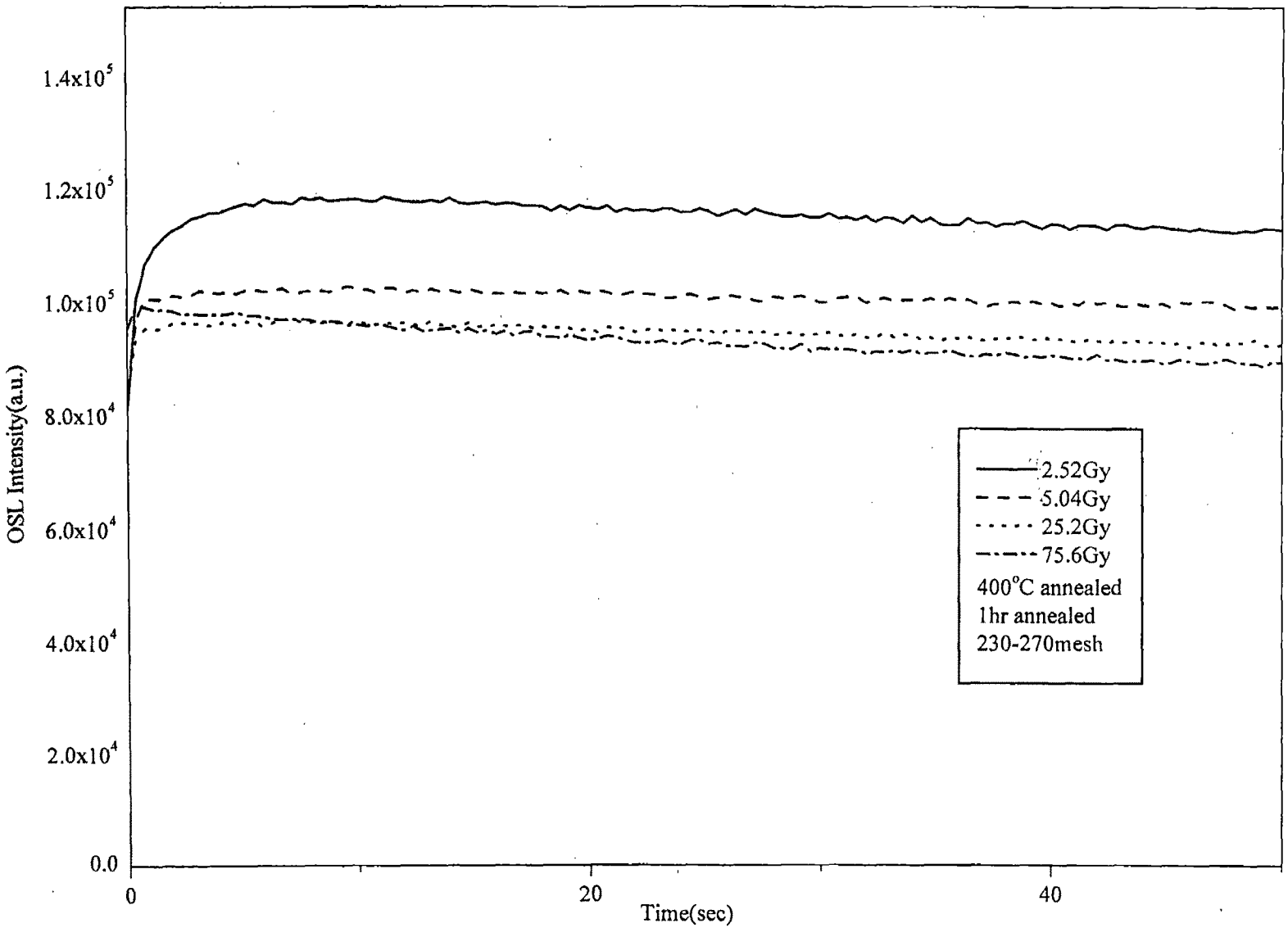
As already mentioned, the OSL technique has many advantages over TL. The specimens were optically stimulated at 470nm, which corresponds to high temperature as well as low temperature TL peak. In order to study OSL decay for the specimens pre-heat treated at different temperature and durations, specimens were irradiated for different beta doses. OSL was measured at room temperature for each protocol while correlating with TL at

room temperature for similar protocol. Role of optically sensitive traps can be investigated more systematically with these efforts.

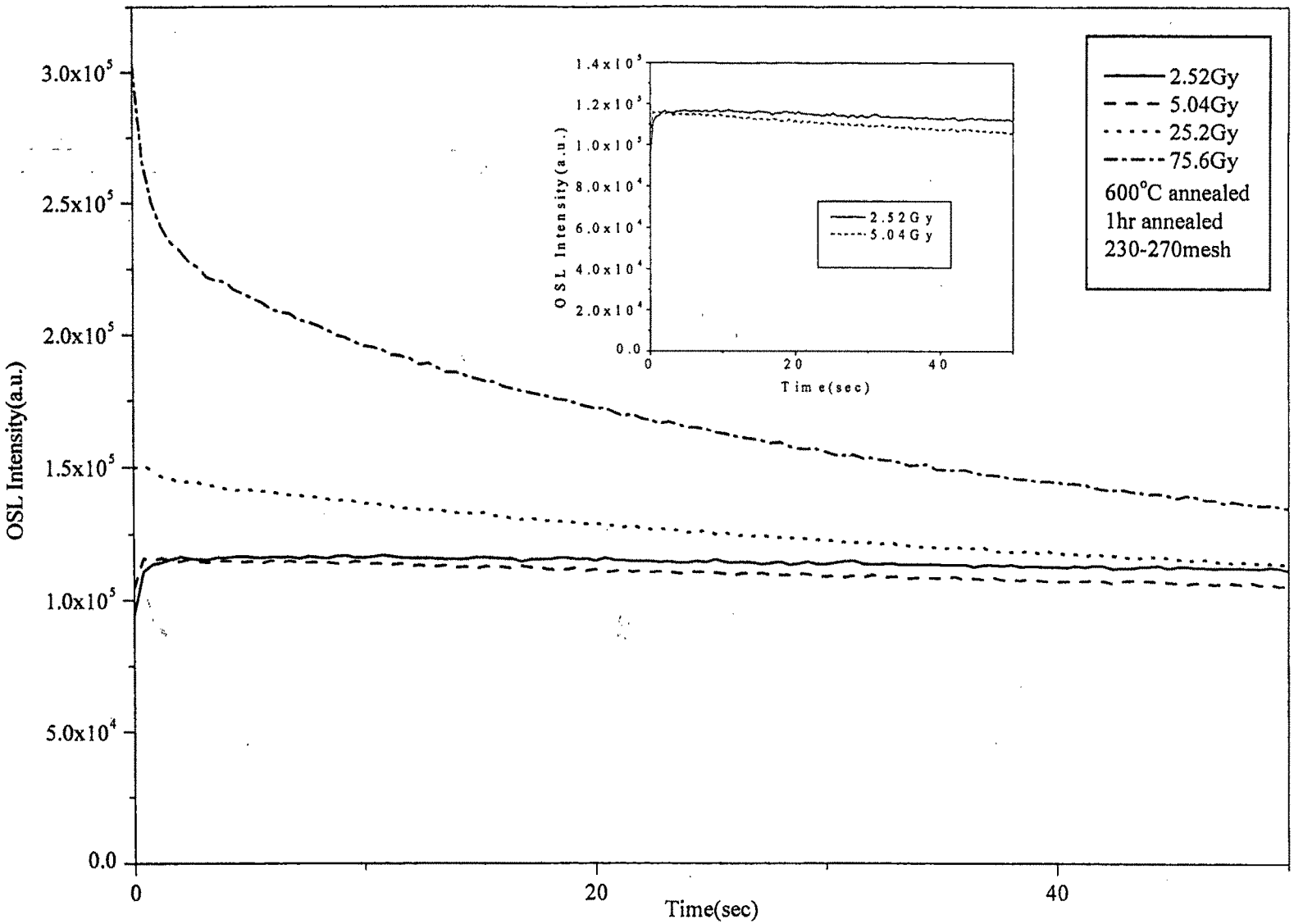
Graph-6 to **Graph-8** show OSL at room temperature for the 230-270mesh powdered specimens, pre-heat treated at 400°C, 600°C and 1000°C for 1hr duration then rapidly air quenched respectively. Prior to OSL decay each specimen were given 2.52Gy to 75.6Gy beta dose. Materials annealed at 400°C do not show exponential decay for any dose of observation. In yet another study, it has been found that initial stimulation peak intensity time reduces from 11.2 to 0.4sec with rise in beta dose. While, material annealed at 600°C with rise in dose, shows similar results but for specimens exposed to 75.6Gy dose, initial stimulation peak time becomes completely zero sec and curve becomes completely exponential in nature.

Material pre-heat treated at 1000°C for 1hr shows that initial stimulation time remains very low and it reduces with rise in beta dose. Specimens were exposed in this case for even higher doses up to 302.4Gy. It is found that exponential OSL decay is discernible for specimens exposed at higher than 75.6Gy beta dose, before this dose initial stimulation time remains very low to 0.4sec.

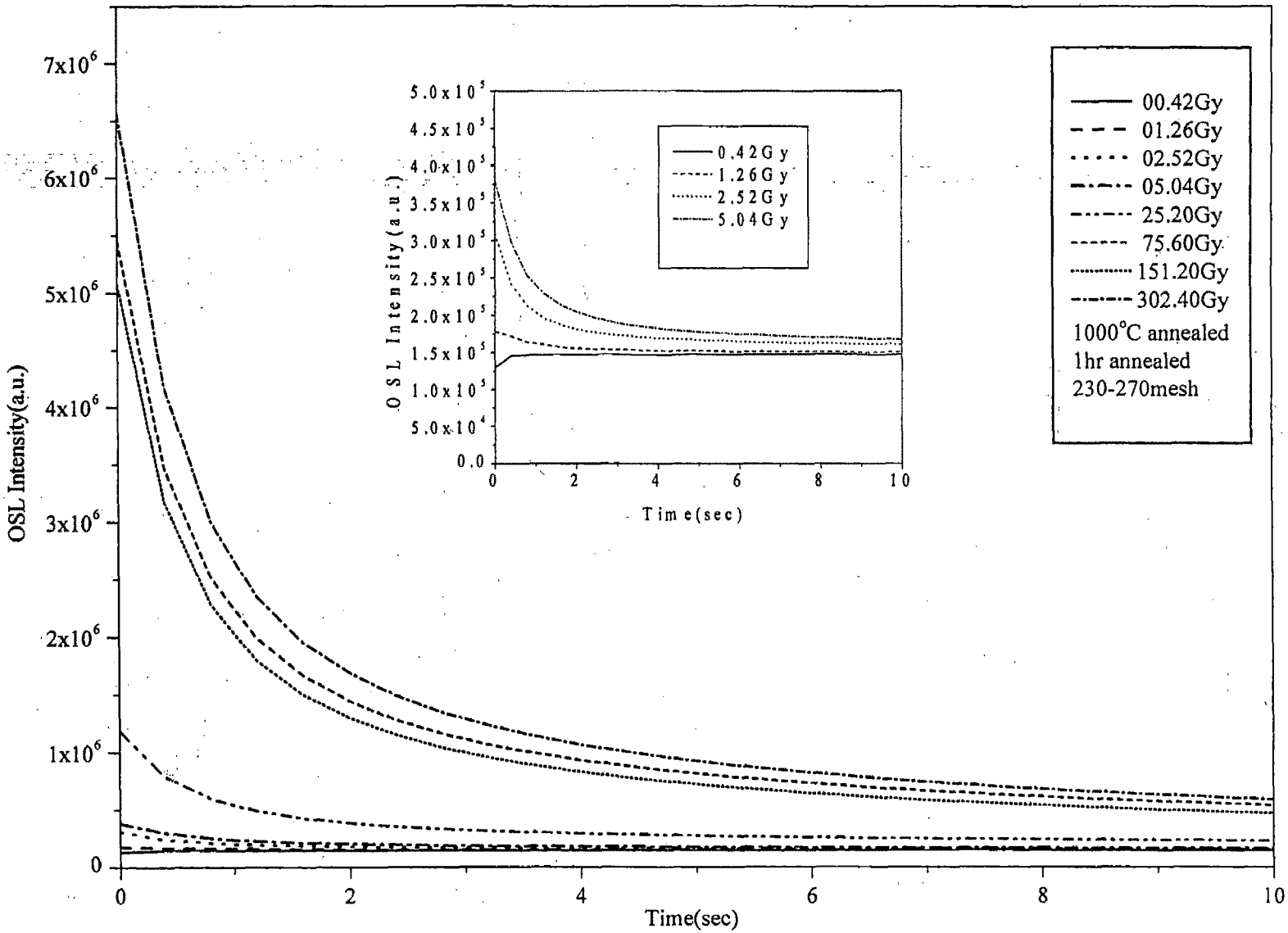
The changes in initial stimulation time for different protocols can be observed from the width of OSL decay curve. The specimen pre-heat treated at 400°C shows no change in width even with rise in beta dose. It means that the curve remains non exponential. However, grains annealed at 600°C show remarkable change in width after 25.2Gy dose, it started decreasing with further rise in beta dose. It is clear that at this point the OSL curve starts becoming exponential. Identical patterns are observed even for specimens annealed at 1000°C. This clearly means the full width of OSL decay is sensitive to the dose as well as pre-heat treatment.



Graph-6 OSL measured at room temperature of 400 °C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-7 OSL measured at room temperature of 600 °C annealed sample for 1hr duration at different doses for 230-270mesh.



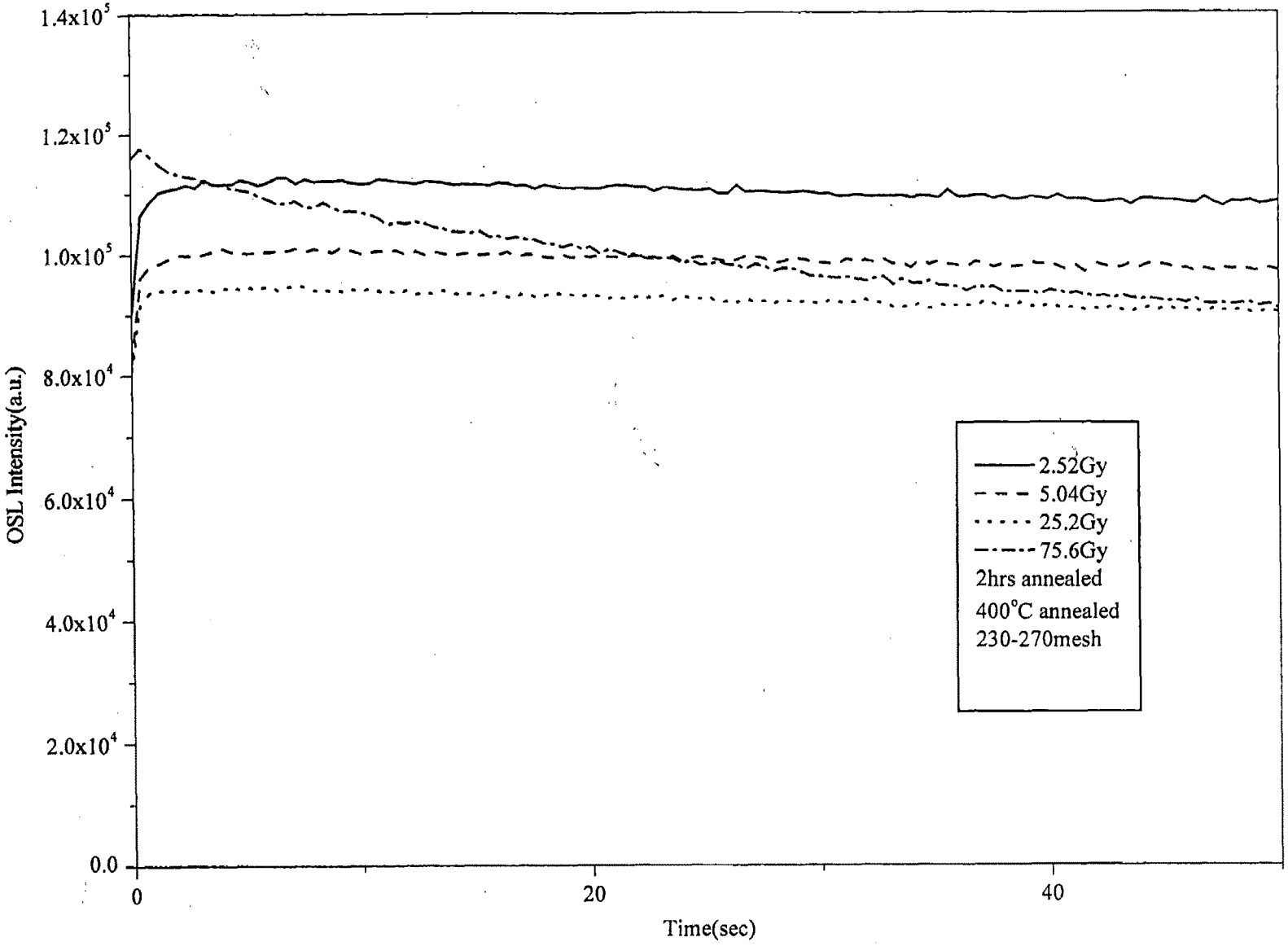
Graph-8 OSL measured at room temperature of 1000 °C annealed sample for 1hr duration at different doses for 230-270mesh

Graph-9 to **Graph-11** show OSL decay curves for higher duration of pre-heat treatment of 2hrs for identical protocol like 1hr, it is noted that 400°C annealed material gives non-exponential characteristics even with rise in beta dose up to 75.6Gy. Whereas 600°C & 1000°C pre-heat treated material show non-exponential decay curve up to 5.04Gy and 2.52Gy beta dose respectively. Beyond this dose OSL decay is exponential with rise in OSL intensity.

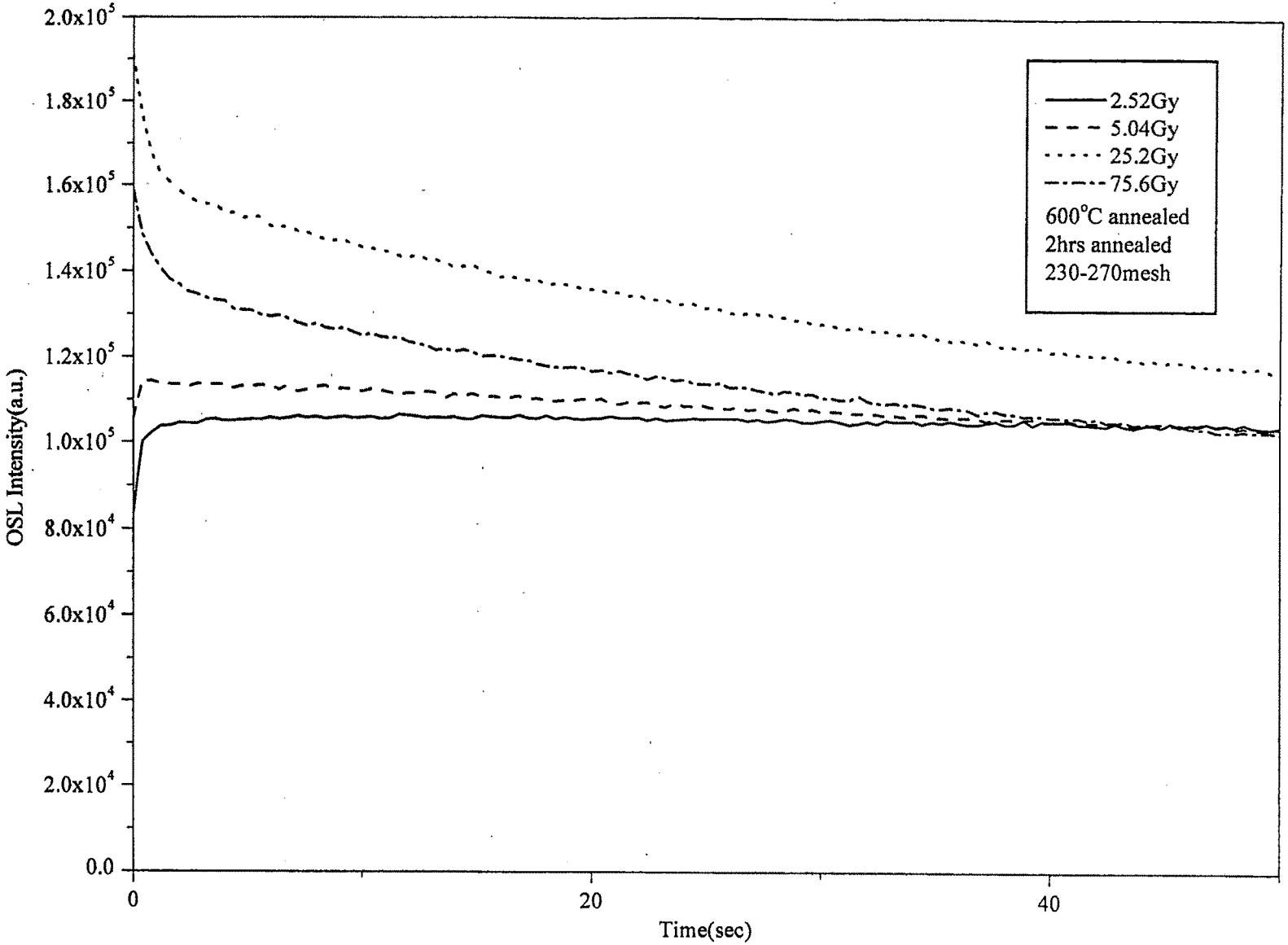
Further, rising the duration of pre-heat treatment to 3hrs for similar protocol indicate that an identical pattern of shape of OSL curve is observed for 400°C annealed and quenched grains. But noticeable changes are seen for 600°C and 1000°C pre-heat treated materials. In this treatment, it is found that the shape of OSL decay curve becomes exponential from 2.52Gy beta dose, which is faster decay compared to 2hrs pre-heat duration results. These results are obviously clear from **Graphs-12** to **Graph-14.**

A comparison made for the OSL characteristics of quartz specimen with another grain size of 120-140mesh at room temperature, which is pre-thermally treated at 1000°C for 1hr and exposed to different beta doses were studied. The results obtained are presented in **Graph-15.** Here it is found that a specimen pre-heat treated at 1000°C for 1hr duration exhibits non-exponential decay up to beta dose ~2.52Gy after that it becomes exponential in nature for each dose up to 302.4Gy beta dose. Further, to understand the effect of pre-thermal treatment on OSL decay curves of this grain size the specimens were given pre-heat treatment at 400°C, 600°C, 800°C & 1000°C for 1hr duration, each specimens was given identical 5.04Gy beta dose before OSL measurement. The decay curves are displayed in **Graph-16.**

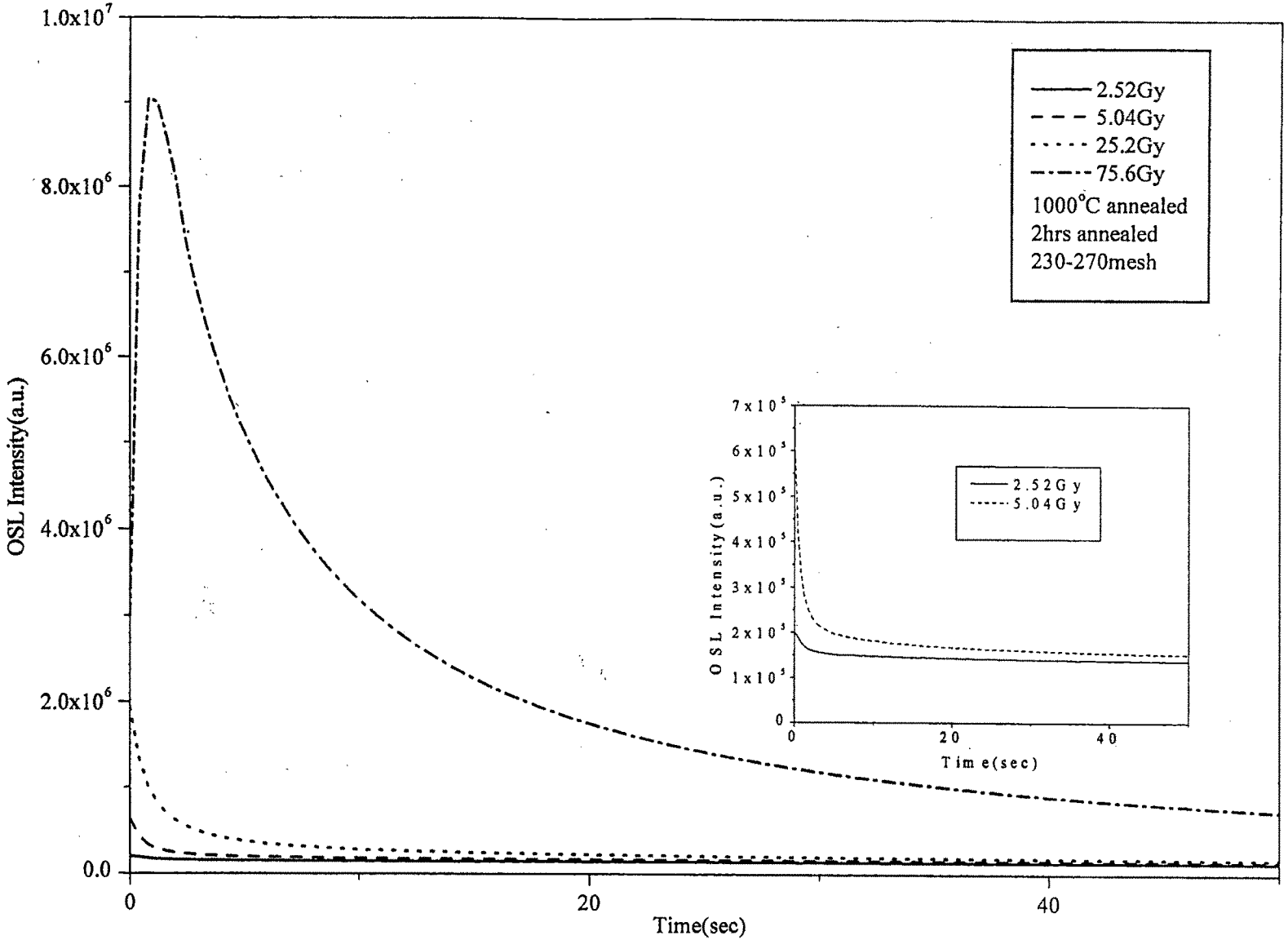
The OSL curves indicate that 1000°C pre-thermal treatment highly influences the shape of OSL decay curve compared to those of other pre-heat



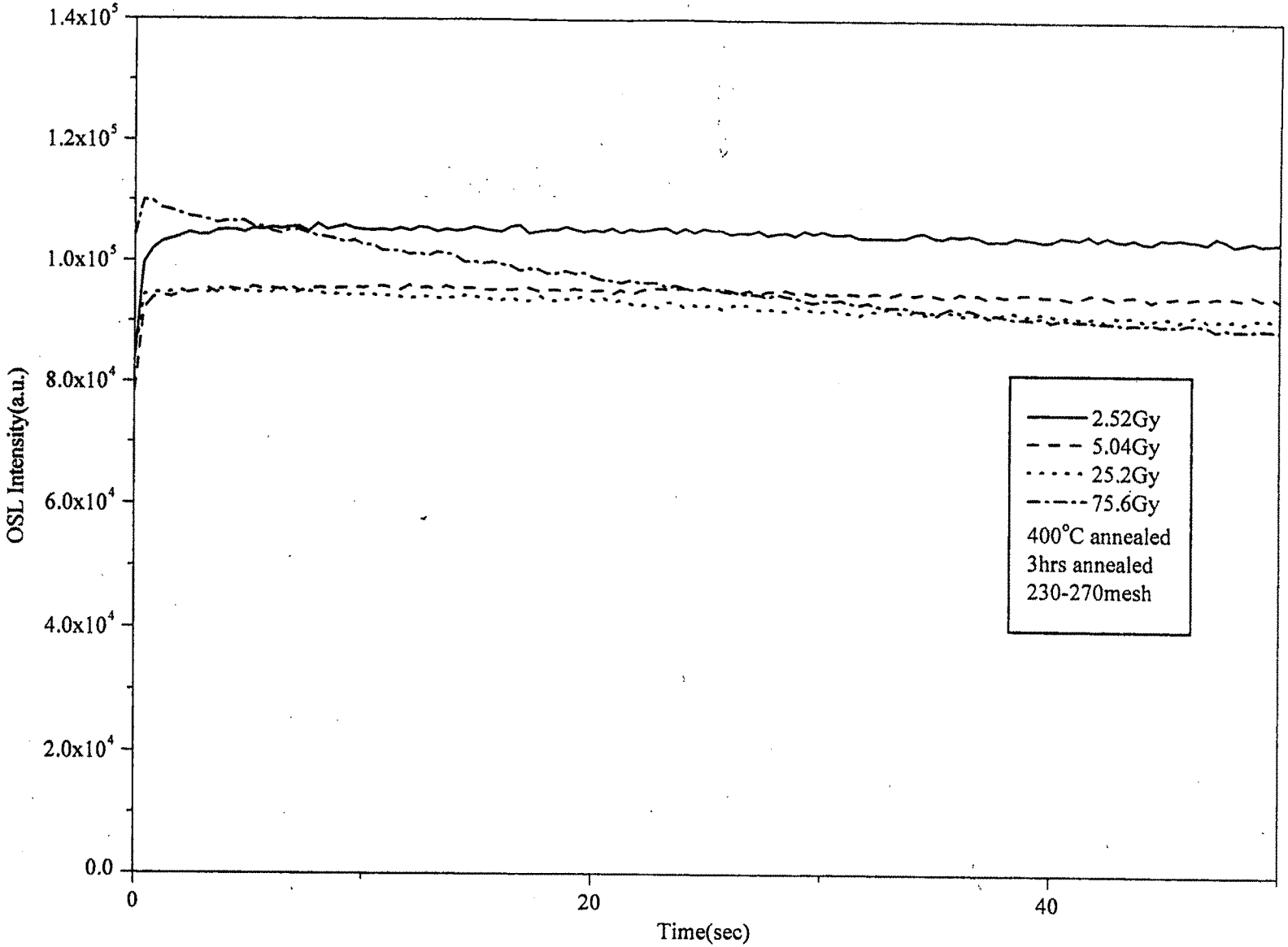
Graph-9 OSL measured at room temperature of 400 °C annealed sample for 2hrs duration at different doses for 230-270mesh.



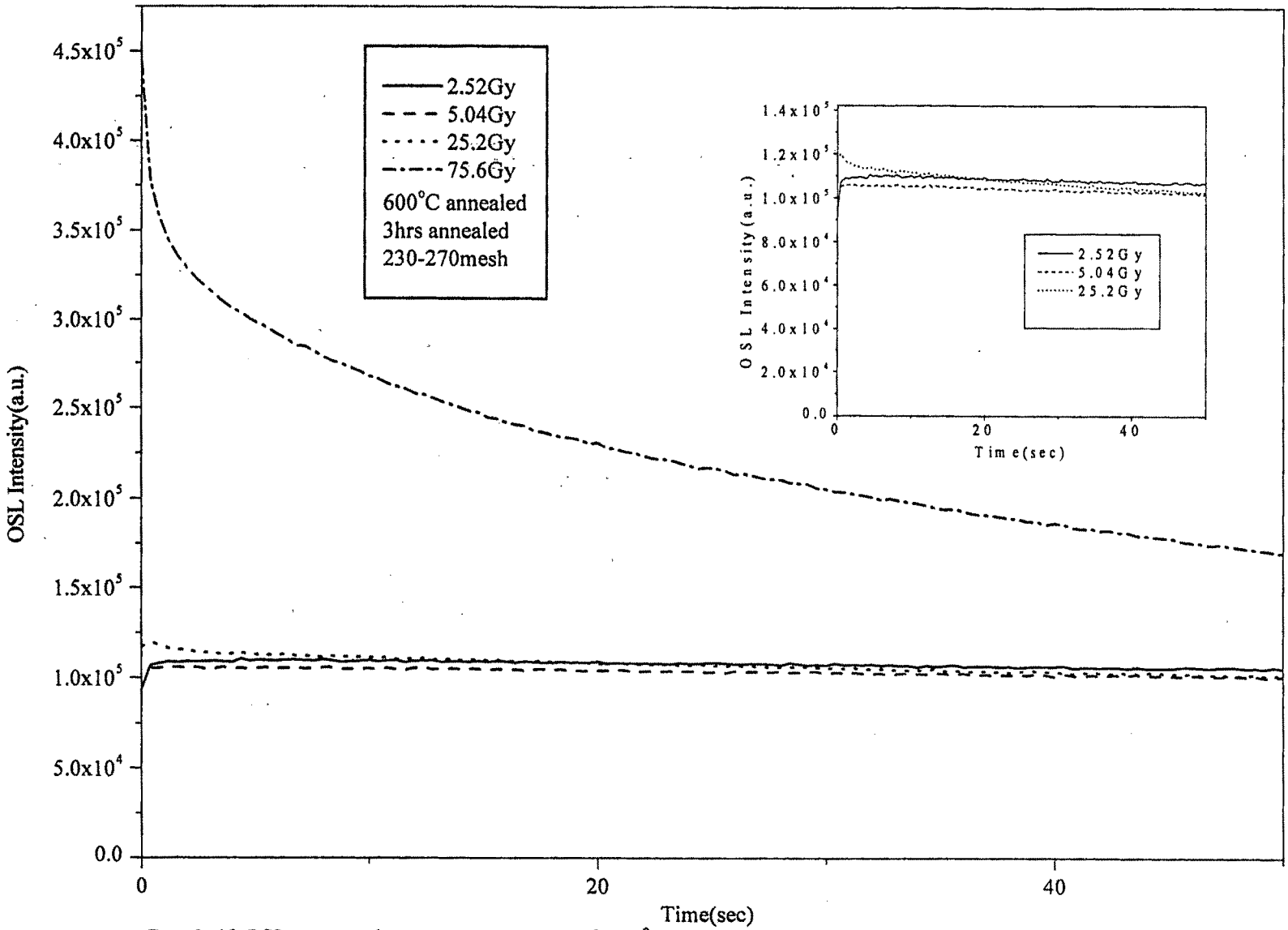
Graph-10 OSL measured at room temperature of 600 °C annealed sample for 2hrs duration at different doses for 230-270mesh.



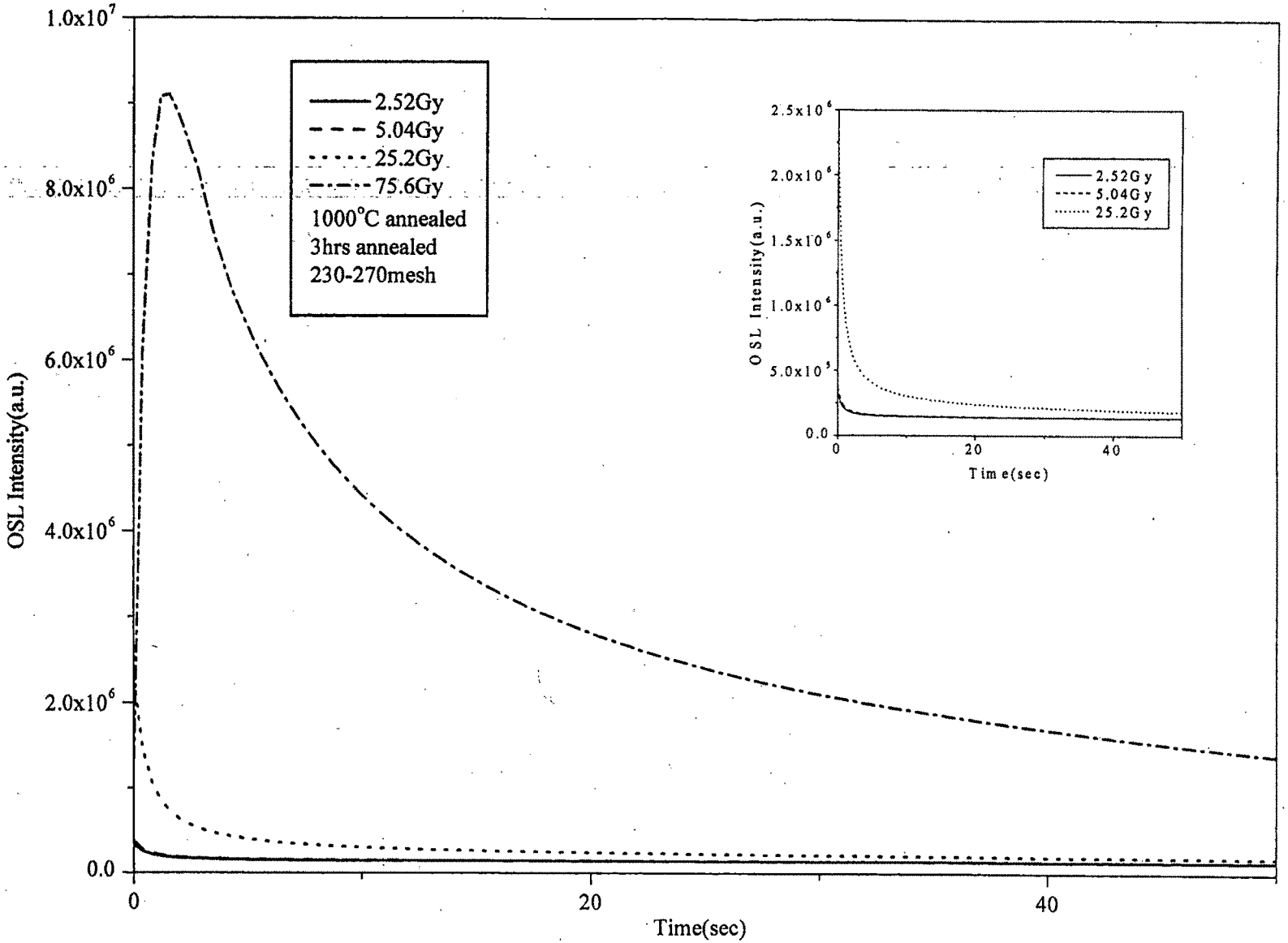
Graph-11 OSL measured at room temperature of 1000 °C annealed sample for 2hrs duration at different doses for 230-270mesh.



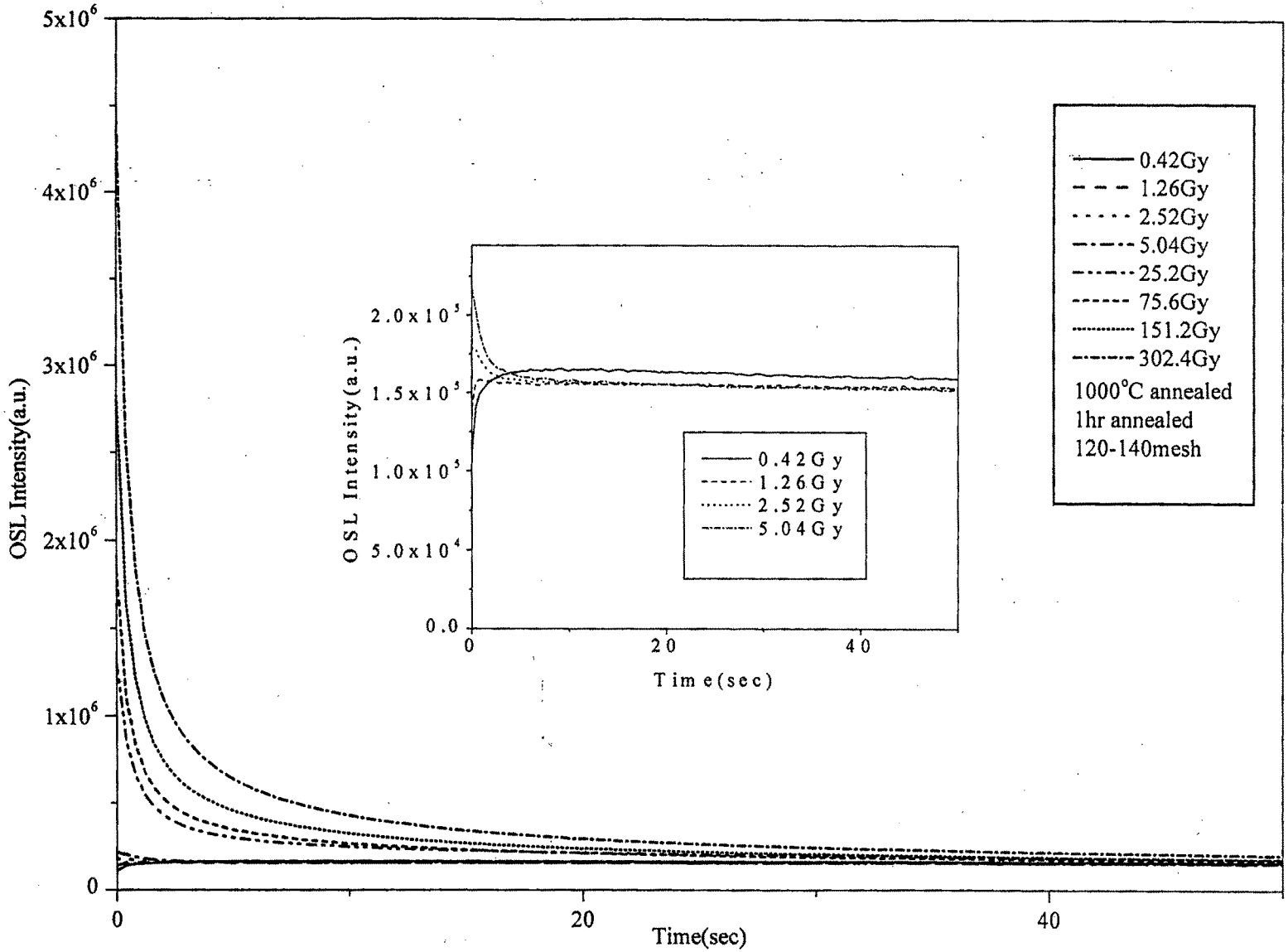
Graph-12 OSL measured at room temperature of 400 °C annealed sample for 3hrs duration at different doses for 230-270mesh.



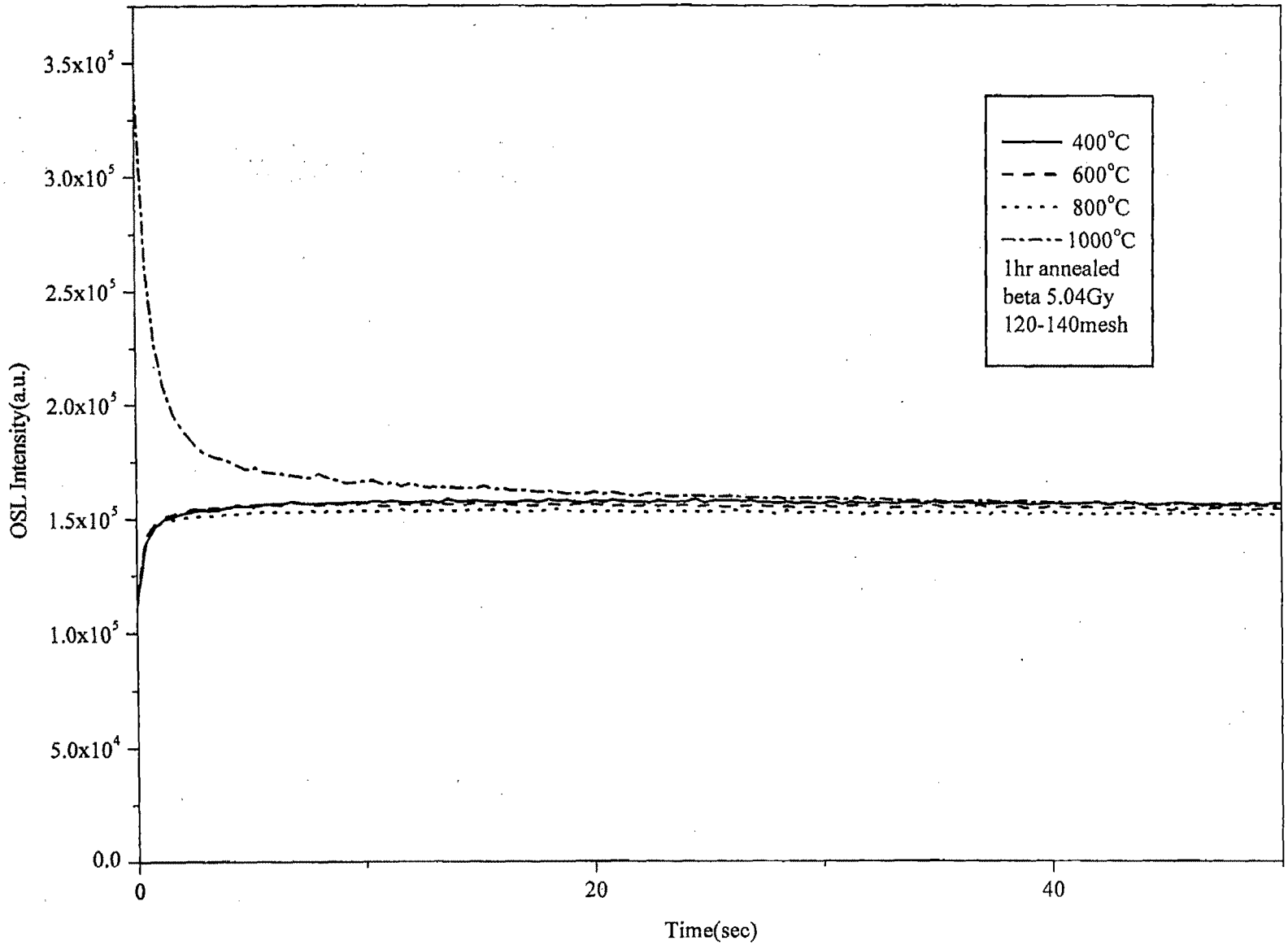
Graph-13 OSL measured at room temperature of 600 °C annealed sample for 3hrs duration at different doses for 230-270mesh.



Graph-14 OSL measured at room temperature of 1000 °C annealed sample for 3hrs duration at different doses for 230-270mesh.



Graph-15 OSL measured at room temperature of 1000 °C annealed sample for 1hr duration at different doses for 120-140mesh.



Graph-16 OSL measured at room temperature of 1hr annealed sample for a given dose of 5.04Gy at different annealing temperatures for 120-140mesh.

treatment specimens. In other words, unlike specimens the 1000°C annealed material gives completely exponential shape of decay curve.

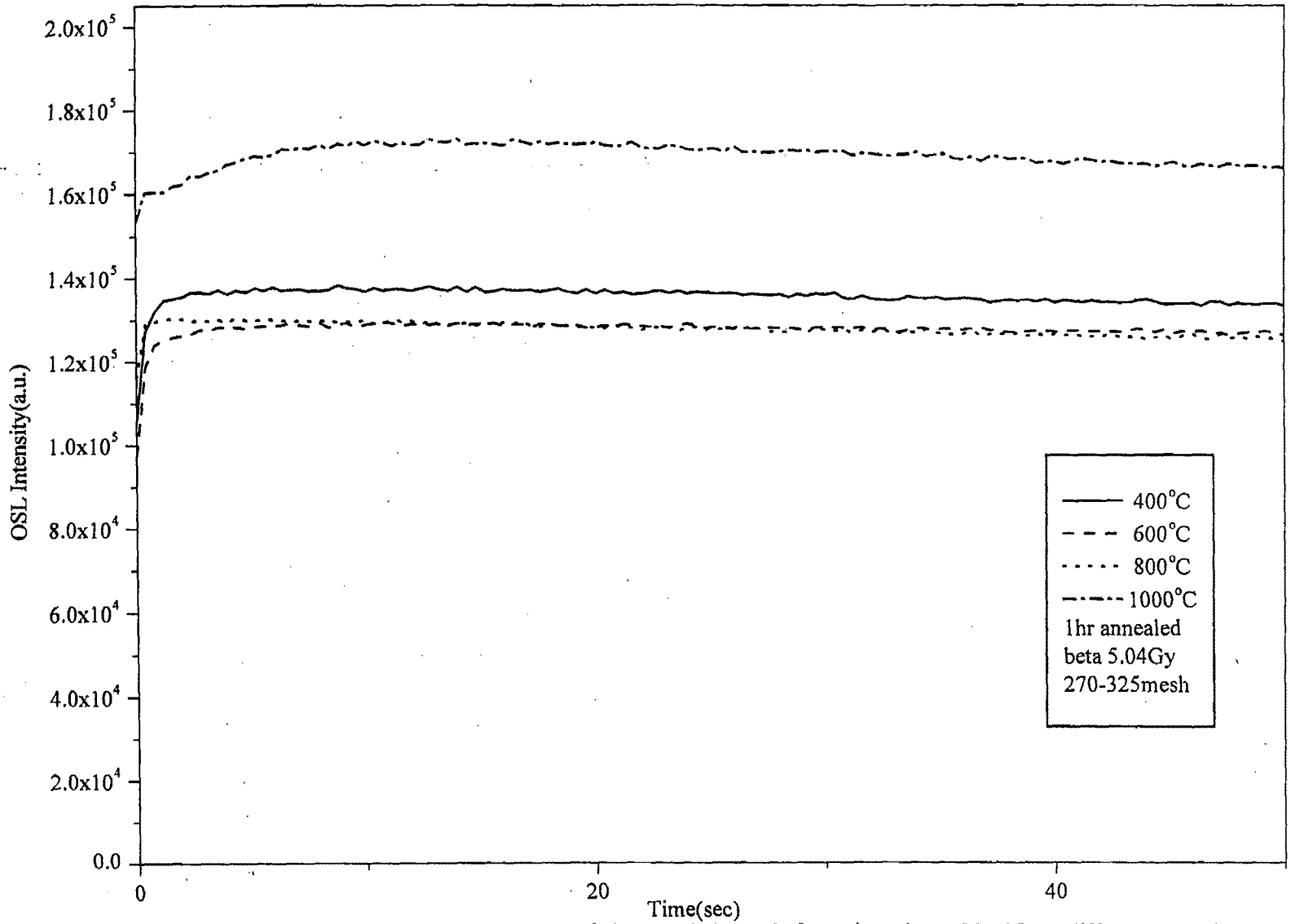
The same protocol was also followed for the finest grains of 270-325mesh. It has shown that the specimens pre-heat treated at 1000°C for 1hr duration gives a noticeable changes like non-exponential shape with faster decay unlike the finer grains. But for both the grain sizes, specimens pre-heat treated below 1000°C show non-exponential decay, having the difference in the decay rates see in **Graph-17**.

In yet another set of observations OSL intensity was compared against the different beta doses given to the specimen pre-heat treated at a temperature for different duration, with a view to understand the effect of doses for different pre-heat treatment duration.

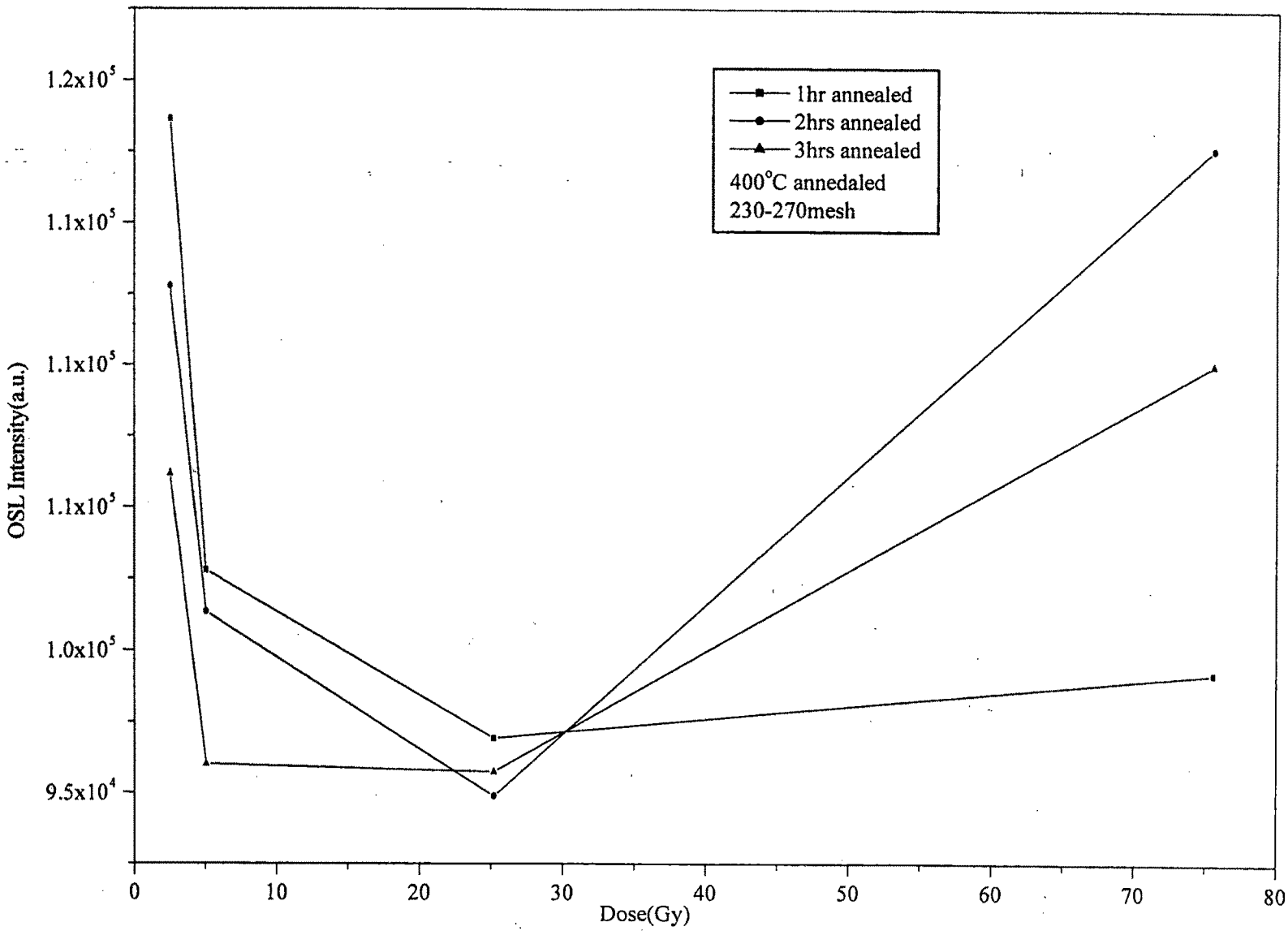
Graph-18 to **Graph-20** display the OSL dose response curves for 230-270mesh size grains annealed at 400°C, 600°C and 1000°C for different durations respectively.

Here it is found that for the specimen pre-heat treated at 400°C, as the beta dose increases, OSL intensity decreases up to 25.2Gy, however, it increases beyond 25.2Gy dose. Similar pattern is observed for 2hrs and 3hrs duration with only change in respective OSL intensity. It is clear that for the same dose the OSL intensity decreases with increase in duration of pre-thermal treatment. However, grains annealed at 600°C shows rise in OSL intensity beyond ~5Gy with the rise in beta dose **Graph-19**, but for the specimen annealed at 1000°C it shows the growth in OSL intensity with the rise in beta dose for all the durations of pre-thermal treatment.

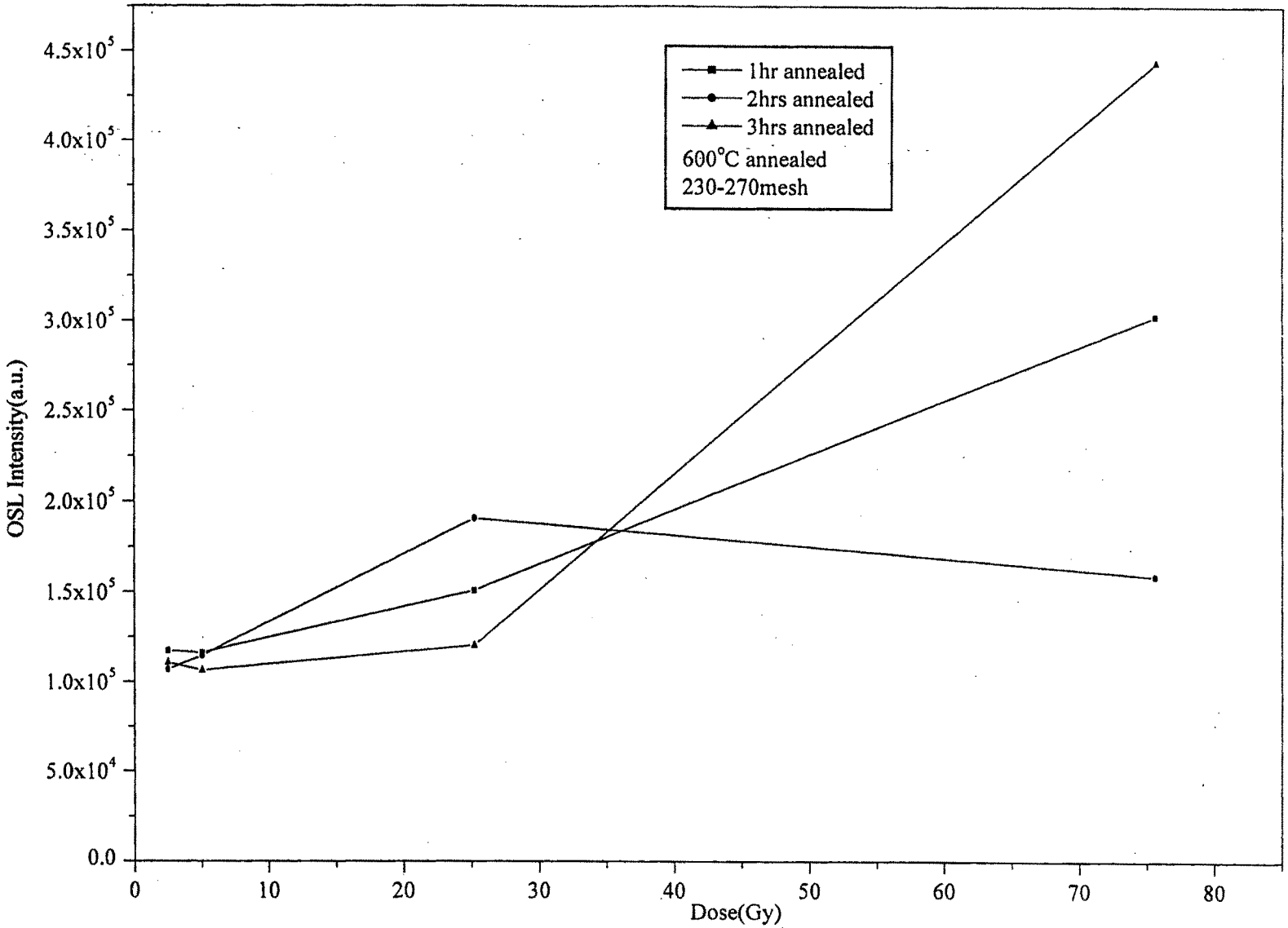
Graph-21 to **Graph-23** display the area under the OSL curves against beta dose. These observations are derived from the actual OSL decay data for three different annealing temperatures 400°C, 600°C and 1000°C respectively. However for the purpose of comparison different duration of



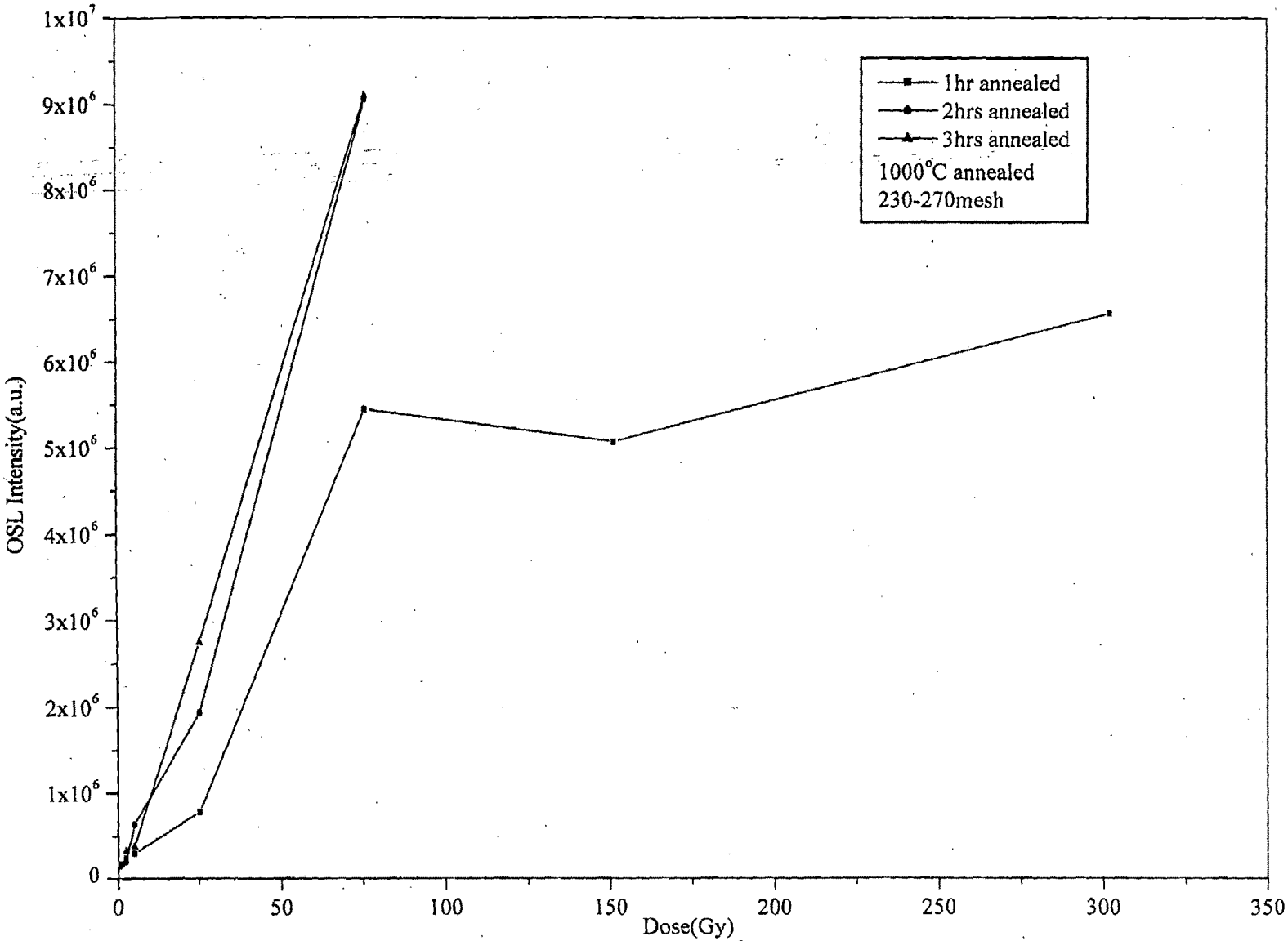
Graph-17 OSL measured at room temperature of 1hr annealed sample for a given dose of 5.04Gy at different annealing temperatures for 270-325mesh.



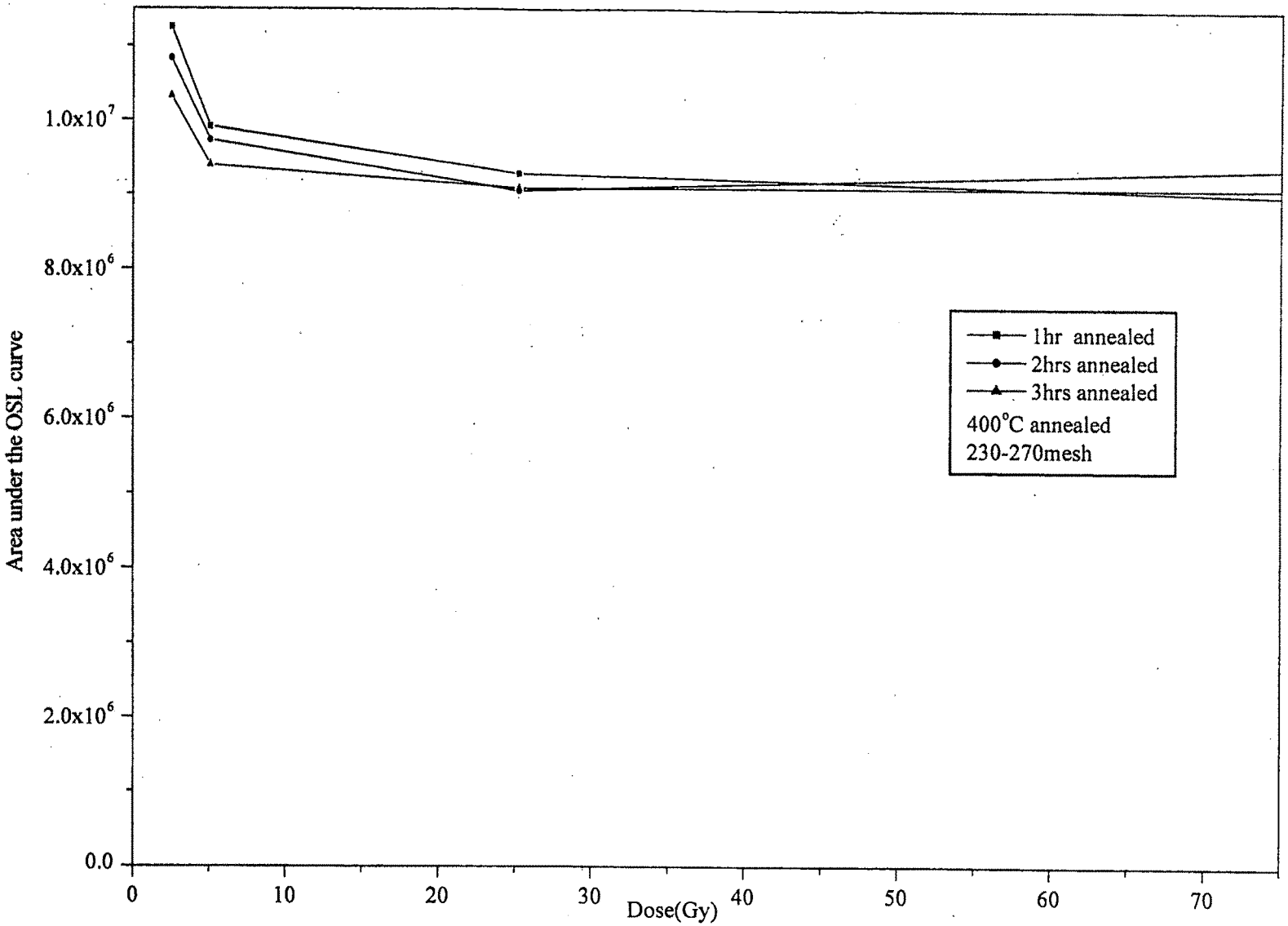
Graph-18 OSL dose response curve at room temperature of 400 °C annealed sample for 230-270mesh at different annealing durations.



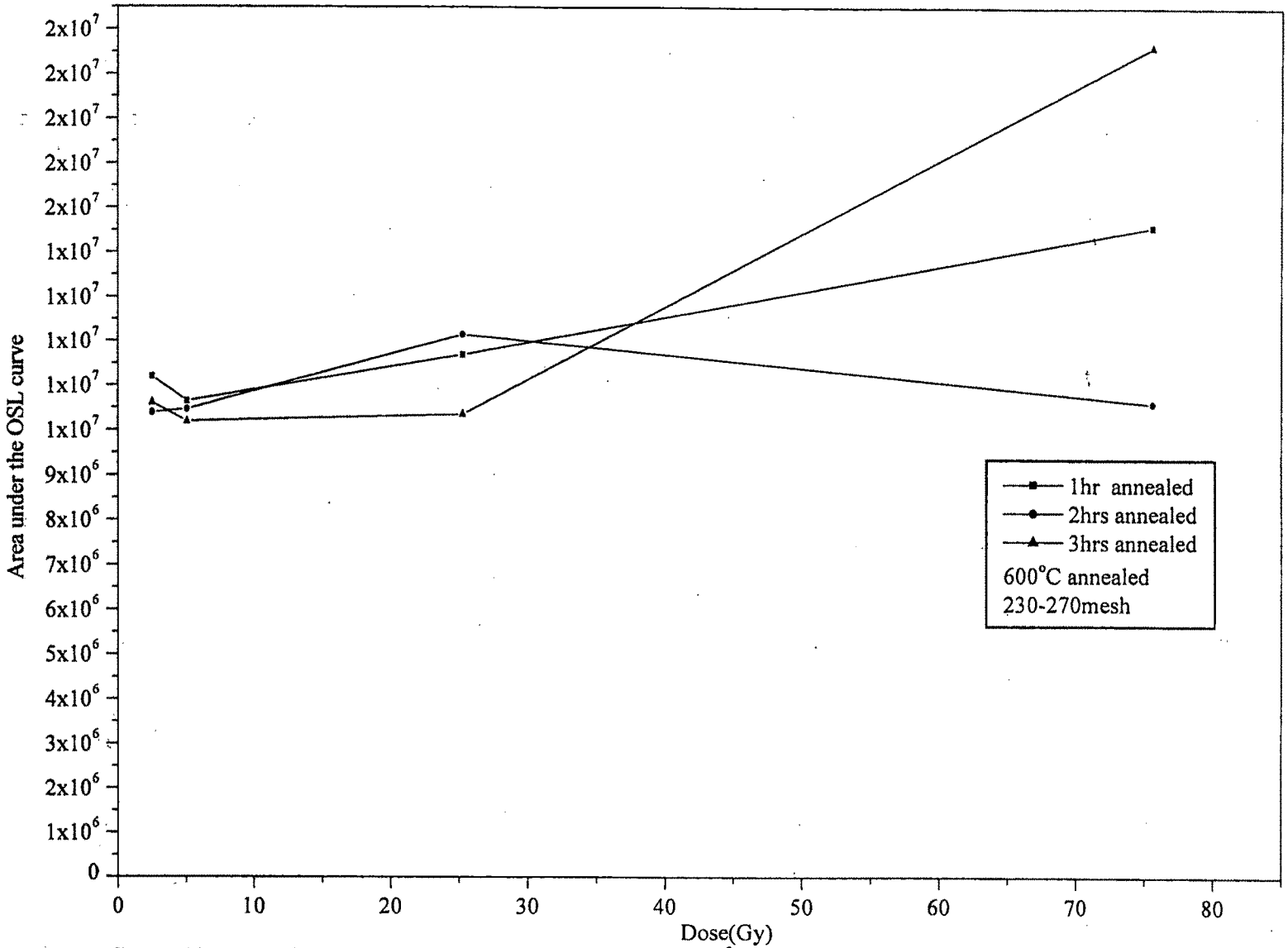
Graph-19 OSL dose response curve at room temperature of 600 °C annealed sample for 230-270mesh at different annealing durations.



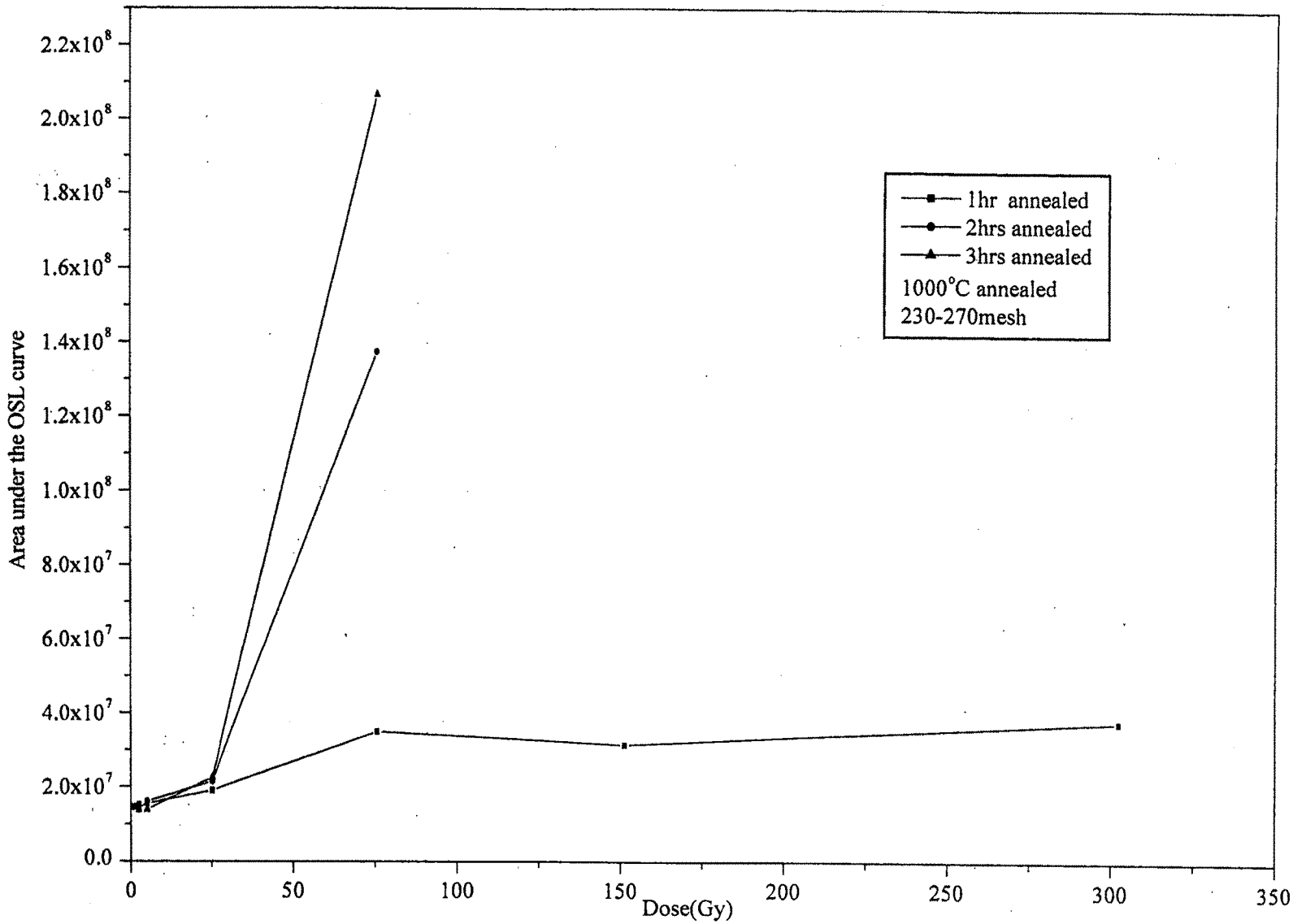
Graph-20 OSL dose response curve at room temperature of 1000 °C annealed sample for 230-270mesh at different annealing durations.



Graph-21 Area under the OSL curve against dose at room temperature of 400 °C annealed sample for 230-270mesh at different annealing durations.



Graph-22 Area under the OSL curve at room temperature of 600 °C annealed sample for 230-270mesh at different annealing durations.



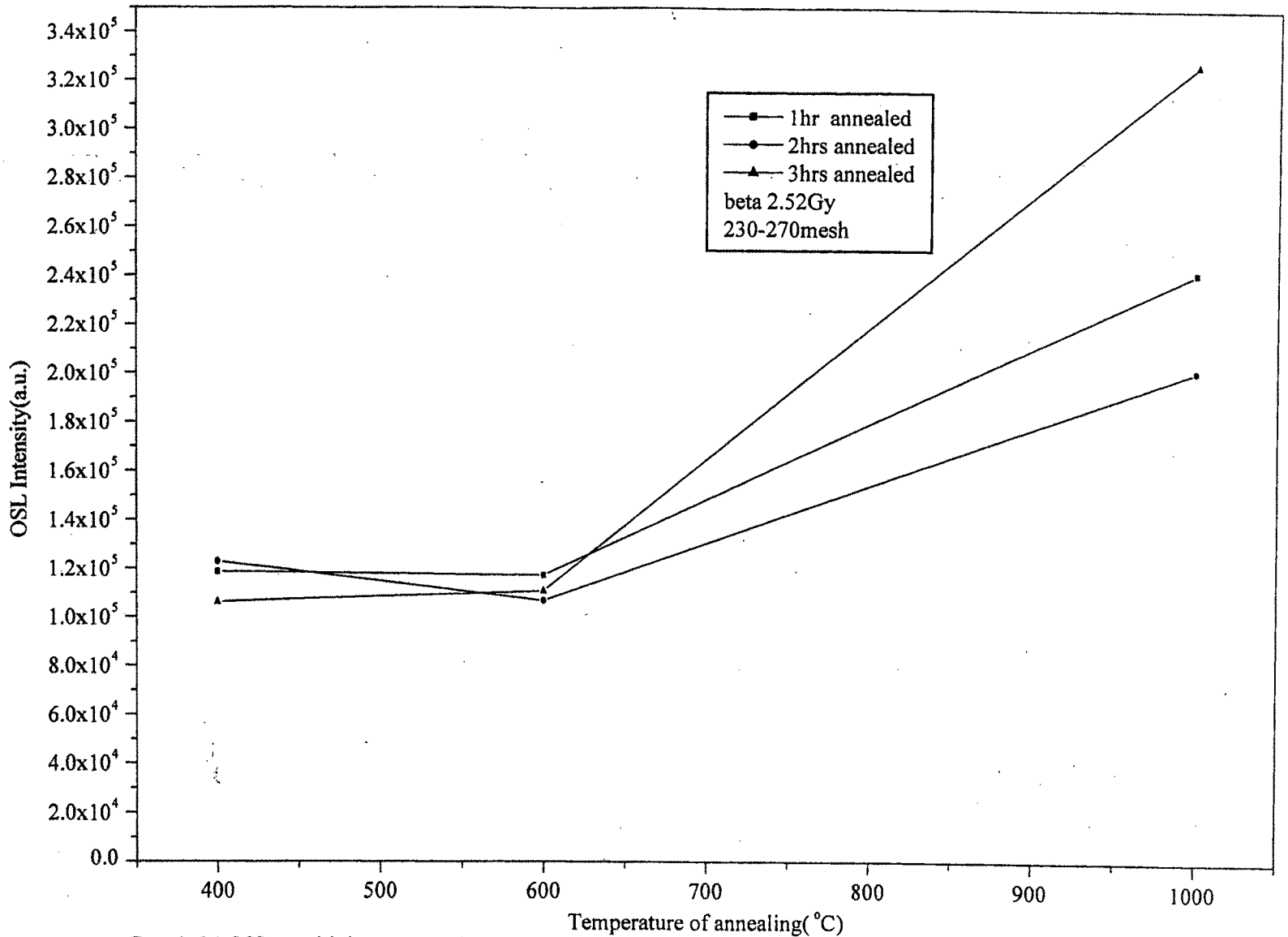
Graph-23 Area under the OSL curve at room temperature of 1000 °C annealed sample for 230-270mesh at different annealing durations.

pre-heat treatment was also considered in each graph. This study is aimed at finding the influence of such protocols on discernible OSL center concentrations.

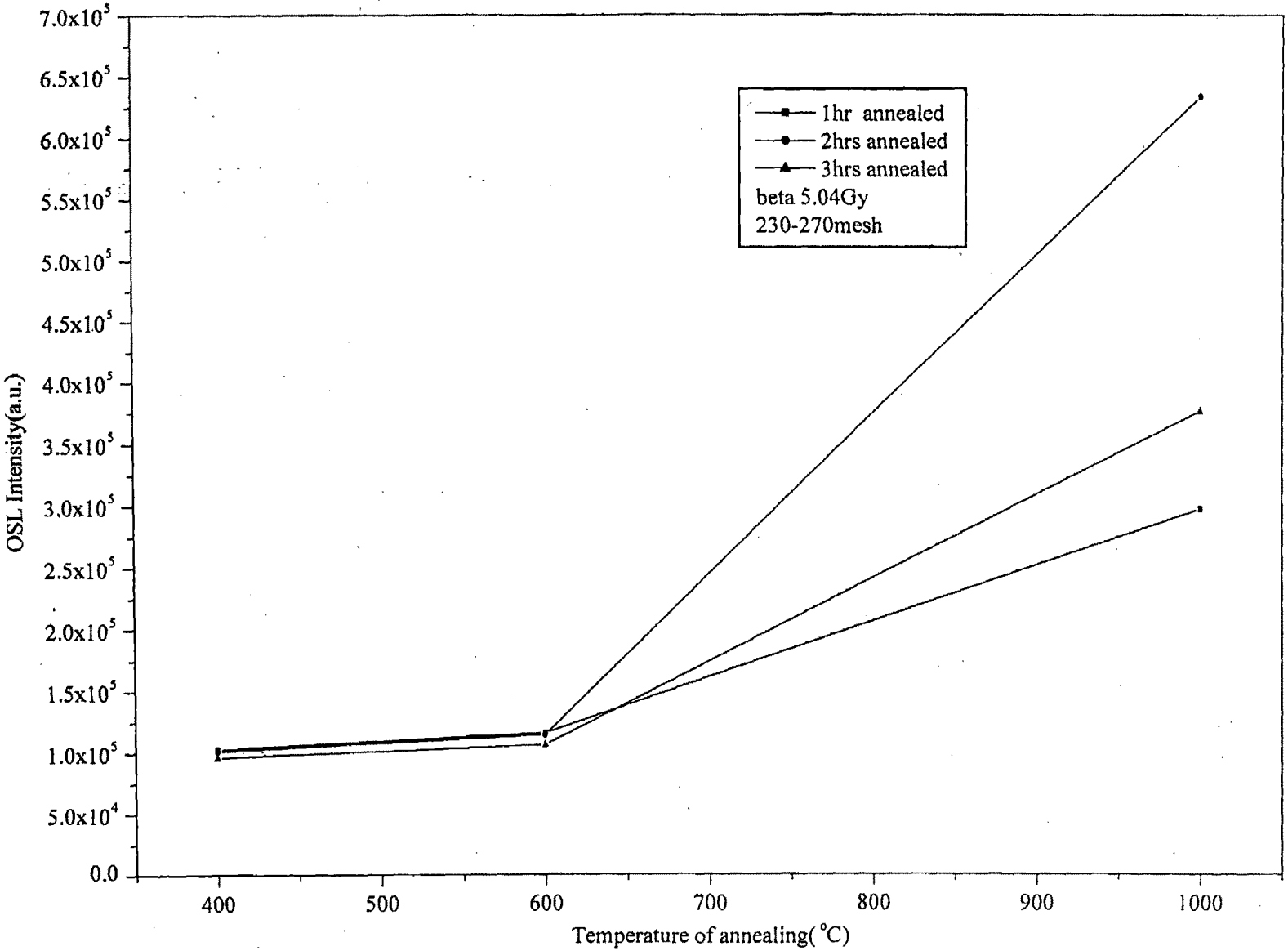
Here, it is found that specimens pre-heat treated at 400°C show decrease in area under OSL curve with rise in beta dose up to the exposure of ~25Gy dose, thereafter it increases on further rise in beta dose. It is important to note that, for the grains annealed at 600°C, such a change takes place for much lower dose (~5Gy). Thereafter, identically the area under OSL curve increases with increase in dose. However, specimens pre-heat treated at 1000°C show such changes with the rise in dose from the lower most doses (**Graph-23**). Identical pattern of observations are found for all durations of pre-heat treatment.

The **Graph-24** to **Graph-27** show data relating to the OSL sensitivity against temperature of annealing for a given constant doses. Here, also the data is compared for different duration for each dose. This study reveals the effect of temperature of annealing for each dose on OSL sensitivity. It is found that OSL intensity was less intense for the specimen pre-heat treated at 400°C and 600°C. Beyond these thermal treatments i.e. at 1000°C there was a sudden rise in OSL intensity. Similar results are observed for higher doses also.

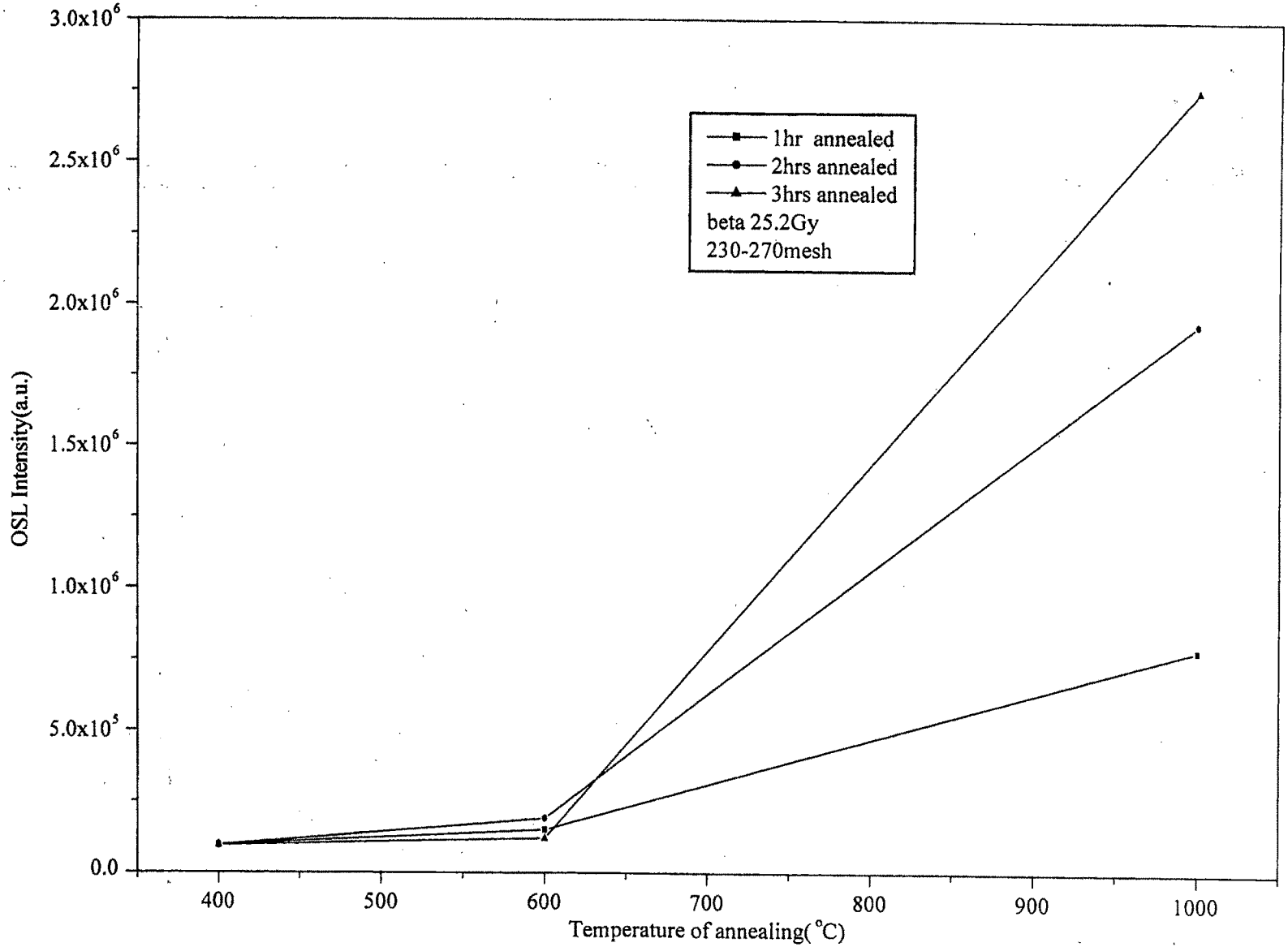
In order to examine, the effect of annealing temperature and duration of pre-heat treatment for a given beta exposure on area under OSL curve each specimen was pre-heat treated at different elevated temperature for different durations. Such specimens were exposed to identical beta dose as seen in **Graph-28** to **Graph-31**. Here it is important to observe that, while raising the temperature of annealing up to 600°C for lower doses <2.52Gy the area under the OSL curve decreases however it is remarkable to note that the area under OSL curve increases for 3hrs duration of pre-heat treatment.



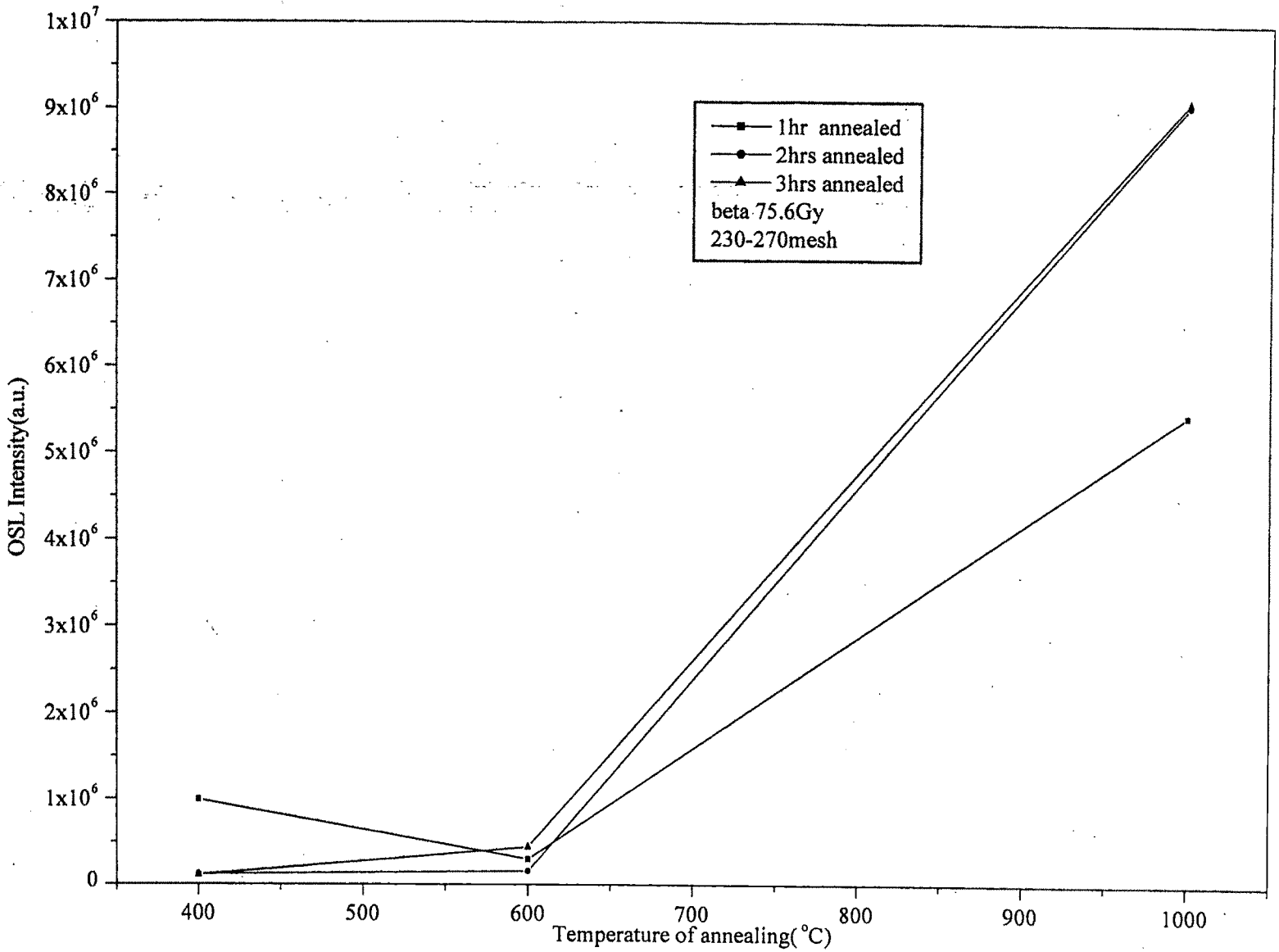
Graph-24 OSL sensitivity measured at room temperature of 2.52Gy beta dose for 230-270mesh at different annealing durations.



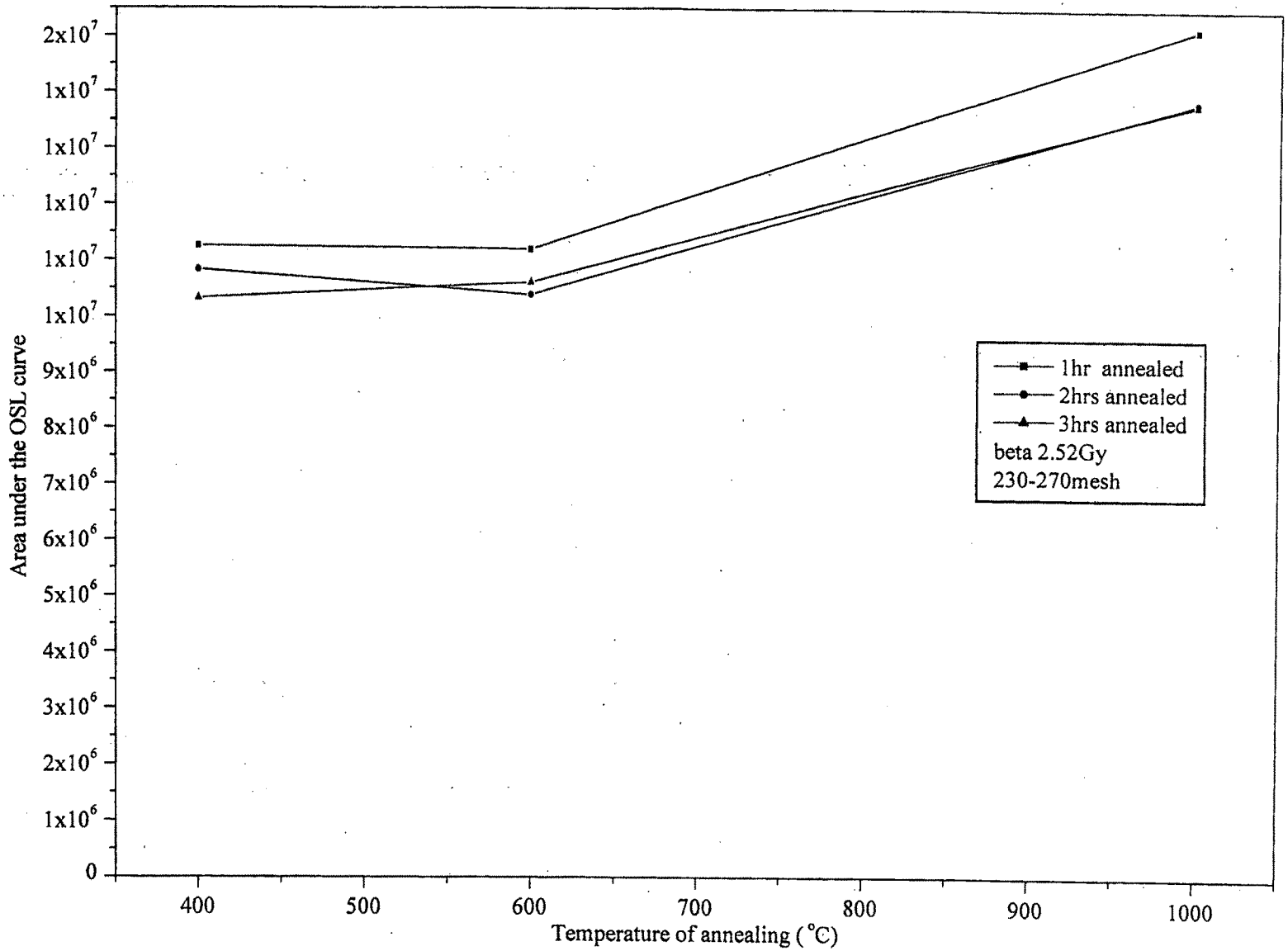
Graph-25 OSL sensitivity measured at room temperature of 5.04Gy beta dose for 230-270mesh at different annealing durations.



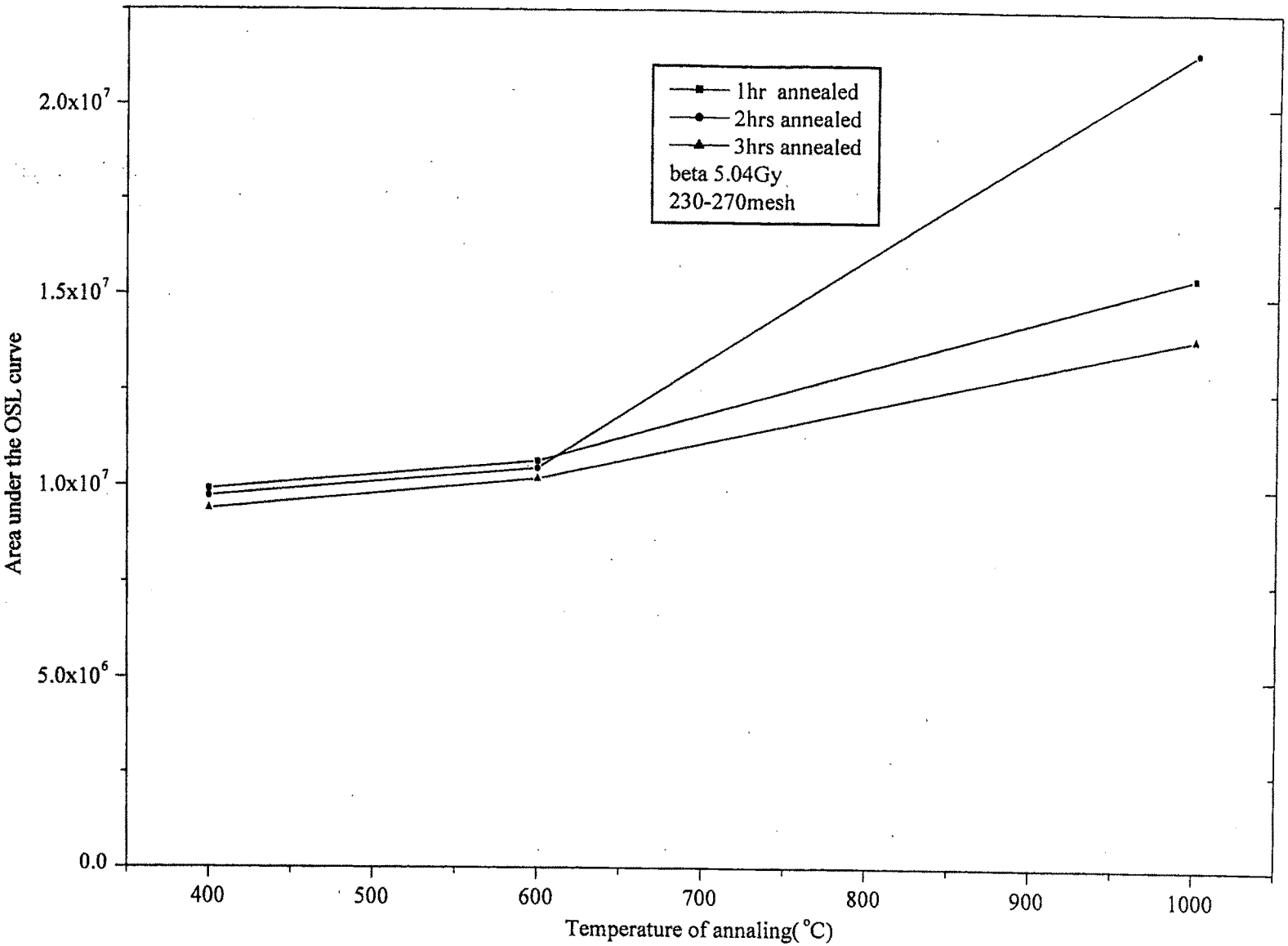
Graph-26 OSL sensitivity measured at room temperature of 25.2Gy beta dose for 230-270mesh at different annealing durations.



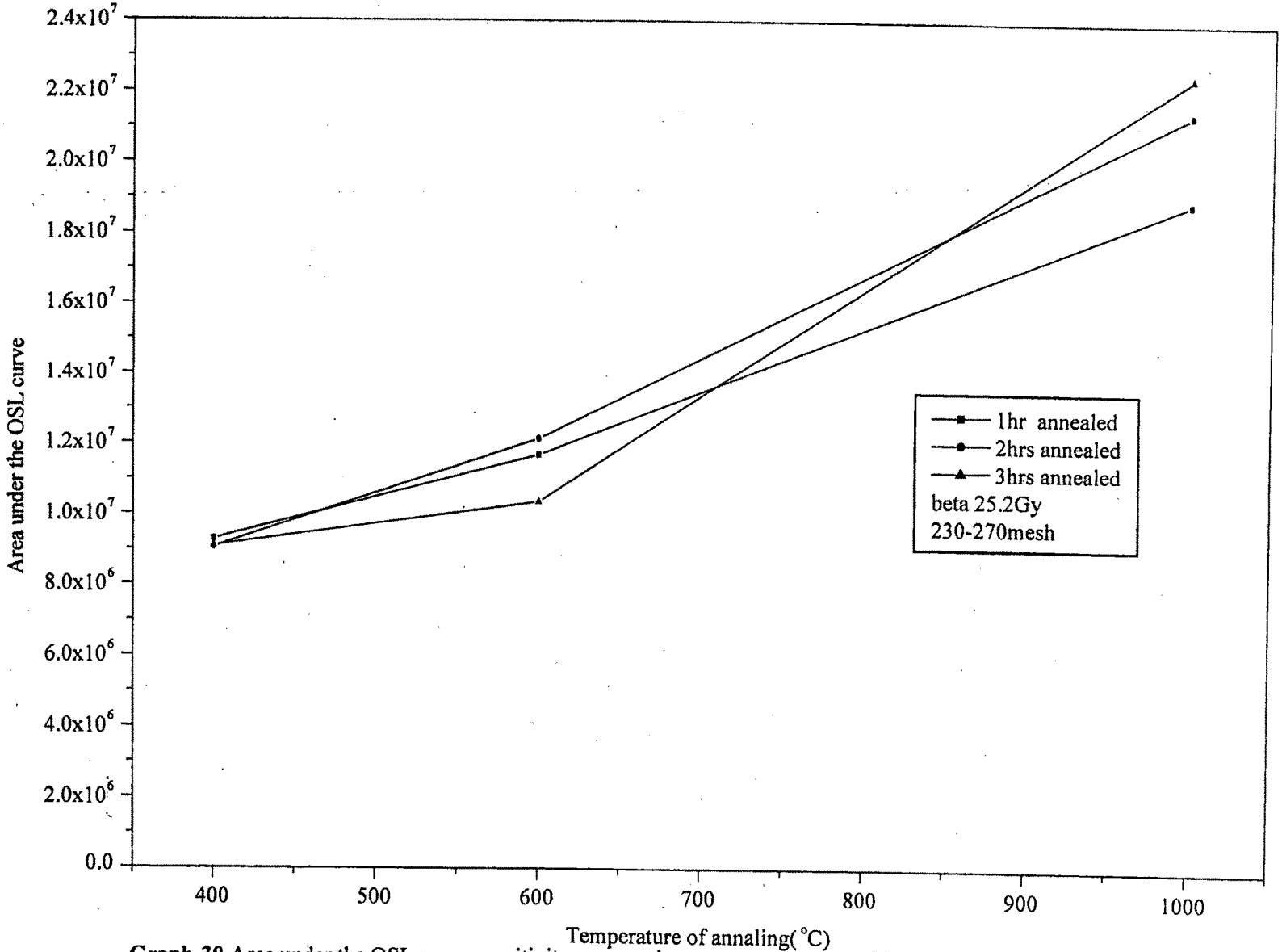
Graph-27 OSL sensitivity measured at room temperature of 75.6Gy beta dose for 230-270mesh at different annealing durations.



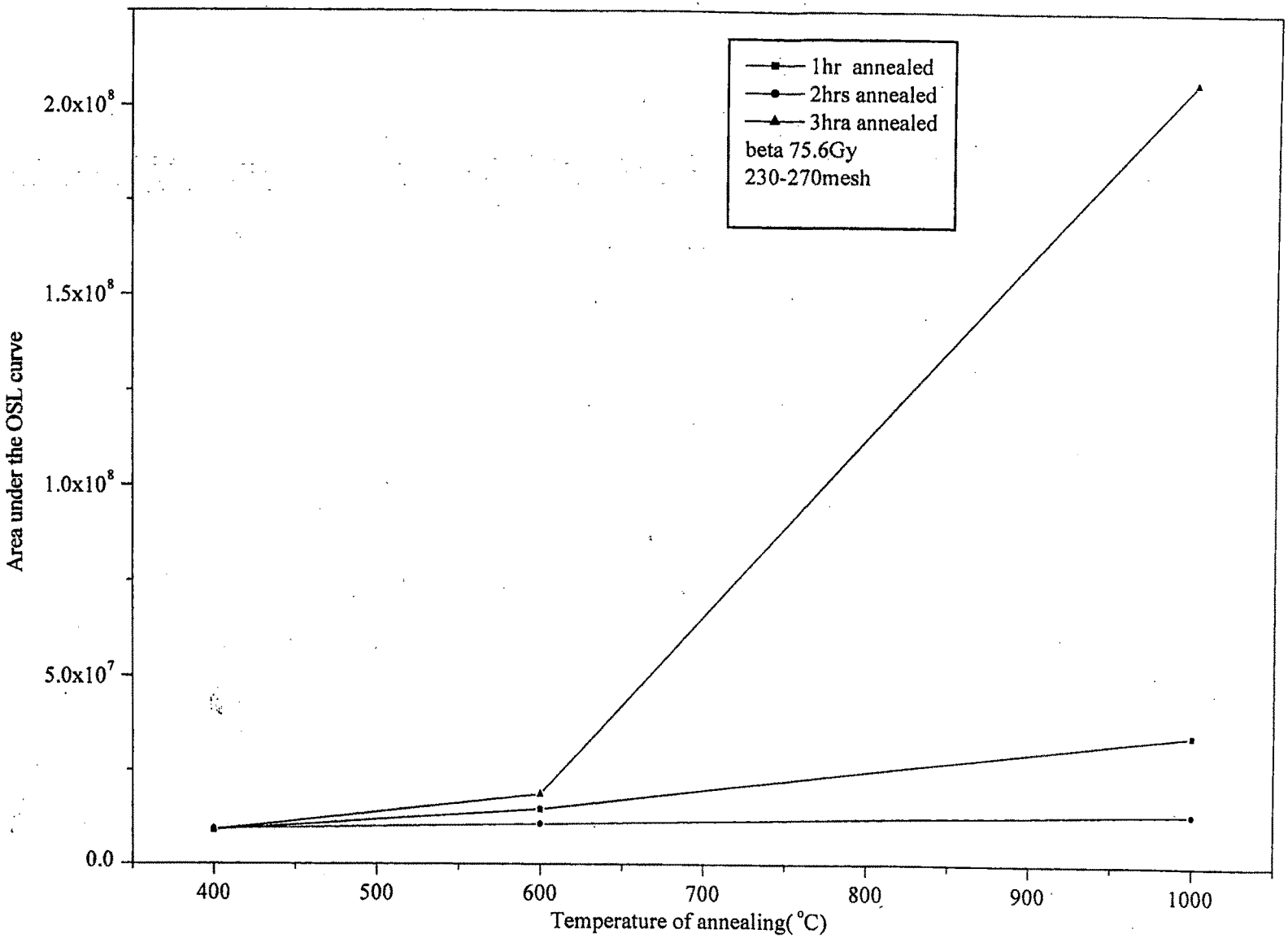
Graph-28 Area under the OSL curve sensitivity measured at room temperature of 2.52Gy beta dose for 230-270mesh at different annealing durations.



Graph-29 Area under the OSL curve sensitivity measured at room temperature of 5.04Gy beta dose for 230-270mesh at different annealing durations.



Graph-30 Area under the OSL curve sensitivity measured at room temperature of 25.2Gy beta dose for 230-270mesh at different annealing durations.

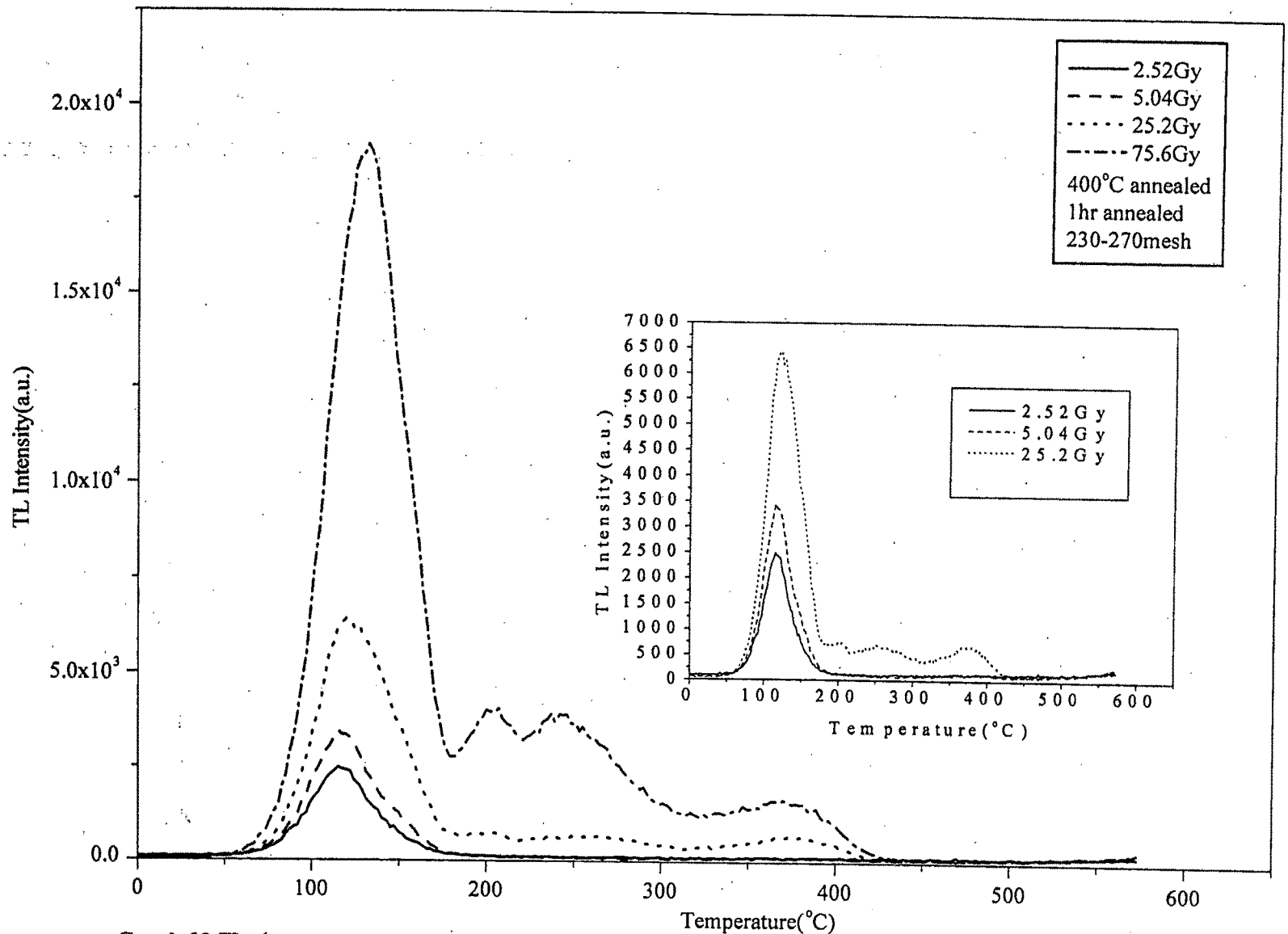


Graph-31 Area under the OSL curve sensitivity measured at room temperature of 75.6Gy beta dose for 230-270mesh at different annealing durations.

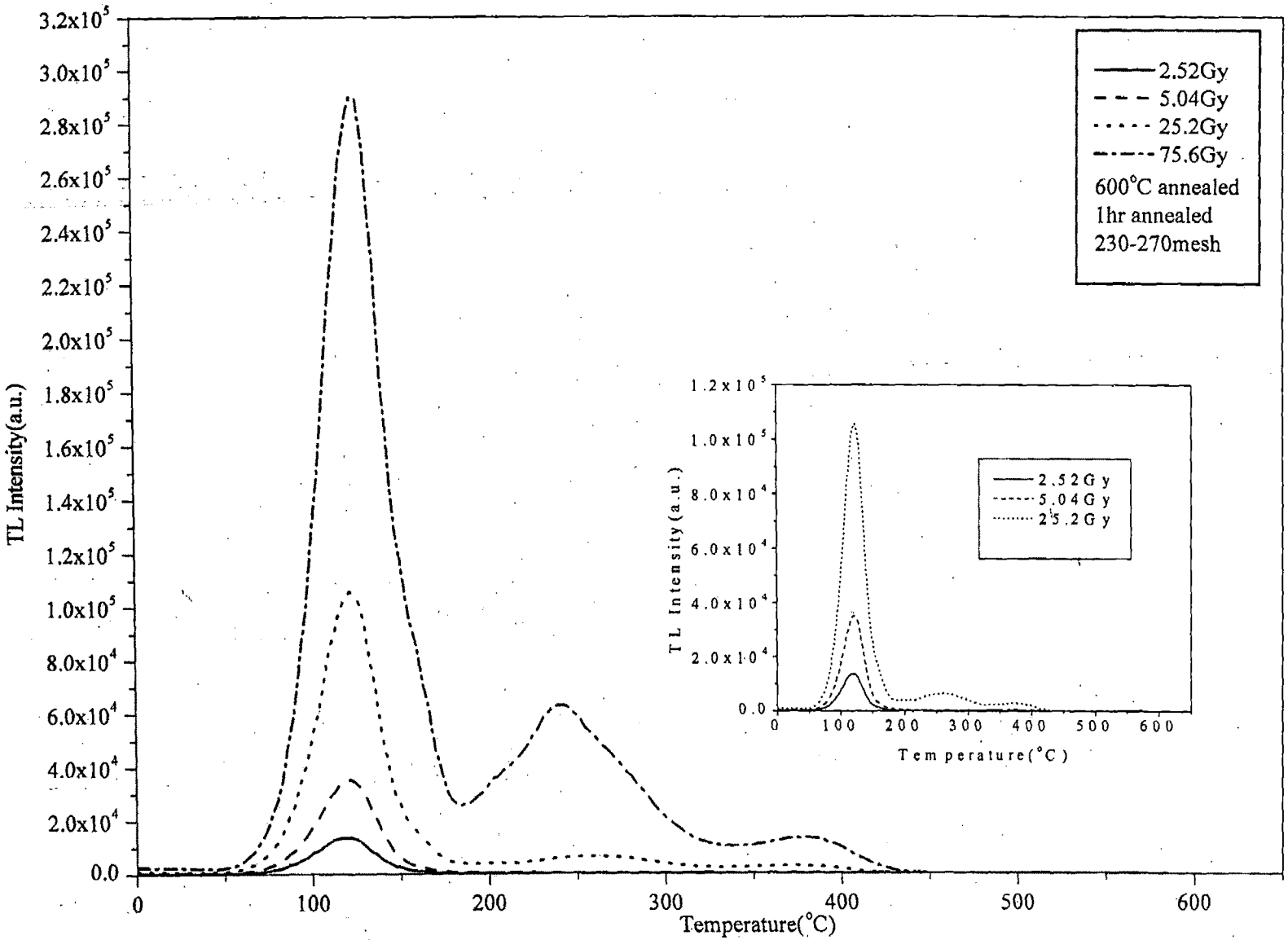
Another interesting observation is that the area under OSL curve increases with the rise in annealing temperature beyond the 2.52Gy beta exposed specimens.

It is already mentioned, earlier that the concept of phototransferred thermoluminescence (PTTL), invokes transfer of trapped carriers from deep traps to shallower traps due to optical stimulation. Hence, TL is recorded after the OSL to know the amount of phototransfer. This will reveal the pre-dose effect, which is significant in dating and dosimetry. This study is also used to understand the role of low temperature TL peak and its thermal stability. Therefore, the TL characteristics after OSL decay for 100sec at room temperature for different protocols were also recorded with a view to understand phototransfer process and thermal stability as well as growth of responsible color centers for such a protocol in quartz which is correlated with low temperature TL peak.

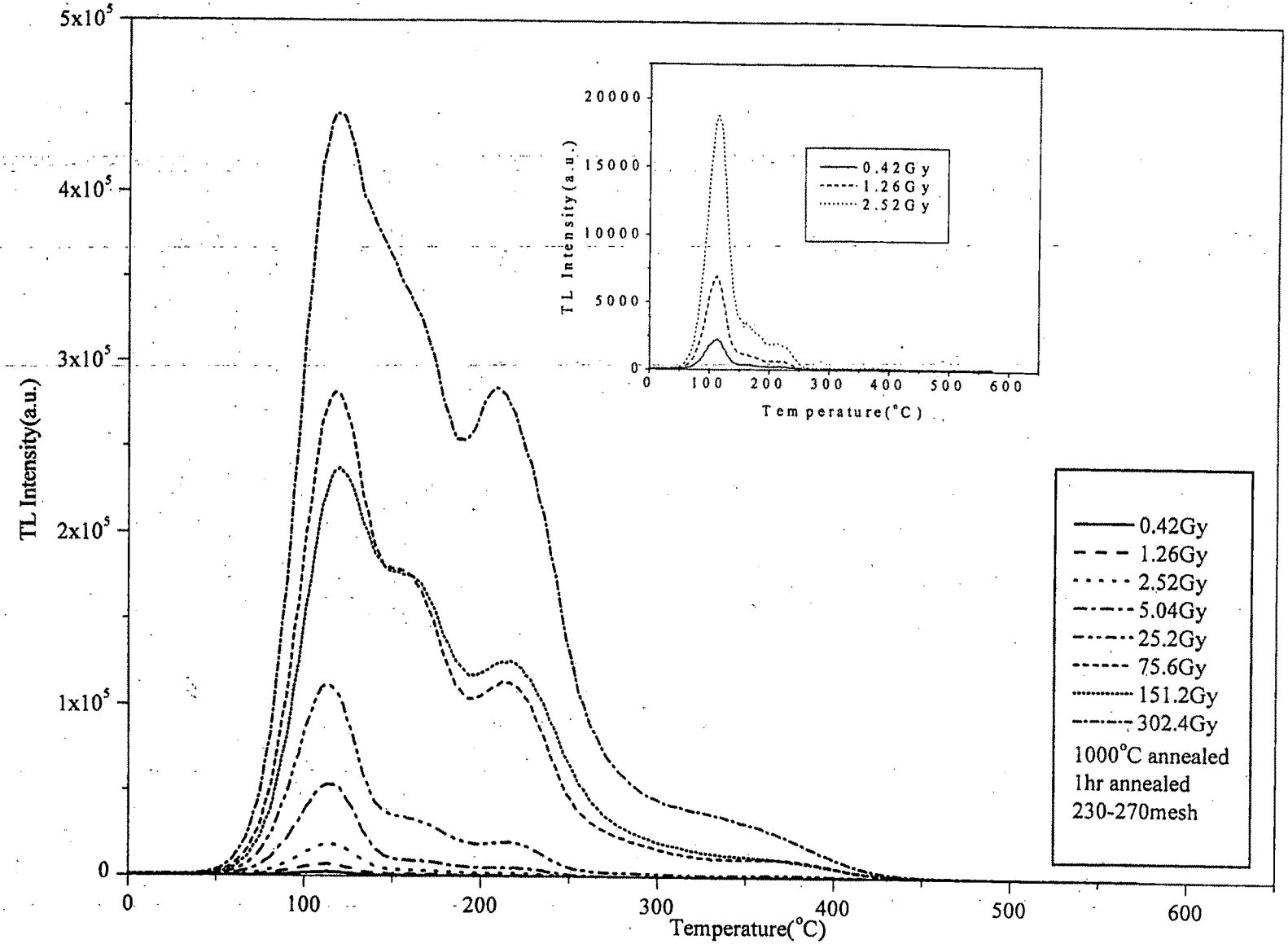
Graph-32 to **Graph-35** are recorded TL readouts after the OSL decay of quartz specimens, which were pre-heat treated at 400°C, 600°C and 1000°C for 1hr duration. Here, it is observed that, a well defined TL glow peak is exhibited at around ~ 120°C for lower doses as much as 2.52Gy for any temperature of pre-heat treatment as well as any duration of pre-heat treatment. However, it is noticed clearly that, the specimens when pre-heat treated at 600°C shows glow peak at around 220°C for higher doses as much as 25.2Gy dose. However, specimens pre-heat treated at 1000°C for 1hr duration exhibits same glow peak even at lower dose (2.52Gy), its intensity changes with the either the rise in beta dose or rise in duration of pre-heat treatment. Further, it is observed that the specimens exposed to ≥ 75.6Gy induce an additional TL glow peak around ~ 370°C. The changes in any glow peak positions are also noticeable for different implemented protocols.



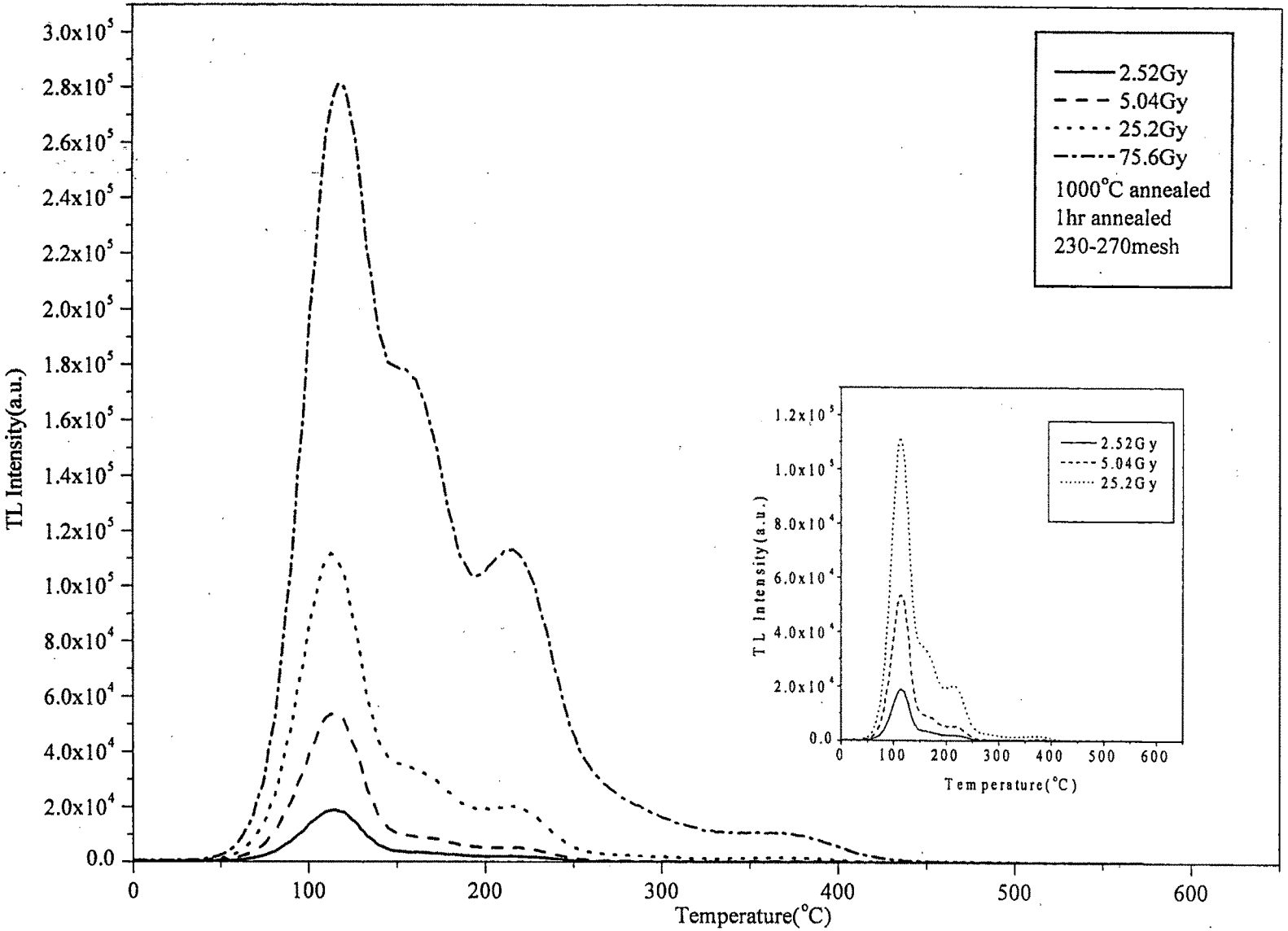
Graph-32 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 400 °C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-33 TL glow curve measured at room temperature (after the OSL measure at room temperature) of 600 °C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-34 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1000 °C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-35 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1000 °C annealed sample for 1hr duration at different doses for 230-270mesh.

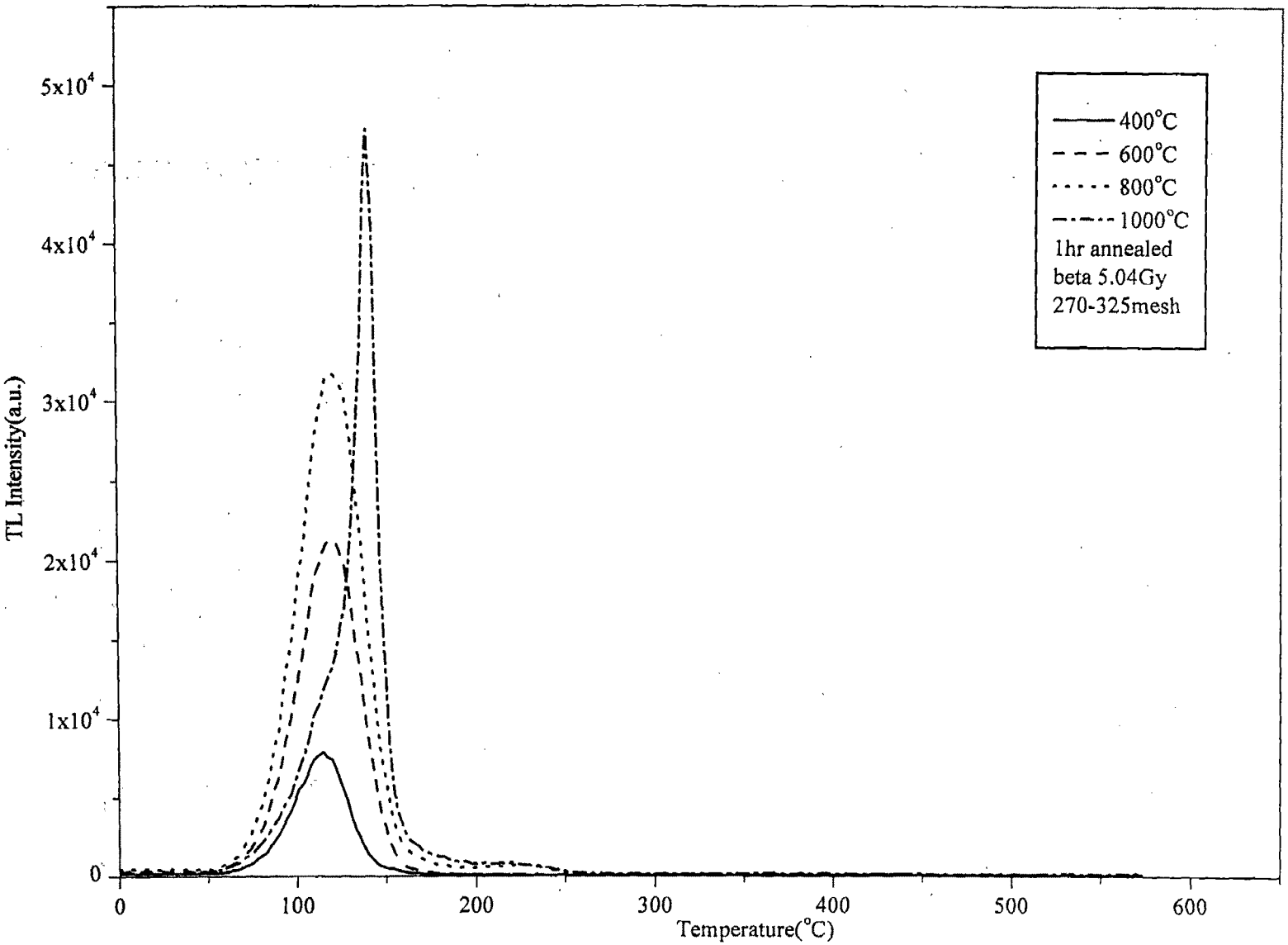
In another noteworthy observation in relation to the different grain sizes for similar protocol, it is clear that the finest grains (270-325Mesh) shows the highest TL sensitivity to the test dose of 5.04Gy (**Graph-36**).

As the grains becomes coarse (120-140mesh) the TL intensity decreases drastically, however TL glow pattern remains identical for each grains sizes (**Graph-37** and **Graph-38**). From the graphs it is also clear that as the temperature of annealing increase while keeping other physical conditions same, TL intensity grows for a grain size as well as for different grain sizes.

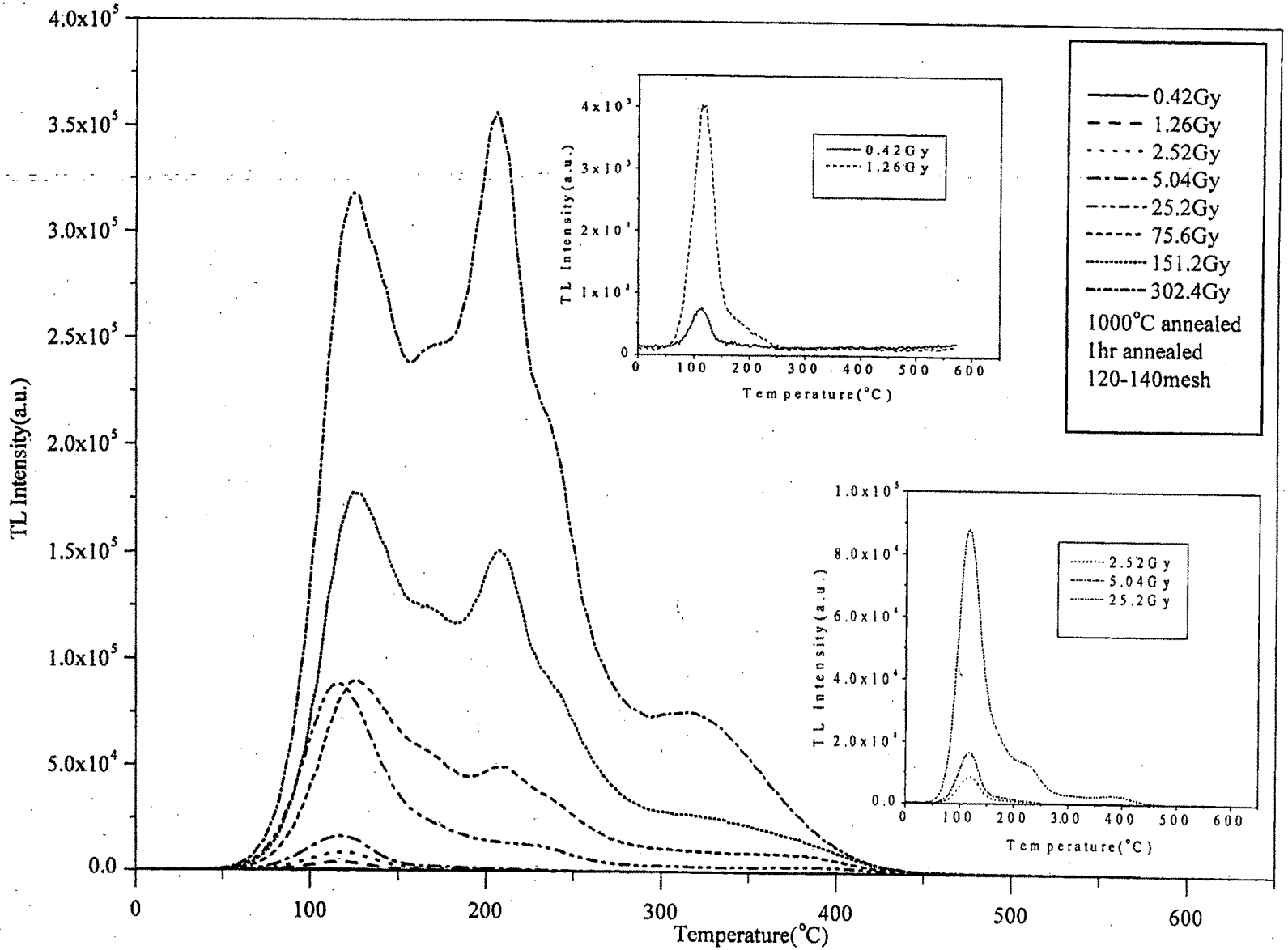
In order to know the effect of increasing the duration of pre-thermal treatment on the moderate grain size i.e. 230-270mesh was chosen due to sufficient TL sensitivity for all the temperature of annealing. From **Graph-39** to **Graph-41** as well as **Graph-42** to **Graph-44** it is clear that TL glow pattern remains unaltered even with the rise in duration of pre-heat treatment namely, 2hrs and 3hrs respectively, but the TL intensity more or less increases.

Thermoluminescence intensity after OSL against different temperature of annealing for a given dose is plotted as **Graph-45** to **Graph-48** and same graphs, the comparison is also made for different durations of pre-heat treatment with a view to establish redistribution of charges in corresponding trap for higher doses too.

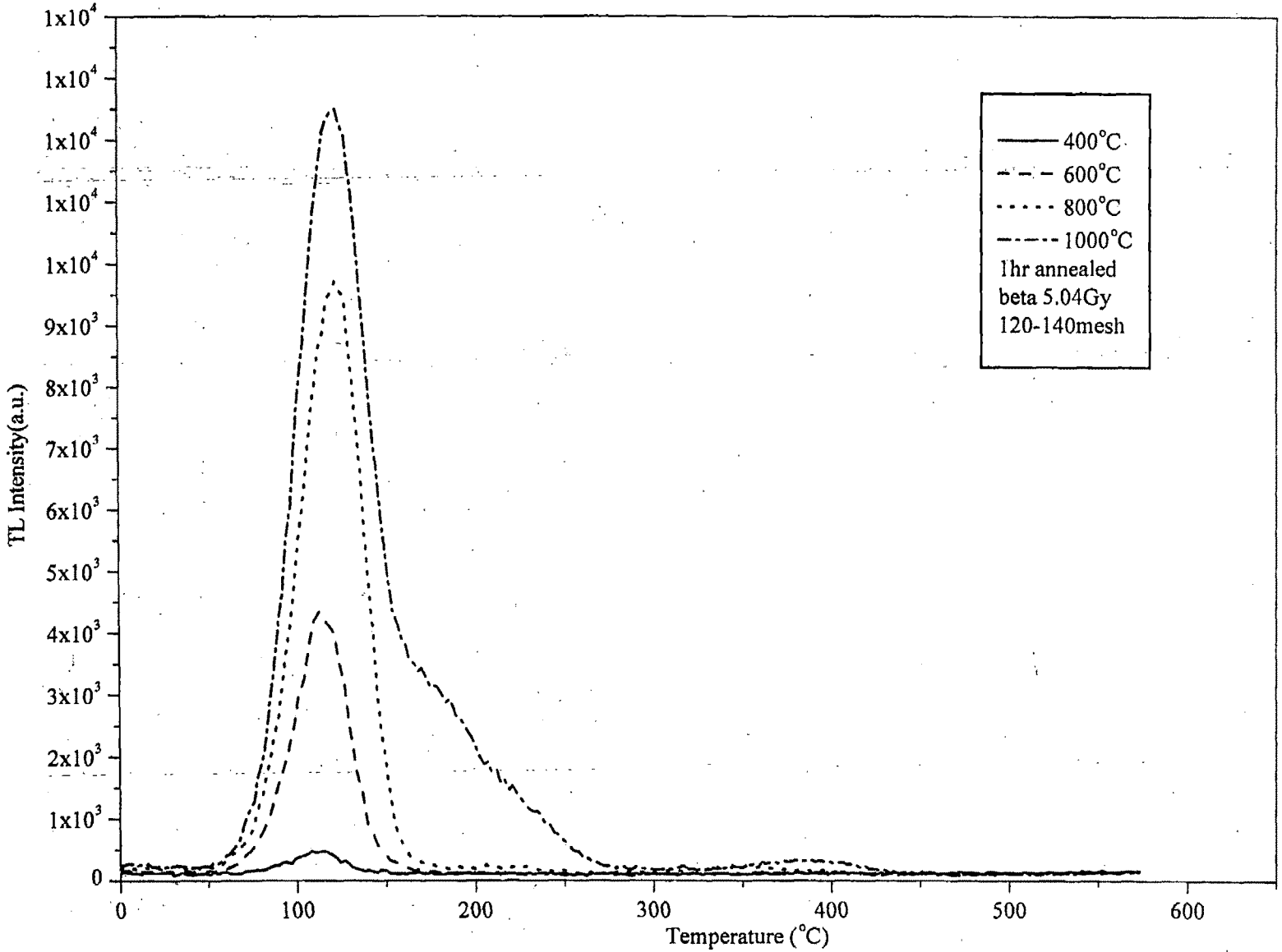
Here, it is noteworthy that the TL intensity grows with the temperature of annealing with a distinct that the specimens pre-heat treated beyond 600°C shows abrupt enhancement in TL intensity. Not only that, on careful investigations it is pointed out that for such a protocol the additional high temperature peaks are induced, which seem to prompt this TL peak to shift toward lower temperature side. Moreover, it should be noted that the peak remains stable for the specimens pre-heat treated up to 600°C even at higher



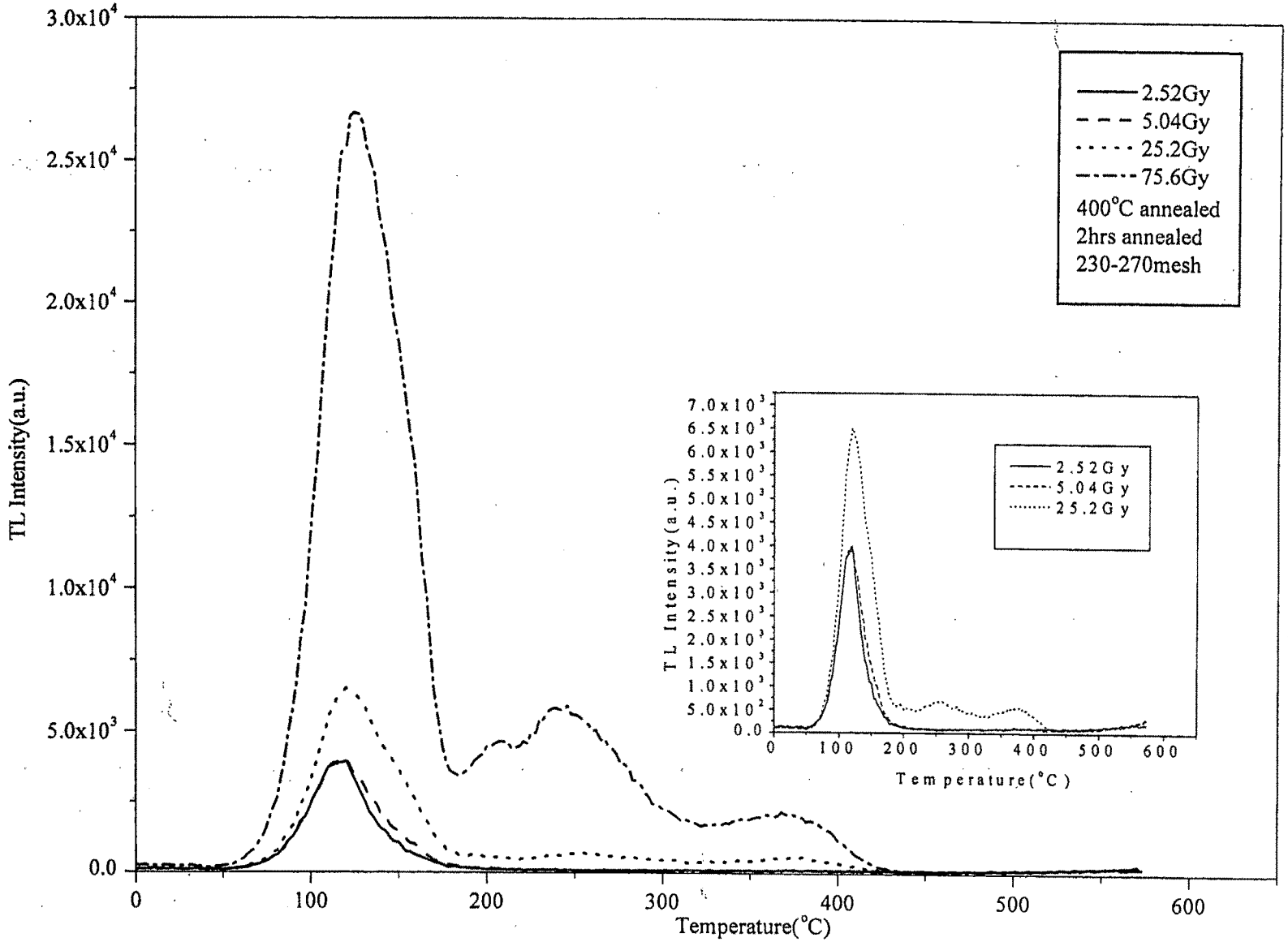
Graph-36 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1hr annealed sample for a given 5.04Gy beta dose at different annealing temperatures for 270-325mesh.



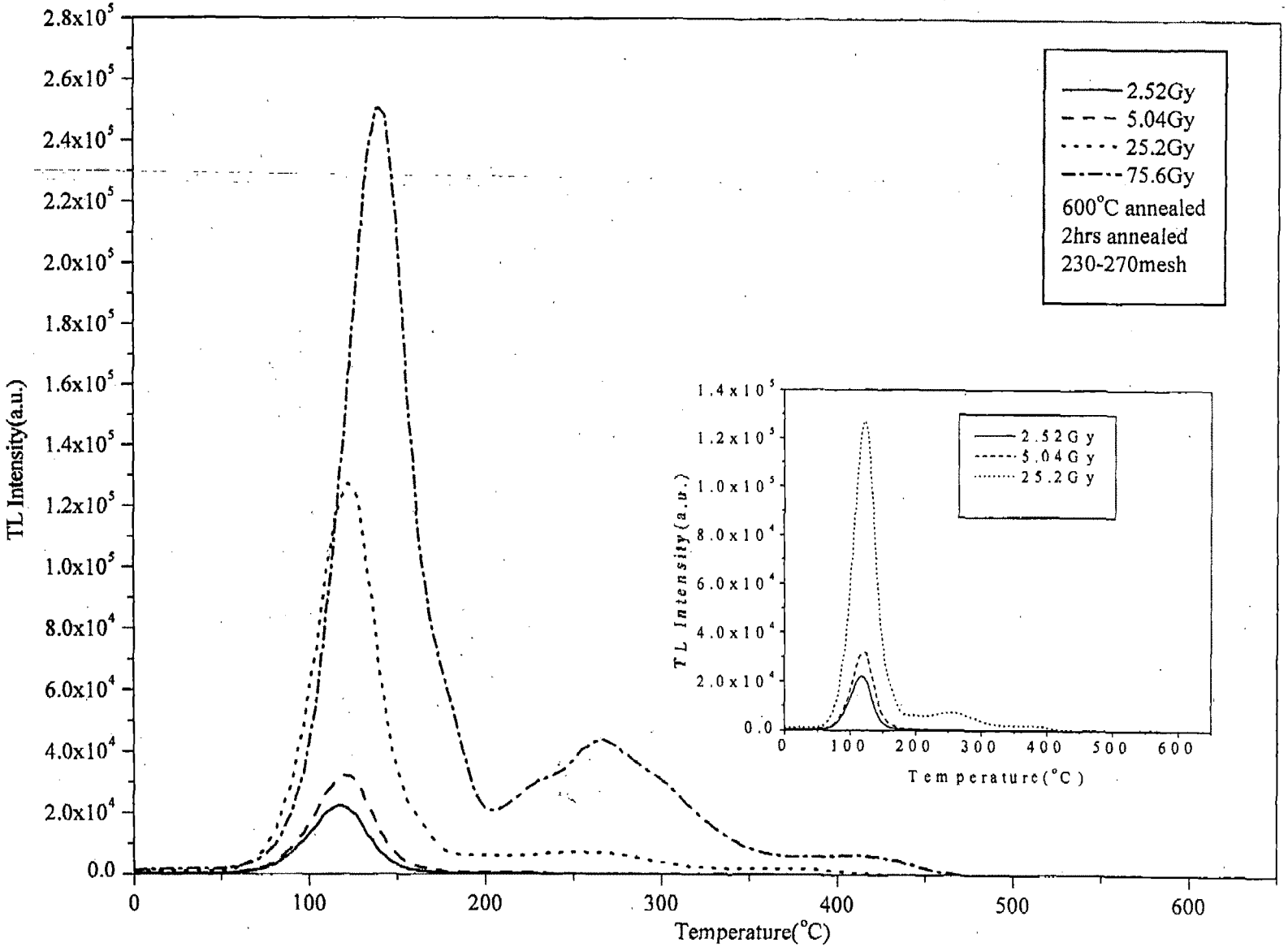
Graph-37 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1000 °C annealed sample for 1hr duration at different doses for 120-140 mesh.



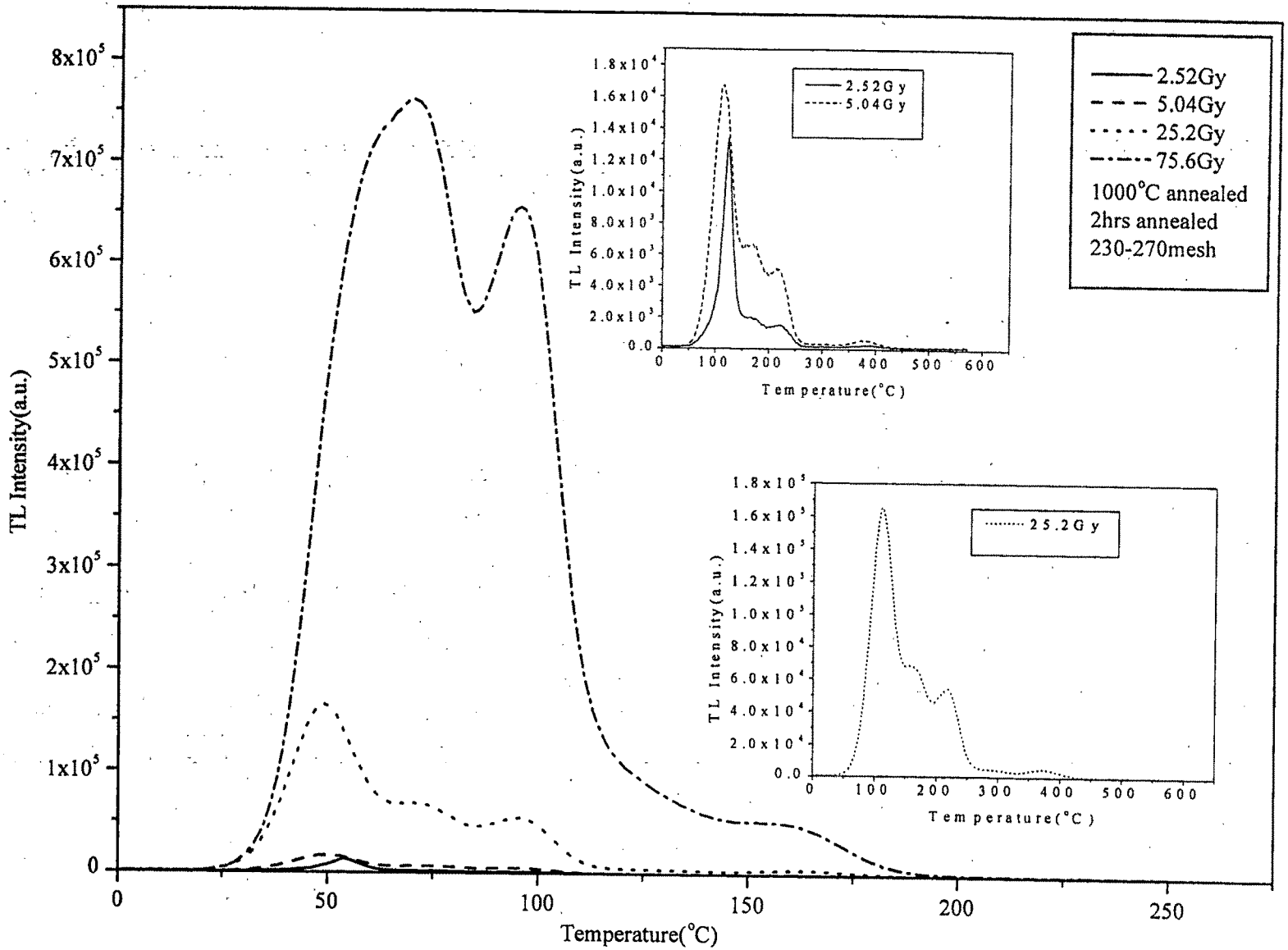
Graph-38 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1hr annealed sample for a given beta dose of 5.04Gy at different annealing temperatures for 120-140mesh.



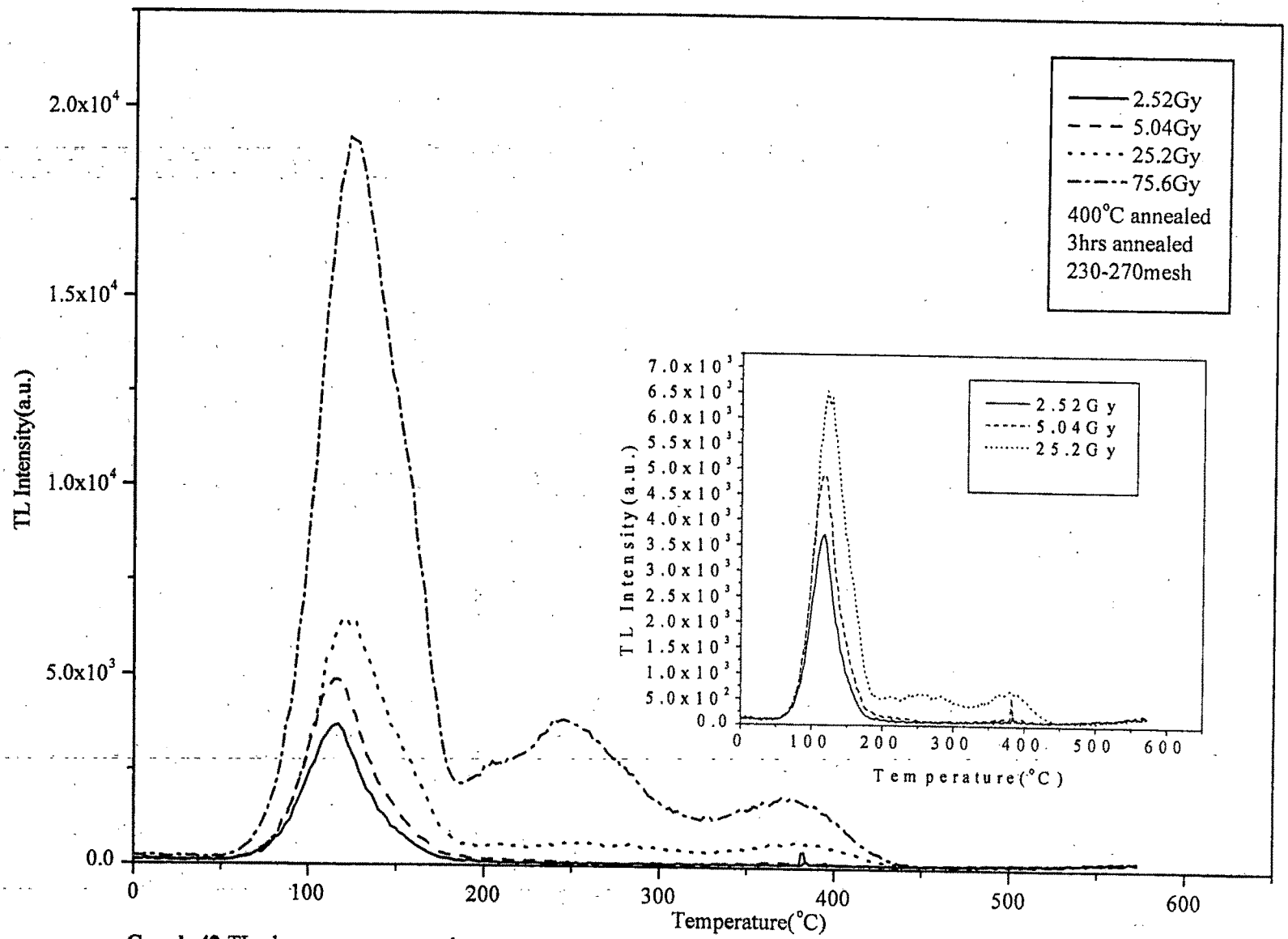
Graph-39 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 400 °C annealed sample for 2hrs duration at different doses for 230-270mesh.



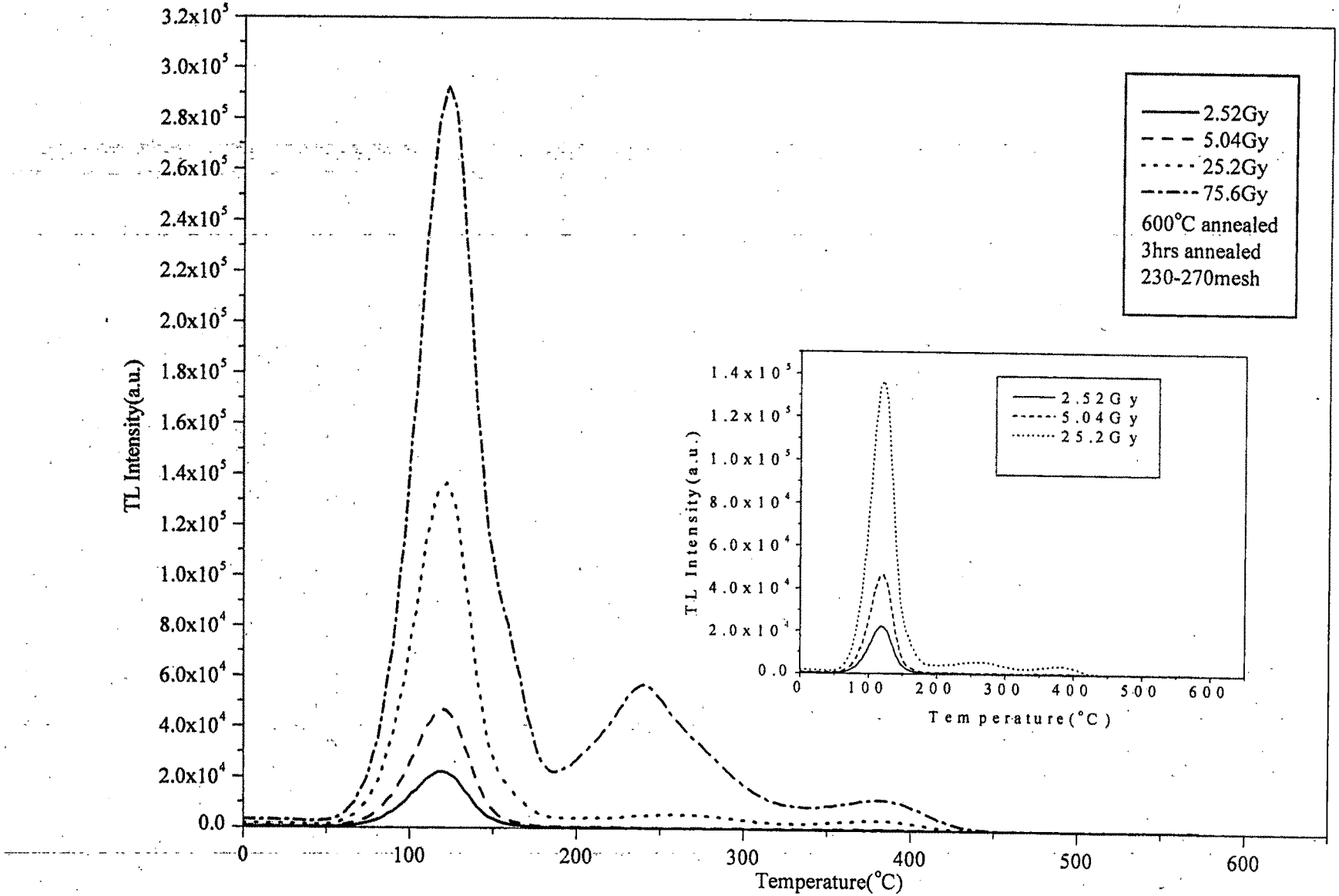
Graph-40 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 600 °C annealed sample for 2hrs duration at different doses for 230-270mesh.



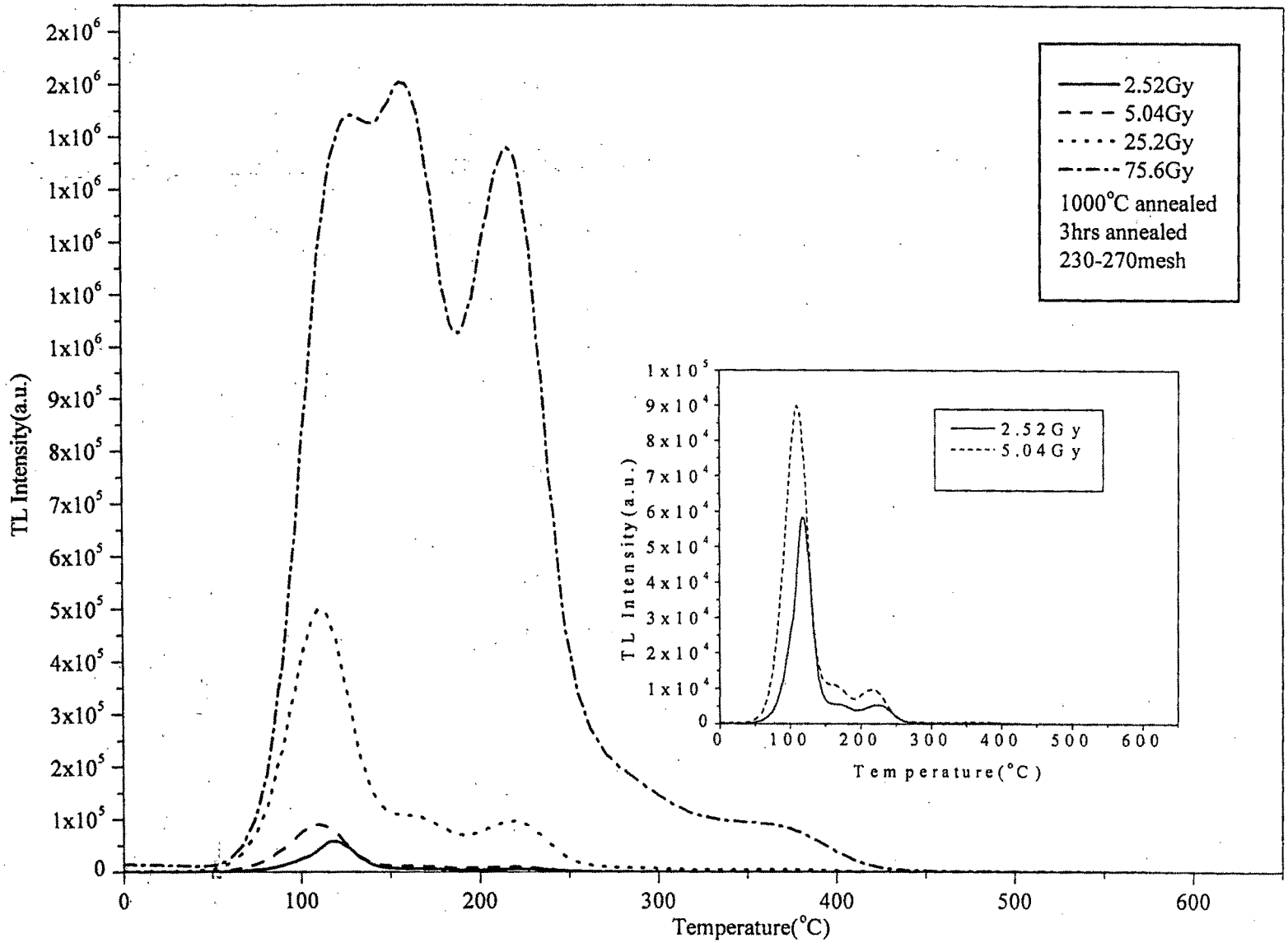
Graph-41 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1000 °C annealed sample for 2hrs duration at different doses for 230-270 mesh.



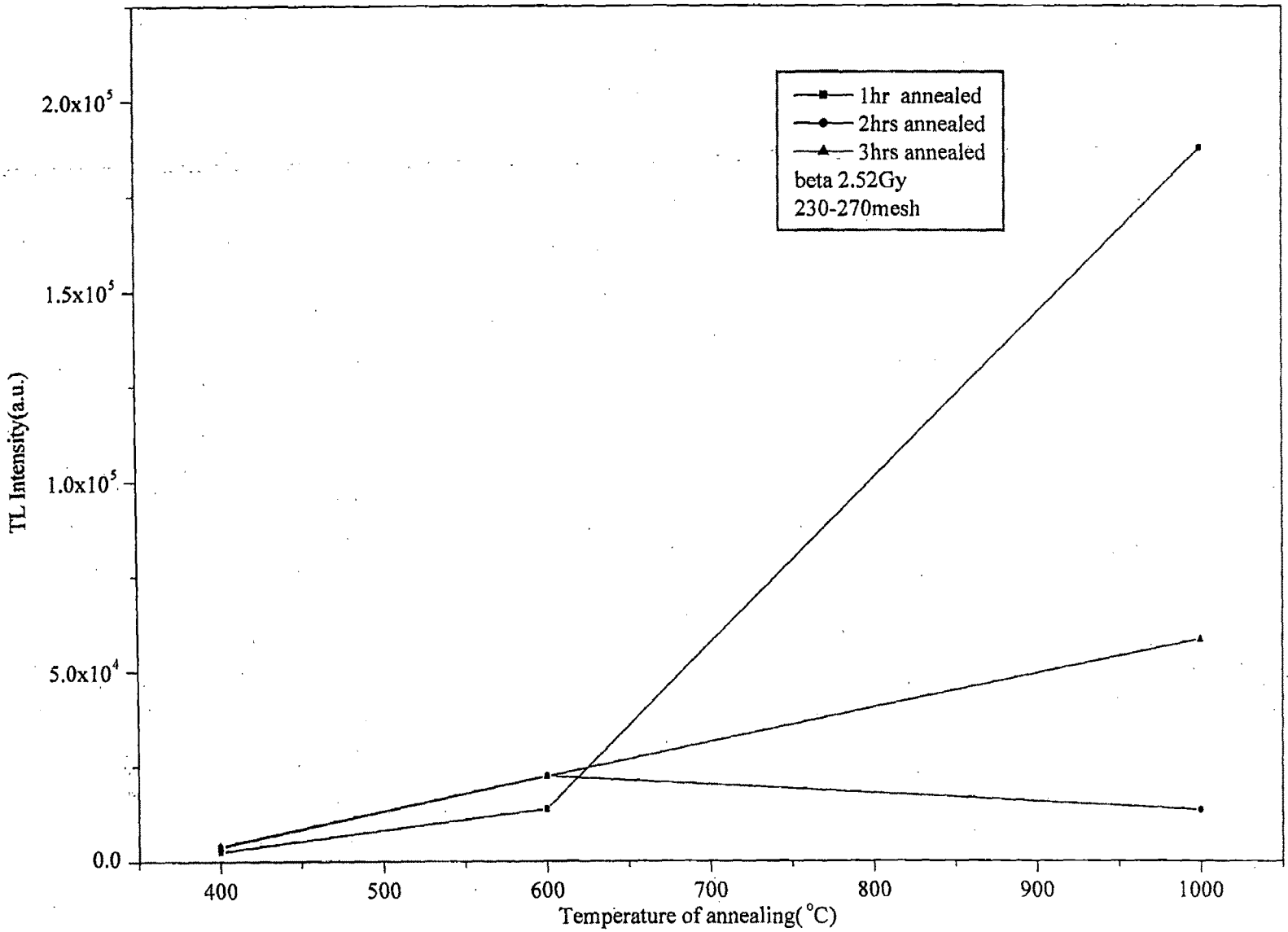
Graph-42 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 400 °C annealed sample for 3hrs duration at different doses for 230-270mesh.



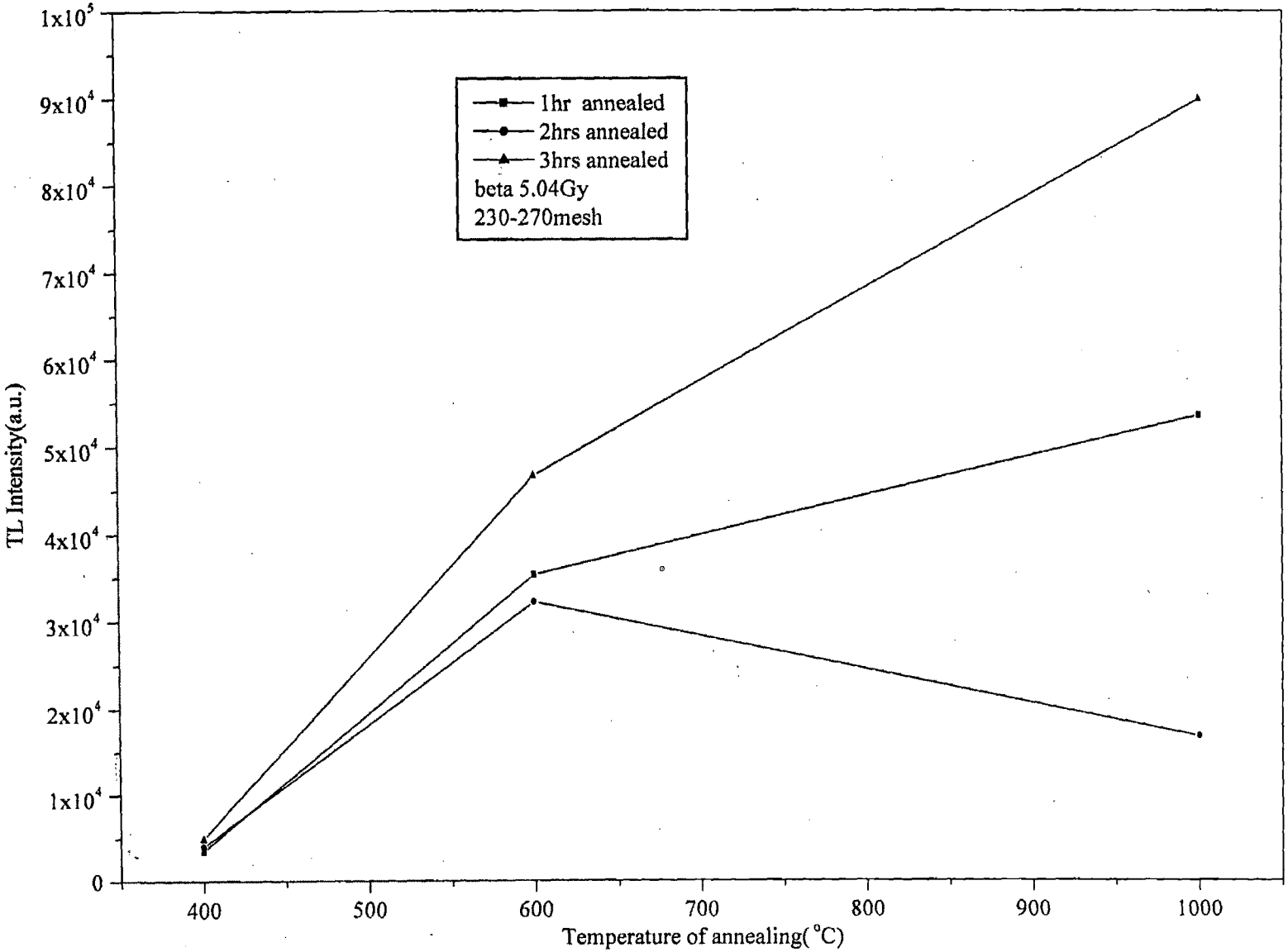
Graph-43 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 600 °C annealed sample for 3hrs duration at different doses for 230-270 mesh.



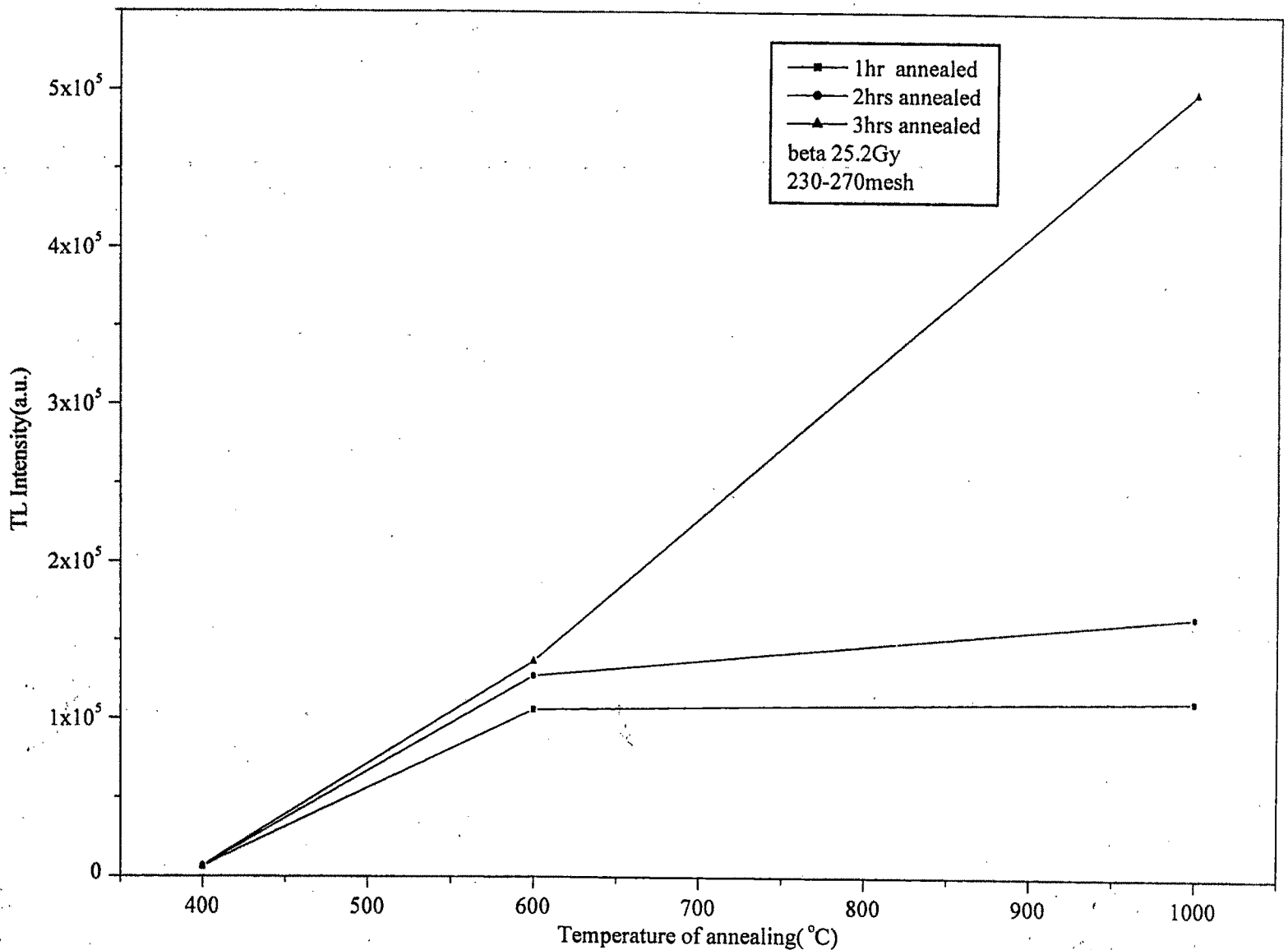
Graph-44 TL glow curve measured at room temperature (after the OSL measured at room temperature) of 1000 °C annealed sample for 3hrs duration at different doses for 230-270 mesh.



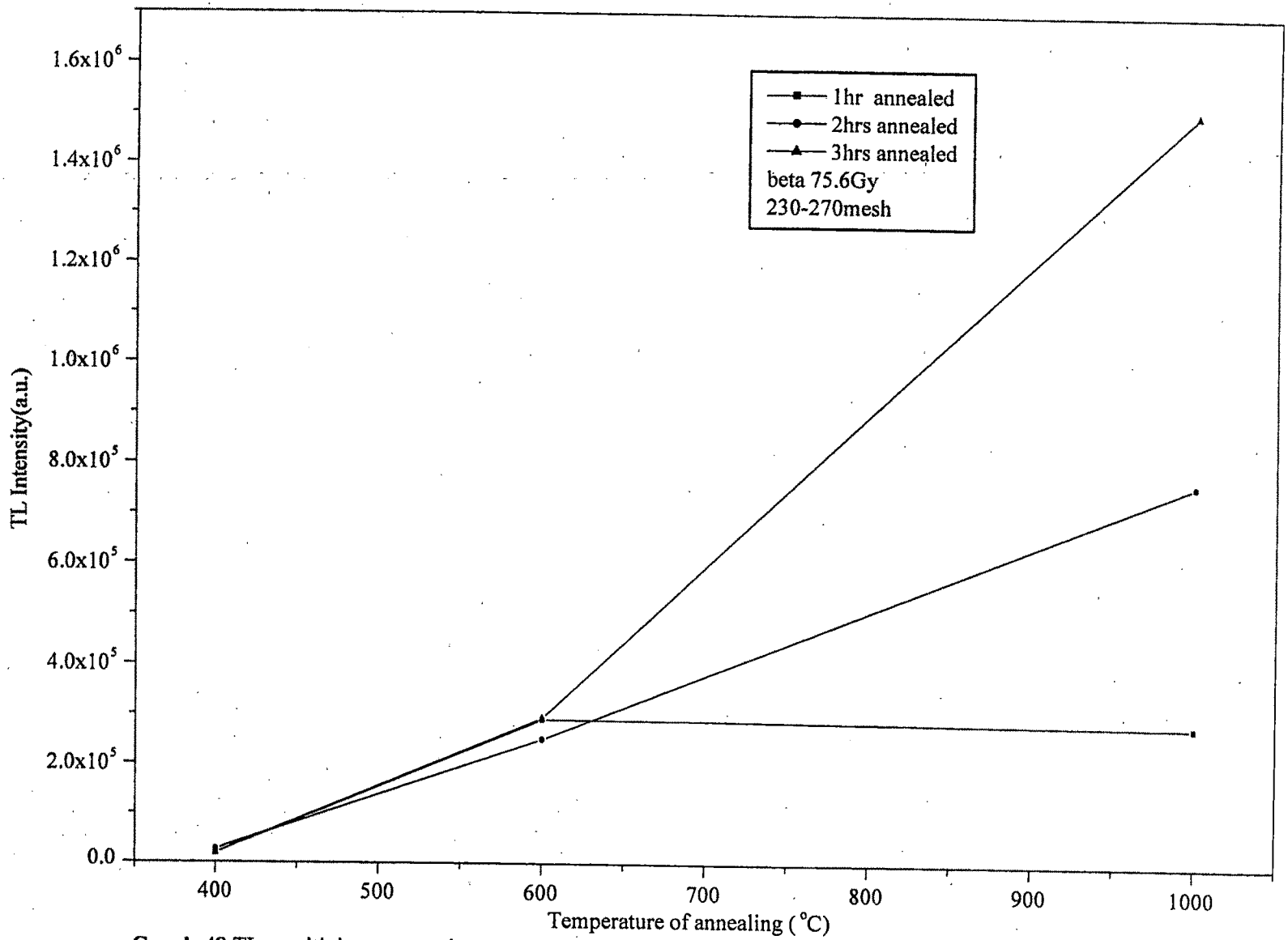
Graph-45 TL sensitivity measured at room temperature (after the OSL measured at room temperature) of 2.52Gy beta dose for 230-270mesh at different annealing durations.



Graph-46 TL sensitivity measured at room temperature (after the OSL measured at room temperature) of 5.04Gy beta dose for 230-270mesh at different annealing durations.



Graph-47 TL sensitivity measured at room temperature (after the OSL measured at room temperature) of 25.2Gy beta dose for 230-270mesh at different annealing durations.



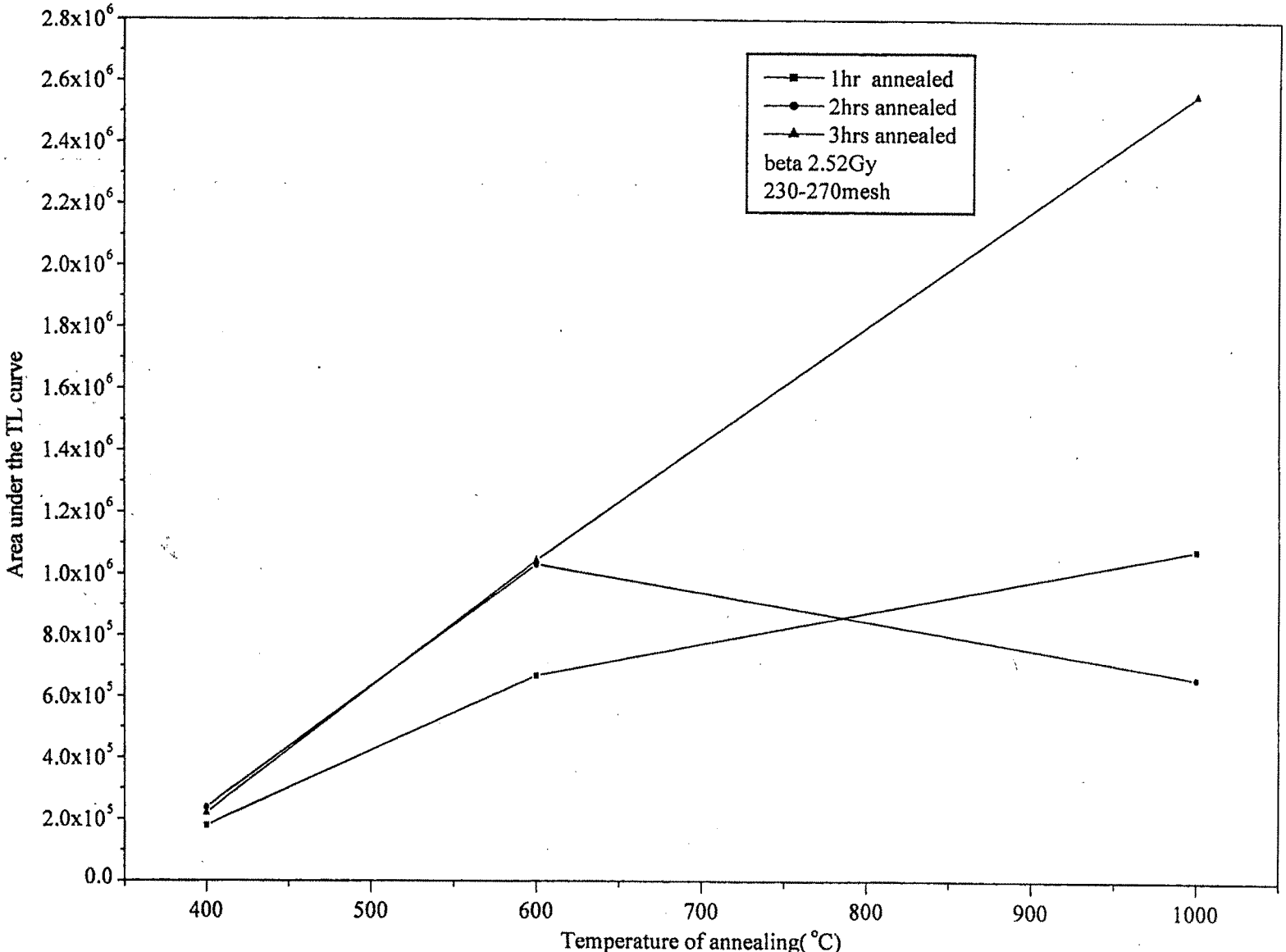
Graph-48 TL sensitivity measured at room temperature (after the OSL measured at room temperature) of 75.6Gy beta dose for 230-270mesh at different annealing durations.

doses. However, the peak shifts towards lower temperature side, when specimens were pre-heat treated beyond 600°C. It should also be noted that, the specimens may even show high temperature TL peaks when specimens were given beta dose ≥25Gy.

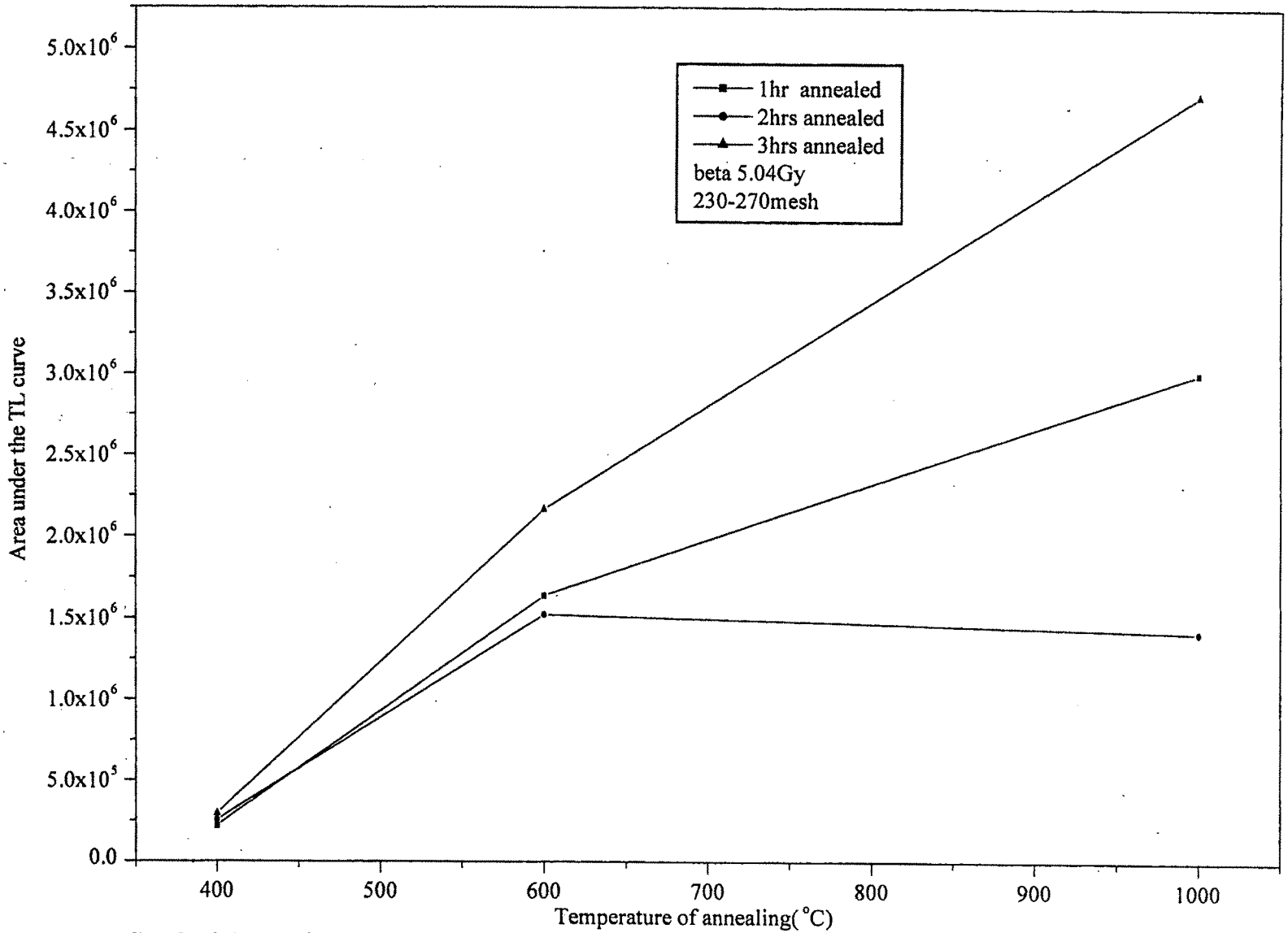
In another part of experiments area under the TL glow curve are plotted at identical beta dose for different annealing temperature 400°C to 1000°C with respect to 1hr, 2hrs and 3hrs pre-heat duration. From **Graph-49** to **Graph-52,** it is observed that at identical beta dose in the range between 2.52Gy to 75.6Gy, for material annealed at temperature between 400°C to 1000°C the area under TL glow curves systematically increases for all pre-heat duration. Also it is found that for beta dose ≥ 5.04Gy and thermal pre-treatments in the range 600°C to 1000°C there is sudden enhancement in the area under the observed TL glow curve.

In general, the shape of the OSL decay curve doesn't lend itself to analysis using simple descriptions. The decay is usually non-exponential, typically exhibiting a long 'tail" to the decay at long illumination times. In some circumstances, and for some samples, the OSL may even display an initial slow increase after the illumination is applied, followed by the more usual decrease at longer times. Overall, the decay shape is dependent upon the sample conditions such as the absorbed dose, the illumination intensity and temperature of pre-heat treatment and temperature of sample etc.[22 & 42]

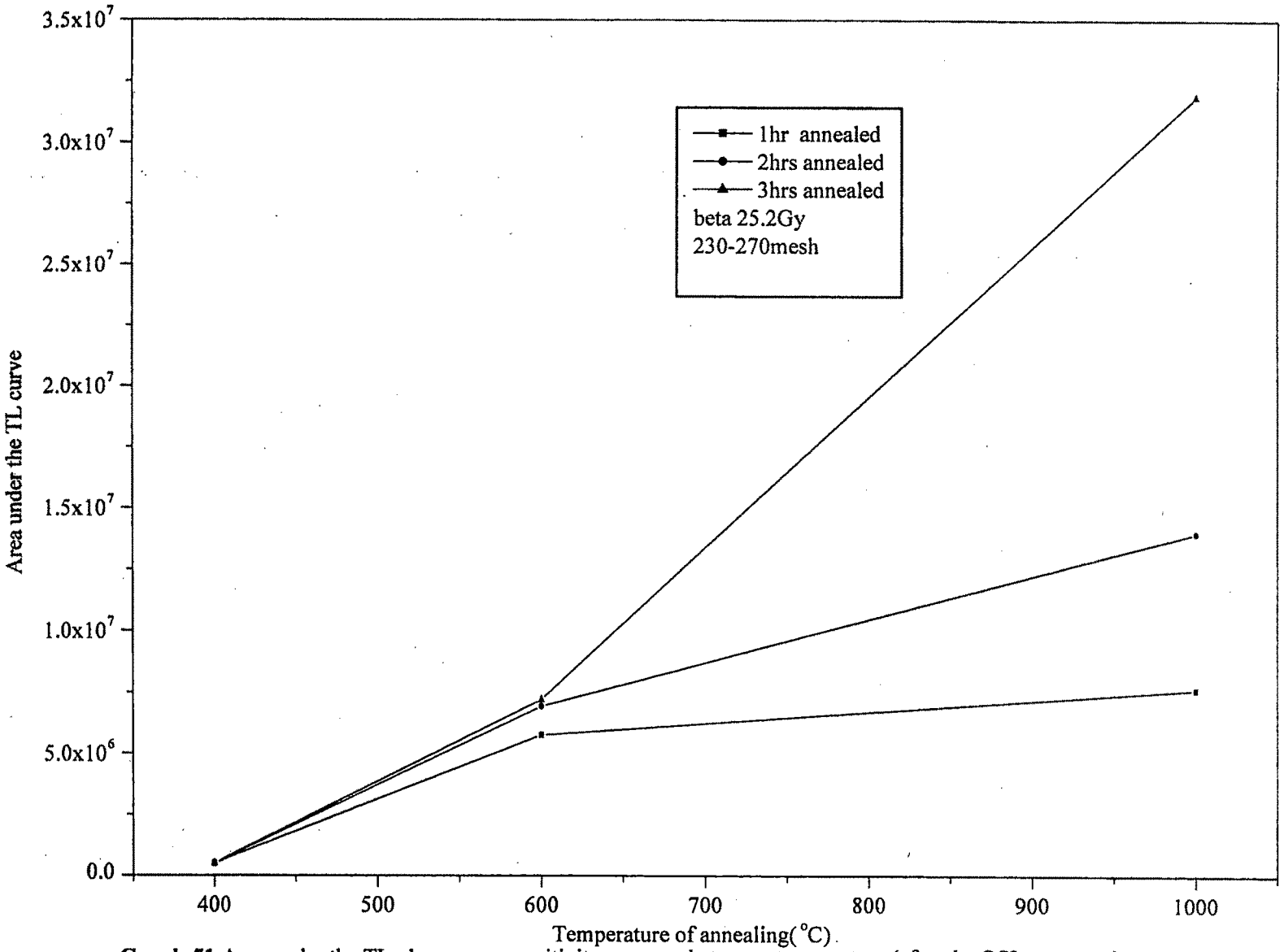
Although, one might expect a multi-component OSL decay curve if more than one trap is being emptied at the illumination wavelength being used, all available data in the present work indicate that at 470nm (the stimulation wavelength) only one trap is being emptied. Through, the causes of these protocol-induced changes in shape and sensitivity of the decay curves are not well understood.[23]



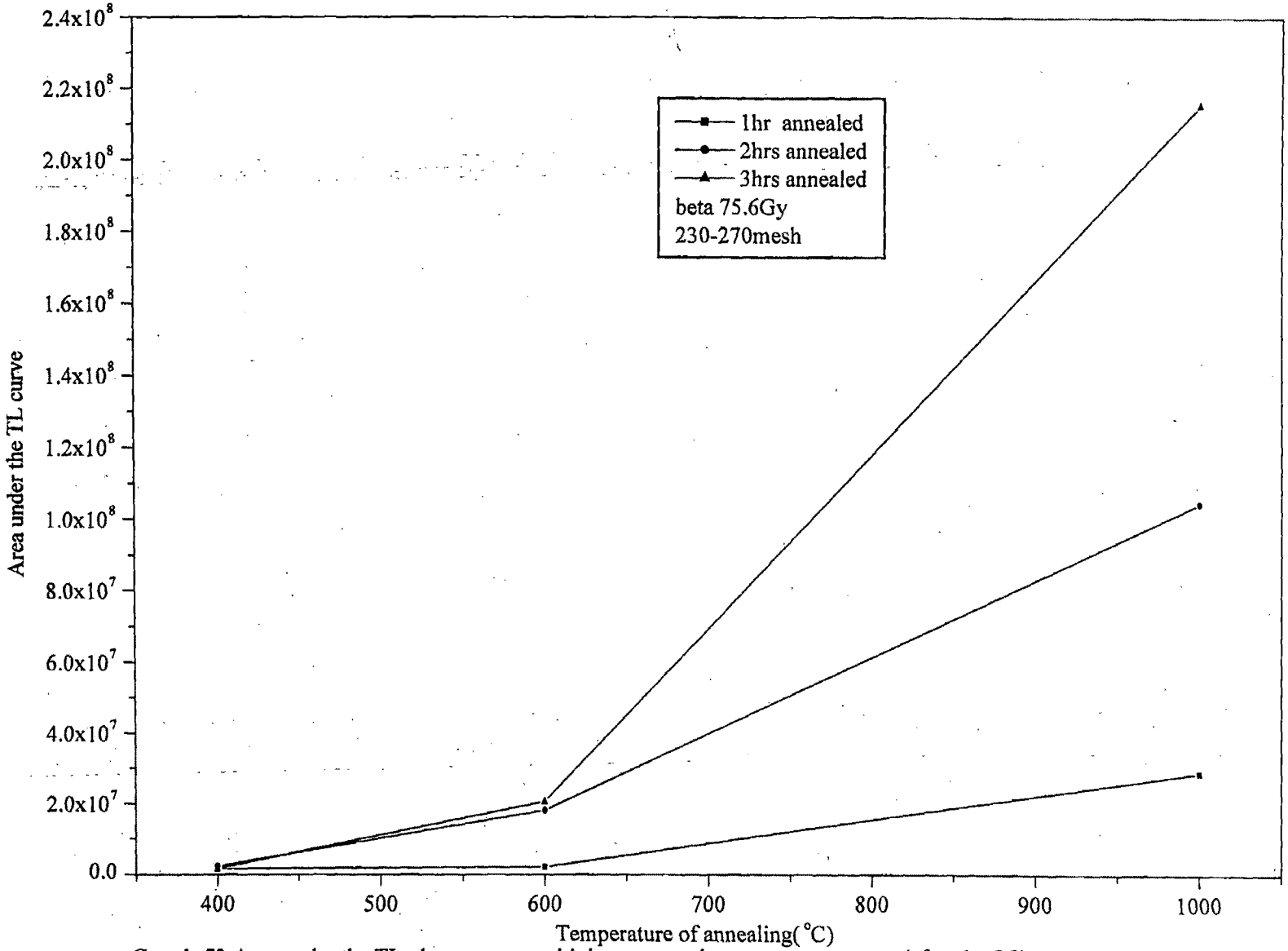
Graph-49 Area under the TL glow curve sensitivity measured at room temperature (after the OSL measured at room temperature) of 2.52Gy beta dose for 230-270mesh at different annealing durations.



Graph-50 Area under the TL glow curve sensitivity measured at room temperature (after the OSL measured at room temperature) of 5.04Gy beta dose for 230-270mesh at different annealing durations.



Graph-51 Area under the TL glow curve sensitivity measured at room temperature (after the OSL measured at room temperature) of 25.2Gy beta dose for 230-270mesh at different annealing durations.



Graph-52 Area under the TL glow curve sensitivity measured at room temperature (after the OSL measured at room temperature) of 75.6Gy beta dose for 230-270mesh at different annealing durations.

Smith and Rhodes attempted to fit green light stimulated OSL decay of quartz and later researchers explained the changes in OSL decay pattern in terms of alterations in the recombination centers for which delocalised transport via the conduction band was attributed.[24, 40]

In the present investigations, it is observed that not only non-multiple component OSL decay nature but the clear increase in OSL as a function of illumination time up to a maximum before the decay starts for the specimens having implemented below critical protocols such as high dose for lower pre-thermal treatment and/or, lower dose for higher pre-thermal treatment.

Several workers have attempted to explain such type of decay nature, which they attributed to the contribution of shallow trap during OSL measurements at room temperature. In this connection Mckeever et al., suggested that several processes are seen to influence the behaviour of OSL curve as a function of temperature, depending upon the precise mechanism by which the OSL is produced. Shallow traps seen to play an important role whenever the OSL process involves stimulation into the delocalized band. The trapping of charge by the shallow traps, followed by the thermal release of this charge back into the conduction band, yields non-exponential tail to the OSL decay curve, reduces the initial intensity of the OSL, and can, in the right circumstances, give rise to a peak in the OSL curve.

Experimental results show that the temperature of the measurement, temperature of pre-heat treatment, irradiation dose and intensity of illumination light govern whether a peak appears or dose not appears in the OSL curves. Mckeever and Morris gave an explanation regarding contribution of low temperature traps during OSL production and on the same basis they also explained the OSL decay results.[22]

In general, however the temperature dependence of OSL is complicated by the presence of shallow tarps e.g. when the OSL stimulation

is performed near room temperature a long tail to the OSL curve is often observed. This is believed to be (at least partially) caused by the influence of shallow traps, which localize charges released during illumination and slowly release them again at a rate determined by the trap depth and the sample temperature. This is a slow, optically stimulated phosphorescence component to the OSL decay curve. In this way, shallow traps may slow down the OSL decay processes. Furthermore, shallow traps give rise to a temperature dependent OSL component since at higher temperatures they become less effective at trapping charge.[25]

In our observations such slow optically stimulated phosphorescence component to the OSL decay curve is clearly noticeable, hence it is believed that the temperature of annealing also contributes to the rate of release of localized charge carriers from shallow traps during illuminations. From the viewpoint of applications of OSL in dosimetry, the OSL dose response curve was investigated for different protocols. The area under the OSL curve was also obtained against the imparted dose to the specimens after implemented protocol. Here it is observed clearly, that for lower (400°C) temperature of annealing OSL reduces with dose up to 25.2Gy dose and it increases beyond this dose. For the specimens pre-heat treated at moderate temperature (600°C) this critical dose moves to the lower side i.e. as much as 5.04Gy. However, the specimens pre-heat treated at higher temperature (~1000°C) OSL doesn't reduce for any dose, but increase with the dose. The similar pattern of behaviour was observed even in the area under the OSL curve.

Mckeever et al., explained the interesting OSL decay pattern for different doses and different temperature of annealing on the premise of transport via the conduction band. They considered first-order kinetics (i.e. negligible re-trapping). This appears to be a reasonable assumption for quartz but it can't be guaranteed to hold throughout the OSL decay curve.

Furthermore a real sample is likely to have more traps than those indicated-including several optically active traps, deep competing traps, shallow competing traps as well as radiative and non-radiative recombination centers.[26]

It is believed that for lower temperature of annealing the lifetime of the charge in the shallow traps is much longer than the decay time giving rise to small OSL signal due to trapping in the low temperature traps, but re-trapping into deeper, competing traps, re-trapping into the optically active traps and non-radiative recombination produces non-exponential decay curve, which is discernible upon increase up to critical dose for low temperature of annealing. Therefore, the decrease in OSL up to critical dose for low temperature annealing is observed.

This can be better explained through Botter-Jensen model concerning the dose response of OSL. The essential elements of this model are shallow electron trap (level-1) that represents all those shallow traps in real materials, which are only partially stable at the temperature of irradiation (e.g.110°C TL peak in quartz).

Level-2 represents all those optically active traps that are emptied during OSL measurement (for example, the traps responsible for the 325°C TL peak in quartz-RBP).

Level-3 represents deep electron traps. These states do not empty, either optically or thermally during OSL or TL measurement. High temperatures thermal annealing may empty them.

In addition, there are two-recombination centers, Level-4 and Level-5. It is assumed, for the purpose of this model, that only recombination at Level-4 produces measurable luminescence. That is, it is assumed that recombination at Level-5 is either non-radiation, or produces

light at a wavelength outside the detection window of TL or OSL measurement.

During irradiation sequence, electrons accumulate in the electron traps and holes accumulate in the recombination centers. During the bleach, electrons are removed from Level-2 (optically active trap) and are redistributed among the other levels. Those that recombine at Level-4 and Level-5 during this process are then 'lost' from the system.

Annealing of the system is stimulated by either (i) removal of Level-5 (center removal) or (ii) the creation of additional Level-4 (center creation). In addition, it assumed that during annealing all surviving levels (including Level-3) are emptied of their trapped charge. Thermal emptying of the levels was thought to be a reasonable assumption since the annealing of the real materials takes place at high temperatures (up to 1000°C in some cases) sufficient to depopulate traps with thermal depths up to several eV. Clearly in a real system, some charge may remain trapped in deep levels depending upon the annealing temperature but one of the critical ideas being tested in the simulations is whether removal of charge from deep levels will affect the dose response of the system.[27, 41, 42]

The above results give rise to two effects- the first effect concerns the concentration of the luminescence centers (Level-4) compared with the concentration of the Level-5. In either of the annealed cases there is a large enhancement of Level-4 concentration in comparison to Level-5 (in one case the concentration of Level-4 is increased; in other the concentration of level-5 reduced). In either case, more electrons recombine with the luminescence centers or more light output (OSL or PTTL) results for the same absorbed dose i.e. the sensitivity are increased. The second effect governs the shape of the growth curves (not just the slope) and concerns the emptying of the deep traps during high temperature annealing. Empty, deep

traps can act as competing levels to the luminescence process during heating (for TL) or during illumination (for OSL). The present studies also show that on raising the temperature of annealing for any dose the OSL increases and also the area under the OSL curve also the increases. These observations are interpreted on the premise of change of concentration of luminescence center as per the above-mentioned model.

Mckeever et al., also noted sensitivity enhancement effect in both the TL and RL of quartz following annealing at high temperatures. In their conclusion they too suggested that the enhancement effects were related to alterations in the recombination centers, since RL and TL were affected in similar ways.[28]

Further, it is also been noticed that as the duration of pre-heat treatment increases, OSL also enhances. This is more predominant at certain temperature of annealing such as 600°C and 1000°C. These effects are also in accordance with the alterations in recombination centers. In addition to that, the structure change in quartz at different temperatures is also believed to play a vital role for their predominant effects at around that temperature. Several other workers also attribute sensitivity enhancement to the alterations in recombination centers and their site using spectral changes, which are due to the structure changes in synthetic quartz at high temperature annealing.[29] However, the phase-changes of SiO_2, namely the alpha-beta quartz transition at 573°C and the beta quartz to β_2-tridymite transition at 870°C are well established. These frozen phase changes due to pre-heat treatment around that temperature is believed to be responsible for the above-mentioned changes in OSL pattern. Thus, the OSL intensity is strongly dependent upon pre-thermal treatment, duration of pre-thermal treatment and laboratory irradiation. The shape of the decay curve also remains sensitive to physical such factors.[39, 43]

Mckeever, Wintle and Morries interpret such types of involvement of low temperate TL traps successfully. This interference of shallow traps during OSL production is interpreted by recuperation (transferring process), role of other than OSL deep traps temperature effect etc. The roles of shallow tap during OSL measurement are discussed by several workers, which are already mentioned earlier. However, researchers proved the role of shallow traps by recuperation process, such a process also called a transferring process of trapped electron from deep OSL trap to shallow trap and vice to versa during optical stimulation and subsequent heating process. It gives a proof of involvements of shallow trap. Aitken and Smith gave an interpretation for the presence of shallow trap and labeled it as recuperation i.e. a transferring process in OSL production.

Wintle and Murray further utilized this idea and explained clear-cut picture of recuperation process, which is highly efficient in OSL creativity. Therefore, in the present investigation, such type of process is highly attributable to growth of OSL and importance of annealing treatment, irradiation of the sample and justification of the role of shallow trap. Further they calculated the charge cycling from, and to, the deep OSL trap.

Wintle and Murray explained the transfer of charge from 110°C to OSL deep trap (during stimulation) by using model in which, the deep OSL trap and the 110°C traps are the only electron traps present. It is assumed that both the OSL and TL used the same recombination centers, and that the ratios of probabilities of luminescent and non-luminescent recombination remain the same and constant during OSL and TL. They described, 't' is the fraction of the total number of electrons from the deep OSL trap stimulated by a 0.1sec exposure to green light. Then the fraction, which goes to recombination is 'st', and the fraction, which is re-trapped by the 110°C trap, is (1-s) t. i.e. during stimulation,

t-st = (1-s) t Where, t - OSL trap; st- recombination center; (1-s) t - 110°C TL trap.

During heating, which completely empties the 110°C TL trap, a fraction r of the electrons thermally stimulated from the 110°C trap will recombine, and a fraction (1-r) will be returned to the deep OSL. Although, green light is believed to stimulate the trapped charge directly into the conduction band there is no evidence of shallow trap effect- in spite of there being a strong 110°C TL peak when the sample is irradiated at room temperature in the laboratory (thus showing that there is a significant concentration of shallow trap present). However, the relative strength of the shallow trap effect depends upon the excitation rate, which, in turn, depends upon the photo-ionization cross-section and illumination intensity. In particular, decreasing the intensity should lead to the effect of the shallow traps becoming more noticeable.[22]

Wintle & Murray explained, over 50sec or so of stimulation the shape of the room temperature OSL decay curve is dominated by the competition between and re-trapping of electrons released both from the deep OSL trap and subsequently from shallow traps. Thus the OSL decay curve shape doesn't contain significant information about the stability of the initial natural dose distribution. Only after a long period of stimulation (about 100sec) is there a significant contribution to the OSL signal from traps deeper than at 325°C or from shallow traps. They deduced that a single trap/luminescence center combination appears to be responsible for the great majority of the OSL signal.

From previous results of the OSL measurement at room temperature, and respective interpretation indicate that the presence of 110°C TL trap is creating an effect on shape and sensitivity of OSL characteristics. It is already mentioned that OSL is best method, which gives an advantage over

TL in its applications for dating and dosimetry. In regard to these applications, worker are more interested in deep traps, which are stable, slowly bleachable and gives a maximum contribution for releasing of an electrons with rapidly (~325°C). But during room temperature measurement of OSL there is availability of shallow traps, which capture electrons released from deep traps. On other hand we can say that during room temperature OSL process there is a loss of OSL efficiency. Therefore, scientific workers are trying to find out a technique to avoid this loss. However, they selected an appropriate elevated temperature OSL measurement technique and explained the importance of this stimulation temperature.

Smith and Rhode suggested the decay of the measured OSL signal from the sample held at 160°C during measurement. When a sample is heated above 100°C, any charge transferred to TL traps in the 100°C region is immediately evicted, simplifying the form of the OSL observed. The chosen temperature for OSL measurement must be sufficiently low that thermal quenching effect are minimal and that the removed of high temperature TL during measurement is insignificant. Being sufficiently high temperature minimizes the effect of re-trapping at lower temperature TL traps during OSL measurement (especially TL traps in the 100°C TL region). A temperature of 160°C was found to be good compromise. No detectable signals were observed when sample was held at 160°C for 100sec[31]. Wintle and Murray also supported Smith and Rhodes statements and they fully utilized such experimental technique for archaeological and geological dating. An advantage of elevated temperature OSL measurements technique is to reduce re-trapping probability and an increase in the efficiency of OSL. In this connection, Wintle and Murray mentioned the importance of isothermal OSL measurement. It was proposed that routine measurements of

OSL on quartz are carried out at a temperature at which the 110°C peak doesn't play a major role. Temperatures of 125°C and above would be appropriate. Therefore, they concluded that it would be an advantage to make the OSL measurements at elevated temperature to prevent the 110°C trap form trapping electrons. The response of different part of OSL decay curve from the same quartz sample are examined relative to one another when different experimental conditions are applied. In particular, experiments were designed to separate the effect of different temperature dependent phenomena, thermal quenching, thermal assistance and cycling of charge through low temperature trap. They pointed out; optical stimulation can be carried out at any temperature below that at which the optically stimulated trap empties because of thermal stimulation.[32]

4.4 OSL measured at 160°C.

In the previous study a role of shallow trap (110°C TL peak) on OSL decay at room temperature was discussed. It was shown that slower and non-exponential OSL decay along with reduction in OSL intensity seems to be due to such traps. The non-exponential OSL decay has been indicated using initial stimulation peak time, and reduction in OSL sensitivity.

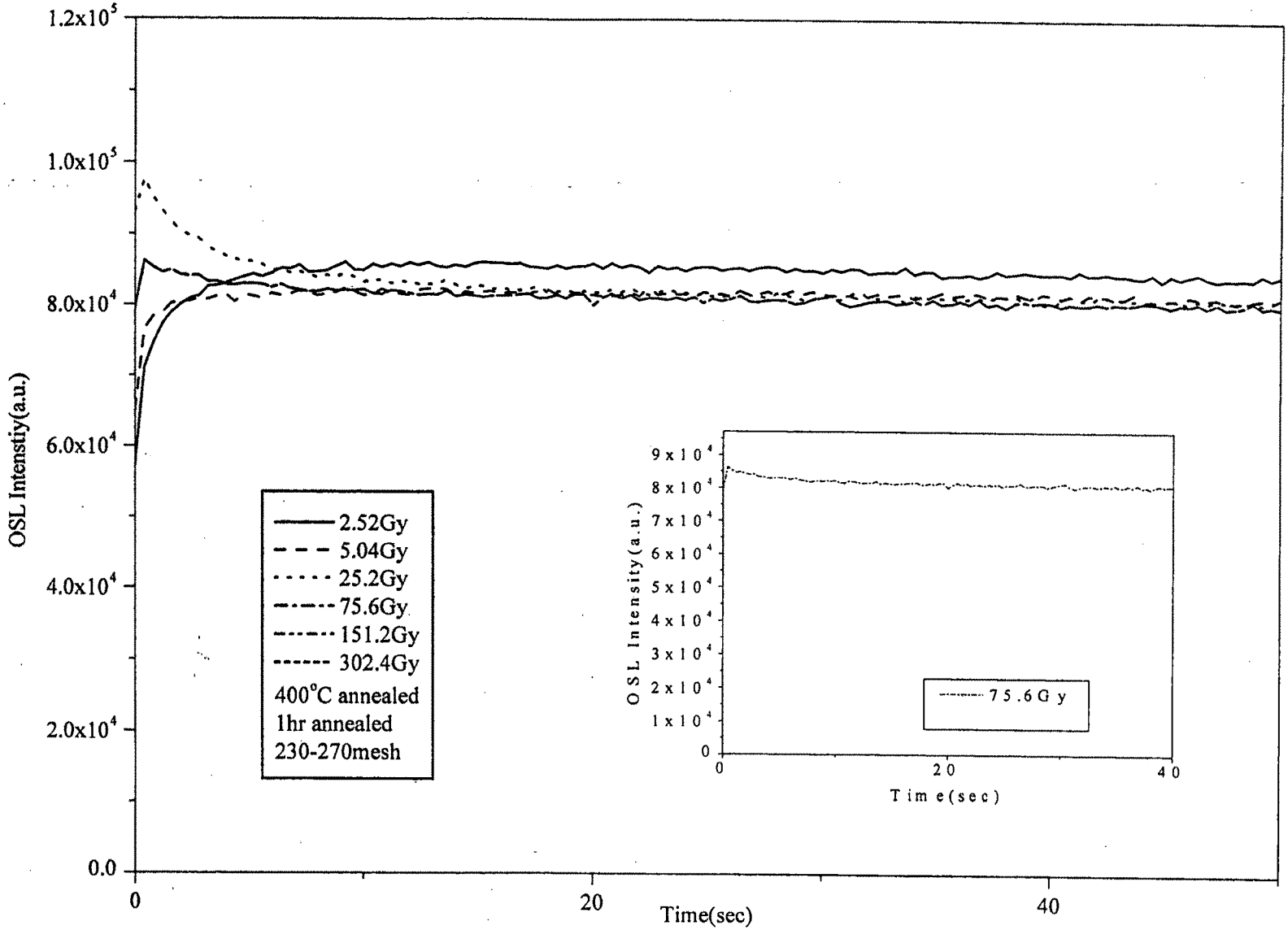
According to Mckeever et al,[22] non-exponential OSL decay is attributed to the re-trapping of optically released charges, which lead to decrease in OSL efficiency, and hence it is not a good candidate for OSL/TL dosimetry.

To avoid these problem researchers suggested that the OSL measurements should be performed at elevated temperature. According to B.W.Smith et al.,[31] this protocol will eliminate the contribution of 110°C TL trap, which in turn results in increased OSL and exponential OSL decay, which is mainly applicable in OSL dating and dosimetry applications of

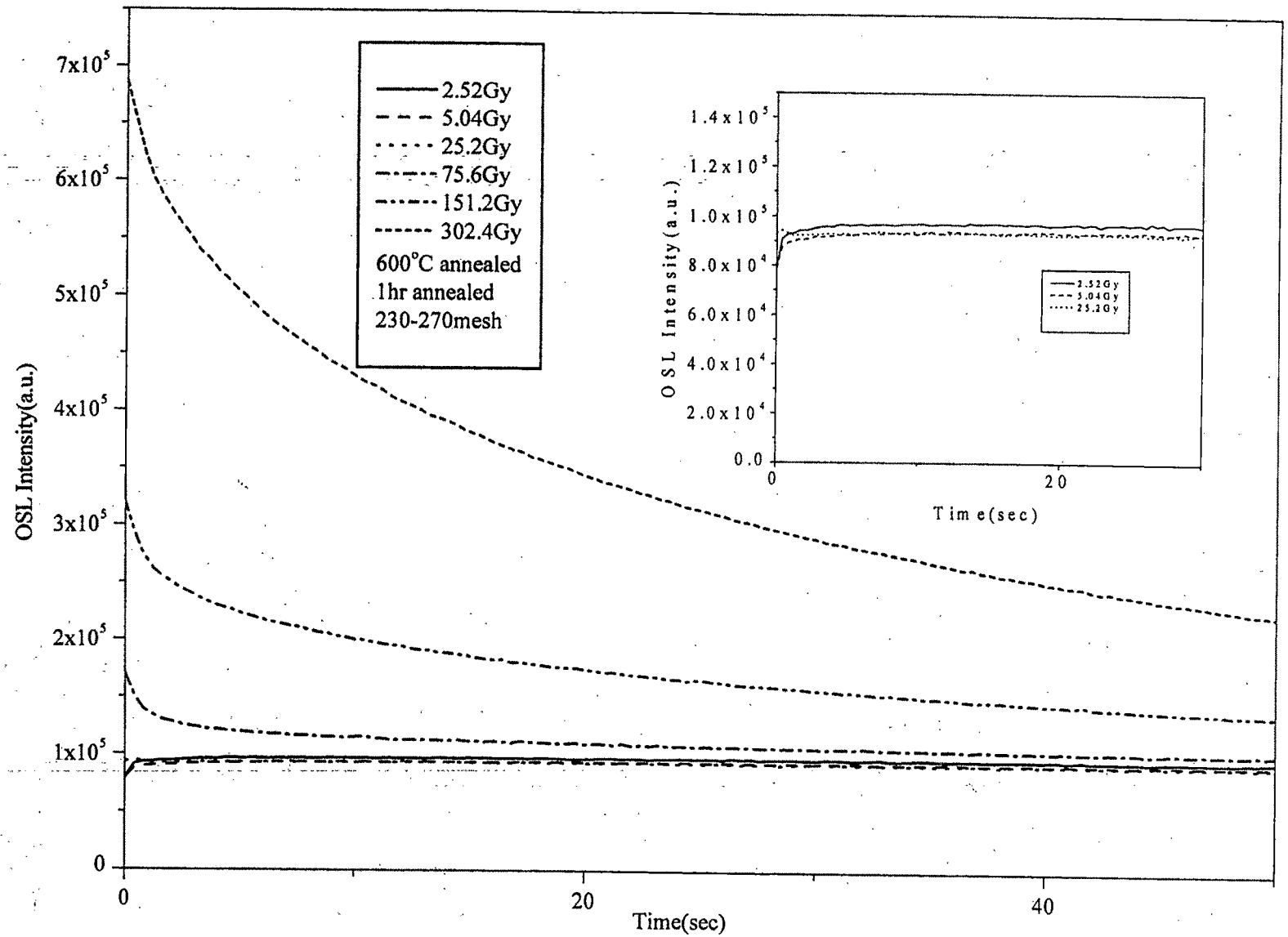
quartz specimen. Therefore, in this part of the study the optical stimulation was performed at an elevated temperature (at 160°C). For the purpose of comparison, OSL decay were recorded at an elevated temperature for different doses as well as for different pre-heat treatments, as similar to that of OSL decay recorded at room temperature.

Here, the specimens were pre-heat treated at 400°C, 600°C and 1000°C for 1hr separately; subsequently each specimen was exposed to beta doses from 2.52Gy to 302.4Gy before recording OSL at 160°C. The results are shown in **Graph-53** to **Graph-55**. It is found that the specimen pre-heat treated at 400°C shows non-exponential OSL decay, even after increasing the dose up to 75.6Gy. However, the specimens given higher doses than 75.6Gy for such a pre-heat treated specimen display exponential decay. This is also clear from the decreased initial stimulation peak time and enhanced OSL intensity. For lower temperature of pre-heat treatment such a dose (75.6Gy) may be called as critical dose. Further, it was observed that when the specimen was pre-heat treated at 600°C the critical dose lowers down to as much as 5.04Gy. At critical beta dose (5.04Gy), initial stimulation peak time is high to 24.4sec and OSL intensity decreases up to critical dose. With the rise in beta dose from 5.04Gy to 302.4Gy, initial stimulation peak time is systematically reduced to 0sec with the rise in OSL intensity up to 687500a.u. from 94172a.u. **(Graph-54)**.

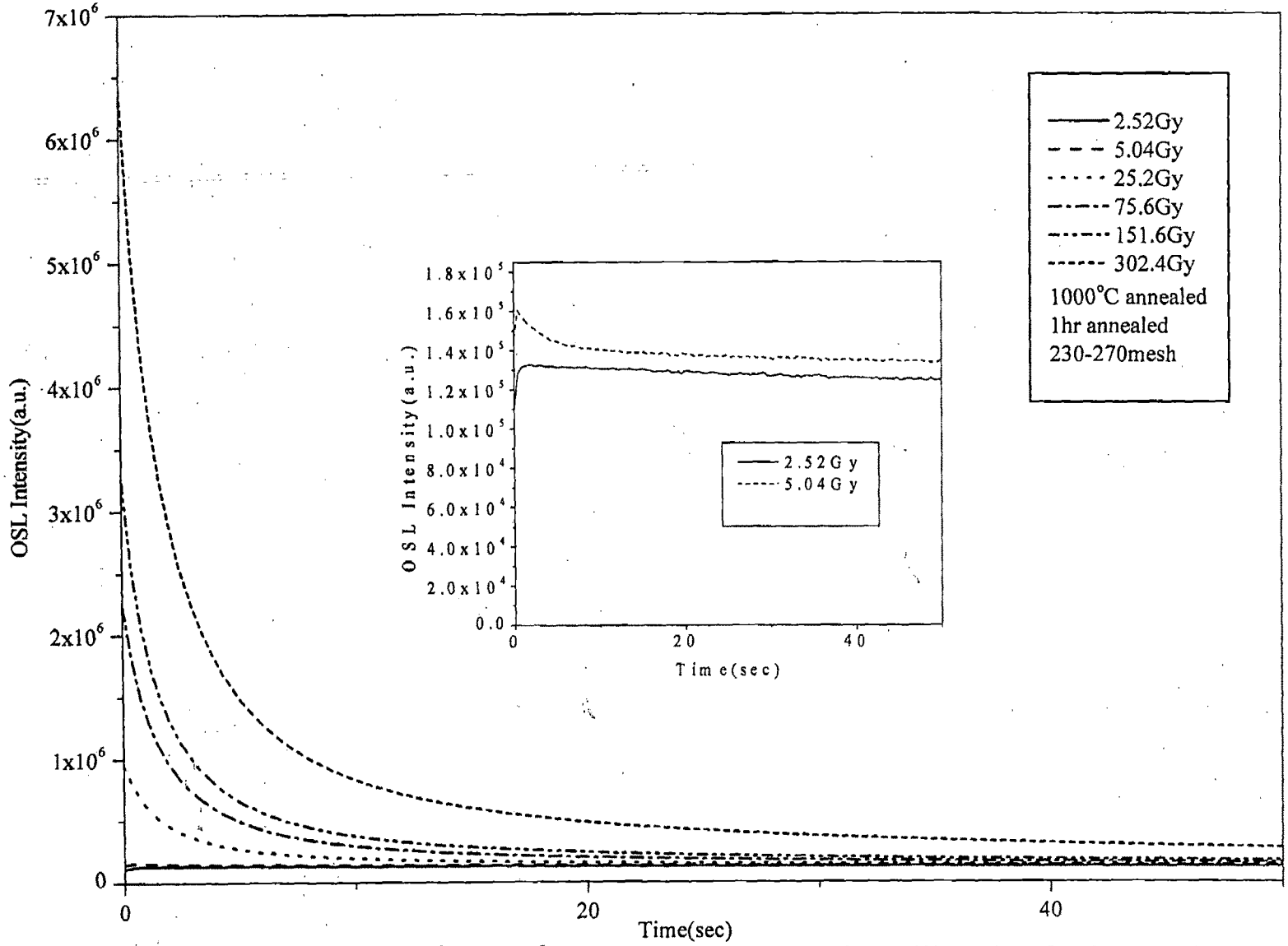
Similar pattern of behaviour was observed for the specimen pre-heat treated at 1000°C, with a distinction in initial stimulation peak time, which was much lower (0.4sec) compared to other pre-heated specimens. Another, significant observation is that at and beyond critical beta doses OSL intensity rises with beta doses for any temperature of pre-heat treatment of the specimen.



Graph-53 OSL measured at 160 °C of 400 °C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-54 OSL measured at 160°C of 600°C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-55 OSL measured at 160°C of 1000°C annealed sample for 1hr duration at different doses for 230-270mesh.

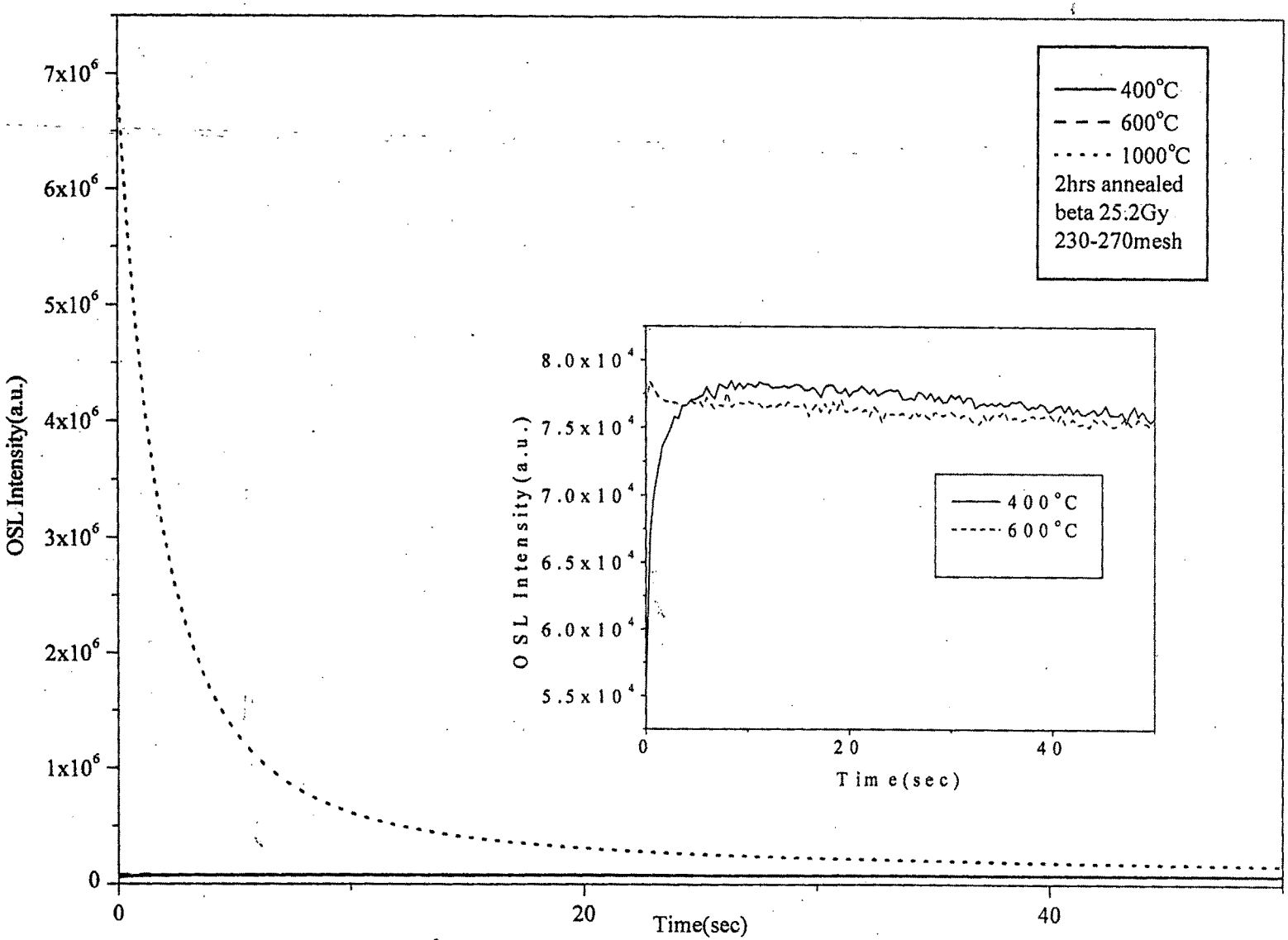
The studies were also done for higher duration of pre-heat treatment keeping other parameters constant. These studies were made for all the beta dose of interest and also for different temperature of pre-heat treatment separately. The results are presented as **Graph-56** and **Graph-57**. It is observed that the specimens pre-heat treated at 400°C, even with higher duration of thermal treatment display non exponential decay, but with a high initial stimulation peak time 8.4sec and 31.2sec and OSL intensity of 78427a.u. to 75638a.u. for 2 and 3 hours duration of pre-heat treatment respectively.

Further it is important to note that the specimens pre-heat treated at 600°C for 2 hours and 3 hours show exponential OSL decay with the reduction in initial peak time to 0.4sec as well as reduction in OSL intensity at 78346 and 75229a.u. respectively. But when specimens were pre-heat treated at 1000°C for higher durations clearly exhibited exponential OSL decay with an initial peak times as zero sec and the OSL intensity grows upto 10^6 a.u.

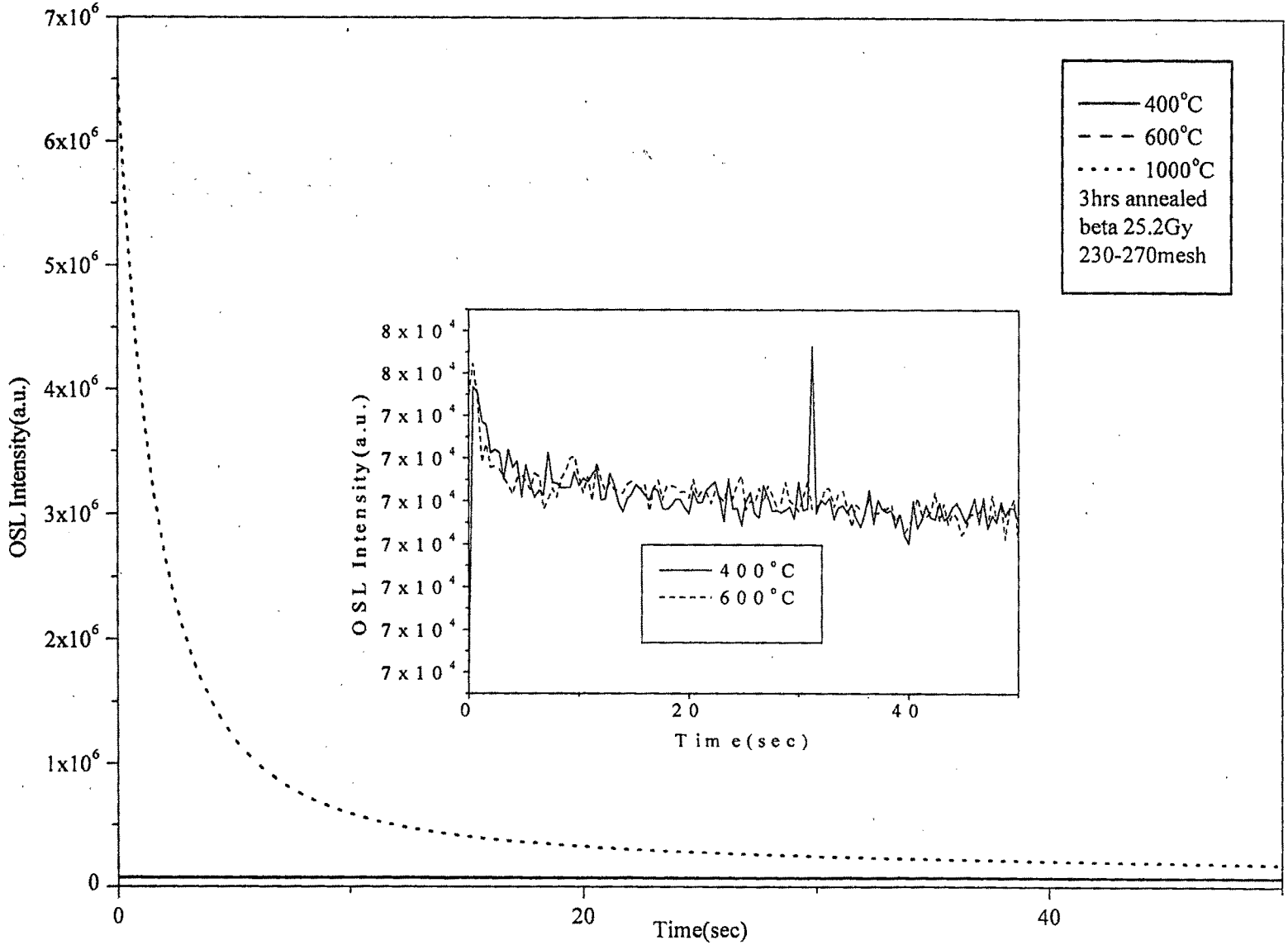
Graphs-58 and **Graph-59** show the OSL dose response for different temperature of annealing for a particular duration of pre-heat treatments. They indicate linear OSL dose response curve after critical dose suggested for different pre-heat treatments. Influence of dose for different pre-heat treatment on area under the OSL decay may be linked with center concentration.

It was pointed out that the area under the OSL decay remains more or less same up to critical dose, and it grows after the critical dose. This is true for any temperature of pre-heat treatment and any duration of pre-heat treatment also.

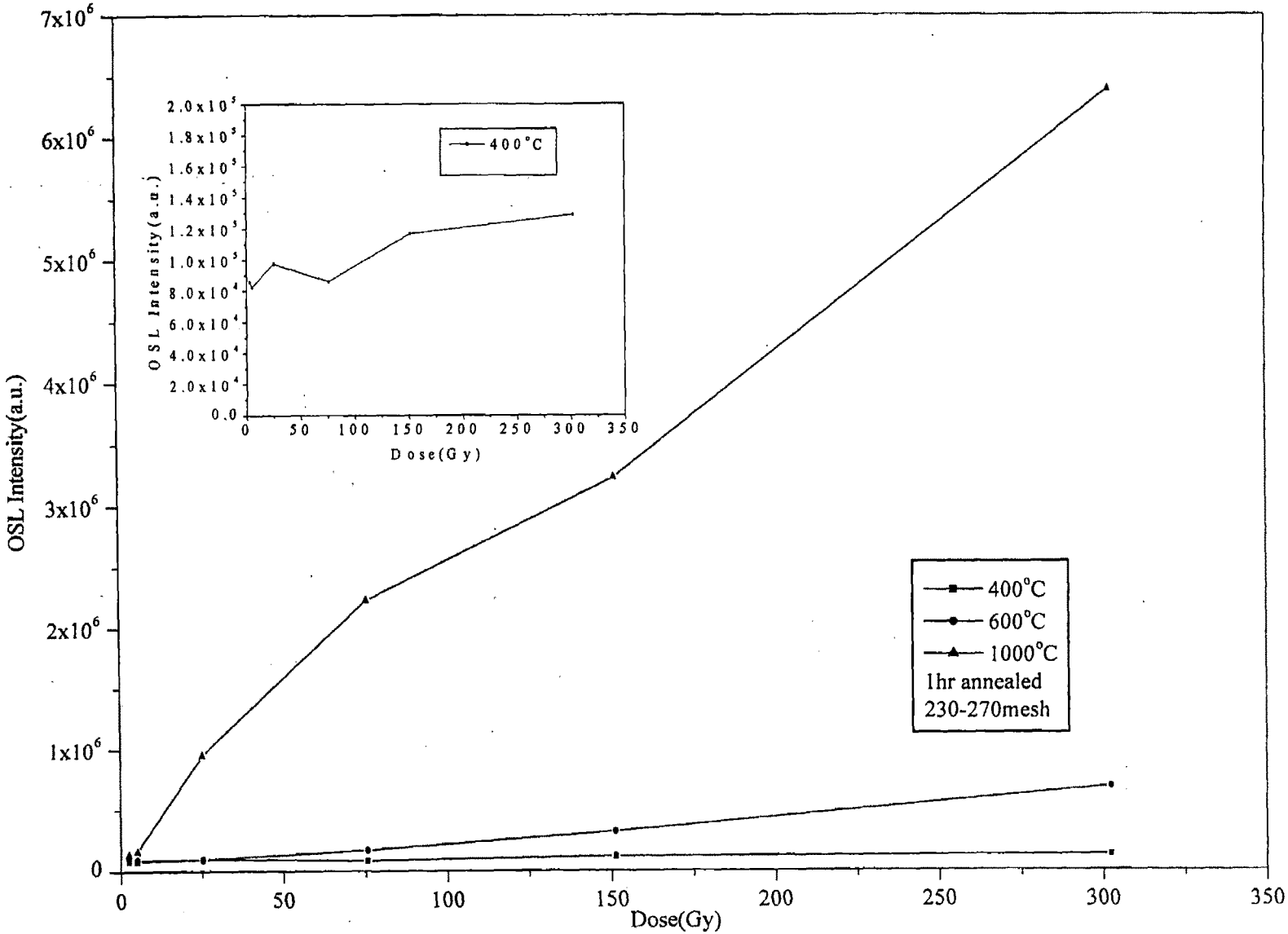
In yet another experiments, the TL glow curves were recorded at room temperature after recording optically stimulated luminescence at elevated



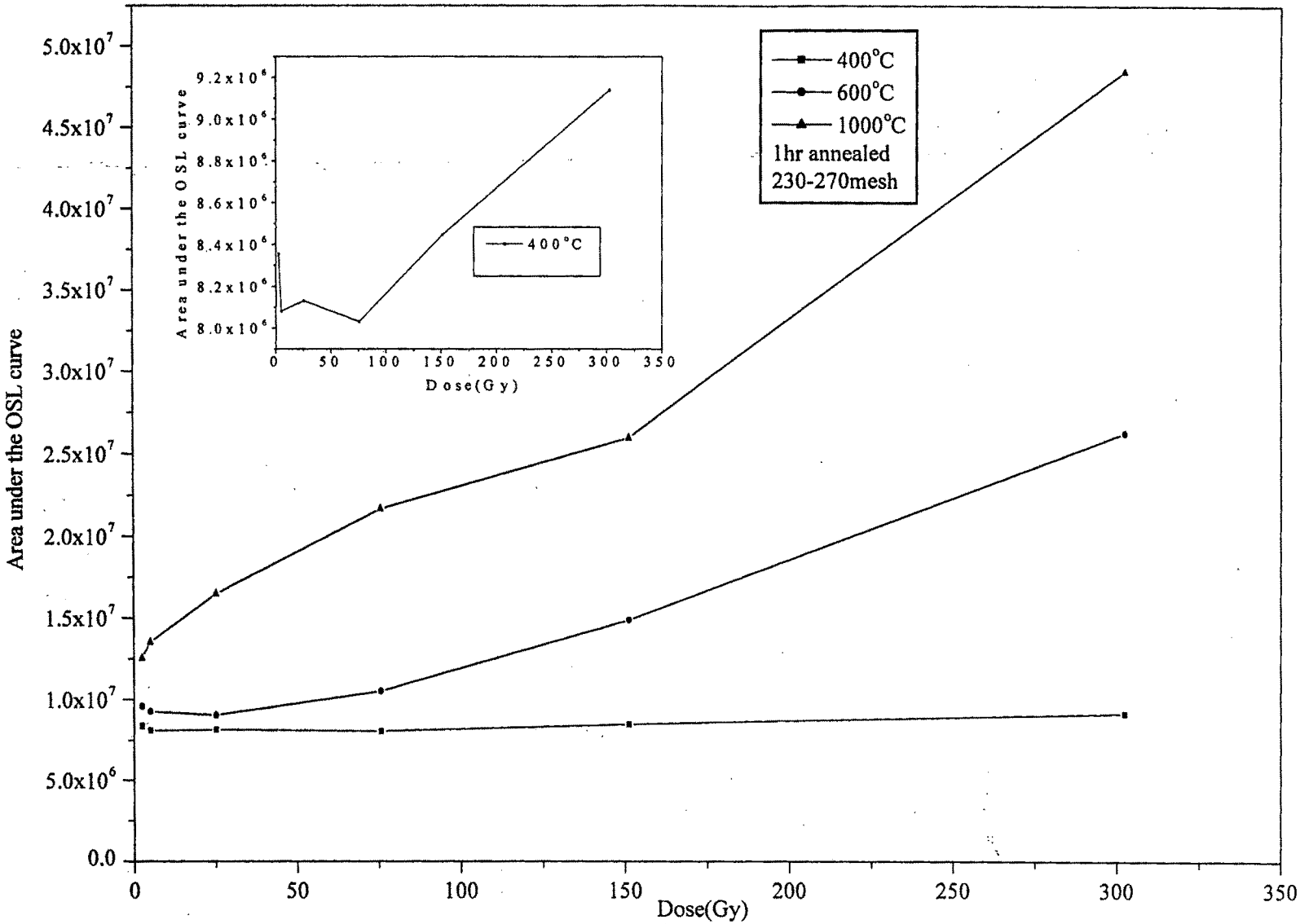
Graph-56 OSL measured at 160°C of 2hrs annealed sample for a given beta dose of 25.2Gy at different annealing temperatures for 230-270mesh.



Graph-57 OSL measured at 160 °C of 3hrs annealed sample for a given beta dose of 25.2Gy at different annealing temperatures for 230-270mesh.



Graph-58 OSL dose response curve at 160 °C of 1hr annealed sample for 230-270mesh at different annealing temperatures.



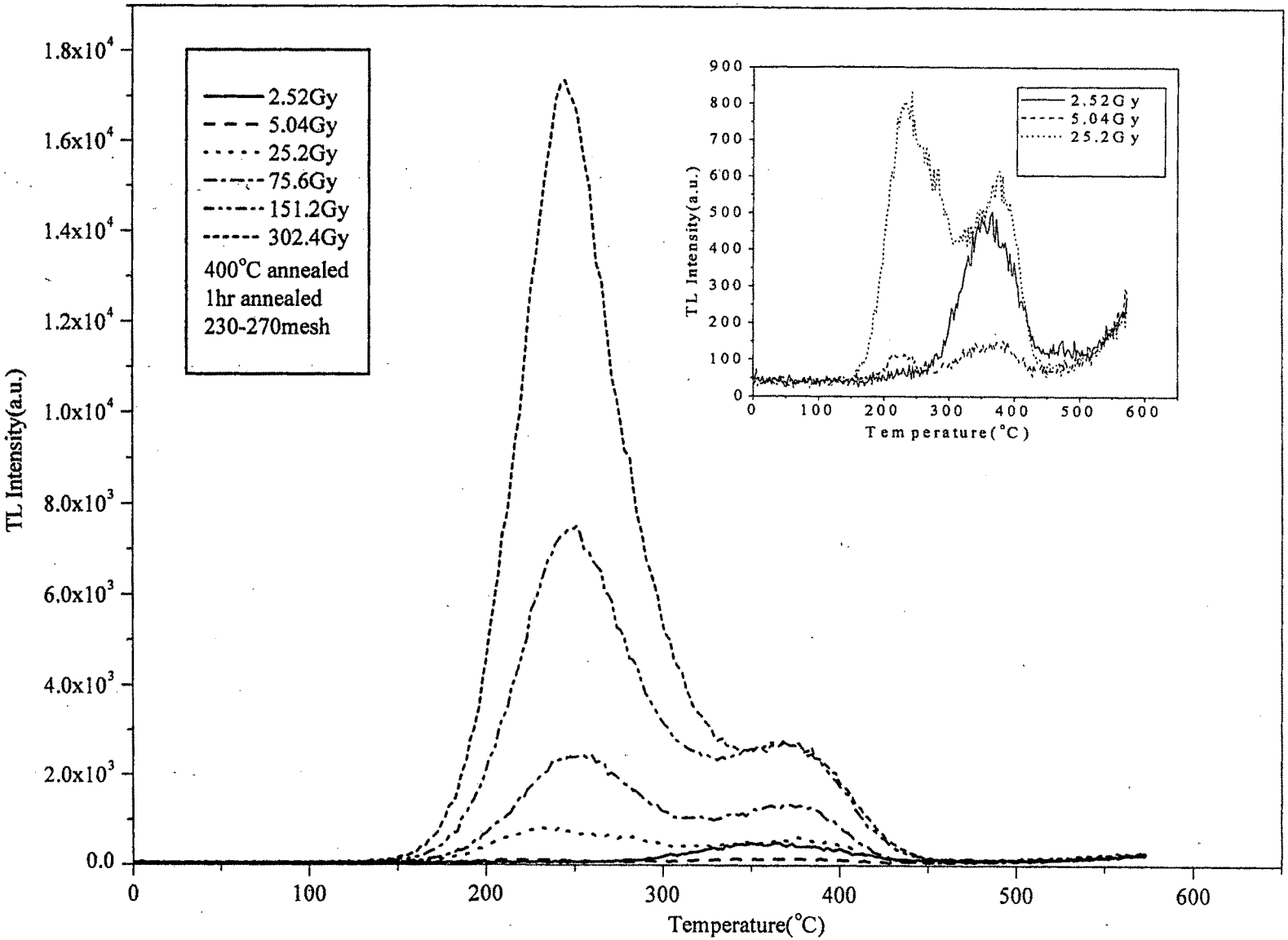
Graph-59 Area under the OSL curve against dose at 160 °C of 1hr annealed sample for 230-270mesh at different annealing temperatures.

temperature for various pre-heat treated and beta-exposed specimens. This study was carried out with a view to correlate the results of OSL records at elevated temperature. This correlation may strengthen the explanation of the OSL results. It is important to note that 110°C TL peak is absent for the specimens pre-heat treated at any pre-heat treatment as well as for any given beta doses. TL measurements for specimens annealed at 400°C for 1hr duration before being subjected to beta doses from 2.52Gy to 302.4Gy, show two well-defined glow peaks at 230°C & 375°C. It may be noted that upto 25.2Gy beta dose no significant TL feature is observed. Beyond this beta dose enhancement in TL intensity with high sensitivity in both the peaks become visible (**Graph-60**). For the specimens annealed at 600°C for 1hr pre-heat duration more or less identical TL behaviour was absent. At this pre-thermal treatment, it was found that a weak signal is observed below 5.04Gy beta dose (**Graph-61**).

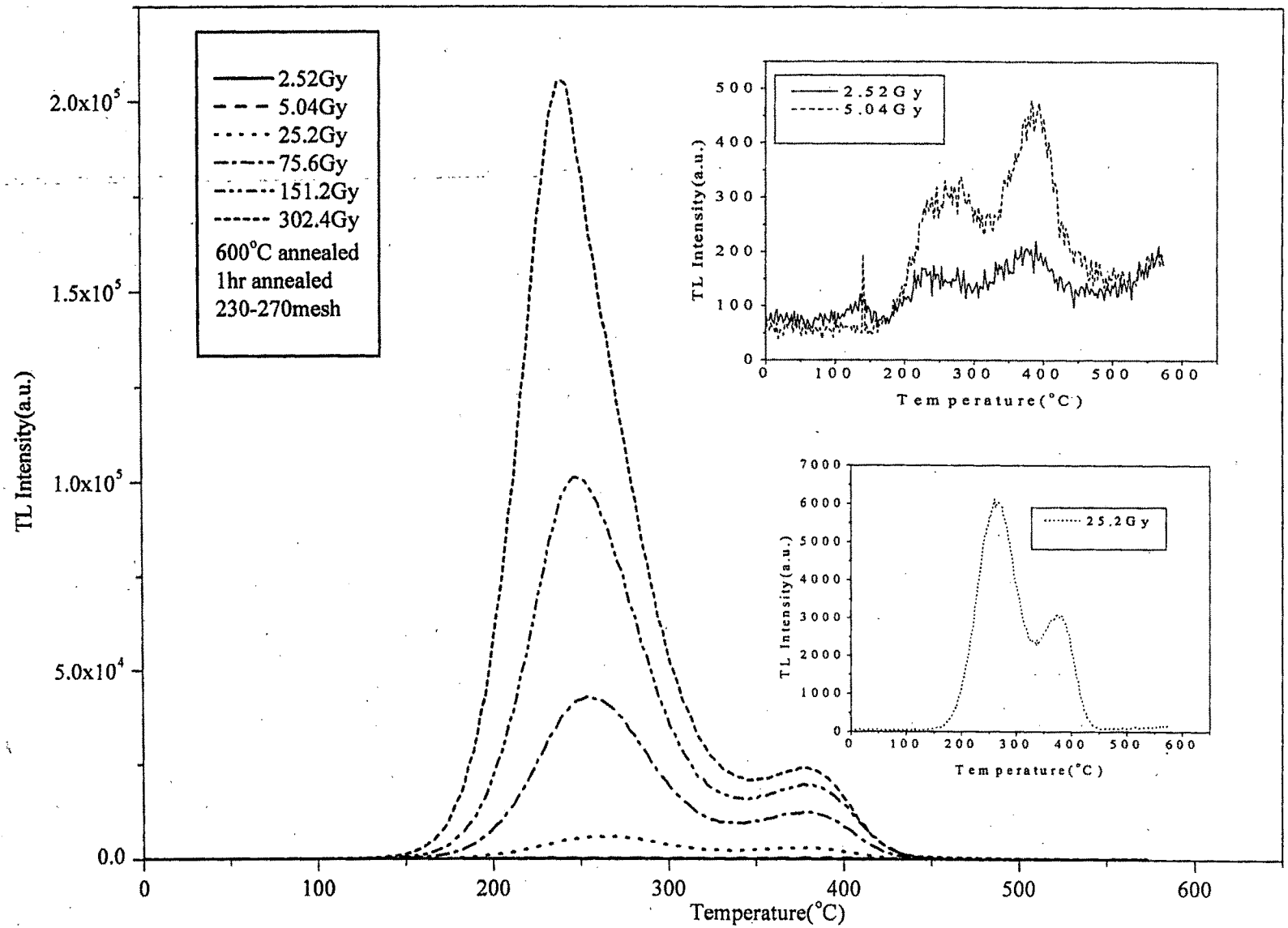
However, 1000°C annealed specimen exhibits weak TL signal at very low beta dose of ~2Gy (**Graph-62**). Both the TL glow peaks grow linearly with the rise in beta exposure. This is observed for all the specimens pre-heat treated at different temperatures and identical duration of annealing. It should be specially noted that the growth of 230°C TL peak is higher than the 375°C TL peak.

The TL glow curves recorded after OSL decay at elevated temperature for the specimens pre-heat treated for higher duration i.e. 2hours and 3 hours at different temperature followed by 25.2Gy identical dose display identical pattern of observations, but with a higher sensitivity in each case (**Graph-63** and **Graph-64**).

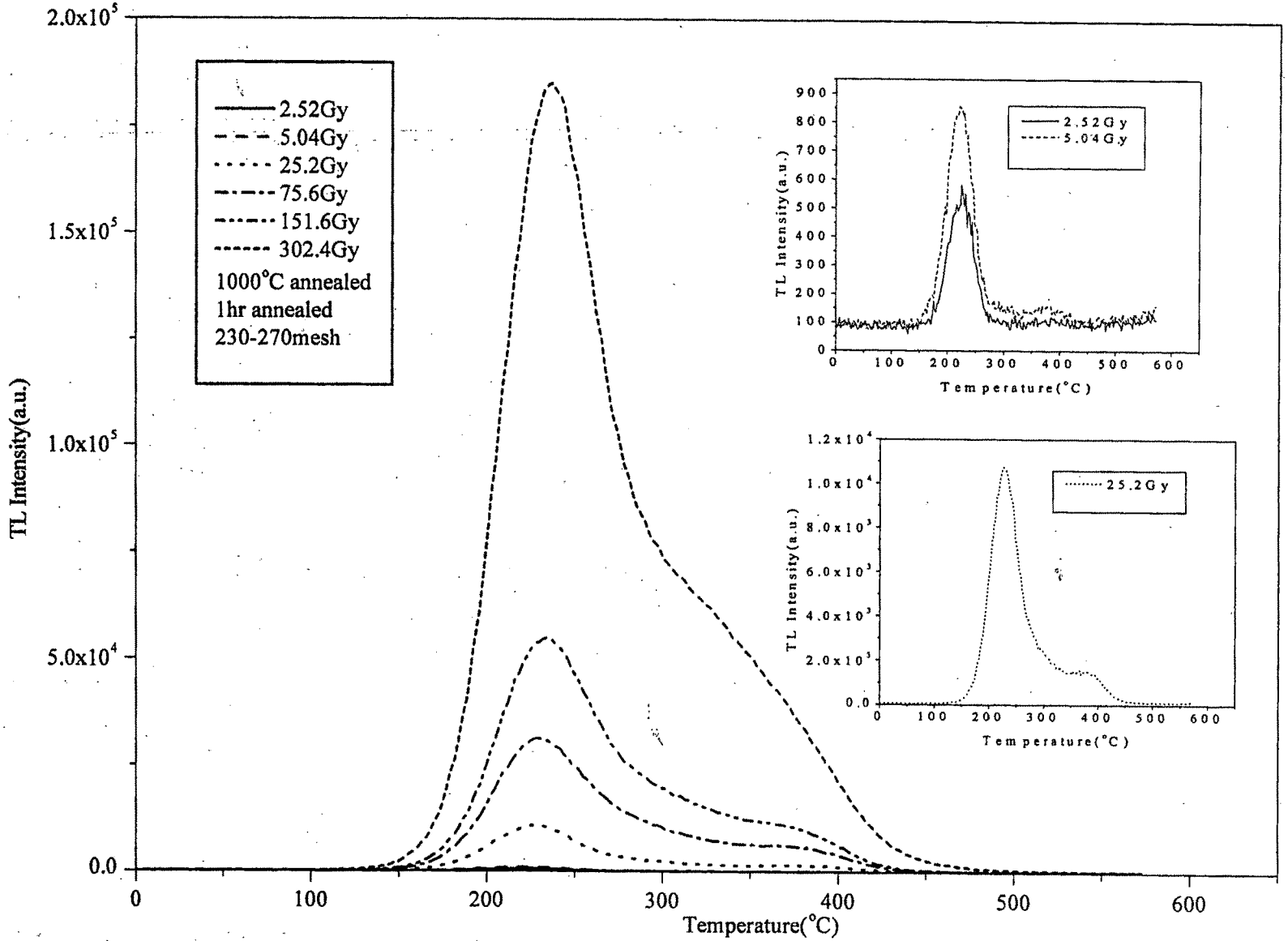
Owing to the special interest of 230°C TL peak for the purpose of dosimetry it is important to find appropriate pre-heat treatment protocol to be given to the specimen. The graphs were plotted relating the TL intensity and



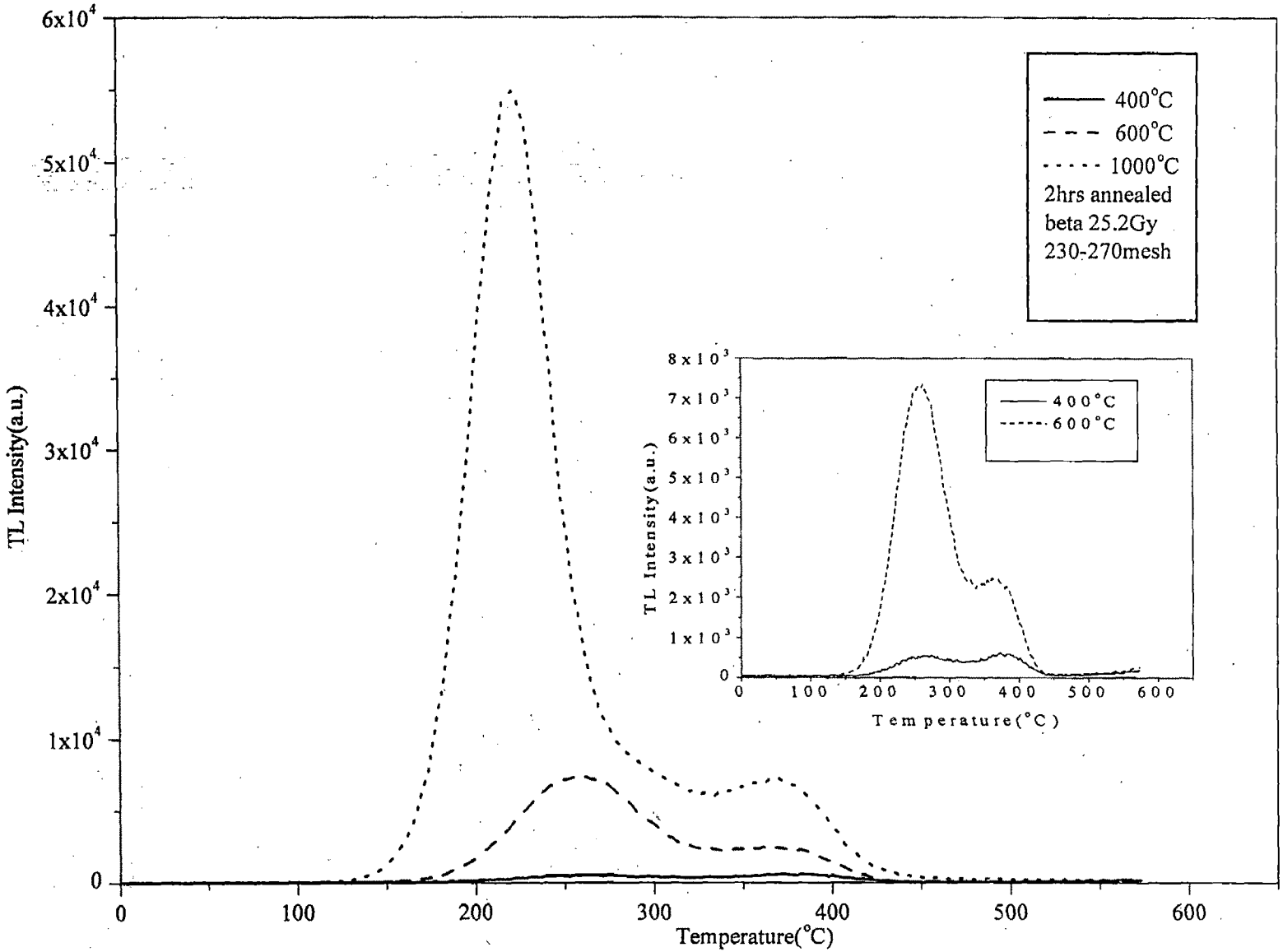
Graph-60 TL glow curve measured at room temperature (after the OSL measured at 160 °C) of 400°C annealed sample for 1hr duration at different doses for 230-270mesh.



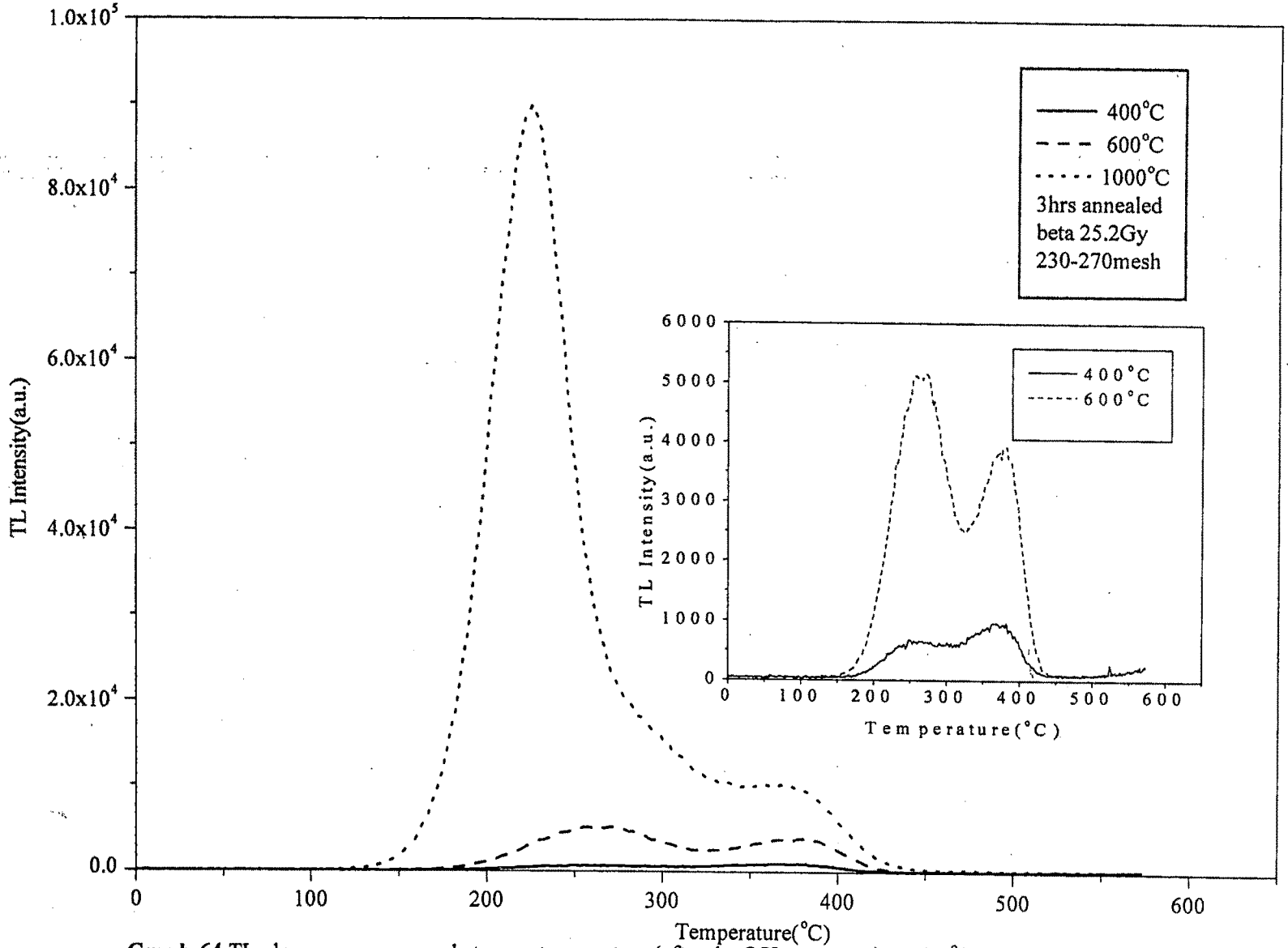
Graph-61 TL glow curve measured at room temperature (after the OSL measured at 160 °C) of 600°C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-62 TL glow curve measured at room temperature (after the OSL measured at 160 °C) of 1000°C annealed sample for 1hr duration at different doses for 230-270mesh.



Graph-63 TL glow curve measured at room temperature (after the OSL measured at 160 °C) of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



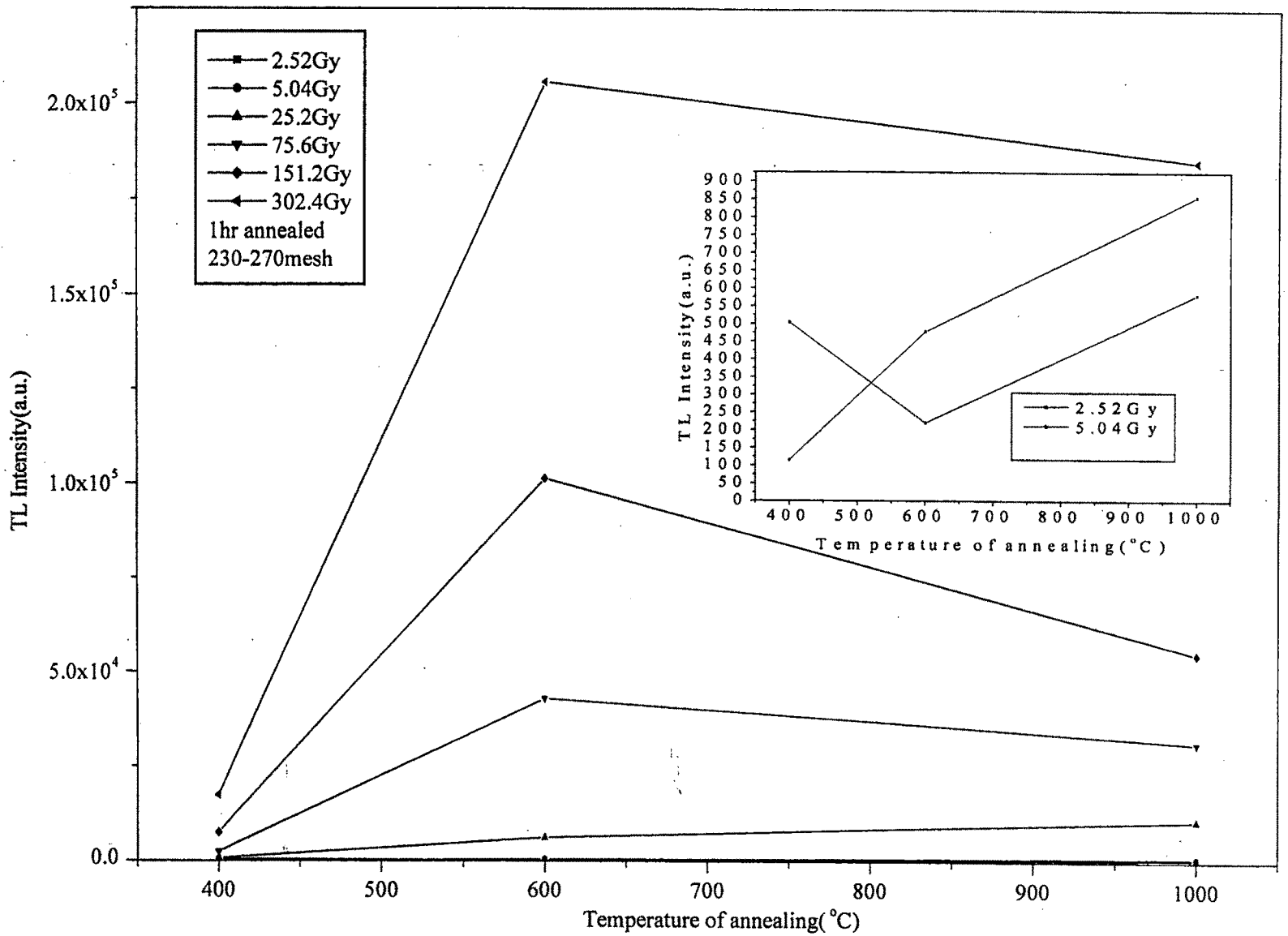
Graph-64 TL glow curve measured at room temperature (after the OSL measured at 160 °C) of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.

temperature of annealing for the given doses. It was found that as temperature of annealing increases TL intensity grows above the critical dose, but below the critical dose TL intensity decreases even with the increase in temperature of annealing. Similar pattern of TL sensitivity was also observed for the specimens pre-heat treated for higher durations. However, TL intensity decreases for the specimens pre-heat treated at 1000°C, for higher doses of exposures (**Graph-65** and **Graph-66**).

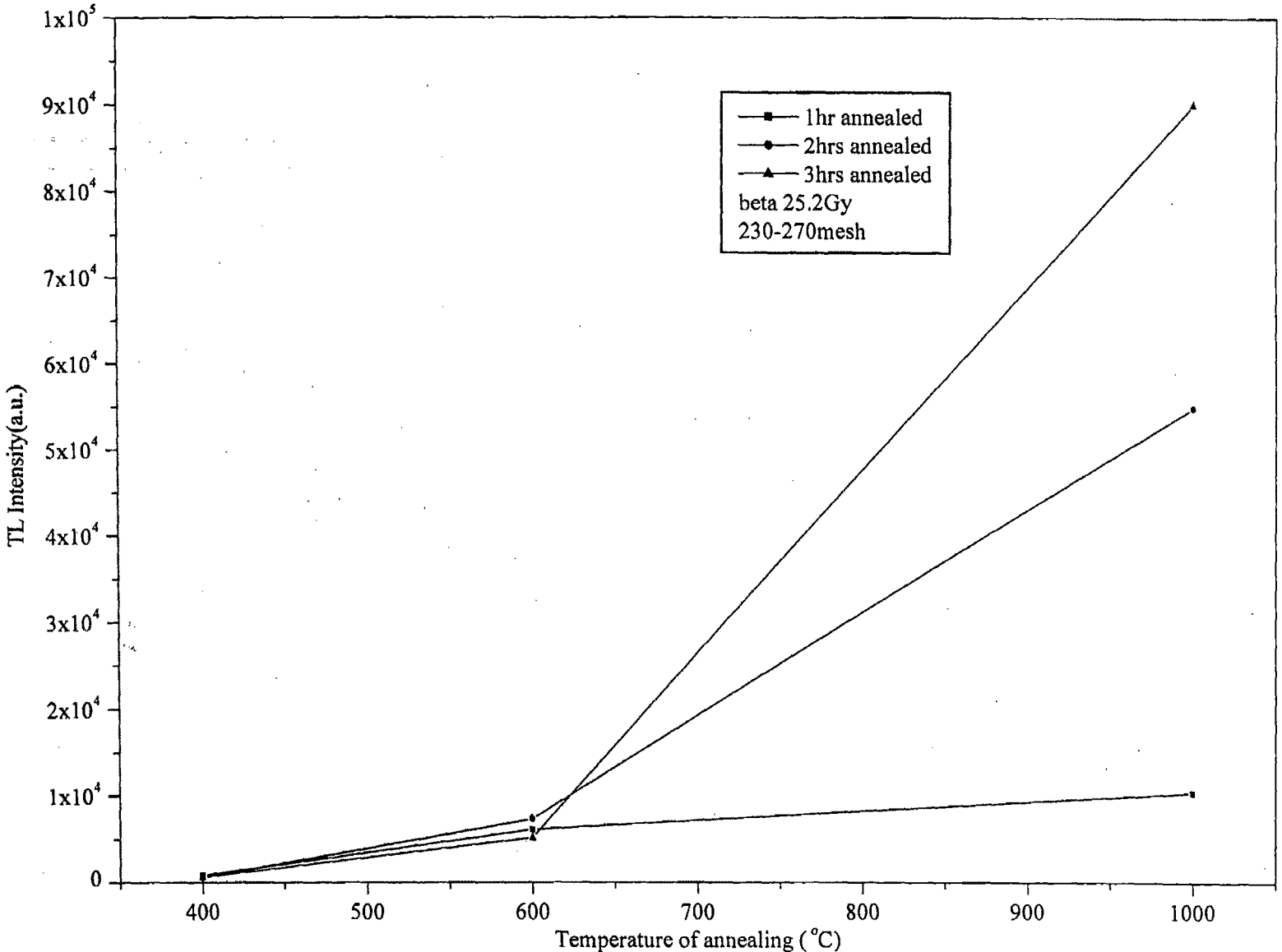
The area under TL grow curves were also plotted for different temperature of annealing. It shows that the area under TL glow curve increases with temperature of annealing for the beta doses less than 75.6Gy (**Graph-67**). Identical pattern of TL behaviour is discernible for higher duration of pre-heat treatment (**Graph-68**).

From the observation, it is clear that the OSL decay remains slow and non exponential even when recorded at elevated temperature of 160°C for the specimens exposed to beta doses lower than the critical dose and the specimens pre-heat treated at temperature lower than critical temperature of annealing and duration. This is in contrast with the suggestion of Mckeever et al.,[22] They believed that the slow and non exponential decay may be due to re-trapping of optically released charges at shallow traps, hence suggested a novel protocol that the OSL be measured at elevated temperature. With the present work it is needless to say that the physical treatments also contribute to the shape of OSL decay curve.

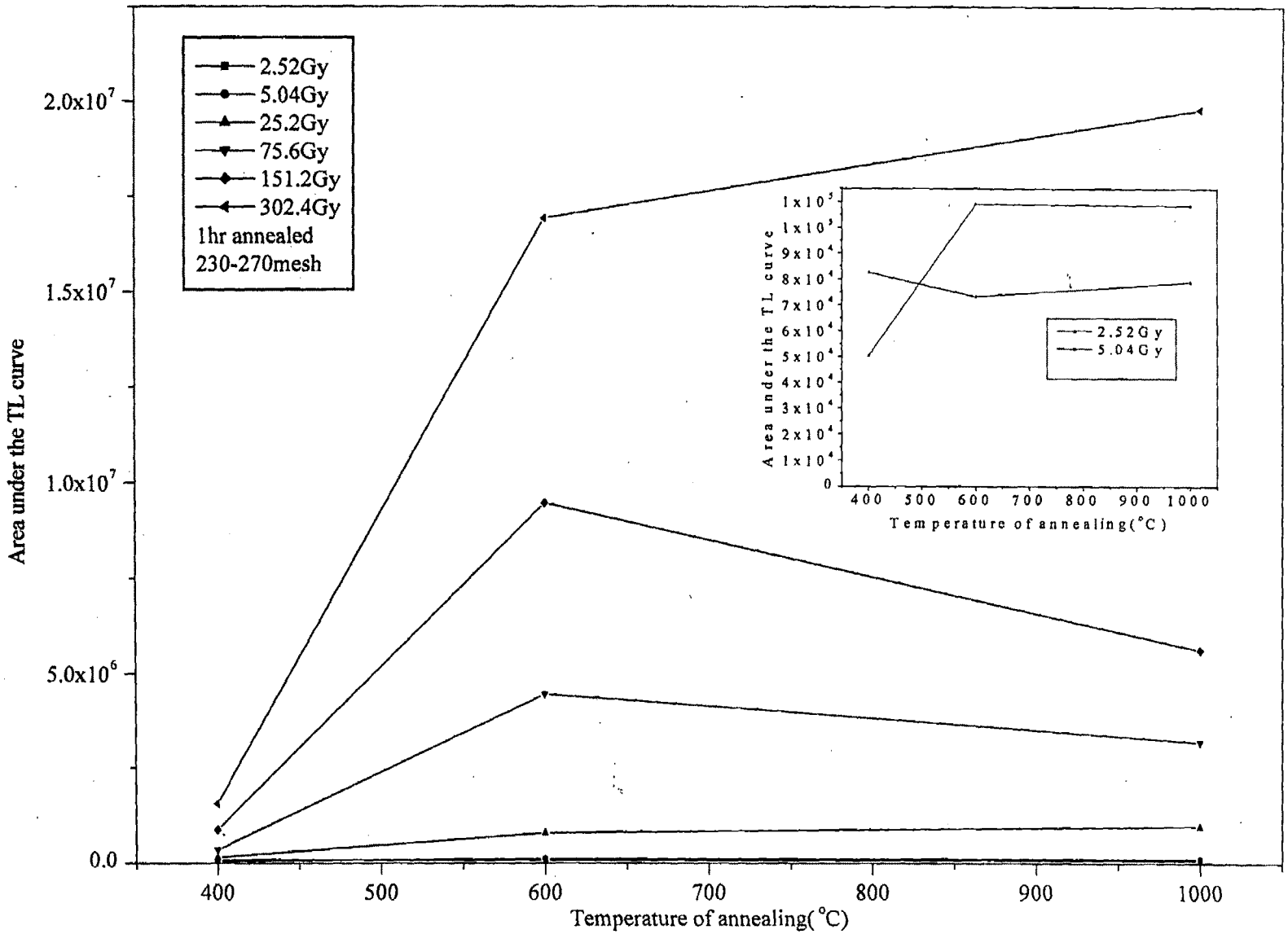
From the measurements of OSL decay at sample temperature greater than 110°C, Smith and Rhodes[31] argue that a multi component OSL decay curve can be expected if more than one trap is being emptied. The OSL data of the present work show single component OSL decay therefore, it is reasonable to believe that only one trap is being emptied for generation of OSL decay records.



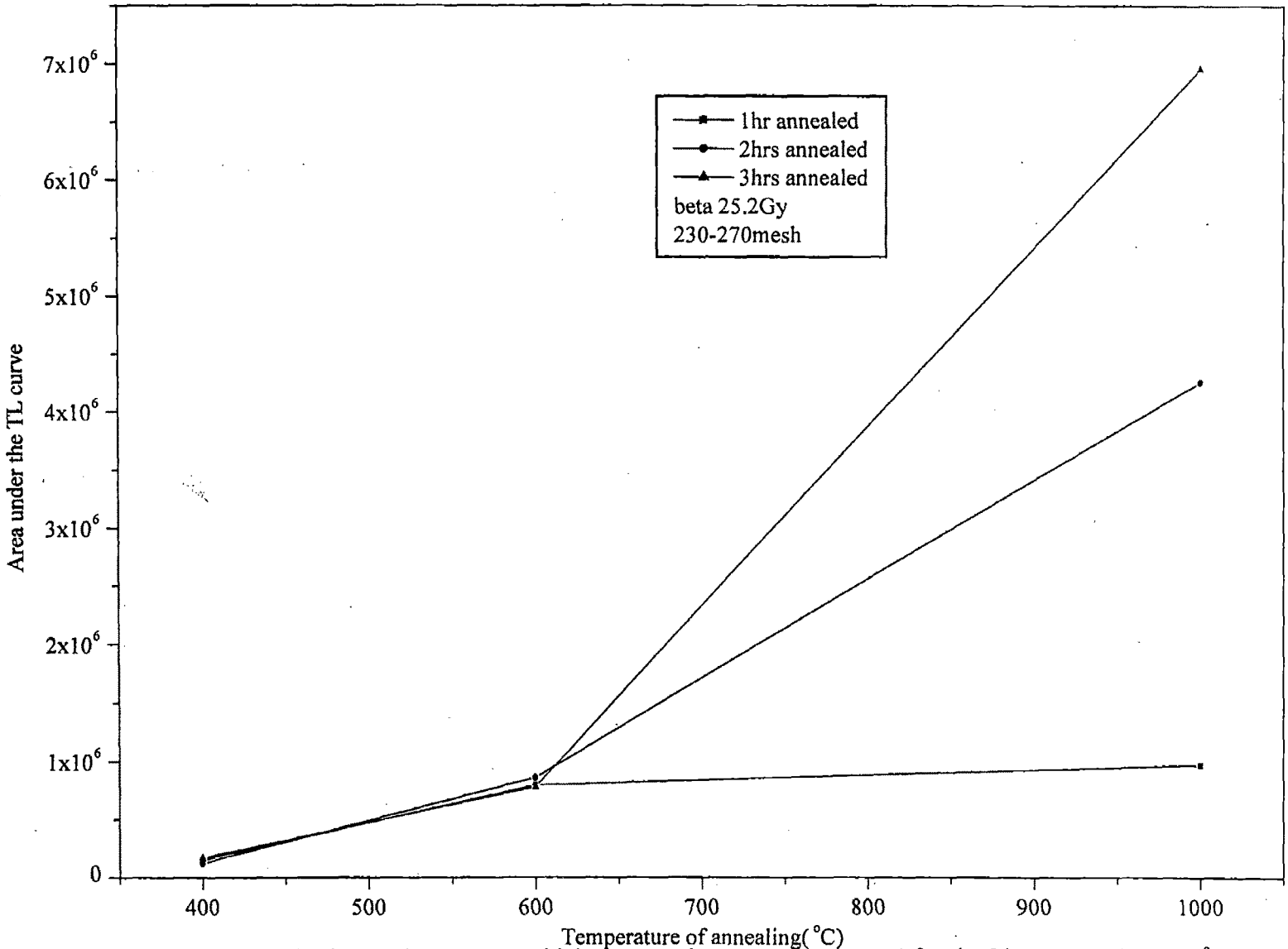
Graph-65 TL sensitivity measured at room temperature (after the OSL measured at 160 °C) of 1hr annealed sample for 230-270mesh at different doses.



Graph-66 TL sensitivity measured at room temperature (after the OSL measured at 160 °C) of 25.2Gy dose for 230-270mesh at different annealing durations.



Graph-67 Area under the TL glow curve sensitivity measured at room temperature (after the OSL measured at 160 °C) of 1hr annealed sample for 230-270mesh at different doses.



Graph-68 Area under the TL glow curve sensitivity measured at room temperature (after the OSL measured at 160 °C) of 25.2Gy beta dose for 230-270mesh at different annealing durations.

The patterns of present OSL decay rates are either fast or slow even when measured at elevated temperature up to 160°C. Regarding these observations researchers explain that there are various factors, which are involved in the production of OSL, especially at elevated temperature measurement; such as a thermally assisted OSL, donor-acceptor pair production and thermal quenching etc. These factors can be responsible influencing the shape and sensitivity of OSL. Smith & Rhodes observed an increase in OSL decay rate as the temperature of measurement increased. The present work reveals that besides measurement of OSL at elevated temperature, measurement of OSL above critical dose and critical pre-heat treatment to the specimen gives rise to faster decay. Researchers explain this phenomenon using the concepts of "thermally assisted process", "thermal quenching process" and "half-life of the charges in the shallow traps".[33] It is to be noted that the re-trapping into deeper and competing traps, re-trapping into optically sensitive traps and non-radiative recombination may result in the non-exponential decay.

Thermally assisted OSL measurements often reveal that more OSL is obtained at an elevated temperature compared to that obtained at lower temperature. It is clear from the discussion, that transport of charges through the delocalised bands if a sample contains a significant number of shallow traps, becomes inactive as the temperature increase. Therefore, thermal assistance affects the rate of optical ejection of charge, and will thus substantially affect the initial luminescence.[30]

The present observations show that the OSL decay does not become slower when measured at elevated temperature compared to that of OSL recorded at room temperature Moreover, for higher pre-heat treatments the OSL decay becomes faster and exponential. These indications reveal that

thermally assisted process may be responsible for this phenomenon as suggested by Rhodes et al.,[31]

Absence of 110°C TL peak, when OSL decay was recorded at 160°C, is found to be due to depopulation of carriers at 110°C during optical stimulation itself. Moreover it prevents re-trapping of carriers corresponding to 110°C TL peak. Further it was observed that the TL glow peaks were exhibited at 230°C & 375°C after recording OSL at an elevated temperature.

The higher growth of 230°C TL peak compared to that of 375°C TL peak with rise in either dose or temperature of annealing is attributed to the redistribution of charges taking place during optical stimulation. Specifically, the redistribution seems to be more probable for the traps corresponding to 230°C. This process finds support of "recuperation process" suggested by Aitken and Smith[34]. This interesting feature of redistribution of electrons among the ascribed traps may also be linked with the same luminescence center responsible for the emission of the TL peaks as suggested by Franklin et al.[19] From the spectral studies of TL records they concluded that rapidly bleached peak (RBP) at 150°C-180°C & 200°C-230°C having emission wavelength at 392 & 410nm respectively use the same luminescence center.

4.5 OSL measured at 160°C after post irradiation heat treatment at 290°C for different durations.

It is already established from the records of OSL decay at elevated temperature for the specimens differently pre-heat treated and irradiated that the OSL decay does not exhibit exponential nature below critical dose and critical pre-heat treatment. To overcome this difficulty researchers suggest "Post Irradiation Heat Treatment (PIHT)" at higher temperature, as this

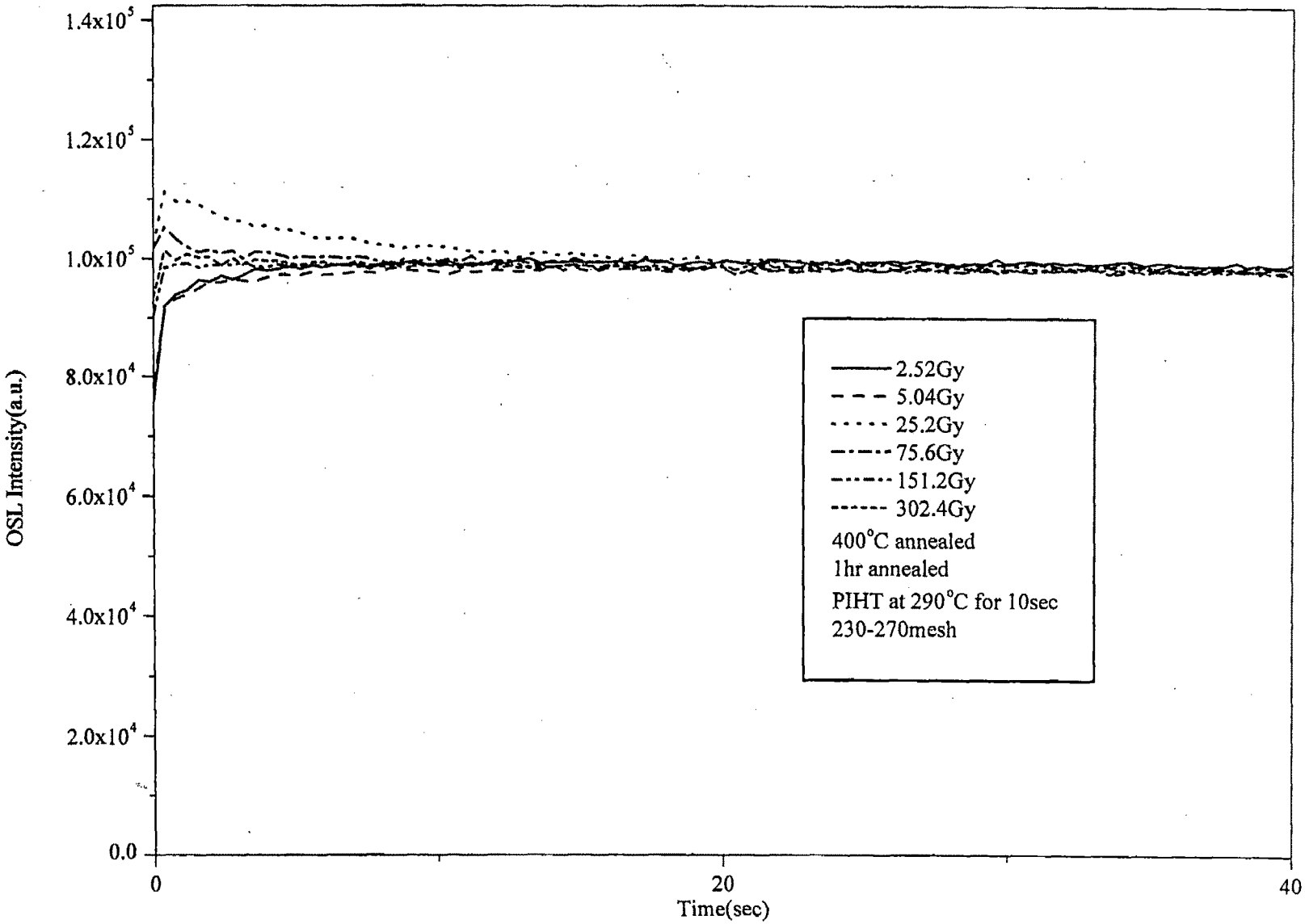
protocol may transfer the charges from shallow traps to the OSL traps, which will enhance the OSL efficiency.

In order to better understand, the effects of long term storage at ambient temperature, the present work also carried out systematically for this protocol, for different duration of post irradiation heat treatment i.e. for 0 sec to 30 sec. The effect of this protocol was examined by comparing with the OSL at elevated temperature but with out PIHT.

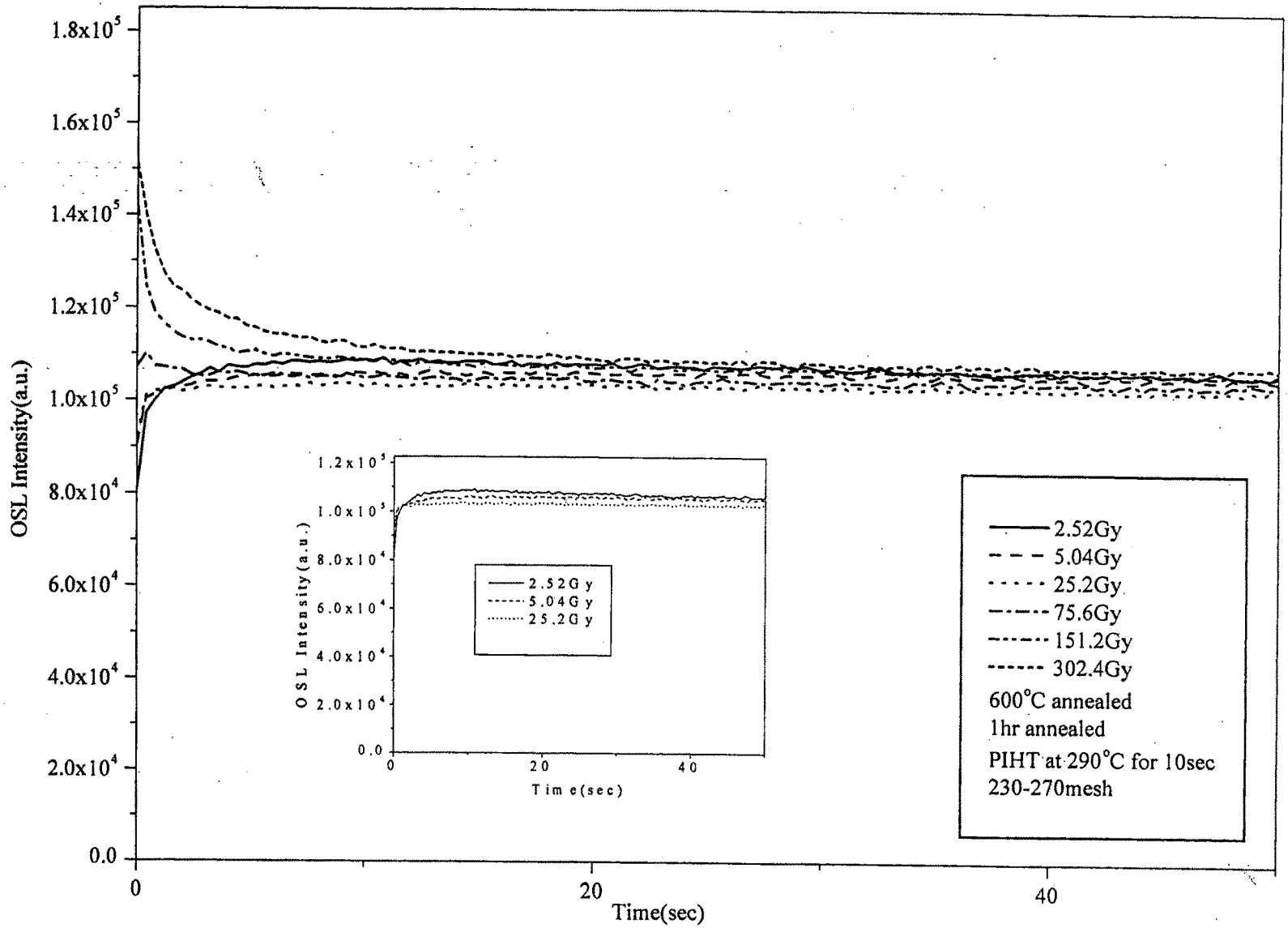
The specimens were annealed at different annealing temperatures such as 400°C, 600°C and 1000°C for 1hr duration. Each pre-heat treated specimen was given different beta doses of 2.52Gy to 302.4Gy followed by PIHT at 290°C for different durations of time namely 0 sec to 30 sec before recording their OSL at 160°C the results obtained are presented as **Graph-69** to **Graph-71.**

The specimens pre-heat treated at 400°C for 1hr duration exhibit non-exponential decay even with the rise in beta dose up to 302.4Gy, while, the specimens pre-heat treated at 600°C for 1 hour duration shows exponential decay after the critical dose of 25.2Gy. It is also clearly seen that the specimens annealed at 1000°C show completely exponential OSL decay. A comparison of OSL intensity for different annealing temperature and for different doses given to the specimen is presented as **Graph-72.** The results are similar to those of specimen without PIHT other physical conditions remaining the same. Further, the OSL studies were carried out to know the effect of rise in PIHT duration after identical dose of 25.2Gy.

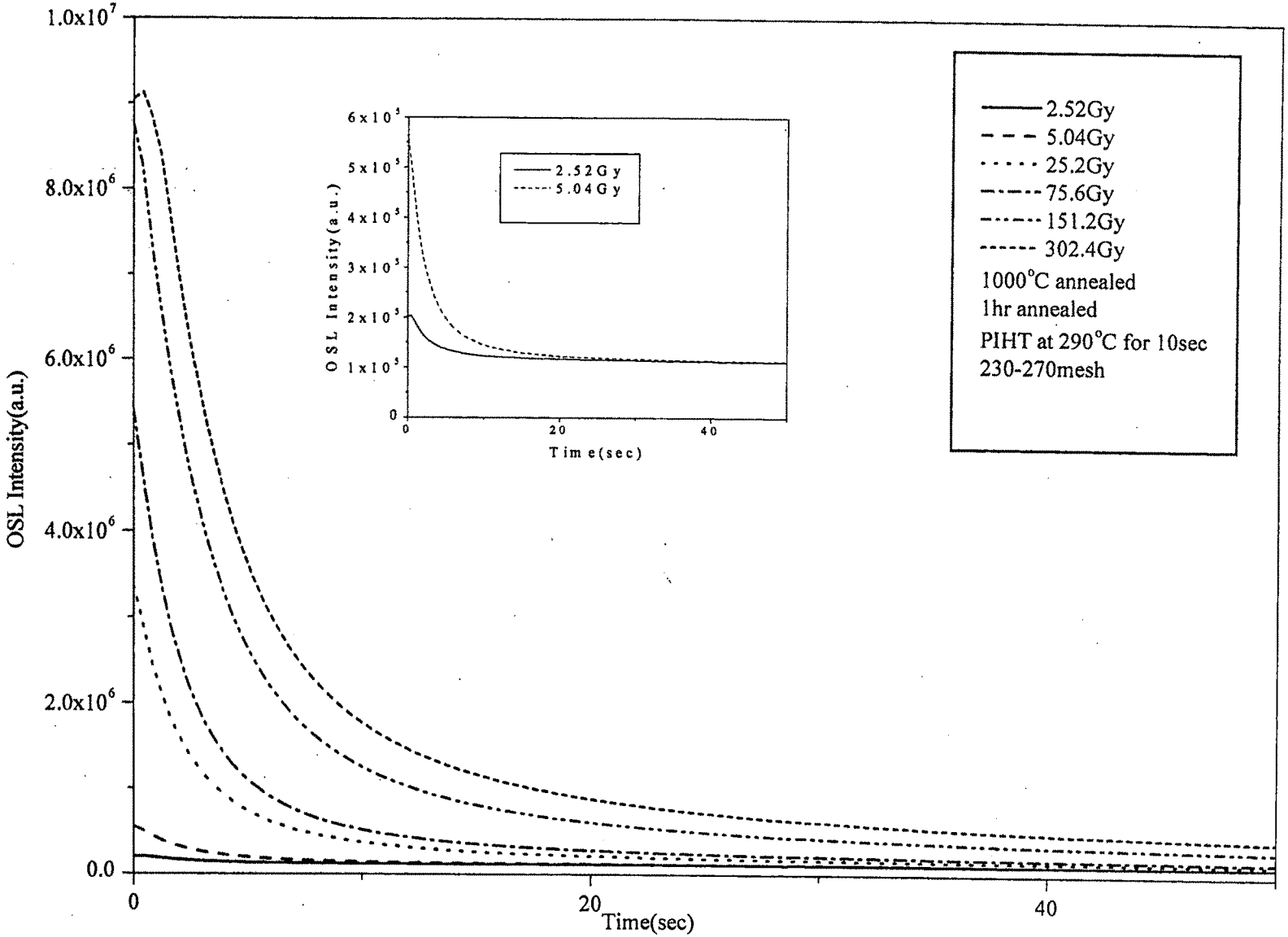
It is observed from **Graph-73** to **Graph-75** and their comparison with **Graph-76,** that, as the length of PIHT increases the OSL intensity increases systematically up to 10sec PIHT for the specimens annealed at 400°C for 1hr and thereafter, it reduces with rise in duration of PIHT. But, the specimens pre-heat treated at 600°C and 1000°C show such a reduction below 5sec of



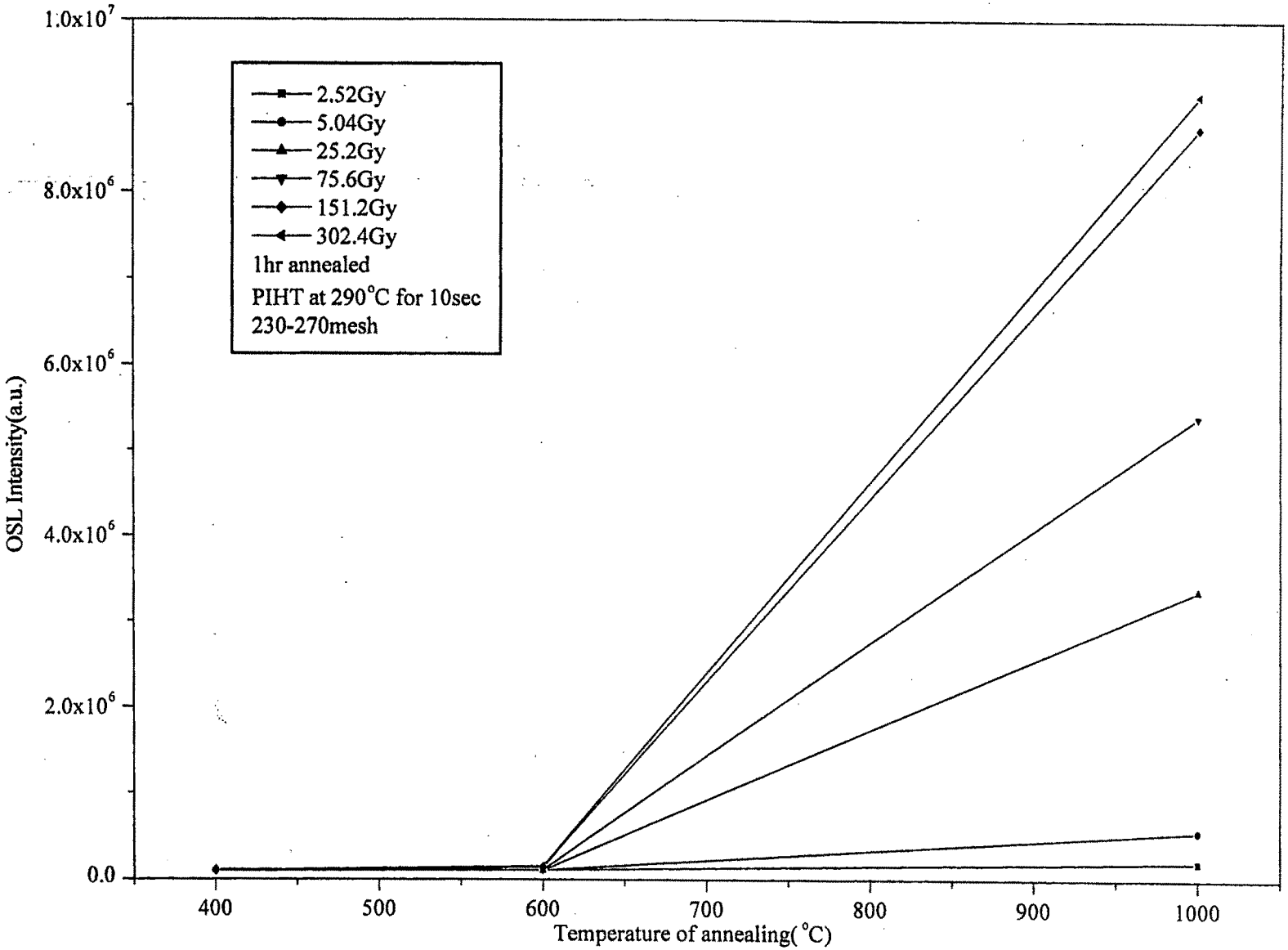
Graph-69 OSL measured at 160 °C(after PIHT for 10sec) of 400 °C annealed sample for 1hr duration at different doses.



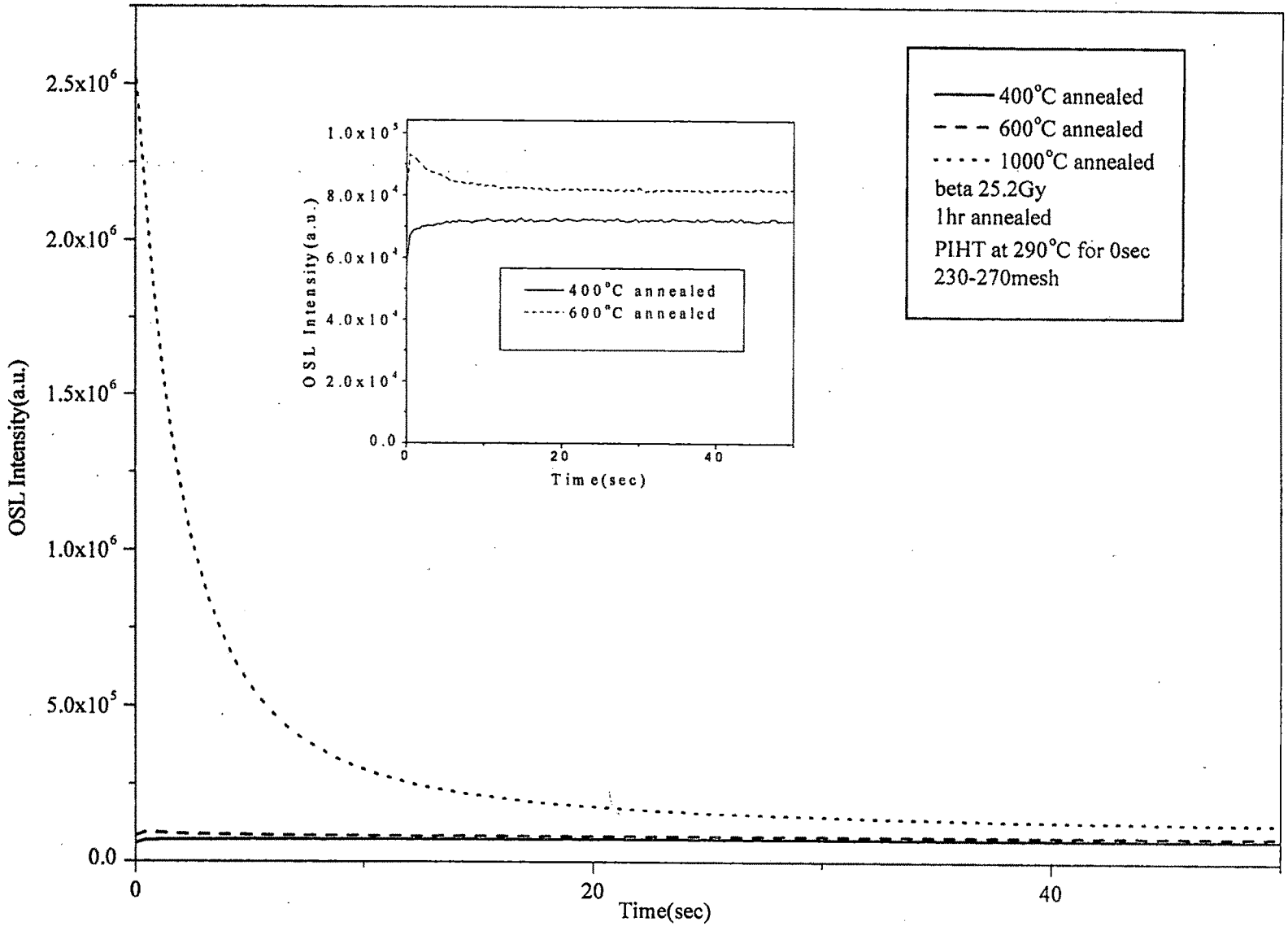
Graph-70 OSL measured at 160 °C(after PIHT for 10sec) of 600 °C annealed sample for 1hr duration at different doses.



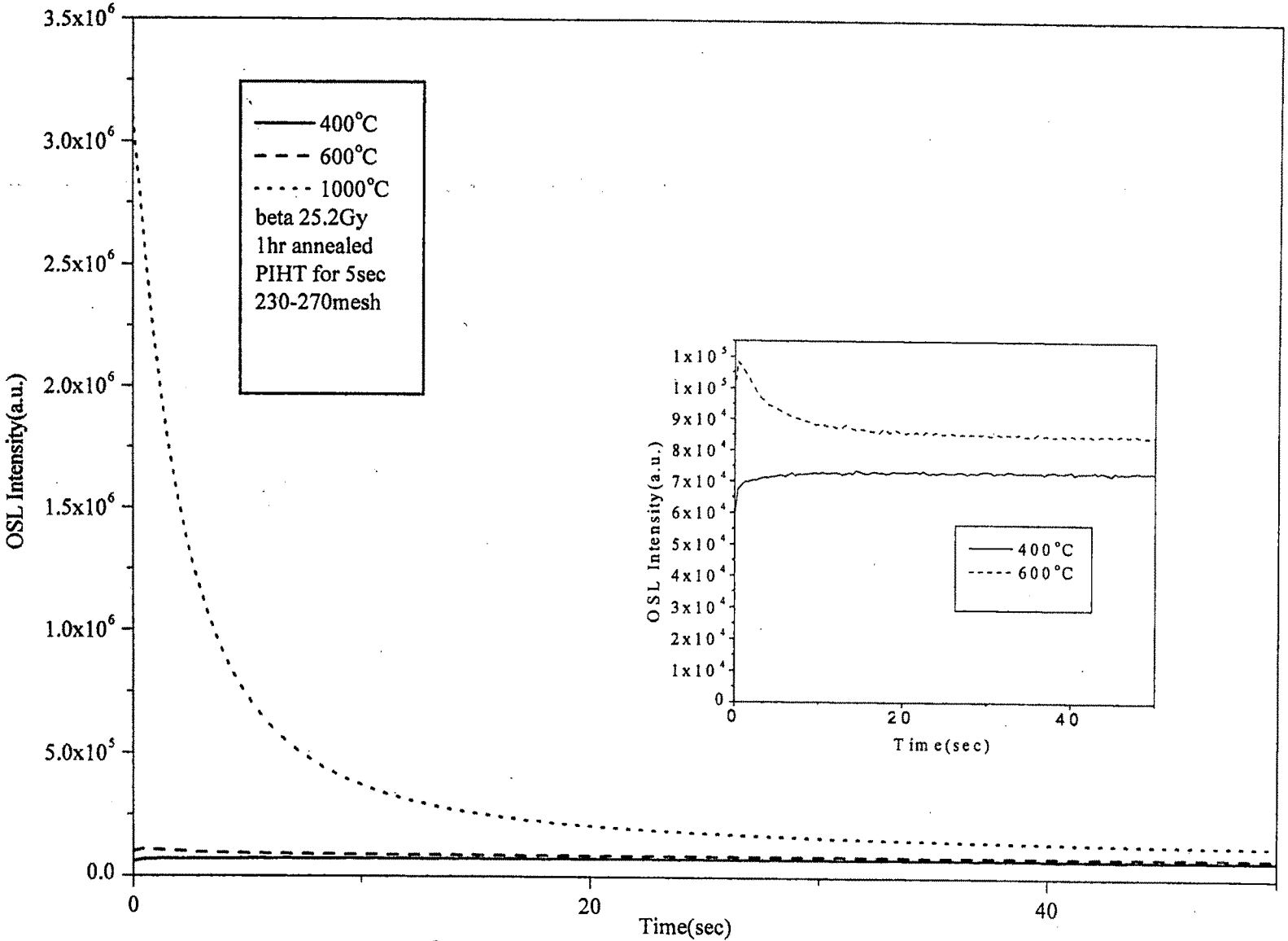
Graph-71 OSL measured at 160 °C(after PIHT for 10sec) of 1000 °C annealed sample for 1hr duration at different doses.



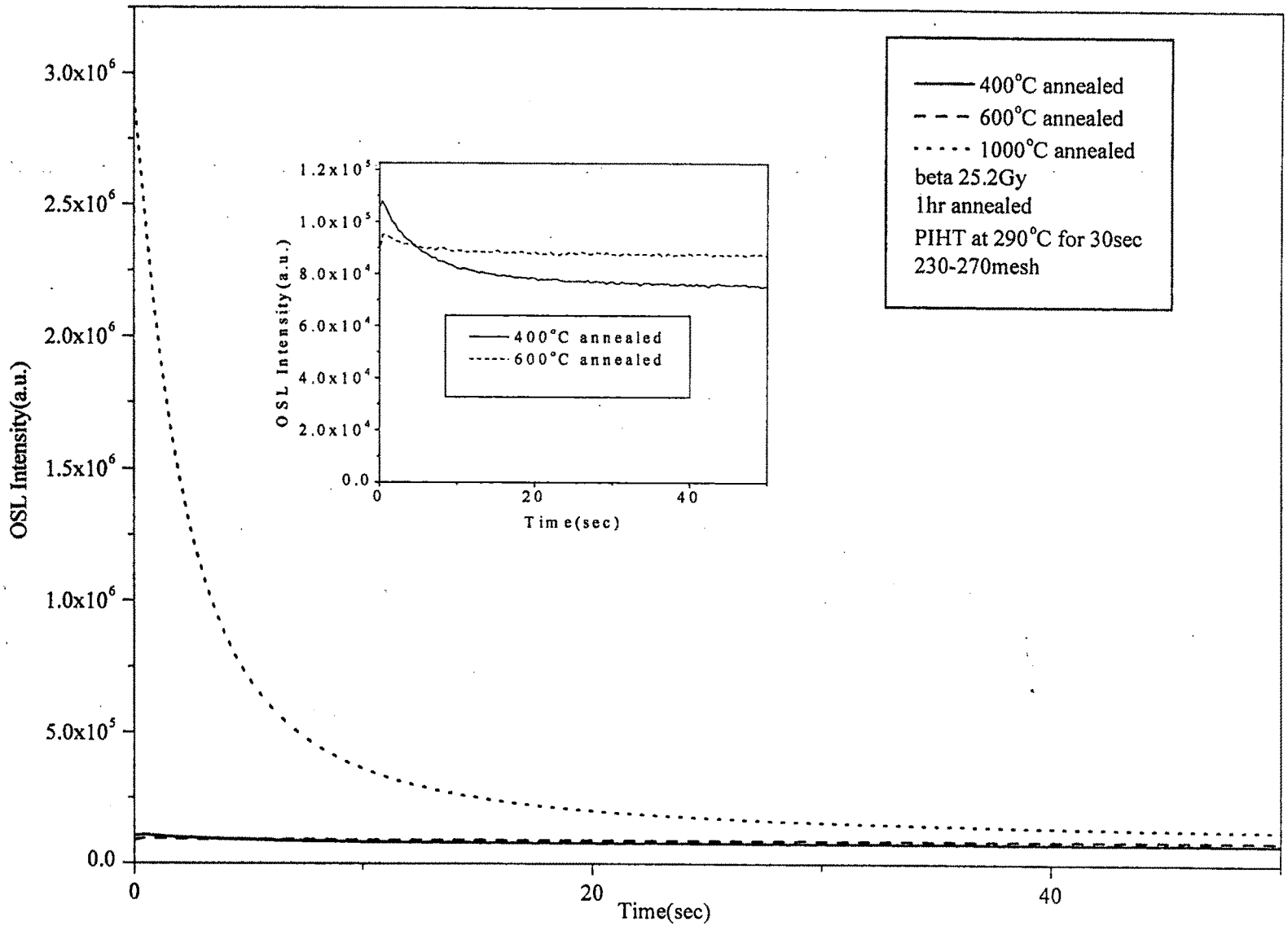
Graph-72 OSL sensitivity measured at 160 °C(after PIHT for 10sec) of 1hr annealed sample for 230-270mesh at different doses.



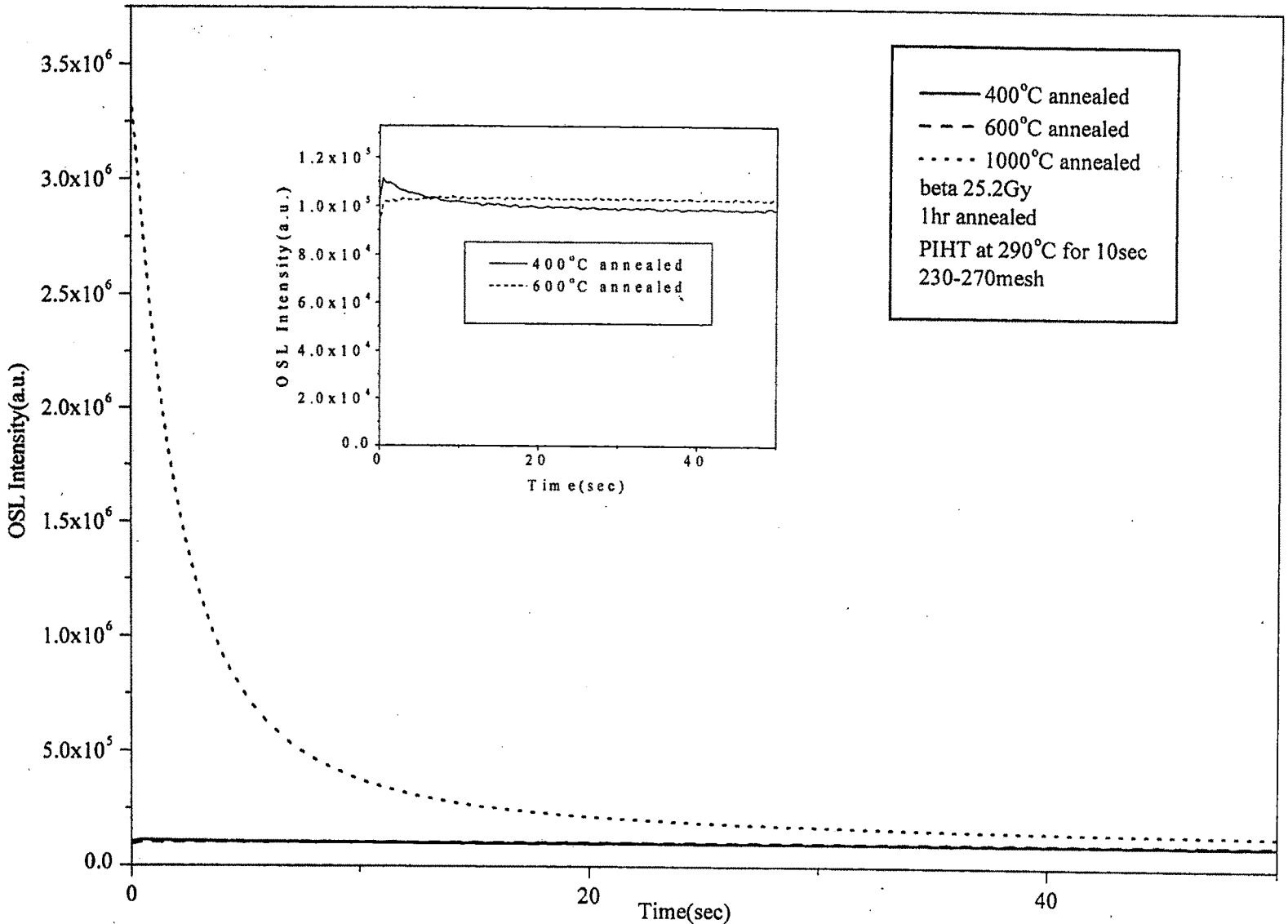
Graph-73 OSL measured at 160 °C(after PIHT for 0sec) of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures.



Graph-74 OSL measured at 160°C(after PIHT for 5sec) of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures.



Graph-75 OSL measured at 160 °C(after PIHT for 30sec) of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures.



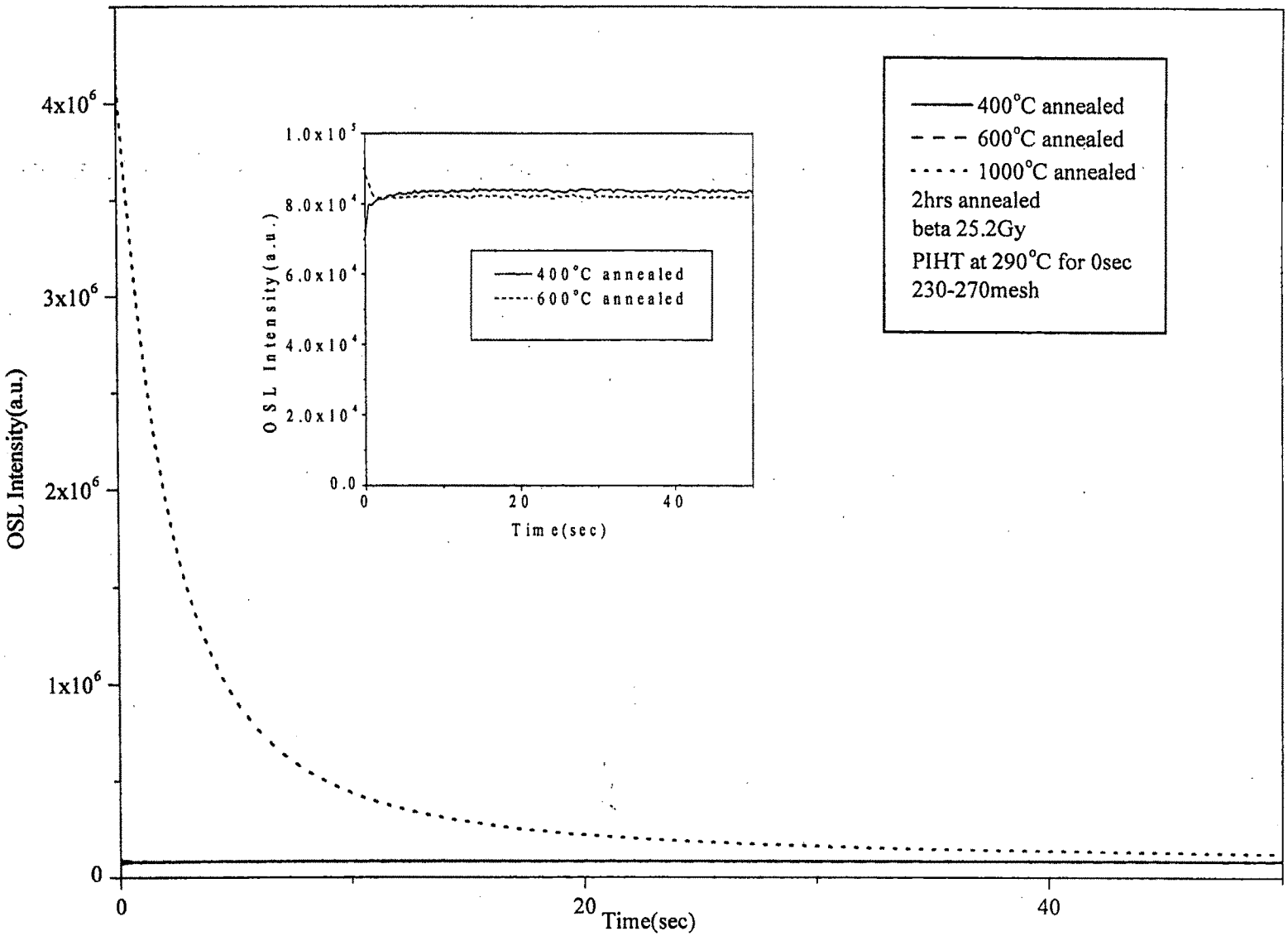
Graph-76 OSL measured at 160°C(after PIHT for 10sec) of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures.

PIHT. The studies were also carried out for 2hrs and 3hrs duration of pre-heat treatment (**Graph-77** to **Graph-80**). It is noticed that OSL intensity decreases with rise in PIHT duration up to 10sec thereafter, it enhances with rise in PIHT duration for 400°C and 600°C annealed material. Whereas, high temperature annealed material (1000°C) shows gradual enhancement in OSL intensity with rise in PIHT duration. The studies were also carried out for 3hrs of pre-heat treatment. The results are presented as **Graph-81** to **Graph-84**.

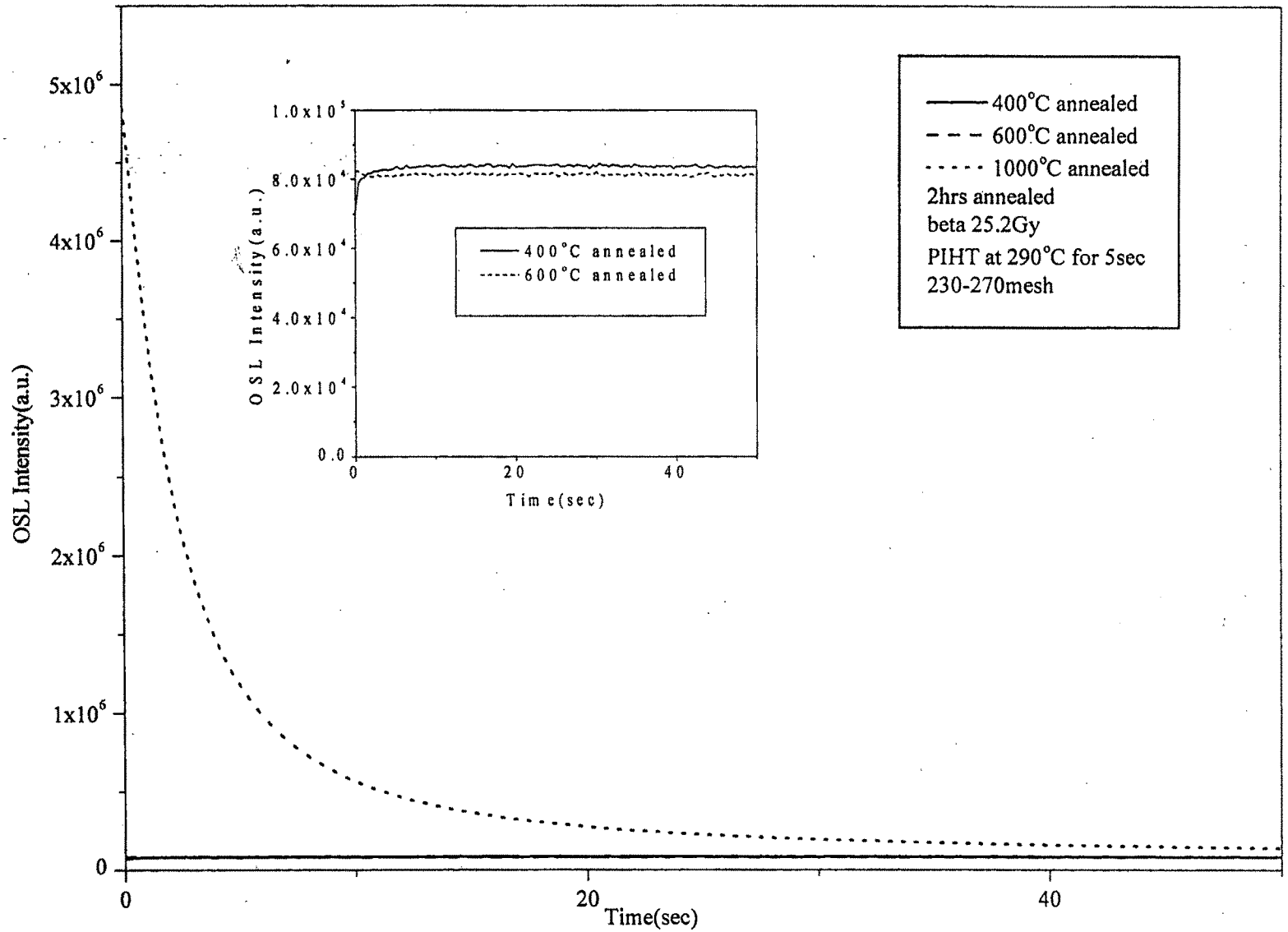
Results are also presented for different duration of pre-heat treatment with PIHT at 290°C for 0 sec, 5 sec, 10 sec and 30 sec. These results are presented in **Graph-85** to **Graph-88** respectively. It is found that the specimens pre-heat treated below 600°C for 1hr followed by 25.2Gy identical dose and given PIHT at 290°C for any duration shows insignificant change in OSL intensity, but it shows remarkable enhancement in OSL intensity for the specimens pre-heat treated beyond 600°C.

Thermoluminensce glow curves were also recorded after OSL measurements at 160°C for identical physical conditions such as beta dose, temperature of annealing and duration of pre-heat treatment for different durations of PIHT. To know the dose response, different doses were given during PIHT for 10 sec. TL glow curves were recorded after OSL measurement at 160°C. Before the beta exposures, the specimens were pre-heat treated at 400°C, 600°C& 1000°C for 1hr duration. Their results are presented in **Graph-89** to **Graph-91**.

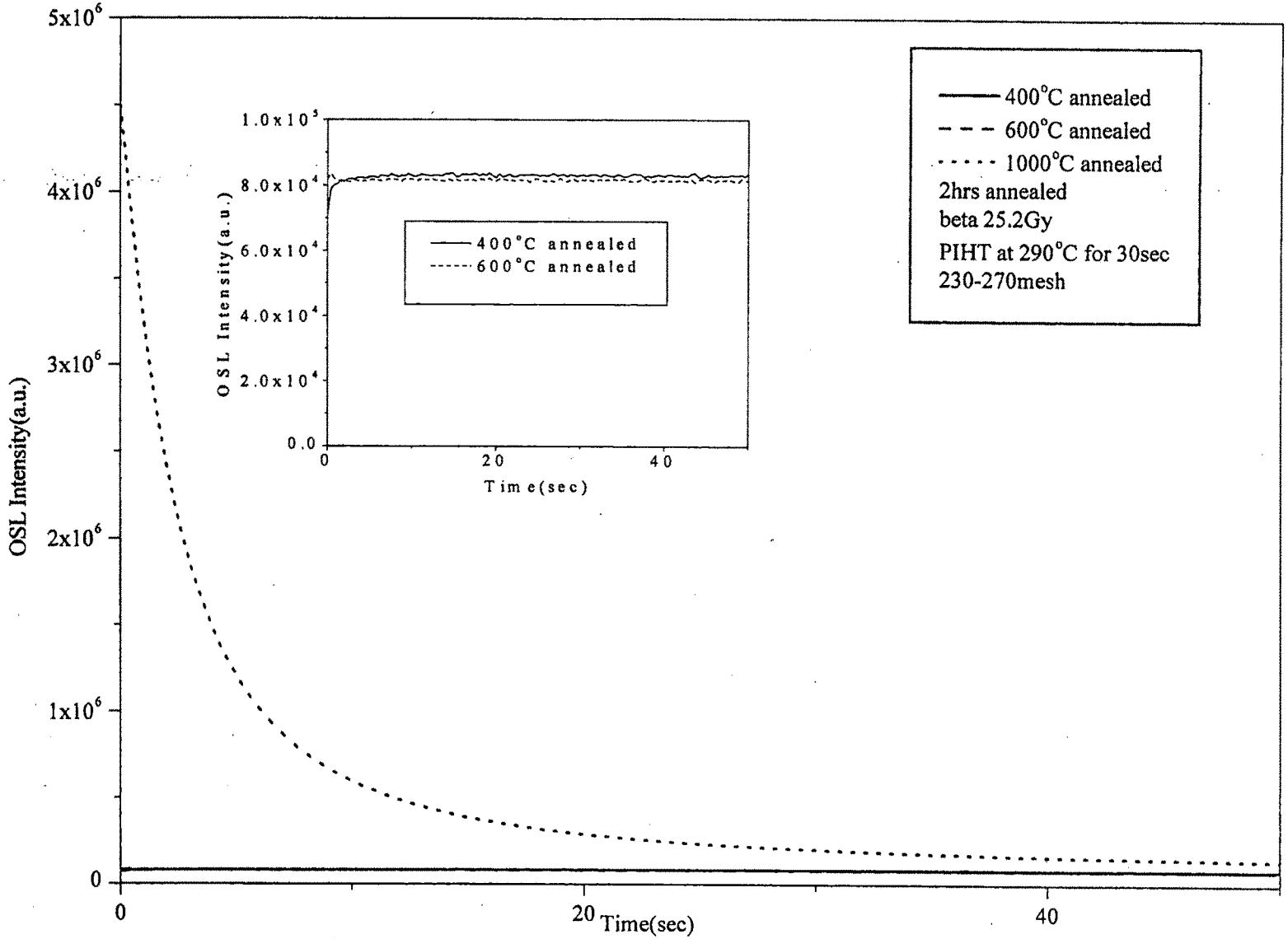
It is clear from the data that the specimens pre-heat treated for 400°C and 600°C show only a well-defined peak at 375°C, which grows linearly with the dose. However, the specimens pre-heat treated at 1000°C exhibit two distinct glow peaks at 220°C and 375°C. Their dose response curve



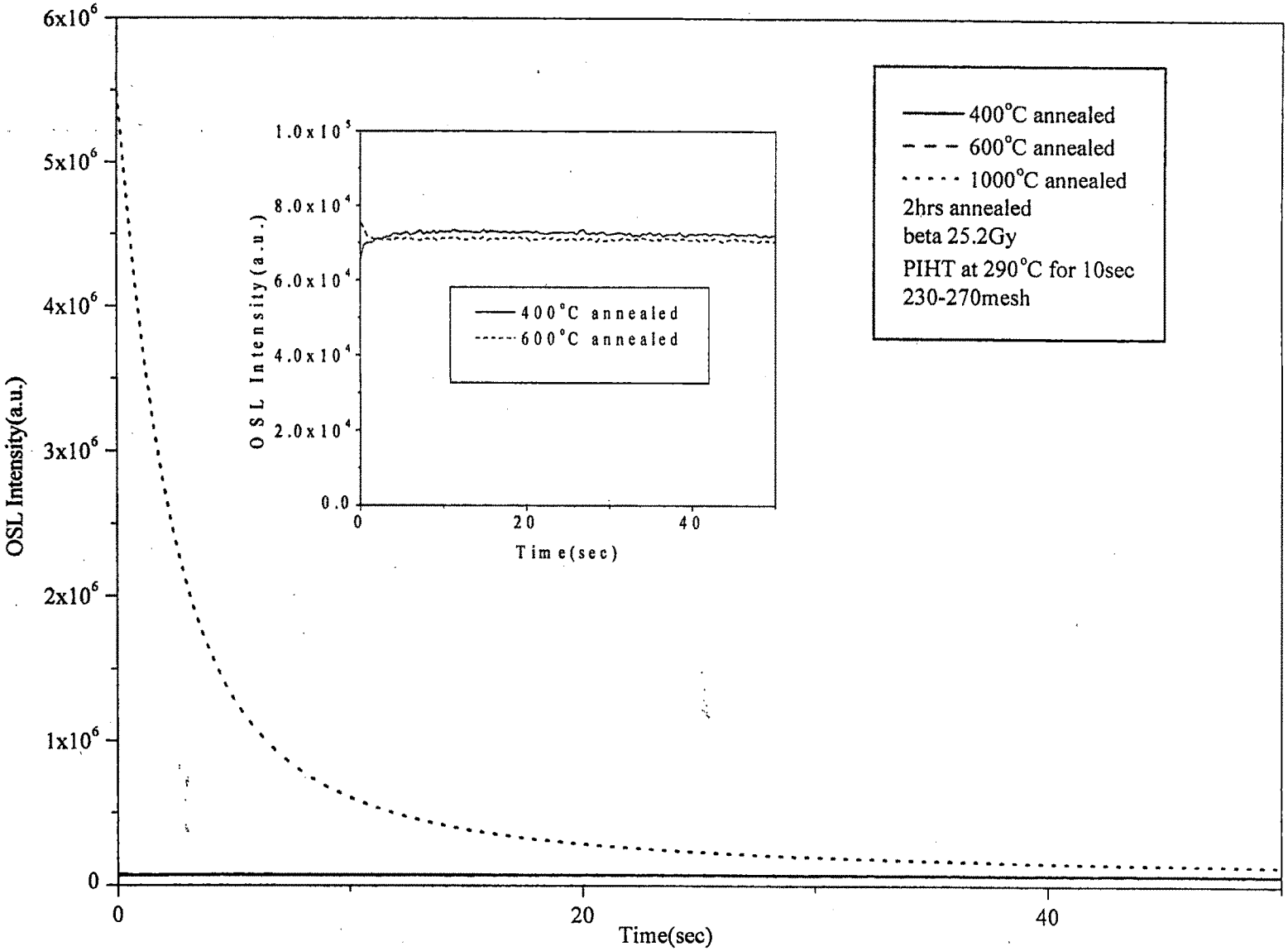
Graph-77 OSL measured at 160°C(after PIHT for 0sec) of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



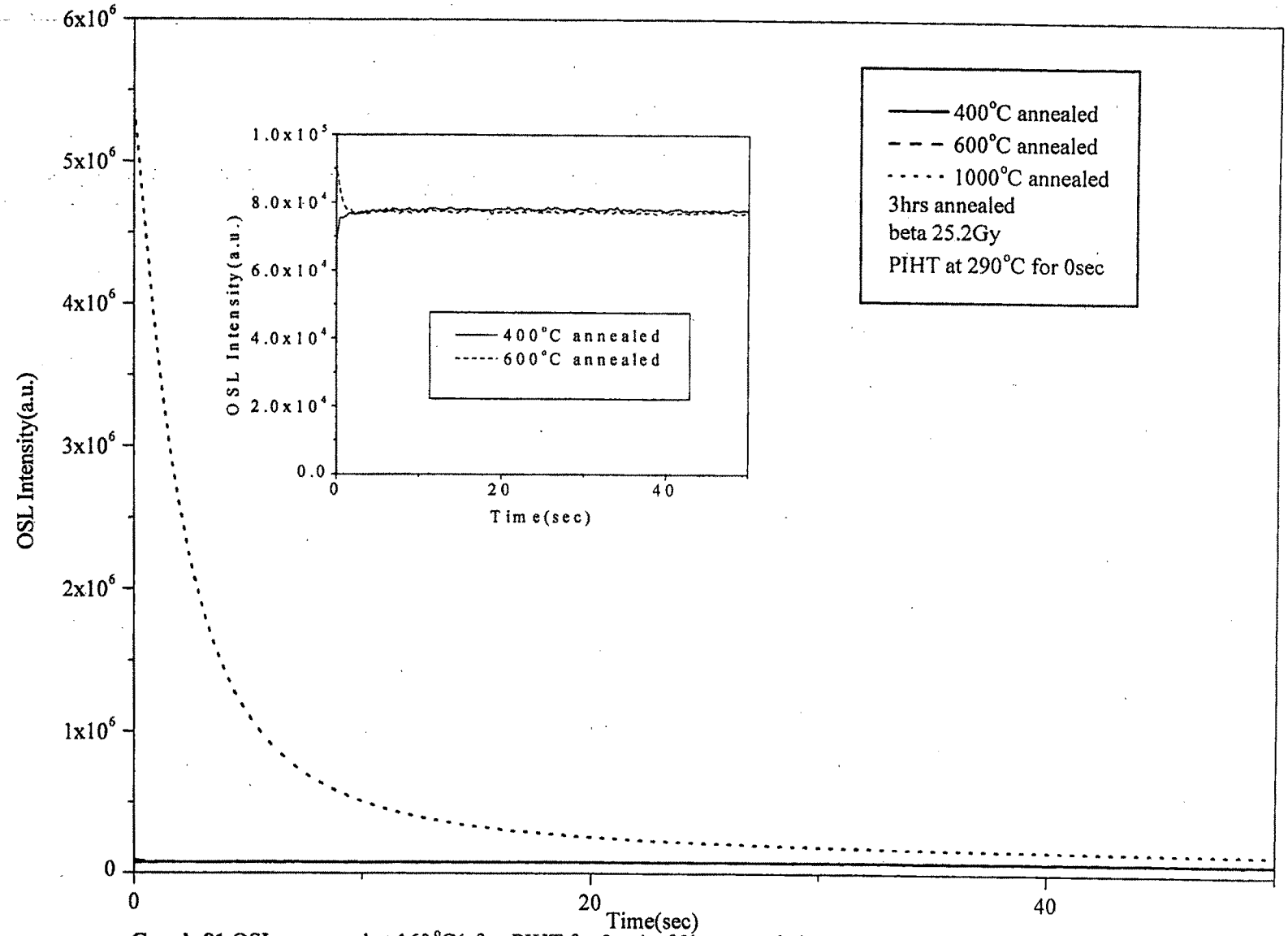
Graph-78 OSL measured at 160°C(after PIHT for 5sec) of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



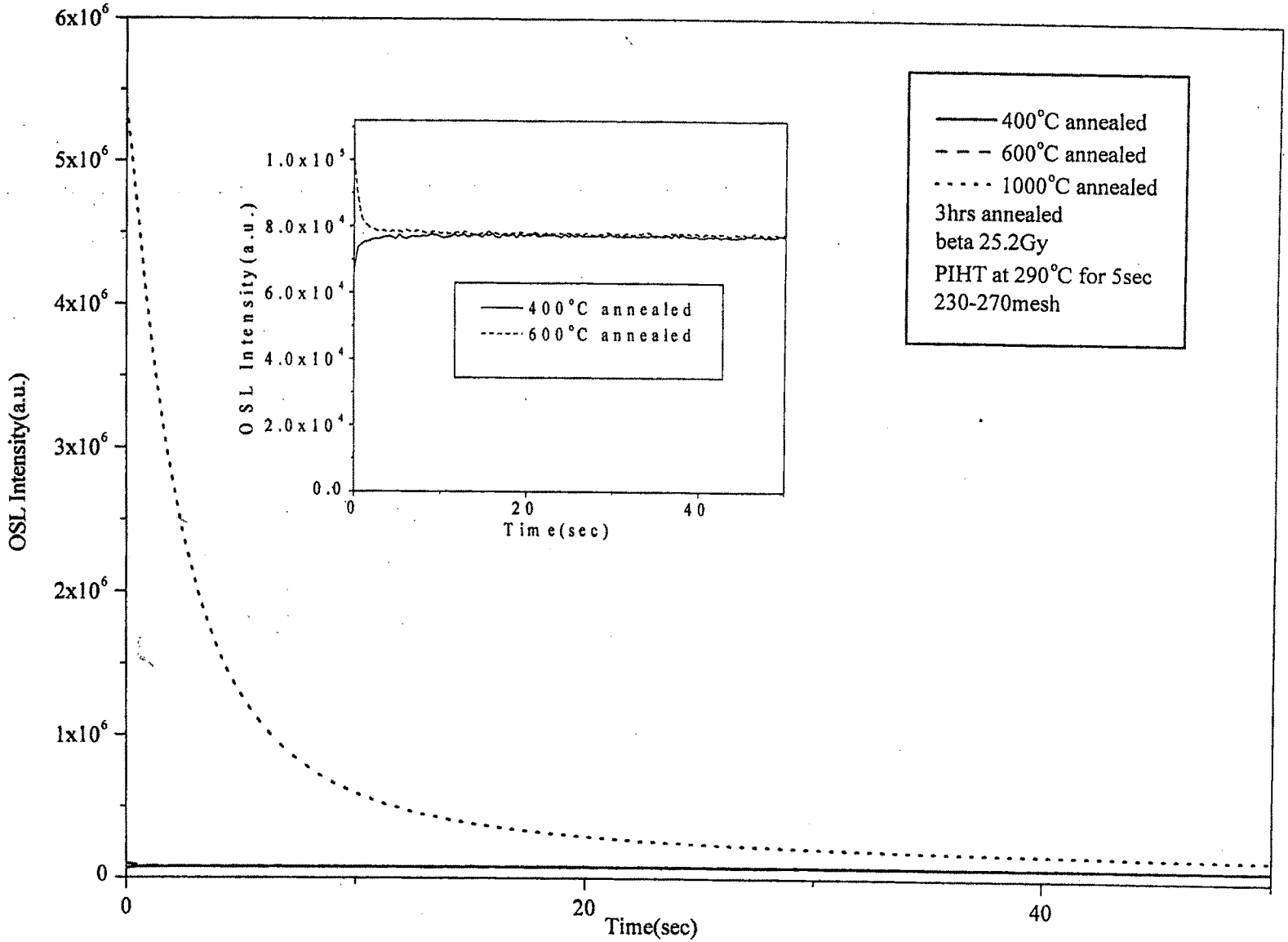
Graph-79 OSL measured at 160°C(after PIHT for 30sec) of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



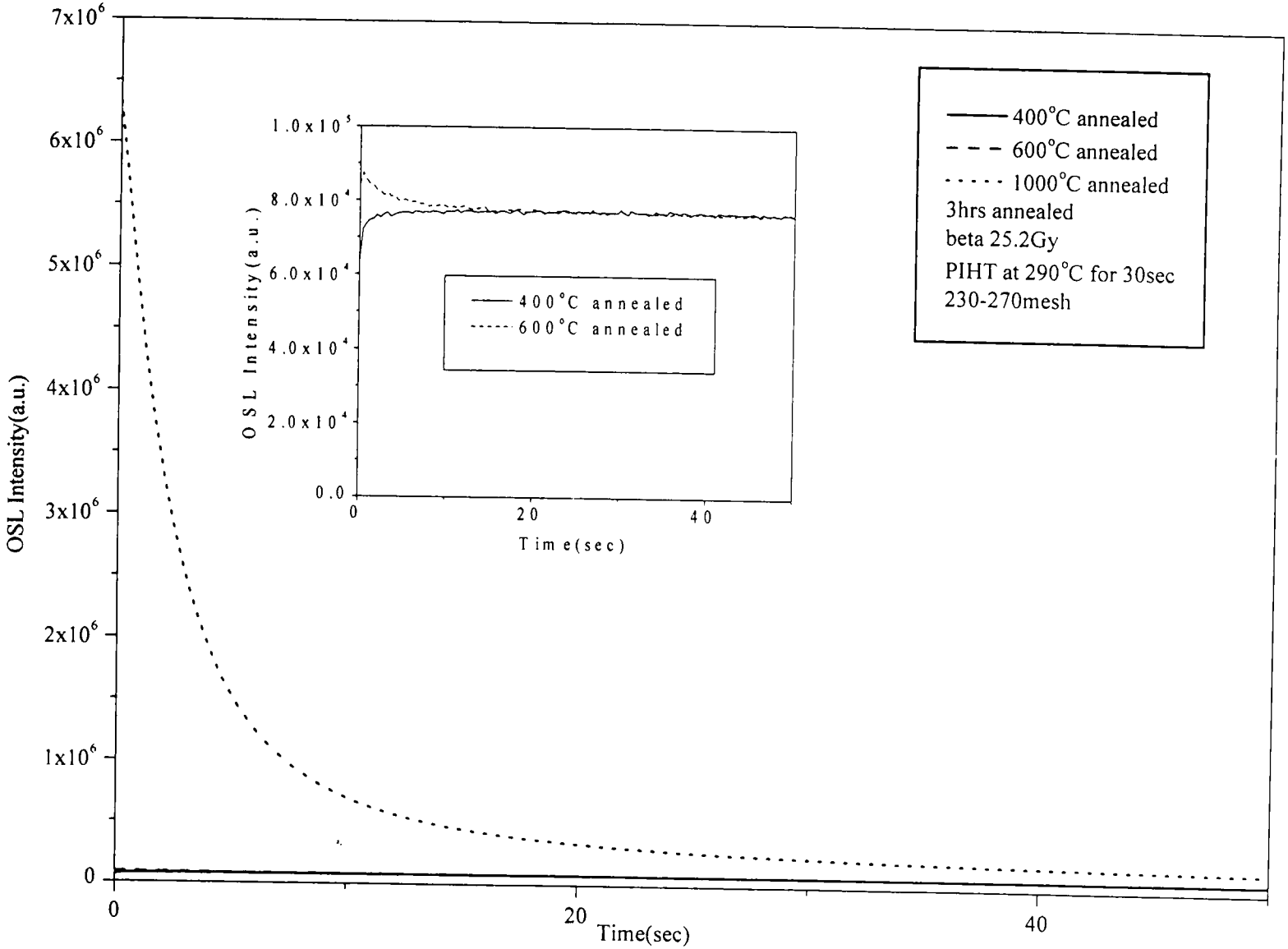
Graph-80 OSL measured at 160°C(after PIHT for 10sec) of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



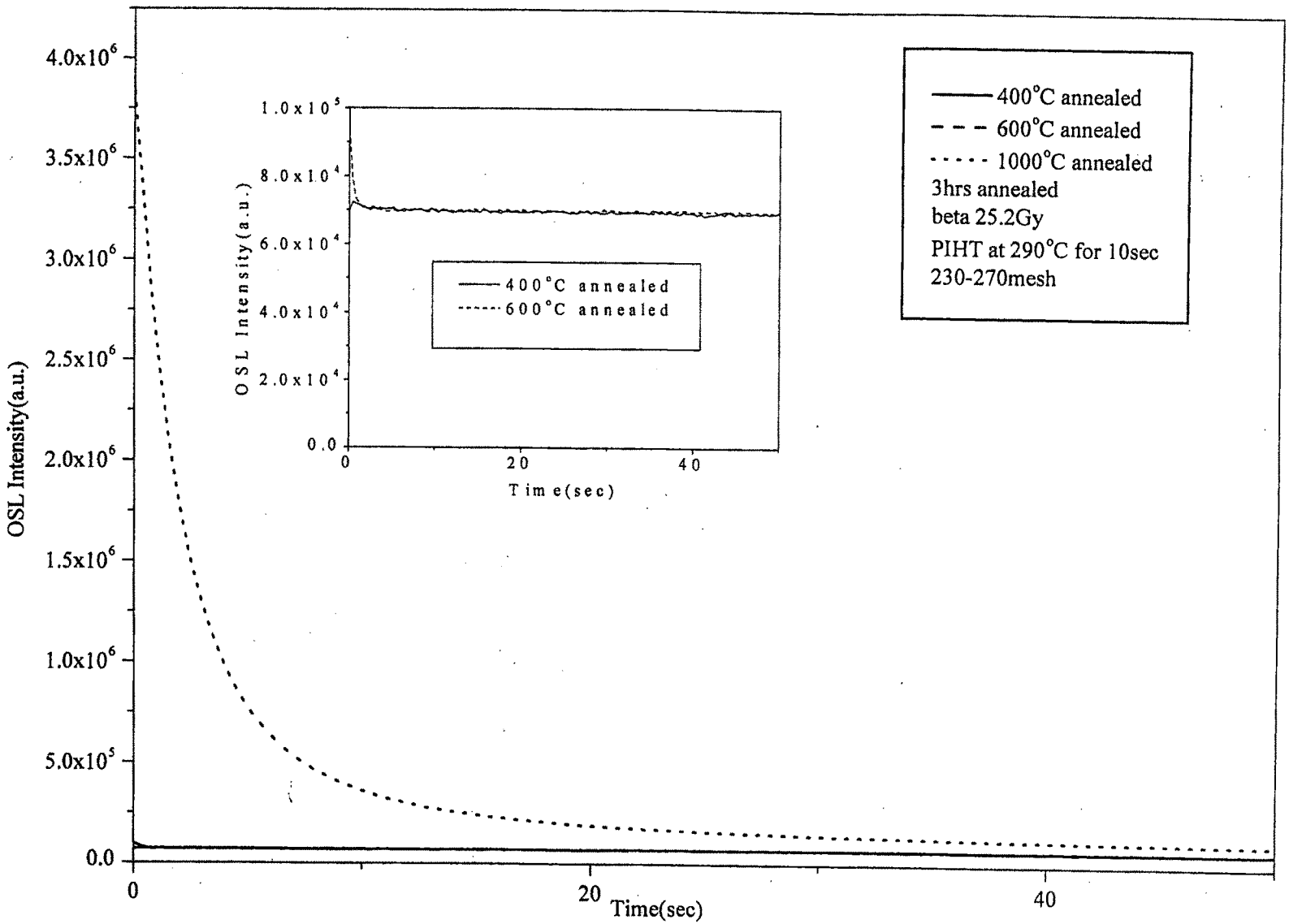
Graph-81 OSL measured at 160 °C(after PIHT for 0sec) of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



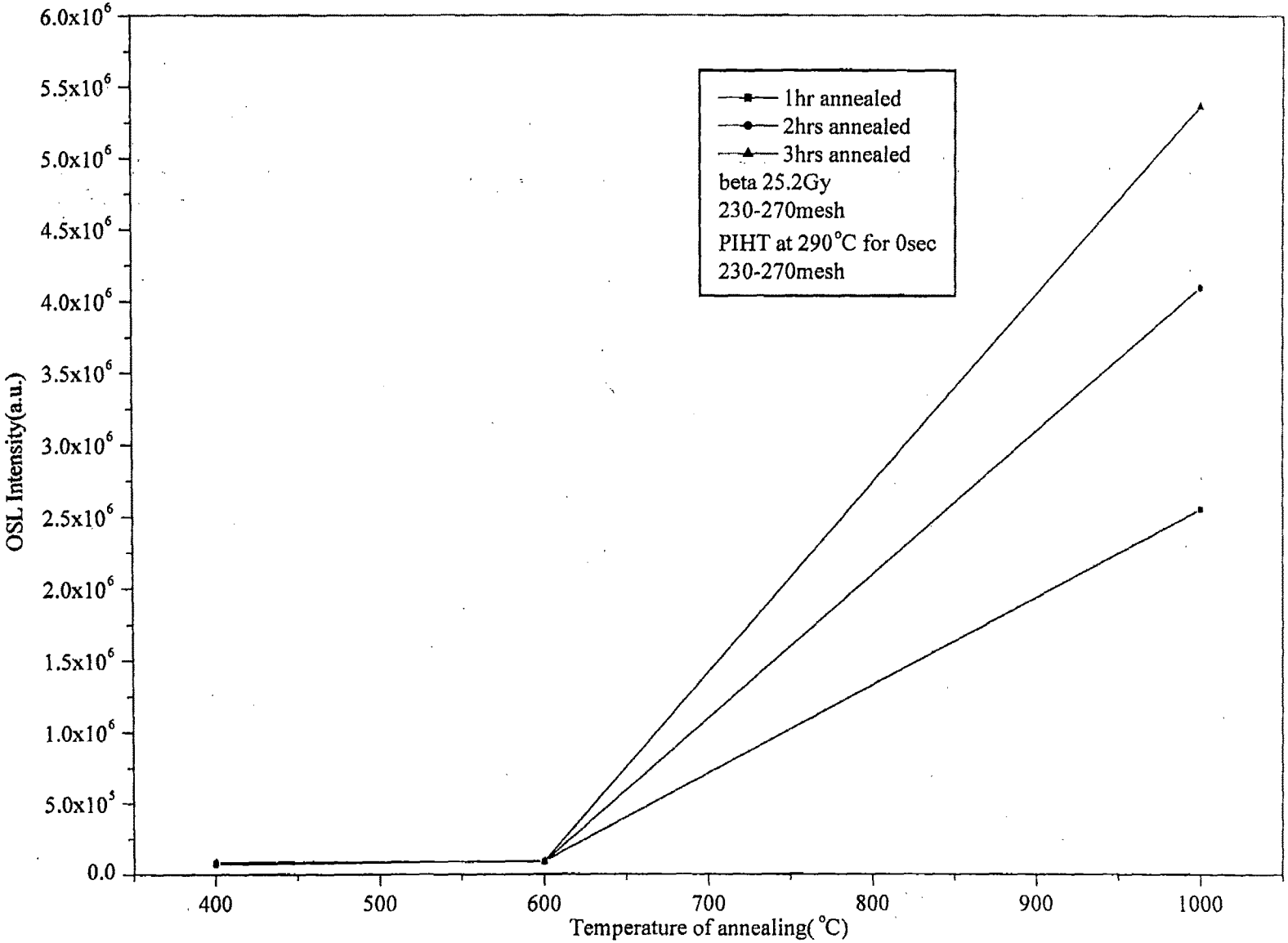
Graph-82 OSL measured at 160°C(after PIHT for 5sec) of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



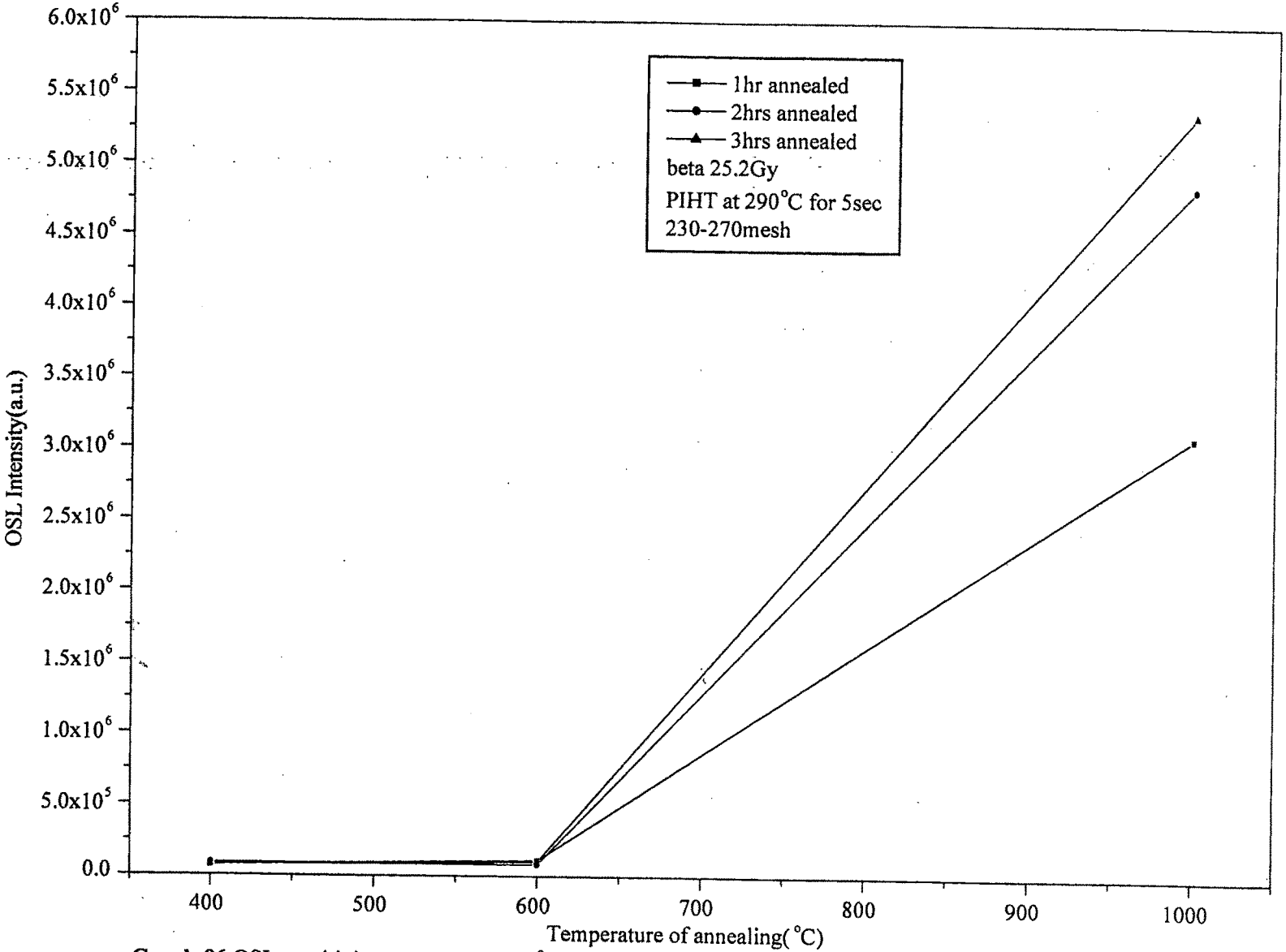
Graph-83 OSL measured at 160°C(after PIHT for 30sec) of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



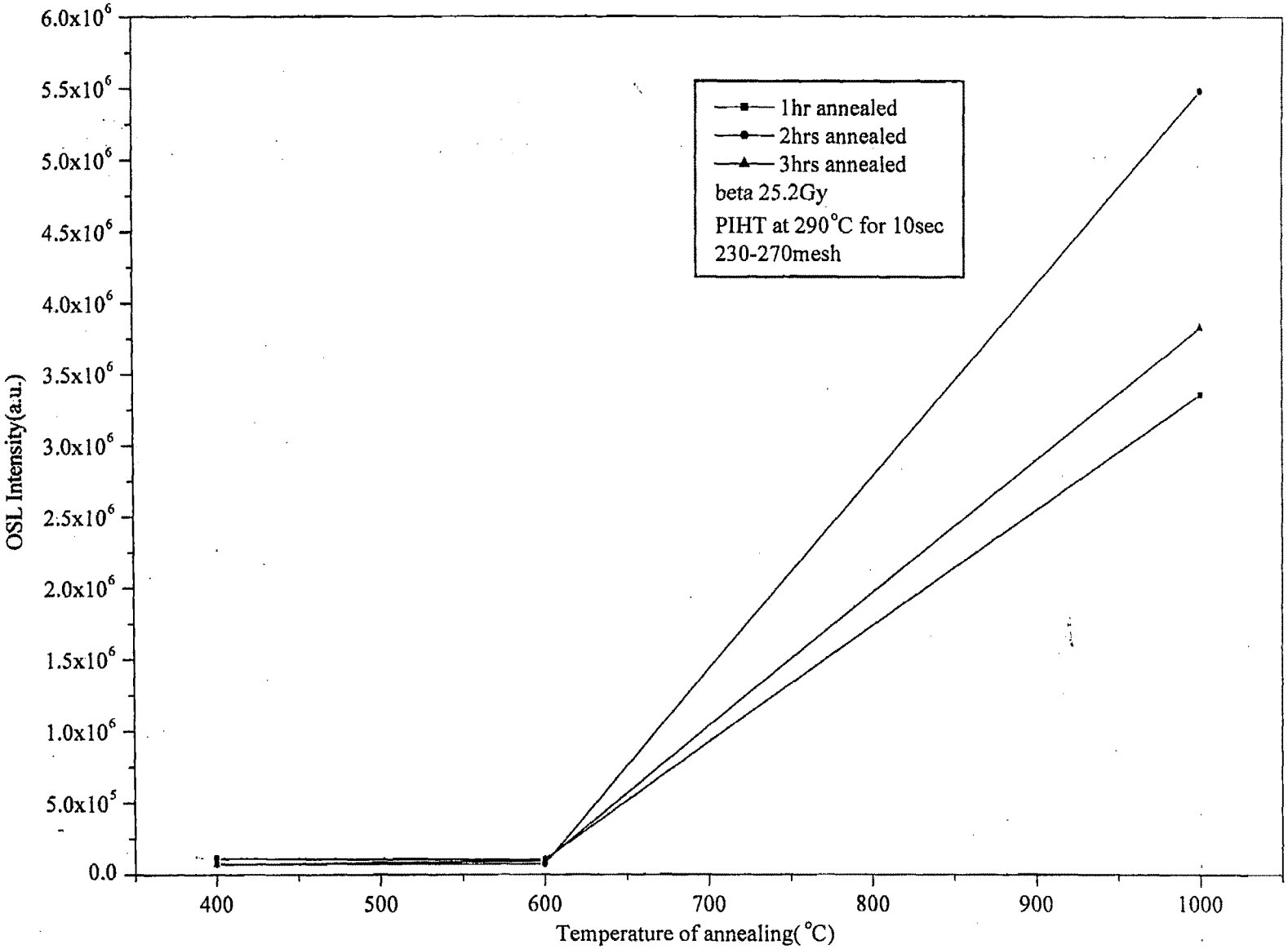
Graph-84 OSL measured at 160 °C(after PIHT for 10sec) of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures.



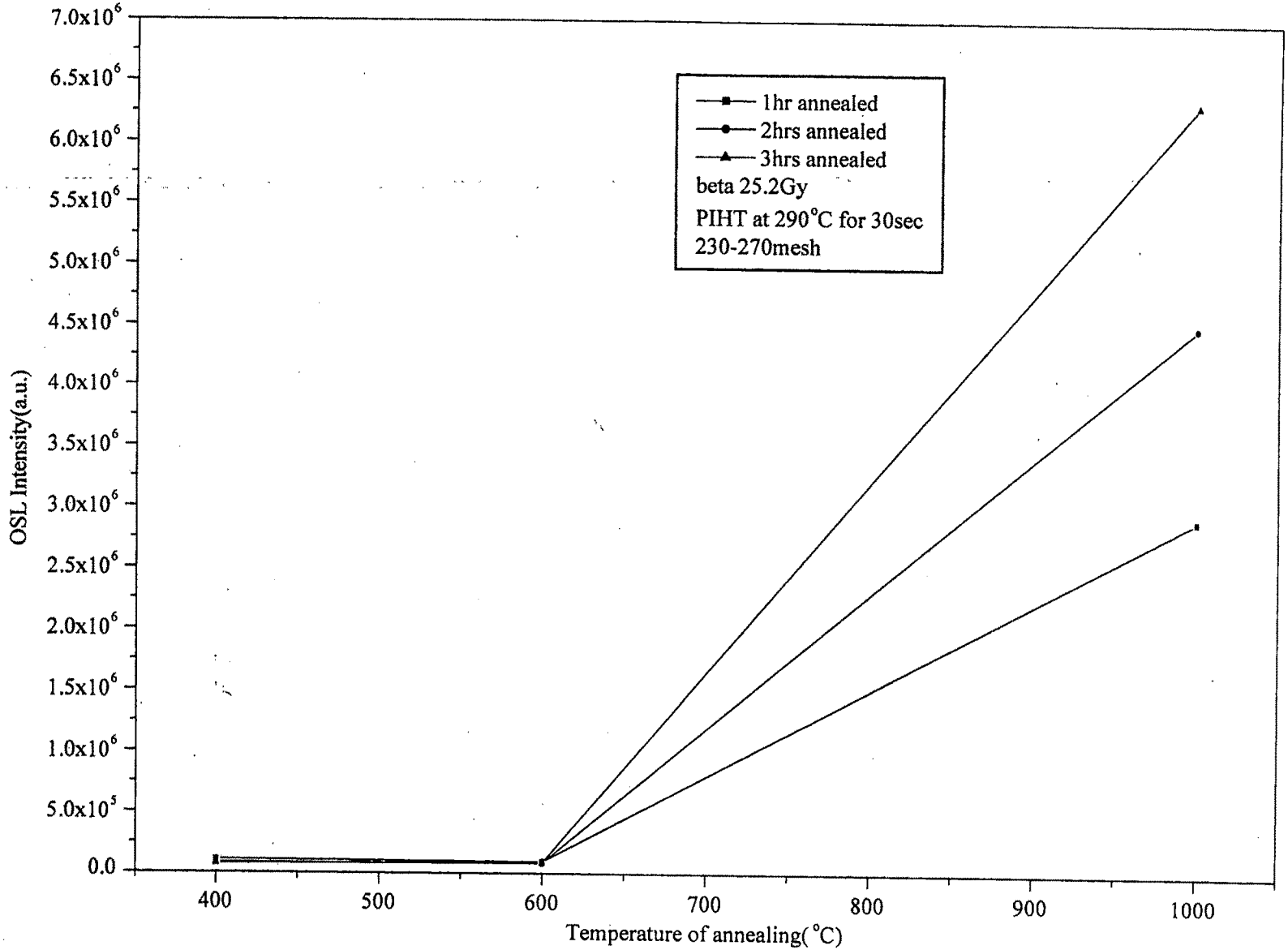
Graph-85 OSL sensitivity measured at 160 °C(after PIHT for 0sec) of 230-270mesh for a given dose of 25.2Gy at different annealing durations.



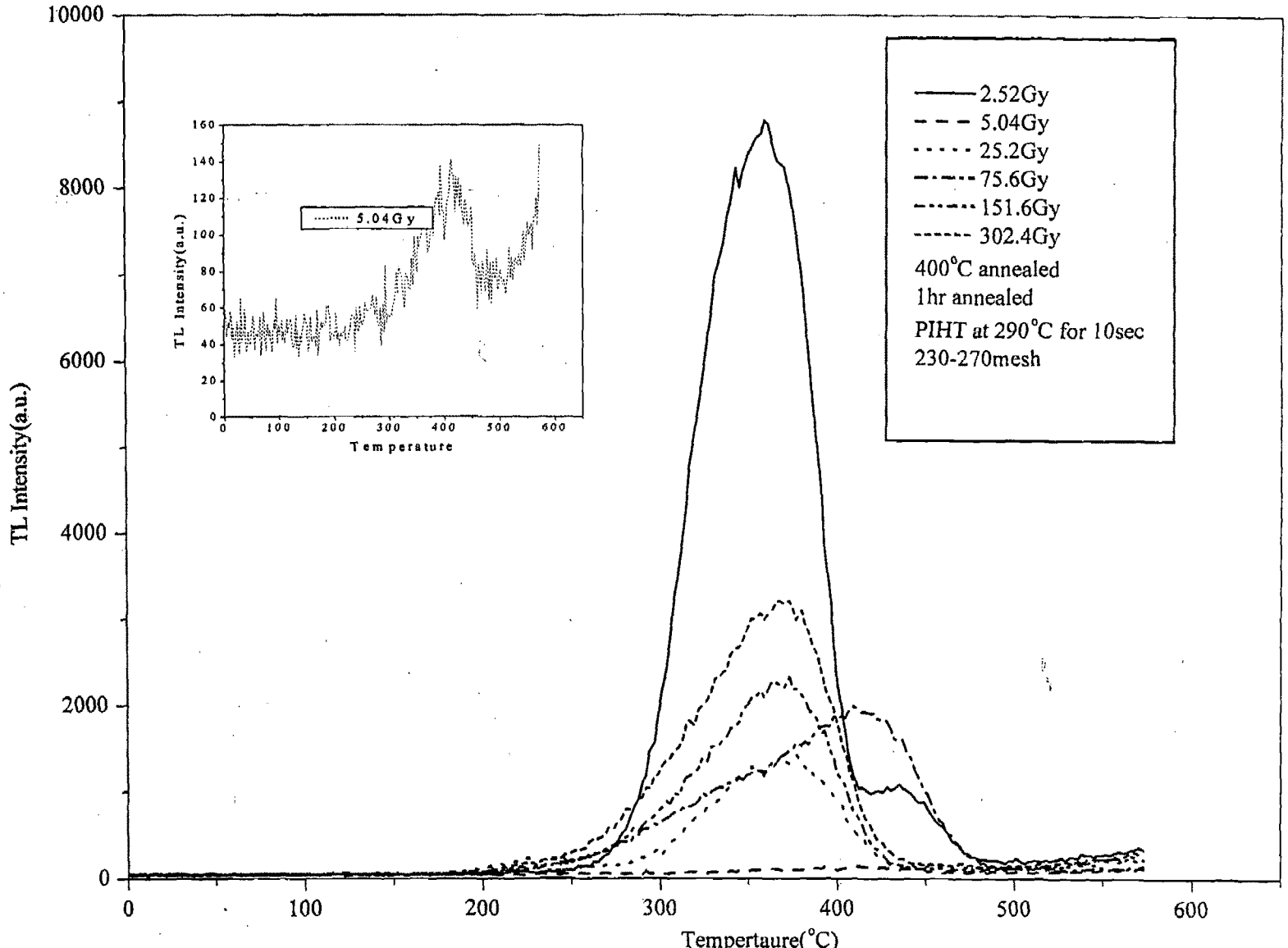
Graph-86 OSL sensitivity measured at 160 °C(after PIHT for 5sec) of 230-270mesh for a given dose of 25.2Gy at different annealing durations.



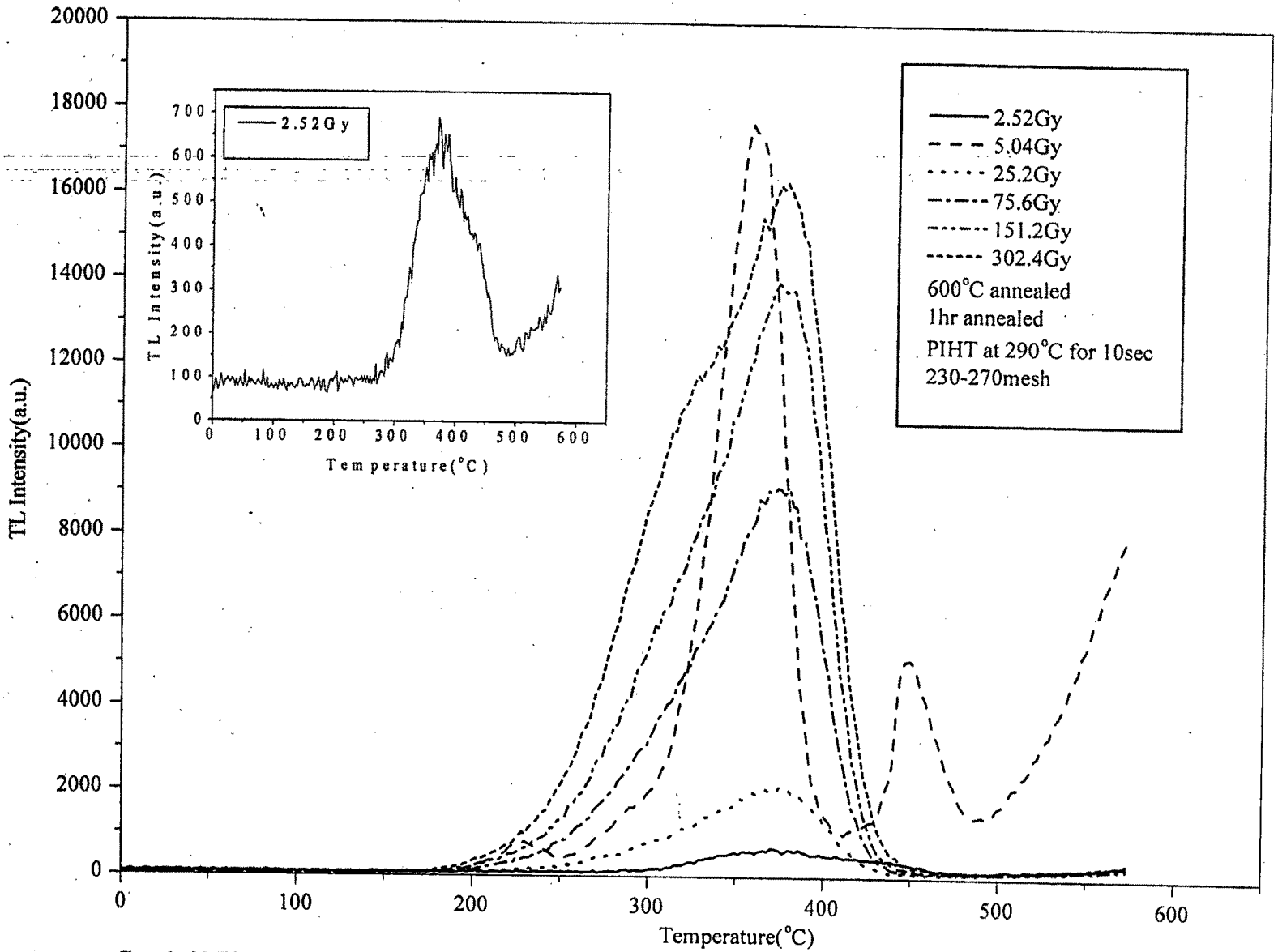
Graph-87 OSL sensitivity measured at 160 °C(after PIHT for 10sec) of 230-270mesh for a given dose of 25.2Gy at different annealing durations.



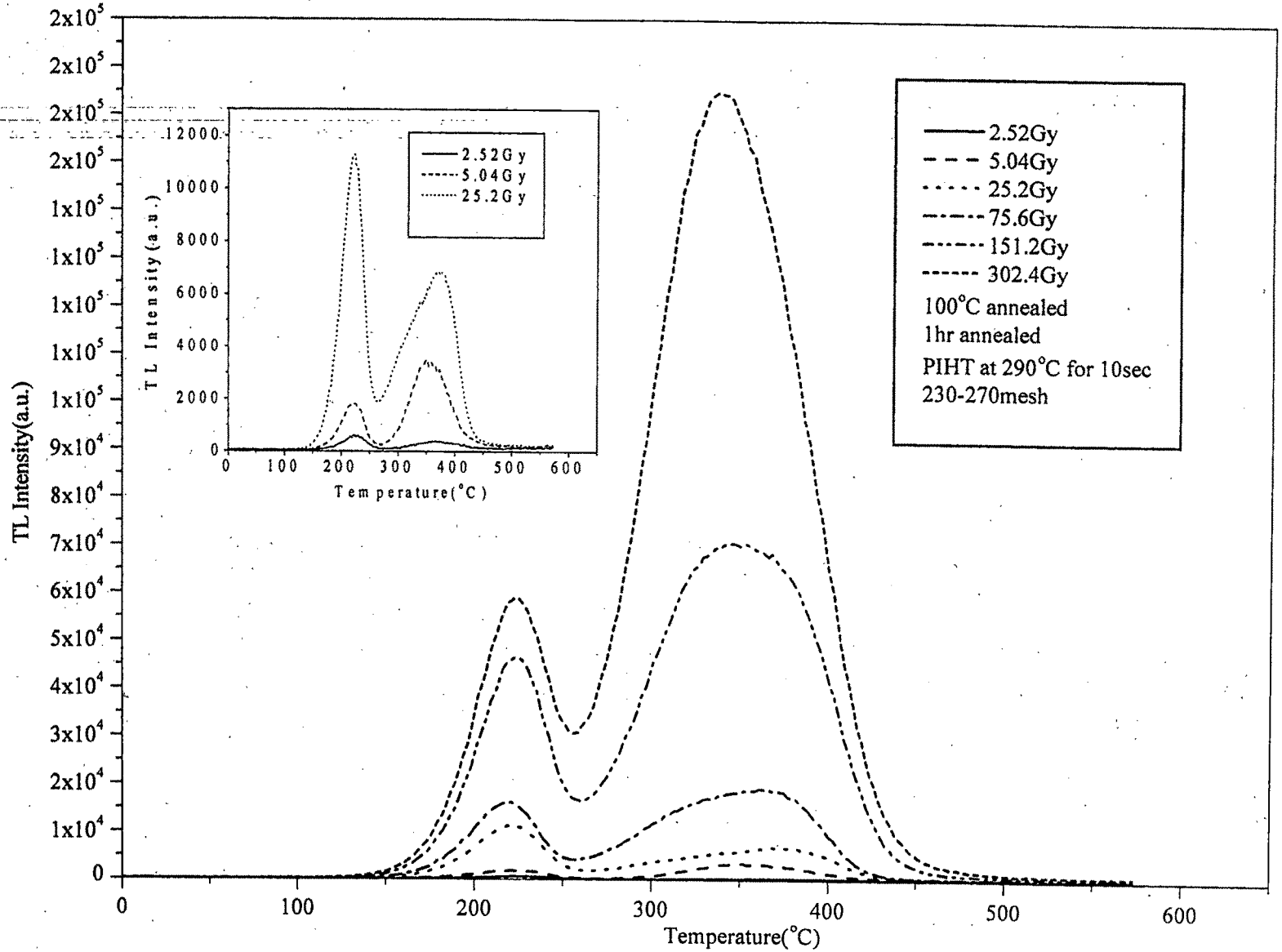
Graph-88 OSL sensitivity measured at 160 °C(after PIHT for 30sec) of 230-270mesh for a given dose of 25.2Gy at different annealing durations.



Graph-89 TL measured at room temperature* of 400 °C annealed sample for 1hr annealing duration at different doses for 230-270mesh. *(After the OSL at 160 °C(after PIHT for 10sec))



Graph-90 TL measured at room temperature* of 600 °C annealed sample for 1hr annealing duration at different doses for 230-270mesh. *(After the OSL at 160 °C(after PIHT for 10sec))



Graph-91 TL measured at room temperature* of 1000 °C annealed sample for 1hr annealing duration at different doses for 230-270mesh. *(After the OSL at 160 °C(after PIHT for 10sec))

(DRC) is also linear, but with a remarkable difference namely, that the growth of 375°C peak is much higher than 230°C TL peak.

Table-4C shows the comparison between different periods of PIHT with different temperature of annealing for an identical beta dose of 25.2Gy. The results were compared also for higher duration of pre-heat treatment. It was found that as the temperature of annealing increases TL sensitivity for the 375°C peak increases. This behaviour is observable for any duration of PIHT. Similar results are observed for 2hrs and 3hrs of annealing durations. It may be noted that a glow peak was induced around 230°C, when the specimens were annealed at 600°C for 1hr. This peak was absent for the specimen pre-heated at 600°C for 2hrs and 3hrs.

From the TL studies it is clear that 375°C TL glow peak grows with the PIHT duration for any pre-heat treatment. Also it is to be noted that the OSL sensitivity changes accordingly. In finding correlation between them it becomes clear that PIHT influences the phenomenon of charge transfer in two ways (i) electrons transferred from easily bleachable shallow traps to optically active OSL trap which in turn improves OSL efficiency and (ii) transfer of electrons from shallow traps to deep traps at 375°C[35-36]. However, the specimens either pre-heat treated at below critical temperature of annealing or below critical dose, influence very weakly on OSL decay. When PIHT followed for any duration. It is clear from the result of OSL decay, that it is weakly sensitive and non- exponential. Further, it is observed that when subjected to conditions higher than critical the specimens exposed to PIHT of different periods display exponential decay with changes in sensitivity. This observation reveals that PIHT influences the charge transfer as mentioned earlier. To know the exact responsible process observations were systematically examined. It was found that before following PIHT and after given PIHT at a constant time of 10 second show almost similar results

Table-4C: A comparison between different times of PIHT with different pre-thermal treatment for an identical dose.

Pre-heat Duration	Beta Dose	PIHT Time	400°C				600°C				1000°C			
Hrs	Gy	Sec	Tm_2 °C	TL_2 Intensity (a.u.)	Tm_3 °C	TL_3 Intensity (a.u.)	Tm_2 °C	TL_2 Intensity (a.u.)	Tm_3 °C	TL_3 Intensity (a.u.)	Tm_2 °C	TL_2 Intensity (a.u.)	Tm_3 °C	TL_3 Intensity (a.u.)
1	25.2	0	-	-	375	600	-	-	375	790	230	6900	375	4200
		5	-	-	375	670	230	250	375	740	230	9000	375	7700
		10	230	330	375	1400	-	-	375	2100	230	11300	375	6800
		30	230	380	375	690	230	150	375	740	230	7800	375	4300
2	25.2	0	-	-	375	690	-	-	375	1600	230	9700	375	4400
		5	-	-	375	850	-	-	375	1700	230	12600	375	5600
		10	-	-	375	1100	-	-	375	1900	230	13700	375	6000
		30	-	-	375	900	-	-	375	2000	230	12900	375	3900
3	25.2	0	-	-	375	690	-	-	375	2400	230	24600	375	10500
		5	-	-	375	580	-	-	375	4100	230	28700	375	12600
		10	-	-	375	780	-	-	375	3900	230	16000	375	5900
		30	-	-	375	790	-	-	375	3200	230	29000	375	8900

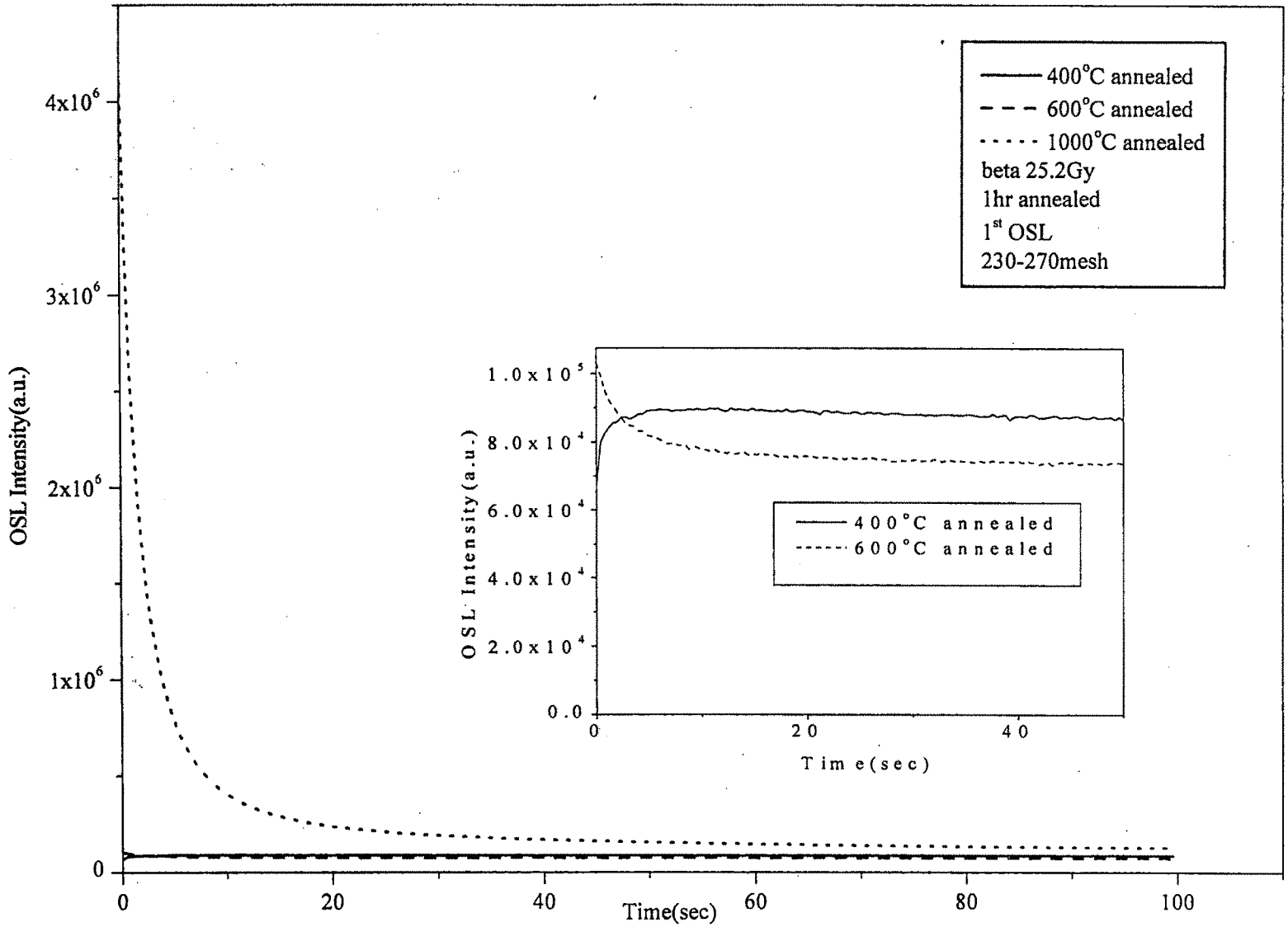
i.e. as temperature of pre-heat treatment increases, OSL increases and yields exponential decay. But when PIHT duration was increased beyond 10 seconds it was found that OSL increases. Thus, it is believable that PIHT may cause the charge transfer from shallow traps to deep traps. This suggestion is supported by TL studies[36].

4.6 Study of OSL under cyclical succession at 160°C measured

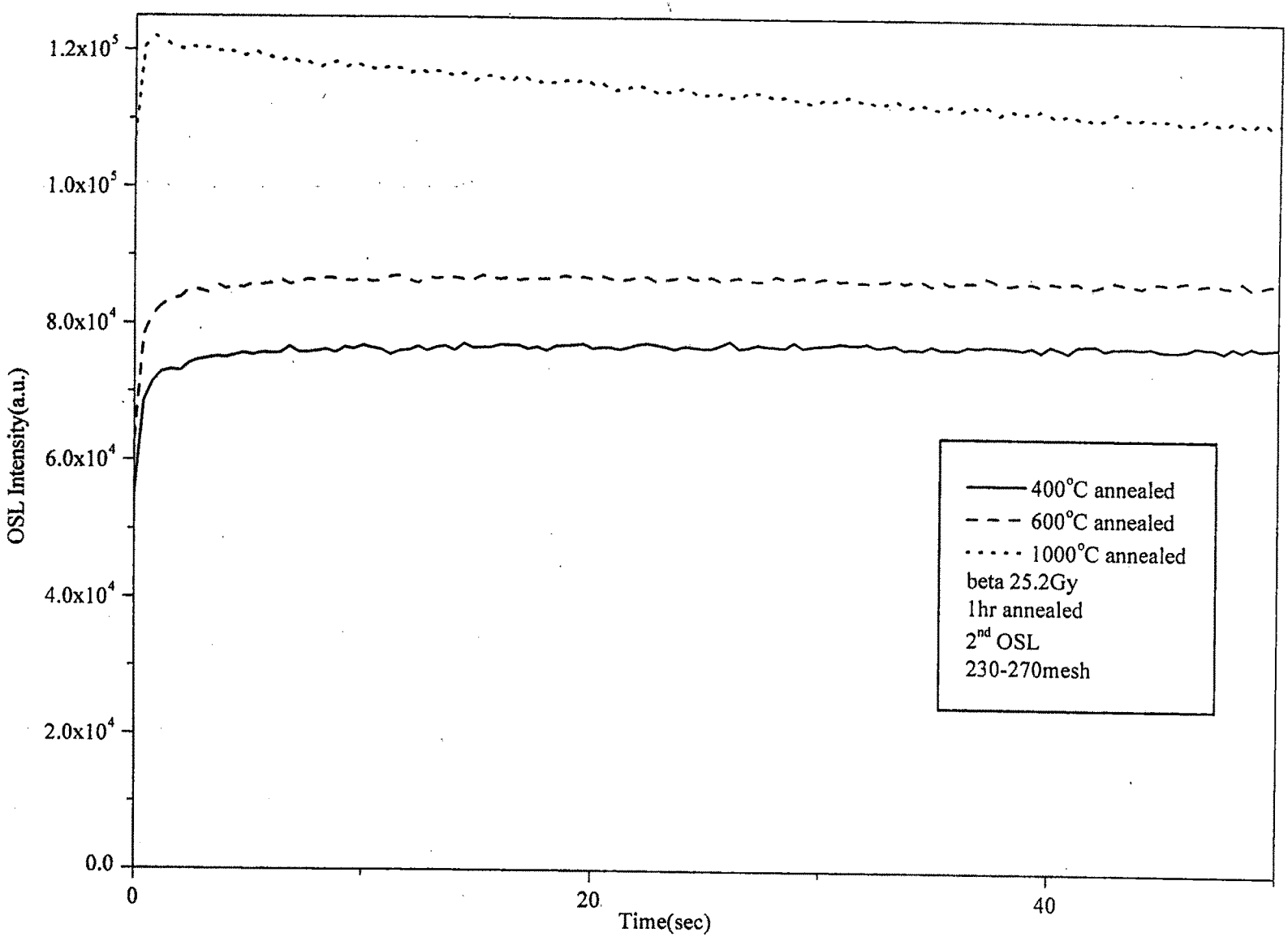
To know the stability of OSL signal, the decay curves were recorded at elevated temperature (160°C) for three cycles. These decay curves were recorded for the specimens after subjecting them to same beta dose 25.2Gy and different physical protocols as followed in earlier studies. The results are presented in **Graph-92** to **Graph-94**.

It is observed that as the temperature of annealing was raised from 400°C to 1000°C for 1hr pre-heat duration, optically stimulated luminescence intensity is enhanced from 89798a.u. to 4.10521×10^{6}a.u. It is also observed that the shape of OSL decay is non-exponential for the specimen pre-heat treated at 400°C but it becomes exponential beyond this temperature of pre-heat treatment. These observations are for the first run of OSL decay. However, following the same when second run (**Graph-93**) and higher runs (**Graph-94**) of OSL decay was recorded, it was found that OSL increases with the rise in temperature of annealing, but the degree of rise in OSL level i.e. the growth in OSL level decrease for higher cycles. This is quite clear in **Graph-93** as OSL increases form 78413a.u. to 122071a.u. for the second run of observation under identical physical conditions.

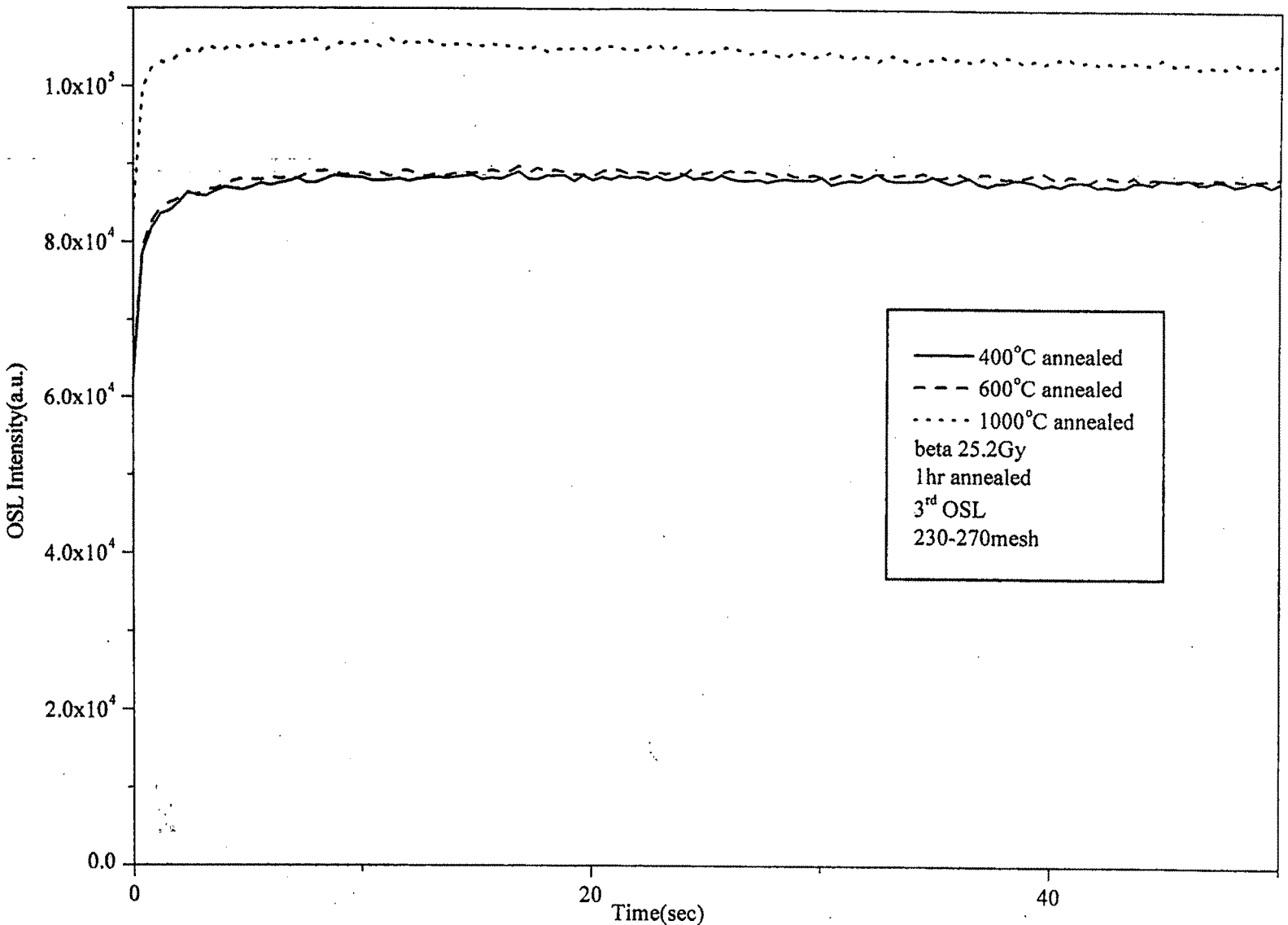
Similar study was also carried out for 2hrs (**Graph-95** to **Graph-97**) and 3hrs (**Graph-98** to **Graph-100**) durations of pre-heat treatment. Almost same behaviour is observed but here it was found that the decay remains non-exponential with either rise in temperature of annealing or higher run of



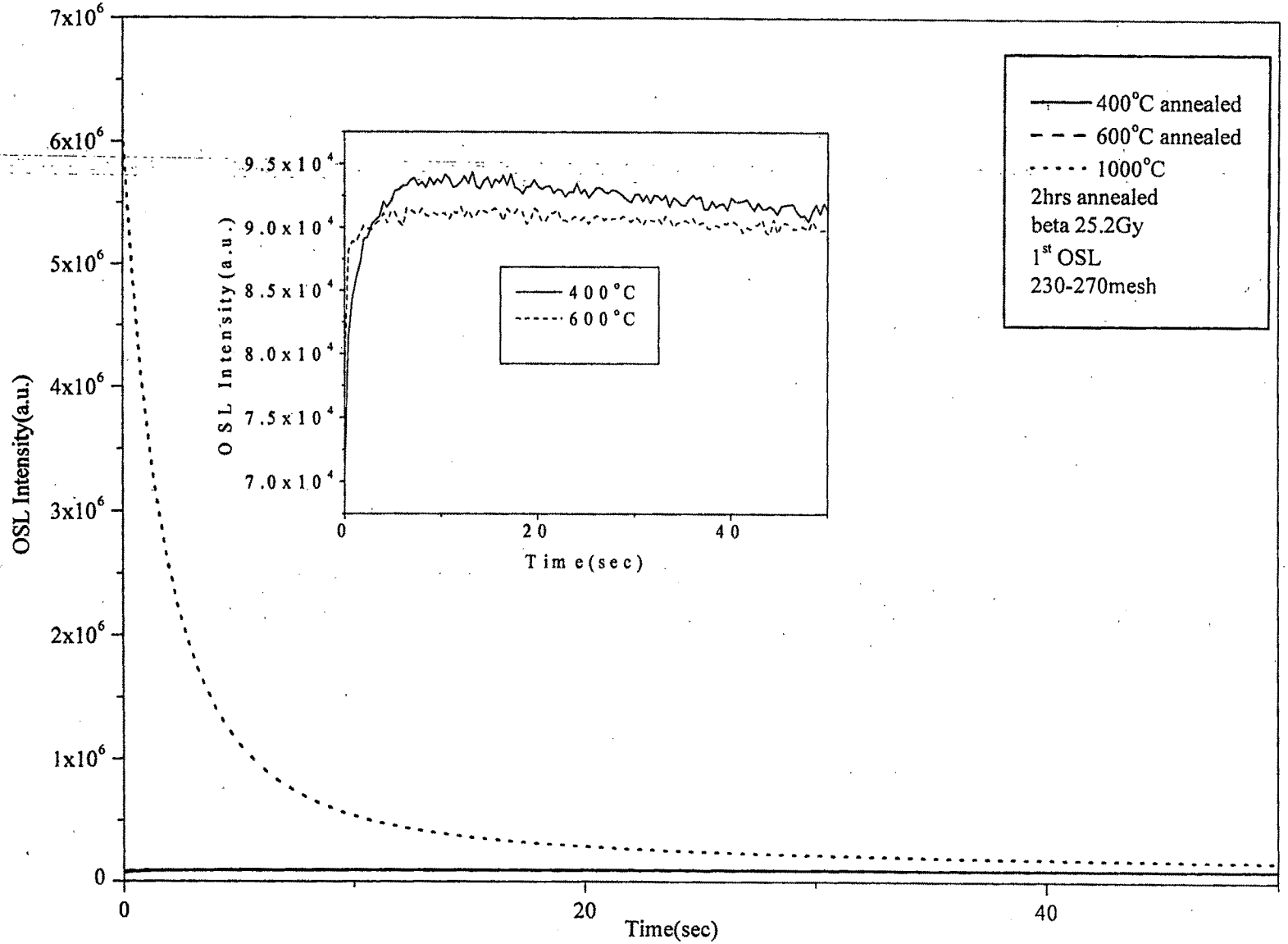
Graph-92 1st OSL measured at 160°C of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



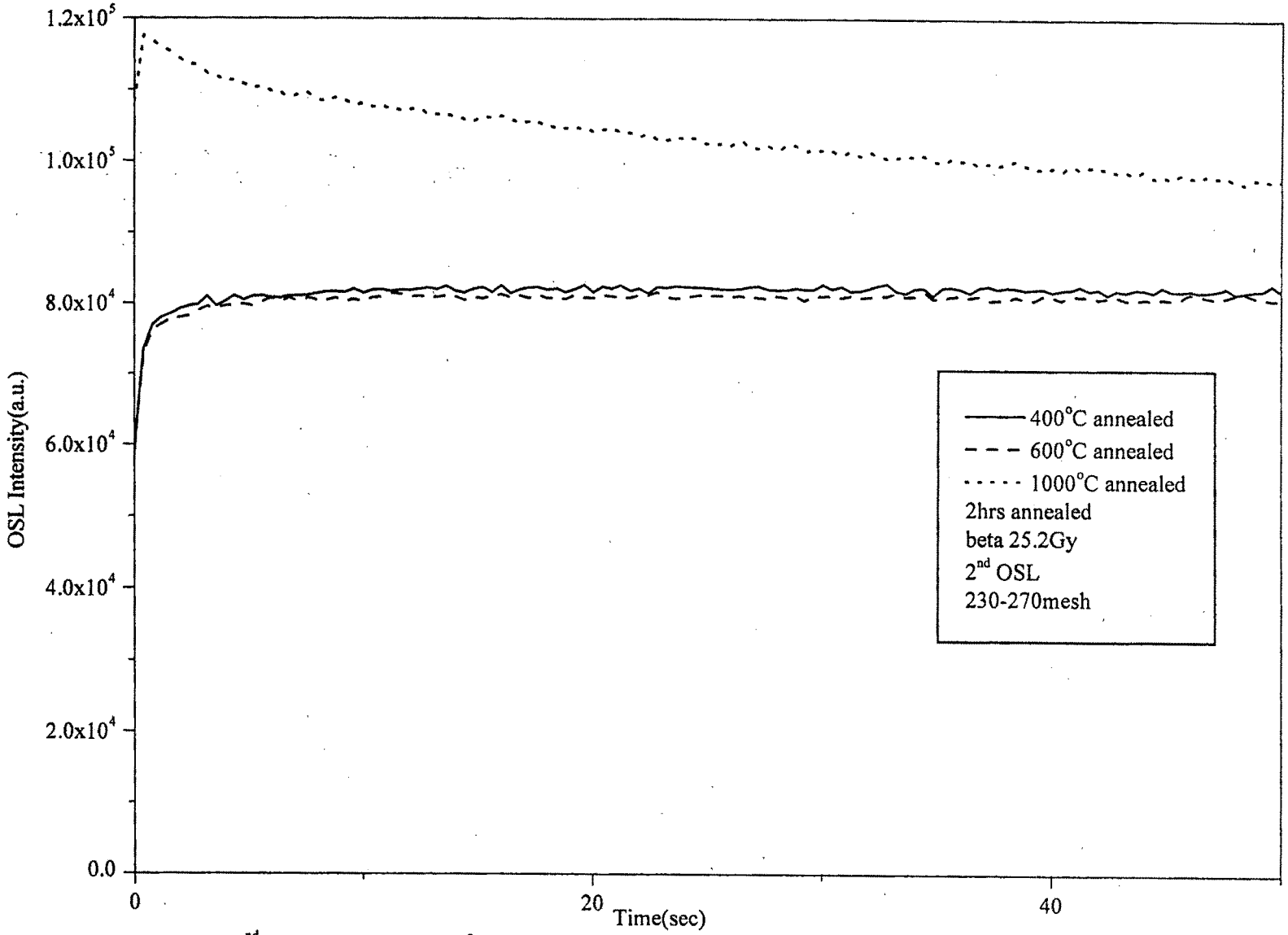
Graph-93 2nd OSL measured at 160 °C of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



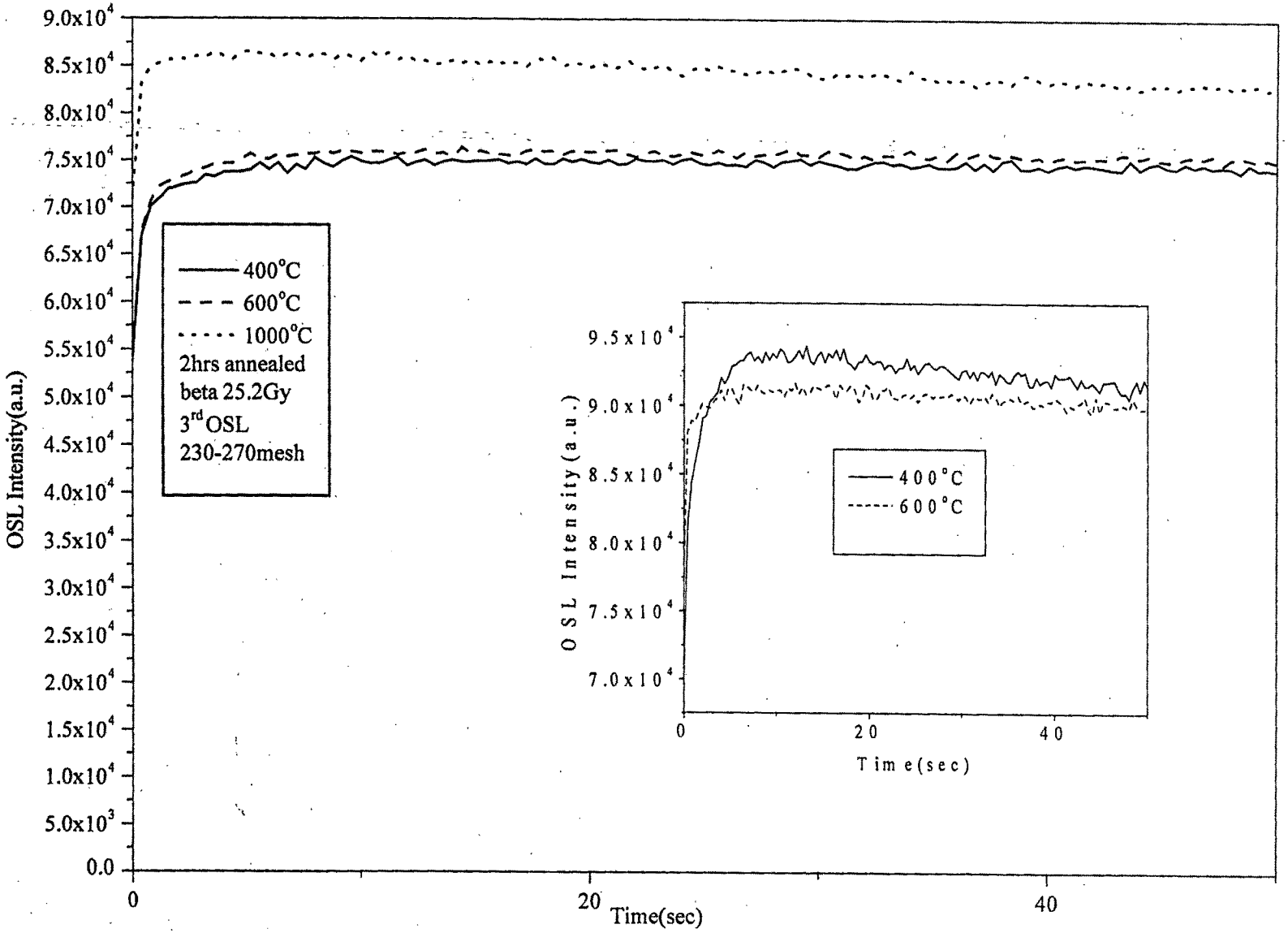
Graph-94 3rd OSL measured at 160°C of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



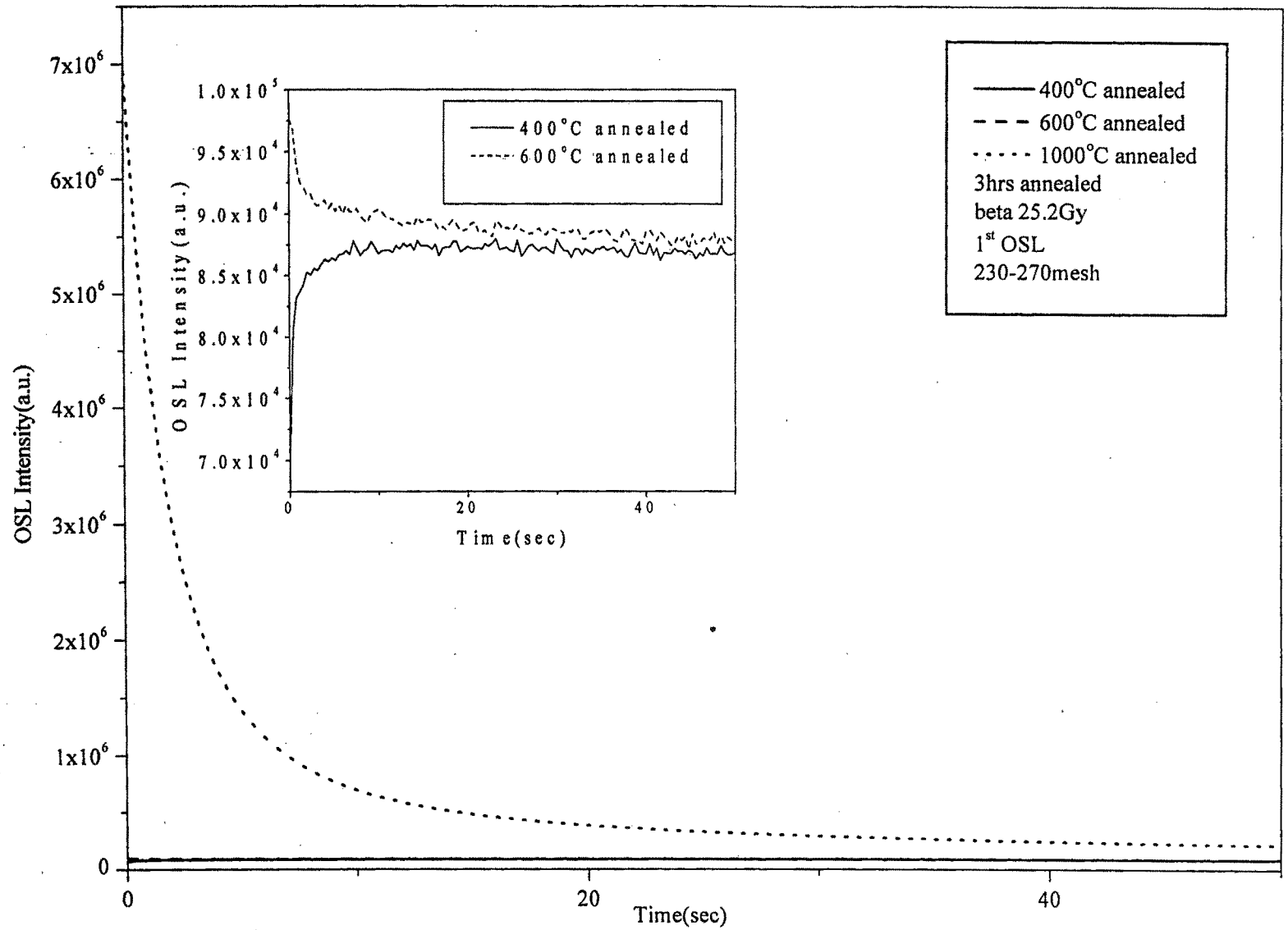
Graph-95 1st OSL measured at 160°C of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



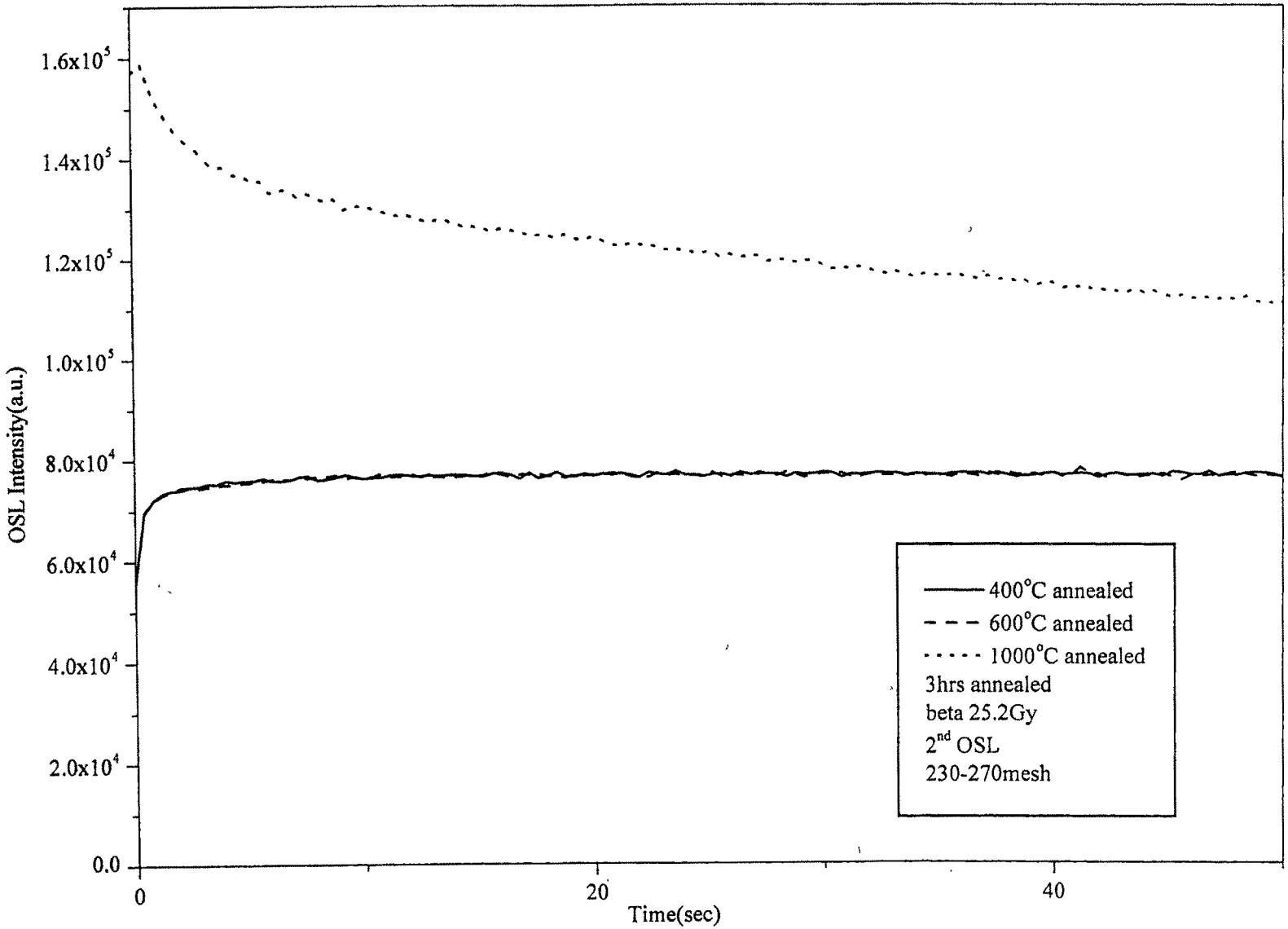
Graph-96 2nd OSL measured at 160°C of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



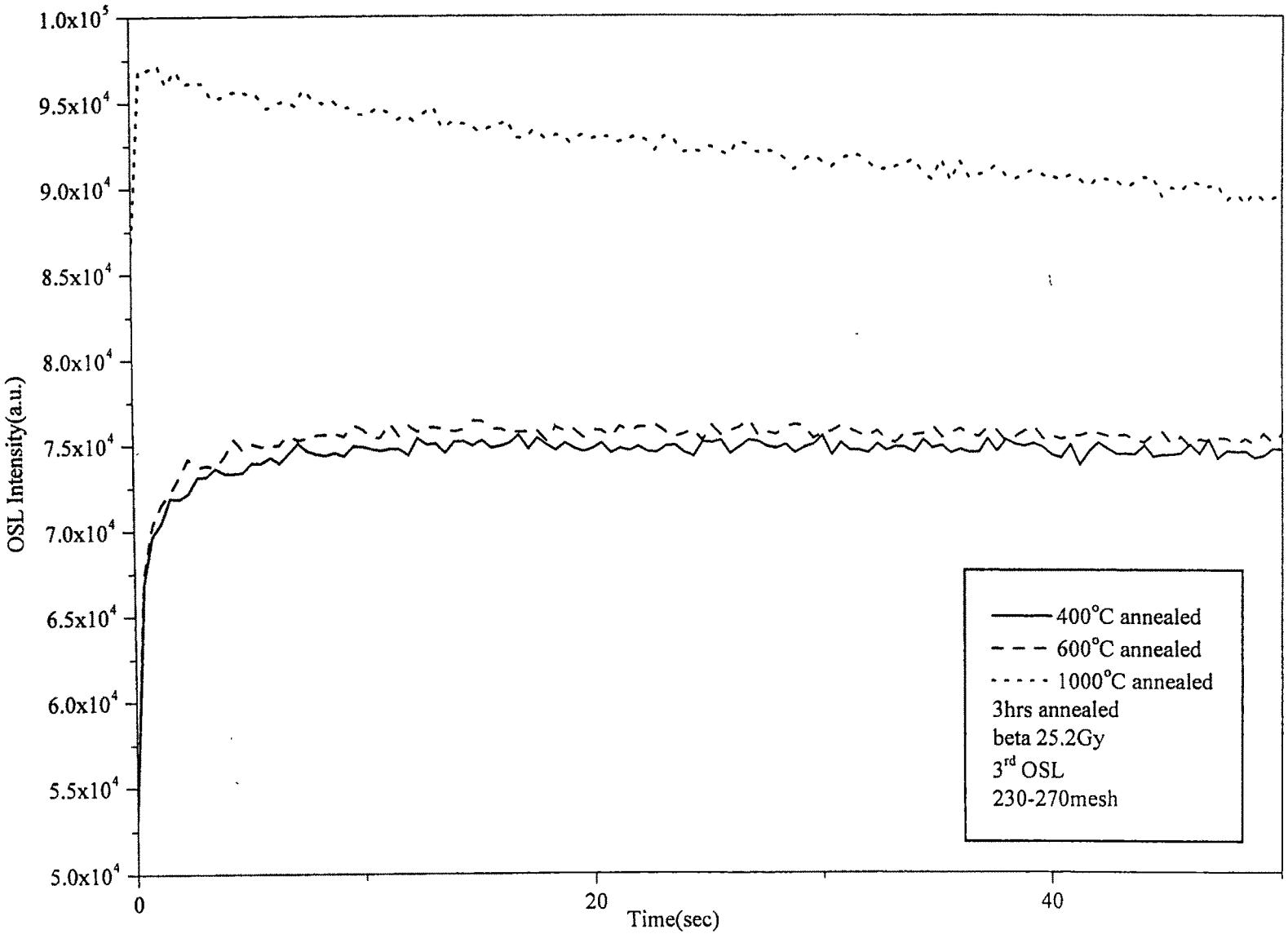
Graph-97 3rd OSL measured at 160 °C of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



Graph-98 1st OSL measured at 160 °C of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



Graph-99 2nd OSL measured at 160 °C of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.



Graph-100 3rd OSL measured at 160 °C of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperatures for 230-270mesh.

OSL record except for the specimens pre-heat treated at 1000°C for 2hrs and 600°C and beyond for 3hrs of pre-heat duration, wherein the OSL decay becomes non-exponential from the exponential shape for the higher cycle of OSL records.

The **Table-4D** gives the information in tabular form that shows the data of OSL intensity and initial stimulation peak time for the different cycles of OSL decay records for the specimens when pre-heat treated differently. It indicates that, as the number of cycles increases OSL level decreases for almost any temperature of annealing. This is also discernible for higher duration of pre-heat treatments. The changes in OSL decay pattern for the higher cycles of OSL decay are worth noting. It is found that the decay becomes slower with the higher runs of OSL decay and the decay remains non- exponential for the specimens pre-heat treated at 400°C (even for higher duration of pre-heat treatment). Further, the decay is almost exponential in nature for the specimens pre-heat treated at and beyond 600°C, but it becomes non-exponential when repeated OSL is performed for the same protocol. This is also true for higher durations of pre-heat treatment.

Being structure sensitive phenomenon and well-established tool to know the stability of the involved centers, thermoluminescence study was carried out and it was co-related with OSL results for different protocols. The comparison is also made between TL measurements made before taking repeated OSL and after measuring repeated OSL in order to know the effect of repeated illuminations on centers and on their locations.

TL measurements before recording OSL decay for a given pre-heat treatment (at 600°C) of interest followed by 5.04Gy beta dose exhibits two well-defined glow peaks around 110°C and 230°C. However when the

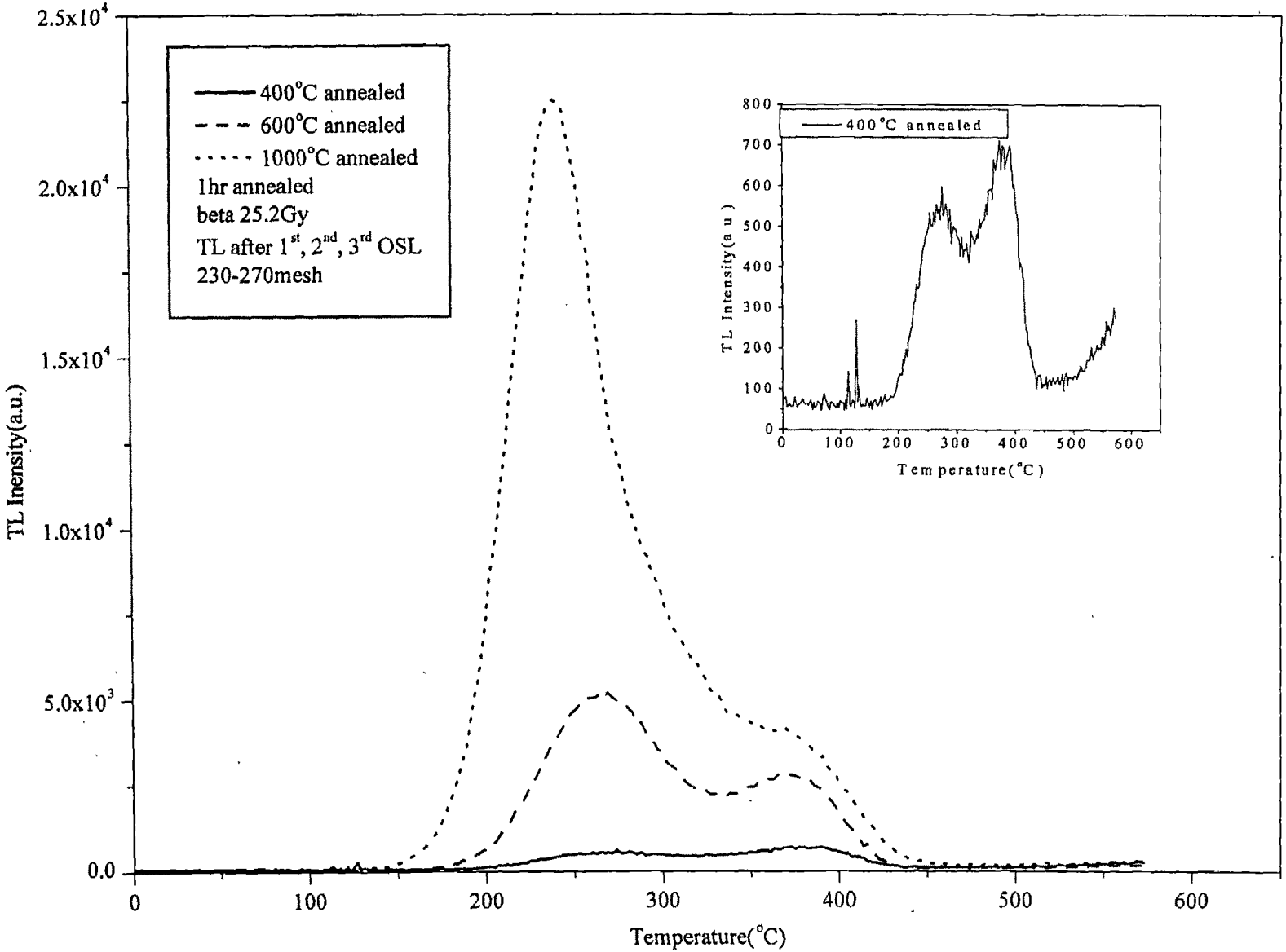
Table-4D: Data table of OSL intensity at initial stimulation peak time for different cycle of OSL for different annealed sample.

Annealing Temperature (°C)	Annealing Duration (Hrs)	Number of Stimulation (at 160 °C for 100 sec)	Initial peak time (sec)	OSL Intensity (a.u.)
400 °C	1hr	1st	10.8	89798
		2nd	66.8	78413
		3rd	16.8	89062
600 °C	1 hr	1st	0.0	102986
		2nd	24.0	87535
		3rd	16.8	89777
1000 °C	1 hr	1st	0.0	4.10521×10^6
		2nd	0.8	12271
		3rd	11.2	106120
400 °C	2 hrs	1st	13.2	94362
		2nd	32.8	82918
		3rd	9.6	75369
600 °C	2 hrs	1st	12.0	91609
		2nd	34.4	81754
		3rd	14.4	76419
1000 °C	2 hrs	1st	0.0	6.08843×10^6
		2nd	0.4	117614
		3rd	10.4	86561
400 °C	3 hrs	1st	23.2	87965
		2nd	23.6	77447
		3rd	16.8	75552
600 °C	3 hrs	1st	0.0	97623
		2nd	41.2	78138
		3rd	26.8	76420
1000 °C	3 hrs	1st	0.0	6.9316×10^6
		2nd	0.4	159075
		3rd	1.2	97232

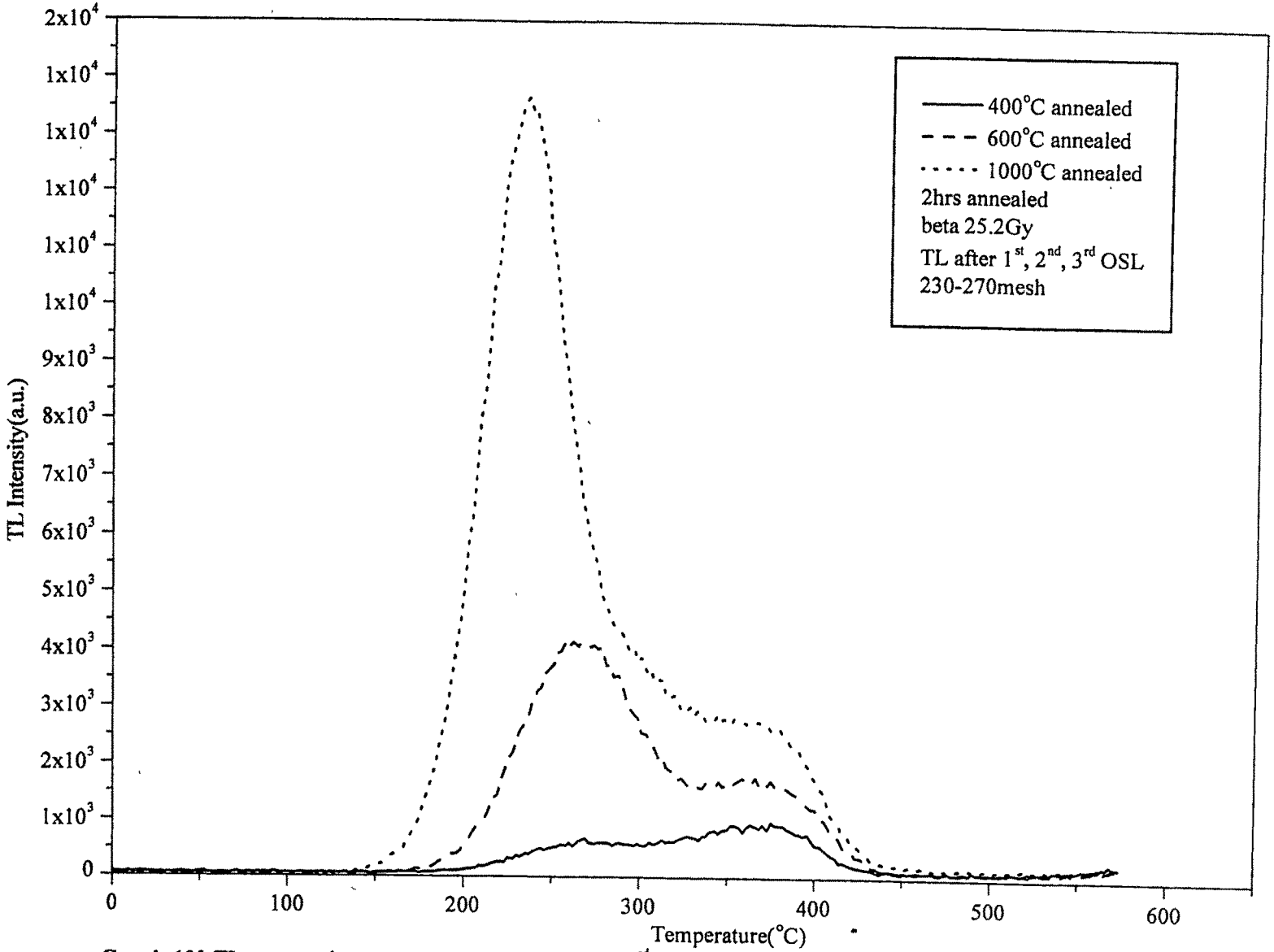
specimens were pre-heat treated at 1000°C they showed three distinct glow peaks at around 110°C, 220°C and 375°C.

Subjected to identical physical conditions the specimens were illuminated for 100sec and then OSL decay was recorded at 160°C. Thereafter repeatedly two more times similar procedure was followed to measure repeated/cyclic OSL decay measurements. Such a specimen was then taken for TL read out to measure TL after repeated OSL measurements. The results are presented as **Graph-101** to **Graph-103**. Such a specimen exhibits well-defined glow peaks at around 240°C and 375°C. It is interesting to note that the 375°C TL glow peak, which was absent for the specimens pre-heat, treated below 1000°C, before, taking OSL now becomes clearly observable after repeated OSL runs. Glow curves after OSL runs show that both the peaks (230°C and 375°C) grow with the rise in temperature of annealing, but more growth is discernible for 230°C TL glow peak. Similar behaviour is observed for the specimens pre-heat treated for higher durations.

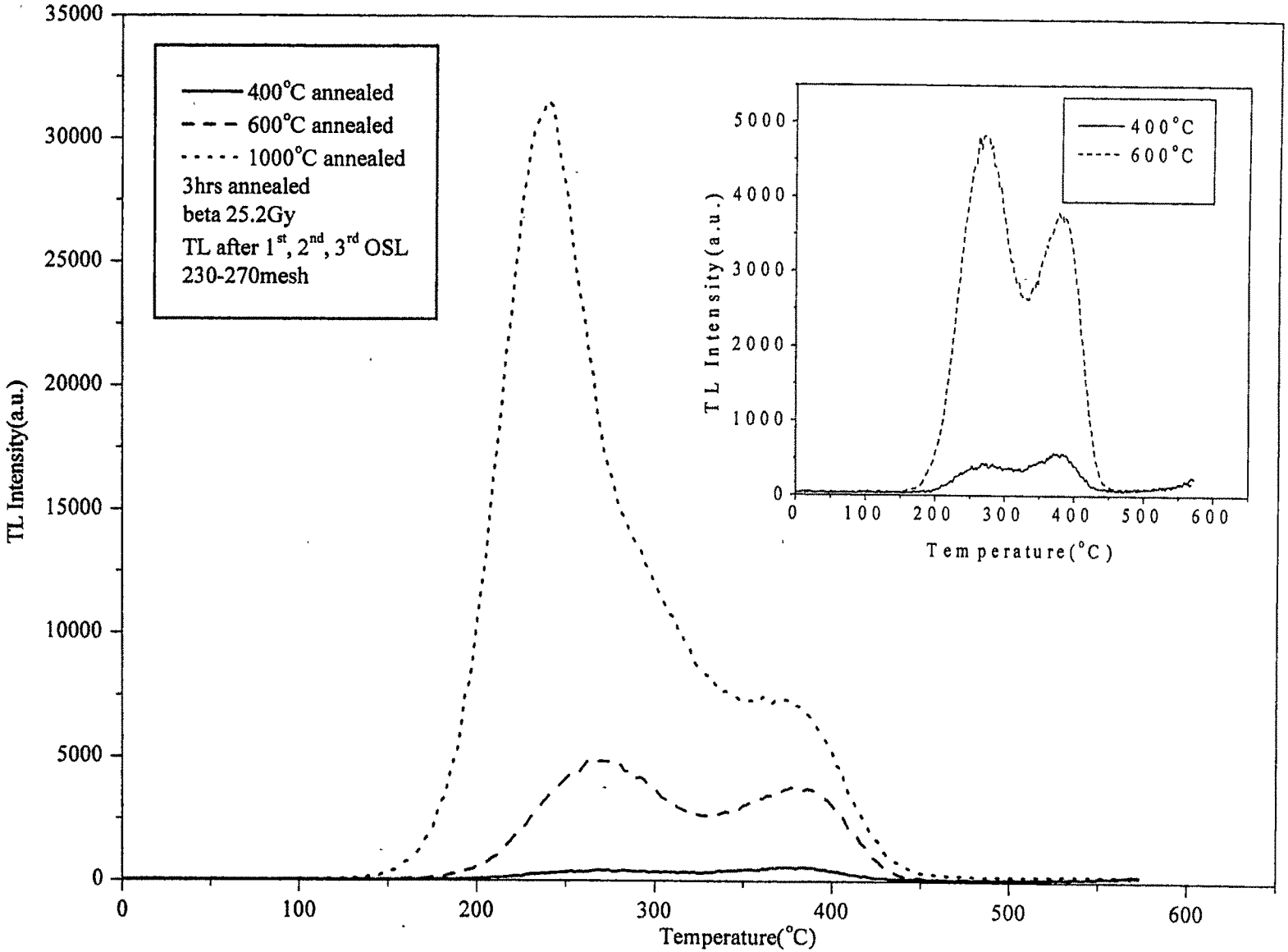
It is already established that OSL can be sensitized with the pre-heat treatment. The remarkable growth in OSL sensitivity for the specimens when pre-heat treated beyond 600°C may be attributed to the phase transformation in quartz i.e.α to β at 573°C.[10] The literatures on TL and spectral studies have revealed that the quartz has rapidly bleachable peak (RBP) (around 325°C) and slowly bleachable peak (SBP) (around 375°C).[16] Further, it is suggested that during optical stimulation, electrons released from optically sensitive traps which corresponds to RBP, major contribution of OSL signal comes from RBP. On the premise of these facts, the decrease in OSL level with repeated cycles of record under identical physical conditions can be explained. It is suggested that the traps corresponding to 325°C TL peak is



Graph-101 TL measured at room temperature (after the 3 rd OSL measured at 160 °C) of 1hr annealed sample for a given dose of 25.2Gy at different annealing temperature for 230-270mesh.



Graph-102 TL measured at room temperature (after the 3 rd OSL measured at 160 °C) of 2hrs annealed sample for a given dose of 25.2Gy at different annealing temperature for 230-270mesh.



Graph-103 TL measured at room temperature (after the 3 rd OSL measured at 160 °C) of 3hrs annealed sample for a given dose of 25.2Gy at different annealing temperature for 230-270mesh.

continuously emptied during repeated optical stimulation process, which causes the decrease in OSL level.

Changes in the shape of OSL decay curve can be explained on the basis of initial stimulation peak time. Except for the specimens pre-heat treated at 400°C the decay becomes non-exponential from the exponential nature with the repetition of OSL record. This may be attributed to the contribution of RBP, but with the repeated optical stimulation the concentration of centers seems to decrease and hence the initial stimulation peak time increases, which gives rise to non-exponential decay for higher run of OSL records.

Further, TL measurements after repeated OSL records at elevated temperature (at 160°C) showed the absence of 110°C peak which otherwise appears predominantly. It is clearly due to sweeping of this peak on account of OSL measurement at 160°C. Significant growth of ~230°C TL peak with the rise in temperature of annealing as compared to 375°C may be attributed to pre-heat treatment and transferring of electrons during optical stimulation. Compare to deeper trap corresponding to 375°C TL peak more electrons appear to be transferred to 230°C TL peak in quartz. The rise in ~230°C TL peak intensity even after repeated OSL records suggest that it is the stable dosimetric peak.[34, 33, 37]

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