Nuclear Instruments and Methods in Physics Research B 383 (2016) 177–182

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Thermoluminescence response of natural white quartz collected from Gelephu, Bhutan

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ARTICLE INFO

Article history:
Received 1 July 2016
Received in revised form 15 July 2016
Accepted 15 July 2016
Available online xxxx

Keywords:
Quartz
Thermoluminescence
Glow curve
Dosimetry

ABSTRACT

TL properties of natural quartz mineral collected from Gelephu, (Bhutan) were studied. With the help of various characterization techniques the quality of the sample was tested. The thermoluminescence (TL) analysis was carried out under X-ray irradiation. The un-irradiated sample showed no TL signal; however, after X-ray irradiation, a composite glow curve was observed. The kinetic analysis of the glow curve was carried out and it was observed that there was five trapping sites at depths ~0.68, 0.90, 0.97, 1.06 and 1.10 eV responsible for five closely spaced glow peaks at ~341, 362, 383, 397 and 426 K respectively. The dosimetric features of the mineral were studied. The response when studied from the whole glow curve was non-linear. However, the dose response studied from the 426 K peak was found to be linear from 10 mGy to 10 Gy. The fading of the TL signal of this 426 K peak was ~12% within 5 days after irradiation and onward it was ~4% up to 30 days. The reproducibility of the results was also good.

1. Introduction

Quartz (SiO_2) is one of a most abundant mineral found in earth surface. The pure quartz is colorless, but due to presence of impurity, it may be found in different colors as orange, purple, pink etc. The luminescence properties of quartz have been studied extensively in both natural and synthetic samples for last five decades because of its importance in dating and retrospective dosimetry [1–13, and references therein]. Rendell et al. [1] investigated the spectral changes occurring in the thermoluminescence (TL) of quartz of various origins namely hydrothermal quartz, synthetic quartz and the samples of volcanic origin. It was reported that the heat treatments affected both the relative intensities of the TL peaks, as well as the relative intensities of the various emission bands. Wintle and Murray [2] studied the TL sensitivity changes that occurred during laboratory heating of 30,000 yr old sedimentary quartz from Australia. Lima et al. [3] studied the effect of various thermal treatments on the TL glow curves and emission spectra of a natural quartz crystal. Pagonis et al. [4] carried out the TL analysis of quartz of various origins and found some common characteristics among the glow curves. It was reported that the main structure (shape, peak position and trapping parameters) of the glow peaks varies within narrow limits even after the application of extreme conditions of temperature and irradiation. Kitis et al. [5] carried out a detail kinetic analysis of the TL glow curve of synthetic quartz using various analysis techniques and found some important results. Further, Kitis et al. [6] studied the cooling rate effects on the TL glow curves of Arkansas quartz. It was reported that a fast cooling rate leads to significant enhancements of the TL intensity for the well-known 383 K (110 °C) TL peak, as well as a change in the ratio of the relative intensities of the main TL peaks. Polymeris et al. [7] studied the effects of annealing and irradiation on the sensitivity and superlinearity properties of the 383 K (110 °C) TL peak of high purity synthetic quartz, natural Arkansas quartz of hydrothermal origin and sedimentary quartz from the coast of the Chalkidiki region in Northern Greece. The thermal quenching effect in quartz of various origins was also studied in detail [8–10]. Topaksu et al. [11] and Nur et al. [12] studied the dosimetric characteristics of some natural hydrothermal quartz from Hakkari and Balikesir–Dursunbey area in Turkey respectively and both reported the supra-linear and linear dose response in the samples. Most recently, Zhou et al. [13] carried out the kinetic analysis of natural quartz from China.

Literatures show that, though there are some common behaviors in the TL characteristics of quartz; but depending on the nature and origin of quartz, the glow curves show a variety of shapes. In the present work, TL properties of natural white quartz collected from Gelephu, Bhutan have been studied. The dosimetric features of the sample also studied in details. Till date, no report is available on the TL properties of this sample collected from this location.

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http://dx.doi.org/10.1016/j.nimb.2016.07.012
0168-583X/

2. Experimental details

The white quartz mineral was procured from Gelephu, Bhutan. Geographically the place is located at latitude 26.88° N and longitude 90.51° E. At first, the mineral was cleaned and washed by distilled water. Then it was crashed into very small pieces and ground in a ceramic mill to powder form. In order to test the quality of the sample X-ray diffraction (XRD) analysis was carried out by a versatile X-ray diffractometer (X'Pert Pro, Philips, PANalytical); scanning electron microscopic (SEM) imaging and energy dispersive X-ray spectroscopic (EDXS) analyses were carried out by a field emission-electron microscope (FE-SEM) coupled with an energy dispersive X-ray spectrometer (ZEISS SIGMA VP). Finally, for compositional analysis X-ray fluorescence spectroscopic (XRFS) analysis were carried out by Philips (PANalytical) make AXIOS spectrometer. For TL analysis, in each aliquot 50 mg of the sample was taken. At first the sample was irradiated under X-ray by an X-ray source under operating voltage 35 kV at 15 mA filament current. According to the specification, under these operating conditions the tube delivers the X-ray dose at a rate of 0.05 mGy/s. TL was recorded within 30 s after X-ray irradiation by a TL reader (TL1009I, NUCLEONIX) at a constant linear heating rate of 2 K/s from 300 K to 520 K. In the TL reader, a quartz filter was inbuilt in front of the photomultiplier tube to pass UV–Vis light but it absorbed the infrared light. The average counts of three aliquots recorded under same experimental conditions were used for TL analysis.

3. Result and discussion

3.1. Sample characterization

Fig. 1 shows the XRD peaks for the powder sample. The sharp and intense peaks clearly show the crystalline nature of the sample. Comparing the XRD peaks with Joint Committee on Powder Diffraction Standard (JCPDS) file, the peaks were indexed by JCPDS number 89-8934. The crystal system was found to be hexagonal. Fig. 2 shows the FE-SEM micrograph of ground sample under different magnifications. The high resolution micrographs show that the sample had irregular grain size. The minimum detected size of a grain was ~873.3 nm however some relatively large grain of ~11.38 μm was also found in the prepared sample. The qualitative as well as the quantitative elemental analyses were carried out by EDXS analysis. Fig. 3 shows the EDXS pattern of the sample. This qualitative report shows the presence of O, Si, Ca, Fe, Ni and Cd in the sample. The quantitative analysis (Table 1) showed that the amount of O and Si was significantly high than the others. Further, for compositional analysis, XRFS analysis was carried out and the result was reported in Table 2. This analysis confirmed that the procured sample contained 92.30% pure quartz (SiO_2).

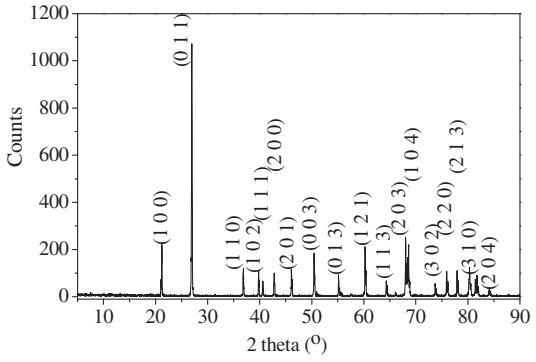

Fig. 1. Powder XRD patterns of the white quartz.

3.2. TL glow curve and kinetic analysis

The un-irradiated sample showed no TL signal. Fig. 4 shows the TL glow curves of natural quartz sample after different dose of X-ray irradiation. The intensity of the glow curves is found to increase with the irradiation dose. However, the positions of the glow peak did not change with the irradiation dose. Apparently three glow peaks were observed at ~346, 386 and 426 K respectively. The increase of peak intensity with the irradiation dose in the first peak was very small, whereas that in the second peak was moderate and in the third peak was highest. In order to identify the total number of peak in the composite glow curve, the well-known '$T_m - T_{Stop}$' analysis [14] was carried out and reported in Fig. 5. This analysis revealed that the glow curve may be composed five peaks located at ~341, 361, 382, 398 and 425 K respectively. The stability of the peaks with the irradiation dose (Fig. 4) along with the variation of peak maximum temperature (T_m) with the T_{Stop} temperature signified that most of the glow peaks followed first order kinetic, however at this stage the kinetic order of the 361 K peak was not clear.

Further, the glow curve was analyzed by Computerized Glow Curve De-convolution (CGCD) technique on the basis of Kitis general order equation (Eq. (1)) [15]:

$$I(T) = I_m b^{b/(b-1)} \exp\left(\frac{E}{kT}\frac{T-T_m}{T_m}\right) \times \left[(b-1)(1-\Delta)\frac{T^2}{T_m^2}\exp\left(\frac{E}{kT}\frac{T-T_m}{T_m}\right) + Z_m\right]^{-b/(b-1)} \quad (1)$$

where I is TL intensity, E (eV) is activation energy, I_m is peak maximum intensity, T_m (K) is peak maximum temperature, b is order of kinetics, k is Boltzmann's constant, T is absolute temperature, $\Delta = 2kT/E$, $\Delta_m = 2kT_m/E$ and $Z_m = 1 + (b-1)\Delta_m$. The goodness of fit was tested by the Figure of Merit (FOM) [16]. The frequency factor, s (s^{-1}) was calculated from the general order equation [17]. The trapping parameters such as E, b and frequency factor s etc. were estimated and reported in Table 3. After proper selection of the fitting parameters it was observed that the glow curves were composed of a unique combination of five overlapping peaks. The de-convolved glow curves of 15, 60 and 90 mGy dose irradiated samples were shown in Fig. 6. Bellow the de-convolved glow curves the residuals of the fit were shown. The low value of FOMs (Table 3) and the residual plot signified that the fittings were excellent.

De-convolution analysis revealed that for all the samples irradiated under different dose, the trapping parameters for a particular peak were approximately same. There were five trapping sites at depth ~0.68, 0.90, 0.97, 1.06 and 1.10 eV responsible for the five closely spaced glow peaks I, II, III, IV and V at ~341.2, 362.3, 383.1, 397.3 and 426.2 K respectively. The values of the order of kinetics signified that all the glow peaks follow the first order kinetic, except the peak II which was found to follow the general order kinetics. These results were consistent with the previous $T_m - T_{Stop}$ report. These results were also consistent with the previous results on geological quartz mineral from different origin [4,7,11–13]. However, some difference in intensity and peak position were observed with them.

3.3. Dosimetric features

3.3.1. Dose response

For dosimetric application the stability of the peak with irradiation dose is very important. From Fig. 4 along with the de-convolution analysis, it was confirmed that there was no