A Summary of Ph.D thesis Entitled

OPTICALLY STIMULATED LUMINESCENCE STUDY OF SYNTHETIC QUARTZ AND THEIR APPLICATIONS

To be submitted to

THE MAHARAJA SAYAJIRAO UNIVERSITY OF

BARODA

For the award of the degree of

DOCTOR OF PHILOSOPHY (IN APPLIED PHYSICS)

By

Sanskriti Parashar

Under the guidance of

DR. D. R. JOSHI



Applied Physics Department Faculty of Technology and Engineering The M. S. University of Baroda Vadodara-390 001.

MAY 2020

Name of the Candidate	:	Mrs. Sanskriti Parashar
Name of the Research	:	Dr. D. R. Joshi
Supervisor		
Subject	:	Applied Physics
Registration Certificate	:	252
Number		
Date of Registration	:	19.12.2011
Title of Thesis	:	Optically Stimulated Luminescence
		Study of Synthetic Quartz and Their
		Applications
Place of work	:	Applied Physics Department
		Faculty of Technology and Engineering
		The Maharaja Sayajirao University of
		Baroda, Vadodara–390001, Gujarat

Sanskriti Parashar

(Research scholar)

Forwarded through

Dr. D. R. Joshi (Research Guide)

Head (Applied Physics Department)

DEAN Faculty of Technology and Engineering The Maharaja Sayajirao University of Baroda Laboratory grown synthetic quartz is pure, with no twins and impurities. The level of defects can be controlled during the production of synthetic quartz crystal by a hydrothermal technique which becomes very easy to understand the structure of the material and luminescence properties. So, it widely used in dating and dosimetry applications due to its high purity, crystalline quality and structure sensitive nature.

For the dating purpose, the grain size of the sample is most significant and crucial. The grain size of the sample depends upon grinding process of the material. It was observed and explained that the significant and noticeable deviation in electronic, magnetic, optical and chemical properties of a molecule with the change in grain size from bulk to nano-metric level. It is also known that the new active/inactive TL and OSL sites were developed due to increased surface area after reduction in grain size of sample by grinding process.

Phosphor powder is attracting considerable attention among different nano materials due to their novel optical properties, which affect emission lifetime and luminescent efficiency. The excellent result related to the luminescence efficiency of nano-ceramic material and silica nanoparticles has reported. We focused on TL and OSL of nano synthetic quartz in present work for its use in future in dating and dosimetric applications.

The Possible parameters were efficiently optimized to minimum mean particle size and poly dispersity index (PDI) for achieve nano size synthetic quartz. After that synthetic quartz nanoparticles were successfully prepared by media milling process in a high energy planetary ball mill. The prepared nano synthetic quartz sample was characterized by particle size analyser, SEM, TEM, XRD, EDX and FTIR. The results obtained were found in good agreement with each other. The optical properties of synthetic quartz nanoparticles were investigated by UV-Visible and PL analysis.

Average grain size and PDI of prepared sample were obtained 87 nm and 0.119, respectively by the particle size analysis. The SEM analysis further confirmed that the particle size was in nano scale but shapes of the particles were irregular and agglomerated. The TEM image also described that the particles of the sample are in nano range (below 100nm). XRD analysis of prepared samples indicates that the sample is in the form of nano synthetic

quartz powder with crystalline phase with 39.41nm crystalline size. Cristobalite phase was appeared with beta phase in 600°C sample but at 1000°C anneal involvement of Cristobalite phase was increased with tridymite phase. Due to preparation by ball milling process of NSQ, there were possibilities of the presence of metallic contamination or process impurities in the material. Therefore, the purity of nano sized samples was checked by EDX technique. The sample was found pure SiO_2 . The presence of hydroxyl group has confirmed by the FTIR analysis in the prepared nano sized synthetic quartz samples. UV spectra of unannealed samples followed by 2.52Gy and 5.04Gy beta doses have exhibited optical absorption intensity around 0.14 to 1.664 a. u for broad range of peaks from 200-258nm. The positions of these peaks remain identical but notable absorption by 2 to 2.5a.u is observed under the influence of pre-heat treatments such as 400°C, 600°C and 1000°C annealed sample followed by 25.2Gy dose than unannealed samples. This absorption peak is due to presence of E₁-centres (defects associated with oxygen vacancies) in synthetic quartz samples. Therefore, UV spectra showed that annealed samples are more optically active in comparison to unannealed samples. PL spectra showed that the NSQ sample exhibits broad emission wavelengths from 371nm to 493nm under 254nm wavelength of excitation. PL study has confirmed the suitable wavelength (470nm) as a stimulation source in OSL process.

TL study was successfully performed for unannealed and different annealed (400°C, 600°C, 1000°C) nano samples followed by beta doses (48.03Gy, 81.43Gy, 120.83Gy, 160.23Gy and 199.63Gy). Below 120.83Gy of beta doses, the unannealed NSQ samples gave significant contribution of traditional 110°C TL glow peak and close to rapidly bleachable TL glow peak at 300°C. Beyond 120.83Gy of doses, the position of 110°C TL glow peak was sustained along with the development of two new TL glow peaks at 165°C and 220°C by fading of 300°C glow peak.

Lower annealed NSQ samples (at 400°C and 600°C) gave the predominant contribution of shallows TL glow peak (at 110°C) and significant strength in TL counts. It attributed to the growth of E_1 ' centres and first phase transformation in NSQ samples. The NSQ samples annealed at 1000°C contributed for shallows traps but it also reduced the TL strength of 110°C glow peak. The higher annealing treatment produced better thermal stability and improved TL dose response of 220° C glow peak. According to ESR results, the Ge centres were developed by loss of E₁' centres.

The TL study after optical bleaching at room temperature supported to growth of 220°C TL glow peak by recuperation process in which the charges were released from optically sensitive traps and transferred to stable 220°C TL trap. While the TL study after optical bleaching at 160°C offered combine effect (recuperation process from optically sensitive traps and thermal erosion of shallow TL traps). These combine physical process of the charges were responsible for growth of new stable TL glow peak at 230°C which suggested widen family of stable TL traps in NSQ sample. Due to influence of optical bleaching temperatures, the NSQ material gave the information about wider family of stable TL trap between 220°C and 235°C.

The 1000°C annealed sample followed beta doses and optical bleaching at RT gives thermal stability of 220°C TL glow peak and gives new development of 300°C TL glow peak accordance with 110°C TL glow peak. The identical sample was optically bleached at 160°C enhances thermal stability of 230°C TL glow peak and new development of 325°C TL glow peak by disappearing of contribution of 110°C TL glow peak.

The lower annealing treatment of 400°C helps to enhance average TL growth of 110°C glow peak by 95.90% over unannealed samples. This average TL growth percentage is too effective than average TL growth percentage of 110°C glow peak of 600°C and 1000°C annealing temperatures.

The TL results of NSQ specimens with optical bleaching at room temperature also revealed re-trapping of optically released charges. While, the development of 230°C TL glow peaks under influence of optical bleaching at 160°C reveals clearly the thermally stable TL glow peaks range within 220°C-230°C along with rapidly bleachable TL glow peaks within 300°C-325°C.

Unannealed NSQ samples were showed super-linear (k=1.57) characteristics of 110° C TL-DRC. Superlinearity was also observed for 110° C TL peak dose response at lower annealed NSQ samples (400°C and 600°C) and superlinearity was changed into sublinearity for the same TL peak as annealing temperature for NSQ raised to 1000°C temperature. On the other side, TL-

DRC nature of 165°C and 220°C TL glow peaks was also showing sub-linear dose response. Hence, it is reasonable to believe that competitors were removed at higher anneal temperature of NSQ sample with decrease in sensitivity. Sublinearity TL–DRC nature of 1000°C annealed NSQ sample was remaining same even after optical bleaching at RT as well as ET. Thus, it can be concluded that competitors may not participate in TL output signals before or after optical bleaching. That means optical bleaching at RT or ET has no roll with trap competitors in any TL process.

Under influence beta doses, the unannealed NSQ samples exhibits the rapid OSL decay within 0 to 0.4 seconds of stimulations and usual OSL decay within 0.4 to 100seconds of stimulation. The nature of rapid OSL decay is responsible to existence of ultra-fast components (UFC) which are associated with availability of shallow unstable traps. The nature of usual OSL decay is responsible to predominant contribution of optically sensitive traps.

The growth of I_{max} OSL counts depends upon strength of ionizing radiations. However, the variations in OSL counts with beta doses are due to either saturation of OSL traps or growth of new OSL traps.

The annealed samples followed by identical beta doses exhibit contribution of UFC and usual components. The 600°C and 1000°C annealed samples gives better uniform OSL growth response with beta doses up to 160.23Gy than OSL growth response with beta doses in 400°C annealed samples. The strength of OSL counts decreases with rise in annealing temperature followed by identical beta doses.

While comparing deconvolution observations of OSL at ET between unannealed and annealed NSQ samples for beta doses, the lower annealed samples enhanced the contribution of fast and medium components of OSL over components of unannealed samples within 0.4 to 100 seconds of stimulation. The contributions of proposed components were diminished by the growth of slower components of OSL in higher annealed samples over unannealed samples. The lower annealed samples enhanced the PIC of fast and medium components of OSL decay curves over PIC of unannealed samples OSL-DRC (at RT) of unannealed NSQ sample showed super linearity with rise in beta radiation dose which turned into sublinearity as sample annealed at different temperatures. The superlinearity of OSL DRC for unannealed NSQ sample with rise in radiation dose might be due to the competition between the trap and the recombination center for free electrons during irradiation. The shifting of superlinearity in unannealed NSQ specimens into sublinearity with thermal treatment to the NSQ specimens indicated that the competitors/electron traps may be removed or non-recombination centers might have been activated due to annealing temperature.

NSQ showed identical pattern of OSL at 160°C ET as similar pattern as obtained for OSL recorded at RT. The strength of OSL counts decreased with the rise in annealing temperature followed by identical beta doses was also observed. The contributions of ultra-fast components (UFC) and usual OSL components (fast, medium and slow) still exist. The annealing temperatures from 400°C to 600°C support to growth of OSL counts at 160°C stimulation. While, the strength of OSL count significantly reduced in higher annealed samples.

While comparing deconvolution observations of OSL at ET between unannealed and annealed NSQ samples for beta doses, the annealing of NSQ samples at 400°C and 600°C increased the average OSL loss percentage than average OSL loss percentage of unannealed samples as a rapid decay. But, the 1000°C annealed samples controlled the rapid loss of OSL percentages. Also, the lower annealed samples enhanced the contribution of medium components of OSL over the contribution of medium components of OSL of unannealed samples. Whereas, the growth of slower OSL components of higher annealed sample found to be more than the growth of slower OSL components of unannealed sample. The lower annealed samples increased the PIC of all components of OSL decay curves over PIC of unannealed sample. However, higher annealed 1000°C samples decreases the PIC of all components of OSL decay curves over PIC of unannealed sample.

OSL-DRC (at 160°C) of samples was showed that the change of super-linear nature of OSL dose response of unannealed sample into sub-linear nature of dose response curves with thermal treatment.

Based on the TL glow peaks of thermally treated specimens, along with the OSL decay for the identical physical treatments, the significant findings were reported. ESR study of prepared samples also confirmed the obtained results. So it can be concluded that higher annealing temperature and higher beta doses showed significant changes in TL glow peaks. The higher annealing treatment forms better thermal stability for 220°C TL glow peak. Along with better TL dose response of 220°C glow peak, TL study after optical bleaching showed that the NSQ (Nano Synthetic Quartz) material gives the information about wider family of stable TL traps between 220°C and 235°C. This is attributed to the developed Ge centers at the cost of E_1 ' centers at higher anneal temperature in NSQ specimens. These findings were clearly confirmed with ESR results also. The higher annealing treatment found be supporting to increase the contribution of usual OSL components outcomes controlling the losses of OSL counts as a rapid decay. Corroborative studies of ESR and XRD also revealed that the thermal treatment affect significantly the centers in nano synthetic quartz specimen. Lower (400°C) annealing temperature is found to be suitable for significant strength in TL and OSL counts in NSQ sample. Such thermally treated NSQ may be the candidate for OSL dosimeter due to better stability of the center.