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THERMOLUMINESCENCE STUDY FOR DIFFERENT GRAIN SIZES OF ANNEALED SYNTHETIC QUARTZ MATERIAL

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Keywords: ***synthetic quartz, grain size, thermal treatment, beta dose, thermally stimulated luminescence***

Abstract

The thermally stimulated luminescence (TSL) glow curves have been recorded at room temperature for different grain sizes of annealed synthetic quartz material followed by beta irradiations. The significant shifting of higher temperature TL peak is observed from 357°C to 245°C with decrease in particle size from 125-106µm to 0.422µm of 600°C annealed sample. It is still appearing up to 45 µm in higher annealed sample of 1000°C material. Below 45µm of grain size, the higher temperature TL peak is disappeared. The TL peak of 110°C still exist both annealed samples. The TL intensity of 110°C and higher temperature peak are increased with decrease in particle size up to 45µm. It is significantly decreased by further reduced in grain size to 0.42µm.

1. Introduction

Researchers have reported that the TL intensity depend upon beta dose, annealing treatment, uv-illumination and particle size **[1]**.

In present paper the TL intensity is recorded to different grain sizes of annealed synthetic quartz. The noticeable changes are observed either in TL intensity or peak position by further decrease in particle size from 45µm to 0.422µm.

2. Experimental Details

Grinding:

(i) With Mortar and Pestle and prepared the grain size of 125-45µm

(ii) With Ball Milling (Planetary Mono Mill, Pulverisette-6, Fritsch, Germany) technique and prepared the grain size of 0.422µm; MBR(Material-Ball-Ratio) was 1:8, RPM-350; for 4hrs

Treatment:

The sample of 0.422µm
↓
Dissolve in HCL+ Washed with distilled water
↓
Filtered by paper and dried at 40°C.
↓
Sample was annealed at 600 and at 1000° for 1hour duration.

Instrument: Nucleonix PC based TL reader

3. Results and Discussion

The 600°C annealed sample shows the growth in TL intensity with decrease in particle size from 125µm to 45µm for 110°C and 350°C TL peaks. It is decreased with particle size to 0.422µm. The 350°C peak is shifted to 250°C with decrease in particle size by keeping an identical position of 110°C TL peak. The 1000°C exhibits an identical pattern of TL growth curve to 600°C. The strength in TL signal is quite noticeable rather than 600°C. The shifting of higher temperature TL peak is observed with particle size from 125µm to 45µm. It is disappeared by further decrease in sample to 0.422µm of grain size **Fig1-5**

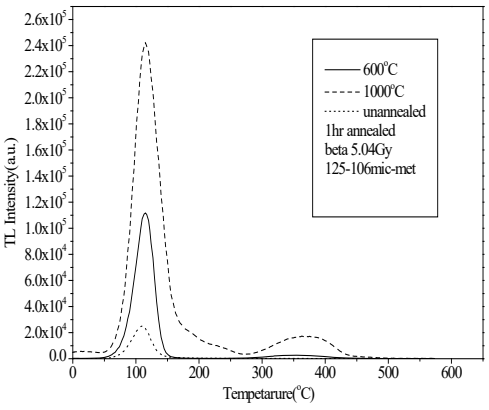

Fig.1 TL glow curve measured at room temperature for annealed and unannealed sample of 125-106mic-met.

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Optical and Photoluminescence Studies of Synthetic Quartz Nanoparticles Prepared by High Energy Planetary Ball Milling Technique

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Abstract— *In the present study, synthetic quartz nanoparticles were prepared by mechanical media milling process in a high energy planetary ball mill using micron sized synthetic quartz as starting material, acetone as medium and stearic acid as surfactant. Additionally tungsten carbide balls were used as milling media at 350 rpm with BPR (ball to powder weight ratio) 10:1 for total duration of 48 hours. The sampling was carried out at 6, 12, 24 and 48 hours. The collected samples were washed with diluted HCL followed by distilled water and methanol to remove impurities and dried in oven at 50°C for 4 hours. The prepared synthetic quartz nanoparticles were characterized by particle size analyzer, scanning electron microscopy, X-ray diffraction, UV-visible spectrophotometry and fluorescence spectrophotometry. The crystalline size of prepared nano-synthetic quartz was found to be about 39.41 nm. In UV-spectrophotometric characterization of the synthetic quartz nanoparticles, the optical band gap was observed in the range of 4.4-5.1 eV. In photoluminescence (PL) analysis, five strong and broad emission peaks were detected at 240 nm, 295 nm, 448 nm, 480 nm and 665 nm. The PL spectra of the samples showed blue light emission in near blue region using UV-excitation wavelength at 220 nm. The obtained results can be further used for dosimetric application of synthetic quartz.*

Keywords— *Synthetic quartz nanoparticles, Photoluminescence, UV-visible spectroscopy.*

1. INTRODUCTION

Materials in nanometer size exhibit a remarkable amount of variation in electronic, magnetic, optical and chemical properties of a molecule that are significantly different from those of the bulk.[1-2] The desirability criteria of a nanomaterials are its submicron size, narrow size distribution, high level of dispersability and low extent of agglomeration.[3] Various methods have been used for preparation of ceramic or metal nanoparticles such as mechanical alloying, combustion synthesis, plasma forming, explosive forming, electro deposition, sol-gel technique and media milling/ball milling process.[1, 4-5] Among these methods, high energy ball milling (BM) is favourable technique widely used in production of nano particles due its simplicity, user friendly operation, low cost of production and applicability to any class of materials at large quantities.[6] Several workers have used BM method for the synthesis of nano-particles of different martials like Fe_2O_3-SnO_2, ZrO_2-Fe_2O_3, TiO_2-Fe_2O_3, TiO_2-WO_3, SiC, Si_3N_4 and quartz (SiO_2).[7-10] Literature revealed that nanoparticles of natural quartz have been prepared and their optical properties have been studied, [11-13] but no research work have been carried out on preparation of synthetic quartz nanoparticles and its optical properties. Natural quartz is a brittle, hard, water-resistant and chemically stable material with twins and other imperfections whereas laboratory grown synthetic quartz is pure, without any twins and impurities. Therefore, synthetic quartz is used for the optical studies under different physical conditions for dosimetric applications.

In present article, synthetic quartz nano-particles have been prepared by high energy planetary BM process for different durations. The characterization of nano-synthetic quartz material was carried out by particle size analyzer, XRD and SEM. The optical properties were studied by UV-Visible and photoluminescence (PL) analysis.

2. EXPERIMENTAL DETAILS

2.1 Sample Preparation

Synthetic quartz nanoparticles were prepared by a high energy planetary BM (FRITSCH Planetary Mono Mill PULVERISETTE 6 Classic Line, Germany) process using micron sized synthetic quartz as starting material, acetone as dispersing medium and stearic acid as surfactant. Additionally tungsten carbide balls were used as milling media at 350 rpm with BPR (ball to powder weight ratio) 10:1 for total duration of 48 hours. The sampling was carried out at 6 (SQ 1), 12 (SQ 2), 24 (SQ 3) and 48 (SQ 4) hours. The collected samples were washed with diluted HCL followed by distilled water and methanol to remove process impurities and dried in oven at 50°C for 4 hours. The dried samples were used for further investigations.

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A STUDY ON CORRELATION AMONG PARTICLE SIZE, UV-ABSORPTION AND PHOTOLUMINESCENCE OF SYNTHETIC QUARTZ

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Abstract

In this study, different micron size samples were prepared by grinding the synthetic quartz crystal using metallic mortar pestle and nano size samples were prepared by mechanical media milling process in a high energy planetary ball mill. The prepared samples were characterized by Particle size analyzer, Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), Fourier-transfer infrared spectroscopy (FTIR) and Energy Dispersive Spectroscopy (EDS). The micron and nano-sized synthetic quartz samples were further analysed using UV-visible spectrophotometry and fluorescence spectrophotometry. The impurities and defects were examined by FTIR. EDS was performed to detect any in-process impurity and to confirm that elemental composition of the prepared micron and nano sized samples. Particle size analyzer, TEM and XRD confirmed the nano size of prepared synthetic quartz samples. In UV-visible spectrophotometric study, one absorption peak was observed in all micron-size samples in 210-235nm range whereas in nano size sample, a broad absorption peak was seen in 210-480 nm range. In photoluminescence analysis, PL intensity systematically increased with decrease in particle size from 105 µm to 53 µm and reduced with further decrease in particle size from 53 µm to 39.41nm.

Keywords: *Synthetic quartz nanoparticles, UV-visible spectroscopy, Photoluminescence.*

1. INTRODUCTION

Natural quartz is a brittle, hard, water-resistant and chemically stable material with twins and other imperfections, whereas laboratory grown synthetic quartz is pure, without any twins and impurities. Therefore, synthetic quartz is used for the optical studies under different physical conditions for dosimetric applications. Synthetic quartz crystals also have attractive properties for development of various electronic and optical devices, where it is exposed to different optical backgrounds. In recent advancements in research and development, many applications are in control of particle size, morphology, size distribution and dispersion [1]. Materials in nanometer size exhibit a remarkable amount of variation in electronic, magnetic, optical and chemical properties of a molecule that are significantly different from those of the bulk [2]. So it is essential to study the variation in optical properties of synthetic quartz due to changes in particle size and morphology.

In present study, synthetic quartz samples in micron- and nano-size have been prepared by mortar-pestle and high energy planetary BM process, respectively. The characterization of prepared synthetic quartz samples was carried out by particle size analyzer, TEM, XRD, FTIR and EDS. The optical properties were studied by UV-Visible spectrophotometric and photoluminescence (PL) analysis.

2. EXPERIMENTAL DETAILS

2.1 Sample Preparation

2.1.1 Micro-sized sample preparation:

Micron-sized synthetic quartz samples were prepared by grinding the samples using metallic mortar-pestle. The crushed samples were passed through the different sieves having different mesh size (i.e. ASTM No.- 120, 140, 170, 200 ,230, 270, 325, 400, 500), to achieve the samples of desired particle size [125-105µm (SQ1), 105-90µm (SQ2), 90-74µm (SQ3), 74-63µm (SQ4), 63-53µm (SQ5), 53-44µm (SQ6), 44-37µm (SQ7), 37-25µm (SQ8)].

2.1.2 Nano-sized sample preparation:

Synthetic quartz nano-size sample was prepared by a high energy planetary BM (FRITSCH Planetary Mono Mill PULVERISETTE 6 Classic Line, Germany) process using micron sized synthetic quartz as starting material, acetone as dispersing medium and stearic acid as surfactant. Additionally tungsten carbide balls were used as milling media at

The Effect Of Grain Size And β-Radiation Dose On Optically Stimulated Luminescence Of As-Received Synthetic Quartz

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Abstract-In the present investigation, the effect of grain sizes of as received synthetic quartz samples and β-radiation dose on optically stimulated luminescence (OSL) were studied. The batches of different grain sizes (74-63μm, 63-53μm, 53-44μm, 44-37μm, and 37-25μm) were exposed by different β-radiation prior to OSL measurements at room temperature. For each grain size, an exponential shape of OSL decay curve was observed and maximum optically sensitive trap was turned to empty within 0 to 0.4 sec. Results indicated that the OSL intensity was increased with decrease in grain size from 74μm to 53μm whereas for the grain sizes below 53μm, the OSL intensities were started to decrease for each exposed sample. The variation in OSL intensity with grains sizes were due to the combined effect of the change in surface area and strength of radiation dose. The results were further elaborated by Fourier Transform Infra-Red (FTIR) analysis of prepared samples. The dose response curves were plotted and studied over the β-radiations from 2.63Gy to 131.33Gy for each grain size. Each grain size showed the supralinear behaviour of OSL signal over the dose range of 55.16 Gy to 131.33 Gy.

Index Terms- Synthetic quartz, grain size, β-radiation, OSL, FTIR, PACS No. – 78.60.Lc

1 INTRODUCTION

Since last decade, optically stimulated luminescence (OSL) technique had become a popular tool for the dating of archaeological and geological materials (natural quartz, Feldspar, Al_2O_3:C etc.) in comparison to of Thermoluminescence (TL) technique [1]. The OSL technique was first implemented by Huntley et al. (1985) for the optical dating of sediments [2]. Studies showed that materials can be used in powder form for OSL study in dosimetry and dating purpose. Literature also revealed that the new active/inactive TL and OSL sites were observed due to increased surface area after reduction in grain size of sample by grinding with mortar and pestle [3]. Researchers explained the effect of grain size on TL response of non-sensitized and sensitized natural quartz by the increase in specific surface area with decrease in grain size [4]. It was reported that the response of TL and OSL curve with respect to Dose was an essential factor in any dating procedure. These findings could be used to estimate the equivalent dose in dating [5]. It was also observed that the OSL intensity of synthetic quartz was influenced by various physical conditions such as radiation dose, annealing temperature, annealing period and elevated temperature [6], [7], [8].

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But there is no literature available for the comparative study on the effect of different radiation dose on different grain size for OSL intensity of synthetic quartz. The aim of present study was to understand the effect of grain size and different β-radiation dose on OSL intensity of as received synthetic quartz at room temperature for dating application. Five different grain size (74-63μm, 63-53μm, 53-44μm, 44-37μm, and 37-25μm) of synthetic quartz were prepared by using mortar pestle. OSL intensities were measured for every grain size irradiated with different β-radiation. The dose response curves were plotted and analysed over the radiation dose range from 2.63 Gy to 131.33 Gy for each grain size. The obtained results have been supported by corresponding changes in Fourier Transform Infra-red spectra of prepared synthetic quartz samples.

2 EXPERIMENTAL DETAILS

2.1 Method of sample preparation

The synthetic quartz sample was kindly provided by CGCRI, Kolkata. Five different sizes of as received synthetic quartz sample were prepared by grinding the sample using mortar-pestle. The crushed sample was passed through different sieves having different mesh size, to achieve the samples of desired grain sizes [i.e. 74-63μm, 63-53μm, 53-44μm, 44-37μm, and 37-25μm].

2.2 Characterization

Each grain size sample was exposed to different β-radiation dose (2.63 Gy, 15.76Gy, 28.89Gy, 55.16Gy, 81.43 Gy, 107.69 Gy, and 131.33 Gy) at the dose rate of 7.88Gy/min at room temperature. Then OSL decay curve were recorded for 0 to 100 sec at room temperature by using Risø TL/OSL reader (model TL/OSL-DA-20) having 470 nm laser line or broad band stimulation wavelength. OSL intensity was normalized by weight for each grain size sample. Fourier transfer infrared (FTIR) spectroscopy [MIRacle10 Single Reflection ATR Spectroscopy (IRAffinity-1) Shimadju] was used for the examination of impurities and defect in the prepared sample.