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STUDY OF GAMMA IRRADIATION EFFECT ON SOL-GEL DERIVED LITHIUM BORATE GLASSY FILM BASED METAL INSULATOR SEMICONDUCTOR (MIS) STRUCTURE

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ABSTRACT
This work presents a structural investigation of lithium borate (LBO) thin film. Firstly, 100 nm SiO_2 was grown on top of n–type Si substrate by dry oxidation. Secondly, LBO gel was prepared by sol–gel method. After it, thin film of LBO was deposited on functionally graded SiO_2/Si stack by spin coating, and then Raman and infrared spectra were studied for different thickness of the LBO film. Gold dot was deposited on top of the LBO film for electrical characterization. C–V measurements were carried out on MIS structure. Finally, influence of gamma irradiation was studied to investigate the radiation hardness of the deposited film.
Keywords: Metal-insulator-semiconductor (MIS) structure; Sol-gel; Raman & FTIR spectroscopy; C-V measurement; Gamma irradiation

I. INTRODUCTION

Conventionally, thermal neutrons are detected by ^{3}He based gas proportional counter. However, scarcity of ^{3}He, requires alternative technology to detect thermal neutron [1]. Recently, significant amount of works are carried out on Schottky diode and scintillator based thermal neutron detectors [2-6]. Thermal neutrons (charge less particles) cannot be detected by Scintillator or Schottky diode directly. However for detection of thermal neutron using scintillator or Schottky diode technology, first it must be converted into ions e.g. $^3H^{+1}$, $^4He^{+2}$ etc. Fissile material like ^{6}Li and ^{10}B isotope enriched compounds are used to convert thermal neutron into charged particles by following nuclear reactions [4-6].

$$^6Li+n\rightarrow {}^3H+{}^4He$$
$$^{10}B+n\rightarrow {}^4He+{}^7Li$$

Generated ions incident on the Schottky diode or Scintillator and subsequently creates electron-hole pairs (EHPs) or photons. By measuring EHPs or photons, neutron signature can be detected. Although there are some limitations in these kinds of detectors like low detection efficiency, consequential amount of work have been carried out by few research groups on perforate Si based thermal neutron detectors to enhance overall detector efficiency [7]. However challenge in this kind of detectors is to fill pores with fissile materials (^{6}Li, ^{10}B isotope enrich compound) using spattering technique. To overcome this challenge we propose a novel sol–gel chemical route to fill pores of perforated Si by ^{6}Li and ^{10}B based lithium borate (LBO) gel.

In this report, properties of a novel LBO film realized by sol–gel method were investigated. Spectroscopic (Raman and FTIR) analysis were carried out for structural characterization of the synthesized LBO film. Film surface morphology was investigated by SEM image analysis. Subsequently, CV characterization was done for different thickness of the LBO glassy film to find out dielectric constants. Finally gamma irradiation effect on LBO film was studied to examine the radiation hardness of the film.

II. EXPERIMENT

The experimental methodology followed by us, was two-step process, (1) Lithium borate gel preparation and (2) MIS structure fabrication.

II.1 Gel preparation

Lithium borate (LBO) gel was prepared by supersaturated aqueous solutions of lithium hydroxide mono hydrate and boric acid as (precursors) [8]. First, 550 mg of lithium hydroxide monohydrate was dissolved into 10ml DI water and stirred for 30 min at 50^{0}C. After

getting a clear transparent solution, 1.625 g of boric acid was added into it and stirred for another 30 min at 50^0C to realize lithium borate gel.

II.2 MIS structure fabrication

An n–type (100)–oriented 2" Si wafer of resistivity 4 ohm–cm was taken as substrate to carry out subsequent experiments. Substrate was chemically cleaned by a standard RCA process. 100 nm oxide layer was grown on the substrate by dry oxidation at 1150^0C. SiO_2 film was grown to prevent material diffusion for LBO layer. Next lithium borate gel was spin coated on top of SiO_2/Si structure and dried at 100^0C in atmospheric ambient. By varying the rotation speed of the spin coater films with different thicknesses were achieved. The metallic contacts were obtained for MIS structure by depositing Au dot on top of LBO film by sputtering technique (Nordiko).

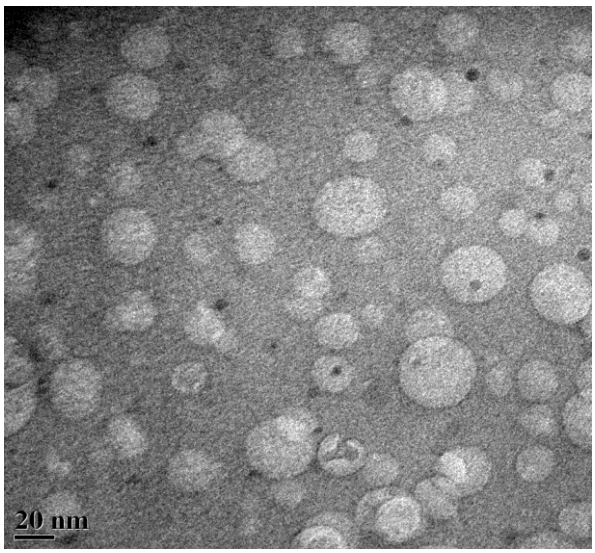

Figure 1: Transmission Electron Micrograph of the lithium borate gel.

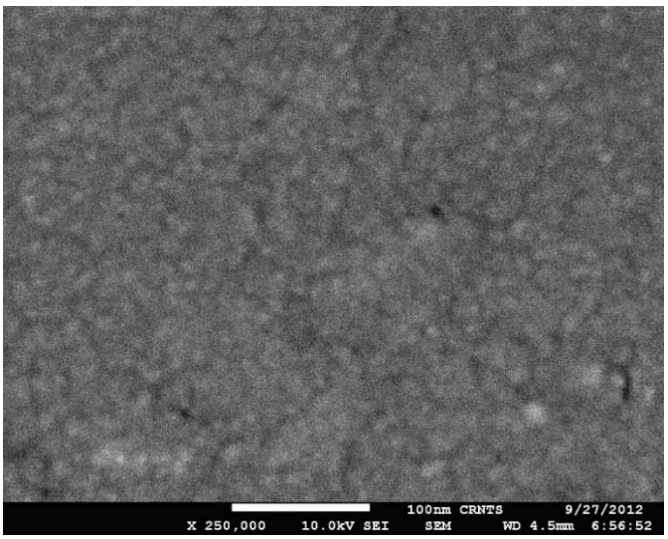

Figure 2: Scanning Electron Micrograph of the lithium borate film.

III. RESULTS AND DISCUSSIONS

III.1 Structure of Lithium borate glassy films

III.1.1 TEM & SEM image analysis

Figure 1 shows a transmission electron micrograph of lithium borate particle synthesized by sol–gel method. The particle size distribution is found to be 15 nm to 40 nm of spherulitic shape. The surface morphology of lithium borate film was investigatedby scanning electron micrograph as displayed in figure 2. According to this figure the surface of lithium borate film is not very smooth, however compact highly dense nano cluster stack of size 35 nm to 60 nm is formed. Hardly any micro size crack were found there.

III.1.2Raman spectroscopy

Raman spectra of LBO glass films with various thicknesses are shown in figure 3. The following bands are present in these spectra: 620 cm^{-1}, 700 cm^{-1}, 790 cm^{-1}, 960 cm^{-1} and 1060 cm^{-1}. Around 790 cm^{-1} peak is due to the trigonal deformation of BO_4 units as di–, tri–, tetra– borate [9]. Raman peak at 790 cm^{-1} decreases with the increase of deposited LBO film thickness which implies the decrease of BO_4 ratio in the LBO matrix. Peak at 960 cm^{-1} is caused by the presence of non–bridging oxygen of pyroborate $[BO_3]^{-}$. Peak around 1060 cm^{-1} is observed due to the Si–O stretching mode vibration [10].

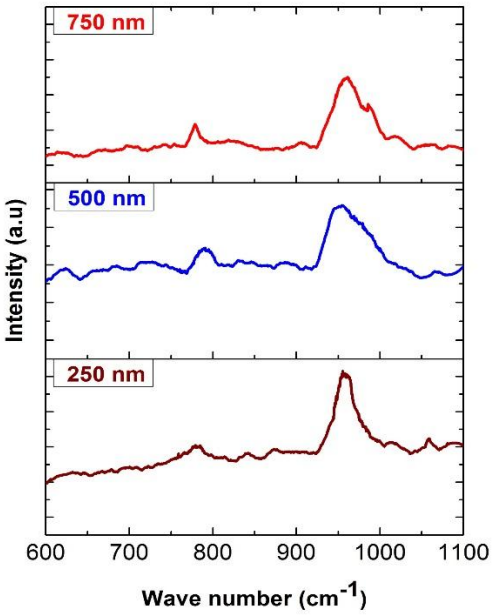

Figure 3: Raman spectra of the lithium borate glassy film on SiO_2/Si structure with varying thickness.

III.1.3 FTIR spectroscopy

The FTIR transmittance spectra of the LBO film with different thickness are recorded in figure.4

Usually the IR spectra of borate glasses are active in 600–750cm^{-1}, 800–1200cm^{-1}, and 1200–1600cm^{-1} regions which are attributed to the B–O–B bond bending vibration, B–O bond stretching vibration of tetragonal BO_4 groups and asymmetric stretching of the B–O band of triangle BO_3 groups respectively [11]. Peak around 1074 cm^{-1} was found due to the BO_4 stretching vibration. With increasing

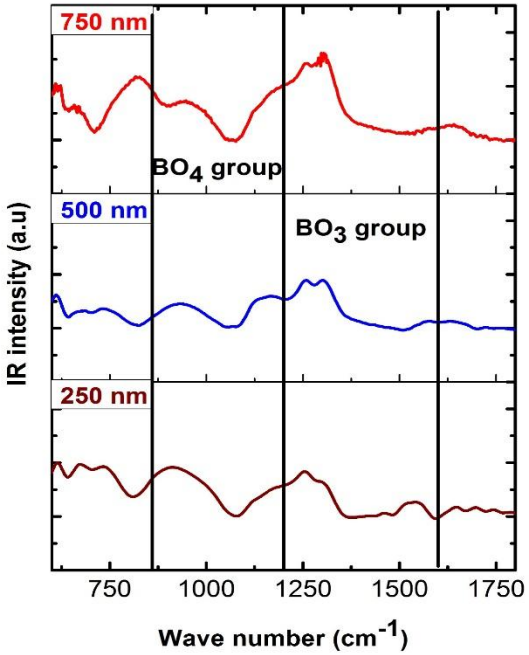

Figure 4: FTIR spectra of the lithium borate glassy film on SiO_2/Si structure with varying thickness.

thickness of the LBO film BO_3 groups are found to be increasing. The band around at 800 cm^{-1} is assigned to the B–O–B bending vibration of bridges containing one trigonal and one tetrahedral boron. Further peak around 1360 cm^{-1} are designated to the meta– borate chain formation of BO_4 on the expense of BO_3 with non–bridging oxygen (NBO).

III.2 Electrical characterization

III.2.1 CV characterization:

C–V measurements of lithium borate film based MIS structure were carried out in light tight enclosure to prevent measurement errors due to extraneous photo–generated currents (Pheonix). High–frequency (40 kHz.) C–V measurements with different dielectric thickness of lithium borate film are shown in fig. 5. Dielectric constant was determined by assuming the dielectric constant of the SiO_2 is 3.9. Thickness of the SiO_2 (t_{SiO2}) and LBO (t_{LBO}) films were measured by ellipsometer (Sentech) and profilometer (Ambios–XP2) respectively. Dielectric constant of the LBO film was found to be around 7.46. However dielectric constant of the LBO film was found to be slightly increasing with increasing thickness. The change of dielectric constant of the film with increasing thickness is due to the reduction of the interfacial dead–layer effect [12-14].

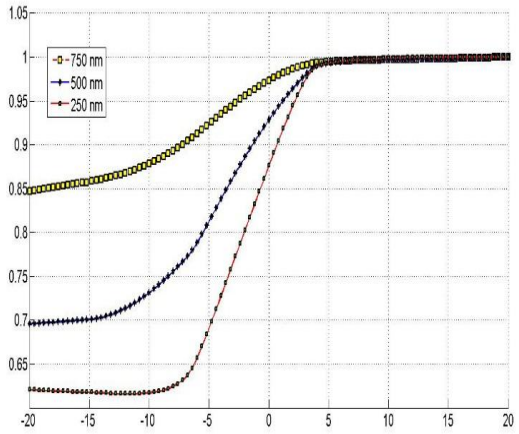

Figure 5: HFCV of lithium borate glassy film based MIS structure with varying film thickness.

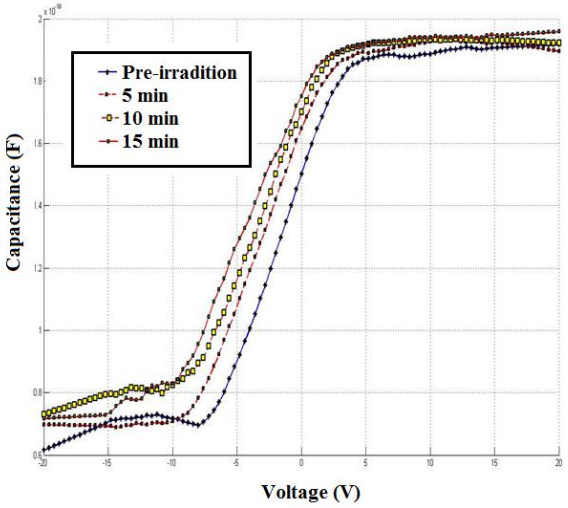

Figure 6: CV shift of lithium borate glassy film based MIS structure due to gamma irradiation.

III.2.2 Influence of γ–irradiation on the MIS structure:

In order to study the response of MIS capacitors to γ–irradiation in a wide range of dose, the 150 nm thick LBO samples were irradiated using the Co–60 γ–source of dose rate 0.5 Gy/min for different time. Fig. 6 shows the high–frequency C–V curves of MIS capacitor before and after γ–irradiation. For LBO based MISCAP structure after irradiation for 5 min the flatband voltage shift is about 1.2 V. However, with increasing irradiation up to 10 min and 15 min flatband voltage shift was found to be 0.4 V only. This result indicates that samples have radiation hardness properties at a limit after exposure to gamma irradiation [15].

IV. CONCLUSION

In this work, we investigate the particles size of the sol-gel derive lithium borate. Surface morphology of the deposited LBO film was studied by SEM image analysis. Raman and FTIR spectroscopic analysis shows conformation of lithium-borate glass thin film. Raman spectra reveled that with increasing LBO film thickness BO_4 unit decreases and non-bridging oxygen atom increases. These LBO film thickness dependent structural results of are consistent with the FTIR spectra. Next C-V characterization was carried out on the LBO based MIS structure. Dielectric constant of the LBO film was found to be changing with the thickness of the LBO film due to effect of interfacial dead–layer. Finally γ–irradiation effect on the LBO film was studied. Due to γ–irradiation for 5 min on 150 nm flat band voltage shift was found to 1.2V. However with increasing the irradiation time the flat band voltage shift was found to be decreasing which shows radiation hardness LBO after a limit of exposure.

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