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Thermal, Optical, and Spectroscopic studies of gel grown Strontium magnesium oxalate crystals

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Abstract : The Thermal, Optical and Spectroscopic studies of Strontium Magnesium Oxalate (SrMgO) crystals were discussed in the present paper. The crystals were grown by silica hydro gel technique. Grown crystals were optimized by different growth parameters. Crystals were examined under various characterizations. The cations present in the crystals Sr^{2+}, Mg^{2+}, C and O identified by Energy Dispersive X-Ray Analysis and confirms the percentage of composed elements. Field Emission Scanning Electron Microscope reveals surface morphology. Thermal studies of grown crystals were carried out by Thermo Gravimetric Analysis, DSC and DTG. The functional groups were identified by employing Fourier Transmission Infrared spectrometer and Raman spectrometer. Absorption coefficient, Band gap energy, Refractive index and electrical susceptibility were computed by UV-Visible spectrum of the crystals. Internal arrangements of atoms in the grown crystals were investigated by X-Ray Diffraction technique.
Keywords : Silica hydrogel, TGA/DSC/DTG, UV-Visible spectrometer and X-Ray diffraction technique.

Introduction

Growth and characterization of single crystals receive increasing importance due to their various applications in solid-state technology and laser technology. With the absence of crystals, there would be no electronic industry and fiber optic communication. Growth of crystals by gel method is a promising technique for growing single crystals of substances which are sparingly soluble in water and decompose before their melting point [1-5]. Now a day's huge attention towards the growth of oxalate crystals because of their enormous applications [6-9]. Oxalate crystals possess applications like dielectric, ferroelectric, piezoelectric and nonlinear optical properties hence they are used in transducers, oscillators, resonators, linear and nonlinear devices [10, 11]. In the present paper growth and spectroscopic investigations of oxalate crystals are discussed in detail. Grown crystals are analyzed and subjected to various characterizations. X-ray powder diffraction

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(XRD) analysis reveals the internal structure of the grown strontium magnesium oxalate crystals. Thermo-gravimetric analysis (TGA) measures the mass of the sample and also measures phase transition and thermal decomposition. Fourier Transform infrared spectroscopy (FTIR) and Raman Spectrum identifies different functional groups associated with the grown crystals. Field Emission Scanning Electron Microscope gives the surface morphology. UV-Visible spectrum used to calculate absorption coefficient, Energy band gap, Reflection, Refractive index, electrical susceptibility.

Materials and methods

Strontium Magnesium Oxalate crystals were grown by silica hydro gel technique. Silica gel was prepared by mixing of Sodium Meta Silicate (SMS) solution with oxalic acid solution. The solution of SMS was prepared by dissolving Sodium Meta Silicate with distilled water. Oxalic acid solution was prepared by dissolving oxalic acid ($C_2H_2O_4$) with 250ml of distilled water. The concentration of oxalic acid was 0.5N. The mixture of SMS solution and solution of oxalic acid was stored in the different test tubes of diameter 1.5cm and length of 12cm. After 5 days gel was set in the test tubes. Mixture of magnesium chloride solution and Strontium chloride solution added to the set gel drop by drop. The concentration of Magnesium chloride solution and Strontium chloride solution was 0.5N. The mixture diffuses through set gel and nucleation starts in the gel, after 15 days crystals were extracted with good morphology. Grown crystals were transparent.

Results and Discussion:

Crystal growth

Strontium Magnesium Oxalate crystals were successfully grown by silica hydro gel technique. Various growth parameters like gel density, concentration of oxalic acid solution (0.5M), concentration of strontium chloride solution (0.5M) and magnesium chloride solution (0.5M), gel set time was optimized. Gel density was observed 1.038g/cc. Time taken for to gel set was 5 days. Crystals were grown in 15 days. Grown crystals were transparent and perfect.

Figure 1. Growth phase and extracted SrMgO crystals

Field Emission Scanning Electron Microscope (FESEM)

Field Emission Scanning Electron Microscope reveals the internal structure of grown crystals. Strontium Magnesium oxalate crystals were show large number of grains of different size [12]. In higher magnification tetragonal and diamond shape structure share were observed. Grown crystals show well defined boundaries [13, 14].

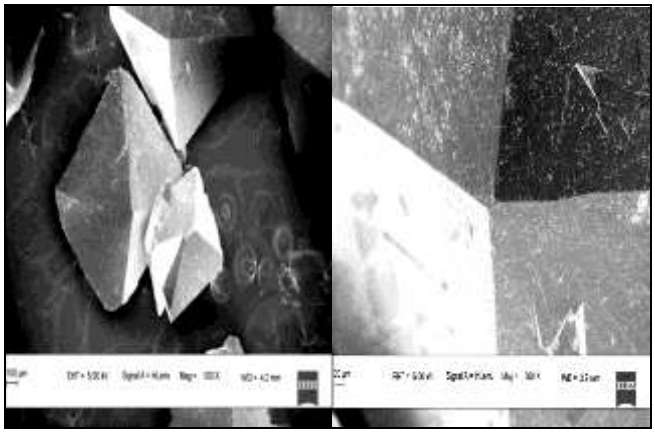

Figure 2. FESEM images of SrMgO crystals

Energy Dispersive X-ray Analysis (EDAX)

Energy dispersive X-ray Analysis identifies the elements present and determines atomic and molecular weight percentage. The Strontium Magnesium oxalate crystals contain Magnesium (Mg), Strontium (Sr), Carbon (C), Oxygen (O) elements. The atomic weight percentage of Magnesium is 0.02, Strontium 7.37, Carbon 30.87, and Oxygen 61.74. The weight percentage of Magnesium is 0.03, Strontium 50.99, Carbon 29.29, and Oxygen 78.04 [15-17].

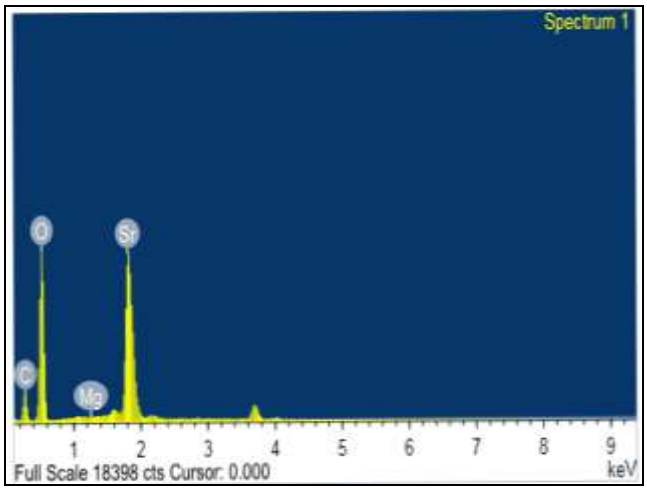

Figure 3. Electron spectra of SrMgO crystals

Thermal studies

The thermal stability of strontium Magnesium oxalate crystals were investigated by thermo gravimetric (TG) studies. Dehydration begins in the first stage at 67.23^{o}C and terminates at 245.80^{o}C. The loss of water molecules observed in the first stage leads 18.75% weight loss [18-20]. Decomposition of CO showed 12.47% weight loss in the second stage. In the third phase loss of CO_2 molecules leads 17.72% weight loss [21,22]. The endothermic peak at 226.14 ^{o}C and exothermic peak at 489.25 ^{o}C shown in the DSC curve. The maximum reaction rates of grown crystals were 217.46^{o}C, 476.08^{o}C, 990.20^{o}C. The chemical formula for grown crystal was $SrMgC_2O_4.2.25H_2O$ and molecular weight 215.99 g/mol [23].

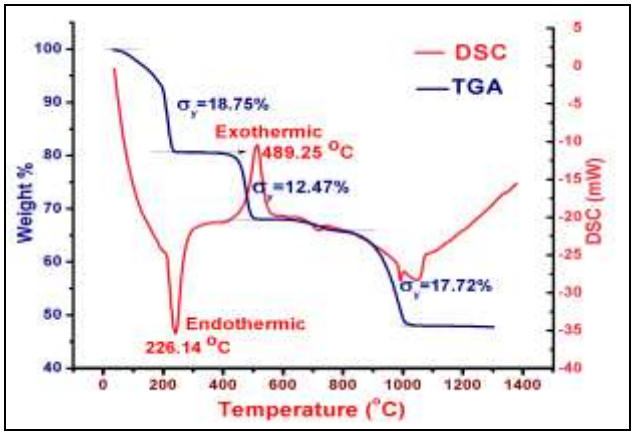

Figure 4. TGA/DSC curves of SrMgO crystals

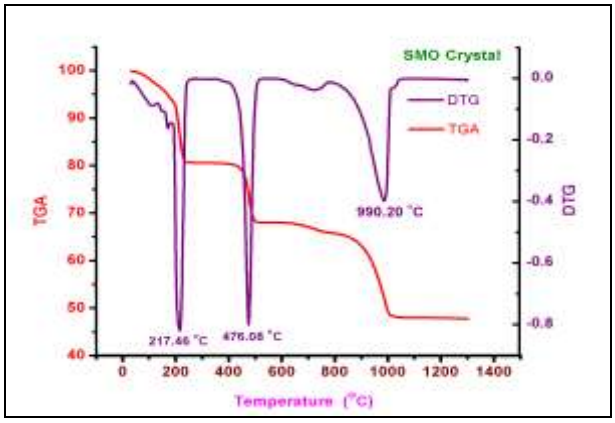

Figure 5 TGA/DTG curves of SrMgO Crystals

Fourier Transformation Infrared spectrum

The Fourier Transformation Infrared spectrum of Strontium Magnesium oxalate crystals was shown in figure 6. The hydrous nature of the compound observed between 3809.48cm^{-1} to 3176.72cm^{-1} it shows the O-H stretching vibrations due to water crystallization associated with the crystals [24,25]. Peak observed at 2775cm^{-1} assigned to the asymmetrical C-H stretching vibration of grown crystals [26]. Peaks observed from 2614.65cm^{-1} to 1999.13cm^{-1} attributed to the asymmetrical stretching vibrations of C-H bonds. The band at 1606.89cm^{-1}is strong and assigned to the C=O of carbonyl group of MSO crystals. Asymmetric stretching of C-O group observed at sharp peak 1321.51cm^{-1}[27]. The absorption bands from 768.9655cm^{-1} to 430.1724 cm^{-1} were assigned to Metal-Oxide stretching modes.

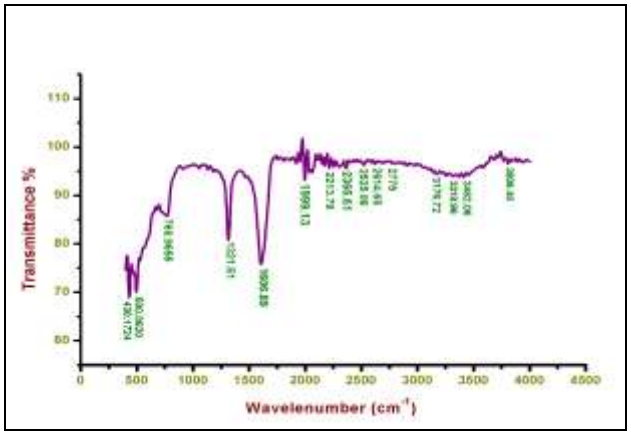

Figure 6. FTIR spectra of SrMgO crystals

Raman Spectrum

The Raman spectrum was obtained by the Horiba scientific instrument in the region 0 to 4000cm^{-1}with a 532nm excitation source. The spectrum exhibits a less intense peak at 3428.63cm^{-1} which is assigned to O-H stretching vibration of hydrogen bonds [28]. The peak corresponds to 1626.63cm^{-1} and most intense 1470cm^{-1} is due to C-O band. Raman band is observed at 912.07cm^{-1} is assigned to the vibration stretching of C-C band [29]. The peaks at 481.440cm^{-1}, 158.71cm^{-1}, 60.28cm^{-1} were vibrations metal-oxygen bands [30].

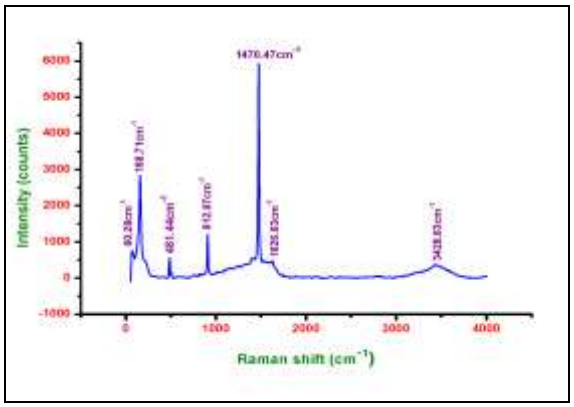

Figure 7. Raman Spectra of SrMgO crystals

UV Visible Spectrum

The optical parameters of SrMgO crystals were determined by UV and Visible spectrum of SrMgO crystals were recorded in the wavelength region 190nm to 1100nm. The absorption spectra grown crystals shows lower cutoff wavelength 227.01nm [31]. The crystals have absorption 2.8 at the wavelength 198.33nm. Lower cutoff wavelength indicates that grown crystals allows the transmission of light radiations. Band gap energy of the crystals were 5.7877eV shown by Tauc's plot [32, 33].

Absorption coefficient of crystals were calculated by $\alpha=2.303A/t$ hence $\alpha=6.4484$.

Refractive index of crystals calculated $E_g e^n=36.3$

Hence n=1.836

The electrical susceptibility determined by using relation $\chi_e= \varepsilon_r-1$

$\chi_e=n^2-1$

$\chi_e=2.370$

The electrical susceptibility is greater than 1, it indicates grown crystals can be easily polarized.

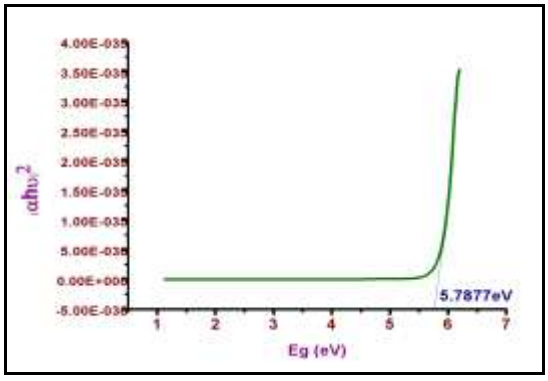

Figure 8.Absorption spectra of SrMgO crystals

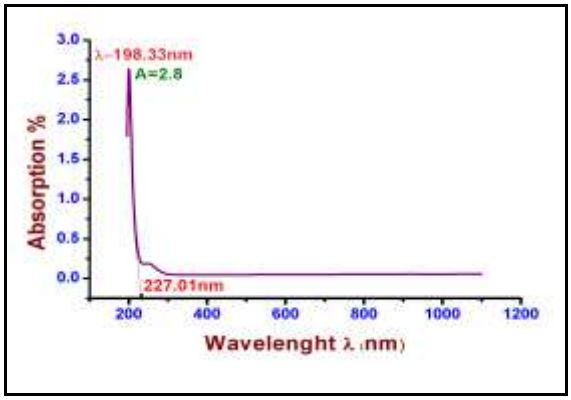

Figure 9. Tauc's plot of SrMgO Crystals

X-Ray Diffraction

X-ray diffraction technique is used to investigate the internal arrangement of atoms or molecules in a crystalline material. The X-ray powder diffraction pattern of the grown SrMgO crystals was obtained using Bruker AXSD8 Advance model Diffractometer with Copper (k alpha 1) radiation of wavelength 1.54056Å, operating at a voltage of 40 kV and a current of 20 mA. The scanning rate was maintained at 1.6° /min over a 2θ range of 10-70° employing the reflection mode for scanning. Well defined peaks at specific 2θ values show high crystallinity of the grown crystals. All the reflections of the powder XRD pattern were indexed using the INDEXING and NTREOR software packages. These results are well in agreement with JCPDS data card no (00-045-0743). The indexed powder X-ray diffractogram of SrMgO crystals is shown in Fig.10

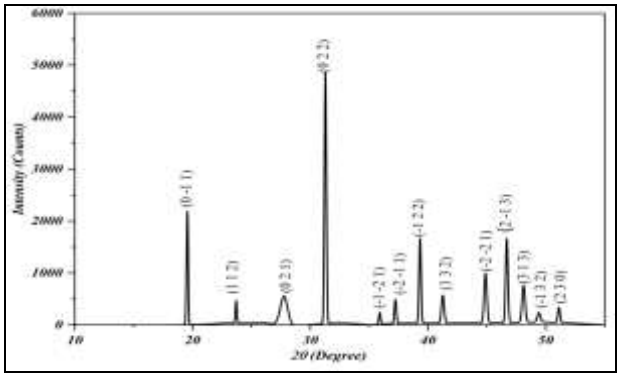

Figure 10. Powder XRD of SrMgO crystal

Conclusion

Gel growth method yielded optically transparent single crystals. Crystals were optimized by various growth parameters. The grown crystals have been subjected to various characterizations. EDAX studies reveal the atomic and weight percentage of grown crystals. Surface analysis confirmed by FESEM. FTIR and Raman spectrum identifies functional groups present in the grown crystals. UV-Visible spectrum determines band gap energy, absorption coefficient, refractive index, electrical susceptibility.

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