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Growth, Thermal and Optical Studies of Zinc Cadmium Thiocyanate Single Crystal (ZCTC)

S.Cynthia[1], P.Sagayaraj[1], B. Milton Boaz[2]*

[1]Department of Physics, Loyola College, Chennai, Tamilnadu, India.
[2*]Department of Physics, Presidency College, Chennai, Tamilnadu, India.

*Corres.author: miltonboazcm@yahoo.co.in
Ph: 09445383612

Abstract: Zinc Cadmium thiocyanate single crystal was grown from aqueous solution by slow cooling technique. The lattice parameters of the crystal have been obtained by single crystal X ray diffraction analysis. Kurtz-Perry test confirms the emission of green radiation from the sample thus exhibiting SHG. Thermal stability and decomposition process were studied by means of TGA and DTA analysis. The surface morphology and grain size are characterized via Atomic Force Microscope, showing the image of the crystal to be uniform and crack-free. The growth features of ZCTC crystals were observed by Field Emission Scanning Electron Microscopy technique. Epifluorescence studies reveal the excitation of the sample around 540nm.
Key words: Organometallic, Thermal, Epifluorescence, Single crystal XRD.

1. Introduction

Nonlinear optical properties have been the subject of numerous investigations by experimentalists in recent years due to the potential applications in optical signal processing and computing. Due to high second order non-linearity, bimetallic ligand based crystals are gaining much attention. Based on the double-radical structural model, a series of efficient metal organic crystals have been reported [1]. In the present paper, ZCTC single crystals have been successfully synthesized and grown by slow cooling technique.

2. Materials and Methods

Single crystals of ZCTC were grown by slow cooling technique using the commercially available $ZnCl_2$, $CdCl_2$ and KSCN from double distilled water. ZCTC have been prepared by taking the raw material in 2:1:1 stoichiometric ratio and synthesized based on the following reaction:

$$ZnCl2 + 4KSCN + CdCl2 \rightarrow ZnCd(SCN)4 + 4KCl$$

About 250 ml of the solution recrystallized with methanol was taken in a beaker and placed in a constant temperature bath. Crystals were grown by slow cooling method by reducing the temperature from 40℃ at the rate of 0.2℃ per day. The solution was periodically inspected and the crystal formation was seen at the

end of the 15th day. Further, the nominal size of the crystal was obtained when the observation was continued for another 11 days. The grown crystal was obtained within a period of 26 days as shown in Figure 1.

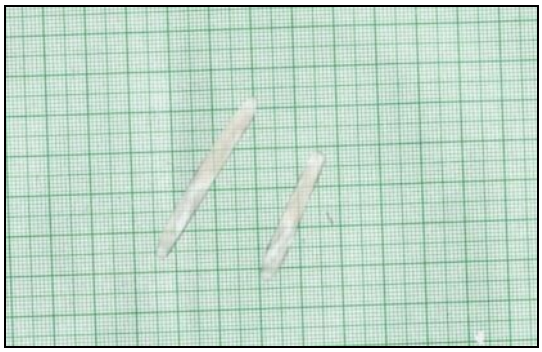


Fig1. As grown ZCTC single crystals

3. Results and Discussion

3.1 Second Order non-linear optical property test

The second harmonic generation efficiency of materials crystallized in non-centrosymmetric crystal structures has been confirmed by the most widely used Kurtz and Perry technique. The sample was tested using Q switched Nd:YAGlaser beam of wavelength 1064nm, Quanta ray series. An input power of 6.8mJ/pulse and pulse width of 8ns with a repetition rate of 10Hz was used. Bright green radiation confirms the second harmonic generation property of the crystal and its conversion efficiency was found to be 2.3 times as that of standard KDP [2].

3.2 Single Crystal X-ray diffraction analysis

The crystal system and lattice parameters were determined by single crystal X-ray diffraction analysis using X-ray diffractometer ENRAF NONIUS CAD 4. The results obtained from the instrument shows that the crystal belongs to the tetragonal system. The determined cell parameters are a= 12.0624 Å, b= 12.145 Å, c= 8.762 Å; α=89.634°, β= 90.143°, γ= 88.73°, V= 1273 (Å)^3 and are in good agreement with the reported value [3].

3.3 Thermal analysis

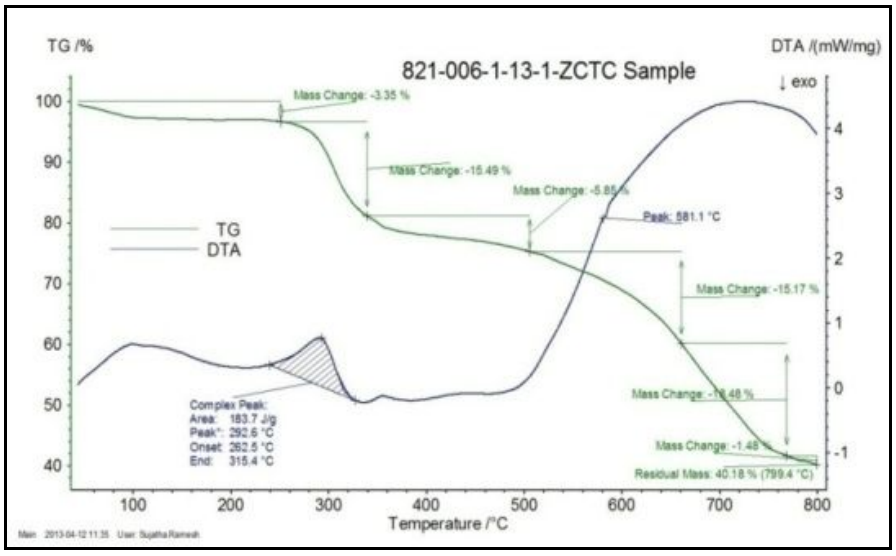


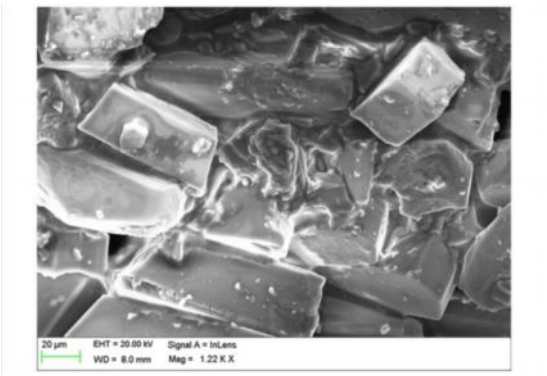
Fig 2. TG/DTA curve of ZCTC single crystal

Simultaneous thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out for the as grown ZCTC crystals. Fine powder of the crystals was used for the TGA/DTA analysis and it was carried out between 30 °C and 850 °C in nitrogen atmosphere at a rate of 20 K/min. From Figure 2 the grown ZCTC single crystal shows five stages of weight loss. First stage of weight loss is between starting temperature and 230 °C resulting in loss of 3.35% of the sample. Second, third, fourth and fifth stage loses15.49%, 5.89%,

15.17%, and 18.48%, of the sample. Finally 40.18% residue of the sample remains. The endothermic peak at 581.1°C in DTA graph shows the melting point of the grown crystal[4].

3.4 Field Emission Scanning Electron Microscope

The Field Emission Scanning Electron microscope was carried out using supra 55, Carl Zeiss, Germany. The FESEM image show step-like growth, which suggests the existence of grain boundaries and striations. The surface is smooth and free from any visible inclusions. The changes on the surfaces and the patterns are due to optimization of pH and temperature oscillations during the growth of the crystal. At higher magnification, cubic micro crystals formation was evident [5].



3.5 Atomic Force Microscopy

Surface morphology plays an important role in various areas of science and technology. Figure 3 shows that the surface contains hillocks with cavities which are probably caused by unequal growth at neighbouring hollow channels [6]. The AFM technique is used to determine the grain size, RMS roughness (0.878163), surface skewness (3.4909), surface kurtosis(37.9252) and maximum peak to valley height.

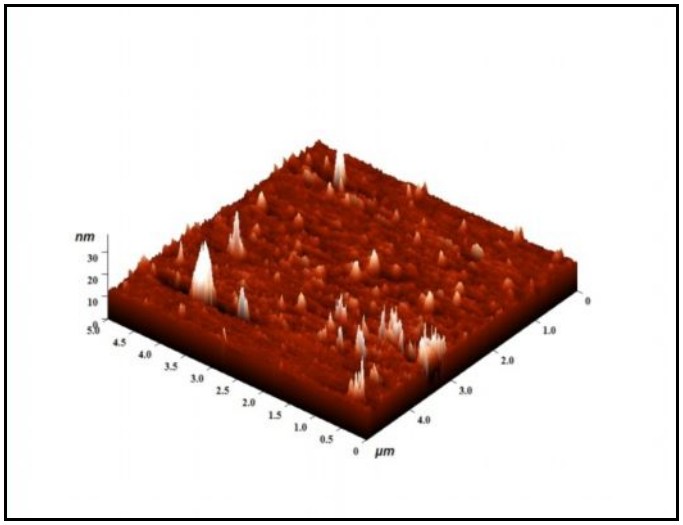


Fig 3.3D AFM image of ZCTC single crystal

3.6 Epifluorescence

The Epifluorescence emission spectrum for ZCTC was recorded using Nikon Eclipse 80i in the wavelength range 400 – 700 nm. The most important advance in fluorescence microscopy was the development of episcopic illumination. Fluorescence finds wide application in the branches of biochemical, medical, and chemical research fields and also used as lighting in fluorescent lamps, LED etc. It is observed from the fluorescent image that the compound shows complete emission [7]. Hence ZCTC possess green fluorescence at 540nm as seen in Figure 4.

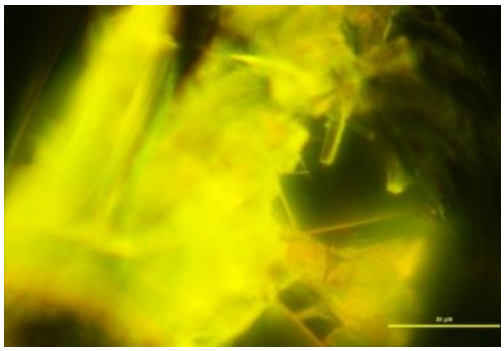


Fig 4. Epifluorescence image of ZCTC crystal

4. Conclusion

Single crystals of ZCTC were grown by slow cooling method in a period of 26 days. Identity of the crystal material was confirmed by single crystal XRD studies. The second harmonic efficiency was confirmed by the emission of green light. The FESEM studies show that the surface is smooth and free from any visible inclusions. The AFM study gives the amplitude analysis, an important parameter to evaluate surface roughness. Finally the Epifluorescence studies reveal that the crystal fluoresces and the compound was excited at 540 nm.

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