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RESEARCH ARTICLE

GROWTH AND CHARACTERIZATION - ORGANIC METAL CRYSTAL OF TETRAMETHYL AMMONIUM CADMIUM IODIDE

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ABSTRACT

The new organo-metallic crystal of tetramethyl ammonium cadmium iodide (TMACI) was grown by slow evaporation technique at room temperature. Crystal structure, lattice parameters and grain size were found using powder XRD technique. To confirm the functional group present in the synthesized grown crystal by FTIR analysis. Absorption spectra and transmittance spectra of the synthesized crystal were studied by UV-VIS method. Surface morphology was analysed by Scanning electron microscope. Dielectric constant is exponentionaly decressed with increase of frequency is observed by dielectric study.

INTRODUCTION

Organic materials with delocalization of electron through conjugated electron systems have been gained considerable attention for chemists, optical physicists, material scientists; because of their excellent performance such as large NLO efficiency, ultra fast nonlinear response time, high optical damage (Zhang, 1994; Aggarwal, 1999; Dongfeng Xue, 1999; Arulchakkaravarthi, 2004; Gupta, 2001; Singh, 1993 and Meenatchi, 2014). Organo metal offers a huge variety of metals with distinctive oxidation states and ligands which could used in optoelectronic application. These non centro symmetric structures may be engineered by using ligands in complex metal centre. They have the general formula $[(CH_3)_4N]$ MX_3 (M=Cd,Zn,Mn,Mg) (X=Cl,Br,I) at room temperature with hexagonal structure (Wang, 2010 and Breezewski, 2001) and possess large number of attractive properties such as optical window , good thermal stability and higher laser damage threshold (Perumal, 2016). Organo-metallic coordination compounds have attracted,because of their functionality of mixing the advantage of both organic and inorganic materials (Boopathi, 2015). The present work, deals with the tetramethyl ammonium cadmium iodide (TMACI) crystal was grown at slow evaporation method. The structural, spectral, optical and electric properties of the grown crystal was studied.

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EXPERIMENTAL METHODS

Crystal Growth: Tetramethyl ammonium cadmium iodide TMACI crystal was prepared by 2:1 ratio of tetramethyl ammonium and cadmium iodide salts, at room temperature by slow evaporation technique. The above two salts were mixed homogeneously with 20ml of water. The prepared solution was stirrerd using magnetic stirrer at room temperature. The saturated solution was transferred to a beaker with a whatman filter paper. The above prepared solution was kept at room temperature without any mechanical disturbance and free of dust particles with the period of 10-15 days, finally colourless transparent crystal harvested, and it in Figure (1). Among them, a few defect and good quality seed crystals were selected for recrystallization.

Chracterization Method

The tetramethyl ammonium cadmium iodide TMACI crystal was subjected to powder X-ray diffraction (PXRD) using Enrof NORNIUS cad 4 X ray diffractometre with CuKα radiation, to detect the lattice parameter and crystal system. The crushed powder sample was scanned over the 2θ range of 10^o-70^o at a rate of 1^o/min. FTIR spectrum of grown crystal TMACI was recorded in the range 4000 to 500 cm^{-1} using Bruker 66v FTIR spectrometer by KBr pellet technique. The UV-VIS absorption spectrum was recorded in the range of 200 to 1100nm. The scanning electron microscope (SEM) studies carried out using MODEL JSM – 6390LV. Dielectric studies on the grown crystal was carried out using a numeric Q impedance analyzer in the frequency range 50 HZ to5MHZ.

RESULTS AND DISCUSSIONS

Structural Analysis

The powder X-ray diffraction pattern of tetramethyl ammonium cadmium iodide (TMACI) shown in figure(1). The lattice parameter of the crystal was determined as a=9.11Å, b=11.26Å and c=11.27Å and interfacial angle α=88.52°, β= γ =66.39°, V= 959°. The orientation angle and hkl plane are indexed and this confirm the purity of the end product. The well developed plane (010) is observed from the Figure (1) and this confirm the crystallinity. The photographs of as grown crystal tetra methylammonium cadmium iodide (TMACI) at show in Figure (2).

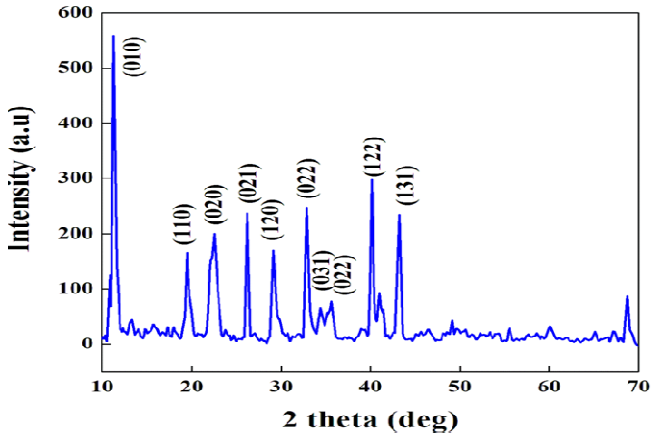

Figure 1. Powder XRD pattern of TMACI crystal

Figure 2. The crystal photo of TMACI crystal

FT IR Analysis

To identify the presents of functional group and determine the molecular structure of grown crystal used the analysis of FT IR spectra shown in Figure (3). The FT IR graph was drawn between transmittance and wave numbers at 500 to 4000cm^{-1}. The peak value around 3022.45cm^{-1} and 3024.34cm^{-1} observed the CH_3 asymmetric stretching in FT IR spectra. The peak occurs 2815.70cm^{-1} denotes the CH_3 symmetric stretching in IR spectra. The peak revealed at strong O-H vibration at 1647.21cm^{-1} in FT IR spectra. The peak values at 1471cm^{-1} and 1446.78cm^{-1} denotes CH_3 symmetric stretching in FT IR spectra. The peak around 947cm^{-1} and 948.26cm^{-1} observed C-N stretching mode of vibration in spectra (Devashankar, 2009).

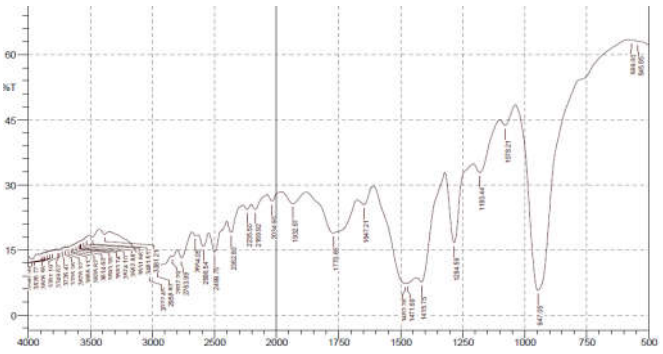

Fig. 3. FTIR spectrum of TMACI crystal

UV-VIS –NIR Analysis

Optical transference of the grown crystal TMACI plays a very important role in the optical technology. For good nonlinear optical material, it is very crucial role to note that the grown crystal free from the intrinsic defects (Dhanabal, 2014). From the graph shows the 90% transmittance to the entire visible region. This enables the crystal to be used for many optoelectronic applications. The lower cut-off wavelength was carried out 375nm from the absorption graph. The grown crystal exhibits low absorption losses with high transmittance and also the UV spectrum does not show any absorption peak in the 400 to 1100nm wavelength range shown in fig (4). The optical absorption coefficient (α) of the grown crystal TMACI was calculated from the following equation (Abdullah, 2013).

$$\alpha = 2.303/d \log (I/T) \quad \text{----------1}$$

Where d is the thickness and T is transmittance of the crystal.

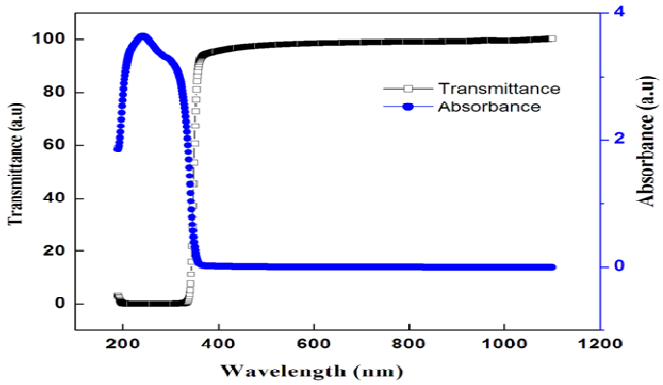

Figure 4. UV-VIS-NIR spectrum for absorbance and transmittance of TMACI

SEM Analysis

SCANNING electron microscope (SEM) JSM-6390 model is used to analysis about the surface of the fabricated crystals. The surface morphology and particle size of the grown crystal TMACI have been shown in Figure (5). Form the SEM micrographs, it can be seen that arrangement of particle distribution SEM image were taken 500x, 1500x, 3500x and 7000x. Magnification with acceleration voltage of 15KV [15]. The particle size increased 18.3µm to 49.7µm by increasing temperature range from 1400°C to 1700°C.

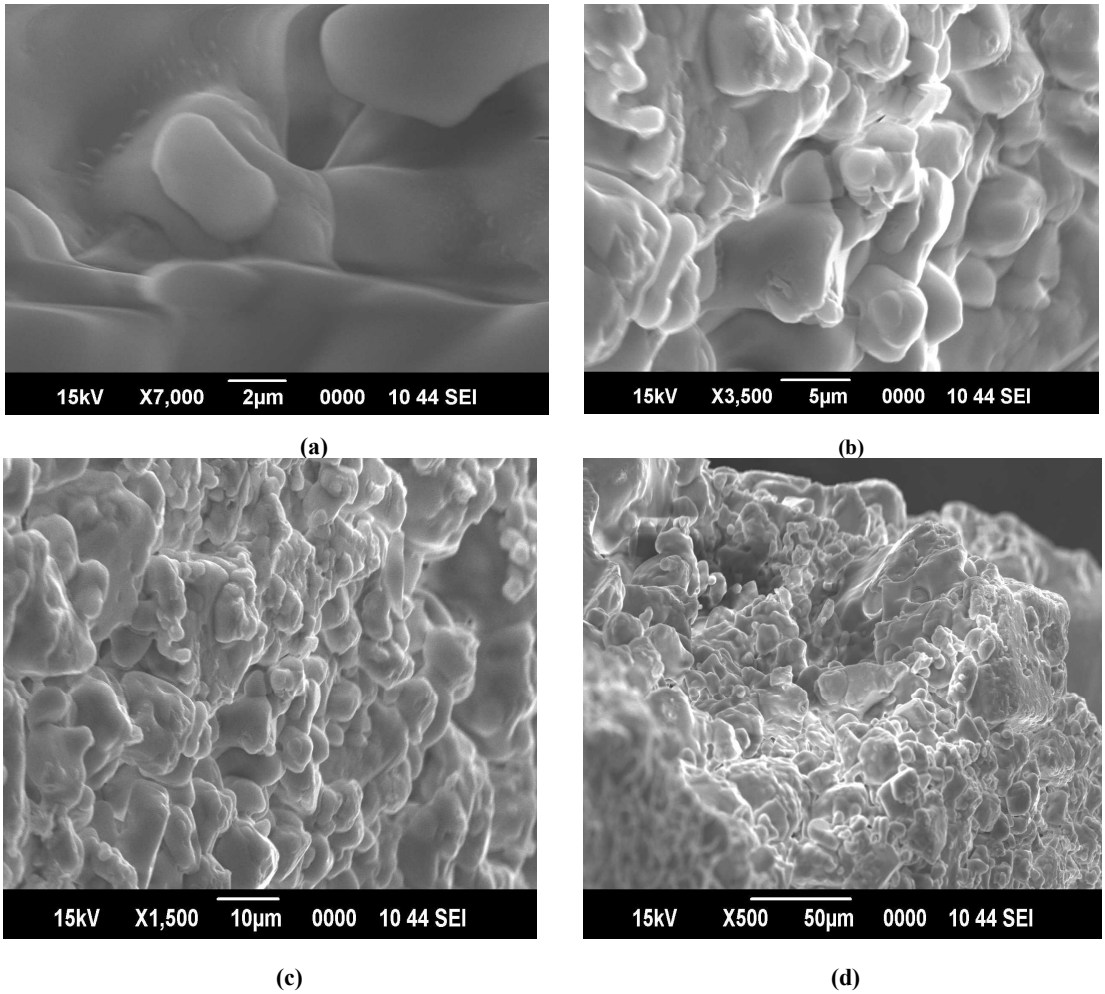

Figure 5 (a,b,c.d) shows SEM Analysis of TMACI crystal

Dielectric Studies

Figure (6) depicts the dielectric constant of TMAI at different temperature range. From the dielectric analysis,dielectric constant is decreased along with increasing frequency for all samples. The TMACI crystal of size (1x1) cm^2 was taken and apply silvar paste on both sides of the crystal and then plasced inside a dielectric cell. The dielectric constant in evaluated using a given relation,

$$\varepsilon = Cd / \varepsilon_o A \quad \text{----------2}$$

Where, C is the capacitance. d is the thickness of the grown crystal , ε_o is the free space permittivity and A is the area of the sample. Under the influence of temperature, dielectric constant value is increased and this is due to hopping conduction. The dielectric constant decreases rapidly in low frequency and slow in high frequency, this behaviour can explain on the basis of koop's theory Maxwell-wagner two layer model.According to koop's theory, material considual to have compound dielectric structure and this explain the decreases of frequency, The higher value of dielectric constant at low frequency are due to interfacial space charge polarization (Kasap, 2006).

Hardness Study

Vickers micro hardness study was observed plane at room temperature using hardness tester fitted with diamond pyramidal indenter. The diagonal length were found using a microscope. The micro hardness (Hv) number was carried from the given relation

$$Hv = 1.8544 (P/d^2)\ kg/mm^2 \quad \text{---------- 3}$$

Where p is the applied load, and d is the average diagonal length of indentation impression in mm. The graph draw between the variation of P versus hardness number (Hv) of the grown crystal TMACI. It is found that the hardness of material increases with increase in the applied load. The phenomenon is called the reverse indentation effect (RISE). According to Meyer's law

$$P = A\ d^n \quad \text{----------4}$$

Where n is the Meyer's index and A is constant for a given material. The graph drawn between log P and log d. The slope of the straight line give the value of n from that value obtained reveals that TMACI crystal belongs to the category

PL Study

Photoluminescence (PL) study used to characterizing vacancies, defects and other imperfection present in the grown crystal (Reena, 2016). This study more challenging, recombination process available transient behaviour of given material. The recombination of electron and hole can in an hydrogen like system by its columbic field as a pair of opposite charges.

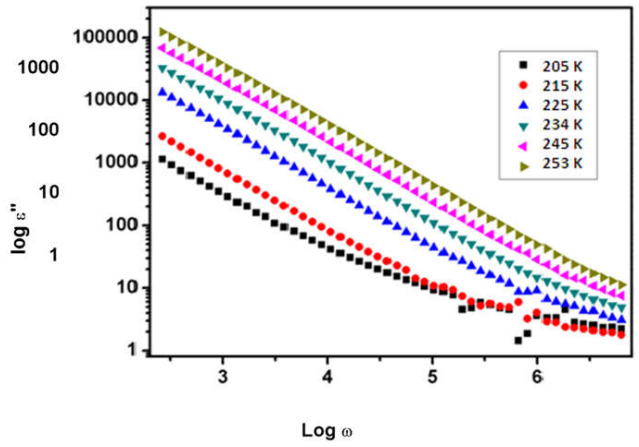

Figure 6. Dielectric analysis of TMACI crystal

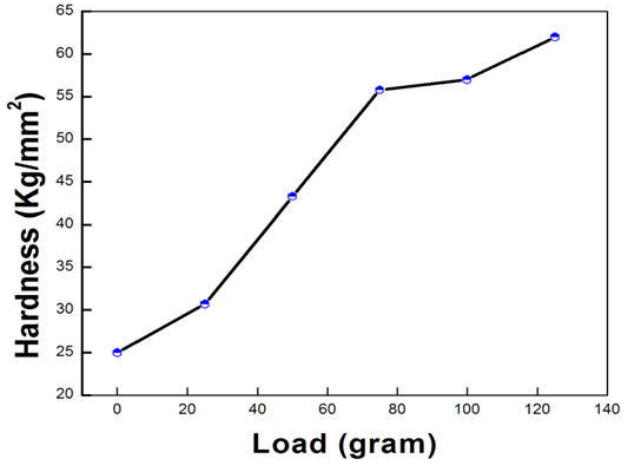

Figure 7. Hardness analysis of TMACI crystal.

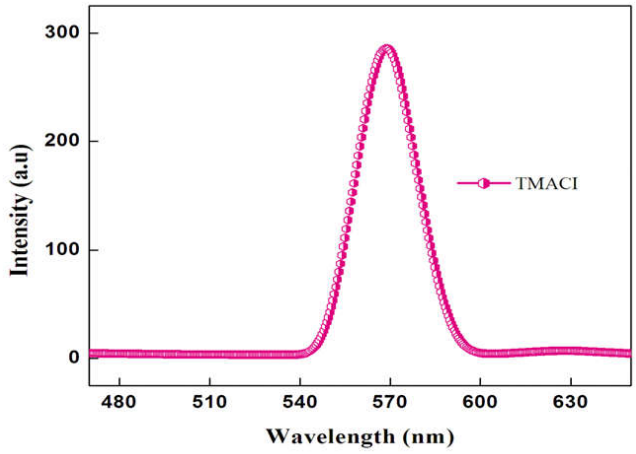

Figure 7. PL spectrum of TMACI crystal

Photoluminescence spectrum was observed at room temperature with an excitation wavelength of 270nm as shown in Figure (7). The broad spectrum was occurs at 570nm. The band gap energy was calculated using the wavelength to energy conversion relation,

$$Eg = hc/\lambda \quad Ev \qquad \text{----------5}$$

Where h is the Planck's constant, c is the velocity of light and λ is the wavelength of peak. From the above equation the band gap of TMACI crystal is 2.16J. The energy band gap of UV transmittance is nearly equal to the value obtained from the photoluminescence (Reena, 2016).

Conclusion

Optically transparent good quality single crystal was prepared by slow evaporation at room temperature. From Single crystal powder X ray diffraction lattice parameter of the grown crystal and well eveloped plan is observed. The morphology and particle size in the range 18.3µm to 49.7µm of is noticed from SEM analysis. To confirm the various functional groups find out using FTIR spectra. The optical absorption study shows the crystal posses large transparency with lower cut off wavelength. Dielectric study was measured the increasing frequency with decreasing ddielectric constant. Vicker's micro hardness study the crystal is subjected and analysis the with stand power of the crystal in the range of value 25grm, 50grm, 100grm. The presence of intrinsic defect observed in photoluminescence (PL) study.

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