

GROWTH AND CHARACTERIZATION OF L-ARGININE ACETATE CRYSTALS

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1. Abstract

Single crystals of pure L- Arginine acetate been grown by slow evaporation technique at room temperature. The crystalline nature of grown crystal was long-established by powder X-ray diffraction analysis (XRD). The structure of the grown crystal determined by X-ray diffraction analysis reveals that it belongs to the monoclinic system. The purposeful group of the grown crystals was established by FTIR analysis. The UV-Vis study was performed to know the optical behavior of the grown crystals. The optical transmittance of the spectrum of solid material was measured in the range of 190 to 800 nm. The crystal shows a high-quality transmittance in the visible region. The transmittance above 74% is observed from 354 to 800 nm, which clearly shows the crystal possesses good optical transparency for the second harmonic generation.

KEYWORD: L- Arginine acetate crystal, XRD, FTIR , UV, optical transparency.

2. INTRODUCTION

Kuang-Chieh Lai et al, have prepared and reported and reported for crystal at first time by conventional methods [1]. They used modified and as-deposited surface properties of thin-film for back contacts on a-Si solar cells. Texturing was performed by simple dry plasma etching in a CVD process chamber. When power is 100W substrate temperature is 190°C, the pressure is 400 Pa and process gas H₂ flow is 700 cm. SEM and TEM were used to evaluate the morphological treatment and induced changes in the deposited films. Comparison of the a-Si solar cells with/without texturing showed both increases in short-circuit current density and fill factor. Consequently, the Si solar cell efficiency was relatively improved by 4.6%.

Li Wei Chang et al .have reported SnO₂ nanowires synthesized by thermal evaporation with ZnO metal as the open-source [2]. The investigator had confirmed, the nanowires are quartzite hexagonal structures. These doped nanowires have the diameters in the range 30-70 nm and several hundred nanometers of length with growth direction along the (100) crystal plane. They found optical properties from the cathode luminescence (CL) and photoluminescence (PL) spectrum showed that nanowires exhibit a relative weak ultraviolet emission (UV) and a strong green emission. Field emission measurements demonstrated that the possesses good performance with a turn-on field of 3.4 V/μm at a current density of 10 μA/cm², a threshold field of 5.4V/μm at a current density of 1 μA /cm² and a field-enhancement factor of 5945. These results are very helpful for the fabrication and optimization of integrated optoelectronic nanodevices using nanowires.

Xun Bie and his co-worker have reported transparent conductive films were deposited on glass substrates by DC reactive magnetron sputtering [3]. Under the optimized deposition

conditions, the films showed acceptable crystal quality, lowest electrical resistivity $2.61 \times 10^{-4} \Omega \text{cm}$ and high transmittance of 90% in the visible region. The surface root-mean-square (RMS) roughness value was about 7.5nm in a scanning area of $10 \mu\text{m} \times 10 \mu\text{m}$ for the film was observed by them.

Bie et al .have been reported transparent conductive films were deposited on glass substrates by DC reactive magnetron sputtering method [4]. The structural, optical; and electrical properties of films were investigated in a wide temperature range from room temperature up to 400°C . The crystalline and surface morphology of the electrical and optical properties of the films are studied by them. The observed films deposited at 350°C exhibited the relatively well crystalline and the lowest resistivity of $3.4 \times 10^{-4} \Omega \text{cm}$. More importantly, the low-resistance and high-transmittance films were also obtained at 150°C and by changing the sputtering powers, having acceptable properties for application as transparent conductive electrodes in LCDs and solar cells are also studied.

Yun-yan Liu et al [5] have reported the films were deposited on quartz substrates by a laser deposition system using a KrF excimer laser at a wavelength of 248nm (200mJ per pulse, 10 ns, and 5Hz). A ceramic with a content of 2.5 at % was used as a target. The XRD pattern was obtained for the films deposited on quartz substrates for 2,4,10 and 30 min and AFM images had been scanned for different films with deposition times=1,4,10 and 30 min. The electrical and optical property, the optimal film prepared in their experimental condition was 10min with a thickness of 200nm, corresponding to the resistivity of $4.8 \times 10^{-4} \Omega \text{cm}$, and its average optical transmittance was 85%.

3. EXPERIMENTAL TECHNIQUE

X-RAY POWER DIFFRACTION

1. X-ray powder diffraction (XRD) is a diffraction technique primarily used for phase identification of a crystalline substance and can provide information about unit cell dimensions. The material is finely ground, homogenized and average bulk composition is determined. X-ray diffractometer consists of three basic elements such as an X-ray tube, a sample holder and an X-ray detector. X-ray is generated in a cathode ray tube by heating a filament to produce electrons and then accelerating the electrons towards a target by applying a voltage and then bombarding the target substance with electrons[1]. As X-ray beam travels through any substance, diffraction takes place whenever Bragg's law is satisfied (fig). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the trellis spacing in a crystalline sample. Solving Bragg's equation gives the d spacing between the crystal lattice planes of atoms that produce productive interference.

X-ray diffraction is based on the constructive interference of a monochromatic X-ray and a crystalline sample. These x-rays are generated by a cathode ray tube. The filters produce monochromatic radiation, the collimator to concentrate directed toward the sample. The interaction of the incident ray with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n = 2d \sin\theta$)

These diffracted X-rays are then detected, processed and counted for analyzing the X-ray diffraction pattern. By scanning the sample through a range of 20 angles, all possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material. So the material has a set of unique d-spacing. Conversion of the diffraction peaks to d-spacing allows identification of the material. Typically, this is obtained by comparison of d-spacing with standard reference patterns[2].

UV-VIS-NIR- SPECTROMETER

The UV-Vis- NIR spectrophotometer. Light from the monochromatic source is passed through the sample and the emergent beam is detected using the detector and recorded. A portion of the beam, absorbed and transmitted are called absorbance and transmittance.

The spectral information contained in the ultraviolet, visible and near-infrared (UV-VIS-NIR) spectrum identifies the intrinsic composition of a liquid and allows for discriminating among similar solids. In particular, the UV band is specific for the electronic transitions, while the VIS band contains information about pigments and chromospheres. Although the true fingerprinting region is the mid-infrared, also the NIR is advantageously used, as it includes overtones of the MIR band and some of their combinations. Absorption spectroscopy is conveniently performed in the wide UV_VIS_NIR spectral range through optical fibers, as they are transparent from 200 to 2500nm. The wide accessibility of dazzling LEDs and compact/portable spectrometers further enhances the purpose areas of absorption spectroscopy and makes it possible for the completion of compact instrumentation dedicated to the monitoring of specific parameters[4].

Here an optical fibers and low-cost spectrometers sources is used to perform UV-VIS-NIR absorption spectrometer with a test case liquid, which is the extra virgin olive oil. The measured hyperspectral signatures are processed through multivariate analysis with the twofold scope to achieve classification maps and also the correlation with important chemo-physical parameters.

Direct Transition

When the substance is free from any imperfection only the direct by allowed transition might take value from valance to conduction band.

For direct transition

$$\alpha = A(h\nu - E_g)^{1/2}$$

For direct forbidden transition

$$\alpha = A(h\nu - e_g)^{3/2}$$

INDIRECT TRANSITION

When the substance has defects such as impurities, dislocation, etc., One can consider the perturbation of the system due to their interaction with photons, for allowed transition

$$\alpha = A(h\nu - E_g)^2$$

For direct forbidden transition

$$\alpha = A(h\nu - E_g)^3$$

DETERMINATION OF BAND GAP

For incident photon energy greater than the bandgap and above the exponential and the optical absorption follows a power law.

$$\alpha = A(h\nu - E_g)^n$$

The optical energy band gap for different substances thickness was obtained by extrapolating the straight-line portion of α^2 vs $h\nu$ curves. The line touching the point will be the bandgap of the material.

FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY

Fourier Transform Infrared Spectroscopy (FTIR) is a dominant tool for identifying types of chemical bonds in a molecule by producing an infrared absorption spectrum that is like a molecular "fingerprint". Because each dissimilar material is a unique grouping of atoms, no two compounds produce the exact same infrared spectrum. Therefore, infrared spectroscopy can result in a positive identification (qualitative analysis) of every dissimilar kind of material. In addition, the size of the peaks in the spectrum is a direct suggestion of the amount of material present. With modern software algorithms, infrared is an outstanding tool for quantitative analysis.

- It can recognize unidentified materials
- It can conclude the excellence or constancy of a sample
- It can conclude the number of components in a fusion

Principle

In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint, no two unique molecular structures produce the same infrared spectrum.

Importance

Fourier Transform Infrared (FT-IR) spectrometry was developed to overcome the limitations encountered with other instruments. The main complexity was the time-consuming scanning

process. Fourier transform infrared spectroscopy is preferred over dispersive or filter methods of infrared spectral analysis for more than a few reasons:

- It is a non-destructive technique
- It provides an accurate measurement method which requires no outside calibration
- It can amplify speed, collecting a scan each second
- It can increase sensitivity – one second scans can be co-added together to ratio out random noise
- It has greater optical throughput
- It is mechanically simple with only one moving part.

SCANNING ELECTRON MICROSCOPE (SEM)

The Scanning Electron Microscope (SEM) has been one of the most versatile and widely used tools of modern science which became commercially available from 1966. Applications are not only the imaging of objects but also metrology and failure analysis. The main benefit of an SEM is the imaging of solid surfaces and morphology with very high spatial resolution and high depth of focus. With an optical microscope, a spatial resolution down to 0.2 μm and a depth of focus of 1 μm is possible. In contrast, the SEM can achieve a resolution down to 5 nm and the depth of focus for a magnification of 100 is about 0.5 mm. Image generation and magnification is carried out electronically, not optically.

Principle

When the accelerated primary electron strikes the sample, it produces secondary electrons. These secondary electrons are collected by a positively charged detector which in turn gives the three-dimensional image of the sample.

Basic Set up and Working

In a Scanning Electron Microscope (SEM) electrons are accelerated by a high voltage. When hitting a solid sample, these **primary electrons** (PE) are generating several interaction products like x-ray, backscattered electrons, and secondary electrons. The SEM mainly uses the **secondary electrons** (SE) for imaging these electrons have energies between 0 eV and 50eV.

Image Formation

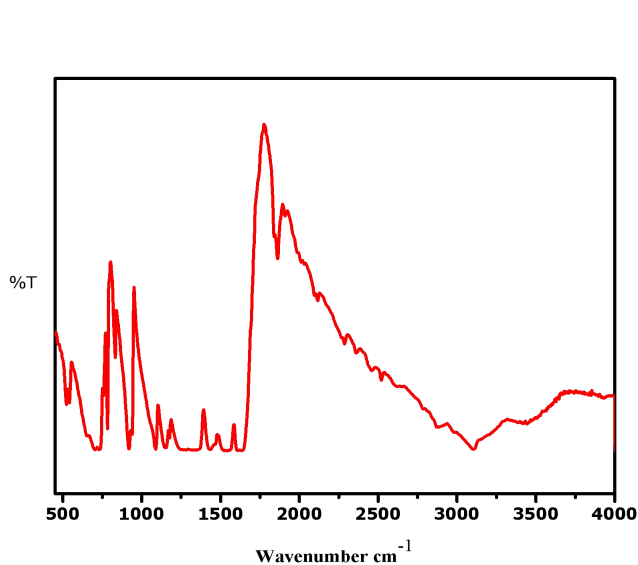
The signal, which is proportional to the amount of radiation leaving an individual point of the sample at any instant, can be used to modulate the brightness of the beam of the display cathode ray tube as it rests on the corresponding point of the image. When the radiation from the point on the specimen is high, the intensity of the point on the display cathode ray tube is also high.

If the probing beam is then moved to an adjacent spot on the specimen, information from that point can be used to modulate the corresponding point on the display cathode ray tube.

Similarly, one-to-one correspondence is necessary for the formation of an image. The image can also be recorded as point-to-point information to build up in sequence on a photographic film.

4.RESULTS AND DISCUSSION

Fourier Transforms Infrared (FT-IR) analysis:



FT-IR of LHP Crystal

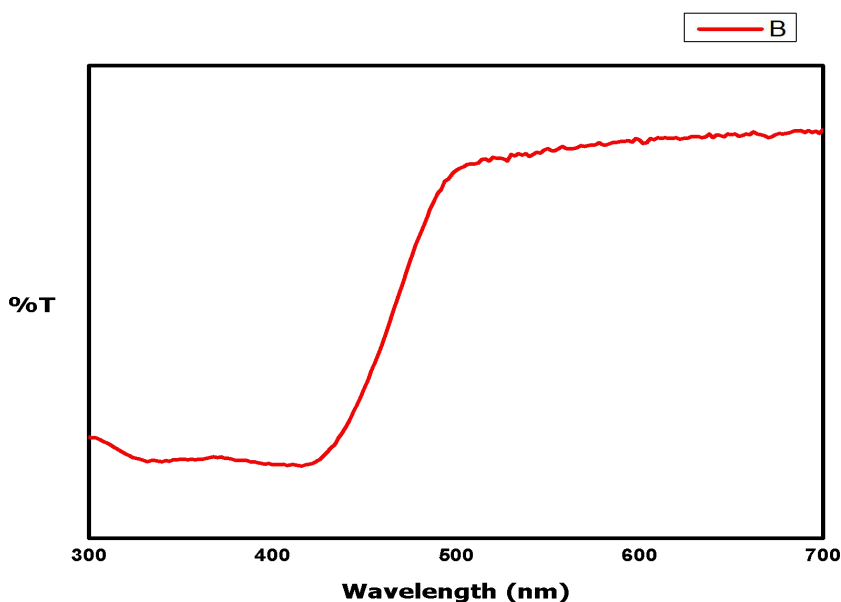
FTIR spectrum show changes in the absorption wavenumber due to change in the bond length between O-H and P=O. It is found that the optical properties of pure and doped ADP changed due to the weak force of attraction of the bond between O-H and P=O. This force of attraction may not be only due to hydrogen bonding but also due to the substitution of NH₄⁺ ion in the crystal lattice of tetragonal ADP crystal, which is in good agreement with the result of Ananda Kumari et al (2009).

The characteristics absorption frequencies of various functional groups are given in the following table.

| S.NO | Frequency Range | Bond Assiment |
|------|-----------------|-------------------------|
| 1 | 3261 | O-H Stretching |
| 2 | 2411.46 | Vibratation of combined |

| | | |
|---|---------|-----------------------------------|
| | | Bond |
| 3 | 1444.30 | Bending Vibration-NH ₂ |
| 4 | 1198 | P-O-H Vibratation |
| 5 | 545.03 | PO ₄ -Vibration |

UV-Visible Spectrum



UV-Visible Transmittance Spectrum of LHP Crystal

The UV-Vis-NIR transmittance spectrum was recorded for the grown crystal using a UV-Vis-NIR spectrophotometer in the range 420-700 nm, to find the suitability of LAA crystal for optical applications. The recorded spectrum is shown in fig.3. The crystal shows good transmittance in the visible region which enables it to be a good material for optoelectronic applications.

5.CONCLUSION

L- Arginine acetate crystal (LAA) crystals were grown by slow evaporation technique. The powder X-ray diffraction studies of pure and L- Arginine acetate doped picric acid showed that crystal posses tetragonal structure having symmetry space group, with lattice parameter has good agreement with JCPDS data card no. 085-0815. Even after the doping crystal system remains unchanged. Intensity peaks of L-Histidine doped picric acid crystal resemble with diffraction angle of pure picric acid crystal with negligible small variation, while intensity variation observed. The FT-IR spectrum confirms the presence of all functional groups and L-

Histidine. As the concentration of L- Histidine increases the peaks shifted towards higher wavenumber side. The UV-Vis-NIR spectral studies confirmed that the grown crystal has wide transparency in the visible region the good transparency shows that LHP crystal can be used for Nonlinear optical applications.

6.REFERENCES

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