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Growth, Spectroscopic, Dielectric & Electrical studies of Glycine Manganous Acetate Single Crystal

P. Geetha[1], K. Satheesh[2]*, V. Chithambaram[1]

[1&2]Research Center Physics, Dhanalakshmi College of Engineering, Chennai, India
[1*]Department of Physics, Sri Krishna Engineering College, Chennai, India

Abstract : A potentially useful semi organic material glycine with Manganous Acetate (GMnAc) has been grown by solution growth slow evaporation technique. Good transparent GMnAccrystals were obtained in a time span of 3 weeks. The grown crystals were characterized by Single crystal X-ray diffraction studies. The grown crystals have been subjected to powder X-ray diffraction to identify the intense peaks on various planes. The UV–VIS-NIR Spectrum of the grown GMn Accrystals shows less optical absorption and good transmittance in the entire visible region enabling its use in optical applications and the lower cut off wavelength as recorded from the absorption spectrum is 276 nm.The band gap energy was found to be 4.5 eV.The grown crystals were thermally stable up to 71.9◦C. Mechanical properties such as micro hardness (Hv) and Meyer's index(n), Yield strength, elastic stiffness constant have been carried out by indentation method.The dielectric constant and dielectric loss measurements of the GMnAc crystal at different temperatures and frequencies of theapplied field are measured and calculated. Electrical properties of the material were investigated over wide frequency and temperature range. The ac/dc conductivity studies are calculated and the activation energy is determined. GMnAc is an electrically non conducting material at room temperature.
Keywords : Semiorganic crystal, Optical properties, Mechanical properties, Dielectric properties , Electrical properties,Spectroscopic.

1. Introduction

The search for new materials with high optical nonlinearity is a potential area for both academic and industrial purpose. These materials have attracted the interests of many theoretical and experimental researchers. Complexes of amino acids with inorganic acids and salts are promising materials for optical second harmonic generation (SHG), as they tend to combine the advantages of the organic amino acid with that of the inorganic acid salt. The organic NLO crystals usually have poor mechanical and thermal properties and are susceptible for damage during processing even though they have large NLO efficiency. Also it is difficult to grow larger size optical quality crystals of these materials for device applications [1, 2].Purely inorganic NLO materials have excellent mechanical and thermal properties butpossess relatively moderate optical nonlinearity. The present fascinating field of research is to synthesize, grow and characterize semi organic NLO crystals. Here an attempt has been made to synthesize and grow NLO semi organic crystals. Hence it may be useful to prepare semi-organic crystals which combine the positive aspects of organic and inorganic materials resulting in useful NLO properties [3, 4].These crystals have higher mechanical strength, chemical stability, and large nonlinearity, high resistance to laser induced damage, low angular sensitivity and good mechanical hardness.

Amino acids are interesting materials, as they contain a proton donor carboxyl acid (-COOH) group and proton acceptor amino (-NH2) group which provide the ground state charge asymmetry of the molecule required for second order nonlinearity [5, 6]. Recently a large number of amino acid based crystals has been reported for successful nonlinear optical applications such as L-alanineacetate, L-alanine cadmium chloride, L-glutamic acidhydro bromide, and L-valinehydrobromide [7-10] in the literature. Glycine is an organic compound with the formula NH_2CH_2COOH.Having a hydrogen substituent as its side-chain, glycine is the smallest of the 20 amino acids commonly found in proteins. Glycine is the simplest amino acid. Unlike other amino acids, it has no asymmetric carbonatom and is optically inactive.It has three polymericcrystalline forms α, β and γ. Glycine and its methylated analogues form complexes with mineral acids exhibitinginteresting physical properties like ferroelastic, ferroelectric or antiferroelectricbehaviour often associated with transitions. According to the structural analysis of ferroelectric triglycine sulfate, there are two kinds of glycine group's glycinium and zwitterions. Such configurations of glycineions interconnected by short O–H-O hydrogen bonds areregarded as significant for the ferroelectric behaviour of thiscrystal.

In the present work we, report on growth of new semi-organic crystal Glycine Manganous Acetate (GMnAc) from amino acid familyand its characterization. To have a full understanding about the structure and its properties for the grown crystal single crystal XRD structure analyses, PXRD, UV- Vis absorption, Thermal analyses (TG/DTA), and SHG measurements were also been carried out. Dielectric studies are also taken for the grown crystal.

2. Experimental Procedure

Commercially available Glycine Manganous Acetate was used for the growth. The solution was prepared by dissolving equimolar amounts of Glycine and Manganous Acetate in double distilled water. The reaction takes place according to the equation given below.

$C_2H_5NO_2 + C_4H_6O_4Mn = C_2H_5NO_2.C_4H_6O_4Mn$

The material was purified by repeated recrystallization.

2.1 Crystal Growth

Glycine, an amino acid was mixed with inorganic salt Manganous Acetate in aqueous solution in the ratio of (1:1) stirred continuously to get a uniform solution. The solution was then filtered twice to remove the suspended impurities and allowed to crystalline by slow evaporation technique at room temperature in a dust-free atmosphere.

The grown crystals are bright, transparent and white in colour. The grown crystals were collected from the mother liquid by using well cleaned forceps. Good transparent crystals were obtained in a period of about 3 weeks as shown in Fig. 1.

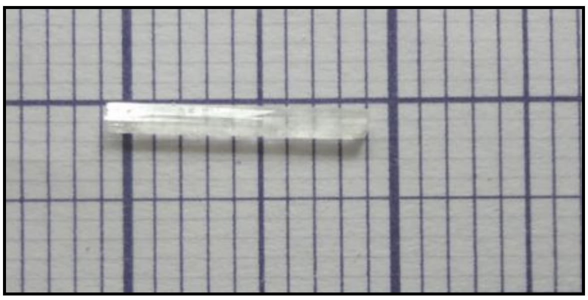


Fig.1 As grown crystal of GMnAc

2.2 Characterization techniques

Single crystal XRD was recorded by Enraf Nonius CAD/MACH3 single crystal X-ray diffractometer to find the molecular structure, atomic coordinates, bond lengths, bond angles and molecular orientation. The powder XRD pattern of crystal was obtained using a BRUKER AXS D8 Advance X-ray diffractometer with

CuKα radiation (λ = 1.54060˚A) at room temperature. The electronic absorption and transmittance spectra of the compound were recorded using Lambda 35 UV–visible spectrophotometer in the wavelength range from 190 to 800 nm. DSC analysis of GMnAc was carried out between 30 and 500 °C in nitrogen atmosphere at a heating rate of 10 °C using Perkin Elmer model. Mechanical properties of the grown GMnA ccrystals were studied using MH–5 hardness testers. The dielectric studies of the compound were recorded at room temperature using a TH 2816 A DIGITAL LCRZ METER in the frequency region 50 Hz–2MHz. The second harmonic generation (SHG) efficiency of the material was carried out by Kurtz–Perry powder technique using Nd: YAG laser beam.

3. Results and discussion

3.1. Single crystal XR Diffraction Analysis

GMnAc was subjected to Single crystal X-ray diffraction and powder X-ray diffraction studies. The X-ray data were collected using an Enraf Nonius CAD/MACH3 single crystal diffractometer instrument for grown Glycine Manganous Acetate crystal. The calculated lattice parameter values are a = 9.11 Å, b =17.54Å and c = 10.41ÅVolume of the unit cell =1552Å^3.The XRD data prove that the crystal is monoclinic in structure with the space group of P. The XRD results are in good agreement with the reported values and thus confirm the grown crystal.

3.2. Powder X-RAY Diffraction

Powder X-ray diffraction study was used for the identification of crystallinity of the grown crystal. The Kα radiations (λ = 1.5418 Å)from a copper target were used. The sample was scanned in the range between 10 and 100°C. Fig. 2 represents the indexed powder diffractogram for the grown crystal of GMnAc. The sharp intensity peaks found in spectra shows good crystalline nature and purity of the grown crystal.

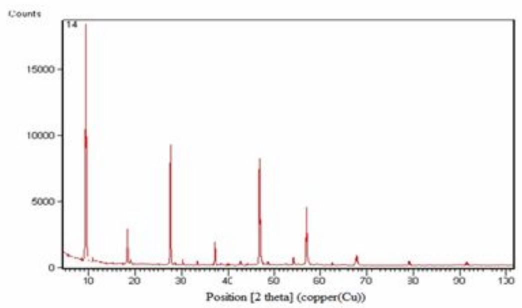


Fig.2 Powder XRD Pattern of GMnAc

3.3. Thermal Studies

DSC was performed using Perkin Elmer (model DSC 7) in the temperature range 20–280°C at a heat rate of 10 K/min. The measurement was recorded at Nitrogen atmosphere. The sharp endothermic peak upwards at 71.9°C shown in Fig.3 confirms the melting point of GMnAc crystal.

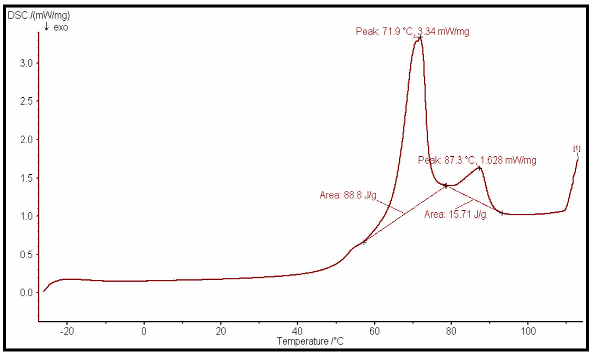
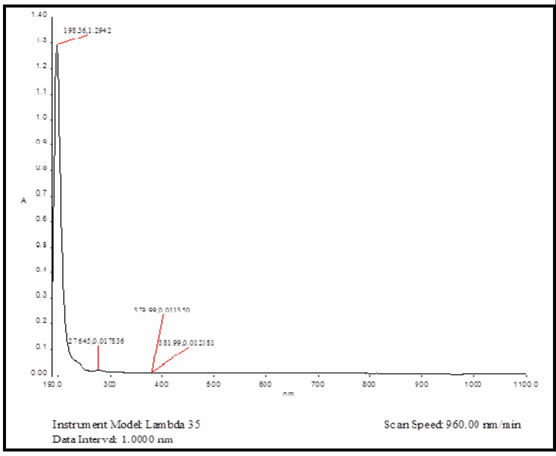


Fig. 3 DSC curve

3.4. Optical studies

The crystal GMnAcis well polished and the sample is dissolved in water to measure the absorption and transmission spectrum in the spectral region shown in Fig.4&5 using LAMBDA-35 UV–Vis spectrometer 190 – 1100nm with Data interval 1.0000nm and Scan Speed 960.00nm/min. For optical fabrication, the crystal should be highly transparent over a considerable region of wavelength [11, 12].In the UV Vis spectrometer the lower cut off wavelength as recorded from the absorption spectrum is 276 nm.From the absorption spectrum the crystal is transparent in the range 381nm-1100nm without any absorption peak, which is an essential parameter of NLO crystals. Absence of absorption in the region 400–1100 nm is an advantage and it is the key requirement for materials exhibiting NLO property.Using the relation,



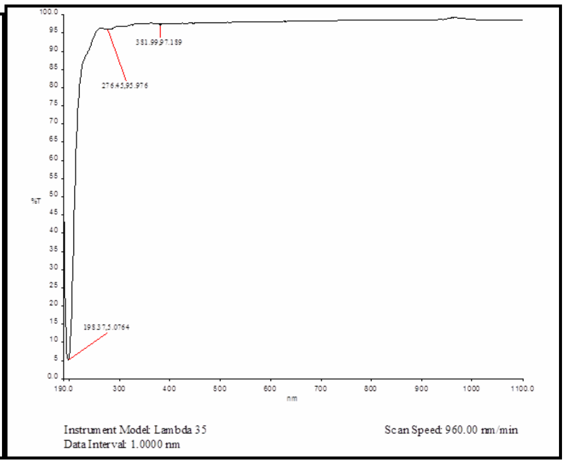


Fig. 4 Absorption spectra **Fig.5 Transmission spectra**

$Eg = 1240/\lambda$ →(1)

The band gap energy was found to be 4.5 eV. The optical absorption spectrum shows that absorption was very less in the entire visible region and part of IR region. The optical absorption coefficient (α) was calculated using the following relation

$\alpha = (2.303/d) \log (1/T)$ →(2)

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

Using the Tauc's relation, a graph (Fig. 6) has been plotted between $h\upsilon$ and $(\alpha h\upsilon)^2$ to estimate the direct band gap value, where α is absorption coefficient and $h\upsilon$ is the energy of the incident photon ($E = h\upsilon$). The energy gap (E_g) is determined by extrapolating the straight line portion of the curve to $(\alpha h\upsilon)^2 = 0$. From the plot, theband gap of GMnAc is found to be 4.2 eV.

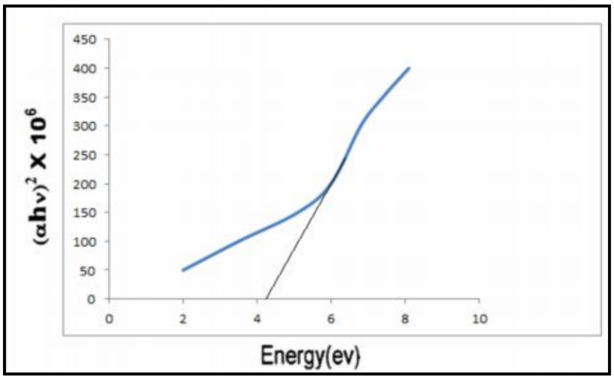


Fig.6 Plot of $(\alpha h\upsilon)^2$vs Photon energy of GMnAc

Transmission spectra are very important for any NLO material because a nonlinear optical material can be of practical use only if it has wide transparency window. The optical transmission spectrum for the wavelengths between 190 and 1100 nm were recorded. From the transmittance spectra, it is observed that GMnAc crystal have high transmittance in the entirevisible and near IR region. The UV cut-off wavelength of the crystal is found to be at 198 nm. It is found that the maximum transmittance of the grown GMnAc single crystal is 52% and it has almost more than 45% transmittance from 276 to 1100 nm.The advantage of using amino acids shows the absence of strongly conjugated bonds leads to wide transparency range in the visible and UV spectral regions. The transparency isan added advantage for this crystal to be utilized in the field of optoelectronic devices.

3.5Mechanical Studies

One of the methods to determine the mechanical behaviour of the grown crystal is micro hardness test**.** The hardness of a material is a measure of its resistance to local deformation. It is correlated with other mechanical properties like elastic constants, yield strength, brittleness index and temperature of cracking. The indentation marks were made on the surface of GMnAc single crystal at room temperature by applying load of 25, 50 and 100 g. The distance between two indentation points has been maintained to more than three times the diagonal length so as to avoid the mutual interference of indentations. The Hv is found to increase with increase inthe load from 25 to 100 g and crack occurs at higher loads. Mechanical properties of the grown GMnAc crystal were studied using M H–5 hardness testers. The diagonal lengths of the indented impression were measured using calibrated micrometre attached to the eyepiece of the microscope.

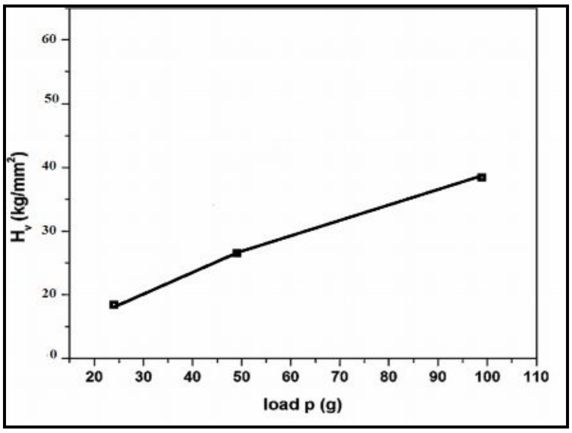


Fig.7 plotof load p vs.hardnessnumber

TheVickersmicro hardnessvalueswerecalculated from the standardformula [13]

$Hv = 1.8544 (P/d^2)$ kg/mm^2 →(3)

Where P is the applied load and d is the mean diagonal length of the indentation. The corresponding trace is shown in the Fig.7, from which it is observed that the hardness increases with the increase of load up to 100g and crack occurs at that load.The Micro hardness value was taken as the average of the several impressions made. According to the normal indentation size effect (ISE), microhardness of crystals decreases with increasing load and in reverse indentation size effect (RISE) hardness increases with applied load. In our case, Hv increases with applied load.

To find the work hardening coefficient (n) of the grown crystal, another graph (Fig. 8) was drawn between logarithmic values of load and diagonal length of indentation.

From Meyer's law [14]

$P = ad^n$ →(4)

Here, is the constant for the given material. It connecting the relationship between applied load and diagonal length of the in dentation, work hardening coefficient or the Meyer index was calculated. The work hardening coefficient ‘‘n’’ was calculated as 3.49 from the Fig. 8.According to Onitsch [15], if n lies between1and 1.6, then the grown crystal will be aharder material and it is more than 1.6 for soft materials [16]. Since the calculated work hardening coefficient ‘n’ is more than1.6, the grown crystal is suggested that it comes under the category of soft material.

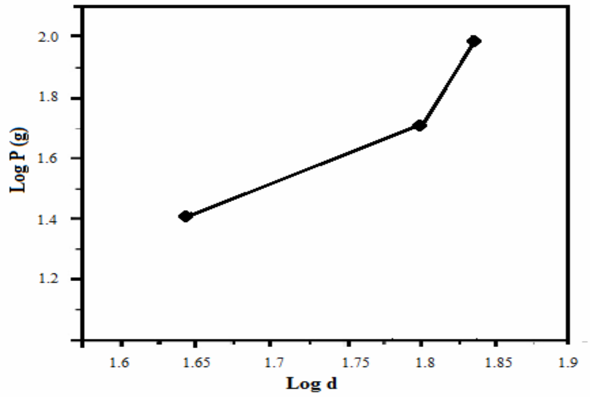


Fig. 8 plot of Log d vs. Log P (g)

Yield strength of the grown GMnAc crystalline material was also calculated using the formula,

$\sigma_y = (Hv/3)(0.1)^{n-2}$ $\rightarrow$(5)

Where σ_y is the yield strength, Hv is Vicker's hardness and n is the logarithmic exponent. It was found to be 3MP from the relation and hence the grown GMnAc single crystal has low mechanical strength.

The elastic stiffness constant(C_{11}) for different loads calculated using Wooster's [17] empirical formula C_{11}= Hv $^{(7/4)}$ is given in Table 1which gives an idea about tightness of bonding between neighbouring atoms.

Table 1 Stiffness constant

Load P	C_{11} (x10^{14} Pa)
25	171.32
50	299.36
100	595.05

3.7. Dielectric studies

Dielectric materials are used in the fabrication of capacitors, thin film transistors, resistors, insulators, tunnelling devices. Good quality single crystal of GMnAc was selected for the dielectric measurements using TH 2816 A DIGITAL LCRZ METER in the frequency from 50 Hz to 2 MHz. The increase in dielectric constant at low frequency is attributed to the space charge polarisation. The sample was polished by soft tissue paper. Silver paste was applied on both opposite faces to make a capacitor with the crystal as a dielectric medium. The sample was placed between two cooper electrodes, which act as a parallel plate capacitor. Dielectric studies give useful information about charge transport mechanism inside the crystal. In normal dielectric behaviour, the dielectric constant decreases with increasing frequency reaching a constant value, depending on the fact that beyond certain frequency of the electric field [18,19] and the dipole does not follow the alternating field. This type of dielectric response may be of great interest in electrical applications. The presence of dielectric between the capacitor plates increases the capacitance of the capacitor. The capacitance and dielectric loss were measured for different frequencies. Variation of dielectric loss vs. log frequency of GMnAc crystal is shown in fig.9.The dielectric constant was calculated using the following relation

$$C_p d = \frac{\varepsilon_r}{A\varepsilon_0} \quad \rightarrow (6)$$

Where ‘Cp’ is the capacitance of parallel plate capacitor, ‘A’ is thearea of overlap of the two plates and‘d’ is the separation between the plates.As the frequency increases, the dipoles do not comply with the external field resulting in the decrease of polarization and hence thedielectric constant decreases as frequency increases.In accordance with Miller rule, the lower value of dielectric constant at higher frequencies is a suitable parameter for the enhancement of SHG coefficient. The variation of dielectric loss with frequency isshown in Fig. 10.

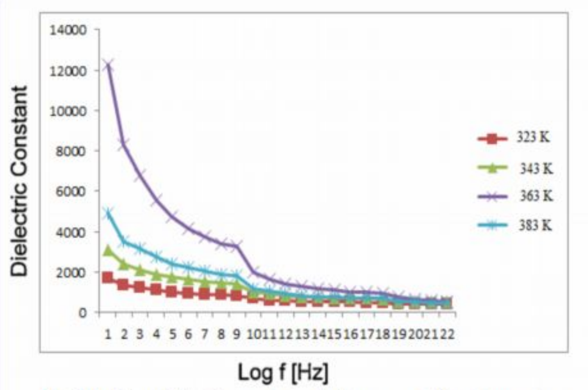


Fig 9 Variation of dielectric constant vs. log frequency at different temperatures

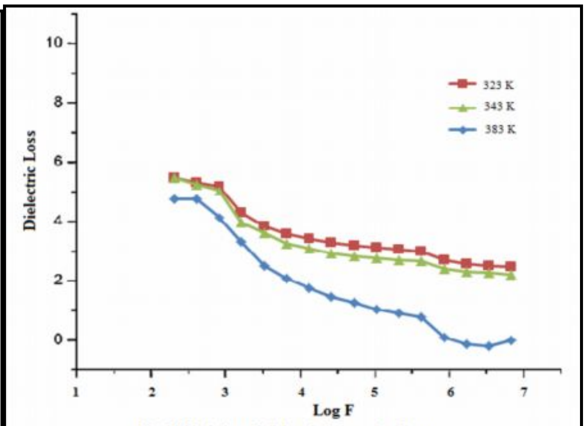


Fig. 10 Variation of dielectric loss vs. log frequency

The characteristic of low dielectric loss with high frequency for the sample suggests that the crystal possess enhanced optical quality with lesser defects and this plays a vital role for the fabrication of ferroelectric devices.The variation of Dielectric constant with temperature for GMnAc crystal is shown in Fig. 11.

3.8 AC conductivity studies

The a.c. conductivity (σ_{ac}) was calculated from dielectric datausing the empirical relation

$\sigma_{ac} = \varepsilon_0 \varepsilon_r \omega \tan \delta$ →(7)

Where ε_0 is the permittivity of free space, $\varepsilon_r$relative permittivity, ω is the angular frequency ($2\Pi f$) and $\tan \delta$ is the dielectric loss.From the graph (12) it is clear that conductivity decreases with temperature. The plot(13) of $\ln\sigma_{ac}T$ versus 1000/T obeys Arrhenius relationship

$\sigma_{ac} = \sigma_o \exp(-E_a/kT)$. →(8)

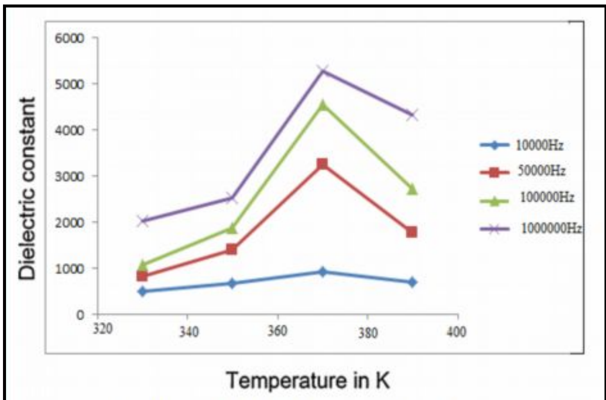


Fig. 11 Variation of Dielectric constant vs. Temperature

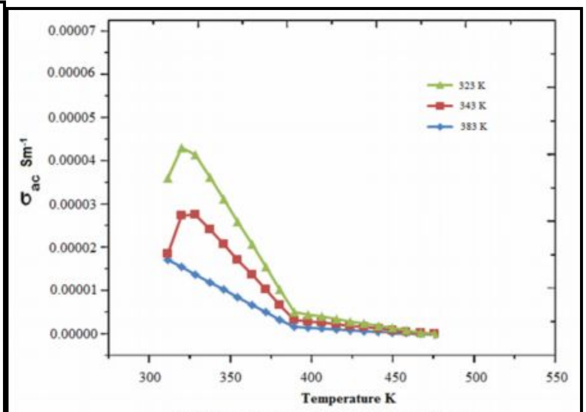


Fig. 12 Temperature variation of a.c. conductivity

WhereEa is the activation energy, k is the Boltzmann constant and T is the temperature (in Kelvin).Therefore, the sample exhibits Arrhenius type conductivity. The sudden increase of ac conductivity is due to the good response of the charge carriers with the applied field.Activation energy wascalculated using the slopes of the above line plots (E = - (slope) k × 1000). The activation energy of GMnAc, calculated from the plot is found to be 0.239eV.

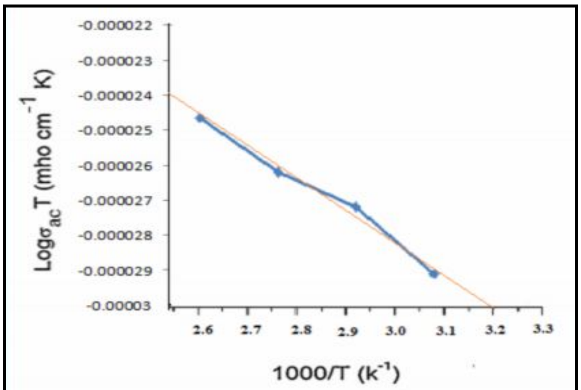


Fig. 13 Plot of Logσ_{ac}T vs. 1000/T

At high temperatures, the curve indicates a frequency independent d.c. conduction phenomenon. On the low temperature side, the conductivity curves branch out with weak dependence on temperature for lower frequencies.

3.9. D.C. Conductivity Studies

The current voltage plots of crystal are shown in Fig. 14. The DC conductivity of the material was calculated using the relation σ= d/RA, d =thickness of sample crystal, A= area of the face of the crystal in contact with electrode. The Arrhenius plot can be drawn by taking ln (σ) along x-axis and(1000/T) along y-axis. The conductivity values can be fitted to the relation $\sigma = \sigma_0 \exp(-E/KT)$, where E is the activation energy, K is the Boltzmann constant, T represent absolute temperature and $\sigma_0$is the parameter depending on the material. Activation energy can be estimated from the slope of above graph using the formula E =-(slope) K x 1000. The ploto fln (σ) T vs. (1000/T) is given in the Fig.15. The value of activation energy was found to be 0.286eV. The crystal GMnAc has the activation energy value which is more than reported value of KDP(0.22eV). The value of the activation energy provides the information that GMnAc crystal is non-semiconducting in nature.

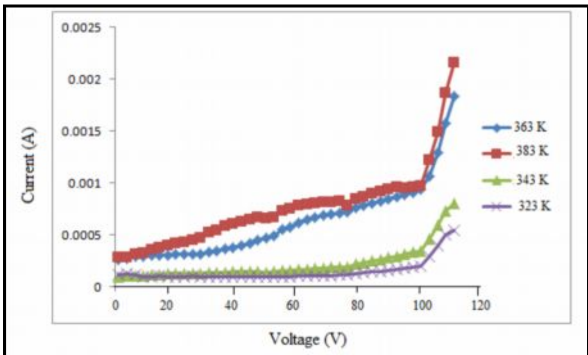


Fig. 14 Plot of Current vs. Voltage

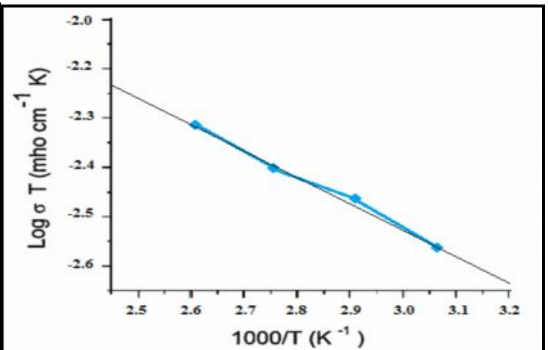


Fig. 15 Variation of σ_{dc} with 1000/T

3.10 a.c resistivity and conductivity

The a.c resistivity and a.c conductivity were calculated using the following relation

P = A/2ΠfCd →(9)

σ_ρ= 1/ρ →(10)

Where C is the capacitance, d is the thickness, A is the area of the crystal, and f is the frequency of the applied field. As shown in Fig.16, a.c resistivity decreased rapidly as frequency increased. Obviously reverse trend was observed for a.c conductivity Fig. 17 of the grown crystals [20].Pyroelectricity is the ability of certain materials to generate temporary voltage when they are heated or cooled [21]. The pyroelectric material shows change in the direction of spontaneous polarization when electric field is applied on them. The pyroelectric current measurement technique can be regarded as complementary to hysteres is loop measurement and applied to study of the Curie point transition.

From the dielectric study, the variation of current with temperature for 1 KHz is shown in Figure 18, from the graph it is clear that current increases with temperature, and finally a critical temperature is reached above which pyroelectric current begins to decrease.

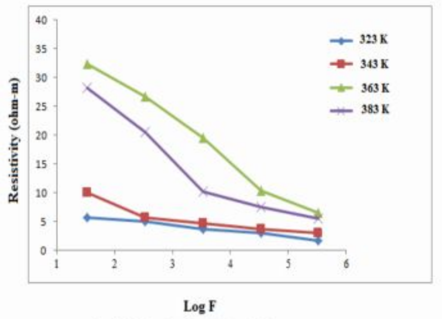


Fig. 16 Variation of resistivity with frequency

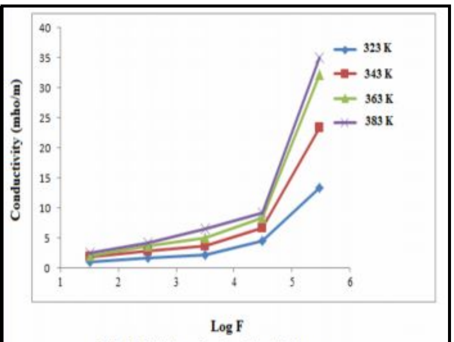


Fig. 17 Variation of conductivity with Frequency

In GMnAc crystal, the critical temperature is found to be 90^{o}Cwhich closely agrees with the literature [22].

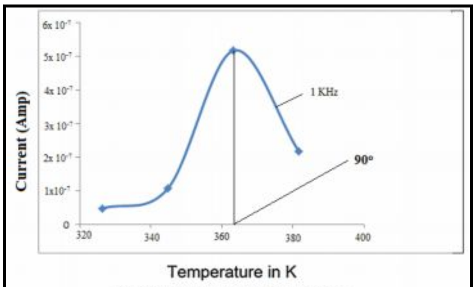


Fig. 18 Variation in current with Temperature

4. Conclusion

Glycine Manganous Acetate optical quality single crystal have been grown by slow evaporation technique. The grown crystals are colourless and transparent in appearance. Single crystal X-ray diffraction study of the crystal revealed that the crystal belongs to non-Centro symmetric monoclinic system with space group Pand crystalline nature was confirmed by powder X-ray diffraction study. Its thermal behaviour was examined by DSC. The optical transmission study confirms that the GMnAc had good transparency in the entire visible region and there was no absorption of light to any appreciable extent in the visible range and the lower cut-off wavelength was found to be 276 nm. Vickers microhardness was calculated in order to understand the mechanical stability of the grown crystals. Dielectric measurements indicate that the dielectric constant of the crystal decreases with increasing frequency significantly to make the crystal a more interesting material in the microelectronics industry. The ac/dc conductivity studies are measured and the activation energy is determined. GMnAc is an electrically non conducting material at room temperature. Hence GMnAc crystal is found to be more beneficial from an application point of view.

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