# Growth, Mechanical and Impedance Studies of BETA-Alanine Single Crystals

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*Abstract*— **Beta-alanine (β-alanine) is an organic crystal that was grown by slow evaporation technique. The structure of the grown crystals was estimated by single crystal XRD analysis. The mechanical properties of β-alanine crystals were evaluated using Vickers microhardness tester by applying load from 25 to 100 g. The hardness parameters such as microhardness number, work hardening coefficient, yield strength and elastic stiffness constant of the grown β-alanine crystals were calculated from the microhardness studies. The impedance, Nyquist plot and conductivities were investigated for the β-alanine crystal as a function of frequency. The observed results are discussed.**

**Keywords**- **Beta-alanine; yield strength; elastic stiffness constant; impedance, Nyquist plot**

## I. INTRODUCTION

Amino acids are bifunctional organic molecules that contain a carboxyl group (–COOH) as well as an amino group ($NH_2$). Beta-alanine is an amino acid in which the amine group is at the β--position from the carboxylate group. β-alanine is also known as 3-aminopropionic acid and it is a positional isomer of L-alanine. Beta-alanine gives strength to athletic sports persons and raises the level of carnosine in the blood, reducing levels of lactic acid in the body during exercise [1, 2]. It occurs within the human central nervous system and some authors have suggested that it functions probably as a neuromodulator or perhaps even a neurotransmitter. It forms crystalline complexes with organic, inorganic acids and salts [3]. Beta-alanine and their complexes are being prepared by our research group and in this paper, investigations on growth, hardness parameters, impedance and conductivity of beta-alanine crystals were carried out.

## II. EXPERIMENTAL PROCEDURE

### *Crystal growth*

Beta-alanine crystals were grown by slow evaporation technique. The saturated solution of the re-crystallized salt of beta-alanine was prepared in accordance with the solubility and the optimized nucleation kinetic data. The solution was stirred well using a hot plate magnetic stirrer for 2 h and it was filtered using a Whatmann filter paper. Then the solution was allowed to evaporate and numerous tiny crystals were formed at the bottom of the container due to spontaneous nucleation. Seed immersion technique was used to obtain big-sized crystals. It took about 20 days to grow a beta-alanine crystal and the photograph of the harvested beta alanine crystal grown by slow evaporation technique is shown in figure 1.

Figure 1. Harvested β-alanine crystal

## III. RESULTS AND DISCUSSION

### *A. Single crystal X-ray Diffraction analysis*

The grown crystals were subjected to single crystal X-ray diffraction studies using an ENRAF NONIUS CAD4 diffractometer with Mo $K_\alpha$ radiation (λ = 0.71073 Å) to determine the lattice parameters. The obtained unit cell parameters are a=9.875(3) Å, b = 13.842(2) Å, c = 6.094(2) Å, α = β = γ = 90° and the volume of the unit cell is 833.24 $Å^3$. From the data, the grown beta-alanine crystal belongs to the orthorhombic system with the space group Pbca. The obtained values of cell parameters in this work are found to be in close agreement with the data reported in literature [4].

### *B. Microhardness studies*

The hardness of the crystals depends on type of chemical bonding, lattice energy, Debye temperature, heat of formation and interatomic spacing, which may differ along the crystallographic directions. Microhardness test provides useful information about the mechanical properties like elastic constants, yield strength, resistance pressure etc of the materials. Vickers hardness test is the most common and reliable method for hardness measurement of solid surfaces. The microhardness of the grown β-alanine crystals were measured using a Shimadzu Microhardness Tester with a diamond indenter. Well polished crystal was mounted on a platform of the microhardness tester and different loads were applied over a fixed interval of time (10 s). The length of the two diagonals of diamond indenter was measured by a calibrated micrometer attached to the eyepiece of the microscope after unloading and the average was found out. For a particular load, at least two well defined indentations were considered and the average value (d) was selected. Vickers hardness number was determined using the expression

$$H_v = 1.8544 \, P / d^2$$

where, ‘P’ is the applied load, ‘d’ is the average diagonal length of the indentation marks and the result is plotted (Figure 2). From the graph, the hardness value increases with the increasing load and hence the beta-alanine crystal exhibits the reverse indentation size effect (RISE). According to the Meyer's law, the relation between the load and size of indentation as $P=k_1d^{n,}$ where ‘$k_1$’ is the material constant, ‘n’ is the Meyer’s index or work hardening coefficient. The plot of log P against log d is shown in inset figure 2 which gives a straight line (after least square fitting). The slope of the line is 3.13. According to Onitsch and Hanneman, ‘n’ should lie between 1 and 1.6 for hard materials and should be above 1.6 for softer materials and the sample is a soft material.

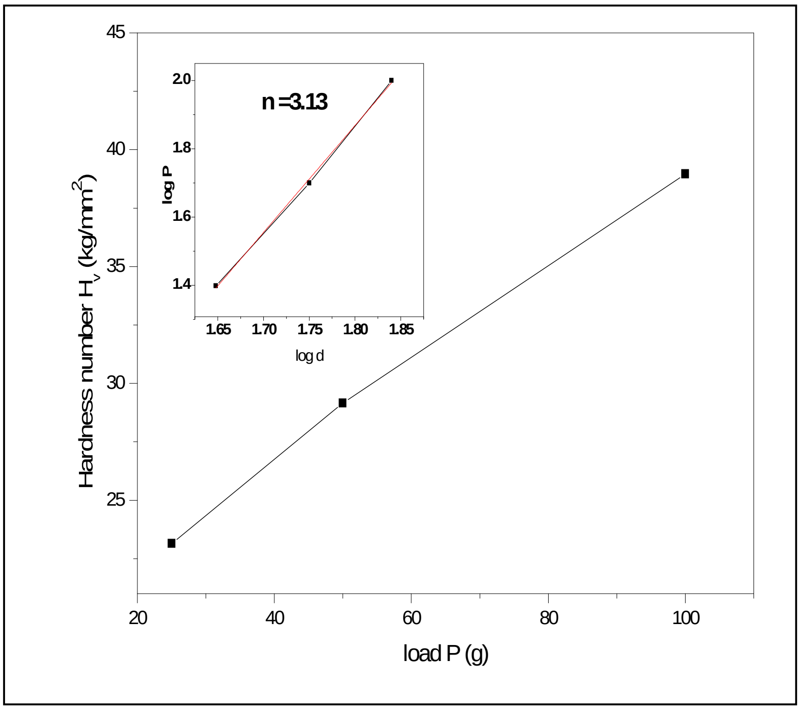


Figure 2. Variation of hardness number with load for beta-alanine crystal
Inset. Plot of log P versus log d for the beta-alanine crystal

Yield strength is the important property for the device fabrication and it is the maximum stress that can be developed in a material without causing plastic deformation. otherwise yield strength is a practical approximation of elastic limit. The yield strength ($\sigma_y$) can be calculated using the relation for n>2, [5]

$$\text{Yield strength } (\sigma_y) = (H_v/3)\ (0.1)^{n-2}$$

The yield strength for different loads is listed in table 1 and it is observed that yield strength increases with load. Hence the grown beta-alanine crystal has relatively high mechanical strength.

Elastic stiffness constant ($C_{11}$) was calculated by Wooster's empirical relation as

$$C_{11} = H_v^{7/4}$$

Stiffness constant for different loads calculated from Vickers hardness values are given in table 1. From the table, it is clear that the stiffness constant increases with increase of load. Hence the high value of $C_{11}$ indicates that the binding forces between the ions are quite strong.

Table 1: Values of yield strength and elastic stiffness constant of beta-alanine crystal

| Load P (g) | $\sigma_y$ (M Pa) | $C_{11}$ x $10^{14}$ Pa |
|---|---|---|
| 25 | 5.606 | 4.194 |
| 50 | 7.059 | 6.277 |
| 100 | 9.432 | 10.424 |

*C. Impedance studies*

Complex impedance spectroscopy is a powerful technique to investigate the electrical properties and process of the materials. This technique is based on analyzing the ac response of a system to a sinusoidal perturbation, and subsequent calculation of impedance and related parameters as a function of frequency of the perturbation. The motion of charges could occurs dipole reorientation, space charge formalism and charge displacement. The impedance measurements on a material give us data having both resistive (real part) and reactive (imaginary part) components. Electrical impedances were measured in a frequency ranging from 1 Hz to 1 MHz using an impedance analyzer with automatic bridge monitored by a microcomputer at room temperature. Transparent good quality crystal was polished and electronic grade silver paste was applied on either surface of the sample, which acts as an electrode so that it behaves like parallel plate capacitor.

The complex impedance Z* measurement [6, 7] can be expressed as a function of resistance R and capacitance C using the equation

$$Z^* = Z' - jZ''$$

where Z', Z'' are the real and imaginary parts of the impedance respectively. Figure 3 and its inset show the variation of real and imaginary parts of impedance of the grown beta-alanine crystal as a function of frequency at room temperature (30$^o$C). The results indicate that the real part of impedance decreases with rise in frequency and provides an indication of increasing conduction with frequency. High impedance behavior is noticed in the low frequency region. This suggests of the manifestation of the mixed nature of polarization behavior in the material. Inset (figure 3) shows the variation of imaginary part of impedance with frequency at room temperature and it is observed that imaginary part of impedance decreases with frequency. This graph is suitable for evaluation of the relaxation frequency of most resistive contribution which is temperature dependent.

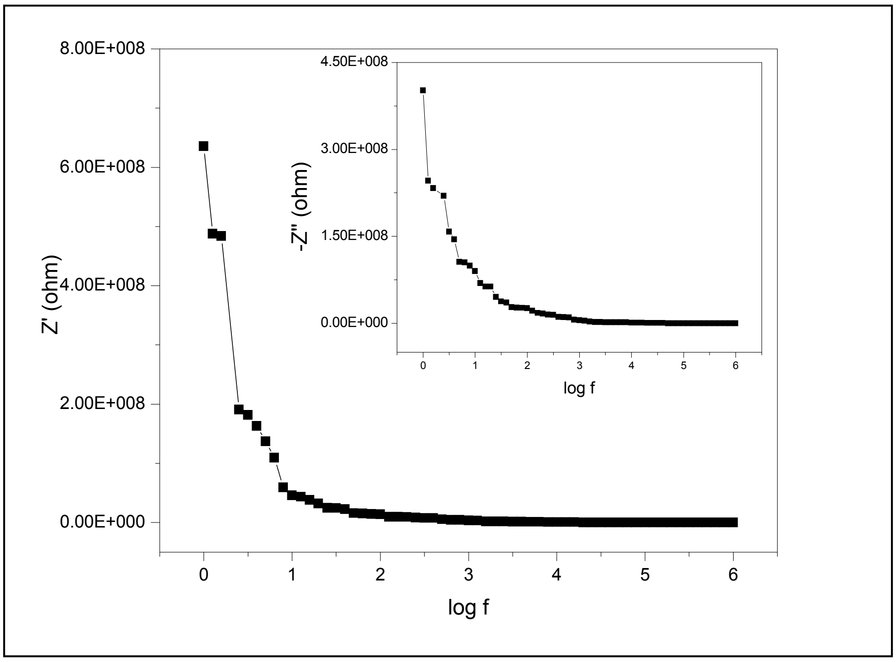


Figure 3.Variation of real part of impedance of the beta-alanine crystal with frequency.
Inset. Variation of imaginary part of impedance of the beta-alanine crystal with frequency.

Figure 4 shows the Nyquist plot between Z' and Z'' of the beta-alanine crystal at room temperature and this graph explains the transport response function. From the graph, one partial semicircular arc has been observed and this depicts the bulk effect. This bulk effect arises due to the parallel combination of bulk resistance ($R_b$). The value of bulk resistance is found to be $1.107 \times 10^8$ ohm and the observed value is typical for an insulating material.

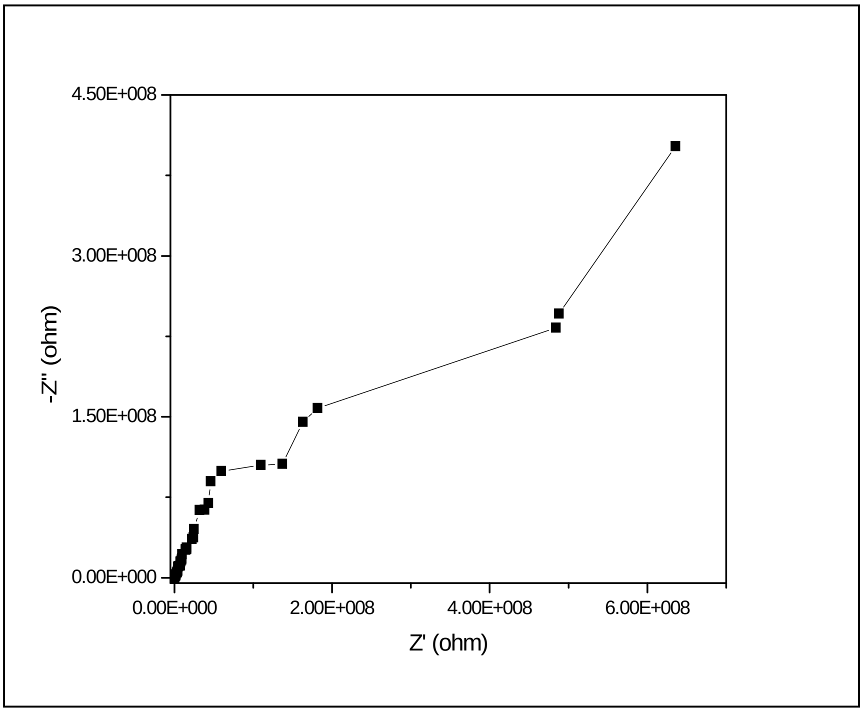


Figure 4. Nyquist plot or complex impedance plot for β-alanine crystal

The dc conductivity ($\sigma_{dc}$) of the crystal is calculated using the formula

$$\sigma_{dc}= d/R_bA$$

where A is the area of crystal surface and d is the thickness of the crystal. The dc conductivity was found to be $2.553x10^{-7}$ $m^{-1}$mho at $30^oC$. The low dc conductivity suggests that the number of defects or impurities present in the grown beta-alanine crystal is low.

The ac conductivity of the crystal at room temperature is evaluated from the real part of the impedance using the relation

$$\sigma_{ac}= d/A\ Z'$$

The ac conductivity of the beta-alanine crystal with different frequencies is given in table 2 and it is observed that ac conductivity increases with frequency. Thus the electrical conduction in the crystal takes place as a result of electrons jumping from the ions with low valence state to high valence state as well as the movement of ions due to the influence of an electric field.

Table 2: Values of ac conductivity of the beta-alanine crystal

| Frequency (Hz) | ac conductivity ( x $10^{-5}$ $m^{-1}$ mho) |
|---|---|
| 1 | 0.0044 |
| 10 | 0.0614 |
| $10^2$ | 0.1997 |
| $10^3$ | 0.7904 |
| $10^4$ | 4.8246 |
| $10^5$ | 129.9091 |
| $10^6$ | 22814.12 |

## V. CONCLUSION

Good quality single crystals of beta-alanine have been successfully grown by solution method with slow evaporation techniques. The cell parameters of the β-alanine were identified by single crystal XRD. The various hardness parameters such as hardness number, Meyer index, yield strength and elastic stiffness constant have been estimated from the various loads using Vickers microhardness tester. The electrical parameters such as the real part and imaginary part of impedance as a function of frequency at room temperature have been investigated by impedance spectroscopy. The ac conductivity of the grown crystal has been carried out. From the Nyquist plot, the dc conductivity of the β-alanine crystal was calculated.

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