CHAPTER – IV

THERMAL AND MECHANICAL PROPERTIES

4.1 Introduction

4.2 Thermal Analysis
   4.2.1 Thermal Analysis of LVZA
   4.2.2 Thermal Analysis of GM
   4.2.3 Thermal Analysis of TGBDD
   4.2.4 Thermal Analysis of GT
   4.2.5 Thermal Analysis of TuTGZC
   4.2.6 Thermal Analysis of BTSN
   4.2.7 Thermal Analysis of TPMS

4.3 Mechanical Analysis

4.4 Vickers hardness measurement
   4.4.1 Meyer’s Index (n)
   4.4.2 Resistance pressure (w) and correction factor(x)
   4.4.3 Elastic stiffness constant (C_{11}) and yield strength (\sigma_y)

4.5 Conclusion
CHAPTER - IV

THERMAL AND MECHANICAL PROPERTIES

4.1. Introduction

Thermal studies are very essential to understand the thermal stability of the material, for the device fabrication. Among several methods, thermo gravimetric (TG) and differential thermal analysis (DTA) are widely used to explore chemical as well as physical information about the sample. Mechanical properties of crystalline solids are intimately connected with their other physical and electrical properties and determine the performance of devices prepared from the solids. Consequently, there is a constant interest in assessing the mechanical properties of solids. Among the various experimental techniques for the determination of the mechanical properties, hardness testing is frequently used to assess the mechanical properties of solids in the form of bulk samples. Hardness may be termed as a measure of the resistance against lattice destruction or the resistance offered to permanent deformation or damage. Micro hardness studies are applied to understand the strength and deformation characteristics of a material since the hardness properties are basically related to the crystal structure of the material. Micro hardness indentation technique is used to study anisotropy, deformation, glide, state of dispersions of impurity, quench hardening, effect of irradiation and environment of dislocation mobility.

This chapter explains thermal studies to understand the thermal stability of the grown NLO crystals and a detailed description of mechanical properties.
4.2. Thermal Analysis

Thermal analysis is a group of techniques that study the properties of materials as they change with temperature. It is used to establish thermodynamic properties which are essential for understanding the behaviour of material under different heating and cooling rates, under inert, reduction or oxidation atmosphere or under different gas pressure. All materials, as they experience changes in temperatures, undergo changes in their physical and chemical properties. These changes can be identified by suitable transducers which convert the changes into electrical signals which are collected and analyzed to give thermograms showing properly change as function of temperature. To determine the thermo-physical properties the following methods are commonly used. They are:

1. Thermogravimetric analysis (TGA)
2. Differential thermal analysis (DTA)
3. Differential scanning calorimetry (DSC)

Thermal gravimetric analyzer measures the change in mass of a sample as a function of time or temperature in inert or oxidative atmosphere. TGA relies on a high degree of precision in three measurements: weight, temperature and temperature change. As many weight loss curves look similar, the weight loss curve may require transformation before results may be interpreted. A derivative weight loss curve can be used to tell the point at which weight loss is most apparent. TGA is commonly used in research to determine the physical and chemical characteristics of materials, to determine degradation temperatures,
absorbed moisture content of materials, the level of inorganic and organinc components in materials and solvent residues.

A simultaneous TGA-DTA measures both heat flow and mass changes in a material as a function of temperature or time in a controlled atmosphere. Simultaneous measurement of these two material properties not only improves productivity but also simplifies interpretation of the results. In differential thermal analysis (DTA), the material under study and an inert reference are made to undergo identical thermal cycles, while recording any temperature difference between sample and reference. The reference material is an inert substance such as alumina, silicon carbide or glass beads. The differential temperature is then plotted against time, or against temperature (DTA curve or thermogram), changes in the sample, either exothermic or endothermic, can be detected relative to the inert reference. Thus, a DTA curve provides data on the transformations that have occurred, such as crystallization, melting and sublimation.

Differential scanning calorimetry is a dynamic thermal analysis technique which is used to study the thermal behaviour of any sample, over a wide temperature range, under non isothermal conditions. Whenever a material under goes a physical change such as melting or transition from one crystalline form to another or whenever it reacts chemically, heat is either liberated or absorbed.

In the present study thermal analysis was carried out using **SDT Q600V 8.3 build 101 simultaneous DTA/TGA analyzer** in the nitrogen atmosphere. The thermo gravimetric analysis (TGA) and differential thermal analysis
(DTA) were carried out for all samples in the temperature range 20-1200°C at a heating rate of 20°C/min in nitrogen atmosphere. The heat capacity at constant pressure $C_p$ of crystal was measured by differential scanning calorimetric (DSC) analysis in the temperature range 20-1200°C at the heating rate for the system calibration.

4.2.1. Thermal analysis of LVZA

The recorded TGA/DTA spectra of the LVZA sample are shown in Fig. 4.1. Heating was carried out at a rate of 20°C/min. The initial mass of the material subjected to analysis was 2.9970 mg and the final was only 0.3630% of the initial mass at temperature of about 1100°C indicating bulk decomposition occurring into LVZA crystals. From the TGA curve, the thermal stability of the sample is realized up to 163°C and thereafter the material shows loss in weight due to the molecules, which are loosely bounded to the metal ion. The major 70% of weight loss between 208°C to 279°C is due to the liberation of volatile substances.

![Fig. 4.1: TGA/DTA curves of LVZA](image)
It is observed from DTA curve, that the material undergoes exothermic transition at about 217°C where the decomposition starts. The material is fully decomposed at an irreversible endothermic transition at about 286°C. It is inferred that the melting point of the material takes place in the vicinity of 217°C. The sharpness of the endothermic peak shows good degree of crystallinity of the grown LVZA. The observed DSC spectrum of the LVZA crystal is shown in Fig. 4.2. From DSC spectrum, the specific heat capacity of the LVZA crystal is 649 J/g°C observed at 286°C.

Fig. 4.2: DSC curve of LVZA

4.2.2. Thermal analysis of GM

A small piece of crystal weighing 2.0280 mg was used for this investigation. From Fig. 4.3, it is evident that the compound GM has good thermal stability up to 153.47°C as there is no major weight loss below that temperature. The TGA curve also shows that there was a weight loss of about
59% in the temperature range 153-520°C due to the liberation of volatile substance in the compound. The DTA curve shows the initial exothermic peak at 144.85°C, the melting point of the substance. Then it undergoes an irreversible endothermic at 167.66°C which shows that the decomposition of the material. The second endothermic peak at 308.68°C may be due to the liberation of glycine in GM, as glycine is known to decompose at 233°C [195]. Major decomposition of the compound is indicated by endothermic peak at 521.78°C. The heat capacity at constant pressure $C_p$ of GM crystal was measured by differential scanning calorimetric (DSC) analysis. Crystal weighing about 2.0280 mg was placed in a sealed alumina DSC pan. The DSC curve of GM is shown in Fig. 4.4. The specific heat of GM crystal at 167.53°C was found to be 543.3 J/g/°C. This endothermic peak temperature is well agreed with DTA curve.

**Fig. 4.3: TGA/DTA curves of GM**
4.2.3. Thermal analysis of TGBDD

TG/DTA analysis of the TGBDD crystal was carried out in the temperature range 10 - 800°C. The recorded thermogram is shown in Fig. 4.5. TGA curve shows the weight loss at three stages, one occurring below 163°C due to weakly entrapped lattice water and the other one occurring above it due
to the removal of strongly entrapped lattice water. This is followed by a major weight loss at 317°C due to 46% decomposition of the substance.

There is an endothermic transition between 163 and 317°C which is in good agreement with the TGA trace. The first sharp endothermic peak at around 163°C is assigned the melting point of the TGBDD compound and the second sharp endothermic peak at around 317°C corresponds to the major decomposition of the material.

4.2.4. Thermal analysis of GT

The thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) were done for the sample of weight 2.2760 mg. GT thermogram is shown in Fig. 4.6. From TGA it is evident that the compound GT has good thermal stability up to 180.57°C as there is no weight loss below that temperature. The TGA curve also shows that there was a weight loss of about 81.95% in the temperature range 180.57 - 286.50°C due to the liberation of volatile substance in the compound. The DTA curve shows the first exothermic peak at 156.34°C, the melting point of the substance and then it undergoes two irreversible endothermic transitions. The first endothermic peak at 182.42°C in the DTA curve may be due to the liberation of thiourea and the second endothermic peak at 229.84°C may be due to the liberation of glycine in GT, as glycine is known to decompose at 233°C [195]. The second exothermic peak at 296.97°C in the DTA curve indicates the major decomposition of the material.

The heat capacity at constant pressure, Cp, of GT crystal was measured by
differential scanning calorimetric (DSC) analysis. Powdered sample weight of 2.2760 mg was placed in a sealed aluminum DSC pan. The DSC curve of GT is shown in Fig. 4.7. The specific heat of GT crystal at 230.03°C was found to be 1000 J/g/°C.

Fig. 4.6: TGA / DTA curves of GT

Fig. 4.7: DSC curve of GT
4.2.5. Thermal analysis of TuTGZC

TGA/DTA analysis of the TuTGZC crystal was carried out in the temperature range 10 - 800°C. The recorded thermogram is shown in Fig. 4.8. From TGA curve it is observed that the weight loss start from 192°C. There is 53% weight loss between 269°C and 472°C. This weight loss is due to the liberation of chlorine atoms and glycine molecule. As there is no weight loss below 192°C, it indicates that the crystal is devoid of any physically adsorbed water in it. The DTA curve shows a sharp endothermic peak at 244.6°C which indicates the melting point of the crystal. This endothermic event is in good agreement with the TGA trace.

![TGA/DTA curves of TuTGZC](image)

**Fig. 4.8: TGA/DTA curves of TuTGZC**
4.2.6. Thermal analysis of BTSN

A powdered sample of BTSN weighing 2.3150 mg was used for the analyses. TGA Fig. 4.9 shows a weight loss at 53.69°C which may be due to the presence of physically adsorbed water molecule in the sample [88]. The TGA curve also shows that there was a major weight loss of about 65.81% in the temperature range 182 - 242°C. The nature of the weight loss indicates the decomposition point of the material. The DTA curve shows that the first exothermic peak at 164.82°C and then the sample undergoes two irreversible endothermic transitions. The first endothermic peak at 186.40°C may be due to the liberation of volatile substances and the second intense endothermic peak at 235.4°C indicates the major decomposition point of the material. The decomposition temperature of thiourea is 182°C [196]. The decomposition temperature of BTSN at 235.4°C shows the formation of metal complex of thiourea. The decomposition temperature of BTSN is higher than that of other
semi organic crystals of thiourea like BTNC [197] (221.08°C), TTPI [187] (183.3°C) and CTA [198] (206°C). The increase in decomposition temperature of semi organic materials when compared with other organic crystals arises due to the stronger bonding existing between the conjugation layers of thiourea molecules and metal ion [199]. High temperature differential scanning calorimetry spectrum of the BTSN is shown in Fig. 4.10. The scan was run from 20°C temperature to 1200°C, two peaks were observed. The specific heat of BTSN crystal at 234.64°C was found to be 1314 J/g/°C.

![DSC curve of BTSN](image)

**Fig. 4.10: DSC curve of BTSN**

### 4.2.7. Thermal analysis of TPMS

Thermal analysis was carried out for a sample of weight 2.5510 mg. TGA/DTA thermogram of TPMS is shown in Fig. 4.11. From TGA it is observed that the crystal TPMS has good thermal stability up to 223.68°C as there is no weight loss below that temperature. The absence of weight loss
around 100°C showed that the absence of water molecule in TPMS crystal during the crystallization process [145]. The TGA curve also shows that there was a weight loss of about 55.30% in the temperature range 223.68 - 362.88°C due to the liberation of volatile substance in the compound. The DTA curve shows the exothermic peak at 218.75°C, the melting point of the substance and it undergoes irreversible endothermic transition. The second exothermic peak at 306.78°C indicates the major decomposition of the material. The sharpness of the endothermic peak show good degree of crystallinity of the grown crystal TPMS. The heat capacity at constant pressure $C_p$ of TPMS crystal was measured by differential scanning calorimetric (DSC) analysis in the temperature range 20 - 1200°C at the heating rate for the system calibration. Powdered sample weight of 2.5510 mg was placed in a sealed alumina DSC pan. The curve of TPMS is shown in Fig. 4.12. The specific heat of TPMS crystal at 246.32°C was found to be 347.6 J/g/°C.

![Fig. 4.11: TGA/DTA curves of TPMS](image-url)
4.3. Mechanical Analysis

Hardness is a technique, in which a crystal is subjected to a relatively high pressure within a localized area. By suitable choice of indenter material and relatively simple equipment construction, hardness tests can be easily carried out on all bulk materials under various conditions of temperature and pressure. There are three general type of hardness measurements used. They are

1. Scratch hardness: Scratch hardness is the ability of material to scratch on one another. Scratch hardness measurement is important to mineralogists.
2. Rebound or dynamic hardness: In dynamic hardness measurement, the indenter is dropped onto the metal surface and the hardness is expressed as the energy of impact.
3. Indentation hardness: Indentation hardness is the major important engineering interest for metals and crystalline structures. Since the technique is based on the production of indentations in small areas on the surface by pressing a hard indenter of specified geometry into the solid, it is essentially non-destructive in contrast to the methods involving compression, extension or bending of bulk samples. Indentation hardness testing has also become increasingly important to industries involved with micro machines, micro electronics and magnetic recording.

The term micro hardness test usually refers to static indentations made with loads not exceeding 1 kg force. In static indentation test, the specific geometry is pressed into the surface of a test specimen under a known load. The indenter may be diamond cone or diamond pyramid. Upon removal of the indenter, a permanent impression is retained in the specimen. The hardness is calculated from the area or depth of indentation produced. The variables are the type of the indenter or load. In the pyramid shape indentation test, the indenter is pressed perpendicularly in the surface of the sample by means of an applied load. Then by measuring the cross sectional area or the depth of the indentation and knowing the applied load an empirical hardness number may be calculated. The most popular methods of the static indentation test are [200],

- **Brinell hardness measurement**

  This test is used for metal surfaces. The Brinell hardness number is expressed as the load divided by surface area of the indentation. This test has
limitations on small specimen or in critically stressed parts where indentation could be a possible site of failure.

- **Meyer hardness measurement**

  Meyer hardness is a more fundamental measure of indentation hardness. The hardness is expressed as the mean pressure between the surface of the indenter and the indentation. This type is less sensitive to the applied load than Brinell hardness and is rarely used for practical harness measurement.

- **Knoop indentation hardness measurement**

  This method is used for measuring in a small area, particularly useful for the study of highly brittle materials due to small depth of penetration for a given indenter load. The Knoop hardness number is the applied load divided by the unrecovered projected area of the indentation.

- **Rockwell hardness measurement**

  This method is the most widely used hardness test and the hardness is measured according to the depth of indentation under a constant load.

- **Vickers hardness measurement**

  Vickers hardness test provide a fairly wide acceptance for research work because it provides a continuous scale of hardness, for a given load.

4.4. **Vickers hardness measurement**

  Vickers hardness tester, Leitz mini load fitted with a diamond pyramidal indenter attached to an incident light microscope is used for the measurement.
In this method, micro indentation is made on the surface of a specimen with the help of a diamond indenter. The Vickers pyramid indenter where opposite faces contain an angle ($\alpha = 136^\circ$) is most widely accepted pyramid indenter. A pyramid is suited for hardness tests due to the following two reasons.

1. The contact pressure for a pyramid indenter is independent of indent size.
2. Pyramid indenters are less affected by elastic release than other indenters.

In the present case micro hardness measurements were made using Shimadzu HMV-2 micro hardness tester fitted with a Vickers diamond pyramidal indenter. The transparent polished crystal free from cracks was selected for hardness measurements. The indentation time was kept as 5s for all the loads. The Vickers hardness ($H_v$) was calculated from the relation [201],

$$H_v = \frac{1.8544P}{d^2} \text{kg/mm}^2 \quad \ldots \quad (4.1)$$

where $P$ is the applied load and $d$ the average length of the diagonal of the indentation mark. With $P$ in g and $d$ in $\mu$m, the units of $H_v$ turned out to be kg/mm$^2$. Indentations were carried out at different loads in the range 25 - 100 g. The variations of micro hardness with applied load for the prominent (100) plane of all the crystals are shown in Fig. 4.13. In all the cases, the hardness values increase with the increase of the applied load. Usually a decrease in apparent micro hardness with increasing applied load which may be arised due to work hardening, initiate plastic deformation, size of dislocation loops etc., And this phenomenon known as normal indentation size effect (ISE). In the
present studies, the hardness number increases with the increasing of applied loads. This behaviour of increasing micro hardness with the load known as reverse indentation size effect (RISE) [202], which is also attributed due to existence of distorted zone near crystal medium interface, effect of vibrations, specimen chipping, etc., and hence the plastic deformation is dominant. At low loads or strains, plastic deformation of crystals mainly involves the nucleation of dislocations along a particular slip system.

The RISE effect can be qualitatively explained on the basis of the depth of penetration of the indenter [203]. At small loads, the indenter penetrates only the surface layers and therefore, the effect is shown sharply at the early stages. When the applied load increases, the penetration depth also increases and the overall effect must be due to the surface and inner layers. Consequently in this stage, indentation depth increases proportionally with applied pressure.

![Fig. 4.13: Variation of Vickers constant as a function of load](image)
4.4.1. Meyer’s index (n)

Kick law proposed the relation between load $P$ and indentation length $d$ is given by,

$$P = k_1 d^n$$

...(4.2)

where, $n$ is the Meyer’s index or work hardening coefficient which is found out from the slope of the plot of log $P$ and log $d$ (after the least square curve fitting) gives a straight line as shown in Fig. 4.14 and $k_1$ is the standard hardness, which is found by the intercept.

In the present investigation, the work hardening coefficient ($n$) for all the crystal samples is given in Table 4.1. Kick’s analysis for hardness postulates, a constant value of $n = 2$ for all indenters and impressions geometrically similar to each other. However, Kick’s law (eq.(4.2)) has not been widely accepted due to the fact that $n$ usually has a value less than 2, is specially in the low hardness region. Onitsch pointed out that an $n$ lies between 1 and 1.6 for hard materials and it is more than 1.6 for soft materials [204]. The $n$ values observed in the present analysis suggesting that all crystals are moderately softer substances except TGBDD. Also it is found that, the work hardening index of LVZA, GM and GT amino acid crystals are greater than the thiourea crystals TuTGZC, BTSN and TPMS. It shows that the amino acid crystals are softer than the thiourea based crystals.
4.4.2. Resistance pressure \((w)\) and Correction factor \((x)\)

By Hays and Kendall’s theory of resistance pressure, there is a minimum level of indentation \((w)\), also known as resistance pressure, below which no plastic deformation occurs. Based on this, a relationship between indentation test load and indentation size by modifying Kick’s law is given by,

\[
P - w = k_2d^2 \tag{4.3}
\]

where \(k_2\) is another constant and \((P - w)\) is the effective indentation test load considered in the micro hardness calculation. Thus

\[
w = k_1d^n - k_2d^2
\]

\[
d^n = \frac{w}{k_1} + \left(\frac{k_2}{k_1}\right)d^2 \tag{4.4}
\]
The plot of $d^n$ versus $d^2$ is a straight line having a slope $k_2/k_1$ and an intercept $w/k_1$. From the Fig. 4.15 - 4.17, the value of $w$, which is the material resistance to the initiation of plastic flow is calculated and are given in Table 4.1.

It is known that material takes some time to revert to elastic mode after the applied load is removed. So a correction $x$ is applied to the observed $d$ value. Kick’s law may be satisfied as given below:

\[ P = k_2(d + x)^2 \quad \ldots (4.5) \]

\[ k_1d^n = k_2(d + x)^2 \quad \ldots (4.6) \]

Therefore,

\[ d^n = \left( \frac{k_2}{k_1} \right)^{1/2} d + \left( \frac{k_2}{k_1} \right)^{1/2} x \quad \ldots (4.7) \]

The value of $x$ was determined from intercept of straight line obtained by plotting $d$ versus $d^{n/2}$, slope yields $\left( \frac{k_2}{k_1} \right)^{1/2}$. The value of $x$ gives an idea about the nature of the dislocations (edge and screw) in a particular orientation of the crystal [205]. The calculated correction factor $x$ for all crystal are given in Table 4.1.
Fig. 4.15: $d^2$ versus $d^n$ of TuTGZC, BTSN & TPMS

Fig. 4.16: $d^2$ versus $d^n$ of LVZA & TGBDD

Fig. 4.17: $d^2$ versus $d^n$ of GM & GT
Table 4.1: Mechanical parameters of the chosen compounds

<table>
<thead>
<tr>
<th>Mechanical Parameter</th>
<th>LVZA</th>
<th>GM</th>
<th>TGBDD</th>
<th>GT</th>
<th>TuTGZC</th>
<th>BTSN</th>
<th>TPMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness at 25 g</td>
<td>27.90</td>
<td>36.39</td>
<td>63.03</td>
<td>58.44</td>
<td>64.3</td>
<td>79.58</td>
<td>65.4</td>
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<tr>
<td>Meyer’s Index (n)</td>
<td>4.17</td>
<td>5.10</td>
<td>1.09</td>
<td>4.26</td>
<td>2.31</td>
<td>2.67</td>
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<td>$k_1$ (kg/m)</td>
<td>$1.48 \times 10^7$</td>
<td>$5.62 \times 10^8$</td>
<td>$2.03 \times 10^3$</td>
<td>$7.46 \times 10^7$</td>
<td>$1.19 \times 10^5$</td>
<td>$4.94 \times 10^5$</td>
<td>$8.41 \times 10^5$</td>
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<td>$k_2$ (kg/m)</td>
<td>$4.88 \times 10^4$</td>
<td>$3.39 \times 10^5$</td>
<td>$-11.90 \times 10^3$</td>
<td>$9.70 \times 10^4$</td>
<td>$0.48 \times 10^5$</td>
<td>$1.46 \times 10^6$</td>
<td>$0.68 \times 10^5$</td>
</tr>
<tr>
<td>Material Resistance $W$ (g)</td>
<td>$-6.07 \times 10^3$</td>
<td>$-86.98 \times 10^3$</td>
<td>$15.09 \times 10^3$</td>
<td>$-64.22 \times 10^3$</td>
<td>$-85.4 \times 10^3$</td>
<td>$-15.64 \times 10^3$</td>
<td>$-24.05 \times 10^3$</td>
</tr>
<tr>
<td>Correction Factor $X$ (µm)</td>
<td>24.25</td>
<td>25.48</td>
<td>-29.38</td>
<td>18.27</td>
<td>4.95</td>
<td>7.66</td>
<td>10.29</td>
</tr>
<tr>
<td>Stiffness Constant $C_{11}$ (GPa) at 25 g</td>
<td>0.103</td>
<td>0.164</td>
<td>0.430</td>
<td>0.377</td>
<td>0.446</td>
<td>0.648</td>
<td>0.459</td>
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<tr>
<td>Yield Strength $\sigma_y$ (MPa) at 25 g</td>
<td>91.2</td>
<td>118.9</td>
<td>206</td>
<td>191</td>
<td>210.1</td>
<td>256.8</td>
<td>213.7</td>
</tr>
</tbody>
</table>
4.4.3. Elastic stiffness constant \( (C_{11}) \) and yield strength \( (\sigma_y) \)

The elastic stiffness constant \( C_{11} \) gives an idea about tightness of bonding between neighbouring atoms. The stiffness constant for different loads has been calculated using Wooster’s empirical formula [206],

\[
C_{11} = H_v^{7/4}
\]  \hspace{1cm} \text{... (4.8)}

The calculated stiffness constant at 25 g for all crystals are given in Table 4.1. It is identified that the stiffness constant increases with the increase of load and are shown in Figs. 4.18 - 4.19. High value of \( C_{11} \) indicates that the binding forces between the ions are quite strong.

Fig. 4.18: Variation of stiffness constant with hardness number for LVZA, GM, TGBDD & GT

Fig. 4.19: Variation of stiffness constant with hardness number for TuTGZC, BTSN & TPMS
The microhardness value correlates with other mechanical property namely yield strength $\sigma_y$. Yield strength is a point at which material exceeds the elastic limit and will not return to its origin shape or length if the stress is removed. Yield strength is one of the important property for device fabrication which can be calculated by the relation,

$$\sigma_y = \frac{H_v}{3}$$  \hspace{1cm} ... (4.9)

The calculated values of yield strength of all crystals at 25 g are presented in Table 4.1. The variation of the yield strength with hardness number for all the chosen compounds are shown in Figs. 4.20 - 4.21.

![Fig. 4.20: Variation of yield strength with hardness number for LVZA, GM, TGBDD & GT](image)

![Fig. 4.21: Variation of yield strength with hardness number for TuTGZC, BTSN & TPMS](image)
4.5. Conclusion

The thermal stability of the grown NLO crystals is investigated in detail with TGA/DTA/DSC thermograms. The melting point of the compounds is found from DTA analysis. The specific heat capacity of the samples is found from DSC studies. The Vickers hardness values are measured for all the crystals and the variations of hardness with load are discussed. The mechanical parameters such as Meyer’s index, material resistance, correction factor, stiffness constant and yield strength are calculated. Thus the thermal and mechanical studies reveal the thermal and mechanical strength of the grown NLO crystals.