EFFECT OF Sn INCORPORATION ON STRUCTURAL, PHYSICAL AND ELECTRICAL PROPERTIES OF Ge-Se-Sb-Sn THIN FILMS

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The present work reports the analysis of physical, structural and electrical properties alongwith their compositional dependence for (Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{100-x}Sb\textsubscript{x}Sn\textsubscript{x} (x = 2, 4, 6, 10 at %) chalcogenide glassy system. XRD spectra show the amorphous nature of the prepared samples with the possibility of short-range ordering in the samples. Chemically ordered network model, consistent with (8 − N) rule, has been utilized in estimating various physical parameters which reveals that Ge-Se-Sb-Sn glassy systems are thermally stable glasses. The current transport mechanisms have been investigated as a function of temperature in the temperature range 293 K − 363 K revealing that dc-conduction occurs through an activated process with single activation energy. Meyer-Neldel rule is found to be obeyed. The present study suggests Ge-Se-Sb-Sn glassy system to be an optimal material for utilization in optical devices.

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1. Introduction

The group 16 elements of the periodic table – sulphur (S), selenium (Se) and tellurium (Te) are referred to as chalcogens. The group 16 elements doped with elements of group 13 to 15 of the periodic table forms chalcogenide glasses\textsuperscript{[1]}. Chalcogenide glasses, having an energy gap of 3 eV, show semiconducting behaviour\textsuperscript{[2]}. Semiconductors were presumed earlier to exhibit long-range order. For analyzing band structure in amorphous semiconductors, the chemical bonding environment is given more importance than the periodicity\textsuperscript{[3]}. The molecular bonding in chalcogenide glasses is covalent in nature and the components are kept together with attractive van der waals forces. In chalcogenide glasses, the lone pairs enhance the bending of the bond angles and hence reduce the strain energy of the system\textsuperscript{[4]}. Hence the atoms form amorphous structures readily when doped with other elements. Elemental or binary glassy system exhibit one or two dimensional chain-like or layered structures and thus possess short-range order over a larger extent. In contrast ternary, quaternary and higher multi-component glassy systems exhibit rigid three-dimensional structure and thus possess short-range order over a shorter extent\textsuperscript{[5]}

Chalcogenide glasses are attracting a rigorous research essentially due to their excellent chemical stability in aggressive environments, transparency in the near and far-infrared region, low optical losses, high refraction coefficient and relatively high ion conductivity [6]. The distinctive properties viz. lesser phonon energy, larger and wider bandgap, high values of linear as well as non-linear refractive index, extraordinary transmittance range, etc. of these glasses find them a wide usage in photo-detectors, LED, IR sensors, holography and waveguides\textsuperscript{[7]}. Owing to high refractive index values and high non-linearity (~ 10\textsuperscript{2} times of silica), chalcogenides are utilized as an ultrafast switch\textsuperscript{[8]}. Due to high chemical and thermal stability, chalcogenide materials ease fabrication of optical devices. Metallic doping in chalcogenides alters the average coordination number as well as produce structural changes viz. flexible ↔ intermediate ↔ rigid, in

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the glassy matrix[9]. In chalcogenide glasses, almost all the properties are compositional dependent. Therefore the physical, optical, electrical or structural properties can be altered and controlled depending upon utilization requirement[10].

Amorphous Se has been reported to have vast application in various fields like solar technology, lubricants, pharmaceuticals as well as metal coating[11]. It also shows a remarkable feature of reversible phase transformation (amorphous to crystalline transformation). This feature is very useful for optical memory devices. But pure and unalloyed Se has few shortcomings because of its lesser sensitivity, smaller lifetime, poor thermal stability and higher aging effects. To overcome these drawbacks, pure Se is doped with elements like gallium (Ga), germanium (Ge), tin (Sn), arsenic (As), bismuth (Bi), antimony (Sb), lead (Pb), etc. which gives smaller aging effects, higher crystallization temperature, greater hardness, significantly higher sensitivity and high conductivity[12, 13].

The Ge-Se system is being studied extensively these days. When Ge is added to Se, Ge atoms serve as bond modifiers. Ge-atoms cross-link the Se chain structures, enhancing the average bond strength and hence increasing the glass transition temperature[14]. The mechanical constraints theory of Phillips and Thorpe predicts the critical composition for binary chalcogenide glasses of IVₓ−VI₁₀₀₋ₓ group, at around x = 20 at % with an average coordination number2.4. For all known Ge-Se systems, GeₓSe₂₀ with average coordination number 2.4, is the most critical composition[15]. The addition of Sb in Ge-Se system favours the glass formation and has a remarkable influence on the electrical conductivity of Ge-Se binary chalcogenide glasses. Moreover, Ge-Se-Sb glasses have been selected in the manufacturing of optical devices for the reason of having high transparency in the near and mid-infrared regions as well as for possessing great thermal, chemical and elastic properties[16].

In the present work, Sn has been selected as a chemical modifier in Ge-Se-Sb ternary system which may further broaden the glass forming area and can also produce structural and compositional disorder in the system in respect of the ternary glassy system. (Geₓ₂₀Se₈₀)₉₀₋ₓSbxSnₓ (x = 2, 4, 6, 10 at %) glassy systems have been synthesized using melt-quenching technique. The thin films, for the mentioned glassy systems, have been synthesized on glass substrates using the vacuum evaporation technique. The thin films are studied structurally by X-ray diffraction. The physical parameters – mean bond energy, average coordination number, glass transition temperature, average number of constraints, cohesive energy, lone pair electrons, heat of atomization, etc. are deduced using differential empirical approaches and their compositional dependence is also discussed in detail. The current conduction mechanism has also been analyzed as a function of temperature within the temperature range 293 K – 363 K of Ge-Se-Sb-Sn thin films.

2. Experimental details

Glassy alloys of (Geₓ₂₀Se₈₀)₉₀₋ₓSbxSnₓ (x = 2, 4, 6, 10 at %) were prepared using conventional melt-quenching technique. The constituent elements (5N pure) were weighed according to their atomic percentages and vacuum-sealed in quartz ampoules (outer diameter ~ 1 cm, inner diameter ~ 0.8 cm and length ~ 12 cm) in a vacuum of 2 × 10⁻⁵ mbar. The vacuum-sealed ampoules were placed inside a furnace and the temperature was raised upto 1100 °C at a constant heating rate of 3–4 °C min⁻¹. The ampoules were wobbled regularly for 24 hours at the maximum temperature for making the melt uniform. The quenching was performed in the ice-cold water to get the ingots that were crushed later in the mortar pestle assembly to get the fine powder of the glassy material.

For the preparation of the thin films, Corning 7059 glass slides were utilized as substrates which were cleaned to wash out any particulate contamination. Cleaning methodology included initial washing with water, further cleaning with acetone or ethanol and lastly rinsing with deionized water. Thin films of the glassy alloys were prepared using vacuum evaporation technique [vacuum coating unit HINDHIVAC, MODEL: VS-65D] using the inert gas condensation (IGC) technique in the presence of argon (Ar) as inert gas at room temperature and base pressure of
2 × 10\(^{-5}\) mbar. The thin films were placed inside the deposition chamber for 24 hours before taking measurements for achieving thermodynamic equilibrium. The powdered sample was evaporated by using molybdenum (Mo) boats. The deposition rate was kept slow so that the composition of the films nearly equals the composition of the starting bulk material. X-ray diffraction pattern (XRD) of the prepared thin films were recorded using Rigaku Miniflex-600 X-ray diffractometer operated at 40 kV using CuK\(_\alpha\) – wavelength (\(\lambda = 1.54056\) Å) at a scanning speed of 0.05°s\(^{-1}\) in the 2\(\theta\) range from 10° – 80°. For the electrical measurements, indium (In) electrodes are deposited on thin films (electrode spacing 0.8 mm) by thermal evaporation technique under base pressure of 2 × 10\(^{-5}\) mbar. Planar geometry of In electrodes is utilized because it establishes good and stable ohmic contacts for electrical measurements. The electrical measurements of the samples are studied using the two-probe method under vacuum conditions. These measurements are done by mounting the thin films in an especially devised metallic sample holder, under a vacuum of 2 × 10\(^{-3}\) mbar. The temperature dependence of dark conductivity (\(\sigma_d\)) is determined by fitting a small heater with a metallic base below the thin film in the sample holder. A direct current voltage is applied across the thin film using Keithley Electrometer, Model 6517-A, and current is noted using Pico-ammeter (DPM-111 Model). The variation of the temperature is controlled with a variac and recorded using Copper-Constantan thermocouple with one end attached to the sample and other end attached to the temperature sensor.

3. Results and discussion

3.1. Phase-identification

XRD techniques utilized for investigating the structural details of (Ge\(_{20}\)Se\(_{80}\))\(_{90-x}\)Sb\(_{10}\)Sn\(_x\) (\(x = 2, 4, 6, 10\) at %) chalcogenide glassy system. Fig. 1 shows XRD spectra of as-prepared (Ge\(_{20}\)Se\(_{80}\))\(_{90-x}\)Sb\(_{10}\)Sn\(_x\) (\(x = 2, 4, 6, 10\) at %) samples. It is clearly observed from Fig. 1 that the diffractions have no sharp peaks present for as-prepared samples, which verifies the amorphous nature of the prepared samples. The humps in the 2\(\theta\) range viz 25° – 35° and 45° – 55° indicate the possibility of short-range ordering in the glassy systems.

![Fig. 1. XRD spectra of (Ge\(_{20}\)Se\(_{80}\))\(_{90-x}\)Sb\(_{10}\)Sn\(_x\) (\(x = 2, 4, 6, 10\) at %) chalcogenide glasses.](image)

3.2. Physical properties

3.2.1. Number of constraints and average coordination number
The average coordination number (< r >) is described as the average atoms coordinated with its nearest neighbour of the constituents. It is quite helpful in understanding the cross-linking. For the composition (Ge$_2$Se$_{80}$)$_{80}$,Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %), < r > is given by[17]:

$$< r > = \frac{\alpha Z_{Ge} + \beta Z_{Se} + \gamma Z_{Sb} + \delta Z_{Sn}}{a + \beta + \gamma + \delta}$$  \hspace{1cm} (1)

where $\alpha$, $\beta$, $\gamma$, and $\delta$ are the atomic percentages of Ge, Se, Sb and Sn respectively; $Z_{Ge} = 4$, $Z_{Se} = 2$, $Z_{Sb} = 3$ and $Z_{Sn} = 4$ represent the coordination numbers (according to 8 − N rule of chemical bond approach with N being the number of valence electrons of respective element) of Ge, Se, Sb and Sn respectively. The values of < r >, as determined by Eq. 1, are listed in Table 1. It is observed that < r > increases as Sn-content increases. It shows that the cross-linking of chains among the atoms increases as the concentration of Sn increases.

In a glassy system, covalent networks are constrained mechanically by the inter-atomic valence forces namely bond bending and bond stretching. In optimal glass formation, the bond stretching constraints ($N_a$), the bond bending constraints ($N_b$) and the average number of constraints ($N_c$) are given as[18]:

$$N_a = \frac{< r >}{2} \hspace{1cm} (2)$$

$$N_b = 2 \times < r > - 3 \hspace{1cm} (3)$$

$$N_c = N_a + N_b \hspace{1cm} (4)$$

The calculated values of $N_a$, $N_b$ and $N_c$ for (Ge$_2$Se$_{80}$)$_{80}$,Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) systems are reported in Table 1. The chalcogenide glassy systems can be grouped into three categories viz. (i) over-coordinated or rigid glassy system having < r > greater than 2.4 and $N_c$ greater than 3; (ii) fully coordinated or ideal glassy system having < r > = 2.4 and $N_c = 3$; (iii) under-coordinated or spongy glassy system having < r > less than 2.4 and $N_c$ less than 3. The values of < r > and $N_c$ from Table 1 show that (Ge$_2$Se$_{80}$)$_{80}$,Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) glassy systems are rigid or over-coordinated. From literature[17, 18], it is inferred that the rigid systems have higher glass transition temperature and hence appears to be more thermally stable.

### 3.2.2. Lone pair of electrons and deviation of stoichiometry

In a chalcogenide glassy system, the number of lone-pair electrons (L) is given as[19]:

$$L = V - < r > \hspace{1cm} (5)$$

where $V$ corresponds to the valence electrons and is given as:

$$V = \frac{\alpha w + \beta x + \gamma y + \delta z}{a + \beta + \gamma + \delta} \hspace{1cm} (6)$$

where $\alpha$, $\beta$, $\gamma$, and $\delta$ are the atomic weight percentages of Ge, Se, Sb and Sn respectively; $w = 4$, $x = 6$, $y = 5$, $z = 4$ are the number of valence electrons of Ge, Se, Sb and Sn respectively. The presence of enough lone pairs leads to a stable non-crystalline state. The glass-forming ability is highly influenced by the interaction between the lone pair electrons of the crosslinking chalcogen atom and the cations present in the chalcogenide glassy system. The determined values of L and V are mentioned in Table 1. It is observed that the values of L decreases as Sn-concentration increases which is attributed to the enhanced interaction of Sn ions with the bridging Se atoms. The high values of L (L > 1) show that the investigated systems are good glass former[19].

The deviation of stoichiometry (R) is formulated as the ratio of probabilities of covalent bonding of chalcogen atoms to probabilities of covalent bonding of non-chalcogen atoms. For (Ge$_{20}$Se$_{80}$)$_{80}$,Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) glassy systems, R is defined as[19]:
\[ R = \frac{\beta Z_{\text{Se}}}{\alpha Z_{\text{Ge}} + \gamma Z_{\text{Sb}} + \delta Z_{\text{Sn}}} \]  
(7)

where \( \alpha, \beta, \gamma \) and \( \delta \) are the atomic weight percentages of Ge, Se, Sb and Sn respectively; \( Z_{\text{Ge}} = 4, Z_{\text{Se}} = 2, Z_{\text{Sb}} = 3 \) and \( Z_{\text{Sn}} = 4 \) represent the coordination numbers of Ge, Se, Sb and Sn respectively.

The occurrence of only heteropolar bonds is characterized by the critical value, \( R = 1 \). Also the critical value \( (R = 1) \) determines the least chalcogen concentration for which a chemically ordered glassy network can exist without any metal–metal bond formation. The values of \( R < 1 \) is correlated as a chalcogen-poor system whereas the values of \( R > 1 \) is correlated as a chalcogen-rich system.

The values of \( R \) are depicted in Table 1. From Table 1, it is observed that \((\text{Ge}_{20}\text{Se}_{80})_{90-x}\text{Sb}_{10}\text{Sn}_x\) (\( x = 2, 4, 6 \) and \( 10 \) at %) glassy systems are chalcogen-rich up to \( x = 6 \) at\% and become chalcogen-poor with the further increase in the concentration of Sn to \( x = 10 \) at\%.

**Fig. 2.** (a) Plot of lone pair electrons (L) with Sn-content for \((\text{Ge}_{20}\text{Se}_{80})_{90-x}\text{Sb}_{10}\text{Sn}_x\) (\( x = 2, 4, 6 \) and \( 10 \) at %) glassy systems. (b) Compositional dependence of deviation of stoichiometry (\( R \)) for \((\text{Ge}_{20}\text{Se}_{80})_{90-x}\text{Sb}_{10}\text{Sn}_x\) (\( x = 2, 4, 6 \) and \( 10 \) at %) glassy systems.

**Table 1.** Values of average coordination number \( (< r >) \), number of bond stretching constraints \( (N_a) \), number of bond bending constraints \( (N_b) \), total number of constraints \( (N_c) \), valence electrons \( (V) \), lone pair electrons \( (L) \) and deviation of stoichiometry \( (R) \) for \((\text{Ge}_{20}\text{Se}_{80})_{90-x}\text{Sb}_{10}\text{Sn}_x\) (\( x = 2, 4, 6 \) and \( 10 \) at %) glassy systems.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(&lt; r &gt;)</th>
<th>( N_a )</th>
<th>( N_b )</th>
<th>( N_c )</th>
<th>( V )</th>
<th>( L )</th>
<th>( R )</th>
</tr>
</thead>
<tbody>
<tr>
<td>((\text{Ge}<em>{20}\text{Se}</em>{80})<em>{90}\text{Sb}</em>{10}\text{Sn}_2)</td>
<td>2.492</td>
<td>1.246</td>
<td>1.984</td>
<td>3.230</td>
<td>5.508</td>
<td>3.016</td>
<td>1.299</td>
</tr>
<tr>
<td>((\text{Ge}<em>{20}\text{Se}</em>{80})<em>{90}\text{Sb}</em>{10}\text{Sn}_4)</td>
<td>2.524</td>
<td>1.262</td>
<td>2.048</td>
<td>3.310</td>
<td>5.476</td>
<td>2.952</td>
<td>1.199</td>
</tr>
<tr>
<td>((\text{Ge}<em>{20}\text{Se}</em>{80})<em>{90}\text{Sb}</em>{10}\text{Sn}_6)</td>
<td>2.556</td>
<td>1.278</td>
<td>2.112</td>
<td>3.390</td>
<td>5.444</td>
<td>2.888</td>
<td>1.109</td>
</tr>
<tr>
<td>((\text{Ge}<em>{20}\text{Se}</em>{80})<em>{90}\text{Sb}</em>{10}\text{Sn}_{10})</td>
<td>2.620</td>
<td>1.310</td>
<td>2.240</td>
<td>3.550</td>
<td>5.380</td>
<td>2.760</td>
<td>0.955</td>
</tr>
</tbody>
</table>

### 3.2.3. Glass transition temperature, mean bond energy and electronegativity

For any glassy system, the values of mean bond energy depend upon certain factors including average coordination number, different types of (homopolar or heteropolar) bonds, the degree of cross-linking per atom, etc. Mean bond energy is represented by \(< E >\) and is calculated using the relation described by Ticha et al[20, 21]:

\[ < E > = E_{\text{cl}} + E_{\text{rm}} \]  
(8)

where \( E_{\text{cl}} \) is the contribution emerging from strong heteropolar bonds and \( E_{\text{rm}} \) is contribution emerging from weaker bonds that form once the strong bonds are maximized. The average energy of cross-linking is calculated as:
where $E_{hb}$ is the average heteropolar bond energy for glasses with composition $\text{Ge}_x\text{Se}_y\text{Sb}_z\text{Sn}_\delta$. It is calculated using the relation:

$$E_{hb} = \frac{\alpha Z_{\text{Ge}}E_{\text{Ge-Se}} + \gamma Z_{\text{Sb}}E_{\text{Sb-Se}} + \delta Z_{\text{Sn}}E_{\text{Sn-Se}}}{\alpha Z_{\text{Ge}} + \gamma Z_{\text{Sb}} + \delta Z_{\text{Sn}}}$$  \hspace{1cm} (8c)

where $E_{\text{Ge-Se}}$, $E_{\text{Sb-Se}}$ and $E_{\text{Sn-Se}}$ are the heteropolar bond energies for Ge-Se, Sb-Se and Sn-Se respectively. The bond energies of the homopolar bonds are taken as $E_{\alpha}$ for Ge-Se, 44.00 kcal/mol, $E_{\beta}$ for Sb-Se and $E_{\gamma}$ for Sn-Se respectively. The degrees of cross-linking $P_p$ (for $R < 1$) and $P_r$ (for $R > 1$) are given by:

$$P_p = \frac{\beta Z_{\text{Se}}}{\alpha + \beta + \gamma + \delta}$$  \hspace{1cm} (8d)

$$P_r = \frac{\alpha Z_{\text{Ge}} + \gamma Z_{\text{Sb}} + \delta Z_{\text{Sn}}}{\alpha + \beta + \gamma + \delta}$$  \hspace{1cm} (8e)

The average energy of the remaining matrix is calculated as:

$$E_{\text{rm}} = \frac{2(0.5 < r > P_r)E_{\text{Se-Se}}}{< r >} \hspace{1cm} \text{for } R > 1$$  \hspace{1cm} (8f)

$$E_{\text{rm}} = \frac{2(0.5 < r > P_p)E_{\text{Se-Se}}}{< r >} \hspace{1cm} \text{for } R < 1$$  \hspace{1cm} (8g)

Hence in $\text{Ge}_x\text{Se}_y\text{Sb}_z\text{Sn}_\delta$ glassy system, for selenium-rich region ($R > 1$), we evaluate as:

$$E_{\text{cl}} = \alpha Z_{\text{Ge}}E_{\text{Ge-Se}} + \gamma Z_{\text{Sb}}E_{\text{Sb-Se}} + \delta Z_{\text{Sn}}E_{\text{Sn-Se}} = 4\alpha E_{\text{Ge-Se}} + 3\gamma E_{\text{Sb-Se}} + 4\delta E_{\text{Sn-Se}}$$  \hspace{1cm} (9a)

$$E_{\text{rm}} = \frac{(\beta Z_{\text{Se}} - \alpha Z_{\text{Ge}} - \gamma Z_{\text{Sb}} - \delta Z_{\text{Sn}})E_{\text{Se-Se}}}{< r >} = \frac{(2\beta - 4\alpha - 3\gamma - 4\delta)E_{\text{Se-Se}}}{< r >}$$  \hspace{1cm} (9b)

and for selenium-poor region ($R < 1$), we evaluate as:

$$E_{\text{cl}} = \frac{\beta Z_{\text{Se}}(\alpha Z_{\text{Ge}}E_{\text{Ge-Se}} + \gamma Z_{\text{Sb}}E_{\text{Sb-Se}} + \delta Z_{\text{Sn}}E_{\text{Sn-Se}})}{\alpha Z_{\text{Ge}} + \gamma Z_{\text{Sb}} + \delta Z_{\text{Sn}}} = \frac{2\beta(4\alpha E_{\text{Ge-Se}} + 3\gamma E_{\text{Sb-Se}} + 4\delta E_{\text{Sn-Se}})}{4\alpha + 3\gamma + 4\delta}$$  \hspace{1cm} (10a)

$$E_{\text{rm}} = \frac{(\alpha Z_{\text{Ge}} + \gamma Z_{\text{Sb}} + \delta Z_{\text{Sn}} - \beta Z_{\text{Se}})E_{\text{Se-Se}}}{< r >} = \frac{(4\alpha + 3\gamma + 4\delta - 2\beta)E_{\text{Se-Se}}}{< r >}$$  \hspace{1cm} (10b)

The bond energy of heteropolar bonds are calculated by the method suggested by Pauling[22]:

$$E_{\text{A-B}} = (E_{\text{A-A}} \times E_{\text{B-B}})^{1/2} + 30(\chi_A - \chi_B)^2$$  \hspace{1cm} (11)

where $E_{\text{A-B}}$ is the bond energy of heteropolar bond, $E_{\text{A-A}}$ and $E_{\text{B-B}}$ are the bond energies of homopolar bonds. The bond energies of the homopolar bonds are taken as $E_{\text{Ge-Ge}} = 37.60$ kcal/mol, $E_{\text{Se-Se}} = 44.00$ kcal/mol, $E_{\text{Sb-Sb}} = 30.20$ kcal/mol and $E_{\text{Sn-Sn}} = 34.20$ kcal/mol respectively. $\chi_{\text{Ge}} = 2.01$, $\chi_{\text{Se}} = 2.55$, $\chi_{\text{Sb}} = 2.05$ and $\chi_{\text{Sn}} = 1.96$ are the electronegativity values of Ge, Se, Sb and Sn respectively.

Table 2 lists the evaluated heteropolar bond energies.

Table 2. Bond energies of heteropolar bonds for (Ge$_{20}$Se$_{30}$)$_x$Sb$_{y}$Sn$_z$ ($x = 2, 4, 6$ and $10$ at %) glasses.
Using Eq. 8, 9 and 10, the values of \(< E >\) for (Ge\(_{20}\)Se\(_{80}\))\(_{30}\), Sb\(_{10}\)Sn\(_{x}\) (x = 2, 4, 6 and 10 at%) glassy systems have been estimated and are depicted in Table 3. It is observed that \(< E >\) increases as Sn-content increases. The increased values of \(< E >\) are due to the higher bond energies of Sn–Se bonds as compared to the weaker Se–Se bonds. The calculated values of \(< E >\) are utilized in assessing the glass transition temperature (\(T_g\)) using the empirical relation proposed by Tichy-Tichal[20, 21]:

\[
T_g = 311 (< E > - 0.9)
\]  

(12)

Table 3 lists the calculated values of \(T_g\) using Eq. 12. \(T_g\) values are observed to increase with the increasing Sn-concentration. The increased values of \(T_g\) owe to the increase in density of samples with Sn-content along with the cause for the increase in \(< E >\). The higher glass transition temperatures suggest that (Ge\(_{20}\)Se\(_{80}\))\(_{30}\), Sb\(_{10}\)Sn\(_x\) (x = 2, 4, 6 and 10 at%) are thermally stable glasses.

The electronegativity (\(\chi\)) has been calculated using Sanderson’s principle[23]. According to this principle, \(\chi\) of the glassy system is determined by taking geometric mean of the electronegativity of its constituent elements. For (Ge\(_{20}\)Se\(_{80}\))\(_{30}\), Sb\(_{10}\)Sn\(_x\) (x = 2, 4, 6 and 10 at%) glassy systems, \(\chi\) is given as:

\[
\chi = [(\chi_{Ge})^a (\chi_{Se})^b (\chi_{Sb})^c (\chi_{Sn})^d]^{1/a + b + c + d}
\]  

(13)

where \(a\), \(b\), \(c\) and \(d\) are the atomic fractions of Ge, Se, Sb and Sn respectively. \(\chi_{Ge} = 2.01\), \(\chi_{Se} = 2.55\), \(\chi_{Sb} = 2.05\) and \(\chi_{Sn} = 1.96\) are the electronegativity values of Ge, Se, Sb and Sn respectively. The evaluated values of \(\chi\) for Ge-Se-Sb-Sn glassy system is mentioned in Table 4. \(\chi\) of the glassy system decreases with the increase in Sn-content.

**3.2.4. Theoretical bandgap and average heat of atomization**

The heat of atomization is defined as the energy required to dissociate one mole of a substance into isolated atoms. The average heat of atomization characterizes cohesive energy and hence determines the relative bond strength between the isostructural materials. The values of average heat of atomization \(< H_s >\) for Ge-Se-Sb-Sn glasses are estimated by using the relation[24]:

\[
< H_s > = \frac{a H_{Ge}^s + b H_{Se}^s + c H_{Sb}^s + d H_{Sn}^s}{a + b + c + d}
\]  

(14)

where \(a\), \(b\), \(c\) and \(d\) are the atomic weight percentages of Ge, Se, Sb and Sn respectively; \(H_{Ge}^s = 377\) kJ/mol, \(H_{Se}^s = 227\) kJ/mol, \(H_{Sb}^s = 262\) kJ/mol and \(H_{Sn}^s = 302\) kJ/mol are the values of heat of atomization of Ge, Se, Sb and Sn respectively. The evaluated values of \(< H_s >\) are depicted in Table 3. It has been seen that the values of \(< H_s >\) increases with the incorporation of Sn which supports the enhanced rigidity of the present Ge-Se-Sb-Sn glassy systems.

The values of the theoretical bandgap (\(E^\text{th}_g\)) for the present glassy alloys are evaluated by using the empirical relation[25]:

\[
E^\text{th}_g = v_{Ge}E_g(Ge) + v_{Se}E_g(Se) + v_{Sb}E_g(Sb) + v_{Sn}E_g(Sn)
\]  

(15)
where \( v_{\text{Ge}}, v_{\text{Se}}, v_{\text{Sb}} \) and \( v_{\text{Sn}} \) are the volume fractions of Ge (13.63 \( \text{cm}^3/\text{mol} \)), Se (16.42 \( \text{cm}^3/\text{mol} \)), Sb (18.19 \( \text{cm}^3/\text{mol} \)) and Sn (16.24 \( \text{cm}^3/\text{mol} \)) respectively; \( E_g(\text{Ge}) = 0.67 \text{ eV} \), \( E_g(\text{Se}) = 1.95 \text{ eV} \), \( E_g(\text{Sb}) = 0.101 \text{ eV} \) and \( E_g(\text{Sn}) = 0.08 \text{ eV} \) are the energy gaps of Ge, Se, Sb and Sn respectively. The assessed values of \( E_{g}^{th} \) are depicted in Table 3 and are observed to decrease as Sn-content is increased. These results are supported by the decrease in average single bond energy defined by the ratio of \( < H_s > / < r > \)[26]. The values of average single bond energy are also found to decrease with the incorporation of Sn. The absorption edge \( (\lambda) \) is estimated from \( E_{g}^{th} \). The values of \( \lambda \) are recorded in Table 3, suggesting that the optical transmission will be above 900nm. This makes Ge-Se-Sb-Sn glassy systems optimal material for new generation infrared optical systems.

### Table 3. Values of mean bond energy \( (E) \), glass transition temperature \( (T_g) \), average heat of atomization \( (H_s) \), average single bond energy \( (H_s/r) \), theoretical band gap \( (E_{g}^{th}) \) and absorption edge \( (\lambda) \) for \((\text{Ge}_{20}\text{Se}_{80})_{10-}x\text{Sb}_{10}\text{Sn}_{x} \) \((x = 2, 4, 6 \text{ and } 10 \text{ at } \%) \) glassy systems.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(&lt; E &gt;/\text{per atom} ) (eV)</th>
<th>( T_g ) (K)</th>
<th>(&lt; H_s &gt; ) (kJ/mol)</th>
<th>(&lt; H_s&gt;/ &lt; r &gt; ) (kJ/mol)</th>
<th>( E_{g}^{th} ) (eV)</th>
<th>( \lambda ) (nm)</th>
</tr>
</thead>
</table>
| \((\text{Ge}_{20}\text{Se}_{80})_{10-}x\text{Sb}_{10}\text{Sn}_{2} \) | 2.50                           | 497.45        | 258.40               | 103.69                    | 1.51                  | 817
| \((\text{Ge}_{20}\text{Se}_{80})_{10-}x\text{Sb}_{10}\text{Sn}_{4} \) | 2.56                           | 516.35        | 259.30               | 102.73                    | 1.48                  | 835
| \((\text{Ge}_{20}\text{Se}_{80})_{10-}x\text{Sb}_{10}\text{Sn}_{6} \) | 2.62                           | 535.85        | 260.20               | 101.80                    | 1.45                  | 854
| \((\text{Ge}_{20}\text{Se}_{80})_{10-}x\text{Sb}_{10}\text{Sn}_{10} \) | 2.71                           | 561.66        | 262.00               | 100.00                    | 1.38                  | 895

#### 3.2.5. Density, molar volume, packing density and compactness

Density \( (\rho) \) measures the rigidity of the system. For Ge-Se-Sb-Sn glassy systems, \( \rho \) has been determined using the formula[27]:

\[
\rho = \left( \frac{\sum m_i}{d_i} \right)^{-1} = \left( \frac{m_{\text{Ge}}}{d_{\text{Ge}}} + \frac{m_{\text{Se}}}{d_{\text{Se}}} + \frac{m_{\text{Sb}}}{d_{\text{Sb}}} + \frac{m_{\text{Sn}}}{d_{\text{Sn}}} \right)^{-1}
\]  

where \( m_{\text{Ge}} \), \( m_{\text{Se}} \), \( m_{\text{Sb}} \) and \( m_{\text{Sn}} \) are the mass fractions of Ge (72.64 amu), Se (78.96 amu), Sb (121.76 amu) and Sn (118.71 amu) respectively. The values of \( d_{\text{Ge}} \), \( d_{\text{Se}} \), \( d_{\text{Sb}} \) and \( d_{\text{Sn}} \) are taken as 5.323 \( \text{g/cm}^3 \), 4.819 \( \text{g/cm}^3 \), 6.684 \( \text{g/cm}^3 \) and 7.31 \( \text{g/cm}^3 \) respectively.

Molar volume \( (V_m) \) has been evaluated using the value of density expression[27]:

\[
V_m = \frac{\Sigma x_i M_i}{p} = \frac{a M_{\text{Ge}} + b M_{\text{Se}} + c M_{\text{Sb}} + d M_{\text{Sn}}}{p}
\]
where a, b, c and d are the atomic fractions and $M_{\text{Ge}}$, $M_{\text{Se}}$, $M_{\text{Sb}}$ and $M_{\text{Sn}}$ are the molar masses of Ge, Se, Sb and Sn respectively.

Packing density is formulated as the ratio of used space to the allocated space and is calculated using the relation[27]:

$$\text{Packing density} = \frac{N \times \rho}{M}$$

(18)

where N is the Avogadro’s number, $\rho$ is the calculated density and M is the molecular weight of the respective composition of (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ ($x = 2, 4, 6$ and $10$ at %) glassy systems.

Compactness is formulated as the measure of normalized change of mean atomic volume caused by the chemical interaction of elements forming the glassy network. Compactness is more sensitive to changes in the structure of the glass network as compared to the mean atomic volume. The compactness of structure is determined according to formulae[27]:

$$\text{Compactness} = \frac{\sum x_i M_i \rho_i - \sum x_i M_i \rho}{\sum x_i M_i \rho}$$

(19)

where $x_i$, $M_i$, and $\rho_i$ are atomic fraction, atomic weight and atomic density of $i^{th}$ element of glassy system. The evaluated values of $\rho$, $V_m$, packing density and compactness from Eq. 16, 17, 18 and 19 respectively are depicted in Table 4. It is inferred that $\rho$ and $V_m$ values for Ge-Se-Sb-Sn glasses increases with the increase in Sn content. The density (5.323 g cm$^{-3}$) and mass (72.64 amu) of Ge as well as the density (4.819 g cm$^{-3}$) and mass (78.96 amu) of Se are very low as compared to heavier Sn with density (7.310 g cm$^{-3}$) and mass (118.71 amu). So as the concentration of Sn is increased, Ge and Se atoms are replaced by heavier and dense Sn, which leads to the increase in $\rho$ and $V_m$. Further, the addition of Sn content enhances the cross-linking in the glassy network and hence rigidity increases due to which $\rho$ increases also. Fig. 4 shows the variation of $\rho$ and $V_m$ with $<r>$. 

![Fig. 4. Variation of density ($\rho$) and molar volume ($V_m$) for (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ ($x = 2, 4, 6$ and $10$ at %) glasses as a function of average coordination number ($<r>$).](image)

The calculated values of compactness show a decrease in the value with the incorporation of Sn (up to $x = 4$ at%) and an increase with the further addition of Sn ($x = 6$ and $10$ at%). Thus one can expect that the micro-hardness of the samples increases with the increase in its compactness. The negative values of compactness correspond to higher free volumes and flexibilities. The average coordination number dependence of the compactness for Ge-Se-Sb-Sn glassy systems is shown in Fig. 5. From Fig. 5, it is evident that the minima of the compactness occur at $<r> = 2.524$ for the investigated glassy system. This shows that the system has the highest mean atomic volume of the network for $x = 4$ at % composition of (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ glassy system.
Fig. 5. Variation of compactness with the average coordination number (\( < r > \)) for (Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{90}\textsubscript{x}Sb\textsubscript{10}Sn\textsubscript{x} (x = 2, 4, 6 and 10 at \%) glassy systems showing that the minima of the compactness occur at x = 4 at \%.

Table 4. Values of electronegativity (\( \chi \)), density (\( \rho \)), molar volume (\( V_m \)), packing density and compactness for (Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{90}\textsubscript{x}Sb\textsubscript{10}Sn\textsubscript{x} (x = 2, 4, 6 and 10 at \%) glassy systems.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( \chi )</th>
<th>( \rho ) (g/cm(^3))</th>
<th>( V_m ) (cm(^3)/mol)</th>
<th>Packing density (10(^{22}) atoms/cm(^3))</th>
<th>Compactness</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{88}Sb\textsubscript{10}Sn\textsubscript{2}</td>
<td>2.38</td>
<td>5.16</td>
<td>16.08</td>
<td>3.744</td>
<td>0.00081</td>
</tr>
<tr>
<td>(Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{86}Sb\textsubscript{10}Sn\textsubscript{4}</td>
<td>2.37</td>
<td>5.20</td>
<td>16.09</td>
<td>3.742</td>
<td>-0.00080</td>
</tr>
<tr>
<td>(Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{84}Sb\textsubscript{10}Sn\textsubscript{6}</td>
<td>2.36</td>
<td>5.25</td>
<td>16.10</td>
<td>3.740</td>
<td>-0.00049</td>
</tr>
<tr>
<td>(Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{80}Sb\textsubscript{10}Sn\textsubscript{10}</td>
<td>2.34</td>
<td>5.35</td>
<td>16.11</td>
<td>3.737</td>
<td>0.00016</td>
</tr>
</tbody>
</table>

3.2.6. Distribution of bonds and cohesive energy

The cohesive energy (CE) of any system is specified as the energy required in the stabilization of an infinitely large collection of materials per atom which allows the determination of the number of probable bonds. It helps in determining the magnitude of the bond strength. In general, it is found that Zachariasen assumption [28] is obeyed in a glass structure which suggests that the probability of bond-formation among dissimilar atoms is higher than the probability of bond-formation among similar atoms. Therefore, according to this assumption, bonds between similar atoms will form only when there is an excess of similar atoms. Also the bond-formation occur in the decreasing order of bond energies until all the existing valences for the atoms are saturated. So according to this model, the bond energies are assumed to be additive. Thus CE is evaluated by adding the bond energies of the overall bond expected in the alloy[27]:

\[
CE = \sum C_iD_i
\]  

where C\(_i\) is the number of expected chemical bond and D\(_i\) is the corresponding bond energy of the occurring bond in the glassy system. The distribution of bonds and CE for (Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{90}\textsubscript{x}Sb\textsubscript{10}Sn\textsubscript{x} (x = 2, 4, 6 and 10 at \%) chalcogenides are depicted in Table 5. From the data, it is very evident that CE increases with the incorporation of Sn at the cost of Ge and Se up to x = 6 at\% due to the formation of stronger heteropolar Sb−Se bonds. With further incorporation of Sn (up to x = 10 at\%), the formation of Sb−Sb bonds lower down CE. The large and increased values of CE confirm that (Ge\textsubscript{20}Se\textsubscript{80})\textsubscript{90}\textsubscript{x}Sb\textsubscript{10}Sn\textsubscript{x} glasses are highly stable glasses making them optimal materials for utilization in optoelectronic devices.
Table 5. Distribution of chemical bonds and cohesive energy (CE) for \((\text{Ge}_{20} \text{Se}_{80})_{x0} \text{Sb}_{10} \text{Sn}_x\) \((x = 2, 4, 6 \text{ and } 10 \text{ at\%})\) glassy systems.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Distribution of chemical bonds</th>
<th>CE (kcal/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ge−Se</td>
<td>0.59935</td>
</tr>
<tr>
<td></td>
<td>Sn−Se</td>
<td>0.06813</td>
</tr>
<tr>
<td></td>
<td>Sb−Se</td>
<td>0.19333</td>
</tr>
<tr>
<td></td>
<td>Se−Se</td>
<td>0.13919</td>
</tr>
<tr>
<td></td>
<td>Sb−Sb</td>
<td>−</td>
</tr>
</tbody>
</table>

3.3. Electrical properties

Fig. 6 depicts the current-voltage plots on a log-log scale for \((\text{Ge}_{20} \text{Se}_{80})_{90} \text{Sb}_{10} \text{Sn}_x\) \((x = 2, 4, 6 \text{ and } 10 \text{ at\%})\) thin films. The plots are linear with slope almost unity up to the operating range of applied voltage \((0 \text{ V} \text{−} 100 \text{ V})\) and hence indicating the ohmic behaviour of the contacts.

\[
\sigma_d T^{1/2} = \sigma_0 \exp \left( -\frac{E_a}{k_B T} \right)
\]  

where \(\sigma_0\) is the pre-exponential factor, \(E_a\) is the conduction activation energy, \(T\) is the absolute temperature and \(k_B\) is the Boltzmann constant. The values of \(E_a\) and \(\sigma_0\) are calculated from the slope and intercept of \(\ln (\sigma_d T^{1/2})\) against \(T^{-1}\) plot. Fig. 7 depicts the variation of \(\ln (\sigma_d T^{1/2})\) against
T$^{-1}$ for Ge-Se-Sb-Sn thin films within the temperature range 293 K – 363K. The straight line behaviour of the plots depicted in Fig. 7 in the studied temperature range suggests that the conduction in the glassy systems occurs via an activated process exhibiting single activation energy in this particular temperature range.

As the activation energies and temperature range are too high to observe hopping so thermally assisted tunneling has been investigated, which is given as[29]:

$$\sigma_d = \sigma_o \left(1 + \frac{F^2}{6}T^2 \right)$$

(22)

where $\sigma_o$ and $F$ are constants. Fig. 8(a) represents the variation of $\sigma_d$ versus $T^2$ for Ge-Se-Sb-Sn thin films. The non-linear plots clearly indicate that the tunneling mechanism is not the dominant conduction mechanism in Ge-Se-Sb-Sn thin films in this temperature range. Therefore the conduction mechanism predominating the mentioned temperature range is thermionic emission only. It is factual that thermally activated phenomena like dc-conduction in chalcogenide glasses obeys Meyer-Neldel (MN) rule which is given as[30]:

$$\sigma_o = \sigma_{oo} \exp\left(\frac{E_a}{E_{MN}}\right)$$

(23)

where $\sigma_{oo}$ is MN pre-exponent factor and $E_{MN}$ is MN characteristic energy. Fig. 8(b) depicts the plot of $\ln (\sigma_o)$ against $E_a$ for Ge-Se-Sb-Sn thin films. The linear plot infers that $\sigma_o$ varies exponentially with $E_a$ according to Eq. 23 and hence MN rule is obeyed in all compositions of Ge-Se-Sb-Sn thin films. The slope and intercept of the straight-line plot in Fig. 8(b) yields MN characteristic energy ($E_{MN} = 33.64$ meV) and MN pre-exponential factor ($\sigma_{oo} = 1.779 \times 10^{-4} \Omega^{-1}cm^{-1}$). The values are close to the range suggested by Shimakawa and Abdel-Wahab for chalcogenide glasses[30].

Fig. 7. Plots of $\ln (\sigma_d T^{1/2})$ against $T^{-1}$ in the temperature range (293 K – 363 K) for (Ge$_{20}$Se$_{80}$)$_{100-x}$Sb$_x$Sn$_x$ ($x = 2, 4, 6$ and $10$ at %) thin films depicting the values of activation energies also.
Fig. 8. (a) Plots of $\sigma_d$ against $T^2$ for (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) thin films in the temperature range (293 K – 363 K). (b) Validation of Meyer-Neldel rule in (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) thin films.

Table 6. Values of conductivity ($\sigma_d$) at 298 K, activation energy ($E_a$) and pre-exponent factor ($\sigma_o$) for all compositions of (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) thin films.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\sigma_d$ ($\Omega^{-1}$cm$^{-1}$) at 298 K</th>
<th>$E_a$ (eV)</th>
<th>$\sigma_o$ ($\Omega^{-1}$cm$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Ge$<em>{20}$Se$</em>{80}$)$<em>{90}$Sb$</em>{10}$Sn$_2$</td>
<td>1.06 x 10$^{-8}$</td>
<td>0.77</td>
<td>1.87 x 10$^6$</td>
</tr>
<tr>
<td>(Ge$<em>{20}$Se$</em>{80}$)$<em>{90}$Sb$</em>{10}$Sn$_4$</td>
<td>1.59 x 10$^{-8}$</td>
<td>0.73</td>
<td>5.75 x 10$^5$</td>
</tr>
<tr>
<td>(Ge$<em>{20}$Se$</em>{80}$)$<em>{90}$Sb$</em>{10}$Sn$_6$</td>
<td>4.75 x 10$^{-9}$</td>
<td>0.87</td>
<td>3.46 x 10$^7$</td>
</tr>
<tr>
<td>(Ge$<em>{20}$Se$</em>{80}$)$<em>{90}$Sb$</em>{10}$Sn$_{10}$</td>
<td>5.84 x 10$^{-9}$</td>
<td>0.81</td>
<td>3.80 x 10$^6$</td>
</tr>
</tbody>
</table>

Table 6 represents the values of $\sigma_d$ at 293 K, $E_a$ and $\sigma_o$ for Ge-Se-Sb-Sn thin films. It is observed that $\sigma_o$ is not constant for the series under investigation. Further the values of $\sigma_o$ increases as the value of $E_a$ increases. Moreover, the variation in the values of $\sigma_d$ as well as $\sigma_o$ is very low with the incorporation of Sn. $\sigma_d$ values increase as Sn-concentration is increased to x = 4 at%. With the further addition of Sn (to x = 6 at% and 10 at%) $\sigma_d$ values decrease.

The results are interpreted with reference to the structural changes taking place after the addition of Sn. The incorporation of Sn leads to the substitution of Ge-atoms and the formation of Sn–Se bonds. The Sn–Se ionic-covalent bonds (as Sn is more electropositive than Ge), in contrast to Ge–Se bonds, offer more conducting path in the system and hence $\sigma_d$ increases as Sn concentration increases to x = 4 at%. This rise in $\sigma_d$ is responsible for the decrease in $E_a$. With the further increment in Sn content (to x = 6 at% and 10 at %) weaker Sb–Sb bonds formation occurs that leads to more disordering in the system and thereby decreasing $\sigma_d$ (hence $E_a$ also increases). From Table 6, it is very evident that (Ge$_{20}$Se$_{80}$)$_{90}$Sb$_{10}$Sn$_4$ is the most conducting as compared to others in the series.

4. Conclusions

In the present work, the effect of the addition of Sn on the structural, physical and electrical properties of vacuum evaporated (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) thin films has been studied. The samples have been found amorphous in nature by XRD technique with the possibility of short-range ordering in the glassy systems. The temperature-dependent dark conductivity measurements in the temperature range 293 K–363 K affirms that the conduction in
Ge-Se-Sb-Sn thin films is through an activated process with single activation energy and (Ge$_{20}$Se$_{80}$)$_x$Sb$_{10}$Sn$_4$ composition has the highest conductivity as compared to the other compositions in the series. The formation of weaker Sb−Sb bonds leads to a decrease in conductivity for the higher concentration of Sn element. Meyer-Neldel rule is obeyed by (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) glassy systems.

The number of constraints and average coordination number values confirm Ge-Se-Sb-Sn glassy systems to be rigid and over-coordinated. The prepared (Ge$_{20}$Se$_{80}$)$_{90-x}$Sb$_{10}$Sn$_x$ (x = 2, 4, 6 and 10 at %) chalcogenides are selenium-rich up to x = 6 at% and turns to selenium-poor for x = 10 at%. The high lone pair electrons suggest that Ge-Se-Sb-Sn system a good glass former. The high glass transition temperatures confirm Ge-Se-Sb-Sn glasses to be thermally stable. The deep analysis of the physical, electrical and structural properties and the variation of these properties as a function of Sn-content suggest the utilization of these glasses in optical devices (in the near and mid-IR region).

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References