Investigation of the effect of Au$_2$O$_3$ dopant on elastic properties of PbO-B$_2$O$_3$-SeO$_2$: Er$_2$O$_3$ glass ceramics by ultrasonic techniques

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**Article Info**

**Abstract**

Various elastic coefficients of Au$_2$O$_3$ doped PbO-B$_2$O$_3$-SeO$_2$:Er$_2$O$_3$ (PBSE) glass ceramics were evaluated as functions of Au$_2$O$_3$ content using ultrasonic velocity measurements. The elastic coefficients and micro-hardness showed a decreasing tendency with the concentration of Au$_2$O$_3$. Such decrease is attributed to the increasing concentration of gold metallic particles and [SeO$_3$]$^2-$ groups that acted as modifiers and induced imperfections in these samples. Obtained results were observed to be consistent with the conclusions drawn from spectroscopic studies that include X-ray photoelectron spectroscopy (XPS), infrared (IR), photoluminescence (PL) and positron annihilation (PAL) spectroscopy studies. Overall, these studies have revealed that even though, the presence of gold metallic particles is preferable for achieving superior luminescence and electrical properties, presence of such particles caused to decrease the elastic coefficients and micro-hardness of these glass ceramics. However, when the concentration of Au$_2$O$_3$ is increased beyond 0.075 mol%, we have observed a slight increase of elastic coefficients and micro-hardness.

**Keywords:**

Au$^0$ metallic particles; Elastic coefficients; PBSE glass ceramics; Ultrasonic velocity

1. Introduction

Various elastic coefficients like Young’s modulus (Y), shear modulus (G), Poisson’s ratio (ν), micro-hardness (H) and various other parameters like Debye temperature (θD) etc., can be evaluated by determining sound velocities in glasses and glass ceramics. Knowledge of such parameters not only helps in assessing their elastic behavior but also throw useful information on structural variations occurring in the glasses and glass ceramics. In the recent past, a large number of studies on elastic properties of different glasses and glass ceramics were investigated by measuring ultrasonic velocities in them [1-5].

Among various glass systems, selenium oxide (SeO$_2$) is a rare glass system. As such it is a feeble glass forming oxide and forms the glass when the modifying oxides like PbO and different traditional glass formers like borate, silicate, etc. are added. Interestingly, in the process of glass forming, the coordination number of Se ions with oxygen varies from 4 to 3 and the oxide of selenium transforms from [SeO$_4$]$^4-$ (selenate) structural units to isolated [SeO$_3$]$^3-$ (selenite) pyramidal units [6, 7]. Among these two groups, the [SeO$_3$]$^3-$ do participate in the glass formation, whereas [SeO$_4$]$^4-$ act as modifying ions and generate different imperfections and cause to decrease elastic coefficients of the samples. Hence, the elastic parameters of these samples depend on relative proportions of these two groups. Quite recently, we have reported the luminescence properties of Er$^{3+}$ doped lead borosilicate glass ceramics admixed with Au$_2$O$_3$ [8]. Au$_2$O$_3$ is added with a view to enhance PL efficiency of rare earth dopant ions since the gold particles emit PL in the visible region and also bring important changes in the elastic properties of the host glass ceramic. The studies further suggested that higher the fraction of [SeO$_3$]$^3-$ units (determined by XPS studies) larger is the PL efficiency of erbium ions. Such higher efficiency was attributed to the decreased phonon losses because of higher concentration of structural defects induced by [SeO$_3$]$^3-$ units. We have added about 40 mol% of PbO to the glass system to enhance the density, refractive index (which cause to increase PL efficiency) and also to minimize the sublimity and hygroscopic nature of SeO$_2$. We have also added a minimal concentration of B$_2$O$_3$ (10 mol%) to this glass system in order to ensure good glass formation. Further, it may be noted that the physical characteristics of amorphous systems containing SeO$_2$ are very

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limitedly investigated and in most of such systems small concentration of SeO₂ was added as a dopant. Such studies as on the glasses or glass ceramics mixed with relatively large concentration of SeO₂ are very rare in spite of their high suitability for the several applications for example, in electrical and optical devices as sensors and various non-linear optical (NLO) devices etc. [9-13].

The current investigation is aimed to investigate the influence of Au₂O₃ on elastic characteristics of PbO-B₂O₃-SeO₂:Er₂O₃ glass ceramics. As mentioned earlier, addition of traces of Au₂O₃ to these makes them useful for the designing of miniature (nanosized) optoelectronic circuits and memory devices with large data storage capacity [14]. Owing these reasons the knowledge of elastic properties of SeO₂ based glass system doped with Au₂O₃ is highly helpful. In general, Au₂O₃ is a volatile oxide and breaks into AuO, Au₃S and Au₃ metallic particles (MPS) during the synthesis of the glasses. The presence of larger content of Au³⁺ MPS will have a strong bearing on elastic properties as well as other physical characteristics of the glasses. For obtaining the glass samples with larger fraction of Au³⁺ MPS (possible by reducing trivalent gold ions), in this study the synthesized glasses were heated for a time interval of 24 h at the crystallization temperature. Such treatment reduces Au³⁺ ions into Au⁰ particles; in addition, it is quite likely for the formation of anisotropic crystalline phases e.g., Au₂(Se⁴⁺O₂)₃ and Au₂(Se⁴⁺O₂)Au₂(Se⁺ IVO₄) of selenium ions [15,16] in the samples. Glasses with such crystallities are useful for NLO devices [16]. Further, the development of such crystalline phases along with Au³⁺ MPS in the glasses causes to entrench several nanosized free volume defects in the material which alter the elastic properties. We propose to determine the concentration of such defects by means of positron annihilation lifetime (PAL) spectroscopy in this study [17,18].

Thus, in continuation to our earlier investigations on the impact of Au₂O₃ on photoluminescence features of PbO-B₂O₃-SeO₂:Er₂O₃ glass ceramics, the current investigation is devoted to study the elastic properties (in combination with PAL characteristics in detail) as a function of Au₂O₃ content and to correlate these results with those of luminescence characteristics.

2. Experimental

The chemical composition chosen for the present is: 40PbO-10B₂O₃-50-x)SeO₂-0.5Er₂O₃-(Au₂O₃, where x = 0, 0.025, 0.050, 0.075 and 0.1, all in mol%) and the samples are labeled as EA0, EA25, EA50, EA75 and EA100. PbO, B₂O₃, SeO₂, Au₂O₃ and Er₂O₃ compounds were mixed thoroughly and melted in Pt crucibles in the range of temperature 1000–1050 °C (for about 40 min) in a thermally controlled furnace. The resultant molten liquid was casted in to slabs of rectangular shape and were annealed at 350 °C. For crystallization, the samples were held at 630 °C (crystallization temperature-onset, identified from DSC studies) for 24 h and then chilled to room temperature. The densities of the samples were measured by Archimedes’ principle (using o-xylene as the buoyant liquid) to an accuracy of ±0.001. The other details of the preparation of the samples and characterization techniques e.g., X-ray diffraction (XRD), IR and XPS spectra etc. were reported in our earlier communication [8]. Transmission electron microscopy (TEM) images of the prepared samples were recorded on a JEOL 2200FS instrument with FE gun operated at 200 kV. XPS measurements were performed on PHI 5000 Versa Probe ULVAC instrument with a monochromatic Al Kα (hν = 1486.6 eV) as the source. Spectra were recorded with respect to C 1s peak. Positron annihilation spectra were recorded on “Ortec” positron lifetime system with a ²²Na isotope (100 kBq) source. Lifetime τ of the positroniums (from which the size of the free volume space defects is determined) is evaluated to a precision of 230 ps at 18 °C in the atmosphere of RH = 35%.

For measuring the longitudinal velocity in the samples, the OPBOX 2.1 measuring device with software was used. This device controls the operation of ultrasonic heads (transmitting and receiving). The frequency of the transmitted signal was 20 MHZ. The speed of the sound is determined by measuring the passage time of the ultrasonic impulse and its echo through the glass ceramic. Shear velocities were measured with a piezoelectric composite oscillator technique [19,20] using an X-cut (0.13 MHz) cylindrical quartz transducer (2.0 cm long and 0.25 cm diameter). The dimensions of the glass ceramics used for these measurements are approximately the same as those of quartz transducer.

3. Results and discussion

X-ray diffractogram of the glass ceramic EA25 is presented in Fig. 1. The diffractogram exhibited two diffraction peaks at 2θ = 29.9° and 44.0° due to the reflections from Au₂(SeO₂)₃ crystal phase and Au³⁺ MPs, respectively [14,16,21]. The diffractograms of all the other samples have exhibited similar peaks. Out of these two peaks, the peak due to Au³⁺ MPs exhibited a gradual growth up to 0.075 mol% of Au₂O₃ (inset of Fig. 1); such growth of the peak suggests an increase in the concentration of Au³⁺ metallic particles. It may be noted here that in the diffractogram of the as-prepared sample no peaks were noticed and indicated the amorphous character of such samples.

In order to prove further, the crystallinity in the samples, we have recorded TEM images and presented in Fig. 2, for the samples EA0, and EA25 with different magnifications. The pictures indicated a large number segregated crystal grains of nanosize embedded in the glass samples.

Further, we have recorded positron annihilation decay profiles in these glass ceramics in order to estimate the free volume defects/free volume space entrenched in the glass ceramics. Fig. 3(a) represents the positron decay profiles of one of the glass ceramic samples viz., EA100. The decay curve is deconvoluted in to I₁, I₂ and I₃ components. By using the lifetime (τ₃) of the third component (long lived component), we have estimated radius R of free cavities in the glass ceramics with the standard expression [22,23]:

\[
\tau_3 = \frac{1}{2} \left[ 1 - \frac{R}{R_0} \right] \frac{\sinh \left( \frac{2\pi R}{R_0} \right)}{\sinh \left( \frac{\pi R}{R_0} \right)}
\]

Using the value of I₃ (the intensity of the 3rd component of the decay profiles) and the radius R of free cavities (found to be in the range of 2.6 to 2.8 Å), the free volume space fraction fᵥ trapped in the sample is determined with the equation.

![Fig. 1. X-ray diffractogram of EA20 sample [Ref. 8]. Inset represents the variation of the intensity of the diffraction peak related to Au³⁺ metallic particles and Au₂(SeO₂)₃ crystalline phase with the concentration of Au₂O₃. The patterns were recorded in continuous scan mode with a scan speed of 2 deg/min.](image-url)
glass ceramics leading to decrease the values of elastic parameters.

In order to have additional confirmation of the presence of Au⁴ MPs (which act as modifiers) and to have an idea over their variation with the concentration of Au₂O₃ in these samples we have recorded XPS spectra. Fig. 4 represents such spectrum for the sample EA₁₀₀. The spectrum exhibited the peaks corresponding to the binding energy (BE) of 4f Au⁴⁺ ions at 85.8 eV and 89.6 eV and two more peaks at 84.0 eV and 87.6 eV connected with the BE of Au⁵⁺ MPs [24,25]. Fig. 4 (inset) represents variation of concentration of Au⁴⁺ ions and Au⁵⁺ MPs with the content of Au₂O₃. The figure showed an increase of Au⁴⁺ MPs (at the expense of Au⁵⁺ ions) up to 0.075 mol% of Au₂O₃. These gold MPs behave as modifiers and produce structural imperfections as mentioned earlier [8,17].

Fig. 5 represents the infrared (IR) spectrum of PbO–B₂O₃–SeO₂–Er₂O₃ glass ceramic containing 0.05 mol% of Au₂O₃. Spectrum exhibited the vibrational bands due to [SeO₄]²⁻ units (at nearly 950 cm⁻¹) and stretching (symmetrical) vibrational band of quartzine selenite [SeO₃]²⁻ pyramidal groups in the wavenumber range 850-900 cm⁻¹ [11]. In addition, the spectrum also exhibited the conventional vibrational bands of BO₃ and BO₄ units as shown in the figure. With increase of Au₂O₃ content up to 0.075 mol%, the vibrational band of [SeO₃]²⁻ groups exhibited a gradual growth, whereas that of [SeO₄]²⁻ groups showed a decaying trend (inset of Fig. 5). The selenite [SeO₃]²⁻ groups mainly act as modifiers [12] and induce structural defects in the glass ceramic. In support of this argument, we have observed the growth of BO₃ vibrational band, whereas that of BO₄ units exhibited a decreasing trend.

Fig. 6(a) and (b) represent propagation of longitudinal waves (by which the velocity ν₁ is determined) in the samples EA₀ and EA₁₀₀. In these figures, measuring gates are represented by the two arrows. Such arrows in fact, indicate the starting and end point of the measurements between two consecutive reflected pulse.

Using the values of ν₁ and ν₅, various elastic coefficients of PBSE glass ceramics doped with different concentrations of Au₂O₃ are estimated with the standard expressions and tabulated in Table 1. Their variations with the concentration of Au₂O₃ are presented in Fig. 7.

All these parameters showed a decreasing trend with the content of Au₂O₃ up to 0.075 mol%. In Fig. 8(a) and (b), Young’s and shear moduli variations with the content of Au₂O₃ are presented. The variations showed the gradual decrement of these parameters (and also the micro-hardness H of the samples (Table 1)) with the content of Au₂O₃.

Zhang et al. [26] have established a general empirical relation between Vickers hardness Hv and yield strength σy as

\[ H_v = 3\sigma_y \]  

The obtained values of σy evaluated based on this relation are presented.

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**Fig. 2.** TEM images of EA₅₀ and EA₇₅ glass ceramic samples recorded with different magnifications.

\[ f_r = \frac{A_1(\%)}{3.14R^2} \]  

In Eq. (2), relative fraction of the \( f_r \) in the titled samples (without using the value of A whose value is predicted for polymers as 0.0018/Å²) is estimated with respect to that of the pure glass, using following equation

\[ \frac{\Delta f}{f} = \left( \frac{f_v - f_0}{f_0} \right) \times 100\% \]  

In Eq. (3), \( f_0 \) represents free volume of the titled glass ceramics mixed with different contents of gold oxide and \( f_v \) denotes free volume in the pure glass ceramic.

The free volume fraction of the free space \( (\Delta f/f_0) \) obtained relative to that of the undoped samples is plotted against Au₂O₃ concentration in Fig. 3(b). The plot showed a gradual increase of the free space fraction in the samples up to 0.075 mol% of Au₂O₃. Such increase in magnitude of free space obviously causes to enhance the acoustic impedance of the material.

**Fig. 3.** (a) Positron annihilation lifetime decay profiles of the glass ceramics EA₁₀₀. Fig. 3(b) Variations of free volume fraction \( \Delta f/f_0 \) with concentrations of Au₂O₃ doped PBSE glass ceramics.
in Table 1. The value of $\sigma_y$ exhibited a decreasing trend with the concentration of $\text{Au}_2\text{O}_3$ up to 0.075 mol% similar to Vickers hardness $H_v$ and subsequently, Aila and Yin [27] have applied this relation to evaluate the dependence of contact hardness, $H_c$, on the yield strength, $\sigma_y$, for lithium metasilicate glass-ceramics. According to these people the value of $H_v$ is given by

$$H_v = C\sigma_y$$

In Eq. (5), C is a constraint factor which is found to be 3 for materials that exhibit a combination of piling-up and shear-banding phenomenon such as lithium metasilicate glass-ceramics [26]. In the present case for PBSE glass ceramics the evaluated value of $H_v$ gives an approximate idea over the dependence of yield strength $\sigma_y$ on the concentration of $\text{Au}_2\text{O}_3$. In other words it can be said that the yield strength is minimal for the glass ceramic $\text{EA}_{75}$.

The IR spectra have clearly suggested that an increasing concentration of $[\text{SeO}_3]^2-$ structural groups with increase of $\text{Au}_2\text{O}_3$. XPS spectra have demonstrated a steady growth in the concentration of Au$^{3+}$ metallic particles.

Both of these acted as modifiers and de-augment Se-O-Se linkages in the glass network and also introduce free volume defects in the glass ceramics. Such enhanced degree of de-fracturation in the glass network facilitated a decrease of ultrasonic speed and caused to decrease elastic parameters and micro-hardness.

Our earlier results on photoluminescence studies of these glass ceramics have also indicated an increase in the luminescence efficiency of green emission ($^{4}S_{3/2} \rightarrow ^{4}I_{15/2}$) and orange emission ($^{4}F_{9/2} \rightarrow ^{4}I_{15/2}$) of erbium ions, as mentioned earlier, with increase of $\text{Au}_2\text{O}_3$ content [8] and patterns of PL emission and band positions obtained were found to be in accordance with the reported data in the literature [28–30]. Such enhancement was attributed to the decrease of phonon losses or due to the rise in the degree of internal structural chaos in the glass ceramic network. In conclusion even though, the introduction of $\text{Au}_2\text{O}_3$ is proved to improve the optical properties and also the electrical characteristics in various glasses and glass ceramics, it caused to decrease the elastic coefficients and micro-hardness of PbO-B$_2$O$_3$-SeO$_2$-Er$_2$O$_3$ glass ceramics.

4. Conclusions

Different elastic coefficients and micro-hardness H/yield strength $\sigma_y$ of $\text{Au}_2\text{O}_3$ mixed PbO-B$_2$O$_3$-SeO$_2$-Er$_2$O$_3$ glass ceramics were estimated using ultrasonic velocities. The coefficients were found to decrease with $\text{Au}_2\text{O}_3$ concentration up to 0.075 mol%. Such decrease was ascribed to growing presence of $[\text{SeO}_3]^2-$ structural groups and also Au$^{3+}$ metallic particles that produce structural imperfections like free volume defects by acting as modifiers. This argument was further supported by XPS, IR and positron annihilation spectroscopy studies. Such increased degree of disorder or increased magnitude of free volume space with increase of $\text{Au}_2\text{O}_3$ content was projected to be an obstruction for the passage of ultrasonic waves in the samples and hence responsible for the decrement in the elastic coefficients and micro-hardness of the studied glass ceramics. Finally, it is concluded that even though, the $\text{Au}_2\text{O}_3$ is a favorable dopant for superior optical and electrical properties of the studied glass ceramics it caused to have an adverse effect on the elastic coefficients. However, when the concentration of $\text{Au}_2\text{O}_3$ is increased beyond 0.075 mol%, we have observed a slight increase of elastic coefficients and micro-hardness.

Fig. 4. XPS spectrum of $\text{EA}_{100}$ sample. Inset shows the relative variations of intensities of the peaks of Au$^{2+}$ and Au$^{3+}$ ions with content of $\text{Au}_2\text{O}_3$.

Fig. 5. IR spectrum $\text{EA}_{50}$ glass ceramic. In the inset relative variations of intensities of the various vibrational groups are presented with the content of $\text{Au}_2\text{O}_3$. 

\[ \text{ EA}_{75} \]
Fig. 6. Longitudinal wave propagation in the samples (a) EA₀ and (b) EA₅₀.

### Table 1

| Sample  | Long. Vol. V₁ (m/s) | Density ρ (g/cm³) | Long. coeff. L = ρν₁² | Shear Vol. V₅ (m/s) | Shear Modulus, G (x10¹⁰ N/m²) | Young’s Module, Y (x10¹⁰ N/m²) | Avg. Vol. V₆ (m/s) | Debye Temp. θ₀ (K) | Micro hardness H (GPa) | Yield strength σₚ (GPa) |
|---------|-----------------------|------------------|----------------------|-------------------|-----------------------------|-----------------------------|------------------|------------------|---------------------|-------------------|
| EA₀     | 3478                  | 5.87             | 7.10                 | 2333              | 3.19                        | 7.00                        | 2200             | 99.6             | 8.71                | 2.90              |
| EA₂₀    | 3404                  | 5.92             | 6.66                 | 2203              | 3.09                        | 6.73                        | 2153             | 97.9             | 8.42                | 2.81              |
| EA₅₀    | 3377                  | 5.94             | 6.77                 | 2265              | 3.05                        | 6.65                        | 2136             | 97.3             | 8.31                | 2.77              |
| EA₁₀₀   | 3375                  | 6.10             | 6.95                 | 2264              | 3.13                        | 6.72                        | 2134             | 98.1             | 8.53                | 2.76              |

"σ is the Poisson’s ratio, V₅₆ = ν₅₆ = [ν₁² - (4/3)ν₁²]¹/₂ and θ₀ = θ₀ = h/[(3Mₑ/4πkₐ)¹/²]ν₆."
CRediT authorship contribution statement

A. Siva Sesa Reddy: Conceptualization, Methodology, Investigation.
A.V. Kityk: Methodology, Data curation, Formal analysis, Software, Writing – original draft. J. Jedryka: Methodology, Data curation, Formal analysis, Software, Writing – original draft. P. Rakus: Methodology, Data curation, Formal analysis, Software, Writing – original draft. A. Wojciechowski: Methodology, Data curation, Formal analysis, Software, Writing – original draft. N. Venkatramalai: Methodology, Data curation, Formal analysis. V. Ravi Kumar: Conceptualization, Methodology, Investigation, Supervision, Writing – original draft, Writing – review & editing. N. Veeralah: Supervision, Writing – original draft, Writing – review & editing.

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