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# Microstructure, thermal, optical and dielectric properties of new glass nanocomposites of SrTiO<sub>3</sub> nanoparticles/clusters in tellurite glass matrix

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## ABSTRACT

New glass nanocomposites (GNCs) of the nominal compositions [(100-x) TeO<sub>2</sub>-xSrTiO<sub>3</sub>] with x = 5, 10 and 15 (in mol%) were prepared. The glassy phase in the GNCs samples was established by X-ray diffraction (XRD) and Differential thermal analysis (DTA) studies. The SrTiO<sub>3</sub> nanoparticles/clusters embedded in tellurite glass matrix for GNCs were identified by transmission electron microscopic (TEM) and confirmed by Fourier transform infrared (FTIR) study. The optical transmission (at x = 5) and the electron spin resonance (ESR) studies for the GNCs samples indicated that the presence of Ti<sup>3+</sup> ions in tetragonal distorted octahedral sites. The variation of the thermal, density (ρ) and the molar volume (V<sub>m</sub>) of the GNCs samples with increasing SrTiO<sub>3</sub> were discussed in terms of the structural modifications that take place in the glass matrix. The dielectric properties and ac conductivity of the GNCs samples cannot be explained on the basis of the proportion of Ti<sup>3+</sup> ions only.

## 1. Introduction

Recently, glass nanocomposites (GNCs) have attracted much attention due to their design flexibility and technological applications [1–3]. These applications of GNCs are due to the host glass matrix containing at least one of the phases in the nanometer size [2,3]. Among the types of GNCs, the glass matrix containing ferroelectric nanoparticle phases such as Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>-BaTiO<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>-TeO<sub>2</sub>-BaTiO<sub>3</sub> and LiBO<sub>2</sub>-LiNbO<sub>3</sub> plays an important role in the development of modern electronics [4–6]. The properties of the GNCs are dependent on the characters of the host glass matrix and the ferroelectric phase [7]. Among the host glass matrix, TeO<sub>2</sub> is one of the best host glass matrixes where TeO<sub>2</sub> has low melting point, high density and its ability to form a glass with a modifier or other glass former by conventional melt quenching techniques [5,8]. In addition to, the tellurite based glasses have good infrared transmission, high dielectric constant and electrical conductivity which due to the unshared pair of electrons of the TeO<sub>4</sub> group [5,9]. Among the different types of ferroelectric phases, strontium titanate (SrTiO<sub>3</sub>) has cubic perovskite structure at room temperature, high melting point, relatively low electronic and ionic conductivity [10,11].

Recently, Haenil et al. [12] have found for the first time the ferroelectricity in strained SrTiO<sub>3</sub> grown on DyScO<sub>3</sub> substrates at room temperature. This strain induced during the growth process of nanoparticles in a host matrix used to increase T<sub>c</sub> by hundreds of degrees. Yuan et al. [13] studied the SrTiO<sub>3</sub> nanoparticles embedded in

amorphous Lu<sub>2</sub>O<sub>3</sub> matrix and they found that the structural phase transition from cubic to tetragonal symmetry occurs at room temperature. In addition to the strain from amorphous matrix can cause the rotation of the TiO<sub>6</sub> octahedra. The crystallization process and dielectric properties of 2SrTiO<sub>3</sub>-SiO<sub>2</sub> glass were investigated by Kim et al. [14]. The glass exhibits two exothermic peaks which represent the crystallization of SrTiO<sub>3</sub> and Sr<sub>2</sub>TiSi<sub>2</sub>O<sub>8</sub> respectively. Swartz et al. [15] studied the crystallization, microstructure and dielectric properties of [68 wt% SrTiO<sub>3</sub>-23 wt% SiO<sub>2</sub>-12 wt% Al<sub>2</sub>O<sub>3</sub>] glass-ceramics. These glass-ceramics exhibit dielectric constant and loss peaks at low temperature, which represent the ferroelectric phenomena of SrTiO<sub>3</sub> phase.

To the best of our knowledge there is no report describing the incorporation of SrTiO<sub>3</sub> nanoparticles in the tellurite glasses (new glass nanocomposites). In view of the aforementioned aspects, we fabricate new glass nanocomposites of the nominal compositions [(100-x) TeO<sub>2</sub>-xSrTiO<sub>3</sub>] with x = 5, 10 and 15 (in mol%). In this study, the microstructure, structural, thermal, optical, electron spin resonance (ESR), Ac electrical conductivity and dielectric properties of glass-nanocomposites (GNCs) have been investigated.

## 2. Experimental procedures

## 2.1. Samples preparation

New glass nanocomposites (GNCs) samples with the molar

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composition [(100-x) TeO<sub>2</sub>-xSrTiO<sub>3</sub>] (where x = 5, 10 and 15 in mol %) were prepared by melt quenching method. The GNCs samples under investigation were prepared from reagent grade TeO<sub>2</sub> (99.99%) and SrTiO<sub>3</sub> (99.95%). The stoichiometric amounts of these chemicals were milled in an agate mortar. These batches were melted in porcelain crucible at 900 °C for 1 h in an electric furnace in air. The melt was quickly poured on a copper plate and then pressed by another one. GNCs plates were obtained from the above process. Finally, the GNCs samples were kept in a desiccator to prevent moisture absorption from air.

## 2.2. Samples characterization

X-ray diffraction (XRD) patterns of the GNCs samples were carried out by Philips type PW3710 diffractometer using CuK $\alpha$  radiation at room temperature. The microstructure of the GNCs samples were carried out by a JEOL JEM-1010 transmission electron microscopic (TEM). The differential thermal analysis (DTA) of the GNCs samples were carried out on a Shimadzu DTA-50 analyzer with heating rate 5 K/min and an accuracy of  $\pm 1$  K. The density ( $\rho$ ) of the GNCs samples were determined at room temperature using the Archimedes principle with a toluene as an immersion liquid ( $\rho_{\text{toluene}} = 0.866 \text{ g/cm}^3$ ). The molar volume ( $V_m$ ) of the GNCs samples were calculated from the relation  $V_m = (x_i M_T) / \rho$ , where  $x_i$  is the molar concentration and  $M_T$  is the total molecular weight of the  $i$ th component. The random errors in the density measurements are evaluated by taking the standard deviation of the mean of the density values for three specimens of each concentration. The FTIR absorption spectra of the GNCs samples were recorded in the range of 400–860  $\text{cm}^{-1}$  by a JASCO FTIR-430 spectrophotometer using KBr pellets with a resolution of 2  $\text{cm}^{-1}$ . The optical transmission spectrum of the GNCs sample at x = 5 was recorded in the 200–800 nm wavelength range using a Jasco 570 spectrophotometer with a resolution of  $\pm 1$  nm. The electron spin resonance (ESR) measurements of the GNCs samples were recorded at room temperature by X-band EMX spectrometer (Bruker Germany) using a standard rectangular cavity of ER 4102. The magnetic field was scanned in the range 2550–4000 G. Ac electrical conductivity and dielectric properties of the GNCs samples were carried out using a Stanford Research RCL Bridge (Model: SR-720) in the temperature range of 300–673 K and in the frequency range of 1–100 kHz.

## 3. Results and discussion

### 3.1. XRD and TEM studies

Fig. 1 shows the XRD patterns of the synthesized [(100-x) TeO<sub>2</sub>-xSrTiO<sub>3</sub>] with x = 5, 10 and 15 (in mol%) samples at room temperature. From this figure, it is noticed that the XRD patterns for all samples exhibit broad humps without any sharp peaks which indicating the amorphous nature for these samples [16].

Fig. 2 shows the transmission electron microscope (TEM) micrographs for the [(100-x) TeO<sub>2</sub>-xSrTiO<sub>3</sub>] with x = 5, 10 and 15 (in mol %) samples. From this figure, it is noticed that the micrographs show the presence of SrTiO<sub>3</sub> nanoparticles/clusters embedded in the glass matrix which precipitated during glass formation [17,18]. These nanoparticles are nearly spherical in shape. Hence, these observations indicate that the present samples are glass-nanocomposite (GNCs) [18]. These nanoparticles/clusters embedded in the glass matrix are not detected in XRD patterns (Fig. 1) because of the low concentration and small size of SrTiO<sub>3</sub> in the glass matrix [17–19]. Fig. 3 shows the enlarged cluster of a selected region and the distribution of particle size histograms of GNCs samples were displayed in the insets. The average particle sizes were calculated from the histograms of GNCs samples are 19.7, 27.5 and 26.4 nm for the compositions x = 5, 10 and 15 mol% respectively. The values of average particle sizes increase with the increase of SrTiO<sub>3</sub> content up to x = 10, then decrease with further increase of SrTiO<sub>3</sub> content. The increase in the average particle sizes can be attributed to the increase of the amount of SrTiO<sub>3</sub> where the particles at x = 5 have not enough thermal energy and time to grow [20,21]. On the other hand, the decrease in the average particle sizes can be attributed to the increase of agglomeration of clustering of SrTiO<sub>3</sub> nanoparticles where the presence of agglomerated particles may be attributed to the high surface energy of small-sized particles [22,23]. In addition to, the decrease in the average particle sizes can be explained by the inhibiting of grain growth at the grain boundaries [24].

### 3.2. DTA, density and molar volume studies

Fig. 4 shows the DTA curves for the obtained GNCs samples. From this figure, it is noticed that the DTA curves exhibit one endothermic dip which represents the glass transition temperature ( $T_g$ ). These curves exhibit also two separated obviously exothermic peaks ( $T_{p1}$  and  $T_{p2}$ ) due to the crystal growth followed by two endothermic peaks ( $T_{m1}$  and

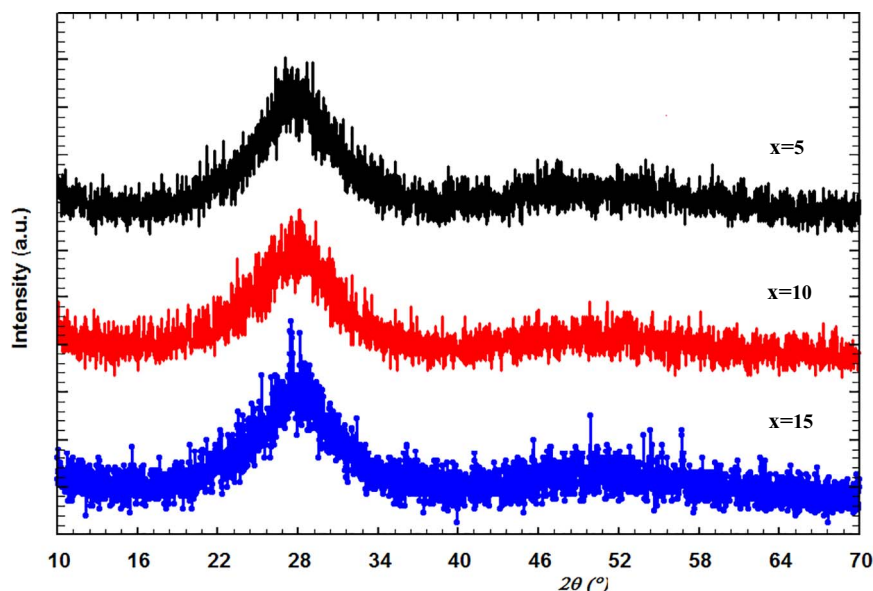


Fig. 1. XRD pattern of the GNCs samples as a function of the SrTiO<sub>3</sub> content.

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