

Electrical and dielectric properties of carbon nanotube containing $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites

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ABSTRACT

The electrical and dielectric properties of 3 and 5 wt. % of multi-walled carbon nanotube (MWCNT) containing $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites have been investigated in the frequency range 20 Hz to 2 MHz from room temperature to 387 K. It has been found that the electrical conductivity is enhanced by 6 to 9 orders of magnitude up to 5 wt. % of MWCNT content resulting in transition for insulating to conducting behaviour. The activation energies for all samples have also been evaluated from the Arrhenius plot of the DC conductivity which shows a decreasing trend up to 5 wt. % MWCNT content. The largest dielectric constant of 142 (almost 10 times greater than pristine $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy alloy) has been observed for 5 wt. % of MWCNT content at room temperature in low frequency range. Therefore it can be inferred that the electrical and dielectric properties of the chalcogenide glasses can be altered by the admixing or doping of carbon nanotubes (CNTs). The enhanced dielectric constant and electrical conductivity can be attributed to interface effect between MWCNT and the glassy matrix. Copyright © 2015 VBRI Press.

Keywords: Multi-walled carbon nanotube; glassy composite; dielectric constant; electrical conductivity.

Introduction

Recently, carbon nanotubes (CNTs) have attracted meticulous interest for high performance chalcogenide glassy composites [1, 2] because of their remarkable mechanical and electrical properties, very low density and high aspect ratio [3]. Now days, incorporation of carbon nanotubes in glasses, polymers and ceramic materials [2, 4, 5] to alter their properties and to enlarge advanced engineering composites have become an interesting area of research. As known, chalcogenide glass is a widely studied material having unique mechanical, electrical, thermal, and outstanding optical properties [6, 7].

However, in order to use chalcogenide amorphous semiconductor as a functional material in some applications such as batteries, sensors and display [1, 3, 8] a higher electrical conductivity is required. In addition, a high dielectric constant is also needed for use in energy storage devices [9, 10]. The usual method to improve the electrical properties of chalcogenide glasses is to compose them with metal (such as Ag, Cu and Li) [11-13].

Nevertheless it might be interesting to incorporate multi-walled carbon nanotube inside the host matrix of glassy materials by melt-quench technique. This being a novel fabrication, our primary interest is to translate a hardcore insulator into a conducting composite by introduction of multi-walled carbon nanotube inside the

host glassy matrix in a well dispersed manner in order to examine its applicability for a conductor-insulator interface device [14, 15]. Although agglomeration of CNTs is a big problem during synthesis of chalcogenide glassy composites, still researchers are trying to synthesize glassy composites with different techniques [14, 15]. However, many researchers have shown that even at low level of CNT doping or admixing the electrical, mechanical, thermal and optical properties [1, 2] have been altered. This is because CNTs provide an alternative way due to its small size and particular physical properties [3]. In recent years, most of the studies have also been focused on single walled carbon nanotube (SWCNT)/ceramic composites, SWCNT/polymer composites [16, 17]. The conductivity of SWCNT depends on chirality of the graphene sheet [18] whereas MWCNTs are always electrically conductive, even at low temperatures [19]. Furthermore, MWCNTs have been found to improve the electrical conductivity and dielectric constant in glasses, polymers and ceramics. This enhancement in dielectric constant value becomes fruitful in improving electromagnetic interference (EMI) shielding and decoupling capacitors in composite materials and polymers [20, 21]. In the present study, we have investigated electrical conductivity and dielectric properties of MWCNT containing $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ chalcogenide glassy composite in range of frequency 20 Hz to 2 MHz at different temperature. It has been observed that the

electrical conductivity (AC/DC) and dielectric constants are enhanced significantly by several orders of magnitude up to 5 wt. % of MWCNT incorporation. Such huge enhancement in electrical conductivity and dielectric constants accounts for electrical property transition from insulator to conductor.

Experimental

Material synthesis

High purity (99.999 %) Selenium (Se), Tellurium (Te), Copper (Cu as flakes) were purchased from Alfa-aesar group. Fullerene carbon nanotube, multi-walled, 3-20 nm outer-diameter (O.D), 1-3 nm inner-diameter (I.D), 0.1-10 micron long having 95 % purity were also purchased from Alfa-aesar group. Synthesis of CNT containing $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites were carried out by melt-quenched technique which has already been described in our published article [1, 2].

Characterizations

Scanning electron microscopy (SEM) characterization was carried out using a QUANTA-200 (SEM) model employing an e-beam voltage of 25KV at 40000 magnifications. Electrical measurements were performed at temperatures between 297 K to 387 K. The Electrical and dielectric studies were experimentally performed by using an Agilent E-4980A LCR meter. LCR meter is a microprocessor based fully automatic inductance (L), series and parallel capacitance (C) and resistance (R) measurement device. For electrical conductivity and dielectric measurement first of all the electrical contacts were made by painting glassy pellets on both sides with silver paste, simulating a parallel plate capacitor. The surface area of glassy pellets were about 0.80 cm^2 and distance between the electrodes were 2 to 3 mm. Dielectric measurements were performed in an alternative current mode (AC) in range of frequencies between 20 Hz and 2 MHz, at the temperatures between 297 K and 387 K, in a neutral atmosphere.

Results and discussion

Microstructural analysis

Fig. 1 (a-c) shows SEM images of pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ sample, 3 % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ and 5 % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites. The presence of glassy microstructure can be observed from the SEM images, shown in **Fig. 1 (a-c)**. The featureless microstructure does not show evidence of any contrast change which is an indicative of absence of grain boundary. In addition, **Fig. 1(b, c)** show the presence of CNTs in glassy microstructure which are indicated within the red circles. The incorporated CNTs in glassy matrix appear to be somewhere bent, which may be attributed to the strain arisen during synthesis, quenching processes. **Fig. 1(d)** shows the energy-dispersive X-ray spectroscopy (EDAX) pattern of 5 % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites. The EDAX pattern clearly shows the presence of elemental composition such as Selenium (Se), Tellurium (Te), Silver (Ag) and carbon (C) (due to CNT) in our glassy composites. An extra peak of gold (Au) is also observed due to gold pasting on glassy pellets, employed in SEM characterization.

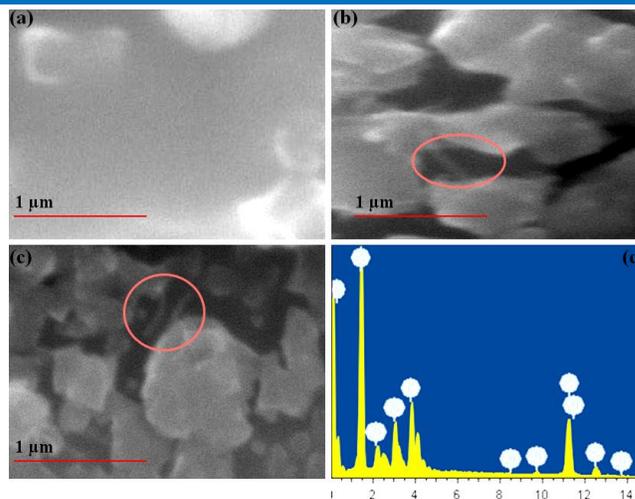


Fig. 1. SEM images of (a) pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glass and (b-c) 3, 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite.

AC conductivity spectral analysis

The variation of electrical conductivity (σ) with frequency (f) obeys Jonscher's power law and known to be the "universal dynamic response" (UDR) [22, 23]. This is because of a wide variety of materials (polymers, ceramics, glasses, composites etc.) that displayed such behaviour which is found to vary with frequency according to eqn. given below.

$$\sigma(\omega) = \sigma(0) + \sigma_{ac}(\omega) = \sigma_{dc} + A\omega^n \quad (1)$$

Here, σ_{dc} is the dc conductivity (at $\omega \rightarrow 0$) and A is a constant. Both these parameters are associated with thermal activation process. n is an exponent dependent on both frequency (ω) and temperature (T) having values in the range of $0 < n < 1$ [24].

The frequency dependence of the ac conductivity at different temperatures for the pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy sample is depicted in **Fig. 2(a)**. It can be observed that each curve has a low frequency plateau, corresponding to a DC conductivity (frequency-independent) $\sigma(0)$ of the material and a dispersive region (frequency-dependent) at high frequency, which corresponds to the AC conductivity. As observed in **Fig. 2(a)**, the frequency at which AC conductivity start off (crossover frequency), is temperature dependent. The value of this crossover frequency increases with increasing temperature. In addition, figure also confirms the dominance of DC conductivity at high temperatures which is an indicative of non-dielectric behaviour. At higher frequencies there is a second plateau in **Fig. 2(a)** showing a sharp decrease in conductivity, also observed by other workers [24] which are believed to be due to excitation of transverse optical phonons. Moreover, at higher frequency, inductive effects [25] are also accounted, which makes any attempt of applied frequency a slower response.

Fig. 2(b) shows the frequency dependence behaviour of ac conductivity for 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite at various temperatures. An inset for 387K has been shown in **Fig. 2(b)** as AC regimes for higher temperatures appear to be suppressed. Other 3 wt. % MWCNT- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite sample exhibited

similar behaviour. With the incorporation of 3 and 5 wt. % CNT concentration in pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy sample, the conductivity of the glassy composites increases with increasing frequency and temperature as well. As temperature ($T > 342$ K) increases, the conductivity indicates the dominance of dc conductivity which is an indicative of non-dielectric behaviour. The electrode polarization is assumed to be negligible [26, 27]. The conductivity of $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glass is increased by several orders of magnitude with increasing CNT content up to 5 wt. %.

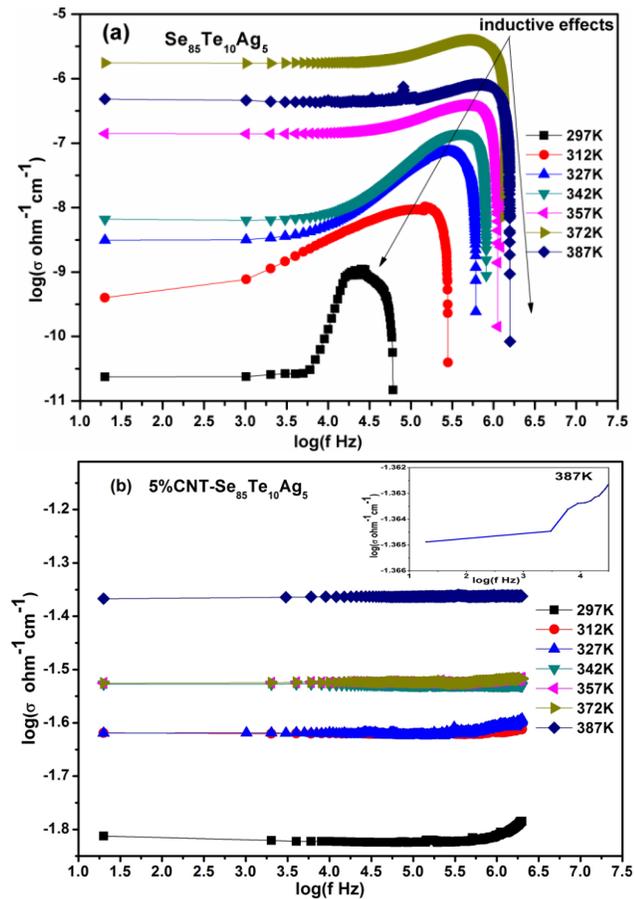


Fig. 2. (a) Relation between ac electrical conductivity (σ_{ac}) and frequency for pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glass and (b) Relation between ac electrical conductivity (σ_{ac}) and frequency for 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite.

Two distinct domains can be identified in the plots of AC conductivity of pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy sample and 3, 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites. There are several models which have been proposed to interpret this phenomenon (frequency dispersion of ac conductivity) such as CBH (correlated barrier hopping) model (including bipolaron and single polaron hopping depending on low and high temperature) [28, 29], DRT model (distribution relaxation time) [30, 22] and random resistor-capacitor networks model given by Almond *et al.* [31, 32]. According to the random R-C networks model, materials are suggested to have microstructural networks consisting of conducting and insulating regions [31-34] which are exhibited by a complex random network of resistors and capacitors. The low frequency plateau in Fig. 2(a, b) corresponds to the

one or more randomly configured percolation paths of resistors across the network. In low frequency region, the capacitor admittances (ωc) are very small and it makes the region to act as effective insulators, leaving the percolation paths of the resistors alone to determine the low frequency conductivity $\sigma(\omega)$ response. At higher frequencies, the R-C network capacitance falls as fractional powers of frequency giving rise to ac conductivity. These effects occur at frequencies where the magnitude of admittance (ωc) of capacitors turns out to be comparable to that of the resistors (R^{-1}). As a result, at these frequencies, ac currents starts to flow via a complex set of paths with all components contributing to the network conductivity. Various researchers [35, 17] have suggested that, the full network of resistors and capacitors are randomly connected in series and parallel at sufficiently higher frequencies, giving rise to power law dispersions (ω^n) in electrical response. Thus, a logarithmic mixing rule should account for the characteristics of such complex arrangements of R-C components.

Careful examination of Fig. 2(a, b) shows shifting of onset frequency (ω_0) on going towards higher temperatures. This onset frequency (ω_0) at which the ac conductivity switches over from frequency-independent region (DC region) at low frequency to the frequency-dependent region at higher frequencies give information about conductivity relaxation phenomenon [30] shifting towards higher frequencies with increasing temperature.

Fig. 3 shows the ac conductivity behaviour for pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy sample and CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites as a function of 3, 5 wt. % CNT fractions, at room temperature (R.T.) between 20Hz to 2MHz. An inset in Fig. 3 for 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ sample has been shown as the AC regime is suppressed on showing altogether in one graph. It can be observed from the Fig. 3 that onset frequency ω_0 shifts towards higher frequencies with increasing CNT content.

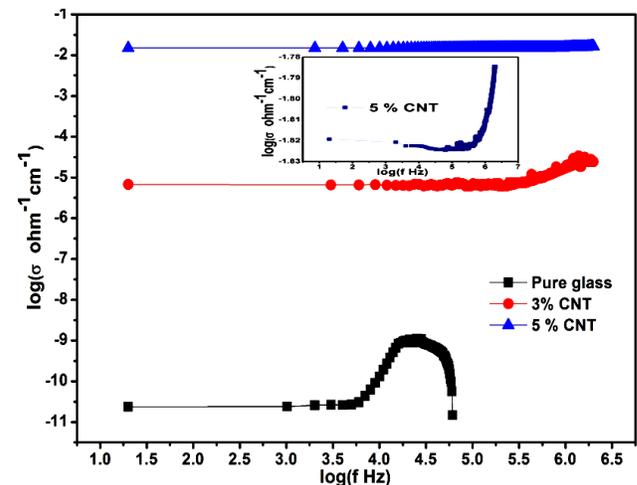


Fig. 3. Relation between ac electrical conductivity (σ_{ac}) and frequency as a function of CNT content (CNT wt. %).

Higher the value of onset frequency, shorter is the correlation length (λ) [36]. In disordered systems, the correlation length (λ) exhibits the distance between connections, suggested by Kilbride *et al.* [37]. As stated by Sangeeth *et al.* [36], the onset frequency ω_0 is that

frequency at which charge carrier can travel a distance approximately equal to λ and also the onset frequency scales inversely proportionally to some power of the correlation length (λ). As frequency exceeds the ω_0 , charge carrier travels a shorter distance within a well-connected region without being suffered by difficult hopping processes.

Thus, the observed enhancement in ω_0 with the increase of CNT content implies that connectivity was improved with greater CNT addition in the glassy matrix [37, 36]. Therefore, the value of onset frequency (ω_0) might be considered as a useful parameter to investigate the level of connectivity in the $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy system, so that the performance of CNT composites can be optimized.

The temperature dependence of the dc conductivity in temperature range (297K-387K) has been evaluated from the low frequency plateau region for pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glass and 3, 5 wt.% CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites under investigation. As shown in Fig. 4, the variation of $\log \sigma$ vs. $1000/T$ for pure and 3, 5 wt.% CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites show straight lines indicative of the fact that d.c. conductivity (σ_{dc}) increases with increasing temperature. In addition, it also suggests that dc conduction mechanism is a thermally activated process.

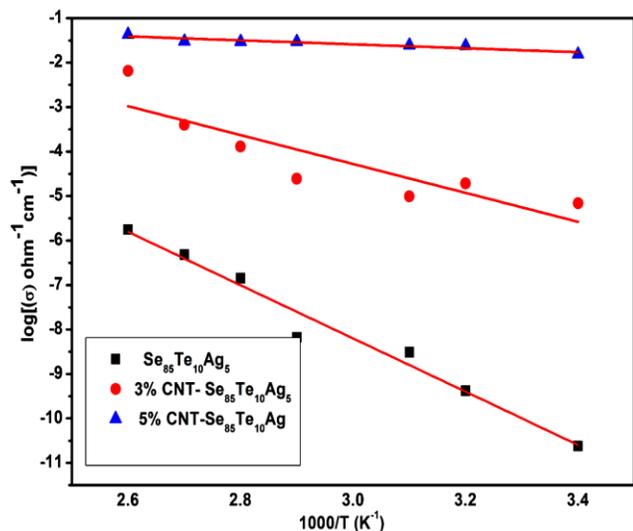


Fig. 4. $\log(\sigma_{dc})$ as a function of $1000/T$ for pure and 3, 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites.

The mechanism for such a conduction process can be explained in light of small polaron hopping (SPH) conduction model [38]. The dc conductivity and activation energy (ΔE_{dc}) for dc conductivity for all three samples have been calculated from Arrhenius equation given below

$$\sigma_{dc} = \sigma_0 \exp(\Delta E_{dc}/KT) \quad (2)$$

where, σ_0 is the pre exponential factor which is constant for a particular glass composition, K is the Boltzmann constant. The obtained ΔE_{dc} and dc conductivity values are given in Table 1.

It can be observed from the Table 1, that the values of activation energy (ΔE_{dc}) for dc conduction decrease with the increase of CNT content. The dc conductivity for 3, 5 wt. % CNT content sample increased by 6 and 9 orders of

magnitude respectively in comparison to pure glassy alloy. This dramatic increase in the dc conductivity value with 3 and 5 wt. % of CNT content exhibits a typical insulator-conductor transition. When CNT content is incorporated in pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy alloy, the internanotube connections are expected to increase with increasing CNT content upto 5 wt. %, giving rise to many conductive paths in glassy matrix. Thus, the significant carriers which travel through the entire glass network may follow the percolating paths that avoid larger barriers in their conducting paths [38].

Table 1. Calculated values of dc conductivity (σ_{dc}) and activation energy (ΔE_{dc}) for pure and CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composites.

| Sample | σ_{dc} (S/cm) | ΔE_{dc} (eV) |
|--|-------------------------|-------------------------|
| $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ | 2.37669E-11 | 0.517 |
| 3%CNT- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ | 6.00122E-5 | 0.281 |
| 5%CNT- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ | 1.541E-2 | 0.038 |

Dielectric properties

The chalcogenide glassy composites with large dielectric constant have been paid more attentions in recent years. Usually, the dielectric constants of the glassy matrix can be increased by two methods: by admixing or doping of high dielectric constant component [39, 40] or admixing/doping of conductive component [39-43].

Fig. 5(a) shows the frequency dependence of the dielectric constants at different temperatures for pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy sample under investigation. It can be observed from Fig. 5(a) that dielectric constant increases with increase of temperature and shows frequency independent behaviour from low to intermediate frequencies, and further sharply decreases at higher frequencies. One of the advantages of base $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glass is that it has a weak dependency of dielectric constant in low to intermediate frequency range. The sharp decrease in dielectric constant value at frequencies above 10^5 Hz may be attributed to the dielectric relaxation behaviour [5].

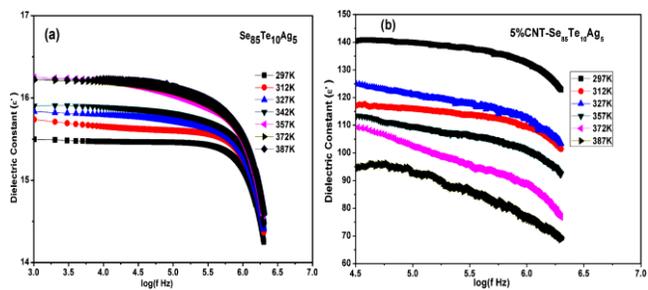


Fig. 5. (a) Relation between dielectric constant (ϵ') and frequency for pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glass. (b) Relation between dielectric constant (ϵ') and frequency for 5 wt.% CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite.

Fig. 5(b) shows the frequency dependence behaviour of dielectric constants at different temperatures for 5 wt. % CNTs- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite. It can be observed from figure that the dielectric constant shows largest value

at room temperature (297K) and further decreases with increase of temperature. Similar behaviour is also observed in case of 3 wt. % MWCNT- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite with small dielectric constant value. Ahamad *et al.* [5] and Li *et al.* [44] have reported that incorporation of CNTs produces many inter conducting clusters which are isolated by thin dielectric layers. These conducting clusters serve as many minicapacitors. Further, the polarization effects acting between conducting clusters inside the percolation network (based on Inter cluster Polarization model) improve the electric charge storage of glassy alloy.

In this way, these factors contribute to the enhancement of dielectric constant with CNT incorporation. This indicates that carbon nanotubes do not behave as an inert conducting phase mixed in the base $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy matrix but that they interact with glassy matrix affecting its properties. Fig. 6 shows an enhancement in dielectric constant with increasing CNTs content in pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy alloy at room temperature. As shown in Fig. 3 conductivity increases with increasing CNTs content in pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy alloy which implies that the availability of CNTs created pathways suitable for the migration of more charge carriers. In this respect, space-charge polarization takes place, leading to an increase in the dielectric constant.

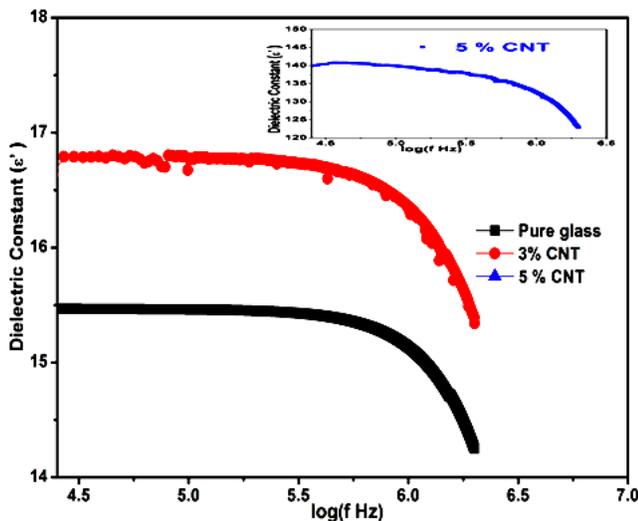


Fig. 6. Relation between dielectric constant (ϵ') and frequency as a function of CNT content (CNT wt. %).

Fig. 7(a and b) shows the dependence of dielectric constant and dielectric loss as function of CNT concentration at room temperature (297K) and 50 KHz. As CNT concentration increases dielectric constant as well as dielectric loss increases and such kind of results have also been reported by other researchers [5, 41, 43].

The largest dielectric constant and loss is observed for 5 wt. % CNT- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite at 50 KHz. The enhancement in dielectric constant values has already been discussed by mini capacitor principal [5, 44] whereas enhancement in dielectric loss can be attributed to typical structure of CNTs. It has been reported [44] that CNTs produce electric current under the application of electric field which further causes electrical energy conversion to thermal energy. This energy conversion increases with

increasing CNT concentration, giving rise to more dielectric loss.

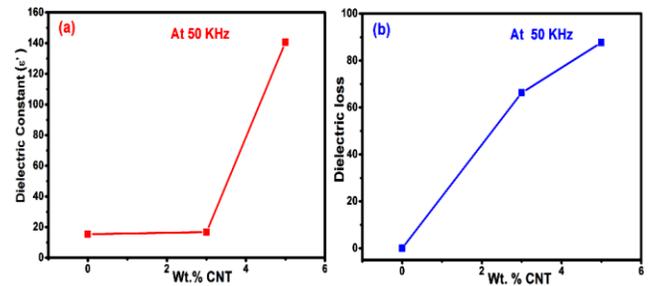


Fig. 7. Dependence of the (a) dielectric constant and (b) dielectric loss as a function of CNT content (CNT wt. %) at a frequency of 50 KHz.

Conclusion

The following conclusions have been drawn based on experimental results:

- The ac conductivity exhibited a low-frequency plateau and a high-frequency dispersion regime. The onset of conductivity relaxation (switch over frequency) shifted to higher frequencies with increasing CNT content in pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy alloy indicating a better CNT-CNT connectivity in pure glassy alloy due to CNT incorporation.
- A significant increase in the dc conductivity value was observed with increasing CNT concentration up to 5 wt. %, the conductivity increased by 6 to 9 orders of magnitude. This may be attributed to the effect of internanotube connections for increased CNT content in the pure $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy alloy.
- The dielectric constant values significantly enhanced with increasing CNT content and shows enhancement by 10th order for 5 wt. % CNT content. The largest dielectric constant of 142 is obtained for 5 wt. % CNT- $\text{Se}_{85}\text{Te}_{10}\text{Ag}_5$ glassy composite in low frequency range at room temperature. This large dielectric constant behaviour of CNT containing glassy composite is an indication of providing pathways for more charge carriers by the CNTs.

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Author contributions

Conceived the plan: Kedar Singh; Performed the experiments: A.N.Upadhyay; Data analysis: A.N.Upadhyay, Kedar Singh; Wrote the paper: A.N.Upadhyay, Kedar Singh, R.S.Tiwari. Authors have no competing financial interests.

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