Investigation of Dielectric and Electrical Properties of $BiFeO_3$-$PbZrTiO_3$ (BFO-PZT)-Polyvinyl Alcohol (PVA) Composites

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Abstract

The composites of PVA-BFO-PZT-Ag were prepared through solution casting technique by dissolving polyvinyl alcohol in water and stirring for 2h at 80^0C*. Then, the desired amount of Ag and 10 vol % of* $BiFeO_3$*-*$PbZrTiO_3$ *(BFO-PZT) were mixed with water under ultrasonication at room temperature for 1 h to disperse the BFO-PZT-Ag particles homogeneously. Prior to this,* $BiFeO_3$*-* $PbZrTiO_3$ *ceramic was prepared by the conventional solid state reaction method by mixing stoichiometric amount of powders of Bismuth oxide (*Bi_2O_3*), Iron oxide (*Fe_2O_3*), Lead Oxide (*Pb_3O_4*), Titanium dioxide (*TiO_2*), and Zirconium oxide (*ZrO_2*) thoroughly in agate mortar in an air atmosphere for 2 h and in alcohol for 2 h and then subjected to calcination at 950* ^0C *for 4 h..Ag nano particles can also be synthesized by taking Silver nitrate (1mM) and Hydrazine hydrate as a metal salt precursor and a stabilizing agent respectively along with CTAB. The suspension of BFO-PZT-Ag was poured into the mixture of PVA solution. The mixture so obtained was stirred with mechanical stirrer to ensure that the Ag particles are well dispersed in the polymer matrix. The resulting composite films were made by casting onto a glass substrate and heating in an oven at* 80^0C *for 12 h to remove any traces of the solvent. Composites containing 10, 20, 30 and 40 wt% of BFO-PZT were prepared by following the same procedure in the absence of Ag content for comparison. The absorption spectra of silver sol consisting of a single sharp peak at* λ_{max} *= 418 nm due to the excitation of surface plasmon resonance band in the UV visible regions has been observed. The use of three component systems i.e. PVA-BFO-PZT in presence of Ag nanoparticles has enhanced the interfacial adhesion of two component systems and the particles are well coated and dispersed in PVA matrix as compared to two component PVA-BFO-PZT systems as revealed by SEM analysis. The dielectric constant of the composite has gradually increased upto ~373 at 100 Hz for 1.1vol% Ag which is nearly 34 times higher than that of pristine PVA. The AC conductivity of the composites has also increased with the increase in frequency for all the concentrations of Ag content, which is slightly higher than that of pristine PVA indicating that there is no conductive network and the prepared films can be used for making suitable insulators.*

Key words: PVA-BFO-PZT-Ag, $BiFeO_3$***-***$PbZrTiO_3$ ***(BFO-PZT), Surface Plasmon Resonance, Insulators.***

1. Introduction

Recently, there has been a constant search for new dielectric materials with high dielectric constant which has attracted great interest for their wide range of potential applications [1] in capacitors [2], gate dielectrics [3] and electric energy storage devices [4]. Polymer based materials with high dielectric constant have become more prominent for dielectric applications due to their low cost processing, easier to fabricate, better dielectric breakdown strength and mechanically more amenable than the ceramics. However, the dielectric constant of pure polymers is very low (i.e., <10), thus, it is a key issue to improve the dielectric constant of polymer nanocomposites [5-6]. So far, various strategies have been adopted for the improvement of dielectric constant of polymer nanocomposites. One common approach is to introduce high dielectric constant ceramic particles (e.g., $BaTiO_3$ [7], $CaCu_3Ti_4O_{12}$ (CCTO) [8], $PbZrTiO_3$ [9]) into flexible polymer matrix. However, the enhancement of the dielectric constant of the composite can only be done by the addition of certain amount of ceramic fillers. Normally at typical ceramic filler loading over 50 vol % may result in damaging of mechanical performance of the polymeric composites [4, 10]. For example, Pingkai et al [11] demonstrated that the dielectric constant enhancement of pristine PMMA (3.49) at 10^3 Hz to 14.6 from PMMA/$BaTiO_3$ composite when $BaTiO_3$ content is as high as 76.88 wt%. To overcome these issues, dispersing conductive fillers have been incorporated into polymer based ceramic composite systems as the third component, in which whole filler contents is much lesser. The three phase composite containing metal particles (Ag, Zn, Cu and Ni) [2, 12], carbon nanotube [13-14] carbon fibre [14], graphene [15-16], conductive oxide [17] and conductive polymer [18], into the polymer matrix, has been used to achieve high dielectric constant. According to percolation theory [19], the dielectric constant and electrical conductivity of such composites can be increased dramatically, when the weight percentages of filler content increases to near the percolation threshold [19, 20]. But, the conductive fillers (i.e., silver nanoparticles) have significant importance because of their excellent electrical, thermal and optical properties. Moreover, these types of composites have usually quite high dielectric loss, which may be due to the formation of conductive network near the percolation threshold [10, 21]. The uniform dispersion of conductive fillers in the polymer matrix can improve the dielectric constant of the resultant composite to achieve percolation threshold and can be attributed as a potential candidate in micro capacitors. These micro-capacitor are produced by separating the nearest conductive fillers with an insulating polymer based ceramic systems [21]. So, the most critical role in formulating composite with high dielectric constant while maintaining the low dielectric loss is still a great challenge.

Nowadays polymer composites are being made with incorporation of nano fillers like nano clays, nano particles, nano tubes, nano fibres, etc. This reinforcement of nano fillers into polymers enhances their mechanical and thermal barrier properties along with their processing behaviour considerably. Also, better dispersion of these fillers within the matrix provides high performance nanocomposites. [22, 23]

If we add ceramic fillers into the polymer matrix, ‘then the dielectric property of the composites increases. But if we add more amount of fillers (say 40%) the mechanical properties of the composites decrease and also possess poor processing [24-26]. To overcome this issue the idea of three phase composite involving nanoparticles as conductive fillers comes in to play. Nanoparticles like carbon nanotubes, nanofibers, nanoplates, grapheme nanoparticles and many metal nanoparticles like Pd, Ag, Ni etc. have been used as conductive fillers to modulate the mechanical strength of the composites. Recently, there are various strategies reported on the three phase composites such as PVDF/$BaTiO_3$-Ni [27], PVDF/$BaTiO_3$-CNT [28], PVDF/$BaTiO_3$-Carbonfiber [29], PVDF/$BaTiO_3$-Ag [30], PMMA/$BaTiO_3$-Ni [31], Epoxy /$BaTiO_3$-CNT [32], PS/$BaTiO_3$-GNS [33].

Polyvinyl alcohol (PVA) is a good insulating material with low conductivity; therefore it is very useful in microelectronic industry [34]. Electrical conductivity of PVA generally depends on the thermally generated carriers and also with the addition of suitable dopants. Also the PVA polymer is a semi crystalline material with several interesting physical properties which are very useful in technological applications [35].

Lead-zirconate-titanate, $PbZr_xTi_{1-x}O_3$ or PZT, is considered as an important ferroelectric material. Thin films of PZT are widely used for preparing electronic devices like non-volatile memories, infrared sensors, optical shutters and modulator, micro-sensors and micro-actuators [36-39]. Though PZT is a good ferroelectric material having high dielectric constants, it is brittle in nature and is not suitable for high frequency applications. Its poor mechanical strength and lack of ductility possess problems for applications requiring mouldable sensors. As we know polymers do not have desirable electric properties but they serve as excellent host media to incorporate ceramic fillers. Different PZT-polymer composites have been prepared and properties were studied for their dielectric applications [40, 41].

In the present work, the polymeric materials are used to prepare nanocomposite films (PVA-$BiFeO_3$-$PbZrTiO_3$-Ag) by taking 10 wt% of BFO-PZT and several weight percentage of Ag *via* solution casting techniques. Scanning electron microscope (SEM) was investigated to study the surface morphology of resultant PVA-BFO-PZT-Ag nanocomposites. The electric and dielectric properties of PVA-BFO-PZT-Ag nanocomposites were examined and percolation theory was used to study their electric and dielectric behaviours of the composites.

2. Materials and Methods

2.1 Materials

Bismuth Oxide (Bi_2O_3), Iron oxide (Fe_2O_3), Lead Oxide (Pb_3O_4) 99.5% purity, Zirconium Oxide (ZrO_2), Titanium Oxide (TiO_2) 99% purity, were purchased from Merck, India. Polyvinyl alcohol (PVA) was purchased from Alfa Aesar. Silver nitrate ($AgNO_3$), Hydrazine hydrate, CTAB, all these chemicals were used without further purification. Millipore water was used throughout the experiments.

2.2 Preparation of $BiFeO_3$- $PbZrTiO_3$ (BFO-PZT)

$BiFeO_3$- $PbZrTiO_3$ ceramic was prepared by the conventional solid state reaction method. Stoichiometric amount of starting powders Bismuth oxide (Bi_2O_3), Iron oxide (Fe_2O_3), Lead

Oxide (Pb_3O_4), Titanium dioxide (TiO_2), and Zirconium oxide (ZrO_2) was mixed thoroughly in agate mortar in an air atmosphere for 2 h and then in alcohol for 2 h. The mixed powders were calcined at 950^o C for 4 h. in a high-purity alumina crucible. The obtained calcined BFO-PZT powders were grinded to powder form and taken for XRD to confirm.

2.3 Preparation of Ag Nanoparticles

Silver nitrate (1mM) and Hydrazine hydrate were used as a metal salt precursor and a stabilizing agent respectively. First, the CTAB was dissolved in Millipore water and then few drops of hydrazine hydrate were added to the solution. Silver nitrate solution was added drop wise to the above solution with constant stirring at 60^{0}c. Yellow colour appearance of the solution confirms the formation of silver nanoparticles. UV-Absorption spectra were taken for confirmation.

2.4 Preparation of PVA-BFO-PZT Composites

The fabrication of composites was done by using solution casting techniques. In this method, the desired amount of PVA was dissolved in water by constant stirring at 70-80^{o}C. The ceramic particles (BFO-PZT) dispersed in water are sonicated for half an hour. It was then mixed with PVA solution with constant stirring in magnetic stirrer. After complete dissolution of composite, the solution was casted at 70^{0}C in oven to remove any traces of the solvent.

2.5 Preparation of PVA-BFO-PZT-Ag Composite Films

The composites of PVA-BFO-PZT-Ag were prepared through a solution casting techniques, described as follows. Firstly, polyvinyl alcohol was dissolved in water and stirred for 2h at 80^{0}C. Then, the desired amount of Ag and 10 vol% of $BiFeO_3$-$PbZrTiO_3$ was mixed into water under ultrasonication at room temperature for 1 h to disperse the BFO-PZT-Ag particles homogeneously. Then, the suspension was poured into the mixture of PVA solution. The obtained mixture was stirred with mechanical stirrer to ensure that the Ag particles are well dispersed in the polymer matrix. The resulting composite films were made by casting onto a glass substrate and heating in an oven at 80^{0}C for 12 h to remove any traces of the solvent. For the purpose of comparison, composites containing 10, 20, 30 and 40 wt% of BFO-PZT were prepared by same procedure in the absence of Ag contents.

2.6 Characterization Techniques

UV spectra were obtained using a Shimadzu 220V (E) UV-vis spectrophotometer. Fourier-transform infrared (FTIR) spectroscopy was recorded with a 5700 FTIR (Nicolet) to examine the modification of BFO nanoparticles. The surface morphology was analyzed by scanning electron microscope (SEM, Zeiss EVO 18). The dielectric properties of the nanocomposite films were carried out by using an impedance analyzer (HIOKI 3532 LCR HiTESTER) at a frequency range of (100Hz–1 MHz) at room temperature**.**

3. Results and Discussion

The preparation of PVA-BFO-PZT-Ag composites was carried out by solvent casting technique using polyvinyl alcohol as matrices. To investigate the electrical, dielectric and Impedance properties of such composites, the preparation was made at various volume percentages ranging from 0.1 to 1.1 vol% of conductive filler.

3.1 Morphology of PVA-BFO-PZT-Ag Composites

Dispersion of particles into the polymer matrix is one of the crucial factors to have improved dielectric properties of the composites. The dispersion of fillers in a polymer matrix is not an easy process, particularly if someone is using nanoparticles, as they have a strong tendency to agglomerate. It might be worth mentioning here that the use of three component systems i.e. PVA-BFO-PZT in presence of Ag nanoparticles enhanced the interfacial adhesion that of two component systems. As shown in Fig. 1(a), one can find that the particles are well coated and dispersed in PVA matrix as compared to two component PVA-BFO-PZT systems. From Fig. 1(b) one can find that there are partial clusters even through the cluster are not serious, which may be due to the agglomeration of ceramic particles over the polymer matrix.

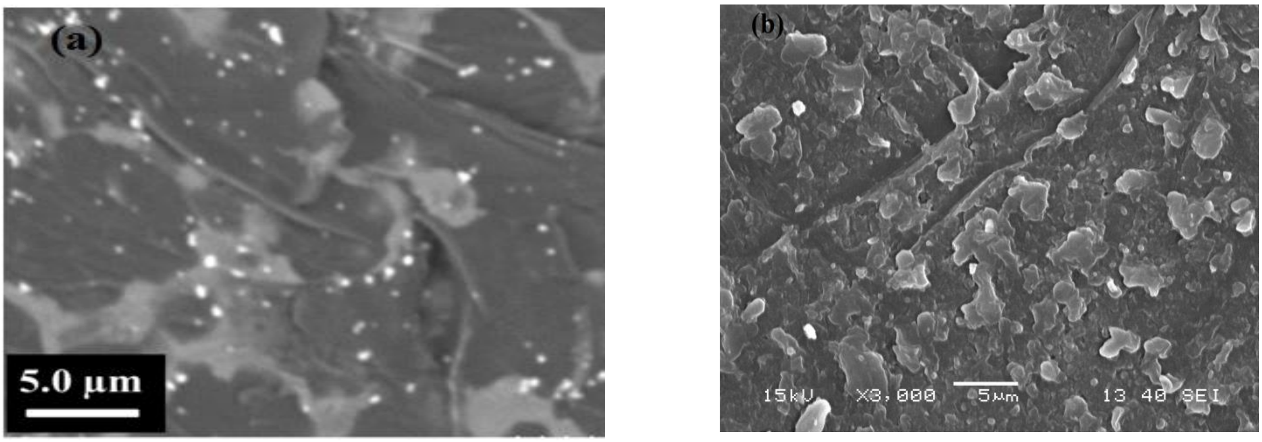

Fig.1. SEM Micrographs of the (a) PVA-BFO-PZT (b) PVA-$BiFeO_3$-$PbZrTiO_3$-Ag composites.

3.2 UV-Visible Spectra Analysis

UV-visible spectroscopy is one of the most commonly used techniques for structural analysis of silver nanoparticles. As the hydrazine hydrate solution was mixed with aqueous solution of the silver nitrate and CTAB as stabilizing agent, it starts appearing as pale-yellow colour due to the reduction of silver ion, which indicates the formation of silver nanoparticles. It generally shows that, the UV–Vis spectroscopy could be used to study the shape and size controlled nanoparticles in aqueous solution. The absorption spectra of silver sol consists of a single sharp at λ_{max} 418nm (Fig. 2) due to the excitation of surface plasmon resonance band in the UV visible regions. The most characteristic part of silver sol is a narrow plasmon absorption band observable in the 350–600 nm ranges. A broad surface plasmon resonance band is due to aggregation or adsorption of hydrazine hydrate onto the surface of Ag-nanocrystals.

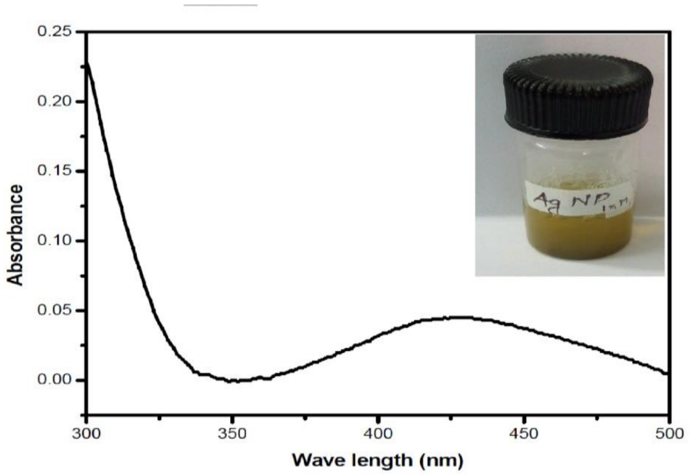

Fig.2. UV-Visible absorption spectrum of silver nanoparticles

3.3 Dielectric and Electrical Properties of three component PVA- BFO-PZT-Ag Composites

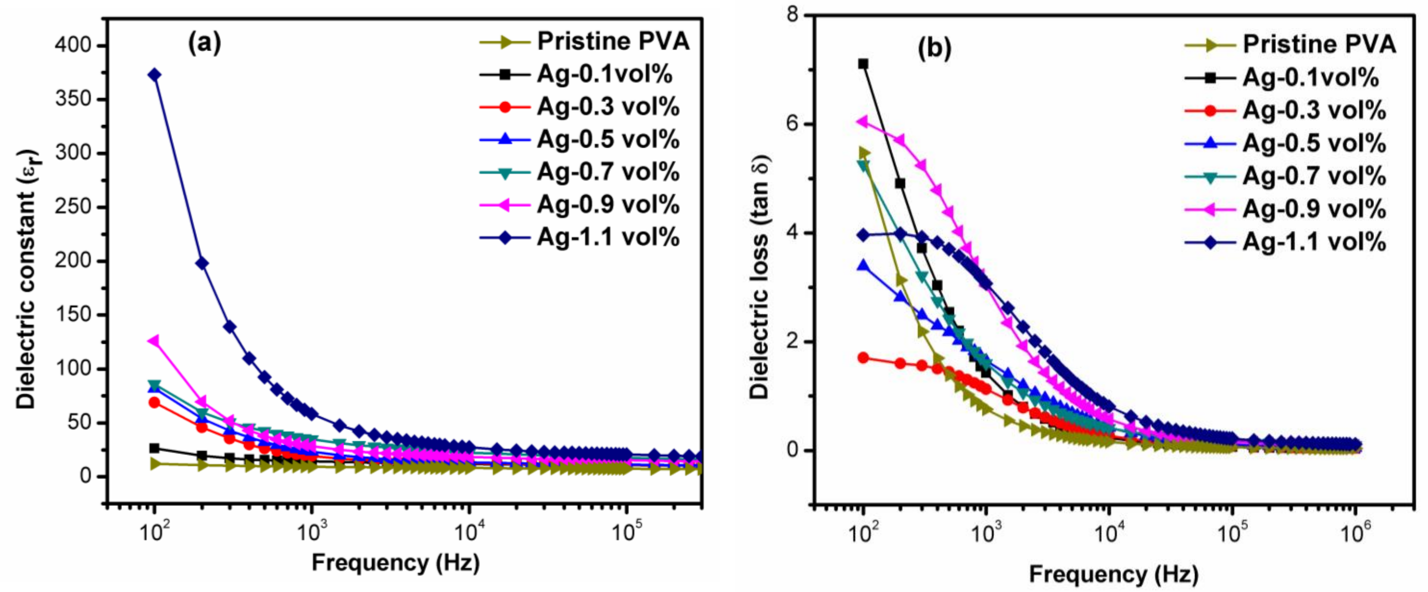

Fig.3 Frequency dependence of (a) dielectric constant and (b) dielectric loss (tan δ) of PVA-BFO-PZT-Ag composite films at various volume percentages of Ag contents.

Figure 3 illustrates the dielectric properties of the PVA-BFO-PZT-Ag composite films as a function of frequency at different contents of Ag nanoparticles. From Fig.3 (a), we can see that, the dielectric constant of the composite gradually increases with the increase of Ag nanoparticles loading over all frequency range. The dielectric constant of the composite with 1.1vol% Ag nanoparticles reaches ~373 at 100 Hz, which is nearly 34 times higher than that of pristine PVA. As shown in Fig. 3(a), the homogeneous distributions of Ag nanoparticles are isolated by a thin PVA layer, results a number of micro–capacitors and it may be due to the high dielectric constant of the composites. In addition, charge can be accumulated at the interface between Ag, BFO, PZT and the polymer matrix due to their various dielectric constants as well as conductivity (Fig.5). These dielectric properties of the composite are closely associated to Maxwell-wagner-sillars (MWS) interfacial polarization, which can make a significant role to the increase in dielectric constant at low frequency regions [41]. Moreover, the dielectric constant increases sharply as the Ag nanoparticles content approaches the percolation threshold. When Ag nanoparticles loading increases, more micro-capacitors are formed and results superior interfacial area in the composite, which exhibits a steady increase in dielectric constant. However, the dielectric constant decreases with the increase in frequency, which is due to the decrease in dipolar contribution at high frequencies and also shows slow dielectric relaxation of the matrix and the interface of the composite [42].

Fig.3 (b) shows the dielectric loss of PVA-BFO-PZT-Ag composites with various contents of Ag nanoparticles as a function of frequency. As shown in Fig. 3(b), the dielectric loss is generally determined by electrical conduction and dipolar polarization at larger frequency. The low dielectric loss is observed at high frequency region which is due to the highly restricted polymer chain motion in high frequency ranges. However, the dielectric loss increases slowly at low loading of Ag nanoparticles, which is due to the typical insulating materials. When the Ag content is 0.1 vol% and above, the dielectric loss exhibits a significant increase. For example, at higher concentration of Ag i.e. 1.1 vol%, the value is 3.97 (100 Hz) but, the corresponding value for lower concentration of Ag i.e. 0.1 vol% is 7.1 (100 Hz). Thus, an insulator-conductor transition is formed at 0.1 vol% for Ag as conductive fillers and there is a homogeneous distribution of Ag nanoparticles in the PVA matrix. As the Ag nanoparticles increases beyond the percolation threshold, the dielectric constant rises (Fig. 3(a)) and dielectric loss is also high. Both high dielectric constant and high dielectric loss composites may be used in the electromagnetic wave absorption fields [43].

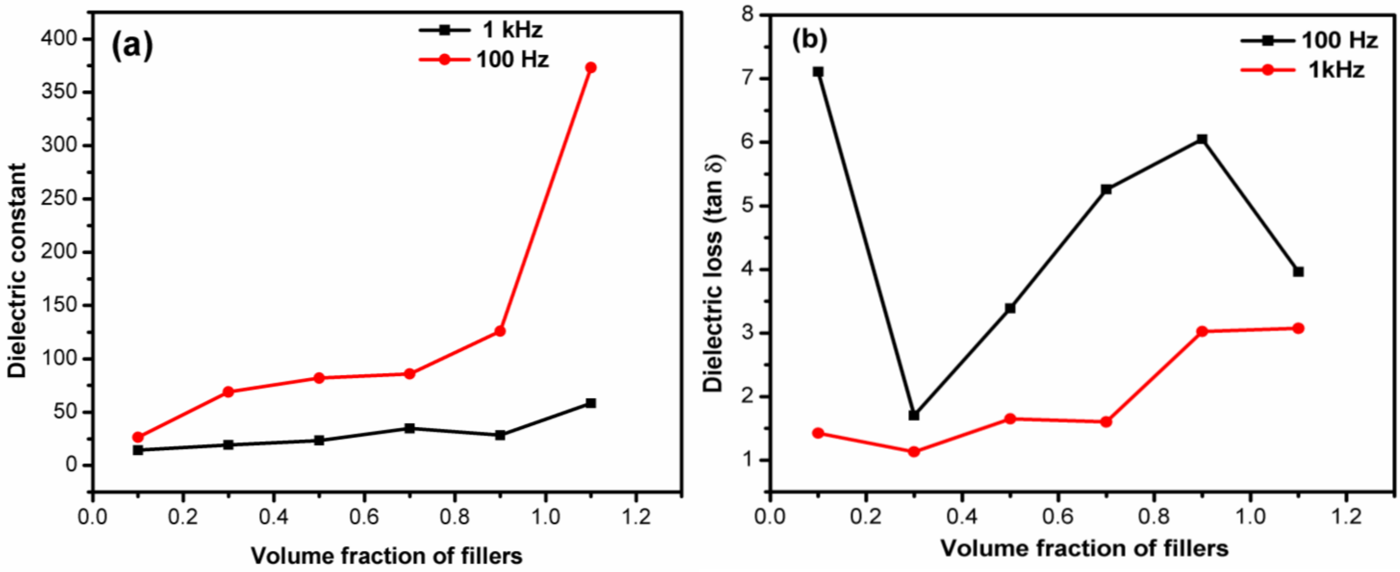

Fig.4 (a,b) Comparison of (a) dielectric constant and (b) dielectric loss of PVA-BFO-PZT-Ag composite films with Ag loading at 100 Hz and 1 kHz.

Fig.4 presents the dielectric properties of PVA-BFO-PZT-Ag composite films with Ag contents at 10^2 Hz and 1 kHz, respectively. As shown in Fig. 4(a), it is found that, the dielectric constant of the composites gradually increases as the increase in Ag contents. When the content of Ag is 1.1vol%, the dielectric constant of the three component composite can reach 373.14 (10^2 Hz) and 58.36 (1 kHz), which is much higher than that of the PVA-BFO-PZT composite without Ag contents.

3.4 AC Conductivity of PVA-BFO-PZT-Ag Composites

The AC conductivity study is performed to incorporate the effect of conduction mechanism and different type of charge carrier in the materials. The value of AC conductivity (σ_{ac}) of the composite is evaluated by using empirical formula:

$$\sigma_{ac} = \varepsilon_0 \varepsilon_r \omega \tan\delta$$

Where the symbols have their usual meaning: σ_{ac} = AC Conductivity, ε_0 = Permittivity in free space, ε_r = Relative dielectric constant, ω = Angular frequency, $\tan\delta$ = Loss tangent

Fig.5 presents the AC conductivity of PVA-BFO-PZT-Ag composites with various Ag contents in the wide range of frequency. The AC conductivity of the composites increases with the increase in frequency for all the concentration of Ag contents, which is slightly higher than that of pristine PVA. Moreover, it also indicates there is no conductive network is obtained and the prepared films to make well as insulators [44].

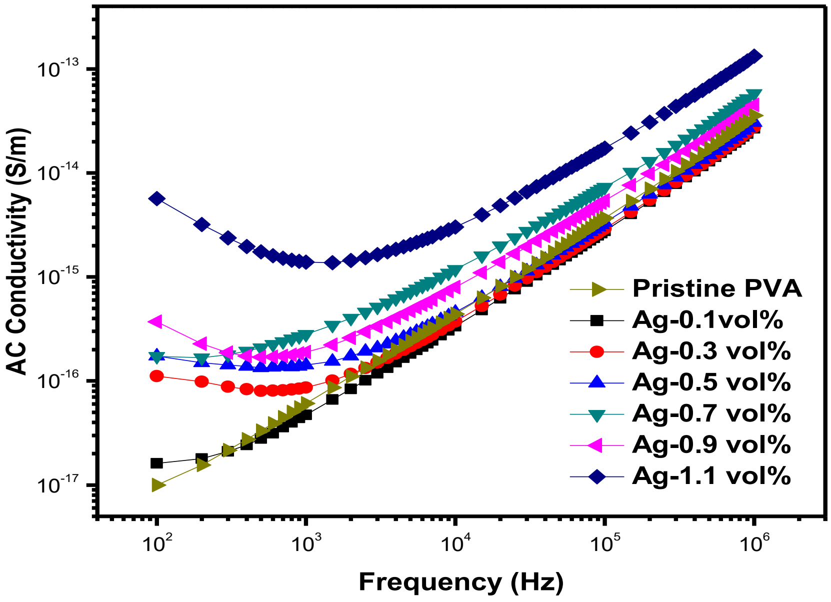

Fig.5 Frequency dependence of AC conductivity of PVA-BFO-PZT-Ag composite films at various content of Ag nanoparticles.

4. Conclusion

A novel three component PVA-BFO-PZT-Ag composites were prepared by solution casting techniques. These composites have high dielectric constant, high loss and better flexibility. The AC conductivity increases exponentially with frequency and Ag nanoparticles loading due to formation of conductive path in the polymer matrix The easy processing, flexibility and good dielectric behaviour of such three component composites potentially will make the composites more attractive for practical applications.

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