Yttrium Barium Copper Oxide-Filled Polystyrene as a Dielectric Material

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ABSTRACT: Electrical impedance measurements are carried out on high temperature superconducting ceramic Yttrium Barium Copper Oxide (YBCO)–Polystyrene (PS) composite materials, in which superconducting particles are embedded in polystyrene matrix. The results of impedance versus frequency (100 Hz–13 MHz), phase angle versus temperature for volume percentage of superconductor (0–40%) are presented. No marked transition in phase angle is observed when the material goes through the superconducting transition temperature of the filler. The dielectric constant and losses increase with increasing YBCO content. However the increase in losses is modest and the excellent dielectric properties of the composites are not adversely affected. The system conforms to Clausius-Mossotti equation. Dipole moment of YBCO particles and polarizability of the composites are calculated using the Clausius-Mossotti approaches. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 120: 2233–2241, 2011

Key words: composites; modeling; dielectric properties; Claussis Mossotti equations; polystyrene

INTRODUCTION

Hard, brittle, high temperature superconducting ceramics can be incorporated into polymer matrices[1,2] for preparing composite materials with superior mechanical properties, greater processability, and flexibility. They can be easily molded into various useful shapes by versatile polymer processing techniques such as compression, extrusion, and injection molding. In particular, the composites, in which superconducting ceramic powders embedded in polymer matrices with (0-3) connectivity, are extremely flexible.

The practical application of high temperature superconductors for electronic applications required the development of novel processes and devices. The development of capacitors that are compatible with superconducting and manufacturing materials capable of high frequency operations will be required for integrated devices and that can be prepared by the suitable inclusion of high temperature superconducting materials with polymers.

When polymeric materials serve for practical uses, they are commonly mixed with other materials to achieve desired performances. Dielectric and conductive properties can be controlled over a broad range by ceramic inclusions.[3] It is important to study the "AC" electrical properties of these new materials, which would give information about their microstructure, composite dielectric behavior, frequency, and temperature dependence.

In our previous paper a detailed study of the mechanical properties of the aforementioned composites are reported.[4] The pace of research on thermal and dielectric properties of heterogeneous materials has accelerated in recent years. This is because electronics packaging has continuously provided the impetus pushing in the development of new materials in a fascinating and rich variety of applications.[5] In this article, we report a study on the dielectric properties of Yttrium Barium Copper Oxide (YBCO)–Polystyrene (PS) composites. Dielectric mixing laws are examined for the composites consisting of superconductor ceramic particles (YBCO) dispersed in the (PS) matrix with (0-3) connectivity. Clausius-Mossotti approximation is one of the most commonly used equations for calculating the bulk dielectric properties of inhomogeneous materials. It is useful when one of the components can be considered as a host in which inclusions of the other components are embedded. It involves an exact calculation of the

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field induced in the uniform host by a single spherical or ellipsoidal inclusion and an approximate treatment of its distortion by the electrostatic interaction between the different inclusions.

AFM (atomic force microscope) pictures of selected composites have been made to characterize the morphology of the composites.

EXPERIMENTAL

Materials

Solid-state reaction technique was adopted for the preparation of the material (YBCO).[6] The starting materials were $BaCO_3$ (Merck, Mumbai, India), Y_2O_3 (CDH, New Delhi, India), and CuO (Merck, Mumbai, India). The specific properties of the Polystyrene (Merck, Mumbai, India) needed for this study are dielectric constant and glass transition temperature and the values are 2.5 and 108°C, respectively.

Method of composite preparation

The melt mixing technique was chosen for preparing the composites because it allowed solvent-free mixing for the ceramic filler. By melting (PS) at high temperature, molten polystyrene can easily penetrate between filler particles, which facilitate suitable mixing and allow avoiding air trapping into the composites. Consequently void free composites are obtained.

YBCO-PS composites are prepared in a brabender plasticoder. The compositions of the samples are 10, 20, 30, and 40% by the volume of the filler.[7] The mixed samples are compression molded into sheets of desired thickness by hydraulic press at a temperature of 180°C and are used for different studies. The composites are named as YBCO10, YBCO 20, YBCO 30, and YBCO 40 where 10, 20, 30, and 40 represents the vol % of the filler in the matrix.

Measurements

Tapping mode atomic force microscope measurements are carried out in air at ambient conditions (28°C) with a Veeco Nanoscope 3D, made by Digital Instruments, USA. The characteristics of the measurements are scan rate, scan size, and data points. Images are analyzed using a Nanoscope image processing software. Particle size is calculated by the particle analysis software "Nanoscope V531r1."

Dielectric measurements are performed with Hewlett–Packard-Japan HP 4192A impedance analyzer at a temperature range of 28–120°C and at a frequency range of 100 kHz to 13 MHz. For low temperature studies the sample under test was located in a model C22 condensed gas cycling cryostat which could control the temperature between 10 and 300 K. Well polished pellets of samples are given silver metallization for the measurement. The dielectric constants of the composites are calculated from the sample geometry and their capacitance using the equation,

$$\varepsilon_c = \frac{Ct}{\varepsilon_0 A} \tag{1}$$

where ε_c is the dielectric constant of the sample, ε_0, is the absolute permittivity of vacuum (8.85×10^{-12} F m^{-1}) and "A" the area, and "t" the thickness of the sample. C is the measured value of the capacitance of the sample.[8]

RESULTS AND DISCUSSION

AFM studies of the composites

The morphology and particle size of the composites are carefully characterized by AFM. Figures 1 and 2 show the AFM images of the selected composites YBCO10 and YBCO40. These images reveal that microfillers are well dispersed and embedded uniformly through out the PS matrix. The calculated particle size of YBCO is of the order of 600 nm. The images reveal that the embedded particles have an average particle size of about 600 nm, and the distribution in grain sizes is ranged from 500 to 700 nm.

Dielectric constant and mixing laws

In the composite materials under study, the YBCO particles which are separated by polystyrene matrix, each pair of filler-polystyrene combination represent a capacitor. The dielectric constant of the prepared YBCO remained constant within the experimental temperature interval and its measured value is 24. Superconductivity of the YBCO phase was confirmed after firing using field exclusion technique. The dielectric constant of YBCO is greater than that of PS, so the addition of YBCO to polymer matrix will result in an increase in dielectric constant. At room temperature, as in Figure 3, the dielectric constant of the prepared composites increases with filler volume fractions. This behavior is attributed mainly due to the addition of YBCO, which introduces the molecular dipoles in the system. Since the values of "ε_f" and "ε_p," dielectric constant of filler, and matrix, are respectively, 24 and 2.55. It is also clear that dielectric mixing do not obey the law of physical mixtures, as shown by eq. (2) and Figure 3.

$$\varepsilon_c = \varepsilon_f v_1 + \varepsilon_p (1 - v_1) \tag{2}$$

where v_1 is the volume fraction of filler.

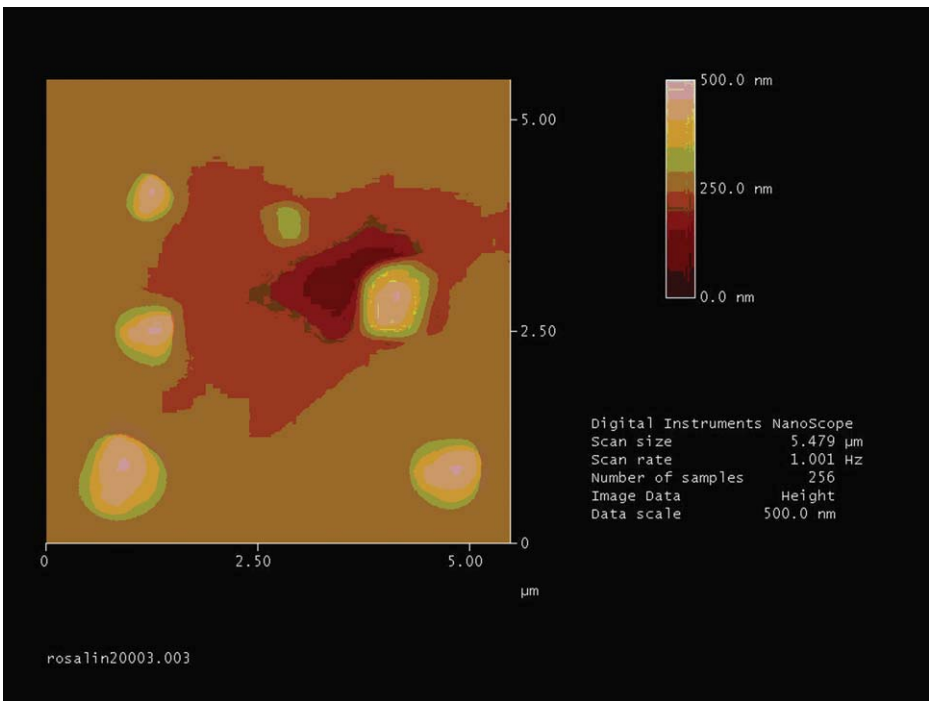

Figure 1 AFM image of YBCO10. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The present system of composites is a diphase mixture of two dielectrically different materials where YBCO is ionic and polycrystalline and polystyrene is amorphous and atactic. A great variety of formulae has been suggested for the calculation of permittivity of heterogeneous mixtures.[9,10] They were derived on the basis of various theoretical assumptions and experimental data.

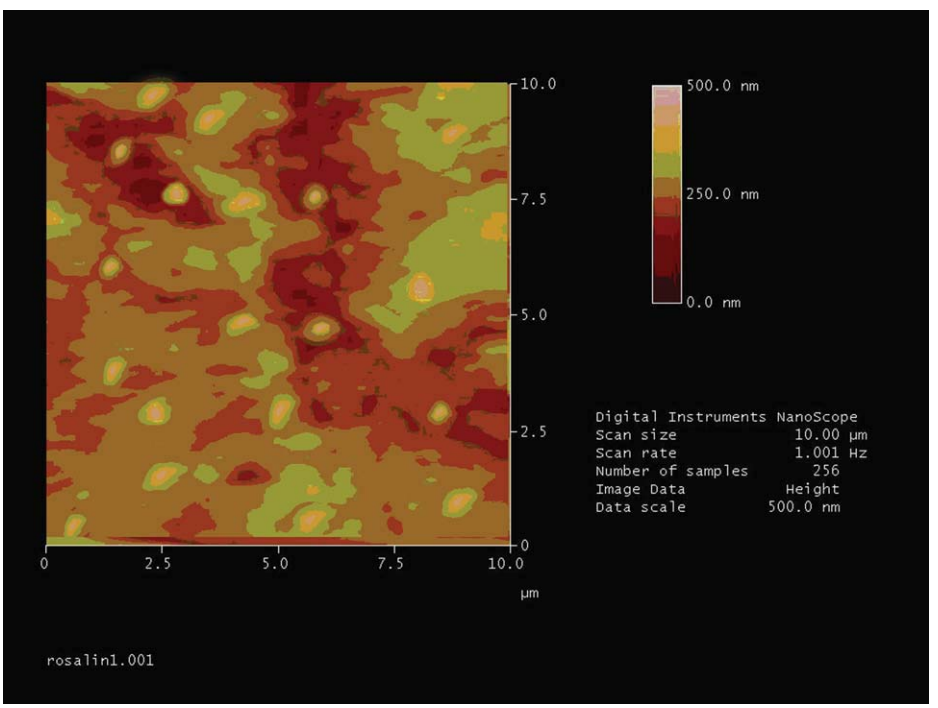

Figure 2 AFM image of YBCO 40 (low magnification). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

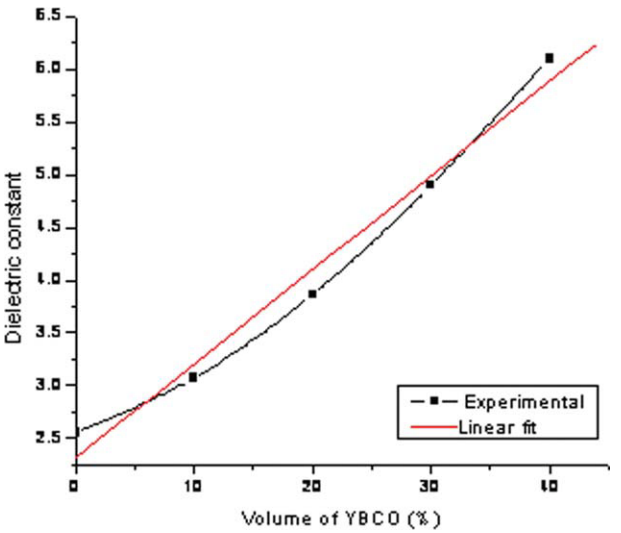

Figure 3 Dielectric constant versus volume percentage of filler for YBCO-PS composites at 1 MHz. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Dielectric mixture rules

Mixtures of ideal dielectrics can be most simply considered on the basis of layer materials with the layers either parallel or normal to the applied field. When the layers are placed normal to the applied field, the capacitance are additive. When layers are parallel to the applied field, the structure corresponds to capacitive elements in series, and the inverse capacitance are additive. It is found from Figure 3 that ε_c, the dielectric constants of the composites are nonlinearly dependent on vol % of YBCO.

Suppose we have a particulate two phase material we could use the model of composites to predict upper and lower bounds of the composite capacitances. One can choose to model composites as having capacitance in parallel (upper bound) or in series (lower bound). In practice the answer will lie somewhere between the two. Upper bound and lower bound equations are the special cases of a general empirical relationship

$$\varepsilon_c^n = \sum_i v_i \varepsilon_i^n \tag{3}$$

where "n" is a constant (n is (1) for upper bound and n is (-1) for lower bound) and "v_i" is the volume fraction of phase "i." As "n" approaches zero, we have

$$\log \varepsilon_c = \sum_i v_i \log \varepsilon_i. \tag{4}$$

This is the so called simple logarithmic rule of dielectric mixing. This gives a value intermediate between the extremes considered.

Figure 4 illustrates the dielectric interaction pattern of the mixed system with respect to composition.

The logarithm of the dielectric constant and $[(\varepsilon_c - 1)/(\varepsilon_c + 2)]$, the specific polarization of YBCO-PS composites are linearly proportional to the vol % of YBCO at all compositions as in Figure 4. Hence it is assumed that the composites follow the "log law" relationship, originally proposed by Lichtenecker[11] in which the dielectric constant of clean two component system can be represented by [eq. (5)], where v_1, is the vol % of filler. The logarithmic law of mixtures [eq. (5)] firmly confirms a logarithmic dependence of the dielectric constant of the composite on the volume fraction of the filler.

$$\log \varepsilon_c = \log \varepsilon_p + v_1 \log\left(\frac{\varepsilon_f}{\varepsilon_p}\right) \tag{5}$$

The plot of specific polarization $[(\varepsilon_c - 1)/(\varepsilon_c + 2)]$, where ε_c, is the dielectric constant of the composite, versus vol % of filler as presented in Figure 4 is also linear with slope and intercepts of 0.77 and 0.333, respectively, are as expected in accordance with Clausius-Mossotti equation modified by Lorentz and Lorentz[12,13] applicable to the overall composite dielectrics. It implies that not only the square of the dipole moment per unit particle of the combination but also the polarizability of a unit particle directly decrease proportionally to the increase of the quantity of the polystyrene. The Clausius-Mossotti equation itself does not consider any interaction between filler and matrix. This approach has been extensively used for studying the properties of two-component mixtures in which both the host and the inclusions possess different dielectric properties. In recent

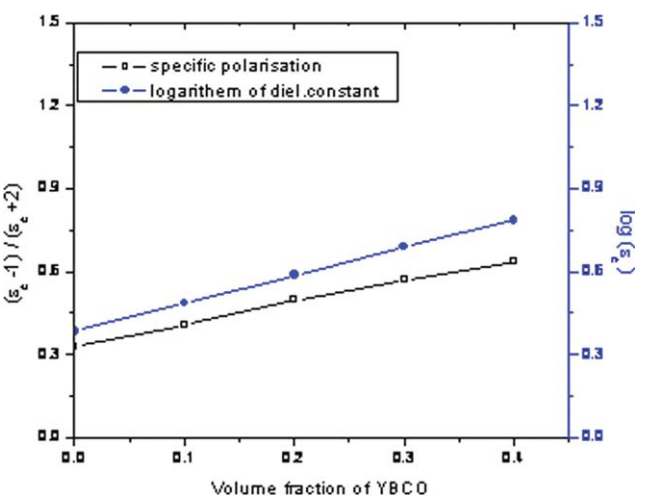

Figure 4 Specific polarization and Logarithm of dielectric constant versus filler volume fraction of YBCO-PS composites 1 MHz. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

years, this approximation has been extensively applied to composites involving high-temperature superconductors. This discussion may be further extended to calculate dipole moments of YBCO particles.

Calculation of dipole moment of Ybco particle

The Clausius-Mossotti[14,15] equation for a single-component system can be written as

$$\frac{\varepsilon_c - 1}{\varepsilon_c + 2} = \frac{N}{3\varepsilon_0}\left(\alpha + \frac{(\bar{\mu})^2}{3K_BT}\right) \tag{6}$$

where "N" is the number of molecules per unit volume, "α" is the deformational polarizability (both electronic and ionic polarization.), $(\bar{\mu})^2/(3K_BT)$ is the dipole polarizability, $(\bar{\mu})$ is the dipole moment, K_B is the Boltzman's constant, and T is absolute temperature.

Equation (6) is an appropriate one and becomes precise when polar molecules are separated from each other, i.e., when polar molecules are distributed in a plastic environment.

The present system more or less conforms to this situation, where ionic YBCO particles are distributed in a polystyrene matrix. For such a system μ is the average dipole moment of YBCO particles in the polystyrene matrix. Substituting the values as $N = (d_1/M_1)\, N_A\, v_1$ where density of YBCO $d_1 = 5200$ kg m^{-3}, molecular weight of YBCO is $M_1 = 0.66625$ kg, N_a, Avogadro number, and v_1 is the volume fraction of YBCO [eq. (6)] changes to

$$\frac{\varepsilon_c - 1}{\varepsilon_c + 2} = \frac{N}{3\varepsilon_0}\left(\alpha + \frac{(\bar{\mu})^2}{3K_BT}\right) \rightarrow \frac{N}{3\varepsilon_0}\left(\alpha + \frac{N_A d_1 \overline{(\mu)}^{-2}}{9\varepsilon_0 K_B T M_1} * v_1\right) \tag{7}$$

The L H S of eq. (7) is called specific polarization and the plot of specific polarization versus v_1, volume fraction of filler is linear and its slope and intercepts are proportional to both dipole moment and polarizability respectively, as in Figure 4, the slope is

$$\text{Slope} = \frac{N_A d_1\,(\bar{\mu})^2}{9K_BTM_1\varepsilon_0} \tag{8}$$

$$\mu = \left(\frac{N_A d_1}{9K_BTM_1\varepsilon_0}\right)^{-1/2} * (\text{Slope})^{1/2} \tag{9}$$

Now K_B is Boltzman's constant, $T = 300$ K and the slope is 0.77. The calculated average dipole moment of YBCO in polystyrene matrix is 7.32×10^{-30} Cm.

From the value of intercept one can calculate the polarizability of composite and the value we got is 0.29×10^{-35} C^2 N^{-1} m^{-1}. This dipole moment of YBCO particles and the induced polarization of YBCO in polystyrene matrix under electric field contribute for the dielectric constant of YBCO-PS.

Modeling

The two phase mixtures are also represented by the Bottcher-Bruggeman[16] formula based on the spherical particle model. Here the filler is interacting with matrix. According to this formulae

$$\varepsilon_c = \frac{1}{4}\left(H + (H^2 + 8\varepsilon_f\varepsilon_p)^{1/2}\right) \tag{10}$$

where

$$H = (3v_1 - 1)\varepsilon_f + (2 - 3v_1)\varepsilon_p$$

with $\varepsilon_f = 24$, dielectric constant of YBCO and $\varepsilon_p = 2.55$, dielectric constant of polystyrene. The values of the dielectric constant of the composite may be calculated from this equation and plotted against v_1, the filler fraction. However, the Maxwell-Wagner-Sillars equation predicts as a complete solution of the Wagner-Raleigh theory for a system of one spherical particle uniformly distributed in another.[17]

$$\varepsilon_c = \varepsilon_p \frac{2\varepsilon_p + \varepsilon_f + 2v_1(\varepsilon_f - \varepsilon_p)}{2\varepsilon_p + \varepsilon_f - v_1(\varepsilon_f - \varepsilon_p)} \tag{11}$$

If we consider the dispersion of spherical particles of dielectric constant much greater than that of matrix, Maxwell has derived a relationship.

$$\varepsilon_c = \frac{v_m\varepsilon_p\left[\frac{2}{3} + \frac{\varepsilon_f}{3\varepsilon_p}\right] + v_f\varepsilon_f}{v_m\left[\frac{2}{3} + \frac{\varepsilon_f}{3\varepsilon_p}\right] + v_f} \tag{12}$$

The experimentally obtained plot of (ε_c) versus filler volume fraction lie in between the three plots (Fig. 5) and almost coinciding with Logarithmic laws.

In the present system it is clear that the shape, particle size distribution, and concentration of the dispersed component do not permit a very high degree of chemical interaction as envisaged by Bottcher-Bruggeman.

The composite under investigation consists of ionic YBCO dispersed in polystyrene medium. Hence it is likely that the magnitudes of both the short range and the long range interactions possible between the ions in the filled matrix are lessened by the imposition of a plastic environment on the ionic

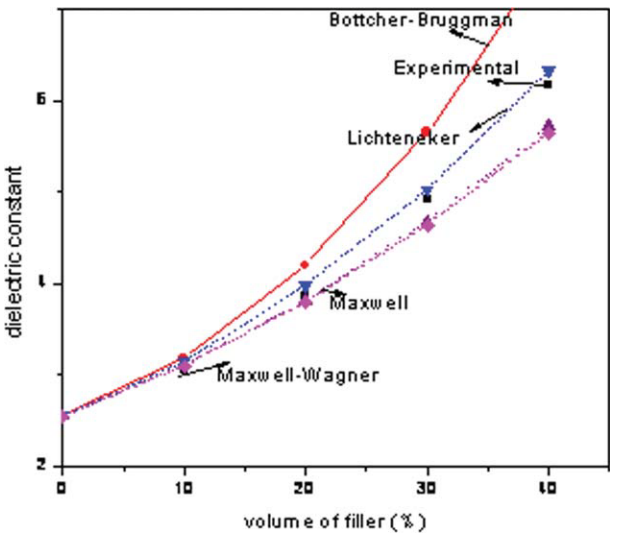

Figure 5 Modeling of the dielectric constant of YBCO-PS composites at 1 MHz. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

YBCO. The best reproduction of dielectric constant was obtained by the use of "Lichteneker relations".

Effect of temperature on dielectric properties of polymer and the composites

The dielectric constant of the composites increases with increase in temperature (Fig. 6). It is essentially due to the different thermal expansion of the polymer (50×10^{-6} K^{-1} to 300×10^{-6} K^{-1}) on one hand and the ceramics (0.5×10^{-6} K^{-1} to 15×10^{-6} K^{-1}) on the other.[18] The increase in dielectric constant is attributed to the higher orientation polarization of the polymer at higher temperature due to the greater mobility of molecules. It is observed that the rate of variation of dielectric constant with temperature is steeper for higher volume fraction samples. This is attributed to the internal field generated by the YBCO particles, which favors the orientation of polymer molecules. For YBCO, the electronic and ionic polarizabilities are practically independent of temperature for normal temperatures where as orientation polarization and space charge polarization are affected by temperature. In orientation polarization, the randomizing action of thermal energy decreases the tendency of the permanent dipoles to align themselves in the applied field. This results in a decrease in dielectric constant with temperature at and above 100°C, that is the glass transition temperature of polystyrene[19,20]

Impedance measurements

For an ideal dielectric the AC current leads the EMF by exactly 90° and the energy loss due to joule heating effect is zero i.e., the vector product "$I \times V$" is zero for a phase difference of 90° between I and V. In such cases energy is transmitted through the sample without dielectric losses. At higher temperature and high frequencies, a stage is reached where the polarization can no longer keep up with the alternating EMF. Consequently current leads the voltage by less than 90°, i.e., by an angle (90- δ), that is called "θ" the phase angle. The current has a component (I sinδ) that is in phase with the voltage as in Figure 7. This give rise to dissipation of energy as heat and it is regarded as the dielectric losses of the composites.[21]

The impedance spectra obtained for the composites are processed through computer assisted electrochemical data analysis software that ideally fit to the experimental data and it is found that the dielectric combination looks like a parallel combination of capacitance resistance circuit. We prefer [eq. (13)] for the suitable explanation of the impedance value "Z".

$$Z = \frac{RX_c}{(R^2 + X_c^2)^{1/2}} \tag{13}$$

where $(R^2 + X_c^2)^{1/2}$ is the vector addition of the resistance and capacitive reactance. The impedance of a parallel RC circuit is always less than the resistance or capacitive reactance of the individual branches. The relative values of "X_c" and "R" determine how capacitive or resistive the circuit line current is. The one that is the smallest and therefore allows more branch current to flow is the determining factor. Thus if "X_c" is smaller than "R," the current in the capacitive branch is larger than current in

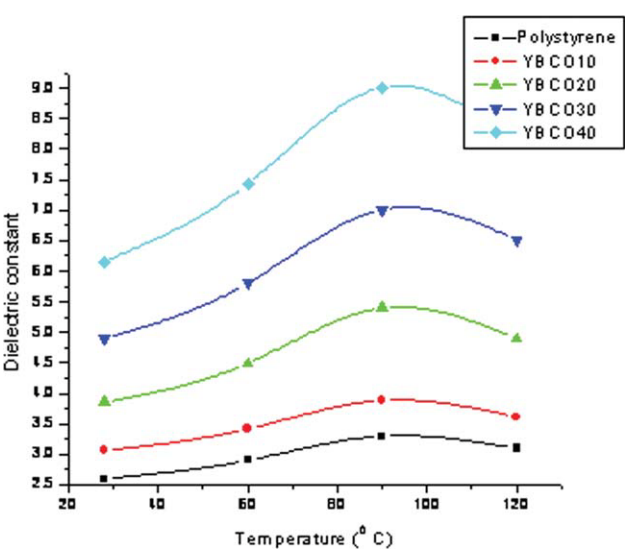

Figure 6 Dielectric constant versus temperature for YBCO-PS composites at 1 MHz. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

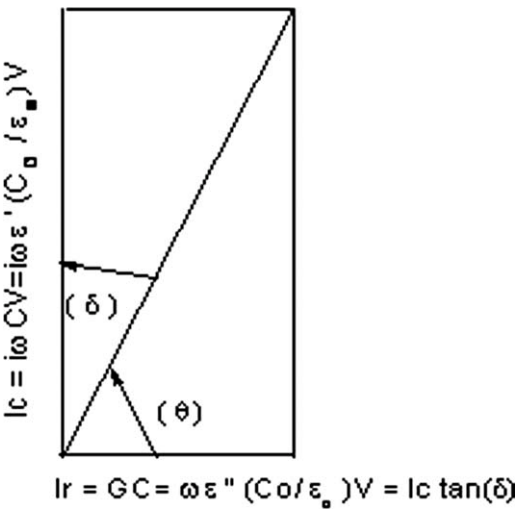

Figure 7 Charging and loss current for a capacitor.

the resistive branch, and the line current tends to be more capacitive (Fig. 7).

The impedance measurement of the composites revealed that when the volume fraction increased from 10 to 40%, the composites remained in the capacitive characteristic only. The logarithm of impedance Z versus frequency curves were parallel curved lines and the phase angle θ about 90° which were like ideal capacitors (Fig. 8)

$$Z \leq \frac{1}{2\pi\, jfC} \tag{14}$$

The composites are highly insulating between 10 and 40 vol %. fraction because the particles are completely separated from each other by the polymer. Frequency affects the value of the capacitive reactance and so also affects the circuit impedance, line current, and phase angle, since they are determined to some extent by the value of "X_c." The higher the frequency of a parallel RC circuit, the lower is the value of "X_c." This means that for a given value of R, the impedance is also lower, making the line current larger and more capacitive.[22,23]

Below the transition temperature of the superconductor YBCO (91K), the conduction electrons divide into two classes, some behaving as super electrons which can pass through the material without resistance (suffering no collision), the remainder behaving as normal electrons which can be scattered and so experience just like conduction electrons in a normal metal. The fraction of super electrons appears to decrease as the temperature is raised toward transition temperature and on further heating the proportion of normal electrons increases. Eventually above superconducting transition temperature, all electrons have become normal electrons and the composite material losses its superconducting properties.[24] With model of a parallel resistor and capacitor, the temperature and frequency dependence of the phase angles can be easily explained. The phase angle "θ" is proportional to capacitive current/resistive current

$$\text{i.e., } \theta = \tan^{-1}\left(\frac{B}{G}\right) = \tan^{-1}(2\pi fCR) \tag{15}$$

where $G = 1/R$ is the conductance and $B = 2\pi$fC is the susceptance. Therefore the frequency "f" increases, the phase angle increases, and "Z" decreases as in Figure 8.

The AC conductivity (σ) of the prepared composites at room temperature is calculated using the formulae

$$\sigma = 2\pi f \varepsilon_0 \varepsilon_r \tan(\delta) \tag{16}$$

where "ε_0" is the dielectric constant of vacuum, ε_r the relative dielectric constant of the composites, and tan(δ)[25] is the dielectric loss factor. For polycrystalline materials and composites the overall sample resistance may be a combination of the intragranular resistance, or bulk composite resistance and the intergranular or grain boundary resistance. Grain boundary resistances have an associated capacitance in parallel with whose magnitude depends upon the filler volume fraction of the composites. If we have two layers of material of different conductivity, motion of charge carriers occurs readily through one phase but is interrupted when it reaches a phase boundary. This causes a build up of charges at the interfaces which to an outside observer, corresponds

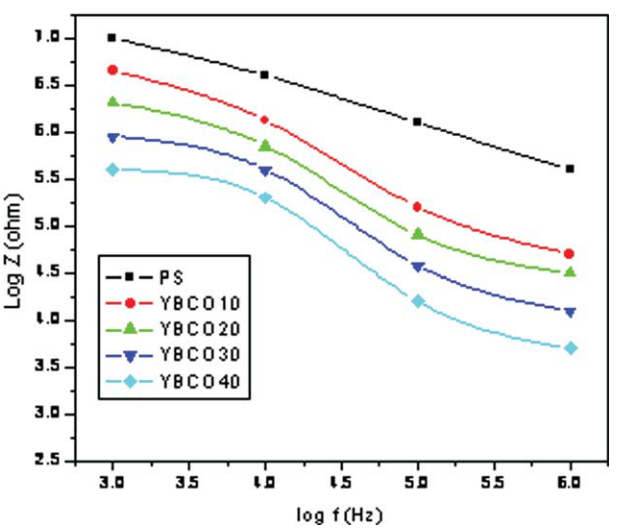

Figure 8 Log Z versus log f plot for PS and composites at room temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

to a large polarization and high dielectric constant.[26] This process is schematically expressed in Figure 9.

Low temperature studies

The low temperature studies of the prepared composites are done and the results are graphically shown in Figure 10. The temperature dependence of the phase angles for various composites is studied at low and high temperature, between 80 and 400 K. We wanted to observe whether any sudden change occurred around the superconducting transition temperature of YBCO, which was found to be around 91 K by susceptibility measurement of YBCO. It is noted that even high loaded composite YBCO40 showed no transition in the phase angle when the samples went through superconducting transition temperature of YBCO. This suggests that the resistive coupling between the superconductor particles, which is the dominant factor for the high loaded composites, is not strong enough to produce super electrons and super current. Because of both the very thin dielectric layers between these particles and the large surface contact resistance of the particles, the composites have a conductive behavior with temperature.[27–29]

The interpretation of the impedance spectra and their electrical characteristics support the statement that the composites with low filler concentration are essentially capacitive, although a small conductivity is observed with increasing filler concentration. This conductivity may be attributable by the tunnel effect, that allow the electrons to flow from one conductor particle to the next through the polymer film sandwiches between the particles, thus establishing an electric current. When temperature decreases, the effective resistance of the composites, R, increases, phase angle θ, changes. No noticeable sudden change was noted for either the impedance magnitude (not shown graphically) or the phase angle during the temperature changes from 400 to 80 K.

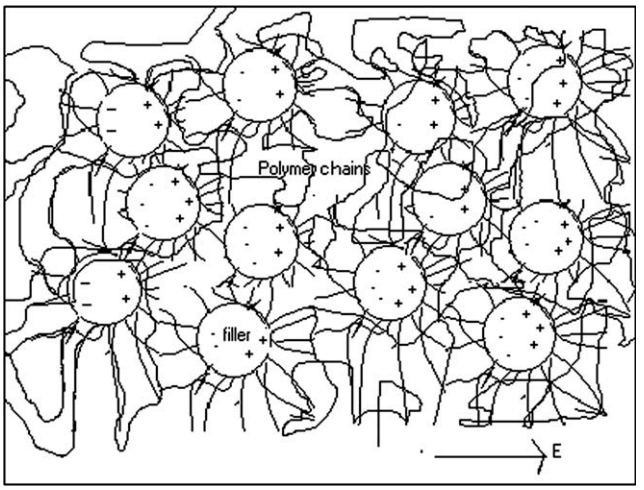

Figure 9 The schematic representation of YBCO-PS at room temperature.

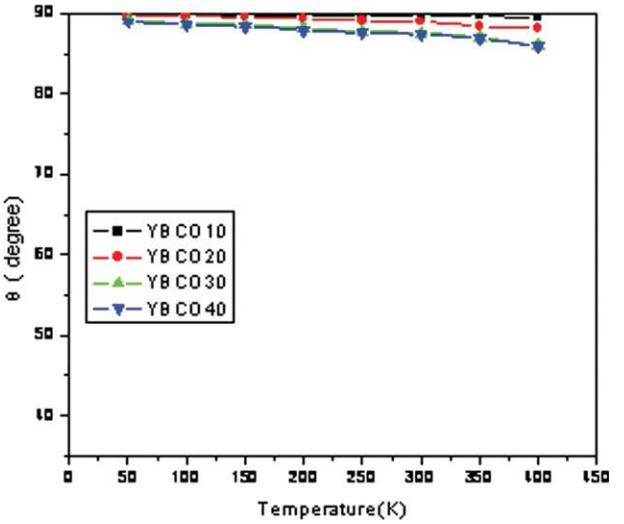

Figure 10 Phase angle versus temperature of the YBCO-PS composites at 1 MHz. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSIONS

Dielectric mixing laws are examined for the composites consisting of superconducting ceramic particles dispersed in the polystyrene matrix. The dielectric constant of the composites follows a logarithmic mixing rule at all the studied filler volume fractions. The dielectric constant variations with ceramic content are consistent with Lichteneker relations. The Clausius-Mossotti approximation is used for calculating the polarizability and dipole moment of the composites and the filler particles. The slope and intercept of the relationships depend on the dielectric characteristics of the composite constituents. For a particular composite system, these parameters also depend on the measurement frequency. The frequency dependence of the composite impedance revealed that the composites posses the characteristics of ideal capacitors.

There were no marked sudden transition in impedance and phase angle when the composites went through the superconducting transition temperature of YBCO.

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