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Article *in* Materials Physics and Mechanics · July 2022

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Materials Physics and Mechanics. 2022;48(3): 328-341
Research article
http://dx.doi.org/10.18149/MPM.4832022_4

Submitted: February 3, 2022
Approved: April 20, 2022
Accepted: April 25, 2022

A study on mechanical and dielectric properties of B_4C and Al dispersed single-layered epoxy-based polymer composites fabricated through molding and curing route

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Abstract. The work aims in developing epoxy composites with different compositions of boron carbide (5, 10, 15, and 20 wt.%) added with aluminium (10 wt %) for studying the mechanical and dielectric behavior. It was found that the tensile strength of the samples initially increased with an increase in reinforcements and then decreased with an increase in B_4C content beyond 10 wt. %. Flexural strength, on the other hand, increased with the addition of B_4C particles. Moreover, the impact strength of the samples decreased with an increase in the addition of the B_4C particles. The dielectric properties were studied by considering various factors like temperature and frequency. The samples were subjected to frequencies 100Hz, 1kHz, 10kHz, 100kHz, and 1MHz, and temperatures ranged from 40°C to 150°C. As a result, it was found that the dielectric loss factor was increased with increasing temperature. Further, at higher frequencies, an increase in the dielectric constant and a decrease in the dielectric loss factor were evidenced which concluded the material's suitability for energy storage applications.

Keywords: conducting polymers, dielectric properties, mechanical properties, molding, particle dispersed

Acknowledgements. *The author acknowledges the support rendered by King Mongkut's University of Technology North Bangkok, Bangkok, Thailand and The S.T. Hindu College, Nagercoil, for providing necessary research support.*

Citation: Vignesh S., Winowlin Jappes J.T., Nagaveena S., Sankaranarayanan K., Krishna Sharma R., Brintha N.C. A study on mechanical and dielectric properties of B_4C and Al dispersed single-layered epoxy-based polymer composites fabricated through molding and curing route. *Materials Physics and Mechanics*. 2022;48(3): 328-341. DOI: 10.18149/MPM.4832022_4.

1. Introduction

Increasing demand in the field of industrial packaging and microelectronics particularly in energy storage has led to the quest in searching for advanced novel materials. In industrial packaging, the materials are expected to be mechanically excellent in their characteristics, and in electronics, the need is for electrically conductive polymeric materials with a conductive core and an insulating shell. Several kinds of research are underway in particulate reinforced polymer-based composites in which the primary phase is the polymer matrix whereas, the secondary phase possessing a good dielectric constant is made to disperse in the primary phase through conventional fabrication routes. Researchers have revealed the possibility of consuming these polymers possessing electrical conductivity with the aid of their secondary phases but also not trading off the weight of the material [1-2]. Epoxy, is a polymeric hinderer of the flow of electrons owing to the inherent low dielectric constant but mechanically possesses wide characteristics [3-5]. However, scientists revealed the ability to increase the dielectric constant of the polymer through the mode of particulate addition utmost to 50 vol.% [6]. On the other hand, reports revealed the detriment of mechanical properties owing to the augmented addition of the fillers [7-9]. Works suggest the addition of particles improves the dielectric properties through the following two notions such as (i) establishing contact between particles, and (ii) making the electron move across a hindrance, widely called electron tunneling. However the former and the latter mechanism can be made possible by generating a 3D-conductive network facilitated by the closeness of the filler particles and transportation of electrons amidst closest particles through a thin layer hindrance by polymer [10].

Here the subject of concern is not restricted to conductivity but also the mechanical property of the material being produced. Hence the proper choice of dispersing reinforcement should be taken. Particularly Boron carbide (B_4C) based hard materials are seeking attention in the field of structural, tribological-interactive applications [11-20]. Moreover, its excellent hardness and density make it suitable for ballistic applications as well. Anyhow, B_4C founds less use owing to its increased brittleness [21-23].

Also, aluminum (Al), possessing very less weight, excellent electrical conductivity, and good thermal conductivity can be used in electrical and electronic applications as it has the ability to withstand heat resulting from high operating voltage. The electrical and mechanical characteristics of aluminium along with epoxy were studied and revealed the yield in dielectric properties with respect to that of filler concentrations [24]. Nevertheless, the works are almost void in the case of augmentation of B_4C and aluminium for the dielectric property through the molding and curing route. Hence in this work, an attempt is made to study the mechanical and electrical properties of the B_4C and Al dispersed epoxy-based single-layered polymer composite of varying compositions of B_4C and Al.

2. Materials and Methods

Materials. The work was carried out using commercially available B_4C (10µm), Al powder (10µm), epoxy LY566, and its corresponding hardener. The procured metallic powders were subjected to a scanning electron microscope equipped with an electro dispersive spectrum analyzer using JEOL-JSM-5600LV scanning electron microscope equipped with EDS under 20kV and exposure time of 50s and are depicted in Fig. 1 and Fig. 2. The matrix and particle reinforcement were made with different compositions tabulated as shown in Table 1. The different samples were fabricated through the molding and curing method which is depicted in Fig. 3.

Before the fabrication of the composite material, the epoxy matrix and the reinforcement particles were mixed mechanically and were subjected to the ultrasonic dispersion technique for 45 minutes. The initial mixing was then followed by the addition of

hardener to the mixture and was further stirred mechanically for 15 minutes at a uniform speed of 200rpm. The epoxy matrix and the hardener used were mixed in a ratio of 2:1. Subsequently, the liquid mixture of matrix and dispersed particles was poured into a glass mold of 300mm × 130mm × 5mm. Further, the samples from the composite were cut as per the ASTM standard and were subjected to mechanical, electrical, and fractography studies and the result are discussed in the forthcoming chapters.

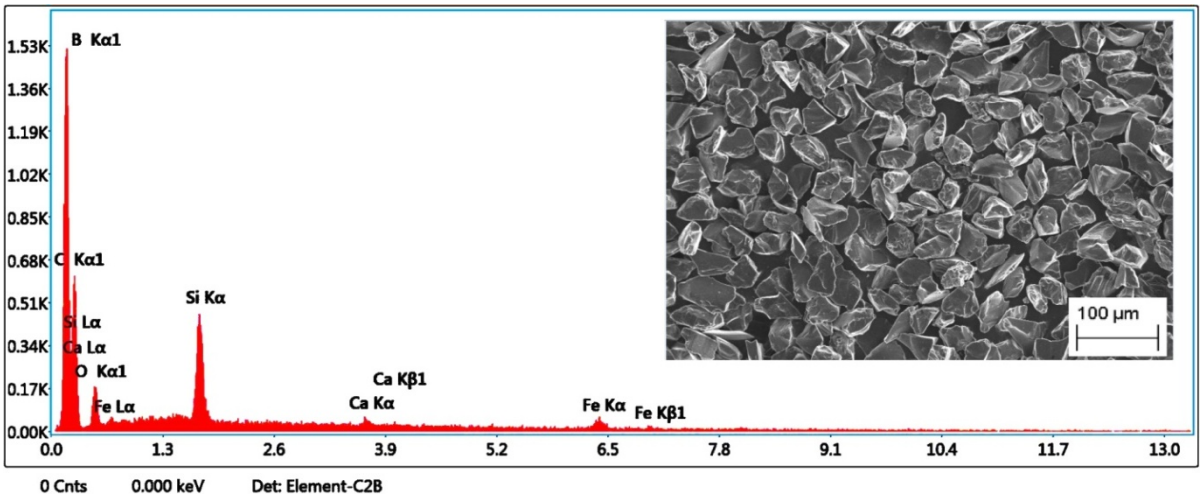

Fig. 1. SEM & EDS spectra depicting the particle size and composition of B_4C particles

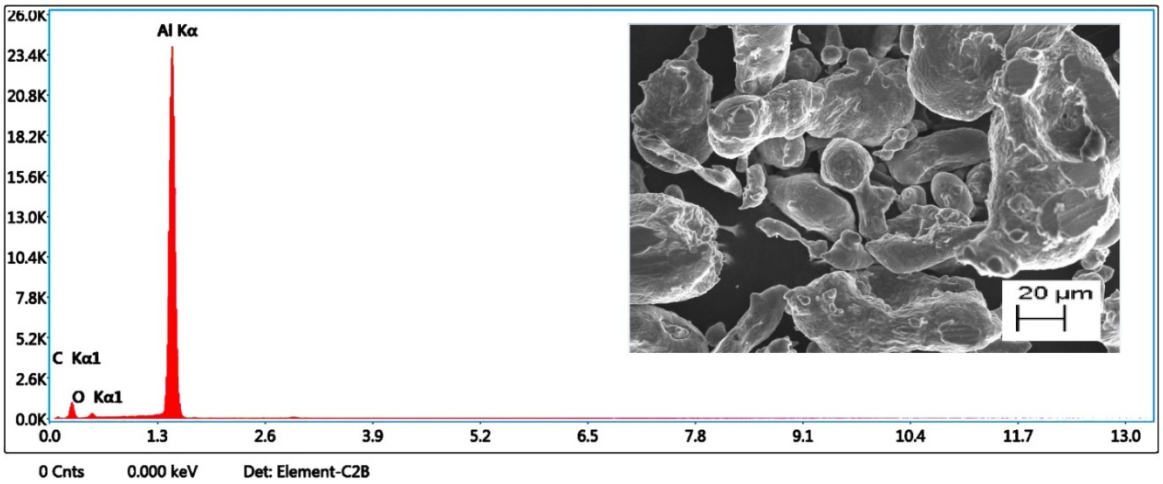

Fig.2. SEM & EDS spectra depicting the particle size and composition of Al particles

Table 1. Composition of the samples prepared

Samples	Composition (in wt %)				Weight (g)			
	B_4C	Al	Epoxy	Hardener	B_4C	Al	Epoxy	Hardener
A	5	10	57	28	6	12	120	60
B	10	10	54	26	12	12	120	60
C	15	10	50	25	18	12	120	60
D	20	10	47	23	24	12	120	60

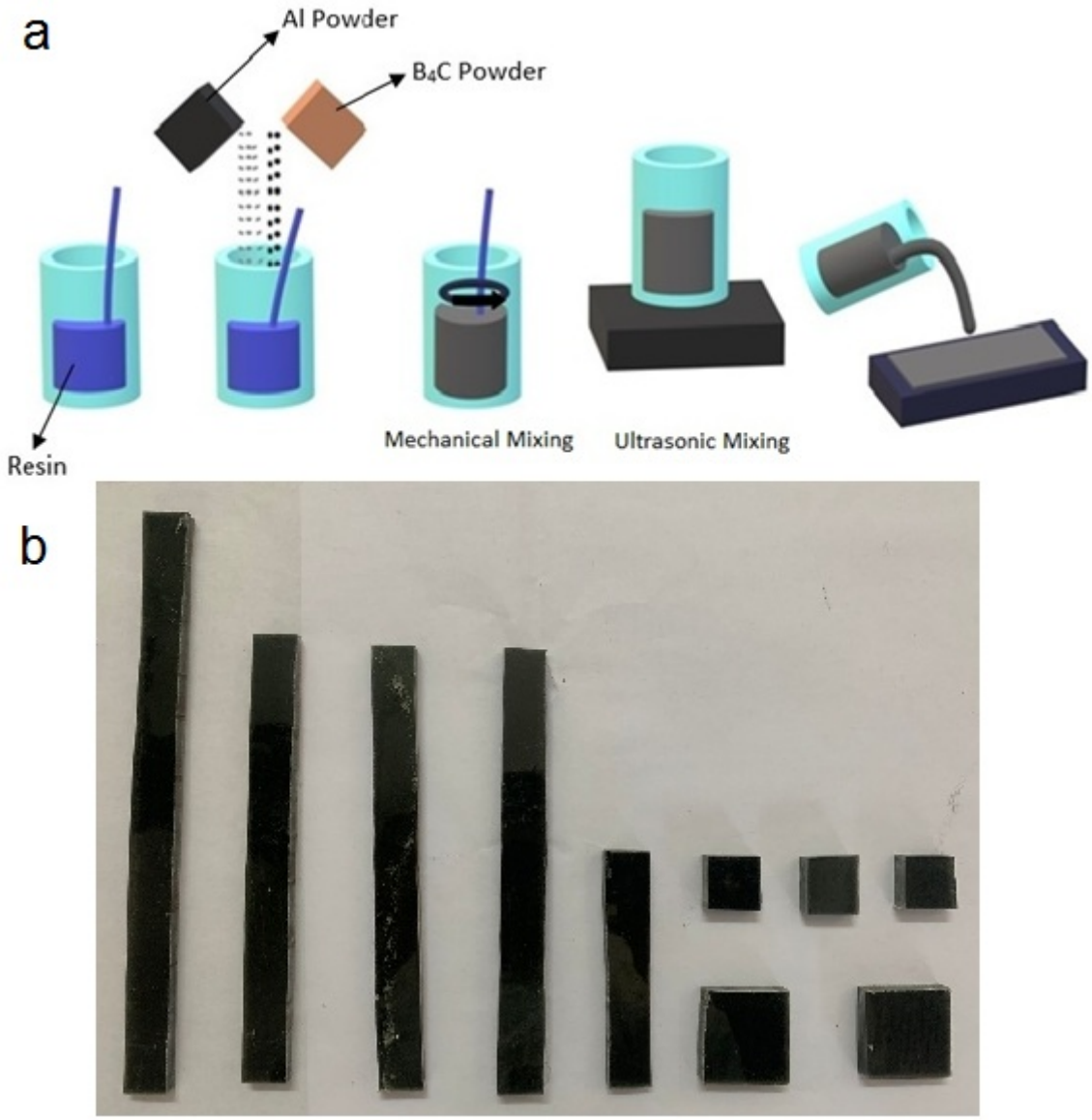

Fig. 3. a – composite fabrication method through molding and curing,
b – as prepared composites specimen

Mechanical Testing. The samples prepared were tested for their tensile strength flexural strength and impact strength (Izod-v notch samples) after carefully adhering to the ASTM D638, ASTM D790, and ASTM D256 standards respectively. The mechanical studies were carried out in 4 samples and the results are discussed in the forthcoming sections. The fractography of the tested samples was done by JEOL-JSM-5600LV scanning electron microscope under 15kV and exposure time of 50s. The fractured sections were carefully cut from the samples and were subjected to SEM in order to study the fractography which is depicted in Fig. 3b.

Electrical Characterization. The prepared samples were subjected to electrical characterization to study the capacitance C_p and dielectric loss factor tanδ at various temperatures ranging from 40°C to 150°C along with a range of frequencies from 100Hz, 1kHz, 10kHz, 100kHz, and 1MHz using an LCR Meter Aglient 4284A through parallel plate capacitor method. Annealing was done to the samples while holding the same in the sample holder at 160°C prior to observations. The readings were taken during the cooling process of the sample and it should be noted that the temperature was controlled with an accuracy of

±0.5°C. Air capacitance C_a was measured and using the relation $\varepsilon_r = C_p/C_a$ the dielectric constant of the samples was measured. It should be also noted that the AC conductivity of the samples was determined by the relation

$$\sigma_{AC} = \varepsilon_0 \varepsilon_r \omega \tan\delta,$$

where ε_0 is the permittivity of free space and ω is the angular frequency and is given by $\omega = 2\pi f$, f is the applied frequency.

3. Results &discussions

Tensile, flexural & impact properties. Figure 4 depicts the tensile and flexural characteristics of the samples prepared. The tensile strength of the sample clearly notifies that the addition of B_4C beyond 10 wt. % decrements the tensile characteristics of the samples whereas an increase in addition of B_4C from 5 wt. % to 10 wt. % increases the tensile strength. On the other hand, the ductility of the samples was reduced upon increased augmentation of B_4C. The increment in tensile strength was attributed due to the better dispersion of the particles in the sample. It is also possible to achieve the tensile characteristics, as the addition of aluminium could result in an improved modulus of tensile. However, the rest of the samples with the increased addition of B_4C resulted in reduced tensile properties which could be attributed due to the following causes: Primary contribution to the reduction could be the agglomeration of the reinforcement particles owing to the increased addition of the same [25] and the secondary contribution could be a decline in the surface area of contact between the particle and the matrix because of the agglomeration. The former reason was found to be there in the samples and is evident from the tensile fractography as in Fig. 5.

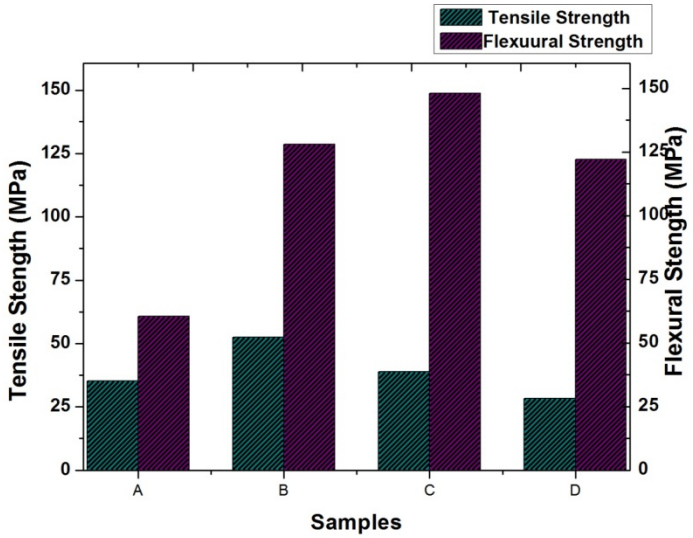

Fig. 4. Tensile & Flexural Strength of samples A, B, C & D

Moreover, it is also evident from Fig. 6 that some known pattern like chevron is significantly visible in the tested sample. It is obvious that the chevron-like pattern signifies the failure due to brittleness [26] as the presence of Al and B_4C in epoxy will have both ductility and brittleness which is inherent by their nature. It is also noted from the fractography that the sample exhibited forceful separation between epoxy and reinforcement particulates. Moreover, the sharp protruded shapes as evident from Fig. 7, that emerged upon the action of loading led to cracking and crack propagation as a result ended with brittle fracture even though with the presence of ductile aluminium particles [27].

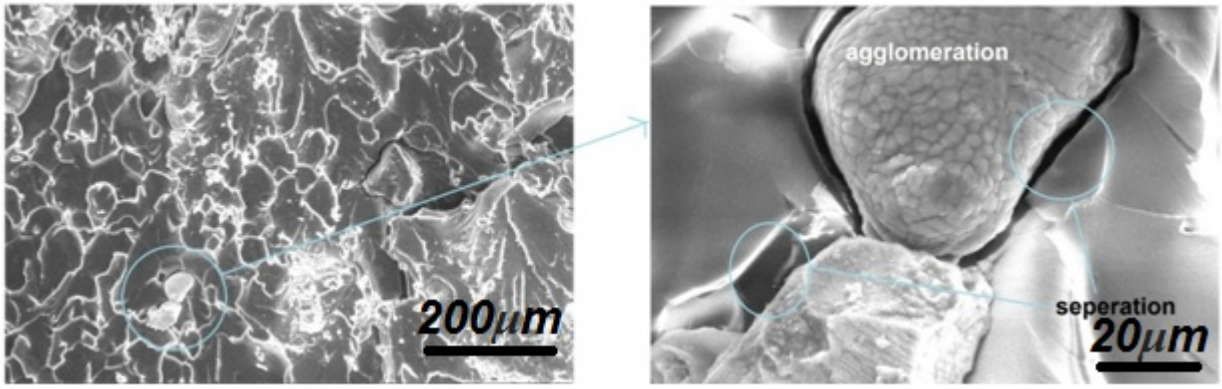

Fig. 5. SEM micrograph depicting agglomeration and separation of B_4C particles (after tensile testing)

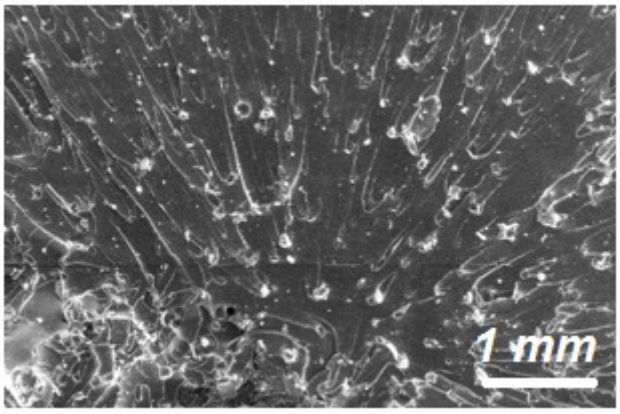

Fig. 6. SEM micrograph depicting Chevron-like patterns of B_4C particles

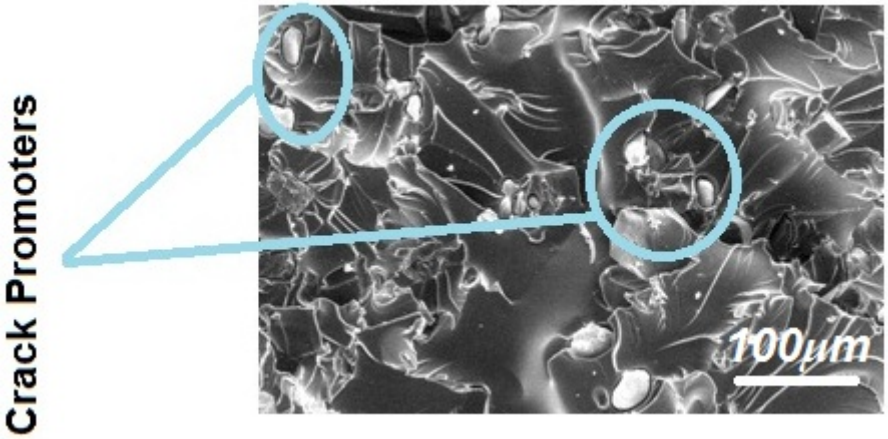

Fig. 7. SEM micrograph depicting sharp-edged crack promoters

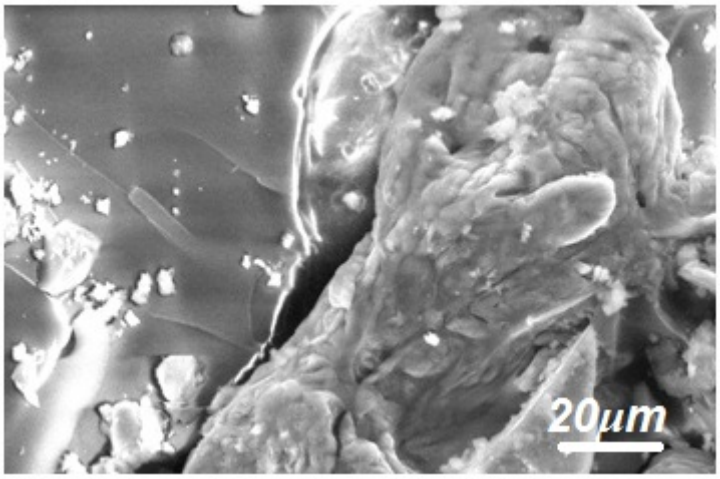

Fig. 8. SEM micrograph depicting the separation of B_4C & Al particles from the epoxy matrix

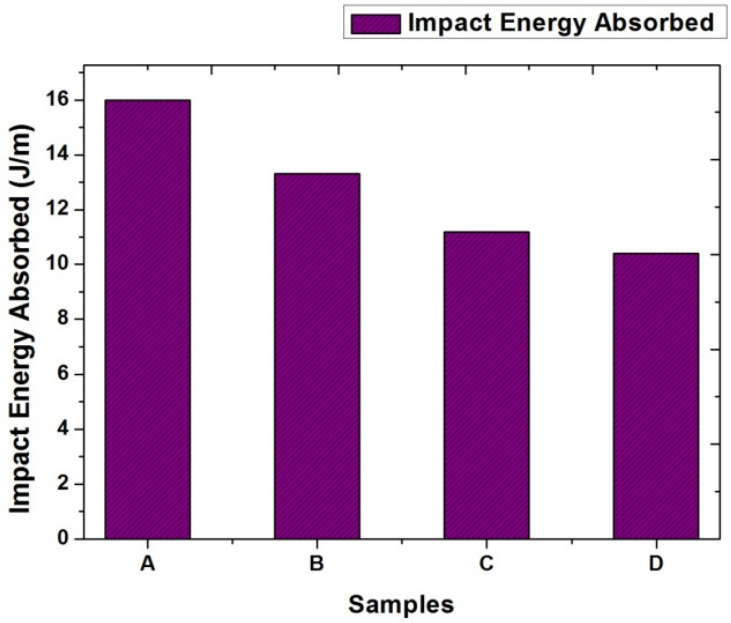

Fig. 9. Impact Energy Absorbed of different samples

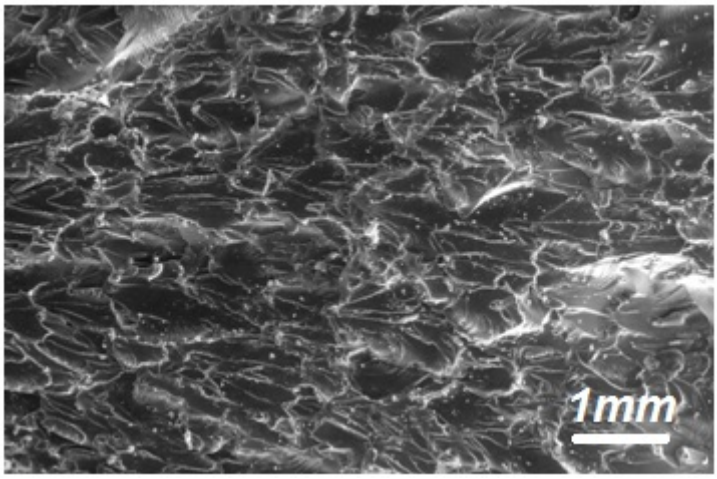

Fig. 10. SEM micrograph depicting mountain-like pattern causing brittle failure

The flexural strength of the specimen depicted in Fig. 4 clearly shows the increase in flexural strength with the increased addition of B_4C particles. The added particles, upon loading, during the test, resulted in the de-lamination of the reinforcements from the epoxy matrix as evident from the Fig. 8, and thus contributed to the reduced flexural strength of the sample A. Since, the sample A incorporated with the least amount of B_4C has resulted with the least dispersion and is more prone to separation at the zone where de-lamination take place. On the other hand, the impact strength of the samples decreased with an increase in the addition of the B_4C particles as in Fig. 9. It is because the increase in tensile strength results in the detrimental value of impact strength of the specimen. It is also evident from Fig. 10 that the fractography resembles mountain-like patterns tend to propagate cracks through the v-notch resulting in the brittle failure of the specimen.

Electrical Properties. The fabricated samples A to D were subjected to AC conductivity studies to measure the dielectric constant, dielectric loss factor, and electrical conductivity for five different frequencies at different temperatures, and the results are tabulated in Table 2. The parameters were tested with respect to their temperature dependency and are shown in Figs. 11-13. In general, the dielectric properties were found to increase with respect to that temperature. It was also observed from Figs. 11-12, the dielectric constant tends to increase with respect to frequency and the dielectric loss factor decreased with augmented frequency. However, on the other hand, the AC conductivity of the samples increased with an increase in frequency.

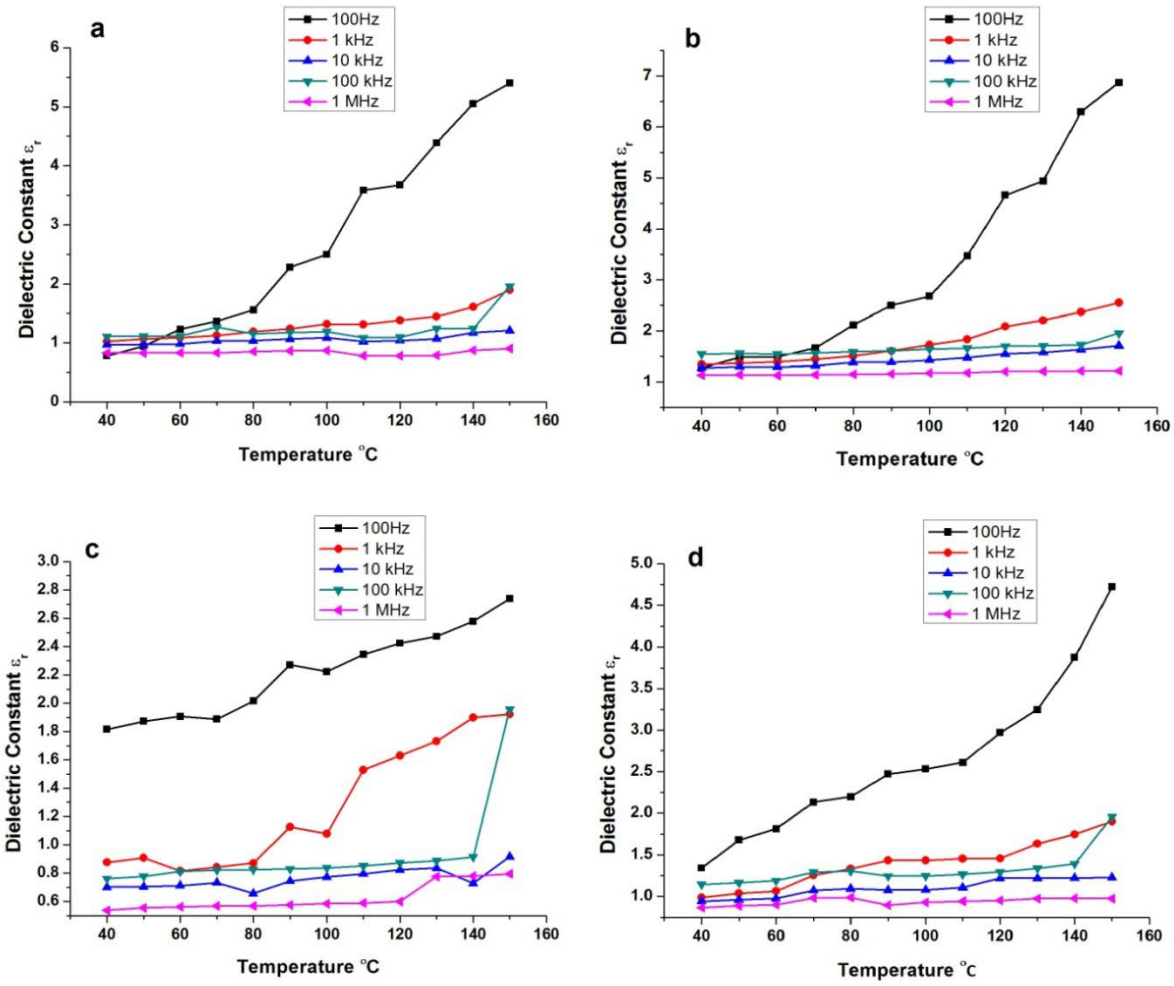

Fig. 11. Dielectric constants for samples viz., a) Sample A, b) Sample B, c) Sample C and d) Sample D

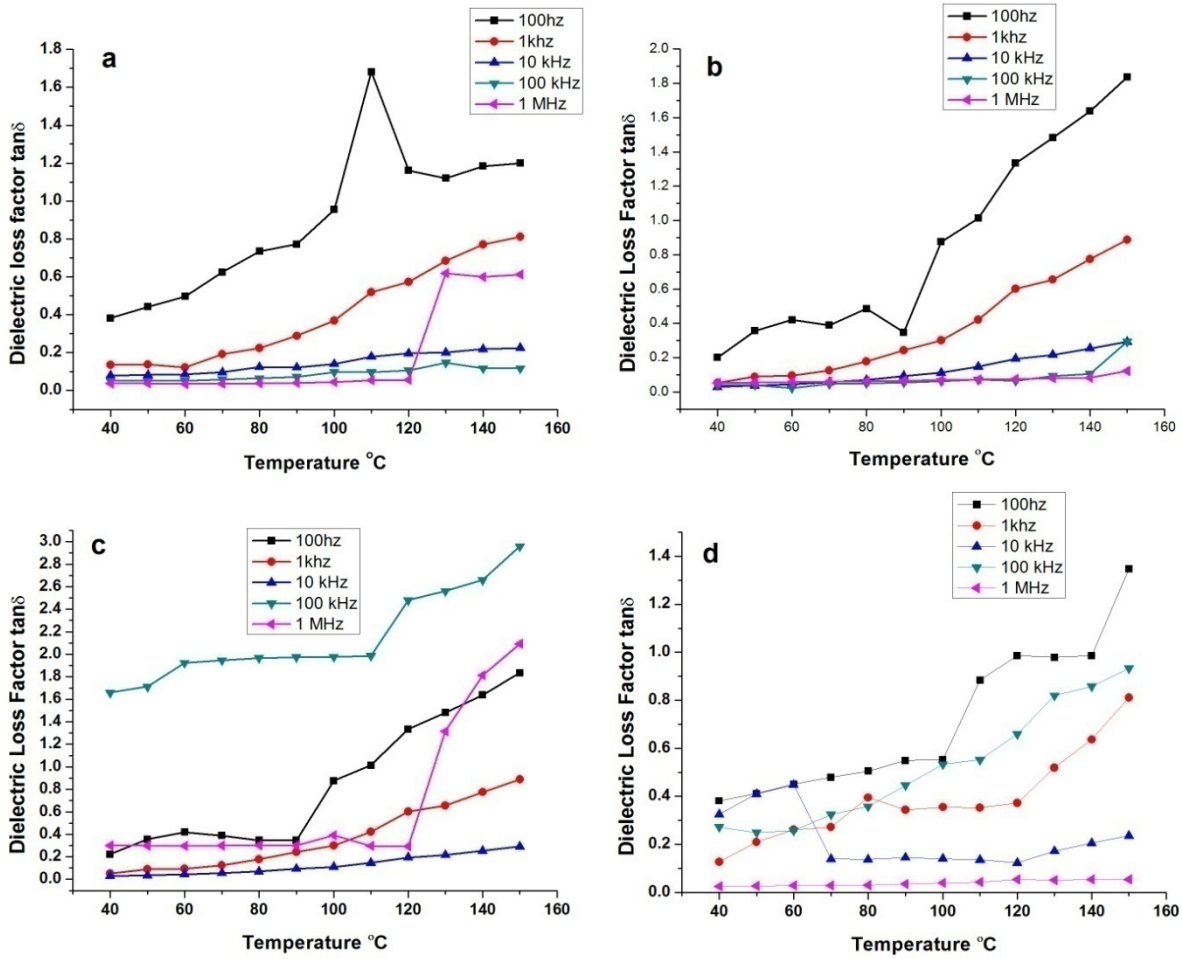

Fig. 12. Dielectric loss factor for samples viz., a) Sample A, b) Sample B, c) Sample C and d) Sample D

The presence of different varieties of polarizations in the materials, in general, contributes to the dielectric behavior of the same [28]. Moreover, it is well known that the dielectric constant of epoxy increases predominantly by reducing the free volume of the matrix and is accompanied by improving the polarization of the matrix [29-30]. However, in all the samples, the presence of reinforcement particles contributes to the reduction in the free volume of the epoxy matrix by occupying the volume owing to uniform dispersion. It should be also noted that the existence of these particles provides polarization change thus resulting in a high dielectric constant. Moreover, the higher dielectric constant at lower frequencies is owing to space charge polarization and rotational polarization as well [31]. It should be also noted that the orientation of dipoles would be enhanced upon the effect of temperature, tending to yield the dipole moment [32]. The increase in frequency will give rise to electronic polarization thereby reducing the dielectric constant values in some samples. Further, research suggests that an increase in the dielectric constant of the materials makes the material suitable for energy storage applications to a larger extent. Also, the dielectric characteristics of polymers greatly rely upon the mechanisms of polarization that includes electronic, atomic, dipolar interfacial, and ionic [39]. Moreover, the polarization due to orientation is a predominant factor in achieving better dielectric properties, particularly epoxy-based composites [40] and this could be the possible reason for the improvement of the dielectric characteristics of the sample.

It should be taken into account that dielectric loss factors, are a phenomenon of dissipating heat at the expense of electric energy. Insulating material will tend to have less dielectric loss [33-34]. However, the increase in dielectric constant and dielectric loss could not be explained as a dependence function of filler content concentration. In general, particularly in the case of insulator-conductor composites, it is obvious that tunneling of electrons from a bunch to another bunch would be possible when separated by polymer layers having less number of conductive particles [35-36]. In the case of insulator-conductor composites, it is obvious that the tunneling of electrons is the underlying reason for the AC conductivity performance, due to which the conductivity of the samples increased with an increase in frequency [37-38].

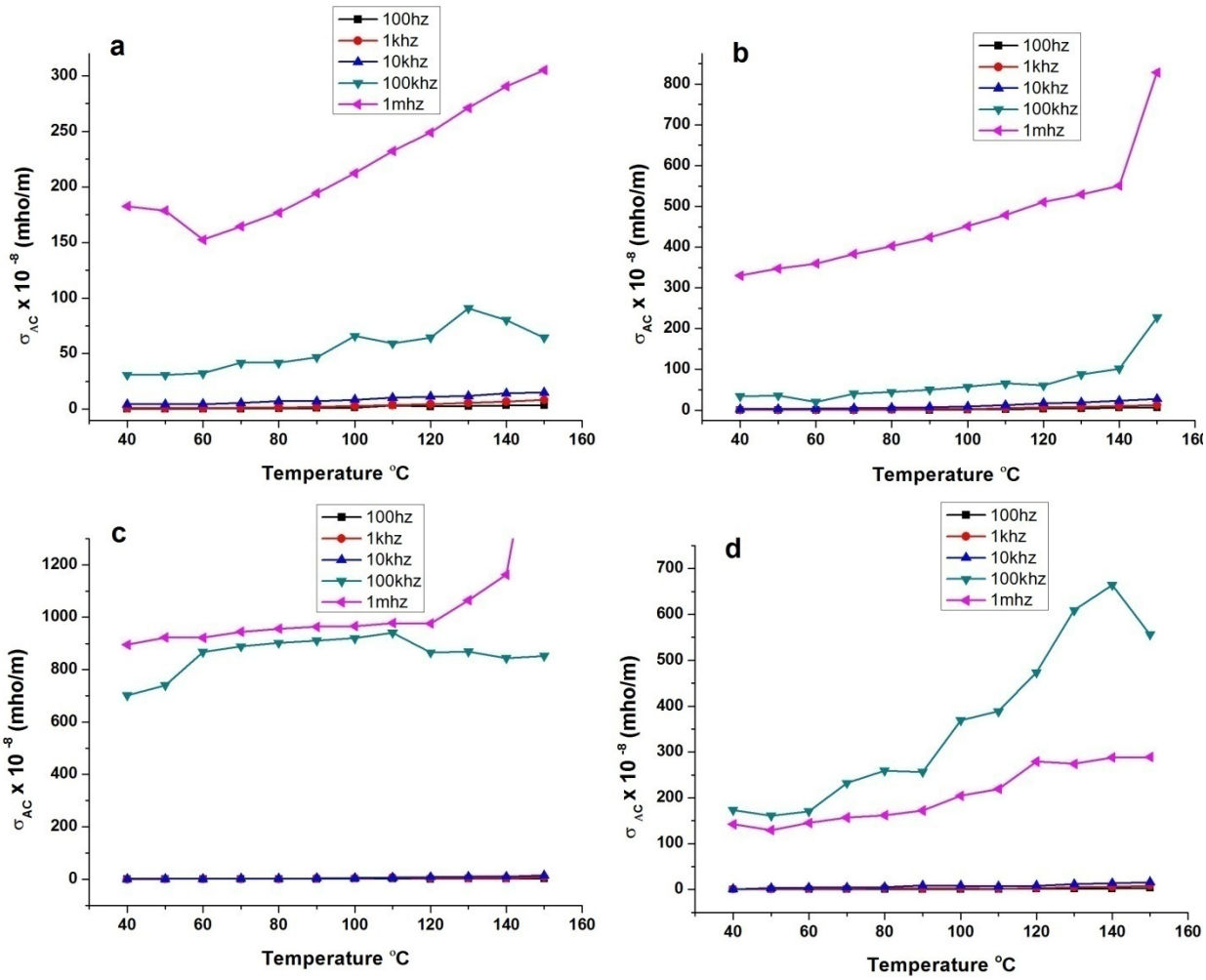

Fig. 13. AC Electrical Conductivity (σ_{AC} x 10^{-8} mho/m) for samples viz., a) Sample A, b) Sample B, c) Sample C and d) Sample D

Table 2. Dielectric constant, dielectric loss factor, and AC conductivity of the prepared samples

Temp	**Dielectric Constant**					**Dielectric loss factor**					**AC conductivity**				
	100Hz	**1 kHz**	**10 kHz**	**100 kHz**	**1 MHz**	**100hz**	**1khz**	**10 kHz**	**100 kHz**	**1 MHz**	**100Hz**	**1 kHz**	**10 kHz**	**100 kHz**	**1 MHz**
150	6.871	2.554	1.707	1.958	1.219	1.835	0.887	0.293	0.293	0.122	7.010	12.597	27.769	227.068	828.516
140	6.295	2.374	1.633	1.729	1.215	1.638	0.775	0.254	0.106	0.082	5.734	10.225	23.046	101.410	550.772
130	4.940	2.208	1.577	1.708	1.208	1.482	0.655	0.216	0.092	0.079	4.071	8.044	18.924	87.681	529.371
120	4.661	2.091	1.550	1.698	1.203	1.334	0.602	0.194	0.064	0.076	3.458	6.999	16.740	60.602	510.416
110	3.470	1.836	1.475	1.662	1.180	1.012	0.421	0.146	0.071	0.073	1.953	4.300	11.995	65.704	478.426
100	2.683	1.726	1.427	1.639	1.176	0.876	0.301	0.111	0.063	0.069	1.307	2.886	8.775	57.127	451.343
90	2.503	1.606	1.386	1.612	1.162	0.347	0.244	0.094	0.056	0.066	0.483	2.179	7.243	50.475	423.915
80	2.118	1.511	1.381	1.589	1.151	0.485	0.179	0.069	0.050	0.063	0.571	1.500	5.274	44.260	402.598
70	1.662	1.445	1.319	1.566	1.139	0.388	0.125	0.057	0.046	0.061	0.359	1.005	4.145	40.144	383.280
60	1.495	1.394	1.294	1.546	1.129	0.420	0.095	0.046	0.023	0.057	0.349	0.736	3.289	19.858	359.595
50	1.484	1.370	1.302	1.564	1.145	0.356	0.089	0.037	0.041	0.055	0.294	0.676	2.643	35.474	346.971
40	1.273	1.345	1.278	1.541	1.134	0.201	0.052	0.030	0.040	0.052	0.142	0.392	2.118	33.926	329.850

4. Conclusions

In summary, the tensile strength of the samples initially increased with an increase in reinforcements and then decreased with an increase in B_4C content beyond 10 wt %. Flexural strength, on the other hand, is found to increase with the addition of B_4C particles. The impact strength of the samples decreased with an increase in the addition of the B_4C particles. The dielectric constant increased and the dielectric loss factor decreased with increased frequency. However, the AC conductivity of the samples increased with an increase in frequency. The trend in the increase in dielectric constant with higher frequencies and temperature makes the material suitable for energy storage applications.

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