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Thermal Expansion of Aluminum/Fly Ash Cenosphere Composites Synthesized by Pressure Infiltration Technique

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Abstract
The coefficients of thermal expansions (CTEs) of commercially available pure aluminum and aluminum alloy composites containing 125 μm average size hollow fly ash particles (cenospheres) were measured using a dilatometer. Three types of composites were made using the pressure infiltration technique at applied pressure and infiltration times of 35 kPa and 3 minutes, 35 kPa and 7 minutes and 62 kPa and 7 minutes. The volume fractions of the fly ash cenospheres in the composites were around 65%. The CTE of the composites was measured to be in the range of 13.1×10^{-6} to 11×10^{-6} °/C, which is lower than that of pure aluminum (25.3×10^{-6}/°C). The infiltration processing conditions were found to influence the CTE of the composites. A higher applied pressure and a longer infiltration time led to a lower CTE. The theoretical value of the CTE of fly ash cenospheres was estimated to be 6.1×10^{-6}/°C.

1 Introduction

Metal-matrix composites (MMCs) have enhanced properties including higher strength, lower thermal expansion, higher fatigue life, and higher wear properties, as compared to those of their matrix alloys [1-4]. Ceramic particles have a lower CTE than metallic alloys, and therefore the incorporation of the particles in the matrices can reduce the CTEs of the resulting composite [5-7]. The volume fraction of the particles in the matrix can be controlled by utilizing proper processing techniques. The stir mixing technique is advantageous for making of composites containing lower particle volume fraction of reinforcements [1], while the pressure infiltration technique is appropriate for synthesizing composites with higher particle volume fraction [8].

Of the reinforcements used for synthesizing MMCs, hollow particles, including carbon, glass and alumina, have been used as a reinforcement to make syntactic foam composites. These foams are observed to have excellent energy absorption properties [9]. Dunand studied Al 6061-hollow mullite particles reinforced syntactic foams using the pressure infiltration technique [10]. The compressive stress-strain curves of syntactic foams show a plateau region, which corresponds to the energy absorption in crushing of hollow particles. Similar stress-strain behavior is observed in polymer matrix syntactic foam composites [11-13]. Considerable research has been conducted on measuring the mechanical properties of metal matrix syntactic foams [9, 10, 14, 15]. However, studies on the thermal expansion of syntactic foams have been rarely conducted.

In this study, Al-hollow fly ash hollow particulate (cenospheres) composites were synthesized using the pressure infiltration technique to make composites with high volume fraction of cenospheres (~65%). To identify the effect of processing conditions, specimens were synthesized at different infiltration pressures and times, and their CTEs were measured in the range of 30°C to 400°C. The primary objective of the work is to measure the reduction in CTE of Al due to the incorporation of large volume fraction of ceramic particles. Reduction in CTE enhances the thermal stability of the composite. The effect of thermal cycling on the CTE of the composites was also observed. Several studies have characterized various types of Al based composites for thermal cycling effects for 2 to 2000 cycles [7, 16-18]. Thermal cycling affects the CTE of composites. However, many of these studies test composites for 2 or 3 thermal cycles because a comparison of these studies shows that most of the effect of thermal cycling takes place in the initial few cycles [16, 17].

Direct experimental measurement of the CTE of cenospheres is not possible because of the experimental difficulties arising from the small particle size and variation in the internal structure of these particles. Hence, the measured CTE values for composites were used to estimate the effective CTE of the cenospheres using Rule of Mixtures and Turner model [19, 20].

2 Experimental Procedure

The chemical composition of cenospheres is shown in Table 1. Cenospheres were sieved to obtain particle in the size used in the range of 100-150 μm. To synthesize specimens, a 6 mm diameter quartz tube was filled with cenospheres and tapped to improve the packing density. The fly ash cenospheres were dried in an oven at 150°C prior to filling them in the tube. The packing density was calculated to be 0.36 g/cm^3 by dividing the measured net weight of the particles inside the tube by the total volume of the bed of particles. It is known that the closely packed particles of the same size assume random closed packing (RCP) arrangement with a packing efficiency of 64% [21]. Hence, the fabricated composites will have around 64% of cenospheres by volume.

Table 1. Chemical Compositions of Hollow Cenosphere Fly Ash Particle.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	MgO	K_2O	Na_2O	TiO_2
Wt%	61.00	25.80	4.99	0.82	0.31	1.58	3.59	0.74	1.00

Both ends of the tube were sealed with Kaowool and the packed tubes were dried for at least 30 minutes in an oven at 200°C. Pure Al melt was infiltrated into the bed by introducing nitrogen gas into a pressure chamber at a melt temperature of 750°C, and the gas pressure was increased to the desired values. As a result of pressurization the melt rises into the quartz tube containing cenospheres infiltrating the spaces between particles and later solidifying to form the composite. The samples for CTE tests were made at three different infiltration pressures and two different infiltration times as shown in Table 2. CTE of pure Al used as matrix material was also measured.

Table 2. Infiltration conditions for synthesizing aluminum/hollow fly ash composites and their volume fraction.

Composite #	Applied pressure (kPa)	Infiltration time (min)
1	35	3
2	35	7
3	62	7

Metallographic samples were taken from the Al-fly ash composites and polished to observe their microstructures using an optical microscope (Olympus/BH2-UMA). Standard polishing procedures were followed using SiC grinding papers to 600 grit. The final polishing was carried out on a micropolishing cloth with 0.5 μm SiO_2 slurry.

The specimens for the CTE testing had length and diameter of 50 mm and 6 mm, respectively. All the specimens were annealed for stress relief before CTE tests at 340°C for two hours. The linear thermal expansion tests were performed over the temperature range of 30°C to 400°C using a dilatometer. The specimens placed in the dilatometer were heated at a rate of 3°C/min for 125 minutes. The specimens were held for 10 minutes at 400°C, and then the CTE test apparatus was shut off automatically. The specimens were cooled naturally in the CTE apparatus. The specimens were passed through two successive CTE test cycles to determine the effect of thermal cycling.

3 Result and Discussions

3.1 Microstructure of Pressure Infiltrated Syntactic Composites

The microstructure of the syntactic composite fabricated by infiltrating a bed of fly ash cenospheres at an applied pressure of 35 kPa for 3 minutes is shown in Figure 1. Similar structure was observed all over the specimen cross section and clustering of cenospheres was not observed in the specimen. The microstructure of the composite made by infiltrating a bed of cenospheres at an applied pressure of 62 kPa for 7 minutes is shown in Figure 2, which also illustrates a uniform distribution of the particles. When particles are densely packed in a tube, their movement is restricted during infiltration, leading to a uniform particle distribution. A common feature observed in these microstructures is the presence of entrapped air, called voids, near the areas of contact of neighboring cenospheres.

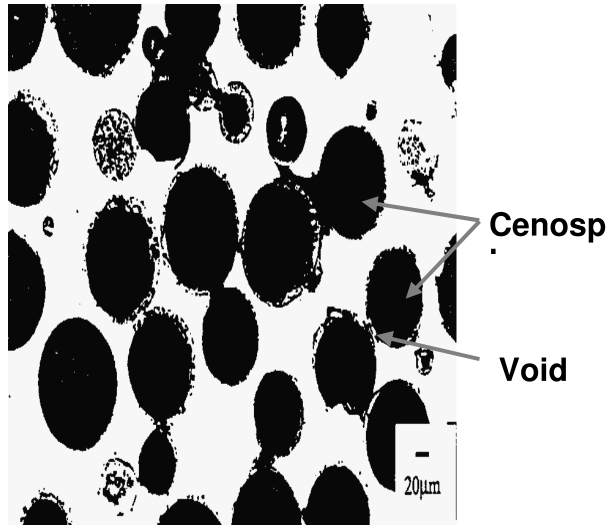

Figure 1. Microstructure of the middle portion of pure aluminum-fly ash cenosphere composite, infiltrated at a pressure of 35 kPa for 3 minutes.

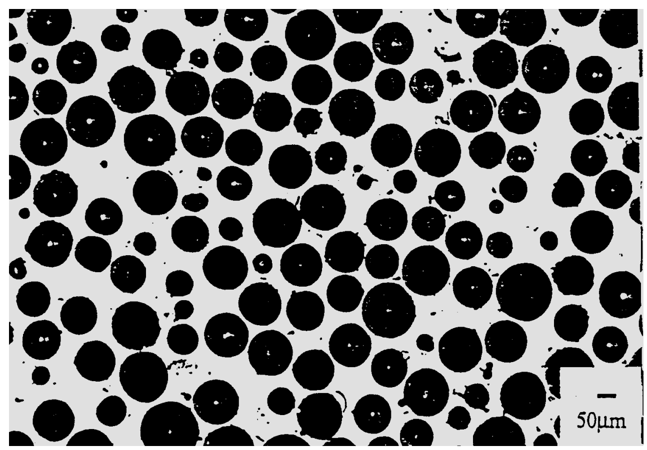

Figure 2. Microstructure of the middle portion of pure aluminum-fly ash cenosphere composite, infiltrated at a pressure of 62 kPa for 7 minutes

Some voids are indicated in higher magnification micrograph in Figure 1. The melt flow near the particle contact areas requires significantly higher infiltration pressures due to the high capillary forces existing in this region. In view of this, the microstructures illustrating voids near the particle-particle contacts indicate that applied pressures of the order of 62 kPa for 7 min were insufficiently high for the melt to infiltrate the voids. However, the infiltration pressure was not increased further because it may lead to fracture of cenospheres. Voids near the contact area of particles or fibers have been observed in many composite materials [22, 23].

The volume fraction of voids present in the samples is likely to decrease with an increase in applied pressures and infiltration times. It is expected that the sample made at the higher applied pressure of 62 kPa and the longer infiltration time of 7 minutes will have lower void content than the samples that were infiltrated at the lower pressure or for the shorter infiltration time.

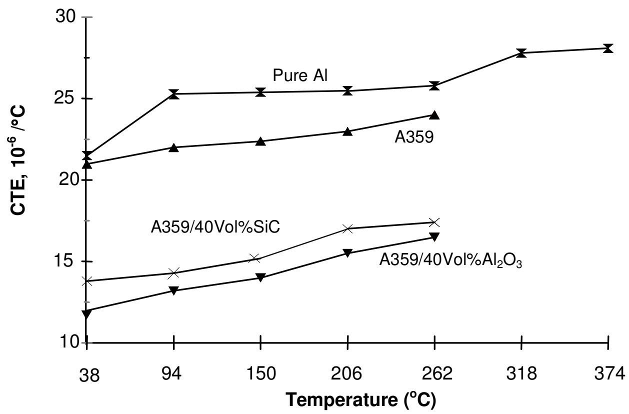

Figure 3. Plot of CTE with temperature for pure aluminum, A359 alloy and different composites.

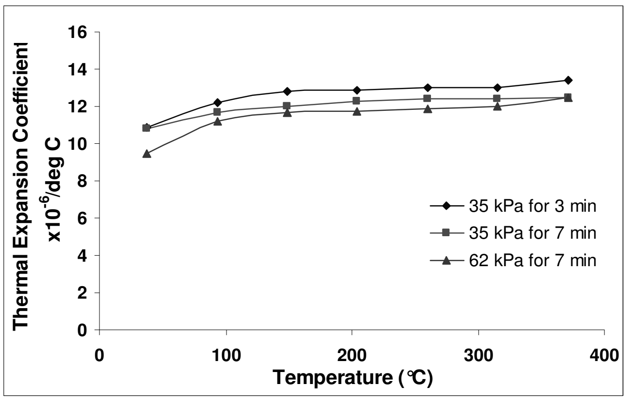

Figure 4. Coefficient of thermal expansion of the pure aluminum-fly ash composites as a function of temperature for various applied pressures and infiltration time.

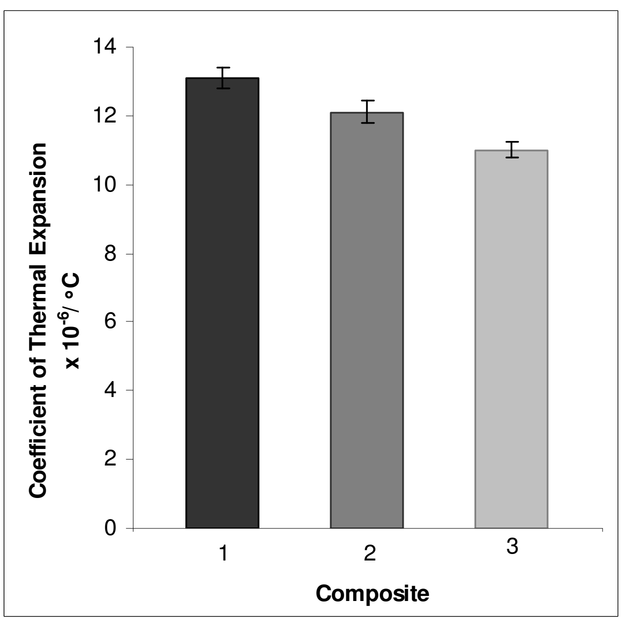

Figure 5. Variation in the CTE of aluminum-fly ash composites (average of three specimens each) with infiltration conditions: Composite 1 (35 kPa for 3 minutes), Composite 2 (35 kPa for 7 minutes), and Composite 3 (62 kPa for 7 minutes).

However, it is difficult to determine the void content in each sample by a difference in their densities calculated using Rule of Mixtures and the measured densities, because some cenospheres fracture during processing and get filled with the infiltrating melt.

3.2 Measurement of Thermal Expansion Coefficient

The variation in the CTEs as a function of temperature for pure Al specimen, measured in this study, is shown in Figure 3. The variation in CTEs of A359 alloy, A359/SiC composite and A359/Al_2O_3 composite, obtained from the literature [24] is also shown in Figure 3. The CTE of pure Al was found to vary in the range of 21.5×10^{-6}/°C to 27.9×10^{-6}/°C at temperatures between 100°C and 400°C. These values are close to the reported value of 25.3×10^{-6}/°C at temperatures between 20 and 300°C [25]. It is also shown in Figure 3 that the CTEs of the composites are lower than that of pure Al and the Al alloy. The lower CTE of the composites is due to the presence of ceramic particles which have a lower CTE than pure Al and its alloys shown in Table 3 [26]. Likewise, incorporation of fly ash cenospheres, which are composed of alumino-silicates, in pure Al is expected to decrease its CTE.

Table 3. Thermal expansion coefficient of various particles and alloy.

Reinforcement particle and alloy	Thermal Expansion (10^{-6}/°C)
SiC	4.8
Al_2O_3	7.5
A2014	23
A6061	22

The variations in the CTEs of Al-fly ash composites synthesized under various conditions, as a function of temperature, are shown in Figure 4. It is shown that the CTEs of the composites increases between 50 and 100°C and remain relatively constant in the range of 100 to 300°C. The repeat measurement showed variations within only 2%, which is smaller than over 4% difference between average CTE values for these composites. Chang et al. also observed similar trend in an Ag/SiC composites [27]. They related this trend to internal stresses that occur due to the CTE difference between the matrix and particles. The magnitude of internal stresses decreases with increasing temperature. In view of this, it can be expected that in the temperature range of 100 to 300°C, the expansion of the composites synthesized under the current experimental conditions is almost linear. Elomari et al. showed that for an Al-SiC composite, the CTE increases rapidly above certain temperatures [28].

The CTEs of Al-fly ash composites, averaged for three specimens of each type, are shown in Figure 5. These values are as follows; 13.1×10^{-6}/°C (±2.2%) (for 35 kPa and 3 minutes of infiltration, 12.1×10^{-6}/°C (±2.7%) for 35 kPa and 7 minutes for infiltration, 11.0×10^{-6}/°C (±2.1%) for 62 kPa and 7 minutes of infiltration. These values are lower than the measured average CTE of pure Al, 24.7×10^{-6}/°C. Guo et al. have observed the formation of a ceramic phase at the cenosphere-matrix interface due to the reaction between Al of matrix and the Si present in the cenospheres [29]. The formation of a ceramic phase also contributes to a decreased CTE in fly ash filled composites.

The difference in the average CTE of the composites suggests that the CTE is influenced by applied pressure and infiltration time. The sample synthesized at the lower applied pressure of 35 kPa and the shorter infiltration time of 3 minutes has a higher CTE,

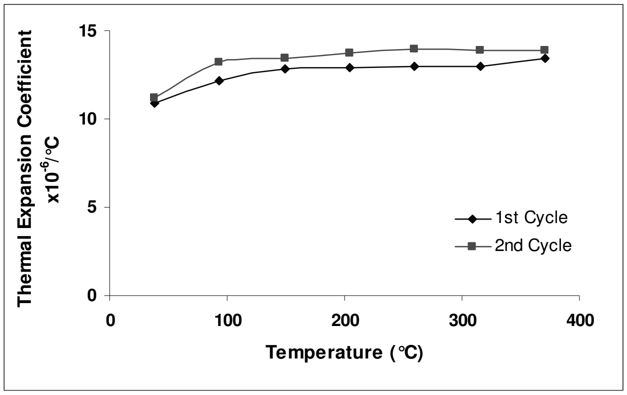

Figure 6. Effect of thermal cycling on the CTE of the composites as a function of the temperature for an infiltration pressure of 35 kPa for a time of 3 min.

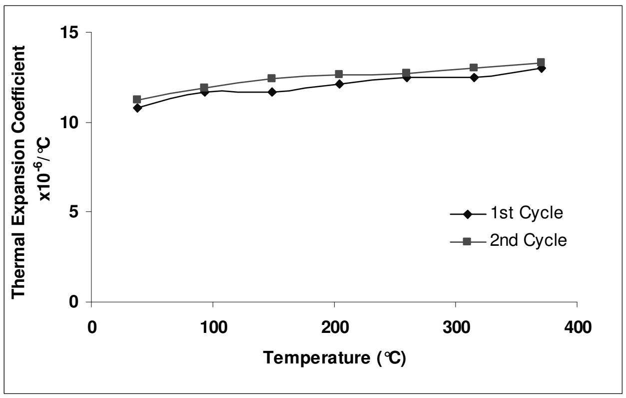

Figure 7. Effect of thermal cycling on the CTE of the composites as a function of the temperature for an infiltration pressure of 35 kPa and time of 7 min.

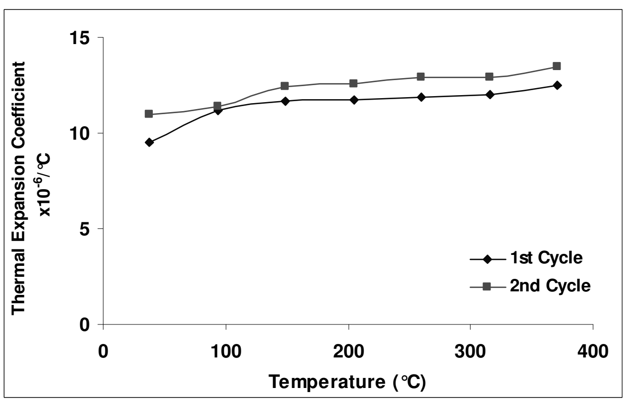

Figure 8. Effect of thermal cycling on the CTE of the composites as a function of the temperature for an applied pressure of 62 kPa and the infiltration time of 7 min.

as compared to other samples synthesized at the higher applied pressure of 62 kPa or a longer infiltration time of 7 minute. In general, processing conditions influence the microstructural features in the pressure infiltration technique, especially the void content. In composites, the presence of voids is known to increase the CTE [30]. The higher applied pressure and the longer infiltration time lead to lower void content in the sample resulting in lower CTE.

3.3 Coefficient of Thermal Expansion of Fly Ash Cenospheres

The rule of mixtures (ROM) is used to express CTE as a function of volume fractions and the CTEs of the matrix and the reinforcement as given in Equation 1:

$$\alpha_c = V_r \alpha_r + V_m \alpha_m \qquad (1)$$

where α is the CTE, and V is the volume fraction. The subscripts c, r, and m denote composite, reinforcement, and matrix, respectively. This model is simple but ignores the influence of voids, mechanical properties of matrix and reinforcements, and the particle-matrix interfacial bonding characteristics. In addition, ROM does not account for the particle-particle interactions. Hence, the Turner model has also been used to calculate the CTE of composites [19]:

$$\alpha_c = \frac{V_r K_r \alpha_r + V_m K_m \alpha_m}{V_r K_r + V_m K_m} \qquad (2)$$

where K is the Bulk modulus. The Turner model assumes that homogeneous strain is present throughout the composite. In Equations 2 the α_c is shown to be influenced by several factors, including K_m and K_r. The values of these factors depend on the type of reinforcement (solid and hollow). For hollow particles the CTE also depend on the wall thickness, due to the dependence of mechanical properties of the particles on wall thickness. An estimate of the average CTE of cenospheres present in the composite can be obtained by using Equations 1 and 2. The calculated values will present a lower bound of the fly ash CTE values because a thin interfacial layer of ceramic phase is generated due to the reaction between cenosphere surface and the matrix material, which can contribute to lowering the CTE of the composite. The CTE of the specimen synthesized at 65 kPa for 7 minutes is used for the calculations because this specimen has the least amount of voids and will give more precise values. In the case where ROM is used, α_r is calculated as 3.3×10^{-6}/°C. This value is considerably lower then that of other ceramics listed in Table 3. A better estimate can be obtained from Equation 2 if K is calculated by the following equation [31].

$$K = \frac{E}{3(1-2\nu)} \qquad (3)$$

where E and ν are Young's modulus and Poisson's ratio, respectively. The bulk modulus of Al matrix is calculated to be 68.6 GPa, using E_m = 70 GPa and ν_m=0.33 [32]. The bulk modulus of the fly ash is calculated to be 107.8 GPa, using E_r = 110 GPa and ν_r=0.33 [33]. The calculated CTE of cenospheres from Equation 2 comes out to be 6.1×10^{-6}/°C. This value is close the values of other ceramic particles given in Table 3. Any improvement in determining the mechanical properties of cenospheres will reflect as better accuracy of the calculated CTE.

3.4 Thermal cycles

The variations in the CTEs of the composites in the 1st and 2nd thermal cycles as a function of temperature are shown in Figures 6 through 8. The CTEs in the 2nd cycle are higher than those at the 1st cycle for the three samples. The difference between the average CTE of the composite in the first and the second cycle in these figures are 5.8, 3.3 and 7.8%. The experimental variations between various specimens of one type of composite are within 2.0, 1.1 and 1.0%, respectively, which are smaller than the difference between the values for the first and the second thermal cycle. Elomari et al. showed that the CTEs in the 2nd cycle are higher than those in the 1st

cycle for Al-SiC composites containing 40 µm size particles but the tendency was reversed for composites containing smaller particles [28]. In the present study, average 125 µm diameter particles are used to make composites and the results show that higher CTEs are observed for the for 2nd cycle, which is in line with Elomari's observations. During the thermal expansion of MMCs, internal stresses are developed around the particles due to a difference in the CTE of the matrix and the particle, and they are relieved by formation and movement of dislocations. The stress built around the particles may not be completely relieved, and therefore it can influence the 2nd thermal cycle.

Some other factors may also influence the CTE of composites during thermal cycling. CTE mismatch between matrix and reinforcement can cause the formation of microvoids or cracks at the particle-matrix interfaces [34, 35]. Elomari et al. observed that the prestrained Al-SiC composites have a higher CTE due to crack formation around the particles [36]. In Al-fly ash composites, the internal stresses developed around the particles due to the difference in CTE between the particle and matrix in the temperature range from 100°C to 200°C is in the order of 131 MPa ($\Delta\alpha \times \Delta T \times E_m$ = (25.3–6.6)×10^{-6}×(200–100)×70 GPa = 131 MPa), which is much higher than the yield strength of a pure Al (around 100 MPa). Thus, during the CTE measurement, yielding takes place adjacent to the particles, which is likely to influence the CTE of the composites undergoing the thermal cycles. Since thermal cycles may lead to formation of microvoids increasing the number of thermal cycles may lead to a higher CTE [29].

In the composites produced by pressure infiltration method the entrapped air voids are mostly attached to the particles. In such cases it is difficult to observe the effects of the stress and the void separately. This is because the stress generated around the particles is influenced by the presence of voids.

In this study, the CTE of pure Al containing 65 vol.% of hollow fly ash particles was studied. The results have suggested that composites with a lower CTE can be made by incorporating cenospheres and controlling the processing conditions for a given volume fraction of reinforcement.

4 Conclusions

An experimental study is conducted to measure the effect of fly ash cenosphere particles on the CTE of aluminum composites. The important findings of the study are:

1. The presence of fly ash cenospheres in pure Al matrix decreases its CTE. The average CTE of Al-fly ash composites is in the order of 12×10^{-6} /°C within the temperatures range of 100 and 400°C.
2. The CTE of the composite synthesized at 62 kPa pressure for 7 minutes is lower than that of the composites infiltrated at 35 kPa for 3 minutes time. The increase in applied pressure from 35 kPa to 62 kPa and the increase in infiltration time from 3 to 7 minutes led to a 16% decrease in the CTE. Increase in infiltration pressure and temperature improves the infiltration and decreases the entrapped air voids, which reflects as lower CTE.
3. The CTE of fly ash cenospheres was calculated using Rule of Mixtures and Turner's model. The CTE of fly ash cenospheres calculated using the Rule of Mixture is 3.3×10^{-6}/°C. The CTE obtained using the Turner's model was 6.1×10^{-6}/°C.
4. It was observed that the CTE in the second thermal cycles is higher than that in the first thermal cycle. This appears to be related to yielding due to the thermal expansion mismatch of cenosphere and matrix during thermal cycling.

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