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Research Article

Mechanical Loading and Tribological Studies on Boron Carbide (B_4C) and Lead (Pb) Particles Dispersed Epoxy-Based Multilayered Composites

S. Vignesh,[1] **J. T. Winowlin Jappes**,[1] **Khan M. Adam**,[1] **and Temal Varol**[2]

[1]*Department of Mechanical Engineering, Kalasalingam Academy of Research and Education, Krishnankoil, India*
[2]*Department of Metallurgical Engineering, Karadeniz Technical University, Trabzon, Turkey*

Correspondence should be addressed to J. T. Winowlin Jappes; winowlin@klu.ac.in

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The study aims in the development of functionally graded epoxy-based layered composites dispersed with B_4C and lead particles. The development route adopted for the composites is a novel route called layered molding and curing. Various compositions of single and trilayered composites were prepared through the abovementioned route. The samples prepared were subjected to mechanical and tribological studies, and the results were reported in this article. It is found that the mechanical properties of the single-layered composites consisting of 20% lead and 20% B4C show superior characteristics than those of the samples with increased addition of lead. However, the trilayered samples with lead core showcased excellent mechanical properties. On the other hand, the wear rate and mass loss of the trilayered samples with B_4C cladding show minimum wear rate than the samples with lead cladding. Furthermore, the coefficient of friction of the samples also showcases the better performance of single-layered samples with 20% lead. The worn surface analysis done through scanning electron microscopy and stereo zoom microscopy reveals the reason for the low specific wear rate of 20% lead sample as the self-hindrance of wear debris evolved during the wear study.

1. Introduction

There is an increasing need in material research to meet out the demand of high-performance materials in the global automotive, aerospace, structural, and other sophisticated sectors. This led to the development of mechanically strong and tribologically better composite materials. Composite materials are even preferred for its multifunctionality, for its life span, for its economical prices, or even its role in improving the material aesthetically. Composite materials, the product of mixing or stacking two or more materials, are a promising attraction to researchers in replacing conventional metallic materials. Although the problem of the product lies in its weak matrix constituting polymers that result in weak interlaminar shear strength, the best use of composite is because of its high specific strength, stiffness, fatigue behavior, and density [1–4]. There is a greater need for a better material in order to transport and store the spent nuclear fuel, since the conventional lead caskets are found to be heavy and not safe due to its chemical characteristics. Epoxy is proposed by several researchers for its greater performance in mechanical and structural applications. It possesses excellent hardness, fatigue resistance, and very small contraction during curing that makes it more suitable for mechanical applications. Similarly, the epoxy serves as an excellent matrix material owing to the adhesion behavior to varieties of reinforcements. Furthermore, it also possesses excellent stability and resilience behavior when exposed to neutrons which is also an obvious reason to use epoxy in many sophisticated applications [5]. However, it lags in antiwear and antiabrasive properties which are not up to the mark. Nowadays, boron carbide (B_4C), a ceramic material, has created attention among the research groups owing to its excellent mechanical, tribological, and structural properties.

Moreover, the properties such as hardness and density make it more suitable for some rugged applications like abrasives, products with good wear resistance, and even in ballistics. Similarly, B_4C has good neutron absorption cross section for catering the thermal neutrons and also possesses an atomic number density of 0.11 $Å^{-3}$, owing to which, it is nowadays used in nuclear industries for effective shielding of ionizing radiations [6]. Research studies support the development of B_4C reinforced epoxy composites for the improvement of mechanical and tribological properties. Furthermore, the reports suggest the improvement of wear resistance of the B_4C composites through increased particle addition by facilitating the anchoring mechanism. However, research suggests the effective addition in the epoxy environment as the increased addition of the same will detriment the mechanical properties [7]. Similarly, lead (Pb) is a widely used potential radiation shielding material which can also be used for packaging, storage, and transportation of radioactive fuels. Pb finds a wide variety of applications particularly in the field of radioactivity and shielding of radioactive environment. In nuclear power industries, the storage of cooled spent nuclear fuel plays an uncompromised role in terms of safety of both biotic and abiotic species [8, 9]. Presently, lead caskets serve the purpose of storing and transporting the cooled spent nuclear fuel. However, because of its high density leading to enormous weight and poor mechanical and tribological properties, there is a profound need of a suitable alternative material that can cater the above needs. Hence, in this work, an attempt is made to develop high mechanically and tribologically efficient B_4C particles along with lead (Pb) dispersed epoxy laminates. Furthermore, the need of irradiation shielding can be ensured by incorporating lead (Pb) particles. Moreover, a storage container is expected to have good mechanical and tribological properties, particularly wear resistance [10]. On the other hand, owing to safety issues, the research on pure lead-based epoxy laminates is almost a void. Hence, in this work, an attempt is made to produce epoxy-based single- and trilayered composites dispersed with varying composition of B_4C and Pb particles through layered molding and curing route for industrial packaging and transportation caskets.

2. Materials and Methods

2.1. Materials. The matrix epoxy LY566 and its corresponding hardener were procured from Vasavi Resins Pvt. Ltd., Chennai. The powdered boron carbide (B_4C) particles (99.9%) with particle size 10 μm were procured from Neena Metal Mart, New Delhi, and the powdered lead (Pb) particles (99.9%) of particle size 10 μm were procured from Alpha Chemika, Mumbai. The metallic particles were subjected to SEM to confirm the particle size.

2.2. Experimental Methods. The required composite material using lead, boron carbide, and epoxy is made using the layered molding and curing method as shown in Figure 1. For this, powdered lead and boron carbide were mixed and made into several composite samples in a mold with several

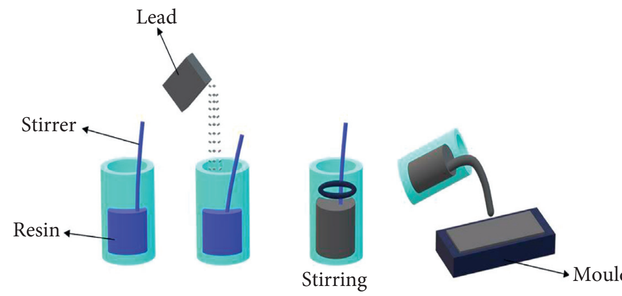


Figure 1: Schematic of layered molding and curing route.

Table 1: Composition of single-layered samples and the density of the samples.

Samples	Composition of single-layered composites		Density (g/cm^3)
	B_4C	Pb	
A	20	10	1.38
B	20	20	1.55
C	20	30	1.78
D	20	40	1.89

different propositions. Six samples were made of various propositions with different processes. Few glass molds were made for preparing the composites.

The dimension of the mold is 300 mm × 137 mm × 3 mm, and all the samples were made with a thickness of 3 mm. OHP sheets were placed over the molds for easy removal of the material. Wax is applied to the mold and over the OHP sheets before every sample is made, for easy removal of the composite material after curing. After getting the molds ready, 6 samples were made, each one had a different proposition of lead, boron carbide, and epoxy. The measurements of the powders and the development of the composites were done with proper care using masks and gloves and in an isolated chamber, respectively, to avoid the contact of any lead or boron carbide powder, which when contacted directly becomes hazardous. The details of the samples prepared and the density are tabulated in Tables 1 and 2.

Thorough mixing is done through mechanical mixing and ultrasonication process as well in order to avoid agglomeration of the particles. The whole mixing process is done at a slow pace to avoid any air bubble formation, which might affect the material quality. After mixing the liquid mixture for 45 mins, the mixture is poured in the glass mold and made to spread uniformly. Similarly, layered samples were made in a definite interval of 45 minutes after the pouring of first and the subsequent layers. The samples were allowed to cure for 24 hours before removing from the mold. The schematic of the layered samples is depicted in Figure 2.

2.3. Mechanical Studies. The samples prepared were tested for its tensile strength after carefully adhering to the ASTM standard ASTM D638 ($165 \times 13 \times 5\,mm^3$). The flexural testing was carried out in accordance to the ASTM standard

Table 2: Composition of trilayered samples and the density of the samples.

Samples	Layer layout of the samples			Composition of the trilayered samples		Density (g/cm^3)
	Layer 1	Layer 2	Layer 3	B_4C	Pb	
E	B_4C	Pb	B_4C	20	20	1.58
F	Pb	B_4C	Pb	20	20	1.88

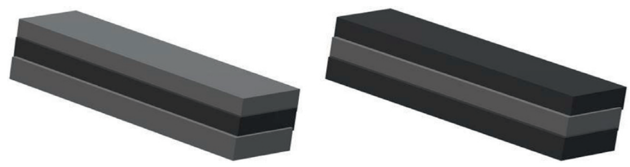


Figure 2: Schematic of the trilayered samples fabricated through layered molding and curing.

of dimensions $127 \times 13 \times 5\,mm^3$, and the impact test was done in accordance with ASTM D256 with specifications $60 \times 13 \times 5\,mm^3$ using an Izod impact tester with a v-notch cut in the samples. The fractography of the tested samples were done by using a JEOL-JSM-5600LV scanning electron microscope equipped with EDS.

2.4. Tribological Study. A pin-on-disc wear tester (Magnum Engineers, Bengaluru, as per ASTM G-99 standard) was used to carry out the friction and wear test for all the samples. Five sets of tests were conducted on each sample for a span of 30 min. The ambient temperature and the relative humidity during the run were 23°C and $50 \pm 5\%$, respectively. The specimen used for the test was $10\,mm \times 10\,mm \times 3\,mm$ and was made in contact with a hardened alloy steel (62 HRC and surface roughness $R_a = 0.54\,\mu m$) disc. The samples were kept in such a way that the thickness of the sample was perpendicular to the counter body that rotates. The samples were initially recorded for its weight and also after the conduction of the test in order to calculate the specific wear rate of the samples. The test conditions of the wear studies are given in Table 3. The specific wear rate was then calculated using the following equation:

$$K_s = \frac{\Delta V}{L d} \frac{m^3}{Nm}, \quad (1)$$

where ΔV is the volume loss of the material during the wear test, L is the load, and d is the distance of sliding or abrading distance.

2.5. Scanning Electron Microscope and Stereo Zoom Microscope Images. The tensile and flexural fractured samples and the worn surface of the samples were scanned and analyzed using a JEOL-JSM-6390 scanning electron microscope with X250–X1000 magnification. Furthermore, the counterpart surface was inspected using a Motic SMZ-168 stereo zoom microscope in order to identify the residues and debris resulting from the wear studies.

Table 3: Wear test conditions.

Normal load (N)	Sliding velocity (m/s)	Sliding distance (m)
20	3.5	2000
20	4.5	2000
20	5.5	2000

3. Results and Discussion

3.1. Tensile, Flexural, and Impact Characteristics. The tensile strength of the samples is depicted in Figures 3(a) and 3(b). It is evident from the figures that the maximum tensile strength of 49.5 MPa is obtained for the sample with 20% Pb. It is further evident that the addition of Pb content beyond 20% reduces the tensile strength of the sample. It may be attributed to the increase in brittleness of the sample beyond the addition of 20%. Furthermore, it should be noted that some research studies revealed the similar scenario of embrittlement upon the addition of B_4C beyond 20%. Also, the trilayered samples exhibit different nature of tensile properties which is evident from the figure shown above. The sample E layered with 20% Pb as core and with B_4C cladding exhibited good tensile strength. On the other hand, the sample F with Pb cladding was found to have poor tensile strength.

Similarly, the elastic modulus of the samples decreased with an increase in the addition of the particles. The reduction in elastic modulus is as evident from the stress-strain diagram depicted in Figure 3(a) which further shows the similar trend for the trilayered samples. It is well obvious from the stress-strain curve that the embrittlement of the material took place and the same behaved more like a brittle material.

Figure 3(c) clearly depicts the trend of flexural strength of the various samples. It is obvious from the figure that the single-layered sample loaded with 20% of Pb and B_4C exhibited a good flexural strength of 50.4 MPa. Furthermore, it is evident that the increase in the composition of lead beyond 30% decreases the flexural strength. On the other hand, the triple-layered sample with B_4C core exhibits lower flexural strength. However, the sample with B_4C cladding has witnessed the increase in flexural strength which may be attributed to superior properties of B_4C. It is evident from Figure 3(d) that the impact performance of the samples yields more at 20% of lead. As the composition of lead increases, the energy absorbed by the specimen decreases, and it decreases steeply with 40% addition of lead.

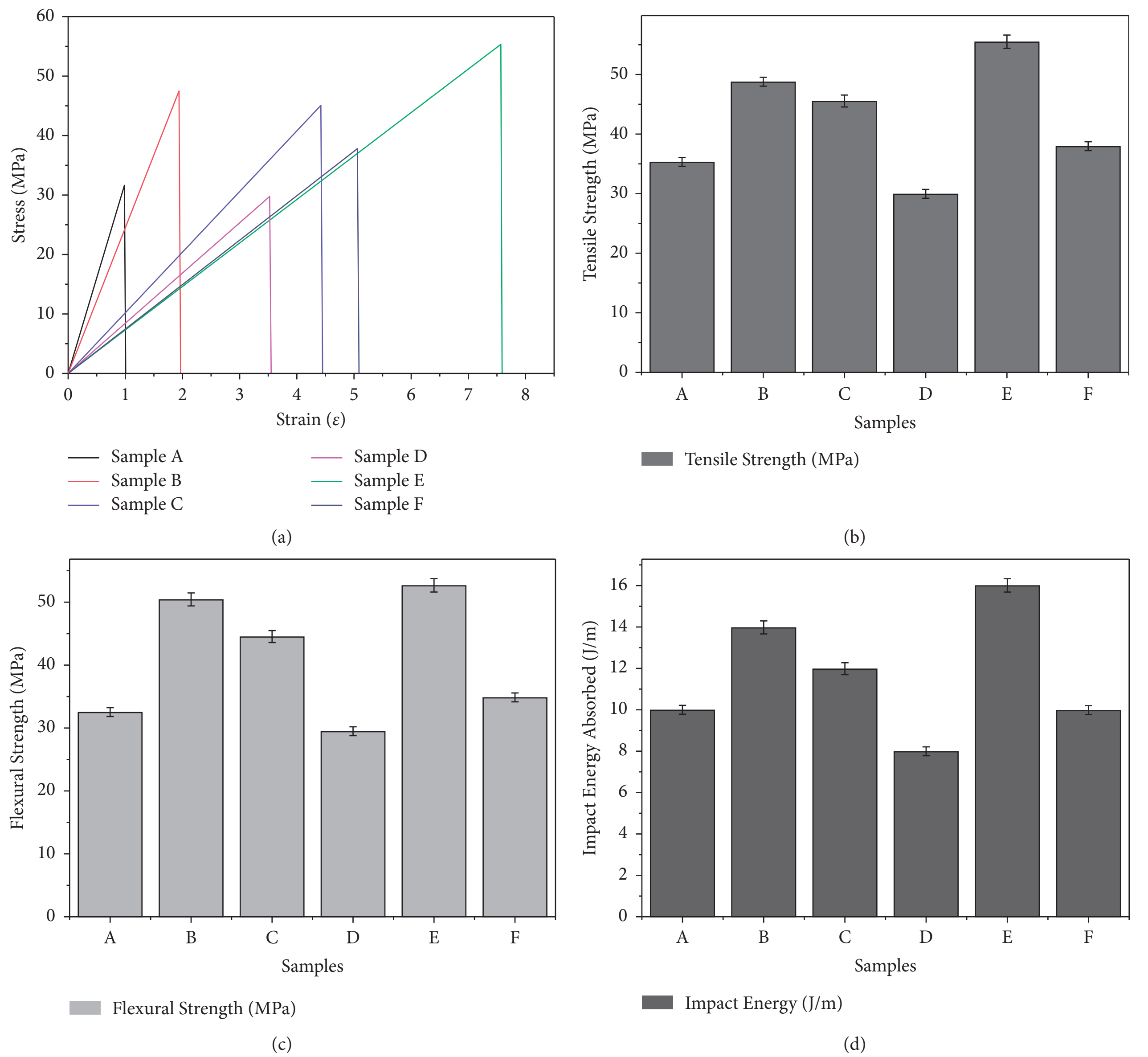
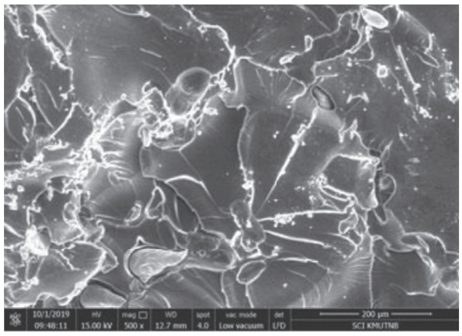


FIGURE 3: (a) Stress-strain curve. (b) Tensile strength of the samples. (c) Flexural strength of the samples. (d) Impact energy absorbed.

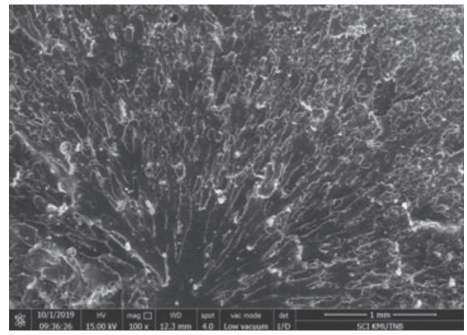
3.2. Fractography Studies. The tensile fractography in Figure 4(a) reveals the presence of agglomeration of the particles which may also be a reason for failure due to embrittlement. Furthermore, the chevron-like pattern in Figure 4(b) indicates the fracture of the sample due to brittleness [11–14]. On the other hand, the reason for the above failure can be evident from the fractograph shown in Figure 4(c). It shows the failure of the sample due to poor adhesion at higher loadings particularly in the sample with lead cladding. Figure 5 depicts the flexural fractograph, and it is evident from the figure that the added particles while loading tend to pullout from the epoxy matrix. This could be the obvious reason behind the failure of the specimen. On the other hand, the triple-layered sample with B_4C core exhibits lower flexural strength. However, the sample with B_4C cladding has witnessed the increase in flexural strength which may be attributed to superior properties of B_4C.

The impact energy absorbed by the specimen showcased superior results by the sample with 20% of lead, whereas the impact performance reduced significantly as the addition of lead is increased. This may be because of the poor particle distribution in the matrix and is evident from the SEM microscopy depicted in Figure 6. However, the trilayered samples witnessed similar trend as that of the tensile and flexural studies, that is, the sample with lead core absorbed energy to a greater extent while compared with the sample with B_4C core. This could be attributed because of the uniform dispersion of the lead particles and also because of softer nature of lead.

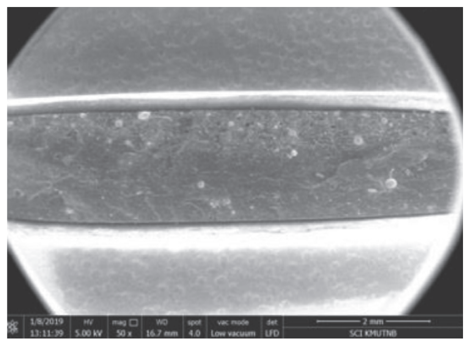
3.3. Tribological Studies. Figure 7(a) shows the specific wear rate of the samples and also the comparison with other composites. It is evident from the graph that the samples



(a)



(b)



(c)

Figure 4: (a) SEM image depicting the agglomeration of dispersed particles. (b) SEM image depicting chevron pattern. (c) Delamination of trilayered specimen.

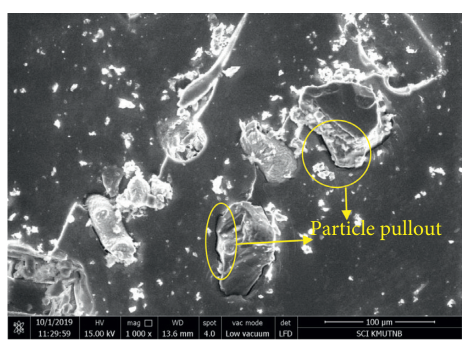


Figure 5: SEM image depicting the particle pullout from the matrix.

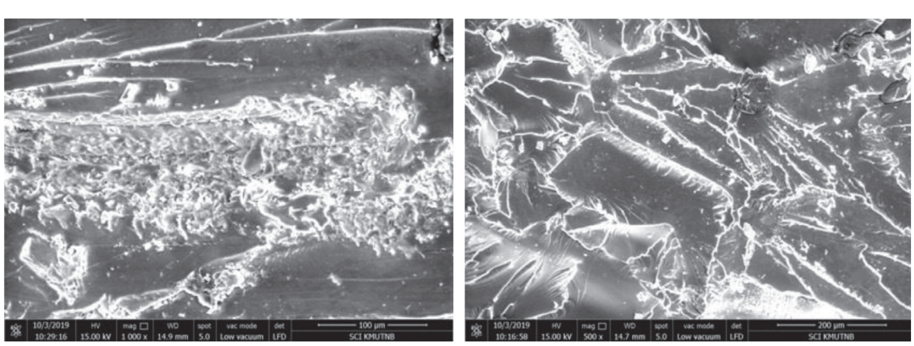


Figure 6: Impact fracture SEM image depicting agglomeration of the particles.

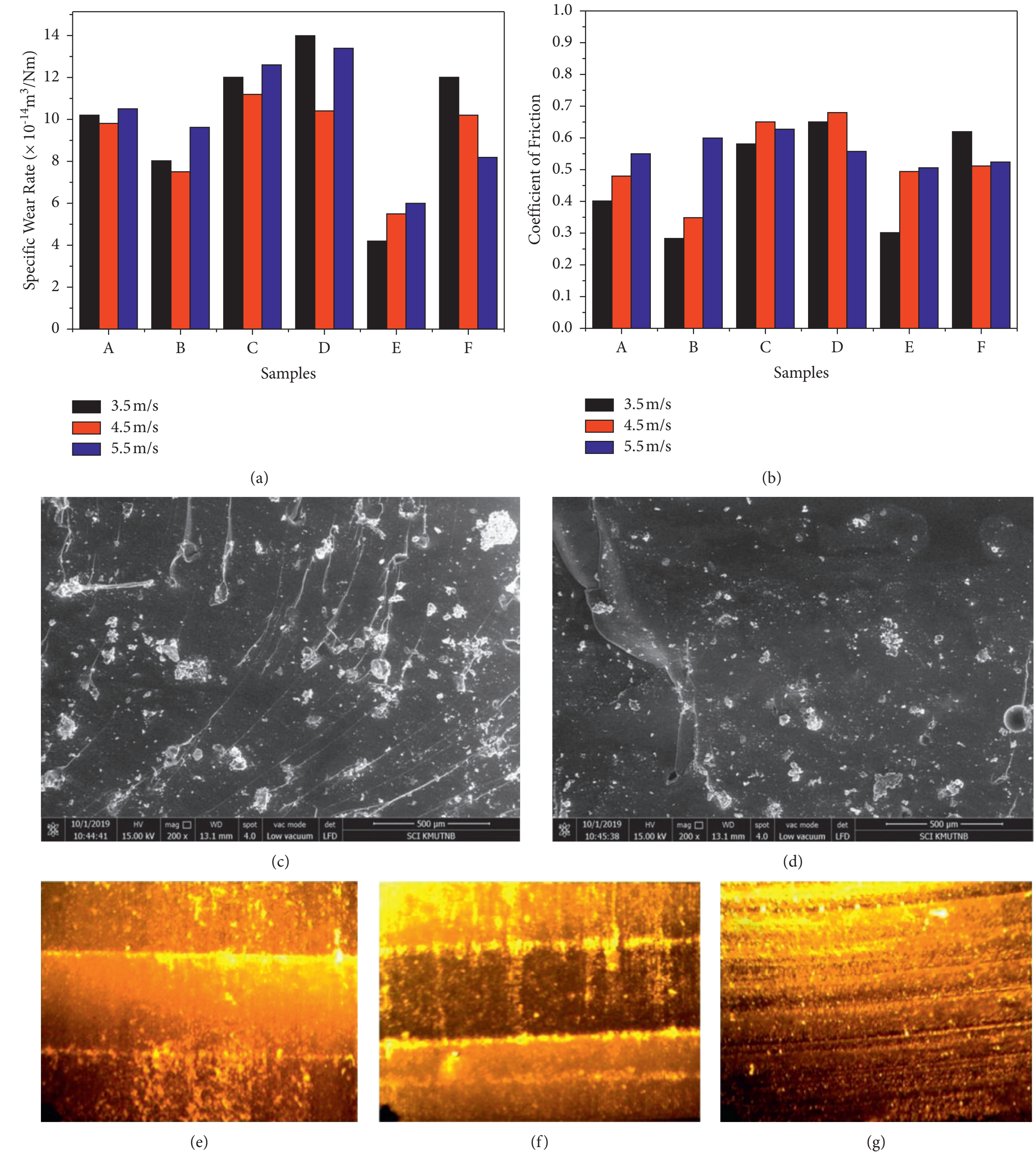


Figure 7: (a) Specific wear rate of the samples. (b) Coefficient of friction of the samples. (c, d) SEM image depicting the wear surfaces. (e–g) Stereo microscope images of the worn surface of the trilayered samples.

with increasing lead content reduce the wear performance of the composites. The specific wear rate of the sample with 20% lead content showcases excellent wear resistance. This may be owing to the strong bond existing between the epoxy matrix and the boron carbide particles. However, the scanning electron microscopic image depicted in Figures 7(c) and 7(d) clearly shows the length trace of wear path. This may be because of the inherent resistant behavior of the wear debris from forming further wear in the surface owing to its strong interfacial bonding with the matrix [15–17]. This could be the reason that the wear debris itself acts as an inhibitor to the wear. Furthermore, Figure 7(b) elucidates the coefficient of friction of the various samples under study. It clearly confirms that the sample with 20%

lead possesses excellent wear resistance. Furthermore, Figures 7(e)–7(g) clearly illustrate the worn surface of the trilayered samples. The trilayered sample with Pb core possesses excellent wear resistance which may be due to the fact that boron carbide, being a very hard material, possesses inherent wear resistance which acted as a protective layer. This avoided the wear track to reach the inner core of the sample and is clearly evident from Figure 7(e). At high speeds, the samples showcased lower wear resistance owing to the generation of frictional heat which makes the epoxy matrix weaker [18–22]. On the other hand, the sample with lead cladding had resulted poor wear resistance even at low speeds which may be attributed to the softer nature of the lead. Furthermore, it is evident from the SEM analysis that the formation of surface fatigue, grooving, and crazing could also be the reason for the poor wear resistance in the samples with increased lead content [6, 22–26].

4. Conclusion

In this work, different layered composite samples of epoxy matrix dispersed with boron carbide and lead were fabricated through the layered molding and curing technique. The samples were studied for the mechanical and tribological properties, and the conclusion is as follows:

(i) The tensile strength, flexural strength, and impact energy of the single-layered sample with 20% B_4C and 20% Pb exhibited good performance owing to the even particle distribution during the dispersion process.

(ii) However, the mechanical properties were found to decrease with an increase in addition of lead content. This could be because of the softer nature of lead and the agglomeration of the particle around the B_4C particles as well as around the matrix.

(iii) The mechanical properties of the trilayered sample exhibited different trend. The sample with B_4C cladding showcased better performance than the sample with Pb cladding. It was because of the softer core with a harder cladding. Furthermore, the major phenomenon of the failure of the specimen is brittle failure.

(iv) The trilayered sample with B_4C cladding showcased poor mechanical properties which may be because of the hard and brittle nature of B_4C.

(v) On the other hand, all the samples exhibited good specific wear properties. The single-layered composites with 20% Pb and 20% B_4C evidenced the better wear performance compared to all the other single-layered samples.

(vi) The reason for the wear performance could be the self-hindering property of the wear debris which was evident from the worn surface analysis. The wear debris had followed deep and long tracks which shows the strong interfacial bonding of the particles around the matrix.

(vii) Furthermore, the trilayered samples with B_4C cladding exhibited good wear resistance as the hard B_4C cladding hindered the propagation of wear tracks to the core.

(viii) On the other hand, the sample with Pb cladding showcased poor wear resistance due to the soft nature of the Pb cladding.

(ix) Moreover, from the wear surface analysis, it is evident that the surface fatigue, grooving, and crazing were the reasons of failure of the samples due to wear.

Data Availability

No data were used to support this study.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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