

Tribolayer Behaviour and Wear of Artificially Aged Al6061 Hybrid Composites

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ABSTRACT – The current work focuses on enhancing wear resistance due to the presence of reinforcements and the effect of ageing treatment on hybrid composites of Al6061-SiC+B₄C. By varying weight percentage, two kinds of reinforcements, viz. silicon carbide and boron carbide, were prepared for hybrid composites by the liquid state process known as the method of stir casting. The solutionising temperature of 550 °C for 2 hours and ageing temperature of 100-200 °C at different time intervals were used for both Al6061 alloy and its composites during heat treatment. Microstructural and mechanical characterisation were carried out using a standard testing procedure. Compared to Al6061 matrix alloy, artificially peak aged composites show 100-140% improvement in hardness due to harder reinforcements and precipitation of solute rich secondary phases during ageing treatment. Overall, an 80-100% increase in wear resistance observed during peak ageing of hybrid composites. Analysis of Al6061 matrix alloy wear out surface shows extensive grooving and ploughing of the surface with the matrix material smear at many spots. The presence of tribolayer in Al6061-SiC+B₄C composites shows a smoother surface than the Al6061 matrix alloy, which results in an excellent lubrication effect during an improvement in wear resistance. The wear surface of base aluminium alloy doesn't show the existence of iron in the tribolayer. The research work is significant in forming a thermally activated wear-resistant metallic tribolayer with good tribological properties.

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INTRODUCTION

For decades Al6061 alloy has been extensively utilised in automobile and aerospace applications due to its moderate temperature, high strength, and excellent corrosion resistance. Aluminium, copper, lead, magnesium, nickel, and titanium, and their alloys are significant non-ferrous metals and alloys. Age hardening is the prominent heat treatment process used to increase the specific strength of several aluminium alloys [1–3]. The primary purpose of ageing treatment of these alloys (Al-Mg-Si) is to obtain a systematically growing enormous number of uniformly distributed fine intermediate precipitates (Mg₂Si) in the matrix. The formation of the critical-sized stable intermediate phase during the chain transformation is known as coherent and semi-coherent stages responsible for improved wear properties [4, 5]. From the last two to three decades, efforts have been focused on exploring lower density reinforcements for developing good tribological composites. Hybrid metal matrix composites (HMMC) have several tasks like deprived wettability and indecorous distribution of reinforcements.

Attaining homogeneous dispersal of reinforcement during the manufacturing of HMMC is the essential primary work. The manufacturing techniques of HMMC portray a vital role in upgrading the mechanical and tribological properties. Compared with powder metallurgy, stir cast composites characteristics indicate a higher strength [6]. The comprehensive literature shows that boron carbide (B₄C) is an alternative for composites reinforced with silicon carbide (SiC) due to better interfacial bonding with matrix alloy. Applying HMMCs in aerospace components and automobile engine parts viz. piston, driveshafts, brake rotors, and cylinders consequently creates interest in studying the tribological effect of the structural components.

It is reported that the interfacial bonding between the aluminium matrix and B₄C reinforcement is better than that of SiC reinforced composites [7, 8]. The presence of reinforcements demonstrates a major increase in wear resistance and reduces the degree of effective contact between composite surface and counter surface asperities, leading to a decrease in the wear rate of composite surfaces [9]. The formation of a stronger tribolayer in composite shows the presence of iron (Fe) and aluminium oxide when compared to the base alloy. Rosenberger et al. [10] studied the formation behaviour of tribolayer during wear test of aluminium metal matrix composites. Enhancement in wear resistance observed due to the existence of tribolayer formed at the specimen's contacting surface by the elements present at the counterface disc material. During the wear test, the reinforced particles pick up iron from the counterface, and these iron particles undergo oxidation and are transferred onto the composite surface. Tang et al. [11] focused on tribological properties of aluminium composites reinforced with B₄C particulate produced by cryo-milling and consolidating pressing. Tribolayer is observed due to material transfer from steel disc (iron and oxygen) to the pin surface. The friction coefficient between the base alloy and hard steel disc is insufficient to diffuse the iron (Fe) atoms to the base alloy. So it requires a high frictional coefficient at the interface. The hard reinforcements present in the composites help this process by increasing the interface

temperature. Compared to the unreinforced aluminium matrix, the presence of reinforcement particles and intermediate phases in composites reveals a lower coefficient of friction.

Limited research work is observed in studying the improvements in wear resistance and effect of tribolayer on artificially aged Al6061 HMMCs. In view of the above, it is of attentiveness to analyse and compare the wear resistance of the Al6061-SiC+ B₄C HMMCs with Al6061 based alloy. To improve wear resistance of the composites, the novelty of the present work is to explore the importance of the formation of hard metallic tribolayers at the interface. The current research focuses on studying tribological properties and tribolayer formation of the Al6061-SiC+ B₄C HMMC's during artificial ageing.

METHODOLOGY

Scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX) of SiC and B₄C (of 35-40 µm size) were conducted for the present work shown in Figure 1 and 2 respectively. The appearance of carbon peaks in B₄C is due to the inexorable carbon impurity on the surface during the commercial B₄C particles synthesis (as in Figure 2). The stir casting method is used to manufacture Al6061-SiC+ B₄C composites by varying the wt%. of SiC particles range of 3,4 and 5 wt% and B₄C particulates of 1, 2, and 3 wt%. Aluminium alloys were melted in a furnace at 740 °C, and the whole melt was degassed by adding a dry hexachloroethane (C₂Cl₆) tablet. The B₄C and SiC particles were preheated at 300 °C and 750 °C for 2 hours to increase the wettability of the reinforcement surface [2]. B₄C and SiC particles were poured into the vortex created during stirring in varying proportions. The specific details of hybrid composite compositions are shown in Table 1. The total weight percentage of reinforcing particles for all HMMCs was limited to 6 wt. % preclude improper distribution and agglomeration during stirring. The stirring speed was sustained at 200 rpm for 10 minutes, and the melt was then poured into cast iron moulds.

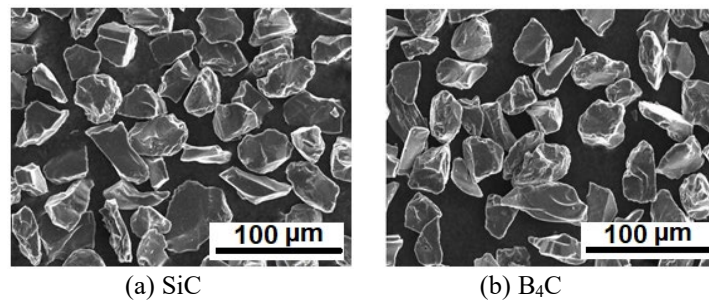


Figure 1. SEM micrographs of SiC and B₄C reinforcement particles used in the present work.

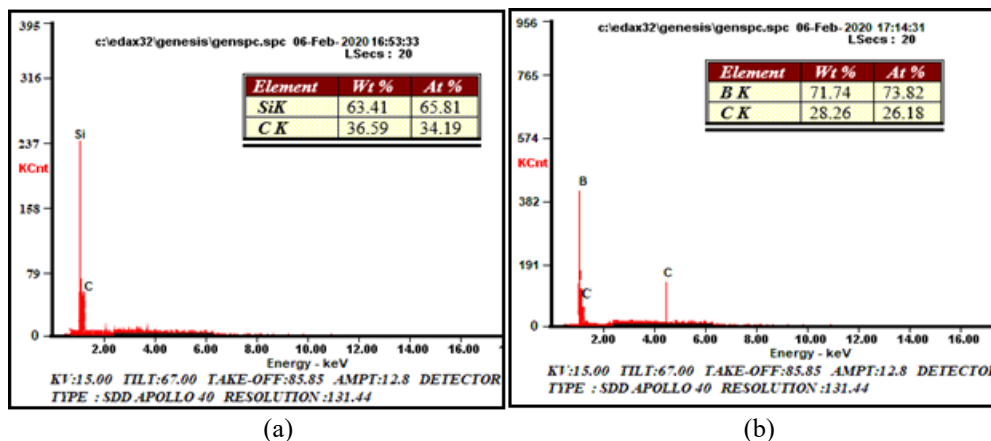


Figure 2. EDXS analysis of (a) SiC and (b) B₄C reinforcement particles.

The as-cast specimens are solutionised at 558 °C for 2 hours and artificially aged at 100, 150, and 200 °C for different time intervals. It is stated that the 6061Al / SiC composite samples, with a solution heat-treated at 558 °C, show better strength compared to the 530 °C sample solution after ageing treatment [12]. Brinell hardness tests were carried out for both as-cast and age hardened HMMC's in adherence to ASTM E10-00 standard. Both as-cast and age hardened Al6061 alloy and its HMMCs were subjected to Brinell hardness test (BHN-ASTM E 10-00) and wear test (ASTM G-99), using DUCOM pin-on-disc machine (India make; Model: TR-201CL) with a disc size of Ø100×6 mm thickness, constant track diameter with a sliding distance of 60 mm and 60 HRC material (EN-31). Three different loads of 15, 30, and 45 N, and speeds of 150, 300, and 450 rpm, were applied via a deadweight loading system during the wear test. The sample sizes used for wear tests were 10 mm in diameter and 40 mm in height. SEM micrographs of the worn surface were taken at the end of each test.

Table 1. Designation of composition and description of Al6061 HMMCs

SiC (S) and B ₄ C (B) combinations	wt%. of reinforcements with Al6061 alloy
Al6061-BS15	Al6061- 1 wt.% B ₄ C + 5 wt.% SiC
Al6061-BS24	Al6061-2 wt.% B ₄ C + 4 wt.% SiC
Al6061-BS33	Al6061-3 wt.% B ₄ C + 3 wt.% SiC

RESULTS AND DISCUSSION

Microstructural Analysis

Figure 3 to 5 show the SEM images and EDX of Al6061-BS15, BS24, and BS33 HMMC, respectively. Irrespective of composition, all HMMCs show homogeneous distribution of the reinforcements in the matrix alloy. Also, the presence of reinforcement agglomeration in the matrix is not revealed in the microstructure. Hence, efficient stirring action and appropriate process parameters during MMC manufacturing can be related to this microstructural analysis.

Effect of Reinforcement and Ageing Temperature on Hardness Properties

The matrix Al6061 alloy and HMMCs were measured for Brinell hardness number (BHN). Figure 6 shows the as-cast and artificially aged peak hardness values under different ageing temperatures. Compared to the Al6061 matrix alloy, the presence of reinforcements in the as-cast HMMCs demonstrates significant hardness improvements. As-cast BS33 HMMC showed an increase in BHN value of 92 BHN than the as-cast matrix alloy, BS15, and BS24.

During the solidification process, HMMCs result in higher dislocation density due to different thermal expansion coefficient of SiC, B₄C reinforcement, and matrix alloy [2]. This promotes higher internal residual thermal stress and strain by changing the alloy matrix microstructure and enhancing the property of the composite. The matrix plastic deformation accommodates the reinforcement results in increased dislocation density, resulting in an additional composite strength increase. It was apparent that SiC and B₄C particles in the Al6061 alloy accelerated the ageing kinetics compared to unreinforced alloy during age-hardening treatment. This is primarily due to the existence of areas with a large dislocation concentration between the matrix alloy and the reinforcement interface, which contributes to heterogeneous nucleation sites and a high diffusivity path for the precipitation of secondary rich precipitates [3, 13, 14].

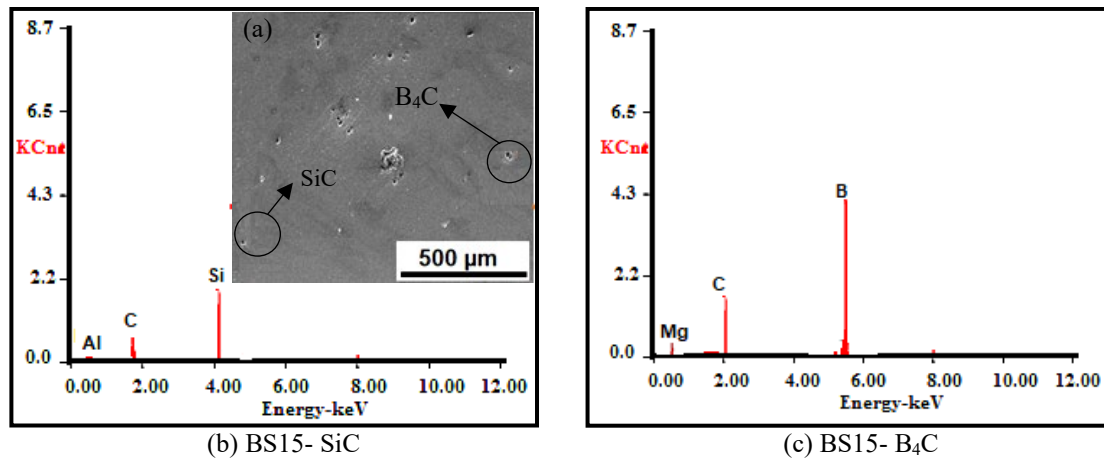


Figure 3. (a) SEM micrographs of BS15 HMMC and the corresponding EDX of (b) SiC and (c) B₄C.

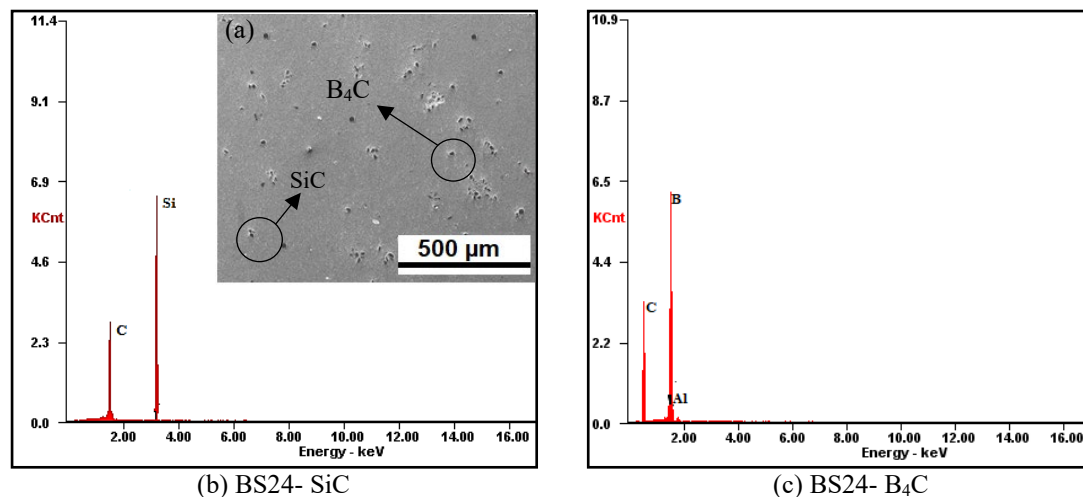


Figure 4. (a) SEM micrograph of BS24 HMMC and the corresponding EDX of (b) SiC and (c) B₄C.

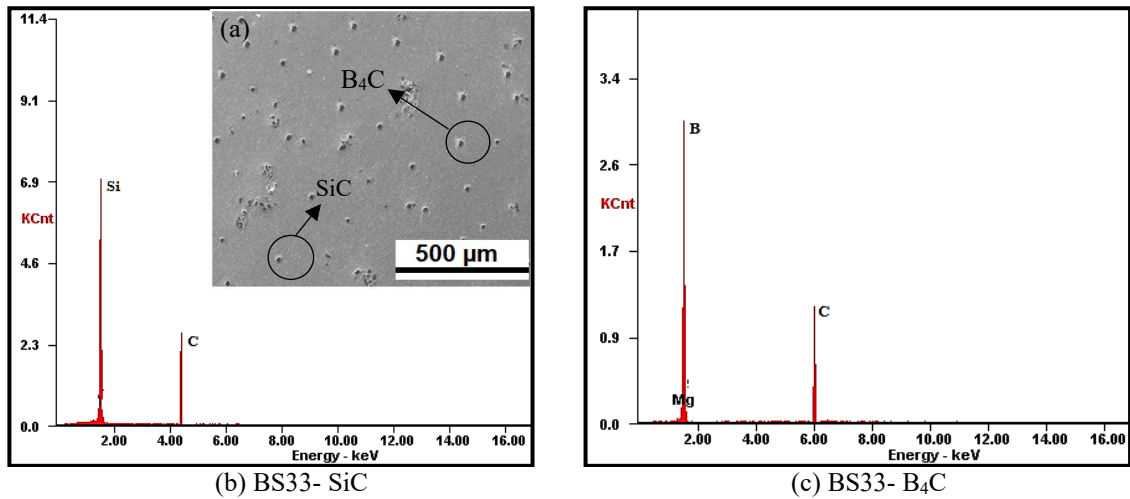


Figure 5. (a) SEM micrographs of BS33 HMMC and the corresponding EDXS of (b) SiC and (c) B₄C.

Similar to Al6061 matrix alloy, HMMC is very profound to age hardening regardless of 100 or 200 °C temperature. Ageing at 100 °C in both the base alloy and the HMMC showed the highest peak hardness values. Hence, lower ageing temperature results in more finely dispersed intermetallics obtained by the number of metastable transition stages. During the formation of transition phases, the lattice is getting strained each time, which reflected as property enhancement, especially hardness [2]. Lower temperature ageing (at 100 °C) contributed to the increased hardness; due to an escalation in the number of intermediate regions during ageing treatment. With increasing B₄C and reduction in SiC wt. %, the hardness was found to increase in both as-cast and aged specimens. The higher the ageing temperature (at 200 °C), the faster is the diffusion kinetics.

The kinetics of the diffusion process is always temperature-dependent, and the Arrhenius-type equation diffusion coefficient is related to the temperature [15].

$$D = D_0 \exp\left(-\frac{Q}{RT}\right) \tag{1}$$

where, *Q* is activation energy (cal/mol) for diffusion, *D*₀ is the pre-exponential term (diffusion coefficient at 1/*T*=0 or *T*=∞), *R* is gas constant at 1.987 cal/mol·K, and *T* is absolute temp (K).

The diffusion coefficient, *D*, increases with the flux of atoms and temperature of the material. The energy supplied for the diffusion of the atoms at higher temperatures overcomes the obstacle for activation energy, which results in the atomic arrangement of atoms at new sites. As per the general rule, it may be stated that the diffusion coefficient doubles for every 20 °C rise in temperature. If the diffusion rate was faster, the time required to form the semi-coherent precipitates from the supersaturated solid solution is lesser. Hence time required to attain peak hardness is lesser [16, 17]. The density of dislocation largely depends on the size of the reinforcements used in hybrid composites. While the sizes of both B₄C and SiC were the same in the current research (35~40 μm), a higher B₄C volume fraction of reinforcement particles results in a smaller inter-particle distance and higher density of dislocation than SiC particles [18, 19]. Given the above explanation, Al6061-BS33 HMMC shows excellent peak hardness of 168 BHN at 100 °C ageing temperature (as shown in Figure 6).

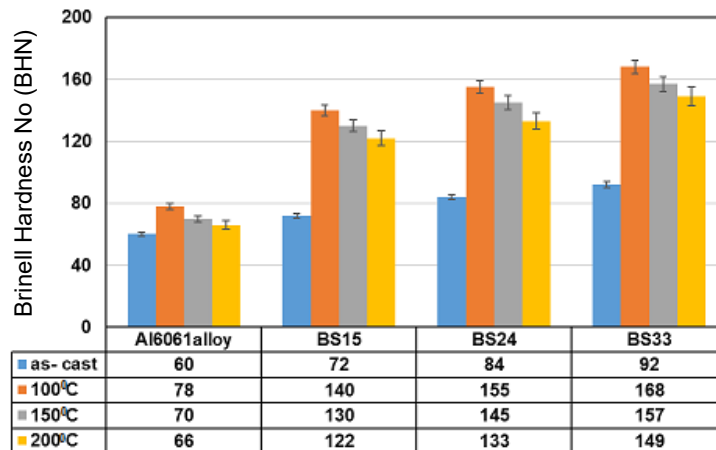


Figure 6. As-cast and artificially aged hardness of Al6061 alloy and HMMCs.

Tribological Characteristics

HMMC used for tribological applications should withstand higher wear resistance, low friction coefficient, and able to resist load without undue distortion. The wear performance of HMMC mainly depends on microstructural changes, type of matrix alloy, the quantity of reinforcements, and process parameters viz. applied load, sliding speed, reinforcements type [20]. It is a well-known fact that the presence of harder reinforcements shows the considerable improvement of the HMMC. Variation in wear resistance with reinforcements addition at different load on Al6061 alloy and its HMMCs is shown in Figure 7. In general, the wear resistance of the Al6061 alloy and its HMMC decreases with an increase in the applied specific load. The phenomena of decrease in wear resistance are mainly accredited to the more considerable extent of plastic deformation, which in turn results in the formation of wear debris. The degree of plastic deformation raises subsurface cracking risk, leading to significant material removal [21, 22].

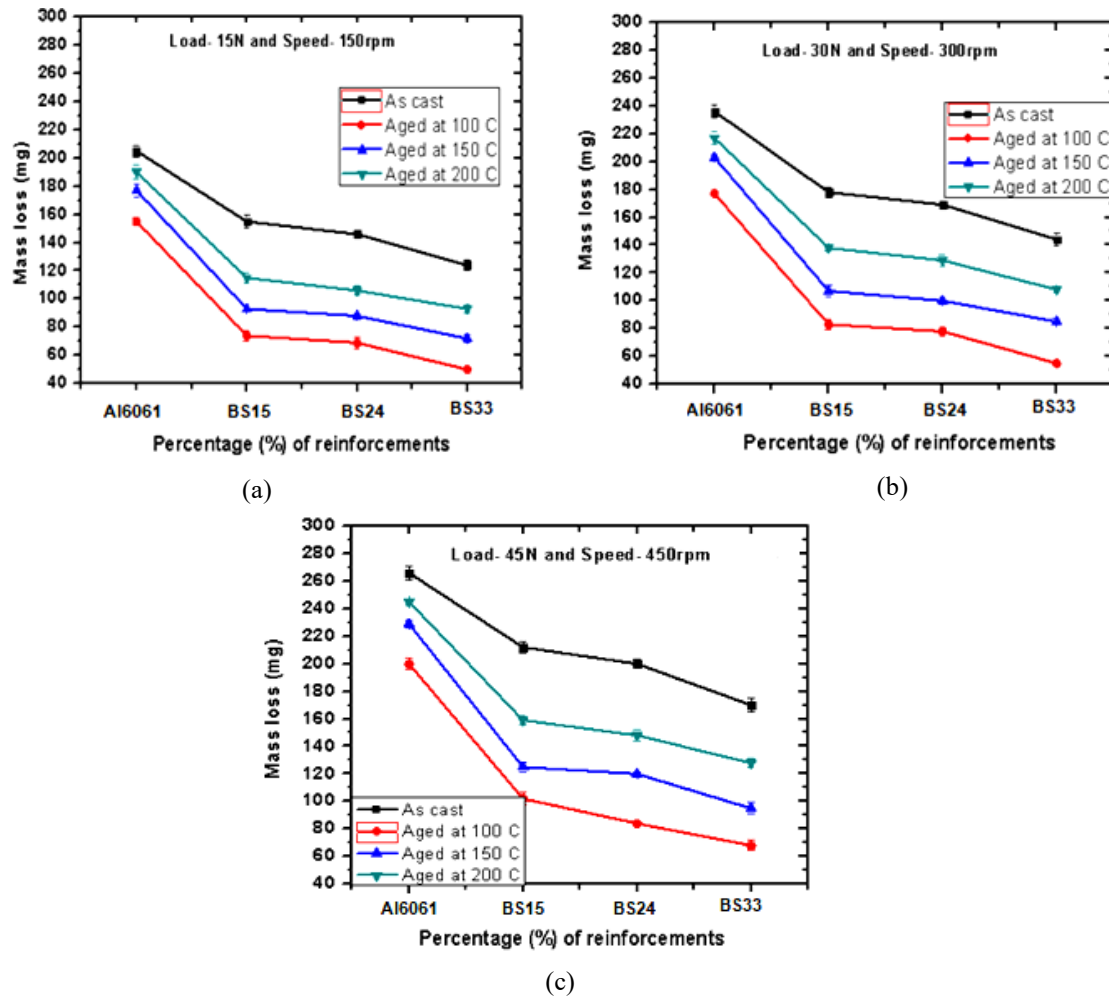


Figure 7. Mass loss variation with the increase in B_4C percentage of as-cast and artificially peak aged Al6061 alloy and HMMCs under different load and speed condition of (a) 15 N, 150 rpm (b) 30 N, 300 rpm (c) 45 N, 450 rpm.

According to Uvaraja et al. [23, 24], the metal surface undergoes oxidation by forming oxide films during the wear test, affecting friction resistance. At lower load of 15 N, the metal-metal contact reduces due to lower ductility and shear strength of the oxide films, which leads to higher wear resistance due to a lower coefficient of friction. With an increase in load from 15 to 45 N, oxide films presence fails to prevent metallic contact, and oxide films may get damaged, leading to escalation of friction coefficient, which in turn reduces the wear resistance. It is also observed that the wear resistance of the Al6061 alloy matrix is minimum irrespective of load and speed under all testing conditions. The trend of wear resistance decreases with increased applied load, owing to direct metal to metal interaction resulting from large-scale plastic deformation during wear test. In the case of HMMCs, penetration depth by the harder asperities of hardened steel disc is primarily superintended by the protruded reinforcement particles. Also, reinforcement particles in HMMC carry a significant portion of the applied load in the alloy matrix. The amount of material removal rate reduces mainly due to the existence of SiC and B_4C reinforcements, which support the metal to metal contact stresses by averting a higher rate of plastic deformation and abrasion between the contact surfaces [25]. The increase in wear resistance of Al6061-BS33 HMMC's, even at higher loads, can be attributed to the fact that SiC and B_4C being hard refractories may contest themselves to the abrasion resulting in increased wear resistance and decreased in seizure resistance of HMMC's. The presence of reinforcements in Al6061 alloy resists the delamination process and has excellent lubricating properties, leading to improved wear resistance [26].

In Figure 7, Al6061-BS33 HMMC shows better wear resistance even at higher loads compared to the Al6061 matrix alloy. Enhancement in wear resistance is attributed to the fact that B₄C itself being hard and contains traces of beneficial free carbon (graphite), which may serve as a lubricating agent and provide a cushioning effect between the contacting surfaces; thereby resulting in lower wear and seizure resistance, as shown in Figure 2(b). Moreover, a huge difference in the density of two reinforcements (B₄C is lighter) increases the quantity of lighter reinforcements and contributes to more volume percentage in the composites. Therefore increase in reinforcement quantity increases the interface density for the nucleation of intermetallics [7].

Al6061 alloy and HMMCs show substantial improvement in wear resistance during ageing treatment. Peak aged specimens at a lower temperature (100 °C) show superior wear resistance. With increasing ageing temperature from 100 to 200 °C, the wear resistance of the Al6061 alloy or HMMCs decreases. Ageing at 100 °C (lower ageing) results in a higher rate of coherency strain in the matrix alloy with the precipitation of finer secondary solute rich phases (intermediate phases) with lesser interparticle distance. Whereas, ageing at 200 °C results in lesser hardness due to the minimum number of intermediate phases by reducing the lower strain rate in the alloy matrix during the formation of coherent precipitates [5, 21, 27]. The precipitates formed during ageing treatment are presumed to improve the interfacial bond between the reinforcements and the matrix and serve as refractory materials to improve wear resistance by enhancing composites hardness [27–29].

Al6061 alloy and HMMCs show less wear rate at lower load and speed than higher load and speed conditions. The wear rate at 15 N with sliding distance and ageing conditions is not too sensitive. This is mainly due to the lower intensity of strain hardening at lower loads. Whereas at 45 N, substantial changes perceived by decreasing the wear resistance. Irrespective of all speeds, there is a decrease in wear resistance with an increase in normal load from 15 to 45 N. As the load on the pin increases, superior debonding and fracture occur due to an increase in the force of friction. The surface layer of the pin breaks during the initial stage and augments the strain hardening effect by increasing the area of contact between the pin and disc surfaces. An increase in frictional force leads to higher metal removal, mainly due to the rise in temperature between the contact surfaces. This effect results in adhesion and increases deformation strength on the pin surface, leading to further loss of material. Minimum wear loss was observed at 150 rpm, whereas an increase in speed and load results in a drastic increase in wear loss. This observation is mainly due to the softening effect of material with an increase in frictional force and temperature. Ageing treatment and reinforcement combination (SiC and B₄C) is beneficial for the overall improvement in wear behaviour of Al6061-BS33 HMMC. Improvement in wear resistance of HMMCs in as-cast and age hardened at 100 and 200 °C compared to Al6061 alloy can be summarised as: 60-90% increase in wear resistance aged at 200 °C and 100-200% (BS15, BS24, and BS33) aged at 100 °C. Figure 8 to 11 show SEM wear surface of Al6061 matrix alloy, Al6061-BS15, BS24, BS33 HMMCs at 45 N with the speed of 450 rpm.

The Al6061 alloy surface shows erosion wear, as in Figure 8(a) and is primarily due to metal-to-metal contact between the base alloy and disc counterface. The combination of higher sliding speed and stresses generated during wear test leads to increased frictional energy, which causes softening of the matrix by increasing the metal removal rate. The morphology of the wear surfaces depends on the sliding speed and applied load conditions [21]. Extensive plastic grooving and ploughing with massive surface damage at many spots are reflected in the Al6061 alloy with the smearing of the matrix material. The grooves of wear surfaces are coarse, and randomly oriented serrations are formed due to the severe plastic deformation at the edges. This pattern is due to the lower strength, hardness, and less strain hardening effect of base alloy compared to HMMCs.

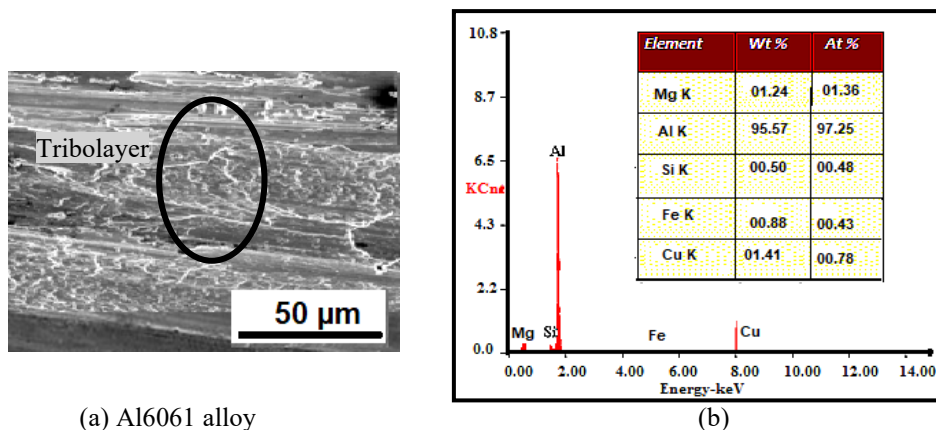
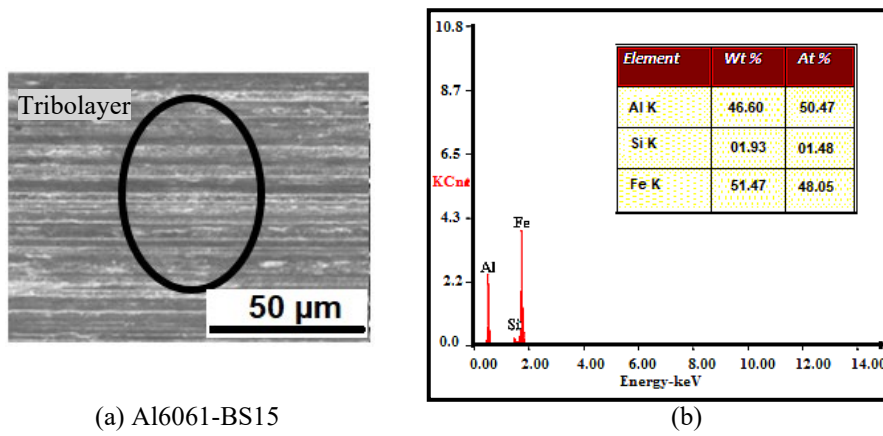


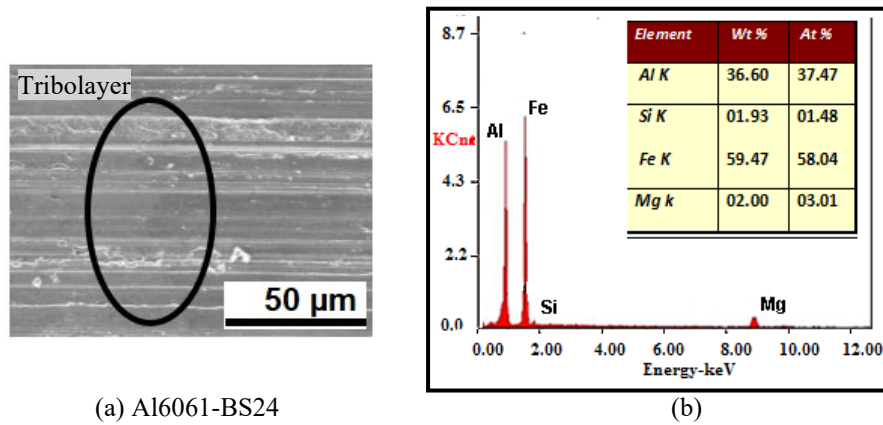
Figure 8. (a) SEM micrograph and the corresponding (b) EDX of Al6061 alloy wear surface.



(a) Al6061-BS15

(b)

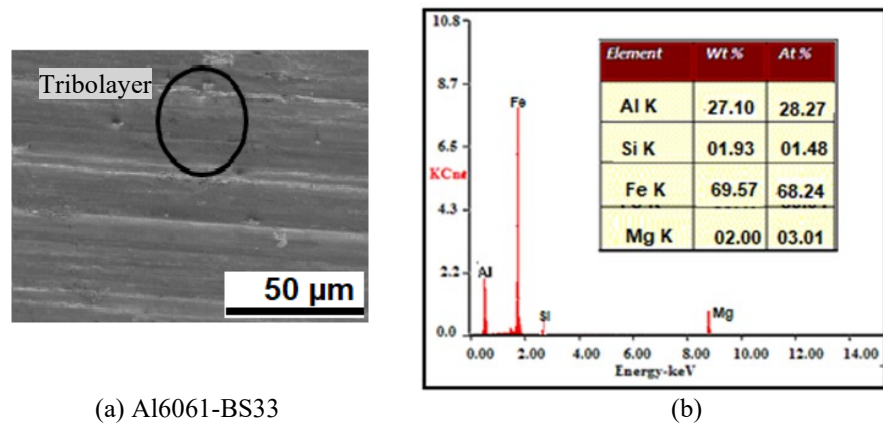
Figure 9. (a) SEM micrograph (b) corresponding EDX of Al6061-BS15 HMMC wear surface.



(a) Al6061-BS24

(b)

Figure 10. (a) SEM micrograph (b) corresponding EDX of Al6061-BS24 HMMC wear surface.



(a) Al6061-BS33

(b)

Figure 11. (a) SEM micrograph (b) corresponding EDX of Al6061-BS33 HMMC wear surface.

The wear surface of all three HMMCs in Figure 9 to 11 shows tribolayer (TL), which is moulded due to the transfer of counterface steel disc material. EDX analysis of the wear surface of the three HMMCs shows a higher concentration of Fe, which may come from the counterface. The EDX spectra clearly show TL formation at the wear surface and abrasion of the counter surface. The existence of hard SiC and B₄C reinforcements withstand the applied load and scratch profoundly on the counter surface. While the pin is in contact with the counterface, TL formation on the pin surface is mainly due to the severe churning process and plough out of the iron material from the counterface [10, 24, 30]. TL presence converts the two-body wear into three-body wear patterns and acts as a solid lubricant, decreasing the wear rate of the composite [31]. Al6061 matrix alloy does not reveal any traces of TL, and the EDX pattern also shows no Fe concentration on the wear surface, as in Figure 8(b). Since TL hardness is three to five times higher than matrix alloy, Al6061 HMMCs show superior wear resistance compared to Al6061 alloy [10]. Therefore, the wear resistance of Al6061 matrix alloy improved due to the addition of reinforcements. The wear surfaces of all three HMMCs are relatively smooth when compared with the Al6061 alloy. The chopping off reinforcement particles over the entire area also observed. Cavitation seems to be low, but cracks and furrows are noticed in Al6061-BS15 HMMC (as in Figure 9).

Some particles were chopped off during sliding, and smaller particles in some areas have emerged from the Al6061-BS24 composite matrix (as shown in Figure 10). Finer shallow grooves, plastically deformed at the edges, also noticed

in Al6061-BS33 (of Figure 11). In all HMMCs, less surface damage and the creation of fine grooves aligned in the direction of sliding observed with fewer surface cracks. The presence of TL shows a smoother surface compared to Al6061 alloy (as in Figure 9 to 11). Dislocation density generated during casting also depends upon the interface area formed by the reinforcements. The finer the reinforcements are, the more total interfacial area is, which increased the dislocation density and results in a large strain hardening effect [2, 11, 20].

CONCLUSION

In this work, the main objective is to increase the hardness and wear resistance of hybrid reinforced composites through precipitation hardening treatment. Al6061 alloy and HMMCs positively respond to age hardening treatment with considerable improvement in hardness values. Generally, in contrast with as-cast alloy, increased hardness is observed during peak ageing of hybrid reinforcement composites, respectively. Al6061 alloy shows substantial improvement in wear resistance with the addition of reinforcements. Peak aged samples of alloy and its composites (BS33) display superior wear resistance. Overall, 10-90% and 60-200% increase in wear resistance observed in peak ageing of hybrid reinforcement composites compared to as-cast alloy. The presence of tribolayer on the composite wear surface is the main role player in regulating the composites wear properties. Finally, the Al6061-BS33 hybrid composite that aged at a lower temperature of 100°C displays higher mechanical and tribological properties in appraisal with Al6061 matrix alloy, BS15 and BS24 HMMCs.

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