

Dry sliding-friction and wear behavior of hot-extruded Al6061/Si₃N₄/C_f hybrid MMC

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Abstract

The effects of reinforcement addition and hot extrusion on the microstructures, micro hardness, friction, and wear behavior of aluminium (Al) hybrid composite were investigated. Al6061 dispersed with electroless nickel-coated Si₃N₄ (6wt.%) and copper-coated carbon fiber (C_f) (1wt.%) hybrid composites was developed through stir casting followed by hot extrusion. Optical micro structural studies confirmed that the size of reinforcements decreased, and their orientations were in the extrusion direction. The decrease in the grain size (29%) of hybrid composites was larger than that in the grain size of matrix alloys under hot-extruded conditions. The synthesized hot-extruded Al6061 hybrid composite exhibited a lower coefficient of friction (51%) and high wear resistance (39%) compared with the hot-extruded Al6061base alloy.

Key Words: Hybrid composite, hot extrusion, Si₃N₄, carbon fibers, coefficient of friction (COF), wear rates.

1. Introduction

Aluminium (Al) alloy-based metal matrix composites (MMCs) dispersed with particles and fibers are widely applied in several fields such as aerospace, automotive, high precision military because of their excellent tribological properties. However, challenges faced during secondary forming processes, such as forging and extrusion, limit their applications. Among all of Al alloys, Al6061 is the most demanded matrix alloy used for fabricating MMCs because of its excellent formability [1-2]. By contrast, aluminium oxide (Al₂O₃), silicon

carbide (SiC), silicon nitride (Si_3N_4), and carbon fibers (C_f) are the most preferred reinforcements in Al alloy-based MMCs [3-5].

Studies have reported adding Si_3N_4 in Al alloys improves their mechanical properties such as hardness and wear resistance [6-9].

Veeresh kumar et al. reported the tribological characteristics of Si_3N_4 -reinforced Al6063 composites. An increased load and sliding distances caused a higher volume wear loss in both a base alloy and composite. However, Al6063/10wt% Si_3N_4 exhibited an excellent wear resistance compared with unreinforced alloys [10]. Chenxu zhang et al. reported the mechanical and tribological behavior of Al- β Si_3N_4 whiskers developed through powder metallurgy. With an increase in the whisker content the wear rate of composite decreased, and the formation of mechanical mixing layer (MML) was reported to cause reduction in both the coefficient of friction (COF) and wear rate of composites [11]. Mir Irfan Ul Haq and Ankush Anand reported the friction and wear behavior of Si_3N_4 /AA7075 composites. An improved wear resistance and a reduced COF, especially at a high load of 50 N, for developed composites was achieved [12].

C_f has been widely used as reinforcement in Al alloys because of its high-specific modulus, high-specific strength, low expansion coefficient, excellent lubrication properties, wear resistance, and high thermal and electric conductivity [13-15].

Anil Alten et al. fabricated nickel-coated carbon fibers reinforced with Al6063 composites by using squeeze casting. An increased in the coating thickness of nickel from 0.9 to 4.2 μm caused a decrease in the impact strength [16]. Sree Manu et al. developed self-lubricating bi-directional C_f reinforced Al composites by using squeeze infiltration [17]. Xiong Cao et al. investigated the tribological behavior of AA5052- C_f composites fabricated through friction stir welding. Adding C_f in Al prevents the nucleation and propagation of micro cracks, leading to an improvement of tribological properties. Additionally, MML formation is a factor contributing to the improvement of the wear resistance [18].

To obtain more satisfactory bonding and to minimize reaction at the matrix-reinforcement interface, some researchers have employed electroless nickel/copper coatings around reinforcements [19, 20]. The toughness coupled with higher load-bearing capacity of copper-coated carbon fibers is higher than that of carbon fibers deposited with nickel. C_f deposited with nickel exhibits low toughness because of the formation of brittle phases at the interface of a base alloy and fiber [13, 21]. Thus, to overcome this problem, in this study, copper-coated C_f was employed to synthesize hybrid MMCs. Metallic coatings on reinforcement will enhance the bond strength and reduce the interfacial reaction, which in turn substantially improved the yield strength leading to superior tribological properties.

Since last three decades, researchers have focused on MMCs preparation using a single reinforcement. However, these MMCs exhibit seizures at high speeds and some other drawbacks such as low load-bearing capacity, poor machinability, and poor thermal conductivity. To overcome these limitations, researchers are currently investigating the hybridization of composites reinforced with particulates and fibers such as Si_3N_4 and C_f . Studies have reported considerable improvement in the wear resistance of hybrid composites compared with that of conventional composites [22-28]. Furthermore, Ajith Arul Daniel et al. have reported the beneficial effects of hybrid reinforcements in improved tribological properties of Aluminium 5059/SiC/MoS₂ when compared with single reinforcement [29]. Mallikarjuna et al. have reported the beneficial effect of hybrid reinforcements of SiC and MWCNTs in wear resistance of copper composites [30]. Sabry I et al. have reported that use of hybrid reinforcement of SiC-Gr in Al6061 alloy has significantly improved the tribological properties when compared with single reinforced composites [31]. Silicon nitride was selected because of its considerably low coefficient of thermal expansion and excellent wear resistance. By contrast, carbon fibers exhibit a high-specific modulus, and low density coupled with excellent thermal conductivity and tribological properties. Thus, a combination

of silicon nitride and carbon fibers can be incorporated as reinforcements in Al and its alloys to develop hybrid MMCs with high performance, lightweight, and high strength.

Therefore, in this study, Al-hybrid composites were developed by adding electroless nickel-coated Si_3N_4 particles and copper-coated chopped-carbon fibers as reinforcements in an Al6061 alloy matrix by using stir casting followed by hot extrusion. Dry sliding-friction and wear behavior of synthesized hybrid composite were studied.

2. Experimental details

2.1 Synthesis of composites and extrusion

Si_3N_4 powders were coated with electroless nickel adopting the procedure as described in our earlier work [19]. Carbon fibers were coated with electroless copper using the standard procedure as reported by Urena et al. [20].

Al6061 dispersed separately with nickel coated Si_3N_4 (6 wt. %) composite and with nickel coated Si_3N_4 (6 wt. %) and copper coated C_f (1 wt. %) hybrid composite were synthesized using stir casting technique as described in our earlier work [19]. After casting, the composites were hot-extruded with an extrusion ratio of 1:10 using forward extrusion method. The operating temperature and extrusion pressure were 550 °C and 175 kg/cm², respectively.

2.2 Characterization

The grain sizes of all polished hot-extruded samples were determined according to ASTM: E112-96 using an optical microscope (Nikon LV150 equipped with Clemex Image Analyser, Japan)

The microhardness of all specimens was measured under hot-extruded conditions before and after a wear test at 10 g load, and 10 sec test duration by using Vickers micro hardness testing machine. The average of five set of hardness measurements taken at different locations are being reported.

Dry sliding-wear tests were performed for all polished specimens under hot-extruded conditions by using computerized pin-on-disc machine according to the ASTM G99-95 standard. The hot-extruded samples were machined to the required diameter and height of 10 and 20mm, respectively. All specimens tested under hot-extruded conditions were slide against a counter disk comprising steel EN-31of RC 60 hardness. Wear tests were performed at different sliding velocities of 0.314 – 1.884 ms⁻¹, varying loads of 10 – 60N, and a constant time interval of 30 min. These test parameters related to automotive brake drum applications. The relative humidity during tribo tests was maintained at 55%. During the test, the height loss of specimens was measured using linear variable differential transducer of 1- μ m accuracy. In addition, a load cell of 0.1-N accuracy was employed to measure the frictional forces. Wear rates were determined using the equation $w_r=(h.a)/(v.t)$, where w_r , h , a , v and t represents wear rate(mm³m⁻¹), height loss(mm), contact area(mm²), sliding speed(ms⁻¹), t-time(s), respectively.

The composition of worn surfaces of the hybrid composite was measured using an X-ray diffractometer (XRD, D8 ADVANCE Eco, Bruker). The surface characteristics of the hybrid composite was determined using an X-ray photoelectron spectroscope (XPS, Kratos Axis Ultra DLD).

The surface features of reinforcements, initial and worn hot-extruded composites, and hybrid composites were analyzed using a scanning electron microscope (SEM, JOEL JSM 840A). Surface elements were analyzed using an X-ray energy dispersive spectroscopy (EDS) detector.

3. Results and discussion

3.1.1. Characterization of coatings on reinforcements

Figure 1a illustrates the SEM micrograph of uncoated silicon nitride particles. The size of uncoated particles was 1 – 3 μ m and that of nickel-coated Si₃N₄ particles was from 1 – 4 μ m (Fig. 1c). EDS spectrum (Fig. 1d), evidenced the presence of nickel on Si₃N₄ particles.

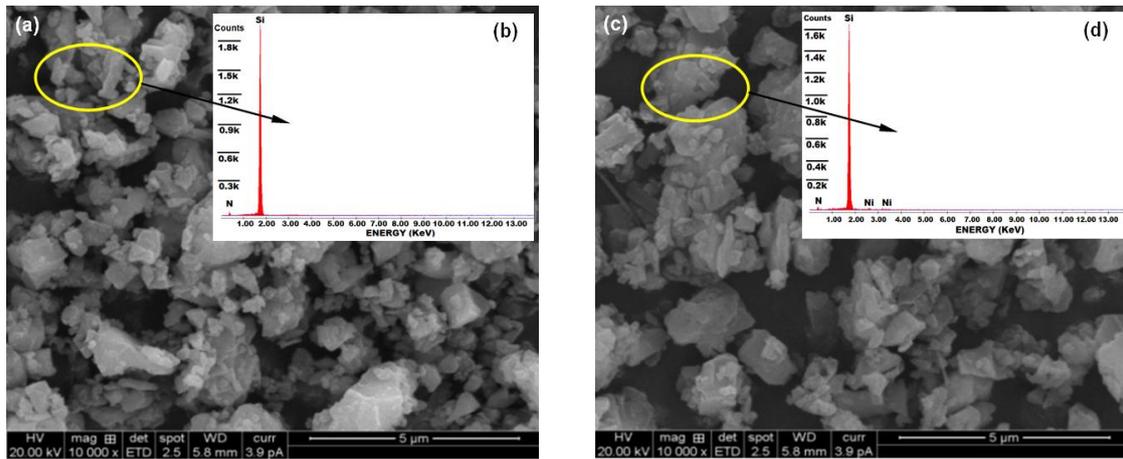


Fig. 1. SEM-EDS images of (a,b) uncoated Si_3N_4 powders and (c,d) Ni coated Si_3N_4 powders

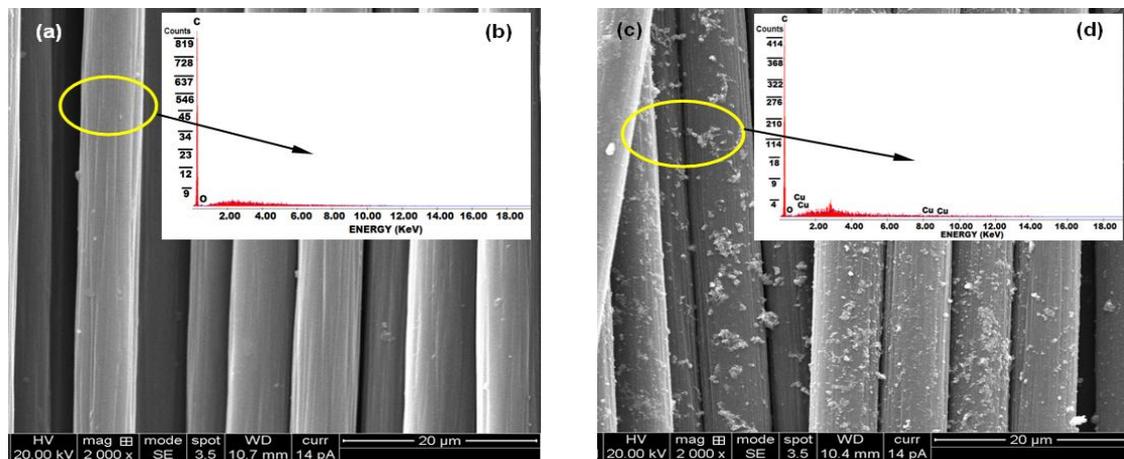


Fig. 2. SEM-EDS images of (a, b) uncoated C_f and (c, d) copper coated C_f

The sizes of uncoated and copper-coated carbon fibers were 7 and 9 μm , respectively, (Fig. 2a and c). EDS spectrum (Fig. 2d), evidenced the presence of Cu which indicated the existence of Cu deposition on carbon fibers.

3.1.2. Microstructural studies

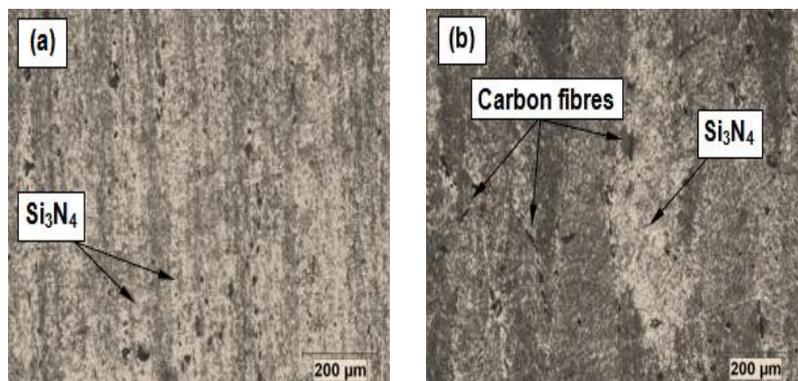


Fig. 3. Optical Photomicrographs of hot-extruded (a) composite (b) hybrid composite

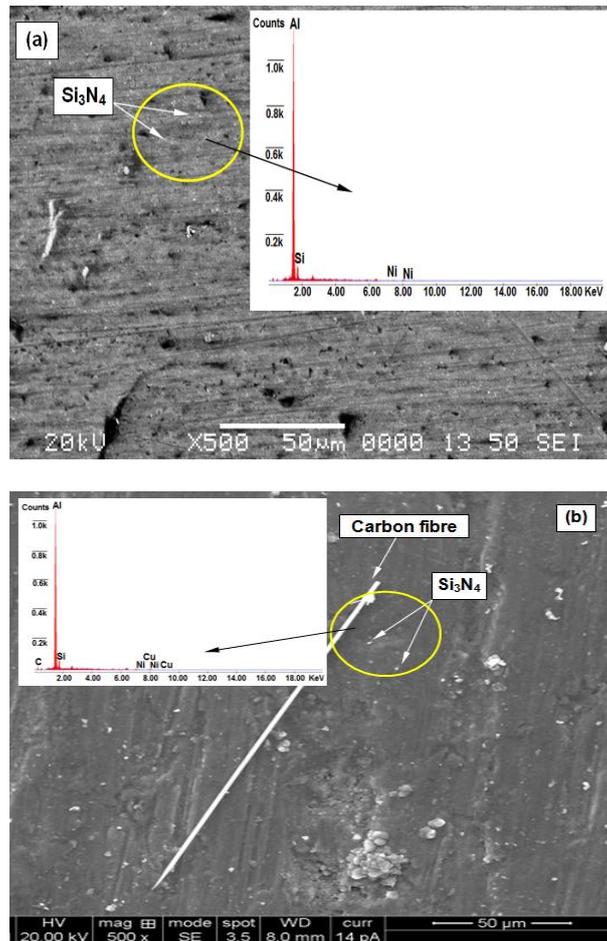


Fig. 4. SEM-EDS of hot-extruded (a) composite (b) hybrid composite

Figure 3a and 3b illustrates the optical micrographs of composites produced under hot-extruded conditions. Reinforcement distribution in the matrix alloy was fairly homogeneous. Reinforcements were oriented along the extrusion direction. During hot extrusion, the high compressive stresses developed between a die and extruded materials resulted in a decrease in the grain size of reinforcements. A small size of reinforcements with a homogenous distribution of particles within the matrix leads to an improved wear resistance. These results are in agreement with the findings of other studies [32]. Figure 4a and 4b presents the SEM with EDS images of composites produced under hot-extruded conditions. Silicon nitride particles were homogeneously distributed. No apparent change in the particle size of silicon nitride obtained after extrusion. Furthermore, Ni and Cu were present on Si₃N₄ and C_f respectively, even after hot extrusion indicating the absence of intermetallic precipitates.

3.1.3. Grain size analysis

Figure 5 (a–c) presents photomicrographs of grain structures with the histograms of Al6061 and its composites under hot-extruded conditions. The average grain size of the hot-extruded Al6061 matrix alloy and hybrid composite was 63.5 and 45 μm , respectively. The grain size of hybrid composites exhibited a decrease of 29% compared with that exhibited by the unreinforced alloy after hot extrusion. This remarkable decrease in the grain size improved the strength of hybrid composite. Similar results have been reported in studies on secondary-processed and cast composites [33, 38].

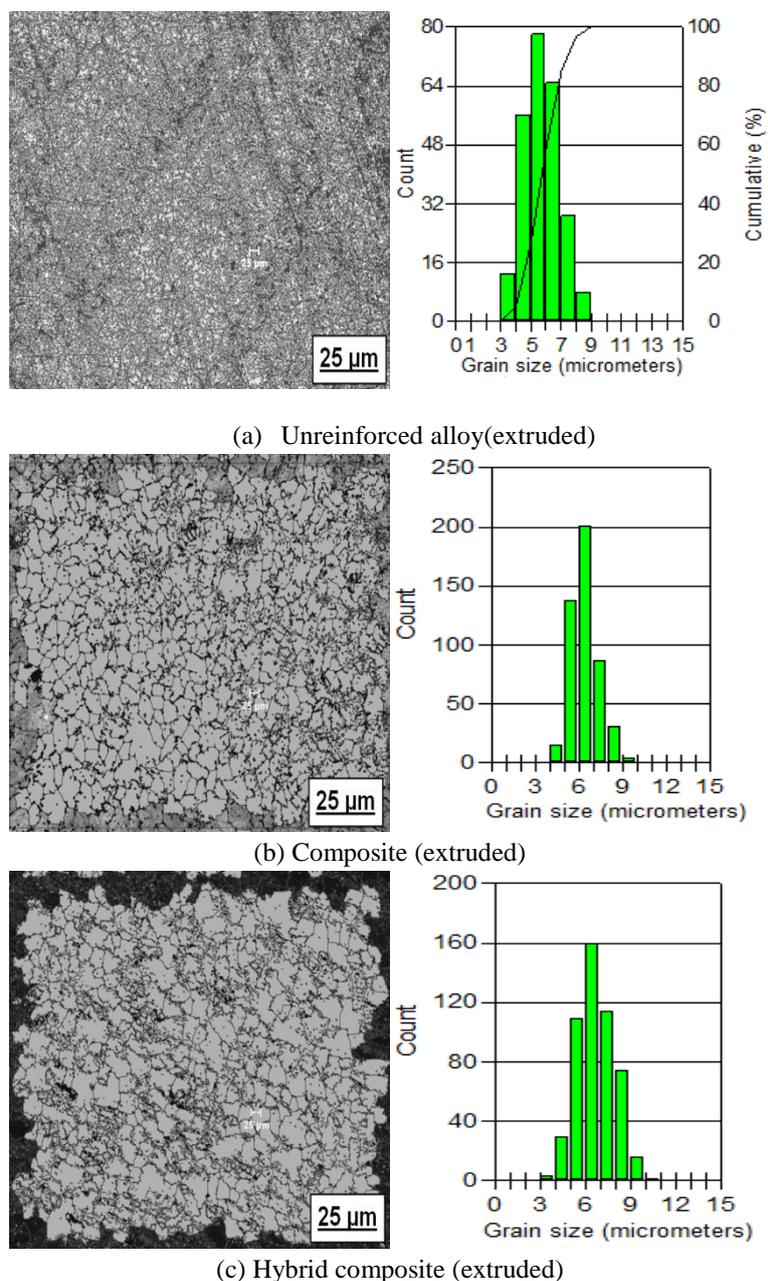


Fig. 5. Photomicrographs indicating grain size with histogram

3.2 Friction and wear studies

3.2.1. Analysis of COF

3.2.1.1. Effect of reinforcements

The COFs at 60-N load, 30-min test duration, and 0.314-ms^{-1} velocity for the extruded base alloy (6061), composite, and the hybrid composite were 0.65 ± 0.03 , 0.63 ± 0.03 , and 0.32 ± 0.03 , respectively. The COF of hybrid composite exhibited a decrease of 51% compared with that exhibited by the base alloy under hot-extruded conditions. The COF of hybrid composite improved under hot-extruded conditions because of the lubricating property of reinforcements (Si_3N_4 and C_f). Silicon nitride is hard; however, after exposure to humidity it forms thin and adherent silicon oxide, which acts as a solid lubricant [34]. The mechanism of wear of Si_3N_4 is predominantly a tribochemical oxidation of Si_3N_4 to silicon oxide in presence of moisture as reported by Gee et al. [35]. Furthermore, during sliding, copper-coated carbon fibers were squeezed between friction pairs, leading to the formation of a lubricating film [36]. In addition, the formed MML comprising metal oxides as indicated by XRD analysis discussed in section 3.2.2.1 was another contributing solid lubricant. A detailed study of MML with a low COF of copper-based hybrid MMCs was conducted [37].

3.2.1.2. Effect of applied load

Figure 6 presents the COF of Al6061 and its composites fabricated under hot-extruded conditions with varying loads. The plots revealed that the COF of an all material system studied under hot-extruded conditions decreased with an increase in the load. With an increase in the applied load, the contact areas at contact surfaces increased which improved the formation degree of thin lubricating films. This phenomenon tended to cause a decrease in COF with the load increase. However, hybrid composites exhibited the lowest COF compared with the base alloy and composite under hot-extruded conditions at all loads. The COF of the hybrid composite exhibited the decrease of 21% and 51% compared with that of exhibited by the base alloy after extrusion at minimum and maximum loads, respectively, of

10 and 60N, respectively. The enhancement in the COF of the hybrid composite at increased loads was caused by the lubricating effects of silicon nitride and carbon fibers [34, 38].

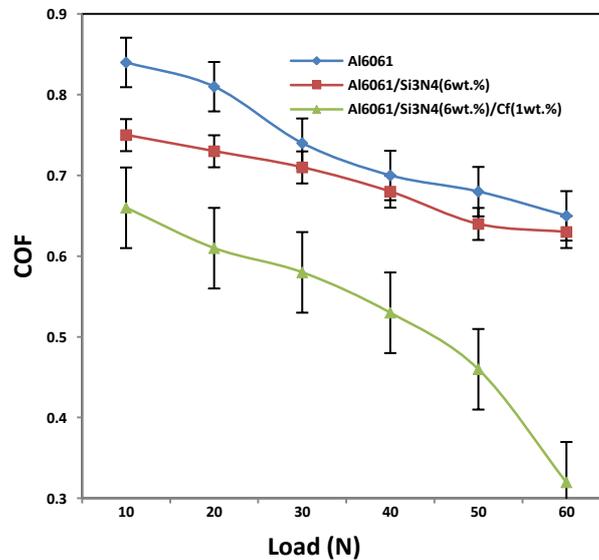


Fig. 6. COF of Al6061 and its produced composites after extrusion as a function of load at 0.314 ms^{-1} velocity and 30 min test duration.

3.2.1.3. Effect of sliding velocity

Figure 7 presents the COF of Al6061 and its composites produced under hot-extruded conditions with different sliding velocities. A decrease in the COF of the all material system studied was obtained under hot-extruded conditions with an increase in the sliding velocity. An increased sliding velocity caused an increase in the temperature of contact surfaces, which led to the softening of materials and formation of thin and adherent tribo films. This phenomenon tended to cause a decrease in the COF with the increase in sliding velocities. However, the COF of the hybrid composite was the lowest among the base alloy, hybrid composite and composite under hot-extruded conditions at all sliding speeds. The decrease in the COF of the hybrid composite of 22% and 13% was obtained compared with that of the base alloy under hot-extruded condition at minimum and maximum sliding speeds, respectively, of 0.314 and 1.884 ms^{-1} , respectively. The enhancement in the COF of the hybrid composite was caused by the improved thermal conductivity of Cu-coated C_f . An enhanced thermal conductivity resulted in the superior heat dissipation from sliding elements reducing softening effects.

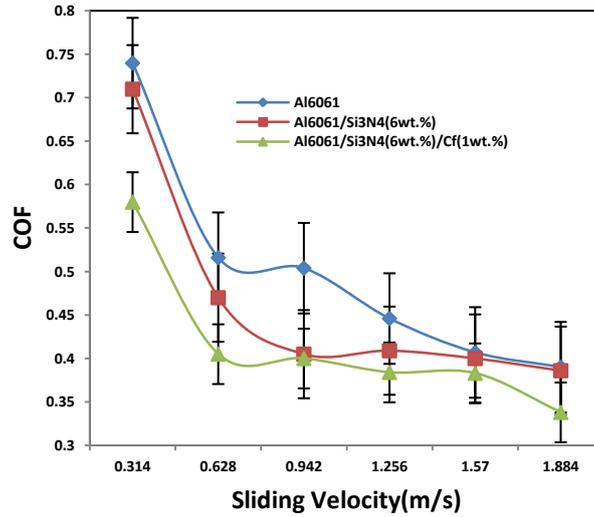


Fig. 7. COF of Al6061 and its produced composites after extrusion as a function of sliding velocity at 30 N load and 30 min test duration.

3.2.2 Analysis of Wear rate

3.2.2.1. Effect of reinforcements

Wear rates at 60-N load, 30-min test duration, and 0.314-ms^{-1} velocity for the extruded base alloy (Al6061), composite and hybrid composite were $2.6 \times 10^{-3} \pm 1 \times 10^{-3}$, $1.67 \times 10^{-3} \pm 1 \times 10^{-3}$ and $1.58 \times 10^{-3} \pm 1 \times 10^{-3} \text{ mm}^3/\text{m}$, respectively. The wear rate of the hybrid composite was low when compared with unreinforced alloy under hot-extruded conditions. The wear rate of the hybrid composite decreased by 39% compared with that of the Al alloy after hot extrusion.

The wear resistance of the hybrid composite improved after extrusion because of the following reasons:

1) Decrease in the grain size:

The grain size of the hybrid composite under hot-extruded conditions decreased, as discussed in section 3.1.3. In the hybrid composite, a considerable decrease in the grain size improved toughness which further restricted surface and subsurface cracking during dry sliding process. Similar observations have been reported for Al6061/C_f rod MMCs [38].

2) Solid lubrication effects of reinforcements (Si₃N₄ and C_f).

3) Formation of MML.

Figure 8 illustrates the results of the SEM with EDS of the worn surface and debris of hybrid composite obtained after extrusion. The SEM image of wear debris revealed the presence of C_f fragments. EDS spectra confirmed the presence of elements such as Fe, Cu, Ni, C, Si, and Al. These elements combined with O_2 to form respective metal oxides. In addition, the XRD results (Fig. 9) confirmed the formation of different metal oxides. This finding was supported by XPS results of the worn surface of the hybrid composite (Fig. 10). The presence of carbonaceous film was evidenced (Fig. 10b). A mixture of different metal oxides and fragmented C_f results in the development of MML at contact surfaces, which improved the wear resistance of the hybrid composite. A similar observation was obtained in a study regarding MML formation on tribological characteristics of AA5052/ C_f composites [39]. Furthermore, several other researchers have reported the beneficial effects of MMLs for reducing the wear rates of MMCs [11, 40-42]. The beneficial effects of carbonaceous film as solid lubricant and enhancing the wear resistance of hybrid Al/ Al_2O_3 /C MMCs have been reported [43].

4) Increased microhardness:

Figure 11 illustrates the microhardness of the base alloy and its composites produced under hot-extruded conditions before and after wear tests. After wear tests, the load increased leading to an increase in the hardness of all the developed specimens. However, the hardness of the hybrid composite was higher than that of the base alloy and composite under hot-extruded conditions after wear at all loads. The hardness of the hybrid composite was improved because of strain hardening effects. Additionally, the presence of C_f in the hybrid composite can cause a decrease in plastic deformation at increased loads. Similar results were reported by Umanath et al. for the improved microhardness of hybrid composites after wear tests [23].

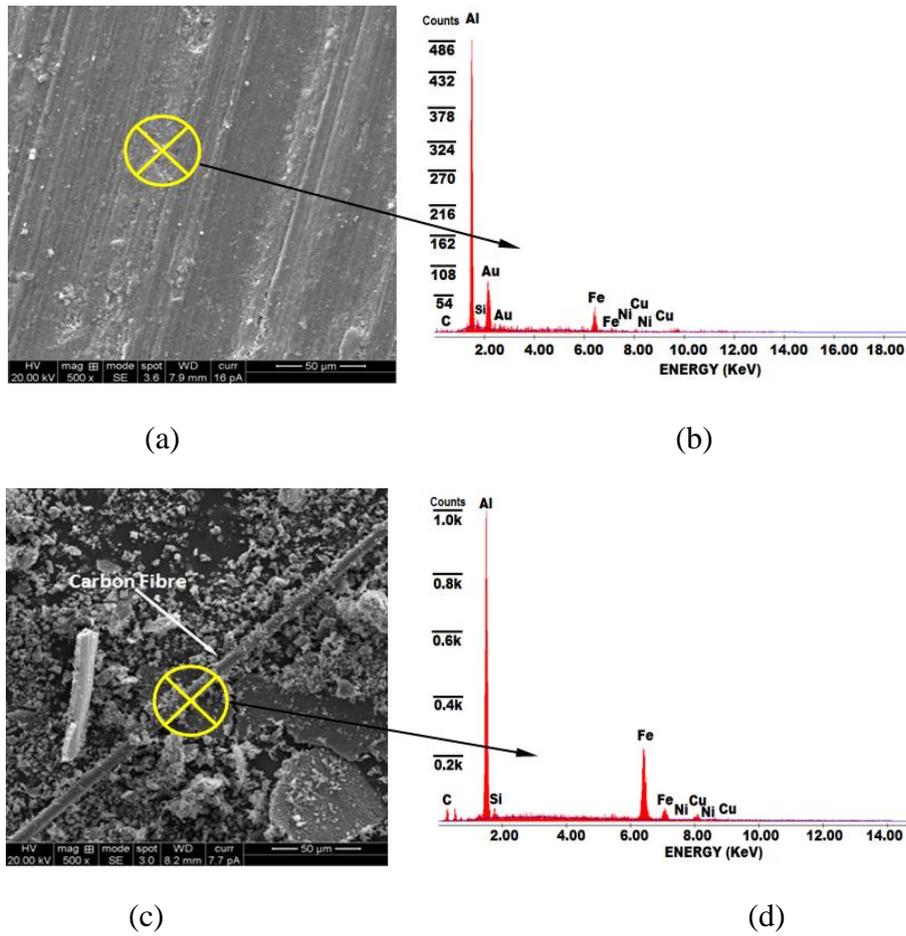


Fig. 8. SEM-EDS images of worn surface (a, b) and debris (c, d) of hybrid composite after extrusion at 60 N load, 30 min test duration and 0.314 ms^{-1} velocity.

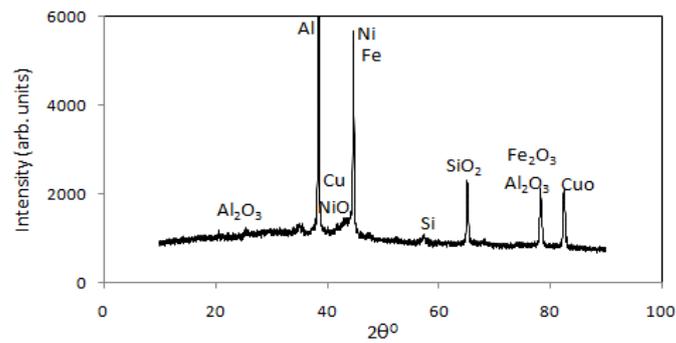


Fig. 9. XRD diffraction pattern of worn surface of hybrid composite after extrusion at 60 N load, 30 min test duration and 0.314 ms^{-1} velocity.

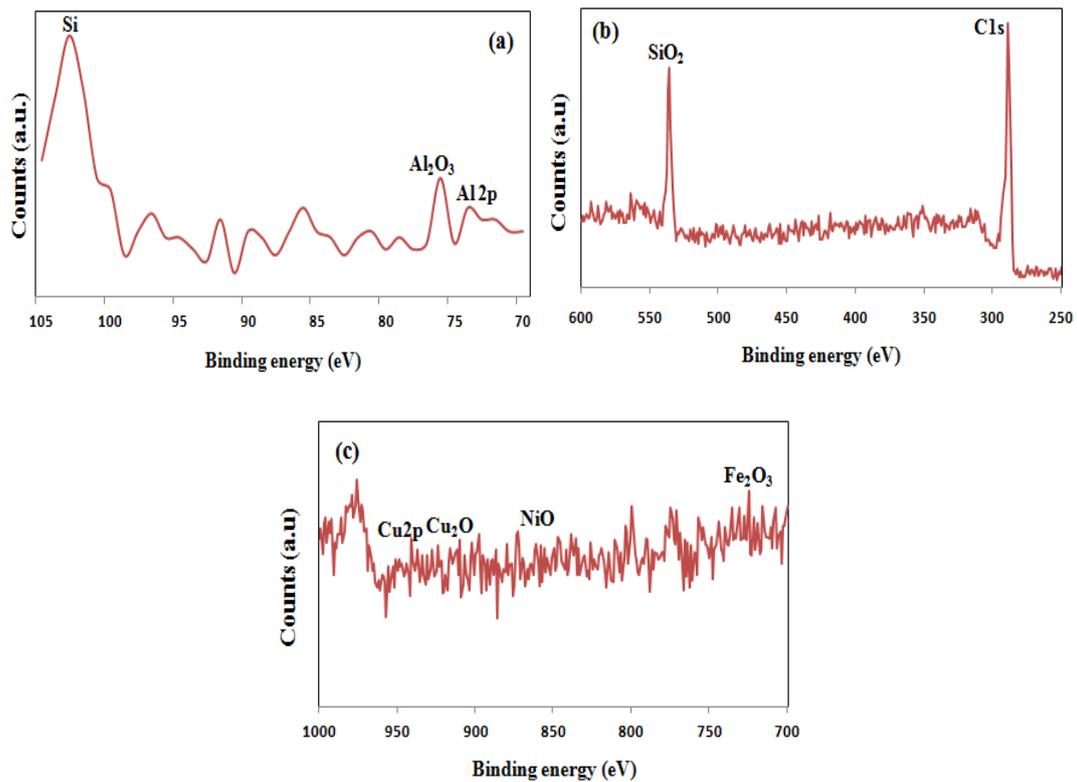


Fig. 10. XPS spectra of worn surface of hybrid composite after extrusion at 60 N load, 30 min test duration and 0.314 ms^{-1} velocity.

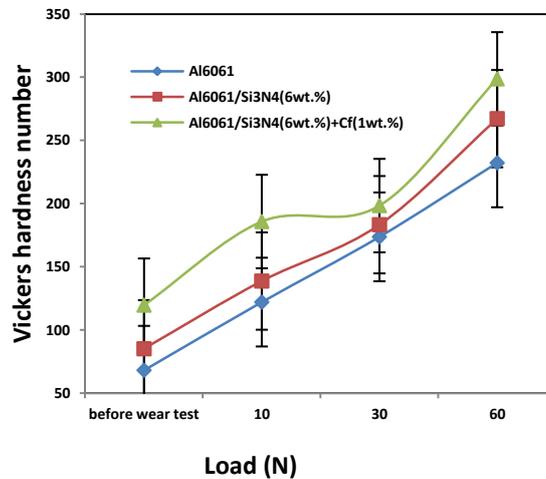


Fig. 11. Vickers microhardness values of Al6061 and its composites produced under hot-extruded conditions before and after wear.

3.2.2.2. Effect of applied load

Figure 12 presents the wear rate of Al6061 and its composites fabricated after extrusion with varying load. The wear rates of the all material system increased with the increase in applied load. The increased load led to an increase in the degree of plastic deformation at the

subsurface and surface of the tested specimens, resulting in larger wear rates. However, the wear resistance of the hybrid composite was higher than that of the base alloy and composite after extrusion at all loads. The wear rates of the hybrid composite were reduced by 49% and 39% compared with those of the base alloy after extrusion at minimum and maximum loads, respectively, of 10 and 60N respectively. The wear resistance of the hybrid composite at higher loads improved because of the presence of C_f , which can retard the propagation of subsurface cracking leading to decrease in the formation of wear debris. This finding can be attributed to the high load-bearing capacity of carbon fibers which was demonstrated through the fragmentation of carbon fibers (Fig. 8c).

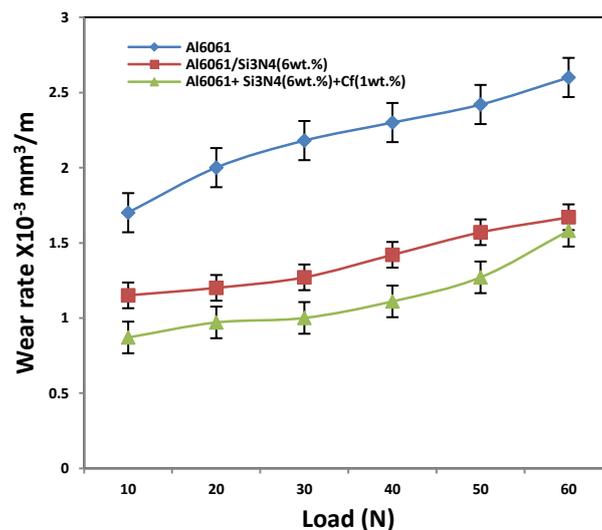


Fig. 12. Wear rate of base alloy and its composites produced after extrusion as a function of load at 0.314 ms^{-1} velocity and 30min test duration.

3.2.2.3. Effect of sliding velocity

Figure 13 presents the wear rate of Al6061 and its composites fabricated under hot-extruded conditions with different sliding velocities. The wear rates of the extruded base alloy and its composites increased with an increase in the sliding velocity. The increased sliding velocity caused an increase in the temperature of contact surfaces, which led to material softening. This phenomenon caused an increase in wear rates at high velocities. However, the hybrid

composite exhibited high wear resistance under hot-extruded conditions at all sliding velocities. The wear rate of the hybrid composite exhibited the decrease of 85% and 54% under hot-extruded conditions at minimum and maximum sliding speeds, respectively, of 0.314 and 1.884 ms⁻¹, respectively, compared with that exhibited by the alloy. The wear resistance of hybrid composite improved even at high sliding speeds because of the enhanced thermal conductivity of copper-coated C_f which caused superior heat dissipation from sliding elements. The improved heat dissipation caused a decrease in thermal softening effects.

Figure 14 illustrates the SEM morphology of the worn surfaces of Al6061 and its composites produced under hot-extruded conditions. The width of grooves in the hybrid composite was smaller than that in the base alloy. This finding indicated that material removal was lower in the hybrid composite. Moreover, the hybrid composite dispersed with carbon fiber exhibited the lowest plough width because of the lubrication effect of C_f. These observations strongly support the experimental results.

Figure 15 presents the SEM results of the wear debris of Al6061 and its composites fabricated under hot-extruded conditions. It is observed that the wear debris of hybrid composite consist of smaller size particles of base alloy and fibres, while the debris of base alloy and composite contains comparatively larger particles size. The large size of wear debris indicated high plastic deformation, which led to larger material loss.

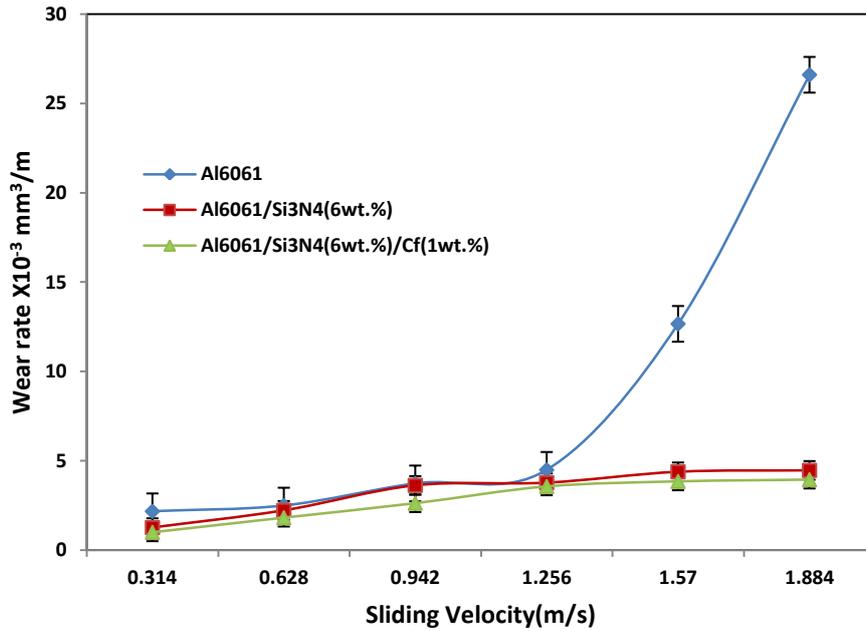


Fig. 13. Wear rate of base alloy and its composites produced after extrusion as a function of sliding velocity at 30 N load and 30 min test duration.

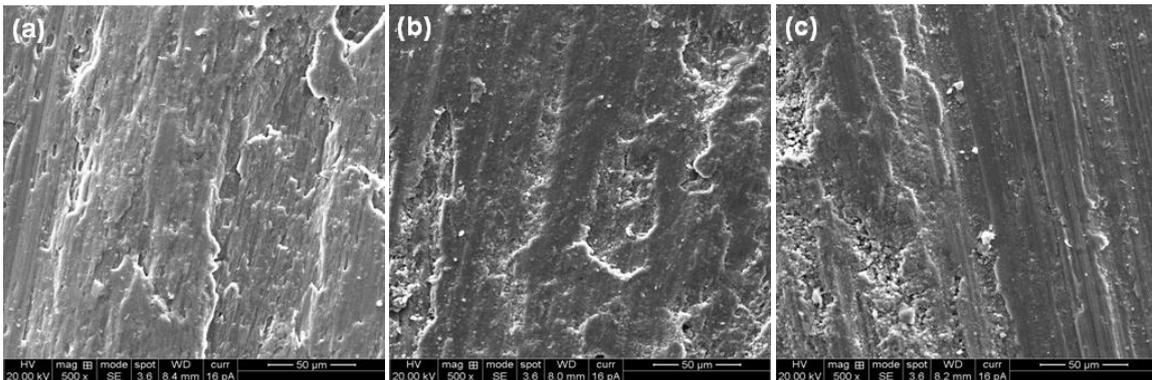


Fig. 14. Worn surface SEM images of (a) base alloy (b) composite (c) hybrid composite after extrusion at 30 N load, 30 min test duration and 1.884 ms^{-1} velocity.

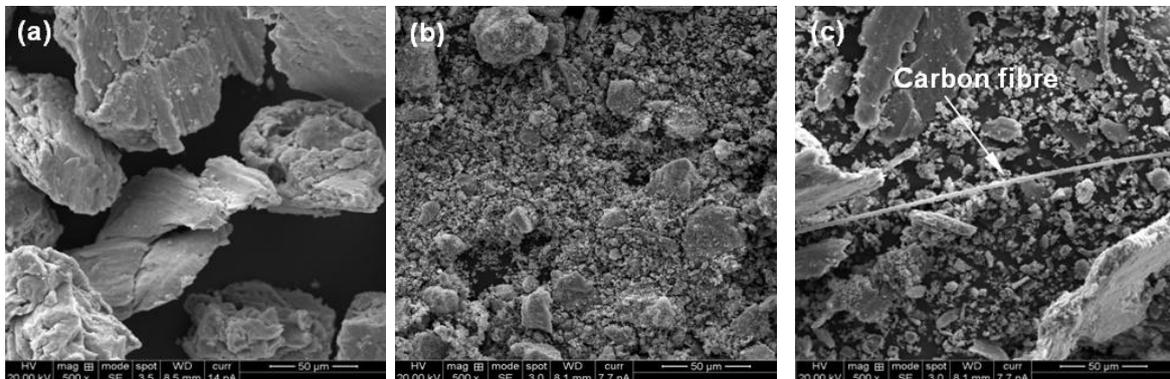


Fig. 15. Wear debris SEM images of (a) base alloy (b) composite (c) hybrid composite after extrusion at 30 N load, 30 min test duration and 1.884 ms^{-1} velocity.

4. Conclusions

1. The grain size of the hybrid composite exhibited a decrease of 29% compared with that exhibited by the unreinforced alloy under hot-extruded conditions.
2. The COF of the hybrid composite exhibited the decrease of 21% and 51% compared with that exhibited by unreinforced alloy after extrusion at minimum and maximum loads, respectively, of 10 and 60N, respectively.
3. The COF of the hybrid composite exhibited the decrease of 22% and 13% compared with that exhibited by the base alloy under hot-extruded conditions at minimum and maximum sliding speeds, respectively, of 0.314 and 1.884 ms⁻¹, respectively.
4. The wear rates of the hybrid composite were reduced by 49% and 39% compared with those of the base alloy after extrusion at minimum and maximum loads, respectively, of 10 and 60N, respectively.
5. The wear rate of the hybrid composite exhibited the decrease of 85% and 54% compared with the alloy under hot-extruded conditions at minimum and maximum sliding speeds, respectively, of 0.314 and 1.884 ms⁻¹, respectively.

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