Mechanical and tribological properties of AA7075-T6 metal matrix composite reinforced with ceramic particles and aloevera ash via Friction stir processing

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Abstract
Mechanical and wear properties of AA7075-T6 reinforced with SiC and Aloevera ash, fabricated using Friction stir processing (FSP) are investigated in this study. Due to less density, easy availability, and cost-effectiveness, aloevera ash is considered as one of the reinforcements. FSP is done using a square tool pin profile, at different tool rotational speeds. To study wear behaviour, pin on disc test is carried out on High Temperature Rotary Tribometer at 20N, 30N and 40N applied load. Wear increases on increasing the applied load and at 20N load Al+SiC/AIDS vera ash composite, processed at 600 tool rpm gave the best results due to the formation of oxide tribolayer. At 30N and 40N applied load Al+SiC composite, processed at 900 tool rpm showed the least wear because of proper scattering of ceramic particles due to high tool rotational speed. Coefficient of friction increases on increasing the applied load and all fabricated composite samples showed a lesser coefficient of friction than the base metal. Microhardness, ductility and Ultimate tensile strength increases on the addition of reinforcement and had a direct relation with tool rpm. Wear morphology was analysed using Scanning Electron Microscope (SEM). Energy Dispersive Spectroscopy (EDS) analysis after wear shows the presence of C, Fe, O, Mg, Zn, Si, Al elements and confirms the formation of an oxide layer which is responsible for decreasing wear loss.

1. Introduction

Aluminium is one of the most abundant metal found on the Earth. It finds wide application in the automobile sector, defence sector, nuclear industry for neutron absorption etc [1]. Aluminium parts are widely used in machines and aluminium composites are widely used in industries [2]. Wear of aluminium is one the serious concern, while it cannot be stopped but can be minimised. In order to minimise the wear Aluminium Matrix Composites (AMMC) are being used instead of Aluminium metal.

In recent years, many researchers have been conducted to fabricate aluminium composite with desirable mechanical and tribological properties. Zawawi et al [3] discussed the wear rate and coefficient of friction effect of Al2O3-SiO2 composite nano lubricant. They found that pure lubricant had a higher coefficient of friction and wear rate than composite nano lubricant. Naveed et al [4] used Graphite as reinforcement keeping SiC content as constant and Aluminium as the base metal. They concluded that wear properties of Aluminium Matrix Composite were enhanced and properties further improved on heat treatment. Raghavendra et al [5] coated Ni-Al2O3 nano particles by electro-deposition process on aluminium, Ni-Al2O3 electrodeposited composite showed better wear resistance than Ni coating. Razzaq et al [6] on observing AA6063 alloy reinforced with different wt.% of fly ash, found that wear rate decreased on increasing fly ash content and it increased on increasing the applied load. Nagaral et al [7] used SiC and Graphite as reinforcement and Al6061 as the base metal. They stated that Al6061%-6%SiC-6%graphite showed less volumetric wear than Al6061 lattice. They also concluded that on increasing applied load, sliding distance and disc speed volumetric wear increases. Gireesh
et al [8] reinforced Al6061 Metal Matrix Composite with aloevera and found that wear resistance, density and ultimate tensile strength showed no significant difference whereas ultimate yield strength and hardness increased as compared to pure Aluminium based Metal Matrix Composite reinforced with aloevera. Mittal et al [9] used SiC, Al2O3 and red mud as reinforcement, AA7075 as the base metal and found that wear rate decreased with increasing wt.% of Al2O3 and SiC with red mud it decreases till 7.5% wt. and on further adding wear rate increases. Baradeswaran et al [10] found that wear resistance increased on increasing B4C content which was used as reinforcement and AA7075 as base alloy.

Raman et al [11] fabricated AMMC using SiC as reinforcement, processed through stir casting and observed that wear rate decreased and wear resistance increased on increasing SiC content. Sun et al [12] on evaluating the microstructure of AA206 during FSP, they observed that grain size and pore morphology of processed material was decreased. Ning Sun et al [13] investigated FSP effect on mechanical and structural properties of age hardenable AA206, tensile and ductile properties were observed to enhance and solidification shrinkage porosity almost eliminated. Singla et al [14] used SiC as reinforcement and Al as base metal. They observed that volume wear increased linearly with sliding distance for both composite and base metal. Wear Rate increased with normal load, but the increment was less for composite than that for pure aluminium. Alizadeh et al [15] used B4C nanoparticles as reinforcement and Al as the base metal. They observed that mechanical properties i.e. yield strength, hardness and tensile strength improved and wear rate was observed to be less than that coarse grain Al matrix. Gireesh et al [16] reinforced aluminium with fly ash as well as with aloevera separately and concluded that aluminium reinforced with aloevera provided better wear resistance than aluminium reinforced with fly ash. Butola et al [17] found that tool profile in FSP is the least influential factor, whereas tool rotational speed of FSP tool microhardness the most.

Miranda et al [18] formed aluminium based FGM composite using FSP. They concluded that because of FSP there were decreased number of cracks and voids, and surface hardness of composite increased. Vijayarit et al [19] reinforced LM25AA with 5% SiC by stir casting and then FSP was carried out on this composite with different shoulder diameter. They concluded that for better microhardness and tensile strength shoulder diameter to pin diameter (D/d) ratio should be 3 and there was very fine distribution of SiC particles in the matrix after FSP. Dinaharam [20] reinforced AA6082 with ceramic particles i.e. SiC, Al2O3, B4C, TiC and WC via friction stir processing. He found that hardness and tensile strength increased in all reinforced samples. Best hardnes and tensile strength results were observed for AA6082/TiC AMC. Khodabakshi et al [21] fabricated Al-matrix nanocomposite ultra-fine grain by FSP. Al-Mg alloy matrix 3.5 vol% SiC and multiple FSP passes were done. Tensile properties improved and hardness increased for ultra-fine grained Al-matrix. Due to multiple passes, SiC was well distributed throughout the matrix. Salehi et al [22] by FSP fabricated AA6061/SiC nanocomposite and determined the factors that influenced ultimate tensile strength. Optimisation of parameters was done by Taguchi. The factor that influenced the most was rotational speed, followed by transverse speed, then pin profile and the factor that influenced the least is tool penetration depth. 1600 rpm rotational speed, 40 mm min$^{-1}$ transverse speed, 0.3 mm tool penetration depth and threaded type tool profile were the optimum parameters obtained for maximum tensile strength.

Rathee et al [23] fabricated AA6061/SiC composite via FSP and to obtain maximum microhardness optimised the design of experiment. Maximum microhardness was obtained at nugget zone and optimum parameters obtained were 50 mm min$^{-1}$ transverse speed, 2.50 tilt angle and 1400 rpm of tool rotational speed. Deepak et al [24] by FSP fabricated Al5083 with nano SiC as reinforcement and observed that the hardness of fabricated composite was more than of base metal, but wear resistance was less due to higher frictional force and coefficient of friction. Salehi et al [25] by FSP fabricated Al6061 composite having SiC nanoparticles, with SiC wt% varying from 0%−18%. Microhardness of the composite was more than that of base metal. They also proposed that microhardness is more when inter-particles spacing is less. Kurt et al [26] by FSP fabricated commercially pure aluminium with SiC particles at different tool rotational and transverse rate. The decrease in grain size and increase in hardness was observed. 1000 RPM and 20 mm min$^{-1}$ were the optimum parameters for the particle’s distribution throughout the matrix. Paidar et al [27] reinforced AA5182 with WC using FSW at different tool traverse speed. They found that the composite had enhanced microhardness, wear-resistance and grain refinement at decreased traverse speed. Paidar et al [28] fabricated Al/B4C composite via FSP, by varying number of passes. Grain size and wear loss decreased, whereas UTS value increased with the increasing number of passes. Devaraju et al [29] by FSP, with SiC and Gr as reinforcement formed aluminium composite. To optimise vol% of reinforcement and tool rotational speed Taguchi was used in order to enhance the mechanical and tribological properties. Wear and hardness increased at respective optimum conditions but tensile properties were lower than base metal at its optimum conditions. Izadi et al [30] by FSP formed Al-SiC composite with vol% varying from 4−16 vol% and found that there were pores and lack of consolidation when processed 16 vol% SiC and all processed samples showed increased hardness and it increased with increase in SiC content. Adilokht et al [31] reinforced AA350 with SiC and MoS2 using FSP at constant tool rotational and travel speed. Hardness and wear resistance of processed composite was more than base metal due to the presence.
of reinforcement. Butola et al [32] reinforced AA6061 with variety of agro-waste materials i.e. jute, bagasse and banana, in order to improve its mechanical properties by stir casting process. Mechanical properties of the material were enhanced with the introduction of these materials and the best results obtained were for composite reinforced with banana ash.

In the present study, mechanical and tribological properties of aluminium reinforced with ceramic and natural reinforcement (Aloevera ash) is studied. Effect of FSP tool rotational speed on the distribution of reinforcement particle is studied. One of the most important factors for any industry is the cost-effectiveness of its product without compromising with its properties. In manufacturing industry constant attempts are being made in order to produce a low-cost AMMC to achieve this several attempts have been made by researchers like taking bagasse ash, red mud, fly ash, rice husk etc as reinforcement [16]. Aloevera is less dense, eco-friendly, easily available material and it also costs less these properties of aloevera attracted us to use aloevera ash as reinforcement as it can be a good alternative for various reinforcements.

2. Experimental procedure

2.1. Matrix material
Aluminium alloy 7075-T6 is used as a matrix material in this study. On aluminium, process like FSP can easily be performed [33]. The plate of length 200 mm, width 80 mm and height 6 mm is used as the base plate. AA-7075-T6 has properties like high fatigue strength, creep resistance, high formability [34]. The chemical composition of aluminium 7075-T6 is mentioned in table 1.

2.2. Reinforcement
Powdered SiC and Aloevera ash are used as reinforcement. SiC due to its semiconductor characteristics possesses interesting electrical properties. Fine powder of SiC having a particle size of 40 \( \mu \)m is used; SEM image of the SiC powder is shown in figure 1(a). Aloevera is a naturally occurring medicinal plant. It has high water content and is rich in nutrients, minerals and vitamins. It is easy to grow plant, which can easily be found throughout the world. The aloevera is first dried in sunlight and then it is burned. The powder left after burning of aloevera is aloevera ash, which is composed of preserved minerals like Ca, Cl, Cr, Cu, Fe, Mg, K, Na, Zn and Mn, most of which act as antioxidants [35, 36]. After burning the resulting white ash is collected and ball milled for 30 h in order to produce a fine powder of size 40 \( \mu \)m. The SEM image of the resulting aloevera ash used is shown in figure 1(b).

2.3. Fabrication of composites
All samples are fabricated using Friction stir processing at different rpm i.e. 600, 900. FSP is the method of fabricating composite by plastic deformation of the matrix surface by rotating a tool at very high rpm, the set up

| Table 1. Composition of AA7075-T6. |
|-----------------------------------|
| Element       | Si  | Mg  | Fe  | Zn  | Al  |
| Weight % composition | 1.514 | 1.664 | 1.744 | 4.156 | Balance |

![Figure 1. SEM images of (a) SiC particles at 20 \( \mu \)m (b) Aloevera ash particles at 20 \( \mu \)m.](image-url)
can be seen in figure 2. Different properties of friction stir processed alloy are due to the different speed and angle of the FSP tool [37]. The square tip tool made of H13 tool steel of total length of 117 mm and tip length of 3.5 mm is used for fabricating the above samples, as square tip minimises the defects in the matrix, uniformly distributes the reinforcement and increases hardness [21, 38]. Summary of groove design is shown in table 2. H13 tool steel is also used in friction stir welding and manufacturing welding joints [39]. The other process parameters are mentioned in table 3. Even sized grooves of depth 3.5 mm and width 2 mm are machined on aluminium 7075-T6 plates. Various calculations related to grooves i.e. volume of fraction, area of groove and projected tool pin area are done in equations (1)–(3). In these grooves, reinforcement is filled in powdered form and by using FSP these are mixed.

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\text{Area of groove} = \text{Depth of groove} \times \text{Width of groove} \quad (1) \\
\text{Projection area of tool pin} = \text{diagonal of square base} \times \text{tool pin length} \quad (2) \\
\text{Fraction Volume} = \frac{\text{Area of groove}}{\text{Projection area of tool pin}} \quad (3)
\]

Sample 1 contains 2 wt.% SiC reinforcement with FSP done at 600 rpm. In Sample 2 FSP is done at 600 rpm and SiC + Aloevera ash (2 wt.%) are used as reinforcements. In sample 3 and sample 4 FSP is done at 900 rpm,

Table 2. Groove design.

| Parameters          | Values                     |
|---------------------|----------------------------|
| Dimension of plate  | 200 $\times$ 80 $\times$ 6 (mm$^3$) |
| Groove length       | 160 mm                     |
| Groove width        | 2 mm                       |
| Groove depth        | 3.5 mm                     |

Table 3. Process parameters.

| Process parameters | Values                      |
|--------------------|-----------------------------|
| Tool material      | H13 tool steel              |
| Tool pin profile   | Square                      |
| Tool shoulder profile | Flat                     |
| Tool pin length    | 3.5 mm                     |
| Tool shoulder diameter | 19.95 mm                 |
| Tool plunge depth  | 0.20 mm                    |
| Tilt angle         | 2$^\circ$ constant          |
| Reinforcement      | SiC, Aloevera ash           |

Figure 2. (a) FSP set up (b) FSP tool and processed plate design.
used SiC (2 wt.%) and SiC + Aloe vera ash (2 wt.%) as reinforcements respectively. Table 4 shows the chemical composition of all tested samples.

2.4. Pin on disc set up
Wear is studied using high temperature rotary tribometer manufactured by DUCOM. Disc of 100 mm diameter made of EN24 metal is used and this disc is cleaned using acetone before the experiment. Samples used to perform testing are taken in the shape of cylindrical pins with 8 mm diameter and 6 mm height. Test conditions and set up used for the pin on disc experiment are shown in table 5 and figure 3 respectively. Wear and coefficient of friction are calculated from the data and analysed in this study.

2.5. Tensile testing
Samples of length 80 mm and thickness 3.5 mm with flat base are prepared according to ASTM standard shape for the tensile testing, shown in figure 4. Samples are gripped from both sides and a constantly increasing load is applied until the breaking point is reached. Changes in the sample are recorded throughout the testing and then

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Table 4. Composition of tested samples.

| Sample  | Composition                  | Tool rpm |
|---------|------------------------------|----------|
| Sample 1| AA7075-T6 + 2wt% SiC         | 600      |
| Sample 2| AA7075-T6 + 1wt% SiC + 1wt% Aloe vera ash | 600 |
| Sample 3| AA7075-T6 + 2wt % SiC       | 900      |
| Sample 4| AA7075-T6 + 1wt% SiC + 1wt% Aloe vera ash | 900 |

Table 5. Test conditions for pin on disc experiment.

| S. No. | Test Conditions | Values |
|--------|----------------|--------|
| 1.     | Load           | 20 N, 30 N, 40 N |
| 2.     | Pin diameter   | 8 mm   |
| 3.     | Pin height     | 6 mm   |
| 4.     | Track diameter | 30 mm, 40 mm, 50 mm |
| 5.     | Sliding distance | 950 m  |
| 6.     | Rpm            | 637, 478, 382 |
| 7.     | Time           | 16 min |
| 8.     | Disc roughness | 0.2 μm |
| 9.     | Disc hardness  | 57 HRC |

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analysed. All samples i.e. base metal, sample 1, sample 2, sample 3, sample 4 has been tested and the parameters recorded were ultimate tensile strength and stress-strain curve.

2.6. Microhardness
Microhardness is tested using Fischer scope HM2000 S microhardness tester according to ASTM: E8/E8M-011 standards. By analysing the indentation depth formed by indenter due to a constant force of 200.5 KN, applied on it for 20 s, microhardness value at a point is obtained. Microhardness is examined for 4 different points on a sample and an average of them is considered as the true microhardness of the sample. Microhardness is analysed for base metal, sample 1, sample 2, sample 3 and sample 4.

2.7. Wear morphology
The worn-out samples are examined using Scanning Electron Microscope (SEM) focused electron beam is used to produce the image in order to examine the wear surface of the samples. SEM images are produced in order to analyse the wear effect and Energy Dispersive x-ray Spectroscopy (EDS or EDX) is done to find the compound present after wear. EDS basic principle is that every element has a unique atomic structure, it works on interaction between the x-rays and samples.

3. Results and discussion

3.1. Wear
From figure 5 it can be concluded that with increasing load wear increases for all samples, the increase is different for samples depending upon the reinforcement and tool rpm of FSP. Similar pattern was observed when SiC and Al₂O₃ were reinforced with aluminium matrix and an increase in wear volume was observed with an increase in load [40].

With the addition of reinforcement wear decreases, as load is distributed on uniformly scattered hard ceramic particles. Base metal shows the maximum wear loss for all tested load. Of all tested samples, minimum wear at 20 N load is observed for Al+SiC/Aloevera ash processed at 600 tool rpm, due to less ageing and formation of the oxide layer which acts as a solid lubricant and reduces wear [41]. For 30 N and 40 N load, optimum results are given by Al+SiC processed at 900 tool rpm, as hard ceramic SiC particles are properly dispersed throughout the matrix and prevent direct contact of aluminium with the disc. On comparing wear loss of Al+SiC composite i.e. sample 1 and sample 3, it can be observed that wear is less for sample processed at 900 rpm than sample processed at 600 rpm for all tested loads. This is due to proper dispersion of fine hard SiC particles in the matrix at higher tool rpm, with the increased FSP tool rpm number of cavities and defects in the metal matrix are minimum [42]. For Al+SiC/Aloevera ash composites i.e. sample 2 and sample 4, the samples processed at 600 rpm showed less wear than sample processed at 900 rpm. This might be attributed to the softening of the matrix because of heat generated in the nugget zone, due to over ageing it causes matrix softening [43].

3.2. Coefficient of friction with sliding distance
Variation of coefficient of friction as a function sliding distance of AMMCs is displayed in figure 6, the total sliding distance was 950 m. There is a large rise in the initial coefficient of friction value of the base metal. This can be due to the large force required to overcome the adhesion between the contact surfaces [40]. After 100 m there is large fluctuation and friction coefficient value goes down and fluctuates within the limits. Ceramic SiC particles dispersed throughout the matrix assist the load and reduce the contact between the aluminium surface and disc surface. There is a sudden rise in the friction coefficient value of Al+SiC composite i.e. sample 1 and 3. The reinforcement particles are very well scattered throughout the matrix and when the disc surface comes in
contact with these well dispersed hard SiC particles the coefficient of friction increases and this results in less wear loss and more wear resistance [44]. Sudden fluctuation can be seen in coefficient of friction of Al+SiC/Aloe vera ash composite for both 600 and 900 tool rpm. This fluctuation is due to ploughing off of oxide layer which results in the generation of surface pits. Presence of these pits causes mismatching of the surface resulting in fluctuating value of coefficient of friction. SiC prevent the drastic metal removal and ploughing off of layers hence preventing pits formation, so less fluctuation in the value of friction coefficient is recorded [44].

3.3. Effect of load
Variation of coefficient of friction with load can be seen in figure 7(a), it is more pronounced at lower load. Base metal shows the highest coefficient of friction at all tested loads. Coefficient of friction increases with increasing load. For composite processed at higher tool rpm there is a consistent increase in the coefficient of friction with increasing load. Samples containing aloe vera ash as one of their reinforcement i.e. sample 2 and 4, showed comparatively lower coefficient of friction due to the formation of the oxide layer, it acts as a lubricating hence reduces the coefficient of friction. Effect of load on wear rate is shown in figure 7(b). It can be concluded that with increasing load wear rate increases and base metal shows the highest wear rate at all tested load. Due to well distribution of SiC particles throughout the matrix sample 3 has least wear rate at 30 N and 40 N and sample 2 showed minimum wear rate at 20 N due to formation of a lubricating oxide layer. Sample 4 showed the highest wear rate of all reinforced sample justifying the matrix softening due to ageing by FSP.

3.4. Tensile properties
Selection of engineering material is an important task and it all depends on the properties of the material. The tensile test is important in determining the properties of a material. Tensile strength is tested for the base metal as well as for processed composites and, on the basis of the readings obtained the stress-strain curve of the respected samples is presented in figure 8.

From figure 8 it can be observed that ductility increased for all fabricated composite. The composites fabricated at higher tool rpm i.e. sample 3 and sample 4, are more ductile than composite fabricated at low rpm i.e. sample 1 and sample 2. It can be summarised that ductility and UTS are enhanced with addition of reinforcements (SiC and aloe vera ash), and further enhancement was observed on processing the material at higher tool rpm. Similar type of results were reported by Zhou et al [45] when reinforced Al7085 with TiC via FSP. Arab et al [46] in their study showed that ductility improved when AA1100 was FSPed without any reinforcement, and further enhanced when AA1100 was reinforced with various novel fibres through FSP, reflecting that reinforcement plays a vital role in enhancing the properties along with the process.
3.5. Microhardness

Hardness refers to the property of the material to resist the deformation due to the force applied. Microhardness of all samples is shown in figure 9. Microhardness for base metal is 102 Hv and for sample 1 is 121.3 Hv which is
19% more than base metal, as well dispersed hard reinforcement particles support the matrix base and thus increase the microhardness [29]. Sample 2 has 18% more microhardness than base metal due to the presence of well-dispersed reinforcement particle. For sample 3 microhardness value is 152 H and for sample 4 it is 137.82
which is more than the recorded value for base metal, sample 1 and sample 2. It can be observed that on adding reinforcement microhardness of material increases and it further increases on increasing the tool rpm on which it is processed, as material FSPed at higher tool rpm have finer grains than those FSPed at lower rpm [21] and microhardness is enhanced due to uniform distribution of these fine grains throughout the matrix [26].

3.6. Wear morphology
SEM analysis is done in order to discern the effect of reinforcements, tool rotational speed and applied load on wear behaviour of the fabricated AMMCs. Figures 10(a)–(h) illustrates the worn surface of all fabricated composites at different applied load i.e. 20 N and 40 N. Wear tracks can be seen in the SEM images (figures 10(b)–(e), (h)) and the white particles in the images shows the formation of carbide in the samples (figures 10(b), (d), (e), (g)) during wear of disc materials. Traces of adhesive wear can be seen in the images majorly at higher applied load. Figure 11 shows the EDS analysis of the fabricated composite, it is done in order to find the constituent of the samples after wear. Presence of C, O, Mg, Al, Si, Fe, and Zn can be seen in the EDS of the wear debris. Presence of oxygen confirms the formation of oxide tribolayer. This tribolayer is formed by the reaction of oxygen and aluminium or iron. This reaction might have occurred during the wear test due to the large heat generation between the surfaces. Fe in EDS signifies the formation of Fe layer which also acts as a solid lubricant, hence reducing wear and coefficient of friction. Such high content of Fe in the samples is due to the transfer of elements between wear pin and disc in the early phase of wear testing. Similar types of results were reflected by Alam et al and Coyal et al [47, 48] in their study.

4. Conclusions

• Wear resistance of all fabricated composite was more than that of base metal.
• At 20N applied load Al+SiC/Aloe vera ash composite processed at 600 tool rpm showed the least wear loss, due to oxide tribolayer formation. At higher applied load Al+SiC, processed at 900 rpm had the best wear resistance, as at higher tool rpm there is proper dispersion of reinforcement.
• Wear rate and coefficient of friction both increases with increasing the applied load.
• Of all tested samples base metal had the maximum value for both coefficient of friction and wear rate for all applied load.
• Ductility and ultimate tensile strength increased with the addition of reinforcement.
• processed at higher tool rpm are more ductile and have higher ultimate tensile strength than those processed at lower tool rpm.
• Microhardness increased with the introduction of reinforcement. Microhardness is more for samples processed at higher tool rpm due to the finer grains obtained because of high tool rotation.
• SEM analysis of wear surface shows the proper distribution of reinforcement and traces of adhesive wear.
EDS of the sample after wear indicates the presence of C, O, Fe, Zn, Si and Al, confirming the formation of oxide tribolayer and validation the reduced wear loss and coefficient of friction.

Figure 10. (a) SEM of sample 1 (20 N load) (b) SEM of sample 1 (40 N load) (c) SEM of sample 2 (20 N load) (d) SEM of sample 2 (40 N load) (e) SEM of sample 3 (20 N load) (f) SEM of sample 3 (40 N load) (g) SEM of sample 4 (20 N load) (h) SEM of sample 4 (40 N load).

- EDS of the sample after wear indicates the presence of C, O, Fe, Zn, Si and Al, confirming the formation of oxide tribolayer and validation the reduced wear loss and coefficient of friction.
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