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Corina Julieta Birleanu

Technical University of Cluj-Napoca

Horea-Stefan Goia

Technical University of Cluj-Napoca

Florin Popister

florin.popister@muri.utcluj.ro

Technical University of Cluj-Napoca

Ramon Pamies

Universidad Politécnica de Cartagena

María Dolores Aviles

Universidad Politécnica de Cartagena

Razvan Udrioiu

Transylvania University of Braşov

Marius Pustan

Technical University of Cluj-Napoca

Mircea Cioaza

Technical University of Cluj-Napoca

Mihai Dragomir

Technical University of Cluj-Napoca

Florin Popa

Technical University of Cluj-Napoca

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Synergistic Friction and Wear Reduction in CuSn10-Modified Basalt Fiber-Reinforced Epoxy Composites via Protic Ionic Liquid and Graphene Nanolubrication

Corina Birleanu¹, Horea-Stefan Goia^{1,*}, Florin Popister^{1,*}, Ramon Pamies², María Dolores Avilés², Razvan Udrioiu³, Marius Pustan¹, Mircea Cioaza¹, Mihai Dragomir⁴ and Florin Popa⁵

¹ MicroNano Systems Laboratory, Mechanical Systems Engineering Department, EUT+—Institute for Nanoscience and Nanotechnology of the European University of Technology (EUTINN), Technical University of Cluj-Napoca, Bvd. Muncii no. 103–105, 400641 Cluj-Napoca, Romania; corina.barleanu@omt.utcluj.ro (C.B.); marius.pustan@omt.utcluj.ro (M.P.); mircea.cioaza@staff.utcluj.ro.

² Grupo de Ciencia de Materiales e Ingeniería Metalúrgica, Technical University of Cartagena (member of European University of Technology EuT+), Campus Muralla del Mar, C/ Doctor Fleming s/n, 30202 Cartagena (Murcia), Spain; ramon.pamies@upct.es (R.P.); mdolores.aviles@upct.es (MD.A.)

³ Manufacturing Engineering Department, Transilvania University of Brasov, Blv. Eroilor Nr. 29, 500036 Braşov, Romania; udrioiu.r@unitbv.ro (R.U.)

⁴ Design Engineering and Robotics Department, Technical University of Cluj-Napoca, Bvd. Muncii no. 103–105, 400641 Cluj-Napoca, Romania; mihai.dragomir@muri.utcluj.ro (M.D.)

⁵ Materials Science and Engineering Department, Technical University of Cluj-Napoca, Bvd. Muncii no. 103–105, 400641 Cluj-Napoca, Romania; florin.popa@stm.utcluj.ro; (F.P.a.)

* Correspondence: horea.goia@campus.utcluj.ro (H.G.); florin.popister@muri.utcluj.ro (F.P.r.)

Highlights

- CuSn10-modified BFRP composites combined with PIL nanolubricants studied systematically
- PIL nanofluids reduced friction by up to 94% and wear by up to 99.6% vs. dry sliding
- Graphene-dispersed citrate PIL (MCi+0.1%G) gave the best overall tribological performance
- CuSn10 bronze filler acts as a load-bearing phase, cutting wear even without lubrication
- GLM-ANOVA quantified the relative contribution of material and lubricant to COF and wear

Abstract: Basalt fiber-reinforced epoxy (BFRP) composites combine a favorable strength-to-weight ratio with relatively low cost, which makes them attractive for structural applications. However, the epoxy matrix has limited abrasion resistance, and this restricts how well these composites perform under sustained sliding contact. In this study, two autoclave-processed BFRP variants were tested: an unfilled composite (60 wt% basalt fiber) and a hybrid composite modified with 10 wt% CuSn10 bronze powder. The two samples were evaluated under dry sliding and under lubrication with four 2-hydroxyethylammonium (2-HEA)-based protic ionic liquid (PIL) nanofluids two used neat (MSu, MCi) and two dispersed with 0.1 wt% graphene (MSu+0.1%G, MCi+0.1%G). Pin-on-disc tests were performed under a normal load of 10 N and a sliding speed of 0.25 m·s⁻¹, and worn surfaces were characterized by SEM-EDS. All four PIL nanofluids reduced the steady-state coefficient of friction (COF) by 83–94% relative to dry sliding, while specific wear rate reductions ranged from 14% to over 99%, with the graphene-dispersed citrate PIL (MCi+0.1%G) consistently yielding the best performance. Notably, the CuSn10-modified composite showed a wear rate comparable to the best-lubricated unfilled composite even under dry sliding, indicating that the bronze particles act as a load-bearing phase independent of lubrication. GLM-ANOVA confirmed that lubricant chemistry dominates friction (92.7% contribution) while material composition and lubricant contribute comparably to wear resistance (38.1% and 47.9%, respectively). It is acknowledged by the authors that this is the first report of its kind to combine a metallic load-bearing filler (CuSn10) with graphene-dispersed protic ionic liquid nanolubricants on a basalt fiber-reinforced polymer composite, evaluated under a unified, statistically validated experimental framework. These findings establish this combined material–lubricant strategy as an effective approach for improving the tribological durability of basalt-fiber composites in sustained sliding applications.

Keywords: Basalt fiber-reinforced composite; Protic ionic liquid; Graphene nanolubricant; CuSn10 bronze filler; Tribological performance; Pin-on-disc testing

Abbreviations

- BFRP - Basalt fiber-reinforced polymer
- PIL - Protic ionic liquid
- 2-HEA - 2-hydroxyethylammonium
- MSu – Di(2-hydroxyethylammonium) succinate
- MCi – Tri(2-hydroxyethylammonium) citrate
- G - Graphene
- COF - Coefficient of friction
- k - Specific wear rate
- SEM - Scanning electron microscopy
- EDS - Energy-dispersive X-ray spectroscopy
- BSE - Backscattered electron (imaging mode)
- GLM-ANOVA - General Linear Model analysis of variance
- PC - Personal contribution (statistical, ANOVA)

- SD - Standard deviation
- CV - Coefficient of variation

1. Introduction

The reduction of friction and wear in sliding contacts is one of the enduring challenges in materials engineering, with direct consequences for energy efficiency and operational reliability across a wide range of industrial systems [1, 2]. Fiber-reinforced polymer composites (FRPCs) have become increasingly important in this regard, largely because they offer a favorable strength-to-weight ratio alongside good chemical stability and considerable flexibility in tailoring mechanical and tribological properties to specific service requirements [3, 4]. Among the reinforcement fibers that have attracted growing attention, basalt fibers are notable for a particularly advantageous combination of characteristics: namely, mechanical strength that exceeds that of glass fibers, good resistance to both thermal and chemical degradation, a naturally occurring mineral origin that limits environmental impact, and a cost that is substantially lower than that of carbon fibers [5, 6]. The epoxy matrix, a material frequently employed in the fabrication of structural composites, confers rigidity and chemical inertness. However, its inherent limitations with respect to resistance to abrasive and adhesive wear are well-documented. This shortcoming is of particular relevance in applications involving sustained tribological loading, where matrix degradation can compromise the integrity of the composite and necessitates either compositional modification or effective external lubrication [7].

In the preceding two decades, there have been several research initiatives directed towards enhancing the tribological performance of basalt fiber-reinforced epoxy (BFRP) composites. These research efforts have encompassed various approaches, including the modification of the composite matrix with micro- and nanoscale particles, the application of surface coatings, and the utilization of external lubricants. In their study, Zhang et al. [8] examined basalt/phenolic composites that had been filled with solid lubricants. The researchers found that the addition of molybdenum disulfide (MoS_2) led to a significant reduction in the coefficient of friction. Graphite, on the other hand, was found to enhance wear resistance. The outcome was found to be particularly dependent on the specific sliding conditions that were present. In their research, Jamali et al. [9] utilized multiscale basalt/epoxy composites incorporating silanized graphene oxide. At a loading of 0.4wt%, which was identified as optimal, there was a 62% decrease in wear rate and a 44% decrease in the friction coefficient compared with the unfilled composite. A related approach was adopted by Liu et al. [10], who incorporated graphene nanoplatelets (GNPs) into the epoxy matrix of BFRP composites. This resulted in a 23% decrease in the friction coefficient and a 60% enhancement in wear resistance. The authors attributed this phenomenon to two distinct effects: the solid-lubricant behavior of the GNPs themselves, and the enhanced thermal conductivity of the modified matrix. Basalt fibers also compare favorably to glass fibers in this respect. Fiber composites, comparative testing has shown that basalt variants retain better wear resistance under elevated pressure-velocity (PV) conditions, which makes them the more suitable choice when tribological demands are severe [11]. Incorporating metallic powders into the polymer matrix has proven to be a further step toward improving BFRP tribological performance. Earlier work by Birleanu et al. [12] looked at how copper (Cu) powder additions 5, 10, and 15 wt% affected both the mechanical and tribological behavior of BFRP composites made by vacuum bagging. Wear rate and friction coefficient both improved measurably, and a protective copper film was seen to develop at the sliding interface during testing. This work was later extended by Birleanu et al. [13], who used bronze powder (CuSn10) instead, at the same concentration range, in hybrid BFRP composites produced by vacuum-assisted processing. Tests were run under dry sliding, across normal loads of 10, 20, and 30 N and sliding speeds of 0.1, 0.25, and 0.36 m/s. Tensile strength peaked at 440.6 MPa for the 10 wt% CuSn10 composition, while the best tribological results came at 15 wt%, where friction coefficient dropped by up to 38% and specific wear rate fell by more than 50%, relative to the unreinforced BFRP baseline. It appears that the bronze particles cushion the abrasive action on the basalt fibers while also helping stable tribofilms form at the sliding interface together, these two effects likely explain the improved wear behavior reported [13].

Beyond modifying the composite structure itself, researchers have also pursued external lubrication as a complementary route to better tribological performance, and this direction has proven increasingly productive. Ionic liquids (ILs) in particular have drawn considerable attention, largely due to their negligible vapor pressure, wide range of thermal stability, and their ability to form stable boundary films that adhere well to both metallic and polymeric surfaces [14, 15]. Among the various IL families, protic ionic liquids (PILs) offer a distinct set of practical advantages: they are synthesized in a single acid-base reaction, they are relatively inexpensive, they are free of halogens, and many of them have demonstrated reasonable biodegradability, which gives them a more favorable safety and sustainability profile compared with aprotic counterparts [16, 17].

Espinosa et al. [18] tested di(2-hydroxyethylammonium) succinate (MSu) as a lubricant for the sapphire/AISI 316L stainless steel contact and reported that neat MSu reduced the run-in friction coefficient by 88% relative to water. When used as a 1 wt% additive in water, MSu allowed a PIL boundary film to form upon water evaporation, yielding a minimum friction coefficient of 0.0001, which is 97% lower than that of water and 78% lower than that of neat MSu; surface analysis by FTIR, SEM-EDX, and XPS confirmed the presence of a stable adsorbed film responsible for this behavior [18]. The closely related PIL tri[bis(2-hydroxyethyl)ammonium] citrate (DCi) was evaluated as a neat lubricant and as an additive in mineral oil for steel-steel and aluminium-steel contacts; at 2 wt% in mineral oil it reduced the friction coefficient by up to 16% and the wear volume by up to 40% [19]. Aviles et al. [20] subsequently showed that incorporating 9 wt% DCi directly into an epoxy matrix produced a self-lubricating material with substantially lower friction and wear rates than the neat resin.

Dispersing graphene in these PILs opened a further class of lubricants, referred to as ionanocarbon systems, in which the two components act synergistically to produce tribological outcomes that neither achieves alone. Aviles et al. [21] demonstrated that depositing a thin (IL + graphene) film by spin coating reduced the friction coefficient by 40% and completely prevented wear, whereas a film of the IL alone showed friction independent of film thickness. Saurin et al. [22] found that adding graphene to an epoxy matrix alongside an imidazolium IL produced friction reductions of up to 70% with negligible surface damage. A systematic account of the underlying mechanisms was provided by Aviles et al. [23], who explained that the IL promotes graphene dispersion and surface adsorption while graphene provides low-shear solid lubrication, the combination yielding better friction and wear reduction than either component separately. A similar approach was taken by Aviles et al. [24], who used the diprotic PIL di(bis(2-hydroxyethyl)ammonium) succinate (DSu) as a base fluid for graphene dispersions tested on sapphire/steel contacts. Adding just 0.05 wt% graphene was enough to eliminate the high-friction run-in period seen at the start of testing, and it also prevented damage to the steel surface reducing the wear rate by two orders of magnitude compared with lubrication using the full fluid alone. Martinez-Rubio et al. [25] took a more characterization-focused approach, studying the physicochemical properties of nanofluids made by dispersing graphene in 2-hydroxyethylammonium lactate (ML), at concentrations up to 1 wt%. Their 0.1 wt% dispersion turned out to be stable for at least two months, behaved as a Newtonian fluid, and showed a strong graphene-IL interaction under Raman spectroscopy all of which pointed toward its suitability for boundary lubrication applications. Against this background, the present work undertakes a systematic investigation of the tribological behavior of basalt fiber-reinforced epoxy composites based on a twill weave fabric (60 wt% fiber content), with and without the addition of CuSn10 bronze powder (10 wt%), lubricated by four 2-HEA-type PIL nanofluids: MSu di(2-hydroxyethylammonium) succinate, MCi tri(2-hydroxyethylammonium) citrate, MSu+0.1%G (MSu with 0.1 wt% dispersed graphene), and MCi+0.1%G (MCi with 0.1 wt% dispersed graphene). The specimens are manufactured by autoclave processing and subsequently tested on a pin-on-disc tribometer under both dry sliding and boundary lubrication conditions for each of the four nanolubricants. Friction and wear behavior are assessed comparatively, and the worn surfaces are examined by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) to identify the operative wear mechanisms and the nature of lubricant-material interactions at the interface. Statistical analysis of the experimental data is used to quantify the influence of composite type and lubricant formulation on the friction coefficient and specific wear rate.

The main objective of this work is to establish, within a single unified experimental framework, how bronze (CuSn10) reinforcement of the epoxy matrix and external lubrication with 2-HEA-type protic ionic liquid nanofluids independently and jointly govern the friction and wear response of basalt fiber-reinforced epoxy composites. The principal novelty of the study lies in the combined use of a metallic load-bearing filler with graphene-dispersed PIL nanolubricants on a structural polymer composite substrate a pairing not previously reported together with a statistically validated (GLM-ANOVA) quantification of the relative contribution of material composition versus lubricant chemistry to each tribological response variable. To our knowledge, this is the first published systematic study of this PIL-BFRP combination with and without CuSn10 bronze powder, and the results are expected to broaden the understanding of ionic nanofluid lubricants applied to structural polymer composites.

2. Materials

2.1 Polymer matrix

The polymer matrix used throughout this study is a two-component epoxy system consisting of EPIKOTE(TM) Resin MGS(TM) LR 135 and EPIKURE(TM) MGS(TM) Curing Agent LH 136, both supplied by HEXION GmbH (Duisburg, Germany). This system is well established in structural composite manufacturing and is widely used in aerospace and high-performance engineering applications owing to its combination of high mechanical strength, good chemical resistance, and low viscosity in the uncured state [26, 27]. The resin-to-hardener mass ratio applied in this work was 100:35.2, as specified by the manufacturer for achieving complete, uniform crosslinking. At this stoichiometry the cured matrix exhibits good stiffness-to-toughness balance while maintaining adequate adhesion to basalt fiber surfaces. The moderate viscosity of the uncured mixture facilitates homogeneous impregnation of the reinforcement fabric and minimizes the formation of air voids or dry fiber zones during autoclave consolidation, which is critical for obtaining reproducible mechanical and tribological properties in the final composite.

2.2 Basalt fiber reinforcement

The reinforcement in all composite specimens consists of a 2/2 twill-weave basalt fiber fabric with an areal density of 220 g·m⁻². Twill-weave architectures are preferred for tribological applications because the interlaced fiber pattern distributes contact stresses more evenly across the laminate surface, promotes uniform wear, and suppresses crack propagation relative to plain-weave variants [12, 13]. Basalt fibers are produced by melting and drawing naturally occurring basaltic rock, a process that yields a product with mechanical properties exceeding those of standard E-glass fibers while remaining substantially less expensive than carbon fibers [5, 6]. Their inorganic composition also confers notable thermal stability, with continuous service capability up to 550°C and ignition resistance to temperatures approaching 1200°C, making them attractive for applications where both structural performance and thermal protection are required.

The typical chemical composition of the basalt fibers used in this investigation is presented in Table 1. Silicon dioxide (SiO₂, 45-55 wt%) is the primary constituent and is responsible for the high mechanical strength and thermal stability of the fiber. Aluminium oxide (Al₂O₃, 10 - 15wt%) contributes to stiffness and elevated-temperature performance, while the iron oxides (Fe₂O₃ + FeO, 10 - 15wt%) account for the characteristic dark color and assist in load transfer within the fiber

structure. The remaining oxides calcium, magnesium, sodium, potassium, and titanium play secondary roles in controlling melt rheology during fiber drawing, stabilizing the amorphous structure, and imparting chemical resistance to the finished fiber [5].

Table 1. Typical chemical composition of basalt fibers used as reinforcement.

Oxide	Formula	Content (wt%)	Role in fiber properties
Silicon dioxide	SiO ₂	45-55	Thermal stability and mechanical strength
Aluminium oxide	Al ₂ O ₃	10-15	Stiffness and high-temperature resistance
Iron oxides	Fe ₂ O ₃ +FeO	10-15	Mechanical strength; dark fiber color
Calcium oxide	CaO	5-12	Structural stabilization; impact resistance
Magnesium oxide	MgO	3-10	Thermal shock resistance; elasticity
Sodium oxide	Na ₂ O	1-5	Lowers melting point; controls melt viscosity
Potassium oxide	K ₂ O	1-5	Processability; thermal stability
Titanium dioxide	TiO ₂	0.5-2	Hardness; UV resistance

2.3 CuSn10 bronze powder

A proportion of the composite specimens was modified by incorporating CuSn10 tin-bronze powder into the epoxy matrix prior to lamination. The powder was supplied by Easy Composites Ltd (Stoke-on-Trent ST3 5XA, UK) and has a nominal particle size of approximately 74.5 µm. Its chemical composition falls within the range of 10.0-12.0wt% tin with the balance being copper, which is consistent with the CuSn10 designation under standard bronze specifications. The rationale for selecting this metallic filler rests on several previously documented effects in fiber-reinforced composites. Bronze powder increases the thermal conductivity of the epoxy matrix, facilitating heat dissipation from the sliding contact and reducing the likelihood of thermally induced softening during wear testing [13]. At the tribological interface, bronze particles can contribute to the formation of a ductile protective film on the counterface, which lowers the coefficient of friction and shields the basalt fibers from direct abrasive contact with the steel ball [12, 13]. In the present study, a fixed addition of 10 wt% CuSn10 was selected for the modified composite variant, based on the previously published finding from our research group that this concentration yields the highest tensile strength (440.6 MPa) among the tested compositions, while offering the second-best tribological performance, with the optimal tribological behavior observed at 15 wt% CuSn10 [13]. Given that mechanical strength was the primary requirement for the present application, 10 wt% was considered the most suitable overall compromise.

2.4 Steel ball counterpart

Tribological testing was conducted using AISI 52100 high-carbon chromium bearing steel balls with a nominal diameter of 12.7 mm, obtained from RKB Bearing Industries Group (Balerna, Switzerland). AISI 52100 is the most widely used material for anti-friction bearing elements and is specified under ASTM A295; its high carbon content (0.98-1.10 wt%) together with the chromium addition (1.40-1.60 wt%) results in a through-hardened martensitic microstructure with excellent fatigue and wear resistance [28, 29]. The principal mechanical and physical properties of these balls are listed in Table 2. The low surface roughness ($R_a = 0.028\text{--}0.030\text{ }\mu\text{m}$) of the as-received balls ensures that the initial contact conditions are well defined and reproducible across all test specimens, which is important when comparing results obtained with different composite formulations and lubricants.

Table 2. Chemical composition and mechanical/physical properties of the AISI 52100 steel balls (Ø12.7 mm) used as counterparts.

Property	Value	Property	Value
Chemical composition (wt%)		Mechanical properties	
Fe	balance (96.5–97.3)	Hardness, HRC	54–58
C	0.98–1.10	Compressive strength (MPa)	2100–2200
Si	0.15–0.35	Tensile strength (MPa)	2000
Cr	1.40–1.60	Young's modulus, E (GPa)	200
Mn	0.25–0.45	Poisson's ratio, ν	0.30
Physical properties			
Surface roughness, R_a (µm)	0.028–0.030	Density, ρ (g·cm ⁻³)	7.81

Note: values represent manufacturer-specified ranges; Fe content given as balance.

2.5 Protic ionic liquid nanofluids

Four 2-hydroxyethylammonium (2-HEA)-based protic ionic liquid formulations were selected as external lubricants in this study: MSu di(2-hydroxyethylammonium) succinate, MCi tri(2-hydroxyethylammonium) citrate, MSu+0.1%G, and MCi+0.1%G, the latter two being dispersions of 0.1 wt% few-layer graphene in the respective neat PILs. All four formulations share the same 2-HEA cation, differing only in the anion structure and the presence or absence of graphene. This design allows the role of the anion (diacid succinate vs. triacid citrate) and of the graphene additive to be assessed independently from one another, which is one of the aims of the present investigation.

MSu is synthesized by a simple acid-base neutralization between monoethanolamine and succinic acid, yielding a halogen-free, fully organic compound (C, H, N, O only) with documented biodegradability and low ecotoxicity [16, 17]. Its tribological performance has been characterized in detail by Espinosa et al. [18], who reported that a boundary film of MSu formed on AISI 316L stainless steel after water evaporation produced a minimum friction coefficient of 0.0001 under pin-on-disc conditions, representing a 97% reduction relative to pure water. The main lubrication mechanism involves ionic adsorption of the PIL on the metal surface, which suppresses iron oxidation inside the wear track [18].

MCi is the triprotic analogue, combining three (2-hydroxyethylammonium) cations with a single citrate anion. The higher number of carboxylate and hydroxyl groups in the citrate anion results in a higher viscosity than that of succinate-based ionic providing more adsorption sites per molecule on metallic surfaces, which translates into a higher load-carrying capacity [19, 26]. Guo and Iglesias [19] demonstrated that DCi, bis-(2-hydroxyethylammonium) citrate at 2 wt% to mineral oil reduced the friction coefficient and the wear volume on a steel-on-steel contact by 16% and 40%, respectively, while Jimenez et al. [26] showed that neat DCi and related diprotic citrate PILs reduce friction by up to 70% compared with water on Ti6Al4V-sapphire contacts.

The graphene-containing nanofluids, MSu+0.1%G and MCi+0.1%G, are prepared by mechanical blending followed by ultrasonication of few-layer graphene (purity > 99%, 1-10 layers, supplied by IoLiTec Ionic Liquids Technologies GmbH, Heilbronn, Germany) in the corresponding neat PIL. The 0.1 wt% loading was chosen deliberately: at this concentration the dispersions remain Newtonian fluids (flow index n approximately 1), show no yield stress, and remain physically stable for at least two months, as confirmed by Raman spectroscopy and rheological characterization for the analogous ML+0.1%G nanofluid [25]. This contrasts with loadings of 0.5 wt% and above, which produce shear-thinning behavior and risk graphene agglomeration during tribological testing [25]. The lubrication mechanism in these ionanocarbon systems relies on the synergy between the PIL boundary film and the low-shear lamellar graphene tribofilm: the PIL promotes graphene dispersion and surface adsorption, while graphene sheets exfoliate under contact stress and create a protective carbon-rich layer on the worn surface, as confirmed by Raman and EDX analysis in related systems [21, 23, 24]. Table 3 summarizes the key characteristics and reported tribological performance of the four nanofluids.

Table 3. Summary of the four 2-HEA-based PIL nanofluids used as external lubricants in this investigation.

Code	Full name	Composition	Key tribological feature
MSu	Di(2-hydroxyethylammonium) succinate	2-HEA cation + succinate anion (diacid)	Ultralow COF (0.0001); 97% reduction vs. water; boundary film on steel [18]
MCi	Tri(2-hydroxyethylammonium) citrate	2-HEA cation + citrate anion (triacid)	Up to 70% COF reduction vs. water; enhanced load-carrying capacity [19, 26]
MSu+0.1%G	MSu with 0.1 wt% dispersed graphene	MSu + few-layer graphene, Newtonian at 0.1 wt%	Eliminates run-in friction; prevents steel surface damage [24, 25]
MCi+0.1%G	MCi with 0.1 wt% dispersed graphene	MCi + few-layer graphene; stable >2 months [25]	Prevents wear loss; deposits graphene-containing tribofilm [24]

3. Experimental Methodology

The experimental programme consisted of four stages: fabrication of composite specimens by autoclave processing, mechanical characterization under tensile and bending loading, tribological evaluation on a pin-on-disc tribometer under dry and lubricated conditions, and post-test surface analysis by scanning electron microscopy and energy-dispersive X-ray spectroscopy. The fabrication procedure and the tribological testing protocol are described in full in the following subsections, as they directly govern the quality and reproducibility of the specimens and the conditions under which the friction and wear data were generated. Mechanical testing, surface analysis, and statistical treatment of the results are described concisely, as their outcomes are reported in the dedicated results section.

3.1 Composite specimen fabrication

Both composite variants were manufactured using a hand lay-up wet impregnation process, followed by vacuum bagging and an autoclave cure cycle. This processing sequence ensured uniform fiber wet-out, controlled resin content, and minimal void formation. The fabrication sequence is illustrated in Figure 1. Seven plies of 2/2 twill-weave basalt fiber fabric (220 g·m⁻², 500 mm x 300 mm per ply) were stacked on a flat mold surface and individually impregnated using the EPIKOTE MGS LR135 / EPIKURE LH136 two-part epoxy system at a resin-to-hardener mass ratio of 100:35.2, as specified by the supplier. Resin was distributed over each ply by manual rolling (Figure 1), working from the center outward to displace trapped air and achieve consistent fiber coverage before the next ply was laid.

For the hybrid composite variant, CuSn10 bronze powder (10wt% of the total laminate mass) was pre-dispersed in the liquid resin by mechanical stirring for at least 15 minutes before impregnation commenced, to obtain the most uniform distribution of metallic particles throughout the laminate thickness that the hand lay-up process permits. Once all seven plies had been stacked and impregnated, the surface of the laminate was covered with a perforated high-density polyethylene release film (thickness 0.027 mm). This film permits excess resin to bleed outward under vacuum while preventing the overlying bleeder fabric from bonding to the laminate surface. A layer of porous bleeder felt was then placed over the release film; it absorbed the surplus resin and simultaneously provided a continuous evacuation path for any residual air remaining between the plies.

The entire stack mold, laminate, release film, and bleeder were enclosed in a vacuum bag sealed at the edges with sealant tape, as shown in Figure 1. A vacuum of no less than 0.9 bar was drawn from the bag before the assembly was transferred to the Maroso autoclave. The autoclave cure cycle, including applied pressure, vacuum level, heating and cooling rates, was tailored to the laminate configuration and the processing requirements of the composite material [12, 13]. Autoclave pressure acting on the vacuum bag during cure consolidates the laminate, compresses out remaining voids, and maintains the fiber volume fraction within the target range throughout the crosslinking reaction.

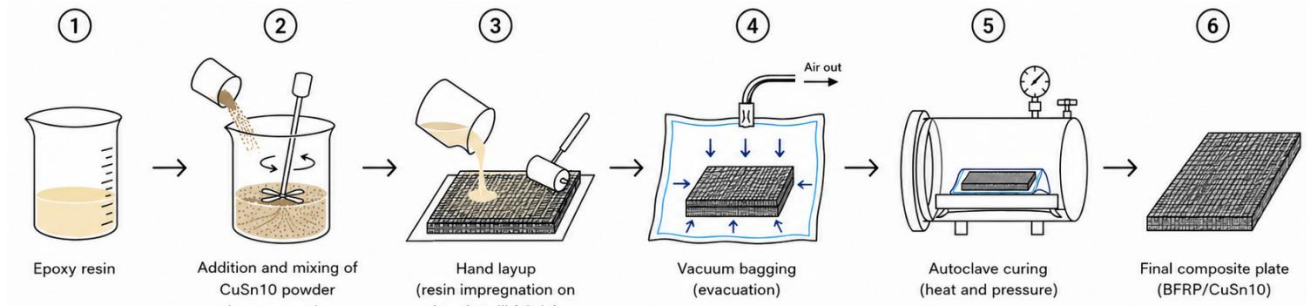


Figure 1. Schematic lay-up sequence showing ply stacking and vacuum bag assembly

After demolding and ambient-temperature post-cure, the consolidated laminates were cut and machined to the specimen geometries required for each test type. Tribological disc specimens were machined to a diameter of 32 mm and a thickness of 4 mm. Tensile specimens followed the rectangular geometry of ASTM D3039-17 and ISO 527-1:2019 (250 mm x 25 mm x 2 mm), and three-point bending specimens conformed to ASTM D7264 and ISO 178:2003 (80 mm x 13 mm x 2 mm). The fiber weight fraction of every batch was verified by mass measurement with a Kern ACJ 200-4M analytical balance (resolution 0.0001 g); acceptable ranges were defined as 57-63wt% for the 60% nominal target and 47-53wt% for the 50% target. Any batch falling outside these limits was rejected and remanufactured. The coefficient of variation for repeated mass measurements within a single batch was kept below 3%. The nominal compositions of the two variants produced are summarized in Table 4.

Table 4. Designation and nominal composition of the two composite variants fabricated in this study.

Designation	Basalt fiber (wt%)	Epoxy resin (wt%)	CuSn10 powder (wt%)
BFRP_60_CuSn10_0 (sample A)	60	40	0
BFRP_50_CuSn10_10 (sample B)	50	40	10

3.2 Pin-on-disc tribological testing

Friction and wear tests were performed on an Anton Paar TRB³ rotary tribometer (Anton Paar GmbH, Graz, Austria), shown in Figure 2, in compliance with ASTM G99 [32] and DIN 50324 [33]. The composite discs were mounted as the stationary lower specimen. The upper sliding counterpart was an AISI 52100 high-carbon chromium steel ball of 12.7 mm diameter. Before each test, disc surfaces were cleaned ultrasonically in acetone for 10 minutes and dried under a nitrogen stream to eliminate surface contamination that could interfere with the initial contact conditions or with the lubricant film formation.

The TRB³ is equipped with two independent friction force sensors and an elastic measurement arm designed to compensate for thermal drift during prolonged tests, which is particularly relevant for the long duration runs at the lowest sliding speed [37]. Friction force, normal load, temperature, and relative humidity were recorded continuously throughout each test. The coefficient of friction (COF) was computed in real time, and a mean steady-state value was extracted over the final 1800 m of each test, after the run-in transient had stabilized. A schematic of the pin-on-disc contact geometry and the tribometer setup is provided in Figure 3.



Figure 2. Anton Paar TRB³ pin-on-disc tribometer: (a) instrument overview; (b) close-up of the contact zone with composite disc (lower) and steel ball holder (upper).

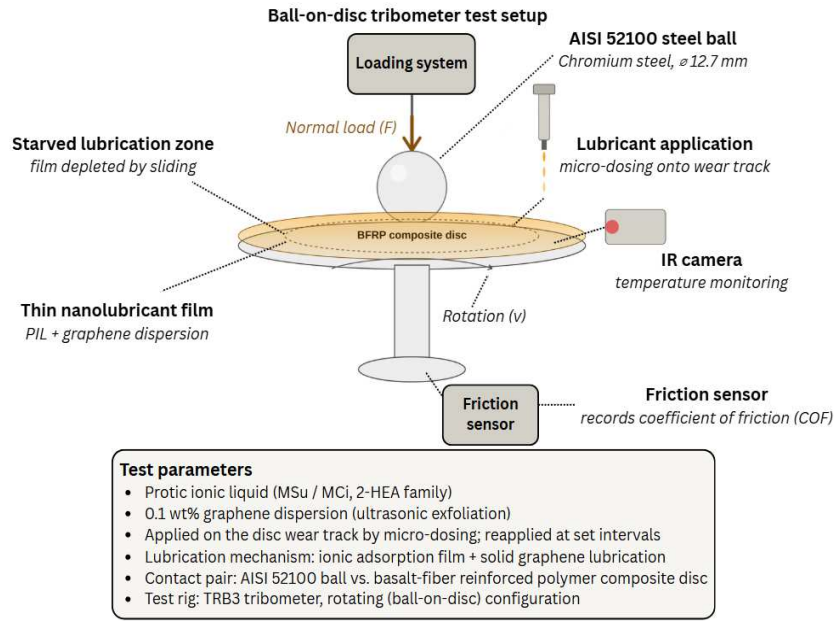


Figure 3. Schematic of the ball-on-disc contact geometry: normal load F applied through the AISI 52100 steel ball ($\varnothing$ 12.7 mm), wear track radius $r = 10$ mm, sliding direction indicated by the rotation arrow, and the PIL + graphene nanolubricant film in the contact zone.

The normal load (10 N) and sliding speed ($0.25 \text{ m}\cdot\text{s}^{-1}$) selected for the present study were informed by the prior tribological characterization of Cu- and CuSn10-filled BFRP composites carried out by our research group [12,13], in which dry-sliding tests were conducted across a range of normal loads (10, 20, and 30 N) and sliding speeds (0.1, 0.25, and $0.36 \text{ m}\cdot\text{s}^{-1}$). Among these conditions, the combination of 10 N and $0.25 \text{ m}\cdot\text{s}^{-1}$ consistently produced the most favorable dry-sliding tribological response, characterized by the lowest friction coefficient and specific wear rate for the CuSn10-reinforced composite [13]. This condition was therefore adopted as the fixed testing parameter set for the present investigation, ensuring that the baseline dry-sliding behavior of both composite variants could be directly compared with previously published data, while isolating the effect of PIL nanofluid lubrication as the primary experimental variable under optimal mechanical loading conditions.

Tests were conducted under a full factorial combination of 10N normal load and $0.25\text{m}\cdot\text{s}^{-1}$ sliding speed for 120 minutes. The wear track radius was fixed at 10 mm, and each test covered a total sliding distance of 1800 m. Five lubrication conditions were examined: dry sliding and four external PIL nanofluid lubricants (MSu, MCI, MSu+0.1%G, MCI+0.1%G). For lubricated tests, an initial volume of approximately 50-100 μL of each nanofluid was deposited by microsyringe directly onto the wear track immediately before the start of each test. A small additional quantity (approximately 30 μL) was periodically reapplied every 10 minutes to maintain a continuous boundary film over the full 1800 m sliding distance. For each test, three different batches of raw material were used (e.g., A-Dry1, A-Dry2, A-Dry3), with five measurements performed on each batch. The standard deviation was calculated based on these five measurements for each batch.

The complete test matrix is presented in Table 5.

Table 5. Full pin-on-disc test matrix showing lubrication conditions, normal load, sliding speed, sliding distance, and composite variants examined.

Common test parameters for all conditions: load 10 N, sliding speed $0.25 \text{ m}\cdot\text{s}^{-1}$, sliding distance 1800 m.

Lubricant / condition	Composites tested
Dry (no lubricant)	BFRP 60 / BFRP 50+CuSn10
MSu	BFRP 60 / BFRP 50+CuSn10
MCI	BFRP 60 / BFRP 50+CuSn10
MSu+0.1%G	BFRP 60 / BFRP 50+CuSn10
MCI+0.1%G	BFRP 60 / BFRP 50+CuSn10

Each of the 30 test conditions represents the meaning of five independent tribometer runs, so that the tribological campaign alone comprised of 150 individual pin-on-disc tests, providing a robust statistical basis for the GLM-ANOVA analysis presented in Section 4.3.

Post-test wear quantification was carried out using the optical profilometry module integrated into the TRB³ system. Cross-sectional profiles of the wear track were acquired at eight angular positions (Fig. 8b) on each disc. The mean cross-sectional wear area was determined by numerical integration of the four profiles, and the volumetric material loss V was calculated by multiplying the mean area by the circumferential length of the wear track. The specific wear rate k ($\text{mm}^3 \text{ N}^{-1} \text{ m}^{-1}$) was then obtained from the expression $k = V / (F \times d)$, where F is the applied normal force (N) and d the total sliding distance (m). All data acquisition and processing were carried out with TRB³ V10.0.x software.

3.3 Complementary characterization methods

Mechanical characterization, tensile tests on an INSTRON 8801 machine (ISO 527-1:2019 [34], ASTM D3039-17 [36]) and three-point bending tests on an INSTRON 3366-10 kN frame (ISO 178:2003, ASTM D7264 [35]) was carried out to verify the structural integrity of the fabricated composites and to confirm that the targeted fiber and filler contents were achieved. The results of this characterization are reported in reference [13] and are cited in the results section where they provide context for the tribological findings.

Worn disc surfaces were examined after tribological testing by scanning electron microscopy (SEM) on a JEOL JSM-5600LV instrument and by energy-dispersive X-ray spectroscopy (EDS) using an Oxford Instruments ULTIM MAX 65 detector running AZtec 4.2 software. These analyses served to identify the wear mechanisms active under each test condition and to characterize the composition of any tribofilms or transfer layers at the sliding interface. The SEM-EDS results are presented and discussed in full in the results section.

The tensile and flexural properties of both composite variants, determined according to ISO 527-1:2019 and ISO 178:2003 respectively, are summarized in Table 6 and figure 4. These results are reported in full reference [13] and are reproduced here in condensed form to establish the structural baseline of the materials subjected to tribological testing.

Table 6. Summary of tensile and flexural properties of the two composite variants. CV = coefficient of variation. Data from reference [13].

Sample	Tensile strength (MPa) / CV (%)	Strain at max. load (%) / CV (%)	Elastic modulus E (GPa) / CV (%)	Flexural strength (MPa) / CV (%)	Flexural modulus (GPa) / CV (%)
BFRP_60_CuSn10_0	329.2 / 8.89	1.84 / 2.70	20.84 / 10.73	284.31 / 9.42	20.20 / 11.89
BFRP_50_CuSn10_10	440.6 / 4.22	2.62 / 5.68	18.40 / 2.04	280.41 / 4.00	21.74 / 9.69

The incorporation of 10wt% CuSn10 bronze powder into the basalt fiber-reinforced laminate (BFRP_50_CuSn10_10) produced a substantial increase in tensile strength relative to the unmodified composite (BFRP_60_CuSn10_0), rising from 329.2 MPa to 440.6 MPa, an improvement of approximately 34%. The strain at maximum tensile load also increased, from 1.84% to 2.62%, indicating that the bronze-modified composite accommodates greater deformation before failure. In contrast, the elastic modulus decreased slightly (from 20.84 GPa to 18.40 GPa), which reflects the substitution of a portion of the stiff basalt fiber fraction by the more ductile metallic filler. The flexural properties showed a different trend: flexural strength remained practically unchanged between the two variants (284.31 MPa vs. 280.41 MPa), while the flexural modulus of BFRP_50_CuSn10_10 (21.74 GPa) marginally exceeded that of BFRP_60_CuSn10_0 (20.20 GPa). The lower coefficients of variation obtained for BFRP_50_CuSn10_10 in both tensile and flexural tests suggest that the bronze particles contribute to greater compositional homogeneity across the laminate, consistent with the improved particle distribution achieved by pre-dispersing the powder in the resin before impregnation [13].

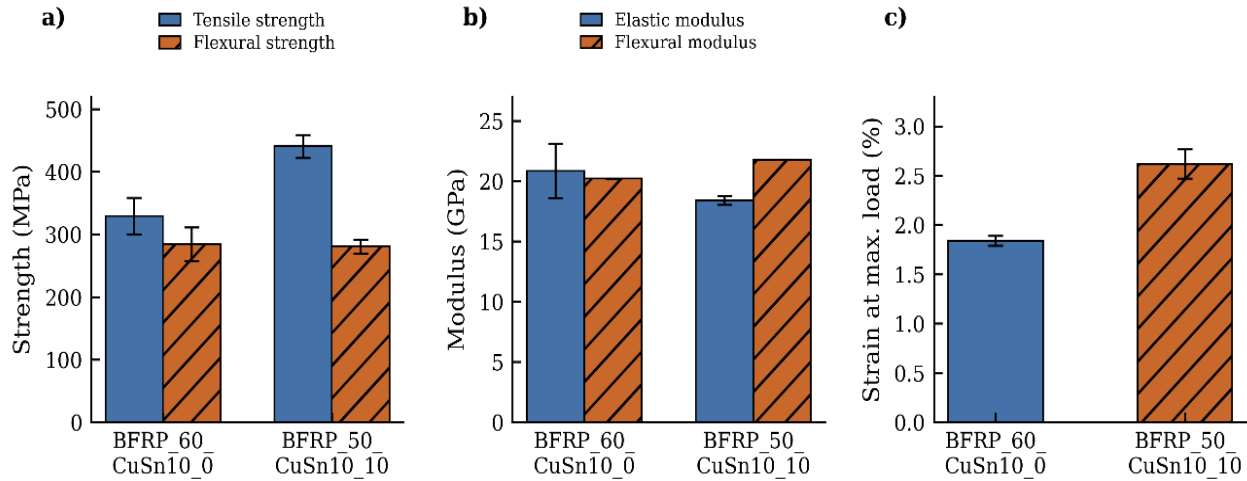


Figure 4. Comparison of tensile and flexural properties of BFRP_60_CuSn10_0 and BFRP_50_CuSn10_10 composites (data from [13]).

A design of experiments with two factors, lubricant condition with five levels and composite material type with two levels was performed, with each condition replicated at least three times as described in Section 3.2 (Table 7). Statistical analysis was performed within the Minitab 19 software (Coventry, UK). Statistical significance of the effects of composite material type and lubricant condition on the COF and specific wear rate (k) was assessed by General Linear Model (GLM) analysis of variance (ANOVA) with Box-Cox transformation of data. The Box-Cox transformation statistical technique was used to convert non-normal data into a normal distribution. Thus, it was used as an optimal parameter λ of 0.032135 in the case of COF, and 0.13265 for k , to stabilize variance. The statistical outputs, main effects plots, interaction plots, and ANOVA tables are included in the results section alongside the tribological data they interpret. The normality assumption of ANOVA was verified by examining the residuals.

4. Results

This section presents the experimental results obtained for the two composite variants BFRP_60_CuSn10_0 and BFRP_50_CuSn10_10 across all test conditions. The section is organized into two parts: tribological results under dry sliding and PIL nanofluid lubrication, comprising coefficient of friction (COF) and specific wear rate data; and surface characterization by SEM and EDS of selected worn specimens. The discussion of these results considering existing literature is reserved for section 5.

4.1 Tribological results

Tribological testing was carried out under the conditions described in Section 3.2: a normal load of 10 N, a sliding speed of $0.25 \text{ m}\cdot\text{s}^{-1}$, a total sliding distance of 1800 m (120 minutes), and ambient conditions of $23 \pm 2^\circ\text{C}$ and $45 \pm 3\%$ relative humidity. Five lubrication conditions were examined: dry sliding and four external 2-HEA PIL nanofluids (MSu, MCI, MSu+0.1%G, MCI+0.1%G), each applied to both composite variants. For every condition, at least three replicate tests were performed, and mean values of COF and specific wear rate k are reported together with their coefficients of variation. The wear of the AISI 52100 steel ball counterpart was negligible under all test conditions and is not discussed further.

4.1.1 Coefficient of friction

Figure 5 presents representative COF versus sliding distance curves for BFRP_60_CuSn10_0 (Figure 5a) and BFRP_50_CuSn10_10 (Figure 5b) under all five lubrication conditions. The dry sliding curves are included as a reference baseline. All four PIL nanofluids reduced the COF substantially relative to dry sliding for both composite variants, though the magnitude and temporal stability of the reduction differed between lubricants and between composites, as described below.

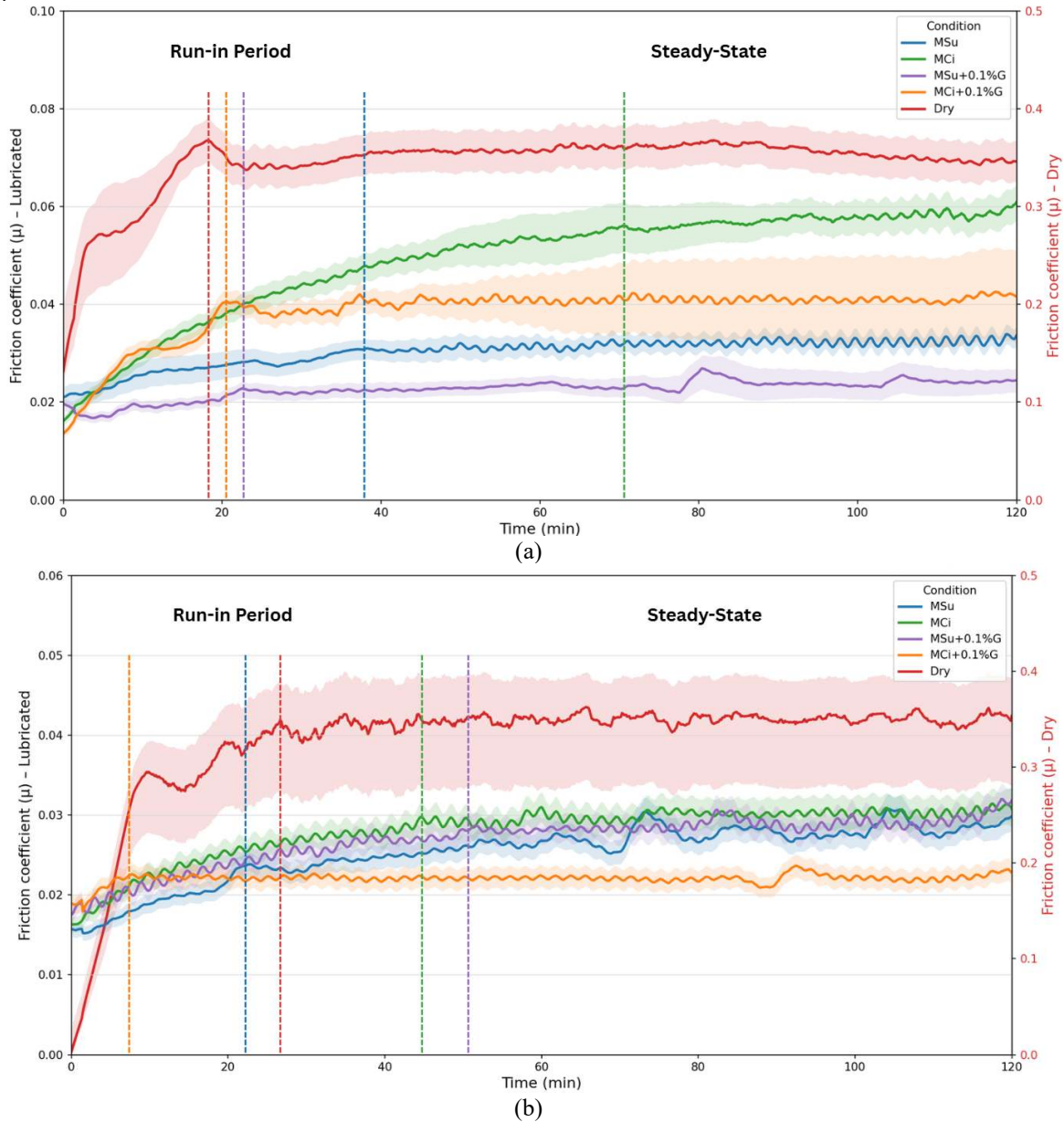


Figure 5. COF as a function of sliding distance under dry and PIL nanofluid lubrication: (a) BFRP_60_CuSn10_0; (b) BFRP_50_CuSn10_10. Curves represent the mean of at least three replicates.

Under dry sliding, both composites exhibited a pronounced run-in period characterized by a rapid initial rise in COF followed by gradual stabilization. Mean steady-state COF values and their variability for all conditions are compiled in Table 7. The graphene-containing nanofluids (MSu+0.1%G and MCi+0.1%G) notably shortened or eliminated the run-in transient compared with the neat PILs, in agreement with the behavior reported for analogous succinate-based PIL-graphene systems on metal contacts [24]. The steady-state COF values under PIL lubrication were consistently lower than those recorded under dry conditions for both composites, with the relative ranking of the four lubricants discussed in Section 5. Table 7 presents results for three independent batches per sample (denoted 1, 2, 3), each based on five measurements used to compute the standard deviation.

Table 7. Tribological test results for Sample A (60% basalt fiber composite) and Sample B (50% basalt fiber + 10% CuSn composite) under dry and lubricated conditions ($F = 10 \text{ N}$, $v = 0.25 \text{ m/s}$, $t = 120 \text{ min}$, $n = 3 \text{ batches} \times 5 \text{ measurements}$).

No.	Test Code	Condition	Lubricant / Medium	COF $\pm$ SD	Wear Rate, $k [\text{mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}] \pm \text{SD}$
Sample A – Composite reinforced with 60% basalt fiber ($F = 10 \text{ N}$, $v = 0.25 \text{ m/s}$, $t = 120 \text{ min}$)					
1	A-Dry-1	Dry	No lubricant	0.3608 ± 0.0126	$3.291 \times 10^{-5} \pm 1.045 \times 10^{-6}$
2	A-Dry-2	Dry	No lubricant	0.3292 ± 0.0137	$3.148 \times 10^{-5} \pm 1.084 \times 10^{-6}$
3	A-Dry-3	Dry	No lubricant	0.3439 ± 0.0129	$3.186 \times 10^{-5} \pm 0.972 \times 10^{-6}$
4	A-MSu-1	Lubricated	MSu (2-HEA succinate)	0.0267 ± 0.0010	$2.262 \times 10^{-5} \pm 0.408 \times 10^{-6}$
5	A-MSu-2	Lubricated	MSu (2-HEA succinate)	0.0382 ± 0.0018	$3.076 \times 10^{-5} \pm 1.5347 \times 10^{-6}$
6	A-MSu-3	Lubricated	MSu (2-HEA succinate)	0.0326 ± 0.0015	$2.987 \times 10^{-5} \pm 1.1946 \times 10^{-6}$
7	A-MCi-1	Lubricated	MCi (2-HEA citrate)	0.0655 ± 0.0028	$1.399 \times 10^{-5} \pm 0.6607 \times 10^{-6}$
8	A-MCi-2	Lubricated	MCi (2-HEA citrate)	0.0514 ± 0.0022	$1.209 \times 10^{-5} \pm 0.3846 \times 10^{-6}$
9	A-MCi-3	Lubricated	MCi (2-HEA citrate)	0.0599 ± 0.0020	$1.416 \times 10^{-5} \pm 0.6938 \times 10^{-6}$
10	A-MSu+G-1	Lubricated	MSu + 0.1% graphene	0.0295 ± 0.0014	$4.565 \times 10^{-6} \pm 2.1816 \times 10^{-7}$
11	A-MSu+G-2	Lubricated	MSu + 0.1% graphene	0.0178 ± 0.0007	$2.306 \times 10^{-6} \pm 0.775 \times 10^{-7}$
12	A-MSu+G-3	Lubricated	MSu + 0.1% graphene	0.0254 ± 0.0010	$3.201 \times 10^{-6} \pm 1.126 \times 10^{-7}$
13	A-MCi+G-1	Lubricated	MCi + 0.1% graphene	0.0365 ± 0.0018	$1.671 \times 10^{-6} \pm 0.776 \times 10^{-7}$
14	A-MCi+G-2	Lubricated	MCi + 0.1% graphene	0.0475 ± 0.0016	$4.657 \times 10^{-6} \pm 2.169 \times 10^{-7}$
15	A-MCi+G-3	Lubricated	MCi + 0.1% graphene	0.0387 ± 0.0015	$2.987 \times 10^{-6} \pm 0.932 \times 10^{-7}$
Sample B – Composite 50% basalt fiber + 10% CuSn powder ($F = 10 \text{ N}$, $v = 0.25 \text{ m/s}$, $t = 120 \text{ min}$)					
16	B-Dry-1	Dry	No lubricant	0.2403 ± 0.0073	$1.547 \times 10^{-5} \pm 0.6303 \times 10^{-6}$
17	B-Dry-2	Dry	No lubricant	0.4237 ± 0.0197	$1.012 \times 10^{-5} \pm 0.474 \times 10^{-6}$
18	B-Dry-3	Dry	No lubricant	0.3876 ± 0.0169	$1.465 \times 10^{-5} \pm 0.551 \times 10^{-6}$
19	B-MSu-1	Lubricated	MSu (2-HEA succinate)	0.0382 ± 0.0016	$1.479 \times 10^{-6} \pm 0.649 \times 10^{-7}$
20	B-MSu-2	Lubricated	MSu (2-HEA succinate)	0.0189 ± 0.0008	$0.9983 \times 10^{-6} \pm 0.443 \times 10^{-7}$
21	B-MSu-3	Lubricated	MSu (2-HEA succinate)	0.0287 ± 0.0014	$1.2560 \times 10^{-6} \pm 0.489 \times 10^{-7}$
22	B-MCi-1	Lubricated	MCi (2-HEA citrate)	0.0275 ± 0.0009	$0.835 \times 10^{-6} \pm 0.282 \times 10^{-7}$
23	B-MCi-2	Lubricated	MCi (2-HEA citrate)	0.0338 ± 0.0013	$0.526 \times 10^{-6} \pm 0.172 \times 10^{-7}$
24	B-MCi-3	Lubricated	MCi (2-HEA citrate)	0.0298 ± 0.0014	$0.783 \times 10^{-6} \pm 0.328 \times 10^{-7}$
25	B-MSu+G-1	Lubricated	MSu + 0.1% graphene	0.0275 ± 0.0013	$1.2040 \times 10^{-6} \pm 0.4545 \times 10^{-7}$
26	B-MSu+G-2	Lubricated	MSu + 0.1% graphene	0.0307 ± 0.0011	$2.3880 \times 10^{-6} \pm 0.8463 \times 10^{-7}$
27	B-MSu+G-3	Lubricated	MSu + 0.1% graphene	0.0301 ± 0.0009	$1.9870 \times 10^{-6} \pm 0.706 \times 10^{-7}$
28	B-MCi+G-1	Lubricated	MCi + 0.1% graphene	0.0200 ± 0.0007	$0.457 \times 10^{-7} \pm 0.218 \times 10^{-8}$
29	B-MCi+G-2	Lubricated	MCi + 0.1% graphene	0.0284 ± 0.0014	$0.568 \times 10^{-7} \pm 0.1923 \times 10^{-8}$
30	B-MCi+G-3	Lubricated	MCi + 0.1% graphene	0.0180 ± 0.0009	$0.602 \times 10^{-7} \pm 0.251 \times 10^{-8}$

Abbreviations: MSu = 2-hydroxyethylammonium succinate (PIL) | MCi = 2-hydroxyethylammonium citrate (PIL) | G = graphene | COF = friction coefficient (mean over full test) | SD = standard deviation (instrument repeatability, 3–5%).

As shown in Figures 6 and 7, the introduction of PIL lubrication produced a dramatic reduction in COF for both composite variants, from approximately 0.34–0.35 under dry sliding to values below 0.06 under all lubricated conditions, a reduction exceeding one order of magnitude. For Sample A, the succinate-based PIL (MSu) yielded the lowest COF among the neat lubricants (≈ 0.033), while MCi produced a moderately higher value (≈ 0.059). The addition of 0.1% graphene further reduced friction relative to MSu alone (≈ 0.024), confirming the beneficial tribofilm-forming effect of graphene nanoparticles observed in Section 4. A similar trend was noted for Sample B, where the CuSn10 addition appears to narrow the gap between the four lubricated conditions (0.022–0.030), with MCi+0.1%G giving the lowest overall COF (≈ 0.022). Notably, Sample B also displayed larger scatter in the dry condition ($\text{SD} \approx 0.097$), attributable to the heterogeneous, metal-containing surface interacting with the counterface before lubricant film formation. Overall, both figures confirm that PIL lubrication particularly when combined with graphene consistently lowers and stabilizes the friction response compared with dry sliding, for both basalt- fiber composite formulations.

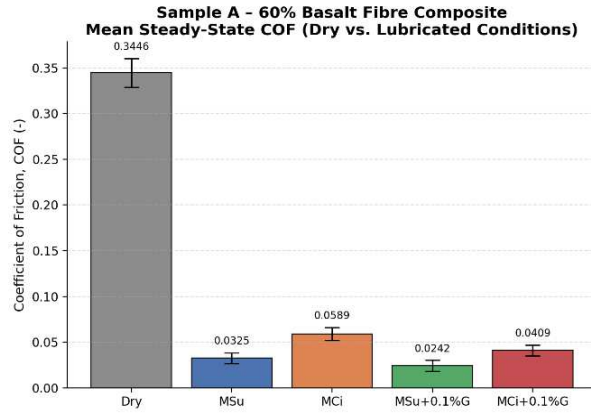


Figure 6. Mean steady-state COF ($\pm$ SD) for Sample A (60% basalt fiber composite) under dry sliding and PIL-based lubrication (MSu, MCI, MSu+0.1%G, MCI+0.1%G)

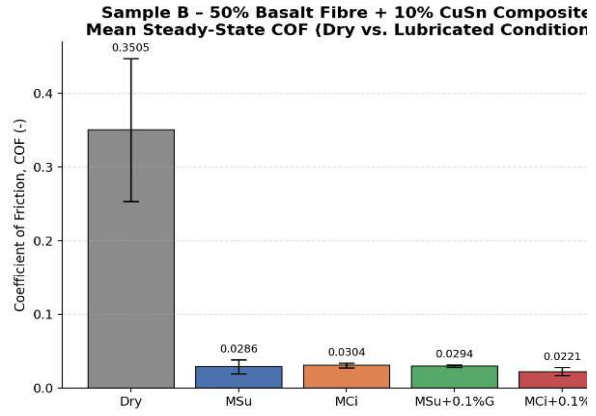


Figure 7. Mean steady-state COF ($\pm$ SD) for Sample B (50% basalt fiber + 10% CuSn composite) under dry sliding and PIL-based lubrication (MSu, MCI, MSu+0.1%G, MCI+0.1%G).

4.1.2 Specific wear rate

Figure 8 illustrates the methodology used to quantify volumetric wear on the composite disc specimens. Panel (a) shows a representative cross-sectional profile extracted from the wear track, highlighting the characteristic groove geometry, including edge pile-up and the central wear-mark area (A) used for volumetric wear calculations while panel (b) depicts the angular scanning scheme, whereby eight profilometric cross-sections were acquired at 45° intervals around the circular wear track (positions 1–8) to minimize the influence of local topographic heterogeneity, such as fiber pull-out or matrix cracking, on the estimated wear volume.

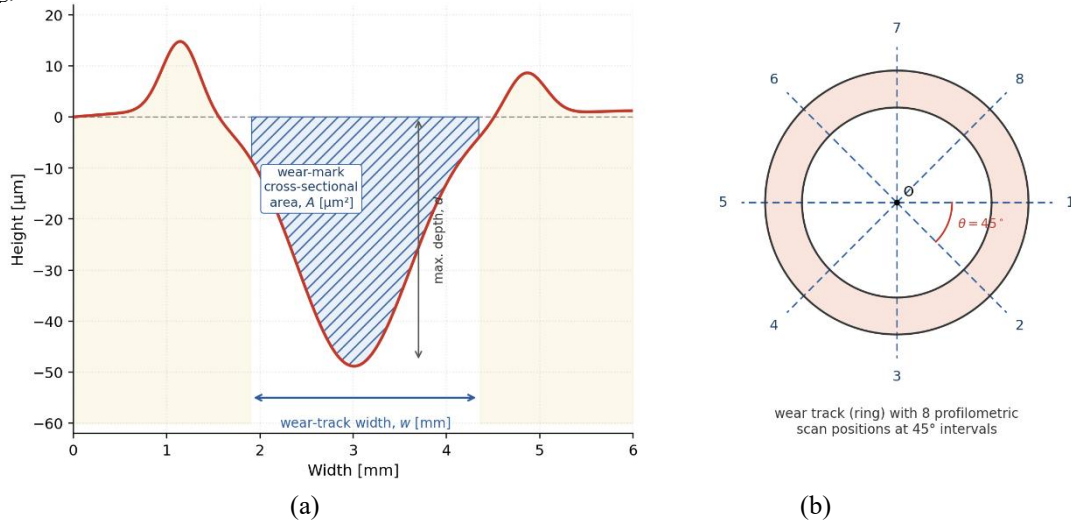


Figure 8. Schematic of the wear-track cross-sectional profile (a) and the eight-position profilometric-scanning scheme used for volumetric wear determination (b).

As illustrated in Figures 9 and 10, PIL lubrication reduced the specific wear rate substantially relative to dry sliding for both composite variants, confirming the trend already observed for the friction coefficient. For Sample A, the specific wear rate dropped from $3.208 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ under dry sliding to 2.775×10^{-5} with MSu and 1.341×10^{-5} with MCI, while the graphene-dispersed nanofluids produced the lowest wear rates overall (0.336×10^{-5} for MSu+0.1%G and 0.317×10^{-5} for MCI+0.1%G) nearly an order of magnitude below the neat PILs. This indicates that graphene nanoparticles not only lower friction but also contribute a distinct protective mechanism at the sliding interface, most likely by forming a lubricious tribofilm that limits direct fiber–counterface contact and suppresses abrasive fiber pull-out.

The wear behaviour of Sample B followed a markedly different pattern. Even under dry sliding, its specific wear rate ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) was already comparable to the MCI-lubricated condition of Sample A, and it decreased further to values below 0.19×10^{-5} under all lubricated conditions, reaching a minimum of $0.0054 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ for MCI+0.1%G over two orders of magnitude lower than the dry Sample A baseline. This behaviour is attributed to the CuSn10 particles embedded in the composite, which appear to act as a secondary solid-lubricant/load-bearing phase at the tribological interface, complementing the PIL and graphene lubrication mechanisms and further restricting fiber-matrix degradation. The comparatively higher scatter observed for Sample A under dry sliding and MSu lubrication (Figure 8) is consistent with the heterogeneous, fiber-pull-out-dominated wear mechanism typical of unreinforced-matrix dry contact, whereas the CuSn10-containing composite (Sample B) shows more consistent, lower-magnitude wear across all conditions, reflecting a more stable and protective interfacial response. A detailed discussion of the role of CuSn10 particles is provided in Section 5.

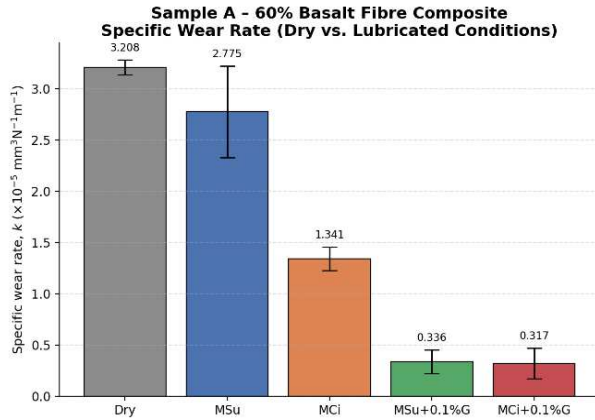


Figure 9. Specific wear rate k ($\times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, $\pm$ SD) for Sample A (60% basalt fiber composite) under dry sliding and PIL nanofluid lubrication.

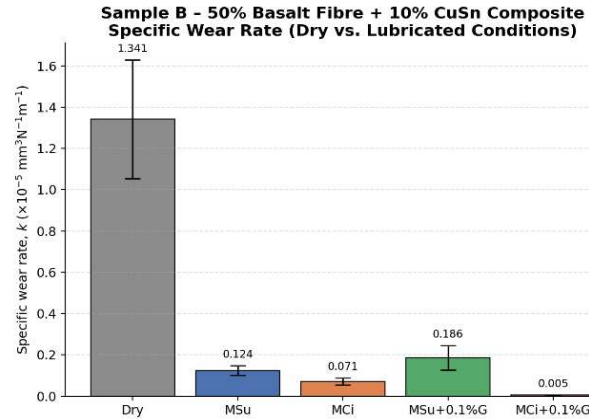


Figure 10. Specific wear rate k ($\times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, $\pm$ SD) for Sample B (50% basalt fiber + 10% CuSn composite) under dry sliding and PIL nanofluid lubrication.

4.1.3 Contact temperature

Contact temperature was continuously monitored throughout each test using the integrated sensor of the TRB³ tribometer. Across all tested conditions, the recorded values did not show significant variations relative to the ambient (room) temperature at which the experiments were conducted. Consequently, no further interpretation of the contact temperature data was considered necessary in the context of this study.

4.2 Worn surface analysis: SEM and EDS

Representative SEM micrographs of the worn disc surfaces after dry sliding and after lubrication with each of the four PIL nanofluids are presented in Figures 11 and 12 for BFRP_60_CuSn10_0 and BFRP_50_CuSn10_10, respectively. The images were acquired in BSE (Backscattered Electron mode) mode to maximize compositional contrast between the epoxy matrix, the basalt fibers, the CuSn10 particles (where present), and any tribofilms deposited during sliding.

Figure 11(a) provides a representative BSE-SEM view of the BFRP_60_CuSn10_0 wear track after dry sliding, with EDS point analyses correlated to the observed surface morphology. The bright, longitudinally striated region on the left side of the micrograph corresponds to an exposed and fractured basalt fiber bundle; the parallel striations running across this area are abrasive grooves produced by directional sliding contact with the steel counterface. This assignment is supported by the EDS analysis at Spectrum 8, which shows a fiber-like signature (Si 12.0%, Al 4.1%, Fe 3.6%, Mg 1.6%, Ca 1.8%). The smoother, mid-gray patch sampled at Spectrum 9 is dominated by carbon and oxygen (C 73.5%, O 24.3%) with only trace mineral content, identifying it as an epoxy-rich region where the matrix is recessed relative to the harder, protruding fiber ends. Between these two zones, the mottled, porous-looking area sampled at Spectrum 10 shows an intermediate composition (O 46.6%, C 42.4%, Si 5.7%, Al 2.5%), consistent with a mixed fiber-matrix debris layer formed by fractured fiber fragments intermixed with abraded resin. Together, these features corroborate the combined abrasive and adhesive wear mechanisms expected under dry sliding, with basalt fiber fracture, matrix recession, and debris redistribution all evident within a single field of view.

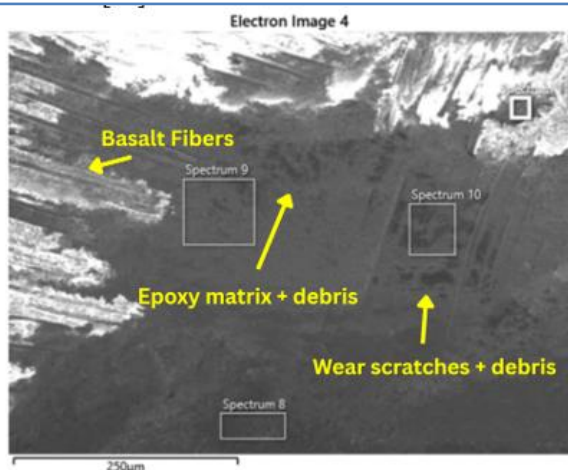
Figure 11(b) shows the BFRP_60_CuSn10_0 wear track after lubrication with the neat MSu nanofluid. Compared with the dry sliding condition, the surface retains a much more continuous, longitudinally oriented fiber morphology, with markedly less fragmentation and a smoother overall appearance, indicating that the PIL boundary film substantially suppressed abrasive damage. The fiber ridge sampled at Spectrum 3 still shows a basalt-related signature but with a comparatively higher oxygen contribution and reduced silicon relative to the dry-sliding fiber regions, consistent with a thin, adsorbed PIL film covering the exposed fiber surface rather than a fully bare, freshly fractured fiber. The area sampled at Spectrum 4, dominated by carbon with only trace mineral content, corresponds to a resin-rich region where lubricant and matrix material have been smeared along the sliding direction, forming a continuous protective layer over the underlying fibers. These observations are consistent with the substantial reduction in COF and specific wear rate recorded for the MSu condition relative to dry sliding (Section 4.1) and support a lubrication mechanism dominated by PIL boundary-film adsorption rather than solid-lubricant action, in agreement with the mechanism reported for 2-HEA-type PILs on metallic and polymeric surfaces [18].

Figure 11(c) shows the BFRP_60_CuSn10_0 wear track after lubrication with the neat MCI nanofluid. The micrograph is dominated by two distinct zones: a rough, textured resin matrix occupying roughly the left half of the field, and a region of exposed, rod-shaped basalt fiber sections, corresponding to fibers sectioned transverse to their length, on the right. Unlike the continuous, longitudinally intact fiber ridges observed under MSu lubrication (Figure 11b), the fibers here appear as discrete, individually smooth-faced fragments rather than one unbroken bundle, consistent with localized fiber breakage accompanying matrix wear. EDS analysis at Spectrum 6 and Spectrum 7, both located within the resin-dominated zone, is dominated by oxygen and nitrogen (O 57.5%, N 15.8% and O 46.0%, N 23.1%, respectively), a signature consistent with an adsorbed 2-HEA PIL boundary film rather than bare matrix. Both spectra additionally show minor copper and zinc (Cu 10.6%, Zn 2.9% and Cu 12.9%, Zn 6.0%) without any detectable tin, indicating that this signal does not originate from the CuSn10 filler used in the companion composite, but rather from trace metallic contact transfer,

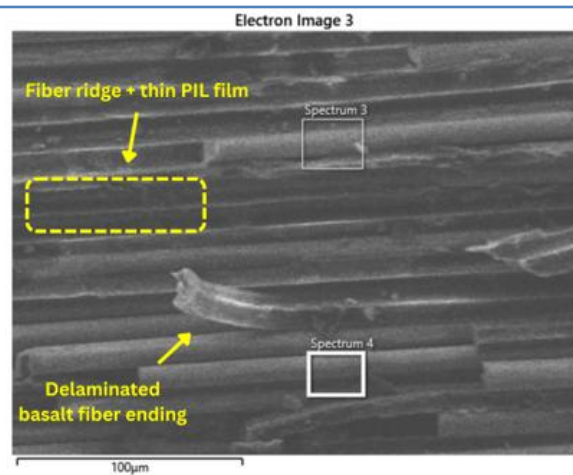
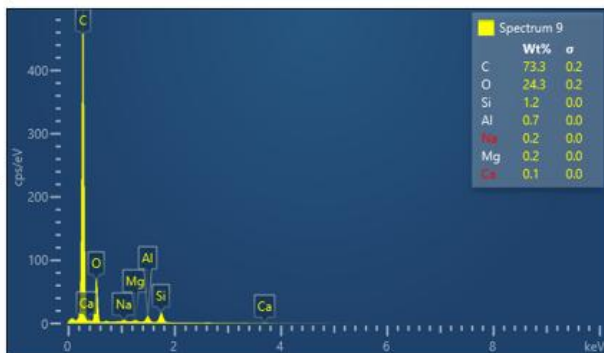
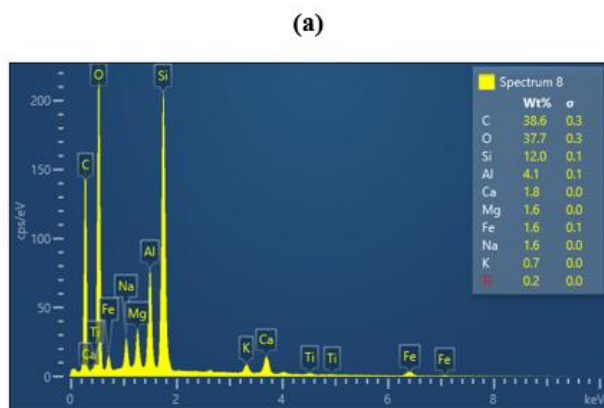
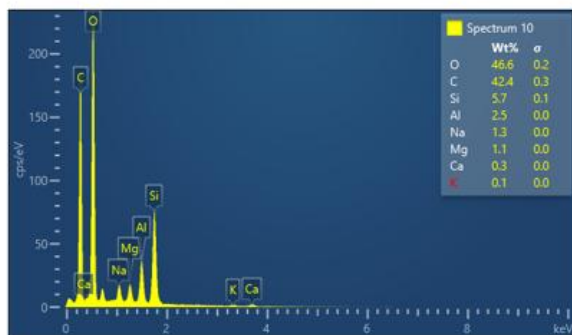
consistent with brass-type contamination unrelated to the reinforcement itself. This is compatible with the friction and wear data reported in Section 4.1: although MCI produced a moderately higher COF than MSu for this composite (≈ 0.059 vs. ≈ 0.033), its specific wear rate was nevertheless lower (1.341×10^{-5} vs. $2.775 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$), suggesting that the citrate-based PIL film, while less effective at reducing friction, still limited material removal, in line with SEM/EDS-based tribofilm evidence reported previously for DCI/MCI lubrication of metallic contacts [19].

Figure 11(d) shows the BFRP_60_CuSn10_0 wear track after lubrication with MSu+0.1%G. Four distinct morphological zones are identifiable within the field of view. In the upper region, a resin layer presenting fine scratch marks was sampled at Spectrum 2, showing a higher oxygen contribution and reduced silicon relative to the fiber regions (O 73.5%, Si 12.9%), together with a small chlorine contribution not detected at Spectrum 1. This pattern mirrors the fiber-surface signature attributed to an adsorbed PIL film under MSu lubrication in Figure 11(b), and is consistent with a thin, graphene-containing PIL boundary layer covering the resin surface. Adjacent to this, a visible boundary separates the resin layer from the underlying fiber matrix, marking the transition between the two phases. Toward the center of the micrograph, a wavy, folded fiber segment is identifiable as a delaminated and turned-over basalt fiber, a feature comparable to the raised, delaminated fiber ending reported for Figure 11(b), indicating that localized fiber lifting and turnover persisted under this lubrication condition despite the added graphene. Finally, the fractured basalt fibers oriented along the wear track, sampled at Spectrum 1, retain a clean basalt-related signature (O 64.2%, Si 18.9%, Al 6.8%, Na 3.3%, Mg 3.0%, Ca 1.9%, Fe 1.0%, K 0.9%), comparable to the fiber compositions reported for the dry-sliding and MSu conditions. These observations correlate with the friction and wear data reported in Section 4.1: MSu+0.1%G produced the lowest COF recorded for this composite among all lubricated conditions (≈ 0.024 , versus ≈ 0.033 for neat MSu), together with a specific wear rate of $0.336 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, nearly an order of magnitude below the neat PIL nanofluids. The persistence of fiber fracture and delamination alongside this substantial reduction in both friction and wear suggests that the graphene-containing PIL film primarily suppresses further material removal rather than eliminating pre-existing fiber damage, consistent with the protective tribofilm-forming role attributed to graphene nanoparticles at the sliding interface [22, 30].

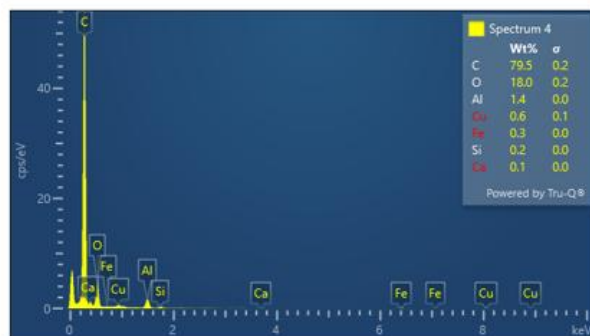
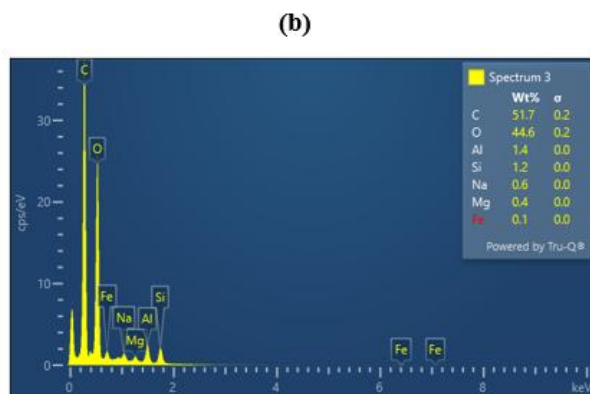
Figure 11(e) shows the BFRP_60_CuSn10_0 wear track after lubrication with MCI+0.1%G. The micrograph presents a resin debris region alongside fractured and worn basalt fibers. The resin-debris area sampled at Spectrum 6 is dominated by oxygen and nitrogen (O 71.2%, N 21.2%), with only a minor mineral contribution (Al 6.1%, Si 1.6%), consistent with a thick, continuous PIL boundary film covering the underlying debris. A comparable N+O-dominated signature was recorded at Spectrum 8 (O 79.0%, N 17.8%, Al 3.2%), located within the basalt fiber + debris area, where silicon is essentially absent; this suggests that the graphene-containing citrate PIL film extends continuously over the fiber region as well, largely masking the underlying basalt signature at this sampling point. By contrast, Spectrum 7, positioned in the transitional zone between the debris-covered matrix and the fiber bundle, shows no detectable nitrogen and a clear basalt-related signature (O 70.3%, Si 15.5%, Al 7.8%, Na 3.4%, Mg 2.9%, Fe 0.1%). This absence of a nitrogen signal at Spectrum 7 may reflect a local thinning or discontinuity in the tribofilm at this position, allowing the underlying fiber material to be sampled directly, though this cannot be confirmed without additional point analyses in the same region. This distribution, a near-continuous graphene-PIL film over both resin and fiber regions, interrupted only locally, is consistent with the friction and wear data reported in Section 4.1: MCI+0.1%G produced the lowest specific wear rate recorded for this composite among all conditions ($0.317 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, compared with 3.208×10^{-5} under dry sliding), alongside a substantially reduced COF (≈ 0.041) relative to neat MCI (≈ 0.059). These results support the interpretation that graphene incorporation promotes a more continuous, thin-film boundary layer at the sliding interface, in agreement with the coverage and load-bearing behavior reported for graphene-ionic liquid thin-film nanolubricants on metallic substrates [21].



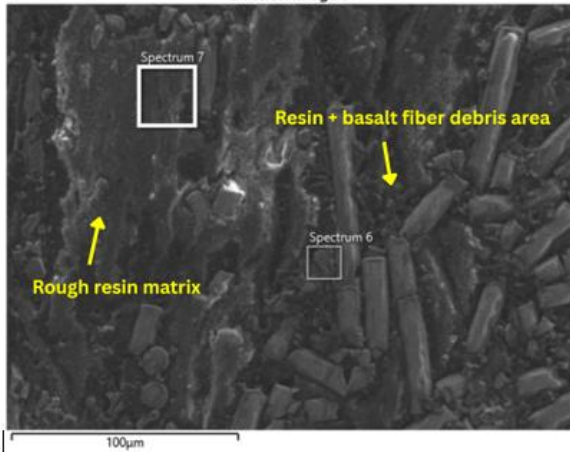
Annotated regions: fractured basalt fiber bundle (abrasive striations), recessed epoxy matrix, abrasive striations, and mixed fiber-matrix debris layer, correlated with EDS point analyses (Spectrum 8, 9, 10).



Annotated regions: basalt fiber ridge covered by a thin PIL boundary film and a raised delaminated basalt fiber ending.

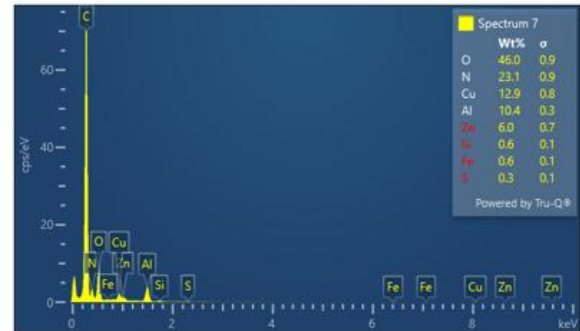
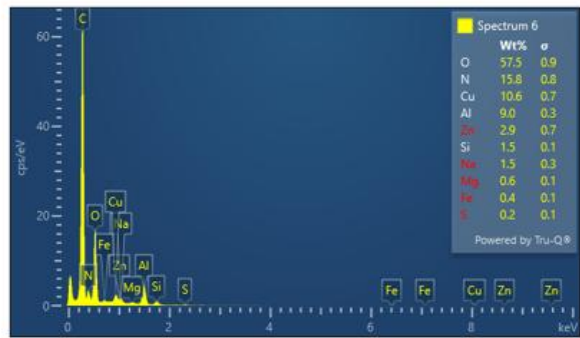


Electron Image 3

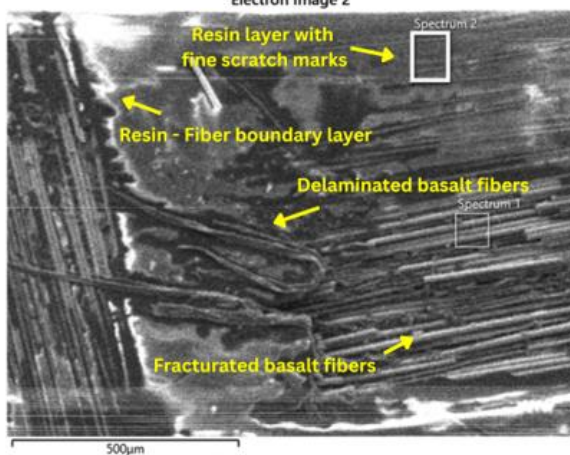


Annotated regions: Area presenting a rough resin matrix and debris composed of resin particles and larger basalt fiber sections.

(c)

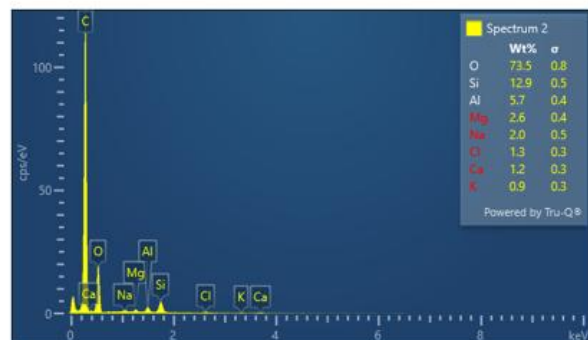
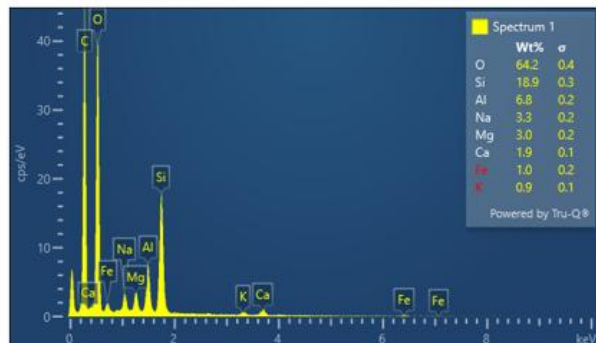


Electron Image 2



Annotated regions: visible boundary between resin and fiber layers, resin matrix presenting abrasive striations, fractured basalt fibers oriented along the wear track, and delaminated and turned-over basalt fibers.

(d)



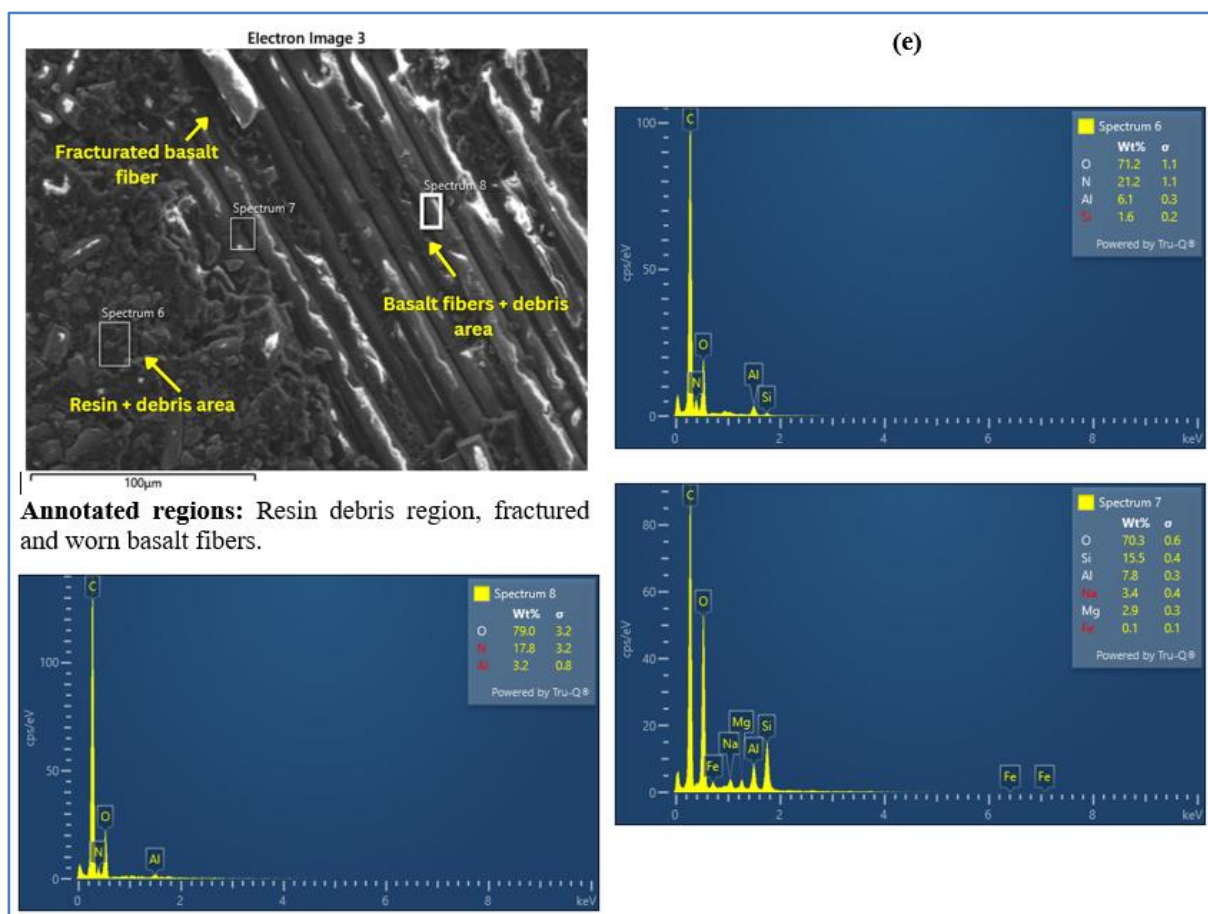


Figure 11. BSE-SEM micrographs of BFRP_60_CuSn10_0 wear tracks and EDS elemental maps of the wear track after (a) dry sliding, (b) MSu, (c) MCI, (d) MSu+0.1%G, and (e) MCI+0.1%G lubrication.

Figure 12(a) presents the BFRP_50_CuSn10_10 wear track after dry sliding. Three distinct features are identifiable within the field of view. A discrete fragment sampled at Spectrum 4 shows a metallic, low-oxygen composition dominated by copper and tin (Cu 85.2%, Sn 7.1%), with a Cu:Sn ratio close to the nominal CuSn10 filler composition, confirming this as an intact bronze particle embedded at the worn surface. Surrounding this fragment, a rough resin matrix was sampled at Spectrum 6, showing a substantial copper contribution but no detectable tin (O 41.1%, Cu 35.8%, Si 10.4%, Al 4.8%, Ca 2.2%, Fe 2.1%, Cl 1.8%, Mg 1.1%, Ti 0.8%). The absence of tin at this level of copper suggests that this signal may not originate directly from the intact CuSn10 filler, though the precise origin of this tin-depleted copper signature cannot be established from the present data alone and would benefit from further investigation. Toward the upper right of the micrograph, an exposed basalt fiber layer is visible, and the adjacent area sampled at Spectrum 5 shows a mixed signature combining basalt-consistent elements with both copper and tin (O 43.3%, Si 20.1%, Cu 16.1%, Al 8.3%, Ca 3.6%, Fe 2.8%, Mg 2.1%, Sn 1.7%, K 0.9%, Cl 0.5%, Ti 0.5%), at a Cu:Sn ratio consistent with the bronze filler, indicating that fine CuSn10 debris has been smeared onto or intermixed with the fiber-matrix material at this boundary. Together, these observations are consistent with the general description of this condition, bronze particles remained largely intact and partially proud of the worn surface, carrying a portion of the contact load and reducing direct abrasive action on the basalt fibers. This is compatible with the wear data reported in Section 4.1: even under dry sliding, Sample B's specific wear rate ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) was already comparable to the MCI-lubricated condition of Sample A, despite both samples exhibiting similar COF values under dry sliding (≈ 0.34 – 0.35), suggesting that the load-bearing role of the bronze particles measurably limited material removal from the earliest stage of sliding contact, independent of any lubrication effect.

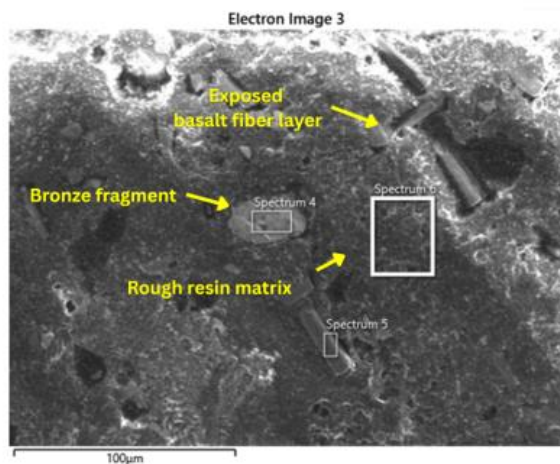
Figure 12(b) shows the BFRP_50_CuSn10_10 wear track after lubrication with the neat MSu nanofluid. The micrograph shows fine scratch marks across a fiber matrix covered by a thin PIL film, together with a resin matrix in the lower right of the field, and multiple circular features with raised edges and a central copper-rich particle. The fiber region sampled at Spectrum 1 shows a basalt-related signature with a small copper contribution (O 49.8%, Si 24.3%, Al 9.5%, Ca 4.5%, Fe 3.8%, Mg 2.4%, Cu 2.1%, Na 2.0%, K 1.0%, Ti 0.5%), while Spectrum 3, sampled slightly further from the metallic particle discussed, shows a comparable basalt signature without any detectable copper (O 62.1%, Si 18.5%, Al 7.8%, Ca 3.2%, Mg 2.1%, Fe 2.0%, Na 2.0%, Cl 1.6%, K 0.7%). Spectrum 2, sampled on an isolated point-like feature within this fiber region, shows a metallic, low-oxygen composition dominated by copper and tin (Cu 76.7%, Sn 12.6%), at a Cu:Sn ratio consistent with the CuSn10 filler, confirming this as an intact bronze particle embedded within the fiber bundle rather than a resin- or fiber-associated feature. The presence of an intact bronze particle together with only trace copper at the adjacent fiber point (Spectrum 1) suggests localized redistribution of copper away from the particle itself into the

immediately surrounding fiber-matrix material, consistent with the general pattern of Cu/Sn redistribution at fiber-matrix boundaries described for the lubricated bronze-filled condition. The circular raised-edge feature with its central copper-rich particle is seemingly associated with localized surface topography developing around a hard, load-bearing inclusion at the sliding interface. This raised, delimited rim could indicate some form of localized matrix response to the presence of the particle during sliding, whether thermal, mechanical, or otherwise. These observations correlate with the friction and wear data reported in Section 4.1: for Sample B, MSu lubrication produced a mean COF of ≈ 0.029 and a specific wear rate of $\approx 0.124 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \cdot \text{m}^{-1}$, well below the dry-sliding value for this composite ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) and consistent with the interpretation that the CuSn10 particles act as a secondary load-bearing phase that, together with the PIL boundary film, restricts further fiber-matrix degradation under lubricated sliding.

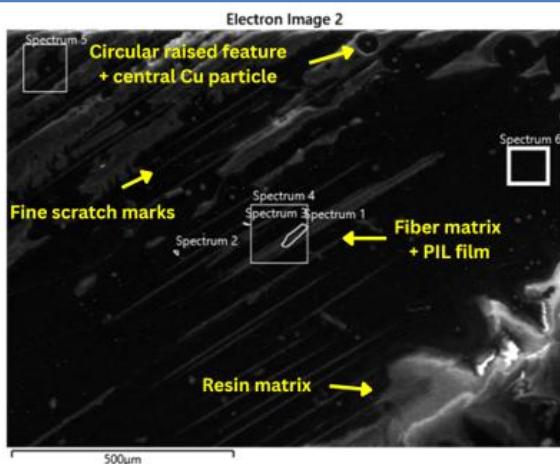
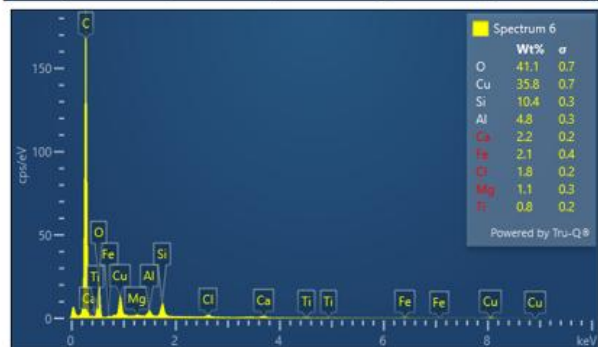
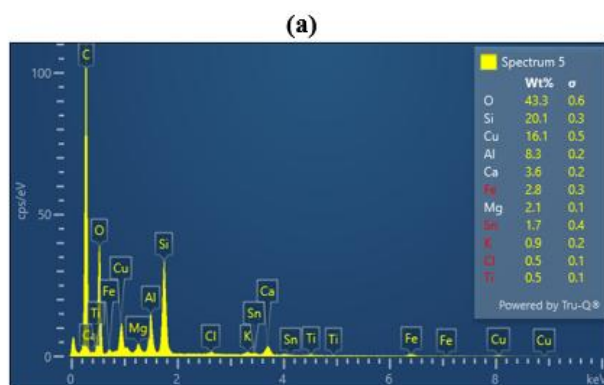
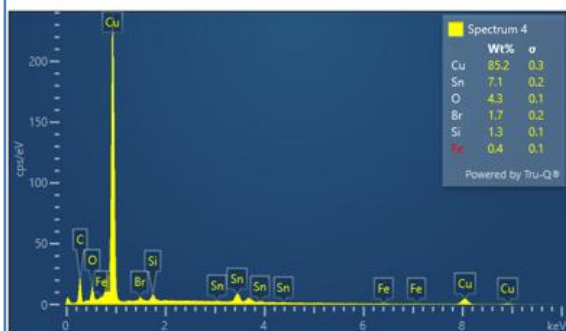
Figure 12(c) shows the BFRP_50_CuSn10_10 wear track after lubrication with the neat MCi nanofluid. The field of view contains a basalt fiber region, a resin matrix area, and a circular feature with a raised edge and an apparent central copper-rich particle, comparable to the feature described for Figure 12(b). The fiber region sampled at Spectrum 1 shows a clean basalt-related signature (O 66.0%, Si 18.6%, Al 8.3%, Na 2.3%, Mg 2.2%, Ca 1.8%, K 0.5%, Ti 0.2%, Fe 0.1%). Spectrum 2, sampled on the circular feature, shows an oxide-dominated composition with no detectable copper (O 95.5%, Al 3.4%, Cl 1.0%), consistent with a position on the raised rim rather than the central particle. The whole-field Map Sum Spectrum confirms that copper is present within this field. The recurrence of this raised-edge, centrally-particled morphology in two independent fields of view (Figures 12b and 12c) suggests that it is a genuine, repeatable feature of the worn surface under PIL lubrication rather than an isolated artifact. These observations are broadly consistent with the friction and wear data reported in Section 4.1: for Sample B, MCi lubrication produced a mean COF of ≈ 0.030 and a specific wear rate of $\approx 0.072 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \cdot \text{m}^{-1}$, both comparable to the MSu condition and substantially below the dry-sliding baseline for this composite, supporting the broader interpretation that the CuSn10 particles contribute a load-bearing, protective role that persists across the different PIL lubricants tested.

Figure 12(d) shows the BFRP_50_CuSn10_10 wear track after lubrication with MSu+0.1%G. Four morphological zones are marked within the field of view. In the upper region, a resin matrix presenting fine scratches was sampled at Spectrum 3, showing a clear CuSn10-consistent signature (O 69.1%, Cu 24.3%, Sn 3.6%, Cl 3.0%), confirming that bronze debris has been smeared into the resin surface at this location. Toward the center of the micrograph, a needle-like, raised feature is identifiable as a delaminated basalt fiber ending, comparable to the raised, turned-over fiber feature reported for Figure 11(b), indicating that localized fiber lifting persisted in this bronze-filled composite under MSu+0.1%G lubrication. Below this, in the fractured basalt fiber matrix, Spectrum 2 shows an unusually high titanium content alongside a bronze-derived copper signal (O 49.2%, Ti 33.9%, Cu 6.9%, Si 3.8%, Al 3.1% etc). This titanium level is well above the trace amounts typically associated with basalt fiber bulk composition, and most plausibly reflects a localized accessory Ti-oxide mineral inclusion, such as ilmenite, intersected directly by the analysis point; the co-occurrence of iron at this location is consistent with this interpretation, since ilmenite is an iron-titanium oxide commonly present as an accessory phase in basaltic rock. To the right, the fiber region covered by a CuSn tribofilm was sampled at Spectrum 1, showing a basalt-related signature together with a PIL film contribution and a bronze-derived copper signal (O 45.2%, Si 19.5%, Al 7.5%, N 6.8%, Cu 5.7%, etc). An additional point, Spectrum 4, sampled in the upper left of the field, shows a strong PIL contribution alongside a bronze-derived copper signal and a notable sodium and chlorine content (O 34.7%, N 18.7%, Cu 15.8%, Na 14.2%, Cl 7.6%, etc). Taken together, these four spectra indicate that the CuSn10 tribofilm extends across both the resin and fiber regions of this field under MSu+0.1%G lubrication. These observations are consistent with the friction and wear data reported in Section 4.1: for Sample B, MSu+0.1%G produced a mean COF of ≈ 0.029 and a specific wear rate of $\approx 0.186 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \cdot \text{m}^{-1}$, both substantially below the dry-sliding baseline for this composite ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$), supporting the combined protective role of the graphene-containing PIL film and the redistributed CuSn10 phase at the sliding interface.

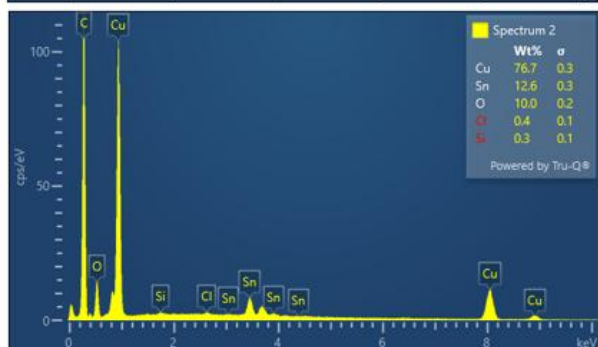
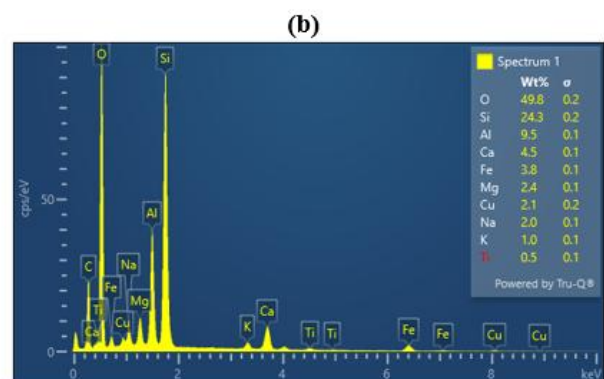
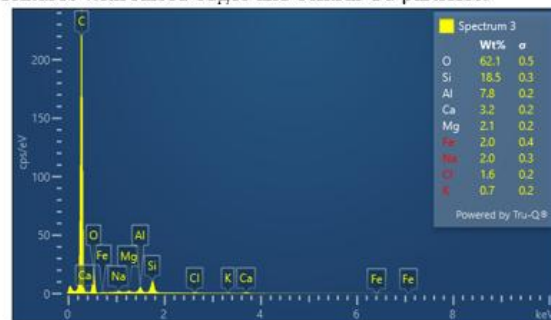
Figure 12(e) shows the BFRP_50_CuSn10_10 wear track after lubrication with MCi+0.1%G, the most protective condition tested in this study. The field of view is characterized by a crossing basalt fiber architecture, visible as a diagonal boundary running from the lower-left to the upper-right of the micrograph, consistent with the twill-weave structure of the basalt fabric reinforcement. Within one of the individual fiber sections in the upper portion of the field, Spectrum 4 shows a clean basalt-related signature (O 47.1%, Si 27.3%, Al 10.3%, Ca 4.7%, Fe 3.7%, Mg 2.5%, Na 2.1%, K 1.1%, Ti 0.7%, Cl 0.4%). At the resin matrix boundary, Spectrum 3 shows a bronze-derived copper signal together with a substantial PIL contribution (Cu 44.9%, O 17.4%, N 14.7%, Si 11.3%, Al 3.8%, etc), indicating that CuSn10 debris has been smeared into the resin at this location, consistent with the tribofilm pattern observed in this study. The whole-field Map Sum Spectrum confirms that copper is present within this field at the bulk level, consistent with the debris area contributing to the overall Cu signal alongside the resin boundary. These observations are consistent with the friction and wear data reported in Section 4.1: MCi+0.1%G produced the lowest COF (≈ 0.022) and the lowest specific wear rate ($0.0054 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \cdot \text{m}^{-1}$) recorded for Sample B among all conditions tested, over two orders of magnitude below the dry Sample A baseline. This is consistent with the combined protective mechanism proposed in Section 4.1 for this composite, the CuSn10 particles acting as a secondary load-bearing phase alongside the graphene-containing PIL boundary film, jointly restricting fiber-matrix degradation under the most favorable lubrication condition examined in this study.

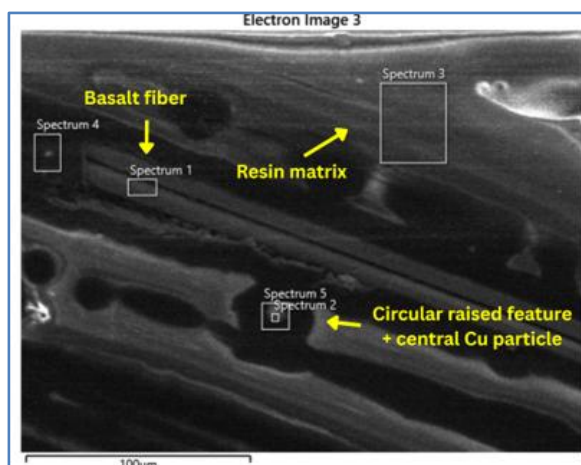


Annotated regions: Rough resin matrix, bronze fragment indented in the resin surface, basalt fibers exposed due to wear.



Annotated regions: Fiber matrix presenting a thin PIL film and fine scratch marks, resin layer boundary, and circular features with raised edges and central Cu particles.

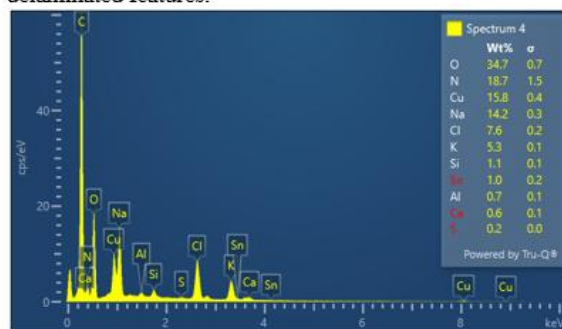




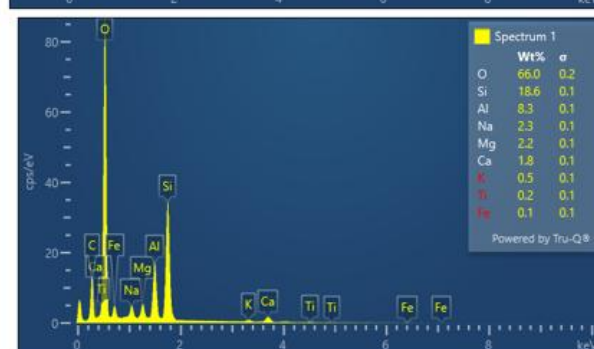
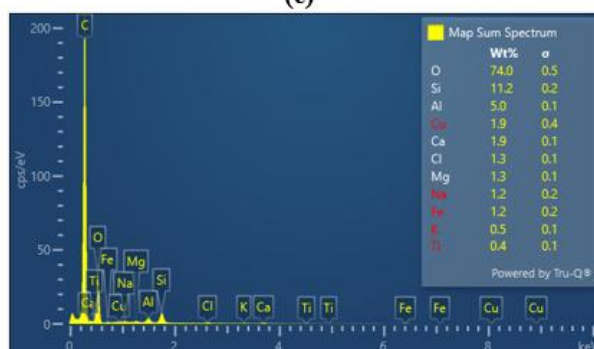
Annotated regions: Resin matrix presenting Circular features, varying in size, having raised edges and central Cu particles, basalt fibers, correlated with EDS point analyses (Spectrum 1, 2, 3, 5).



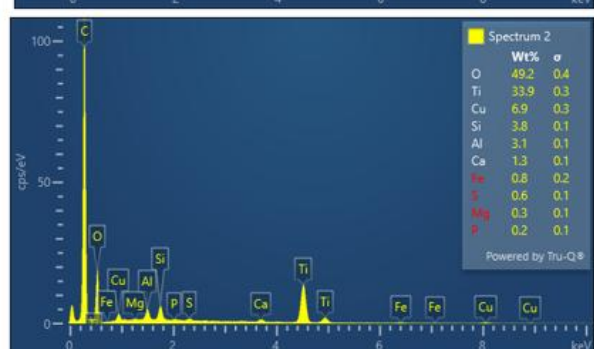
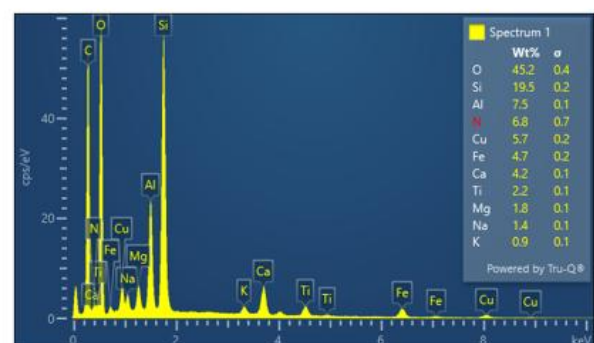
Annotated regions: Resin matrix presenting fine abrasive striations, basalt fiber matrix with fractured and delaminated features.



(c)



(d)



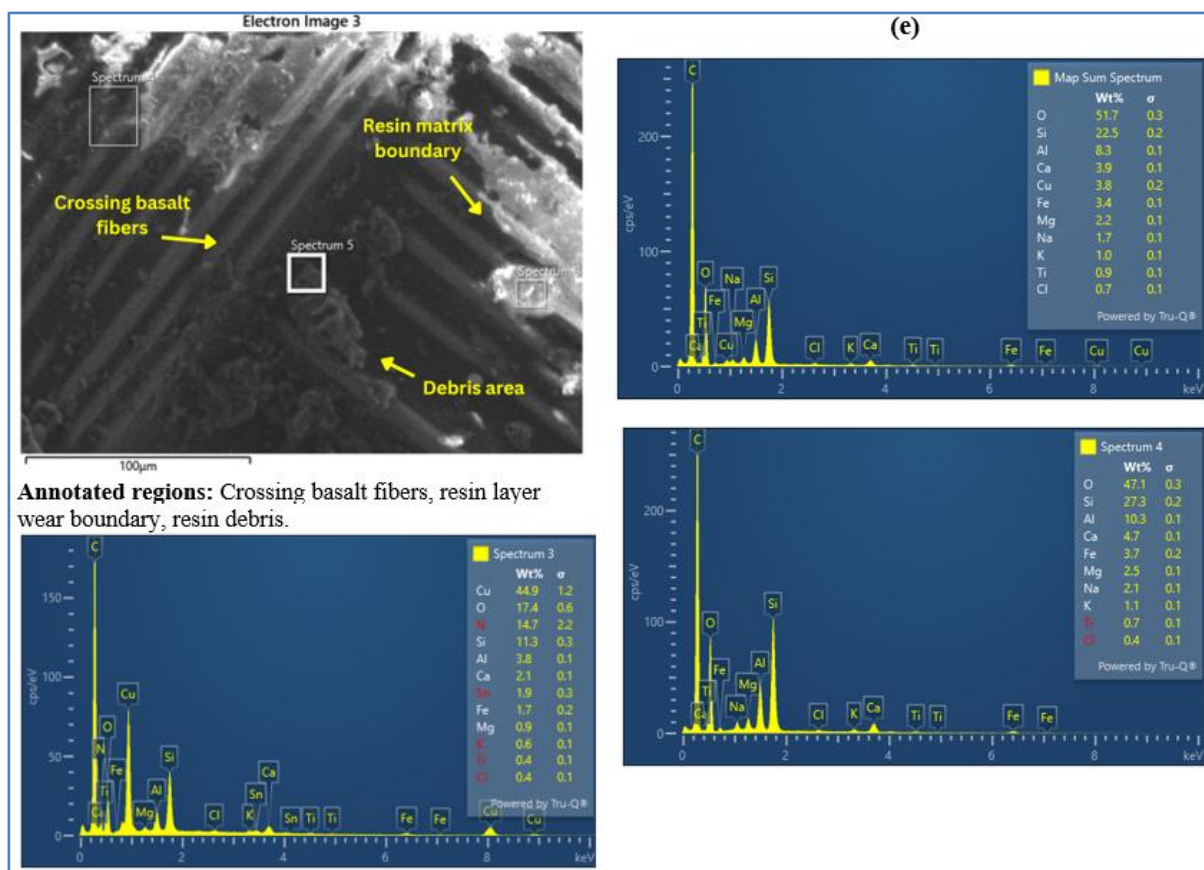


Figure 12. BSE-SEM micrographs of BFRP_50_CuSn10_10 wear tracks and EDS elemental maps of the wear track after (a) dry sliding, (b) MSu, (c) MCI, (d) MSu+0.1%G, and (e) MCI+0.1%G lubrication.

Under dry sliding, the worn surfaces of both composites showed clear evidence of fiber fracture, matrix cracking, and debonding at the fiber-matrix interface, consistent with the combined abrasive and adhesive wear mechanisms expected at this contact severity. In BFRP_60_CuSn10_0, fragmented basalt fibers were visible along the wear track, with the epoxy matrix recessed relative to the harder fiber ends. In BFRP_50_CuSn10_10, similar fiber damage was observed but the bronze particles remained largely intact and partially proud of the worn surface, suggesting that they carry a portion of the contact load and reduce the direct abrasive action on the fibers.

Under PIL nanofluid lubrication the character of the worn surfaces changed substantially. The extent of fiber fracture and matrix removal was reduced for all four lubricants relative to dry sliding, and the degree of surface protection increased progressively from the neat PILs to the graphene-containing nanofluids. For the MSu+0.1%G and MCI+0.1%G conditions, a continuous dark layer was observed on the worn surface by BSE imaging, interpreted as a graphene-rich tribofilm deposited during sliding. EDS mapping confirmed the presence of this carbon-enriched layer, as shown in Figure 11 and 12.

EDS analysis of the wear tracks on BFRP_50_CuSn10_10 after lubricated sliding additionally revealed the redistribution of copper and tin within and around the contact zone, as shown in Figure 12. The elevated Cu and Sn signals concentrated along the edges of the wear track and at the surface of fractured fiber ends suggest that CuSn10 particles were plastically smeared across the contact interface during sliding, contributing to the formation of a mixed metal-PIL tribofilm. This mechanism consists of the role of bronze particles in reducing adhesive wear reported for metal-filled polymer composites [13] and is discussed further in Chapter 5 in relation to the interaction between the PIL molecules and the exposed copper surface.

4.3 Statistical analysis of tribological results

The results of the statistical analysis, consisting in ANOVA table, main effect plots, and interaction plots for COF and specific wear rate are presented in Table 8, Figures 13-18, respectively.

The generalized linear models for COF and k were checked for model adequacy, showing a normal distribution, as is presented in Figure 13 and 14.

The lubricant condition emerged as the factor with the strongest influence on both tribological response with a contribution of 92.72% for COF and 47.85% for k (Table 8). The interaction between factors Lubricant and Material had the smallest individual effect in both cases. The complete ANOVA tables, including F-values, p-values, PC- personal contributions, and R-squared effect sizes, are presented in Table 8. Also, the influence of control factors (lubricant and material) on COF and k was statistically evaluated using graphical methods, as is shown in Figure 15-18.

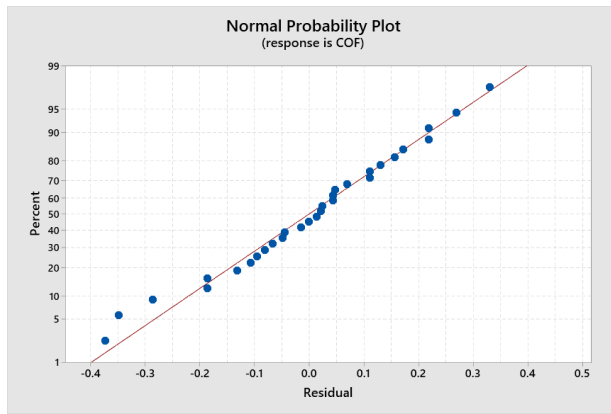


Figure 13. Normal probability plots of residuals for coefficient of friction.

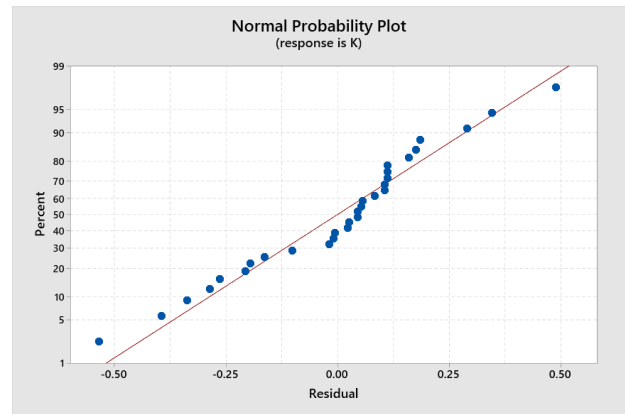


Figure 14. Normal probability plots of residuals for specific wear rate k.

Table 8. GLM-ANOVA summary: F-values, p-values, PC- personal contribution, and R-squared on mean steady-state COF and specific wear rate k

Source	COF			k		
	F-Value	p-value	PC [%]	F-Value	p-value	PC [%]
Material	10.9	0.004	1.53%	552.14	<0.001	38.13%
Lubricant	165.04	<0.001	92.72%	173.22	<0.001	47.85%
Material*Lubricant	5.23	0.005	2.94%	45.72	<0.001	12.63%
Error			2.81%			1.38%
	R_sq=97.19%			R_sq=98.62%		

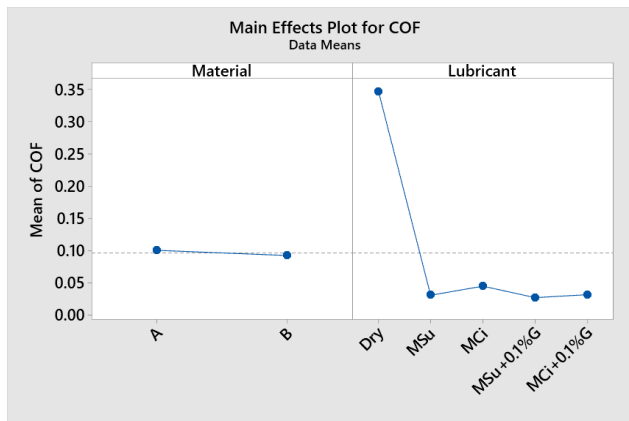


Figure 15. Main effects plots (GLM-ANOVA) for mean steady state COF showing the individual influence of each experimental factor.

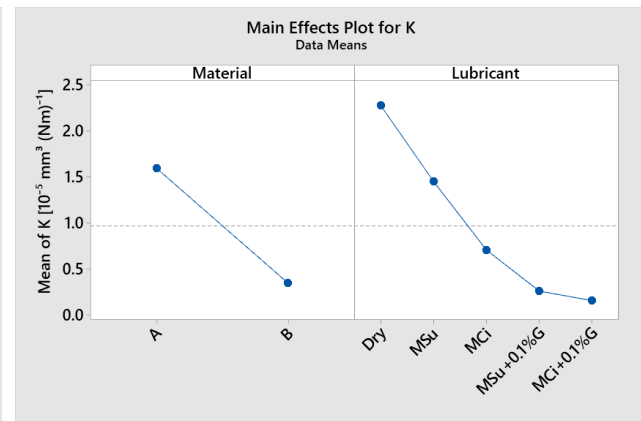


Figure 16. Main effects plots (GLM-ANOVA) for specific wear rate k showing the individual influence of each experimental factor.

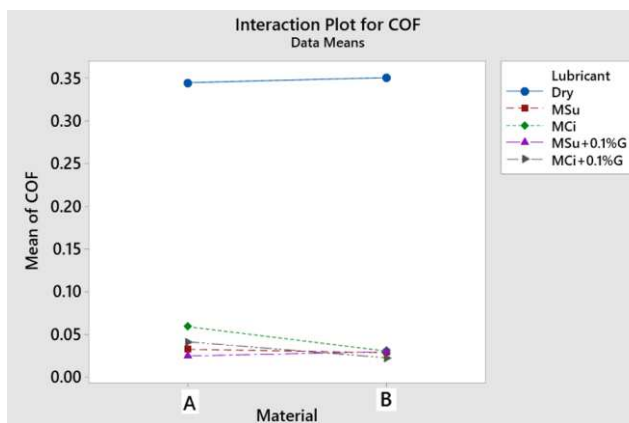


Figure 17. Interaction effects plot of lubricant factor with material type for coefficient of friction.

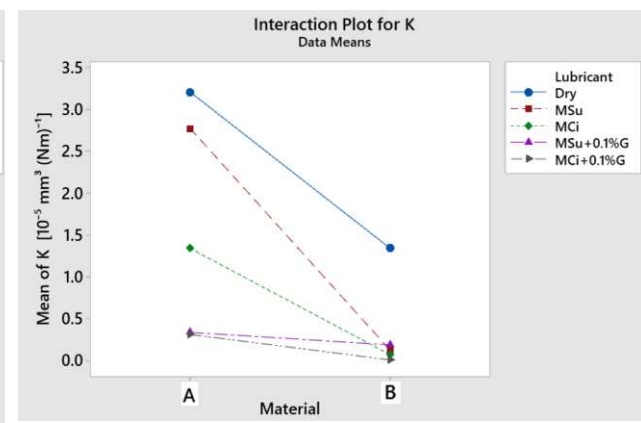


Figure 18. Interaction effects plot of lubricant factor with material type for specific wear rate.

The main effects plot indicates that lubricant type is the dominant factor governing the coefficient of friction. The material factor exhibits only a minor influence on COF, as the mean values for Materials A and B are very close. Material B shows a slightly lower average COF than Material A, suggesting that the choice of material alone has a limited effect on friction under the investigated conditions. In contrast, the lubricant factor has a pronounced effect on COF. Dry sliding produces by the highest coefficient of friction, with a mean value of approximately 0.35, highlighting the absence of effective lubrication.

The main effects plot demonstrates that both material and lubricant significantly influence the specific wear rate, with the lubricant exerting the strongest effect. The material factor has a significant influence on the wear rate. Material A exhibits a substantially higher mean specific wear rate than Material B, indicating lower wear resistance. The lubricant factor has an even more pronounced effect on the wear behavior. Under dry sliding, the highest mean wear rate is observed. The introduction of lubricants results in a progressive reduction in the wear rate. Among the lubricants, MSu + 0.1%G provides the greatest reduction in wear, followed closely by MCi + 0.1%G.

The minimum mean value of COF was obtained for MSu+0.1%G lubricant (Figure 15). MCi+0.1%G provide the minimum k (Figure 16). The effectiveness of conventional lubricants, MSu and MCi is lower than that of the graphene-reinforced formulations.

Figures 17 and 18 shows the interaction plot for the mean of lubricant and material factors, which shows no interaction for parallel lines, and shows interaction for intersecting lines. The results show a small statistical interaction between MSu, MSu+0.1%G and MCi+0.1% lubricants for COF (Figure 17). In addition, an interaction was found between MCi and MSu+0.1%G lubricants for k (Figure 18). The steep slopes observed for Dry, MSu, and MCi indicate that the material has a pronounced influence on wear under these conditions, with Material B benefiting from considerably lower wear rates than Material A. In contrast, the nearly horizontal profiles corresponding to MSu + 0.1%G and MCi + 0.1%G indicate that the addition of graphene minimizes the differences between the two materials.

5. Discussions

5.1 Effect of CuSn10 incorporation on the mechanical response. The mechanical data summarized in Table 6 and Figure 4 show that substituting 10wt% of the basalt fiber fraction with CuSn10 bronze powder produced a markedly asymmetric effect on the composite's load-bearing behavior. Tensile strength increased by approximately 34% ($329.2 \rightarrow 440.6$ MPa) and the strain at maximum load rose from 1.84% to 2.62%, while the elastic modulus decreased slightly ($20.84 \rightarrow 18.40$ GPa). This pattern is consistent with a ductile metallic phase acting as a secondary reinforcement that redistributes local stress concentrations around fiber breaks and delays catastrophic failure, at the cost of the overall stiffness normally provided by the stiffer basalt fraction being partially displaced by the filler [12,13]. The flexural response followed a different trend: strength was essentially unchanged between the two variants (284.31 vs. 280.41 MPa) while the flexural modulus increased marginally ($20.20 \rightarrow 21.74$ GPa) for the CuSn10-modified laminate. Because flexural loading is dominated by the outer plies of the laminate, this divergence between tensile and flexural behavior suggests that the bronze particles are not uniformly rate limiting across all loading modes, and that their principal mechanical contribution is to in-plane load transfer rather than to bending stiffness. The lower coefficients of variation recorded for BFRP_50_CuSn10_10 in both test modes further indicate that pre-dispersing the metallic powder in the resin before impregnation improved compositional homogeneity across the laminate thickness, corroborating the fabrication rationale described in Section 3.1 and the findings previously reported for the same filler system [13].

5.2 Friction reduction under PIL and ionanocarbon lubrication. The friction data in Table 7 and Figures 5 - 7 demonstrate that all four PIL nanofluids reduced the steady-state COF by more than an order of magnitude relative to dry sliding, from approximately 0.34 - 0.35 to below 0.06 for both composite variants. This drop is consistent with the boundary-film-forming mechanism established for 2-HEA-type PILs on both metallic and polymeric counterfaces, whereby the ionic species adsorb onto the sliding surface and separate the asperities of the composite and the steel ball, suppressing direct fiber-counterface contact [18,20]. Two distinct trends within the lubricated conditions are worth noting. First, the diacid succinate PIL (MSu) consistently outperformed the triacid citrate PIL (MCi) on Sample A (COF ≈ 0.033 vs. ≈ 0.059), a difference attributable to the higher viscosity and more strongly adsorbing carboxylate/hydroxyl architecture of the citrate anion, which increases load-carrying capacity but does not necessarily translate into a thinner, lower-shear boundary film [19,26]. Second, dispersing 0.1wt% graphene in either PIL produced a further, measurable reduction in friction and, more importantly, shortened or eliminated the initial run-in transient visible in Figure 5, in agreement with the behavior reported for analogous succinate-based PIL graphene systems on metal contacts [24]. This is consistent with the synergistic lubrication mechanism proposed for ionanocarbon fluids, in which the ionic liquid promotes graphene dispersion and adsorption while the graphene sheets themselves provide low-shear solid lubrication once exfoliated under contact pressure [21,23]. Notably, the gap between the four lubricated conditions narrowed considerably for Sample B (0.022 - 0.030) compared with Sample A (0.024 - 0.059), indicating that once a CuSn10-containing surface is present, the specific chemistry of the PIL becomes a secondary factor relative to dry sliding, while remaining a statistically significant one (Section 5.5).

5.3 Wear resistance and the dual protective role of CuSn10. The specific wear rate results (Table 7, Figures 9 - 10) reveal a more complex picture than the friction data alone. For Sample A, PIL lubrication reduced wear from 3.208×10^{-5} mm³·N⁻¹·m⁻¹ under dry sliding to 2.775×10^{-5} (MSu) and 1.341×10^{-5} (MCi), with the graphene-containing nanofluids achieving a further order-of-magnitude reduction (0.336×10^{-5} for MSu+0.1%G and 0.317×10^{-5} for MCi+0.1%G). This confirms that graphene acts through a mechanism distinct from simple friction reduction: it forms a lubricious tribofilm

that physically limits direct fiber–counterface contact and suppresses abrasive fiber pull-out, an interpretation directly supported by the SEM-EDS evidence discussed in Section 5.4.

The behavior of the CuSn10-modified composite (Sample B) departs from this pattern in an important way. Even under dry sliding, its wear rate ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) was already comparable to the best neat-PIL condition recorded for Sample A, despite both samples exhibiting statistically indistinguishable dry-sliding COF values ($\approx 0.34 - 0.35$). This dissociation between friction and wear a condition with a high coefficient of friction but a comparatively low material removal rate is the clearest evidence in this study that the bronze particles act primarily as a load-bearing phase rather than as a friction modifier: partially proud, largely intact CuSn10 particles carry a portion of the normal load at the sliding interface and shield the more brittle basalt fibers from direct abrasive contact, even before any external lubricant is introduced. Under PIL lubrication this protective role compounds with the boundary-film and tribofilm mechanisms already active for Sample A, driving the wear rate of Sample B down to values below $0.19 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ for all four lubricants and to a minimum of $0.0054 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ for MCI+0.1%G over two orders of magnitude below the dry Sample A baseline. This combination of a hard, load-bearing metallic filler with a graphene-containing ionic nanofluid therefore appears to represent a genuinely synergistic protective system rather than the simple superposition of two independent effects, consistent with previously reported findings for metal-filled BFRP composites lubricated under boundary conditions [12,13] and for graphene PIL systems on bare metallic counterfaces [21,24,38,39].

5.4 Microstructural evidence of tribofilm formation and wear mechanisms. The SEM-EDS analysis (Figures 11 - 12) provides direct microstructural support for the friction and wear trends discussed above. Under dry sliding, both composites showed clear evidence of basalt fiber fracture, matrix recession, and debris redistribution, consistent with a combined abrasive–adhesive wear mechanism operating at the contact severity used in this study. Under PIL lubrication, the worn surfaces retained a substantially more continuous, longitudinally intact fiber morphology, and EDS spectra consistent with an adsorbed 2-HEA boundary film (elevated oxygen/nitrogen content with markedly reduced exposed-fiber signatures) were recorded on both fiber ridges and resin-rich regions. The degree of surface protection increased progressively from the neat PILs to the graphene-containing nanofluids: for MSu+0.1%G and MCI+0.1%G, a continuous dark layer was observed by backscattered-electron imaging and confirmed by EDS mapping as a carbon-enriched tribofilm, directly corroborating the order-of-magnitude drop-in wear rate recorded for these two conditions.

For the CuSn10-modified composite, EDS point analyses additionally revealed the redistribution of copper and tin across the contact zone under both dry and lubricated sliding intact bronze fragments with a Cu:Sn ratio close to the nominal CuSn10 composition were repeatedly identified alongside adjacent zones showing smeared copper and tin signals of comparable ratio, indicating plastic smearing of the metallic filler into the surrounding resin and fiber-debris regions during sliding. This is consistent with the formation of a mixed metal PIL tribofilm, in which the redistributed bronze phase complements the ionic and graphene-based boundary lubrication mechanisms already established for the unfilled composite. The recurrence, across two independent lubricated conditions (Figures 12b, 12c), of a distinct raised-rim morphology surrounding embedded copper-rich particles further suggests a reproducible, rather than incidental, local surface response to the presence of load-bearing metallic inclusions during sliding contact.

5.5 Statistical significance of material and lubricant effects. The GLM-ANOVA results (Table 8, Figures 13 - 14) place the qualitative trends discussed above on a quantitative footing and reveal an instructive asymmetry between the two response variables. For the coefficient of friction, the lubricant condition was overwhelmingly dominant, accounting for 92.72% of the explained variance, while the composite material contributed only 1.53%; this is consistent with friction being governed primarily by the chemistry of the interfacial film rather than by the underlying composite composition, in line with the boundary-lubrication mechanism discussed in Section 5.2. For the specific wear rate, by contrast, the material factor accounted for 38.13% of the variance nearly as much as the lubricant condition itself (47.85%) confirming statistically that the CuSn10 filler exerts an influence on wear that is largely independent of, and comparable in magnitude to, the effect of the external lubricant. The material $\times$ lubricant interaction was also more pronounced for wear (12.63%) than for friction (2.94%), indicating that the protective contribution of the bronze filler is not simply additive but depends on which lubricant is present, consistent with the synergistic mixed metal PIL graphene tribofilm mechanism proposed in Section 5.3–5.4. Both models achieved high explanatory power ($R^2 = 97.19\%$ for COF, 98.62% for K), supporting the reliability of these conclusions within the tested factorial design.

5.6 Comparison with previous studies and practical relevance. The friction reductions obtained here for the neat PILs (up to $\sim 90\%$ relative to dry sliding) and for the graphene-containing nanofluids are broadly consistent with, and in several cases exceed, values reported for 2-HEA PILs and PIL–graphene systems on metallic contacts [18,19,21,24,26], confirming that the lubrication mechanisms established for hard, non-porous counterfaces transfer effectively to a fiber-reinforced polymer composite substrate. The wear reductions observed for the CuSn10-modified composite are similarly in line with, and extend, previous reports on copper- and bronze-filled BFRP laminates under dry and boundary-lubricated sliding [12,13], while the magnitude of the combined CuSn10–graphene–PIL effect (over two orders of magnitude below the unmodified, dry baseline) has not, to the authors' knowledge, been previously documented for this specific material combination. From an applications perspective, these results suggest that BFRP components intended for sustained tribological service, for example, bushings, guide elements, or lightly loaded bearing surfaces can achieve substantial friction and wear improvements either through metallic particle modification of the matrix alone, which is advantageous where external lubricant reapplication is impractical, or through the combined use of a bronze-modified matrix with periodic PIL graphene lubrication, which is preferable where the lowest achievable wear rate is the priority and lubricant maintenance is feasible.

The combination of results obtained here points to two distinct application strategies depending on service constraints. Where periodic lubricant maintenance is impractical or undesirable such as in enclosed or hard-to-access mechanical assemblies, dry-running bushings, or lightly loaded guide rails the CuSn10-modified composite alone offers a substantial wear-resistance benefit even without external lubrication, since its dry-sliding wear rate ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) already matches the best neat-PIL-lubricated performance of the unfilled composite. Where periodic lubrication is feasible, such as in accessible sliding or rotating machine elements (e.g., pin joints, low-speed bearing liners, conveyor guides), the combined use of the CuSn10-modified composite with graphene-dispersed PIL lubrication (MCi+0.1%G) offers the lowest achievable friction and wear observed in this study, making it particularly suited to structural components subject to sustained boundary-lubricated sliding, such as robotic joint bushings, precision guide elements in packaging or textile machinery, or lightweight bearing surfaces in transport applications where weight reduction relative to metallic alternatives is also a design priority. The halogen-free, biodegradable nature of the PIL formulations further supports their use in applications where lubricant leakage or disposal is an environmental concern, such as food-processing or outdoor equipment.

5.7 Limitations and directions for further work. Several aspects of the present study warrant caution when generalizing the findings. The tribological tests were conducted at a single normal load and sliding speed (10 N , $0.25 \text{ m} \cdot \text{s}^{-1}$), selected on the basis of the previously identified optimal dry-sliding condition for CuSn10-filled BFRP composites [13]; while this choice enables direct comparison with prior baseline data, the relative ranking of lubricants and the magnitude of the CuSn10 contribution may shift under more severe pressure-velocity conditions, particularly given that basalt/glass composite comparisons in the literature have shown condition-dependent wear behavior [11]. Future work should therefore extend the present factorial design across a broader range of loads and speeds to establish whether the lubrication and load-bearing mechanisms identified here remain dominant outside the optimal condition used in this study. The contact temperature did not vary significantly from ambient across the tested conditions (Section 4.1.3), and it remains to be established whether the protective mechanisms identified here persist at elevated bulk or flash temperatures. Finally, the origin of the tin-depleted copper signal recorded in some EDS spectra of the CuSn10 modified composite (Section 4.3) could not be unambiguously attributed to the intact filler versus a secondary redistribution process and would benefit from targeted cross-sectional or depth-resolved analysis in future work.

It should be noted that the lowest wear rates reported (e.g., Sample B under MCi+0.1%G lubrication, $0.0054 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) approach the resolution limit of the profilometric system used. This is consistent with the elevated coefficient of variation ($\sim 34\%$) observed at these low wear rates, compared to the low single-digit CVs at higher wear rates, and suggests these values should be interpreted as indicative of very low wear rather than precise absolute measurements.

6. Conclusions

This study carried out a systematic investigation of the mechanical and tribological behavior of basalt fiber-reinforced epoxy composites, with and without a 10wt% CuSn10 bronze filler, under dry sliding and four external protic ionic liquid (PIL) nanolubricants, two neat and two graphene-dispersed. Friction and wear were assessed on a pin-on-disc tribometer and interpreted alongside mechanical testing, SEM-EDS surface analysis, and GLM-ANOVA statistical modeling. The following conclusions can be drawn.

1. All four PIL nanofluids reduced the steady-state coefficient of friction by more than an order of magnitude relative to dry sliding, from approximately 0.34–0.35 to below 0.06 for both composite variants, confirming that 2-HEA-type protic ionic liquids form an effective boundary film on basalt fiber-reinforced epoxy surfaces. The diacid succinate PIL (MSu) outperformed the triacid citrate PIL (MCi) on the unfilled composite, and dispersing 0.1 wt% graphene in either fluid produced a further friction reduction together with a shortened or eliminated run-in transient.
2. Expressed relative to dry sliding, PIL lubrication reduced the steady-state COF by 83–93% for the unfilled composite and by 92–94% for the CuSn10-modified composite, depending on the lubricant. The specific wear rate was reduced by 14–90% for the unfilled composite and by 86–99.6% for the CuSn10-modified composite. Across both composites and both response variables, MCi+0.1%G consistently produced the largest improvement, combining the lowest COF for the CuSn10-modified composite (93.7% reduction) with the lowest wear rate overall (99.6% reduction), identifying it as the most effective lubricant tested in this study.
3. Specific wear rate followed the same general trend but with a distinct role for the metallic filler. For the unfilled composite, wear dropped from 3.208×10^{-5} to $1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ with the neat PILs and to approximately $0.32 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ with the graphene-containing nanofluids. For the CuSn10-modified composite, wear was already low under dry sliding ($1.341 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$) and fell to a minimum of $0.0054 \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$ under MCi+0.1%G lubrication, over two orders of magnitude below the unmodified, dry baseline.
4. The dissociation between friction and wear observed for the CuSn10-modified composite under dry sliding, a coefficient of friction comparable to the unfilled composite but a markedly lower wear rate indicates that the bronze particles act principally as a load-bearing phase that shields the basalt fibers from direct abrasive contact, rather than as a friction modifier in their own right.
5. SEM-EDS analysis of the worn surfaces corroborated these tribological trends: dry sliding produced clear basalt fiber fracture and matrix recession, PIL lubrication preserved a substantially more continuous fiber morphology consistent with an adsorbed boundary film, and the graphene-containing nanofluids generated a continuous, carbon-

enriched tribofilm. In the CuSn10-modified composite, EDS additionally revealed plastic smearing and redistribution of copper and tin across the contact zone, consistent with the formation of a mixed metal–PIL tribofilm that reinforces the ionic and graphene-based lubrication mechanisms.

6. GLM-ANOVA confirmed these mechanistic interpretations statistically. The lubricant condition dominated the coefficient of friction (92.72% contribution), while the composite material and the lubricant contributed comparably to the specific wear rate (38.13% and 47.85%, respectively), with a non-negligible material $\times$ lubricant interaction (12.63%) reflecting the synergy between the bronze filler and the PIL–graphene nanofluids. Both models achieved high explanatory power ($R^2 = 97.19\%$ for COF and 98.62% for wear rate).

Taken together, these results show that friction and wear in basalt fiber-reinforced epoxy composites can be controlled through two complementary, largely independent routes: external boundary lubrication with 2-HEA PIL nanofluids, which governs friction almost irrespective of the underlying composite composition, and internal modification of the matrix with a load-bearing metallic filler, which governs wear resistance to a degree comparable with that of the lubricant itself. Their combination, evidenced here by the CuSn10 composite lubricated with graphene-dispersed PILs, yields a wear rate roughly two orders of magnitude below the unlubricated, unfilled baseline, suggesting that this material–lubricant pairing warrants further evaluation as a design strategy for lightly loaded basalt-fiber composite components operating under sustained sliding contact, such as bushings and guide elements. Future work should extend the present factorial design to a wider range of normal loads and sliding speeds and should examine the durability of the identified tribofilms under prolonged or thermally elevated service conditions.

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Data Availability Statement

The datasets generated and/or analyzed during the current study are not publicly available as they form part of the ongoing doctoral thesis of Eng. Horea Goia and cannot be disseminated until the thesis is completed and defended. However, the data are available from the corresponding authors (including Horea Goia) on reasonable request. Following completion of the doctoral thesis, the data are expected to be deposited in a public repository as part of the broader research project.

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Additional information: Correspondence and requests for materials should be addressed to H.G. and F.P.r.

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