

Surface Chemistry and Tribological Performance of Al–SiC–TiO₂ Composites under Cryogenic Conditions

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The increasing demand for materials with enhanced strength, wear resistance, and thermal stability drives the development of hybrid metal matrix composites. This study investigates the tribological behavior of Al6061 reinforced with SiC and TiO₂ particles, fabricated using the stir casting technique, under both cryogenic and non-cryogenic conditions. A Taguchi L9 orthogonal array was employed to optimize key parameters, including reinforcement content, applied load, and sliding distance. Results revealed that increasing the weight percentage of SiC and TiO₂ significantly reduced wear loss, with cryogenic-treated composites exhibiting superior wear resistance compared to untreated ones. The optimum wear resistance was achieved at 7.5% SiC, 7.5% TiO₂, 5 N load, and 600 m sliding distance. This study addresses the research gap concerning the combined effect of SiC–TiO₂ reinforcements under cryogenic conditions and highlights the role of cryogenic treatment in enhancing composite performance. Future work will explore other reinforcement combinations and the long-term durability of these composites under dynamic loading conditions.

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The search for new materials has been prompted by the growing need in advanced material sciences for materials with increased strength, toughness, thermal resistance, and wear resistance [1-3]. Metal Matrix Composites (MMCs) are an emerging sophisticated material developed largely due to the question of excellence in material performance [4]. Regarding traditional alloys, metal matrix composites typically offer better wear resistance and superior strength at higher temperatures than their monolithic alloy equivalents [5]. MMCs are appropriate for the nuclear, aerospace, defense, and automotive industries despite their high manufacturing costs due to their exceptional wear characteristics and strong mechanical qualities at high temperatures [6]. The wear characteristics mainly depend on the reinforcement percentage, size, applied load sliding velocity, and sliding distance. By optimizing these parameters, the researchers can reduce the wear loss [7]. Discontinuous reinforcement is gaining popularity even though continuous and discontinuous fiber reinforcements have already been well-studied. Such dispersed reinforced metal matrix composites can improve their mechanical and tribological properties by further processing [8]. Pitchayapillai et al. used stir casting to create an aluminum (6061) matrix composite with nanosilver (Ag). Compared to cast Al6061 alloy, they reported that Al6061 with 2% Ag composite had a maximum compressive and tensile strength [9]. Using the stir casting method, Jawhar et al. investigated the impact of various weight percentages of SiC in aluminum (6061) alloy composites for use in automotive and

aerospace applications. They found that an increase in SiC content improves the mechanical characteristics of the composite [10]. Superior mechanical and wear resistance was discovered by Sharma et al. upon adding 2.5 weight percent cerium oxide to an aluminum (6061) hybrid composite that also contained 15 weight percent SiC and Al₂O₃. There have been reports on the wear behavior of Al6061-SiC composites and an increase in wear resistance when the base alloy's SiC content increases. Increased wear resistance was found in a 15 vol. % SiC sample produced using powder metallurgical procedures [11]. It was shown that when TiC particles were used as reinforcements, Al₃Ti needles developed during the crystallization of MMCs, and the TiC particulates acted as nucleates, improving the matrix's strength and elastic modulus greatly. The impact of adding TiC ceramic particles on the in situ composites' high temperature sliding wear resistance, which is made from a molten metal mixture of K₂TiF₆ and graphite powder. They saw that the wear rate rose as the applied load grew and fell as the weight percentage of TiC increased [12]. By forming an oxidation transfer layer, monolithic and composite materials demonstrated resilience to thermal softening. A few researchers have also established the reaction between Al and TiC at an equilibrium temperature of 100 °C. These results suggest that the aluminum matrix has increased TiC stability [13]. Multiple research studies show TiC seems to have respectable wettability and regulated reactivity. Magnesium did not participate in reaction-aided wetting since it has minimal tendency to form carbides in Al6061.

However, in addition to CuAl₂, Mg₂Si synthesis was also found. Stronger and harder MMCs were seen when the TiC weight content rose. A linear relationship was found between the specific wear rate of the TiC-reinforced composites and the weight fraction of the reinforcements. The average coefficient of friction decreases linearly as the normal load and TiC volume fraction increase [14]. Moreover, many researchers are focusing on cryogenic-treated composite materials. The structure of cryogenically cooled MMCs is finer than that of the unchilled matrix alloy, as confirmed by microstructural investigations. When the composite melt is cryogenically solidified, the interaction occurs between the chill's below-freezing temperature, and the melt undergoes extreme super cooling and grows incredibly personal at the same time [15]. Despite the growing use of chills in Al alloy foundation, research on the impact of chills on the mechanical characteristics of Al–B₄C composites is currently lacking, despite some studies on the impact of chills on the solidification and soundness of wide-freezing-range alloy castings [16]. Moreover, research has demonstrated that chilling affects Al alloy castings' composition, characteristics, and soundness. The researchers found that deep cryogenic treatments enhanced stiffness and tribological properties. Using Taguchi's optimization technique, the researchers computed the parametric adaption of DCT to investigate the wear and tribological output of the implant solid material UNS R56700. Research shows that soaking time, sliding speed, and contact pressure may impact wear rate. The optimization of the wear response of aluminum MMCs (356/B₄Cp) was investigated using Taguchi's S/N approach [18]. Despite extensive studies on the tribological behavior of Al6061-based composites reinforced with SiC and TiC, a notable lack of research focuses on the combined effect of SiC–TiO₂ hybrid reinforcements fabricated through cryogenic treatment under dry sliding conditions. Prior works mainly investigated individual reinforcements or focused on conventional casting methods without cryogenic solidification. However, the synergistic effect of SiC and TiO₂ particles and cryogenic processing on wear behavior and surface chemistry remains underexplored. Addressing this gap is crucial for developing advanced metal matrix composites with superior mechanical and tribological performance for critical aerospace, automotive, and defense applications.

Using a systematic Taguchi optimization approach, the present study uniquely investigates the tribochemical behavior of Al6061 composites reinforced with varying percentages of SiC and TiO₂ under cryogenically treated conditions. By correlating microstructural changes to wear resistance improvements, this work offers novel insights into designing high-performance composites for extreme service environments.

EXPERIMENTAL

Aluminium alloy Al6061 is utilized as a matrix material, and silicon carbide (SiC) and Titanium dioxide (TiO₂) materials are used as reinforcement materials. The compositions of Al6061 aluminium alloy are tabulated (Weight percent) in Table 1. Silicon carbide (SiC) and titanium dioxide (TiO₂) are utilized as the molecule fortification material for diverse volume portions. The normal molecule size of silicon carbide is 25µm, and Titanium dioxide is 50 µm. Reinforcement SiC and TiO₂ particulates are calculated for an equal volume of reinforcements and an equal volume of reinforcements (2.5 %, 5 %, 7.5 %). The stir-casting procedure involves preparing a mild steel mold measuring 300 mm in length and 50 mm in diameter to collect and solidify the composite material cast in the induction furnace. The stir casting method entails mixing SiC and TiO₂ reinforcement particles into a liquid Al6061 aluminum melt and solidifying the mixture. At the same time, the vortex technique introduces preheated reinforcement into the molten alloy via a vortex created by rotating the impeller [19]. In the vortex method, the base material Al6061 is melted and vigorously mixed by a high-speed stirrer to create a vortex at the surface of the melt, and the reinforcement materials SiC and TiO₂ are then added at the side of the vortex. Deep cryogenic treatment (DCT) is applied to the composition specimens prepared for wear testing. The stir-cast Al6061 hybrid metal composite is processed in a cryogenic environment. Samples are cryogenically treated by putting an Al6061/SiC-TiO₂ specimen within a cryogenic chamber. In a nitrogen reservoir, the cryogenic treatment is carried out [20]. A stepper motor was utilized to lower the sample and maintain a temperature fall of 1°C/min while a K-type thermistor monitored the temperature of the material. The mercury has dropped to -196°C. It takes almost four hours to get there.

Table 1. Chemical Composition of Al6061 alloy.

Elements	Cu	Si	Mg	Mn	Fe	Ti	Ni	Zn	Sn	Al
Percentage	0.17	0.8	1.2	0.15	0.15	0.13	0.006	0.06	<0.001	Balance

Design of Experiments

Taguchi's technique is a resilient design method frequently used in engineering applications and typically aims to offer high-quality products to the producer at an economical cost. The current study used a Taguchi L9 orthogonal array for experimental trials and a pin-on-disc apparatus for dry sliding wear tests. Using a CNC turning machine, the wear test specimens with dimensions of 30 mm in height and 10 mm in diameter were created by ASTM standards G99-95 [21]. The test specimen was loaded and forced up against a revolving 65 HRC AISI D2 steel disc. To experiment, the initial and final weights were measured to an accuracy of 0.001g.

RESULTS AND DISCUSSION

The Figure shows the S N ratio graph for non-cryogenic treated composites. From the graph, we can find the optimum combination for minimum wear loss. The best combination is at 7.5 % SiC and 7.5 % TiO₂, 5 N of Load, and 600m of sliding distance. The main scenario behind these values is the hardness

of the composites. The statement suggests that an increase in the weight percentage of silicon carbide (SiC) and titanium carbide (TiO₂) is correlated with a reduction in wear loss. This implies that incorporating a higher concentration of SiC and TiO₂ into a material or coating can enhance its resistance to wear [22]. Both SiC and TiO₂ are known for their hardness and wear-resistant properties. When these materials are added to a composite or coating, they can improve the material's overall hardness and wear resistance, making it more durable and less prone to wear and tear.

At 7.5 % of SiC and 7.5 % of TiO₂, 5 N of Load and 600m of sliding distance is the best combination for the composites at cryogenic condition shown in Figure 2. This observation aligns with a well-known phenomenon in materials science and engineering where certain materials exhibit improved mechanical properties, including wear resistance, at lower temperatures [23]. At such low temperatures, the thermal expansion of materials is minimized, and their hardness and toughness may be enhanced. Several factors contribute to the reduced wear loss in cryogenic conditions

Table 2. Design of Experiments.

	Cryogenic Treated			Non Cryogenic Treated		
SiC	2.5	5	7.5	2.5	5	7.5
TiO ₂	2.5	5	7.5	2.5	5	7.5
Load	5	10	15	5	10	15
Sliding Distance	200	400	600	200	400	600

Table 3. L9 Orthogonal Array with results.

S.No	SiC	TiO ₂	Load	Sliding Distance	Non Cryogenic Treated	Cryogenic Treated
1	2.5	2.5	5	200	0.00748	0.00713
2	2.5	5	10	400	0.01015	0.0088
3	2.5	7.5	15	600	0.00535	0.0051
4	5	2.5	10	600	0.00579	0.0049
5	5	5	15	200	0.00915	0.0078
6	5	7.5	5	400	0.00671	0.0053
7	7.5	2.5	15	400	0.00655	0.0052
8	7.5	5	5	600	0.00532	0.00397
9	7.5	7.5	10	200	0.00705	0.0057

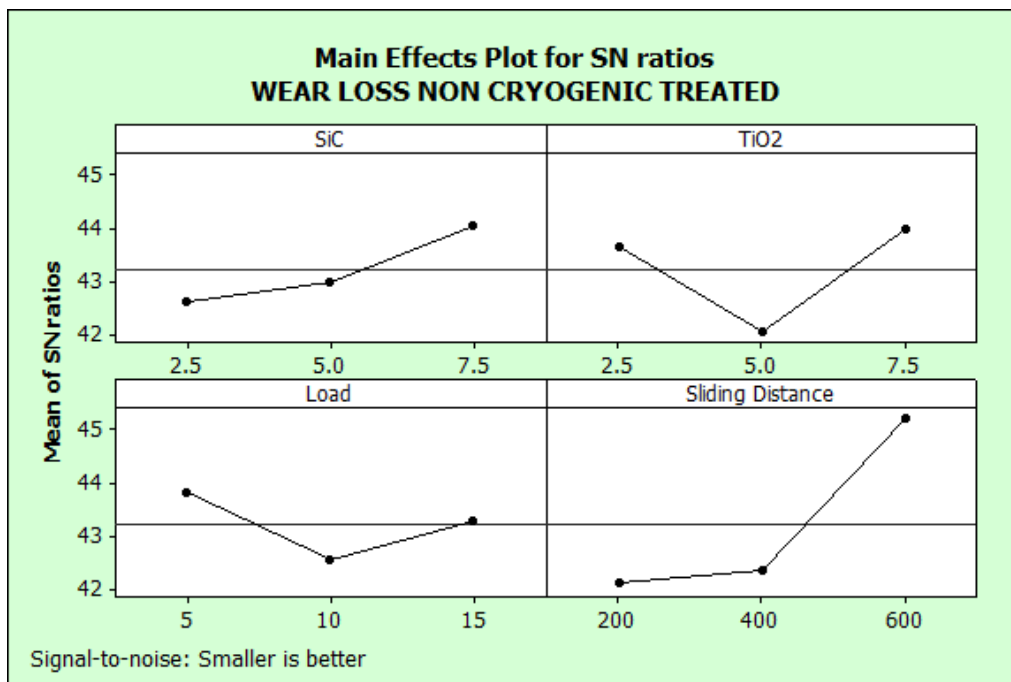


Figure 1. S/N ratio for wear loss under non cryogenic treated condition.

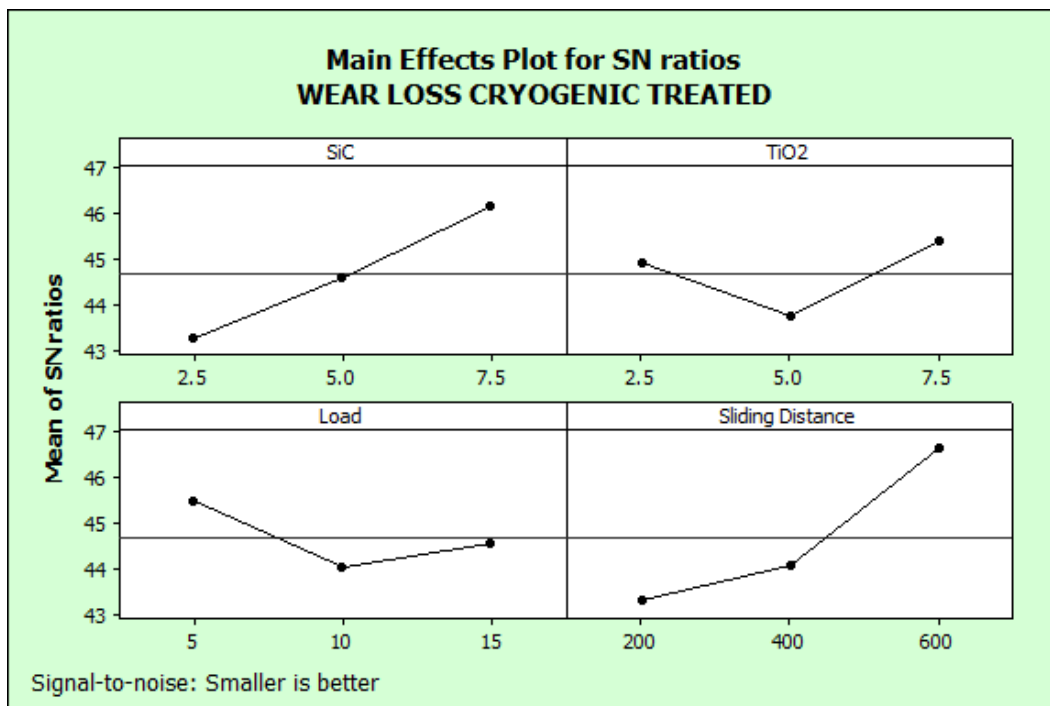


Figure 2. S/N ratio for wear loss under cryogenic treated condition.

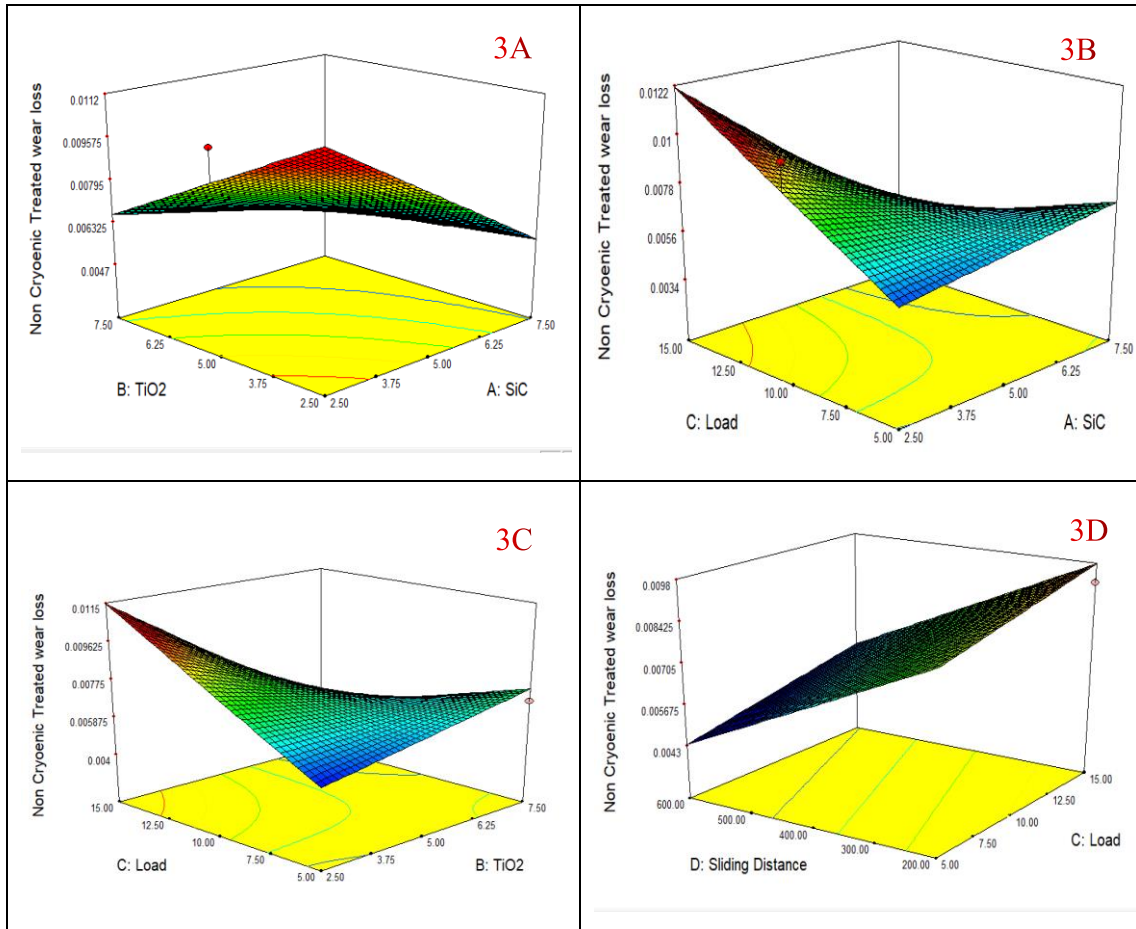


Figure 3. [A -D] Shows the contour plot for various input parameters VS wear loss in non-cryogenic treated conditions.

Figure 3A shows the relationship between the weight percentage of SiC and TiO₂ over wear loss in non-chilled conditions. In non-cryogenic conditions, the increase in the weight percentage of the reinforcement has minimum wear loss. The mechanism behind this is often related to the ability of these hard particles to act as reinforcements within the material, forming a protective barrier against abrasive forces. The contour plot of the Load and SiC percentage is shown in Figure 3b. The maximum wear loss, i.e., 0.0112g, is obtained at a maximum load of 15N. Increased load increases wear loss" reflects a common observation in tribology, the study of friction, wear, and

lubrication. In mechanical systems, the load refers to the force applied to the surfaces in contact. As the load on a system increases, the contact pressure between interacting surfaces also rises [24]. Higher contact pressure can increase friction and wear between the materials, which causes higher wear loss. Figures 3C and 3D show the wear loss of the composite at maximum sliding distance. A maximum load of 15 N and 600m of sliding distance offer minimum wear loss. This is because when you continuously increase the sliding velocity, the kinematic coefficient of friction decreases. This is the cause of decreasing wear loss at higher speeds [25].

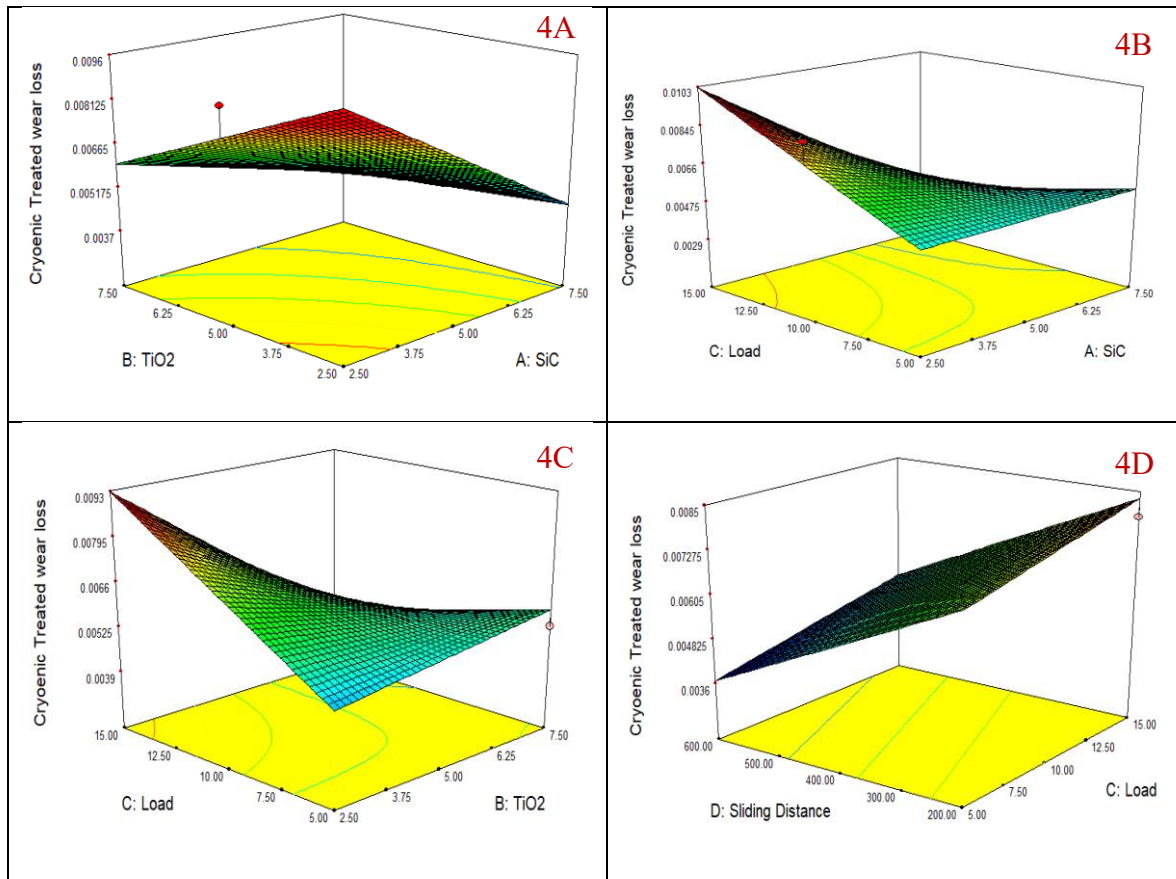


Figure 4. [A -D] Shows the contour plot for various input parameters VS wear loss in cryogenic treated condition.

Figure 4A shows the relationship between the weight percentage of SiC and TiO₂ for wear loss in cryogenically treated conditions. The hardness and wear resistance of TiO₂ and SiC may contribute to reduced friction and heat generation during sliding or abrasive conditions, further decreasing wear loss [26]. The cryogenic effect during solidification strengthens the connection between the matrix and dispersion. This can be attributed to the improved wettability of the particles and matrix, a direct result of the cryogenic effects [27]. When comparing chilled Metal Matrix Composites (MMCs) to their un-chilled alloy counterparts, the result is a more uniform distribution of particles and a finer matrix structure, leading to elevated mechanical properties. Figures 4B and 4C show the significant relation between the reinforcement and load. Cryogenic temperatures can reduce the ductility of materials. Reduced ductility may make materials more susceptible to cracking and fracture, contributing to increased wear loss under heavier loads [28].

WORNOUT SURFACE ANALYSIS

Figure 5a shows the after-wear test samples of 7.5 % SiC 7.5 % TiO₂, 15N Load, and 600 m Sliding

Distance, without cryo treatment. It is observed that when the volume content of the reinforcement increases, wear loss generally decreases. When the amount of TiO₂ and SiC increases, wear loss also lowers compared to unreinforced aluminum alloy. This illustrates how the lubricating properties of TiO₂ and the creation of a mechanically mixed layer of reinforcing particles occur when sliding temperatures rise.

Figure 5 b shows the after-wear test samples of 7.5 % SiC, 7.5 % TiO₂, 5N Load, and 200m Sliding Distance, with cryo-treated condition. The treated composite appears to begin with localized surface melting and then progress through delaminations caused by significant matrix plastic deformation [29]. When compared to Figure 5c, Figure 5d shows less wear loss. At the maximum load, adhesion, and slide phenomena become visible as the sliding speed rises. Here, materials that have been removed can be used to measure adhesive wear, and material wave patterns can also reveal slip occurrences [30]. The material appears to be removed more quickly due to the fracture of the matrix and short TiO₂ particles, which may be caused by the high friction force on the wear surface, increasing the wear loss.

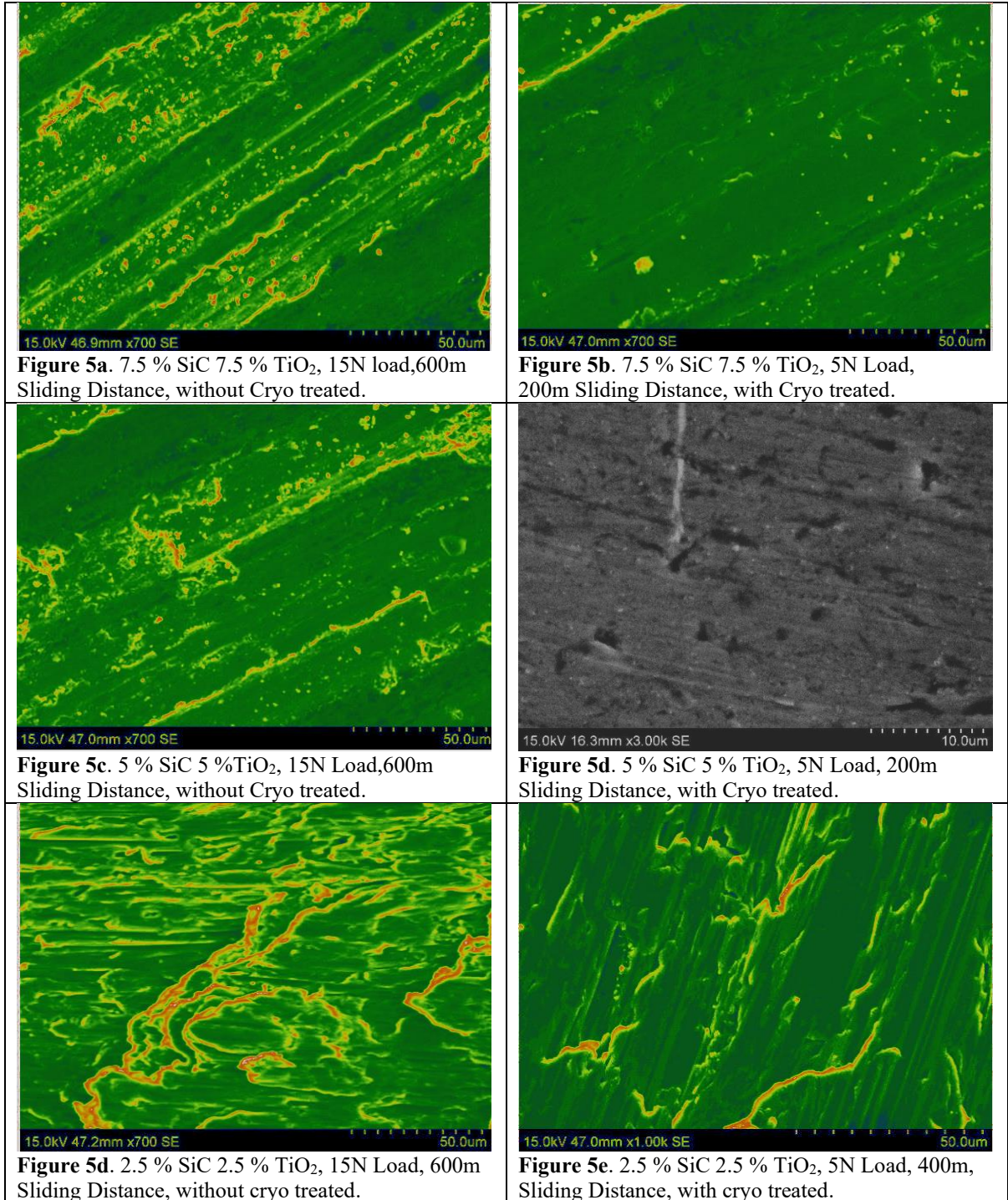


Figure 5c. Plow and groove were shown to be caused mainly by abrasive wear at high sliding distances of 600 m. That is, because of the abrasive action between the wear specimen and counter material, possibly due to the high friction force applied to the wear surface, the forms of the stripes on the wear surface appear to increase the wear loss gradually. Compared to Figure 5e, larger and deeper grooves are shown on the surface of the composite material. Compared to unprocessed cryogenic samples of hybrid composite, the results of this reduced

volume % of hybrid composite subjected to cryogenic processing also have improved wear resistance properties. As a result, when creating a hybrid composite, the reinforcement particle's quantity component is also determined by its properties.

CONCLUSION

The Present paper reveals the tribological aspects of Aluminium 6061 reinforced with SiC and TiO₂ at different weight percentages fabricated through

the stir casting technique. The composites are subjected to cryogenic and non-cryogenic treatment to find the best solution for minimum wear loss. Taguchi L9 orthogonal array was chosen to experiment, and an S/N ratio analysis was conducted to find the input parameters. The optimum combination for minimum wear loss is at 7.5 % of SiC and 7.5 % of TiO₂, 5N of Load, and 600m of sliding distance is the best combination for both cryogenic and non-cryogenic treated conditions. The load is the maximum influencing parameter for increasing wear loss in both cryogenic and non-cryogenic treated condition. The cryogenic processing refined the microstructure of the composites, resulting in a finer and more uniform distribution of reinforcement particles. Extreme cooling rates promoted higher dislocation density and suppressed grain growth, increasing matrix hardness. This microstructural refinement minimized localized plastic deformation during sliding, improving wear resistance. Increasing the applied load (from 5 N to 15 N) generally increased the wear loss, especially in non-cryogenic treated samples. Higher loads elevated the contact stresses, promoting material removal by microploughing and delamination. However, the improved hardness and strong particle–matrix bonding mitigated these effects in cryogenic-treated samples, showing relatively lower wear rates even at higher loads. At longer sliding distances, the wear loss initially increased due to cumulative damage, but eventually stabilized or decreased slightly at high reinforcement levels and cryogenic treatment. This stabilization is likely due to establishing a stable mechanically mixed layer (MML) and wear debris compaction, which protected the underlying material. Also, the SEM analysis of worn surfaces revealed that non-cryogenic treated samples exhibited deeper grooves, ploughing marks, and delamination pits, indicating severe abrasive and adhesive wear mechanisms. In contrast, cryogenic-treated composites showed smoother surfaces with finer grooves and less severe material removal, confirming improved tribological behavior. The presence of TiO₂-rich tribofilms was evident in cryo-treated samples, which likely contributed to reduced friction and wear by serving as a solid lubricant.

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