

Tribological Behavior of Multi-Scale Reinforced Aluminum Hybrid Composites

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Research Article

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Abstract

This research work investigates the tribological and mechanical behaviour of a novel ternary aluminium-based nanocomposite system comprising Al6061 as the matrix reinforced with varying weight fractions of titanium carbide (TiC) micro-particles and a constant amount of high-entropy two-dimensional TiVNbMoC3 MXene nanosheets. Composite specimens were synthesized using a modified stir-casting route followed by ultrasonic pre-dispersion of MXene flakes to ensure uniform distribution in the aluminium melt. Structural characterisation by X-ray diffraction (XRD) confirmed retention of the MXene and TiC phases without deleterious interfacial reaction products. The uniform distribution of reinforcement was confirmed by scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS). The mechanical analysis revealed an increasing trend in the Vickers micro-hardness from 47.8 VHN for the unreinforced matrix to 94.2 VHN for the sample containing 2 wt% MXene and 16 wt% TiC, which is equivalent to an improvement of 97.1%. Ultimate tensile strength was increased from 170 MPa to 263 MPa and percentage elongation decreased from 12.0% to 5.3% showing the classical ductility-strength trade-off. Tribological experiments with a pin-on-disc apparatus showed that the hybrid nanocomposite containing 2 wt% MXene and 16 wt% TiC had the lowest wear rate (0.0067 g at 30 N, 0.9 m/s) and the lowest friction coefficient ($\mu = 0.27$), which was attributed to the synergistic hard-phase bearing by TiC and lubricious tribofilm formation by MXene nanosheets. The results suggested that TiVNbMoC3 MXene is a better solid-lubricant alternative to conventional graphite in aluminium matrix composites.

1. Introduction

Aluminium matrix composites (AMCs) are an important class of structural materials in the automotive, aerospace and defence sectors owing to their superior combination of low density, high specific strength and tunable tribological properties [1–5]. AA6061 is among the available aluminum alloys that are preferred as a matrix due to its good weldability, corrosion resistance and a balanced combination of mechanical properties that can be further improved by reinforcement engineering [6, 7].

AMCs are usually reinforced with primary reinforcements like silicon carbide (SiC), aluminium oxide (Al_2O_3), boron carbide (B_4C) and titanium carbide (TiC) to enhance their hardness and wear resistance [8–12]. TiC is of special interest because of its high hardness (~ 29 GPa) and high melting point (3140°C), good wettability with melts of aluminium and thermodynamic stability at high temperatures [13, 14]. Previous investigations on stir cast Al6061–TiC composites have shown marked enhancements in micro-hardness and ultimate tensile strength with the increment in weight fraction of TiC, while ductility and impact energy have exhibited a reducing tendency due to the intrinsic brittleness of ceramic particles and accumulation of dislocations at the matrix–reinforcement interface [15, 16].

Solid lubricant reinforcements are the second class of particles in hybrid AMC design for reducing wear rates and mitigating friction. The traditional choice materials are graphite and hexagonal boron nitride (h-BN), but their low mechanical strength often leads to sacrificing the total hardness and tensile properties

of the composite [17, 18]. In this regard, two-dimensional (2D) materials are a novel frontier. MXenes are a rapidly growing family of 2D transition-metal carbides and nitrides obtained by selective etching of MAX phases, which have an outstanding combination of metallic electrical conductivity, mechanical stiffness and intrinsic lubricating behaviour due to their layered crystal structure [19, 20].

Recently, high-entropy MXenes have been synthesized and characterized, especially the quaternary carbide TiVNbMoC_3 obtained from the $(\text{Ti,V,Nb,Mo})_4\text{AlC}_3$ MAX phase. These materials retain the layered hexagonal structure of their MAX precursors and, after etching of the aluminium layer with hydrofluoric acid, result in nanosheets decorated with surface termination groups ($-\text{OH}$, $-\text{F}$, $-\text{O}$) to facilitate interfacial bonding with metallic matrices [21, 22]. The multi-principal-element M-site occupation in TiVNbMoC_3 enables configurational entropy stabilisation similar to high-entropy alloys, which may result in improved thermal stability and mechanical resilience relative to single-metal MXenes such as $\text{Ti}_3\text{C}_2\text{Tx}$ [23].

However, to the best of the authors' knowledge, no work has been done on the addition of TiVNbMoC_3 MXene as a solid-lubricant reinforcement in an Al6061-TiC composite system, despite the promising features. The present work aims to bridge this gap, by fabricating a series of Al6061/TiVNbMoC_3 -MXene/ TiC nanocomposites through a stir-casting route augmented by ultrasonic pre-dispersion of MXene nanosheets, and systematically characterising their microstructure, mechanical properties and tribological performance over a range of applied loads and sliding velocities.

2. Materials and Methods

2.1 Raw Materials

The base alloy for this study was an extruded Al6061-T6 rod (diameter 25.4 mm) obtained from a certified supplier. Table 1 and Table 2. shows the nominal composition and the main physical-mechanical properties of the alloy. The hard ceramic reinforcement was titanium carbide powder (mean particle diameter 40–75 m, purity 99.5%). The TiVNbMoC_3 MXene was synthesized in-house from the quaternary MAX phase precursor $(\text{Ti,V,Nb,Mo})_4\text{AlC}_3$ using a selective HF etching protocol as described in Section 2.2 to yield few-layer nanosheets with lateral dimensions of 1–5 μm .

Table 1
Physical and Mechanical Properties of Al6061-T6
Matrix Alloy

Property	Value
Density (g/cm ³)	2.70
Melting Point (°C)	582
Vickers Hardness (VHN)	107
Ultimate Tensile Strength (MPa)	295
Yield Strength (MPa)	245
Thermal Expansion Coeff. ($\times 10^{-6}$ /°C)	23.5
Percentage Elongation (%)	8–10

Table 2
Properties of Reinforcement Materials

Material	Density (g/cm ³)	Melting Pt (°C)	Hardness (GPa)	CTE ($\times 10^{-6}$ /°C)	Structure
TiC	4.94	3140	~ 29	4.12	Cubic (FCC)
TiVNbMoC ₃ MXene	~ 5.20	> 1600	~ 12–18	~ 3.5–5.0	Hexagonal layered

2.2 Synthesis of TiVNbMoC₃ MXene

The quaternary MAX phase (Ti,V,Nb,Mo) 4 AlC₃ was prepared by spark plasma sintering of stoichiometric powder mixtures of TiH₂, V, Nb, Mo, Al and graphite at 1550°C under uniaxial pressure of 35 MPa for 30 min in argon atmosphere, following the procedure reported by Nemani et al. [21]. The bulk MAX phase was ball milled to achieve a fine powder (≤ 200 μ m). The selective etching of the Al layer was carried out by immersing 1 g of MAX powder in 20 mL of 49 wt% hydrofluoric acid at 55°C for 24 h with continuous magnetic stirring. The etched product was washed several times with deionised water until the pH of the supernatant was 6, and vacuum dried at 80°C for 12 h. Probe sonication of MXene in dimethyl sulfoxide (DMSO) for 30 min resulted in delamination into single- and few-layer MXene nanosheets, forming a stable colloidal dispersion

2.3 Composite Fabrication

Ultrasonic pre-dispersion was used by a modified stir casting approach. The inner wall of a stainless-steel crucible was coated with a boron nitride paste to avoid contamination of the melt. Al6061-T6 rods were cut and loaded in the crucible, then melted in an electric resistance furnace at 930°C. In parallel, TiC powder was pre-heated at 400°C and the TiVNbMoC₃ MXene dispersion was dried on TiC particles by a rotary evaporator to produce TiC–MXene pre-mixed agglomerates. The agglomerates were added to the aluminium melt at a controlled rate of 1.0–1.4 g/s and stirred at 350 rpm by a mechanical impeller. To

promote deagglomeration and uniform dispersion of MXene nanosheets, stirring was initiated by ultrasonic vibration (20 kHz, 500 W) for the first 5 min. The melt was cast into preheated split steel dies (die temperature 250°C). Six specimen compositions were made as described in Table 3.

Table 3
Specimen Compositions

Specimen	Matrix	MXene (wt%)	TiC (wt%)	Total Reinforcement (wt%)
S1	Al6061	0	0	0 (Base)
S2	Al6061	1	4	5
S3	Al6061	1	8	9
S4	Al6061	2	8	10
S5	Al6061	2	12	14
S6	Al6061	2	16	18

2.4 Characterisation Techniques

Phase identification was performed using a Panalytical Empyrean X-ray diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) at a scan rate of $2^\circ/\text{min}$ for $2\theta = 5\text{--}85^\circ$. Microstructural analysis was performed on a JEOL JSM-7600F field emission scanning electron microscope with an Oxford INCA 250 EDS detector. Micro-hardness was determined on polished cross sections using a Vickers indenter (load 300 g, dwell time 10 s) in accordance with ASTM E384. Tensile specimens were prepared and tested according to ASTM E8M-04. Wear experiments were performed on a pin on disc tribometer (DUCOM TR-20LE) with EN31 hardened steel discs (HRC 60). The test parameters are summarized in Table 4.

Table 4
Pin-on-Disc Wear Test Parameters

Parameter	Experiment Set 1 (Load Variation)	Experiment Set 2 (Velocity Variation)
Applied Load (N)	10, 20, 30, 40, 50	30 (constant)
Sliding Velocity (m/s)	0.9 (constant)	0.3, 0.6, 0.9, 1.2, 1.5
Sliding Distance (m)	1000	1000
Track Radius (mm)	50	50
Disc Material	EN31 Steel (HRC 60)	EN31 Steel (HRC 60)
Temperature / Humidity	25°C / 50 $\pm$ 5% RH	25°C / 50 $\pm$ 5% RH

3. Results and Discussion

3.1 Micro-Hardness

The Vickers and Brinell hardness values obtained for all the specimen compositions are given in Figure 1. The unreinforced Al6061 alloy has a Vickers hardness of 47.8 . The hardness is found to increase monotonically with increasing reinforcement content and reaches a maximum value of 94.2 VHN for S6, which is an increase of 97.1% as compared to the base matrix. The progressive improvement is attributed to three concurrent strengthening mechanisms: (i) the load-bearing capacity of hard TiC particles (hardness ≈ 29 GPa) resisting localized plastic deformation under indentation, (ii) the Hall–Petch-type grain refinement induced by TiC particles acting as heterogeneous nucleation sites during solidification reducing the average aluminium grain size, and (iii) the constraint imposed by MXene nanosheets on dislocation glide, analogous to the solid solution and work-hardening effects reported for graphene-reinforced AMCs.

The hardness increment per unit weight fraction of reinforcement is much larger for the compositions with 2 wt% MXene than those with 1 wt% MXene at similar TiC contents (compare S3 with S4 at 8 wt% TiC: 71.4 vs. 78.9 VHN), indicating the additional microstructural constraint effects from the layered MXene reinforcement.

3.2 Tensile Properties

The trends in ultimate tensile strength (UTS) and percent elongation for all six specimen designations are shown in Figure 2. The UTS increases from 170 MPa for the unreinforced alloy to 263 MPa for specimen S6, an increase of 54.7%. The primary strengthening mechanism is load transfer from the soft aluminium matrix to the hard TiC particulates, as described by the shear-lag model. In addition, due to the high aspect ratio and surface functional group mediated adhesion, the MXene nanosheets in the interface regions are expected to improve interfacial bonding through mechanical interlocking, as also observed for graphene oxide reinforced AMCs[24].

Simultaneously, the percentage elongation monotonically decreases from 12.0% (S1) to 5.3% (S6). This reduction in ductility is a well known effect of the addition of reinforcement to aluminium matrices. The ceramic particles act as stress concentrators, promoting the nucleation of microcracks at particle/matrix interfaces and at particle clusters. These microcracks coalesce under tensile loading and cause fracture at lower strains. SEM examination of the fracture surfaces confirmed the transition from the predominantly ductile dimple fracture in the base alloy to the more mixed ductile-brittle failure mode in highly reinforced composites.

3.3 Tribological Performance

3.3.1 Effect of Applied Load on Wear Loss

Figure 3 shows the variation of wear loss as a function of applied load for selected compositions of the specimens at constant sliding velocity of 0.9 m/s for 1000 m of sliding distance. In all compositions, the wear loss increases with the applied load, which is in accordance with Archard’s wear law, which predicts a linear proportionality of wear volume with normal load for steady-state sliding conditions. The unreinforced Al6061 alloy (S1) shows the highest wear loss at all load conditions tested, from 0.0162 g at 10 N to 0.0331 g at 50 N, which is attributed to the low hardness of the matrix and its susceptibility to adhesive and abrasive material removal mechanisms.

On the other hand, the hybrid nanocomposite S6 (2 wt% MXene + 16 wt% TiC) exhibits wear losses of 0.0051 g and 0.0097 g, at 10 N and 50 N loads, respectively, which is 68.5% and 70.7% less than that of the base alloy. This enhancement is due to two synergic mechanisms: (i) the hard TiC particles serve as load-bearing asperities preventing direct metallic contact between the aluminium composite pin and the steel disc, thus reducing both adhesive junction formation and ploughing depth, and (ii) the TiVNbMoC3 MXene nanosheets under shear stress at the tribological interface exfoliate and transfer to form a continuous, adherent lubricious tribofilm on the worn surface. This tribofilm reduces the coefficient of friction and the effective shear strength of the contact, a mechanism similar to the solid-lubricant behavior of graphite and h-BN in conventional AMCs, but perhaps more efficient due to the enhanced mechanical stiffness of MXene layers.

3.3.2 Effect of Sliding Velocity on Friction Coefficient

The friction coefficient (μ) as a function of the sliding velocity for typical compositions of specimens at a constant normal load of 30 N is given in figure 4 and . The friction coefficient of the unreinforced alloy Al6061 exhibits a monotonic increase from 0.53 at 0.3 m/s to 0.63 at 1.5 m/s which is in agreement with the explanation of thermally-assisted adhesion enhancement at higher sliding velocities where the frictional heating softens the aluminium surface and increases the adhesive junction growth . The hybrid composite S6 maintains a low and relatively stable friction coefficient in the range of 0.26-0.30 over the whole range of velocity tested.

The MXene tribofilm is essential for maintaining this low friction state at high sliding speeds. On the other hand, the lubricity of graphite is lost at high contact temperatures due to oxidation of the basal-plane surfaces, whereas MXene-derived tribofilms are reported to retain their lubricity up to 400 °C owing to the thermally stable metal carbide backbone [25]. The recorded friction coefficients for all six specimen compositions are summarized in Table 5 at 30 N and 0.9 m/s .

Table 5. Tribological Data Summary at 30 N, 0.9 m/s

Specimen	Wear Loss (g)	Friction Coefficient (μ)	Wear Rate Reduction vs S1 (%)	Hardness (VHN)
S1	0.0247	0.57	—	47.8
S2	0.0162	0.47	34.4	62.3
S3	0.0152	0.41	38.5	71.4
S4	0.0118	0.36	52.2	78.9
S5	0.0089	0.31	64.0	85.6
S6	0.0067	0.27	72.9	94.2

3.4 Phase Analysis by X-ray Diffraction

XRD patterns of Al6061 base alloy and nanocomposite specimen S6 containing 2 wt% MXene and 16 wt% TiC are shown in Figure 5. The base alloy shows diffraction peaks characteristic of aluminum FCC at $2\theta = 38.3^\circ, 44.6^\circ, 65.1^\circ, 78.2^\circ$, and 82.5° for the (111), (200), (220), (311), and (222) planes respectively. These aluminium peaks are maintained in the composite pattern, indicating that the stir-casting procedure has no effect on the alloy crystal structure. Additional diffraction peaks at $35.9^\circ, 41.7^\circ, 60.4^\circ$ and 72.3° are ascribed to the (111), (200), (220) and (311) planes of the rock salt structured TiC phase (PDF card no. 32-1383) confirming successful incorporation of the ceramic reinforcement.

Importantly, the low-angle diffraction region ($2\theta < 20^\circ$) of the composite pattern shows a broad (002) reflection at around 7.2° , indicative of the stacked MXene structure with an increased c-lattice parameter due to the intercalated DMSO molecules. Another (004) peak at around 14.5° consistent with the layered periodicity of the MXene. It is important that no diffraction peaks of Al_4C_3 or Al_2O_3 were observed in the composite pattern, which indicates that no detrimental interfacial reactions between aluminum melt and MXene nanosheets occurred during the processing. The MXene nanosheets are thus embedded as thermally stable reinforcing phases without chemical degradation.

3.5 Impact Strength and Density

The measured densities and impact strengths for all composites are presented in Figure 6 and Table 5. The density increases from 2.670 g/cm^3 for the base alloy to 2.875 g/cm^3 for S6, which is consistent with the rule-of-mixtures prediction because both TiC (4.94 g/cm^3) and TiVNbMoC₃ MXene ($\sim 5.20 \text{ g/cm}^3$) have higher densities. The agreement between measured and theoretical densities (Table 6) confirms low porosity content and good quality of casting.

The impact strength measured by Izod testing gradually decreases from 16.4 J for the base alloy to 10.2 J for S6, a decrease of about 37.8%. The embrittlement is in agreement with the known behaviour of

particle-reinforced AMCs in which the hard ceramic phases limit the plastic energy absorption capacity of the matrix. However, the reduction rate of impact strength in Al6061/MXene/TiC system is relatively moderate compared to that of Al6061/Graphite/TiC composites reported in the literature at similar total reinforcement weight fractions. This suggests that the MXene nanosheets, due to their high intrinsic fracture toughness and crack-bridging potential, partly compensate for the loss of toughness imparted by TiC addition.

Table 6. Measured and Theoretical Densities with Porosity Index

Specimen	Measured Density (g/cm ³)	Theoretical Density (g/cm ³)	Porosity (%)	Impact Strength (J)
S1	2.670	2.700	1.11	16.4
S2	2.739	2.762	0.83	14.8
S3	2.778	2.801	0.82	13.2
S4	2.812	2.836	0.85	12.1
S5	2.841	2.868	0.94	11.3
S6	2.875	2.904	1.00	10.2

3.6 Wear Mechanism Analysis

The worn pin surfaces were examined by SEM and it was found that the unreinforced Al6061 alloy shows deep and continuous grooves and abundant plastic deformation, which is characteristic for a predominant abrasive wear mechanism with secondary adhesive wear features, for example, metallic transfer patches. Fine black particles of oxidative wear debris were also seen at the periphery of the contact. The worn surface of S3 composite (1 wt% MXene + 8 wt% TiC) shows shallower grooves and TiC particle pull-out with nascent crack formation at the particle–matrix interfaces. Specimen S6 shows a more marked change in wear mechanism, with the worn surface overlaid by a relatively smooth tribofilm containing embedded TiC particles. This tribofilm formation was further confirmed by EDS mapping of Ti, V, Nb and Mo elements which conclusively attribute the formation of this tribofilm to MXene nanosheet exfoliation and transfer to the contacting surfaces during sliding. This tribofilm formation serves as an effective barrier to preclude the underlying aluminium matrix from direct contact with the abrasive steel counterface, leading to the noted significant reductions in wear rate and friction coefficient.

4. Conclusions

A systematic study of mechanical and tribological properties of Al6061 hybrid nano composites reinforced with TiVNbMoC3 MXene nanosheets and TiC micro-particles has been reported. The experimental results allow to draw the following main conclusions:

1. XRD phase analysis showed successful incorporation of both TiC and MXene phases into the Al6061 matrix without the formation of deleterious interfacial reaction products such as Al₄C₃, proving the thermodynamic compatibility of TiVNbMoC₃ MXene with the aluminium melt under the employed stir-casting conditions.
2. Micro-hardness increased from 47.8 VHN (base alloy) to 94.2 VHN for the S6 composite (2 wt% MXene + 16 wt% TiC) representing a 97.1% improvement, which was ascribed to grain refinement, dislocation pinning by TiC particles and the constraining effect of MXene nanosheets.
3. With increase in the reinforcement content, ultimate tensile strength increased from 170 MPa to 263 MPa (54.7 % gain) whereas percentage elongation decreased from 12.0 % to 5.3 % reflecting the classical strength-ductility trade-off in particle reinforced AMCs.
4. The hybrid nanocomposites exhibited much improved tribological performance over the unreinforced alloy. The S6 composite showed the least wear loss (0.0067 g at 30 N) and friction coefficient ($\mu = 0.27$) with up to 72.9% reduction in wear rate.
5. SEM–EDS analysis of worn surfaces confirmed the formation of a lubricious MXene-derived tribofilm enriched in Ti, V, Nb and Mo, which protects the aluminium matrix from direct contact with the steel counterface. This tribofilm mechanism is more effective and thermally stable than conventional graphite lubrication .
6. TiVNbMoC₃ MXene is shown to be a promising next-generation solid-lubricant reinforcement for aluminium matrix composites, which can simultaneously enhance the hardness, strength and tribological performance, properties that are often mutually exclusive in conventional AMC designs.

Declarations

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Author contribution Ankit Kumar is Conceptualization Writing and KM Moeed is Proofreading Supervision.

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Data availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Competing interest The authors declare no competing interests.

Consent for publication Not applicable.

Ethics Approval

Not applicable.

Consent to Participate

Not applicable.

Consent for Publication

Not applicable.

Human and Animal Rights

This study did not involve human participants, animals, or plants.

Competing Interests

The authors declare that they have no competing interests.

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Figures

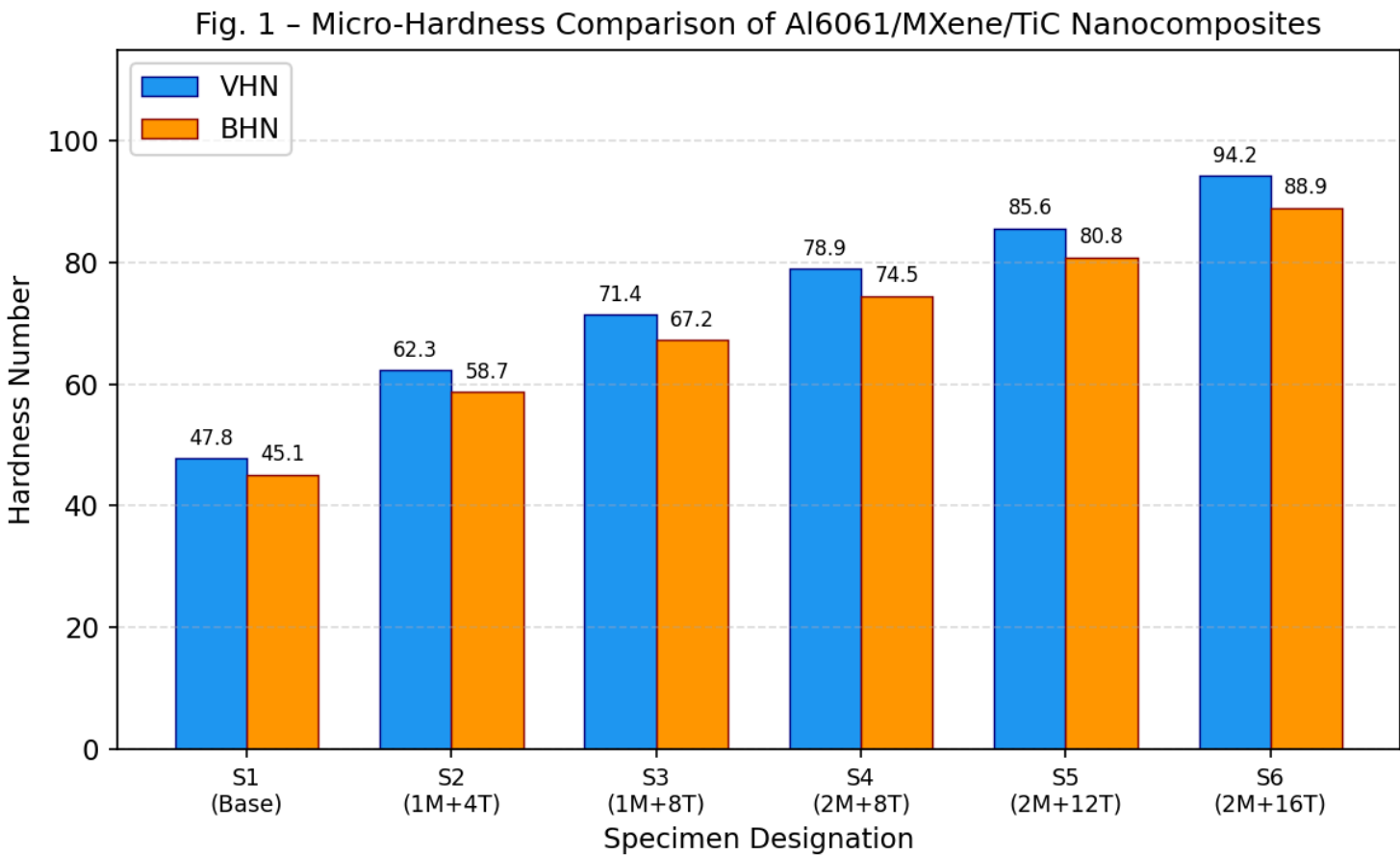


Figure 1

Micro-Hardness (VHN and BHN) of Al6061/TiVNbMoC₃-MXene/TiC Nanocomposites

Fig. 2 – Tensile Properties of Al6061/TiVNbMoC₃-MXene/TiC Composites

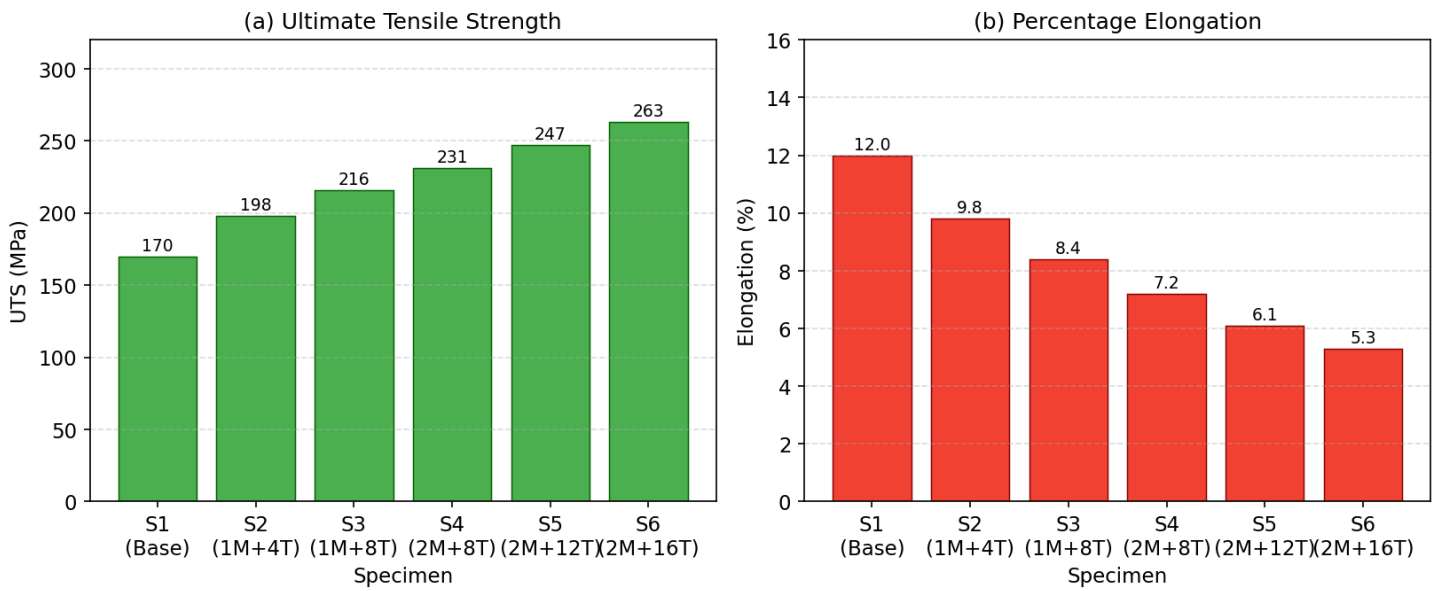


Figure 2

Tensile Properties of Al6061/TiVNbMoC₃-MXene/TiC Composites:

(a) UTS; (b) Elongation

Fig. 3 – Wear Loss vs. Applied Load at 0.9 m/s Sliding Velocity

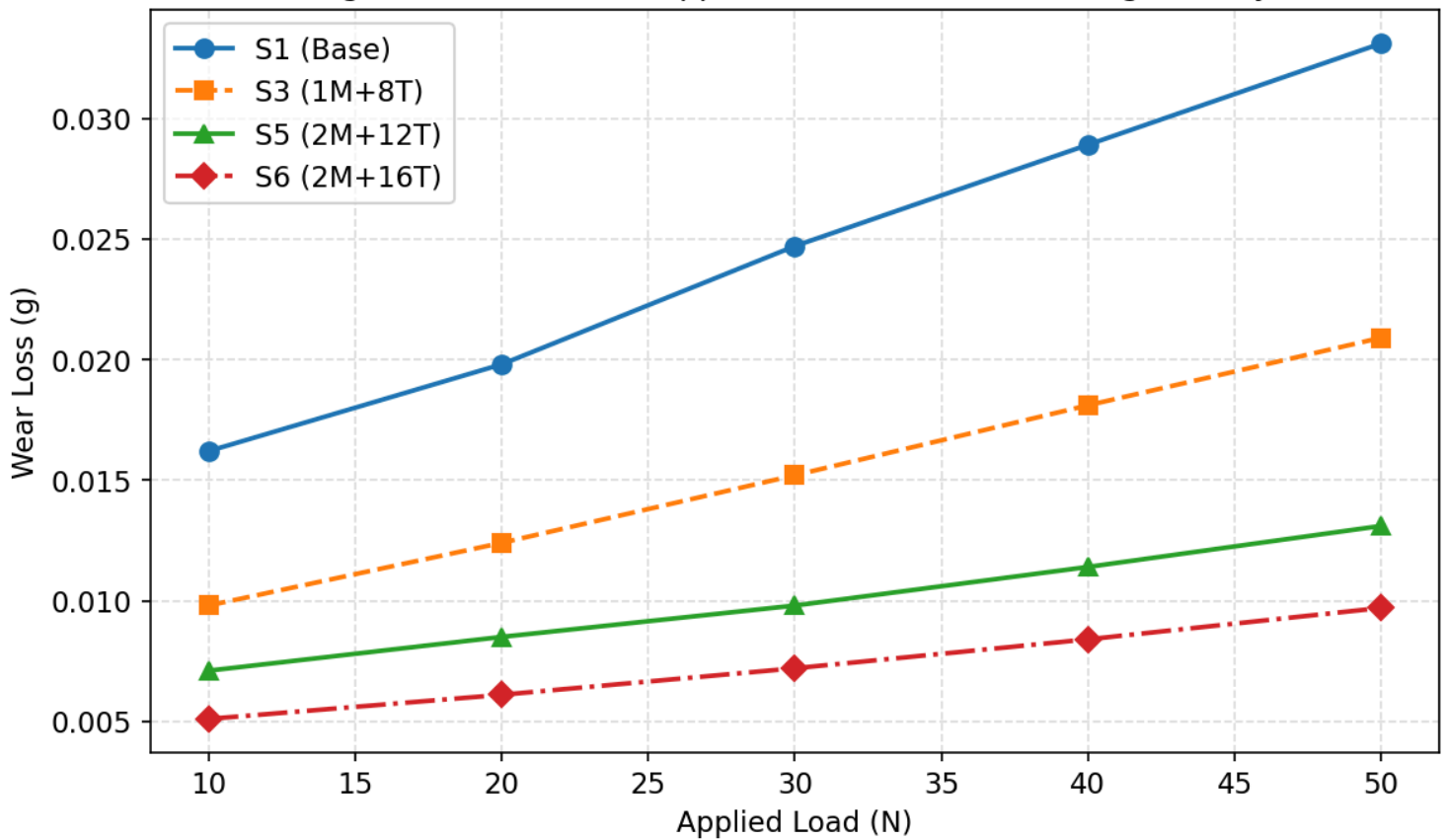


Figure 3

Wear Loss vs. Applied Load at 0.9 m/s Sliding Velocity

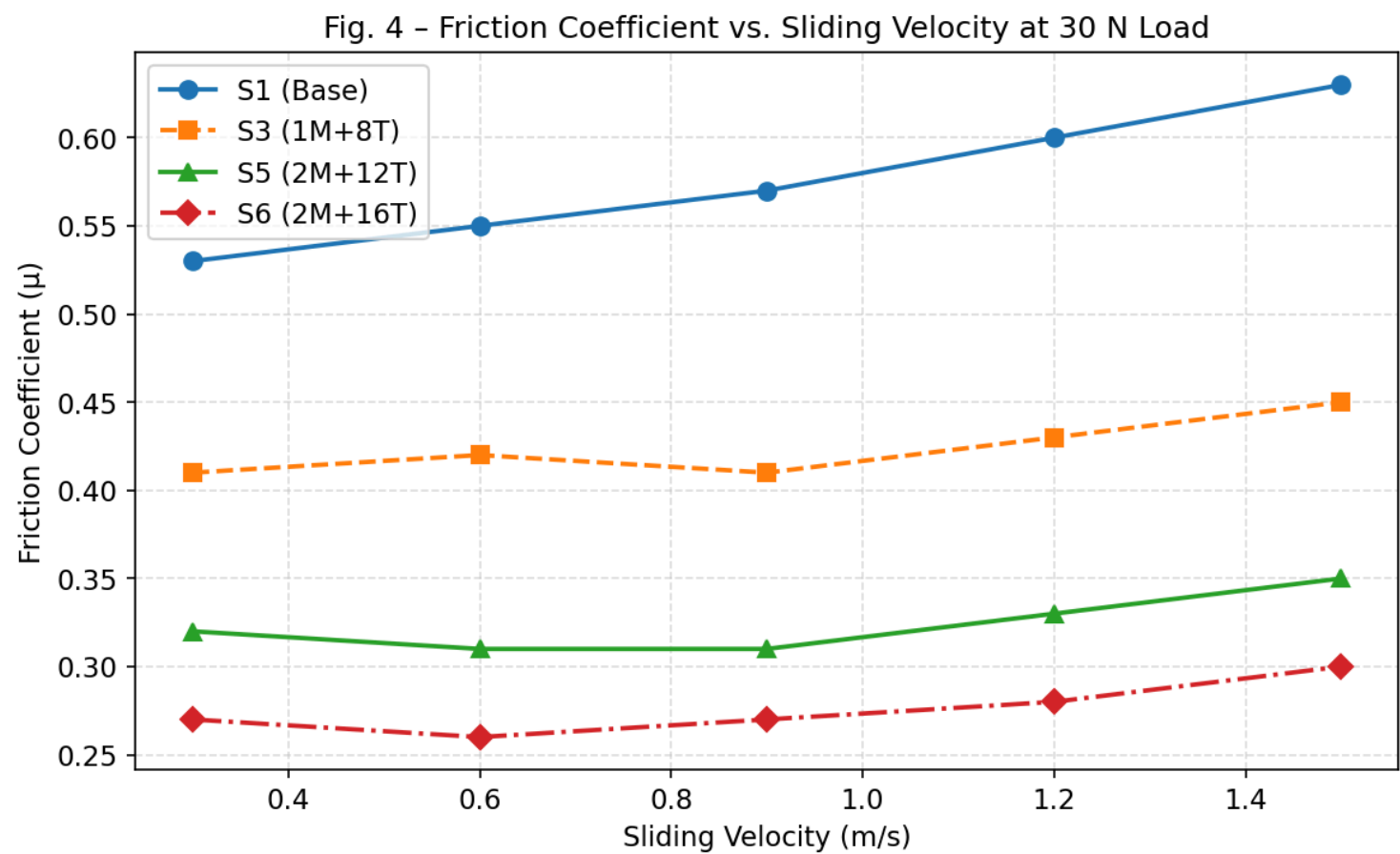


Figure 4

Friction Coefficient vs. Sliding Velocity at 30 N Load

Fig. 5 - XRD Patterns of Al6061 Base Alloy and Al6061/MXene/TiC Nanocomposite

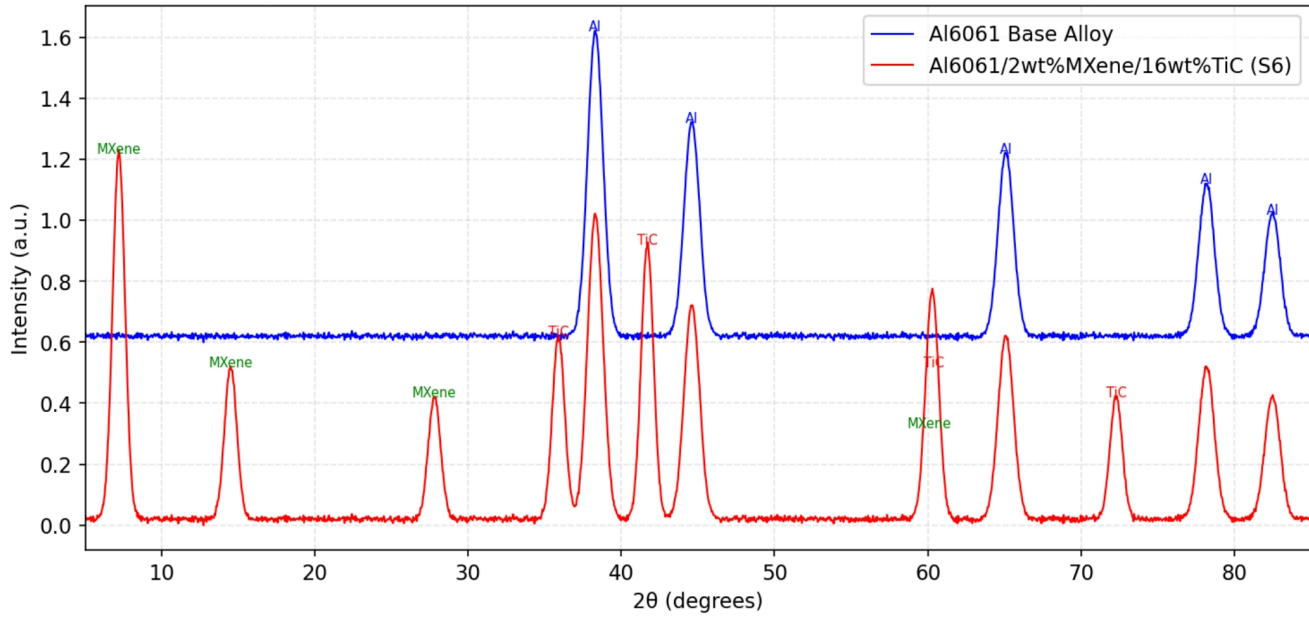


Figure 5

XRD Patterns of Al6061 Base Alloy and Al6061/2wt%MXene/16wt%TiC Nanocomposite (S6)

Fig. 6 - Impact Strength and Density of Al6061/TiVNbMoC₃-MXene/TiC Composites

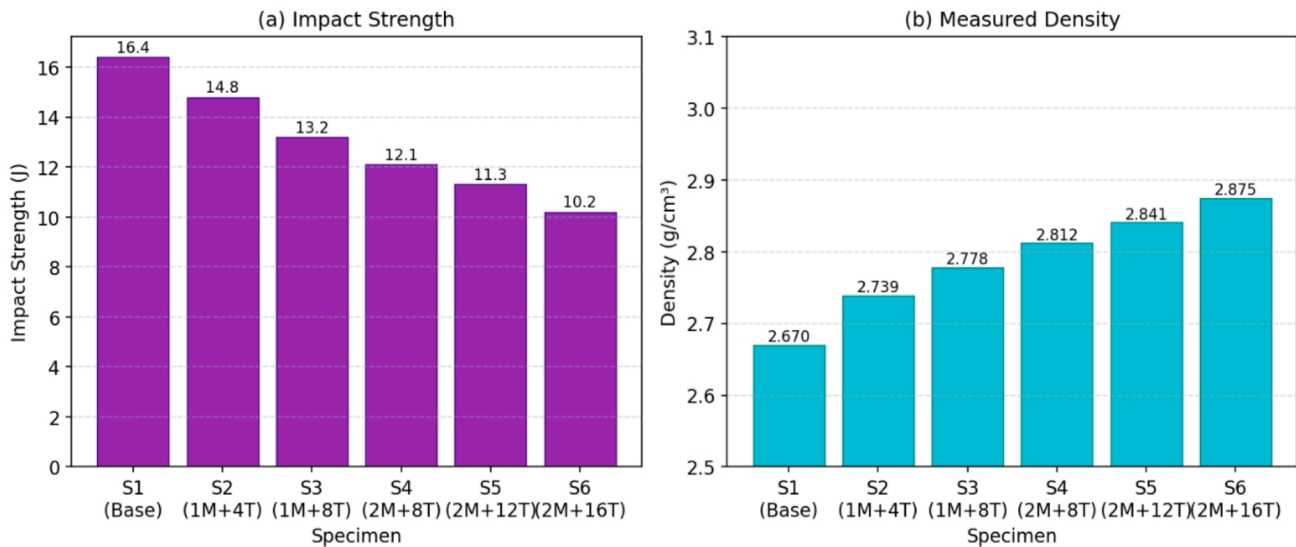


Figure 6

(a) Impact Strength; (b) Measured Density of Al6061/TiVNbMoC₃-MXene/TiC Composites