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Microstructural Evolution and Mechanical Properties of Hybrid Al6060/TiB₂–MWCNT Composites Fabricated by Ultrasonically Assisted Stir Casting and Radial-Shear Rolling

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Abstract

This work presents a comprehensive study on the fabrication, microstructural evolution, and mechanical performance of hybrid aluminum matrix composites based on Al6060 alloy reinforced with ~2 wt.% TiB₂ and ~1 wt.% multi-walled carbon nanotubes (MWCNTs). The composites were produced via ultrasonically assisted stir casting followed by radial-shear rolling (RSR). The combined processing route enabled a uniform distribution of reinforcing phases and significant grain refinement in the aluminum matrix. SEM, EDS, XRD, and EBSD analyses revealed that TiB₂ particles acted as nucleation centers and grain boundary pinning agents, while MWCNTs provided a network structure that suppressed agglomeration of ceramic particles and enhanced interfacial load transfer. As a result, hybrid composites demonstrated a submicron-grained structure with reduced anisotropy. Mechanical testing confirmed that yield strength (YS) and ultimate tensile strength (UTS) increased by 67% and 38%, respectively, in the cast state compared to unreinforced Al6060, while after RSR processing, YS exceeded 115 MPa and UTS reached 164 MPa, with elongation preserved at 14%. Microhardness increased from 50.2 HV0.2 (base alloy) to 82.2 HV0.2 (hybrid composite after RSR). The combination of ultrasonic melt treatment and RSR thus provided a synergistic effect, enabling simultaneous strengthening and ductility retention. These findings highlight the potential of hybrid Al6060/TiB₂–MWCNT composites for structural applications requiring a balance of strength, ductility, and wear resistance.

Keywords: hybrid Al6060/TiB₂–MWCNT composites; ultrasonically assisted stir casting; radial-shear rolling (RSR); microstructure; mechanical properties



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1. Introduction

Aluminum alloys are widely used as structural materials in transportation [1], aerospace [2], energy [3], and other high-performance applications due to their low density, high specific strength, and excellent corrosion resistance. Nevertheless, their functional properties—particularly wear resistance and strength under high loads—often fall short of the requirements for advanced engineering structures [4]. This has driven significant interest in aluminum matrix composites (AMCs), where the metallic matrix is reinforced

with dispersed ceramic particles or carbon-based nanostructures. These composites combine the inherent ductility of the aluminum matrix with the high stiffness and strength of the reinforcement, achieving a property balance that is difficult to attain with conventional alloys [5–7].

Over the past decades, aluminum alloy-based composites have been extensively investigated with various reinforcing agents, including carbides [8,9], oxides [10,11], nitrides [12,13], borides [14], and agricultural or industrial by-products [15,16]. Among these, titanium diboride (TiB_2) has received particular attention, as it can be incorporated *ex situ* as preformed particles [17–19]—offering high hardness, elastic modulus, and thermal stability—or synthesized *in situ* directly in molten aluminum [20–22]. The addition of nanoscale TiB_2 effectively enhances the wear resistance and yield strength of aluminum alloys while maintaining acceptable ductility. Nevertheless, relying on a single ceramic phase does not always yield an optimal balance of properties, since high reinforcement levels may compromise ductility and impact toughness. This challenge has motivated the development of hybrid aluminum composites, which combine two or more reinforcing phases with distinct characteristics and matrix interaction mechanisms, enabling synergistic enhancements in overall performance [23,24]. Carbon nanomaterials, in particular, have attracted significant interest due to their low density, high thermal conductivity, excellent tribological performance, and efficient load transfer through strong interfacial bonding. Incorporating carbon nanostructures such as graphene [25,26] or carbon nanotubes [27,28] further improves stiffness, wear resistance, and thermal stability of aluminum alloys while reducing weight compared to traditional ceramic reinforcements. Multi-walled carbon nanotubes (MWCNTs) are especially notable for their extremely high elastic modulus (on the order of terapascals), hardness, and tensile strength. Their high specific strength, large aspect ratio, and extensive surface area not only contribute to additional reinforcement but also facilitate strong interfacial bonding with the aluminum matrix, ensuring efficient load transfer and minimizing localized deformation [29–31].

Despite considerable advances in the production and modification of aluminum composites, achieving a uniform distribution of reinforcing particles within the matrix remains a major challenge. This issue is especially pronounced for liquid-state stir casting, one of the most widely employed and cost-effective methods for fabricating aluminum composites [32–35]. While stir casting offers simplicity and low production costs, it often struggles with nanoparticle dispersion and uniform microstructure formation, resulting in local stress concentrations and reduced mechanical performance. Ultrasonic treatment of the melt has emerged as a promising solution, promoting degassing, breaking particle agglomerates, and improving nanoparticle distribution throughout the aluminum matrix [36–38]. Plastic deformation provides an additional means of controlling composite structure and properties, particularly through processes that induce favorable deformation modes such as shear [39,40] or compression [41]. Rolling processes are of particular relevance, forming an essential part of modern metallurgical production [42,43], as continuous casting is typically integrated with subsequent rolling within a single production cycle [44,45]. Rolling not only achieves the desired geometrical dimensions but also serves as a critical tool for microstructural control, including grain refinement, activation of interfacial interactions, and development of internal stresses that strengthen the matrix. Among rolling techniques, radial-shear rolling (RSR) is especially effective, combining compression and shear in a single pass to generate high-intensity plastic deformation [46–48]. This enables efficient formation of submicron- and nanostructures in metallic materials and offers extensive opportunities for property optimization. Nevertheless, systematic studies on the effects of RSR on the microstructure and properties of cast aluminum composites remain scarce,

limiting understanding of the underlying strengthening mechanisms and constraining the targeted optimization of processing parameters to achieve the desired performance.

The novelty of this study lies in a combined approach for producing Al6060-based aluminum composites reinforced with ~2 wt.% TiB₂ and an additional ~1 wt.% MWCNTs. The integration of ultrasonic treatment during melt processing with subsequent radial-shear rolling (RSR) promotes uniform dispersion of the reinforcing phases and the development of a fine-grained matrix, resulting in a synergistic enhancement of mechanical properties. For the first time, this work establishes the correlation between processing parameters, microstructure, and the comprehensive set of performance characteristics of the composites. The objective of the study is to elucidate the effects of ultrasonic melt treatment and RSR on the microstructural evolution, reinforcement distribution, and mechanical behavior of Al6060/TiB₂-MWCNTs hybrid aluminum composites.

2. Materials and Methods

2.1. Raw Materials

Aluminum alloy Al6060 was selected as the matrix material for this study. Plate specimens measuring 3 × 30 × 160 mm³ were machined from commercially available long-length Al6060 sheets obtained from Metall-Komplekt LLP (Karaganda, Kazakhstan). The chemical composition of the alloy is summarized in Table 1.

Table 1. Chemical composition of Al6060 alloy (wt.%).

Mg	Si	Mn	Fe	Cu	Zn	Ti	Others	Al
0.35–0.50	0.30–0.60	≤0.10	0.10–0.30	≤0.10	≤0.15	≤0.10	≤0.15	97.65–99.35

Titanium diboride (TiB₂) powder, gray-black in color with a commercial purity of approximately 99.5%, was supplied by Luoyang Tongrun Info Technology Co., Ltd. (Luoyang, China). Multi-walled carbon nanotubes (MWCNTs), black in color with a purity exceeding 99%, were obtained from Guangzhou Hongwu Material Technology Co., Ltd. (Guangzhou, China). The morphology of both powders was characterized using scanning (SEM, Crossbeam 540 microscope, Carl Zeiss, Oberkochen, Germany) and transmission electron microscopy (TEM, JEM-1400 Plus microscope, Jeol Ltd., Tokyo, Japan).

SEM analysis of the TiB₂ powder (Figure 1a) revealed predominantly irregular, angular particles with sharp edges and fractures, indicative of mechanical milling. Particle sizes ranged from 5 to 15 µm, with an average of 6–7 µm, and an aspect ratio of 1.3–1.6, suggesting primarily isometric but occasionally slightly elongated shapes.

MWCNTs exhibited a characteristic network of entangled fibrous structures forming a three-dimensional, “web-like” morphology, as observed in SEM (Figure 1b) and TEM (Figure 1c) images. High-resolution TEM (×800,000) allowed determination of the outer diameter of the hollow multi-walled nanotubes (20 ± 1 nm) and the interlayer spacing (~0.36 nm).

The elemental composition of the reinforcing materials was analyzed by energy-dispersive spectroscopy (EDS, JSM-IT200 microscope, Jeol Ltd., Tokyo, Japan). The EDS spectrum of TiB₂ (Figure 2a) confirmed the presence of titanium (Ti) and boron (B), whereas the spectrum of MWCNTs (Figure 2b) showed exclusively carbon (C), indicating high purity and absence of detectable impurities.

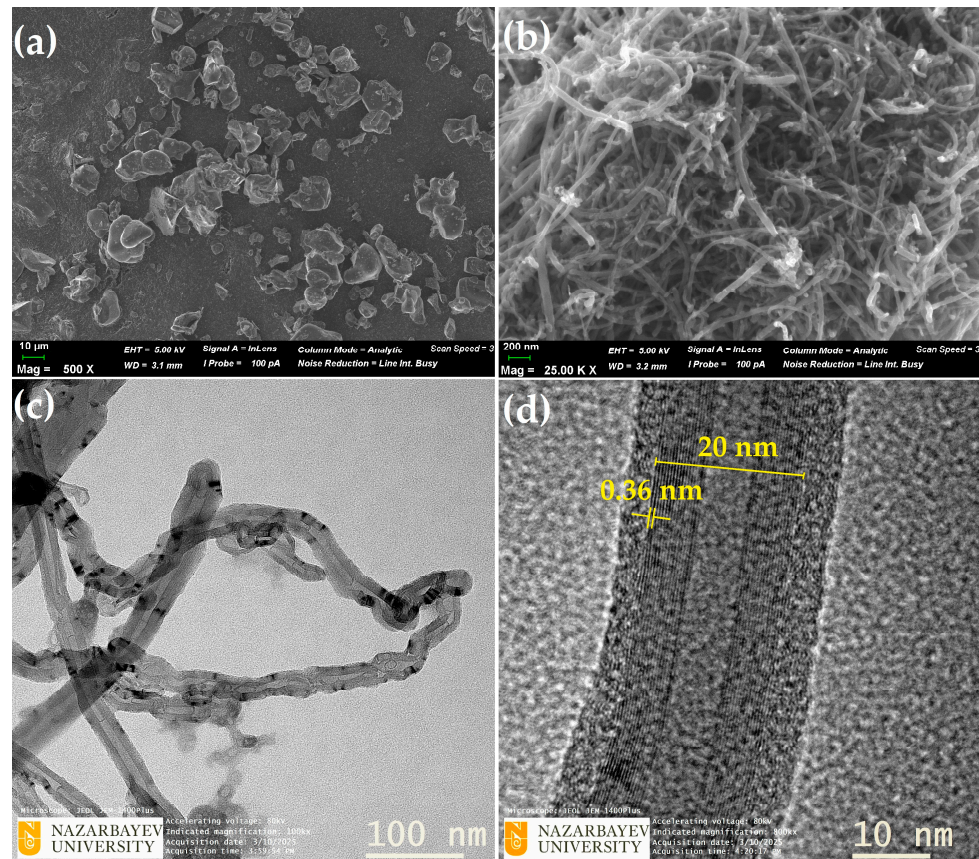


Figure 1. SEM and TEM characterization of TiB_2 and MWCNT powders: (a) SEM image of TiB_2 particles at $\times 500$ magnification; (b) SEM image of entangled MWCNT network at $\times 25,000$ magnification; (c) TEM image of MWCNT at $\times 100,000$ magnification; (d) TEM image of MWCNT at $\times 800,000$ magnification.

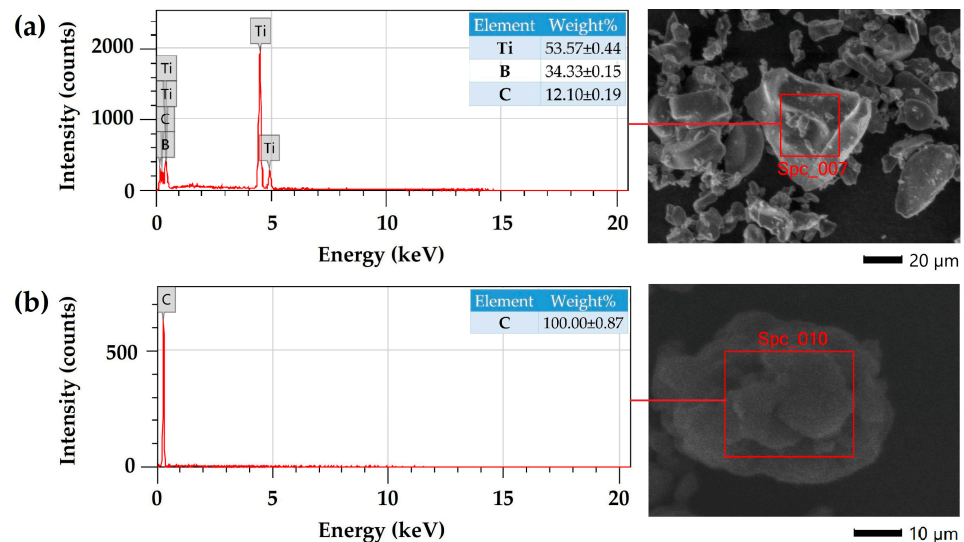


Figure 2. Energy-dispersive X-ray spectroscopy (EDS) spectra and the corresponding elemental compositions of the powders: (a) TiB_2 , and (b) multi-walled carbon nanotubes (MWCNTs).

The phase composition of the reinforcing materials was analyzed by X-ray diffraction (XRD, Rigaku SmartLab X-ray diffractometer, Rigaku Corporation, Tokyo, Japan), as shown in Figure 3a,b. For TiB_2 , intense diffraction peaks were observed at $2\theta \approx 27.74^\circ$, 34.30° , 44.61° , 57.13° , 61.31° , 68.27° , 68.52° , 72.11° , 78.85° , and 88.57° , corresponding to the (001),

(100), (101), (002), (110), (102), (111), (200), (201), and (202) planes of the hexagonal crystal lattice (JCPDF card No. 35-0741) [49,50]. These results confirm the high phase purity of the TiB_2 powders.

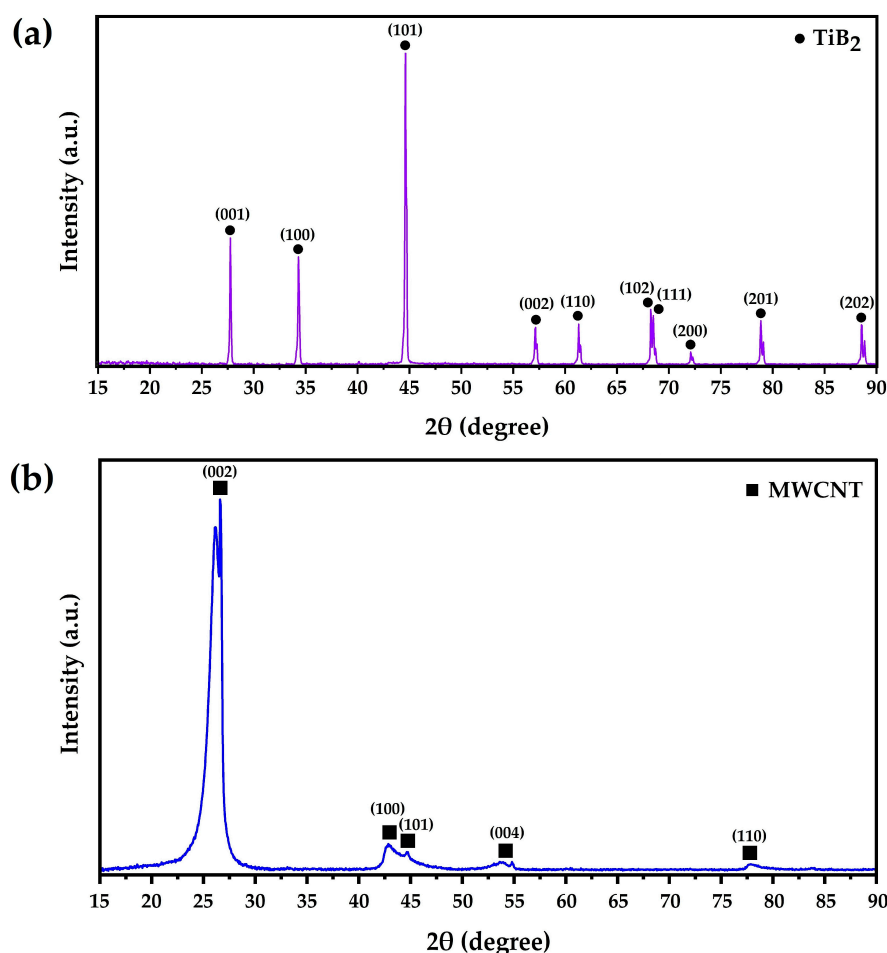


Figure 3. X-ray diffraction (XRD) patterns of the powders: (a) TiB_2 and (b) multi-walled carbon nanotubes (MWCNTs).

The XRD pattern of MWCNTs, presented in Figure 3b, exhibits characteristic diffraction peaks at $2\theta \approx 26.14^\circ$ (002), 42.82° (100), 44.46° (101), 53.91° (004), and 77.81° (110), typical of graphite-like carbon (JCPDS card No. 96-101-1061) [51,52]. The most prominent peak at $2\theta \approx 26.14^\circ$, corresponding to the (002) plane, reflects the interlayer spacing of the graphite-like layers, confirming the multi-walled structure of the nanotubes. Other reflections are considerably weaker. Compared to TiB_2 , the MWCNT peaks are broader and less intense, reflecting their nanoscale dimensions and the presence of structural defects.

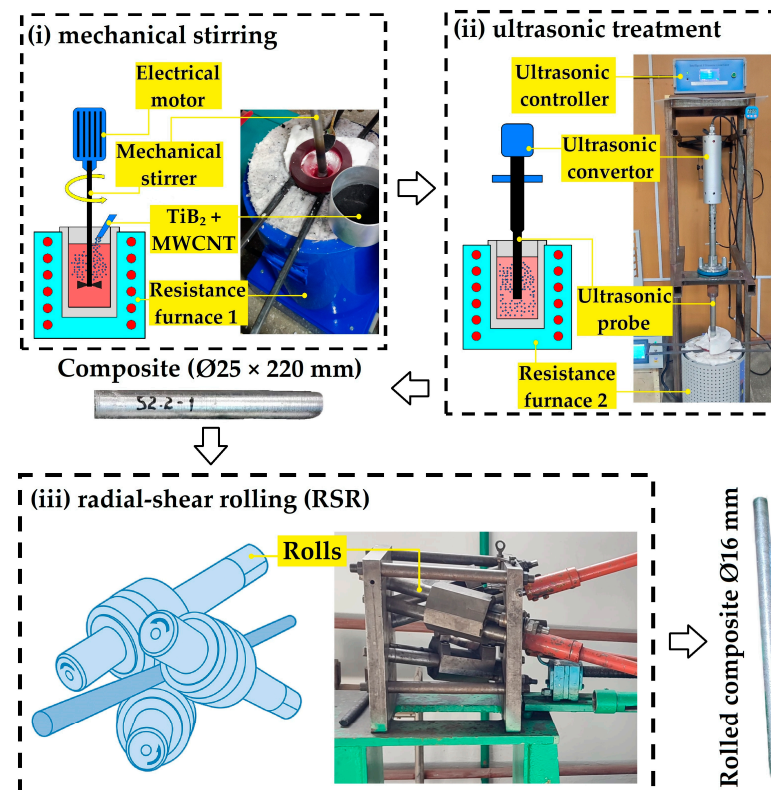
2.2. Composite Fabrication Procedure

The designation of the fabricated materials, along with their corresponding weight fractions (wt.%), is summarized in Table 2.

The materials listed in Table 2 were fabricated using a multi-stage process comprising three sequential steps: (i) mechanical stirring, involving the initial blending of TiB_2 and MWCNT particles in molten aluminum; (ii) ultrasonic treatment, applied to achieve uniform particle dispersion and to deagglomerate TiB_2 and MWCNT within the melt; and (iii) radial-shear rolling (RSR), employed for additional strengthening through grain refinement and densification of the particle distribution within the matrix. These steps are schematically presented in Figure 4.

Table 2. Nomenclature and description of the fabricated composites.

Nomenclature	Designation	Al6060 (wt.%)	TiB ₂ (wt.%)	MWCNTs (wt.%)
Al6060–Cast	S0-C	100	0	0
Al6060–Rolled	S0-R	100	0	0
Al6060/TiB ₂ –Cast	S1-C	98	2	0
Al6060/TiB ₂ –Rolled	S1-R	98	2	0
Al6060/TiB ₂ /MWCNT–Cast	S2-C	97	2	1
Al6060/TiB ₂ /MWCNT–Rolled	S2-R	97	2	1

**Figure 4.** Schematic representation of aluminum material fabrication steps.

Aluminum plates (3 × 30 × 160 mm, 1694–1710 g) were loaded into a graphite crucible (Ø84 × 181 mm) and melted in resistance furnace 1 at 750 ± 10 °C. Temperature was controlled using a dedicated furnace controller and monitored with a K-type thermocouple. To minimize hydrogen content and gas porosity, the melt was purged with argon (2–3 L/min). After melting approximately half the crucible depth, a steel four-blade mechanical stirrer, driven by an electric motor, was introduced. Stirring speed was gradually increased to ~600 rpm. Pre-prepared TiB₂ and MWCNT powders were added into the melt vortex. Aluminum powder mass was increased by 0.25–0.35% relative to Table 2 to compensate for non-mixing particles and ensure the target composition. To reduce thermal mismatch, the powders were preheated to 500 °C at 3.75 °C/min, preventing agglomeration and moisture contamination. For the hybrid composite S2-C, TiB₂ and MWCNT powders were pre-mixed in a laboratory mini-mixer and gradually incorporated into the melt, followed by 15 min of stirring at ~600 rpm. The melt was then transferred to a preheated resistance furnace 2, stabilized at 750 ± 10 °C, for ultrasonic treatment using a JH-LRT30 system (Hangzhou Precision Machinery Co., Ltd., Hangzhou, China). The system comprised an ultrasonic converter and a titanium-alloy probe inserted to approximately half of the crucible depth.

The ultrasonic controller recorded a frequency range of 20.53–20.72 kHz depending on slight melt temperature fluctuations (average value ~ 20.6 kHz). In accordance with the manufacturer's recommendations, 85% of the system's maximum rated power (≈ 1.7 kW out of 2 kW) was applied to ensure stable cavitation and prevent premature probe degradation. Sonication was conducted for 15 min, which promoted particle deagglomeration and uniform dispersion [37,38]. The treated melt was cast into a preheated (450°C) seven-channel steel mold ($\text{Ø}25 \times 220$ mm) and cooled to room temperature. Five samples were prepared per composition, with the top sections removed to eliminate external shrinkage. The resulting billets ($\text{Ø}25$ mm) were subsequently subjected to radial-shear rolling to a final diameter of 16 mm.

The rolling mill featured three rolls arranged symmetrically around the rolling axis at 120° intervals. The process was conducted at a roll speed of 100 rpm and a temperature of 200°C , corresponding to warm deformation conditions for the Al6060 alloy. Each pass achieved a nominal reduction of $\sim 20\%$, and four consecutive passes were applied, yielding a cumulative true reduction of approximately 59%, corresponding to a total true strain of ~ 0.89 .

2.3. Material Characterization

The microstructure and properties of ultrasonically assisted stir-cast and RSR-processed samples were characterized using SEM, EDS, XRD, electron backscatter diffraction (EBSD), tensile testing, hardness, and density measurements. Microhardness was measured on cylindrical specimens ($\text{Ø}16 \times 30$ mm) according to ASTM E384 [53] using an HVT-1000A microhardness tester (Laizhou Laihua Testing Instrument Factory, Laizhou, China) equipped with a Vickers diamond indenter (136°). A 0.2 kgf load was applied for 15 s, and at least five measurements were performed at different locations on each sample.

Density was determined by hydrostatic weighing on $\text{Ø}16 \times 12$ mm samples at $23 \pm 1^\circ\text{C}$ using an MH-300A electronic densitometer (Shenzhen Omena Technology Co., Ltd., Shenzhen, China), following ASTM procedures [54,55]. Theoretical (ρ_{th}) and experimental (ρ_{exp}) densities, as well as porosity, were calculated using the nominal densities of the aluminum matrix (~ 2.71 g/cm³), TiB₂ (~ 4.52 g/cm³), and MWCNT (~ 2.10 g/cm³), with composition values given in Table 2.

Tensile specimens were machined into dog-bone shapes (gauge diameter 9 mm, length 60 mm) according to ASTM E8 [56]. Tests were conducted on a WDW-100 kN universal testing machine (Jinan Xinluchang Testing Machine Co., Ltd., Jinan, China) at a constant crosshead speed of 1 mm/min ($\sim 10^{-3}$ s⁻¹ strain rate) at room temperature, measuring yield strength (YS), ultimate tensile strength (UTS), and elongation.

For microstructural and phase analyses, specimens were cut along the diametral section using a Brilliant 220 precision saw (ATM Qness GmbH, Mammelzen, Germany) to a thickness of 3 mm, hot-mounted in carbon-containing resin (Struers PolyFast) using a Citopress-5 press (Struers ApS, Ballerup, Denmark), and mechanically polished. Grinding was performed with SiC papers (320–2000 grit, Shenzhen Sun Abrasives Imp. & Exp. Corp., Shenzhen, China), followed by diamond polishing ($9 \rightarrow 3 \rightarrow 1$ μm) and final polishing with Eposil M suspension (0.06 μm , pH 9.5, ATM Qness GmbH, Mammelzen, Germany).

SEM and EDS analyses were carried out on a JSM-IT200 SEM (Jeol Ltd., Tokyo, Japan) at 20 kV. Phase composition was examined using XRD on a Rigaku SmartLab diffractometer, Rigaku Corporation, Tokyo, Japan ($\text{Cu K}\alpha$, $\lambda = 1.5406$ Å, $2\theta = 5\text{--}90^\circ$, step 0.05°). EBSD analysis was conducted on a Thermo Fisher Helios 5 UX SEM (Thermo Fisher Scientific, Waltham, MA, USA) to obtain inverse pole figure (IPF) maps in the RD–ND and TD–ND planes [57–59].

3. Results and Discussion

3.1. Microstructural Observations

SEM images and EDS spectra of the as-cast samples S0-C, S1-C, and S2-C are presented in Figure 5.

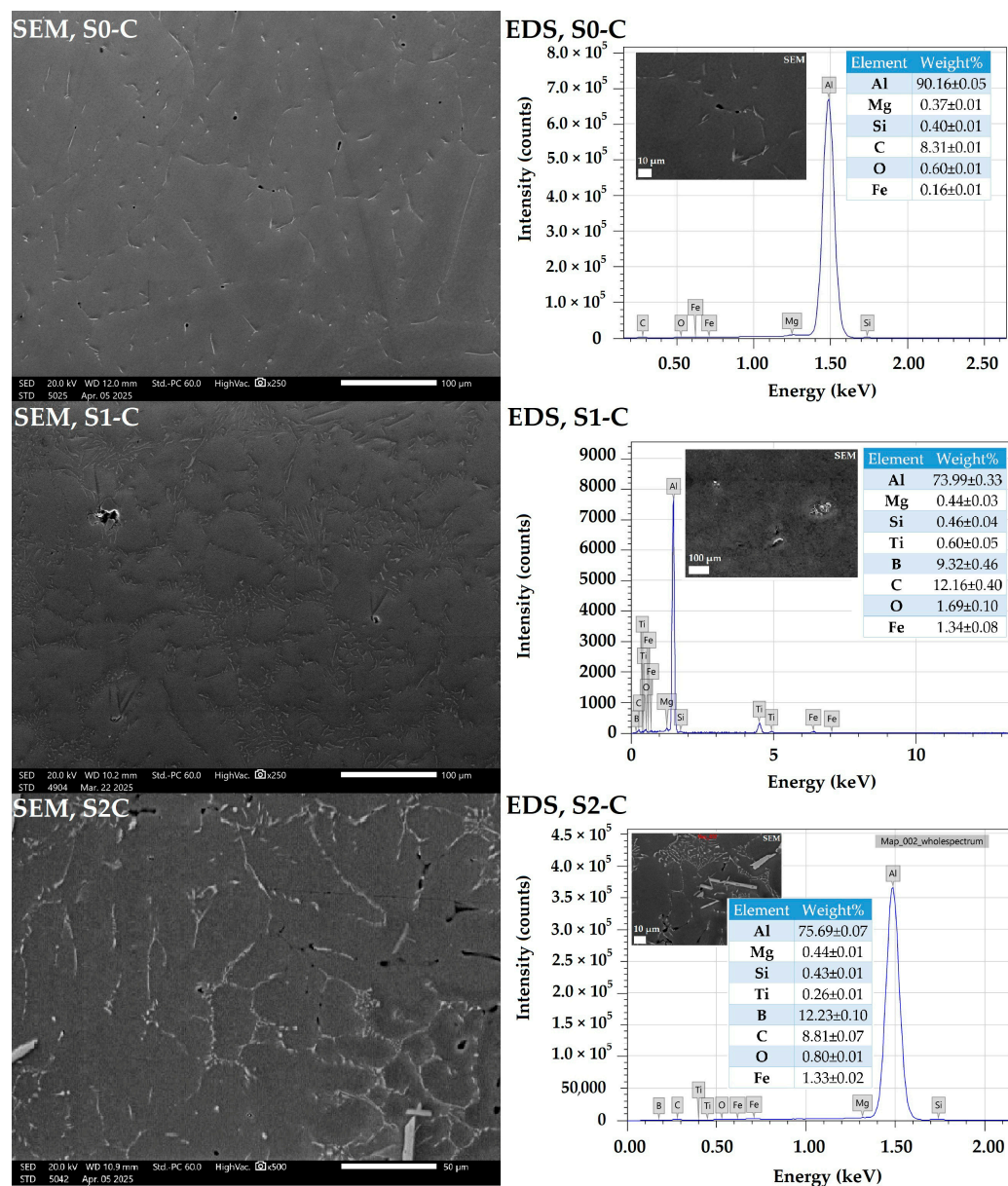


Figure 5. SEM images and EDS spectra of the as-cast samples S0-C, S1-C, and S2-C.

Sample S0-C exhibits a typical dendritic morphology of cast aluminum alloys [60,61], with relatively large primary dendritic cells measuring 50–100 μm , separated by interdendritic regions. These regions consist of eutectic phases enriched in silicon and iron, as confirmed by local EDS analysis. The spectrum shows dominant peaks for aluminum (matrix), silicon, and magnesium (major alloying elements), along with traces of carbon, oxygen, and iron. Iron is attributed to residual impurities and the formation of intermetallic Al-Fe-Si phases, commonly found in cast aluminum alloys [62,63]. The S0-C microstructure shows limited homogeneity, with microshrinkage defects and gas pores of 5–15 μm in some regions, resulting from hydrogen evolution during solidification [64,65].

In S1-C, the addition of 2 wt.% TiB₂ significantly modified the alloy morphology. The dendritic structure is less pronounced, reflecting the role of TiB₂ particles as heterogeneous nucleation sites [66,67]. Sharp-edged gray particles identified as TiB₂ are dispersed throughout the matrix, ranging from 5 to 15 µm, consistent with the original powder size. Particle distribution is generally uniform, though small agglomerates of 3–5 particles appear mainly in interdendritic regions with lower solidification rates. Ultrasonic treatment effectively minimized global segregation and ensured good dispersion. EDS confirmed the presence of titanium (peak ~4.5 keV) and boron (peak ~0.18 keV), alongside aluminum and its alloying elements, indicating that the chemical integrity of the reinforcing phase was maintained. Notably, local grain refinement occurs near TiB₂ particles, reducing aluminum grain sizes to 40–60 µm, while more distant areas retain coarser grains. This observation aligns with the grain refinement mechanism, where dispersed particles serve as nucleation sites for new aluminum crystals [68,69].

Microstructural evolution was also investigated in the hybrid sample S2-C. SEM analysis revealed that, compared to the as-cast condition, the microstructure became significantly more homogeneous and fine-grained, with an average grain size of 25–35 µm, reflecting the combined effect of both reinforcing phases.

The incorporation of an additional carbon phase in the form of 1 wt.% MWCNTs further enhanced grain refinement. SEM images demonstrated a more uniform microstructure with grain sizes of approximately 35–45 µm. The MWCNTs formed a three-dimensional network that inhibited local TiB₂ coalescence and promoted a more uniform particle distribution. Elongated regions, attributed to MWCNT agglomerates, were observed in the S2-C structure, typically 1–2 µm wide and 5–10 µm long, indicating partial compaction during solidification. However, their overall fraction was low and did not compromise the macroscopic homogeneity of the composite. TiB₂ particles retained their morphology and remained uniformly dispersed, while the presence of MWCNTs further reduced their tendency to agglomerate. This suggests that the nanotubes stabilize TiB₂ particles and hinder coalescence during crystallization [70,71]. EDS analysis confirmed the presence of carbon (~0.28 keV), in addition to aluminum, titanium, and boron, while the absence of other peaks indicated the high chemical purity of MWCNTs and the lack of undesirable reactions with the aluminum matrix.

SEM investigation of RSR-processed samples (Figure 6) revealed significant differences in microstructural evolution depending on composition and the presence of reinforcing phases. Analyses were conducted on two perpendicular sections, RD-ND (rolling direction-normal direction) and TD-ND (transverse direction-normal direction), allowing evaluation of microstructural anisotropy induced by shear deformation.

For the S0-R sample, SEM images show relatively indistinct grain boundaries compared to S1-R and S2-R, with blurred interfaces between adjacent grains and weakly developed deformation bands. The absence of reinforcing particles permits relatively free grain boundary migration, resulting in grain sizes of approximately 25–45 µm, far from submicron scales. Occasional larger recrystallized grains indicate the onset of static recrystallization at 200 °C.

The addition of TiB₂ particles markedly modifies the microstructure. In the RD-ND section, TiB₂ particles are uniformly distributed along grain and subgrain boundaries, acting as effective barriers to grain growth [72,73]. In the TD-ND section, fine-grained structures develop, with TiB₂ particles generating local stresses and promoting activation of additional slip systems. Distorted microstructural zones near the particles reflect thermomechanical mismatches between the aluminum matrix and ceramic reinforcement, enhancing strength while potentially reducing local ductility [74,75].

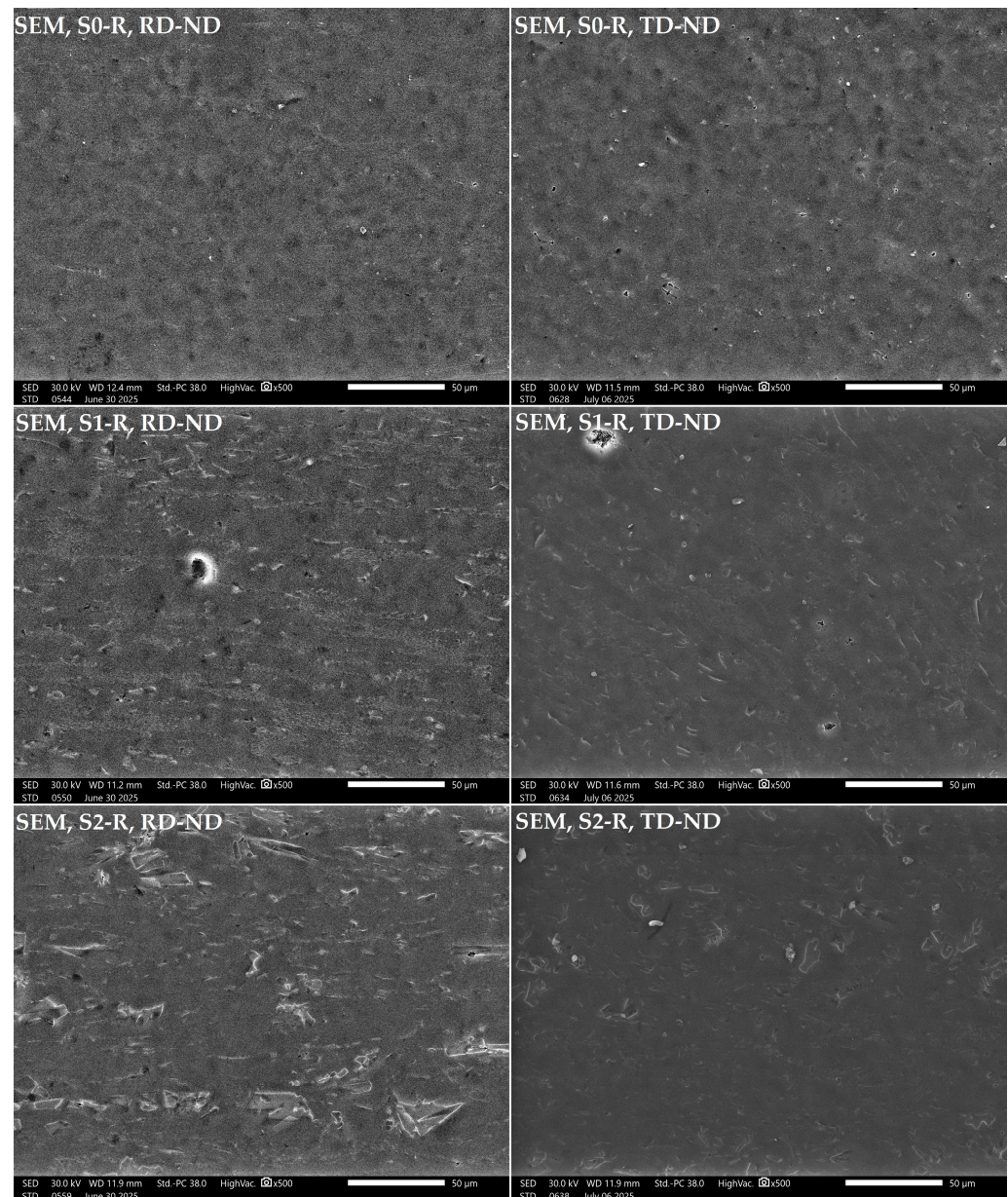


Figure 6. SEM images of samples S0-R, S1-R, and S2-R after RSR in the RD–ND and TD–ND planes.

In the S2-R sample, RD–ND images reveal a fine-grained network with TiB_2 particles uniformly dispersed and frequently associated with localized MWCNT agglomerates. The fibrous morphology of MWCNTs forms spatial bridges between aluminum grains and TiB_2 particles, visible as elongated lines in SEM images, facilitating load transfer and restricting matrix plasticity. The TD–ND plane exhibits a submicron structure with additional fine-grain fragmentation and formation of substructural cells, resulting from MWCNTs impeding dislocation motion and inducing dislocation accumulation and boundary polarization [76,77]. Overall, the uniform distribution of reinforcing phases highlights the synergistic effect of ultrasonic treatment and RSR in producing a refined and mechanically strengthened microstructure.

To gain deeper insight into the microstructural evolution induced by RSR, EBSD analyses were conducted on samples S0-R, S1-R, and S2-R in two orthogonal planes: RD–ND and TD–ND. The results are shown as Inverse Pole Figure (IPF) maps, with colors representing the orientation of individual grains relative to the ND (Figure 7).

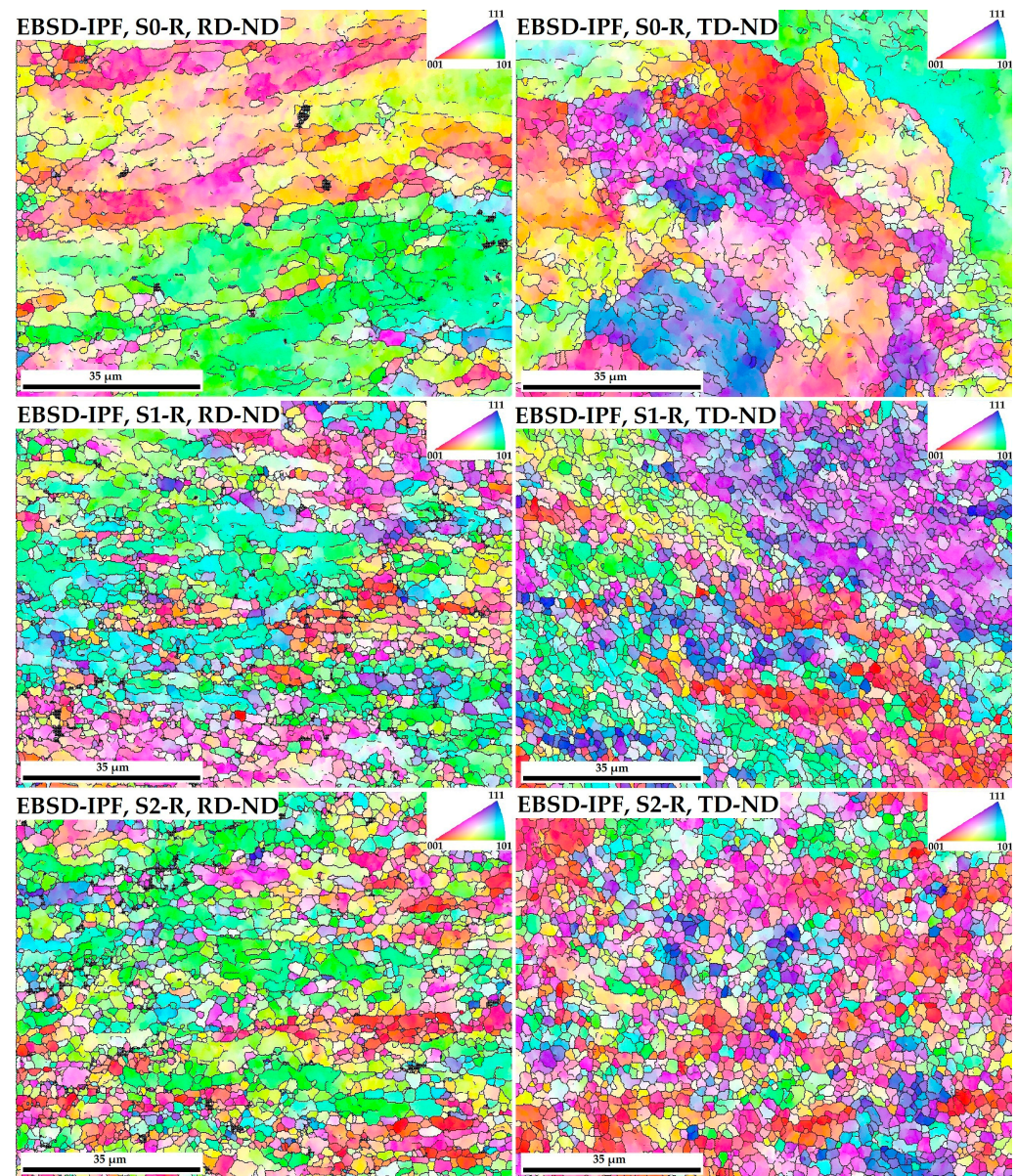


Figure 7. EBSD-IPF maps of S0-R, S1-R, and S2-R samples after RSR in RD-ND and TD-ND planes.

The unreinforced sample (S0-R) exhibited a relatively homogeneous yet coarse-grained structure, with an average grain size of 15–20 μm , most grains being elongated along the rolling direction. The IPF maps revealed a $\{001\}\langle 100 \rangle$ and $\{111\}\langle 110 \rangle$ texture, typical of aluminum alloys subjected to plastic deformation [78,79]. In the RD-ND plane, the orientation distribution was heterogeneous: some grains preserved the original as-cast texture, while others formed elongated bands with consistent crystallographic orientation. This suggests that RSR promoted active dislocation substructuring, though without substantial refinement to the submicron scale. In the TD-ND plane, a stronger anisotropy was evident, with elongated grains aligned along the rolling direction, indicating the presence of a pronounced crystallographic texture and limited recrystallization.

The incorporation of dispersed TiB_2 particles led to pronounced microstructural refinement. The average grain size decreased to 8–12 μm , and the overall structure became more fragmented. IPF maps highlighted regions of higher density, indicative of dynamic recovery and dislocation accumulation [80,81]. In the RD-ND plane, grain orientations were more homogeneous compared to S0-R, although elongated grains along the rolling direction remained. Notably, grains constrained by TiB_2 particles were observed, which

inhibited grain growth and stabilized the microstructure. In the TD–ND plane, the structure appeared even finer, with sub-5 μm grains clustered around TiB_2 particles, and a tendency toward $\{111\}\langle 110 \rangle$ texture formation was noted, a configuration known to be stable under deformation in FCC metals [82,83].

In the S2-R hybrid composite, similar trends were observed. The average grain size decreased further to 7–9 μm , reaching the submicron-crystalline regime. IPF maps revealed mosaic-like regions with uniformly colored grains, each exhibiting a unique orientation, indicating effective suppression of texture development and the formation of a quasi-isotropic structure. In the RD–ND plane, grains displayed a wide distribution of crystallographic orientations without any dominant texture, suggesting that the combined presence of TiB_2 and MWCNTs effectively counteracted the anisotropy typically observed in pure or single-component reinforced alloys. Furthermore, nascent recrystallized grains were detected near MWCNT agglomerates, pointing to localized stress redistribution.

X-ray diffraction (XRD) analysis (Figure 8) confirmed the successful incorporation of TiB_2 and MWCNT reinforcing phases into the aluminum matrix and provided insight into the phase composition of all investigated sample series.

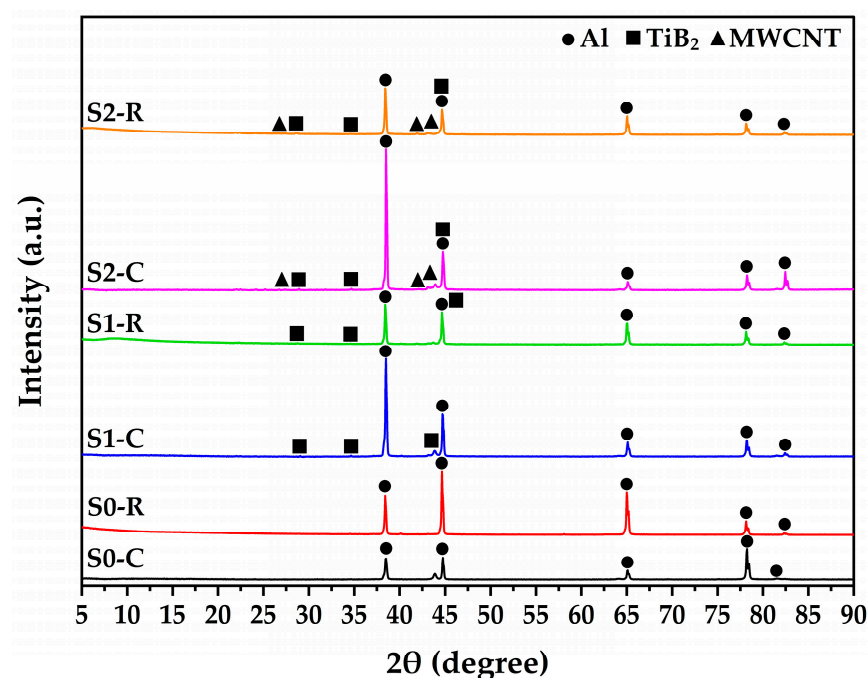


Figure 8. X-ray diffraction (XRD) patterns of the samples.

The X-ray diffraction (XRD) patterns of the base Al6060 alloy (S0-C and S0-R) display dominant aluminum reflections at $2\theta = 38.4^\circ$, 44.7° , 65.1° , and 78.2° , corresponding to the (111), (200), (220), and (311) planes of the face-centered cubic (FCC) structure. These reflections confirm the preservation of the aluminum crystal lattice following both casting and deformation. After RSR (S0-R), noticeable peak broadening occurs, particularly for the (111) and (200) reflections, indicating reduced average crystallite size and increased lattice defect density, in agreement with EBSD observations of submicron-grained microstructures.

In TiB_2 -reinforced composites (S1-C and S1-R), additional reflections appear at $2\theta \approx 27.7^\circ$ (001), 34.3° (100), 44.6° (101), and 61.3° (110), corresponding to the hexagonal lattice of titanium diboride (JCPDF card No. 35-0741). These peaks confirm the phase stability of TiB_2 under ultrasonic treatment and subsequent deformation. The increased intensity of these reflections after RSR (S1-R) suggests a more uniform particle distribution

and enhanced matrix texture, with preferential orientation of specific TiB_2 crystallographic planes along the rolling direction.

The hybrid composites containing MWCNTs (S2-C and S2-R) exhibit, in addition to aluminum and TiB_2 peaks, a weak but distinct broad peak at $2\theta \approx 26.1^\circ$, corresponding to the (002) interlayer diffraction of graphite-like carbon (JCPDS card No. 96-101-1061). This feature directly confirms the presence of MWCNTs after casting and RSR. The peak's increased full width at half maximum (FWHM) reflects the nanoscale dimensions of the carbon nanotubes and the presence of structural defects.

Following RSR (S2-R), the MWCNT peak at $2\theta \approx 26.1^\circ$ becomes less intense and shifts slightly toward higher angles ($\sim 26.2\text{--}26.3^\circ$). This behavior is attributed to increased internal stresses in the matrix, transmitted to the carbon nanotubes via interfacial interactions, partial misorientation of MWCNTs during plastic deformation, and overlapping signals from the aluminum matrix and TiB_2 . Nevertheless, the peak's persistence unambiguously demonstrates the thermal and mechanical stability of the carbon phase under high-temperature and severe plastic deformation conditions.

3.2. Mechanical Properties

Figure 9 summarizes the mechanical performance of all sample series. In the as-cast condition (S0-C), both yield strength (YS) and ultimate tensile strength (UTS) were minimal, characteristic of cast aluminum alloys without thermal or mechanical treatment.

Elongation was the highest ($\sim 20\%$), reflecting comparatively high ductility at low reinforcement levels. Following radial-shear rolling (S0-R), the strength increased markedly, with YS rising by $\sim 59.8\%$ and UTS by $\sim 40.5\%$ relative to S0-C, while ductility decreased by over 25%, highlighting the classical strength–ductility trade-off under intensive deformation [84].

The incorporation of 2 wt.% TiB_2 (S1-C) in the as-cast state led to notable strength gains: YS increased by $\sim 47.1\%$ and UTS by 31% compared to S0-C. However, ductility decreased nearly 1.6-fold, reflecting the restrictive influence of rigid ceramic particles on matrix deformability. Subsequent rolling (S1-R) further enhanced the strength, with UTS exceeding 150 MPa—almost 1.5 times higher than the as-cast alloy—while ductility remained around 9%, suitable for various structural applications.

The hybrid Al6060/ TiB_2 /MWCNTs composite exhibited even more pronounced improvements. In the as-cast state (S2-C), YS and UTS increased by 67% and 38%, respectively, relative to S0-C. Despite the presence of dual reinforcing phases, the reduction in ductility was less severe than in S1-C, suggesting a mitigating effect of MWCNTs on deformation behavior. After RSR (S2-R), YS exceeded 115 MPa and UTS reached 164 MPa, nearly 1.5–2 times higher than the unreinforced alloy, while elongation remained high ($\sim 14\%$), demonstrating an excellent balance between strength and ductility.

Microhardness measurements corroborate these trends, ranging from 50.2 HV0.2 in S0-C to 82.2 HV0.2 in S2-R. The difference of ~ 6 HV0.2 between S1-R and S2-R indicates the additional reinforcing contribution of carbon nanotubes.

Density measurements showed only minor deviations from the theoretical values, indicating low porosity ($<1\%$), which was achieved through ultrasonic treatment during melt processing. Figure 9 illustrates the sequential evolution of material properties: from the low-strength, highly ductile as-cast Al6060 (S0-C), to a stronger but less ductile deformed state (S0-R), to TiB_2 -reinforced composites (S1) and their deformed variants, and finally to the most balanced hybrid composite incorporating TiB_2 and MWCNTs after RSR (S2-R). The observed increase in mechanical performance arises from several complementary mechanisms. TiB_2 particles provide efficient load transfer and promote grain refinement during solidification, while MWCNTs act as nanoscale barriers to dislocation motion, contributing

to Orowan strengthening and enhancing ductility [85–87]. In addition, the application of ultrasonic treatment minimized porosity and ensured uniform dispersion of reinforcements, thereby preventing premature failure [37,60]. Radial-shear rolling further refined the microstructure and densified the composites, producing a synergistic strengthening–ductility balance. Collectively, these factors explain the superior mechanical properties achieved in the hybrid Al6060/TiB₂–MWCNT composites.

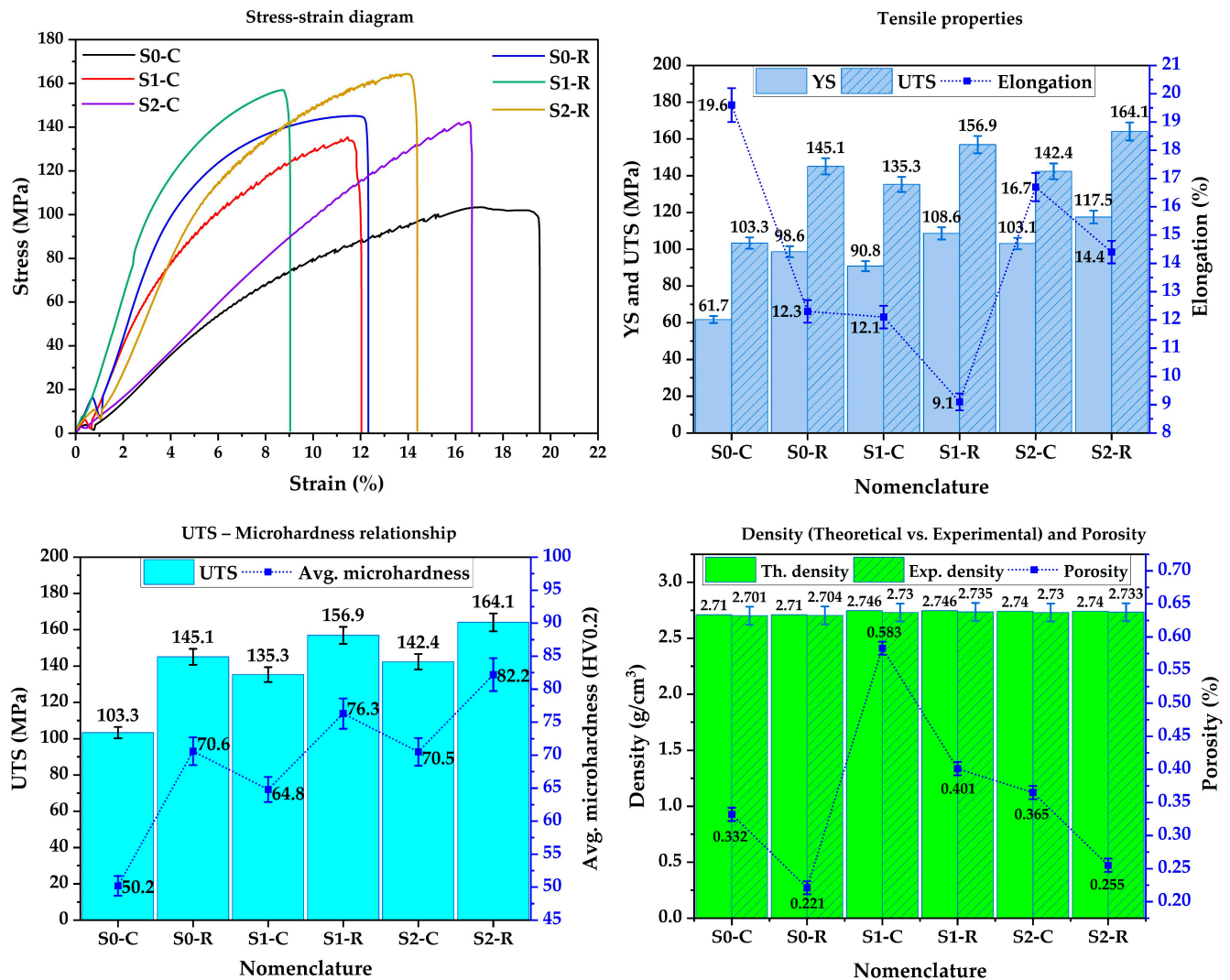


Figure 9. Comparison of the properties of the investigated samples.

4. Conclusions

Hybrid Al6060/TiB₂–MWCNT composites were successfully fabricated via ultrasonically assisted stir casting followed by radial-shear rolling, which ensured effective dispersion of reinforcement phases and minimized porosity (<1%).

TiB₂ particles acted as heterogeneous nucleation sites and grain growth inhibitors, while MWCNTs enhanced load transfer and suppressed agglomeration, resulting in a refined, homogeneous microstructure.

EBSD analysis confirmed significant grain refinement to 7–9 μm and suppression of strong deformation texture, leading to a quasi-isotropic microstructure.

The synergistic effect of dual reinforcement and severe plastic deformation provided a remarkable balance of properties: after RSR, the hybrid composite reached YS > 115 MPa, UTS ≈ 164 MPa, and elongation ≈ 14%, with microhardness up to 82.2 HV0.2.

The proposed processing route demonstrates high potential for producing lightweight structural materials with enhanced strength–ductility synergy. For example, in the aerospace sector, such composites could be applied in secondary load-bearing components where both strength and ductility are critical for safety. In transport applications, they may be suitable for lightweight suspension or chassis elements, while in the energy sector, they could enhance durability of structural parts in wind turbine housings or protective casings for energy storage systems.

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