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The Effect of carbon nanotubes on microstructure and mechanical properties of Al/TiH_2/CNT foam precursor produced via continual annealing and roll-bonding process

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A B S T R A C T

Carbon nanotube (CNT)-reinforced aluminum matrix composites offer significant potential for lightweight, high-strength applications but face challenges in achieving uniform CNT dispersion. This study investigates the microstructural and mechanical enhancements of Al/TiH_2/CNT foam precursors fabricated through eight cycles of the Continual Annealing and Roll-Bonding (CAR) process. Aluminum strips (AA1050) were combined with 0.65 wt.% multi-walled CNTs and 0.75 wt.% TiH_2 as the foaming agent. Scanning electron microscopy (SEM) and field emission SEM (FESEM) were used to analyze CNT distribution, while tensile testing and Vickers microhardness assessed mechanical properties. Results revealed improved CNT dispersion with increasing CAR cycles, though minor agglomerations persisted due to van der Waals forces. The composite exhibited a 3.87-fold increase in microhardness (88.62 Vickers in the RD–TD plane) and a 3.49-fold increase in tensile strength (171 MPa) compared to annealed pure aluminum. These enhancements stem from effective load transfer and grain refinement facilitated by the CAR process. The findings highlight the CAR process's efficacy in producing Al/TiH_2/CNT precursors with superior mechanical properties, making them promising for structural applications requiring high strength-to-weight ratios.

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1. Introduction

Metal matrix composites (MMCs) and metallic foams are two distinct classes of advanced materials with unique properties and applications. MMCs consist of a metallic matrix (e.g., aluminum, magnesium) reinforced with secondary phases, such as ceramics, to enhance mechanical properties like strength, stiffness, and wear resistance. These composites are widely used in aerospace, automotive, and structural applications where high strength-to-weight ratios are critical [1, 2]. Aluminum-based composites are particularly valued for their low density, high ductility, and excellent mechanical performance. However, achieving sufficient strength for advanced applications remains challenging despite advancements in foam manufacturing [2, 3].

Carbon nanotubes (CNTs) are promising reinforcements due to their exceptional stiffness, strength, and flexibility, with a Young's modulus of ~1 TPa and tensile strength exceeding 30 GPa [4, 5]. However, their poor wettability and tendency to agglomerate due to van der Waals forces hinder uniform dispersion in metal matrices [6, 7]. Recent studies emphasize that uniform CNT distribution is critical for maximizing composite properties, achievable through mechanical processes and CNT functionalization [8, 9]. For instance, Deng et al. [10], reported a 41.3% increase in Young's modulus (from 73 GPa to 103 GPa) in a 2024Al matrix with 1% CNTs, though agglomeration limited further gains.

Various methods, including powder metallurgy (PM), electroplating, and sputtering, are used to produce CNT/MMCs. PM methods often result in grain coarsening and the formation of brittle phases (e.g., Al_4C_3) [11, 12], which can degrade precursor properties critical for subsequent foaming. That is why the severe plastic deformation (SPD) techniques are particularly considered for producing composite. SPD techniques can refine grain structures, but achieving homogeneity requires precise control of strain and temperature. Studies demonstrate that high strain promotes stronger bonding between carbon nanotubes (CNTs) and the matrix, which enhances load transfer and improves the mechanical properties of the composite [13, 14].

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While the Continual Annealing and Roll-Bonding process (CAR) has been previously established for refining microstructure in monolithic metals and simple composites [15], the novelty of this work lies in its application to the hybrid Al/TiH_2/CNT foam precursor system. This system uniquely combines three critical challenges: (1) dispersing CNTs in an aluminum matrix prone to agglomeration, (2) preserving the structural integrity of thermally sensitive TiH_2 blowing agents during processing, and (3) balancing grain refinement (via SPD) with controlled residual stress to optimize precursor foaming behavior. Due to the accumulated strain in the conventional Accumulative Roll Bounding (ARB) process, which lacks intermediate annealing steps, there is a risk of CNTs fragmentation.

In contrast, CAR process involves elevated annealing temperatures, which may lead to the premature decomposition of TiH_2. TiH_2 begins to decompose at temperatures above 400 °C, releasing hydrogen gas. If the annealing temperature exceeds this threshold, the released hydrogen may interact with the CNTs or the matrix during the annealing stage, potentially altering interfacial bonding characteristics or promoting pore nucleation [16, 17]. By adapting CAR to this system, intermediate annealing cycles are strategically utilized to relax residual stresses, mitigate CNT damage, and stabilize TiH_2 prior to forming a synergy not previously achieved in either MMC or foam precursor fabrication. Controlled residual stress in the precursor ensures uniform pore nucleation during foaming, as excessive stress can lead to localized pore collapse or density gradients [18].

2. Materials and methods

The study utilized aluminum strips Aluminum strips (AA1050, Arak Aluminum, Iran) (5×15 cm, 1 mm thick) as the matrix, titanium hydride powder (TiH_2) (Merck, Germany) as the foaming agent, and Multi Walled Carbon Nanotubes (MWCNT) (US Research Nanomaterials, Inc.) as the reinforcing agent.

The characteristics of TiH_2 and MWCNT are presented in Tables 1 and 2.

Table 1

Characteristics of (TiH_2) titanium hydride powder.

Particle Size (μm)	Melting Temperature (°C)	Density (g/cm^3)
<40	Up to 400	3.91

Table 2

Specifications of reinforcing MWCNT.

Specific Surface Area (m^2/g)	Length (μm)	Diameter (nm)
270	10	10-30

2.1. Fabrication of Al/TiH_2/CNT

Initially, five aluminum strips were fully annealed at 350 °C for one hour. Following annealing, their surfaces were degreased using acetone (Sigma-Aldrich, Germany) and mechanically roughened using a wire brush with 3 mm diameter bristles. To disperse MWCNTs, 0.65 wt.% relative to the total weight of the five aluminum strips were ultrasonically dispersed in acetone, for 30 minutes using an ultrasonic homogenizer (BENDELINE, Germany; model: 3200) and then sprayed evenly onto the strip surfaces using a spray bottle.

The rapid evaporation of acetone facilitated a temporary uniform distribution of the CNTs on the metal surfaces. After applying MWCNTs, 0.75 wt.% of TiH_2 foaming agent, relative to the total weight of the five aluminum strips, was evenly distributed using a 325-mesh U.S. standard sieve. The treated strips were then stacked and subjected to rolling, reducing the overall thickness from 5.5 mm to 2 mm (a 64% reduction), forming the precursor corresponding to cycle zero.

In the subsequent processing steps, the roll-bonded laminate was sectioned into halves, followed by degreasing and mechanical surface treatment via wire brushing to remove contaminants and activate the surfaces. The cleaned strips were then restacked and subjected to further roll bonding with a 50% thickness reduction per cycle, without the addition of additional powders. After each rolling cycle, the bonded laminates underwent inter-cycle annealing at 240 °C for 30 minutes. This annealing step facilitates atomic diffusion across interfaces, promoting diffusion bonding and thereby enhancing metallurgical bonding and interfacial strength between the layers [19, 20].

The selection of eight CAR cycles was based on optimizing CNT dispersion and mechanical properties while balancing processing efficiency and material integrity. Previous studies on SPD processes, such as ARB, indicate that increasing cycle numbers enhances particle dispersion by breaking down agglomerates through cumulative strain [19, 21].

In this study, preliminary trials showed that MWCNT agglomerates significantly reduced in size and number between cycles 4 and 6, with near-optimal dispersion achieved by cycle 8. The annealing step (240 °C for 30 minutes) after each cycle mitigated residual stresses and preserved MWCNT integrity, allowing eight cycles to achieve a uniform microstructure without compromising the foaming agent's efficiency [15]. This cycle number was thus selected to maximize MWCNT distribution, interlayer bonding, and mechanical enhancements while maintaining process scalability.

2.2. Characterization techniques

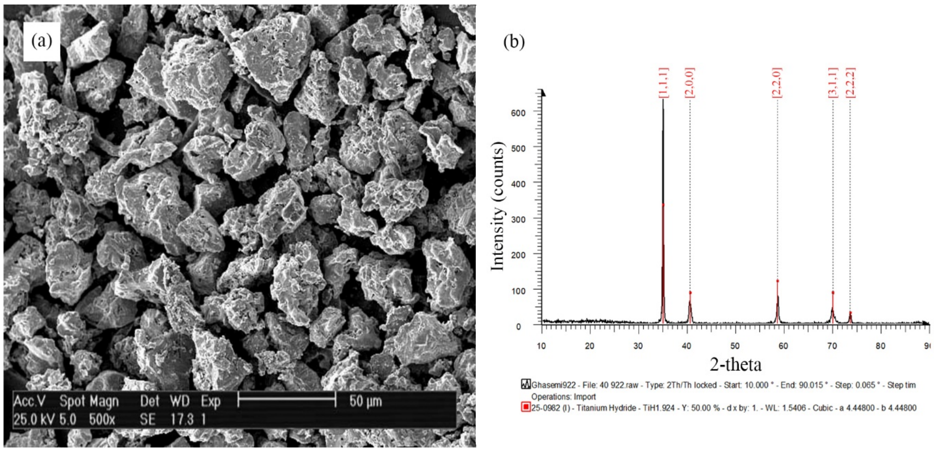
The distribution of the foaming agent and MWCNT within the aluminum matrix of the fabricated precursors was examined using scanning electron microscopy (SEM; Philips XL30) and field emission scanning electron microscopy (FESEM; Tescan Mira 3-XMU). To evaluate mechanical performance, tensile strength measurements were conducted on specimens with a gauge length of 25 mm and a width of 6 mm, in accordance with ASTM E8 standards. Vickers microhardness testing was performed on the RD–TD and ND–TD planes under a load of 50 g and a dwell time of 10 seconds. Hardness values were obtained from ten randomly selected points on each sample to ensure statistical relevance. Additionally, to monitor the precursor fabrication process, the thermal behavior of the foaming agent was analyzed by differential thermal analysis (DTA) in air, with a heating rate of 10 °C/min from room temperature up to 1000 °C.

3. Results and discussion

3.1. Characteristics of the micro-sized and nano-sized powders

SEM image and XRD analysis of TiH_2 foaming agent is shown in Fig. 1. Fig. 1a shows TiH_2 particles (<40 μm), with XRD (Fig. 1b) verifying the $TiH_{1.924}$ phase (card number 982-25). Fig. 2 illustrates MWCNTs with diameters of 10–30 nm and lengths of ~10 μm.

It is well established that the incorporation of CNTs into metal matrix composites can enhance mechanical properties, provided a strong interface is formed between the matrix and the nanotubes. However, the inherently poor wettability of CNTs with aluminum presents a significant challenge. To enhance wettability, MWCNTs were functionalized with 3 M HNO_3, introducing surface functional groups that improve interfacial bonding with the aluminum matrix, as supported by Malaki et al. [22, 23].



(a) (b)

Fig. 1. (a) SEM image of TiH_2 particles (<40 μm), Scale bar: 50 μm; Magnification: 500x (b) XRD analysis of TiH_2.

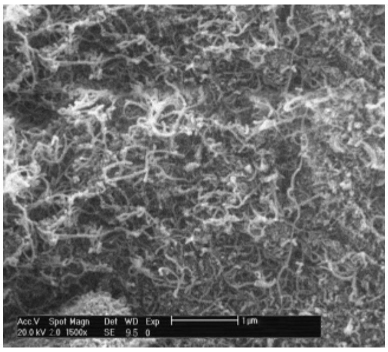


Fig. 2. SEM image of MWCNT, Scale bar: 1 μm; Magnification: 1,500x.

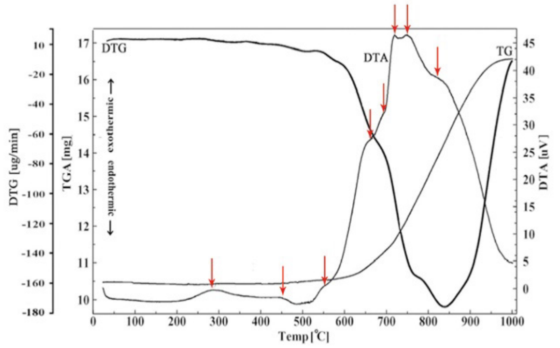


Fig. 3. TG, STA, DTA, and DTG charts of TiH_2 in air (10 °C/min, ambient to 1000 °C).

Specifically, MWCNT were immersed in a 3 M HNO_3 solution (Merck, Germany) and refluxed at 90 °C overnight to introduce functional groups onto their surfaces. The treated MWCNT were then rinsed thoroughly with distilled water (local, Iran) using a 0.2 μm polycarbonate membrane filter to remove residual acid. Subsequently, the functionalized MWCNT were dried in an oven at 110 °C for one hour [24, 25].

In addition, due to strong van der Waals forces, CNTs have a natural tendency to agglomerate, which hinders their effective dispersion and the transfer of their exceptional properties to the matrix.

To mitigate this issue, the CNTs were ultrasonically dispersed in acetone for 30 minutes and then sprayed uniformly onto the aluminum strips prior to stacking and rolling [26].

It is important to note that excessive annealing time or temperature can adversely affect the performance of the foaming agent. Specifically, prolonged or high-temperature annealing may allow the foaming agent to prematurely reach the initial stage of gas release, thereby reducing its effectiveness during the subsequent foaming process.

Differential thermal analysis (DTA, Fig. 3) revealed TiH_2's hydrogen release onset at ~290 °C, guiding the selection of a 240 °C annealing temperature to preserve foaming efficiency, unlike higher-temperature processes that risk premature gas release [17]. Compared to Sun et al. [26], who used similar dispersion techniques for CNTs in organic solvents, our approach achieved comparable uniformity but avoided high-temperature processing that could degrade TiH_2.

This balance is critical for foam precursor applications, as premature decomposition could compromise pore formation, a challenge not addressed in traditional CNT composite studies.

3.2. Characterization of foamable precursors

3.2.1. Micro structure

Fig. 4 presents SEM images of the RD–TD cross-sections of the Al/TiH_2/CNT composite precursor after peeling, corresponding to cycles 4, 6, and 8 of the CAR process. Owing to the high aspect ratio, low density, and strong van der Waals interactions between MWCNT, achieving uniform dispersion is inherently challenging. Nonetheless, in this study, a relatively favorable distribution of MWCNT within the aluminum matrix was observed.

As illustrated in the Fig. 4, with progressive CAR cycles, both the dispersion and surface concentration of reinforcement particles and foaming agent improved. Additionally, the particles increasingly aligned along the rolling direction. In earlier cycles, MWCNT were present as large agglomerates; however, after eight cycles, these clusters were broken down into smaller agglomerates. Despite the improvement, van der Waals forces and surface tension continued to promote the formation of elongated clusters.

Tajzad et al. [21] reported that the internal pressure within CNT agglomerates facilitates the infiltration of aluminum into the clusters. Therefore, at higher CAR cycles, increased internal pressure combined with severe plastic deformation enhances matrix penetration, contributing to a more homogeneous distribution of reinforcement particles. Furthermore, intense plastic strain facilitates the flow of aluminum into the nanoporous regions within MWCNT clusters, promoting a more uniform reinforcement dispersion throughout the matrix. However, minor agglomerations persisted due to van der Waals forces, consistent with Nie et al. [6], who reported similar challenges in CNT-reinforced aluminum composites.

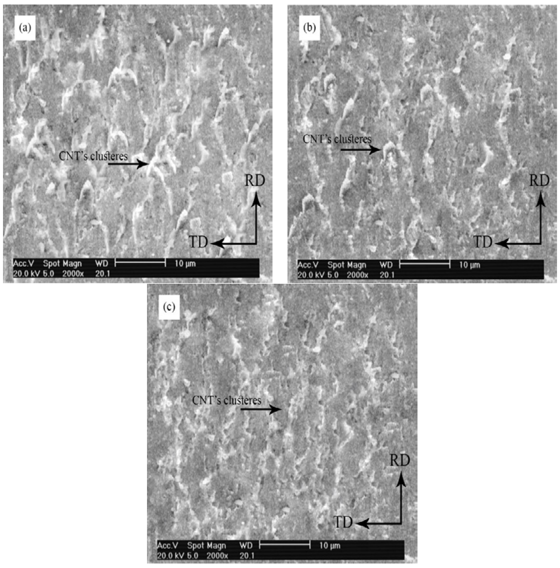


Fig. 4. SEM images of RD-TD cross-section of Al/TiH_2/CNT precursors in cycles (a) 4, (b) 6, and (c) 8.

Fig.5 displays the SEM image of the RD–TD cross-section of the composite sample after peeling, corresponding to cycle 8 of the CAR process. As shown, MWCNT reinforcement particles are predominantly aligned along the rolling direction in the form of clusters. Notably, fractured titanium hydride (TiH_2) particles are observed to be entrapped within these MWCNT clusters.

The image also highlights protruded ends of MWCNT that are deeply embedded in the aluminum matrix, indicating the formation of strong interfacial bonding between the reinforcement phase and the matrix. This interfacial integrity contributes to more effective load transfer and enhanced mechanical reinforcement by the MWCNT.

However, the presence of some cracks and fractures within the MWCNT particularly those not fully embedded in the matrix suggests that localized stress concentrations and compressive forces during the rolling process may have contributed to partial MWCNT damage.

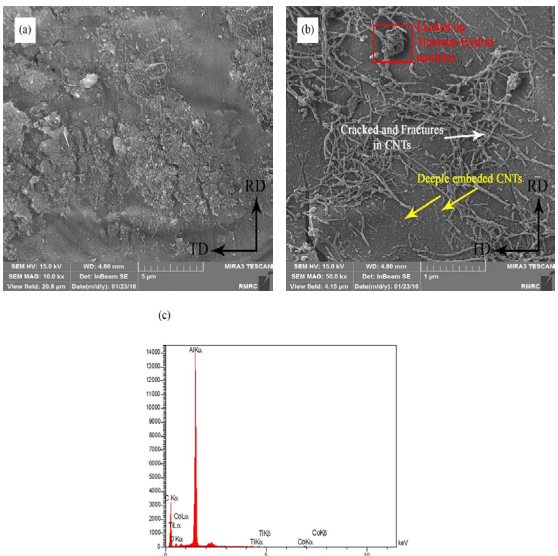


Fig. 5. FESEM images (a , b) and EDS analysis of RD-TD cross-section in cycle 8 (c).

Fig. 6 presents SEM images of the RD–ND cross-sections of the Al/TiH_2/CNT composite samples after 4, 6, and 8 cycles of the CAR process. As the number of cycles increases, a more uniform distribution of reinforcement particles throughout the composite volume becomes evident. However, due to persistent van der Waals interactions between MWCNT, some agglomerates still remain, albeit smaller and more scattered compared to earlier cycles. Several mechanisms have been proposed to explain the disruption of the initial layer structure and the progressive dispersion of particles within the metallic matrix. According to Jma'ati et al. [19], during the initial rolling cycle, powder layers are fragmented into smaller segments, and aluminum from the matrix is extruded into the interparticle spaces. With additional cycles, the flow of the aluminum matrix increasingly infiltrates these fragmented regions, generating shear flows and intensifying plastic deformation. As strain accumulates, the particle morphology becomes increasingly elongated, and the reinforcement particles become more homogeneously distributed.

Furthermore, the destruction of MWCNT agglomerates and their transformation into extended clusters is facilitated by shear-induced twisting and alignment along the direction of applied strain. These elongated clusters, driven by surface energy and van der Waals forces, tend to orient parallel to the rolling direction, contributing to improved dispersion but also revealing the intrinsic challenge of completely eliminating agglomeration.

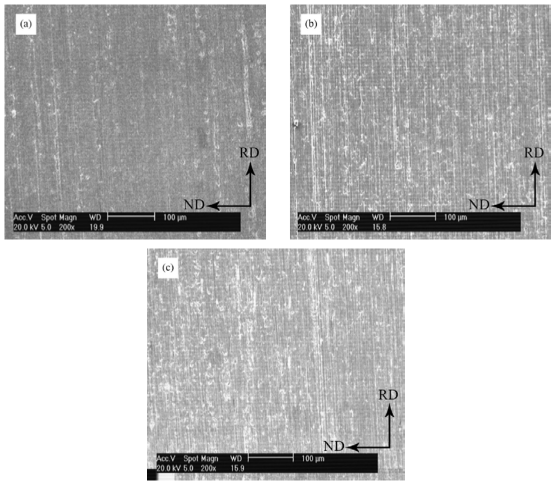


Fig. 6. SEM images of RD-ND cross-section in cycles (a) 4, (b) 6, and (c) 8.

In contrast to Deng et al. [10], who observed significant CNT agglomeration in a 2024Al matrix processed via powder metallurgy (PM), our CAR process achieved better dispersion through iterative rolling and annealing. The intermediate annealing at 240 °C mitigated residual stresses and CNT damage, unlike accumulative roll bonding (ARB), which risks CNT fragmentation due to high strain [14]. The strong interfacial bonding observed in Fig. 5, with MWCNTs embedded in the matrix, facilitates effective load transfer, a key advantage over PM methods that often form brittle Al_4C_3 phases [11]. This microstructure underscores the CAR process's suitability for producing uniform Al/TiH_2/CNT precursors, advancing foam composite fabrication for high-strength applications.

3.2.2. Mechanical properties

In this section, the mechanical properties of the Al/TiH_2/CNT nanocomposite are evaluated, with a focus on two key aspects: microhardness and tensile strength.

3.2.2.1. Hardness of the roll bonded Al/TiH$_2$/CNT composite

Fig. 7 illustrates the average Vickers microhardness values measured at 10 randomly selected points in the RD–TD and ND–TD sections of the Al/TiH$_2$/CNT composite precursors. The indentations were aligned along the transverse direction (TD), with an approximate spacing of 50 μm. In cycle 8 of the CAR process, the microhardness in the RD–TD section reached 88.62 Vickers representing a 3.87-fold increase compared to annealed pure aluminum (23.4 Vickers). Similarly, the microhardness in the ND TD section reached 69.51 Vickers, indicating a 2.88-fold improvement over pure aluminum. This anisotropy reflects the preferred orientation of MWCNTs along the rolling direction, as confirmed by SEM. These significant increases underscore the reinforcing effect of MWCNT within the aluminum matrix. A more uniform dispersion of MWCNT contributes to consistent hardening across the matrix, as regions in proximity to MWCNT exhibit higher hardness due to effective load transfer. Conversely, non-uniform distribution can lead to localized variations in hardness. SEM analysis confirms that MWCNT are relatively well-dispersed within the matrix in later cycles, which supports the observed enhancements in hardness.

Furthermore, despite the small addition of only 0.65 wt.% MWCNT, a dramatic increase in microhardness was achieved, highlighting the efficacy of MWCNT reinforcement. Comparative analysis of CNT-reinforced and non-reinforced precursors clearly demonstrates that the incorporation of MWCNT significantly enhances the hardness of the composite. The uniform MWCNT dispersion, achieved through CAR's iterative deformation, contrasts with the localized hardness variations reported in ARB-processed composites by Najjar et al. [27].

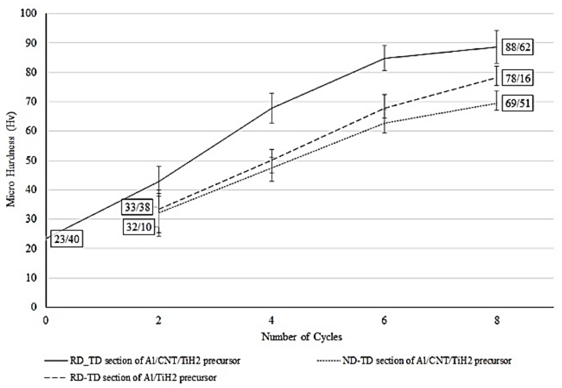


Fig. 7. Microhardness of RD-TD and ND-TD sections of Al/TiH$_2$/CNT and Al/TiH$_2$ precursors in cycles 4, 6, and 8.

3.2.2.2. Tensile strength of the roll bonded Al/TiH$_2$/CNT composites

Fig. 8 presents the engineering stress–strain curves for annealed pure aluminum, Al/TiH$_2$, and Al/TiH$_2$/CNT composite precursors after eight cycles of the CAR process. As shown, both reinforced precursors exhibit significant improvements in tensile strength compared to annealed pure aluminum.

The Al/TiH$_2$ composite precursor demonstrates a tensile strength of 122 MPa, representing an approximate 2.49-fold increase over annealed aluminum. In contrast, the Al/TiH$_2$/CNT composite precursor achieves a tensile strength of 171 MPa equivalent to a 3.49-fold enhancement. This substantial improvement in mechanical performance is attributed to the synergistic effect of the uniformly dispersed carbon nanotubes and TiH$_2$ particles, which contribute to load transfer, dislocation pinning, and matrix strengthening during deformation. Compared to Deng et al. [10], who reported a 41.3% increase in Young's modulus with 1% CNTs in a 2024Al matrix, our 3.49-fold tensile strength gain is notably higher, likely due to CAR's superior dispersion and interfacial bonding.

These results clearly highlight the reinforcing capability of MWCNT, even at low concentrations, and the effectiveness of the CAR process in enhancing the tensile properties of the aluminum-based composite system.

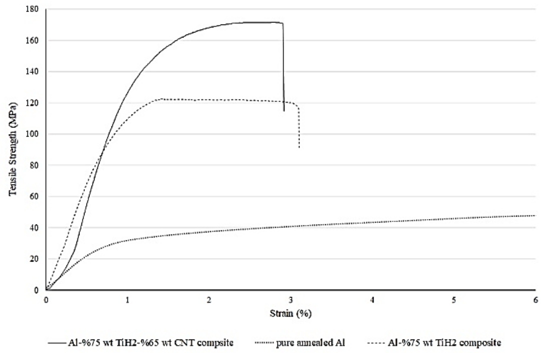


Fig. 8. Engineering stress–strain curves of annealed aluminum, Al/TiH$_2$, and Al/TiH$_2$/CNT precursors after cycle 8.

In general, the strength enhancement in CAR and ARB processed composites arises from two primary factors: (1) the improved bonding strength between interlayers, and (2) the inherent hardness of the reinforcing particles [27]. According to the load transfer strengthening mechanism, the extent of strength improvement depends on the modulus, volume fraction, and aspect ratio of the reinforcement particles. Due to the high modulus and large aspect ratio of CNTs, efficient load transfer occurs predominantly along the CNTs axis during mechanical loading. The Orowan strengthening mechanism, where MWCNTs pin dislocations, significantly enhances strength, as supported by Song et al. [28]. Moreover, the loading direction plays a critical role in determining the reinforcing efficiency of MWCNT. In composites exhibiting preferred orientation of MWCNT such as those aligned along the rolling direction tensile strength is significantly enhanced parallel to the MWCNT axis, which correlates with the observed anisotropy in hardness results.

Three interfacial interaction types are typically considered in Al/CNT composites: (I) adhesion interface, (II) friction interface, and (III) cohesive interface [29]. SEM and FESEM observations confirm that MWCNT are coherently embedded within the aluminum matrix, indicating a dominant adhesion interface that facilitates effective stress transfer from the matrix to the reinforcement particles. Given the superior Young's modulus and mechanical strength of MWCNT, this strong interfacial bonding leads to a substantial increase in composite strength compared to the unreinforced matrix. Fig. 9 illustrates the tensile strength values of Al/TiH$_2$/CNT and Al/TiH$_2$ precursors across different CAR cycles. As the CAR process progresses, the precursor strength increases, primarily due to two factors: grain refinement and strain hardening from dislocation accumulation, along with the reinforcing effect of particles. In the initial cycles, strain hardening dominates, while grain refinement becomes more influential with increasing cycle number. This is attributed to finer grain structures and more uniform reinforcement particle distribution within the matrix. A more homogeneous particle dispersion reduces interparticle spacing, thereby impeding crack propagation and enhancing strength. Additionally, improved bonding between the matrix and reinforcement facilitates efficient load transfer, further increasing mechanical performance. However, the impact of grain refinement diminishes in later cycles due to intermediate annealing steps. Since annealing occurs at temperatures lower than aluminum's recovery threshold, partial recovery likely takes place at the surface, moderating strength gains in higher cycles.

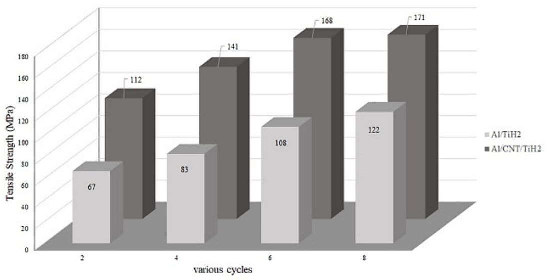


Fig. 9. Tensile strength values in various cycles of CAR precursor of $Al/TiH_2/CNT$ and Al/TiH_2.

The elongation of the precursors decreased compared to annealed pure aluminum. During the ARB process, three-dimensional stresses and residual plastic strains arise from the mismatch in thermal expansion coefficients between the reinforcement particles and matrix during cooling to room temperature.

These stresses induce microcracks at the particle-matrix interfaces, making elongation sensitive to the distribution of MWCNT. MWCNT clusters generate microvoids that reduce ductility, while well-dispersed MWCNT hinder dislocation motion, also lowering ductility. In this production method, MWCNT clustering diminishes at higher cycles, but dispersed MWCNT continue to contribute to ductility reduction. However, elongation decreased to 2.89% for $Al/TiH_2/CNT$ and 3.09% for Al/TiH_2, attributed to micro-cracks at MWCNT-matrix interfaces and dislocation pinning, as noted by Shi et al. [29]. This buckling behavior allows the composite to sustain higher stress levels before failure; however, the associated formation of microcracks limits elongation, preventing significant ductility improvement.

Comparing the strength of the $Al/TiH_2/CNT$ precursor with the Al/TiH_2 precursor, the significantly reduced interparticle spacing in the reinforced composite leads to a substantial increase in tensile strength. This enhancement primarily arises from two key factors: grain refinement and strain hardening, both driven by the accumulation of dislocations and the intrinsic characteristics of the reinforcement particles. Unlike ARB-processed composites, where high strain reduces ductility further [14], CAR's annealing steps moderated residual stresses, preserving some ductility. These results position the CAR process as a superior method for producing high-strength, low-density foam precursors, with potential applications in aerospace and automotive industries, where uniform reinforcement and controlled foaming are critical.

4. Conclusion

This study aimed to investigate the effect of MWCNT distribution on the microstructure and mechanical properties of $Al/TiH_2/CNT$ foam precursors produced via the CAR process. The CAR process's ability to balance CNT dispersion, TiH_2 stability, and grain refinement addresses key limitations of PM and ARB methods, such as grain coarsening and CNT damage. SEM analysis confirmed that eight CAR cycles achieved relatively uniform MWCNT distribution, with particles aligned in the rolling direction and crushed TiH_2 particles embedded in MWCNT clusters. By achieving a 3.87-fold hardness increase and 3.49-fold tensile strength improvement with only 0.65 wt.% MWCNT, this study demonstrates a scalable approach for fabricating $Al/TiH_2/CNT$ foam precursors. These findings advance the field of metal matrix composites by offering a robust method for producing lightweight, high-strength materials, with implications for structural applications requiring high strength-to-weight ratios. These findings advance the field of metal matrix composites by offering a robust method for producing lightweight, high-strength materials, with implications for structural applications requiring high strength-to-weight ratios.

Author contributions

Maryam Qasemi: Data Curation, Resources, Writing—Original Draft Preparation, Writing—Review and Editing.
Ali Habibolahzadeh: Investigation, Data analysis and interpretation, Writing—Original Draft Preparation.

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Conflict of interest

The authors declare no conflict of interest.

Data availability

The datasets generated and analyzed during the current study are available from the corresponding author upon reasonable request.

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