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Reinforcement of Ultra-High-Temperature Zirconium Diboride Composites with Silicon Carbide Nanowires for Aerospace Applications

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Abstract
The development of ultra-high-temperature ceramics (UHTCs) is critical for hypersonic and aerospace systems. This work presents the fabrication and characterization of ZrB_2–SiC nanowire composites with improved thermal stability and oxidation resistance. Hot pressing and spark plasma sintering were used to achieve dense microstructures with controlled nanowire dispersion. Mechanical testing at 1800°C demonstrates enhanced flexural strength and thermal shock resistance, attributed to crack deflection and load transfer mechanisms. These composites show promise for reusable thermal protection systems and high-enthalpy propulsion components.

Keywords: Zirconium diboride; Silicon carbide; Hypersonic systems; Nanomaterials
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1. Introduction

Nanowires are shifting the boundaries of materials science primarily because their one-dimensional structure unlocks behaviors (physical, chemical, and mechanical) that simply aren't possible in bulk materials [1,2]. Their defining feature is an extreme aspect ratio; by maximizing surface area relative to volume, these wires can accelerate chemical reactions and heat dissipation far beyond standard limits [3]. Mechanically, they are nearly peerless, with tensile strengths that often hit the material theoretical maximum. This makes them a go-to choice for reinforcing ceramic or metal matrices where high strength is required without the penalty of extra weight [4,5].

In the aerospace sector, silicon carbide (SiC) nanowires are now critical for the thermal shields used on supersonic craft. They function as a microscopic reinforcement layer, effectively "pinning" cracks to prevent them from spreading. This boost in fracture toughness is what allows a spacecraft to survive the brutal thermal and mechanical loads of atmospheric reentry [6-8]. Beyond structural use, their sensitivity makes them ideal for space-grade sensors, capable of detecting trace gases or radiation levels with almost instant response times [9,10].

More broadly, nanowires are the engines behind smaller, faster transistors in nanoelectronics and higher-efficiency cells in the renewable energy sector [11]. As we get better at controlling their growth patterns, we move closer to a new era of flexible, wearable tech. Ultimately, nanowires provide the foundation for a generation of "smart" materials that refuse to compromise between lightness and durability [12-14].

Zirconium diboride (ZrB2) is a central material in the push for next-generation aerospace and defense systems. As an ultra-high-temperature ceramic (UHTC) with a melting point north of 3000°C, it is one of the few viable candidates for the leading edges of hypersonic vehicles and rocket nozzles [15,16]. Its value comes from a rare overlap of metal-like thermal and electrical conductivity paired with high hardness and chemical stability under oxidation [17]. Despite these strengths, ZrB_2 is hindered by low fracture toughness and a high susceptibility to thermal shock-vulnerabilities that risk catastrophic failure during the intense mechanical and thermal loading of atmospheric reentry [18,19]. This study addresses these gaps by engineering a hybrid microstructure that pairs the inherent stability of the ZrB_2 matrix with nano-reinforcement to ensure reliability in extreme environments.

The core of this strategy is the integration of silicon carbide (SiC) nanowires as reinforcing agents. While SiC is typically added to improve oxidation resistance via the formation of a protective silica layer, using it in nanowire form introduces a significant mechanical advantage [20]. Because of their high aspect ratio, these nanowires function as microscopic bridges across crack tips. By forcing crack deflection and consuming energy through "nanowire pull-out," they prevent brittle fracture propagation.

This transition in the failure mode of the material is essential for maintaining structural integrity under the dynamic stresses encountered throughout different flight phases [21,22].

However, the performance of these composites is highly sensitive to processing conditions. This research investigates how reinforcement ratios and nanowire distribution affect the "thermo-mechanical synergy" of the final part [23]. A major hurdle is preserving the matrix's high thermal conductivity, necessary for shedding aerodynamic heat, while protecting the delicate nanowires from degrading during high-pressure sintering, such as hot pressing or Spark Plasma Sintering (SPS) [24].

This work aims to map the relationship between nanoscale architecture and macro-scale performance under simulated long-duration flight. By bridging this gap, we hope to advance a new class of resilient materials capable of meeting the rigorous mechanical demands of outer space and high-speed transit.

2. Experimental Part

The fabrication of the ZrB_2-SiC nanowire composites followed a multi-stage process designed to ensure a uniform distribution of the reinforcement phase within the ceramic matrix. Initially, high-purity ZrB_2 powder was blended with SiC nanowires at specific weight percentages. To mitigate the risk of oxidation and ensure a consistent mix, high-energy ball milling was performed in a liquid ethanol medium. This milling phase was maintained for several hours; the goal was to achieve a homogeneous dispersion and break up any nanowire agglomerates, which could otherwise act as stress concentrators and weaken the final structure. Following the mixing stage, the slurry was vacuum-dried to remove the solvent, yielding a refined hybrid powder ready for consolidation. Figure (1) shows a block diagram of the experimental methodology for ZrB_2-SiC nanowire composite fabrication.

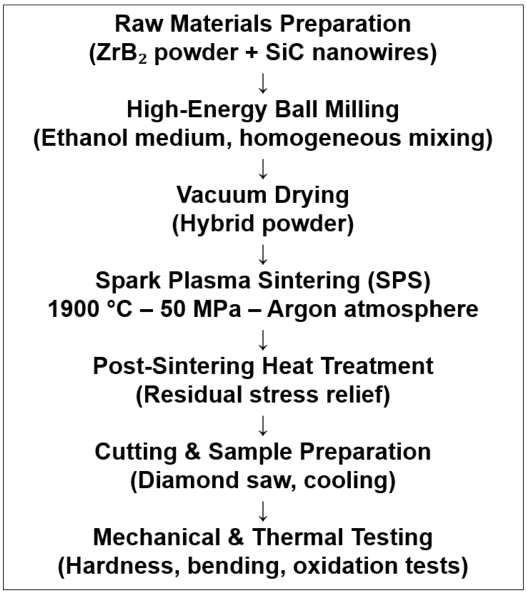

Fig. (1) Block diagram of the experimental methodology for ZrB_2–SiC nanowire composite fabrication

Consolidation was carried out via spark plasma sintering (SPS), utilizing high-strength graphite dies. The hybrid powder was processed at 1900°C under a uniaxial pressure of 50 MPa within an inert argon atmosphere. SPS was selected specifically to achieve near-theoretical density (approximately 99%) while minimizing the thermal window; this rapid densification is vital to preserve the SiC nanowires' morphology and suppress grain growth within the ZrB_2 matrix. Post-sintering, the samples underwent heat treatment to mitigate residual thermal stresses before being precision-machined into test specimens using a diamond-wafering saw. The final phase involved a comprehensive characterization of the composite s mechanical and thermal limits. Mechanical properties were quantified through Vickers indentation and four-point bending tests, determining fracture toughness and elastic modulus from room temperature up to 1500°C to simulate hypersonic flight conditions. To assess environmental durability, oxidation resistance was evaluated using oxy-acetylene torch testing. This allowed for a direct observation of the protective silica (SiO_2) layer formation, providing the necessary data to correlate sintering parameters with real-world performance in aerospace environments.

3. Results and Discussion

Figure (2) plots the correlation between SiC nanowire volume fraction and the resulting fracture toughness and Vickers hardness of the ZrB_2 composites. The data indicates a clear, progressive enhancement in both properties as the nanowire content increases from 0 to 30 vol.%. The baseline sample (0 vol.% SiC) reflects the typical brittle behavior of monolithic ZrB_2, with a fracture toughness of roughly 3.2 MPa\m$^{1/2}$ and a hardness of 15 GPa. Upon introducing 10 vol.% SiC nanowires, these values climb to 4.5 MPa\m$^{1/2}$ and 17.5 GPa, respectively. This initial jump suggests the activation of toughening mechanisms, specifically crack deflection and bridging, driven by the high aspect ratio of the nanowire reinforcements.

As the content reaches 20 vol.%, the performance gains become more pronounced; toughness exceeds 6 MPa/m$^{1/2}$ while hardness reaches the 20-21 GPa range. At this concentration, the more uniform distribution of the nanowires within the ceramic matrix optimizes load transfer. The energy dissipation observed here is largely due to nanowire pull-out and the arrest of micro-cracks, which significantly hampers crack propagation. At the maximum loading of 30 vol.%, fracture toughness peaks at approximately 7.2 MPa/m$^{1/2}$ and hardness at 22-23 GPa. This simultaneous increase in both toughness and hardness is a notable result, as these properties are typically inversely related in standard ceramics. The synergy at 30 vol.% suggests the formation of a semi-continuous reinforcement network that effectively restricts localized deformation and prevents the rapid coalescence of micro-cracks [25].

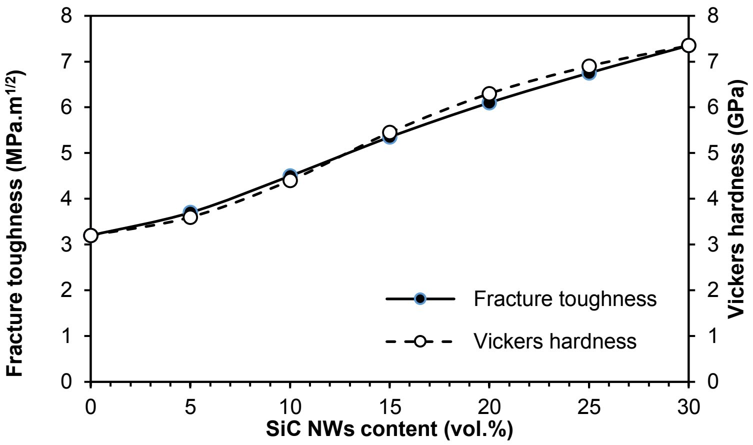

Fig. (2) A comparison between the pure ZrB_2 matrix and the composite after adding different proportions of nanowires

The data in Figure (2) confirms that SiC nanowire reinforcement is a viable strategy for bypassing the traditional trade-offs in ceramic engineering. Typically, hardness and toughness are mutually exclusive – increasing one often compromises the other. However, these composites demonstrate a simultaneous upward trend in both metrics. This dual enhancement is critical for the aerospace sector, specifically for ultra-high-temperature environments. In these contexts, a material needs high hardness to resist erosive wear and high fracture toughness to survive the thermal shock and mechanical stresses of high-velocity flight. The ability of the nanowire network to bolster both properties suggests that these ZrB_2 composites are more than just laboratory curiosities; they are high-performance candidates for the next generation of thermal protection systems (TPS) and structural components in hypersonic vehicles.

The temperature-dependent flexural strength of both the monolithic ZrB_2-SiC composite and the SiC nanowire-reinforced variant is compared in Figure (3), spanning from ambient conditions up to 1800°C. These results highlight how the nanowire architecture mitigates the typical strength degradation seen in ceramics under the extreme thermal loads found in aerospace environments. At room temperature, the nanowire-reinforced composite achieves a flexural strength of approximately 650 MPa, a significant 44% increase over the 450 MPa recorded for the conventional composite. This baseline advantage stems from a more refined microstructure and superior load distribution between the matrix and the reinforcement. The enhanced strength is likely the result of lower residual porosity and stronger interfacial bonding, which allow the nanowires to effectively pin micro-cracks during the early stages of mechanical loading [26].

As the temperature approaches 1000°C, both materials begin to show a reduction in flexural strength, though the nano-reinforced composite proves much more resilient. It retains approximately 620 MPa, while the strength of conventional composite dips to 420 MPa. This stability is largely due to the ability of nanowires to counteract thermal softening within the ZrB_2 matrix. By maintaining the structural integrity of the grain boundaries and interfaces, the SiC network prevents the premature crack expansion that typically occurs as the material's internal bonds begin to weaken under heat. The performance gap widens significantly in the 1200-1500°C range. Here, the monolithic material experiences a precipitous drop in strength, falling to 250 MPa at 1500°C. In contrast, the reinforced composite maintains a robust 480 MPa. At these temperatures, the nanowires serve as mechanical stabilizers that inhibit grain rearrangement and the creep of structural defects, phenomena that usually lead to the rapid degradation of ceramics. Furthermore, the nanowire distribution helps diffuse thermal stresses, preventing the high-stress concentrations at crack tips that would otherwise lead to failure. Even at the 1800°C limit, where thermal effects are most severe, the SiC nanowire-reinforced composite retains roughly 390 MPa – nearly 60% of its room-temperature strength. This stands in stark contrast to the conventional composite, which collapses to about 110 MPa. This superior retention of mechanical properties at ultra-high temperatures is a defining characteristic for materials intended for high-heat-flux environments, where the ability to withstand mechanical load under thermal saturation is non-negotiable [27].

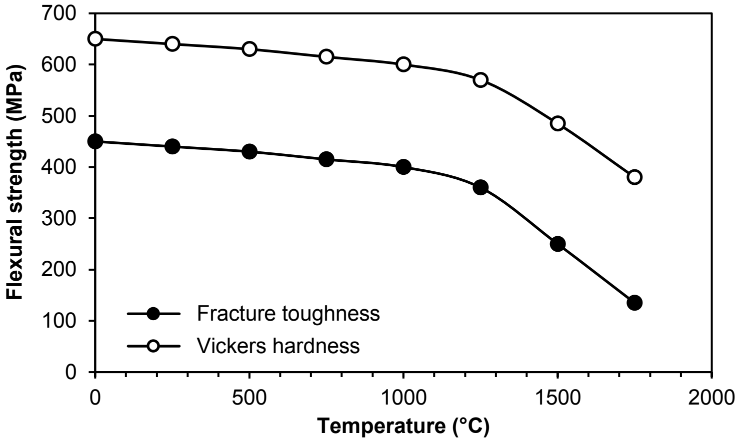

Fig. (3) Flexural strength vs. temperature up to 1800°C, demonstrating the compound's ability to retain its mechanical strength under hypersonic conditions.

Figure (3) confirms that SiC nanowire reinforcement does more than just boost ambient flexural strength; it fundamentally changes the material's performance envelope at extreme temperatures. By significantly improving strength retention and mechanical stability, these composites solve one of the primary challenges of ultra-high-temperature ceramics: the loss of structural load-bearing capacity near the material's melting point. These findings position ZrB_2-SiC nanowire composites as primary candidates for high-enthalpy propulsion components and the leading edges of hypersonic vehicles. In these environments, where thermal saturation is constant, the ability to maintain structural integrity, rather than just surviving the heat, is the critical factor. Ultimately, this reinforcement strategy provides a path toward thermal protection systems that offer both the durability and the reliability required for the next generation of deep-space and high-speed aerospace missions.

The oxidation kinetics of the ZrB_2-SiC system at 1600°C are compared in Fig. (4). The plot tracks mass gain (mg/cm^2) over time, serving as a primary metric for the growth of the oxide scale. Both the monolithic and nanowire-reinforced composites exhibit a continuous increase in mass throughout the exposure period, consistent with a parabolic growth model where surface reactions with oxygen produce a stable oxide layer. While both materials follow this general trend, the divergence in their oxidation rates highlights how microstructural modification influences chemical stability at extreme temperatures. In the standard composite, mass gain is more aggressive, suggesting a less controlled reaction. In contrast, the nano-reinforced material shows a more stabilized mass gain, indicating that the presence of the nanowires alters the formation or the density of the resulting oxide scale, effectively slowing the rate of oxygen diffusion into the bulk material.

As shown by the black dashed line in Fig. (4), the standard ZrB_2-SiC composite exhibits a steeper and more rapid mass gain compared to the reinforced variant. This accelerated kinetics suggests that the resulting oxide scale is either poorly adhered or highly permeable, failing to provide an effective barrier against oxygen ingress. The increased oxidation rate in the monolithic sample is likely linked to its coarser grain structure. Larger grains often result in a lower boundary density and a higher concentration of interconnected micropores or surface defects. These features act as "fast-track" diffusion pathways for oxygen, allowing it to bypass the surface and react more aggressively with the underlying ceramic matrix.

In contrast, the ZrB_2-SiC nanocomposite shows a significantly attenuated mass gain, with the curve flattening as exposure time increases. This parabolic behavior indicates the formation of a superior passive layer. The SiC nanowires appear to facilitate a denser, more cohesive oxide scale, likely a viscous silica-rich glass, which effectively seals the surface and restricts oxygen transport to the bulk material. Beyond chemical barrier formation, the nanowire network provides a mechanical advantage within the oxide scale itself. By reinforcing the growing oxide layer, the nanowires help dissipate residual thermal stresses that often lead to "spallation" or micro-cracking during prolonged heat exposure. This prevents the formation of new pathways for oxygen ingress, ensuring the material remains stable even under cyclic or long-duration thermal loads. The divergence between the two materials is most evident at the end of the test cycle: while the monolithic composite exceeds a mass gain of 10 mg/cm^2, the nanocomposite remains below 7 mg/cm^2. This 30% reduction in oxidation rate is a critical performance indicator for high-heat-flux applications, such as turbine components and hypersonic leading edges, where even minor surface degradation can lead to catastrophic structural failure.

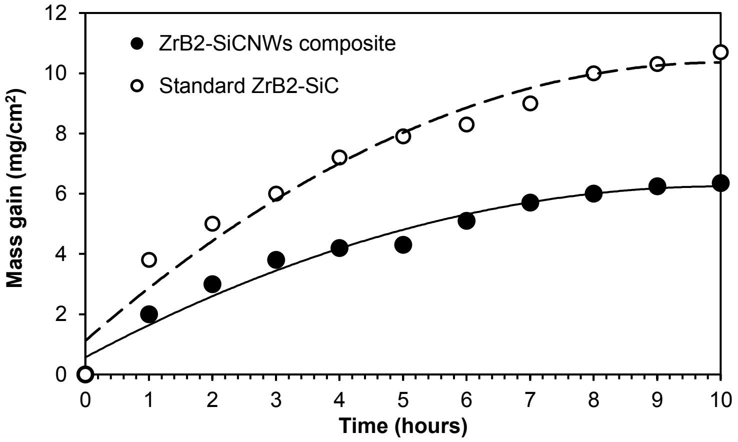

Fig. (4) Isothermal oxidation for assessing the material's lifespan in an atmospheric environment. The diagram illustrates the rate of weight gain or thickness of the oxidation layer formed over time at high temperatures, demonstrating the role of SiC nanowires in forming a protective glassy layer that shields the inner matrix from chemical corrosion

4. Conclusions

This research successfully demonstrates that the integration of SiC nanowires into a ZrB_2 matrix provides a definitive solution to the inherent brittleness and high-temperature degradation that typically limit ultra-high-temperature ceramics. By engineering the microstructure at the nanoscale, the composite activates a suite of reinforcement mechanisms (most notably nanowire pull-out, crack bridging, and deflection) which significantly bolster fracture toughness and maintain a robust flexural strength of 390 MPa even at 1800°C. Furthermore, the nanonetwork facilitates the development of a dense, cohesive silica-rich glass layer during high-temperature exposure, providing a passive chemical barrier that reduced mass gain by 30% compared to monolithic baselines at 1600°C. These findings confirm that ZrB_2-SiC nanowire composites possess the thermal-structural reliability necessary for the most extreme aerospace environments, positioning them as primary candidates for the next generation of hypersonic leading edges and high-performance rocket propulsion systems.

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