

Synthesis, Characterization, and Compositional Impact on the Thermal and Mechanical Properties of High-Entropy Carbides via High-Energy Ball Milling and Spark Plasma Sintering

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Abstract

High-entropy carbides (HECs) hold promise for high-temperature applications due to their potentially low thermal conductivity and strong mechanical properties. This study examines how compositional variations affect the thermal and mechanical properties of three HEC compositions: (Hf-Ta-Ti-Zr-Nb)C, (Ta-Ti-Nb-V-W)C, and (Hf-Ta-Ti-Nb-W)C. Synthesized via high-energy ball milling and spark plasma sintering, Composition A, characterized by its stable single-phase structure, exhibited the highest Vickers hardness (~ 1970 HV), fracture toughness (~ 5.8 MPa·m^{1/2}), and thermal conductivity (~ 13 W/m·K) at room temperature. In contrast, Compositions B and C, which formed secondary phases, showed reduced mechanical properties and thermal conductivity. These results highlight the need for precise compositional control and processing optimization to enhance HEC performance in high-temperature environments.

Keywords: High-entropy carbides, thermal conductivity, mechanical properties, high-energy ball milling, spark plasma sintering.

I. Introduction

The rapid advancement of high-tech industries, such as aerospace and polar marine exploration, has significantly increased the demand for materials that maintain exceptional properties under extreme environmental conditions^{1–4}. Traditionally, metals, alloys, and ceramics have been developed with one or two principal elements, with minor alloying additions to enhance specific attributes^{5, 6}. However, excessive alloying can lead to the formation of undesirable secondary phases, compromising the overall physical and chemical properties of the material⁷. As a result, the development of alloys with complex compositions has been limited, restricting the ability of conventional materials to meet the stringent demands of cutting-edge industries⁸.

To address these limitations, recent research has focused on high-entropy materials, which incorporate three or more principal elements in nearly equimolar proportions³. Among these, high-entropy carbides (HECs) have emerged as a novel class of materials with unique properties and broad potential applications⁹. Unlike traditional carbides, which typically include only one or two metal elements, HECs combine multiple metals and refractory elements, resulting in materials with exceptional hardness, high elastic moduli, and remarkable resistance to thermal shock and chemical corrosion^{10, 11}.

HECs have shown significant potential for advanced applications, particularly in industries that require materials

to withstand extreme temperatures and mechanical stresses. For example, in the aerospace sector, HECs are being explored for use in hypersonic vehicles and thermal protection systems in spacecraft, where materials must resist thermal shock and maintain structural integrity under rapid temperature fluctuations. Despite their promising properties, HECs' industrial application has been limited due to insufficient understanding of how variations in composition affect their mechanical and thermal properties^{12, 13}. This knowledge gap must be addressed to unlock the full potential of HECs in extreme environments¹².

The goal of this study is to assess their potential for applications that require high strength and low thermal conductivity in extreme environments, thereby advancing the understanding of how compositional variations impact these critical properties.

II. Experimental Procedures

In this study, high-entropy carbide (HEC) powders with nominal compositions of (Hf-Ta-Ti-Zr-Nb)C, (Ta-Ti-Nb-V-W)C, and (Hf-Ta-Ti-Nb-W)C were synthesized using commercially available carbide powders. The starting materials included seven carbide powders: HfC (99.5 %, < 15 μ m), TaC (99.5 %, 1 μ m), TiC (99.5 %, 2–3 μ m), ZrC (99.5 %, < 15 μ m), NbC (99 %, < 3 μ m), VC (99 %, 1–3 μ m), and WC (99.5 %, 0.3–0.4 μ m). These powders were selected based on their high purity and appropriate particle sizes, which are critical for achieving uniform alloying and optimal sintering characteristics in HECs (see Table 1 for detailed compositions).

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Table 1: Nominal compositions of the high-entropy carbides studied.

%	HfC	TiC	TaC	ZrC	NbC	VC	WC	Composition
Mole Ratio	1	1	1	1	1	-	-	(Hf-Ta-Ti-Zr-Nb)C
	-	1	1	-	1	1	1	(Ta-Ti-Nb-V-W)C
	1	1	1	-	1	-	1	(Hf-Ta-Ti-Nb-W)C

To prepare the powders for sintering, the carbide powders were subjected to high-energy ball milling. This method was chosen due to its effectiveness in reducing particle size and promoting uniform mixing, both of which are essential for producing homogenous HECs with desirable properties¹³. The milling was conducted in a cylindrical cemented carbide jar (74 mm in diameter, 60 mm in length) with 5.0 mm diameter cemented carbide balls, using a ball-to-powder weight ratio of 10:1. This specific ratio was selected based on previous studies, which indicated that a higher ball-to-powder ratio promotes more efficient milling, leading to finer particles and more uniform mixing, which are crucial for achieving a homogeneous microstructure in the final sintered product¹³. The jar was cooled externally with circulating water to manage the substantial frictional heat generated during the process, as this could otherwise lead to oxidation or unwanted phase transformations. An O-ring seal was used to maintain consistent internal pressure and prevent contamination, ensuring that the powders retained their purity throughout the milling process. The milling parameters were carefully selected to balance particle size reduction and phase homogeneity. A rotation speed of 600 rpm was chosen because it is high enough to ensure significant energy transfer to the particles, promoting effective milling, but not so high as to cause excessive heat generation or mechanical degradation of the powders. The milling time of 60 minutes was selected based on optimization studies that showed this duration to be sufficient for achieving the desired particle size reduction and uniform mixing without leading to excessive cold welding or contamination, which can occur with prolonged milling¹³.

The milled powders were then subjected to spark plasma sintering (SPS) using an SPS system (AGUS, SPS-210SX). Approximately 5 g of each powder mixture was loaded into a graphite die (10 mm inner diameter, 30 mm outer diameter) pre-coated with boron nitride (BN) to facilitate demolding and prevent reaction with the graphite. The sintering temperature was set to 1700 °C with a heating rate of 100 K/min, and a dwell time of 10 minutes was chosen. These sintering conditions were selected based on their ability to achieve full densification without excessive grain growth, which is critical for maintaining the desired mechanical properties^{14,15}. The uniaxial pressure of 50 MPa was applied to ensure good contact between the particles, enhancing the densification process. The cooling rate was controlled at 100 K/min to minimize thermal stresses and prevent crack formation, which could otherwise compromise the mechanical integrity of the sintered HECs.

For microstructural analysis of the milled powders and sintered bulks, a field-emission scanning electron microscope (FE-SEM, JEOL, JSM-7100F) was used. Elemental

compositions were analyzed using energy-dispersive spectroscopy (EDS) coupled with the FE-SEM. Phase identification was performed by means of X-ray diffraction (XRD, Bruker AXS, D8 ADVANCE) with a scan range of 20° to 90° 2θ and a step size of 0.02°, chosen for its ability to accurately identify the phases present in the HECs. Mechanical properties were assessed by measuring the density of the sintered samples using the Archimedes method (Alfa Mirage SD-120L) and determining Vickers hardness with a microhardness tester (Wilson VH3300) under a 10 kgf load. Fracture toughness (K_{IC}) was calculated from the lengths of the radial cracks emanating from the corners of the Vickers indentations using the Anstis equation¹⁶: $K_{IC} = 0.016 (E/H)^{1/2} (P/c^{3/2})$, where E is the Young's modulus (estimated using the rule of mixtures from constituent carbide properties), H is the measured Vickers hardness, P is the indentation load (10 kgf), and c is the average crack length measured from the center of the indent. Additionally, the thermal diffusivity (α) of the HEC samples was measured using the laser flash method with a thermal properties analyzer (NETZSCH, LFA 447). The measurements were performed on disk-shaped samples with approximately 10 mm in diameter and 2–3 mm in thickness. Prior to measurement, both surfaces of the samples were coated with a thin layer of graphite spray to ensure opacity and enhance emissivity/absorptivity. The thermal conductivity (λ) was then calculated using the measured thermal diffusivity (α), bulk density (ρ , determined by the Archimedes method as described above), and specific heat capacity (C_p , measured by means of Differential Scanning Calorimetry (DSC)) according to the equation: $\lambda = \alpha \times \rho \times C_p$.

Throughout this manuscript, the designations Composition A, Composition B, and Composition C refer to the high-entropy carbides with nominal formulas (Hf-Ta-Ti-Zr-Nb)C, (Ta-Ti-Nb-V-W)C, and (Hf-Ta-Ti-Nb-W)C, respectively. These were synthesized using equimolar mole ratios of the corresponding raw carbide powders, as detailed in Table 1.

III. Results and Discussion

High-energy ball milling significantly contributed to the refinement and alloying of powders in the fabrication of high-entropy carbide (HEC) sintered bulks, which is crucial for optimizing the microstructure and properties of the final materials. This process reduced particle size and enhanced powder homogeneity, facilitating densification during sintering and improving the overall performance of the HEC sintered bulks^{15,17}. The initial raw powders, observed using field-emission scanning electron microscopy (FE-SEM), exhibited irregular shapes with average parti-

cle sizes ranging from 1 to 15 μm , depending on the type of powder (Fig. 1(a-f)).

Post-milling FE-SEM images (Fig. 2(a-c)) showed a uniform size distribution with agglomerated nanoscale features, likely due to intensified Van der Waals forces as the specific surface area increased¹⁸. This refinement aligns with previous studies on sinterable materials such as ceramics and superhard materials¹⁹. A 60-minute milling duration was found optimal for achieving adequate refinement and uniform mixing, avoiding the risks of contamination and cold welding associated with longer milling times¹⁵. While some coarse particles remained unrefined, energy-dispersive spectroscopy (EDS) mapping confirmed that the overall mixture achieved a relatively uniform blend after 60 minutes of milling.

X-ray diffraction (XRD) analysis of the powders and sintered bulks (Fig. 3) revealed significant phase transformations across all compositions. The monocarbide phases present in the milled powders (Fig. 3(a)) gradually transformed into a rock-salt phase during the sintering process (Fig. 3(b))^{19,20}. Composition A formed a stable high-entropy single phase, whereas Compositions B and C exhibited secondary phases, particularly with the addition of tungsten (W). This was evidenced by a rightward shift in the 2-theta value of the major peaks in Compositions B and C, likely due to lattice distortion from the hexagonal close-packed (HCP) structure of WC²⁰. Additionally, in Composition C, peaks corresponding to HfO_2 and carbon were observed, indicating phase segregation or the formation of binary phases due to the differing constituent elements²¹.

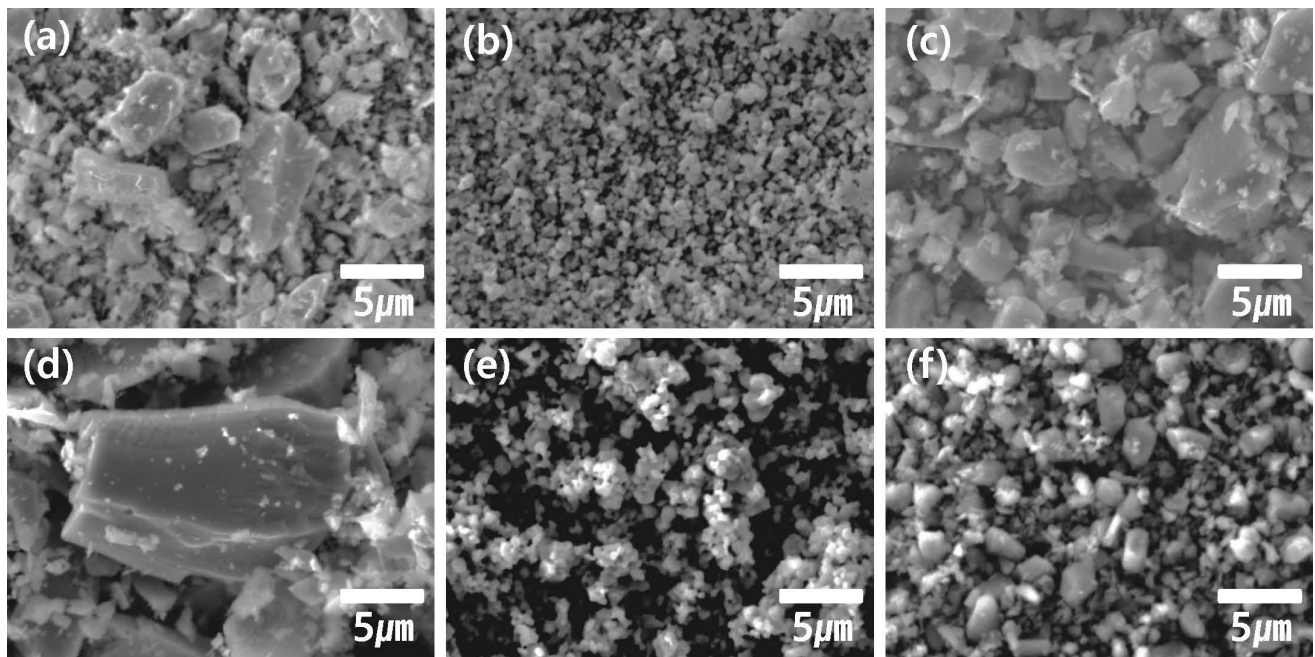


Fig. 1: FE-SEM images showing the morphology of raw carbide powders: (a) HfC ($< 15 \mu\text{m}$), (b) TaC ($1 \mu\text{m}$), (c) TiC ($2-3 \mu\text{m}$), (d) ZrC ($< 15 \mu\text{m}$), (e) NbC ($< 3 \mu\text{m}$), and (f) VC ($1-3 \mu\text{m}$).

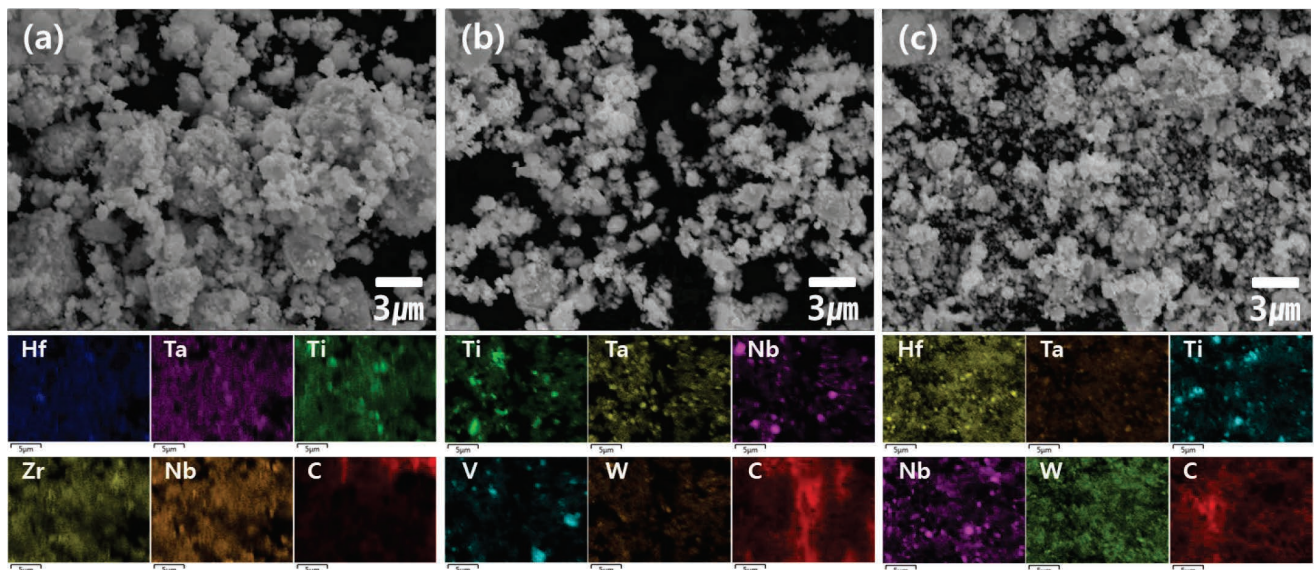


Fig. 2: FE-SEM images and EDS mapping results of high-entropy carbide powders after 60 minutes of milling: (a) (Hf-Ta-Ti-Zr-Nb)C, (b) (Ta-Ti-Nb-V-W)C, and (c) (Hf-Ta-Ti-Nb-W)C.

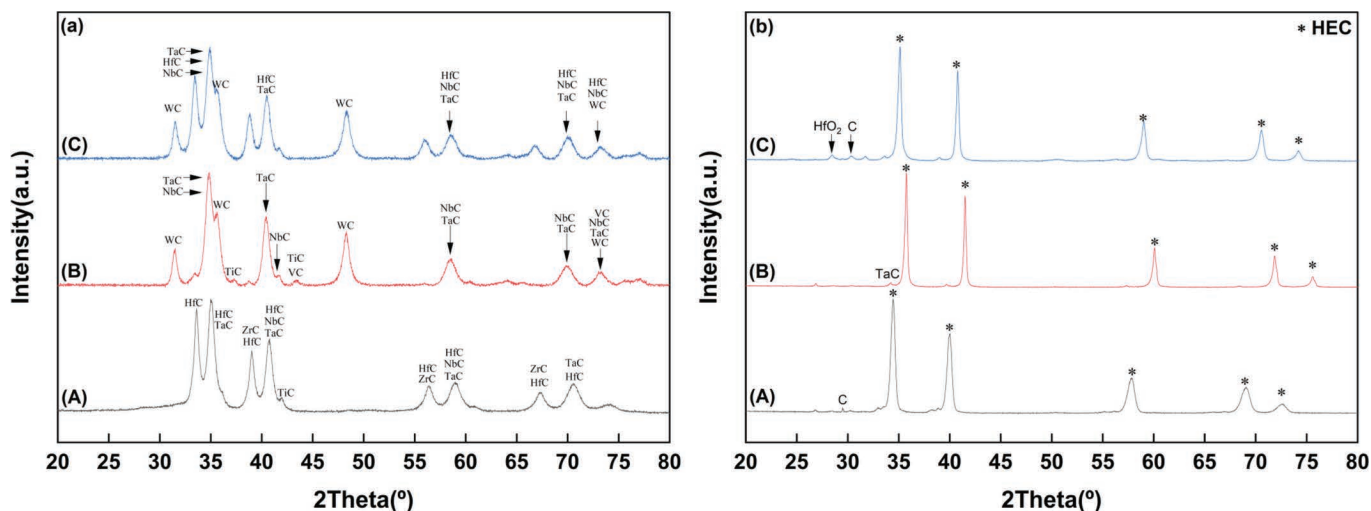


Fig. 3: XRD patterns of high-entropy carbide sintered bulks with different compositions: (a) (Hf-Ta-Ti-Zr-Nb)C, (b) (Ta-Ti-Nb-V-W)C, and (c) (Hf-Ta-Ti-Nb-W)C.

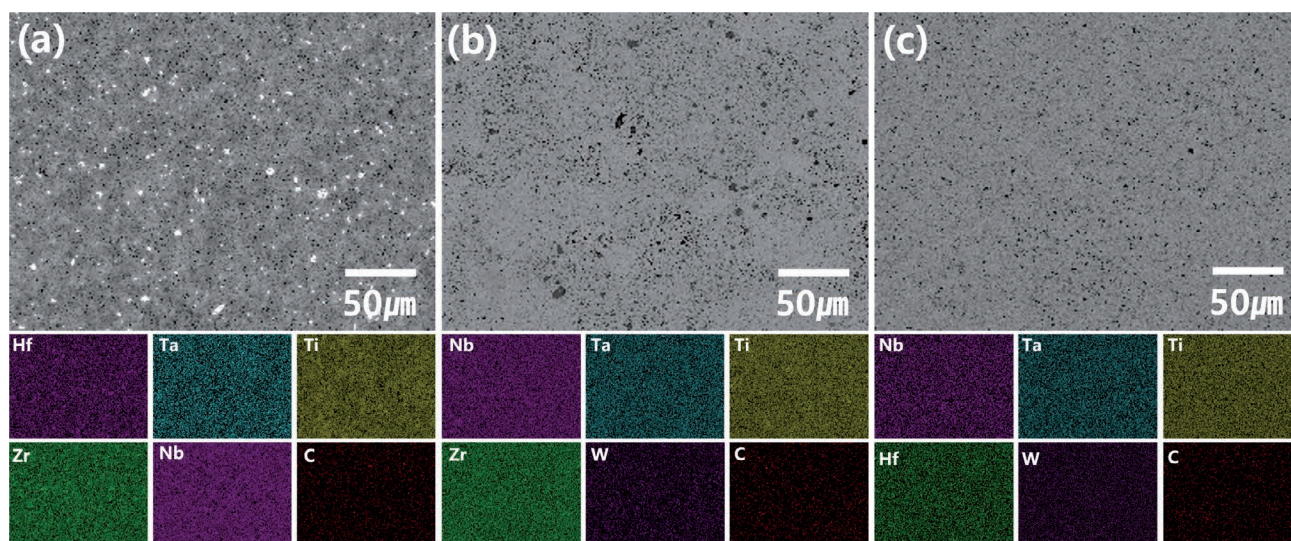


Fig. 4: Low-magnification cross-sectional FE-SEM images and EDS mapping results of high-entropy carbide sintered bulks processed at 1700 °C: (a, e) (Hf-Ta-Ti-Zr-Nb)C, and (b, f) (Ta-Ti-Nb-V-W)C.

Cross-sectional microstructural observations and compositional analysis of the sintered bodies (Figs. 4 and 5) confirmed that all compositions produced sintered bulks that were free from macroscopic defects such as cracks or large voids, although they contained residual porosity consistent with the measured relative densities (93 % – 97.5 %). However, higher magnification observations (Fig. 5) revealed secondary phases, such as TiC-rich regions and $\text{HfO}_2 + \text{C}$, in Compositions B and C, where tungsten (W) was added, unlike Composition A, which maintained a single-phase structure²². In the SEM micrograph of Composition A (Fig. 5a), the predominant gray matrix represents the homogeneous HEC phase. The minor, isolated white specks likely correspond to regions with slightly higher average atomic number, potentially due to minor enrichment of heavier elements like Hf and Ta, while the dark spots are residual pores. These secondary phases can impact the material's properties, as discussed below.

The formation of a stable high-entropy single phase in Composition A can be attributed to the compatibility of

its constituent elements, all of which share an FCC structure²². This structural compatibility likely contributed to its stability. In contrast, the addition of WC with an HCP structure in Compositions B and C introduced structural instability, leading to the formation of secondary phases during sintering^{17,20}. These observations are consistent with findings in other studies, where the specific composition of high-entropy carbides influenced the formation of secondary phases²⁰. The presence of TiC-rich phases in Composition B and $\text{HfO}_2 + \text{C}$ in Composition C suggests that Ti and Hf, alongside W, were more prone to phase segregation to achieve structural stability.

The relative densities of the sintered bulks (Fig. 6) ranged from approximately 93 % to 97.5 %. While all compositions showed high densities, the formation of secondary phases in Compositions B and C likely contributed to their slightly lower densities compared to Composition A. In particular, the lower relative density of Composition C can be attributed to the formation of HfO_2 and segregated graphite, which decreased the overall density.

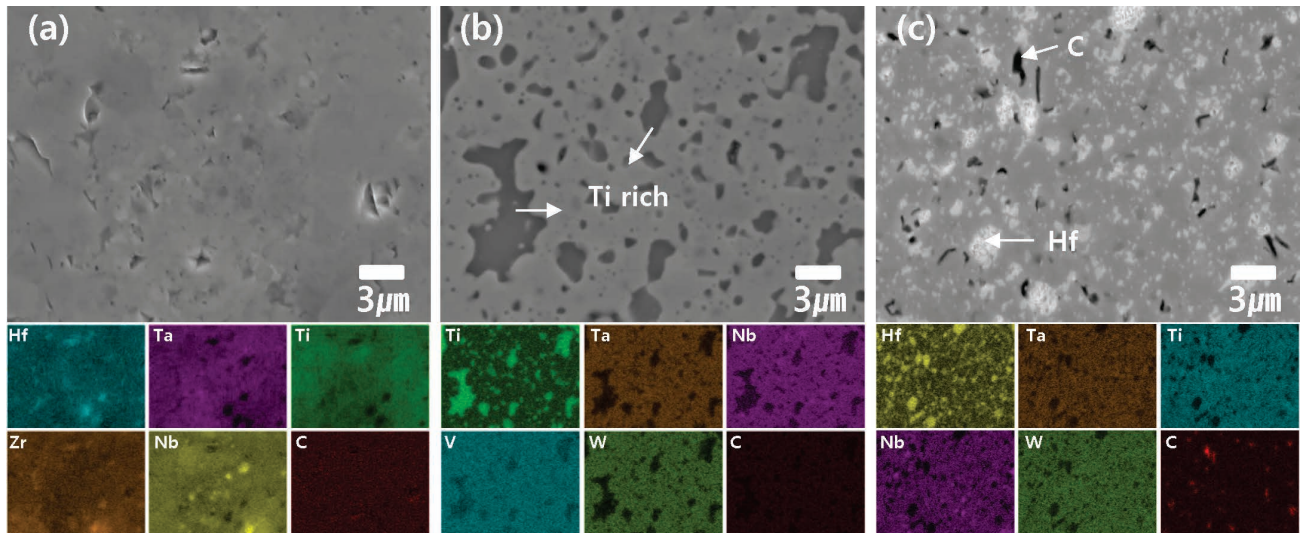


Fig. 5: High-magnification cross-sectional FE-SEM images and EDS mapping results of high-entropy carbide sintered bulks processed at 1700 °C: (a, e) (Hf-Ta-Ti-Zr-Nb)C, (b, f) (Ta-Ti-Nb-V-W)C, and (c, g) (Hf-Ta-Ti-Nb-W)C.

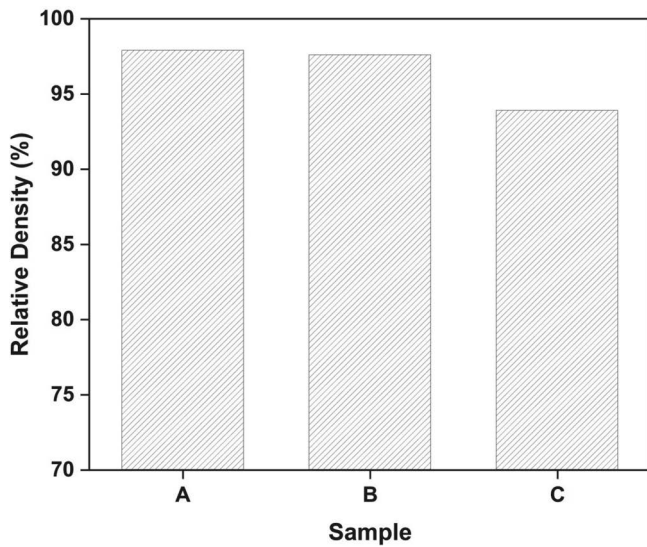


Fig. 6: Relative density of high-entropy carbide sintered bulks with different compositions: (a) (Hf-Ta-Ti-Zr-Nb)C, (b) (Ta-Ti-Nb-V-W)C, and (c) (Hf-Ta-Ti-Nb-W)C.

The mechanical properties of the HECs were strongly influenced by their microstructural composition. As shown in Fig. 7, the Vickers hardness of the compositions was approximately 1620 HV (Composition C), 1750 HV (Composition B), and 1970 HV (Composition A). Composition A, with its single-phase structure, exhibited the highest hardness. In contrast, Compositions B and C, which contained secondary phases, showed lower hardness and fracture toughness (K_{IC}) values: 5.4 $\text{MPa}\cdot\text{m}^{1/2}$ for Composition B and 5.7 $\text{MPa}\cdot\text{m}^{1/2}$ for Composition C, compared to 5.8 $\text{MPa}\cdot\text{m}^{1/2}$ for Composition A. These results align with the known impact of secondary phases, which act as stress concentrators and promote crack initiation and propagation.

Fig. 8 illustrates the variation in thermal conductivity of high-entropy carbide compositions with different compositions as a function of temperature. The thermal properties can be calculated using the following formula:

$$\alpha = \lambda / (\rho \times C_p)$$

where α is thermal diffusivity, λ is thermal conductivity, ρ is density, and C_p is specific heat capacity.

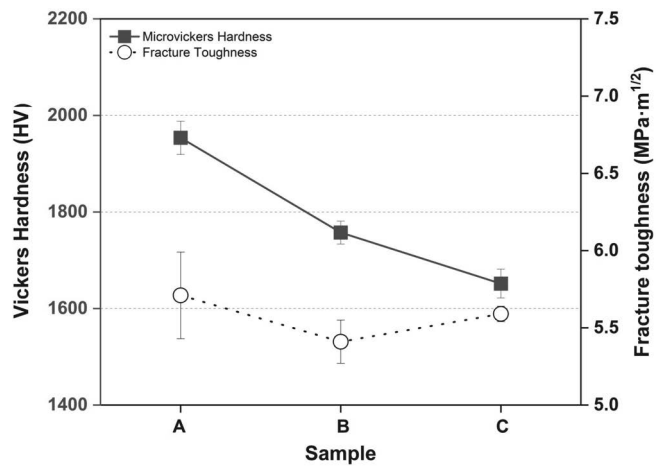


Fig. 7: Vickers hardness (left axis, ■) and fracture toughness (K_{IC} , right axis, ○) of high-entropy carbide sintered bulks with different compositions (A, B, C). Hardness was measured under a 10 kgf load.

Thermal conductivity measurements (Fig. 8) showed that the HEC compositions exhibited values of 7.0 $\text{W}/\text{m}\cdot\text{K}$ (Composition C), 11 $\text{W}/\text{m}\cdot\text{K}$ (Composition B), and 13 $\text{W}/\text{m}\cdot\text{K}$ (Composition A) at room temperature, with Composition A again showing the highest values due to its single-phase structure²³. The presence of secondary phases in Compositions B and C reduced thermal conductivity, particularly in Composition C, which showed the lowest value (7.0 $\text{W}/\text{m}\cdot\text{K}$). This reduction is attributed to increased phonon scattering at the interfaces of secondary phases, which disrupt the regular atomic structure²³. Even at elevated temperatures (up to 873.15 K), the thermal conductivity of all the samples remained lower than that of conventional shielding materials such as oxides^{19, 23}.

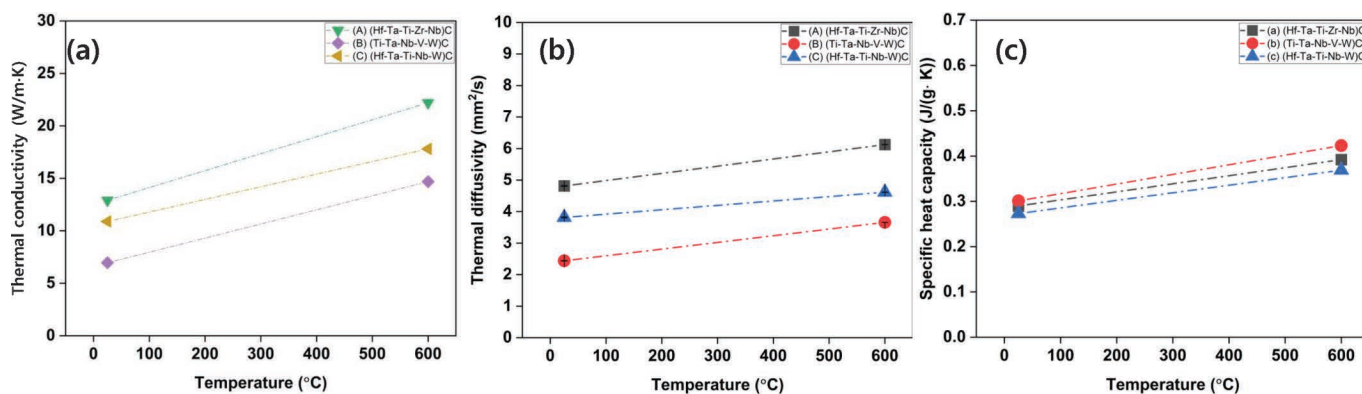


Fig. 8: Thermal properties of high-entropy carbide sintered bulks as a function of temperature: (a) Thermal conductivity, (b) Thermal diffusivity, and (c) Specific heat capacity for Compositions A ((Hf-Ta-Ti-Zr-Nb)C), B ((Ta-Ti-Nb-V-W)C), and C ((Hf-Ta-Ti-Nb-W)C).

In conclusion, the formation of secondary phases in HECs, while often inevitable due to their complex chemistry, has a critical impact on both mechanical and thermal properties. However, with careful control of the milling and sintering conditions, the formation of these secondary phases can be managed, allowing for the fine-tuning of mechanical and thermal properties to meet specific application requirements^{15,23}. Future research should focus on optimizing these processing conditions to balance the trade-offs between mechanical strength and thermal conductivity, thereby tailoring HECs for specific high-performance applications²².

IV. Conclusions

In this study, high-entropy carbides (HECs) with varying compositions were successfully synthesized and characterized using high-energy ball milling followed by spark plasma sintering (SPS). The key findings are summarized as follows:

1. **Synthesis and Microstructure:** Composition A ((Hf-Ta-Ti-Zr-Nb)C) maintained a stable single-phase structure after sintering at 1700 °C, while Compositions B ((Ta-Ti-Nb-V-W)C) and C ((Hf-Ta-Ti-Nb-W)C) developed secondary phases (e.g., TiC-rich regions, HfO₂+C), particularly due to the addition of tungsten (W).
2. **Mechanical Properties:** Composition A exhibited superior mechanical properties with a Vickers hardness of ~1970 HV and a fracture toughness (K_{IC}) of ~5.8 MPa·m^{1/2}. The formation of secondary phases in Compositions B and C led to reduced hardness (~1750 HV and ~1620 HV, respectively) and lower fracture toughness (~5.4 MPa·m^{1/2} and ~5.7 MPa·m^{1/2}, respectively).
3. **Thermal Properties:** Consistent with its single-phase structure, Composition A displayed the highest thermal conductivity (~13 W/(m·K) at room temperature). The presence of secondary phases and interfaces in Compositions B and C resulted in lower thermal conductivity (~11 W/(m·K) and ~7.0 W/(m·K), respectively, at room temperature), attributed to increased phonon scattering. These findings highlight the critical influence of composition on phase stability and the resulting mechanical and thermal properties in HECs.

Precise compositional control and optimization of processing conditions are essential for tailoring these materials for demanding high-temperature applications.

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