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RESEARCH ARTICLE

Brazing ZrB_2 ultra-high-temperature ceramics to Zr metals for hypersonic thermal protection systems

Younes Belrhiti[1] | **M. Watson Grossman**[1] | **R. Hedgecock**[1] | **M. McGilvray**[2] | **L. Vandeperre**[1]

[1]Department of Materials, Imperial College London, London, UK

[2]Oxford Thermofluids Institute, University of Oxford, Oxford, UK

Correspondence
Younes Belrhiti, Department of Materials, Imperial College London, London, UK.
Email: younes.belrhiti@gmail.com

Abstract

Porous and dense zirconium diboride (ZrB_2) materials are promising ultra-high-temperature ceramics (UHTCs) for transpiration cooling in hypersonic applications, providing an effective means to reduce component temperatures and mitigate oxidation effects. While porous ZrB_2 enables coolant flow, dense ZrB_2 offers structural integrity. However, achieving a reliable ceramic-to-metal connection remains a significant challenge due to the inherent differences in their physical, chemical, and mechanical properties. This study investigates the brazing process of both porous and dense ZrB_2 UHTCs to zirconium metal (Zr). Zr was selected due to its thermal expansion properties closely matching those of ZrB_2, reducing thermal stress at the interface and enabling coolant transfer into porous ZrB_2 from the supply reservoir. The brazing temperature of 1650°C was selected based on the Zr–ZrB_2 phase diagram to ensure eutectic melting without compromising the structural integrity of the base materials, and the filler composition was selected to enhance wetting and spreading. The results demonstrate that dense ZrB_2 forms robust and continuous interfaces with Zr metal, while porous ZrB_2 presents greater challenges due to thermal expansion mismatches and paste infiltration. These findings provide valuable insights into ceramic–metal joining for aerospace high-performance thermal protection systems under extreme conditions, which would reinforce the real-world impact.

KEYWORDS

brazing, ceramic–metal joining, hypersonic applications, transpiration cooling, UHTCs

1 | INTRODUCTION

Porous and dense zirconium diboride (ZrB_2) materials are among the most promising ultra-high-temperature ceramics (UHTCs)[1–6] for extreme environment applications requiring alternative engineering approaches, particularly transpiration cooling in hypersonic applications.[7] This cooling technology involves passing a coolant fluid through porous materials to reduce the overall temperature of the component. Additionally, it can mitigate oxidation effects, a major challenge for most UHTCs in air environments.[8,9] This technology is particularly suitable

for high-heat flux regions, such as leading edges or shock impingement zones, which are exposed to extreme thermal loads (>1 MW/m^2) due to the high velocities of hypersonic vehicles traveling through planetary atmospheres. In such extreme conditions, materials must withstand not only thermal loads but also intense mechanical and oxidative stresses encountered during planetary atmospheric re-entry and sustained hypersonic flight.[10,11]

In the studied application, the coolant needed to be transferred into the porous ZrB_2 through a metallic tube connected to the supply reservoir to achieve effective cooling. While porous ZrB_2 is critical for enabling this cooling mechanism, dense ZrB_2 plays a complementary role by providing mechanical strength and thermal compatibility at structural interfaces, such as the junctions with metallic coolant channels. Establishing a reliable bond between ceramics and metals is crucial to fully exploit their complementary properties. While ceramics[12,13] exhibit excellent high-temperature performance, metals[14] provide superior toughness and mechanical strength. Ceramics and metals have considerable differences in physical, chemical, and mechanical properties, and so these advanced materials put forward new requirements for material joining techniques. Indeed, conventional fusion welding methods, such as arc,[15] electron beam,[16] and laser welding,[17] cannot be used to connect ceramics and metals effectively. Various joining techniques have been explored, such as ultrasonic bonding,[18] self-propagating high-temperature synthesis (SHS),[19] and partial transient liquid phase welding,[20] have been developed in recent years. However, current literature and practical applications indicate that brazing and diffusion welding are still the primary methods of connecting ceramics and metals.[21–23]

This paper focuses on the brazing process to join both porous and dense ZrB_2 UHTCs with metals, aiming to create structurally sound interfaces for future deployment in hypersonic thermal protection systems. Zirconium (Zr) was selected due to its thermal expansion coefficient being close to that of ZrB_2, minimizing thermal stress mismatches between Zr and ZrB_2, thereby limiting residual stresses at the interface. The brazing temperature was carefully chosen to ensure that the filler material melts while preventing the base materials from reaching their melting points, thereby preserving their structural integrity and performance. Furthermore, the composition of the metallic filler paste was selected to achieve wetting and spreading at brazing temperature. Given the fundamental differences between ceramics (covalent bonding) and metals (metallic bonding), achieving continuous adhesion requires precise control over wettability, which is influenced by factors such as atmosphere, temperature, thermodynamic stability, alloy composition, and surface conditions to achieve continuous interfaces between ZrB_2 and Zr metal were obtained. The conditions for this brazing process are detailed in this paper.

2 | EXPERIMENTAL

2.1 | Materials

Porous ZrB_2 (35% porosity) were produced using ZrB_2 powder (ABCR, grade A, 3–5 mm). The powder was partially oxidized by heating 540°C for 30 min before sieving and hot-pressing in an argon (Ar) atmosphere at 1700°C under 24 MPa for 15 min to prevent the oxidation of the powder. Pre-oxidizing the powder surface promotes structural coarsening and enhances necking between particles while preventing excessive densification at high temperatures. This results in a more open porous structure, which is desirable for transpiration cooling. Porous discs and porous hollow cylinders were manufactured and presented in Figure 1A.

Dense ZrB_2 was produced by hot-pressing ZrB_2 powders (ABCR, grade B, 1–3 mm) at 2000°C under 24 MPa, achieving a density of 95%. Dense ZrB_2 hollow cylinders were manufactured (Figure 1A). The dense ZrB_2 samples in this work were obtained by joining a dense ZrB_2 cylinder to a porous ZrB_2 disc using compression at high temperature (2100°C) using a small amount of a ZrB_2 paste to facilitate bonding.

The ZrB_2 cylinder was wire-cut using electrical discharge machining (EDM) around the partial thickness drill holes, and the rear collar was rough-cut away using wire EDM. The outer cylindrical surface was finished using a die-sink electrode. Pilot holes are wet-drilled with SiC bits (4 mm, then 6 mm) from RS components (UK supplier), and final finishing was achieved using 6.5 mm thin-wall diamond core drills.

Commercial Zr tubes (ZR00-TB-000200 with a purity of 99.2%) with an outside diameter of 6.35 mm, a wall thickness of 0.9 mm, and an inside diameter of 4.55 mm were used. The final sample geometry is detailed in Figures 1B,C.

2.2 | Paste preparation

Bonding between Zr tubes and ZrB_2 was achieved using a glycerol-based paste containing submicron Zr(m) and ZrB_2 powders in eutectic proportions of 86.3% Zr and 13.7% B which produces a liquid phase when fired at 1650°C, as indicated by the phase diagram represented in Figure 2. Any intermediate paste composition would also form a eutectic melt, but controling the reaction kinetics is crucial to limiting erosion of both the metal and ceramic. The

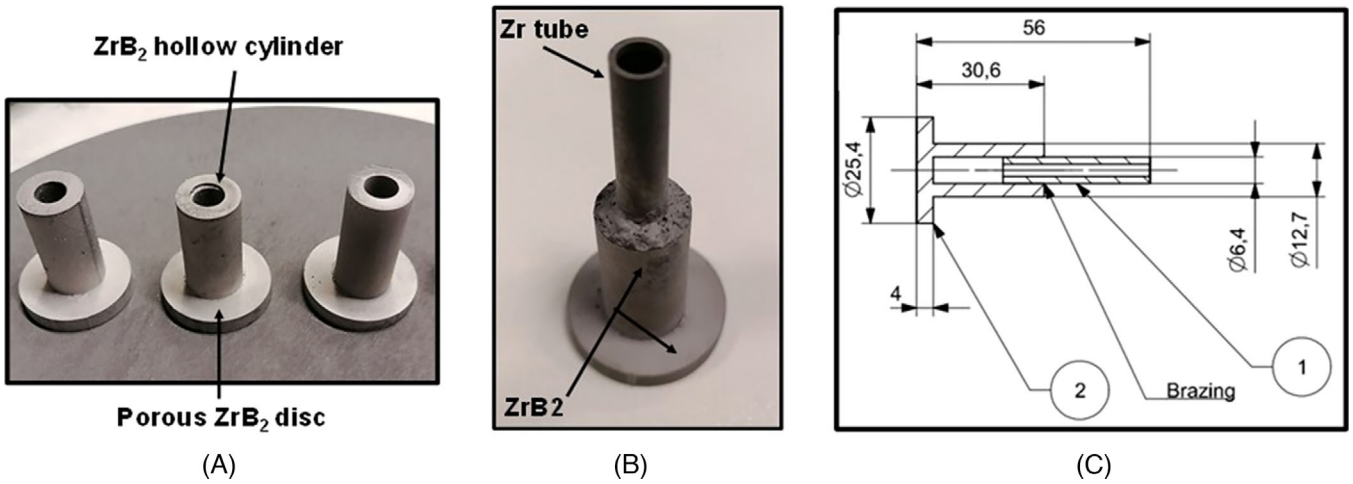

FIGURE 1 Brazed Zr tube with ZrB_2 sample.

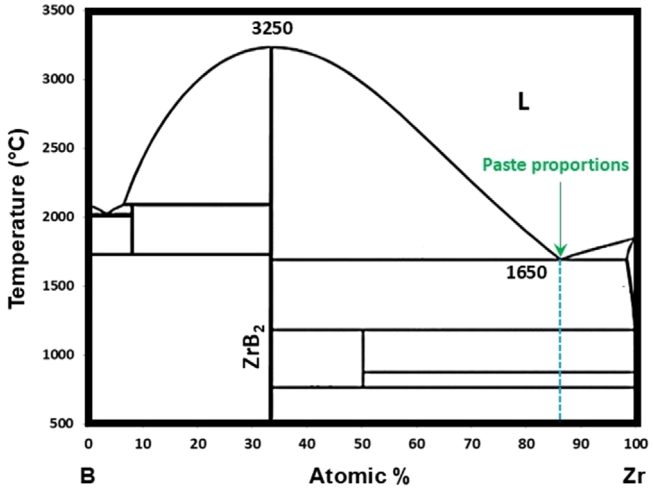

FIGURE 2 Zr–B phase diagram.

empirically optimized paste composition and sintering conditions provided a balance between bonding strength and reaction control at the interface.

Zr powder was mixed with ZrB_2 powder as received (ABCR, grade B, 1–3 μm) using a mortar and pestle. A minimal amount of glycerol was then added to create a thick and sticky paste.

2.3 | Brazing process

A 10 mm section of the Zr tube was coated with the paste and introduced in the rear tube of the dense ZrB_2 sample. Excess paste was scraped off the external surface while introducing the Zr tube inside the ZrB_2 sample (Figure 2B). The sample was sintered at 1650°C (eutectic melt temperature of Zr and ZrB_2) under vacuum (~10^{-5} mbar), using a heating ramp of 20°C/min and a dwell time of 20 min. During cooling to room temperature under vacuum (10°C/min), the molten filler solidified, forming a continuous bond without significant interfacial voids between the ceramic and metal[24] (Figure 1B).

2.4 | Leakage and pressure resistance testing

To validate the effectiveness of the brazed joint, compressed air was injected through the Zr tube into the sample at an applied pressure of 6.5 bar for 10 min, corresponding to the expected operational conditions of the final application. The Zr tube was tightly sealed at the compressed air inlet on one side, while the other end was joined to a dense or porous hollow cylindrical ZrB2 element, which was itself sealed to a porous ZrB_2 disc as shown in Figure 1. A pressure gauge was connected to monitor the internal pressure buildup. The entire system was maintained in a closed configuration to simulate actual in-service conditions where pressurization would occur. Additionally, a soapy water solution was applied around the external interface during pressurization to detect any leakage through the bonding region.

2.5 | Microstructure characterization: scanning electron microscopy analysis

The sample was fully embedded in an epoxy resin (Buehler, EpoThin 2, 20-3440-032) with a resin-to-hardener ratio of 100:45. Sectional cuts were prepared for scanning electron microscopy (SEM) analysis including grinding, polishing, and the gold coating application on the analyzed surface.

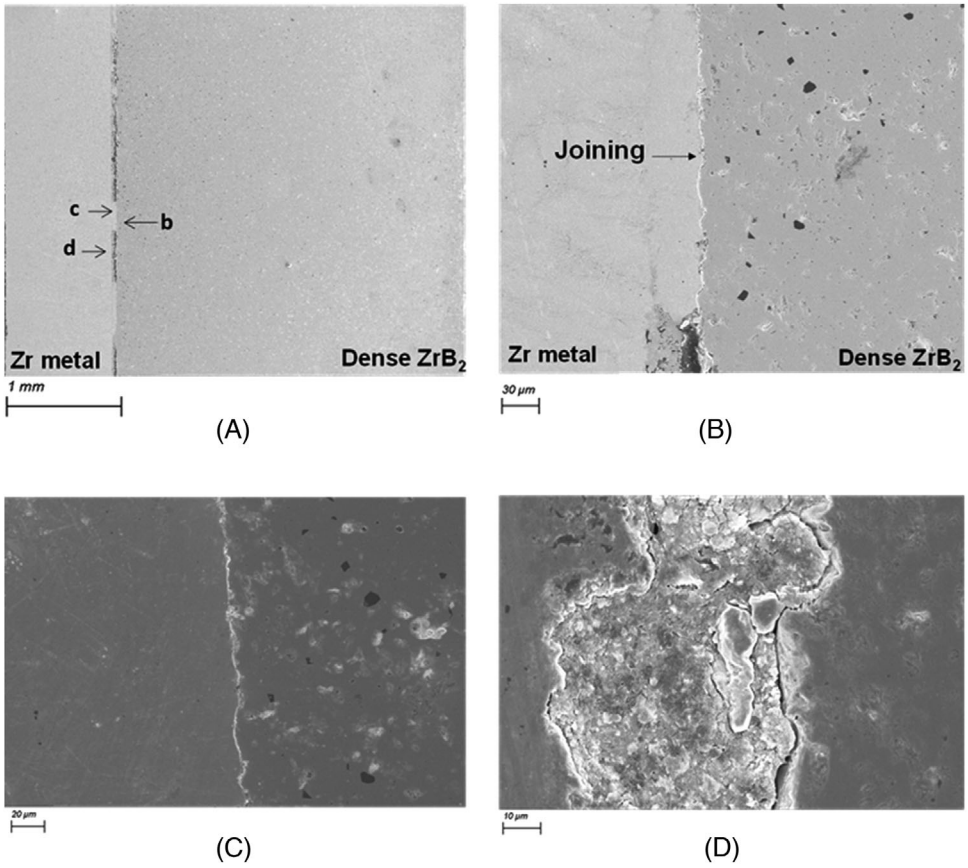

FIGURE 3 SEM analysis of dense ZrB_2 brazed to Zr metal. SEM, scanning electron microscopy.

3 | RESULTS AND DISCUSSION

Figure 3 presents two SEM images of a vertical cross-section of the brazed sample, showing the interface between the Zr tube and the dense ZrB_2.

Figure 3A provides an overview of the central region of the sample with low-magnification SEM images showing the macrostructure of the joint between Zr metal (left) and dense ZrB_2 (right), distinguishable. At a macroscopic scale, the bonding interface appears continuous; however, dark lines are locally observed along the interface, which are attributed to unreacted grains or potential microvoids, likely resulting from incomplete wetting or minor surface contamination. To avoid the presence of unreacted grains in the joint, it would be advantageous to sieve the Zr–ZrB_2 mixture before adding of glycol.

Figure 3B,C highlights higher-magnification SEM images which provide a detailed view of the interface region between the Zr metal and dense ZrB_2 presented in Figure 3A. The continuous brazing line is visible without delamination. Figure 3D represents a close-up of a localized defect observed in Figure 3B. This feature does not correspond to a gap, but rather to unreacted grains that remained during the brazing process likely resulting from a local wetting failure, surface contamination. The presence of such a defect indicates that, despite the overall integrity of the joint, localized bonding imperfections may occur due to the paste mixing homogeneity and so interfacial reactions.

The presence of submicron Zr(m) as an active element with a high melting point enhanced the wettability of the paste, promoting bonding between ceramic and metal, as evidenced by the absence of visible delamination. Additionally, the absence of significant gaps observed at the interface is attributed to the similar thermal behavior of both base materials at the brazing temperature (1650°C) and during the cooling phase.

The ZrB_2–Zr connection remained intact under 6.5 bar air pressure, with no observed leakage or delamination. Besides, no external bubbles from the soapy solution were detected in this region, confirming that the seal was effective and airtight to be used for the final application.

Two porous ZrB_2 cylinders were brazed to the Zr tube to manufacture the porous brazed sample. A sectional cut of the first sample was prepared and analyzed using SEM, as shown in Figure 4.

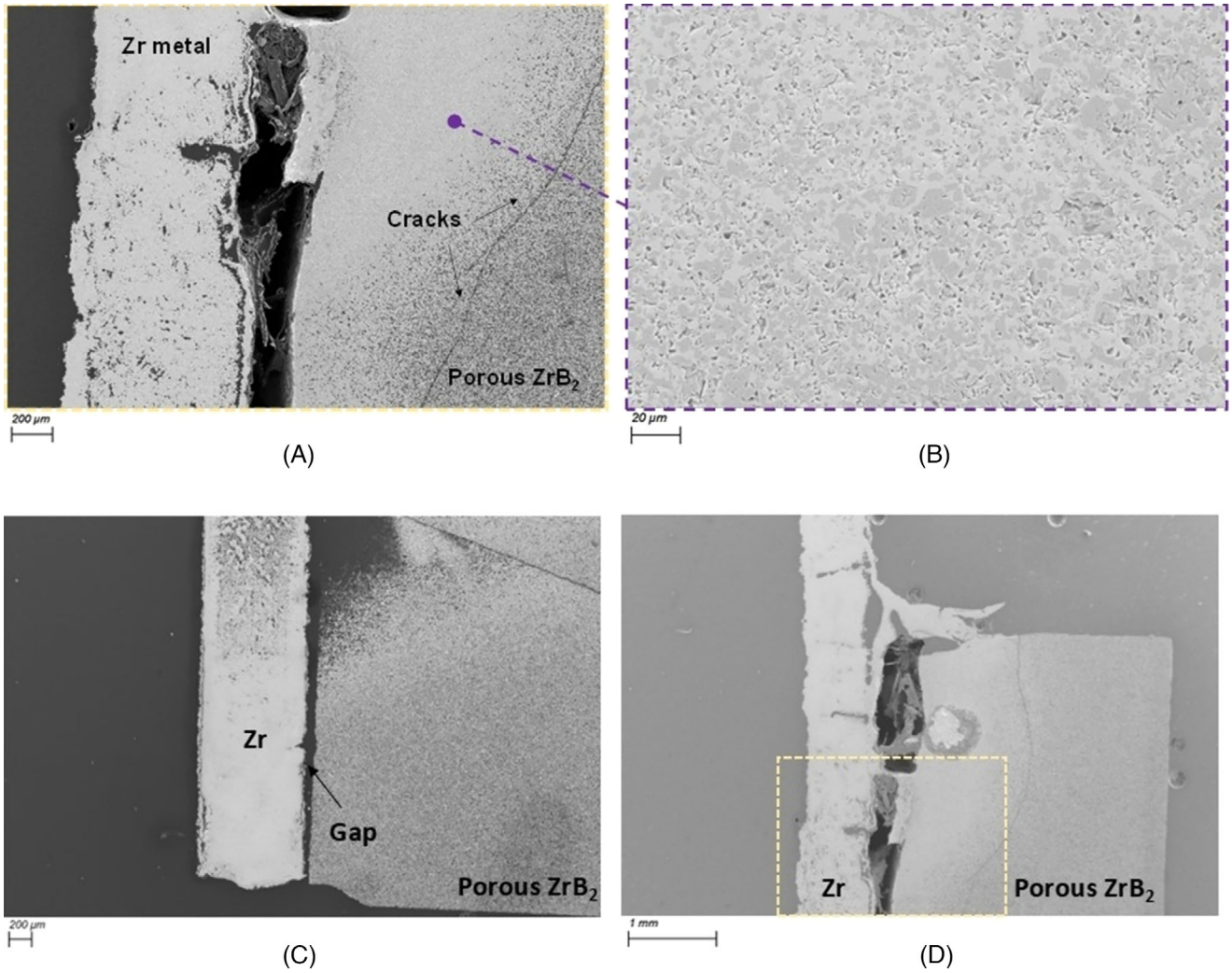

FIGURE 4 SEM analysis of porous ZrB_2 brazed to Zr metal. SEM, scanning electron microscopy.

Figure 4A,C,D shows the macrostructure of the brazed interface. A gap is visible at the interface indicating a failure of the brazed joint. The porous nature of ZrB_2 led to discontinuous adhesion at the interface and the interfacial region shows irregularities.

Figure 4B provides a high-magnification view of the porous ZrB_2 microstructure after brazing. The filler paste infiltrated into the porous structure. The penetrated paste appears light gray in Figure 4B, while ZrB_2 grains are represented in dark gray. This infiltration of the filler paste into the porous ZrB_2 did not improve the mechanical strength of the joint and instead induced localized property modifications. During cooling, the hardening of the infiltrated paste led to the generation of thermal stresses[25] at the interface between the porous zone of ZrB_2 and the infiltrated regions. These stresses induced the formation of cracks around the infiltrated region, as shown in Figure 4A,D. A second porous sample was tested using air compression at 6.5 bar, corresponding to the expected operational conditions of the final application. After a few minutes of air injection, the Zr tube was detached from the porous ZrB_2 sample, indicating that the brazed joint was not suitable for the intended application. The failure mechanism is attributed to the weak adhesion at the interface due to low wetting of the porous ZrB_2, paste infiltration altering the local mechanical properties and the thermal expansion mismatch between porous ZrB_2 and Zr metal.

Figure 5 presents the thermal expansion behavior of dense ZrB_2, porous ZrB_2, and the Zr metal tube, obtained through dilatometry measurements from room temperature up to 1650°C.

Zr metal has a quite similar nonlinear thermal expansion behavior as dense ZrB_2 and a different behavior from the porous ZrB_2. To evaluate the thermal compatibility between the materials, the coefficient of thermal expansion (CTE) was extracted from the thermal strain ($\Delta L/L_0$) data over multiple temperature intervals for dense ZrB_2, porous ZrB_2, and Zr metal. Table 1 summarizes

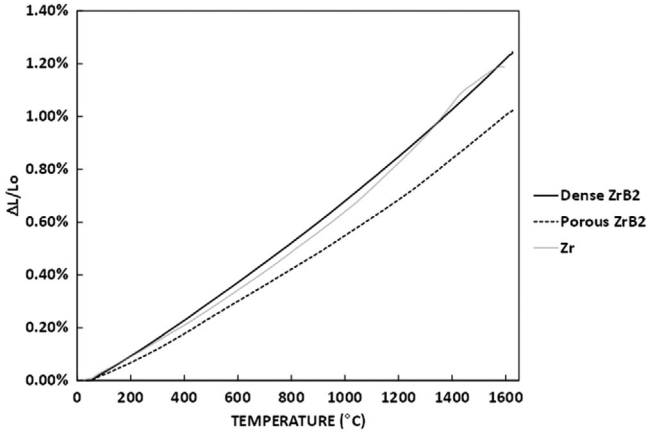

FIGURE 5 Thermal expansion of ZrB_2 materials (porous and dense) and the Zr tube.

TABLE 1 A comparison of CTE values of dense and porous ZrB2 with that of Zr over multiple temperature ranges.

	Zr	Dense ZrB_2	Porous ZrB_2
200°C–400°C	5.92E−06	6.74E−06	5.50E−06
600°C–800°C	7.22E−06	7.50E−06	6.07E−06
1000°C–1200°C	9.26E−06	8.40E−06	6.79E−06
1400°C–1600°C	8.98E−06	9.62E−06	8.18E−06

the CTE values across the 200°C –400°C, 600°C–800°C, 1000°C–1200°C, and 1400°C–1600°C ranges.

Zr[26] exhibited a nonlinear increase in CTE with temperature, rising from $5.92 \times 10^{-6}K^{-1}$ at 200°C–400°C to a peak of $9.26 \times 10^{-6}K^{-1}$ at 1000°C –1200°C, followed by a slight decrease at higher temperatures. Dense ZrB_2 exhibited a more stable thermal expansion trend, with CTE values ranging from 6.74×10^{-6} to $9.62 \times 10^{-6}K^{-1}$, which is consistent with previously reported values in the literature.[27,28] Above 600°C, its CTE closely approaches that of Zr. In contrast, porous ZrB_2 exhibited consistently lower CTEs across all intervals, from $5.50 \times 10^{-6}K^{-1}$ at 200°C–400°C to $8.18 \times 10^{-6}K^{-1}$ at 1400°C–1600°C, due to its lower bulk stiffness.

Zr and dense ZrB_2 exhibit a modest CTE mismatch ($\sim 0.28 \times 10^{-6}$ and 0.86×10^{-6} K^{-1} respectively at 600°C–800°C and 1000°C–1200°C), which minimizes thermal stress and supports interfacial integrity. In contrast, the mismatch with porous ZrB_2 is more pronounced at all temperatures, reaching $\sim 1.1 \times 10^{-6}$ and 2.5×10^{-6} K^{-1} respectively at 600°C–800°C and 1000°C–1200°C. This confirms the interfacial cracking and discontinuous adhesion observed in the Zr-porous ZrB_2 joints.

This study case demonstrates the feasibility of brazing dense ZrB_2 to Zr metal, achieving a robust and continuous bond with no significant gaps at the interface. The successful joining process can be attributed to the similarity in thermal expansion coefficients between Zr and dense ZrB_2, which minimizes stresses during heating and cooling cycles. The continuous interface was observed without measurable discontinuities in the SEM images, along with the absence of delamination or large voids, confirming that the selected brazing parameters were effective in ensuring good wettability and adhesion of the filler material. Additionally, the airtightness test performed under compressed air further validates the mechanical integrity of the joint, highlighting its potential applicability in hypersonic thermal protection systems.[29–34] However, the brazing of porous ZrB_2 presents significant challenges such as thermal expansion mismatch and filler infiltration, which induce a discontinuous bonding of the Zr tube and alter the local material properties, leading to stress accumulation in the ceramic side and generating local cracking while cooling. This issue was exacerbated by the thermal expansion mismatch between Zr and porous ZrB_2. By optimizing various process parameters such as the paste viscosity, the application of interlayers, and the modification of sintering profile, it is possible to improve bonding performance in this case. These developments will be addressed in future work. For the current application, these findings encourage brazing the Zr to the dense ZrB_2 to transfer the coolant into the porous ZrB_2 via Zr from the supply reservoir to mitigate oxidation effects and reduce the component's temperature. On the other hand, these findings suggest that alternative strategies refine the brazing process in this case, such as optimizing paste viscosity and powder mixing, modifying the sintering profile, incorporating advanced filler materials with improved wetting properties,[24,35–37] lowering thermal expansion differentials and introducing interlayer materials to enhance the adhesion and mechanical performance of brazed porous ZrB_2–Zr components. From a broader perspective, the insights gained in this study extend beyond hypersonic applications and are relevant to other extreme environments, such as plasma-facing components used in nuclear fusion systems.[38–42] Additionally, further investigation into the long-term performance of the joints under creep-fatigue thermomechanical loading conditions will be crucial in assessing their reliability for real-world applications.

4 | CONCLUSION

This paper demonstrated the feasibility of the manufacturing process of a UHTC composite by brazing ZrB_2 with Zr tubes, which serve as channels for coolant delivery in the transpiration cooling technology.

A key objective was to establish a continuous and reliable ceramic-to-metal connection, ensuring structural

integrity and performance stability. To achieve this, brazing conditions—including paste composition and sintering temperature—were carefully optimized to promote effective bonding while minimizing adverse effects on the base materials:

- The joint between dense ZrB_2 and the Zr tube exhibited a continuous bond with no significant gaps, attributed to the similar thermal expansion behavior of both materials at brazing and cooling temperatures.
- In contrast, the joint between porous ZrB_2 and the Zr tube displayed discontinuities due to thermal expansion mismatch.
- The presence of Zr as an active element in the filler paste enhanced wettability but also led to paste infiltration into the porous ZrB_2 microstructure, modifying its local properties and potentially compromising mechanical stability.

Beyond hypersonic applications, the insights gained from this study on ceramic–metal joining are also highly relevant to nuclear fusion technology, where materials must withstand extreme thermal loads, neutron irradiation, and plasma exposure. The development of robust ZrB_2–metal joints could contribute to advanced cooling systems and thermal protection barriers in plasma-facing components used in fusion reactors, improving the longevity and performance of structural components in harsh operational environments.

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ORCID

Younes Belrhiti https://orcid.org/0000-0003-4305-1310

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