# Densification and characterization of pressureless sintered ZrB2-20 vol% MoSi2 ultra high temperature ceramic composites

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**Abstract**

In recent times, ultra-high temperature ceramic matrix composites (UHTCMCs) comprising of ultra-high temperature ceramics (UHTCs) such as $ZrB_2$, $HfB_2$ etc. reinforced with additives such as SiC, $MoSi_2$ etc. have been reported to offer tremendous potentials in applications such as Hypersonic Re-entry vehicles, solar thermal absorbers, nuclear fuel claddings etc. Porous ceramics are advantageous in the aforementioned applications areas owing to their high melting points combined with high corrosion and wear resistance. Although $ZrB_2$-SiC UHTCMCs (synthesized using pressureless sintering technique) have been widely investigated for their densification and mechanical properties at both room and elevated temperatures (till 3000°C), there is a very limited information on the densification behaviour and mechanical properties of $ZrB_2$-$MoSi_2$ UHTCMCs prepared using the aforementioned sintering technique. To this end, the present study is aimed at investigating the microstructure and correlating the same with the densification behaviour and mechanical properties of porous sintered $ZrB_2$-20 vol.% $MoSi_2$ (ZM20) UHTCMC. To this end, the composite was ball milled for 2h followed by compaction at different pressures. The compacted samples were sintered at 1700ºC. X-Ray Diffraction (XRD) analysis was performed for the purpose of understanding phase evolution. Subsequent, microstructural investigation was performed under different magnifications in Scanning Electron Microscope (SEM). Moreover, density measurement; hardness and indentation fracture toughness calculations were performed to investigate the room temperature mechanical properties.

**Keywords:** Ultra-high temperature ceramic matrix composites (UHTCMCs), pressureless sintering, X-ray Diffraction, Scanning Electron Microscopy, Vickers microhardness.

Ultra-high temperature ceramics (UHTCs), such as zirconium diboride ($ZrB_2$) and hafnium diboride ($HfB_2$) are a novel class of materials characterized by melting points exceeding 3000ºC. $ZrB_2$-based structural ceramics possess a combination of properties which make them attractive for use in extreme environments [1]. The low electrical resistivity of $ZrB_2$ (6–23 μΩ·cm) contributes to its high thermal conductivity (up to ∼130 W/m·K). and low density (6.14 g/cm$^3$), compared to other UHTCs, renders $ZrB_2$ ceramics as ideal candidates for aerospace components such as engine components and leading edges for hypersonic re-entry vehicles [2]. Other applications include concentrated solar thermal absorbers, nuclear fuel cladding, and neutron absorbers [3]–[6].

Molybdenum disilicide ($MoSi_2$) has been widely investigated as an additive to $ZrB_2$, due to its ability to facilitate densification of $ZrB_2$ at temperatures as low as 1750 °C with or without application of pressure and to improve oxidation resistance of $ZrB_2$ up to ∼1600 °C by forming a glassy borosilicate ($B_2O_3$) surface layer [5], [7–9]. Moreover, $MoSi_2$ has also been reported to enhance the ductility of $ZrB_2$ at elevated temperatures owing to its brittle-to-ductile transition (BDT) upon heating between 900 and 1300 °C. Common sintering routes of ultra-high temperature ceramic matrix composites (UHTCMCs) include hot pressing (HP), pressure-less sintering (PS), spark plasma sintering (SPS) and reactive hot pressing (RHP) [10]–[12]. Even though PS is an attractive route for the fabrication of parts with a high level of design complexity, hot pressing (HP) still remains as the most preferred route due to the high relative density of the final composite [3], [14]-[16].

Porous composites are manufactured because they enable the ceramic product for use in a number of applications such as filtration of molten steel, hot corrosive gases etc. The decomposition of $MoSi_2$ to $Mo_5Si_3$ occurs when $MoSi_2$ is heated to temperatures ranging from 1700° to 2000°C under a vacuum level of $10^{-5}$ mm Hg, leading to structural distortion and subsequent porosity in the microstructure [2],[3]. Porous ceramics are highly advantageous for high-temperature applications due to their high melting point and high corrosion and wear resistance [6]. However, at present, there is limited understanding on the mechanical properties of ZM composites at room temperature. To this end, the present work is aimed at investigating the densification, microstructure, physical and mechanical properties of pressureless sintered porous $ZrB_2$-20vol.% $MoSi_2$ UHTCMCs [6].

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