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An evaluation of the B_4C formation in sintered ZrB_2-SiC ceramic composites at 2100 °C

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ABSTRACT

The present study was concerned with the high temperature sintering behaviour of zirconium diboride ceramics mixed with fine silicon carbide. Zirconium diboride-silicon carbide (ZrB_2-SiC) ceramic composites were pressureless sintered at 2100 °C under flowing argon environment, for a hold time of 150 min. The pressureless sintered ZrB_2-SiC pellets were characterized for microstructure and phase analysis. The sintered microstructure was observed through Scanning Electron Microscopy (SEM) embedded with Energy Dispersive Spectroscopy (EDS) and different phases were analyzed through X-ray Diffraction (XRD). The investigation revealed the presence of boron carbide (B_4C) within the sintered pellets containing 20 mol.% SiC. The B_4C was formed during sintering without the presence of any free carbon as part of initial powder mixture. Possible routes to identify and explain the formation of B_4C in the absence of free carbon were discussed.

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1. Introduction

Zirconium diboride (ZrB_2) has always been chosen for research, either as the main material or as an additive, in mostly ultra-high-temperature ceramics as reported by various researchers [1–8]. The densification of boride ceramics is of particular interest compared to other ceramics due to strong covalent bonding and low self-diffusion coefficient [9]. Because of these properties, silicon carbide (SiC) is added intensively to ZrB_2 as sintering aid or an additive. The addition of SiC improves the oxidation resistance, mechanical properties and enhances the thermal conductivity of the composites [1,2,5,7,10,11]. Moreover, an optimum amount of SiC effectively enhances the densification of ZrB_2 [1,2,5,7]. Both ceramics are remarkably attractive for their ultra-high melting points [3,4,6–8,12].

In most cases ZrB_2 acts as the main ceramic inside the composite, but in some studies, it also has been used as an additive into other ceramics. According to Yang et al., ZrB_2 as an additive also helps to enhance the properties of SiC ceramic by crack deflection and bridging, when added in 5 wt% quantities [13]. On the other hand, in order to improve sintering of ZrB_2, 20 to 30 vol% SiC as an additive has been favourable [3,6–8,12] for composite densification. Boron carbide (B_4C) is also a potential sintering aid to improve the densification of ZrB_2 either by pressureless sintering [3,7,10] or hot pressing as studied by other researchers [3,7,8,14,15]. Generally, B_4C is added within ZrB_2 or ZrB_2-SiC composites sintered at 2000–2300 °C [3,7,8]. B_4C acts to clean the particle surface from oxide impurities and subsequently helps in producing nearly dense ZrB_2 [15]. A study reported by Zhang et al. employs pressureless sintering at 2100 °C, which resulted in ~99.3% densification of ZrB_2-SiC ceramic composites with B_4C and C addition [3].

Recent studies by Harrington et al. have shown that the formation of non-stoichiometric boron carbide ($B_{4.3}C$) takes place in specimens with addition of carbon ≥0.75 wt% during hot-pressing at 2100 °C [1]. Ma et al. reported that 5 vol% of B_4C improves the flexural strength and densification of pressureless sintered ZrB_2 [15]. Without B_4C the highest relative density achieved in sintered ZrB_2 is ~85%.

Previously discussed studies have either employed B_4C as an additive during sintering to promote densification of ZrB_2 or have used C as an additive within the mixture to stabilize B_4C. Therefore, the present work was aimed to identify the formation of B_4C within

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