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Segregation of tungsten atoms at ZrB_2 grain boundaries in strong ZrB_2-SiC-WC ceramics

Hai-Bin Ma [a], Ji Zou [b,*], Jing-Ting Zhu [c], Lei-Feng Liu [b], Guo-Jun Zhang [a,*]

[a] State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, Institute of Functional Materials, Donghua University, Shanghai 201620, China
[b] School of Metallurgy and Materials, University of Birmingham, B15 2TT, UK
[c] SZU-NUS Collaborative Innovation Center for Optoelectronic Science & Technology, International Collaborative Laboratory of 2D Materials for Optoelectronics Science and Technology of Ministry of Education, College of Optoelectronic Engineering, Shenzhen University, Shenzhen 518060, China

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ABSTRACT

Segregation of solute atoms at grain boundaries has been found in many materials and could have a great impact on their properties. However, such important phenomenon has not been observed in early transition metal borides. In the present work, tungsten segregation at grain boundaries was found in ZrB_2-SiC-WC ceramics (ZSW), particularly at ZrB_2/ZrB_2 rather than ZrB_2/ZrC grain boundaries. Besides, densely packed dislocations, mainly in a mixed type, were found in many ZrB_2 grains. The formation mechanism for W segregation and high-density dislocations in ZSW are discussed, both of which are considered to contribute the excellent high-temperature mechanical properties of ZSW.

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Ultra-high temperature ceramics (UHTCs), such as ZrB_2 and HfB_2, are promising candidates for the sharp leading edge and nose cone on the future hypersonic aerospace vehicles, as they exhibit excellent combined chemical-thermal-mechanical properties [1–3]. Severe aerodynamic heating during hypersonic flight are inevitable and can easily result in the temperatures on the sharp components of the aircraft exceeding 2000 °C in a short period. Therefore, these components will have to bear high thermo-mechanical loads for a longer period during reentry, which requires excellent mechanical properties, in terms of flexural strength and creep resistance, at high temperature. Both high-temperature strength and creep resistance of UHTCs are highly relied on the structure and property of their grain boundaries. Firstly, the fracture mode of ZrB_2-based UHTCs changes from transgranular to intergranular with the increase of temperature [4–6] and hence directly affects the flexural strength. Additionally, diffusion creep of polycrystalline materials has to be accompanied by grain-boundary sliding, which accounts for most of the strain during creep process at high temperatures [7–9].

ZrB_2 and ZrB_2-SiC with tungsten carbide (WC) additions have demonstrated the most excellent flexural strength at elevated temperatures in both argon and air atmosphere in the family of UHTCs [10–13], as compared in Fig. S1. ZrB_2-SiC-WC ceramics also show excellent creep resistance compared to their counterparts at 1600 and 1800 °C [14]. One of the positive roles of WC additions on ZrB_2 has been identified [15,16], it could remove different oxide impurities from the ceramic body and results in the ZrB_2 grain boundaries free of amorphous phases, which improves the mechanical properties of ZrB_2-based ceramics at higher temperatures.

Besides, the segregation of W at grain boundaries (GBs) is predicted as another possible reason for the improved mechanical properties. Segregation of solute atoms in the GBs has been found in many material systems, including ceramics and alloys [17–21]. The segregation of W atoms in GB of ZrB_2 ceramic has been predicted via first principles calculation by Zhou et al. In his work, simulations on the GB strength of ZrB_2 doped with different metal atoms were performed via CASTEP with exchange-correlation described by generalized gradient approximation. Three coincident site lattice (CSL) GBs with their rotation axes respectively being [0001], $[11\bar{2}0]/3$ and $[01\bar{1}0]$ of ZrB_2 were selected. The simulation results indicate that the W segregation at the GBs of ZrB_2 improves the strength of GBs by inducing local contractions around GBs due to the short equilibrium W—B bonds [22]. Furthermore, another two separated simulations indicate that dissolution of W into ZrB_2 grains reduces the energy barrier for the dislocation nucleation. This promotes the plastic deformation and hence releases elastic energy accumulated in the materials and suppresses the fracture, which ultimately improves the retention of strength at high temperature

* Corresponding authors.
E-mail addresses: zouji1983@gmail.com, zouji1983@aliyun.com (J. Zou), gjzhang@dhu.edu.cn (G.-J. Zhang).

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[23,24]. However, until now, these hypotheses have not been experimentally validated. On this basis, the aim of the study is to analyze the detailed microstructures of ZrB_2-20SiC ceramics with and without WC additions, particularly at the GB, to understand the complete role of WC on improving the high-temperature mechanical properties of boride ceramics.

ZrB_2-20 vol% SiC (ZS) and ZrB_2-20 vol% SiC-5 vol% WC (ZSW) were consolidated via hot press sintering and detailed information and processes can be found in previous work [10]. The chemistry of GBs and substitution solid solution were investigated by a scanning transmission electron microscope (STEM, Talos, FEI, USA) operating at 200 kV and equipped with energy dispersive X-ray spectroscopy (EDS). The EDS is an FEI Super-X system consisting of four symmetric designed SDDs (Silicon Drift Detectors) with a solid angle of 0.9 srad, an energy resolution of 136 eV for Mn-Ka and 10 k counts per second output. Moreover, EDS maps were collected with a beam current of 300 pA and a collection time of 10–20 min.

At least ten GBs in ZS and ZSW specimens were randomly checked and analyzed. High resolution images of the GB structures in ZS and ZSW were analyzed through a high-resolution transmission electron microscope (HRTEM, JEM-2100F, JEOL, Japan), which was carried out at 200 kV. Dislocations were detected under two beam condition using this TEM. EDS was used to detect the composition of the glassy phase located at the triangular grain boundary of thin ZS slice. Specimens for TEM were prepared conventionally by cutting 3 mm discs from the as-sintered bulk. The specimens were mechanically ground to a thickness of about 20 μm and then further thinned by ion beam milling (Gatan 691, USA) until perforations were observed through an optical microscope.

The removal of ZrO_2 impurities in ZrB_2 based UHTCs by WC addition has been proven in the previous work [12,15,25]. WC addition can also

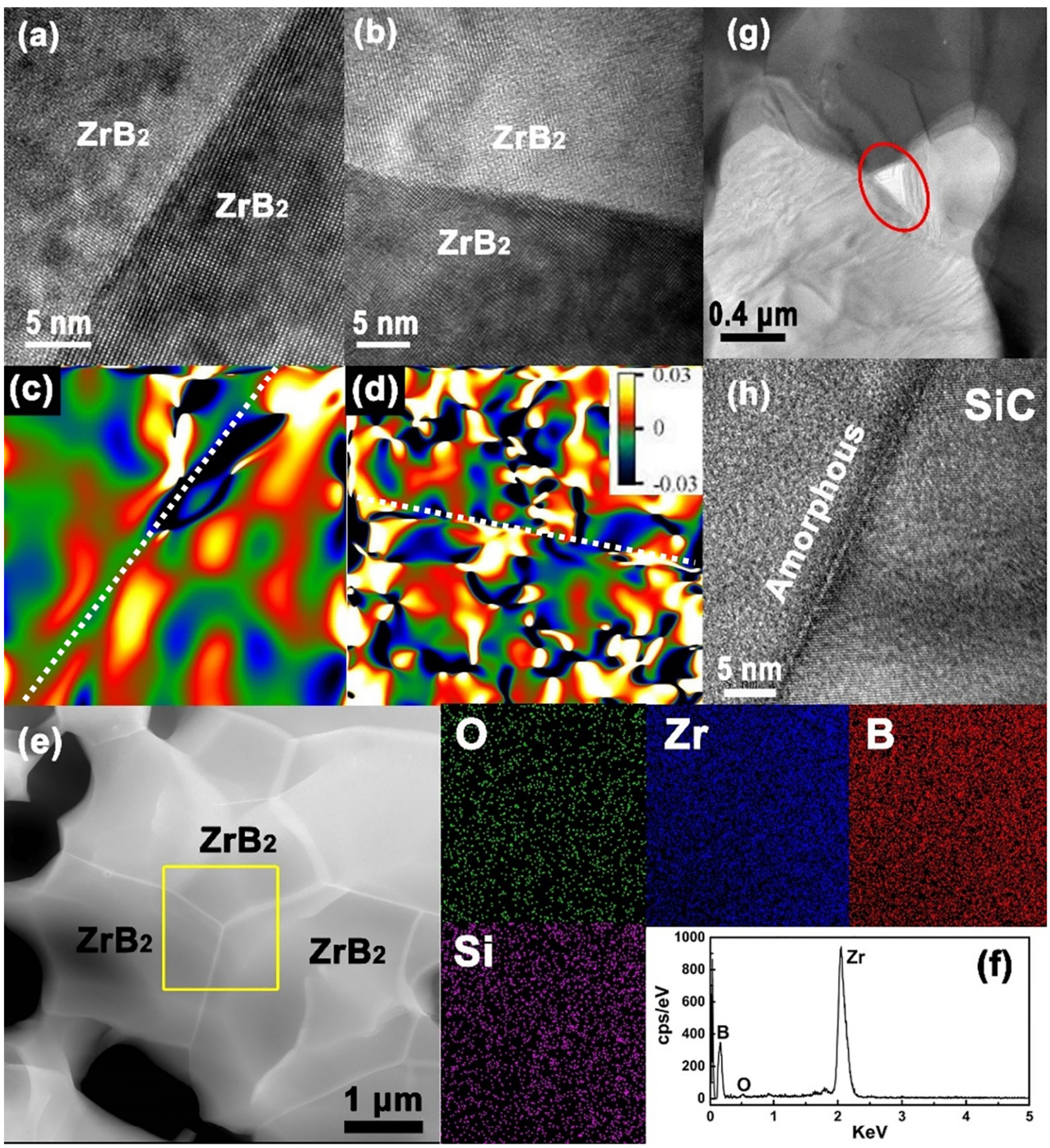

Fig. 1. Typical HRTEM images of ZS (a) and ZSW (b); their strain field images (ε_{xx}) calculated by geometric phase analysis are shown in 1c and 1d, respectively. The colour scales in 1c and 1d are the same (inserted in 1d), indicating the displacement as a fraction of the lattice parameter. High-angle annular dark-field (HAADF) image of ZS (e) and O, Zr, B, Si elemental mappings circled in 1e. The EDS spectrum collected from the area circled in 1e is shown in 1f; Bright field (BF) image of ZS (g) and HRTEM image (h) imply an amorphous nature of the impurities (circled in 1g). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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