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Microstructure and hardness scaling in laser-processed B_4C–TiB_2 eutectic ceramics

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Abstract

Surface layers of the pseudo-binary eutectic comprised of boron carbide (B_4C) and titanium diboride (TiB_2) were directionally solidified via direct laser irradiation in an argon atmosphere. The resulting surface eutectic layers had highly oriented lamellar microstructures, whose scale (i.e. interlamellar spacing) was controlled directly by the laser scan rate, following an inverse square root dependence for lower solidification velocities. Higher velocities (>∼4.2 mm/s) departed from this relationship, although well-ordered microstructures were still achieved. A concomitant increase in the Vickers hardness with decreasing interlamellar spacing was observed, although the trend did not correspond to traditional Hall–Petch behavior. The hardness of the eutectic composites became load-independent at indenter loads greater than 9.81 N, indicating a potential transition from plastic to fractural deformation during indentation. A Vickers hardness of 32 GPa was achieved in the highest solidification velocity samples (42 mm/s) which had interlamellar spacings of 180 nm.
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1. Introduction

Directionally solidified eutectic (DSE) ceramics are of interest for an array of thermo-mechanical applications due to their inherent thermodynamic stability and potential for improved mechanical properties relative to the monolithic end members.[1–6] In addition, the directional solidification process allows for control of the microstructural length scale, which provides some latitude in tailoring the microstructure and properties.[4,6,7] While the majority of ceramic DSE research has been devoted to oxides,[4,6] there is increasing and renewed interest in boride and carbide eutectics because of their very high eutectic temperatures (above 2000 °C), which make them attractive in ultra-high-temperature applications, and for their covalent bonding, which makes them attractive in applications requiring high hardness, including armor and tribological coatings.

Sorrell et al., and more recently Gunjishima et al., reported the directional solidification, microstructure and mechanical properties of several boride/carbide systems, including ZrB_2–ZrC,[1,7] B_4C–SiC[8] and B_4C–TiB_2.[9] These DSEs were grown by a float-zone (FZ) type method, leading to crystallographically and microstructurally well-oriented composites. In the float-zone method, a source material of the eutectic composition is passed through a hot zone at a well-defined rate, allowing for controlled melting and resolidification of the composite. Stubican[10] and Gunjishima,[9] among others,[11] showed that the solidification rate of non-oxide DSE materials has a direct influence on both the resulting microstructure and mechanical properties.

The B_4C–TiB_2 system (75 mol% B_4C),[9,11,12] which displays a lamellar-type eutectic microstructure, is a candidate for armor and tribological coatings where low density and high hardness are stringent requirements. Polotai et al.[11] demonstrated the formation of sub-micron scale B_4C–TiB_2 eutectic surface layers via laser processing of ceramic powders. Several oxide DSEs exhibit an increase in strength with decreasing microstructural scale,[6] and the same has been demonstrated in ZrC–ZrB_2 and ZrC–TiB_2 eutectics by Sorrel et al.[1] This study builds on the work of Polotai et al., investigating further the laser surface processing method, and determining the effect of microstructural scale on the indentation hardness of B_4C–TiB_2 eutectics.

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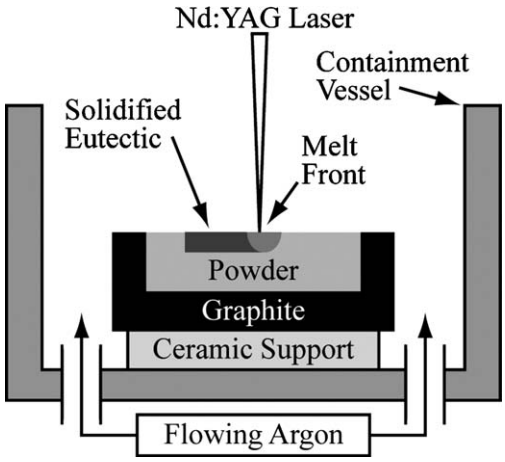

Fig. 1. Laser surface processing schematic.

2. Experimental procedure

Powders of B_4C (ESK, Germany, 96% pure) and TiB_2 (GE Advanced Ceramics, Wilton, CT, 93.5% pure) were combined in a mixture of 25 mol% TiB_2, corresponding to the pseudo-binary eutectic composition.[9,11,12] The powders were shaker-milled in ethanol for 24 h in a low-density polyethylene (LDPE) jar with yttria-stabilized zirconia milling media. The resulting slurry was then dried at 80 °C and passed through a centrifugal mill (Retsch, Germany) to produce a fine particle size suitable for hydraulic pressing. Powders were stored in a sealed LDPE jar and used within 1 week of production to minimize oxidation. Powders were pressed in a graphite crucible using a Carver hydraulic press (Carver Inc., Wabash, IN) and the green density was calculated via the known mass and measured volume of the pressed powder.

Prepared powders were melted and resolidified by scanning a 1064 nm Nd:YAG laser beam (Trumpf Inc., Farmington, CT) over the powder surface. A laser spot of approximately 4 mm in diameter was linearized with an oscillating mirror cycling at approximately 30 Hz. The linearized beam (approximately 12.7 mm long) was then scanned perpendicular to the oscillation direction at laser powers ranging from 500 to 1000 W, as illustrated in Fig. 1. The resolidified coupons were approximately square with 12.7 mm sides. Powder green density, laser power, and processing/resolidification rate were varied to determine relationships between processing variables and microstructure. Materials with the highest solidification rates were grown in the laser processing method described by Polotai et al.[11] No sample backheating was used during the current study, though backheating was used in the experiments performed by Polotai.

To prevent oxidation of the melt pool and resolidified material, processing was performed in an argon atmosphere. A sealed vessel was evacuated with a roughing pump (500 Torr) and backfilled to overpressure with high purity argon gas. This process was repeated three times, after which the top was removed from the vessel. Argon gas was flowed from the bottom of the vessel for the duration of processing and cooling. The containment vessel was evacuated and backfilled with argon as described each time the graphite crucible was changed.

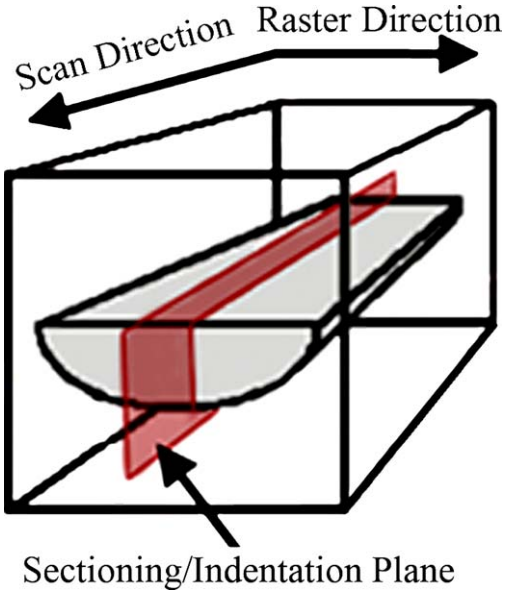

Fig. 2. Post-processing sample sectioning diagram.

The laser-processed coupons were removed from the crucibles and mounted in epoxy to aid in cross-sectioning and polishing. Samples were sectioned along a plane parallel to the growth direction as indicated in Fig. 2. Slices were cut from the remaining coupon with a high speed saw and cross-sectional surfaces were polished with diamond-embedded discs to 1 μm surface finish for observation in the Hitachi 3500-S (Hitachi, Japan) scanning electron microscope (SEM) and for Vickers hardness indentation.

In addition to laser-processed materials, samples of FZ-grown B_4C–TiB_2 were provided by P.I. Loboda and I. Bogmol from the National Technical University of Ukraine (Kiev Polytechnic Institute), grown with a crucible-free floating zone method.[12] Three separate materials were provided with growth rates of 3, 5 and 6 mm/min. These materials were polished to a surface roughness of 1 μm using a South Bay Tech 590 tripod polisher (South Bay Technologies, San Clemente, CA) and diamond lapping films (3M, St. Paul, MN).

Interlamellar spacing of the directionally solidified eutectic materials was measured as the average distance between lamellae. Vickers hardness indentations were performed on a Leica V-100 (Leica, Germany) indentation tester per ASTM C1327-03[13] at loads of 4.91, 9.81 and 19.62 N.

3. Results and discussion

3.1. Microstructure

Cross-sections of the laser-processed materials are analyzed with respect to the microstructural orientation and interlamellar spacing as a function of distance from the free surface. As is evident in Fig. 3, for samples processed at 0.42 mm/s, the orientation of the eutectic lamellae is inclined towards, but not perpendicular to, the sample surface, and there is only a small variation in the interlamellar spacing through the depth of the

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