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Meso-scale microstructural agglomerate quantification in boron carbide using X-ray microcomputed tomography

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ABSTRACT

Failure in brittle materials is governed, in part, by the specific microstructure of that material, with heterogeneous microstructures generally leading to reduced mechanical properties and performance. In ceramics used for armor, an increase in compressive strength can be an indicator of improved ballistic performance. In hot-pressed boron carbide (B_4C) ceramic plates, failure has been attributed to carbonaceous flakes observed on the fracture surfaces of failed specimens. However, this current work shows agglomerates, in addition to the carbonaceous flakes, are present in this boron carbide. The density and distribution of these agglomerates are believed to influence the ballistic performance. Therefore, it is of interest to improve the quantification of meso-scale microstructural agglomerates so that strategies can be developed to mitigate them. This work is focused on the microstructural characterization of dumbbell-shaped compression specimens that were machined from a single tile of pressure-aided densification (PAD) B_4C, using high-resolution micro-computed tomography (microCT). Large clusters of non-B_4C agglomerates, up to 125 μm in diameter, were observed in addition to the typical smaller carbonaceous flakes and aluminum-based phases. Agglomerate size distribution, orientation, morphology, and spacing were quantified both parallel and perpendicular to the pressing direction. This defect quantification provides valuable information for a better understanding of the material microstructure which is a critical step in improving armor materials for the future.

1. Introduction

Boron carbide (B_4C) microstructure and properties in compression have long been of interest since B_4C is an ultra-hard ceramic with a low density ($\rho = 2.54\ g/cm^3$) [1–5]. This makes it ideal for use as a light-weight armor material since hardness is often thought to correlate to ballistic performance [5]. However, due to the additives necessary to produce a fully dense part at reasonable temperatures, even under uniaxial pressure, the B_4C microstructure of armor-grade ceramics is very heterogeneous [1,5,6]. Typically, aluminum nitride (AlN) and a carbonaceous phase are seen on fracture surfaces using light microscopy as identified by energy dispersive spectroscopy (EDS) [1,6]. Attempts to quantify the microstructure have been limited to 2D light microscopy image analysis of fragments produced by mechanical testing as well as scanning electron microscopy (SEM) and EDS analysis on small surface areas [1,6]. However, all of these techniques are limited to the features present on the surface of fractured specimens and can only be performed in 2D. A technique is required to analyze the material in 3D and prior to fracture, to provide additional microstructural details.

Micro-computed tomography systems capable of differentiating among phases with similar X-ray attenuation coefficients at the resolution necessary to differentiate meso-scale microstructural features have become available. This allows for the possibility of examining and quantifying the microstructure of dense materials non-destructively and in three-dimensions, which has been challenging using prior microCT systems due to resolution limitations. While synchrotron- and nano-CT systems with high resolution capabilities are available and have been for some time [7], synchrotron CT is prohibitively expensive requiring beam time at a user facility, and nanoCT has an extremely limited sample size (due to requirement that X-rays be transmitted through the entire sample regardless of imaging area size which is especially difficult at low voltages and high resolutions).

This report describes the analysis of boron carbide dumbbell-shaped specimens, prior to compression strength testing, using microCT. Following initial scans, large agglomerates composed of both AlN and carbon or porosity were observed (see Supplement 1). Since the scale of these agglomerates is much larger than that of the typical individual AlN or carbonaceous flakes often cited in literature [1,6], further quantitative analysis focused solely on these composite features.

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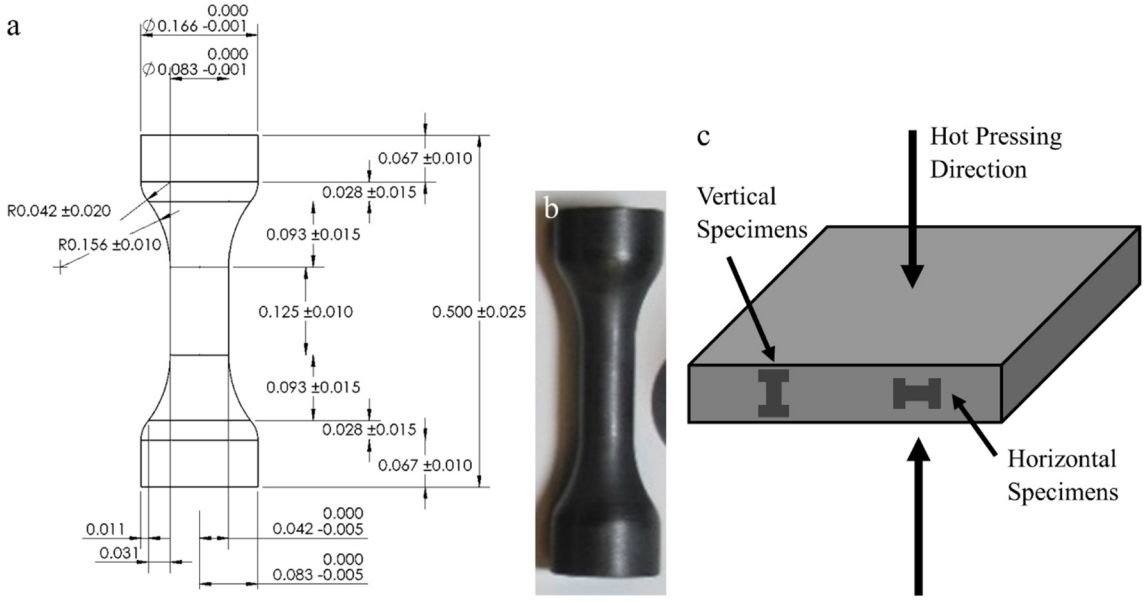

Fig. 1. Schematic (a) and image (b) of machined compression dumbbell samples. (c) Schematic illustration of orientation for each sample group with respect to bulk plate. Dimensions in (a) are in inches.

2. Methods and Materials

2.1. Compression Dumbbell Specimens

All specimens were cut from a single CoorsTek® pressure-aided densification (PAD) boron carbide (B_4C) plate 101.6 mm (4″) square and 12.7 mm (0.5″) thick in dimension. Ten (10) dumbbell-shaped compression specimens, Fig. 1, were machined from the plate. Five (5) specimens were machined parallel to the pressing direction (further referred to as “vertical” specimens), and five (5) specimens of the same dimensions were machined from the plate perpendicular to the pressing direction (further referred to as “horizontal” specimens). The samples were cut in this manner to assess the effect of hot pressing on the microstructure of the final part. All ten (10) specimens were analyzed individually using micro-computed tomography.

2.2. MicroCT Scans

Each specimen was mounted end-on to an aluminum tube of approximately the same diameter as the end of the dumbbell using cyanoacrylate to ensure sample stability during scanning. A Zeiss Xradia 520 Versa MicroCT[1] instrument was used to scan all specimens using the following parameters: 40 kV source voltage, 3 W source power, 8 second exposure time, sample-to-source distance of 7.22 mm, and sample-to-detector distance of 10.00 mm with an LE2 secondary reference source filter. This produced a voxel size of 0.57 μm. A total of 1601 projections were taken on each specimen. Scans were reconstructed using appropriate center shift and beam hardening correction values.

2.3. Image Segmentation

Since the agglomerates observed consisted of a mixture of two phases, images were segmented for both phases, then recombined before performing the analysis. The analysis was conducted by using CTAn (Bruker CT-analyzer, Micro Photonics, Allentown, PA).

First, grayscale, transaxial images or cross sections through the vertical axis of the gauge section, Fig. 2, were then segmented into black, Fig. 3a, and white, Fig. 3b, phases individually by global thresholding.

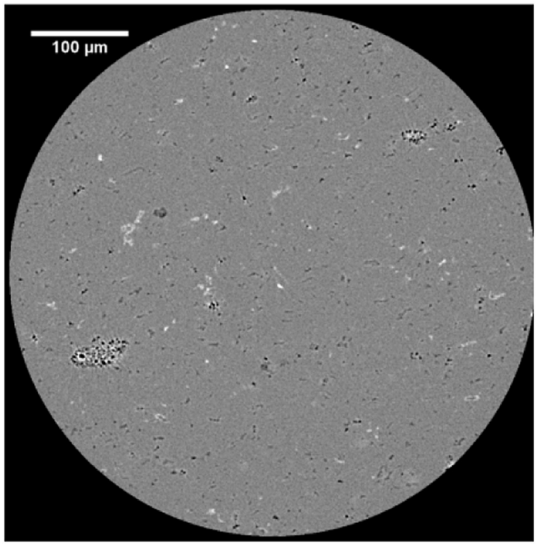

Fig. 2. Grayscale transaxial representative image of quantitative analysis.

The bitmaps produced by global thresholding were then overlaid to produce a mask that isolates both phases simultaneously, Fig. 4.

In order to connect the individual specks produced by the combined mask so that each agglomerate was treated as an individual object instead of a cluster of many smaller individual objects, a dilation (Fig. 5a) and erosion (Fig. 5b) sequence was performed on the combined bitmap. Both dilation and erosion sequences had an equal radius of 5 pixels.

Finally, an image processing technique called “despeckle” was used to remove noise and reduce image processing time. Objects smaller than 150 pixels (approximately 48 μm^2 corresponding to an equivalent sphere radius of 3.9 μm) were removed in 2D and objects smaller than 2000 voxels (approximately 361 μm^3 corresponding to an equivalent sphere radius of 4.4 μm) were removed in 3D. These values were chosen to exclude objects known to be smaller than the average diameter of carbonaceous flakes which are approximately 5–20 μm in diameter [1,6]. Further filtering was performed in MATLAB following analysis in

[1] Mention of specific test equipment, materials, software, or test methodologies does not constitute an official endorsement by the US Army Research Laboratory.