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Determining Grain Size from Backscatter Electron Images

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14. ABSTRACT
A previous literature report by Bennett and Taleff presented a method of measuring grain size using electron backscatter images; if practical, this method will add a new capability to in-house grain size measurements. Determining the grain size of a material is a critical part of characterization because it is correlated to strength and accurately measuring it typically requires capturing images that show the grain boundaries. For some materials (i.e., ceramics), it is not easy to image the grain boundaries using traditional methods such as optical microscopy. Diffraction-based methods, such as electron backscatter diffraction, are also difficult for some ceramic samples due to either inconsistent or unknown crystal structures. Capturing backscatter electron images is an effective and quick method of imaging grains in many materials; however, there must be a method to quantify the grains from the grayscale images. In this work, the method reported by Bennett and Taleff is used to measure the grain size for silicon carbide to determine the effectiveness for future use.

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1. Introduction

Grain size is probably the most common microstructural feature reported for materials when discussing their mechanical properties, but to measure grain size there must first be images of the grains.[1,2] Typical methods of imaging grains involve either optical microscopy or electron backscatter diffraction (EBSD).[3,4] Although these are capable techniques, there are some cases in which they are not practical due to material constraints. For example, ceramic materials can be resistant to traditional chemical etching that reveal grain boundaries for optical microscopy and instead require either thermal or plasma etching or immersion in molten salts, both of which require special equipment.[5,6] EBSD is becoming a more common technique for measuring grain size.[4] This technique is performed inside of a scanning electron microscope (SEM) and does require additional detectors, software, expertise, and sample preparation. In fact, sample preparation is the most important aspect because poor surface quality will negatively affect the intensity of the diffracted beams. Sample preparation of superhard materials, such as ceramics, for EBSD requires mechanical polishing for long periods of time. In addition, the crystal structure of some ceramics makes it difficult to consistently index using EBSD, which makes grain size determination difficult.[7]

Another possible method of obtaining images of grains is backscatter electron imaging (BSE) in an SEM. This technique utilizes SEM images where the signal is formed from backscatter electrons generated by electron channeling. Grains with crystals aligned with the incident beam will channel more effectively and result in less signal (i.e., appear black), whereas grains with crystals that are not aligned with the incident beam will not channel and generate higher backscatter signal (i.e., appear whiter). Due to channeling differences in neighboring grains, the boundaries are clearly visible in these types of images. Identifying and marking the boundaries in the backscatter image makes it possible to measure the grain size. Unfortunately, although the boundaries are easily identified by eye, having a computer find them is difficult. For example, edge finding algorithms (i.e., Sobel and Roberts) do not consistently identify the boundaries.

A recent report published by Bennett and Taleff[8] discusses a method that captures BSE images at different tilt angles, combines the grayscale images into a single, colored image, and then utilizes a modified Felzenszwalb and Huttenlocher[9] (FH) algorithm to identify the boundaries. If successful, this method could be a more effective option for measuring the grain size of ceramics, without needing the extra sample preparation required for EBSD or the specialized equipment for etching. The work in this report is presented to confirm whether the process reported by Bennett and Taleff[8] can be used in house for grain size measurements.

2. Experimental

The material used for this work is Hexoloy silicon carbide (SiC) from Saint-Gobain Refractories, an alpha phase, pressureless sintered SiC that is mounted in poly-fast hot mount epoxy and mechanically polished to a 1.0-µm surface finish.

Backscatter electron images are taken using all segments of the retractable-segmented backscatter detector installed on a Thermo Fisher Scientific APREO SEM operating at a beam voltage of 5 kV and beam current of 3.2 nA. The contrast and brightness of the image is adjusted to highlight the grain contrast. To reduce the noise and make each grain a consistent grayscale, the resolution of the images is set to 1536 × 1094, and the dwell time is 100 µs. To influence the channeling contrast of the grains, an image is collected at three different stage tilt positions: −1°, 0°, and 1°.

After the three images are collected, the grain size analysis follows the procedure detailed in and using the Python code provided in the report by Bennett and Taff,[8] an overview of which is shown in Figure 1.[8] First, the grayscale images taken at different stage tilts are read into the python script, and an alignment step ensures the images are of the same area. Each image represents one of the three Red–Green–Blue channels needed to create a colored image. A colored image is created from these three grayscale images by using the numpy Python module's "stack" function to output the new single image array. Second, a modified FH[9] algorithm identifies the grain boundaries from the colored image. Once identified, the image pixels at boundaries are marked as Boolean values, where boundaries are True (white) and non-boundary or grain pixels are False (black). From this black and white boundary marked image, it is possible to measure grain properties using the Scikit Image "regionprops" function. This function can provide quantitative value for many properties, but the following factors are primarily of interest for the grains in this work: area, equivalent circle diameter, maximum diameter, minimum diameter, and maximum ferret diameter. The average grain size measured from this new method is compared to the grain size calculated using the ASTM-E112[10] line intercept method.

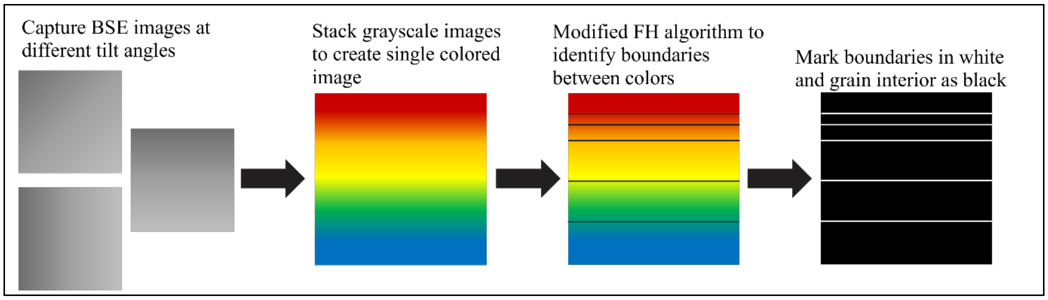

Figure 1. Schematic representing the process for identifying and marking the grain boundaries from backscatter electron images.

3. Results

Figures 2A and 2D show the BSE images and resulting colorized image for the Hexoloy sample. The backscatter electron images are taken at stage tilt angles of 0° (Figure 2A), −1° (Figure 2B), and 1° (Figure 2C). Even these small tilt angles result in noticeable changes in the grayscale values for some of the grains. For example, the grain outlined in the red circle in the BSE images looks very different when tilted between 0° and −1°. There is enough of a difference in the grayscale values between these three images that when used to create a colored image, each grain is a different color, as shown in Figure 2D. The grain circled in the grayscale images appears purple when the images are colorized.

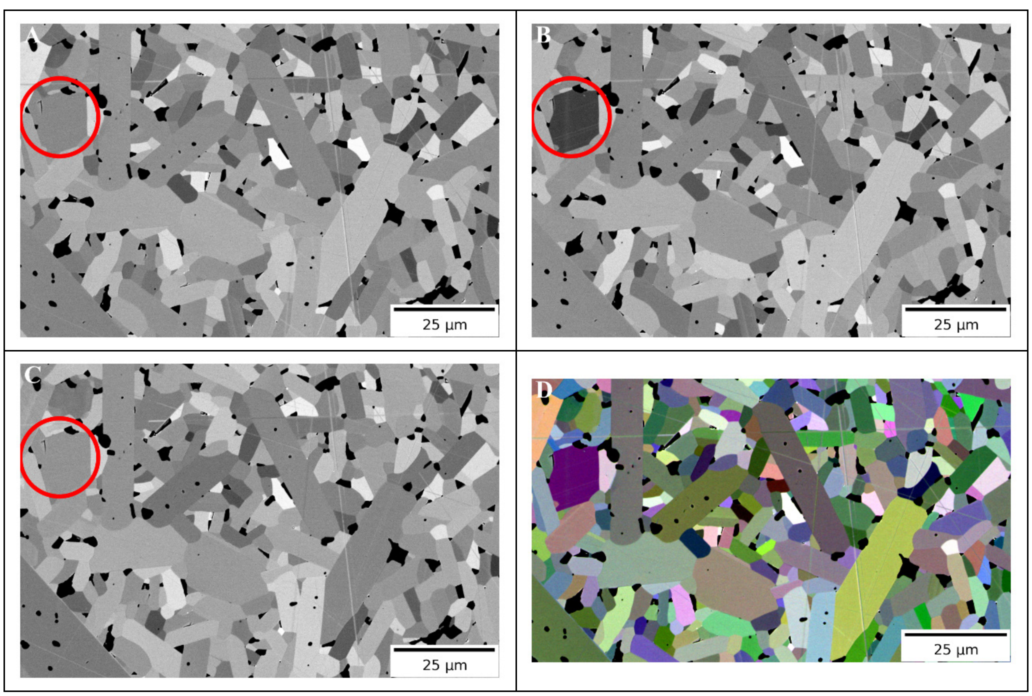

Figure 2. Backscatter electron images of Hexoloy taken at 0° (A), −1° (B), and 1° (C) stage tilt and used to generate the colored image (D).

Figures 3A and 3B show the results of the modified FH algorithm and boundary marking, respectively. The overlay of the boundaries on the colored image (Figure 3A) shows that the algorithm successfully identifies the grain boundaries observed in the BSE images. The boundary image in Figure 3B shows only the identified boundaries, and it is clear to see that it very closely matches the grain boundaries. This sample has grains of varying sizes, and the algorithm can distinguish them all. However, there are some features that are not grains that the algorithm identifies incorrectly. First, there are two large scratches across the surface, which the algorithm identifies as boundaries. This means the larger grains get sectioned into smaller sizes than they are. Second, within those larger grains and at some of the grain boundaries, there are regions that appear as black spots. These are most likely regions of excess carbon, which are still being included as boundaries in this process. Figure 4 shows the results of the area weighted grain size measured from the boundary image in Figure 3B. The distribution plot shows most of the grains fall in approximately the 5.0-µm range, but there are a few grains in the 10- to 20-µm diameter range, which makes the average 8.62 µm, represented by the vertical dashed line. The ASTM-E112[10] line intercept method gives an average grain size value of 4.79 µm, which matches with the peak of grain size distributions using the BSE method.

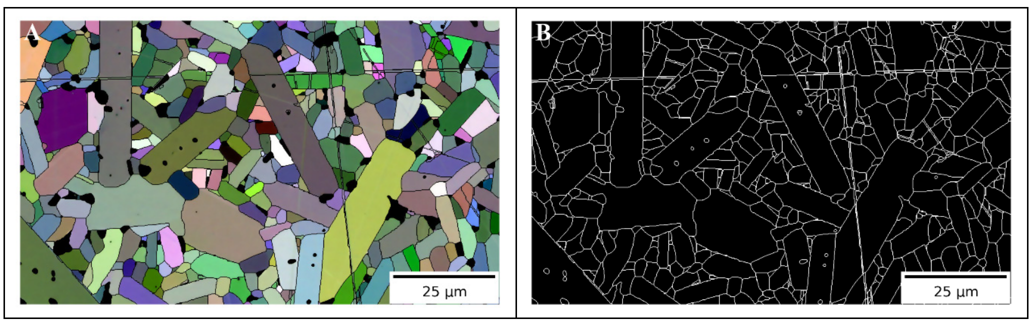

Figure 3. The colored image overlayed with the results of the modified FH algorithm (A) and an image showing just the isolated boundaries (B).

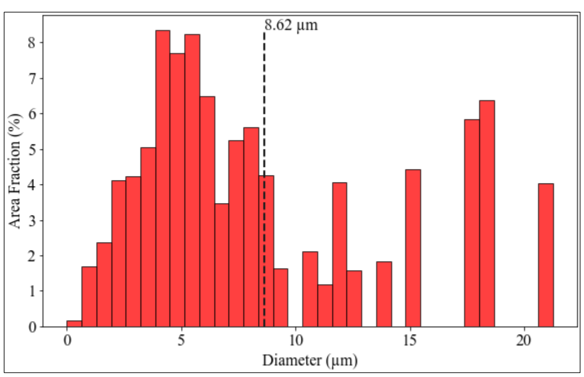

Figure 4. Plot showing the distribution of area weighted grain diameter taken from the current process.

4. Conclusion

Capturing BSE images of ceramics using the retractable detector on the APREO SEM effectively reveals their grain structure without the need for special sample preparation or imaging techniques. Following the procedure outlined in Bennett and Taleff,[8] it is possible to use these BSE images to accurately measure the grain size without the need for EBSD. This technique is advantageous for ceramic samples because the current methods of measuring grain size require manually marking boundaries on captured images. In addition, capturing BSE images requires less user training overall and is ideally suited for automation.

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List of Symbols, Abbreviations, and Acronyms

ASTM	American Society for Testing and Materials
BSE	Backscatter Electron Imaging
EBSD	Electron Backscatter Diffraction
FH	Felzenszwalb and Huttenlocher
SEM	Scanning Electron Microscope
SiC	Silicon Carbide

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