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Time Reduction of the Polishing Procedure for B_4C–SiC Layered Composites

by Tucker W Moore, Kristopher D Behler, and Jerry C LaSalvia

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14. ABSTRACT
Specimens made from a boron carbide and silicon carbide composite were polished using standard metallographic techniques. Scanning electron microscope images were taken at regular intervals during polishing to determine the minimum time for each polishing step before moving on. Using these images, a new procedure was created that reduced the time spent to polish a sample by 45% from the previous method. The new procedure was verified by comparing scanning electron microscopy images of the final polished surface to those of the previous, lengthier procedure. No significant differences were found, so the new procedure was considered suitable for use.

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Contents

List of Figures iv

List of Tables iv

Acknowledgments v

1. Introduction 1

2. Methods 2

3. Results and Discussion 5

4. Conclusions 12

5. References 13

List of Symbols, Abbreviations, and Acronyms 14

Distribution List 15

List of Figures

Fig. 1 Schematic of specimen composition and configuration 3

Fig. 2 SEM images taken after grinding, rough polishing, and final polishing steps for the full time for each step listed in Procedure B. Images A–C taken with BSD, images D–K taken with SED. A) 250-µm grinding pad, B) 63-µm grinding pad, C) 25-µm grinding pad, D) 30-µm diamond slurry, E) 15-µm diamond slurry, F) 9-µm diamond slurry, G) 6-µm diamond slurry, H) 3-µm diamond slurry, I) 1-µm diamond slurry, J) 0.25-µm diamond slurry, and K) 0.02-µm colloidal silica. ... 6

Fig. 3 B_4C/SiC blend after 15-µm diamond grinding 7

Fig. 4 Blend/SiC during 15-µm grinding. Images have very high contrast and negative coloring to highlight the surface porosity created by pullout. A) 30 min, B) 60 min, C) 90 min, D) 120 min, E) 150 min, and F) 180 min. 8

Fig. 5 SiC after CMP with colloidal silica. A) SED and B) BSD. 9

Fig. 6 B_4C/SiC blend after CMP using each polishing procedure. A) Procedure A, B) Procedure B, and C) Procedure C. 10

Fig. 7 Comparison of each layer interface before and after CMP. A) B_4C/blend no CMP, B) B_4C/blend with CMP, C) blend/SiC no CMP, and D) blend/SiC with CMP. 11

List of Tables

Table 1 Polishing procedure details 4

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1. Introduction

Metallographic techniques such as grinding, polishing, and etching are widely used in the field of materials science to prepare the surfaces of material specimens for various characterization techniques, including optical microscopy, electron microscopy, and hardness testing. These techniques sharpen and clarify microstructural features, revealing grains, grain boundaries, second phases, pores, and more.[1] Despite the name, metallography can be applied to all classes of materials, which has led to the coining of alternate terms such as ceramography and plastography to cover the "materialographic" preparation of ceramics and polymers.[2] These names do not merely come from ceramists and polymer scientists wanting more representative terminology, but rather because the particulars of preparing material specimens can vary immensely between different material classes. While the preparation of polymer samples can often be accomplished relatively quickly and easily due to their soft nature, ceramics are often much harder, leading to longer and more difficult preparations.[2]

As the extreme case, preparing diamond requires an arduous, extensive process because it has a very high hardness value (60–100+ GPa).[3] Using typical abrasive techniques is extremely slow as the only media capable of abrading diamond is diamond itself, which will remove material more slowly than is generally desired. Armor ceramics, such as boron carbide (B_4C) and silicon carbide (SiC), have similar difficulties because they also have extreme hardness values (25–40 GPa).[3] As such, diamond is also used as polishing media for these materials as well during ceramographic preparation.[4]

Composite materials provide further challenges for polishing. If the different constituent materials have different hardness values, they will be abraded away at different rates, leading to a feature known as relief.[5] This can cause issues wherein the surface will be recessed out of plane at the sites of the softer material. When viewing a sample under a microscope, this will lead to the recessed areas being out of focus from the rest of the image, reducing clarity of the image.

Ceramic-filled polymer tapes with high solids loading (>40 vol%) is a flexible method for producing layered ceramic composite green bodies of various compositions and layered architectures. In this method, a high volume percentage of ceramic powder (homogeneous or heterogeneous) is mixed into a polymer gel, which is then melt-pressed into "tapes" of desired thickness. These can be stacked into particular layered arrangements and pressed to form the green body. After extraction of the polymer gel, the body can be densified by pressureless or pressure-assisted sintering. Making layered composites based on B_4C and SiC using this

method is of particular interest for armor applications, and metallographic preparation of material variants based on these ceramics is the focus of this study.

Microstructural and chemical characterization of armor ceramics during the materials development stage is often accomplished through scanning electron microscopy (SEM)/electron backscatter diffraction (EBSD)/X-ray energy dispersive spectroscopy (XEDS) and Knoop or Vickers hardness testing. Quantitative SEM/EBSD/XEDS characterization requires a flat, highly polished surface that is parallel to the backside of the sample to be able to take clear, in-focus micrographs with distinct grain and phase boundaries. For imaging, scratches on the surface should be made fine enough to not be visible at magnifications required for observing the microstructure.[2] For hardness testing, the smoothness of the surface is key because the indenter can interact with any surface imperfections, skewing measurements away from a value that is truly representative of the material.[6] To counter this effect, the ASTM standard for hardness testing of advanced ceramics requires that specimens be polished to a surface finish of 1 µm or finer.[7,8]

Thus, as part of the research into ceramic tapes, a multitude of fabricated specimens will need to be polished, but a defined procedure for doing so has not yet been determined. Prior to the present study, specimens of these materials were prepared using an extensive polishing procedure. Polishing longer than necessary was a safe way to ensure that samples had an adequate surface finish as no previous efforts have been undertaken to optimize the process. A more efficient procedure that can still yield a surface finish suitable for characterization is desired.

2. Methods

A hot-pressed specimen made using the ceramic-filled polymer tapes approach, consisting of three distinct layers of B_4C, a blend of B_4C and SiC, and SiC was polished for this study, with densified thicknesses of 1.7, 1.5, and 0.7 mm, respectively, as shown in Fig. 1. The SiC contains 3 wt% Al_2O_3 and 1 wt% B_4C, while the B_4C material contains 2 wt% Al_2O_3 as sintering aids. The blend is a 50/50 mixture of these resulting materials based on volume percent. The sample was sectioned using a low-speed saw (Allied Techcut 4) at 80 rpm with a diamond-embedded wafering blade (Allied 5" Diamond Metal Bond, Low Concentration), exposing a cross section with an area of 85.2 mm^2. It was then mounted into a 38.1-mm-diameter mounting cup using an epoxy (Allied EpoxySet), with the cross section flush with the face of the epoxy disc. Once the epoxy had cured, the newly formed disc was pushed out of the cup. The edges of the disc were chamfered and the face of the disc was ground with a 250-µm grinding disc (TBW

Industries Grid-Abrade, Standard Diamond, 12”) to fully expose the samples surface through the epoxy. Subsequently, the sample was ground finer with 63-µm (Allied Dia-Grid RIGID, 220 Grit Resin Bond, 12”) and 25-µm (Allied Dia-Grid Diamond, 25 µm Resin Bond, 12”) diamond grinding discs to remove damage from sectioning and coarse grinding. Each grinding step was performed on an auto polishing unit (Allied MetPrep 4) using deionized water as lubricant. A 3-lbf load was applied, the platen holding the grinding disc was spun at 60 rpm, and the sample was spun in the opposite direction at 90 rpm. After each grinding step was completed, a series of SEM images were taken. Each time the sample was removed from the polisher, it was rinsed with deionized water and patted dry with a tech-wipe for observation.

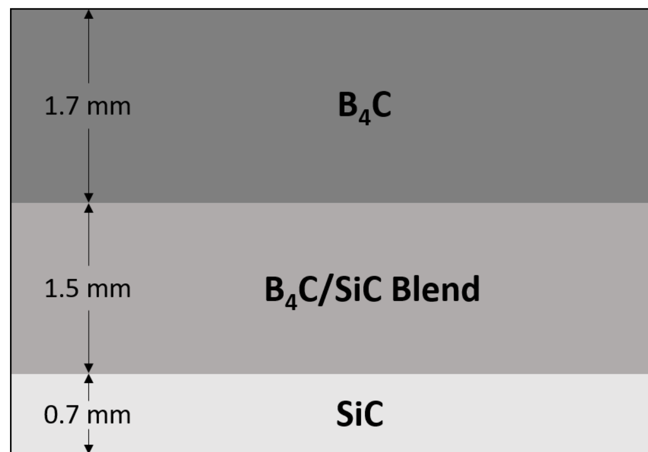

Fig. 1 Schematic of specimen composition and configuration

The sample was then moved to a series of lapping/polishing machines (Lapmaster Wolters Model 12), where the sample was polished using a mixture of progressively finer diamond slurries (Allied DiaLube) from 30 µm down to 0.25 µm and a water-based lubricant (Struers DP-Lubricant Green) dispensed onto various polishing cloths as noted in Table 1. Each polishing cloth was used only for one step of polishing to prevent contamination. The sample was mounted to a disc as part of the Lapmaster system with double sided tape (3M VHB). The platen was spun at 30 rpm with a 5-lbf load. Diamond slurry was dispensed for periods of 2 s at 60-s intervals, while a lubricant was dispensed for periods of 2 s at 90-s intervals. The polishing cloths were sprayed with the diamond slurry and lubricant to saturate the polishing cloth before starting any polishing step. The sample was removed from the polisher at 30-min intervals for SEM analysis. Again, each time the sample was removed, it was rinsed with deionized water and patted dry with a tech-wipe for observation. If little to no change in the size or quantity of surface features was observed based on SEM analysis between 60 and 90 min of polishing, the sample

was moved to the next size down diamond slurry. Otherwise, the sample was polished for 3 h per slurry particle size. The polishing times initially used for each diamond slurry are listed in Table 1 as part of Procedure B. After polishing to a 0.25-µm finish, the sample was chemomechanically polished with 0.02-µm colloidal silica (Allied Colloidal Silica Suspension, Non-Crystallizing, 0.02 Micron) for 15 min using the same rotational speed and load as in the polishing steps. However, the platen was sprayed with the colloidal silica at 3-min intervals using a hand spray bottle instead of using the automatic dispenser.

Table 1 Polishing procedure details

Media particle size (µm)	Media type	Polishing cloth type	Duration (min)		
			Procedure A[a]	Procedure B	Procedure C
250[b]	Diamond impregnated metal	TBW Grid-Abrade	. . .	. . .	. . .
63	Diamond impregnated resin	Allied Dia-Grid Resin Bond	60	60	60
25	Diamond impregnated resin	Allied Dia-Grid Rigid Resin Bond	60	60	60
30	Diamond slurry	Allied Gold Label	180	180	90
15	Diamond slurry	Allied Gold Label	180	180	90
9	Diamond slurry	Allied Gold Label	180	90	90
6	Diamond slurry	Allied White Label	180	90	90
3	Diamond slurry	Allied White Label	180	90	90
1	Diamond slurry	Allied White Label	180	180	90
0.25	Diamond slurry	Allied Red Final C	180	180	90
0.02	Colloidal Silica	Allied Red Final C	15	15	15
Total			1395 (23.25 h)	1125 (18.75 h)	765 (12.75 h)

[a] Procedure A is an estimate of the typical procedure used to polish B_4C-SiC composites before this study was conducted.

[b] The 250-µm grinding step was not carried out for a specific period of time. Instead, it was carried out until the sample was exposed through the mounting epoxy.

Images of the cross-section surface were taken on an SEM (Phenom XL Desktop SEM) using a 10-kV electron source at a variety of magnifications ranging from 250× to 20,000×. Two different methods were used for choosing locations to take images. First, within each layer of the sample, a representative region was found, and one surface feature in the region was centered in the image. Second, at the interface between each layer, a surface feature near a particular crack in the sample was centered in the image to longitudinally track particular regions of the sample

throughout the study. As a result of this systematic approach, over 1,200 SEM images were taken in the course of the study.*

SEM images were initially taken using the backscatter electron detector (BSD). However, this detector provided insufficient topographical contrast for inspection of surface features. The secondary electron detector (SED) was used instead beginning with the 30-µm step. Starting on the 3-µm step, both BSD and SED images were taken to show a worst-case scenario with SED highlighting surface features, as well as a more typical case with BSD somewhat subduing surface features.

Based on observations found during analysis of these SEM images, Procedure C was devised. As indicated by Table 1, this procedure shortened each polishing step to 90 min. A second cross section with an area of 72.3 mm^2 was taken from the same hot-pressed sample, and was polished using Procedure C to verify that it yielded similar results to Procedure A. SEM images using both BSD and SED were taken after polishing was completed, using the same method for image location as before.

3. Results and Discussion

As shown in Fig. 2, drastic changes to the surface of the sample were observed during grinding and polishing. In all of the unedited SEM images presented, the dark material is B_4C, the light material is SiC, and bright spots on the image are areas of sharp topology. The white regions are due to the presence of Al_2O_3, which was used as a sintering aid for both B_4C and SiC. Toward the beginning of grinding and polishing, a phenomenon known as "pull-out" was observed in all three layers of the sample, but most noticeably in the blend section. Pull-out is the removal of grains from the surrounding matrix during the polishing of brittle materials due to wear-induced cracks spreading along grain boundaries.[9] This phenomenon is more common when the size of the abrasive particles are larger than the grains. Once the particles are smaller than the grain size, a transition from grain removal to transgranular fracture may occur.[10]

* The full set of images is available from the authors upon request.

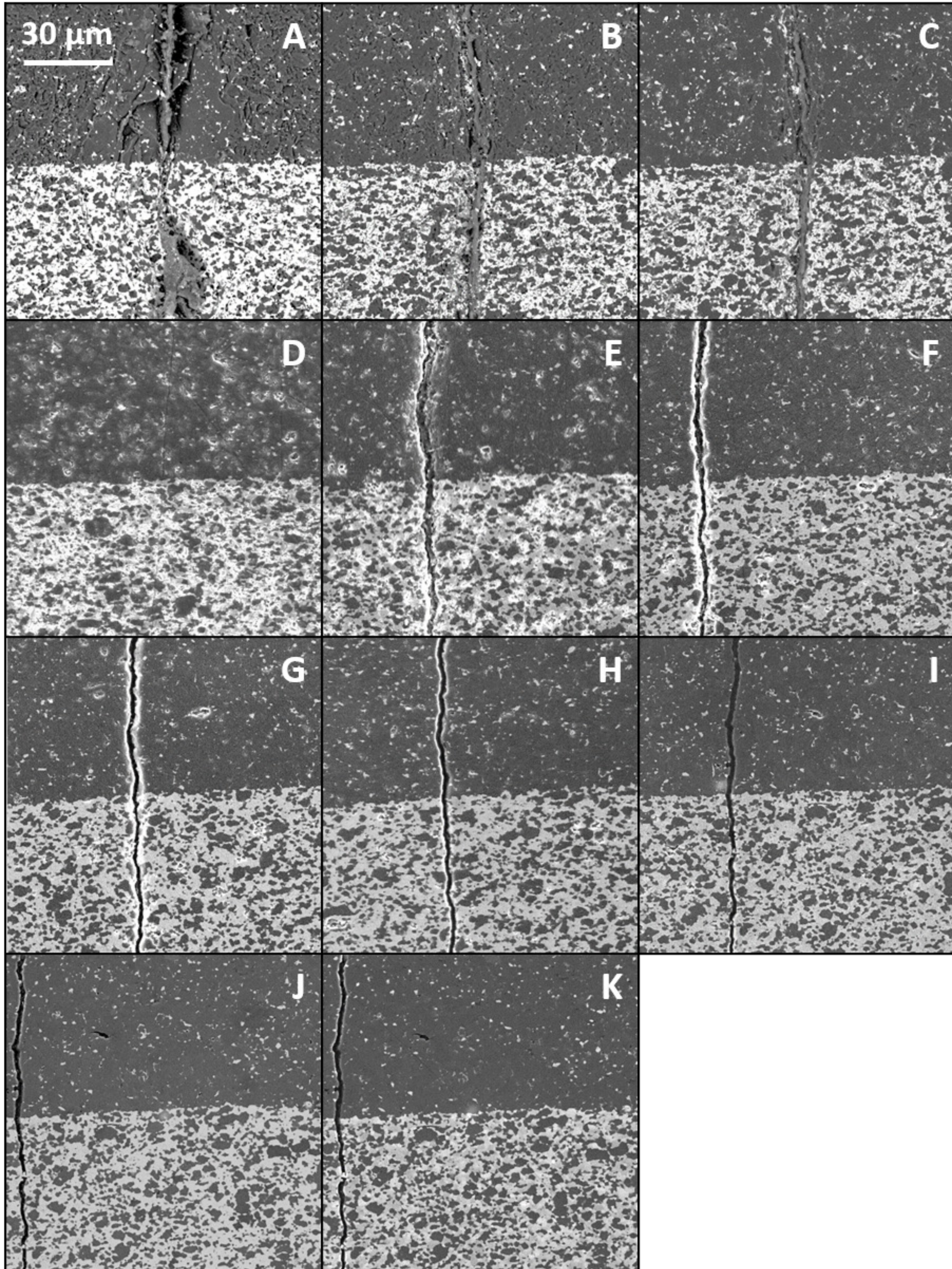

Fig. 2 SEM images taken after grinding, rough polishing, and final polishing steps for the full time for each step listed in Procedure B. Images A–C taken with BSD, images D–K taken with SED. A) 250-µm grinding pad, B) 63-µm grinding pad, C) 25-µm grinding pad, D) 30-µm diamond slurry, E) 15-µm diamond slurry, F) 9-µm diamond slurry, G) 6-µm diamond slurry, H) 3-µm diamond slurry, I) 1-µm diamond slurry, J) 0.25-µm diamond slurry, and K) 0.02-µm colloidal silica.

During the grinding steps using media from 250 to 9 μm (Fig. 2A–F), the largest surface features are the pores created by this pull-out. Figure 3 shows that at a 15-μm finish, these pores can be many times larger than even the deepest scratches created by the same abrasive. In the blend section, the pores are actually larger than the grain sizes of either base material, and thus are not typical single grain pull-out. This suggests that pockets of B_4C, many of which may be polycrystalline, are being removed from the SiC matrix. While it is difficult to determine the grain size in the B_4C layer due to poor contrast between grains, it can be seen that the SiC layer contains much larger grains than the SiC in the blend layer. These grains are closer to the size of the pores, so single grain pull-out seems more likely.

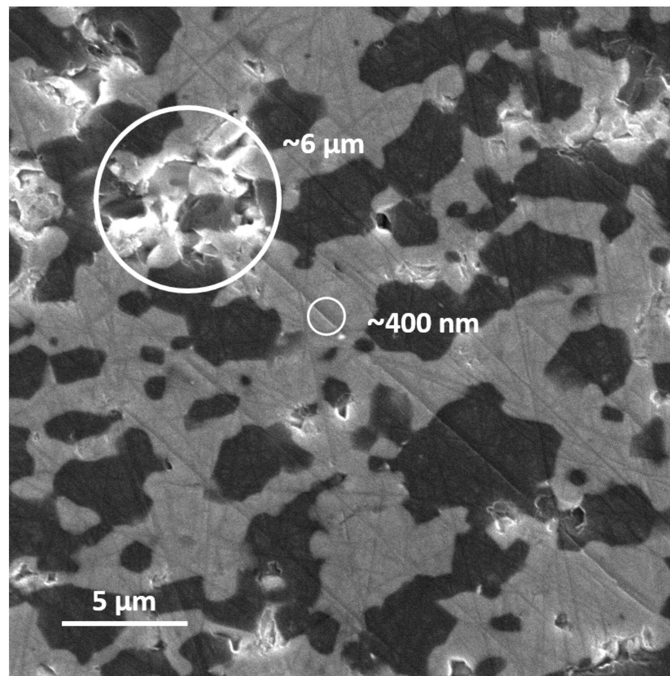

Fig. 3 B_4C/SiC blend after 15-μm diamond grinding

Regardless of whether the pull-out is single grain or not, the surface porosity must be removed by abrasion in order to produce a smooth surface. Figure 4 shows the progression during the 15-μm grinding step, with each image taken after a 30-min period of grinding. To emphasize the appearance of the surface porosity, the images were adjusted to have a very high contrast using Microsoft Photos, then were run through a negative color filter using Microsoft Paint, making the surface pores and cracks the only visible features. During the first 90 min of polishing, a great reduction in the size and number of pores is observed. However, from 90 to 180 min, the amount of surface porosity remains relatively consistent, though new pores do still appear to be created from continued pull-out. This suggests that a steady-state condition is achieved where the abrasive is creating as much new pull-out as it is removing. Similar results were found at each grinding step down to 9 μm,

when the abrasive appears fine enough for no significant amount of pull-out to occur. Reducing the loads and rotational speeds used for the automated polishing could potentially reduce the amount of pull-out at all media levels. However, doing so would likely result in longer polishing times, which is contrary to the goals of this study. As no remnants of the pull-out are seen in the end results, the authors deemed it unnecessary to investigate further.

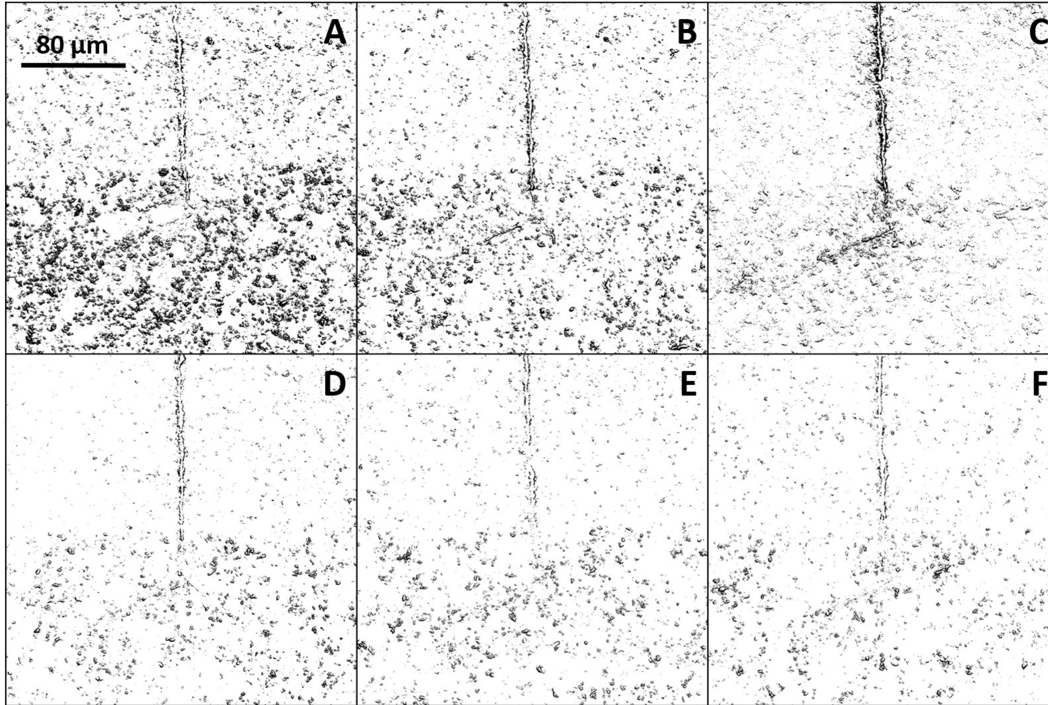

Fig. 4 Blend/SiC during 15-µm grinding. Images have very high contrast and negative coloring to highlight the surface porosity created by pullout. A) 30 min, B) 60 min, C) 90 min, D) 120 min, E) 150 min, and F) 180 min.

Figures 2G–K show that the pull-out has mostly been eliminated beginning with the 6-µm polish. The dominant surface features become the significantly finer scratches, resulting in a much more optically reflective surface. Even at the early, coarse stage shown in Fig. 3, the scratches are very small, with the largest measuring approximately 400 nm wide. At the magnifications and resolution available on the Phenom XL SEM, it is difficult to measure the small differences in the size of scratches between steps. However, once the sample was fully polished with chemomechanical polishing (CMP), the largest visible scratches were approximately 180 nm wide and were only visible using SED on the SEM, as shown in Fig. 5. Using BSD, no scratches are observed, making the surface finish satisfactory for most microscopy needs such as imaging and basic XEDS, as well as Vickers and Knoop hardness testing.[7,8]

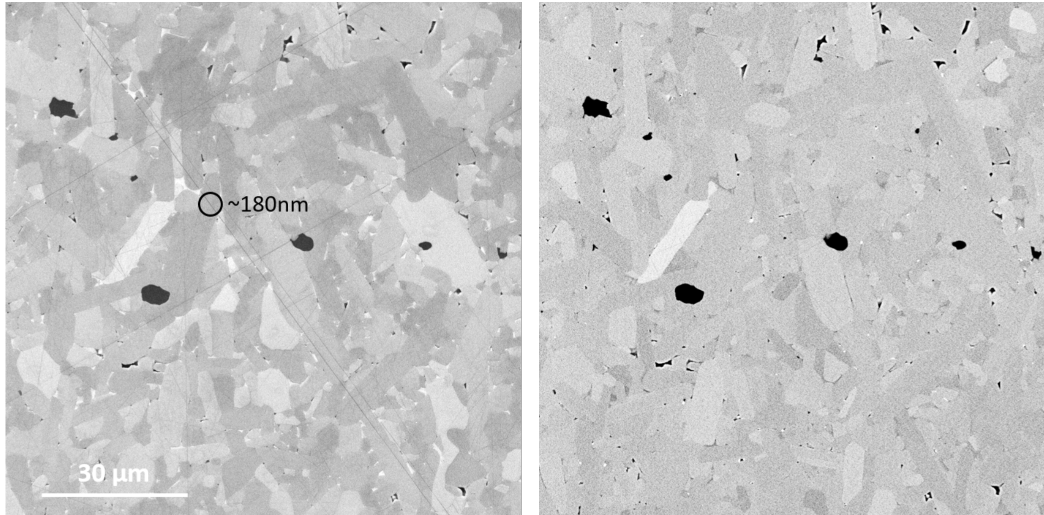

Fig. 5 SiC after CMP with colloidal silica. A) SED and B) BSD.

Figure 6 compares SEM images of the blend section of the material with each of the three polishing procedures. The image shown in Fig. 6A is of a different sample than previously reported, consisting of only a 50/50 vol% B_4C/SiC blend, polished using Procedure A. There is no noticeable difference in the clarity of the image among any of the three procedures, indicating that Procedures B and C are sufficient for obtaining high-quality SEM images.

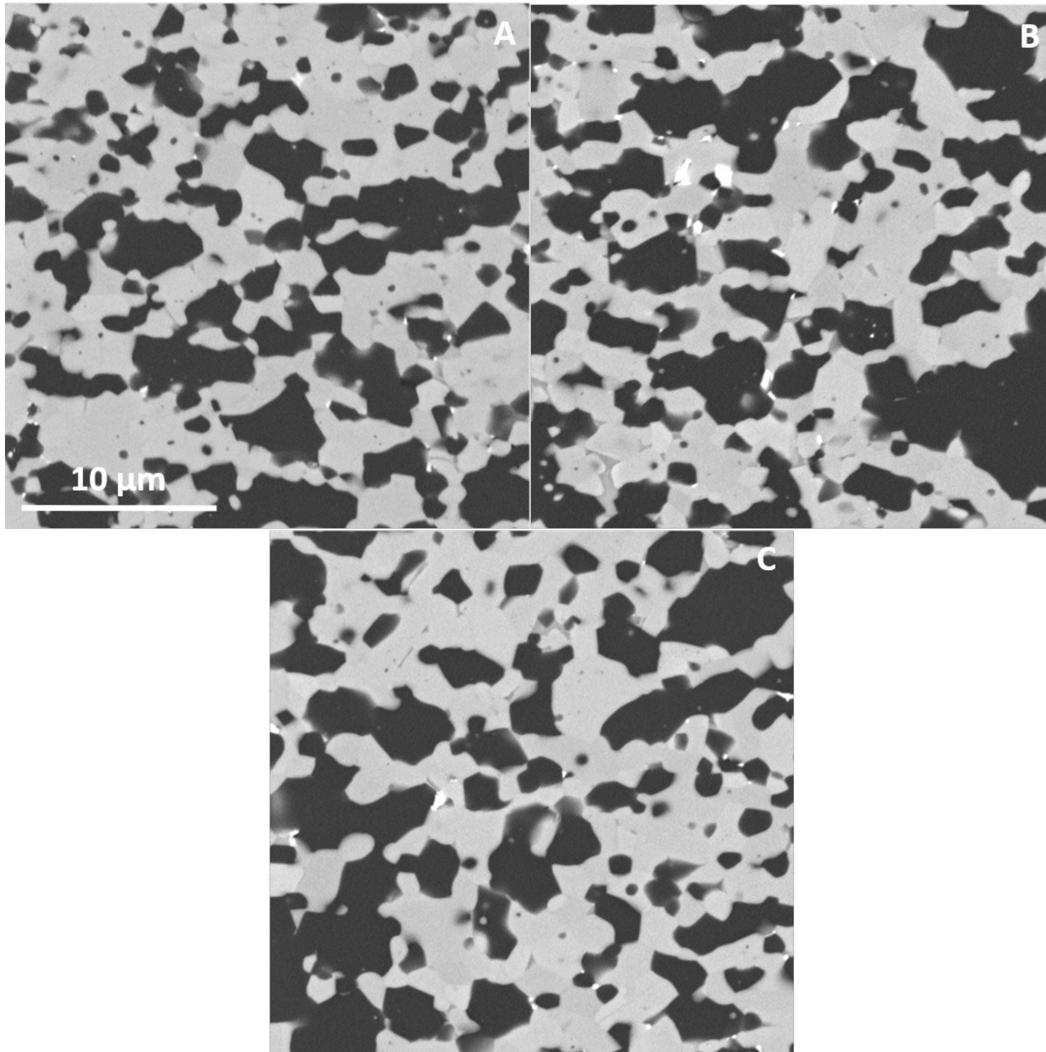

Fig. 6 B_4C/SiC blend after CMP using each polishing procedure. A) Procedure A, B) Procedure B, and C) Procedure C.

Two steps in the procedure that have previously been skipped by some users are the 6-µm diamond and the 0.02-µm colloidal silica CMP. The 6-µm step is overlooked as it is similar in size to the 9-µm step. However, this step does seem necessary for removing pull-out left over from polishing at 9 µm, as seen in Fig. 4. CMP is sometimes skipped as it is not thought to be as useful of a step as the mechanical polishing. The value of CMP is clear when one compares the sample before and after (Fig. 7). The SiC grains are much more evident after CMP in both the monolithic and blend sections, although the B_4C grains appear mostly unaffected. Other etching methods may be more appropriate if improved grain contrast is desired in the B_4C. However, further investigation would be required to find a procedure that would balance the etching rates of both the B_4C and SiC phases and result in a quality surface for each material. The colloidal silica CMP

works by a combination of mechanical and chemical processes. The suspended silica particles act as an abrasive, while the moderately basic medium (pH = 9.8) chemically etches the surface.[11,12] If the sample is being polished only to obtain hardness measurements, skipping CMP may be acceptable. However, if the sample is to be observed with SEM, it is a worthwhile endeavor to get as much contrast between grains as possible for improved observation of grain size and morphology.

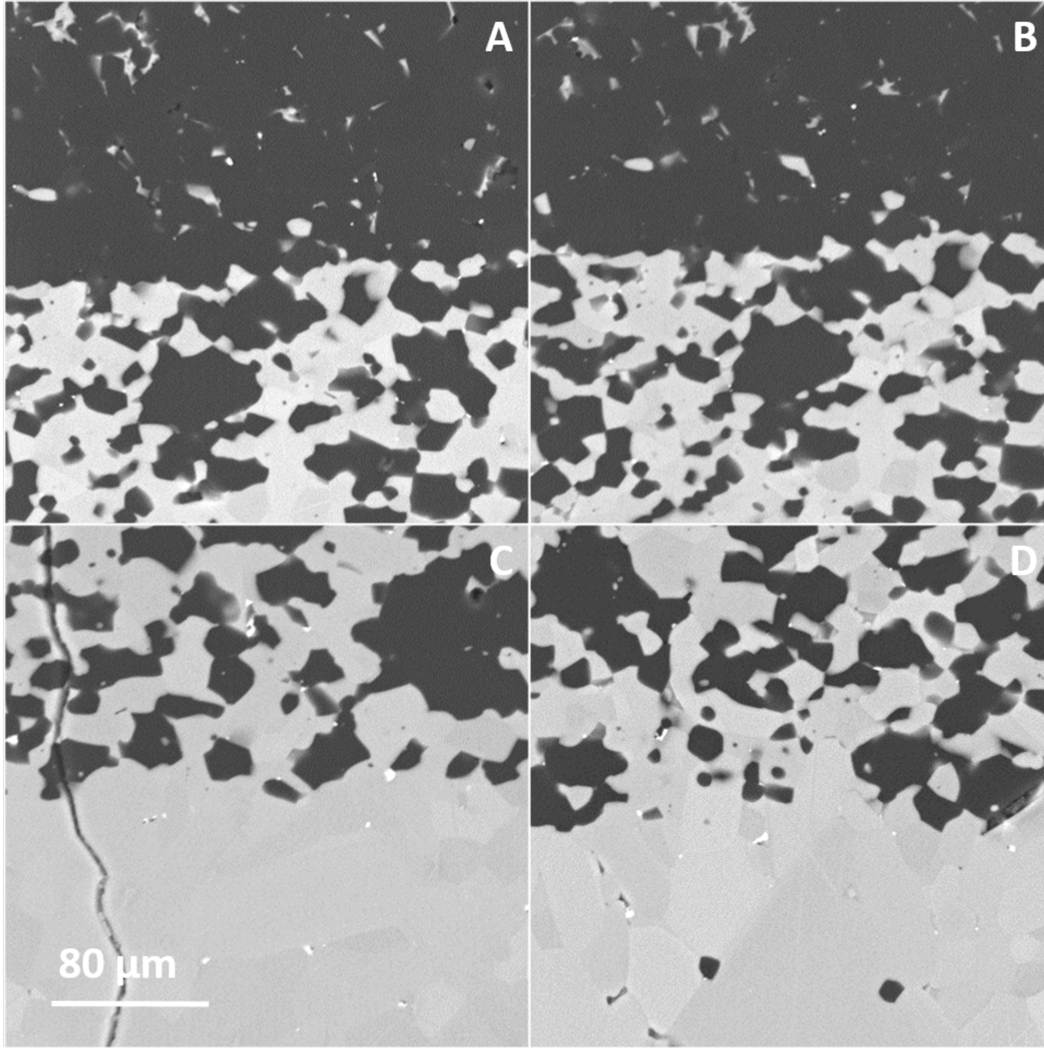

Fig. 7 Comparison of each layer interface before and after CMP. A) B_4C/blend no CMP, B) B_4C/blend with CMP, C) blend/SiC no CMP, and D) blend/SiC with CMP.

Overall Procedure C is significantly more efficient than Procedure A while maintaining a minimal difference in the resultant surface quality, with a 10.5-h (~45%) reduction in polishing time. This reduction will save users a significant number of hours and decrease the time between fabrication and characterization. The variable space for further optimization of this procedure is vast, so additional work could be done. Polishing periods shorter than 30 min could be investigated,

different periods could be used for each polishing step, loads and rotation speeds could be adjusted, and different polishing media could be used. However, these optimizations are beyond the scope of this study due to the significant time investment it would require and the low potential for significant improvements.

4. Conclusions

Ceramographic preparation of B_4C-SiC layered composites is more complex than preparation of B_4C or SiC, both of which are difficult to prepare due to their high hardness. To reduce time spent by users to polish these materials, a progressive study was performed by capturing SEM images at regular intervals during polishing of such a specimen. Using these images, a new, shortened procedure was developed and a second sample was prepared to verify the new procedure's viability. SEM analysis was used to compare the previous procedure to the newly devised procedure, with no significant difference found between the two.

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List of Symbols, Abbreviations, and Acronyms

Al_2O_3	aluminum oxide
ARL	Army Research Laboratory
B_4C	boron carbide
BSD	backscattered electron detector
CMP	chemomechanical polishing
DEVCOM	US Army Combat Capabilities Development Command
EBSD	electron backscatter diffraction
SED	secondary electron detector
SEM	scanning electron microscopy/scanning electron microscope
SiC	silicon carbide
XEDS	X-ray energy dispersive spectroscopy

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A DIGIOVANNI
M GOLT
M GUZIEWSKI
S HIRSCH
C HUBBARD
M IVILL
S KILCZEWSKI
M KORNECKI
N KU
J LASALVIA
T MOORE
T PARKER
P PATEL
S RAJU
A ROSENBERGER
W SHOULDERS
J SWAB
W GAMBLE
FCDD RLW MF
P GOINS
C HAINES
E HORWATH
FCDD RLW MG
J LENHART

R MROZEK
E NAPADENSKY
FCDD RLW S
J CIEZAK-JENKINS
A WEST
FCDD RLW TB
J MCDONALD
S SATAPATHY
T WEERASOORIYA
FCDD RLW TC
J CAZAMIAS
D CASEM
J CLAYTON
R LEAVY
J LLOYD
C MEREDITH
T SCHARF
C WILLIAMS
FCDD RLW TD
R DONEY
K STOFFEL
FCDD-RLW TE
C KRAUTHAUSER
P SWOBODA
FCDD RLW TF
P JANNOTTI
L MAGNESS
D MALLICK