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A Conventional Metallographic Method for Ytterbium Disilicate and Yttrium Ytterbium Disilicate Air Plasma Spray Coatings

by Arturo Villegas, Nickolas Sotiropoulos, Joshua Taggart-Scarff, and Clara Mock

NOTICES

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14. ABSTRACT
Ytterbium-based environmental barrier coatings for silicon carbide (SiC)-based ceramic matrix composites (CMCs) are of great interest to enable gas turbine engines to operate at higher temperatures. Metallographic evaluation of the coatings is challenging due to the hard, brittle nature of the coatings and substrate, causing artifacts such as coating pull-out and uneven polishing. A technique for the metallographic preparation of ytterbium disilicate coatings on monolithic SiC was developed. This enables key features to be measured through optical microscopy, highlighting the effectiveness of the proposed methodology in minimizing artifacts and leading to high-quality microscopy images and robust evaluations. This approach enables enhanced assessment of CMCs for high-temperature aerospace applications.

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air plasma spray, ytterbium disilicate, silicon carbide, metallography, microscopy, Sciences of Extreme Materials

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1. Introduction

Improving the performance of gas turbine engines requires higher engine temperatures and ceramic matrix composite (CMC) components. One of the candidate CMCs is silicon carbide (SiC)-based CMCs, which are ideal because of their high-temperature strength and toughness.[1] SiC CMCs require thermal/environmental barrier coatings to minimize vaporization of the component during exposure to moisture from the combustion process at high temperatures.[2–4] Ytterbium disilicate (YbDS) is one of the candidate coatings for SiC as it has shown promise to reduce silica volatilization of SiC substrates compared to bare SiC.[5] Microcracking and porosity in coatings can reduce the temperature that the substrate is exposed to and therefore reduce the rate of oxidation.[6] The microcracking and porosity paired with the brittle nature of the ceramic make metallographic preparation difficult, where portions of the coating tend to pull out, resulting in an inaccurate evaluation of the coating microstructure, especially for porosity quantification.

2. Experimental

The thermal spray coatings underwent a series of metallographic procedures, including cold mounting, sectioning, remounting, grinding, and polishing. It is imperative to perform each step with precision to prevent damage to the coating and avoid subsequent alterations to the microstructure during examination. A comprehensive understanding of the experimental setup is crucial in developing a resilient recipe for the SiC ceramic matrix composite. A variety of equipment and consumables from manufacturers such as Struers, Buehler, and Allied High Tech was used to perform the laboratory experiments. The details about specific equipment and consumables used can be found in Appendix A.

3. Mounting

To reduce potential damage during the cutting process, the samples were embedded in epoxy prior to any other metallographic preparation. A low-viscosity, high-hardness epoxy with minimal shrinkage was chosen for this work. Mounting in epoxy was assisted by vacuum infiltration allowing for effective filling of porous areas of the coating. The vacuum aids in eliminating trapped air from uncured epoxy, filling open pores and cavities in the samples to ensure optimal bonding and support. This preserves sample integrity during cutting preparation, reducing the risk of cracking or delamination. Further instructions on how to mount with epoxy are as follows:

1) **Preparing Mold Fixtures:**

   a. Clean the mold fixtures using alcohol to remove any contaminants.

   b. Apply a small amount of release agent to the molds to aid in the removal of the cured epoxy mount.

2) **Mixing Epoxy Resin and Hardener:**

   a. Use a precise weight scale, a disposable cup, and a wooden stick for stirring.

   b. Dispense the recommended weight ratio of resin to hardener per the manufacture's recommendations.

   c. Mix the hardener and resin in a disposable plastic cup until smooth and transparent without streaks.

3) **Filling Mold Fixtures:**

   a. Insert the coated ceramic sample into the mold fixtures.

   b. Pour the epoxy mixture into the mold until it reaches the desired level to cover the samples thoroughly.

4) **Curing:**

   a. Allow the filled mold fixtures to cure inside a fume hood.

   b. Since the curing process typically requires at least 8 h, samples may be left overnight and removed the next day.

3.1 Sectioning

We recommend using a metal-bonded diamond blade with a high diamond concentration for cutting ceramics and minerals with hardnesses greater than HV800. In this study, a precision cut-off saw was used to section and reveal the cross-section of the coatings. The setup proceeded as follows:

1) **Choice and Placement of Diamond Blade:**

   a. Choose a 6-inch diamond blade designed for ceramics and minerals with a hardness value exceeding HV800.

   b. Securely lock the diamond blade onto the precision saw.

2) **Preparing Sample and Fixture:**

   a. Secure the ceramic sample onto the fixed holder.

3) **Mounting Fixture onto Specimen Holder Arm:**

   a. Affix the fixture to the specimen holder arm and tighten it securely.

   b. Align the blade with the sample.

   c. Reconfirm the alignment of the cut region desired.

4) **Adjusting Precision Saw Parameters:**

   a. Set parameters for this sample: cut-off wheel speed of 3000 rpm, linear feed rate 0.10–0.20 mm/s, and low-cut force.

   b. Optionally, select the alternative of returning the cut-off wheel to its original position after completing a cut.

3.2 Remount

A secondary epoxy mount is still required for easier mounting in the automatic polisher fixture holder to complete the grinding/polishing process. Follow the steps described in the previous mounting section. The second vacuum infiltration of epoxy is optional.

3.3 Grinding

Initial grinding of the sample is required to produce an even and flat surface for further metallographic preparation. Diamond-coated discs are the preferred grinding disc pads because they are specifically designed to preserve the samples' flat surface and edge retention. Using these pads on a hard substrate, such as SiC, is ideal because the pads contain nickel-plated diamond particles in a unique raised matrix. The design allows for improved sample cooling, which leads to increased grinding rates and efficient debris removal, as well as high durability and excellent edge retention and flatness. Standard SiC papers were not useful because the sample's substrate was effectively the same material as SiC, making the grinding process significantly longer. An automatic polisher equipped with a lubricant and diamond suspension dispenser was used based on local availability. (Operational procedures remain consistent with the equipment in use, but input settings may need to be adjusted due to hardware/software differences). After initial grinding, nail polish was used to fill areas where epoxy resin did not fully infiltrate into the sample, protecting against uneven force during polishing, which could cause material pullout. The sequential steps for this procedure are noted as follows and summarized as a table in Appendix B.

1) **Dressing the Diamond Disk Pad:**

   a. Presoak the dressing stick in water for 3 min.

   b. Use a dressing stick to remove debris buildup on the diamond disk pad; for thorough cleaning, employ a non-metallic brush or follow manufacturer product guides.

   c. Apply firm pressure to the dress stick and perform a horizontal grind sweep from the center to the edge of the rotating pad.

   d. Dressing time varies based on debris removal requirements.

2) **Grinding Duration for Level Plane Surface:**

   a. The recommended grinding time is 1–3 min to achieve a level plane surface using water as a lubricant, which is adjustable based on the ceramic material.

3) **Constant Force Setting:**

   a. Each sample received a constant force of 20 N.

4) **Rotation Speed of Grinding Surface Disk:**

   a. Program each grinding surface disk to rotate at 150 rpm in both the head and plate directions.

5) **Ultrasonication after Grinding and Polishing:**

   a. Ultrasonicate samples for 5 min after each grinding and polishing stage.

6) **Coating with Nail Polish:**

   a. After the first 15-μm grind, apply nail polish and quickly dry under a heat lamp or at room temperature if no heat source is available.

   b. This process allows nail polish to penetrate areas within and around the coating that did not fill during cold mounting, reducing the risk of artifacts affecting the polish process.

7) **Flattening the Nail Polish Layer:**

   a. Perform an additional grinding procedure on the 15-μm pad for 2 min to remove excess nail polish.

3.4 Polishing

Both napless and napped cloths were used in the polishing procedure. The process consisted of two steps: (1) a coarse polish with a napless cloth having a dense woven pattern to maintain excellent sample flatness while also providing a high material removal rate and (2) a fine polish with a medium-napped cloth with synthetic silk. Final polishing was conducted on a porous neoprene pad, which is ideal for providing exceptional final polishing for materials because of its high resilience, soft hardness, and nonstick porous nature, which prevents suction for large specimens. Using colloidal silica with a pH of 9.8 for the final polish results in a clean, impurity-free depiction of the microstructure. These polishing steps are as follows:

1) Apply the 9-µm diamond suspension and lubricant to the dense, woven nylon pad.

2) Set the automated polisher to operate for 12 min. Subsequently, rinse the samples with water and ultrasonicate, following the procedures outlined in the grinding steps.

3) Proceed to readjust the dispense ratio on the dense, medium-nap synthetic silk pad to accommodate the 1-µm diamond suspension.

4) Repeat for five cycles of 5 min each, totaling 30 min of polishing time. This iterative approach facilitates periodic checks of sample progress, enabling adjustments to dosing as necessary.

5) Repeat the same cleaning process as previously executed in the grinding step.

6) Using the porous neoprene final polishing pad (Allied High-Tech Products), dispense a pipet full of 0.04 µm colloidal Si at 5:00, 4:30, 3:45, 3:00, 2:15, 1:30, and 0:45. Flush with water 0:30 for 5 min, adhering to the interval timing specifications provided in Appendix A.

4. Results and Discussions

In Figure 1, a refined depiction of the YbDS-Si-SiC coating is presented, displaying each layer of the substrate devoid of artifacts, such as excessive pullout, which can lead to the formation of large porous regions. Using gentle force during plane and fine grinding is recommended to minimize pullout. Upon examining the coated layer of YbDS, microcracks are observable, consisting of vertical cracks perpendicular to the substrate and horizontal cracks extending parallel to the substrate, resulting from cooling during the air plasma spray deposition. Cracks can be prevented by decreasing thermal stress during the spray process.[7] Figure 2 shows an example of a highly porous yttrium ytterbium disilicate (YYbDS) topcoat that was prepared using this metallographic technique. No microcracking or obvious coating pullout is observed in this sample.

Mount wax is frequently used as a temporary adhesive in the metallographic process, allowing it to bond to specific regions where the epoxy mount did not fully adhere to the sample edge, or porous regions caused by pull-out during the grind process. A new approach was taken, with clear nail polish used to fill in areas where the epoxy was unable to completely encapsulate the unfilled areas. This proved useful during the grinding and polishing stages, resulting in high-quality microscopy images, as illustrated in Figure 2.

Despite the apparent contrast in polishing outcomes in both figures, the true porosity might still be hidden. Other methods in the literature can help determine this.[8,9] Further analysis using energy dispersive X-ray spectroscopy will help to comprehend the phase constituents of the splats, which will aid in identifying the splat morphology of YbDS coating.

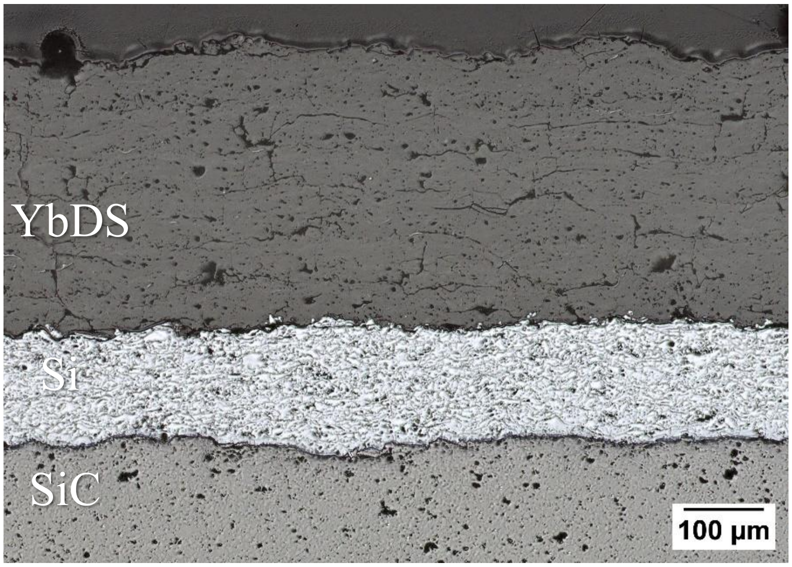

Figure 1. A representative image micrograph of a bilayer YbDS-Si coating on top of monolithic SiC substrate.

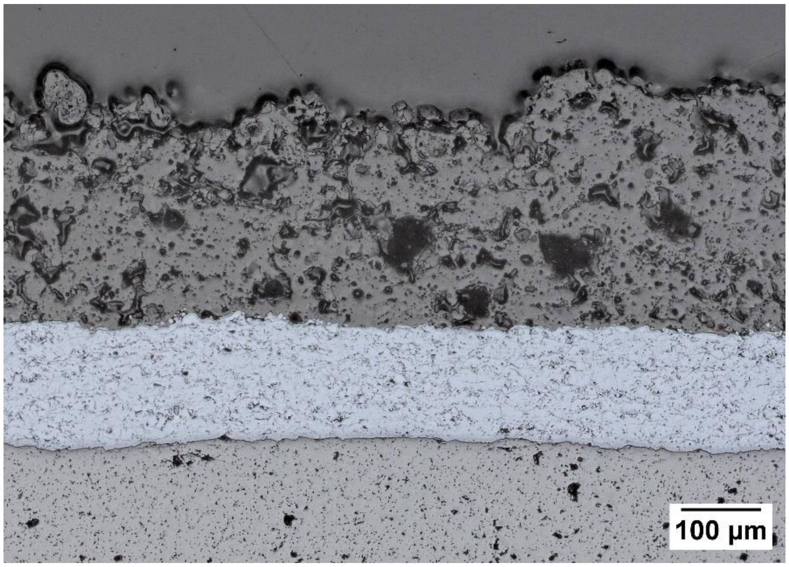

Figure 2. A highly porous YYbDS topcoat with a Si bond coat on SiC substrate.

5. Conclusions

Developing a reliable metallographic technique for evaluating ytterbium-based disilicate coatings on monolithic SiC is crucial for improving the effectiveness and cost-efficiency of ceramic matrix composites used in high-temperature aerospace applications. Careful sample preparation allows us to assess characterization using optical microscopy, examine key factors such as splat particles, porosity, and coating thickness. Each metallographic step is critical, from pre-mounting to polishing, to prevent coating damage and ensure an accurate microstructural evaluation. Hence, creating a methodology that facilitates dependable performance evaluations sets the stage for comprehensive assessments of features, aiding in the development of SiC-based CMCs.

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Appendix A. Products and Equipment Used

Table A-1. List of equipment used in this study.

Equipment type	Manufacturer	Model	Description
Mounting	Allied High Tech	VacuPrep	Epoxy impregnation system
Sectioning	Struers	Accutom-100	Precision cut-off machine
Grinding/Polishing	Struers	LaboSystem	Grinding and polishing system

Table A-2. List of consumables used in this study.

Product Type	Make	Model	Description
Sectioning	Struers	M0D15	Metal-bond, high concentration diamond cut-off wheel
Grinding	Allied High Tech	Dia-Grid Diamond Discs	Diamond particles nickel-plated in a raised matrix, 70, 30, and 15 µm
Mounting	Allied High Tech	EpoxySet Resin and Hardener	Low viscosity, high hardness, clear, and low-shrinkage epoxy
Mounting	Struers	FixiForm	Two-part mounting cup, 1.25inch dia.
Mounting	Buehler	Release Agent	Liquid release agent for mount removal from castable molds
Polishing	Allied High Tech	Gold Label	Dense, woven nylon pad for use with diamond (15–3 µm)
Polishing	Allied High Tech	Red Final C	Dense, medium nap synthetic silk flock pad for use with diamond (3–0.02 µm)
Polishing	Allied High Tech	Polycrystalline, Glycol Based Diamond Suspension	Water-soluable, glycol-based polycrystalline diamond suspension, 9 and 1 µm
Polishing	Allied High Tech	Colloidal silica suspension	Noncrystallizing 0.02µm colloidal silica suspension, 9.8 pH
Polishing	Struers	MD-Chem	Porous neoprene polishing cloth for final polishing
Polishing	Buehler	MetaDi Fluid	Polishing lubricant
Polishing	Buehler	MetaDi Supreme, Poly	Polycrystalline diamond suspension, 9 µm
Polishing	Sally Hansen	Ultimate Shield Base and Topcoat Nail Polish	Clear nail polish

Appendix B. Recipe for the Metallurgical Preparation of YYbSi2O7DS or YYbSi2O7-Si-SiC Air Plasma Spray Coating

Recipe name	Equipment	Step	Surface	Abrasive	Grit/grain size	Grit dosing	Lubricant	Lubricant dosing	Head and plate rotational speed (rpm)	Head and plate rotation direction	Force mode attachment holder	Force (N)	Machine time (min)	Comments
SIC TEBC	Struers Laboforce 100	1	Dia-Grid Diamond, 70 µm	Diamond	No suspension	N/A	Water	ON	150	Comp	Single samples	20	1–3	
		2	Dia-Grid Diamond, 30 µm	Diamond	No suspension	N/A	Water	ON	150	Comp	Single samples	20	1–3	
		3	Dia-Grid Diamond, 15 µm	Diamond	No suspension	N/A	Water	ON	150	Comp	Single samples	20	1–3	
		Coat of nail polish on sample												
		4	Dia-Grid Diamond, 15 µm	Diamond	No suspension	N/A	Water	ON	150	Comp	Single samples	20	2	
		5	Gold Label	Diamond	9 µm	2/4	Meta Di	3/6	150	Contra	Single samples	20	12	
		6	Red Final C	Diamond	1 µm	1/6	Meta Di	3/6	150	Contra	Single samples	20	30	Five iterations of 5 min/ check periodically
		7	MD-Chem	Colloidal Silica	0.04 µm	manual	N/A	N/A	150	Contra	Single samples	20	5	Final 30-s flush pad surface with water

Notes: During the grinding step, both the head and plate of the automatic polisher must be complementary. In the polishing step, only the plate needs to be complementary.
Multisample holder used for all steps.
Rinse and clean samples under water with gentle rub of thumb with nitrile gloves.
After each grind/polish process, ultrasonicate the samples for 5 min.
Clean both the sample holder and the magnetic disk plate after each grind and polish step.
Use a pipet full of colloidal-silica and dispense at 5:00, 4:30, 3:45, 3:00, 2:15, 1:30, and 0:45. Flush with water at 0:30.

List of Symbols, Abbreviations, and Acronyms

ARL	Army Research Laboratory
CMC	ceramic matrix composites
DEVCOM	US Army Combat Capabilities Development Command
SiC	silicon carbide
YbDS	ytterbium disilicate
YYbDS	yttrium ytterbium disilicate

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