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Accepted Practice for Recognizing Artifacts in Air Plasma Spray Thermal Barrier Coating Microstructures

ASM Thermal Spray Society, Accepted Practices Committee

ABSTRACT

Thermal spray coating (TSC) microstructures are prone to interactions with common metallographic procedures that may result in artifacts and misinterpretation of the microstructure. This article identifies metallographic TSC artifacts, specifically in the air plasma spray zirconia-based thermal barrier coating including both the bond coating and the top coating. Artifacts that result from specific sectioning and mounting practices, as well as from different polishing times, are presented. Additionally, factors in optical microscopy and scanning electron microscopy that affect microstructure interpretation are discussed.

INTRODUCTION

Thermal barrier coatings (TBCs) are applied using thermal spray coating (TSC) processes to components that are internally cooled and operate in a heated environment. The thermally insulating effect of a TBC maintains a lower service temperature of the component, reducing cooling requirements and extending component life. Commercial applications for TBCs include hot stages of gas turbine engines and internal combustion engines.

The microstructure of a thermal spray coating (TSC) is typically a primary indicator of the coating quality, its functional properties, and proper deposition and processing. Due to the nature of the TSC deposition process, the microstructures are not homogeneous, comprising multiple phases and phase boundaries, as well as layers and directionality. As a result, the TSC microstructures are prone to interactions with common metallographic procedures that may result in artifacts and misinterpretation of the TSC microstructure. The goal of this paper is to aid in identifying metallographic TSC artifacts, specifically in the air plasma spray (APS) zirconia-based thermal barrier coating (TBC) including both of its common constituents, the bond coating (BC) and the top coating (TC).

Many technical papers have been published on the best practices for metallographic preparation of TSCs [1, 2]. Any of several metallographic preparation methods have been shown to produce a true and reproducible TSC microstructure. For comparative purposes, the TSCs used for this paper were deposited simultaneously and polished using variants of ASTM E 1920-03 Method-II [3]. Artifacts that result from specific sectioning and mounting practices, as well as from different polishing times, are presented. Additionally, factors in optical microscopy and scanning electron microscopy that affect microstructure interpretation are discussed.

BACKGROUND

Typical APS Microstructural Features

The APS TBC is typically a bi-layer coating comprised of (1) a softer, relatively ductile, metallic bond coating (BC) and (2) a harder, porous, lower ductility ceramic top coating (TC). In the TC, porosity and cracks are key to its function as a thermal barrier layer in aero and power generation turbine engines. In the BC, features related to cohesion between splats and adhesion to the substrate (porosity, oxidized boundaries, unmelted particles, cracks) are indicators of TBC service life at high temperature.

A simplified drawing of key microstructural TSC features highlights the degree of inhomogeneity present in these coatings (Fig. 1). In addition to three distinct layers that make up a TBC (the substrate alloy, the BC, and the TC), there are other features common to TSCs in general such as voids or pores, oxidized particles in the metallic BC, metallic inclusions in the TC, unmelted or spherical particles, grit particles at the substrate-BC interface, vertical micro-cracks, and horizontal debonding cracks. Some amount of each of these features are allowable in a TBC TSC and are defined in the specific quality requirements for the coating.

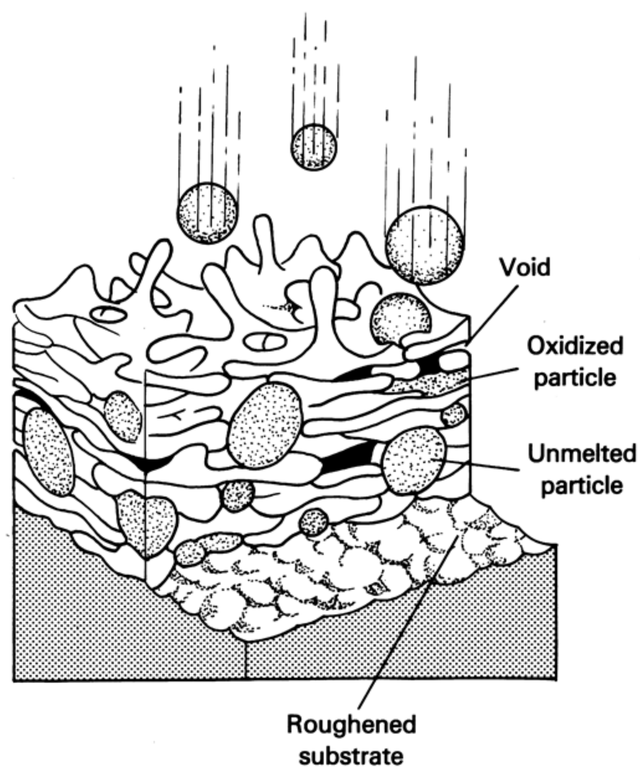


Figure 1: Key TSC microstructural features [1]

TBC Metallographic Process

Metallographic processing of TBCs follows the same general steps as those of wrought material, that is, sectioning, mounting, planar grinding, polishing, microscopy, and analysis. An exception is that chemical etching of the TSC is not typically done prior to microscopy. As described in other sources, the magnitude and direction of forces during sectioning can damage the TBC significantly, such that subsequent metallographic steps cannot remove the damage. Similarly, mounting and polishing methods also affect loading on internal microstructure interfaces and phase boundaries; consequently, these features can be changed by the method of mounting and polishing that is employed. Accepted metallographic practices for thermal spray coatings are well documented [1, 2] and specific deviations from accepted methods are used in this paper to create artifacts in the TBC microstructure. ASTM E1920-03 Method-II [3] is the basis of all polishing in this paper.

FACTORS THAT CAUSE TSC ARTIFACTS

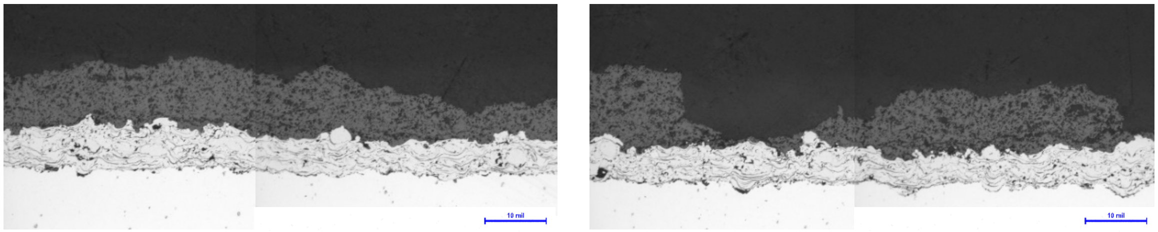
Sectioning

Sectioning involves applying several forces to a part/sample, including clamping pressure, blade incursion, bending, friction, shear, and thermal expansion stresses. Specific to as-deposited TSCs that are layered structures and are not metallurgically bonded to the substrate, the saw, blade, or cutoff wheel applies directional forces on the sample. If the blade enters the substrate and exits through the coating, it applies forces that push the coating away from the substrate.

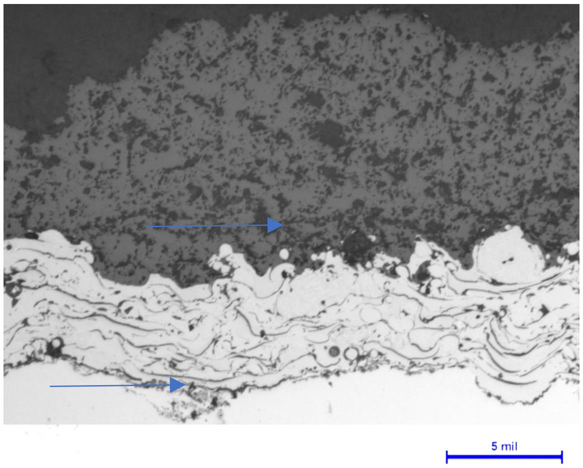
If those forces exceed the local bond strength of the coating, "debonding" can occur. Debonding presents as horizontal cracks or gaps along the or near the substrate interface. Also, specific to TBCs, where the TC is a less-ductile material, sectioning into the substrate and exiting the TC surface can also cause horizontal cracking within the TC. The TC cracks may appear discontinuous as the crack propagates in/out of the plane of view of the image. Debonding and TC cracking are possible artifacts of sectioning.

Subsequent grinding and polishing steps may not remove the artifacts of sectioning. A unique artifact of a TBC TC initiates as horizontal cracking due to sectioning and presents in the final polished structure as large, elongated pores or strings of pores that are not randomly distributed. Misinterpretation of this structure characterizes the TC as having greater pore fraction than actual.

Figure 2 shows a TBC microstructure with artifacts from improper sectioning, i.e., at high incursion rate, using limited coolant, and with the blade oriented to cut into the substrate and exit the coating surface. Figure 2a shows several fields of view demonstrating the crack path in the TC and loss of TC that resulted from cracking during sectioning. Figure 2b shows a higher magnification of this sample with the specific artifacts labelled.



(a)



(b)

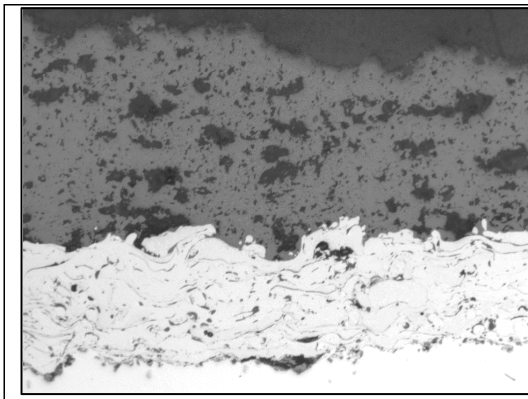
Figure 2: Artifacts in TBC caused by sectioning shown in (a) lower magnification images showing loss of TC by cracking and decohesion during sectioning and (b) high magnification image with arrows indicating additional horizontal cracking in the TC along the substrate interface in the BC. Samples and images provided by FL Institute of Technology, Center for Advanced Coatings, Melbourne, Florida, and Technetics Group, Deland, Florida

For non-porous wrought materials, sectioning is performed prior to mounting. For TBCs, the porosity in the TC is critical to the function of a TBC, and those pores represent internal surfaces that can fracture or yield under applied loads from sectioning and mounting. Because TBCs are porous and easily damaged by the forces involved in sectioning, coatings should be vacuum-infiltrated with epoxy prior to sectioning to support the pore walls during sectioning thus minimizing the risk of artifacts.

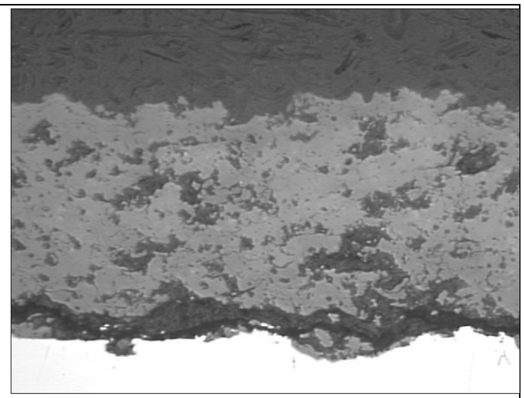
Mounting

Mounting processes can be characterized broadly as hot-mount type and cold-mount type. Among the various mounting materials in either process, cold mount materials applied using vacuum infiltration provide the lowest risk of artifacts for TBCs. All samples presented in this section were polished using ASTM E 1920-03 Method-II, non-modified. The distinctions of polishing using Method-I or Method-III are not discussed in this paper.

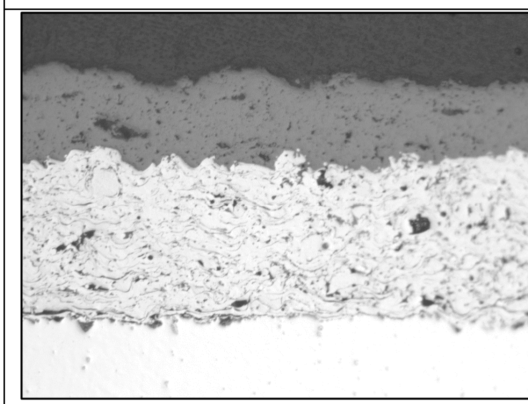
Figure 3 highlights common TBC microstructural artifacts caused during mounting. The images show several distinctive artifacts: 1) a hot-mount acrylic with artificially large pores in the TC, most elongated or aligned horizontally, which is consistent with horizontal cracking in the TC that becomes emphasized in relief during polishing; 2) a hot-mount phenolic with a continuous crack along the TC-BC interface due to elastic-plastic strain, collapse of TC pores under compression, or overall bending forces; 3) cold-mount acrylic samples have no horizontal cracking but may have localized large elongated pores near the bottom of the TC because cold mount acrylic is fast-curing, highly viscous, and may not infiltrate the TC entirely leaving pores unsupported and subject to relief; 4) a vacuum-infiltrated epoxy mount has negligible artifacts due to high internal pore support and low applied stresses.



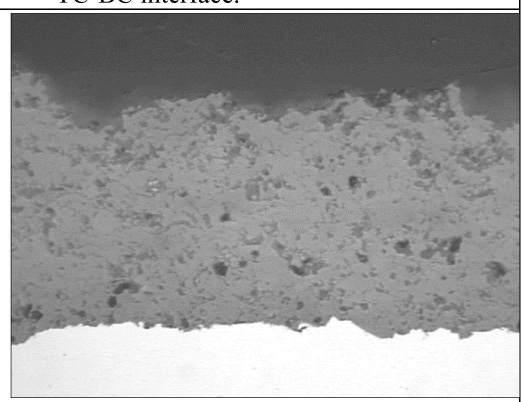
1) Hot-mount acrylic: lack of infiltration into TC pores, edge-rounding or relief of pore edges, inaccurately large pores.



2) Hot-mount phenolic: lack of infiltration into TC pores, edge-rounding or relief of pore edges, inaccurately large pores, horizontal cracking and debonding at the TC-BC interface.



3) Cold-mount acrylic, no vacuum: incomplete infiltration to the lower pores in the TC causing edge rounding and inaccurate pore size.



4) Cold-mount epoxy, vacuum-infiltration: minimal artifacts, possible lack of infiltration of lower TC pores due to thick TBC.

Figure 3: Artifacts in TBC resulting from mounting methods. Images provided by the Center for Advanced Coatings at Florida Institute of Technology.

Grinding and Polishing

Even when proper sectioning and mounting methods are used, minor and localized artifacts will be present near the sectioning plane, and these can be removed by proper grinding and polishing practices. For this reason, the initial planar grinding step is typically longer compared to grinding wrought materials. The grinding abrasive size is also typically finer than those used for wrought materials. One industry practice suggests a removal of 0.060-inch from the mount height during planar grinding of TSCs to ensure artifacts from previous steps have been removed.

Of course, planar grinding uses large abrasive size and the deep abrasion forces can cause artifacts of their own. Subsequent polishing with a series of finer abrasive media can remove the planar grinding artifacts when applied for sufficient time. In this section, all samples were properly sectioned then mounted using vacuum-infiltrated slow-curing epoxy. All were planar ground using 180-grit SiC paper with water coolant, replacing the paper every 30 seconds until each sample was planar. The effect of the supplier of consumables and abrasives is not addressed in this paper, but abrasive quality and polishing surface resiliency may affect artifact formation.

The post-planar polishing steps follow the ASTM E 1920-03 Method-II [3], presented in Table 1; however, polishing times were adjusted as listed in Table 2 to demonstrate how each step contributes to pore structure stabilization and removal of artifacts from the previous step.

Table 1: ASTM E 1920-03 Method-II polishing method for TSCs in this study.

Surface	**Coolant/ lubricant**	**Abrasive size/type ANSI(a) (FEPA)(b)**	**Time, min**	**Force(c)**		**Surface speed, rpm**	**Relative rotation**
				N	**lbf**		
Paper	Water	240- (P220-) grit SiC	Planar grinding, 0.25–0.5	20–30	4.5–6.7	250–350	Comp(d)
Hard woven cloth	Water-based suspension	6–9 µm diamond	Fine grinding, 4–7	20–25	4.5–5.6	100–150	Comp
No/low-nap cloth	Lapping oil or extending fluid	3 µm diamond	Rough polishing, 1–3	20–30	4.5–6.7	100–150	Comp
Synthetic suede	Water(e)	Colloidal silica	Final polishing, 1–3	23–35	5.2–7.9	100–150	Contra(f)

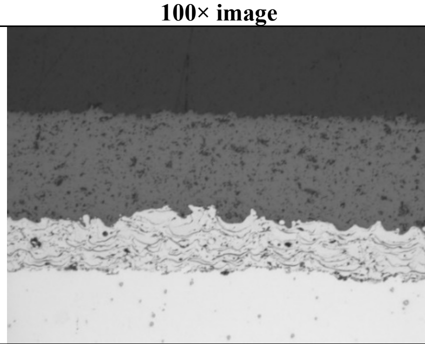
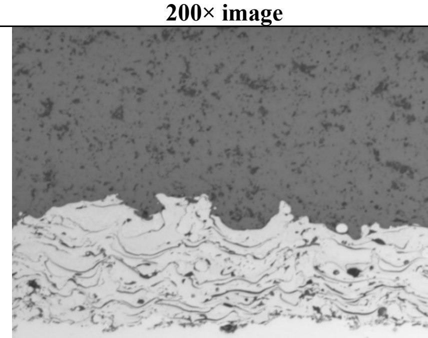
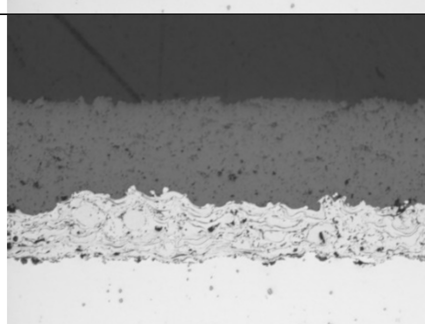
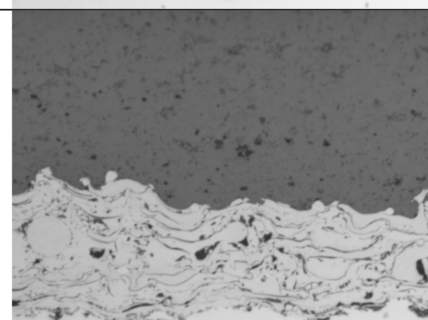
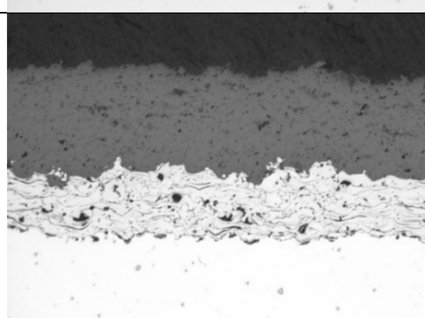
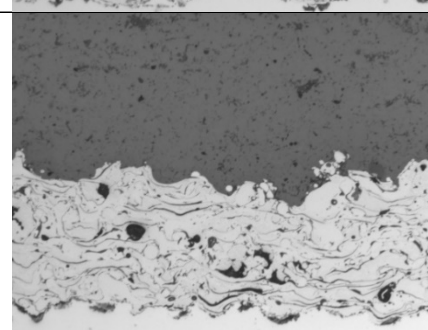
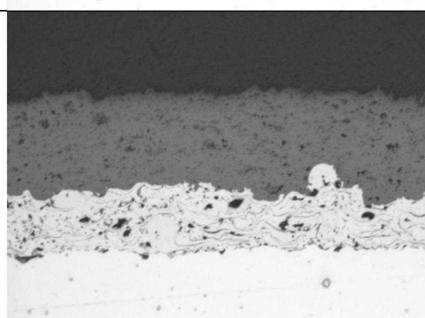
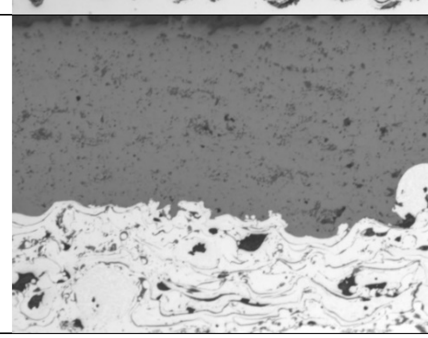
(a) American National Standards Institute (ANSI) designation of grit size. (b) Federation of European Producers of Abrasives (FEPA) designation of grit size. (c) Force per 32 mm (1.25 in.) diameter specimen. (d) Complementary rotation: surface and specimens spin in same direction. (e) Water free of particulate is required; distilled water is recommended. (f) Contrary rotation: surface and specimens spin in opposite directions

Table 2: Sample identification and polishing steps with adjusted polishing times in minutes.

Sample ID	**9 µm diamond suspension**	**3 µm diamond suspension**	**0.25 µm colloidal silica**
A	1	2	1
B	1	4	1
C	4	8	2
D	8	8	2

Table 3 presents the resulting TBC microstructures of samples A–D, along with area fraction of porosity in the TC as measured by image analysis.

Table 3: Images of samples A-D showing the effect of polishing times in removing artifacts from planar grinding.

Sample ID	**100× image**	**200× image**	**Area% porosity in top coat**
A			8.5
B			5.6
C			5.0
D			5.3

Sample A had reduced polishing times from Method-II for all polishing steps; the result was a higher pore fraction in the TC compared to the other samples, demonstrating that a false-high pore fraction is an artifact of planar grinding and insufficient fine grinding time.

Sample B had a reduced fine grinding time but normal subsequent polishing times, resulting in significantly lower pore fraction from Sample A. Sample B demonstrates that a reduced fine grinding time can be compensated by longer subsequent polishing times, but localized planar grinding marks and artifacts may remain.

Sample C had normal fine grinding time but longer polishing times, resulting in slightly lower TC porosity than Sample B. Sample C demonstrates that pore fraction stabilizes (no longer decreases) when all artifacts are removed.

Sample D had extended times for all steps and demonstrates that true microstructure can be verified by extended polishing times where additional polishing causes no change in pore fraction.

Under all polishing conditions here, the BC appears the same, exhibiting clear splat boundaries, voids, and unmelted particles. The BC is far less sensitive to artifacts than the TC under these polishing conditions, but BCs also have artifacts. The most common artifact in a BC is smearing (plastic deformation) of the metal on the polished plane that obscures these BC features. Smearing can be caused by worn consumables, dull abrasives, or improper polishing surface. Also, the BC is softer than the TC and can exhibit scratches more easily. Scratches in the BC can indicate insufficient polishing time to remove artifacts from the previous step. Scratches may also indicate contaminated or worn polishing paper.

An artifact common to any polishing method is edge-rounding. It is typically identified during the optical microscopy analysis as a field of view (FOV) where all features within the FOV cannot be brought into focus. The term "edge-rounding" connotes that the edge of the coating at the mount medium interface is not in plane with the rest of the coating. It can occur when the mount medium is softer than the coating and abrades faster than the coating. It can also be caused by highly resilient polishing papers that yield excessively into pores and along edges, increasing the polishing rate in those locations. The addition of Al_2O_3 particles to the cold-mount epoxy is one method to minimize edge rounding by adding hard particles to support the epoxy.

Figure 4 presents two cases of edge rounding: (1) the top of the TBC is out of focus due as the softer mount medium abraded rapidly; and (2) TC pores are not infiltrated by mount media, are unsupported during polishing, and the resulting edge-rounding of TC pores creates the artifact of inaccurately large pore size.

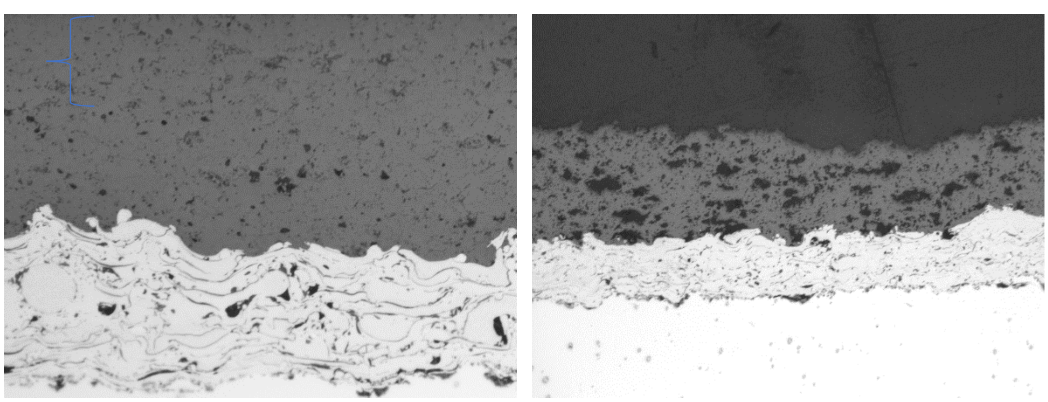
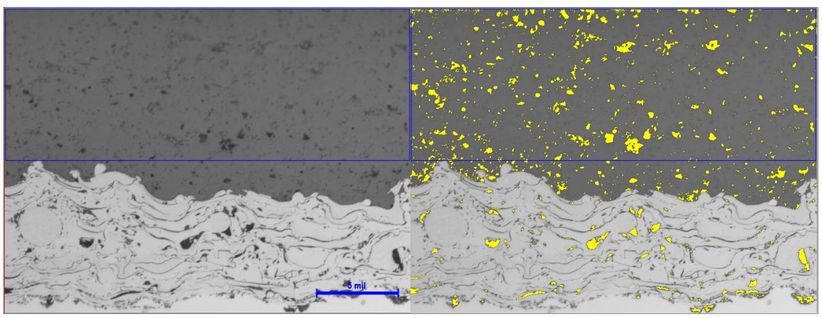


Figure 4: Optical images of edge-rounding: 1) Left, the surface of the TC is out of focus due to edge rounding near the mount medium; 2) Right, non-infiltrated pores in the TC appear darker and larger than infiltrated pores due to rounding of pore edges.

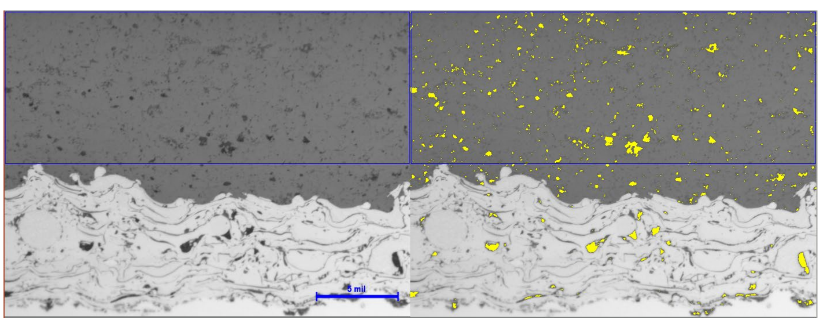
Optical Microscopy and Image Analysis Measurements

Optical microscopy uses visible light to form an image. Visible light can be reflected, transmitted, scattered, or absorbed to different degrees by filters in the microscope, dust or defects on the lenses, the coating materials, the phases in the materials, and the mount medium; as a result, artifacts can be caused by the nature of light interacting with the sample and the microscope. This paper will not address all interactions but will show how filters such as brightness and gamma can affect measurements of the TBC.

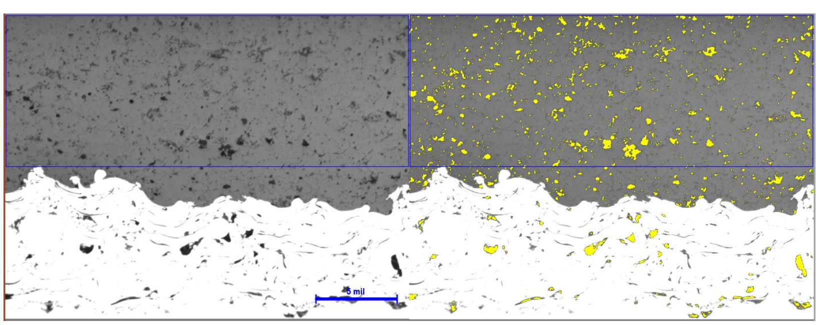
Figure 5 highlights Sample B from the previous section where the light intensity was not uniform, i.e., brighter on the right and darker on the left. Initial thresholding of the TC pores (selecting the shades of gray that correspond to a pore and defining them as yellow in this example) had focused on capturing all the pores on the right, consequently, the pores on the left in the dimmer light were "over-sampled." The calculated pore fraction was falsely high at 5.6 area-%. The next analysis of Sample B focused on thresholding the left pores in the dimmer area accurately, resulting in false low pore fraction measurement of 2.6 area%.



Sample B: 5.6 area% porosity, over-sampled pores on left in dimmer light



Sample B: 2.6 area% porosity, under-sampled pores on right in brighter area



Sample B: higher gamma, 5.0 area% porosity

Figure 5: Artifacts from optical microscopy and image analysis.

The third analysis applied a higher gamma setting to the image, further emphasizing mid-tone gray pixels. Thresholding of the pores across the higher gamma image was more uniform and the measured pore fraction of 5.0 area% was judged the most accurate.

Scanning Electron Microscopy (SEM)

SEM images represent energy interaction between the sample and impinging electrons; the materials and phases present very differently than optical microscopy images. In general, lighter shades of gray represent materials with higher atomic weight and darker grays correlate to low atomic weight materials such as carbon and oxygen. Therefore, carbon-based epoxy, metal oxides in the BC, and SiC or Al_2O_3 grit will appear dark in the image. An artifact of SEM imaging is brighter pixels along sharp edges or of non-conductive materials, where electron density may be higher and provide more signal to the collector. Bright definition of edges can help identify pores or cracks in TC's, also sand or organic fiber particles on the surface, but it can also be mis interpreted as a unique phase. Figure 6 shows the difference in appearance of TBC features in optical versus SEM images.

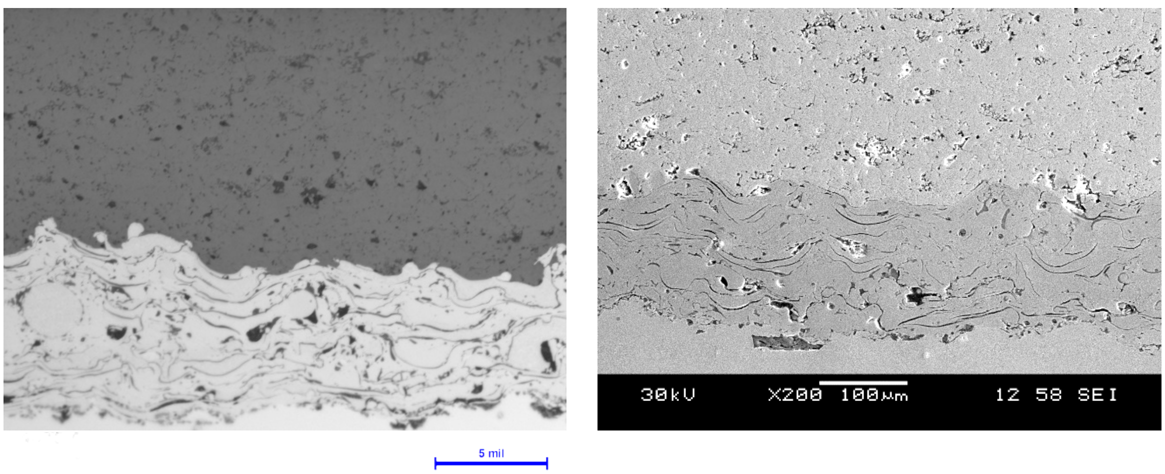


Figure 6: Sample B optical image (left) and SEM image (right), both 200×. SEM images provided courtesy of the Florida Institute of Technology, Center for Advanced Coatings

To distinguish a valid pore or crack from an artifact pore or crack, SEM images combined with energy dispersive spectrometry (EDS) can be useful. Figure 7 highlights the SEM image of Sample B bond coating at higher magnification; the color maps of each element identify the oxides in the BC (circled) as well as epoxy-filled pores/voids (teal arrow) and a SiC grit particle (purple arrow).

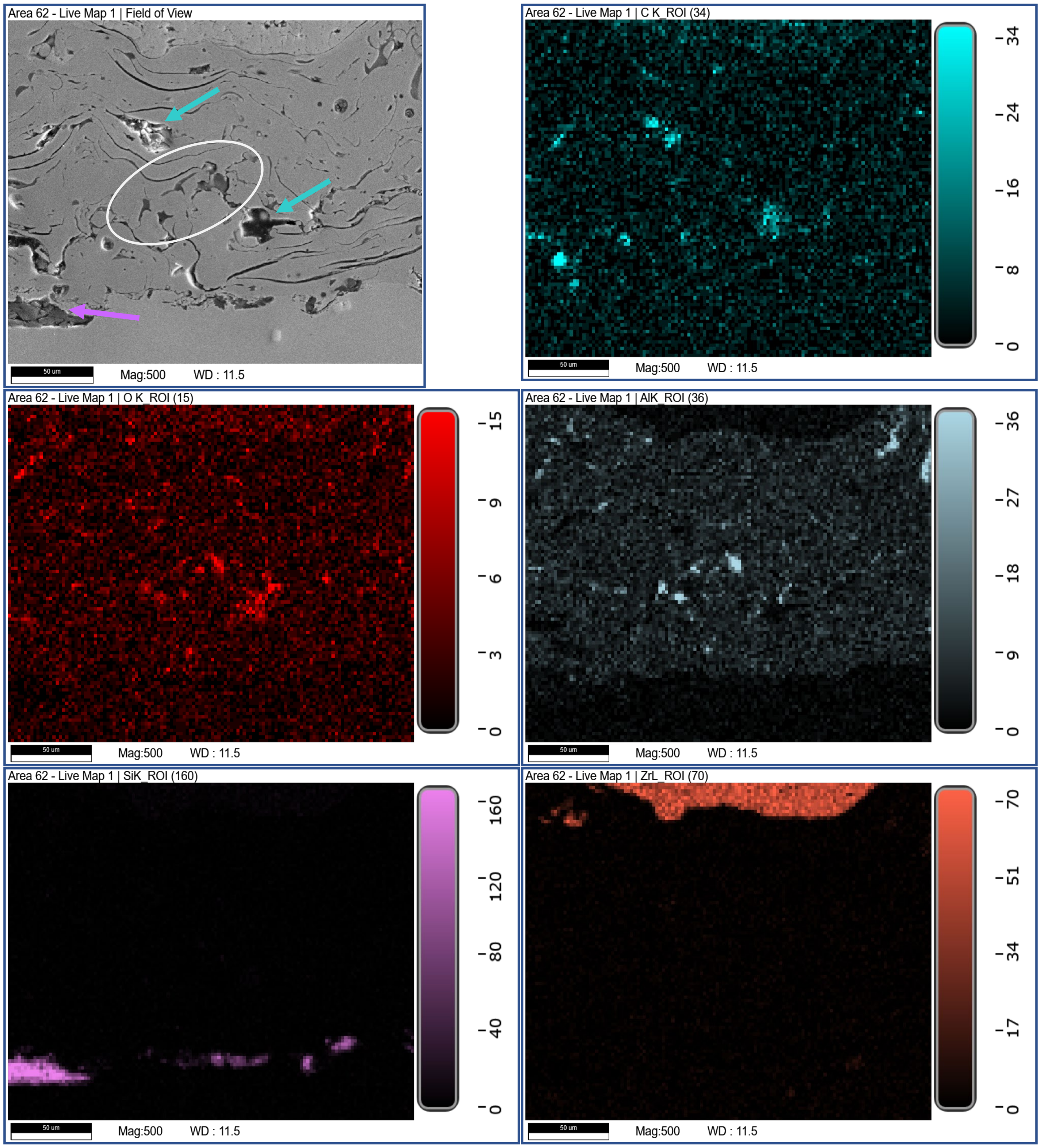


Figure 7 SEM images of Sample B with EDS elemental maps of carbon, oxygen, aluminum, silicon, and zirconium shown in different colors to distinguish BC oxides of Al and O from pores/cracks filled with C-based epoxy.

An aggressively sectioned TBC that was insufficiently polished is presented in Fig. 8 The image shows the bright highlighting along scratch edges and pore edges in the lower conductivity TC. The EDS maps of carbon shows a crack in the TC infiltrated by epoxy, indicating the crack was present prior to epoxy mounting.

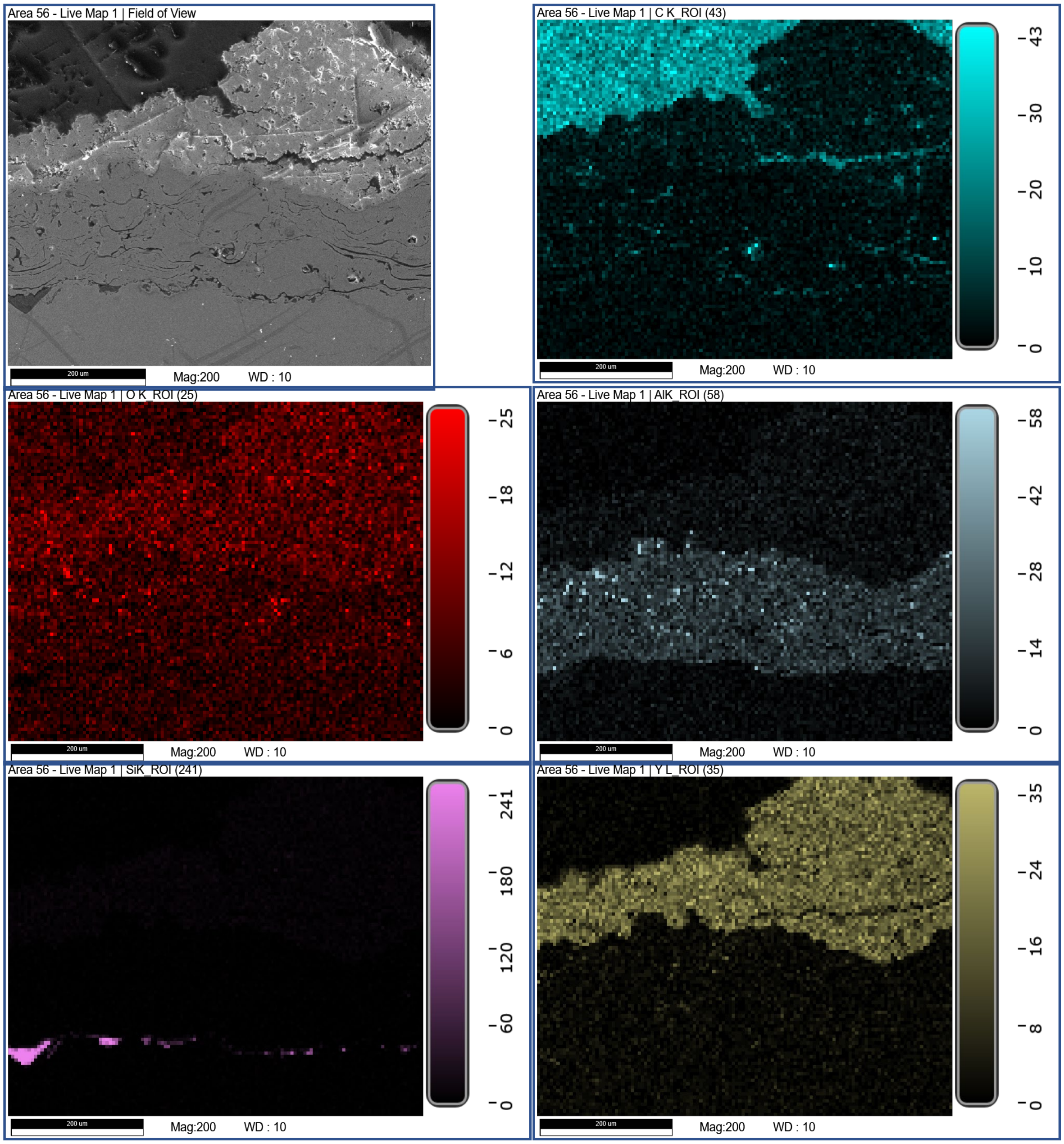


Figure 8: SEM image and EDS element maps of poorly sectioned and polished TBC showing highlighted scratches and carbon in the TC crack.

SUMMARY

For a TSC TBC, the key artifacts due to metallographic methods and their likely causes are summarized in Table 4. A summary of best practices mentioned in this paper are listed in Table 5.

Table 4. Summary of artifacts and possible causes

Observed feature	Likely metallographic causes
Large pores with lack of mounting medium inside them, oriented along a horizontal line in the coating, or predominantly near the BC.	• Poor infiltration of the mount medium into the pores resulting in unsupported pores prone to edge fracture and pullout during polishing. • Insufficient planar grinding to remove sectioning damage or insufficient polishing times to remove prior polishing artifacts, i.e., to stabilize the porosity.
Horizontal cracking within the coating and/or along the TC or BC interface.	• Large bending stress on the sample during sectioning. • Aggressive sectioning that induces sample heating, such as high incursion rate, lack or coolant, cutting direction exiting the coating, or dull blade. • Hot mounting process that collapses TC pores under compression.
Inability to focus on entire field of view; edges of image are out of focus.	• Edge rounding due to softer mount medium wearing faster than the harder sample or due to highly resilient polishing surface. • Sample was not held flat against the polishing surfaces during grinding and/or polishing.
TC pores that appear dark and excessively large.	• Edge rounding or pores due to lack of mount medium infiltration to support the pore edges during polishing.

Table 5. Summary of best practices noted in this paper

Metallographic step	Best practices
Sectioning	• Because TBCs are porous and easily damaged by the forces involved in sectioning, coatings could be vacuum-infiltrated with epoxy prior to sectioning to support the pore walls during sectioning thus minimizing the risk of artifacts.
Mounting	• Vacuum infiltration of low viscosity epoxy is the best method to minimize risk of artifacts due to mounting.
Grinding and polishing	• Consider removal of 0.060-inch from the mount height during planar grinding of TSCs to ensure artifacts from previous steps have been removed.

NOTE

This Accepted Practice is intended to be used as a baseline, but it does not replace local test or laboratory instructions. Additional requirements may apply based on the available equipment, testing materials, customer requirements, and other criteria.

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