Metallographic Preparation of Thermal Spray Coatings: Coating Sensitivity and the Effect of Polishing intangibles

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Abstract

The heterogeneous or composite nature of the thermal spray deposit can make choosing a metallographic preparation process very difficult. Hard vs. soft phases, brittle ceramics, and mixtures of many phases can create a variety of issues. When a procedure is developed, it becomes a necessity to document all the parameters of intangibles that define the process. Examples of procedure definition will be discussed and a suggested list of critical intangibles such as polishing abrasives (concentration vs. frequency of application), lubricant (pH, type), and many other factors will be highlighted.

Metallography: Coatings vs Metals

In analyzing the issues with metallographic preparation of coatings, one could question why there is so much controversy and debate about a test method which has been around for over 100 years. Why do coating laboratory technicians not just take the techniques available in numerous metallographic textbooks or from the varied manufacturers of metallographic preparation equipment and consistently prepare samples to obtain repeatable results? When preparing a coating sample, you are really working on a composite system of a coating and the substrate (primarily metallic based). Does the coating/substrate respond the same way to varied metallographic techniques? The answer is usually no. For the most part, the metallic substrate will show little if no change in structure within a reasonable range of parameters such as speed, pressure, or lubricant/abrasive application rate. The material below the coating is dense, strong, and homogeneous; there is limited sensitivity to the metallographic process.

Many coating materials exhibit an entirely different behavior. Small and relatively subtle changes in preparation parameters can result in a vast range of coating microstructures. Porosity in TBC’s will show significant variation dependent on rotational direction. The porosity in soft coatings will be closed via smearing if the pressure is varied only slightly. Corrosion/etching can occur during polishing if the correct abrasive/lubricant combinations are not used. Why are coatings more sensitive to the preparation process and are all thermal spray materials affected? As indicated earlier, the answer lies in coating characteristics. Coatings are heterogeneous materials composed of varied phases. As compared to the
metallic substrate, coatings can be considered weaker, more brittle, and porous. Coatings can be more sensitive to the metallographic process. However, all coatings are not affected in a similar manner because some are closer in density and strength to metallic materials. It is incumbent upon the engineer/lab technician to consider material properties and the projected sensitivity to the metallographic process.

Metallography: Important Method Parameters and Intangibles

Metallography is very similar to the thermal spray in that there are many variables that define the process. It is critical to define all the important parameters which must be controlled to produce consistent and repeatable results or microstructure. Table I lists the major methods parameters which most everyone will identify as critical items to be monitored. Table II highlights the intangibles which must also be considered as parts of the steps or variable results will be obtained.

Table I - Major Parameters

<table>
<thead>
<tr>
<th>Speed</th>
<th>Direction of Head vs Table Rotation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure</td>
<td>Mounting Method - Hot/Cold</td>
</tr>
<tr>
<td>Lubricant</td>
<td>Frequency of Application</td>
</tr>
<tr>
<td></td>
<td>Automatic vs. Manual Preparation</td>
</tr>
<tr>
<td>Abrasive - Type</td>
<td></td>
</tr>
<tr>
<td>Polishing Cloth</td>
<td>Grinding Format - Papers or Discs</td>
</tr>
</tbody>
</table>

Table II - Major Parameters Plus Intangibles

<table>
<thead>
<tr>
<th>Speed</th>
<th>Size of polishing wheels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orientation of Mounts in the Holder</td>
<td>In compression or tension during grinding or polishing?</td>
</tr>
<tr>
<td>Pressure</td>
<td>Documented per mount or per rack?</td>
</tr>
<tr>
<td>Lubricant/Extender</td>
<td>Variation in pH level?</td>
</tr>
<tr>
<td>Abrasive Type</td>
<td>Should supplied concentration be diluted?</td>
</tr>
<tr>
<td>Polishing Cloth</td>
<td>High nap vs no nap.</td>
</tr>
<tr>
<td>Vendor to Vendor Consumable Differences</td>
<td>Direction of Head vs Table Location of head on the table.</td>
</tr>
<tr>
<td>Cleaning Between Grinding/Polishing Steps</td>
<td>Vacuum impregnation.</td>
</tr>
<tr>
<td>pH of soap.</td>
<td>Control of cooling rate with hot mounting.</td>
</tr>
<tr>
<td>Grinding Format</td>
<td>Fixed abrasive</td>
</tr>
<tr>
<td>Discs</td>
<td>Rolling abrasive</td>
</tr>
<tr>
<td>Papers</td>
<td></td>
</tr>
</tbody>
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778
Wheel Speed and Rotational Direction. For example, speed is a relatively easy parameter to control. Dependent upon your setup, the head is usually controlled to a fixed speed of 150 rpm while the table will either have two to three fixed values or the ability to vary over a prescribed range. Pressure is either applied via pressure feet or through the central stem of the rack loaded with any number of mounts. The head vs table rotation can either be complimentary (same direction) or counter (opposite direction). Why highlight these specific parameters? Head, rotation, speed and pressure combine to define the dynamics of the process. Counter head vs table rotation removes material at a much faster rate or more aggressively than complimentary rotation. It is easy to visualize why counter rotation is more aggressive as the samples push against the rotational direction of the grinding media. Complimentary does remove material due to the interaction of speed and diameter on the head vs table. The table will always be rotating at a faster speed than the head (also smaller diameter) thus creating a drag or cutting action on the specimen although not "ear the removal rate for counter rotation. This can be critical for coatings which are not well bonded or ceramic materials like the TBC in Figure 1 where the aggressive nature of counter rotation significantly altered the result.

Many systems provide the flexibility to "se a variety of table wheel sizes and holders. This can and will result in procedural differences due to wheel speed differences as the diameters change dependent upon the system. It is therefore critical to place the head at the same location every time you polish and "se the same wheels/holders to maintain the same polishing dynamics.

Pressure/Mount Orientation. Applied pressure is a very critical parameter. It must be specified on a per mount basis. This is easy when "ing a “pressure feet” system which applies the force directly to each individual mount. However, with a rack or holder, the pressure rating is sometimes specified as 15 psi or 70N, regardless of whether there are 3 or 9 mounts in the rack. This can substantially change metal removal/polishing rates and must be monitored.

A” area of debate is the necessity to keep specimens in compression during the majority of the grinding/polishing cycle. With a holder arrangement and “ing either counter or complimentary rotation, samples can be oriented in the rack/holder to accomplish this task. Application of pressure to a” individual mount results in rotation of the mount during preparation and orientation in Compression/tension to varying degrees. What is the best method? This will depend upon the preparation system which you are “ing and the coatings which you prepare. The issue is not tight or wrong. The important point is to be aware of the critical parameters/intangibles and control the process to achieve repeatable results.

Consumables Consumables are a very important part of the metallographic process. Grinding papers, polishing cloths, abrasives, extenders/lubricants, etc., must be used to accomplish the end goal of a “true” microstructure. A” important question is: are all consumables equal? Lubricants/extenders can be water/alcohol based and have a pH which varies from 4 to 9. The pH values for pre-mixed alumina abrasive can range from 4 to 8. Grinding papers may be sold as a specific grit size but the concentration of grinding media and therefore the cutting rate will vary. The concentration of diamond in a polishing suspension can also vary. What is the concentration of aluminum oxide or colloidal silica in the polishing suspension? What is the viscosity of the epoxy used for vacuum impregnation? It is very important to know the answer and consistently control the given parameter.

The issue is not to highlight one consumable supplier over another. The critical item is to be aware that differences exist and that the differences can change or alter the final outcome. Consistency in results can be established if the same consumables are used repeatedly in the process. However, when considering changes in consumable products, the preparation process must be analyzed to ascertain if the final result will be affected. Awareness is the key to success.

How to Define a Procedure

With all the parameters to monitor, it can be difficult to control the total metallographic process. Table III represents a framework to begin analyzing and formulating a complete and detailed preparation process. This is only a starting guideline to begin defining the key parameters/intangibles. The requirements for detail and monitoring will vary from system to system dependent upon the degree of automation which a company has in the metallography preparation. It is again critical to identify the manufacturer of consumables and consistently "se the same vendor on a repeated basis.

What Can Go Wrong!

With a” improper procedure, results can be very different. Figure 1 highlighted the change in morphology when counter vs complimentary rotation is used on a TBC material. Figure 2 emphasizes the difference between just simple cold mounting of a TBC vs the “se of vacuum impregnation/pressure mounting to fill the porosity. This impregnation process provides support for the void structure and minimizes the "enlargement” of the porosity.
which can occur during polishing/grinding without epoxy support (as cast). Viscosity of the starting epoxy and consistency in the achievable vacuum is critical in this situation to insure infiltration of porosity/cracks in very dense TBC’s.

Figures 3 and 4 illustrate polishing of a nickel graphite abradable coating. Figure 3 shows that use of insufficient lubricant can result in improper polishing/cutting action and smearing of the bondcoat. With a proper amount of lubricant as dispensed either automatically or manually via proper procedure control, an acceptable microstructure of the bondcoat is revealed. In preparing the topcoat of nickel graphite, an incorrect morphology can also be obtained if solution concentration and a proper pH is not maintained. Figure 4 depicts the same mount finished with final polishing steps of an aluminum oxide slurry and 1μ diamond with extender. A reduction in the white nickel content and pitting can be seen when comparing diamond (top) to aluminum oxide (bottom). This is apparently caused by a corrosion cell which results from the use of aluminum oxide slurry. If the correct concentration of aluminum oxide is used, results similar to the diamond polish can be obtained. With this in mind, it is suggested that the diamond polish be used to minimize the sensitivity to aluminum oxide suspension concentrations.

Summary

Metallographic preparation of thermal spray coatings is a very critical step in the assessment of material characteristics. Thermal spray coatings which are inherently non-homogeneous are clearly more sensitive to the metallographic process than typical metallic (substrate) materials. It is therefore absolutely necessary to define and monitor the critical parameters of the metallographic process. Intangibles which are sometimes overlooked have been highlighted and their impact identified. Procedures may vary somewhat dependent upon the preparation equipment available. Knowledge of the important parameters/ intangibles is the key to success. Control can then be maintained to achieve repeatable and consistent results on a day to day basis.

References


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Katie Evans
Lou Gatto
Table III - Metallographic Procedure Format

<table>
<thead>
<tr>
<th>COATING:</th>
<th>OPERATOR:</th>
<th>RUN #:</th>
<th>DATE:</th>
</tr>
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<tbody>
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<td></td>
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</table>

<table>
<thead>
<tr>
<th>CUTTOFF PROCEDURE:</th>
<th>MOUNTING PROCEDURE:</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>#</td>
</tr>
</tbody>
</table>

1) IDENTITY OF MOUNTS: ____________________________________________________________________

2) # OF MOUNTS IN HOLDER: _______  3) MOUNT ORIENTATION: ____________________________ (CRITICAL IF NOT A COUPON)

4) POLISHER: _________________________________________________________________________

5) ROTATION OF HEAD-VS-TABLE: _______  DIRECTION OF HEAD/TABLE: ____________________________

LOCATION OF HOLDER/RACK ON TABLE: ______________________________________________________

6) WAS A STANDARD RUN: YES OR NO (Circle One)  7) LOADING SKETCH REVIEWED? YES OR NO (Circle One)

IF YES SPECIMEN ID: _________________________________________________________________ REVIEWED BY: ____________________________

8) SIZE OF WHEEL: _________________  9) IS PRESSURE APPLIED VIA INDIVIDUAL FINGERS OR VIA SPECIMEN HOLDER (Circle One)

10) GRINDING: ROUGH OR COARSE (Circle One)  METHOD: GRINDING PAPER/DISC/OTHER (Circle One)

   a) LUBRICANT TYPE - HOW APPLIED AND LOCATION ON WHEEL

   b) SPEED __________________________  c) SIZE OF WHEEL _____________________________

   c) TIME ___________________________  d) PRESSURE PER MOUNT ______________________

   # OF SHEETS USED: ______________________ METAL REMOVAL: ___________________________

   180 180 (Grit)
   240
   220
   400
   600

MANUFACTURER: _______________________________________________________________

11) POLISHING: STEP # __________________________

   a) POLISHING CLOTH WAS CLOTH CHARGED PRIOR TO USE? YES OR NO (Circle One)

   b) LUBRICANT TYPE FREQUENCY APPLIED AND LOCATION ON WHEEL

   MANUFACTURER: __________________________ CONCENTRATION: __________________________

   c) ABRASIVE TYPE FREQUENCY APPLIED AND LOCATION ON WHEEL

   MANUFACTURER: __________________________ CONCENTRATION: __________________________

   IF APPLICABLE, WAS DIAMOND PASTE USED? YES OR NO (Circle One)

   d) SPEED __________________________  e) PRESSURE PER MOUNT ______________________

   f) SIZE OF WHEEL: _____________________  g) TIME: ____________________________

781
Fig. 1. The effects of head rotation on Zirconium Oxide (TBC) void content. Counter rotation of the polishing head vs. table causes excessive pullout.

Top: Counter rotation of head vs. table
Bottom: Same rotation of head vs table.
Mag: 200X

Fig. 2. The effects of mounting on Zirconium Oxide (TBC) void content. Cold mounting only vs cold mounting with vacuum and pressure impregnation. Same polishing procedure for both samples. Lack of impregnation results in pullout.

Top: Cold mounting only.
Bottom: Cold mounting with vacuum and pressure impregnation.
Mag: 200X
Fig. 3. The effects of using a lubricant when polishing bondcoat on Nickel Graphite. An insufficient amount of lubricant during polishing causes smearing.

**Top:** Insufficient amount of lubricant smears bondcoat.

**Bottom:** Sufficient amount of lubricant produces acceptable polish.

**Mag:** 200X

Fig. 4. The effects of final polishing step on nickel content in Nickel Graphite. The use of aluminum oxide vs diamond can cause a significant reduction in nickel and severe pitting.

**Top:** Final polish with 1µ diamond.

**Bottom:** Final polish with aluminum oxide slurry causing reduction in nickel content and substantial pitting.

**Mag:** 200X