TECHNotes

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Accurate Porosity Measurement in Thermal Spray Coatings using ASTM E1920

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Introduction

In most fields of scientific research and industrial quality assurance, we are constantly striving to improve the performance of the products and the materials from which they're made. This can sometimes be achieved through breakthrough technologies, but a significant proportion of progress is through incremental improvement, driven by enhanced understanding of the performance characteristics and material properties of the material. This can lead both to the refinement of key properties, and to reducing the margin of error in design.

In the aerospace and power generation industry, there is a constant push for higher performance and increased efficiencies. The role of coatings has been of great importance to supporting rapid progress in capability. However, core measurements of some coatings is compromised by uncertainty of results – in repeated studies, we have seen large variability in measurement between testing laboratories. While mechanical preparation for coatings analysis can typically be made repeatable, it is far more challenging to make it reproducible and measurement results can be more dependent on the operator than the actual material conditions.

Background

Thermal spray coatings have been around in their many forms for more than 100 years [1]. The principle of Thermal Spray is very simple - heating a feed stock material, typically in the form of powder or wire, such that it is partially or fully melted as it's accelerated towards the substrate at velocity high enough to allow the particles to deform the particle on impact. The particle solidifies and mechanically bonds to the surface of the substrate in the process.

The range of materials that can be applied using thermal spray is broad, although typical applications focus on improvements in temperature, wear and corrosion resistance. Similarly, there are a number of technologies that can be used to apply thermal spray, each with their own characteristics. It's not our intent to review these in this paper, but extensive information is readily available [2, 3]

Coatings manufactured via thermal spray typically have porosity in them. One of the most common processes for characterizing a spray coating is metallographic preparation. However, it's long been established that there can be problems in both finding a correct approach to preparation, and to reproducing this approach over time [4, 5, 6, 7, 8, 9, 10].

One recent 'round robin' experiment [ ] demonstrated variability between laboratories, and showed that:

- Similar polishing consumables did not give the same results
- Similar results did not reflect a particular metallographic approach
- Significant variability originated from the measurement itself

In addition, measured porosity values varied dramatically between participants – all of whom were experience in the metallographic preparation and analysis of samples. It has also been observed that any laboratory can achieve good repeatability in their results. The implication, therefore, is that variation in approach is the main causal factor of error in the preparation and analysis of porous thermal spray coatings.

The purpose of this paper is to look at the metallographic processes and analyse them in sufficient detail to characterize the importance of each stage to measurement result. Other contributory factors, such as microscope and imaging systems, will be investigated in other papers [12].

Experimental Approach

It has been established many times [4-10] that the preparation approach used has a dramatic effect on the results. Some factors that have been previously shown to individually cause problems are:

- Aggressive sectioning (to be avoided)
- Aggressive grinding (commonly used to recover sectioning damage)
- Poor mounting techniques (Insufficient support of porosity)

However, there has been wide disagreement on what the right preparation method is. The tendency has been to list a specific method as the only correct option.

The aim of this work was to test the theory that the sensitivity of porosity measurement variation was less related to the specific preparation approach used, but in fact depends on the consistency of equipment and consumables and knowledge of the preparation process. In order to do this, samples were taken of a T800 (HVOF) coating and a WC-Co (Plasma Spray) coating. We based experimentation around the approaches to preparation recommended in ASTM E1920-03 (2014) [13], Methods I and II shown in Tables 1 and 2 respectively. These methods were chosen as they are representative of the most common approaches found in industry, and are commonly accepted standard of preparation. Method I uses a series of SiC papers for short times, followed by one preparation stage on a Trident cloth and final polishing on a Microcloth using Colloidal

Silica. Method II uses just one SiC grinding stage followed by two diamond polishing stages on no-nap surfaces and polishing with Colloidal Silica.

Experiments were designed to look individually at the effect on apparent (measured) porosity from sectioning, encapsulation and grinding with respect to these two methods.

Effect of Sectioning and Grinding

It can be difficult to characterize the effect of sectioning damage on samples, although it's recognized that using the least damaging method is recommended. In this experiment, we chose to use a low damage sectioning method, but to compare samples that were sectioned and then mounted with samples that were mounted first to protect the coating during sectioning. An Isomet High Speed precision wafering saw was used for both sets of specimens (Figure 1).

This saw has a high-powered motor, which ensures consistency during the automatically controlled cutting. The use of diamond or Cubic Boron Nitride (CBN) wafering blades ensures that cutting load on the sample is kept low during the cutting process, minimizing damage. Best practice recommendations of keeping the coating in compression during cutting, and dressing the blade regularly, were used in all cases.

Table 1: Preparation based on ASTM E1920 Method I

	Surface	Abrasive	Lubricant/Extender	Force (Per Specimen)	Time (min:sec)	Platen Speed	Head Speed (rpm)	Relative Rotation
1	CarbiMet	180 [P180]	Water	25N	Until plane	250	60	>>
2	CarbiMet	240 [P220]	Water	25N	0:30	250	60	>>
3	CarbiMet	320 [P500]	Water	25N	0:30	250	60	>>
4	CarbiMet	600 [P1200]	Water	25N	0:30	250	60	>>
5	CarbiMet	800 [P2400]	Water	25N	0:30	250	60	>>
6	Trident	3µm Metadi Supreme	Metadi Fluid	25N	4:00	150	60	>>
7	Microcloth	0.06 µm MasterMet**		25N	2:00	150	60	><

** last 15-20 second use water only >>Complimentary ><Contra

Table 2: Preparation based on ASTM E1920 Method II

	Surface	Abrasive	Lubricant/Extender	Force (Per Specimen)	Time (min:sec)	Platen Speed	Head Speed (rpm)	Relative Rotation
1	CarbiMet	180 [P180]	Water	25N	Until plane	250	60	>>
2	UltraPad	9µm Metadi Supreme	Metadi Fluid	25N	6:00	250	60	>>
3	Trident	3µm Metadi Supreme	Metadi Fluid	25N	3:00	150	60	>>
4	Microcloth	0.06 µm MasterMet**		25N	2:00	150	60	><

** last 15-20 second use water only >>Complimentary ><Contra

Figure 1: Isomet High Speed saw – used at 4000rpm and 3mm/min cut speed with an Isocut HC blade

Both sets of specimens were prepared using Method II, and the porosity was analyzed. The specimens were then re-ground by repeating Step 1 six times for 1 minute each time, to ensure any remaining sectioning damage was removed, and then repeating Steps 2-4 as before. The specimens were then analyzed again for porosity.

Figure 2 shows the porosity analysis results. We can see that the porosity level was significantly higher in the specimen sectioned prior to mounting, after initial preparation. The measured porosity in this sample dropped significantly after being re-ground and the preparation steps repeated. Conversely, the measured porosity in the sample that had been mounted prior to sectioning remained statistically the same. We can draw the following conclusions from this graph:

1. Encapsulating the sample prior to sectioning protected the sample from damage
2. Sectioning damage could be recovered through sufficient grinding, to give the same porosity result
3. The ASTM method as written will not remove all damage from sectioning if excess damage is incurred

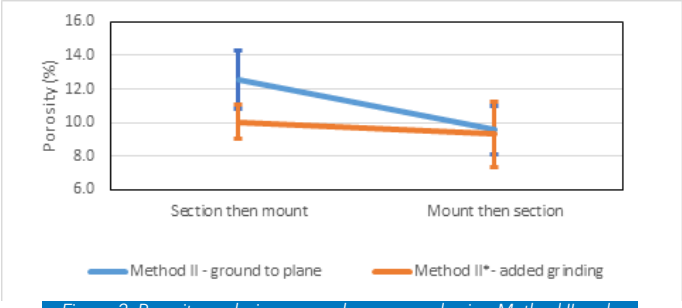


Figure 2: Porosity analysis on samples prepared using Method II and different levels of sectioning damage

Effect of Encapsulation Material

It can be difficult to characterize the effect of sectioning damage on samples, although it's recognized that using the least damaging method is recommended. In this experiment, we chose to use a low damage sectioning method, but to compare samples that were sectioned and then mounted with samples that were mounted first to protect the coating during sectioning. An Isomet High Speed precision wafering saw was used for both sets of specimens (Figure 1).

This saw has a high-powered motor, which ensures consistency during the automatically controlled cutting. The use of diamond or Cubic Boron Nitride (CBN) wafering blades ensures that cutting load on the sample is kept low during the cutting process, minimizing damage. Best practice recommendations of keeping the coating in compression during cutting, and dressing the blade regularly, were used in all cases.

Table 3: mount materials used in encapsulation experiment

Material	Viscosity	Mount Hardness	Shrinkage
EpoThin 2	Very low	78	Low
EpoThin 2 (wet sample)	Very low	78	Low
EpoKwick FC	Very low	82	None
EpoKwick FC + ceramic bead	Very low	>90	None
Epoxicure	Medium	82	None
SamplKwick (acrylic)	High	78	Medium

All the samples were mounted in a single specimen holder, such that they were all prepared identically. The sample sets were prepared using Method I and Method II.

Method I appeared to not be as effective on this material as Method II, especially for the harder samples. This is particularly noticeable in the sample where ceramic bead was added to the mount. In this case, the removal rate during grinding an polishing is greatly reduced –by the lower wear rate of the ceramic. In addition, the ceramic has a blunting effect on SiC, and so the effect is more pronounced in Method I

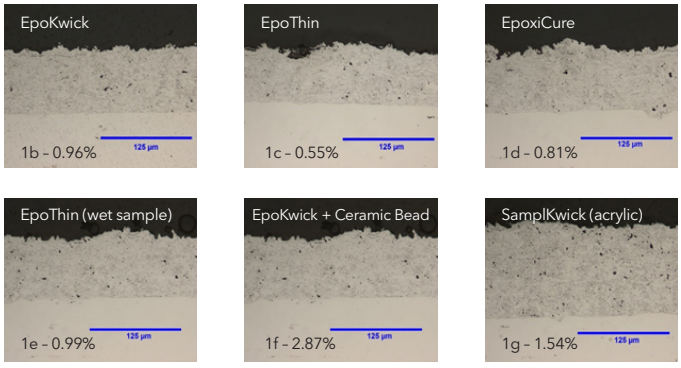


Figure 3: Final polish on each specimen using ASTM Route I

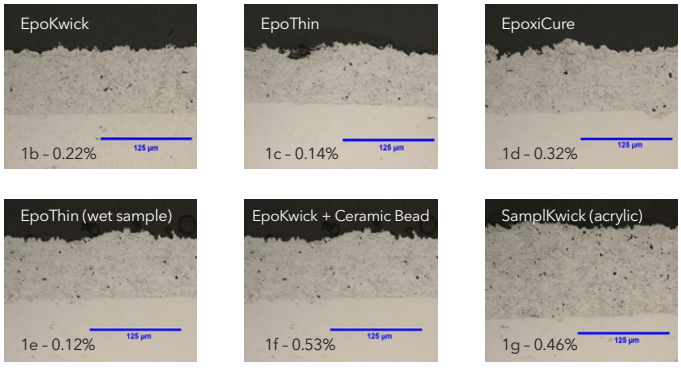


Figure 4: Final polish on each specimen using ASTM Route II

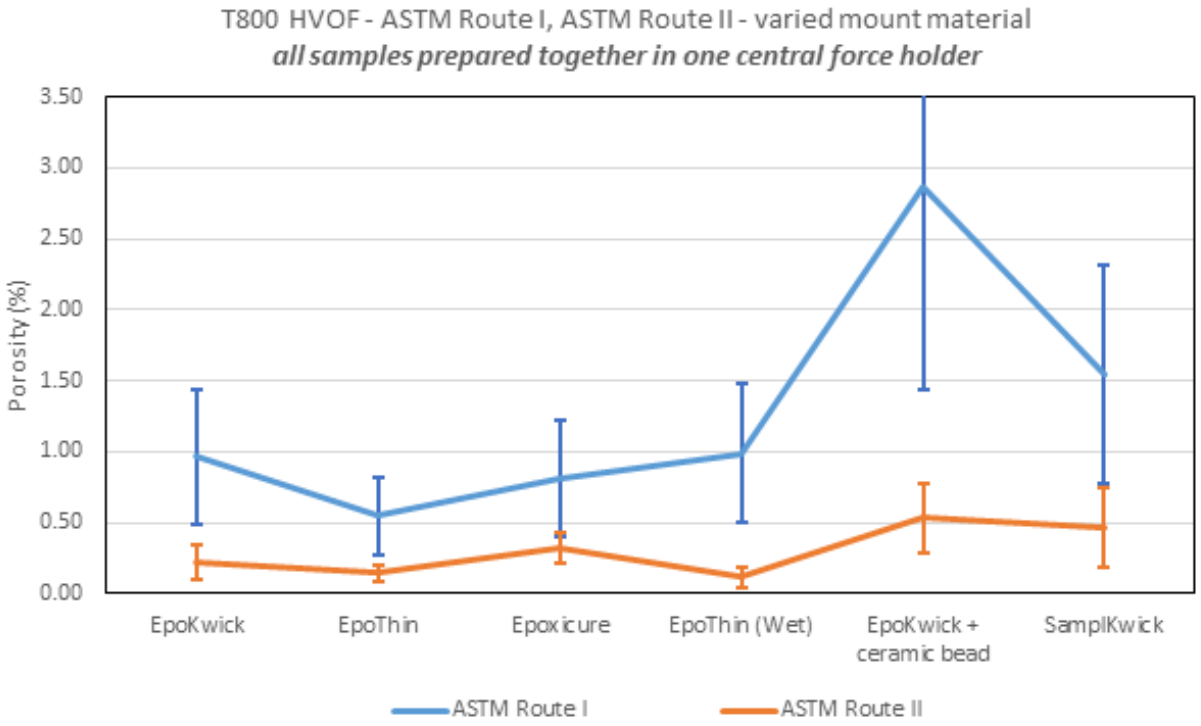


Figure 5: Porosity analysis from all samples with varied mount materials after, Method I and Method II

Low viscosity epoxies performed better (lower values, and less variability).
The acrylic mount (SamplKwick) gave poor results compared with the epoxy mounted samples.
The poorly dried sample was more sensitive to inadequate preparation, although it gave similar results in Method II. It's likely that this low porosity sample has limited connectivity, allowing us to grind through the effect. The impact of mounting poorly cleaned and dried specimens would be expected to be significantly more in higher porosity samples.

At this point, one preparation method would typically be classified as better than the other – but this is often not quite correct, and will be investigated more in the Preparation Method section below.

Preparation Method

In order to look at the direct effect of preparation method, we took our best practice recommendations to this point and mounted two sets of samples as follows:

- Clean/degrease samples
- Rinse samples with water, then soak in ethanol for 10 minutes to absorb water from pores
- Dry thoroughly. Do not handle samples with bare skin, to avoid contamination with oils
- Mount samples in EpoThin and vacuum impregnation using a Cast N Vac 1000
- Section samples on Isomet High Speed Saw
- Re-mount sectioned sample in desired orientation
- Prepare all samples in a Central Force holder for greatest flatness and reproducibility

When we did this, we found that the porosity after preparation was very similar to that shown in Figure 2 (for samples mounted prior to sectioning). During the preparation, however, we analyzed the samples for apparent porosity at each stage. The results of this analysis are shown in Figure 6 below.

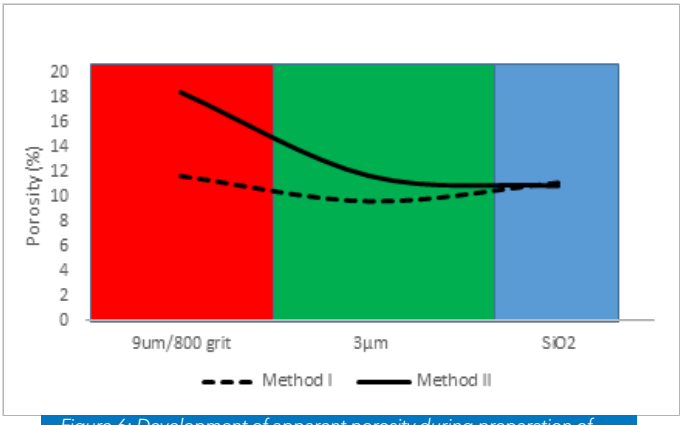


Figure 6: Development of apparent porosity during preparation of WC-Co coating

We can see that the porosity level using Method I hardly seemed to change during the preparation. The implication is that we are not removing sufficient damage during the stages used to properly reveal the true porosity. In order to investigate this, we extended each of the stages in the preparation routes, examining porosity levels at regular intervals, until the measured value stabilized at each stage. This effectively shows us both how long the preparation step should be, and the level of damage associated with the step. This can be significant as with some materials, a particular preparation step may be more damaging than expected.

In this particular sample, apparent porosity levels of 12-15% were typical after the 9um stage. Using the standard ASTM route did not greatly reduce this. Extending the 9µm/800 grit stage did not greatly change this. However, when we extended

the 3µm stage the apparent porosity dropped significantly. We then added a 1um stage on a TexMet C cloth and continued to track the apparent porosity, until it was stable, and then the final polishing was done as in Table I and II. Figure 7 shows the porosity analysis through preparation for this modified Method II, as shown in Table 3. It can immediately be seen that the apparent porosity has been greatly reduced.

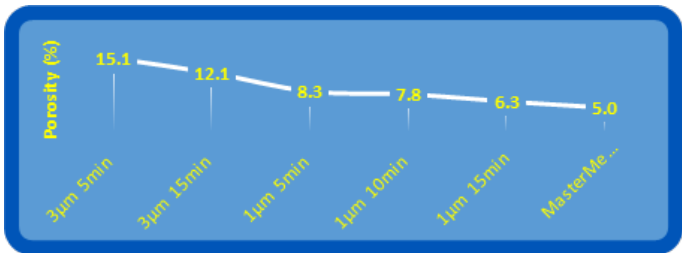
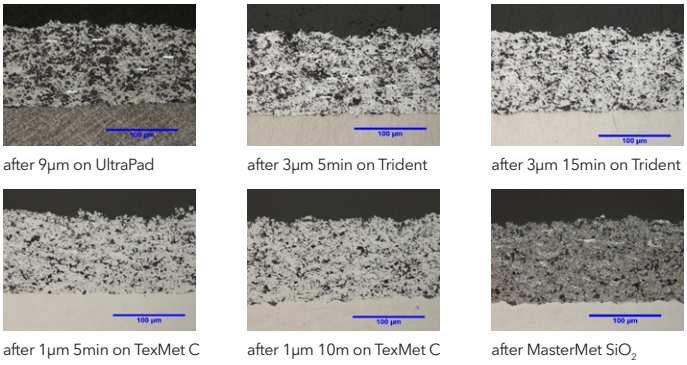


Figure 7: Change in apparent porosity through preparation stages, modified route based on ASTM Method II



after 9µm on UltraPad | after 3µm 5min on Trident | after 3µm 15min on Trident

after 1µm 5min on TexMet C | after 1µm 10m on TexMet C | after MasterMet SiO_2

Figure 8: Change in porosity through preparation, modified Method II

Images from this process are shown in Figure 8. The improvement in the specimen surface both from extending the 3µm stage and from adding the 1µm stage is immediately apparent. Further clues of the necessity of modifying the preparation route can be seen when the micrographs are examined more closely. Figure 9 show areas A and B from Figure 8. In both cases, a particle of grit is present, embedded in the substrate during the surface cleaning/roughening process applied prior to thermal spray. After 5 minutes on the Trident cloth using 3µm diamond, it can be seen that particle A is incomplete, and that the area around it still has deformation from earlier preparation stages.

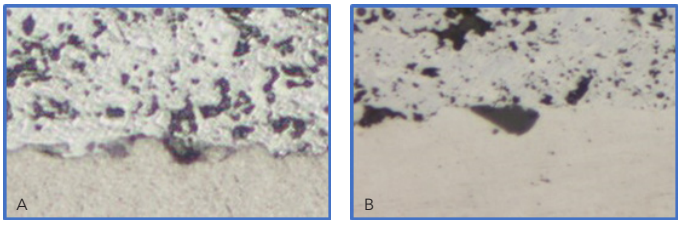


Figure 9: Areas A (3um stage, 5min) and B (1um stage, 10min) from Figure 8 - digitally magnified

The interface between the coating and the substrate also shows signs of grinding damage – this is indicated by the rougher surface (deformation) and rounding at the material interface (grey area), as well as the presence of scratches.

Table 3: Optimized preparation for this sample, based on ASTM E1920 Method II

	Surface	Abrasive	Lubricant/Extender	Force (Per Specimen)	Time (min:sec)	Platen Speed	Head Speed (rpm)	Relative Rotation
1	CarbiMet	180 [P180]	Water	25N	Until plane	250	60	>>
2	UltraPad	9µm Metadi Supreme	Metadi Fluid	25N	5:00	250	60	>>
3	Trident	3µm Metadi Supreme	Metadi Fluid	25N	15:00	150	60	>>
4	TexMet C	1µm Metadi Supreme	Metadi Fluid	25N	15:00	150	60	>>
4	Microcloth	0.06 µm MasterMet**		25N	2:00	150	60	><

** last 15-20 second use water only >>Complimentary ><Contra
[Note: the sample used in this work has relatively high porosity and therefore is more susceptible to grinding damage – typical preparation method for WC-Co coatings would typically use shorter steps]

Conclusions

While it can be agreed that good quality preparation of thermal spray coatings requires best practices in all preparation stages to be followed, it is also apparent that understanding the processes involved and examining the specimen at each stage of preparation is a more reliable approach than simply following a given method.

Specimen preparation following a strict methodology may give highly reproducible results, but may also give reproducibly incorrect results. This was shown in the comparison of Method I and II in Figure 2. Both approaches gave the same porosity when encapsulation and sectioning damage were normalized. However, the porosity level (12 - 15%) was significantly higher than the porosity level observed using the modified method in Table 3 (5 - 6%).

In addition, as the apparent porosity is sensitive to changing conditions, there is a risk that uncontrolled factors can also change the result. This includes variability in the sample itself, but also in the consumable items and equipment used. Buehler equipment and consumables are produced to high standards of quality control, to ensure repeatability and reproducibility of results.

Buehler Application laboratories partner with our customers to develop hundreds of unique, effective and reliable preparation methods in our global applications laboratories every year. Each of these solutions are tailored to individual needs, to ensure high quality and highly reliable preparation solutions. Our application laboratories generate reports and provide advice to highlight best practice in all stages of preparation, as well as how to recognize and adapt to change. Our training courses cover both theory and practice of specimen preparation, aimed at providing attendees with the ability to understand, develop and modify their own preparation methods.

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