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Quality Considerations for the Evaluation of Thermal Spray Coatings

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Revealing the true structural and mechanical properties is of utmost importance for the optimized use of thermal sprayed coatings. Only the true properties can be expected to correlate to the spray parameters. During the recent decade, the gas turbine industry has experienced a focus on the laboratory procedures being the weakest link in a frozen and robust process. This article will show several results indicating that the laboratory procedures are more essential to the evaluation results than the spray parameters themselves. With new and robust laboratory techniques, the true properties of thermal spray coatings are revealed, causing a major problem with respect to the quality standards developed 30-40 years ago. In many cases, these old specifications need updates, which is a difficult task from a cost, time, and quality perspective for OEM's. Coatings that have been successfully used for almost half a century no longer conform to the specification they were optimized to, because of these new appropriate laboratory techniques and procedures. What is actually meant when stating the following? (1) The coating has 5% porosity; (2) No cracks are allowed; (3) Tensile bond is 50 Mpa; (4) Hardness is 1000 HV; and (5) Coating thickness is 100 μm. This article also initiates a discussion on the measurement inaccuracies, for testing of thermally sprayed coatings, with respect to the commonly used general international standards (such as QS9000, ISO17025, AS9003, and ISO10012), as well as with respect to recommendations from the Six Sigma methodology.

Keywords adhesion of TS coatings, hardness and visco-elastic properties, porosity of coatings

1. Introduction

Thermal spray coatings are frequently evaluated regarding a number of features, such as:

- Microstructure
- Hardness
- Tensile strength
- Thickness.

During recent years (last decade) many OEM's have focused on the robustization of the laboratory procedures. Round Robins have been implemented between various laboratories and new standards for testing and preparation for testing have been established.

With the newer laboratory procedures, it is now apparent that many OEM process specifications were developed in a time when laboratory practices were far from optimum (Ref 1). Several examples will be shown below, where unless updated, a new coating (other than the ones that have been used over the last 30-40 years) will be applied.

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It is also necessary to evaluate current measurement techniques and procedures with respect to new guidelines formed through the QS9000 (Ref 2, 3), ISO17025 (Ref 4), AS9003 (Ref 5), ISO10012 (Ref 6), as well as with respect to recommendations from the Six Sigma methodology (Ref 3). Examples of rules being applied are:

- The measurement uncertainty shall be estimated for each measurement process covered by the measurement management system (Ref 6);
- Uncertainty estimations shall be recorded (Ref 6);
- Inaccuracy for the measurement instrument shall be less than 1/10th of actual tolerance to be measured (Ref 7);
- Measurement inaccuracy shall be <30% of tolerance width (at 99.7% confidence) (Ref 3); and
- σ for Measurement inaccuracy divided by σ for production scatter shall be <50% (Ref 3).

2. Microstructure

The microstructure of a thermally sprayed coating typically consists of a multiphase matrix (often a mix between hard, soft, and amorphous), pores, oxides, delaminations, cracks, grit residues, and unmelted particles. Due to this complexity in the structure, there are a number of possible errors that often can be made in metallographic laboratories. Several examples and explanations are given below. It is shown that improper handling in the materials laboratory may lead to smearing and pullout,

etc. This will ruin the results and effectively hide the true microstructure.

2.1 Mounting Procedures

The choice between hot and cold (vacuum) mounting may be the single most important step in the whole metallographic procedure.

A TBC can be significantly altered in its appearance (Fig. 1) (Ref 8, 9).

The influence of the mounting technique was very obvious when Ruckert et al. (Ref 10) evaluated plasma sprayed WC/Co specimens (sprayed by one supplier), but metallographically prepared by 27 different laboratories. The only safe way not to distort the microstructure is vacuum mounting technology (Fig. 2). The true porosity (6%) was achieved and proven through the use of fluorescent dye mixed into the epoxy when performing vacuum mounting.

Using this technology, it is also possible to distinguish between oxide stringers and delaminations. What initially and traditionally used to be considered oxide stringers (Fig. 3), are delaminations or wide splat boundaries (Fig. 4) (the dye lights up all the filled voids). This was also suggested by Wigren et al. (Ref 11) and Leger et al. (Ref 12), showing that the cohesion of the coatings

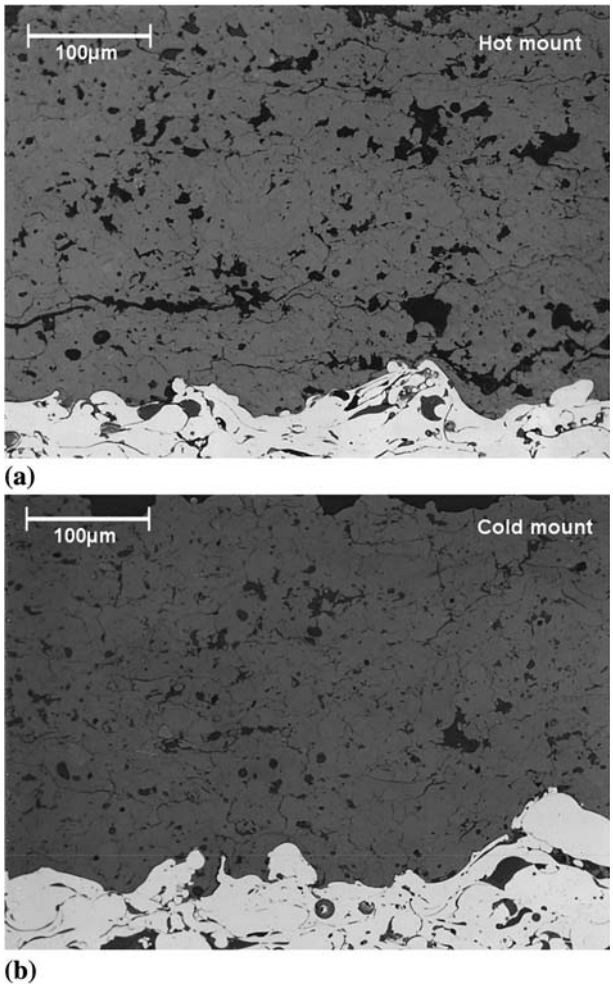

Fig. 1 Difference between hot and cold mounting for a 300 µm zirconia coating (Ref 9)

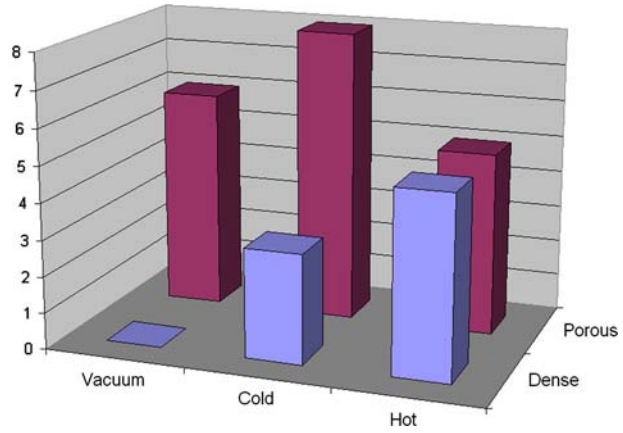

Fig. 2 Achieved porosity of same WC/Co specimen from 27 laboratories using different mounting techniques (Ref 10)

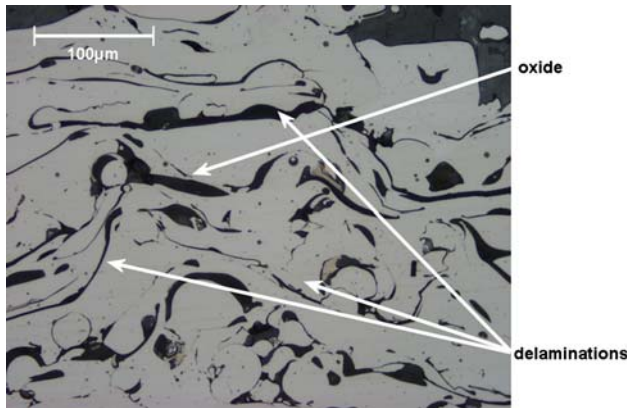

Fig. 3 A typical appearance of an MCrAlY coating in light optical microscopy, 429×343 µm^2

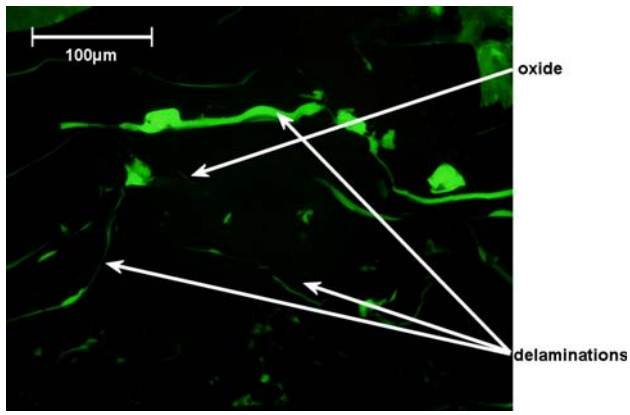

Fig. 4 Same image as Fig. 3, where the fluorescence seen in the splat boundaries and pores indicates a low level of oxide stringers but with a substantial amount of delaminations, 429×343 µm^2

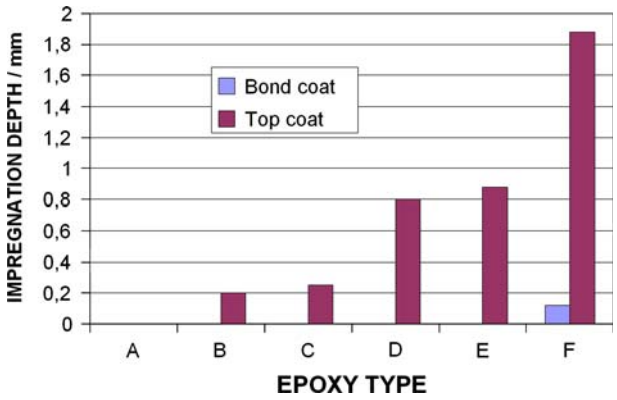

Fig. 5 Impregnation depth in a TBC for different epoxies

strongly correlated to the amount of delaminations (which in turn correlated to the in-flight particle properties).

Thus, penetration of epoxy during mounting is crucial for the following steps in the metallographic procedure, especially the grinding step.

Six (6) commercially available and commonly used cold mounting epoxies were evaluated for impregnation of a 2 mm thick TBC using standard vacuum mounting at 25 mmHg (Ref 8). The depth of impregnation shows a large scatter depending on the choice of epoxy (Fig. 5). In the case of epoxy F, the TBC was completely impregnated all the way through to the bond coat. Epoxy A did not penetrate at all. The penetration efficiency/depth is most likely a result of the viscosity of the mixed epoxy.

2.2 Grinding/Polishing

Many problems can arise from improper grinding/polishing. By far, one of the biggest issues in grinding is the smearing caused by the SiC papers. The most critical are the unfilled pores and cracks in a metallic coating.

Many small cracks were created in a WC/Co coating, by bending the specimen (Fig. 6). On proper metallographic preparation using a grinding disc the crack is visible on a cross section (Fig. 7). However after grinding on SiC papers followed by a brief polishing cycle the crack was effectively hidden (Fig. 8) (Ref 13).

With contrasting mounting and preparation routines, a hot mounted WC/Co specimen ground on SiC-paper followed by short polishing (Fig. 9), shows a dense structure, whereas a vacuum mounted and properly prepared specimen show the true appearance (Fig. 10). The true porosity is easily proven by the use of fluorescence dying techniques. Image analysis reveals 0.2 and 2.3% porosity, respectively.

The explanation is that the matrix material is smeared into the unfilled pores, creating a dense lid on top of the whole coating. As long as the polishing is short enough not to break through this dense lid, the coating will appear dense and perfect. Figures 11 and 12 show this very clearly for an aluminum coating.

It is understood that hot mounting could be acceptable if enough polishing time is applied. However, long polishing

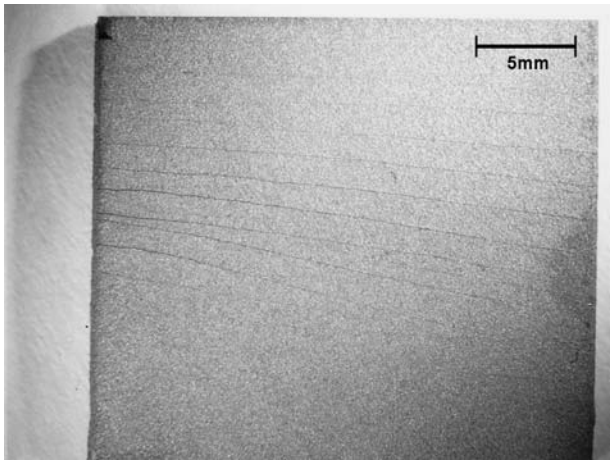

Fig. 6 Cracks initiated (through bend test) in a WC/Co coating. Sample width 26 mm (Ref 13)

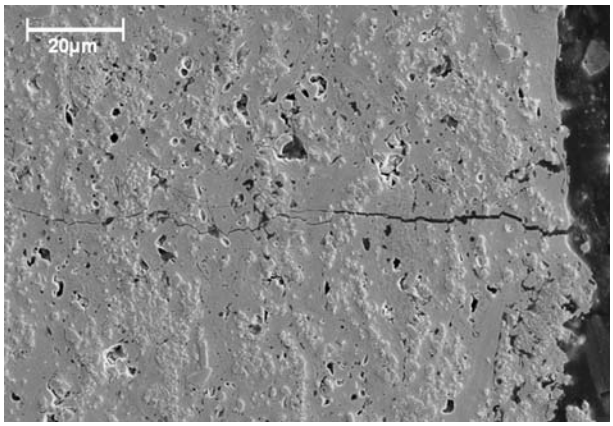

Fig. 7 True microstructure (SEM) of a cracked WC-Co coating (Ref 13). 130×90 µm^2

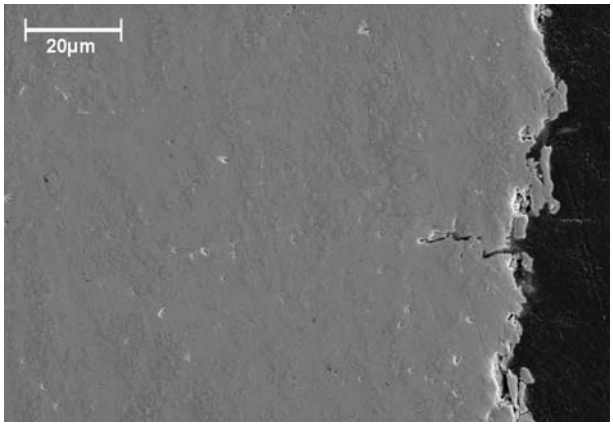

Fig. 8 Same area as Fig. 8, but smeared on SiC paper (Ref 13). 130×90 µm^2

brings forward another problem of edge retention, which causes metallic coatings with oxides and pores to be distorted.

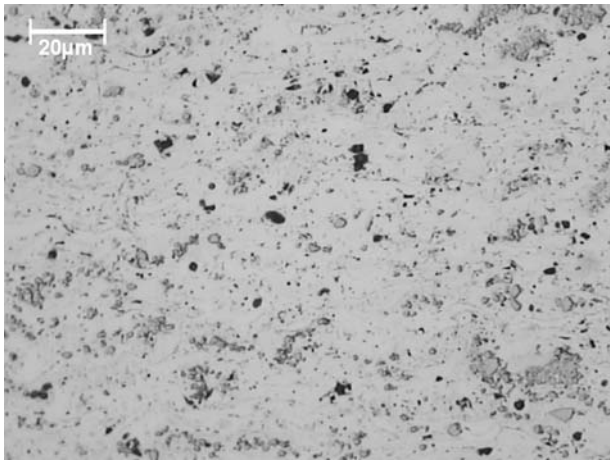

Fig. 9 Hot mounted WC/Co, smeared during grinding and polishing. 169×127 μm^2

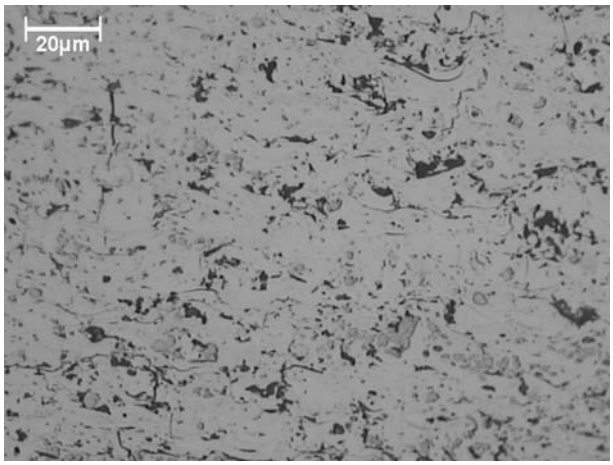

Fig. 10 Vacuum mounted WC-Co properly ground and polished. 169×127 μm^2

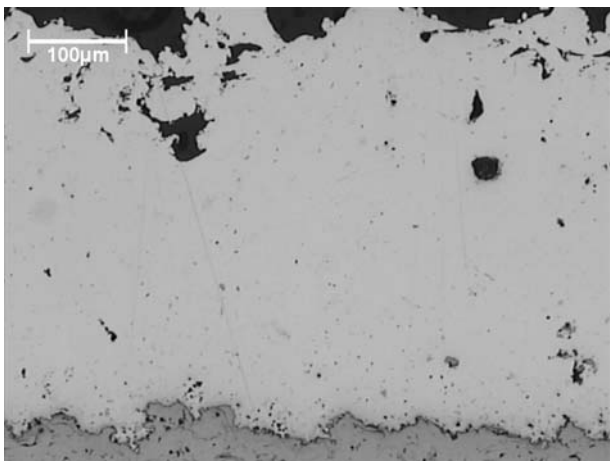

Fig. 11 Smeared aluminum coating by hot mounting. 636×477 μm^2

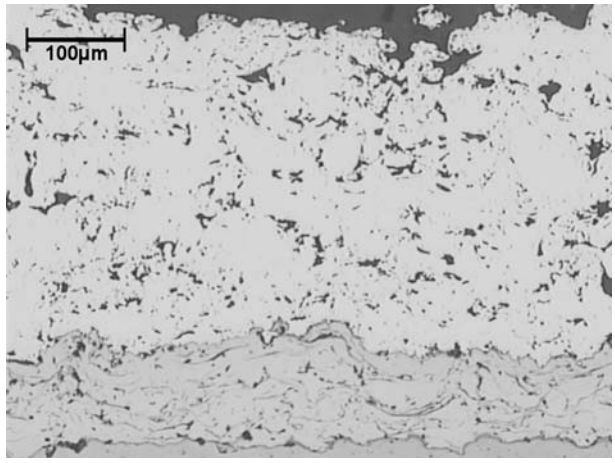

Fig. 12 Same as Fig. 11 but using vacuum mounting. All pores are filled with epoxy (fluorescence) indicating true voids. 636×477 μm^2

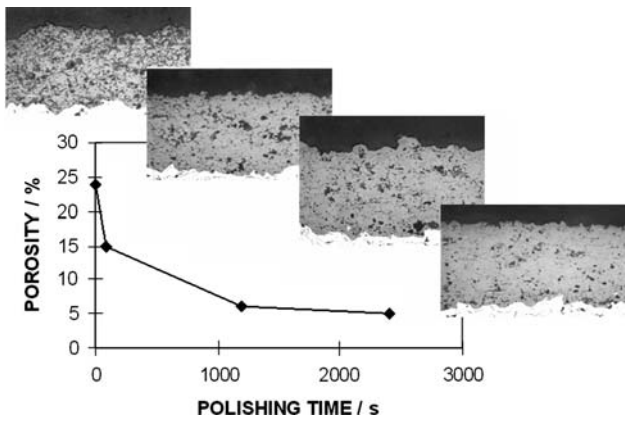

Fig. 13 The effect of polishing on the appearance of porosity in a TBC (Ref 9)

The width of pores and oxides can also be enlarged.

Ceramic coatings can cause other grinding/polishing issues. Many times long polishing times are necessary, because of the damage created by the grinding steps (Fig. 13).

2.3 Measurement Techniques

Measurement system analysis (MSA) using Image analysis indicates that the porosity scatter, 3 sigma, due to the laboratory procedures (polishing, etc.) is 1.42% absolute porosity. This implies that a porosity tolerance width should be at least 9.5% (±4.8%), per the rule of measurement uncertainty to be <30%.

Standard current procedures, however, involves photographic comparisons to reference photographs, which logically is much less accurate. Typically reference photographs at even 5% intervals are used. An estimate is that the measurement uncertainty increases to about a half photo range, which corresponds to 2.5%, which in turn

suggests a tolerance width of about 17%, i.e., the range of at least three photographic standards.

A measurement uncertainty of 2.5% (absolute porosity) at this porosity level (10%) corresponds to a relative measurement uncertainty of 50% (±25% or a half photostandard).

3. Thickness Measurement

Eventually, the method for thickness evaluation can also be questioned. What thickness can one expect of a 100 μm coating? Depending on the technique used, 100 μm can mean several things. The thickness evaluation when plasma spraying a Ni-5Al coating is illustrated in Fig. 14, where a comparison of two microscope techniques (average and maximum readings) and two types of micrometer (flat and ball end) are made. The thicknesses are given stroke (2 passes) by stroke during the coating build up.

A 50 μm thick metallic coating measured with flat micrometer did not have full metallographic coverage in the above investigation, whereas 50 μm measured with a ball end micrometer did.

A measurement system analysis among five well-experienced operators and five different specimens (five readings on each) was conducted as shown in Table 1. It shows that the only way to make current micrometer measurement procedures to conform to state of art guidelines is to use a thickness tolerance width of >200 μm (based on the rule that measurement inaccuracy shall be <30% than the tolerance width (at 99.7% confidence).

In this case, it can also be concluded that a standard micrometer tool for OD measurements can only be used for tolerance widths of 50 μm (based on the requirements that the tool resolution shall be better than 1/10th of the tolerance), just from the tool point of view.

With this example only four solutions can be found:

i) Increase the tolerance width
ii) Major improvement of the micrometer measurement procedure
iii) Averaging from a much larger amount of individual measurements
iv) Find a new tool for measurement.

Table 1 Example of Gage RR on a chrome carbide/nickel chrome plasma sprayed coating

Property	Result	Comment
Tolerance width	280-350 μm	From drawing
σ MSA	9.8 μm	Result of GaugeRR
6σ MSA	58.8 μm	99.7% confidence
6σ/Tolerance width	84%	Req. <30%

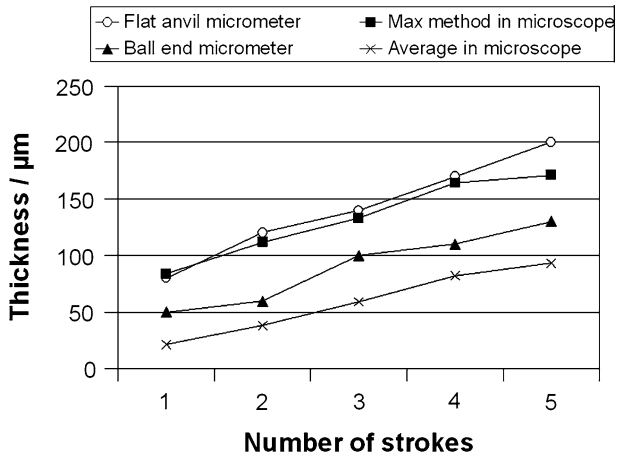

Fig. 14 Thickness evaluation during plasma spraying of a Ni5Al coating as measured by different techniques (1 stroke = 2 passes)

4. Tensile Bond Strength Test

The mechanical disadvantages with the standard tensile bond strength test method (Ref 14) have been previously discussed (Ref 15, 16). Evans et al. described some apparent parameters that need control (Ref 17).

Unfortunately, it appears that the tensile strength achieved on a thermal sprayed coating is very dependent on the penetration of the epoxy and pressure being used during curing. The tensile strength of a Ni-5Al/Alumina system can vary from 60 down to 15 MPa (Fig. 15). In cases, where the coating is dense enough to prevent epoxy penetration (such as WC/Co) of the glue, this tensile variation is not seen.

It is interesting to understand that the failure occurs at different positions depending on the epoxy penetration. Using the low viscous EC2214 (leading to complete penetration) the failure occurs in the epoxy (Fig. 16). With the highly viscous FM1000, however, the failure occurs at the bond-top interface (Fig. 17).

The low robustness of the procedure is also illustrated by the variation at different test occasions (Fig. 18), which ranges from only about 10 MPa up to 30 MPa.

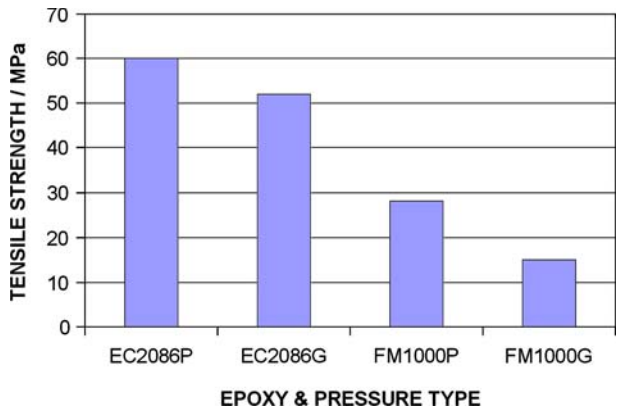

Fig. 15 Tensile strength of a Ni-5Al/Alumina system using different epoxies and gravity (G) vs. pressure bonding (P)

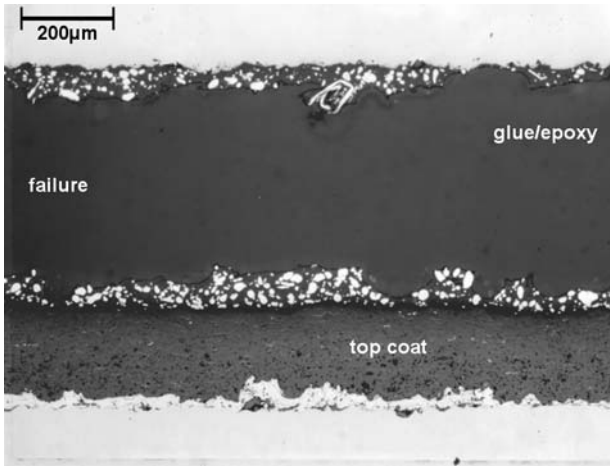

Fig. 16 Failure in epoxy for tensile test of Ni-5Al/Alumina using EC 2214

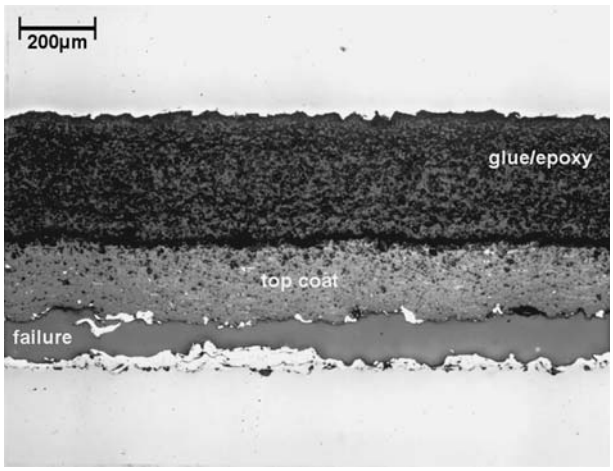

Fig. 17 Failure in the top/bond interface for tensile test of Ni-5Al/Alumina using FM1000

A measurement system analysis (MSA) was performed using 100:s of historical data points for a variety of coatings. This indicates that the measurement inaccuracy (6σ) for tensile strength testing varies from 10 to 30% actual value (depending on the coating and substrate material). Using the rule that measurement inaccuracy shall be <30% of tolerance width (99.7% confidence), this means the tolerance widths should be as listed in Table 2.

5. Hardness

5.1 Micro Hardness

Micro hardness is generally performed as per ASTM E384 (Ref 18). A first general misconception is the number of indentations. It is easily shown that 10 indentations are by far too few (Fig. 19).

A second issue is the metallographic preparation of the specimen before measurement. A preparation routine that tends to smear the coating (i.e., the use of SiC paper) leads to roughly 100 Vickers lower measurement, Fig. 20 and Table 3, compared to a coating prepared by a nonsmearing procedure.

The performed measurement system (based on operator round robin tests) indicated that a tolerance width of 400 $HV_{0.3}$ (±200) is just at the borderline for acceptance, when using the rule that the uncertainty divided by the

Table 2 Minimum required tolerance width on drawing for tensile strength

Level	Minimum tolerance (MPa) width at diff. relative 6σ inaccuracy		
	10%	20%	30%
1 MPa	±0.17	±0.34	±0.5
10 MPa	±1.7	±3.4	±5
50 MPa	±8.5	±17	±25
100 MPa	±17	±34	±50

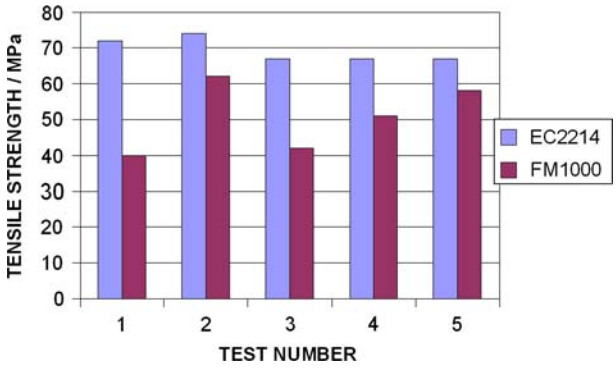

Fig. 18 Difference in tensile strength for five different curing occasions of Ni-5Al coatings

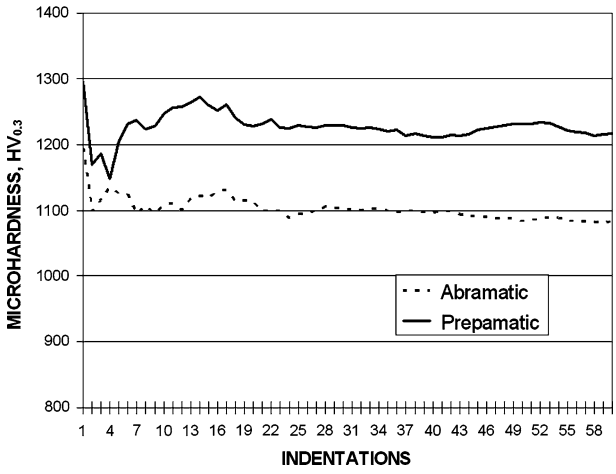

Fig. 19 Micro hardness for a WC/Co showed as running average per indentations

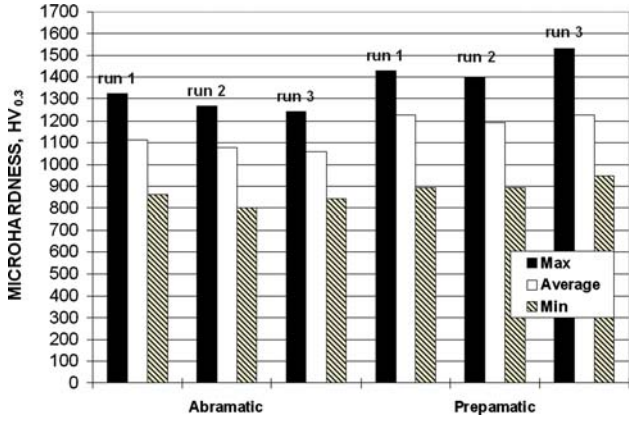

Fig. 20 The effect of the metallographic preparation on micro hardness of a plasma sprayed WC/Co coating, using different (Abramatic and Prepamatic) equipment

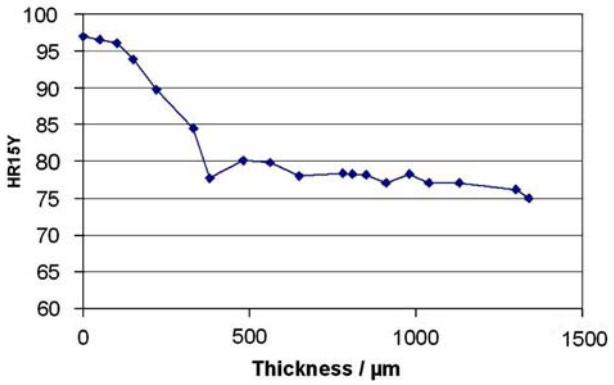

Fig. 21 HR15Y vs. thickness of a NiC coating

Table 3 The effect of metallographic preparation on micro hardness in $HV_{0.3}$ for a WC/Co coated specimen

	Abramatic	Prepamatic
Average	1083	1217
Std Dev	116	141
Std Dev/√n	15	18

tolerance width shall be less than 30% at 99.7% confidence.

5.2 Macro Hardness

It is of utmost importance to measure macro hardness (Ref 19) on the proper coating thickness. Sometimes this is thicker than one may expect. If there is insufficient coating to support the hardness indenter, the reading will be a combination of the coating and coupon substrate material. For example a NiC-graphite coating needs at least about 1 mm (Fig. 21).

A measurement system analysis for HR15Y at the level of 40 shows a standard deviation of 1.08, which suggests that the tolerance width need to be at least 22 to achieve 99.7% confidence.

Similar analysis for HR15N at a level of 80. The obtained standard deviation was 0.59, which suggests a tolerance width of 12 (±6). However, by using 20 indentations instead of 10 the tolerance width requirement is lowered to 6 (±3).

6. Conclusion/Summary

With the robustization and development of optimal laboratory procedures for thermal spray coating evaluation, it is evident that techniques previously used did not reveal the true structure and mechanical properties of the coatings. This new data can and will require older processing specifications with coating property limits to be reviewed and revized.

True microstructure features (cracks, porosities), that never were seen previously, are now revealed with cold mount techniques using low viscosity epoxies, and thus not allowed in the specifications.

Tensile strength requirements initially set by using liquid epoxies need correction to conform to the true readings of high viscous epoxies (such as film adhesive FM1000).

Different grinding techniques (and amount of indentations) will significantly change the micro and macro hardness readings.

The measurement tool (flat or ball point micrometer) used for thickness measurements has a significant effect on the reading.

Tolerances for most properties would have to be adjusted to be able to adapt to the recent requirements on measurement tool and measurement system analysis for the various specified methods and procedures.

If correction/update of specifications is not undertaken, there is a major risk that coating suppliers will have to develop a new process (for the same old application). This could result in coating properties completely different from design intent and years of experience from part performance will be of limited use.

Acknowledgments

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