

TECH NOTES

Preparation of Thermally Sprayed Coatings!

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Thermally Sprayed Coating Metallography

Thermally sprayed coatings are used to improve the resistance of substrate materials to oxidation, corrosion, surface wear and erosion. The accurate characterization of coated metal components requires metallographic examination of their microstructures. Coating thicknesses range from 0.002 to 0.060 inches (0.005 to 1.5mm) and are deposited on substrates using various spray techniques and parameters. The microstructural characteristics must be accurately determined using metallographic preparation techniques. Due to the brittle nature of some coatings and the occurrence of pores and constituents of widely different hardnesses in the layered structure, there is always a danger that metallographic preparation may either fail to expose the true microstructure or induce artifact that will lead to a misinterpretation of the coating characteristics. Light optical microscopy provides a means by which a properly prepared coating specimen can be evaluated to determine the quality of the coating/substrate interface, the amount of porosity, unmelted particles and oxide distribution, coating thickness and other coating characteristics as shown in Figure 1.

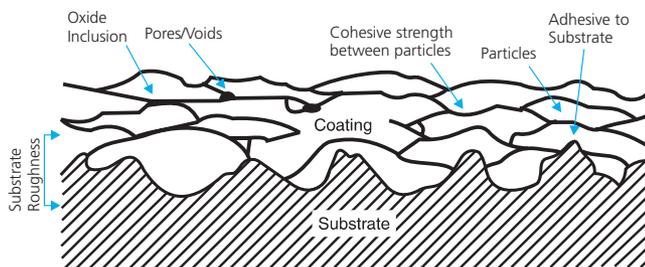


Figure 1. Typical coating cross section to illustrate lamellar structure of oxides and inclusions.

Metallographic techniques used to prepare thermally coated specimens for microstructural evaluation vary from laboratory to laboratory. The variation often produces marginal to acceptable results. These techniques involve the use of silicon carbide abrasive paper, fixed or semi-fixed diamond for the coarse and fine stages of grinding. A graded series of diamond coarse or suspensions on a napless cloth is used for the rough polishing stage and either a fine diamond paste or suspension or sub-micron aluminum oxide powder on either napped or napless cloths is used for the final polishing stage. Improper techniques when using any of these consumables or surfaces will produce less than accurate results.

The purpose of this issue of BUEHLER® Tech-Notes is to provide the

Table 1. Sectioning Parameters

Equipment	ISOMET® 2000
Blade	Diamond (15HC) or Aluminum Oxide (thing)
Speed	2500 rpm or 4000 rpm
Applied Load	600gms or 500gms
Lubricant	ISOCUT® Plus (water soluble)

reader with information pertaining to metallographic preparation procedures for thermally sprayed coatings that permits consistently accurate coating characterizations.

Metallographic Specimen Preparation

Sampling/Sectioning

The various types of thermally sprayed specimens should be sectioned perpendicular to their axis using a precision diamond saw with a metal bonded diamond wafering blade or ultra-thin aluminum oxide blade. The specimen should be clamped in the vise and positioned so that the blade enters the coated side and exits the substrate side, thus substantially reducing coating damage. Figure 2 shows the suggested wheel rotation, specimen position and specimen type when sectioning thermally sprayed coatings. Vacuum impregnating a thin epoxy layer on a porous or friable coating will prevent casting damage during sectioning. The sectioning parameters are shown in Table 1. Each sectioned specimen should be thoroughly cleaned in acetone and dried in a 70°C oven for 5 minutes prior to encapsulation.

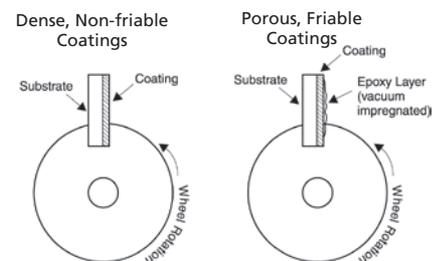


Figure 2. Proper sectioning of thermally sprayed coatings.

Mounting

Mounting protects the coating from destructive attack and provides edge protection during the grinding and polishing procedures. Metallographic encapsulation falls into two categories, compression

molding and castable epoxy. Although there are some coatings that can be mounted in the compressive mode, the castable epoxy practice has become the preferred method of encapsulation. This method is free from high pressure and temperature which tend to cause damage to friable coatings. The use of EPO-KWICK®, Epoxide or EPO-THIN® is acceptable for proper encapsulation. Each specimen should be placed in the Vacuum Impregnation System and the chamber should be evacuated to 26 inches Hg. The epoxy is then poured into each SAMPL-KUP® and held for 5-10 minutes to provide infiltration of the open pores in the coating. Figure 3 is a diagram showing the two encapsulation methods and the suggested type of coating for each method.

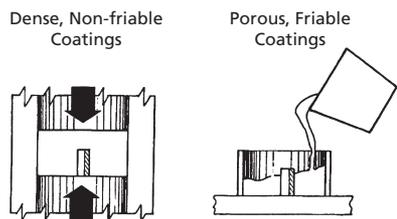


Figure 3. Proper encapsulation of thermally sprayed coatings

Planar Grinding

This first stage prepares the specimen for the subsequent critical Specimen Integrity Stage. It has a powerful influence on the success of the entire specimen preparation process because it removes the initial depth of deformation from cutting in addition to producing a flat surface. When properly performed, it will lead to fewer subsequent steps. The damage produced by each preparation step must be removed by the subsequent step. Thermally sprayed specimens can be planar ground using a coarse (120 or 180 grit) silicon carbide abrasive paper or fixed 45µm diamond grinding disc (ULTRA-PREP™).



ISOMET® 2000 Precision Saw

Each abrasive type will create a planar surface in a short time. The fixed diamond disc will produce high material removal rates while the silicon carbide papers will not be as aggressive. Proper selection of the abrasive surface is important so that serious coating damage is avoided. Thorough cleaning and drying of the specimens should be accomplished prior to proceeding to the next stage. Ultrasonic cleaning is not universally accepted, therefore should be used with minimal times to avoid induced damage.



Vacuum Impregnation Equipment

Specimen Integrity Stage

The ultimate success of the entire procedure depends upon the ability of this stage to remove the deformation produced by planar grinding leaving considerably less surface damage. The true integrity of the coating and substrate should be achieved at the completion of this stage of preparation. The two methods shown in Tables 2 and 3 list two different abrasive methods to achieve the desired specimen integrity results. Silicon carbide abrasive papers of decreasing grit size are effective but costly. Effective use of a semifixed polycrystalline (PC) diamond suspension (9µm) on a hard polyester cloth (ULTRA-PAD™) maintains the planarity that is established during planar grinding and removes the surface deformation from that stage. This process is more cost effective than the use of multiple abrasive papers. The two Specimen Integrity steps listed in Table 3 replace five of the six silicon carbide abrasive papers listed in Table 2 while maintaining a far greater degree of planarity and virtually eliminating microstructural relief or surface deformation. Fine scratches left by the ultra fine abrasive paper and semi-fixed polycrystalline diamond are easily removed by using a TEXTMET® 1000 cloth charged with 3µm polycrystalline diamond suspension.

Final Polishing Stage

Very fine abrasives (sub-micron) are used for the Final Polishing Stage and only remove small amounts of material; therefore, they are incapable of removing substantial surface damage or deformation not removed by the previous steps. Rotating napped cloths exert a selective abrasive action that can cause microstructural relief and edge rounding on harder coatings if performed for extended times. If the Planar Grinding and Specimen Integrity Stages are performed correctly, the Final Polishing Stage will be brief in duration yet extremely effective in producing a clean and undistorted microstructure without causing relief or edge rounding. The use of CHEMOMET® Cloth charged with MASTERPREP™ (0.05µm AL2O3) was used to achieve a scratch-free, deformation-free surface.

Microstructural Evaluation

The use of conventional as well as new abrasive and surface technology produces consistently good results on a variety of thermally sprayed coatings and substrates. The as-polished surfaces

Table 2. Conventional Preparation

Stage	Surface	Lubricant	Abrasive Type/ Size	Time (sec.)	lbs/ sple	Head Speed	Base Speed	Head Rot
Planar Grinding	SiC Paper	Water	180grit	60	5	60 rpm	240 rpm	comp.
	SiC Paper	Water	240grit	25	5	60 rpm	240 rpm	comp.
	SiC Paper	Water	320grit	25	5	60 rpm	240 rpm	comp.
Fine Grinding	SiC Paper	Water	400grit	25	5	60 rpm	240 rpm	comp.
	SiC Paper	Water	600grit	25	5	60 rpm	240 rpm	comp.
	SiC Paper	Water	800grit	25	5	60 rpm	240 rpm	comp.
Rough Polishing	TEXTMET®		3µ PC diamond	180	6	60 rpm	120 rpm	comp.
Final Polishing	CHEMOMET®		MASTERPREP™	90	5	60 rpm	120 rpm	comp.

Table 2. Advanced Preparation

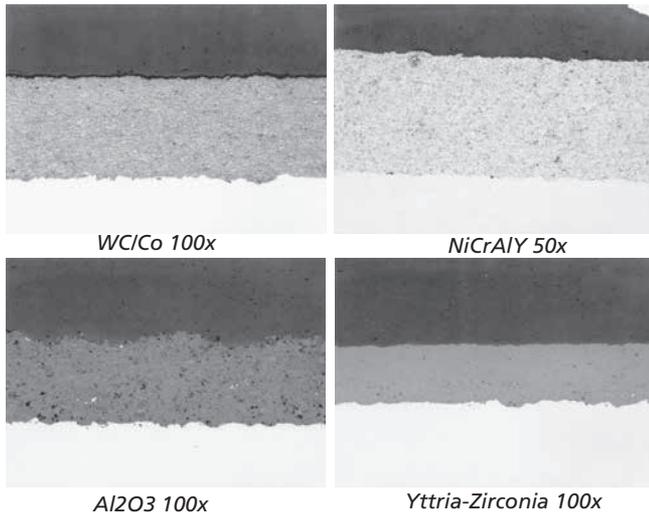
Stage	Surface	Lubricant	Abrasive Type/ Size	Time (sec.)	lbs/ sple	Head Speed	Base Speed	Head Rot
Planar Grinding	ULTRAPREP™	Water	45µm diamond disc	60	5	60 rpm	300 rpm	comp.
Specimen Integrity	ULTRA-PAD™		9µm PC diamond	360	5	60 rpm	200 rpm	comp.
	TEXMET® 1000		3µm PC diamond	180	6	60 rpm	120 rpm	comp.
Final Polishing	CHEMOMET®		MASTERPREP™	90	5	60 rpm	120 rpm	comp.

reveal the integrity of the coating-substrate interface, the amount of porosity and unmelted particles, the presence of oxides and general overall coating thickness. This information provides the evaluator with an accurate picture of the microstructural characteristics of the coating enabling him/her to make decisions pertaining to coating quality with a greater degree of confidence.

Conclusion

Microstructural evaluation of all phases of thermally sprayed coatings can successfully be accomplished using state-of-the-art abrasives, surfaces and equipment. Semi-automatic and fully automatic specimen preparation equipment, in conjunction with new, cost effective abrasives and surfaces, provides consistent, accurate, distortion-free surfaces for the evaluation of the coating characteristics. The use of this equipment and processes will enable thermal spray suppliers, quality assurance laboratories and the end user to have a high confidence level in the final product. Manual preparation of thermally sprayed coatings requires a highly skilled metallographer with special abilities in order to consistently produce accurate results. Due to the absence of such individuals in laboratories today, manual preparation is not recommended.

Thermally Sprayed Coating Characteristics



Tech-Tips

Question: How would I determine the cause of coating damage accurately?

Answer: The best way to eliminate questions as to when the damage occurred is to encapsulate the entire as-received coating

specimen in a castable epoxy (EPO-THIN® or Epoxide) in a vacuum environment of 27" Hg. This procedure will fill all of the open pores, voids and defects prior to the sectioning operation. The pre-encapsulated coating can then be sectioned on a precision saw (ISOMET® 2000 or ISOMET 1000) using a diamond wafering blade (15HC). Production cutters using thick aluminum oxide blades cause localized overheating of the specimen even when the specimen is preencapsulated, thus are not recommended for sectioning coatings for critical evaluation. The sectioned specimen can then be metallography prepared using sequentially diminishing SiC abrasive papers (180, 240, 320, 400, 600 and 800 grit) and a rough (3µm polycrystalline diamond) and final polish (0.05µm Al2O3 -- MASTERPREP™). This procedure will produce reliable results revealing the true nature of the coating.

Question: Is thermosetting mounting of thermally sprayed coatings an acceptable practice for all types of coatings?

Answer: Due to the presence of high temperature (150°C) and high pressure (4200 psi) there is a possibility of damaging the coating. Dense, nonceramic coatings such as WC/Co, or other dense metallic coatings, can successfully be mounted in a mounting press without creating damage. All other coatings should be mounted in a castable epoxy, such as Epoxide, EPO-KWICK®, EPO-THIN® or EPO-COLOR™, in a vacuum of 27" Hg. Castable epoxies set up at low temperatures (75°F to 170°F) and ambient pressure, thus eliminating the possibility of damaging the coating and allowing for the accurate evaluation of the coating characteristics.

Question: Why do some thermal spray laboratories mount the uncut side of a coating specimen as opposed to the cut surface? Is this acceptable practice?

Answer: This practice is performed in laboratories that do not have precision diamond saws to section their thermal spray coating specimens. Production cutters use thick aluminum oxide abrasive blades that create excessive damage to the coating during sectioning and would require excessive grinding to remove the deformation. Mounting the uncut surface was supposed to eliminate this problem, however, this procedure required the removal of 1-2mm of material during the planar grinding step. New, high speed precision diamond saws (ISOMET 2000 or ISOMET 1000) create minimal damage to the coating and allow the laboratory person to mount the specimens on the cut surface. Both methods are acceptable but the diamond precision saw process reduces the amount of work done during the initial stages of metallographic preparation.

Sectioning AbrasiMet • AbrasiMatic • IsoMet	Mounting SimpliMet	Grinding & Polishing EcoMet • AutoMet • MetaServ	Imaging & Analysis OmniMet	Hardness Testing Wilson® Hardness
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