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Standard Guide for Metallographic Preparation of Thermal Sprayed Coatings[1]

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1. Scope

1.1 This guide covers recommendations for sectioning, cleaning, mounting, grinding, and polishing to reveal the microstructural features of thermal sprayed coatings (TSCs) and the substrates to which they are applied when examined microscopically. Because of the diversity of available equipment, the wide variety of coating and substrate combinations, and the sensitivity of these specimens to preparation technique, the existence of a series of recommended methods for metallographic preparation of thermal sprayed coating specimens is helpful. Adherence to this guide will provide practitioners with consistent and reproducible results. Additional information concerning standard practices for metallographic preparation can be found in Practice E3.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *linear detachment, n*—a region within a TSC in which two successively deposited splats of coating material have not metallurgically bonded.

3.2.2 *splat, n*—an individual globule of thermal sprayed material that has been deposited on a substrate.

3.2.3 *taper mount, n*—a metallographic specimen created by mounting a feature, typically an interface or thin coating, at a small angle to the polishing plane, such that the visible width exhibited by the feature is expanded.

3.2.4 *TSC, n*—thermal sprayed coating, including, but not limited to, those formed by plasma, flame, and high velocity oxyfuel.

4. Significance and Use

4.1 TSCs are used in a number of critical industrial components. TSCs can be expected to contain measurable levels of porosity and linear detachment. Accurate and consistent evaluation of specimens is essential to ensure the integrity of the coating and proper adherence to the substrate.

4.1.1 *Example 1:* By use of inappropriate metallographic methods, the apparent amount of porosity and linear detachment displayed by a given specimen can be increased, by excessive edge rounding, or decreased by smearing of material into voids. Therefore inaccurate levels of porosity and linear detachment will be reported even when the accuracy of the measurement technique is acceptable.

4.1.2 *Example 2:* Inconsistent metallographic preparation methods can cause the apparent amount of voids to vary excessively indicating a poorly controlled thermal spray process, while the use of consistent practice will regularly display the true microstructure and verify the consistency of the thermal spray process.

4.2 During the development of TSC procedures, metallographic information is necessary to validate the efficacy of a specific application.

4.3 Cross sections are usually taken perpendicular to the long axis of the specimen and prepared to reveal information concerning the following:

4.3.1 Variations in structure from surface to substrate,

4.3.2 The distribution of unmelted particles throughout the coating,

4.3.3 The distribution of linear detachment throughout the coating,

4.3.4 The distribution of porosity throughout the coating,

4.3.5 The presence of contamination within the coating,

4.3.6 The thickness of the coating (top coat and bond coat, where applicable),

4.3.7 The presence of interfacial contamination,

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4.3.8 The integrity of the interface between the coating and substrate, and,

4.3.9 The integrity of the coating microstructure with respect to chemistry.

5. Selection of Metallographic Specimens

5.1 Selection of specimens for metallographic examination is critical if their interpretation is to be of value. Specimens must be representative of the coating. Generally, the plane of polish should be normal to the coating surface so as to display the entire coating thickness, the substrate, and all interfaces.

6. Sectioning

6.1 Specimens to be mounted for metallographic preparation are generally not larger than 12 mm by 25 mm (0.5 by 1.0 in.). The height of the mounted specimen should be no greater than necessary for convenient handling during polishing.

6.2 In sectioning TSC specimens, care must be exercised to avoid affecting the soundness of the coating and the interface between the coating and the substrate. Sectioning damage of the coating and interface that cannot be removed by subsequent grinding and polishing must be avoided.

6.2.1 Friable, porous, or brittle coatings to be sectioned may be vacuum impregnated with epoxy mounting compound before sectioning to protect the specimen.

6.2.2 Specimens should always be sectioned such that the coating is compressed into the substrate. Sectioning techniques which place the coating and interface in tension are strictly to be avoided. Sectioning in tension may cause the coating to be pulled away from the substrate or result in delamination of the coating. During examination of the polished specimens, it is likely that this type of damage will be mistakenly interpreted. When sectioning some specimens, it may not be possible to avoid placing some areas of the TSC in tension. These areas should be noted and not included in the evaluation of the specimen.

6.2.3 Sectioning with a hack saw will produce significant damage to the coating and interface and is not considered acceptable.

6.2.4 Using an abrasive cutoff blade with a large particle size abrasive produces a smoother surface than a hack saw, but still produces coating damage that may require considerable grinding in subsequent preparation to remove. The choice of cutoff wheel, coolant, cutting conditions, and the type and hardness of the coating and substrate will influence the quality of the cut surface. A poor choice of cutting conditions can easily overheat some TSC specimens rendering the specimens unusable for proper evaluation.

6.2.5 Sectioning can be completed with minimal damage to the cut surface by selection of one of the two following abrasive cutoff blades:

6.2.5.1 Use a diamond wafering blade with a maximum thickness of 0.63 mm (0.025 in.).

6.2.5.2 Use an ultra–thin aluminum oxide abrasive blade approximately 0.76 mm (0.030 in.) thick, which will break down during cutting to help reduce sectioning damage.

6.2.6 Faster blade speeds, 1675 m/min. (5500 surface ft/min.) or greater, produce less coating damage. Slower blade speeds will result in more damage to the cut surface and are not recommended.

6.2.7 Generally, an abrasive cutoff blade selected to cut the substrate effectively will be the best blade for the combination of TSC and substrate.

7. Cleaning

7.1 Cleaning of specimens prior to mounting is essential. All sectioning coolant shall be removed from the surface and from any porosity connected to the surface. Use of an organic solvent to aid in fluid removal and thorough drying is necessary. Drying in an oven at low temperature (60 to 80°C or 140 to 176°F) can accelerate this process. Any liquid residual may impede impregnation of porosity, as well as retard the curing of mounting compounds causing difficulty during grinding and polishing.

7.2 Ultrasonic cleaning of TSC specimens is generally not recommended, especially for fragile or brittle coatings, because coating particles may be lost during this energetic cleaning process. If ultrasonic cleaning is found to be necessary, cleaning time should be kept to a minimum.

8. Mounting

8.1 *General Information:*

8.1.1 It is always necessary to mount TSC specimens to maintain the original structure of the specimen during grinding and polishing. Both compression mounting and castable mounting compounds are commonly used when mounting TSC specimens. However, only castable epoxy mounting compounds should be used in the initial determination of the true characteristics of a coating before considering the use of any other mounting compound. For some TSC specimens castable epoxy may provide the only acceptable mount. Refer to Table 1 and Table 2 and Practice E3 for characteristics of various mounting compounds.

8.1.2 By placing pairs of specimens in the same mount, time and expense can be saved. When using this mounting method,

TABLE 1 Characteristics of Compression Mounting Compounds

Type of Compound[A]	Characteristics
Acrylic	Cure time 10 to 15 min., optically clear, thermoplastic, good impregnation, low hardness, degraded by hot etchants
Diallyl phthalate	Cure time 5 to 10 min., opaque, thermosetting, minimal shrinkage, good resistance to etchants, high hardness
Epoxy	Cure time 5 to 10 min., opaque, thermosetting, minimal shrinkage, good resistance to etchants, high hardness, good impregnation
Phenolic	Cure time 5 to 10 min., opaque, thermosetting, shrinkage during cure can leave crevice at specimen interface, degraded by hot etchants, moderate hardness

[A] These compounds often contain filler materials, such as glass fibers or mineral particulate.

TABLE 2 Characteristics of Castable Mounting Compounds

Type of Compound	Characteristics
Acrylic	Cure time 8 to 15 min, moderate shrinkage, low hardness, opaque
Epoxy	Cure time 1 to 12 h, negligible shrinkage, low level of heat generated during polymerization, moderate hardness, transparent, low viscosity formula is best for vacuum impregnation
Polyester	Cure time 30 to 60 min, high shrinkage, peak curing temperature up to 120°C (248°F), moderate hardness, transparent
Polyester–Acrylic	Cure time 8 to 15 min, negligible shrinkage, peak curing temperature up to 120°C (248°F), high hardness, opaque

the two coated surfaces should face each other. It may be possible to place more than one pair of specimens in a single mount.

8.1.3 Mounting expenses may be reduced by employing the *sandwich* mount technique. A sandwich mount is made by using a small amount of a better, more expensive, mounting compound as the critical layer in contact with the specimen and then topping off the mount with a less expensive compound. Care must be taken to use only mounting materials that are compatible.

8.1.4 Taper mounting may be useful for examination of the interfaces between bond coating and top coating, as well as between coating and substrate.

8.1.5 All components of mounting compounds including resins and catalysts, as well as any dyes or colorants, should be handled in accordance with the manufacturer's instructions. Material Safety Data Sheets are available from the manufacturer.

8.2 *Compression Mounting Compounds:*

8.2.1 Curing of compression mounting compounds is accomplished by the use of a heated press designed for and dedicated to metallographic mounting. Compression mounting compounds require the use of heat (140 to 150°C or 284 to 302°F) and pressure (up to 29 MPa or 4200 psi) to properly cure the mount. Therefore, mounting compounds of this type should only be used to mount dense, non–friable coatings with substrates a minimum of 1.5 mm (0.060 in.) thick.

8.2.2 Compression mounts should be cooled to below 40°C (104°F) while under pressure, preferably by use of a water cooled mounting press, to prevent formation of a crevice between the mounting compound and the specimen as they contract unevenly during cooling.

8.3 *Castable Mounting Compounds:*

8.3.1 Castable mounting compounds may be used for all TSC specimens, but are particularly useful for porous specimens. These mounting compounds are usually prepared by mixing two components just prior to use. The specimen is placed in a mold, usually a cup or ringform, into which the compound is poured.

8.3.2 Vacuum impregnation of porous specimens with an epoxy resin is required. The specimen and mold are put into a vacuum chamber which will allow the castable epoxy to be poured into the mold after the chamber has been evacuated. Vacuum pressure should be in the range of 630 to 760 mm (25 to 30 in.) of mercury. The vacuum should be maintained for about two to ten min before allowing air into the chamber. Low viscosity epoxy resins are recommended for best results during vacuum infiltration and subsequent preparation of TSC specimens.

8.3.3 Addition of fluorescent dyes (**1**) or other agents, such as Rhodamine B (**2**), that produce color response upon fluorescent excitation or with crossed polarized illumination, respectively, to the castable compound during mixing will aid in the identification of impregnated porosity during microscopical examination.

8.3.4 Care should be used when handling castable mounting compounds. Supplier's instructions for the use and handling of these materials should be followed.

9. Grinding and Polishing

9.1 Due to the many different types of TSCs and substrate materials, grinding and polishing sequences will vary. Proper choice of polishing surface, lubricant, abrasive type and size, time, force, polishing wheel speed and relative rotation will produce accurate and consistent results. Some TSCs may require specialized preparation techniques. However, many coatings can be prepared on automatic and semi–automatic polishing equipment by use of one of the three procedures listed in Table 3, Table 4, and Table 5.

9.1.1 Use of automatic and semi–automatic polishing equipment has been found to reduce inconsistency in polished specimens and is therefore required in the metallographic preparation for TSC specimens. These machines provide reproducibility in wheel speed, specimen position on the wheel, applied pressure, relative rotation, and polishing time. Manual grinding and polishing is not recommended because of the inherent inconsistency and the sensitivity of these specimens to preparation technique. (**3**,**4**,**5**,**6**)

9.1.2 Most of the equipment for automatic and semi–automatic grinding and polishing move the specimen around a rotating wheel covered with abrasive so that the specimen follows an epicycloidal path. The scratch pattern consists of random arcs, and upon inspection, should be uniform across the entire specimen surface before proceeding to the next step in the preparation sequence.

9.1.3 Each of the methods outlined in Table 3, Table 4, and Table 5 requires the following four basic preparation steps:

9.1.3.1 Planar grinding is used to remove large amounts of material rapidly and to bring the group of specimens in the holder into coplanarity. This step is necessary to avoid a situation where one or more specimens in the holder do not contact the abrasive while the specimens in contact receive excessive pressure. Planar grinding of specimens also should remove the layer of deformed material resulting from the previous sectioning operation.

9.1.3.2 Fine grinding removes disturbed material from the planar grinding step while introducing a layer of disturbed material acceptable for the next step in the preparation sequence.

9.1.3.3 Rough polishing is accomplished with a 3 to 6 µm diamond abrasive. In some cases, when the surface of the

TABLE 3 Preparation Method I—Silicon Carbide Abrasive Paper Grinding

Surface	Coolant/ Lubricant	Abrasive Size/Type ANSI[A] [FEPA][B]	Time, s	Force[C], n [lbs]	Surface Speed, RPM	Relative Rotation
Planar Grinding						
Paper	Water	180 [P180] grit SiC	15–25	20–30 [4.5–6.7]	200–300	Complementary[D]
Fine Grinding						
Paper	Water	240 [P220] grit SiC	15–25	20–25 [4.5–5.6]	200–300	Complementary
Paper	Water	320 [P500] grit SiC	15–25	20–25 [4.5–5.6]	200–300	Complementary
Paper	Water	600 [P1200] grit SiC	15–25	20–25 [4.5–5.6]	200–300	Complementary
Paper	Water	800 [P2400] grit SiC	15–25	20–25 [4.5–5.6]	200–300	Complementary
Rough Polishing						
No/Low Nap Cloth	Lapping oil or Extending fluid	3–6 µm diamond	120–300	20–30 [4.5–6.7]	100–150	Complementary
Final Polishing						
Synthetic Suede	None - - - - - - - - - - Water[E]	Colloidal Silica - - - - - - - - - 0.04–0.06 µm Al_2O_3	60–120	10–20 [2.2–4.5]	100–150	Contrary[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 used as an extender/diluent, distilled water is recommended.
[F] Contrary rotation, surface and specimens spin in opposite directions.

TABLE 4 Preparation Method II—Diamond Grinding

Surface	Coolant/ Lubricant	Abrasive Size/Type ANSI[A] [FEPA][B]	Time, min	Force[C], n [lbs]	Surface Speed, RPM	Relative Rotation
Planar Grinding						
Paper	Water	240 [P220] grit SiC	0.25–0.5	20–30 [4.5–6.7]	250–350	Complementary[D]
Fine Grinding						
Hard Woven Cloth	Water–base Suspension	6–9 µm diamond	4–7	20–25 [4.5–5.6]	100–150	Complementary
Rough Polishing						
No/Low Nap Cloth	Lapping oil or Extending fluid	3 µm diamond	1–3	20–30 [4.5–6.7]	100–150	Complementary
Final Polishing						
Synthetic Suede	Water[E]	Colloidal Silica	1–3	23–35 [5.2–7.9]	100–150	Contrary[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 used as an extender/diluent, distilled water is recommended.
[F] Contrary rotation, surface and specimens spin in opposite directions.

specimen is evaluated following rough polishing, the surface may be acceptable and the final polishing step is not needed.

9.1.3.4 Final polishing is accomplished with colloidal silica or aluminum oxide abrasive, both 0.04 to 0.06 µm in size.

10. Report

10.1 The laboratory report should include the details and exceptions of the metallographic preparation technique needed to reproduce the microstructure exhibited by the prepared specimen.

11. Precision and Bias

11.1 It is not possible to specify the precision or bias of this guide because no quantitative results are produced. However, quantitative analyses of thermally sprayed specimens may be influenced by the preparation technique.

12. Keywords

12.1 flame spray; flame sprayed coating; high velocity oxy–fuel coatings; HVOF coatings; metallography; plasma spray; plasma sprayed coatings; specimen preparation, metallographic; thermal sprayed coatings; thermal spray

TABLE 5 Preparation Method III—Diamond Grinding

Surface	Coolant/ Lubricant	Abrasive Size/Type	Time, min	Force[A], n [lbs]	Surface Speed, RPM	Relative Rotation
			Planar Grinding			
Fixed Diamond Disc	Water	40–60 µm diamond	2–4	20–25 [4.5–5.6]	250–350	Complementary[B]
			Fine Grinding			
Medium–Hard Composite Disc	Water–base Suspension	6–9 µm diamond	3–5	20–25 [4.5–5.6]	100–150	Complementary
			Rough Polishing			
No/Low Nap Cloth	Water–base Suspension	3 µm diamond	1–3	20–30 [4.5–6.7]	100–150	Complementary
			Final Polishing			
Synthetic Suede	Water[C]	Colloidal Silica	1–3	20–35 [5.2–7.9]	100–150	Contrary[D]

[A] Force per 32 mm (1.25 in.) diameter specimen.

[B] Complementary rotation, surface and specimens spin in same direction.

[C] Water free of particulate is used as an extender/diluent, distilled water is recommended.

[D] Contrary rotation, surface and specimens spin in opposite directions.

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