Designation: E1920 – 03 (Reapproved 2021)

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

This standard is issued under the fixed designation E1920; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 Guide 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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,

[1] This guide is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Specimen Preparation.

Current edition approved Sept. 1, 2021. Published November 2021. Originally approved in 1997. Last previous edition approved in 2014 as E1920–03(2014). DOI: 10.1520/E1920-03R21.

[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.

4.3.3 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,

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 °C to 80 °C or 140 °F 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, 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.