Accepted Practices Committee on Metallography

Accepted Practice to Test Bond Strength of Thermal Spray Coatings

Introduction
The Thermal Spray Society (TSS) Accepted Practices Committee on Metallography and Mechanical Testing has prepared an Accepted Practice for Thermal Spray Coating Bond (Adhesion) Testing. This document defines and highlights critical aspects for representative and reproducible test results based on ASTM C633 and applicable industry standards.

The Accepted Practices Committee, chaired by Dr. Tetyana Shmyreva of Rolls-Royce, is comprised of an international team (25 total members) of scientists and engineers from academia and industry, including notable organizations such as Rolls-Royce Corp. (US), Swinburne University (Australia), Sulzer Metco, and GKN Aerospace (EU). The charter of this committee is to provide the thermal spray industry with technical guidance for common evaluation techniques.

Sufficient adhesion bond between the coating and the surface of a coated part/material (often called the substrate) is critical for the coated part functionality. The coating integrity and durability also directly depends on the cohesion bond strength – bonding of coating internal elements such as the internal layers and individual lamellas.

ASTM C633 “Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings” is the baseline and mandatory process to follow for tensile testing of bond strength of thermal spray coatings. The test is required as a condition of approval for new coatings and their suppliers, and is the core qualification test for coatings for aviation, oil & gas, automotive, power, marine, and many other industries. This practice clarifies details of ASTM C633 requirements and give examples of the best practice confirmed by hundreds of tests performed world-wide, adopted by numerous industrial standards, and requested to comply with international technical standardization and certification organizations such ISO, AS, SAE, and Nadcap.

Test Scope

ASTM C633 test applies tensile stresses to coated system consisting of a coated sample (bond cap/substrate) glued to another cylindrical sample (mating cap) as shown in Figure 1. As an alternative, coating could be applied on a cylindrical “button” which is glued between two cylindrical samples as shown in Figure 2. The load is applied in the direction perpendicular to the interface between the coating and the substrate. The level of tensile load is gradually increased from “0” to the load which results in the sample failure - the coating pulls out from the substrate or fractured in two pieces inside the coating. The coated surface is always a flat circle with a diameter 1 in. + 0/-0.005 in., and the sample dimensions are standardized by ASTM C633. The Bond Strength is calculated as the Load at sample failure divided on the coating failed area.
1. Inspect coating quality on bond caps. (Coating chipping, cracks, delamination, separation, overspray are not allowed.)
2. Prepare mating caps - grit blast their flat surface. (Do not grit blast coating surface).
3. Apply a layer of glue on the coating surface.
4. Place the coated samples with applied glue layer into V groove of curing fixture (see example of V groove gravity fixture in Figure 3).
5. Add mating cap in the fixture with the grit blasted surface facing the glue layer on coated sample.
6. Apply compression pressure to the assembled samples. Note: Fig. 3 doesn’t show the additional weight on top of the assembled sample to keep them in compression during glue curing (see the details below in “Requirements”).

**Figure 1.** Samples assembled for bond test and applied tensile load.

**Figure 2.** Bond Test Failure Map for Coating(s) Applied on Cylindrical Button. (Courtesy of Sulzer Metco Inc.)

### Bond Test Step by Step

<table>
<thead>
<tr>
<th>Possible Failure Locations</th>
<th>Double Coating</th>
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<td>Top Coat/Bond Coat</td>
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<td>Adhesion failure</td>
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<td>Bond Coat Adhesion failure</td>
<td>BB</td>
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7. Place fixture with assembled and compressed samples in the preheated oven.
8. Keep the samples under pressure in heated oven until the glue cures, cool samples, and release the pressure.
9. Carefully remove excess glue from each assembly with grinding. Recommended abrasive size for grinding paper or wheel - not coarser than 120 mesh; grinding direction - parallel to the glued surface; interface damage/removing of the sample material(s) are not allowed.
10. Place assembled samples in grips of tensile machine, and gradually apply tensile load, record the failure load.
11. Measure diameter (D) of sample failed face, and calculate the failure area as follows: \( 3.14 \times \frac{D^2}{4} \)
12. Bond Strength = Load of Failure / Failure Area.

To obtain the representative results and to compare properties of coatings deposited and tested at different facilities, the test conditions and the process require consistency and standardization.

**Important Requirements to Follow**

**Coating Thickness**
Coating thickness 0.01 in. (0.25mm) is recommended if the coating has a porosity below 2%. If the coating has high porosity, increase the coating thickness to 0.015 in. (0.38mm) and above as recommended by ASTM C633 to prevent glue penetration through coating to the substrate, but do not exceed the thickness required for the coated part application. Thicker coating has a lower bond strength because the residual stress accumulation that can lead to failure is not a representative result of the test.

**Coating Thickness Variation within a Sample**
The coating thickness shall not vary across the surface by more than 0.001 in. (0.025mm). The coating surface may be finished by grinding or machining when the thickness variation is excessive. Other treatment such as grit blasting should not be used to level the coating thickness. Coating tapering increases the sample misalignment that results in the failure at lower load.

**Glue**
Polyamide-epoxy FM 1000 Adhesive Film is recommended (mandatory in many industrial specifications) as a bonding glue for coating tensile test. Advantages – allow testing of high porosity coatings (such as abradable and ceramic coatings) without the glue penetration to the substrate. Disadvantages – losing its own strength if stored at temperatures of 85°F (29°C) and above. But can work for years if stored in a refrigerator.

**Curing Cycle**
Recommended cycle for FM 1000 firm: Heat assembled samples in the oven to 340 ± 10 °F (170 +/- 6 °C), and cure them for 90 min ± 10 min. at the temperatures at bond line of 340 ± 10 °F (170 +/- 6 °C). Curing conditions may vary based on the manufacturer recommendations and for different types of glue. Control the curing temperature with calibrated thermocouple touching (or bonding to) assembled samples. Class 5 furnace (per AMS2750) with temperature uniformity in the working zone +/- 25°F is sufficient.

**Fixture**
Figure 3 shows V grooved gravity fixture. This design is working with FM1000 adhesive film. To keep the assembled samples in compression during the glue curing, one solid steel cylinder (1 in. diameter & 2 in. long) must be placed on the top of two assembled samples. Fixture could be made from steel or aluminum alloy, and should support samples at 30 degree to vertical position. Horizontal groove in the middle of the fixture shown in Fig. 3 protects it from contact with excessive glue during the curing process.
Work in Clean and Controlled Environment
Protect samples from contamination: Do not touch coated surface. Keep coated samples in clean (recommended plastic) envelops. Use FILTERED compressed air when removing grit or dry sample surfaces with liquid degreaser. Protect samples from excessive humidity.

Verify Alignment
Sample misalignment always leads to low test results. Verify their alignment in the fixture before and after curing and remove excessive glue from the sample sides. Use tensile machine having universal joints to ensure the samples’ self-alignment when tensile load is applied.

Control Load Applying Speed
Recommended crosshead speed for tensile load - 0.03 – 0.05 in./minute.

Number of Coated Samples
It is recommended to test five (5) samples, and identify the coating bond strength as an average of the five results. For well established dense coatings with high adhesion & cohesion strength (such as cermets) the samples’ number can be reduced to three (3) for 1 data point.

Test Reference Samples (Glue Test)
Always test at least one uncoated reference sample with each set of curing samples to verify/confirm that the glue itself has sufficient bond strength (10000 psi as a minimum for FM1000).

Keep Records
Determine, and keep records about all test conditions and file test reports.
Bond test report should include as a minimum: the glue strength; coating bond strength; % of each mode of coating failure (see Figure 2); test date; operator name/signature; reference to applicable standard(s), and specifications & local instructions documenting the test conditions and requirements.
Note: This Accepted Practice is intended to be used as a baseline for you test process, but doesn’t replace local test/lab instructions. Additional requirements may apply based on the available equipment, testing materials, customer requirements, etc. Acceptance testing should always be performed in accordance with ASTM C633 (latest revision).

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