MASTERBOND®
CASE STUDY

EP15: Utilized to test the bond strength of thermal spray coatings according to ASTM C633

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Overview of EP15

Master Bond EP15 is a one-component, thermally-curing epoxy with a tensile strength in excess of 12,000 psi (82.7 MPa) due to the formation of rigid, dimensionally stable bonds upon curing. This bulk tensile strength makes it ideal for testing the adhesive and cohesive strengths of various thermal spray coatings according to ASTM C633, which requires the use of a high tensile strength adhesive that does not fail until after the coating.

ASTM C633 to Test the Bond Strength of Thermal Spray Coatings

This test evaluates the adhesive strength of a coating to a substrate or the cohesion strength of the coating when tension is applied perpendicular to a surface. During the test, a coating is applied to one side of a substrate, and this coated surface is attached to a loading fixture, followed by the application of a tensile force perpendicular to the coating's plane. This test is specifically designed for examining coatings applied through thermal spray processes, such as combustion flame, plasma arc spraying, two-wire arc, high-velocity oxygen fuel, and detonation methods, using wire, rod, or powder feedstocks. Several parameters may critically affect the test results, including but not limited to:

- **Surface preparation method**
  Regardless of the substrate, its surface typically needs to be prepared to optimize the bonding of the adhesive. To ensure adhesive wettability on the substrate surface, dirt, deposits, and contaminants must be removed, and the surface energy and surface area should be improved. Common techniques for achieving this include degreasing, abrasion, chemical treatment, or physical treatment.

- **Cure schedules**
  To obtain the maximum bond strength with the adhesive, an appropriate cure schedule must be used. When cured at temperatures from 300–400°F for 4-6 hours, EP15 boasts a tensile strength exceeding 12,000 psi (82 MPa).

- **Pull rates**
  The pull rates for ASTM C633 often lie between 0.030 in/min and 0.050 in/min. The appropriate pull rate will depend on the mechanical properties of the coating and substrate (i.e., stiffness, ductility, and strain rate sensitivity). The anticipated failure mode of the coating-substrate system may also influence the choice of pull rate. For example, if a more ductile failure mode is expected to mimic real-world conditions, a slower pull rate may be necessary.

- **Substrates**
  The appropriate surface preparation technique will depend on the substrate used with EP15. For example, in Case Study 4, the authors showed that the substrate porosity affected the amount of EP15 necessary to ensure good bonding.

The Role of EP15 in ASTM C633 Testing

To ensure the success of bonding strength according to ASTM C633, it is necessary to select an adhesive with a bond strength greater than the expected bonding strength so that the specimens fail before the adhesive. As shown below in Table 1, EP15 has been used to test the bond strength of a wide variety of coating and substrate pairs. The case studies below highlight the importance of preparing each substrate surface to optimize the bonding of EP15 for ASTM C633 tensile tests.

Table 1. Overview of the uses of EP15 for ASTM C633 testing

	Thermal Spray Technique	Coating	Substrate	Surface Pre-treatment	Cure Schedule	Measured EP15 Bond Strength
Case Study 1	DC-Magnatron Sputtering	Ti, Nb, or uranium-8 wt% molybdenum alloy	Aluminum, niobium, tantalum, titanium, and zirconium	Polishing, use of high-purity substrate	200°C for 90 minutes	70 MPa
Case Study 2	Cold Gas Dynamic Spraying (CGDS)	Al-Fe-V-Si powder	Al-6061 alloy	Grit-blasting with 20 mesh, 24 grit silica beads, followed by machining on a lathe to obtain a smooth surface	*According to Master Bond's recommended cure schedule*	*Not reported*
Case Study 3	Cold Gas Dynamic Spraying (CGDS)	SiC-reinforced, Al-12Si alloy	6061-T6 aluminum	Grit blasting using 20-grit ebony (ferrosilicate) beads	170°C for 90 minutes	82 ± 10 MPa
Case Study 4	High Velocity Oxy-Fuel (HVOF) Thermal Spraying	Inconel 625	Mild Carbon Stainless Steel (304)	Cleaned, degreased and dried. Acetone etching, followed by mechanical abrasion	176.67°C for 90 minutes	45-60 MPa
Case Study 5	Chemical Densified Coating (CDC)	Cr_2O_3-SiO_2	SS316	Degreasing and Al_2O_3 grit blasting	170°C for 2 hours	*Not reported*
Case Study 6	Arc Spraying	Alpha-1800 (iron-boron cored wire)	Mild steel	Grit blasting	150°C	*Not reported*

Case Study 1: DC-Magnetron Sputtering of Monolithic Two-Metal Layered Systems

Application

In contrast to nuclear power reactors, research nuclear reactors are used for R&D efforts, education, and training. Most early nuclear research reactors were constructed with technology that required highly-enriched uranium (HEU) to perform scientific research. Now, much of this research can be carried out using low-enrichment uranium (LEU), which contains a uranium-235 concentration below 20%. The Reduced Enrichment for Research and Test Reactors (RERTR) program develops new fuel forms to convert research nuclear reactors so they can use LEU instead of HEU. This transition can be facilitated by understanding the in-pile performance of nuclear fuel plates, which is strongly influenced by the mechanical contact between different material pairs used in nuclear fuel plates (fuel/cladding, cladding/diffusion barrier, and fuel/barrier). The quality of mechanical contact between these two-layer systems can be evaluated by their bond strength. An international team of researchers from France and Germany bonded various coatings to substrates and then used EP15 to assess their bond strength according to ASTM C633.

Key Parameters and Requirements

DC-magnetron sputtering was used to fabricate two-layered test specimens consisting of a substrate (aluminum, niobium, tantalum, titanium, and zirconium) and a sputtered layer (Ti, Nb, or uranium-molybdenum alloy with 8 wt.% Mo). These specimens were developed to represent interfaces that may exist in future monolithic fuel plate designs. For tensile tests, each specimen was mounted on top of a stainless steel specimen holder using EP15, which was chosen because it was strong enough so that it did not fail before the coatings. EP15 was cured at 200°C for 90 minutes to maximize its bond strength.

Substrate Surface Preparation

In this study, the authors noted that the bond strengths of two substrate/sputtered layer pairs (Ti/DU-8Mo and Zr/DU-8Mo) varied more than others due to how the substrates were prepared for the Ti and Zr specimens.

1. Ti and Zr substrates were punched-out to obtain a circular specimen, which bent by this procedure, which made adhesive bonding difficult to achieve.
2. The specimens were affected by particles in the sputtering device, which degraded the bonding.
3. The interface with niobium was much better interlocked compared with titanium and zirconium.

Polishing

The authors also noted no significant difference in the bond strength between non-polished and highly-polished Al/DU-8Mo specimens. However, because the distribution of bond strengths was so wide, the authors could not draw definitive conclusions about the effect of polishing.

Substrate quality/purity

The specimens using Al substrates (Al/Ti and Al/Nb) did not reach the same bond strength as the aluminum barrier/fuel specimens because the Al used was of a lower purity and was more susceptible to deformation.

Results

After using SEM to confirm that the curing of EP15 did not damage the samples, the bond strength was measured using a standard procedure, ASTM C633, in which samples were loaded until failure. The authors investigated the following substrate/sputtered layer pairs: Al/Ti, Al/Nb, Al(non-polished)/DU-8Mo, Al(high polished)/DU-8Mo, Ta/DU-8Mo, Zr/DU-8Mo, Ti/DU-8Mo, and Nb/DU-8Mo.

All specimens were compact, with no visible cracks. By optimizing the bond strength of EP15, the authors obtained a bond strength of approximately 70 MPa. When failure occurred, the authors assigned a particular failure mode according to ASTM C633: sputtered layer separated from the substrate, rupture between adhesive and specimen, or failure within the adhesive. The authors also noted that the bond strengths measured during these tests were lower than their true values due to the limiting bond strength of EP15 (70 MPa). Therefore, the “true” bond strengths were higher than those measured during the test. Ultimately, the authors showed that DU-8Mo on niobium and tantalum showed the highest bond strengths, as the only failure observed was adhesive failure, suggesting a bond strength greater than 70 MPa.

Case Study 2: Cold Gas Dynamic Spraying (CGDS) of Metastable Aluminum Alloy Coatings for Automotive Applications

Application

The application of thermal barrier coatings on the pistons of an engine can increase the engine operating temperature, which has been shown to increase efficiency by reducing fuel consumption. However, thermal barrier coatings act as a buffer between the different coefficients of thermal expansion between the substrate and ceramic, and the brittle ceramic substrates may break during operation. Researchers at the University of Ottawa proposed a new method to limit heat losses in the combustion chamber by coating a novel metastable Al-Fe-V-Si alloy powder on the piston crown using cold gas dynamic spraying (CGDS) on an aluminum alloy substrate. Critically, these coatings were required to maintain their mechanical properties at elevated temperatures. The mechanical properties of these coatings were also assessed using microhardness testing and bond strength testing, the latter of which EP15 played a key role.

Key Parameters and Requirements

The bonding strength of the alloy coatings to the substrate surface was assessed using ASTM C633-01 to predict whether the sample would fail and delaminate during its normal operating conditions. The bond test substrates were cylindrical specimens of Al-6061 alloy. Multiple passes were used to completely cover the surface with the Al-Fe-V-Si alloy coating. The coatings were then machined on a lathe to produce a smooth and flat surface normal to the tension applied during the bond test.

Master Bond EP15 was used to coat the machined substrate surface of the cylindrical substrate, which was then mated with an identical uncoated cylinder. During the curing of EP15, the authors used an alignment jig to apply a light pressure to maintain alignment and then cured EP15 according to Master Bond's recommended cure schedule. The authors then performed tensile tests according to ASTM C633-01 and determined the failure mode of each specimen.

Substrate Surface Preparation

The substrates were cut from commercial Al-6061 6.35 mm thick bars before being grit-blasted using silica (quartz) beads with an average particle diameter of 20 mesh and 24 grit to provide sufficient substrate surface roughness to promote mechanical interlocking at the particle-substrate interface without significantly affecting the coating microstructure. Before coating with EP15, the authors machined the substrates on a lathe to produce a smooth and flat surface.

Results

The authors categorized each failure mode as either coating failure, adhesive failure, cohesive failure, or mixed-mode failure. The average bond strength of the alloy coatings sprayed using nitrogen was 44.5 ± 1.8 MPa. From the results, the authors concluded that the failure mode was cohesive failure within the coating itself. In SEM images, the authors noted that both surfaces of the aluminum cylinder pair were covered with the coating, confirming good adhesion at the coating-substrate interface. This bond strength was much higher than previously used coatings, indicating that the composite coating would not fail and detach under normal operating conditions. EP15 ensured that the substrate samples remained securely bonded during the bond strength test.

Case Study 3: SiC-Reinforced Aluminum Alloy Coating Produced by Cold Gas Dynamic Spraying (CGDS)

Application

Metal-matrix composites (MMCs) can be tailored to meet the needs of applications in diverse industries and are composed of particles added to a metallic matrix to provide enhanced properties, such as increased hardness. Aluminum and its alloys are often used to fabricate MMCs, and the incorporation of silicon carbide (SiC) particles into an Al alloy that can be used to coat substrates to improve the surface properties of the underlying Al-12Si alloy without affecting its bulk properties, such as its ductility. Researchers at the University of Ottawa used cold gas dynamic spraying (CGDS) to apply Al-12Si alloy coatings reinforced with dispersed SiC particles and then evaluated the mechanical properties of the coatings. As part of this research, the authors performed bond strength evaluations according to ASTM Standard C 633-01.

Key Parameters and Requirements

The 6061-T6 aluminum substrates were grit-blasted with ferrosilicate beads and then several passes of CGDS were applied to cover the entire surface with SiC-reinforced coating. To evaluate the strength of the coating, bond tests were carried out, in which the top portion of the coating was machined flat and glued to an uncoated test sample using EP15 and then cured at 170°C for 90 minutes in a V-shaped block to ensure proper alignment. To determine the bond strength of the adhesive, the authors performed bond testing using uncoated samples and determined a bond strength of 82 ± 10 MPa.

Substrate Surface Preparation

The 6061-T6 aluminum substrates were grit-blasted at a stand-off distance of 10 mm by 20-grit ferrosilicate beads at a blasting pressure of 400 kPa at a 45° angle.

Results

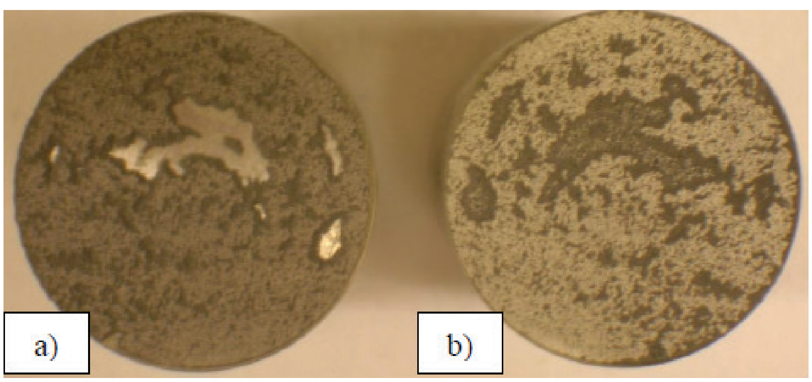

Figure 1. Photographs of the bond strength specimens with a) EP15 and b) the remainder of the Al-12Si coating after the bond test.

The authors successfully obtained SiC-reinforced aluminum alloy coatings by CGDS. An adhesion strength of 49 MPa was obtained for the Al-12Si coating, which slightly decreased to 44 MPa and 43 MPa when adding 20% and 30% SiC to the coatings, highlighting that the inclusion of SiC particles did not greatly change the adhesion of the coatings to the substrates. However, the authors did note that increasing the SiC content increased the porosity of the coatings. The obtained values represented the adhesion strengths of the coatings, as the specimens failed at the substrate-coating interface. The SEM images of the SiC-reinforced coatings revealed substrate-coating interfaces that were free of defects to may have degraded the coating's adhesion. Master Bond's EP15 played a key role in this investigation to determine the failure mode and bond strength of these SiC-reinforced coatings.

Case Study 4: High-Velocity Oxy-Fuel (HVOF) Thermal Spray Coatings of Inconel 625 Powders on 304 Stainless Steel

Application

High-velocity oxy-fuel (HVOF) demonstrates superior high bond strength and lower porosity than other thermal spray techniques, making it attractive and easily adaptable to various industrial processes. In a thesis from Dublin City University, the author compared the performance of welded carbon and stainless steels due to the application of Inconel 625 coatings deposited using HVOF. The dissertation focused on characterizing the mechanical behavior of coatings at the weld-substrate interface when subjected to tensile and fatigue loads, especially after being subjected to a corrosive environment. As part of their characterization, the author used EP15 during tensile tests to determine the bond strength of the coating according to ASTM "Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings."

Key Parameters and Requirements

A combination of coated and uncoated solid round bars specified by ASTM RP C-633-01 was prepared using carbon steel and 304L stainless steel for tensile strength tests. Dummy non-coated specimens were prepared using EP15 to determine the tensile strength of the adhesive. To ensure that the adhesive did not fail before the specimens (and thus invalidate the results), the authors need an adhesive with a high tensile strength. They chose EP15, as its rated strength (~82 MPa) exceeded the expected tensile strength of the coatings.

The authors noted that because their substrate was porous, more adhesive was applied to fill its voids, and had to use 0.5 mm of EP15 between two round specimens (one dummy specimen and one coated specimen) wrapped in aluminum foil. Because of the thickness of the applied EP15 layer, the authors sealed the specimens with high-temperature sealing tape to keep the liquid EP15 in place to maintain an appropriate thickness. Then, the adhesive was cured in an oven set to 176.67°C for 90 minutes. Prior to bond strength testing, the authors allowed the specimens to cool to room temperature.

Substrate Surface Preparation

Before performing adhesion tests, the surfaces were cleaned, degreased, and dried to obtain the maximum bond strength. The authors used acetone to etch the bonding metal (coated and uncoated) surfaces. Mechanical abrasion of the metal surface was also used to roughen the bonding area using emery paper. The authors tested the surface cleanliness by placing a few drops of cool water on the surfaces to be bonded and determined that the surface was sufficiently clean if the water spread over the area to form a continuous film.

Results

The authors determined that an adhesive bond strength greater than 40 MPa would ensure sound bonding during tensile tests. Specimens subjected to a corrosive environment for 3 weeks failed at a lower tensile load than those that were not exposed. Shear deformation of regions adjacent to the coating-substrate interface resulted in total failure of the coating.

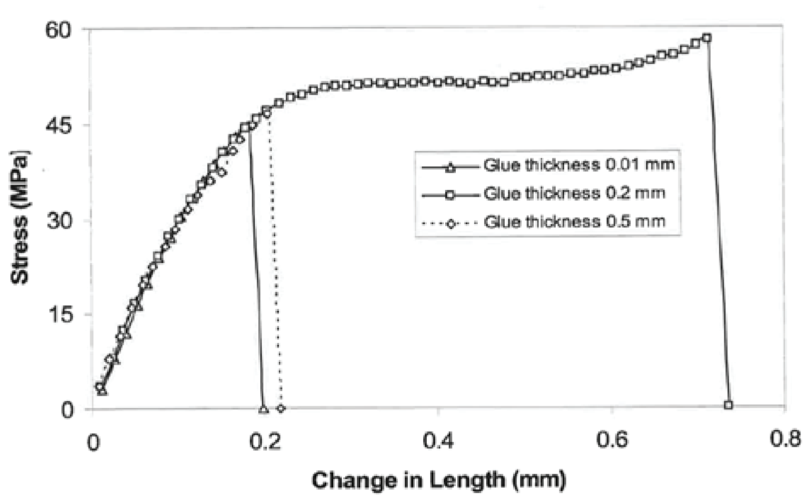

Figure 2. Tensile bond strength of EP15 as a function of adhesive thickness.

When applied at an appropriate thickness, as shown in ***Figure 2***, EP15 provided an appropriate bond strength that allowed the authors to characterize the tensile strength of the coatings applied to mild carbon and stainless steel substrates at pull rates between 0.030 in/min (0.013 mm/s) and 0.050 in/min (0.021 mm/s). The tensile test results showed that the decrease in the bond strength due to corrosion was due to degradation of the coating's adhesion to the substrate surface, likely due to the formation of oxides on carbon steel surfaces during coating. Fatigue tests showed that the coated substrates could endure 15,000 cycles without cracking.

Case Study 5: Chemical Densified Cr_2O_3–SiO_2 Coatings as a Tritium Permeation Barrier

Application

In future fusion reactor designs, fusion blankets are a key design component and act as tritium permeation barriers. However, because of the techniques used to prepare them, these barriers are often porous, which may allow tritium to diffuse through them. Previous efforts produced Cr_2O_3–SiO_2 coatings that were highly porous. Researchers from the Japan Atomic Energy Research Institute and Tocalo Co., Ltd. attempted to densify a Cr_2O_3–SiO_2 coating on stainless steel 316 (SS316) using the chemical densified coating (CDC) method using $CrPO_4$. The CDC method was used because it allowed the fabrication of densified ceramic coatings on either the outer or inner surfaces of a tube. As part of the characterization of the adhesion strength of these coatings, the authors used EP15 during tensile tests.

Key Parameters and Requirements

The authors prepared coatings on SS316 substrates and tested their adhesion strength using an adhesion specimen with the coating and one without the coating. These specimens were joined by EP15, which was cured at 170°C for 2 hours under an ambient atmosphere in an electric furnace. The joined specimen was then loaded into the tensile test machine, as shown in ***Figure 3***, and the coating adhesion was measured at a tensile rate of 2.3 mm/min.

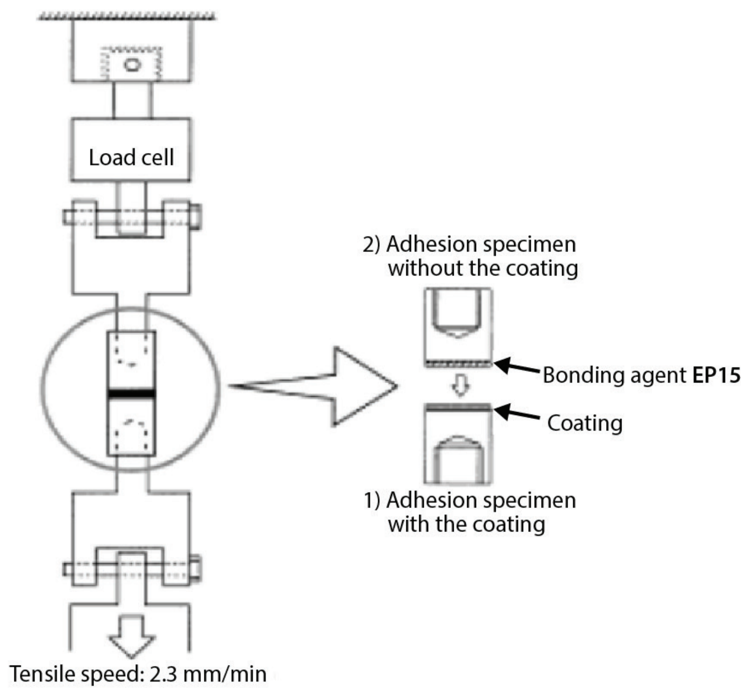

Figure 3. Schematic diagram of the adhesion test using EP15.

Substrate Surface Preparation

Before coating them with the slurry, the authors prepared their substrates by degreasing and then grit-blasting them with Al_2O_3.

Results

The authors performed room-temperature tensile tests at a tensile rate of 2.3 mm/min. Using naked-eye observations, the authors noted that the Type 1 (Cr_2O_3–SiO_2) coating fractured along the interface between the coating and bonding agent, while the Type 2 (Cr_2O_3–SiO_2 + $CrPO_4$) coating fractured within the coating itself. The two coatings showed adhesion strengths of 41.2 MPa and 37.2 MPa, respectively. Due to the interdiffusion of Cr and Fe atoms between the substrate and coating, the CDC coating provided a high adhesive strength compared with the one obtained via plasma spraying previously reported (10–20 MPa). In the deuterium permeation experiments, the coating densified by $CrPO_4$ showed much lower deuterium permeation (permeation reduction factor of ~1000) than the coating without $CrPO_4$ due to the filling-in of porosity. As shown by tensile tests conducted using EP15 as the bonding agent, the adhesive strength of the Type 2 coating was also greater than that of the Type 1 coating.

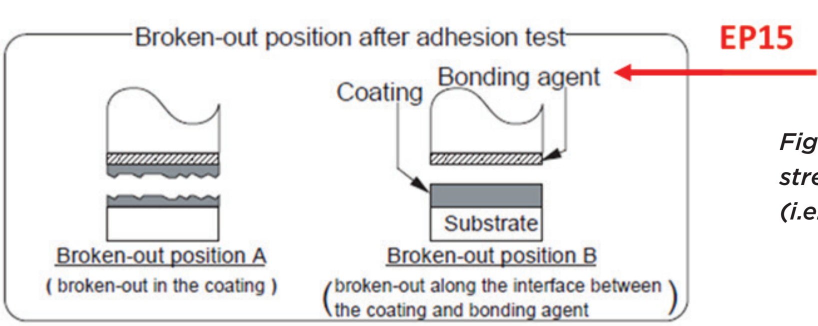

Figure 4. Results of the adhesion strength showing the types of failure (i.e., "broken-out" positions).

Case Study 6: Hard-Arc-Sprayed Coating with Enhanced Erosion and Abrasion Wear Resistance

Application

During their use, metal-sprayed coatings are damaged by micro-machining and ploughing, which may lead to the generation and propagation of subsurface cracks at high impact angles that damage single-phase ceramic coatings. To solve this, multicomponent coatings have been developed to provide wear protection. Researchers at the Industrial Materials Institute of the National Research Council Canada developed a cored wire for producing hard arc-sprayed coatings to resist abrasion and particle erosion at high temperatures. As part of this research, the authors also provided information about the coating's thermal stability and bond strength, in which EP15 was used as the bonding agent during tensile tests.

Key Parameters and Requirements

The adhesive bond strength of Alpha-1800 arc-sprayed coatings was measured according to the ASTM C-633-79 procedure. Alpha-1800 cored wire was sprayed direly on mild steel studs to obtain coatings with a thickness of less than 2 mm. The coatings and opposing studs were grit-blasted and then joined together using EP15 (cured temperature of 150°C), and then tensile tests were performed using these specimens.

Substrate Surface Preparation

The mild steel substrates were grit-blasted to obtain flat surfaces prior to erosion and abrasion testing.

Results

To ensure wear resistance, coatings must remain adhered to their substrates under expected operating conditions. Therefore, the authors investigated the bond strength of the Alpha-1800 arc-sprayed coatings according to ASTM C633-79. The authors observed sound bonding up to a coating thickness of 6 mm, and rupture during tensile tests never occurred at the coating-substrate interface. During cyclic oxidation experiments at 800°C, the Alpha-1800 arc-sprayed coatings gained 8 times less weight than AISI 1020 steel specimens, indicating improved oxidation resistance. The arc-sprayed coatings showed resistance against particle erosion, slurry erosion, and abrasion wear significantly higher than those of structural materials and commercial arc-sprayed coatings. The coatings were tested in industrial fans for 3 years and showed no signs of failure, indicating good resistance.

As part of this research, EP15 played a key role in characterizing the bond strength of these coatings to optimize the coating thickness (***Figure 5***). The developed coatings were used to coat industrial fans and exceeded their predicted lifetime and improved the lifetime of components compared with steels or (Fe-Cr-C) submerged arc overlays used as fan materials.

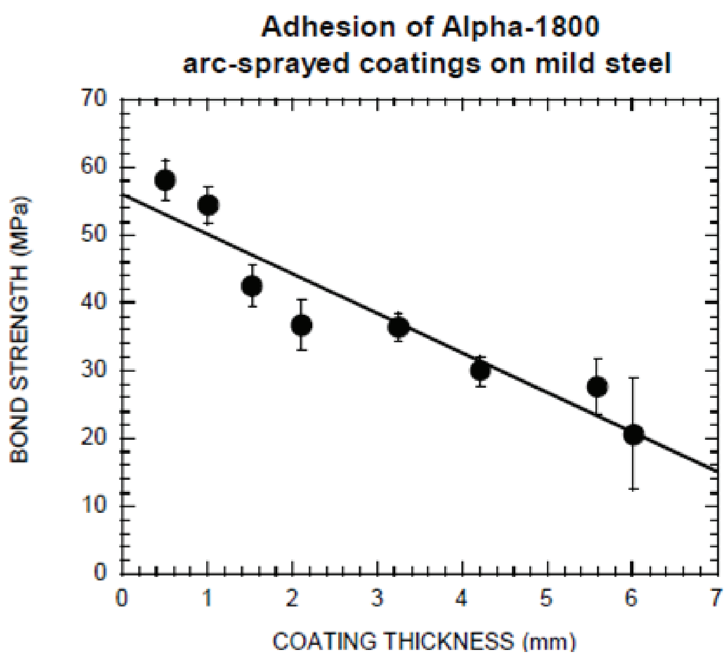

Figure 5. Bond strength of Alpha-1800 arc-sprayed coatings as a function of coating thickness on mild steel.

Concluding remarks

The above case studies highlight the importance of substrate preparation and using an adhesive with an appropriate bonding strength to successfully determine the bonding strength of thermal spray coatings. With a tensile strength exceeding 12,000 psi (82 MPa), EP15 is an ideal choice for performing bond tests according to ASTM C633 on a variety of substrates, including ceramics, alloys, and metals.

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