

Introduction

Thermal spraying is a specific coating technique in which melted material is projected onto a surface to coat it. Compared to other deposition techniques, the thermal sprays offer the ability to cover large area at high deposition rates and provide thicker coatings. Thermal sprays are used in various industries such as aerospace, automobile, marine and heavy machinery. The applications for those coatings cover wear and abrasion resistance, low friction, corrosion protection, altering thermal and electrical conductivity and many others.

Testing Problematic

The projection of melted material onto the substrate surface forms a highly inhomogeneous coating consisting of a multitude of "pancake-like" splats called lamellae formed by the flattening of the material droplet.

The size of those lamellae along with the varying degrees of porosity are typically used to characterize the thermal spray coatings. But this deposition technique results in a unique microstructure that presents properties significantly different than bulk materials.

About Rtec Instruments

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Rtec Instruments develops and manufactures advanced testing solutions for mechanical and optical surface characterization. We specialize in combining techniques that provide a unique perspective in material testing.

Our line of testers include tribometers, scratch testers, indentation, 3D profilometry, fretting testers and hot hardness testing machines.



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The different types of thermal spraying processes (flame, arc, plasma, high velocity oxy fuel and detonation gun spraying) also increase the complexity of the material problematic by yielding different structures.

The durability and functionality of these coatings is highly dependent on the cohesion strength of the resulting coating but also on its adhesion to the substrate. It is therefore necessary to test those coating in their "as deposited" state to investigate the effects of deposition techniques, spraying parameters such as velocities, and substrate surface preparation.

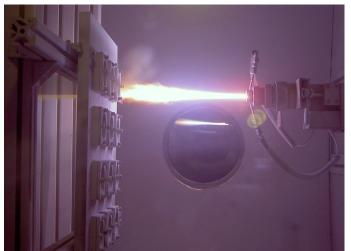
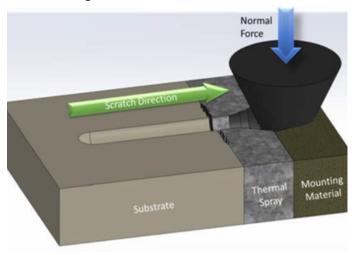


Figure 1: Thermal Spray deposition (Vacuum Plasma Spraying)¹

Test Methodology

The substrate / thermal spray system is mounted as a cross section in a metallographic mount and polished to expose the interface between the substrate and coating. A constant load scratch is generated by dragging a sphero-conical diamond tip perpendicular to the interface and moving from the substrate towards the coating (Figure 2). The scratch is set up to finish in the mounting material.



Two main types of failures can be created as shown in Figure 3:

- Cohesive failure as exhibited by cracks inside the thermal spray coating and conical fracture of the coating at the free surface
- Adhesive failure if some cracks develop at the interface between the substrate and the thermal spray coating.

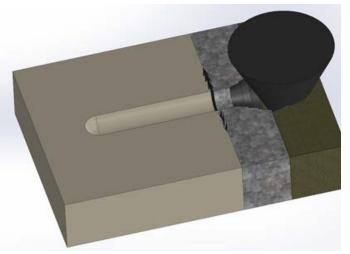


Figure 3: Cross section scratch testing failure

The normal force on the tip is adjusted to create either failure at the free surface of the thermal spray or at the interface between the substrate and the thermal spray coating.

Test Conditions

The Universal Scratch Tester (UST-2) was used to create scratches on different thermal spray coatings. The testing methodology followed ISO-27307. The test parameters are summarized in table 1.

Table 1: Test Conditions			
Load application profile	Constant load		
Scratch length	2mm		
Constant load	20N & 30N		
Scratch speed	4 mm/min		
Stylus	Rockwell with 200µm radius		

Three scratches per load (table 1) are performed on each sample for a total of 6 scratches per sample. The constant loads were chosen to create enough damage and the possibility to test adhesion failure.

Figure 2: Cross section scratch testing principle

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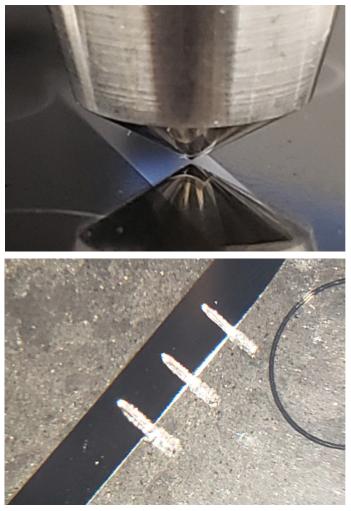


Figure 4 & 5: Cross section scratch testing on the UST-2 platform.

Results

Four different thermal sprays cross sections mounted in metallographic mounts were tested to investigate cohesion and adhesion. Different types of failure can be observed in the coatings:

1. Adhesive failure

In this case, the cracks developed at the interface between the thermal spray and the substrate. This failure is directly related to the strength of adhesion that exists between the thermal spray coating and the substrate. The cracks always originate from the interface and can either propagate along the interface or inside the coating.

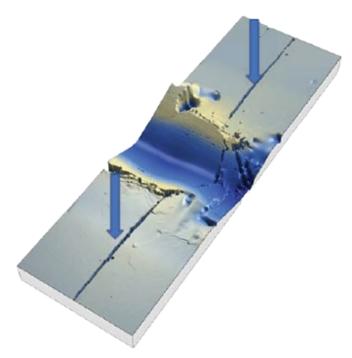


Figure 6: Adhesion failure at the interface.

The lengths of the cracks on either side of the scratch groove can be used to quantify the severity of the adhesion failure. When this failure is observed, no other conclusion can be made as most of the energy of the test goes into failing the coating / substrate interface.

2. Cohesive failure in coating

This type of failure is characterized by some cracks propagating from the side of the scratch groove into the coating itself. Both the origin and end of the cracks are located into the thermal spray coating.

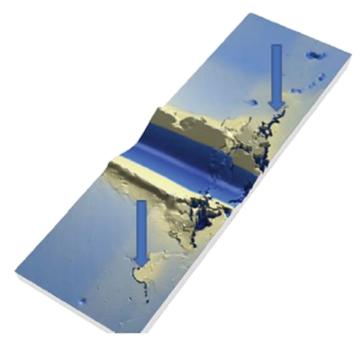


Figure 7: Cohesion failure in the coating.

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3. Cohesive failure at the free surface

The final type of failure is seen at the free surface of the thermal spray coating and is typically in the shape of a cone. This failure relates also to the cohesion of the thermal spray coating but demonstrate a higher cohesion than cracks happening inside the coating. This failure is the result of the thermal spray failing towards its free surface.

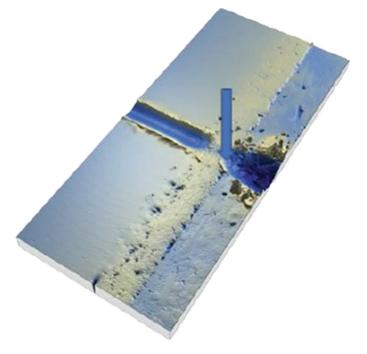


Figure 8: Cohesion failure at the free surface of coating

Granted that no other failure is observed prior to the free surface, the samples exhibiting this failure can be ranked by measuring the angle of the conical failure. A lower angle will correspond to a higher cohesion of the thermal spray.

Comparison of samples

Given the different types of failure, the comparison of samples and their ranking need to follow a rigorous process.

 Can adhesion failure be seen at the interface? If adhesion failure is observed, no other comparison can be made, and these coatings will be the lowest ranking. If the coating cannot adhere to the substrate, the cohesion of such coating does not matter as it will fail at the interface prior to failing cohesively. If several samples exhibit adhesion failures, the length of the cracks at the interface is used to differentiate them.

- 2. If no adhesion failure is seen, can cohesive failure be observed inside the coating? Cohesive failure inside the coating will be the next level of ranking as the interface is strong and the coating is now failing internally. If multiple samples exhibit cohesive failure inside the coating, the length of the cracks inside the coating is used to rank them from small crack to longer crack.
- 3. If no adhesion failure at the interface and no cohesive failure in the coating are observed, the cohesive failure at the free surface becomes the focus. The last point of failure is situated at the free surface of the coating, as the scratch is moving into the mounting material which is often much softer than the coating. In this case the samples are ranked with the measurement of the angle of the facture at the free surface as shown in Figure 6.

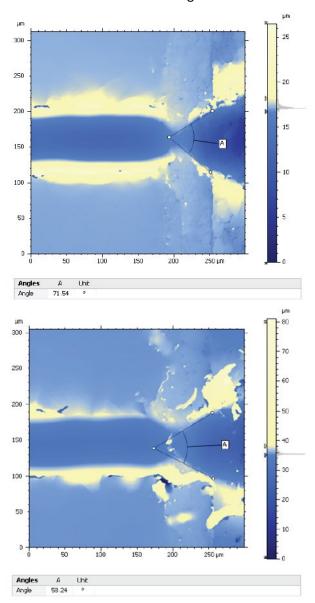


Figure 9: Thermal Spray sample 1 (top) and Thermal Spray sample 3 (bottom) angle measurements.





For the comparison of these two thermal sprays, the "exit" cone angle is measured as the tip scratches from the thermal spray to the mounting material. This angle is representative of the strength of the coating where a stronger coating will exhibit a smaller fractured angle as it is the case for TS 3.

Results summary

In this study, the four thermal spray samples exhibited different failures from adhesion to cohesion. The results of all samples are summarized in Table 2.

		Adhesive failure	
		Y/N	Total crack length
Sample	TS 1	N	
	TS 2	N	
	TS 3	N	
	TS 4	Y	357 μm

		Cohes	ive failure inside coating
		Y/N	Total crack length
Sample	TS 1	Ν	
	TS 2	Ν	124 μm
	TS 3	Ν	
	TS 4		

		Cohesive failure at free surface	
		Y/N	Angle of fracture cone
Sample	TS 1	Y	71.54°
	TS 2		
	TS 3	Y	58.24°
	TS 4		

Table 2: Results of scratches on 4 different thermal spray samples

The adhesion failure of TS 4 brings it to the bottom of the ranking for those samples. TS 2 exhibits good adhesion but cohesion failure in the coating whereas TS 1 and TS 3 are at the top of this samples ranking. As explained above the differentiation between those two is done using the angle of the fractured cone. This measure yields TS 3 as the best coating of this group with a smaller angle.

Conclusion

The scratch testing technique is used here to characterize both adhesion and cohesion of thermal sprays as sprayed on their respective substrates. A single test can yield a deeper understanding of the strength of the thermal spray coatings.

In turn, the observation and quantification of failures helped our customer adjust the substrate surface preparation for TS 4 and the spraying parameters for TS 1 to 3 in order to increase the adhesion and cohesion of their coatings.

Not described in this study, the same thermal sprays were also tested for wear and coefficient of friction using Rtec Instruments MFT-5000 Tribometer. The combination of scratch testing and tribology testing (wear/COF) gives a more complete understanding of those coating and their resistance to mechanical forces. Rtec Instruments is uniquely qualified and positioned for the characterization of such coating.

References:

- By Matthias Zepper Selbst aufgenommen bei Vorführung der Anlage im Institut für Technische Thermodynamik (Pfaffenwaldring 38-40 in D-70569 Stuttgart) des Deutschen Zentrums für Luft und Raumfahrt (DLR)., CC BY-SA 2.5.
- ISO 27307 2015 Thermal spraying Evaluation of adhesion/cohesion of thermal sprayed ceramic coatings by transverse scratch testing.
- Lopez, Zambelli, Cohesion measurement of plasma sprayed ceramic coatings, Surface Modification Technologies (815-821), 1990.

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Suggested instrument for similar testing technique:

Rtec Instruments SMT-5000



The SMT-5000 is the first answer to the increasingly complex testing requirements for surfaces across many industries— from liquid to ultra hard solid materials.

SMT series provides a combination of multiple investigative techniques to measure surfaces on one platform. For example, with one run, a combination of coating adhesion, hardness, thickness, surface roughness, and 3D image data come together for a conclusive comprehensive analysis.

The SMT instruments come standard with a control software and a data analysis software. This allows the user to continue to run the instrument while analysing data from previous tests.

Rtec Instruments MFT-5000



A tribometer allows you to study the interaction of surfaces, from friction and lubrication to wear and abrasion. Interacting surfaces are found everywhere, in ball bearings, between car tires and the road or between a finger and a touch screen.

Friction is vital to breaks, but may damage parts inside an engine. Designing materials to interact in an optimal way, both with regard to performance and life-time, is therefore crucial.

The Rtec MFT-5000 tribometer offered by ST Instruments is a versatile, modular instrument which allows you to experiment even down to nanometer scales, at temperatures ranging from 1200 °C to -120 °C and in controlled environments, e.g. different gasses or a vacuum. A range of sensors and modules allows measurement of many different variables.

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