DEVELOPMENT OF A METHODOLOGY FOR SCRATCH TESTING COLD SPRAY COATINGS

[1]Marek DOUBRAVA, [1]Lukáš ŘEHOŘEK, [1]Libor PANTĚLEJEV

[1]Brno University of Technology, Institute of Materials Science and Engineering, Brno, Czech Republic, EU, marek.doubrava@vutbr.cz, rehorek@fme.vutbr.cz, pantelejev@fme.vutbr.cz

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Abstract

This contribution introduces a comprehensive methodology for evaluating cold spray coatings using scratch testing technique. Unlike traditional thermal spray coatings, cold spray coatings have not been systematically and thoroughly scratch tested, leading to a lack of experimental data and evaluation methods. The primary objective of this research is to evaluate the adhesion or cohesion strength of the coating and to study the damage mechanism. The methodology focuses on the systematic preparation of test procedures. The evaluation of individual scratch marks, performed using both light and electron microscopy, forms the basis for refining the testing methodology. This study addresses a gap in the field, offering a straightforward approach for assessing cold spray coatings. The presented methodology is based on ISO 27307 where the applicability for cold spray coatings is lacking, especially regarding the evaluation of the crack initiation site. Inconel 713LC coatings in as sprayed state showed low energetic ductile fracture represented by tiny and shallow dimples in specific regions of cracks. The quality of the substrate/coating interface was excellent with the adhesion strength correlating with a normal load of 75 N applied to a scratch test indenter.

Keywords: Cold Spray, Inconel, scratch test, adhesion, interface

1. INTRODUCTION

Thermal spraying as a family of technologies commonly serves as a best-practice technique for improving the surface properties of metallic components to mitigate wear, corrosion, and thermal damage, thus extending their service life and/or reducing the maintenance time and cost [1,2]. The significant research and development behind cold spray (CS) technology in the last 15 years aims at overperforming conventional thermal sprayed metal coatings and increasing implementation in automotive, aerospace, military, and energy industries. The microstructure of CS coatings can be fully dense without any signs of oxidation or thermal cracks while exhibiting compressive residual stresses. For any coating, performance hinges critically on the adhesion strength between the substrate and the coating. The cohesion strength is also of great importance, but its functionality relies essentially on adhesion as a crucial coating property and a proper test method for its evaluation [1-5]. Traditional testing methods are ASTM C633 and four-point bend testing, both requiring specific thin or even bulk samples and possessing certain limitations. Scratch testing offers a more versatile approach for the evaluation of thermal spray coatings. Depending on the load (0.3 mN – 200 N), individual splats, interfaces, or entire coatings can be tested while as the sample, standard polished cross section after thickness and/or porosity measurements can be used. Scratch testing of coatings offers valuable information about hardness, wear resistance, tribology, damage mechanism, and especially adhesion and cohesion [1-4,6]. The adhesion of thermally sprayed coatings was previously tested by producing a scratch mark on the coating surface with progressively increasing load in the same manner as thin PVD films are tested to this day. For conventional thermally sprayed coatings and especially CS coatings, this testing method is not suitable due to its high thickness (ASTM C1624). Therefore, Lopez et al. [7] developed a novel scratch testing configuration based on creating a set of scratch marks with constant load on a cross section of the coating. Scratch marks start in the substrate on leads through the substrate/coating, interface, coating, and ends in the resin. Each scratch mark is evaluated based on the damage caused to the substrate/coating system, which can result in creation of no cracks at all, cohesion cracks that start and propagate in the coating and adhesion cracks that initiate on the substrate/coating interface and propagate either through the interface or into the coating. Constant load increases in every set until the deployed force predominantly causes the initiation of adhesion cracks. This methodology was later standardized as ISO 27307 suitable mainly for thermal spray coatings with a thickness of at least 20 μm [2,3]. CS coatings are inherently deposited in solid state without excessive oxidation, phase transformations, and porosity, and exhibit significantly different behavior from conventional thermal sprayed coatings. For this specific reason, this paper outlines a new approach based on the ISO 27307 standard more suitable for the evaluation of adhesion bond strength and behavior of CS coatings in general to gather comprehensive experimental data in systematic manner for future reference and comparison of broad portfolio of materials sprayable by CS [1-3,8-10].

This study follows up on previous work [8] in which IN713LC was deposited onto the same material and similar work by Wu et al. [5], who successfully deposited IN713C on the IN718 substrate with a gas pressure of 45 bar and a temperature of 1000 °C. The resulting coating consisted of 10 layers and was only 0.3 mm thick with a low internal porosity of 1.23 % and a hardness reaching up to 500 HV0.5. The low thickness of the coating suggests a low deposition efficiency of the process. The scientific goal is to comprehensively understand the deformation behavior of CS deposited IN713LC, explain and compare the findings and support the implementation of the scratch test with respect to CS.

2. MATERIALS AND METHODS

In this work, gas atomized Inconel 713LC (Sandvik Osprey Ltd., UK) that had spherical morphology with satellites, agglomerated particles, and powder size in the range from 22 to 53 μm was deposited on the Inconel 713LC substrate in the form of a discs with diameter and thickness of 25 and 7 mm, respectively. The substrate surface was ground and polished to a mirror finish (0.7 μm diamond paste) prior to the deposition to increase the adhesion strength between the substrate and the coating. The coatings were deposited using a nitrogen and Impact Innovations 5/11 cold spray gun (Impact Innovations GmbH, Germany) connected to a six-axis robotic arm (IRB 4600, ABB Ltd., Germany). The “OUT 1” de Laval nozzle (SiC) was chosen for deposition along with the Zig-Zag trajectory and two sets of deposition parameters. Processing gas pressure, scanning speed, standoff distance, and powder feed rate were constant with values of 50 bar, 400 $mm \cdot s^{-1}$, 30 mm, and 3 RPM, respectively. All samples were deposited after the substrate was preheated with hot nitrogen (5 passes of the gun without powder). Sample “A1” was deposited using a gas temperature of 1000 °C and 15 passes, while samples “B1” and “B2” were deposited using a gas temperature of 1025 °C and 10 passes.

The thickness and porosity (area of ~5 mm^2) of coatings were measured on light microscopy micrographs taken on a polished cross section of a standard metallographic samples using Olympus StreamMotion software (Olympus, Japan). The Vickers hardness (HV1) of individual coatings was measured in a cross section perpendicular to the deposition direction (Q10A, QAtm GmbH, Germany). Instrumented hardness was measured to acquire Young’s modulus in the range of loads from 0.2 g to 51 g using a continuous multi-cycle mode (CSM Instruments NHT^2, Anton Paar Group AG, Austria). Adhesion and cohesion strength of the substrate/coating interface and the coating itself were evaluated together with the deformation mechanisms mainly according to the ISO 27307 standard using scratch tester (CSM Instruments Revetest RST^2) equipped with a Rockwell type “C” indenter with radius 200 μm. During the testing, few significant drawbacks of the standard were revealed; therefore, the novel methodology for scratch testing was developed and optimized, especially for CS coatings. The drawbacks and methodology will be explained and discussed later. Quantitative tests of adhesion strength of cold sprayed coatings were evaluated according to ČSN EN ISO 14916 (Zwick Z250, ZwickRoell GmbH, Germany). Fractographic analysis and evaluation of fracture mechanism after adhesion testing was performed using electron microscope (Zeiss Ultra Plus, Carl Zeiss AG, Germany) and for scratch testing by electron microscope (Tescan Vega, Tescan Orsay Holding, Czechia) and light microscope (Zeiss Axio observer Z1m).

3. RESULTS AND DISCUSSION

The nitrogen temperature increased from 1000 °C to 1025 °C, resulted in a significant increase in deposition efficiency expressed in thickness of the coating per pass. The coating thickness of the sample A1 was 204 ± 10 μm, i.e. 13.6 μm/pass while for samples B1 and B2 where the original coating thicknesses prior to the adhesion testing according to ČSN EN ISO 14916 was 300 ± 7 μm, i.e. 30 μm/pass.

The improved deposition efficiency did not significantly influence the overall quality of the coatings, apart from the adhesion strength and hardness. The porosity of the A1, B1, and B2 samples was 0.23; 0.18 and 0.22 %, respectively. In correlation with low porosity, the analysis showed very high hardness without excessive crack propagation from the indent corners. The measured hardness of the individual coatings was 634 ± 44, 532 ± 38 and 522 ± 20 HV1, respectively. The low deposition efficiency resulted in a substantial hammering effect caused by impacting and rebounding the powder particles, effectively decreasing porosity, and causing deformation hardening, especially in sample A1 that underwent the longest deposition. All samples exhibited significantly lower porosity, and sample A1 also significantly higher hardness than IN713C coating in work [5].

Adhesion tests according to the standard ČSN EN ISO 14916 showed promising values in coating strengths. Sample A1 deviates from the standard due to being tested alone and its insufficient thickness. However, there was not enough porosity for the glue to penetrate through the coating during the curing process and cause major deviations. The measured adhesion strength was 38.3 MPa and the failure was of adhesive/cohesive character (**Figure 1**). The substrate surface showed only a limited number of residual splats, and the craters left after the detached particles had only a very small area of shallow dimple morphology characteristic for ductile fracture. No jetting was observed around splats on the substrate or in the coating. The fracture mechanism was determined as a combination of low energetic ductile fracture and decohesion between splats.

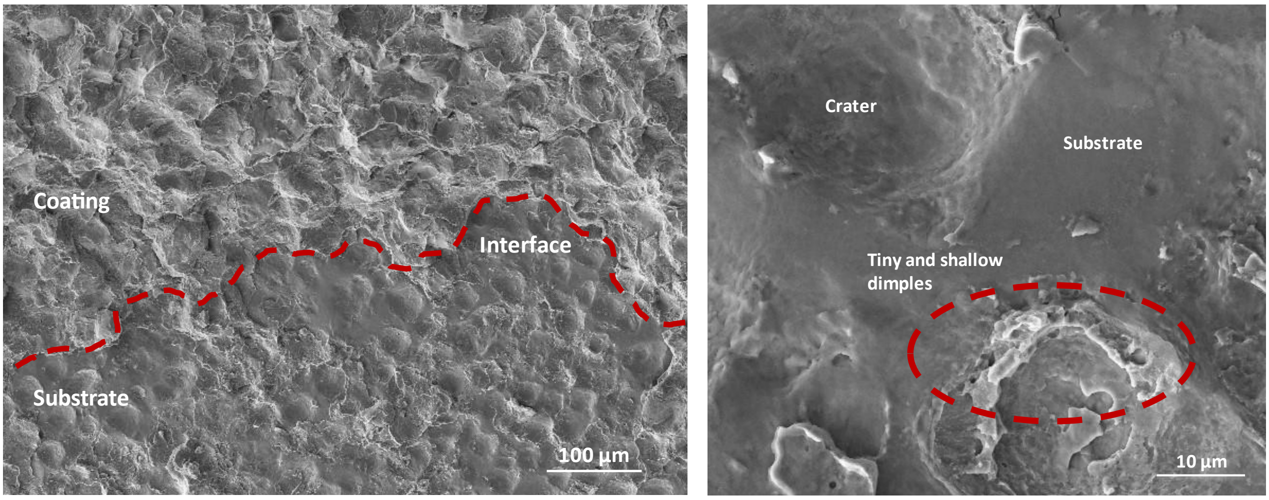

Figure 1 Fractography analysis of sample A1 a) interface between substrate and residual coating; b) shallow dimples on the rim of a crater created by detached powder particle

The adhesion strength of samples B1 and B2 reached up to 51.4 and 65.5 MPa respectively, while the strength of the whole set of samples was 60.4 ± 5.5 MPa. All tested samples exhibited cohesive failure in the glue, resulting in adhesion strengths greater than the measured values.

3.1 SCRATCH TESTING

After tensile adhesion testing, samples A1, B1, and B2 were ground to remove the bonding agent to thicknesses of approximately 200; 230 and 250 µm respectively and used for metallographic analysis and scratch testing. During testing, the normal load (5 – 125 N) increased incrementally with each set of scratch marks (5 – 18 scratch marks per set) until adhesion failure of the substrate/coating interface was predominant. The length of the scratch marks was 1 mm and the scratch speed 2 mm/min. For the final classification, the presented methodology considers the following parameters most important for the quality of bond strength of cold sprayed coatings: i) adhesion strength, ii) length of adhesion cracks, iii) length of cohesion cracks, iv) projected cone area (A_{cn}), v) projected cone angle (α). The testing and optimization of the methodology consisted of an inconsistent number of scratch marks per load throughout the samples. Therefore, all scratch marks were analyzed, the mean lengths of the cracks, the cone areas, and the angles were measured, and results were normalized to 10 scratch marks per sample (mean value multiplied by 10). In the event of a tie between several coatings in adhesion strength that corresponds to the normal load, the length of adhesion and cohesion cracks ranks the coatings accordingly (shorter cracks are better). In the same manner, the evaluation applies to the projected cone area and angle; smaller area and a smaller angle indicate better cohesion behavior of the coating.

According to the standard ISO 27307, scratch marks were evaluated using light microscopy, at first using the scratch tester's objectives and later using the light microscope with extended depth of field (EDF) function that provides images completely in focus. Based on the observations, the damage caused by the indenter during scratch testing of cold sprayed coatings showed characteristics different from those of typical thermal spray coatings (HVOF, APS) when mainly cermet and ceramic and/or metal coatings are deposited. The start of initiation of cohesion and/or adhesion cracks was in the case of a cold sprayed Inconel 713LC coating shifted to higher forces than conventional thermal spray coatings reported in the ISO 27307, while the damage was very complicated to identify and especially assess using only light microscopy tools (**Figure 2**) [3].

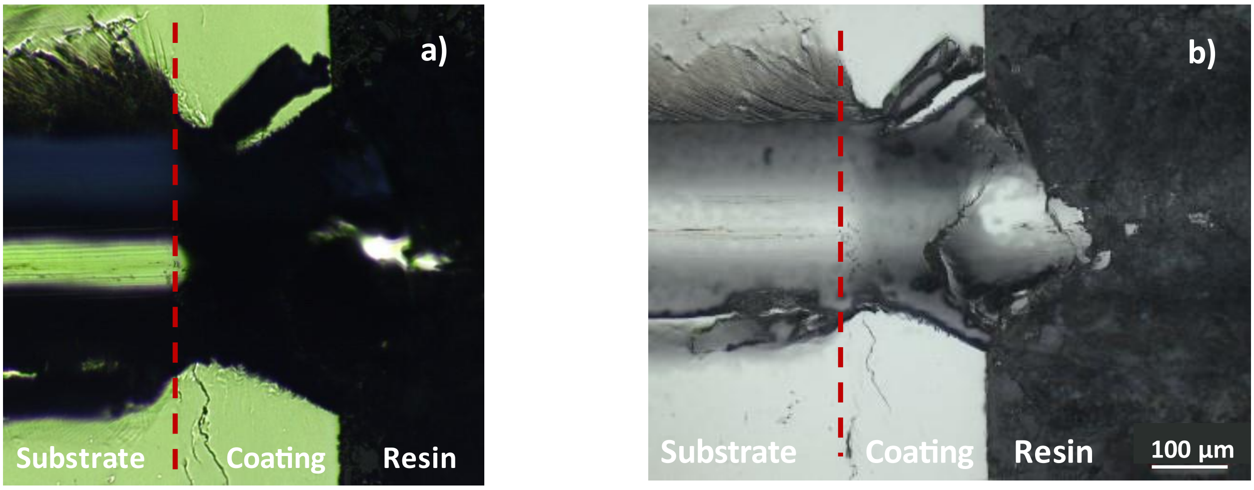

Figure 2 Scratch marks on sample A1 (100 N) a) scratch tester; b) Zeiss Axio Observer with EDF

Scratch tester was equipped with 3 objectives (magnification 5x, 10x, 20x) giving only a limited set of information on the real state of the interface or coating. Ploughed out substrate material on the sides of the groove in the near vicinity of the interface created a shadow that restricts the precise analysis of the substrate/coating interface (**Figure 2)**. In this case (Inconel 713LC deposited on the same alloy), the precise location of the substrate/coating interface was laden with uncertainty. The well-known drawbacks rooting from the principle of light microscopy (low spatial resolution, depth of field) were still present even with the use of an inverted light microscope equipped with the EDF function, resulting in a limited possibility of detecting the crack initiation site. In general, the evaluation of scratch marks using scratch tester objectives, or a light microscope should be indicative only because of the inability to distinguish between, e.g., a distinctive splat interface and adhesion or cohesion crack. Electron microcopy effectively overcame expressed shortcomings and showed the real situation with high magnification and resolution with details enabling us to study fracture surfaces in present cracks, ductile and brittle fracture characteristics and precisely evaluate the scratch marks. Using BSE and/or SE imaging, it was easy to identify the substrate/coating interface resulting in the location of the crack initiation site and measurement of the crack length for further comparison and ranking of individual coatings (**Figure 3)**.

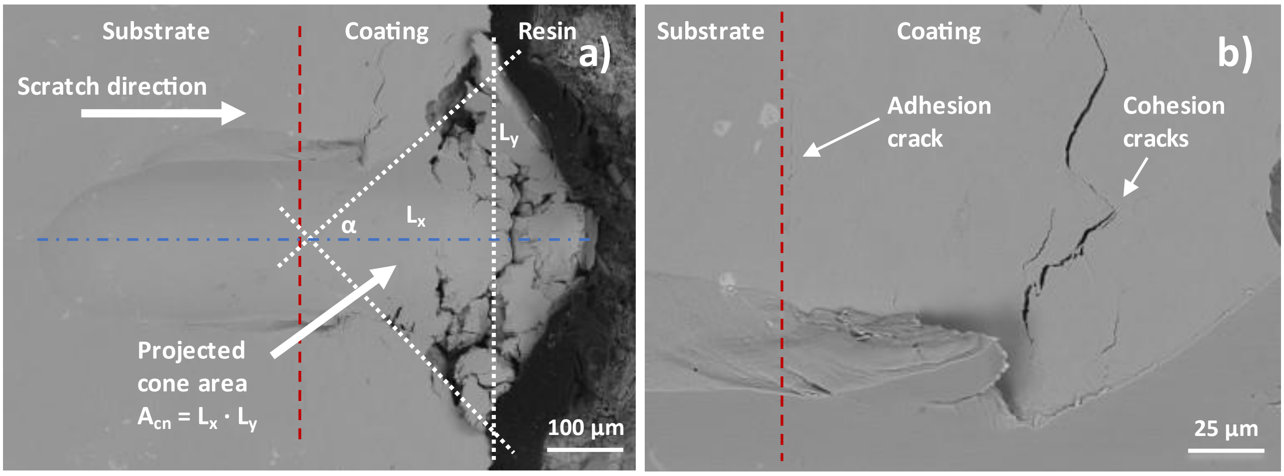

Figure 3 Scratch mark on sample B1 after testing with the load of 75 N (BSE) a) overview; b) detail of the substrate/coating interface

Normalized values of the lengths and cone areas and angles of the cone were used for the final ranking of the samples. Samples A1 and B1 reached up to 75 N as the highest value of normal load where the cohesion failure of the coating was still predominant, while B2 reached only up to 50 N. Based on the methodology presented, the adhesion crack length, as the second most important ranking parameter, in the A1 and B1 were 157 and 543 µm respectively, making A1 the coating with the highest adhesion bond strength. The cohesion crack lengths, projected cone areas and angles of samples A1 and B1 were 1114 µm, 0.5 mm^2, 71.3° and 4829 µm, 0.55 mm^2 and 67.2°, respectively. Similarly, for sample B2 the values of adhesion and cohesion crack lengths, cone area and angle were following: 371 µm, 4686 µm, 0.4 mm^2 and 69.7°, respectively. Hardness as a material property has a strong correlation with wear and abrasion resistance, but regarding adhesion and especially cohesion strength, the correlating material property is Young's modulus [2]. During contact between the sample and the indenter, the deformation rate decreases with increasing Young's modulus, which can result in higher cohesion and adhesion strength. Young's modulus values for samples A1, B1, and B2 were 226 ± 5, 214 ± 11 and 202 ± 22 GPa, respectively, which corresponded perfectly to the ranking according to scratch test.

4. CONCLUSION

The methodology serves as a steppingstone for a more suitable and precise evaluation of the adhesion bond strength of cold sprayed coatings via scratch test according to the ISO 27307. Electron microscopy proved to be a necessary tool for evaluation of individual scratch marks leading to a gain of precise and valuable experimental data, e.g. crack initiation site, adhesion and cohesion crack length, or fracture mechanism.

The difference between individual coatings regarding porosity, adhesion bond strength, and Young's modulus was not marginal. The hardness in the case of sample A1 was higher compared to the rest due to a significantly more pronounced hammering effect during deposition caused by the use of a lower gas temperature during deposition, resulting in a lower deposition efficiency. The mentioned factors played a significant role in the overall highest cohesion and adhesion bond strength of sample A1 examined using the scratch test and the proposed methodology [9].

Based on the different principles of adhesion testing between ISO 27307 and ASTM C633 and experimental data, direct comparison between them seems to be highly uncertain, at least for now [9]. Otherwise, the scratch test is simple, fast, and variable method for evaluation of adhesion/cohesion behavior of cold sprayed coatings requiring only a limited amount of material. For practical use and/or industrial quality control, the methodology could be optimized for even quicker analysis [2,9].

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