Surface & Coatings Technology 344 (2018) 43–51

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Evaluation of adhesion strength between amorphous splat and substrate by micro scratch method

Shu-ying Chen[a], Guo-zheng Ma[a,b,*], Hai-dou Wang[a,**], Peng-fei He[a], Hong-mei Wang[a], Ming Liu[a]

[a] National Key Lab for Remanufacturing, Academy of Armored Forces Engineering, Beijing 100072, China
[b] State Key Laboratory of Tribology, Tsinghua University, Beijing 100084, China.

ARTICLE INFO

Keywords:
Micro scratch
Splat
Plasma spray
Adhesion strength
Debonding mechanism

ABSTRACT

A micro scratch test was performed to evaluate the adhesion strength of plasma sprayed Fe-based amorphous splats deposited on a mirror polished steel substrate preheated at different temperatures. The lateral force (LF), penetration depth (PD) and acoustic emission (AE) signals were recorded to reflect the dislodging process of the splats. The debonding mechanism and the adhesion strength of the individual splat were discussed in details. The results indicate that the adhesion strength of splat was prone to be higher on fully preheated substrate due to sufficient spreading time and good wettability at the droplet/substrate interface. The scratch process contains three stages, i.e., scratching on the bare substrate towards the splat (stage I), contacting and dislodging the splat (stage II), moving away from the splat (stage III). The maximum lateral force (MLF), PD as well as AE signal in stage II vary a lot both in value and range of influenced distance with the increment of substrate preheating temperature. The projected cone areas and residuals onto the scratch can qualitatively reflect the bonding status of splat, whereas the ratio of adhesion force to splat area can quantitatively characterize the adhesion strength of the splat.

1. Introduction

Plasma spray technology has now been widely utilized to fabricate coatings to modify surface properties of components serving in demanding environments, for example, wear resistance coatings in elements with relative motion, anti-corrosion coatings in marine and chemical equipment, thermal barrier coatings (TBCs) in gas turbine, sealing coating in aero-engine compressor, etc. [1–4]. As a heterogeneous material compared with substrate, the adhesion strength plays an important role in the service life and reliability of coatings and components [5,6]. Therefore, complicated experiments need to be done for the improvement of the bonding status at the coating/substrate interface, including the optimization of spraying parameters (i.e., spraying current, spray voltage, spraying distance, gas flow, and powder feeding rate) and substrate pretreatment condition (i.e., coarsening status, preheating status and cleaning status) [7–10].

As is known, mechanical bonding is recognized as the main bonding mechanism for the majority of plasma spraying coatings formed by a stream of molten droplets which undergo rapid deformation and solidification after impinging onto the substrate from the micro perspective. Consequently, two types of interface are naturally formed within the obtained coatings, namely, the first lamellar/substrate interface and the inter-splat interface. The former determines the adhesion strength of the coating, while the latter is related to the cohesion properties of the coating such as micro hardness, elastic modulus and fracture toughness. Therefore, it is important to investigate the impinging and solidifying behavior of molten droplets. According to the published literatures, the wetting process can be influenced by adsorptions, oxide layers and roughness of the substrate, melting degree and velocity of the droplets, reflected by differences in solidification morphologies, residual stress, as well as bonding strength of splats [11–13]. Hence, the evaluation of the adhesion strength of individual splats is conducive to quantitatively comparing the deposition quality of droplets prepared with different parameters. Finally, a more scientific and highly-efficient optimization of experiments can be expected.

Different from entire coatings, the tensile test introduced in the ASTM C433-79 standard can't be performed in the adhesion strength evaluation of individual splats because it is very difficult to apply the glue precisely and exclusively onto a splat. Similarly, the barb pullout method, which applies shear stress parallel to the coating/substrate interface, is not applicable to test the bonding strength of individual splat [14,15]. Therefore, Chromik et al. employed a modified ball bond

---

[*] Correspondence to: G. Z. Ma, National Key Lab for Remanufacturing, Academy of Armored Forces Engineering, Beijing 100072, China.
[**] Corresponding author.
E-mail addresses: magz0929@163.com.cn (G.-z. Ma), wanghaidou@aliyun.com.cn (H.-d. Wang).

https://doi.org/10.1016/j.surfcoat.2018.02.073
Received 22 October 2017; Received in revised form 17 January 2018; Accepted 19 February 2018

shear test method to investigate the adhesion strength of cold spray Ti splat [16]. Balić et al. proposed an indentation method to debond Al_2O_3 splats from the substrate. The strain energy release rate representing the interfacial crack propagation was calculated to characterize the adhesion strength of splats [17]. Nevertheless, the experiment needs to be done with in situ indentation equipment, so that the critical load can be obtained by the observation of interfacial crack propagation.

Alternatively, a lot of research works indicate that micro scratch is promising in testing mechanical properties of films/coatings, such as fracture toughness, adhesion and cohesion strength [18–20], etc. With the micro scratch method, the test location can be accurately selected on the sample surface under an optical microscope. In addition, the operating parameters, e.g., loading force, loading mode (constant or progressive loading) and scratch distance can be adjusted in accordance with different purposes [21]. Recently, Jambagi et al. employ a micro scratch method (MSM) to qualitatively investigate the influence of CNTs on the adhesion behavior of alumina splats deposited on polished NiAl bond coating. The result implies that the adhesion strength of alumina doped with CNTs is larger than that of the original powder because of the bridging and anchorage phenomenon of CNTs [22]. However, the dislodging process of the individual splat was not discussed in detail, such as the quantitative relationship between LF and adhesion strength, the implications of the changes in LF and penetration depth PD of the indenter.

In the paper, well-designed experiments were carried out to clarify the dislodging mechanism through the morphologies of splats before and after scratch testing, and the change rules of lateral force (LF), penetration depth (PD) and acoustic emission (AE) against distance. The adhesion strength of splats deposited on substrates with different preheating temperatures was quantified by the ratio of bonding force to the area of the splat.

2. Experiment procedure

2.1. Operating parameters and deposition of individual splats

The feedstock powder utilized in the paper was a spherical Fe-based amorphous powder (Beijing SunSpray new material Ltd., China) prepared by gas atomization method. The chemical composition of the powder was Fe-48, Cr-15, Mo-14, C-15, B-6, Y-2 (wt%). The median diameter of the powder was approximately 37 μm (± 13 μm) measured by the laser diffraction particle size distribution analyzer (HELOS-RODOS, Germany). Detailed information on the powder was provided in our previous work Ref. [23].

The experiment process is schematically shown in Fig. 1. A highly effective plasma spray system (HEPJet-II, National Key Laboratory for Remanufacturing, China) was employed to prepare individual splats. Argon was used as the primary gas to accelerate the droplets due to many excellent reasons, i.e., ease of ionization, efficiency in momentum transfer and effectiveness in preventing oxidation of the molten alloy droplets [24]. In order to reduce the crystallization of the droplets during solidification, the melting degree of the in-flight particles was controlled by lowering the spraying power and reducing the dwell time of particles in the plasma torch. Thus, the spraying parameters were chosen as follows: current, 380 A; voltage, 160 V; standoff distance, 110 mm; powder feeding rate, 30 g/min; flow rate of Ar, 5.4 m^3/h; flow rate of hydrogen, 0.4 m^3/h.

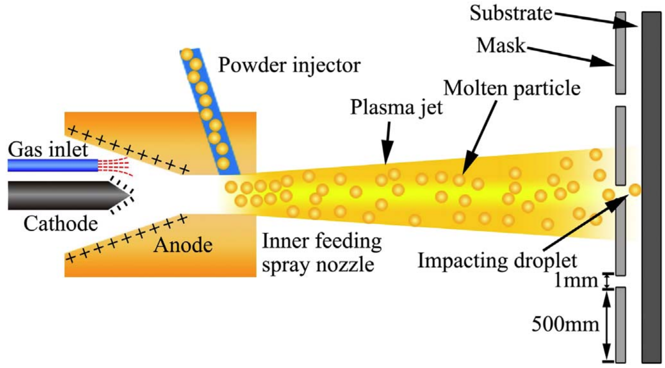

Fig. 1. Schematic of individual splat collecting process.

The droplets were deposited on AISI 1045 steel with a dimension of 60 mm × 20 mm × 5 mm. Prior to spraying, the substrate was polished utilizing 400, 800, 1500, 2000 and 4000 grit SiC papers, then further treated with an abrasive slurry containing 1 μm diamond particles before finishing with 0.04 μm SiO_2 particles. The resulting average surface roughness was $R_a = 0.01$ μm. All the mirror polished substrates were ultrasonically cleaned in acetone for 10 min immediately before splat collection. In order to prevent the splats from connecting with each other and obtain individual splats, the substrates were covered by a baffle drilled with an array of holes with 1 mm diameter and 50 mm interval distance. The traverse speed of the torch was 1300 mm/s with respect to the substrates. The plasma spray gun was employed to preheat substrates to room temperature (25 ± 1 °C), 200 ± 15 °C, 400 ± 35 °C, respectively. A high-precision infrared pyrometer (FLUKE, 62MAX, America) was utilized to monitor the substrate temperature in real-time.

2.2. Micro scratch test method

Scratch tests on individual splats were performed with the Revetest Scratch Tester (CSM Instrument Inc., Switzerland) equipped with a Rockwell-C type conical indenter having an apex angle of 120° and a tip radius of 100 μm. In order to reduce the influence of the elastoplastic deformation behavior of the substrate on the evaluation of the adhesion strength between splat and substrate, the tests were carried out with a normal load of 1 N. The length of the scratch was selected as 400 μm with a velocity of 400 μm/min. During the scratch process, LF and PD of the indenter were recorded by two capacitive transducers, respectively. The acoustic emission (AE) probe was utilized to monitor the signal emitted from cracks propagation during the splat dislodgment process. The digital image correlation (DIC) method was used to analyze the rotation angle and displacement of the splat. For each individual splat deposited on the substrate at different preheating temperatures, a total of 8 to 10 distinct splats were scratch-tested to ensure obtaining 5 effective results, which means the splat was completely displaced from the substrate other than fractured.

2.3. Characterization of splat debonding morphology

The regions where splats were removed by scratch testing were observed by field emission scanning electron microscopy (SEM, FEI Nova Nano SEM 450, Hillsboro, America), equipped with energy dispersive spectroscopy (EDS). The interface morphologies of individual splats were observed by focused ion beam/field-emission scanning electron microscope dual-cross system (FIB/SEM, Zeiss Augriga, Oberkochen, Germany). The width and depth of the scratch were measured by LEXT OLS4000 3D Laser Measuring Microscope (3DLM, Olympus, Japan) with a sampling range of 429.5 × 429.5 μm^2. The area of the debonded splat was calculated by image analysis (IA) method. The micro hardness of coatings and substrate tested by Vickers indentations method is 823HV0.1 (± 45) and 202 HV0.1 (± 18), respectively.

3. Results and discussion

3.1. Morphologies of splat before and after debonding

Fig. 2 shows the typical scratch morphologies of H1 sample (400 °C), M3 sample (200 °C) and L4 sample (25 °C). It can be proved