Effect of substrate roughness on splat formation of thermally sprayed polymer

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Polypropylene (PP) was flame sprayed onto rough mild steel substrates at room temperature (RT) that was preheated at 70 °C, 120 °C, and 170 °C. Single solidified droplets (splats) were collected and analysed to understand how processing variables influenced the thermal spray coating characteristics. The splat morphology was characterized in detail using optical and scanning electron microscopy (SEM).
The splats exhibited a disk-like shape with a large central viscous core and a fully melted wide rim with a thin edge. The splat size increased with increasing substrate temperature. A unique flat microstructure was observed on the surface of the splat deposited onto the RT substrate, whereas a flowing pattern appeared on the splat surfaces deposited onto the preheated substrates and the pattern increased by increasing the substrate temperature. The results of this study revealed improved splat-substrate adhesion by heating the substrate from RT to 170 ºC. On the basis of the result, the influence of substrate parameters on splat morphologies was employed to establish a relationship between the microstructural characteristics and processing variables of flame sprayed polymeric coatings.

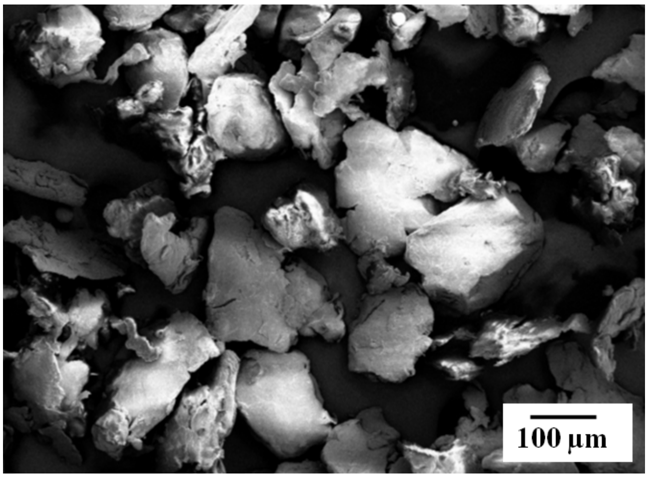
1 Introduction

Flame spray technology has been widely employed for deposition of polymer coatings. Coatings produced by this process can be customized to suit a variety of industrial applications. Fundamentally, the coatings are formed as molten particles impact and spread to form overlapping splats. Thus, the performance of the coatings is closely linked with the way the individual splats are formed [1-3]. It is well known that splat-substrate bonding, splat morphology and spreading in flame spray are depend on substrate temperature [4] and substrate surface [5].
During the last decade, research has demonstrated that the splat morphology and behavior in thermal spray were strongly affected by substrate temperature [6-8]. This work showed that substrates heated above a given temperature, defined as the transition temperature by Fukumoto [9], demonstrate a morphology where a splash changes to a disk. This tendency to change morphology increases with increasing substrate thermal conductivity. In contrast, splats on substrates held below the transition temperature were predominantly irregular or splashed shaped [10-11]. It was also reported that the adhesion of coatings on the substrate preheated above the transition temperature was two to five times better than those on substrates preheated below the transition temperature [12]. There is a good correlation between splat morphology and adhesion strength. However, the underlying cause of the change in splat morphology with substrate temperature is not clearly understood. It has been attributed to changes in substrate surface chemistry [13-14] or changes in substrate surface roughness [15-16].
One of the reported mechanisms to explain the bonding between substrate and splat is mechanical interlocking [17-19] where the coating material penetrates into surface irregularities. Substrate surfaces are often roughened by grit blasting prior to spraying. A polished surface, however, has some roughness at smaller scale. Mechanical interlocking provides higher adhesion strength and roughening of a surface can increase the surface area for more molecular bonding interactions [17, 20]. Upon impact, the droplet spreads out laterally to fill the pits and grooves of the rough surface to form a splat. Good adhesion can be achieved with an increase of surface roughness where more physically active surfaces are present for good bonding. In most cases, the bonding in thermal spray coating is of mechanical nature [18].
The substrate roughness can be distinguished by low substrate roughness when the average surface roughness is Ra < 0.2 μm or high substrate roughness when Ra > 0.2 μm [17]. It was shown experimentally that roughening a substrate can improve adhesion and increase the bond strengths of thermal spray coatings [15]. In the present study, the influence of the substrate temperature and substrate roughness on flame-sprayed PP coating adhesion was investigated with the emphasis on the examination of the first layer of the coating.

2 Experimental

The feedstock was the commercially available Moplen EP203N, PP manufactured by Basell Australia Pty. Ltd. The as-received feedstock powder was in granules that were millimetres in size. The granules were ground using an OMNI mixer homogenizing system, Lomb scientific, AUS into a powder (Fig. 1) with particle size distribution of 70-120 μm (Fig. 2). Liquid nitrogen was used to cool the system and prevent the polymer powder from melting and agglomerating. The four mild steel substrates were grit blasted using aluminium oxide provided by EMAS, Abrasive Salto Ltd, Carborundum, Aloxite, Brazil, type EC31 and grit size 60.
The PP splats were deposited using a Sulzer Metco 6P-II flame spray torch with an acetylene/oxygen gas mixture and air as a carrier gas. An external powder feeder with an internal diameter of 2 mm was used to avoid the high temperature of the torch core that might cause in-flight polymer particle evaporation or decomposition. Powder was fed normal to the flame

and the substrate was held at RT and at 70 °C, 120 °C, and 170 °C. Substrates were mounted perpendicular to the torch and a stand-off distance of 15 cm was selected to produce disk-like splats [4]. The spraying parameters used in this study are shown in Table 1.



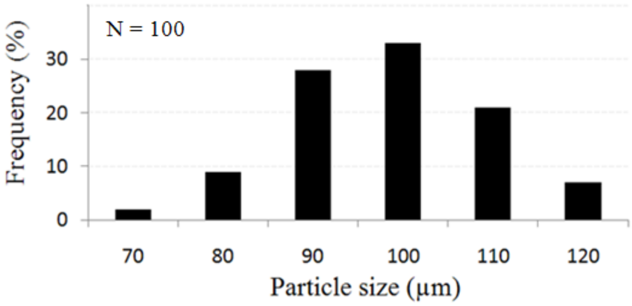


Fig. 1. SEM image of irregular PP feedstock powder

Fig. 2. Particle size distribution of polypropylene used in this study

The exit point of the powder from the feeder is termed as the "insertion port" and was chosen to be 5 cm to prevent premature melting of PP powders within the delivery tube. The morphology and size of powder particles were characterized using a Zeiss Supra 40 VP field emission scanning electron microscopy (SEM). Samples were gold coated with a Dynavac (CS 300) deposition system prior to the SEM analysis. The surface profile roughness of the substrate was obtained and analysed with a VECCO WYKO NT1100 non contact optical surface profilometer and accompanying software (Vision V3.60). The average surface roughness (Ra) was 3.2 µm.

Table 1. Flame spraying parameters used in this study

Torch	Sulzer Metco (Thermospray) torch 6P-II
Stand-off distance, (cm)	15
Oxygen pressure, (KPa)	200
Acetylene pressure, (KPa)	103
Substrate temperature (°C)	RT, 70, 120, 170
Spray angle (deg.)	90

3 Results and discussion

The ability to deposit coating materials onto a wide range of substrate surface finishes with good bonding is highly desirable. In this investigation the substrates are sand blasted for an appropriate adhesion testing observation. The surface roughness of sand blasted substrates is illustrated by a profile scan in Fig. 3. The average surface roughness exceeds Ra 3 µm. The X-Profile (Fig. 3c) and Y-Profile (Fig. 3d) reveal the uniform sand blasted substrates.

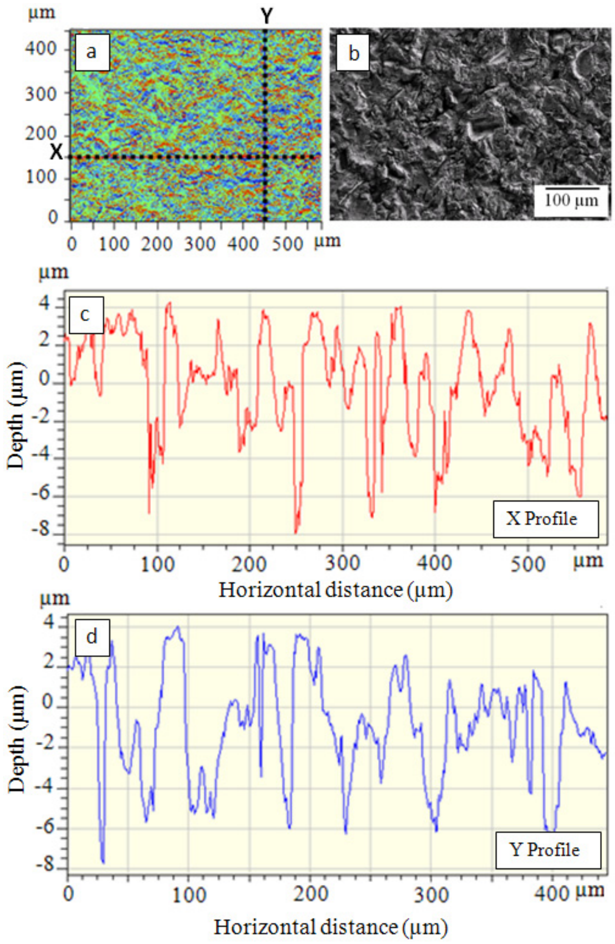


Fig. 3. Surface roughness of the mild steel substrates used in this study obtained by optical surface profilometry (a) is a contour plot data of mild steel substrate surface with lines X and Y indicate the locations of profile measurements (b) a SEM image of the rough surface of mild steel substrate (c and d) plot of depth vs horizontal distance of X and Y profiles, respectively.

The splat observation indicate that the PP splat morphologies vary with preheating temperature. Splat areas and the splat morphology generally increase with increasing substrate temperature (Fig. 4). Increases in splat area, diameter and degree of circularity arise due to the increased time available for splats to spread out and solidify after impact since the temperature difference between the splat and the substrate is decreased.
Disc-like splats, distinguished by a "fried egg" morphology with little splashing evident, were

exhibited on the SEM images. The fried egg phenomenon is exhibited by polymer splats on substrates held at room temperature due to the large radial difference in the flow properties of the molten PP droplets. The lower viscosity of the fully molten rim layer contrasts with the highly viscous core that does not spread upon impact [21-22].

Particles deposited onto the preheated substrates show flattened hemispherical shaped splats that are partially melted forming fried-egg splats with a greater degree of melting and post-deposition material flow, as shown in Fig. 4(a-d). These particles experience a longer cooling time and reflect solidification conditions that are dictated by the substrate temperature and relative surface tensions. Heating up the substrate close to the PP powder melting point range of 160-170 ºC produced fully molten splats with a distinct cusp on the centre of the splat surface. This could be attributed to splat shrinkage during solidification, see Fig. 4d.

The SEM images show patterns with different scales of undulating surface texture in the splat surface mound; i.e., the thicker portion of the splat, Fig. 4(a-d). Surface ripple patterns increased with increasing substrate temperature. It is proposed that there are two forces acting on the droplet flattening and solidification; (i) the effect of the substrate temperature that influences polymer to flow and (ii) the splat surface tension that impedes material flow. The PP flow increased as the substrate temperature increased and is reflected in a decrease in splat thickness. The wave size varies with respect to the substrate preheat from a slight wavy surface texture at a lower preheat temperature (Fig. 4a) to a large texture at the higher temperature (Fig. 4d). This effect would not be observed in the thinner periphery where the shrinkage is restricted by the low thermal expansion of the mild steel.

Good adhesion between the substrate and splat is required and the adhesion mechanism depends on the surface characteristics of the abutting materials. Metallurgical bonding is not relevant in the present case since the polymer is coating a metal substrate. Bonding by mechanical interdigitation of the molten or semi-molten PP with the substrate is the most likely adhesion mechanism. The high pressure of impacting droplets thrust material into surface crevices and form interlocking connection as droplets freeze.

An adhesion defect described as a splat delamination may occurs as a result of splat edge lifting at the interface between the splat and substrate due to inadequate adhesion. Good bonding is expected to occur over the region of the highest pressure within a droplet. The combination of low contact pressure and high surface tension at the droplet edge leads to poor contact (Fig. 5a). If the next deposited droplet cannot fill in the gap caused by the previous curled-up splat, then porosity is created which in the majority of applications is not desirable.

Substrate temperature enables the formation of well-melted splats that can coalesce. Fig. 5 illustrates the differences in splat morphologies due to the changes in substrate temperature. The figure shows splats that are still rounded and partially deformed, indicative of high viscosity with little subsequent deformation at impact due to the relatively high cooling rate on the 70 ºC substrate. A crack was induced around the splat perimeter where the splat was thin enough to break up (Fig. 5b).

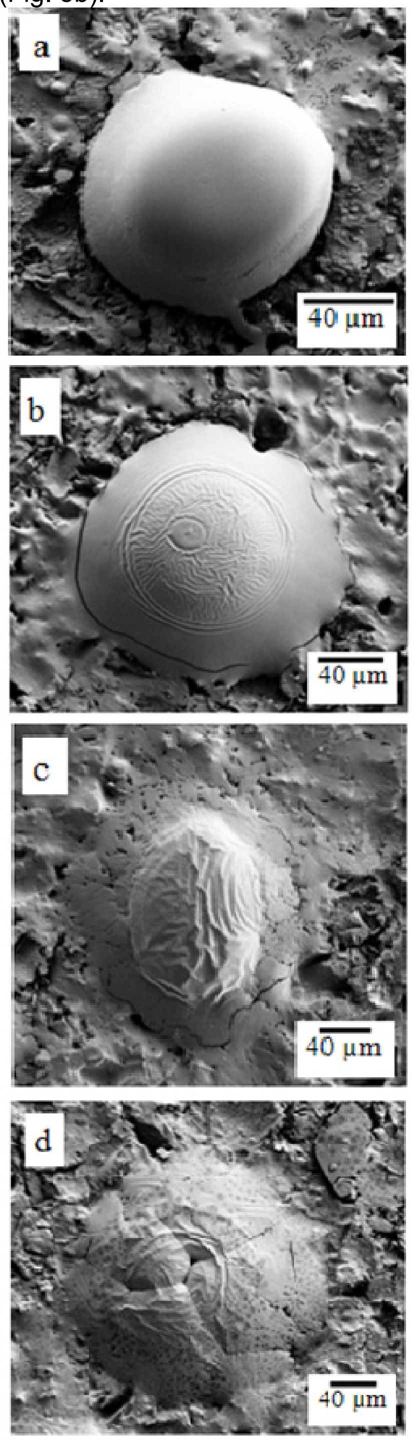


Fig. 4. SEM images of flame sprayed polypropylene particles impacted onto a sand blasted mild steel substrate at different preheating temperatures, showing the effect of substrate temperature on the splat topology, size and surface texture; (a, b, c and d) are the top view of full single splats at different preheating temperatures (i.e., room temperature, 70 ºC, 120 ºC and 170 ºC, respectively).

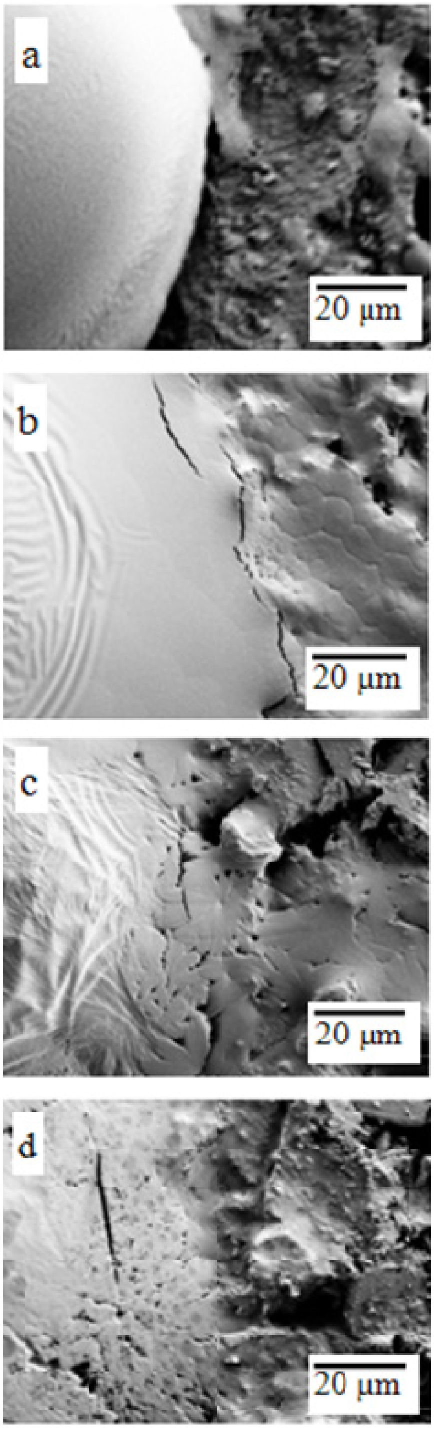


Fig. 5. SEM images of flame sprayed polypropylene particles impacted onto a sand blasted mild steel substrate at different preheating temperatures, showing the effect of substrate temperature on the splat adhesion to the substrate; (a, b, c, and d) are the splat edge in contact with the rough substrates at different preheating temperatures (i.e., room temperature, 70 ºC, 120 ºC and 170 ºC, respectively).

The degree of particle deformation reflects the thermal input into the particle and substrate. Raising the

As the substrate temperature increased to 120 ºC, the flow of material increased to overcome the splat surface tension and increase the thickness of the splat perimeter and reduce the possibility of crack occurrence (Fig 5c). The highly deformed disc-like splats signify particle melting and flow deformation during impact due to a lower cooling rate at the higher substrate temperature of 170 ºC (Fig. 5d). Thus, fully melted splats were formed with no cracks at the splat edge and a more uniform splat thickness.

Altering the substrate temperature caused the most significant change in the degree of splashing compared to other spray parameters. The previous study showed that stand off distance has a very high influence on degree of splashing [4]. However the general splat morpholgies of polypropylene were consistent. They did not demonstrate significant differences with previously research of splats deposited onto different substrate material at different surface conditions (Fig. 6). A preliminary SEM observation of various splat regions indicated that the final splat diameter increased as the substrate temperature increased.

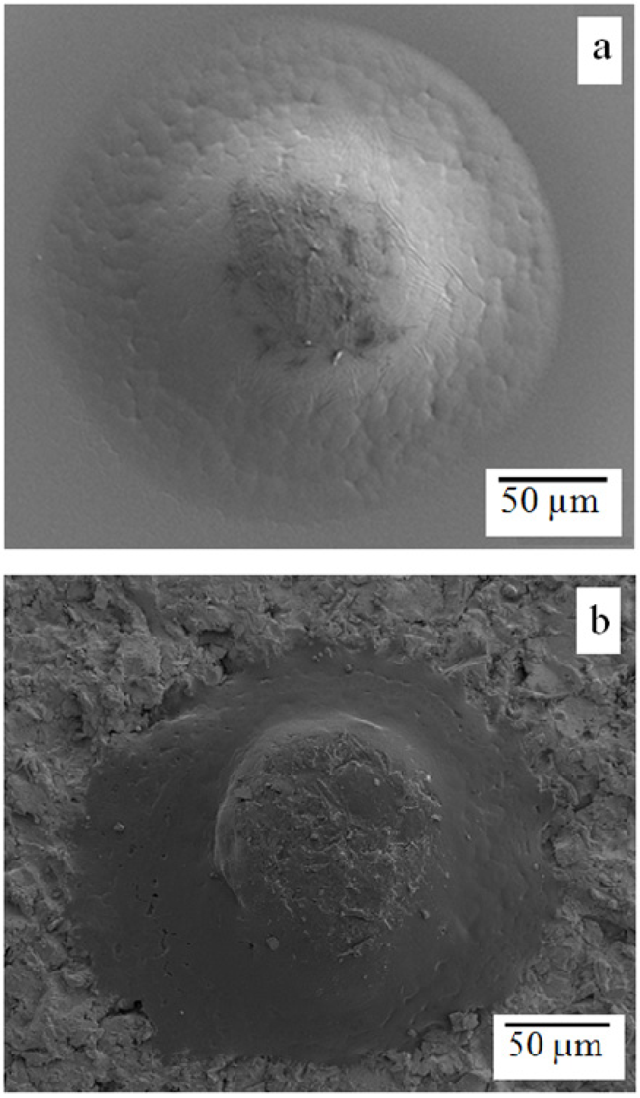


Fig. 6. SEM images of flame sprayed polypropylene particles onto impacted (a) a glass slide substrate at room temperature and (b) a rough mild steel substrate at preheated to 120 ºC.

4 Conclusion

A flame spray process was used to form polypropylene splats against a grit blasted mild steel substrate held at different preheated substrates (i.e., RT, 70, 120, 170°C). The effects of a preheated substrate on splat morphology and microstructure of polypropylene were examined. The PP splat morphologies were shown to be affected by the temperature of the substrate as well as the degree of particle melting. Raising the substrate temperature from RT to 170ºC provided splats that were well melted and which displayed good adhesion to the substrate. Higher substrate temperatures also allowed splat coalesce between lamellar and kept splashes to a minimum.

The size of disk-like splats was increased with increasing substrate temperature. Thus, the flattening ratio increased. The splats exhibited a disk-like shape with a large viscous core in the centre and a fully melted wide rim with a thin edge. The splat shape changed as the substrate temperature increased. The splat edge became thicker since there was more flow of material towards the perimeter. The central regions of the splat were more uniform with a reduced overall thickness.

5 References

1. J.R. Davis, Handbook of Thermal Spray Technology. 2004, USA: ASM International.
2. L. Pawlowski, The Science and Engineering of Thermal Spray Coatings. Second Edition ed, ed. L. John Wiley & Sons. 2008 England: John Wiley & Sons, Ltd. . 647.
3. K. Alamara, S. Saber-Samandari, and C.C. Berndt, Splat taxonomy of polymeric thermal spray coating. Surface and Coatings Technology,In Press, Corrected Proof.
4. K. Alamara, S. Saber-Samandari, and C.C. Berndt, Splat formation of polypropylene flame sprayed onto a flat surface. Surface & Coatings Technology,205 (2010). pp. 2518-2524.
5. K. Alamara, S. Saber-Samandari, P. Stoddart, and C.C. Berndt, Effect of substrate temperature on the splat formation of flame sprayed polypropylene. Surface and Coatings Technology, submitted May,(2011).
6. M. Fukumoto and Y. Huang, Flattening mechanism in thermal sprayed nickel particle impinging on flat substrate surface. Journal of Thermal Spray Technology,8 (1999).3: pp. 427-432.
7. M. Fukumoto, E. Nishioka, and T. Matsubara, Flattening and solidification behavior of a metal droplet on a flat substrate surface held at various temperatures. Surface and Coatings Technology,120-121 (1999). pp. 131-137.
8. A. McDonald, M. Lamontagne, C. Moreau, and S. Chandra, Impact of plasma-sprayed metal particles on hot and cold glass surfaces. Thin Solid Films,514 (2006).1-2: pp. 212-222.
9. M. Fukumoto, E. Nishioka, and T. Matsubara, Effect of interface wetting on flattening of freely fallen metal droplet onto flat substrate surface. Journal of Thermal Spray Technology,11 (2002).1: pp. 69-74.
10. V. Pershin, M. Lufitha, S. Chandra, and J. Mostaghimi, Effect of substrate temperature on adhesion strength of plasma-sprayed nickel coatings. Journal of Thermal Spray Technology,12 (2003).3: pp. 370-376.
11. M. Pasandideh-Fard, V. Pershin, S. Chandra, and J. Mostaghimi, Splat shapes in a thermal spray coating process: Simulations and experiments. Journal of Thermal Spray Technology,11 (2002).2: pp. 206-217.
12. J. Cedelle, M. Vardelle, and P. Fauchais, Influence of stainless steel substrate preheating on surface topography and on millimeter- and micrometer-sized splat formation. Surface and Coatings Technology,201 (2006).3-4: pp. 1373-1382.
13. A. McDonald, C. Moreau, and S. Chandra, Effect of substrate oxidation on spreading of plasma-sprayed nickel on stainless steel. Surface and Coatings Technology,202 (2007).1: pp. 23-33.
14. A.A. Syed, A. Denoirjean, B. Hannoyer, P. Fauchais, P. Denoirjean, A.A. Khan, and J.C. Labbe, Influence of substrate surface conditions on the plasma sprayed ceramic and metallic particles flattening. Surface and Coatings Technology,200 (2005).7: pp. 2317-2331.
15. W.J. Trompetter, M. Hyland, P. Munroe, and A. Markwitz, Evidence of mechanical interlocking of NiCr particles thermally sprayed onto Al substrates. Journal of Thermal Spray Technology,14 (2005).4: pp. 524-529.
16. M. Raessi, J. Mostaghimi, and M. Bussmann, Effect of surface roughness on splat shapes in the plasma spray coating process. Thin Solid Films,506-507 (2006). pp. 133-135.
17. P. Fauchais, M. Fukumoto, A. Vardelle, and M. Vardelle, Knowledge concerning splat formation: An invited review. Journal of Thermal Spray Technology,13 (2004).3: pp. 337-360.
18. S. Chandra and P. Fauchais, Formation of solid splats during thermal spray deposition. Journal of Thermal Spray Technology,18 (2009).2: pp. 148-180.
19. S. Dallaire, Influence of temperature on the bonding mechanism of plasma-sprayed coatings. Thin Solid Films,95 (1982).3: pp. 237-244.
20. F. Awaja, M. Gilbert, G. Kelly, B. Fox, and P.J. Pigram, Adhesion of polymers. Progress in Polymer Science (Oxford),34 (2009).9: pp. 948-968.
21. M. Ivosevic, R.A. Cairncross, and R. Knight, 3D predictions of thermally sprayed polymer splats: Modeling particle acceleration, heating and deformation on impact with a flat substrate. International Journal of Heat and Mass Transfer,49 (2006).19-20: pp. 3285-3297.
22. B.P. Withy, M.M. Hyland, and B.J. James, The Effect of Surface Chemistry and Morphology on the Properties of HVAF PEEK Single Splats. Journal of Thermal Spray Technology,17 (2008).5-6: pp. 631-636.