CERAMIC OXIDE LAYERS MANUFACTURED BY ATMOSPHERIC INDUCTION
PLASMA SPRAYING

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1. Introduction

In the last 10 years, the induction plasma spraying is the subject of intensive theoretical and experimental research. The vacuum induction plasma spraying is the mostly used technology in the field of Radio Frequency (RF) Inductively Coupled Plasma (ICP) applications (e.g. [1-3]). There is a general trend to a high kinetic particle energy at the moment of impact onto the substrate surface.

The operating conditions of the induction plasma spraying (IPS) at atmospheric pressure (AP) differ quite strongly from the conditions at reduced pressure. The powder heating-up and the acceleration conditions disagree with the most other spraying processes. Therefore, the impaction of liquid droplets onto the substrate surface is quite different, and also the coating morphology.

Based on the temperature and velocity profiles of the atmospheric pressure IPS equipment used dwell times and velocities of the powder particles are estimated. The impinging particle conditions at varying substrate temperatures are demonstrated in the paper. The specialties of the particle impact are emphasized, especially the deformation of single droplets and of sprayed layers, the thickness of the lamellae, the porosity and the phase composition using pure alumina as feedstock material. A comparison is drawn to conditions of other thermal spray processes, such as DC plasma spraying and HVOF.

2. Experimental Set-up

2.1 The ICP Torch

As in previous papers described [4,5], the ICP torches developed and always used at the laboratory are composed of quartz tubes (Fig.1). The transparent working gas and sheath gas tubes enable to spy the plasma configuration and the powder flow through the plasma. In the torch head there are the two gas distribution rings. The sheath gas is tangentially injected into the gap between the sheath and working gas tubes being smaller than 1 mm by 18 nozzles with an inner diameter of 1 mm and a slope of 40° to the horizontal. The working gas is axially injected by 12 nozzles. The sheath gas tube confining the plasma is only air-cooled. The six-turned induction coil with a rectangular cross section for a better energy transfer and an inner diameter of 36 mm is supplied by the RF Generator with a maximum plate power of 25 kW at a frequency of 3.7 MHz. The experimental arrangement with the plasma jet is shown in Fig.1.

The also in-house developed watercooled powder feeding probe is made of stainless steel with an inner diameter of 2 mm. It is placed approximately at the second turn of the induction coil and therefore, within the plasma core (Fig.1). Under atmospheric conditions, the plasma has to be ignited by a graphite or tungsten rod. The parameters used for AP IPS are given in table 1.
Table 1: Parameters used for alumina spraying

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate Power</td>
<td>12 kW</td>
</tr>
<tr>
<td>gas flow rate: working gas Argon</td>
<td>6.7 slpm</td>
</tr>
<tr>
<td>sheath gas Argon</td>
<td>33 – 42 slpm</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>0 – 2 slpm</td>
</tr>
<tr>
<td>carrier gas Argon</td>
<td>2 slpm</td>
</tr>
</tbody>
</table>

The visible plasma jet has a diameter of about 20 mm and a length of about 120 mm.

Plasma temperatures and velocities are measured by the TEKNA Enthalpy Probe Measurement System. The enthalpy probe could be immersed into the torch only 40 mm up to the lower turn of the induction coil. The measured temperatures of a specific run is shown in Fig. 2. The highest temperature measured was 10 kK. Outside of the torch, the plasma jet is slightly shifted from the torch axis. Temperatures lower than 5 kK could not be measured because the error exceeds more than 10%.

Fig.1: Visible plasma in the torch

The plasma velocity at the exit of the sheath gas tube amounts to 75 ms\(^{-1}\). That is relatively low compared to the velocities obtained with the DC plasma torch. Therefore, the acceleration of the large powder particles used at the ICP is quite low and, following from that, the particle velocities are only in the range of 10 to 30 ms\(^{-1}\) depending on the parameters used.

The argon content is measured by the BALZERS’ mass spectrometer belonging to the TEKNA’s Enthalpy Probe Measurement System.

The carrier gas flow rate used is the minimum flow rate of the METCO powder feeder MFP-1. This carrier gas flow rate sets the content of nitrogen or oxygen in the plasma axis as is shown in Fig. 3.
2.2 Powder behavior

The feedstock was 99.5 alumina with a powder size of 70 until 90 μm as is shown in Fig. 4. The plasma parameter and powder feeding parameter used are defined by the powder spheroidizing rate. The maximum powder feeding rate at a plate power of 12 kW results to about 10 g/min with a spheroidizing rate of more than 80%. In order to guarantee a nearly complete melting of all powder particles a powder feed rate of 3 g/min is used. Impinging onto the substrate, the liquid droplets splash or spread more or less. At substrate temperatures more than 300 °C spreading without splashing is obtained shown in Fig. 6.

<table>
<thead>
<tr>
<th>100 °C</th>
<th>250 °C</th>
<th>400 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Image]</td>
<td>[Image]</td>
<td>[Image]</td>
</tr>
</tbody>
</table>

Fig. 6: Spreading of ICP sprayed single alumina powder particles on steel substrate
That is in contrast to DC-Plasma sprayed particles. Here, the transformation temperature of alumina on steel substrates is in the range of 100 °C [6, 7].

Substrate temperatures more than 300 °C has to be chosen particularly for ICP spraying with small particle velocities because at higher temperatures splashed droplets predominantly exists, and the contact areas of pancakes formed by spreaded droplets are larger than with splashed ones. Because the incoming liquid alumina droplet temperatures exceed the melting point of the steel substrate in the contact zone, and the mass of the ICP atmospherically sprayed particles is about 30 times higher than the DC-APS particles, the droplets melt down the substrate material within the contact area shown in Fig. 7+8. The penetration of the droplet into the substrate is about 4 µm. By means of these settings a good adhesion of the coatings are attained topping always the adhesion forces of the glue (Ultrabond).

Fig. 7: SEM view of the contact area  
Fig. 8: Cross section of the down-molten substrate

3. Coating Manufacturing and Properties

The opportunity to attain nearly 100% melting of all injected powder particles within the ICP leads to a high deposition efficiency. Especially, a deposition efficiency of about 90% is obtained with round steel probes (diameter 50 mm), and a deposition efficiency of more than 95% is attained with flat steel substrates using pure alumina 99.5 with grain size in the range of -100+80 µm.

In spite of relatively large particles the cooling down times of the lamellae are always in the range of about 160 µs independently of whether they hit on the steel substrate or on the alumina lamella. That is relatively long compared to the DC APS conditions, but always still short enough that all following droplets impinge on already cooled down lamellae. Moreover, the grade of deformation on lamellae differs not substantially from single particles on the substrate surface. The mean lamellae thickness is approximately 12 µm. As is shown in Fig. 9, the ICP sprayed coatings show a clear lamellar structure close to and away from the substrate. There are few or no unmolten particles included in the coating. Regarding the phase composition in the XRD diagram (Fig. 10), the α-phase in the IPS coating is in the same manner predominant as in the feedstock in contrast to other thermal spray coatings.

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As described in previous papers [1], the porosity determined by image analysis is in the same region (2-4%) as in the DC APS coatings. In DC APS coatings there are many and small pores, and in the case of AP IPS few, but larger pores are predominant.

The adhesion force measured analogously to DIN EN 582 is obtained to max. 60 N/mm² which is in the range of the glue adhesion force with 60 N/mm².

Fig. 9: Cross-section of AP IPS alumina coating

Fig. 10: XRD diagrams of Al₂O₃ IPS coatings in comparison to powder and other thermal sprayed coatings

The measured Vicker’s microhardness (HV 0.1, DIN 50103) at sample cross-sections is always higher in the case of IPS related to DC APS. For alumina coatings the microhardness amounts to 1,450 related to about 1,000 and less obtained with other thermal sprayed samples. This higher hardness has to be taken into account for sample grinding and polishing. It is extraordinary difficult to cut the AP IPS samples for crosssectional view with the microscope. If possible, the cutting should always be avoided, otherwise timeconsuming polishing with small loads has to follow. An instruction is imposed in the case of the special compound alumina – steel given in table 2 [8] established in cooperation with the companies Wirtz-Buehler, Duesseldorf, and Struers, Willich, which is gratefully acknowledged. All samples are done with these parameters of table 2.

4. Conclusions

It is shown that alumina coatings could be sprayed very well also by the inductively coupled plasma under atmospheric conditions. The coatings are dense with few, but larger pores than at DC plasma spayed samples. The porosity was in the range of 2-4%, the adhesion amounts up tp 60 N/mm².

The cross sections show a strong lamellar structure. Due to the larger lamellae the microhardness and following from that, also the brittleness are much higher than in DC plasma sprayed coatings. That demands a special preparation procedure of IPS alumina coatings.

The composition of the coating consists predominantly of the α-phase in contrast to other spray techniques. It could be evidenced that this fact is not due to unmoitened particles
integrated in the layer but rather by the lower cooling rate in comparison to other thermal spray processes. Table 2: Grinding instructions of AP IPS alumina coatings on steel substrates

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</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Diamond foil with 40 µm</td>
<td>-</td>
<td>Water</td>
<td>75</td>
<td>200</td>
<td>60</td>
<td>5-10 up to plan</td>
<td>Especially for cutted samples</td>
</tr>
<tr>
<td>2</td>
<td>Diamond foil with 20 µm</td>
<td>-</td>
<td>Water</td>
<td>75</td>
<td>200</td>
<td>60</td>
<td>Up to plan</td>
<td>Start-up if no cutting or remedy of cutting damages</td>
</tr>
<tr>
<td>3</td>
<td>Hard cloth, e.g. Texmet (Wirtz-Buehler) or DP-Plan (Struers)</td>
<td>Diamond suspension, 9 µm, e.g. Metadi (Wirtz-Buehler)</td>
<td>f.e. Dialub (Wirtz-Buehler)</td>
<td>100</td>
<td>150</td>
<td>60</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Hard cloth</td>
<td>Diamond suspension 3 µm, e.g. Metadi (Wirtz-Buehler)</td>
<td>f.e. Dialub (Wirtz-Buehler)</td>
<td>100</td>
<td>150</td>
<td>60</td>
<td>15</td>
<td>Final step, as long as no further improvement</td>
</tr>
</tbody>
</table>

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References

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