Tribological properties of stainless steel/graphite atmospheric plasma spray composite coatings

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Abstract:

The objective of this study is to achieve by Atmospheric Plasma Spraying (APS), on aluminum alloy substrates, a coating exhibiting a higher superficial hardness and an improved tribological property compared to that of the aluminum alloy. The achieved composite coating is constituted of a stainless steel metallic matrix in which are incorporated graphite particles as lubricant. Tribological tests have been achieved on a pin on disk type tribometer. The evolution of the friction coefficient (f) of the composite coating is compared with that of the commonly used stainless steel deposit. For all conditions of tests (three loads and three different slip speeds), the friction coefficient decreased by about 30% for coatings containing graphite.

1. Introduction

Various applications require the utilization of parts with adapted superficial properties, such as a high hardness, a good resistance to wear, friction, oxidization, corrosion and abrasion. In this study, it is wanted to increase the hardness and improve the tribological properties of aluminum parts.

Different surface treatments have been developed and are used industrially to achieve such requirements [1]. For some applications where areas are submitted to high stress and where the mechanical resistance of the substrate is weak, thick coatings (a few hundreds of µm) are required, for which thermal spraying is well adapted. They consist in introducing particles (in the tens of µm range) in a flame or a plasma jet in order to accelerate and melt them before they flatten on to the substrate or the previously deposited layers where the deposit is formed by the resulting splats layering [2], [3].

In order to decrease the friction coefficient between two parts and achieve their lubrication, the deposit sprayed on one of the parts must contain some solid lubricants. Unfortunately the best solid lubricants such as the graphite, CaF\textsubscript{2}, boron nitride or MoS\textsubscript{2}, have no melting point, or are decomposed at a rather low temperature especially in contact with oxygen. Plasma spraying, which is essentially based on the melting of particles within a high temperature (between 8000 and 14000 K) plasma jet core, is therefore not adapted to spray solid lubricants. It is thus necessary to introduce the lubricant within the deposit without melting it while keeping the sprayed coating mechanical resistance. It can be done for example by

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including the solid lubricant in a metallic matrix. The idea was therefore to use plasma spraying to achieve the metallic matrix, by injecting steel particles downstream of the torch nozzle exit, and simultaneously injecting particles of solid lubricant in a zone where they won't be melted or decomposed but will be incorporated in the deposit during its formation. Aiming different domains of applications of mechanics: ship building, automotive and aeronautical, it has been chosen to achieve a deposit composed of a stainless steel matrix (316L) with inclusions of graphite particles and sprayed on a substrate of aluminum alloy. First of all will be presented the used experimental devices, the plasma spraying set up with the graphite powder feeder, and the tribology test configuration. Then, the production of the composite deposits, their morphological characterization, as well as their tribological behavior will be discussed.

2. Experimental devices

Plasma spraying set up
The plasma spraying set up (see figure 1) comprises a d.c. plasma spray torch, a rotating substrate holder (120 mm in diameter), an air barrier slot avoiding the substrate heating by the plasma plume, two powder feeders with their corresponding injectors and a rear air cooling system of samples. Air plasma spraying (APS) is performed with a conventional d.c. plasma gun. The torch is a PTF4 type, and has an anode nozzle made of copper 7 mm in internal diameter. The cathode made of thoriated tungsten (2 wt% of Thoria) is a cylinder 10 mm in diameter with a conical tip with an angle of 40°.

![Diagram of plasma spraying set up](image)

**Figure 15:** device of projection
A stainless steel powder (316L) with a size distribution 60-130 μm is injected perpendicularly to the axis of the plasma jet through an injector 2 mm in internal diameter, located 3 mm downstream of the torch nozzle exit and at 4 mm of the axis of the torch. A vibrating powder feeder, in a spiral shape, is used with 6 Nl/min of argon as carrier gas. For the different tests, stainless steel powder flow rate is varied from 5 to 80 g/min. Aluminum disk shaped substrates (40 mm in diameter, 8.5 mm in thickness) are mounted on a cylindrical sample holder (120 mm in external diameter), which can contain 12 samples. The distance between the nozzle exit and the substrates is fixed at 100 mm. The sample holder rotates at about 2.5 rps and is simultaneously translated orthogonally to the plasma jet axis at a velocity of 20 mm/sec, with an excursion of 80 mm, the plasma torch being stationary. Just before spraying, substrates are grit blasted with corundum with a mean diameter of 400 μm. The blasting pressure is 0.6 MPa with a tungsten nozzle 8 mm in internal diameter. The nozzle-substrate distance is 100 mm. The grit-blasting is made perpendicularly to the
substrate. Then substrates are cleaned in the acetone in an ultrasonic bath. The resulting roughness is $Ra = 7 \pm 1\, \mu m$.

A compressed air barrier machined slot, 28 mm in length and 1 mm in width, is located 80 mm downstream of the nozzle exit perpendicularly to the plasma jet axis and at 15 mm of its axis. Its main goal is to limit the thermal flux of the plasma plume on the surface of the substrate and avoid the blowing out of graphite particles.

Graphite particles with a mean diameter of 8 $\mu$m (size distribution in the range $\pm 12 \pm 2 \mu$m), are injected close to the location of the coating formation with a vibrated fluidized bed feeder where the exit of the injection tube (internal diameter of 4 mm) is located at 30 mm of the substrate surface. The argon carrier gas of the graphite is fixed at 7 Nl/min.

The temperature of the substrate and coating is controlled during spraying by a cooling air jet. This cooling is achieved by means of an air cooling slot (1 mm in width and 28 mm in length) disposed perpendicularly to the sample holder and blowing in the direction opposite to the plasma jet flow. The air flow rate can be adjusted in order to control the surface temperature of the deposit under construction between 200 to 500 °C. This temperature is monitored by a monochromatic pyrometer (of type Ircin Modelin 7000) which wave length is 5.1 $\mu$m and response time 0.1 sec. The typical parameters of the plasma spray torch for the coating deposition are presented in table 1.

<table>
<thead>
<tr>
<th>Plasma gas Ar/H2</th>
<th>Arc current</th>
<th>Torch thermal efficiency</th>
<th>Voltage</th>
<th>Power</th>
</tr>
</thead>
<tbody>
<tr>
<td>46/14 slm</td>
<td>530 A</td>
<td>56 %</td>
<td>62 V</td>
<td>32 kW</td>
</tr>
</tbody>
</table>

**Table 3: conditions of plasma spraying**

The achieved deposits are characterized by scanning electron microscopy (SEM), coupled with an Energy Dispersive Spectroscopy probe (EDS) and by X-ray diffraction (XRD). The microhardness is measured with a tester (Zwick 321200) equipped of a Vickers tip. A load of 2 N has been applied during 10 seconds and the mean value of the obtained microhardness results of an average on 15 measurements.

**Tribological testing configuration**

Tribology tests have been carried on a pin on disk type tribometer (see figure 2). The disk is the substrate with the composite deposit millstone corundum rectified (final Ra about 1.7 $\mu$m), its outside diameter being 40 mm and its thickness 8. 5 mm. The disk rotates at a constant speed. The pin made of 100C6 steel is a shouldered cylinder, which friction surface has a diameter of 6 mm. Three conditions of sliding speed are used with three different applied normal static loads N. The pin is connected to a cell allowing to follow the time evolution of the applied torque.

![Figure 16: tribological test configuration: pin on disk](image)

The friction coefficient is calculated by using the relationship $f = \frac{C}{NR}$, where R is the mean radius of the friction track (16 mm in our case), N the applied load and C the applied torque.

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3. Results and discussion

The achieved deposits
For constant spraying parameters, the thickness of the deposit is conditioned mainly by the stainless steel powder flow rate and the deposition time. The flow rate of the graphite powder has been varied between 6 and 35 vol%, relatively to that of steel and practically it does not modify the thickness of the deposit. For a stainless steel powder flow rate of 80 g/min, the thickness of the coating reaches 450 μm, for a deposition time of 6 min (see. figure 3-A). If the flow rate is only 12 g/min, a deposition time of 20 min is necessary to achieve the same thickness of deposit. The observation with SEM of a polished cross section shows a lamellar structure of the deposit (see. figure 3-A) with inclusions (stain areas) randomly distributed in all the analyzed section (see. figure 3-B). The EDS analysis of these inclusions (see. figure 4) shows prominently the Kα ray of the graphite. But the small diameter of these inclusions (< 12 μm) makes the analysis difficult and the XRD also exhibits iron, chromium and nickel, element lines coming from the stainless steel metallic matrix.

Figure 17: SEM picture of the composite deposit of stainless steel - graphite
For a preheating temperature of the substrate surface of 200 °C, the variation of the flow rate of stainless steel and that of graphite injection in the tested ranges does not modify in an appreciable way the hardness of the deposit which remains constant and equal to 180-210 HV2. This composite deposit on an aluminum alloy substrate surface, permits to double the hardness of the latter (see. table 2).
**Figure 18**: *EDS of the graphite inclusions in a composite coating of stainless steel-graphite*

<table>
<thead>
<tr>
<th>Materials</th>
<th>Hardness HV₂</th>
<th>Dispersion</th>
</tr>
</thead>
<tbody>
<tr>
<td>stainless steel 316L coating</td>
<td>186</td>
<td>18</td>
</tr>
<tr>
<td>stainless steel 316L coating+graphite</td>
<td>190</td>
<td>30</td>
</tr>
<tr>
<td>aluminum alloy AU4G</td>
<td>100</td>
<td>4</td>
</tr>
<tr>
<td>steel 304 L bulk</td>
<td>315</td>
<td>20</td>
</tr>
<tr>
<td>steel 100C6 balk</td>
<td>500</td>
<td>23</td>
</tr>
</tbody>
</table>

**Table 4**: microhardness values of different materials

**Tribological Behavior of deposits**

Tribology tests are achieved on the composite coatings of stainless steel - graphite. These deposits have a roughness (Ra) of about 1.7 μm after grinding with millstone corundum. The evolution of the friction coefficient (f) of the composite deposits of stainless steel - graphite are compared to those of a pure stainless steel deposits (see, figure 5). For all conditions of achieved tribology tests (3 loads for 3 different speeds) the friction coefficient of composite coatings is lower than that of steel coatings. According to conditions and duration time of the tests, this reduction is in the order of 30 to 50% (see, table 3). The analysis of the material collected on the friction track, for the test conditions indicated in figure 5-A and the phase of low friction obtained at the beginning of the test, shows that the 3<sup>rd</sup> body is constituted of graphite compact lamellae in contact with lift zones (see, figure 6). In this case the reduction of the friction coefficient is due to the formation of graphite lamellae that permits the adaptation of the speeds at the level of the third body by shearing of the graphite lamellae [4], [5]. In this phase, graphite permits the elflubrication of the contacts [6], [7]. Its action is time limited and also related to the volumetric fraction of graphite within the metallic matrix. The formation of graphite lamellae is only possible when the topography of surfaces in contact exhibit a very weak roughness and a good planarity, which is not really the case in the described experiments. During the test the elimination of the graphite films can occur especially (if the quantity of graphite in the composite is weak) resulting in interactions between the metal of the pin and that of the matrix. Pulling out that follows deteriorates the contact surfaces and does not permit anymore the formation of transfer graphite films (see, figure 7-A). This situation results in a fast transition of the friction evolution shifting from low to high friction (see, figure 5-A). In this second stage the 3<sup>rd</sup> body is a mixture of metallic and graphite particles.

If the geometry of the contact at the beginning of some test promotes the metallic material adhesion, the low friction phase doesn't appear any more (see, figure 5-B). Nevertheless, the value of the friction coefficient of the composite deposit is lower by 50% relatively to that obtained with the deposit of steel. The graphite particles present in the 3<sup>rd</sup> body particles modify the tribological behavior of the contact. The geometric shortcomings of the pin and the disk promotes localized contacts (see, figure 7-B), resulting in local contact pressures much higher than the theoretical one.

![Graphs A and B showing friction behavior](image_url)
Table 5: *Mean values of the friction coefficient for different test conditions*

<table>
<thead>
<tr>
<th>Friction coefficient</th>
<th>Steel</th>
<th>Steel + graphite</th>
</tr>
</thead>
<tbody>
<tr>
<td>11.7 N 0.1 m/s</td>
<td>0.59</td>
<td>0.40</td>
</tr>
<tr>
<td>18 N 0.1 m/s</td>
<td>0.61</td>
<td>0.40</td>
</tr>
<tr>
<td>28 N 0.5 m/s</td>
<td>0.6</td>
<td>0.45</td>
</tr>
<tr>
<td>11.7 N 0.5 m/s</td>
<td>0.46</td>
<td>0.36</td>
</tr>
<tr>
<td>18 N 0.5 m/s</td>
<td>0.68</td>
<td>0.45</td>
</tr>
<tr>
<td>28 N 0.5 m/s</td>
<td>0.59</td>
<td>0.36</td>
</tr>
</tbody>
</table>

Figure 20: *SEM picture of the friction track*

Figure 21: A: *Track of friction*, B: *rubbed pin*

4. Conclusion

The developed process of co-spraying (plasma molten and solid particles), permits to achieve composite coatings made of graphite particles randomly distributed in a metallic matrix of stainless steel. Such deposits present a lamellar structure and have a thickness in the order of 450 μm for a deposition time of 6 minutes with a stainless steel powder flow rate of 80 g/min. These composite coatings allow to double the hardness of the aluminum alloy substrates on which they are sprayed (the hardness of the composite deposit is in the order of 190 HV2 [±30]). Tribology tests achieved on these deposits show the interest of graphite as solid lubricant, especially when films of transfer can form. Nevertheless, two problems are yet to be solved, on the one hand the high Ra of deposit surfaces, and on the other hand the optimization of the quantity of graphite contained in the deposit.
references
[17]. P. Fauchais, A. Vardelle, B. Dussoubs, Quo Vadis Thermal Sprays, Journal of
Thermal Spray Technology. 10 (2001), (1).
[18]. A Vardelle, C. Moreau, P. Fauchais, The dynamics of deposit formation in thermal-
[19]. P. Reynaud, Y. Berthier, Tribologie Spatiale, Journée du CETIM, Frottement sous
INSA Lyon.
437-452 p.
[22]. P. Fournier, Frottement sec des matériaux monolithiques, composites et réfractaires :