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Microstructure and mechanical properties of yttria partially stabilized zirconia coatings deposited by laser-assisted air plasma spraying

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Abstract

In laser-assisted air plasma spraying (LAAPS), a laser beam interacts simultaneously with the coating surface during plasma spraying in order to increase the coating surface temperature and possibly remelt the coating surface. As a result, the microstructure is partially densified and macrocracks can be inhibited. In this paper, LAAPS was performed to improve the mechanical properties of ZrO$_2$ - 8 wt.% Y$_2$O$_3$ coatings. The coating microstructure was characterized by optical microscopy, SEM and X-ray diffraction. The mechanical characterization was done by hardness measurements, thermal shock resistance and erosive wear tests. Results showed that laser assistance may induce: (1) a decrease of the amount of microcracks and interlamellar pores, (2) a columnar dendritic structure in the coating splats and (3) an improvement of the thermal shock resistance. LAAPS did not affect the hardness or the erosive wear resistance of yttria partially stabilized zirconia coatings.

Introduction

Thermal barrier coatings (TBCs) produced by air plasma spraying (APS) are widely used in aero- and land-based gas turbines and diesel engines. Their benefits result from their ability to sustain high thermal gradients and reduce the substrate surface temperature improving the component lifetime (1, 2). However, TBCs produced by APS contain connected porosities and crack networks due to the volume shrinkage during spraying process and residual tensile stresses. The TBC properties such as high temperature corrosion resistance, mechanical strength and erosion resistance can be, thereby, greatly reduced. To improve these properties, various methods have been proposed such as post-laser irradiation and seal sintering with liquid alloys (2-7). Laser irradiation is an efficient method for densifying the microstructure of ceramic coatings. Nevertheless, post-laser irradiation treatment causes macrocracks due to the rapid cooling and the excessive thermal contraction of the densified layers (1, 3-5, 7-9). On the other hand, laser-assisted air plasma spraying (LAAPS) consists of APS with the simultaneous use of laser beam irradiation. This method has been used for deposition zirconia coatings with nickel-based bond coat (1-4, 8-12). This enables control of the coating microstructure in a single process: melted powders are sprayed from a plasma gun and local heat control of the sample surface is performed by laser irradiation. According to recent studies, LAAPS can prevent the formation of cracks while maintaining an appropriately densified microstructure (1-4, 8, 9, 13).

In this study, zirconia coatings (ZrO$_2$ - 8 wt.% Y$_2$O$_3$) were done by YAG laser-assisted air plasma spraying. Mechanical properties, such as hardness, thermal shock resistance and erosive wear resistance, were evaluated in relation to the microstructure. These properties were compared with those of APS coatings without laser heating.

Experimental set-up

Figure 1 shows the experimental set-up of LAAPS. The laser beam was directed at the spraying point on the substrate surface. The laser head was attached to the plasma gun and was scanned with it at a transverse speed of 6 m/s by a 6-axis robot. The particle stream was almost normal to the surface sample, while the angle between the laser beam and the plasma spray was set at 45°.
LAAPS was carried out using a 2-kW CW Nd:YAG laser at 1.064-μm wavelength. The beam was guided by a 1-mm diameter optical fiber. A lens with a 200-mm focal length was used to focus the laser beam. The laser power was adjusted to 2000 W on the workpiece surface. The laser irradiation density was adjusted by the defocus distance of the laser beam irradiating the specimen surface, whereas the laser power was constant. The relationship between defocus distance, spot diameter of the laser beam and laser irradiation density is given in Table 1. Distribution of the laser beam evolved from a top hat profile at the focus point to a gaussian profile at a defocused position (3).

Table 1. Relationship between defocus distance, spot diameter of the laser beam and laser irradiation density

<table>
<thead>
<tr>
<th>Defocus distance (mm)</th>
<th>Spot diameter (μm)</th>
<th>Power density (W·mm⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>4</td>
<td>144</td>
</tr>
<tr>
<td>31</td>
<td>6.2</td>
<td>66</td>
</tr>
<tr>
<td>40</td>
<td>8</td>
<td>40</td>
</tr>
<tr>
<td>50</td>
<td>10</td>
<td>25</td>
</tr>
<tr>
<td>60</td>
<td>12</td>
<td>18</td>
</tr>
<tr>
<td>70</td>
<td>14</td>
<td>13</td>
</tr>
</tbody>
</table>

The 500 μm thick ZrO₂ – 8 wt.% Y₂O₃ coatings (Metco 204NS powder) and APS Ni-Cr-Al-Y bond coats (Praxair Ni-164-2 powder, thickness of 150 μm) were sprayed on low carbon steel substrates (length: 76.2 mm, width: 25.4 mm, thickness: 12.7 mm). LAAPS was not used during the bond coat deposition. Substrates were grit-blasted before spraying. The spraying parameters are listed in Table 2. During spraying, a cooling system (air jets) was applied to reduce coating temperature, which was monitored by using an optical pyrometer. The maximum coating temperature during spraying did not exceed 170°C.

Table 2. Spray parameters used with the Sulzer-Metco F3 APS torch

<table>
<thead>
<tr>
<th>Parameter</th>
<th>ZrO₂ – 8 wt.% Y₂O₃</th>
<th>Ni-Cr-Al-Y</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary gas (Ar) flow (l.min⁻¹)</td>
<td>35</td>
<td>50</td>
</tr>
<tr>
<td>Secondary gas (H₂) flow (l.min⁻¹)</td>
<td>11</td>
<td>23.6</td>
</tr>
<tr>
<td>Current (A)</td>
<td>550</td>
<td>700</td>
</tr>
<tr>
<td>Voltage (V)</td>
<td>61.8</td>
<td>35.2</td>
</tr>
<tr>
<td>Power (kW)</td>
<td>34</td>
<td>25</td>
</tr>
<tr>
<td>Carrier gas (Ar) flow (l.min⁻¹)</td>
<td>3</td>
<td>6.5</td>
</tr>
<tr>
<td>Powder feed rate (g.min⁻¹)</td>
<td>25</td>
<td>30</td>
</tr>
<tr>
<td>Spray distance (mm)</td>
<td>120</td>
<td>63</td>
</tr>
</tbody>
</table>

The cross section of the coatings was polished to a mirror finish (diamond with grain size of 1 μm). The coating microstructures were evaluated via scanning electron microscopy (SEM). For microstructure characterization, SEM images were digitized and analyzed using an image analysis software. Globular pores, unmelted particles and cracks were isolated by implementing several filtering protocols. This method measured the size difference between pores-unmelted particles (some micrometers of diameter) and cracks (some tenth micrometers long) according to Antou et al. (12). Pores and unmelted particles were then analyzed in terms of relative surface and cracks in terms of cumulated length and orientation. Cracks were classified as perpendicular (within ±10°) or parallel (within ±10°) to the substrate surface. Ten images selected randomly across the cross sections were analysed on each sample and the results were subsequently averaged. Samples were also fractured in order to observe under SEM the failure facieses and the columnar structure of the lamellae. Phases were identified by X-ray diffraction using a Bruker AXS D8-Discover diffractometer (CuKα radiation). Vickers micro-hardness measurements were performed in the cross section with a 3-N load.

The slurry-erosion test was carried out using fully automated test rig. Alumina particles (150-μm diameter) were employed as erodent. The liquid part of the slurry was neutral pH deionised water and the erodent concentration was 0.66 wt.%. The slurry jet had a constant flow rate of 1 l/min (6.6 g/min of alumina particles) and a constant velocity of 20 m/s. The slurry-erosion resistance was evaluated at an impinging angle of 90° during five minutes for each sample. Three samples were tested for each coating type produced in this study. The volume of the material abraded away during the slurry-erosion test was measured via optical profilometry.

Thermal shock resistance of the treated coatings was investigated by cyclic isothermal shock test. Samples were 25.4 x 25.4 x 5 mm³, made of the same low-carbon steel and sprayed following the same protocols (APS and LAAPS). The coated specimens were kept in a furnace for 1 hour at a temperature of 1273 K and then were dropped into icy water (273 K). After each thermal cycle, the coating was inspected by optical microscopy and measured by image analysis. Thermal shock resistance was evaluated with the number of shocks inducing a 5% spallation of the coated surface.

Fig. 2 shows a typical cross section of zirconia coatings obtained by APS (Fig. 2a) and LAAPS (Fig. 2b). With both processes, the microstructure shows lamellar splat morphology with interlamellar pores and microcracks, typical of plasma sprayed coatings. No large microstructure difference is noted between the coatings produced by APS and LAAPS. Indeed, no remelting of the coatings by the laser beam is observed, in contrast with LAAPS of alumina-titania coatings (13). This behaviour can be explain by the relative high reflectance of zirconia coatings at the Nd:YAG wavelength (1.064-μm). This reflectance is about 0.8 (16).
As shown in Fig. 3, a finer observation by image analysis reveals an evolution of the quantity of the microcracks and interlamellar pores. This figure shows the cumulated length of microcracks and interlamellar pores evaluated on the coatings obtained by APS and LAAPS. The cumulated length of cracks and pores parallel to the substrate (Fig. 3.a) on APS coatings is about 90.5 mm. LAAPS use reduces this value, up to a minimum value of 71.1 mm with a laser irradiation density of 25 W.mm\(^{-2}\). This corresponds to a 21\% reduction of the cumulated length of parallel microcracks and interlamellar pores. As shown in the Fig. 3.a, this cumulated length decreases with an increase of the laser irradiation density from 15 to 25 W.mm\(^{-2}\). This improvement can result from the increase of the wettability of the sprayed particles. Indeed, the laser beam increases the sample surface temperature, which results in an increase of the wettability of the sprayed particles and a decrease of the interlamellar pores. Furthermore, the laser irradiation can partially remelt the upper layers of the zirconia coatings during the process. This can also cause the reduction of microcracks. On the other hand, the cumulated length increases between 25 and 102 W.mm\(^{-2}\) and, at 102 W.mm\(^{-2}\), it becomes larger than that of APS coatings. This increase can be explained by the higher laser irradiation density, leading to an excessive melting of the coating. A large contraction of the melted part in an extremely short time occurs in the process of rapid cooling after the laser beam irradiation. This causes the appearance of cracks parallel and normal to the sample surface (13). This phenomenon gets close to the laser remelting of APS coatings studied by many authors in the case of ceramics spraying (3, 4, 8).

Similar phenomena are observed on cracks and interlamellar pores perpendicular to the substrate (Fig. 3.b). In this case, the lower value is obtained with a laser irradiation density of 31 W.mm\(^{-2}\). There is no significant difference in the relative surface of the globular pores and unmelted particles between APS and LAAPS coatings. The relative surface of globular pores is around 2\% and remained constant as function of laser irradiation density. In the case of the unmelted particles, the relative surface is 6.4\% into APS coatings and evolves randomly between 3.7 and 8.8\% into LAAPS coatings.

**Fig. 2. Cross-sectional SEM micrographs of: a) an APS coating and b), c), d) LAAPS coatings at 25 W.mm\(^{-2}\).**

**Fig. 3. Cumulated length of microcracks interlamellar pores in APS and LAAPS coatings as function of the laser irradiation density: a) microcracks parallel to the substrate, b) microcracks perpendicular to the substrate. The cumulated length of APS coatings and its standard deviation are shown respectively by the dark and the dash lines.**
The typical columnar dendritic structure is clearly seen in the individual splats (Fig. 4). The columnar grain diameter is about 1 μm, as shown in Fig. 4b. The grain orientation is typical of the directed solidification and results from the positive thermal gradient generated during particle cooling. This columnar growth is located in individual splats only. There is no evidence of grain grow occurring through several splats even with laser irradiation (Fig. 4a). Nevertheless, this phenomenon is reported by some authors in laser remelting of APS zirconia coatings (3, 4, 8).

As shown in Fig. 5, XRD phase analysis shows that the APS and LAAPS coatings predominantly consist of the t'-ZrO₂ phase. Regarding the relative height and number of diffraction picks, no significant difference is observed between APS and LAAPS coatings.

Fig. 4. Failure facies of LAAPS coating at 66 W.mm⁻².

As shown in Fig. 5, XRD phase analysis shows that the APS and LAAPS coatings predominantly consist of the t'-ZrO₂ phase. Regarding the relative height and number of diffraction picks, no significant difference is observed between APS and LAAPS coatings. The large scattering and standard deviation does not highlight the influence of laser irradiation on the hardness properties. Other studies on ceramic coatings, zirconia (3, 4) and alumina (13), show a hardness increase with the increase of the laser irradiation density and the decrease of the defocus distance. In some cases, authors indicate a linear correlation between hardness and laser irradiation density (13).

Fig. 5. XRD patterns of zirconia coatings obtained by a) LAAPS with a laser irradiation density of 44 W.mm⁻², b) APS without laser.

Fig. 6 shows the hardness in the cross section of APS (dark line) and LAAPS (black plots) coatings with laser irradiation density evolving from 15 to 144 W.mm⁻². Hardness values of LAAPS coatings are roughly similar than one of the APS coating (776 HVₐₙ). The large scattering and standard deviation does not highlight the influence of laser irradiation on the hardness properties. Other studies on ceramic coatings, zirconia (3, 4) and alumina (13), show a hardness increase with the increase of the laser irradiation density and the decrease of the defocus distance. In some cases, authors indicate a linear correlation between hardness and laser irradiation density (13).

Fig. 6. Hardness evolution of APS and LAAPS coatings as function of the laser irradiation density. The hardness of APS coatings and its standard deviation are shown respectively by the dark and the dash lines.

As reported in the Fig. 7, LAAPS does not improve the erosive wear resistance in the laser density range of 15-144 W.mm⁻². The best result is achieved for a laser irradiation density of 66 W.mm⁻², leading to a wear rate of 22.4 mm³.kg⁻¹. This value is similar to the wear rate of APS coatings (22.3 mm³.kg⁻¹), taking account the scattering of measurements. The
reduction of the microcracks and interlamellar pores observed previously does not affect the erosive wear resistance. When the laser irradiation density is higher than 66 W mm\(^{-2}\), the erosive wear rate raises. This phenomenon can be explained by the appearance of cracks parallel and normal to the sample surface (see Fig. 3) due to a higher laser irradiation density. This causes the decrease of the strengthening of the coatings. On the other hand, when the laser irradiation density is lower than 66 W mm\(^{-2}\), the wear resistance decreases also. In this case, the laser irradiation density can be not enough to induce a significant reduction of the microcracks and interlamellar pores. Nevertheless, there is probably a significant increase in the temperature of the coating under the laser spot. It has been demonstrated that thermal spray ceramic coatings deposited on hot coating/substrates tend to exhibit significant more cracking during mechanical solicitation when compared to the same coating deposited on cooler conditions. This is caused by to the presence of high tensile stresses in the coating (14). Therefore, the low performance of the coatings produced at low laser irradiation density is probably explained by their tendency of severe cracking during mechanical loads.

Fig. 7. Relationship between laser irradiation density and erosive wear rate for LAAPS coatings. The erosive wear rate of APS coatings and its standard deviation are shown respectively by the dark and the dash lines.

Fig. 8 shows the thermal shock resistance as function of the laser irradiation density. This resistance is evaluated by the number of isothermal shock cycles leading to a 5% spallation of the coated surface. Each cycle consists of 1 hour at 1273 K following by a rapid quenching into icy water at 273 K. As shown in Fig. 8, APS coating can take 24 thermal cycles before the 5% spallation of its surface. The LAAPS process increases this value up to a maximum number of 40 cycles with a laser irradiation density of 102 W mm\(^{-2}\). The thermal shock resistance is then improved nearly by a factor 2. Moreover, LAAPS coatings with laser irradiation densities between 21 and 102 W mm\(^{-2}\) exhibited better resistance to isothermal shocks than APS coatings. Thermal shock is a complex phenomenon where many parameters take place. We propose here one explanation; nevertheless, future investigation must be performed in order to confirm it. This improvement can be explained by the reduction of microcracks and interlamellar pores for these densities, as shown in Fig. 3. Microcracks and interlamellar pores parallel to the substrate promote the spallation mechanism, while cracks perpendicular to the substrate increases the permeability of zirconia coatings and favor the growth of the TGO (thermally grown oxide). Indeed, the main degradation mechanism during isothermal shocks is the growth of the TGO (15). This mechanism proceeds as follow: (i) cracks are induced into the coating due the thermal stresses, (ii) the cracks propagate around imperfections induced in the TGO, (iii) TGO coalesces and becomes large enough to cause large buckling and coating spallation. In consequence, an increase of interlamellar pores and cracks perpendicular to the substrate increases the coating permeability, limits the TGO growth and increases the thermal shock resistance.

![Fig. 8](image-url)

**Fig. 8.** Number of isothermal shocks inducing a 5% spallation of the coated surface as function of the laser irradiation density. The APS coating measurement is illustrated by laser irradiation density of 0 W mm\(^{-2}\).

**Conclusions**

(i) This study concerns the deposition of zirconia coatings by laser-assisted air plasma spraying using a 2 kW Nd:YAG laser. The possibilities of this hybrid process are investigated, especially the laser irradiation density effects on the microstructure and the mechanical properties of zirconia coatings.

(ii) Microcracks and interlamellar pores are reduced by LAAPS. A reduction of 21% are measured between coatings obtained via APS and LAAPS (laser irradiation density of 25 W mm\(^{-2}\)). A columnar dendritic structure in the coating splats is observed.

(iii) LAAPS does not affect the hardness or the erosive wear resistance of yttria partially stabilized zirconia coatings.

(iv) LAAPS leads to an improvement of the thermal shock resistance (maximum improvement of 90%).
Acknowledgements

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References