# **Role of the Laser Surface Preparation on the Adhesion of Ni-5%Al Coatings Deposited Using the PROTAL Process**

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**The thermal spraying assisted by laser (PROTAL) process combines a laser surface preparation followed by a thermal spray process. This type of surface preparation avoids some drawbacks of the degreasing and sandblasting processes. Previous studies showed that the adhesion of the deposits obtained with the help of the PROTAL process is similar to that achieved by traditional surface preparation. To obtain better insight into the effects of the laser treatment, a Ni-5%Al coating was plasma sprayed using the PROTAL process under different surface conditions. The morphology of the impinging splats and adhesion of the deposits were examined. Removal of contaminants, adsorbates, and oxides at substrate surface is confirmed. The role of the laser irradiation on the coating adhesion is discussed.**

**Keywords** adhesion/cohesion, Ni-5%Al coatings, PROTAL

## **1. Introduction**

The quality of thermal spray coatings depends on a precise control of prespray surface preparation (Ref 1). When grit blasting is usually applied, the entrapment of grit residues should be avoided. Some new prespray methods such as water-jet roughening (Ref 2, 3) or dry-ice-blasting (Ref 4, 5) seem promising to avoid this drawback. Another interesting technique of thermal spraying assisted by laser (PROTAL), which associates a pulsed-laser surface treatment to the spraying operation, was proved to be an efficient technique a couple of years ago (Ref 6-10). The method has economical and technical advantages and therefore have drawn attention from industry.

Past research on the PROTAL process have demonstrated the benefits of the laser cleaning (Ref 6, 7, 10), showing that deposits could be produced directly on raw workpieces by PROTAL, whereas a poor adhesion occurred on untreated substrates. Most of the time, the sprayed coatings elaborated by the PROTAL process exhibit equivalent bond strength with those obtained by grit-blasting (Ref 8, 10, 11). However, a recent study (Ref 12) displayed the opposite result for a Ni-5%Al coating. Although an interdiffusion phenomenon arose as a consequence of the laser irradiation, the statistical results exhibited a negative effect of the laser treatment on the deposit adhesion.

This work was devoted to the study of a Ni-5%Al coating

sprayed by the PROTAL process on TA6V substrate under different conditions. The morphology of the impinging splats on the laser treated surface and the deposit adhesion were especially investigated. It is worth noting that the microstructure characteristics and adhesion mechanisms of a composite powder of nickel and aluminum (Ni-Al) coatings have already been intensively and extensively investigated by many researchers [e.g., in studies (Ref 12-14)]. The current study aims at gaining insight into the role of laser irradiation on the surface preparation and coating elaboration.

## **2. Experimental Procedures**

#### *2.1 Experimental Setup*

The laser source used in the PROTAL process is a Qswitched Nd-yttrium aluminum garnet (YAG) laser from Quantel (France) (laserblast 1000), which operates at the wavelength of 1.064 µm with an average power output of 40 W. The maximum pulse frequency is 120 Hz with a pulse duration full width at half maximum (FWHM) of 10 ns. The laser beam is transferred through  $SiO<sub>2</sub>$  optical fibers and a specific optical arrangement. The final laser beam toward the irradiated substrate has a rectangular shape that allows a homogeneous irradiation, as presented in Fig. 1.

Figure 2(a) schematically illustrates the whole integrated PROTAL system with two superposed laser heads. The laser heads were maintained near the spray torch in front of the sample holder, whose experimental configuration is displayed in Fig. 2(b). Such arrangement (overlaying of the two laser heads) places two rectangular laser beam spots in superposition thus ensuring a sufficiently large area to receive the sprayed particles simultaneously, as shown in Fig. 3. By this way, all sprayed particules can impinge on a laser cleaned surface. During spraying, specimens were fixed on a rotating sample holder. The incidence angle of the laser beams as well as the torch axis was kept constant at an angle of 20° from the normal to the substrate's surface. A two-color pyrometer was fixed at the rear to monitor the

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Fig. 1 Impacts of a single laser shot of superposed two laser heads on photosensitive paper



Fig. 2 Illustration of the integrated system for the PROTAL process (case of atmospheric plasma spraying): (a) schematic view of the whole system and (b) experimental configuration of the laser heads and plasma torch



Fig. 3 Schematic configuration of the laser treated zone and of the location of the sprayed particles jet during the PROTAL process

whole thermal history during coating elaboration. More details about this system can be found in Ref 15.

#### *2.2 Materials*

The investigated substrate was a titanium based alloy TA6V (Ti: 90 wt.%, Al: 6 wt.%, V: 4 wt.%) that is widely used as titanium alloy. Because of the excellent resistance on hightemperature and corrosion, it is frequently used in aerospace industry (Ref 16).

The specimens (25 mm in diameter and 10 mm in thickness) were prepared in the following way: (1) a machined surface finish (mean roughness (Ra) =  $0.25$  to  $0.4 \mu m$ ) was used for thick coatings elaboration; (2) a mirror polished surface finish (Ra < 0.01 µm) was used for surface morphology observation, for capturing the splats as well as preparing thin layers to investigate the elements distribution at the interface.

A commercially available powder, Amdry 956 (Ni-5wt.%Al) with a nominal particle size ranging from  $45$  to  $90 \mu m$ , was used for coating spraying. A fraction with a narrower particle size distribution (63 to 80  $\mu$ m) was sieved out for collecting single splats.

## *2.3 Coating Processing*

Table 1 presents the plasma spraying parameters. To treat the whole surface of the laser without any overlapping, the relative scanning velocity (480 mm/s) was calculated taking into account the size of the laser impact  $(4 \times 16 \text{ mm}^2)$  and its repetition rate (120 Hz; Fig. 3). This way, each point of the surface receives one single laser shot. Only the first pass was concerned by the PROTAL system to treat the substrate surface, the laser being turned off for the following coating build-up.

The 290 to 310  $\mu$ m thick coatings were realized for adhesion tests. Specimens that were prepared using conventional degreasing and grit-blasting were also sprayed under the same condi-



**Fig. 4** SEM observation of irradiated TA6V surface at 2250 mJ/cm2 with (a) 1 pulse and (b) 5 pulses





tions for comparison. A thin 50 µm layer was also sprayed onto a mirror finish substrate for chemical analysis. Moreover, a pass sprayed using low powder feed rate was made for collecting the splats onto mirror finish specimens.

## *2.4 Deposit Characterization*

Morphological observations and microstructural characterizations of the surface and splats were performed using optical microscope and scanning electron microscopy (SEM). Glow



 $(a)$ 



**Fig. 5** SEM observation after chemical etching (a) on initial surface and (b) laser treated surface

discharge spectroscopy (GDS) was used to investigate the distribution of the elements at the interface.

The adhesion of the coating to the substrate was determined according to the standard tensile test (Ref 17). A well-known bonding adhesive (Cytec FM100, Teaneck, NJ) was used for the test (Ref 18).

# **3. Results and Discussion**

## *3.1 Laser-Induced Surface Modification*

A systematic study of the laser irradiation effects on TA6V surfaces and their mechanisms was presented with more details elsewhere (Ref 19), so only a brief description of phenomena will be introduced here. In topographical view, there are generally two opposite aspects that govern the laser irradiation effects on TA6V surfaces: surface roughening that is related to the formation of dispersed craters (Fig. 4a), and surface smoothing (Fig. 4b) that is related to the development of a passive film (Fig. 5). The craters were caused by the local laser ablation at surface defects (asperities, inclusions, etc.) due to their higher sensitivity to the laser irradiation than that of the bulk matrix. Concerning the development of the passive film, it seems to be the result of the rapid melting and cooling of the surface or due to an oxidation of a thin surface layer by increasing the temperature. Consequently, this superficial layer seems to present the characteristics of amorphous titanium or some form of titanium oxide. The results of surface roughness measurements confirmed this double effect (Fig. 6). With the increase in the laser energy density, the craters tend to spread over the whole surface thus resulting in a drastic increase of surface roughness. Increasing the number of laser shots seems to accentuate the superficial film thickness  $(1 \mu m)$  and shield the early formed craters. Consequently, the average roughness decreases with the increase in the number of laser shots.

## *3.2 Influence of the Laser Parameters on Splat Flattening*

It is obvious that the flattening of sprayed droplets plays a crucial role in the quality of sprayed coatings. The mechanism of splat formation has been intensively studed in the past decades (Ref 20, 21). In most cases, the pancake form is quite perferable because such a form of splat is correlated to an improved splat/ substrate contact (Ref 21, 22).

Figure 7 illustrates the representative morphology of Ni-Al splats impinging onto the boundary between a laser treated area and a non-treated area. Significant differences in the splats morphology can be observed after the laser treatment. The particles that impact the polished TA6V surface display a flower shape, and those that impact on the laser treated surface are almost in a pancake shape. Moreover, with an increase in the laser energy, the morphology of the splats improved progressively (more liable to a pancake shape), as shown in Fig. 8. On the untreated surface, a majority of the molten liquid is thrown away at impact. Then, only a small core adheres on the surface with a surrounding radial ring (Fig. 8a and 9a). On the surface treated at a relative low energy density (1000 mJ/cm<sup>2</sup>), although numerous fingers still exist at the periphery, the flattened splats seem to flow completely before its final solidification (Fig. 8b and 9b). Raising the laser energy up to 1500 mJ/  $\text{cm}^2$  or higher leads to suppressing the splashing phenomenon and then the splat pattern is changed from finger shaped to almost a disk-shape (Fig. 8c and d, 9c).

In general, the transition phenomenon in the splat morphology is associated to the surface temperature (Ref 23-25). A drastic change from a finger pattern to a perfect disk shape (pancake) occurs when a certain temperature is achieved (Ref 24, 25). Li initially suggested that splash is dominated by the evaporation of entrapped absorbates/condensates on the surface (Ref 26, 27). More credible evidence was given by Jiang et al. through a series of experiments that were performed in a controlled atmosphere environment (Ref 28). More recently, Escure et al. (Ref 29) indicated that there are two modes of splashing: "impact splashing" that takes place immediately at the impact and "flattening splash," which occurs in the later stage of flattening. The latter case was also noticed by Li et al. (Ref 30) in the case of high energy splats where numerous fingers still arise at the periphery even if the substrate has been preheated upon the "transition temperature." The reason proposed is that the flattened underlayer (solidification may have occurred) could not retain the spreading of excessive impelled upper liquid. Consequently, it can be inferred that splashing would be less severe if the wetting



**Fig. 6** Evolution of the average surface roughness  $(R<sub>a</sub>)$  versus the laser energy density and number of laser shots



**Fig. 7** Morphology of Ni-Al splats sprayed on a cold TA6V surface near the boundary (represented by a dotted line) between a laser treated and untreated areas

between the particles and substrate was improved. Such hypothesis seems to be confirmed by another experimental investigation of Fukumoto et al. (Ref 31, 32) where a better wettability was shown to decrease the splashing phenomon and to promote the occurrence of disk-shaped (pancake) splats.

In the current study, the substrate was kept at room temperature (about  $25 \text{ °C}$ ) for all of the runs, which is much lower than the reported transition temperature. This means the influence of surface modification, which usually happened during preheating operations could be excluded. However, considering the lasermaterials interaction, a surface desorption may happen because the treated surface undergoes rapid heating during irradiation followed by a cooling in a very short time. The whole process is just about a dozen of nanoseconds based on temperature simulation (Ref 8). Moreover, there are only about 30 ms for the treated surface before the first impinged particles. Then, the readsorption can be neglected. In short, the laser irradiation herein seems to play a similar role to the preheating process in reducing the surface adsorbates/condensates level without increasing the temperature. Therefore "impact splashing" can be suppressed by laser irradiation (Fig. 9a and b).



Fig. 8 Morphology of Ni-Al splats on a TA6V surface treated following different parameters: (a) untreated surface, (b) 1000 mJ/cm<sup>2</sup>, (c) 1500  $m\overline{J/cm^2}$ , and (d) 2000 mJ/cm<sup>2</sup>



**Fig. 9** Three representative morphologies of Ni-Al splats on a TA6V surface: (a) "impact splashing" that occurs on an untreated surface, (b) "flattened splashing" on the laser treated surface at 1000 mJ/cm<sup>2</sup>, (c) "disc-shaped" on the laser treated surface at 1500 mJ/cm<sup>2</sup>

Moreover, the previous measurements indicated that surface energy increases monotonously when increasing the laser energy. It is probably linked to the formation of a superficial film because, as mentioned by Feng et al. (Ref. 33), titanium oxides exhibit an enhanced wettability versus titanium metal so that the flattening splash could be reduced with the increase of laser energy.

Figure 10 presents the composition profile of a thin layer (two passes) obtained from GDS analysis. All the samples have been sprayed on a mirror polished surface ( $Ra < 0.1 \mu m$ ). The influence of the interface roughness as well as the layer thickness may be ignored. The distribution of Ni and Ti can then be regarded as the sprayed layer and substrate, respectively. Accordingly, their intersection point can be considered as the de-



**Fig. 10** Distribution of Ni and Ti at the interface between the NiAl deposit and the TA6V substrate

posit-substrate interface. Obviously, it can be observed that the interface signal changed slightly towards the outer layer after laser treatment. The higher the laser energy, the more the interface is shifted towards the surface. This fact suggests that the first sprayed layer is a little thinner if a laser irradiation is used. This supports the former morphology observation that such a laser treatment favors the splat flattening behavior. The small fluctuation of the Ni concentration, which appears at the beginning, is probably due to the roughness variations of the sprayed layer.

According to Bahbou (Ref 11), the presence of oxygen is visible at the interface but in the current study the intensity of the oxygen peak was too low to be distinguished at the interface. Further investigations of interface diffusion are in progress.

## *3.3 Effect of Laser Treatment on Coating Adhesion under Different Surface Conditions*

Figure 11 shows the laser cleaning effect on the splats morphology in case of polluted surface conditions, while the adhesion of the corresponding deposit is presented in Fig. 12. Here the initial mirror-like surface is polluted by predeposited oil and carbon particles, which allows simulation of industrial surface conditions artificially. It can be seen that no adhesion occurs if the substrate is dirty. The PROTAL process, which associated the surface preparation and the spraying operation in one step, allows cleaning of the pollutions and results in an acceptable bond strength. It has to be noted that the present laser parameters were just set to efficiently remove these enhanced contaminations. Although little mechanical bond could arise from the smooth surface, the tensile strength obtained by this preparation reaches almost 80% of the one prepared with traditional sandblasting.

Preheating operations, which are more and more widely used, have a tendency to generate thick oxide on metallic substrates. The nanosecond pulsed laser can be used to remove the thick oxide layer presented on the surface (Ref 34). This laser cleaning effect was also observed in the current study where a yellowish color of preheated sample transformed to a bright white appearance. Consequently, the adhesion of the coating elaborated by PROTAL increased significantly. It should be





Fig. 11 Effect of the laser treatment on the splats collected on polluted surfaces: (a) without laser treatment, (b) treated by laser at  $2 \text{ J/cm}^2$ .



**Fig. 12** Effect of the laser treatment on the deposit adhesion under different surface conditions

pointed out that optimization of the process parameters is not yet achieved. In fact, as indicated by Bahbou et al. (Ref 11), this laser treatment may promote the interdiffusion at the interface implying a possible metallurgical bond. As a result, an improved tensile strength could be expected in optimized conditions.

# **4. Conclusions**

In this paper, experiments were performed to estimate the interplay of laser irradiation and Ni-5Al coating adhesion on TA6V substrate. The results showed that the laser irradiation leads to an improvement in the surface wettability. Consequently, it displays favorable effects on splats flattening. Although this work was primarily focused on an atmospheric plasma system (APS) of NiAl on a titanium based alloy, it can be anticipated that the basic phenomena are applicable to other spraying processes and materials.

The current study also confirmed the laser capability in removing various surface contaminations and oxide layers. The comprehensive work of optimizing the process parameters will be achieved later.

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