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Influence of Surface Laser Cleaning Combined with Substrate Preheating on the Splat Morphology

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The morphology of sprayed splats influences the coating adhesion and properties, which are determined by the spraying parameters. Many studies in this field show that the substrate surface temperature is a very relevant factor for the splat shape: the hypotheses of substrate surface wettability and contamination or adsorption layer on the surfaces are supported by the fact that the near-disk-shaped splat can be obtained by increasing the substrate temperature. In this work, a short-duration pulsed laser was used to ablate the substrate just before powder spraying. This ablation was powerful enough to eliminate the contaminants on the substrate surface and to improve the adhesion. In this study the analyses of NiAl splat morphology on the polished TA6V (Ti-6Al-4V) substrate were carried out using laser ablation with different substrate temperatures and different heating modes: the flame and another laser. Results show that the temperature at which the disk-shaped splat can be obtained decreased dramatically by laser ablation. Moreover, laser ablation combined with another laser increased the adhesion strength of the coatings.

Keywords adherence, flame preheating, laser heating, 95Ni-5Al, PROTAL®, splat, TA6V, Ti-6Al-4V

1. Introduction

Conventional surface-preparation methods consist in successively surface degreasing, protecting the areas not to be sprayed, then grit blasting. Although very largely used in the thermal spraying field, these processes present a number of limits and disadvantages (Ref 1-3). Surface degreasing is generally carried out using solvents such as carbon fluorochloride (CFC) or trichlorethylene (Ref 4). However, their use became more and more regulated considering environment problems, recycling, and operator health protection. Moreover, the grit blasting process is not easily controlled with high precision. It can cause substrate damage that is harmful to future coating performance. In fact, this process can cause a modification of the surface mechanical properties and a decrease in resistance to fatigue for ductile materials such as aluminum and titanium due to the fragility by notch effect or sand encrustation when grit blasting air pressure and incidence angle are incorrectly used. Residues can be entrapped in the substrate; the more ductile the substrate material, the higher the amount of grit particles entrapped. Finally, the very thin substrate can be easily deformed by conventional grit blasting, as well.

To avoid such disadvantages, a technique of ablation by pulsed laser (PROTAL®, for example) seems an efficient technique to substitute the traditional way (Ref 5-8). The surface cleaning and preparation by laser do not release any polluting chemical substance and do not require any waste treatment. It is a clean process, and it is very easy to choose the area that needs to be treated. It is possible to eliminate the surface contamination and to remove oxide layers by shock waves without deteriorat-

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Symbols List

PROTAL®	French acronym of thermal spraying assisted by laser (Projection Thermique Assistée par Laser) developed by the LERMPS laboratory and the IREPA Laser Institute, France
APS	Atmospheric plasma system
Image	Image analysis software of the National Institute of Health
K_{Ic}	Interfacial toughness
NiAl	Composite powder of Ni and Al
TA6V	Titanium-base alloy (Ti: bal., Al: 6 wt.%, V: 4wt.%)
Power density	Laser power delivered on a specific surface substrate, W/cm^2
Energy density	Laser energy on a specific surface substrate, J/cm^2
LERMPS	Member of the Surface Treatment Institute of Franche-Comté (“Institut des Traitements de Surface de Franche-Comté”)
τ	Degree of splashing of flattened particles

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ing the substrate (Ref 9, 10). Used just prior to deposition, the laser irradiation generates a surface condition enhancing the adhesion of the coating and limiting the recontamination of the deposited layers by condensed vapors. The surface roughness is not modified significantly by the laser treatment, and the coating adhesion is no longer the result of mechanical anchoring (Ref 5, 11).

Surface laser ablation also seems favorable to the liquid droplet spreading out onto the substrates thus representing a very good wettability of splats on the substrate, which is one of the most important basic processes during thermal spraying. The study of the mechanisms of particle impact on substrate associated with splat morphology analyses attracts much attention since all the coating properties are connected to these processes. The particle impact and solidification depend on particle properties (kinetic energy, viscosity), as well as substrate properties (temperature, thermal conductivity, surface quality) (Ref 12, 13).

Many studies were performed in this field to better understand the particle impact processes and determine the effects of the various parameters on splat morphologies and coating adhesion (Ref 14-17). More than the surface aspect, studies showed that substrate preheating is favorable to a formation of disk-shape splat that leads to a better coating. Its influence on the wetting might depend on both the oxide formation on the surface and the elimination of adsorbed gas on the surface, which increases drastically the thermal contact resistance and solidification delays (Ref 13, 18). Some studies showed that there was a transition temperature above which splats had a circular disk shape, whereas they splashed and produce irregular splats when deposited on colder substrates (Ref 13, 19). Particles impinging on a cleaned surface, the solidification rate of the splat on the high-temperature substrate is higher than that at room temperature. However, based on the instability theory, the flattening of the droplets appears less rapidly for a high-temperature substrate, which can explain also why splat morphologies change from splash to disk (Ref 20).

Many techniques such as plasma, flame, and so forth can be used to heat the surface. However, due to the large area of the jet, several negative phenomena can be noted (oxidation of the surface, distortion of the substrate, etc.). This way, the laser technology appears also as a good process to treat the surface with a very local impact. With the optical fibers, the beam can be transmitted near the surface and fixed on the robot as well as the plasma torch.

In this work, laser ablation combined with substrate preheating either by flame or by laser was used during deposition of particles and coatings on a substrate. To evaluate the influence of the laser cleaning and the preheating on the coating adhesion, the analysis of the splat morphology was carried out. When flattening and solidifying the particle binds itself to the substrate, the quality of the deposits depends strongly on the quality of this bonding. A change in the splat morphology corresponds quite well with a change in adhesion strength of the coatings. Splat morphologies were also observed to study the combination effects and then the adhesion of the coatings sprayed with this method was estimated by interfacial indentation. To validate such process, metallic particles (Ni-Al) sprayed on titanium alloy substrates were characterized.

Table 1 Standard spray parameters of Ni-Al (Amdry 956) powder

Feedstock	Nickel-aluminum powder (Amdry 956)
Primary gas flow rates, Nl/min	Ar:50 H_2:8
Spray distance, mm	160
Arc current, A	600
Spraying angle, degrees	75
Powder feed rate, g/min	
Particle impacts	2.4
Coating deposition	50
Carrier gas flow rate, Nl/min	
Particle impacts	2
Coating deposition	4
Linear speed, mm/s	30-300 (depending on the ablation laser overlapping rate)

Nl/min, normalized liter per minute

2. Experimental Conditions and Processes

2.1 Material Choice

The material chosen as a substrate for this study is a titanium-base alloy Ti-6Al-4V. The 25 mm diameter and 10 mm thick substrate was polished for splat collection and had no further preparation for the adhesion tests. The Ni/Al 95/5 powder (Amdry 956) (Sulzer-Metco AG, Wholen, Switzerland), selected as spray material, is an agglomerated Al and Ni powder with a mass proportion of 95% Ni, 5% Al. A powder size distribution ranging from 40 to 63 μm was used for splat spraying and from 20 to 63 μm for the coatings for adhesion test.

2.2 Experimental Facilities

An air plasma spraying (APS) system with a F4 gun, manufactured by Sulzer-Metco (Wholen, Switzerland), was used for this study. The particles were sprayed on the titanium substrates using standard (i.e., conventionally used) spray parameters (Table 1).

The laser used for surface ablation before spraying included in the PROTAL® process is a Quantel Q-Switched Nd-YAG laser (Quantel, Les Ulis Cedex, France), with a 1064 nm wavelength, a pulse duration close to 10 ns, and 420 mJ energy per pulse. The laser spot exhibits a rectangular shape with a uniform intensity profile obtained using a special optical system (Ref 8). In this work, an optimized energy density for cleaning Ti substrate (1250 mJ/cm^2) was used for all tests when using this laser (Ref 5). The dimensions of the ablated area on the substrate are also 10 × 5 mm^2. A frequency of 60 Hz was chosen for the tests.

An oxyacetylene flame gun Castodyne DS-8000 (Castolin SA, Courtaboeuf Cedex, France) was used to heat the substrate. This way, a relatively uniform substrate heating can be ensured and the substrate temperature measurement is then easily carried out using a monochromatic pyrometer LAND SYSTEM 4 (LAND Infrared, Dronfield, UK).

Another Nd-YAG laser source, designed by the Cheval company (Cheval, Pirey, France), was also used for the surface pre-

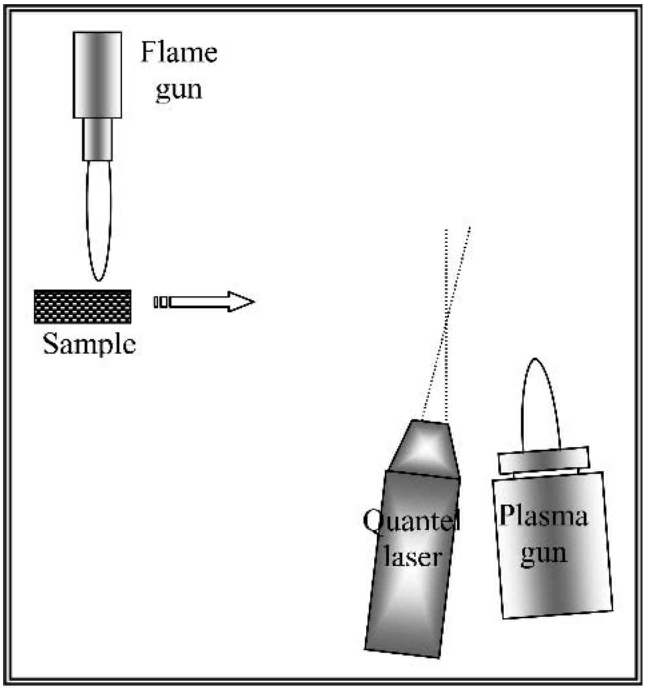

Fig. 1 Experimental setting (without Cheval laser)

heating. It is a strong power laser with a wavelength of 1064 nm and a pulse duration adjustable from 0.5 to 10 ms. With a maximum average power of 1100 W, it delivers pulses of 120 J maximum. The maximum frequency is 200 Hz. To be in accordance with the cleaning laser frequency (Quantel laser), a usual frequency of 60 Hz was also used in this work. Due to the short pulse duration (ms), the substrate temperature measurements cannot be carried out in real time by pyrometer. Then, several power density levels were tested.

2.3 Experimental Setting and Procedure

In this study, three series of experiments were carried out to reach different goals. First, the influence of the substrate temperature on splat morphologies was studied. This test defines a critical temperature beyond which the splat will have a disk shape. Second, the effect of the laser ablation on the critical temperature was tested. Finally, because the first two cases used flame preheating, the effect of the combination of the two lasers for the simultaneous ablation and preheating of the surface on splat morphologies and coating adhesion was tested.

The experimental setup used for the first experiments (case 1) is presented in Fig. 1. The plasma and flame guns are motionless while the sample is held by a robot. During the tests and to avoid an important oxidation of the surface, the sample is heated on the back with the flame. When the temperature measured by the pyrometer reaches the selected value, the robot scans immediately the sample in front of the plasma and powder jets.

For the second experiment (series 2), the Quantel laser used for the substrate ablation is fixed just beside the plasma gun. The same approach for sample displacement is used.

Finally for the third series, the Cheval laser, used for preheating of the substrate, is added on the support of plasma gun and Quantel laser. The samples are still moved by the robot.

2.4 Analysis Methods

The degree of splashing of flattened particles was used to compare splat morphologies obtained in different conditions (Ref 21, 22). This form factor is calculated based on the image analysis of splat micrographs (realized after a "wipe" test), which was carried out with NIH* software using the formula:

$$\tau = \frac{l_{\text{per}}^2}{4\pi S}$$

where τ is the degree of splashing, l_{per} is the splat periphery length, and S is the splat area. All the degrees of splashing measurements correspond to an average of 15 to 20 values.

To quantify the Ni/Al coating adhesion on a titanium alloy substrate, the interfacial indentation test was chosen (Ref 23). For this study a load of 3 kg, sufficient to generate a detectable cracking within the interface, was used. Interfacial toughness K_{Ic} is expressed according to the applied load and the length of crack propagated at the interface by:

$$K_{\text{Ic}} = \frac{P}{\pi^{1.5} \cdot \tan\psi \cdot c^{1.5}}$$

where P is the applied load (N), c is the length of crack (m), and Ψ is the half angle at the pyramid top (degrees), 68° in the case of a Vickers pyramid. The K_{Ic} value represents an average of ten measurements.

3. Results and Discussion

3.1 Influence of Flame Preheating

Figure 2 presents the evolution of the typical morphology of Ni/Al particles impacted on the substrate without laser processing versus the substrate temperature. Figure 3 shows the evolution of degree of splashing for various substrate temperatures. For preheating temperatures lower than 165 °C, the splat morphology is quite similar to that obtained at ambient temperature. This morphology is characterized by irregular forms and shows some very significant peripheral splashes. An approximation was necessary at 25 °C due to the important splashing of the matter (material seems to be ejected and a ring appeared around the particle). Then the biggest diameter was estimated even if the measure is not realistic. On the other hand, for substrate temperatures higher than 250 °C, the impacted particles are regular and have a circular shape. Moreover, a strong dependence of degree of splashing on the substrate temperature was observed as shown in Fig. 3. At 165 °C, the splats present high degrees of splashing, higher than 100. For higher temperatures, the peripheral splash is less marked and the degree of splashing is much lower. For example, the degree of splashing is lower than 40 for

*Image is from a public domain image analysis software of the United States National Institute of Health, available on the http://rsb.info.nih.gov/nih-image/ftp server.

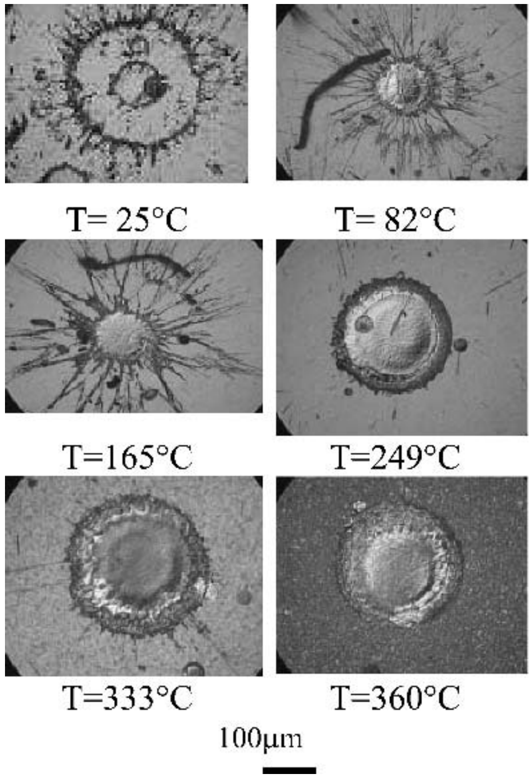

Fig. 2 Typical splat morphologies as function of the preheating substrate temperature

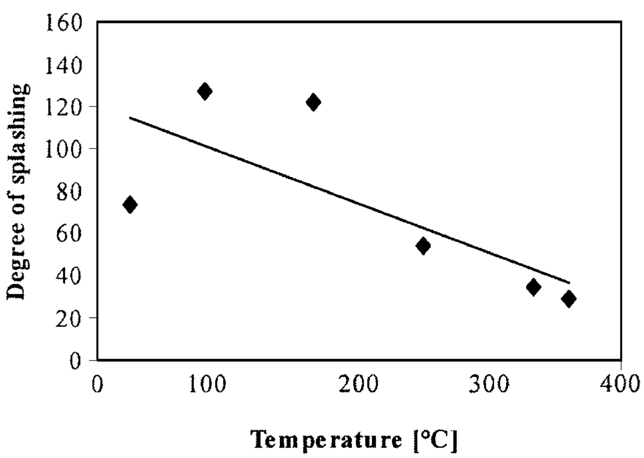

Fig. 3 Degree of splashing as function of substrate temperature (average of 15-20 values)

substrate temperature above 360 °C. Moreover, at ambient temperature, the splat center is often empty indicating that some material rebounded at impact. This could be the reason why the degree of splashing is relatively weak. The evolution of the degree of splashing and splat morphologies agrees with the literature results. Substrate preheating supports the formation of little splashed circular plates. In all cases, transition temperature was determined (specific for each materials association) above which splats had a circular disk shape, whereas they splashed and produced irregular splats when deposited on colder substrates (Ref 19). This is probably due to the vaporization of the contaminants and the absence of moisture on the surface at high temperature. The increase of the substrate temperature limits the thermal transfer to the interface particle/substrate and is also an origin of the improvement of the wettability of the molten particles (Ref 13).

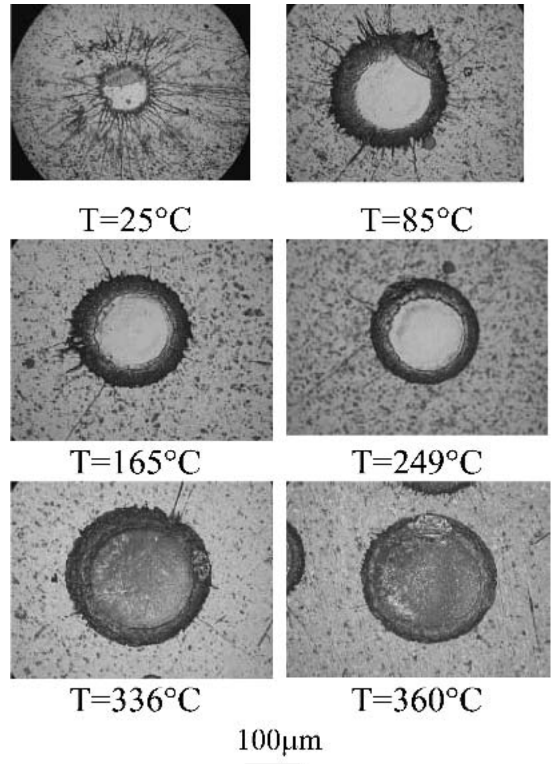

Fig. 4 Typical splat morphologies as function of the preheating substrate temperature with laser ablation

3.2 Effects of Flame Preheating and Laser Ablation

Figure 4 presents the evolution of splat morphologies for various temperatures with laser ablation. During the test, the sample scanning velocity was controlled (300 mm/s) to obtain a laser spot-overlapping rate of 1.1 (ablated area width of 5 mm and laser frequency of 60 Hz). The repetition rate of the laser impact on the surface corresponded also to a unique pulse on each point of the surface. Figure 5 shows the evolution of particle degree of splashing for various substrate temperatures. For a specific energy density of 1250 mJ/cm^2 and a laser spot-overlapping rate 1.1, when the substrate temperature was 25 °C, the splats are characterized by evident peripheral splashes but less marked than those without laser ablation. When the temperature is close to 85 °C, the splash phenomenon attenuates, and the splat form approaches a quasi-perfect disk shape. For higher temperatures, the splashes are less marked and the splats obtained also have perfect circular shape. The preheating and laser ablation combination seems favorable to lower the critical temperature of preheated substrate and the splashes length. The

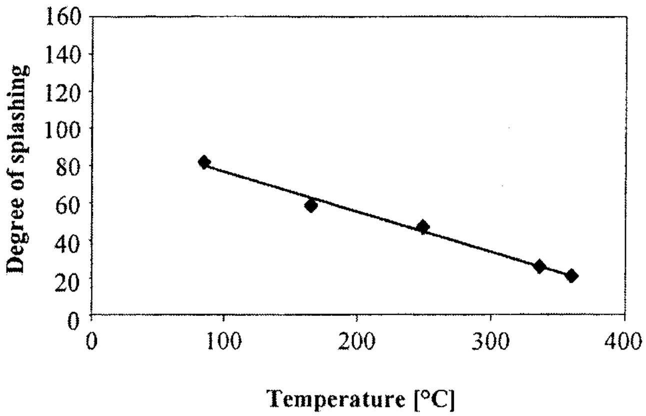

Fig. 5 Degree of splashing as function of substrate temperature with laser ablation (average of 15-20 values)

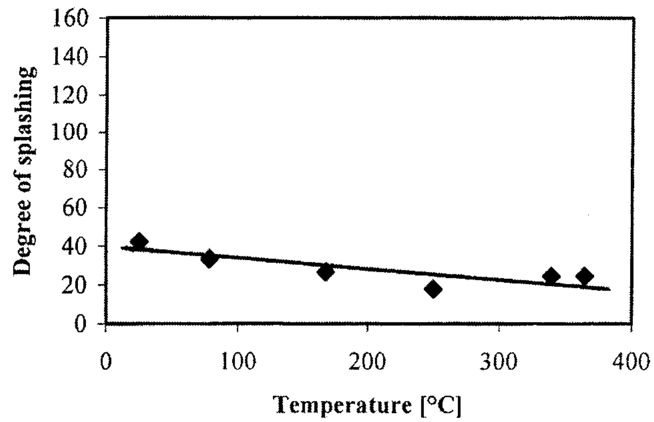

Fig. 6 Degree of splashing as function of temperature with laser ablation with an overlapping of 3.3 (average of 15-20 values)

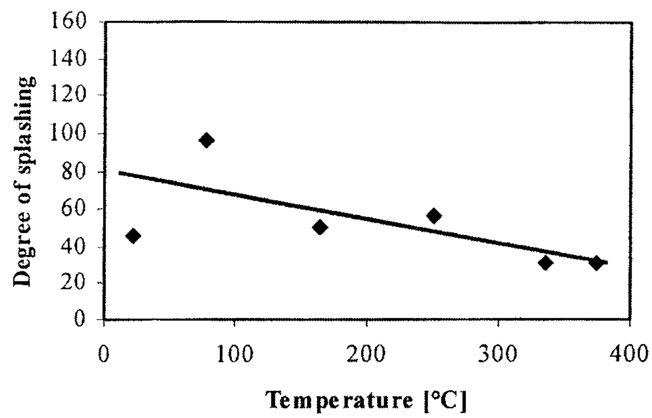

Fig. 7 Degree of splashing as function of temperature with laser ablation with an overlapping of 10 (average of 15-20 values)

degree of splashing is definitely lower than that obtained without laser ablation for all tested temperatures. The laser treatment permitting to remove the contaminants by ablation (e.g., oxides, greases, oils, etc.), the efficiency for the substrate treatment prior plasma spraying permits also to decrease the transition temperature above which the droplets retain its coherence and spread out in the form of disk. Then, particles can impinge the surface in the best conditions (oxide free, perfectly clean, etc.). In laser ablation, removal of matter from the surface results from the contribution of melting, vaporization, and also from the rapid expansion of the plasma and the corresponding shock waves (Ref 24, 25). Main key parameters that influence the ablation threshold of species are pulse duration, beam energy, and absorptivity. Therefore, for a given material, laser-matter interaction can produce various effects, depending on parameters such as beam intensity and surface properties (Ref 26-28).

Figure 6 shows the evolution of the degree of splashing with various substrate temperatures with the same experimental parameters besides a laser spot overlapping rate of 3.3. The sample scanning velocity being 100 mm/s, the repetition rate of the laser impact on the surface corresponds to an overlapping of the ablated surface (the frequency is maintained at 60 Hz and the laser affected area is always equal to 10×5 mm^2). In these conditions it appears that splash phenomenon is considerably attenuated whatever the temperature. In fact, for a substrate temperature of 25 °C, splash is less pronounced than that of the two preceding cases. However, when the laser spot overlapping continue to increase, to 10 for example, the degree of splashing increases again, as observed in Fig. 7.

From these results, it is found that the laser ablation can improve the splat form and this improvement is more remarkable if the substrate is at high temperature. In fact, that means the laser ablation removes the contamination and oxide layer and increases the wettability between the molten particle and the substrate. The benefits of the laser heating (to increase the surface substrate temperature) and the laser cleaning (to prepare the surface and ablate the oxide layer from the heated surface) combination can also be noted whatever the oxidized state of the molten particles during the APS process. In the case of overlapping 3.3, the laser application time is longer and the ablation is more perfect. Moreover, the sample scanning velocity is naturally lower because laser frequency is always kept to 60 Hz. With this slow scanning speed, the substrate surface was probably heated by high-temperature air flowing around plasma jet before arriving in the laser spot. These two phenomena lead to the decrease of the splash rate. For the overlapping 10, the sample scanning velocity is still lower, the time between the laser ablation and spraying particle becomes so long (about 300 ms for overlapping 10 against 30 ms for overlapping rate 1.1) that the effect of the ablation has already disappeared. New oxides can also appear on the surface as well as the recontamination of the deposited layers by condensed vapors during the following step of the coating deposition. As the transition temperature is needed to improve the surface wettability and also the splat morphologies, laser ablation has to be simultaneous with the plasma jet to avoid the recontamination of the substrate.

3.3 Effects of the Combined Action of Two Lasers

3.3.1 Effect of the Preheating Laser. Before performing the test with the two lasers, one test of particle impact on substrate heated by the Cheval laser (preheating) was carried out with a sample scanning speed of 300 mm/s for observing its ef-

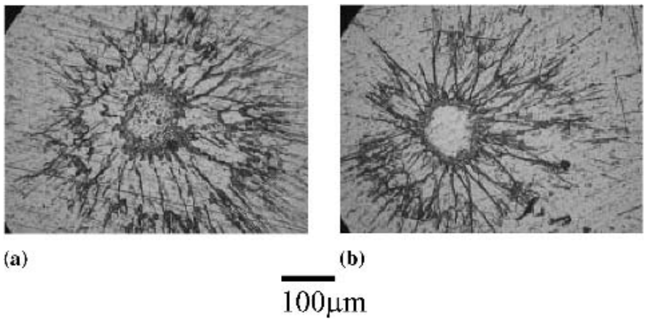

Fig. 8 Typical splat morphologies on substrate treated by Cheval laser with a power of 330/W cm^2 (a) Superposition of the laser and particle spots. (b) Laser spot before particle spot

fect. Figure 8 shows the splat morphology on substrate without laser ablation but with laser treatment with a power density of 330 W/cm^2 and for two positions of the laser spot. In both cases, the splat is characterized by significant peripheral splashes. No differences can be really detected between these two conditions, except maybe in the case of the superposition of the laser impact and the plasma jet where the particle looks more splashed. That means that, with this power, the laser is not able to heat the substrate to a high temperature or to maintain a high temperature for enough time. Moreover, when the two spots are superposed, an attenuation of the laser irradiation power can be detected by reflection or absorption of the beam by the particles. The laser intensity decreases and the real laser power reaching the surface is lower than the initial one (Ref 24).

3.3.2 Combined Action of the Two Lasers. During this experiment, two lasers were used, one for ablation, the other for heating substrate. The power of the Cheval laser was changed, and the positions of two laser spots were also adjusted in the attempt to obtain acceptable results. Figure 9 presents the evolution of the splat morphology versus Cheval laser power density and both laser spots positions.

For a power of 248 W/cm^2 for the Cheval laser and for various laser spot positions, the splat morphology presents irregular forms and very marked splashes. For higher Cheval laser powers, the combined action of the two lasers seems favorable to splat morphology improvement. However, this trend remains less obvious than that of the combination of flame preheating and laser ablation. Better splat morphologies are obtained with superposition of the two lasers spots (for Cheval laser powers higher than 330 W/cm^2). The degrees of splashing mentioned in Fig. 9 confirm this trend.

For a Cheval laser power of 248 W/cm^2, the degrees of splashing are very high whatever the laser spot position. When increasing the power, a clear splashes reduction was found. In fact, for a power of 497 W/cm^2, the degree of splashing obtained in the case of superposition of the laser impact and the particles spot is only 55% that obtained for a power of 248 W/cm^2. During the superposition of the two laser spots, it is close to 28%. In the case of the superposition of particles and heating laser spots, the decrease of the degree of splashing could probably be due to the increase of heating effect on an ablated surface. Even if an attenuation of the laser can be observed by interaction with the powder particles, the heating effect (as small as it could be) combined with the ablation treatment is more favorable than any other treatment. The spectacular reduction in degree of splashing when both laser spots were superposed can be explained by an improvement of ablation effect on the heated surface. Particles also impinged a surface substrate at a higher temperature and more cleanly than in any other conditions.

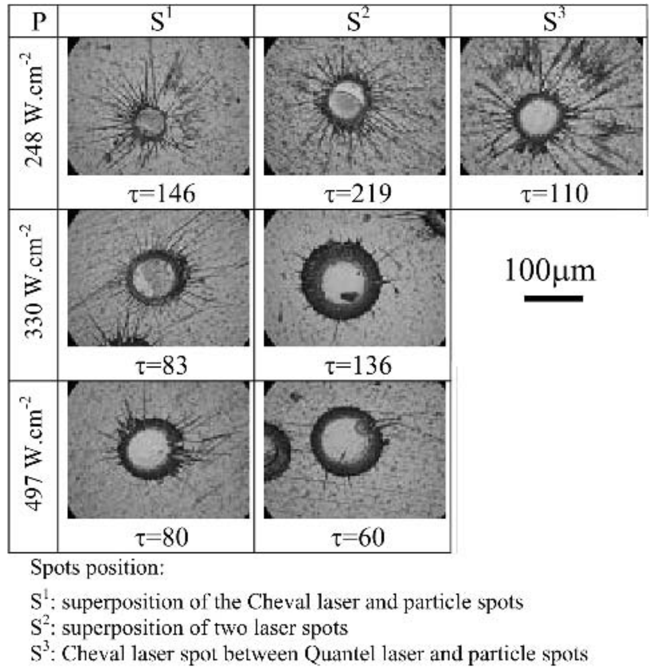

Fig. 9 Typical splat morphologies and degrees of splashing (τ) under different Cheval laser conditions and spot positions

3.4 Effect of Combination of Preheating and Laser Ablation on the Coating Adhesion

To study the effects of the combination of substrate preheating either by Cheval laser or by flame and laser ablation on the coating adhesion, the same setting described above was used, but the sample scanning procedure was repeated about 40 times to obtain a coating of about 300 μm. Both lasers were maintained only for the first and the second cycle. With flame preheating, the sample was heated in the same way as in the case of particle impact test. Table 2 presents the experimental designs and interfacial indentation results.

Compared with the other cases, the reference coating has the highest crack length and the lowest interfacial toughness. All other coatings have higher adhesion. With flame heating, results lead to a rather high adhesion, but it is very difficult to carry out this heating operation in practice due to the sequence treatments. On the other hand, the results obtained by laser treatment appear interesting, particularly when the two laser spots are superposed exactly as in the particle impact test. The laser spot position also seems to be a very important parameter. Whatever the power density used for the laser heating, measurements present toughness obviously higher than others. Comparing these two samples (S^2), the higher the Cheval power, the higher the toughness.

Table 2 Experimental conditions and results of interfacial toughness

Cheval laser power density, W/cm^2	Use of flame, temperature, °C	Spots posit.(a)	Crack length μm (σ)(b)	K_{Ic} MPa m$^{0.5}$ (σ)
330	…	S(c)	198 (50)	0.84 (0.32)
497	…	S^1(c)	203 (44)	0.79 (0.26)
330	…	S^2(d)	184 (18)	0.87 (0.13)
497	…	S^2(d)	180 (51)	0.99 (0.43)
…	…	…	249 (55)	0.58 (0.20)
…	400	…	191 (23)	0.82 (0.15)
…	280	…	199 (18)	0.77 (0.11)

(a) Spots position. (b) σ: standard deviation (average of 10 measurements). (c) S^1: superposition of the Cheval laser and particle spots. (d) S^2: superposition of two laser spots

Then laser treatments to prepare the surface substrate prior thermal spraying appear very interesting for improving the coating adhesion in a single step. After laser heating and laser ablation, it appears that the surface is in a good condition to receive the melted powder (good wettability and oxide-free surface after laser ablation) and permits good spreading of each particle, also ensuring better adhesion. As no significant surface topography modification can be noticed after laser beam impact, the resulting adhesion of sprayed particles does not derive any more from mechanical anchoring, which can explain also the strong influence of the laser power as well as the laser spot positions (Ref 5).

4. Conclusions

Experiments were conducted to estimate the effects of the substrate preheating (either by flame or by laser) combined with laser ablation on the adhesion of a Ni-Al (95-5 wt.%) sprayed onto a TA6V titanium base alloy. To evaluate the laser cleaning and the preheating process on the coating adhesion, the analysis of the morphology of impinged particles was carried out before implementing the interfacial indentation test to measure interfacial toughness.

As a conclusion, a higher substrate temperature (250 °C in that case) leads to a disk-shaped splat when only flame preheating is used. With laser ablation, a circular-shaped splat can be obtained at much lower temperature.

The combined action of two lasers, a preheating laser and an ablation laser, also causes an important improvement of splat morphologies and significant splash rate reduction. This means the use of two lasers simultaneously enhances the effects of both lasers. That was proved by obtaining the best results for both degrees of splash and adhesion when superposition of the two laser spots. The two actions (heating and cleaning) are believed to promote the substrate wettability and the adhesion between the particles and the substrate.

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