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Remelting of Flame Spraying PEEK Coating Using Lasers

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Flame spraying is frequently used for polyether ether ketone (PEEK) and PTFE coating deposition on metallic surfaces. However, this process has a certain number of limitations, particularly on the coating quality such as high porosity, low interfacial adherence, etc. For that reason a thermal post-processing step is often necessary. The objective of this study is to analyze the effects produced during a laser beam heat treatment on morphological structure (compactness) of PEEK coatings and their mechanical properties (adherence and tribology). The influence of the laser beam wavelength (by using a Nd:YAG, CO_2 or diode lasers) on compactness of the flame sprayed PEEK coating deposited on metallic substrate (304L) was analyzed. Since the value of laser light absorption coefficient of the PEEK coating depends on the laser wavelength, an optimization of the operational parameters for each laser has been carried out in order to achieve melting but not burning of the PEEK coating. Nevertheless, whatever the laser wavelengths used, the results showed a good effect of the laser treatment: improvement of both polymer coating compactness and its adherence to the substrate.

Keywords flame spraying, laser remelting, PEEK coating

1. Introduction

In order to answer many industrial requirements (economic, ecological, etc), organic coatings have become more and more attractive to improve the properties of different materials, particularly metallic surfaces. Among these, polyether ether ketone (PEEK) seems to be relevant for their excellent mechanical characteristics (particularly tribological, as low coefficient of friction), their low surface energy and their high working temperature.

Several techniques can be used to make this type of deposits as serigraphy, flame spraying, liquid projection, etc. (Ref 1-4). However, in spite of many efforts made in this area (Ref 5, 6), these processes still have a certain number of limitations, particularly concerning the coating quality such as high porosity, low interfacial adherence, etc. For specific applications such as biomedical, a porous structure can be interesting but in case of applications like corrosion protection, diffusion resistance or wear protection at high temperature, a high coating porosity can introduce bad mechanical properties. In order to improve the coating performances, the materials have to be densified. For that reason, a thermal post-processing step is often necessary, and if possible it would be useful to develop a simultaneous treatment process.

In the last few years, several experimental studies on laser-polymer interactions have been conducted. Polymer behavior during laser transmission welding for different laser beam wavelengths has been studied in (Ref 7-9), while Laurens and Wonga (Ref 10, 11) have examined the influence of laser beam in the process structuring of polymers. The evolution of temperature field within the organic matter during laser-polymer interaction has been analyzed using different numerical models (Ref 12-14).

The aim of this study was also to test the influence of different laser wavelengths on a heating effect on the organic coating. Since polymer coating has different absorption coefficient values for each laser wavelength, laser parameters have also to be adjusted in order to achieve melting but not burning of the polymer. An experimental design matrix has been developed in order to analyze and optimize the laser parameters influence on compactness and adherence of the PEEK coating.

In this experiment, the PEEK coatings have been deposited on stainless steel substrates by flame spraying technique. Three laser beams with different wavelengths have been applied: Nd:YAG ($\lambda = 1.064$ µm), CO_2 ($\lambda = 10.6$ µm) and diode laser ($\lambda = 0.88$ µm).

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2. Experimental Procedure

2.1 Coating Deposition

PEEK coatings were sprayed using a flame gun (Eutectic-Castolin Castodyn 8000 powder flame spray gun) mounted on a robot (ABB 4400). The feedstock material was the commercially available Vitrex PEEK powder (Vitrex Sale Ltd., Lancashire, UK) having a grain of a mean diameter of 25 μm. It has a glass temperature transition of 143 °C and a melting temperature of 343 °C. The substrates were stainless steel 166 × 118 × 2 mm plates.

During the spraying process, the substrates were fixed right in front of the flame torch, which moved with a linear velocity of 150 mm s^{-1}. Then, coatings were built up by multiple melted particles impacting on the stainless steel surface. Of course, several passes are necessary to reach the proper coating thickness of 100 μm.

To improve the coating adherence on the substrate, stainless steel parts were degreased and sandblasted just before the spraying operation. Process parameters are given in Table 1. A view of the surface and a cross-section of the PEEK coating deposited by flame spraying is shown in Fig. 1. The coating exhibits a high porosity and it is simply an accumulation of semi-melted particles.

2.2 Laser Treatments

To achieve the remelting of the coating, a CO_2, a Nd:YAG and a diode laser were used in this work.

The CO_2 laser system uses a ROFIN (ROFIN BASSEL France S.A.) SCX20 pulsed source. Its maximal average power and pulse energy for a spot diameter of 102 μm are 240 W and 360 mJ, respectively. The fusion lines were created using a XYZ translation table.

The Nd:YAG laser system, working in continuous mode, uses a ROFIN SLM40D source. The maximum average power for a spot diameter of 360 μm is 70 W. The displacement of the laser beam on the sample surface was performed using a galvanometric head.

The diode laser system uses a Laserline (Laserline GmbH, Germany) LDL 40-100 continuous source of 100 W maximal power. Using an optical system of 150 mm focal distance, a focused laser spot of 1.3 × 1.3 mm was obtained.

Based on previous tests performed at the laboratory, it was chosen to analyze the influence of three operating parameters for Nd:YAG laser and diode laser (scanning laser speed, defocusing distance, laser incident power) and four operating parameters for CO_2 laser (scanning laser speed, defocusing distance, frequency of pulses, pulse duration) on two objective functions (compactness and adherence of flame sprayed PEEK coating). All tests were conducted in the open air. The levels of laser parameters and their physical values are presented in Table 2. The experiments were performed using an experimental design method (Ref 15, 16). This method allows to study a reduced number of different laser parameter combinations (16 tests for CO_2 and diode lasers and 9 tests for Nd:YAG laser) without decreasing the accuracy of the results. The combinations between laser parameters, as well as observed values of objective functions for each lasers, are presented in Table 3-5.

Table 1 Spray parameters for the PEEK powder

Flame spraying	
Acetylene flow rate, L min^{-1}	6
Oxygen flow rate, L min^{-1}	16
Feedstock carrier gas flow rate (N_2), L min^{-1}	35
Spray distance, mm	140
Spray velocity, mm s^{-1}	150
Scanning step, mm	5

2.3 Laser-PEEK Interaction

The basic structure of matter involves charged particles bound together in many different ways. When laser beam radiation is incident on matter, it causes the charged particles to oscillate and gain energy. The ultimate fate of this energy depends on the situation. It could be immediately re-radiated and appear as scattered (R'), reflected (R), transmitted (T) and/or absorbed (A) radiation.

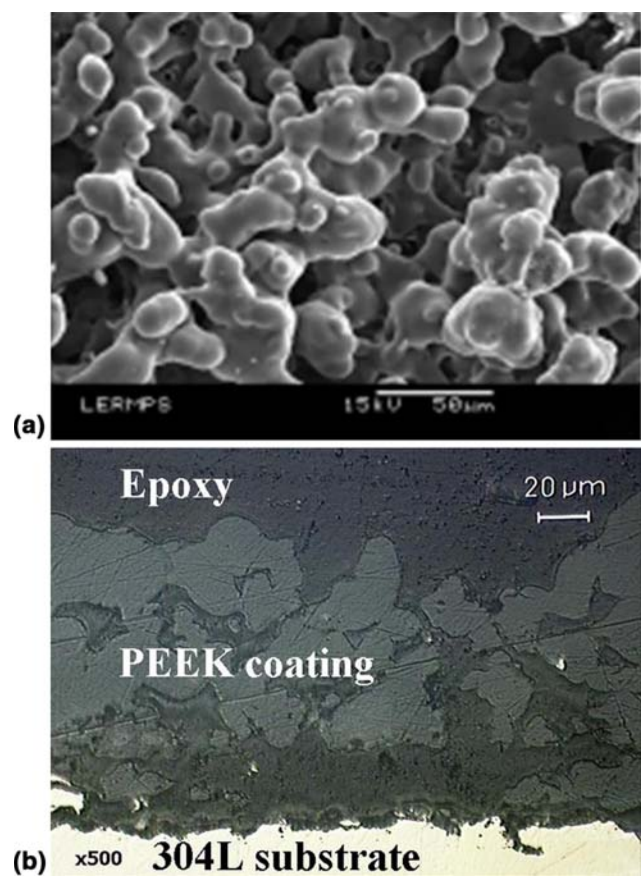

Fig. 1 Surface morphology (a) and structural cross section (b) of as-sprayed PEEK coating

Table 2 Laser operating parameters

Frequency (f), Hz	Pulse duration (t_p), µs	Scanning speed (s), mm s^{-1}	Defocusing (z), mm	Spot diameter (d), mm
CO_2 laser parameters				
1500	20	2	+100	3
3000	50	3	+110	3.3
4500	80	4	+120	3.6
6000	110	5	+130	3.9

Incident laser power (P_i), W	Scanning speed (s), mm s^{-1}	Defocusing (z), mm	Spot diameter (d), mm
Nd:YAG laser parameters			
50	1	+60	3
54	2	+80	4
58	3	+100	5

Incident laser power (P_i), W	Scanning speed (s), mm s^{-1}	Defocusing (z), mm	Spot dimension ($d_x \times d_y$), mm
Diode laser parameters			
70	1	+30	6.3 × 5.8
80	2	+35	6.4 × 6.2
90	3	+40	6.5 × 6.3
100	4	+45	7.0 × 6.5

Table 3 Experimental design matrix and obtained results for CO_2 laser

	Operating parameters				Objective functions	
Test	s, mm s^{-1}	z, mm	f, Hz	t_p, µs	Compactness	Adherence
1	2	100	1500	20	Not melted	Not adherent
2	2	110	3000	50	Melted	Slightly adherent
3	2	120	4500	80	Burned	…
4	2	130	6000	110	Burned	…
5	3	100	3000	80	Burned	Slightly adherent
6	3	110	1500	110	Well melted	Adherent
7	3	120	6000	20	Not melted	Not adherent
8	3	130	4500	50	Well melted	Adherent
9	4	100	4500	110	Burned	…
10	4	110	6000	80	Burned	…
11	4	120	1500	50	Not melted	Not adherent
12	4	130	3000	20	Not melted	Not adherent
13	5	100	6000	50	Burned	…
14	5	110	4500	20	Not melted	Not adherent
15	5	120	3000	110	Burned	…
16	5	130	1500	80	Slightly melted	Not adherent

Table 4 Experimental design matrix and obtained results for Nd:YAG laser

	Operating parameters			Objective function	
Test	P, W	z, mm	s, mm s^{-1}	Compactness	Adherence
1	50	60	1	Burned	…
2	50	80	2	Melted	Adherent (0)
3	50	100	3	Not melted	Not adherent (2)
4	54	60	2	Burned	…
5	54	80	3	Slightly melted	Adherent(0)
6	54	100	1	Well melted	Adherent (0)
7	58	60	3	Burned	…
8	58	80	1	Burned	…
9	58	100	2	Melted	Adherent (0)

Table 5 Experimental design matrix and obtained results for diode laser

	Operating parameters			Objective functions	
Test	v, mm s^{-1}	z, mm	P, W	Compactness	Adherence
1	1	30	70	Burned	…
2	1	35	80	Burned	…
3	1	40	90	Well melted	Adherent
4	1	45	100	Well melted	Adherent
5	2	30	80	Burned	…
6	2	35	70	Melted	Slightly adherent
7	2	40	100	Well melted	Adherent
8	2	45	90	Slightly melted	Slightly adherent
9	3	30	90	Burned	…
10	3	35	100	Burned	…
11	3	40	70	Not melted	Not adherent
12	3	45	80	Not melted	Slightly adherent
13	4	30	100	Burned	…
14	4	35	90	Slightly melted	Slightly adherent
15	4	40	80	Not melted	Not adherent
16	4	45	70	Not melted	Not adherent

The PEEK is more or less transparent at all wavelengths used in this study. In this case, a part of laser power is absorbed by the coating and the other part is transmitted through it. The absorption occurs if the frequency of the incident photons corresponds to the "frequency" associated to the transition energy (electronic, vibrational and rotational) of irradiated molecules. The absorption of laser beam energy leads to system transition from a stationary state to an exited state, including a considerable energy increase at molecular level. Relaxation of excited molecules is done by internal conversion, followed by a vibrational relaxation (about 10^{-13} s). The exceeding energy is transferred to neighbor molecules. The result is the heating up of the substrate. Local temperature increase depends on the material thermal conductivity, laser beam wavelength and intensity.

Hence, before performing the laser treatments, a measurement of laser beam transmission factor through the coating using a powermeter (COHERENT 200 W Ultima LabMaster powermeter) was done for all three laser wavelengths. This consists in measuring both the incident laser beam power and the laser beam power transmitted through the PEEK coating. After that, using Eq 1 the transmission factor was calculated.

$$T\,[\%] = \frac{P_T}{P_i} \times 100 \qquad \text{(Eq 1)}$$

where T is the transmission factor, P_T is the transmitted laser power through PEEK coating and P_i is the incident laser power.

Figure 2 presents the transmission factor variation depending on incident laser power for two laser wavelengths. A transmission factor of 55% for Nd:YAG and 75% for a diode laser has been found. The transmission

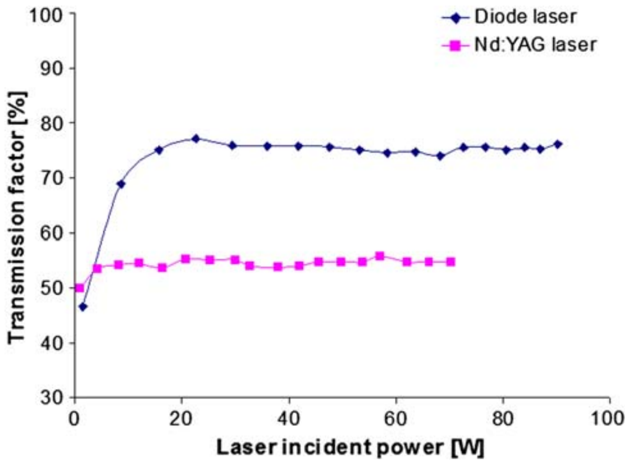

Fig. 2 Transmission factor dependences on laser wavelength

factor for the CO_2 laser beam was supposed to be smaller than 10%, since the PEEK coating used to measure the laser power transmitted to was burned even for a small incident laser power. Hence, in the case of CO_2 laser treatment, the melting of the PEEK coating was mostly done by the action of laser beam. For the other two lasers, since a part of the laser beam reaches the substrate surface, the melting process of PEEK coating is achieved by both absorption of the laser beam and conductive heat transfer between the heated substrate by the laser and the PEEK coating.

2.4 Coating Characterizations

First, to classify the laser treatment effects, the surface and the cross section of the coatings were observed.

After cutting and infiltrating with epoxy (impregnation technique), the samples were polished following standard metallographic techniques (pre-polishing and diamond slurry polishing). The samples were observed using an optical microscopy (Leica® MPS52). It was considered that the PEEK coating shows a good improvement of morphological structure (good compactness) if it is porosity free. In order to perform a statistical analysis, a numerical coefficient indicating the level of compactness has been assigned to each treated sample. The value "0" has been assigned to the samples where the PEEK coating was not melted at all. The value "1" has been assigned to the samples where the PEEK coating was melted and porosity free. The value "2" has been assigned to the samples where the PEEK coating surface was burned. The assessment of the surface of the PEEK as "burned" after laser treatment was done by a simply visual analysis as presented in Fig. 3. In this way it was possible to develop a compactness variation map depending on variation of processing parameters (laser power density and laser-PEEK coating interaction time).

The adhesion strength of the coating to the substrate after the laser treatment was evaluated by implementing a mechanical test. This test is similar to the standard

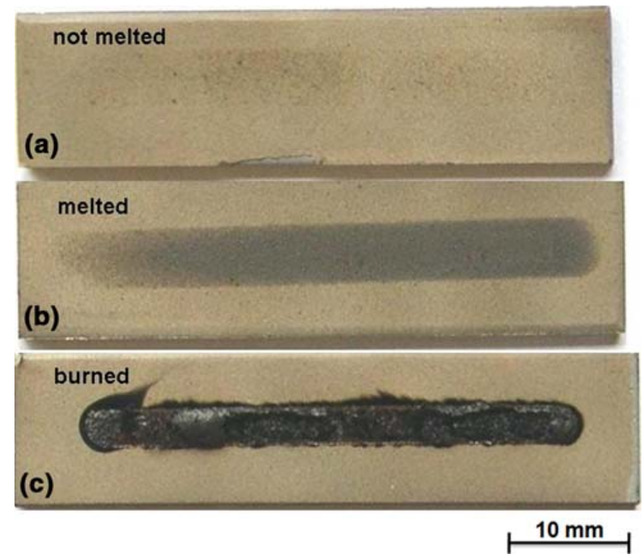

Fig. 3 Visual aspect of PEEK surface coatings after laser treatment

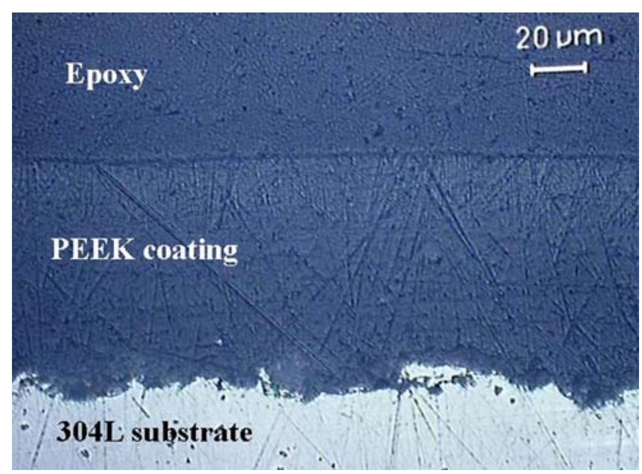

Fig. 4 Typical cross-sectional view of PEEK coating treated by CO_2 laser ($f = 4500$ Hz; $t_p = 50$ μs; $s = 3$ mm s^{-1}; $z = +130$ mm)

ISO2409:2007 (Ref 17) used to verify the paint adherence. For this, equally spaced horizontal and vertical lines were engraved inside the PEEK coating in order to obtain a grid. Then, by optical observation of the surface, a classification of the laser treatments is possible by identifying the peeling-off effects. The PEEK coating shows a good improvement in adherence if the laser treated surface does not show peeling-off effects. Like in the case in compactness, a numerical coefficient indicating the level of adherence has been assigned to each treated sample. The value "1" has been assigned to the samples where there were a lot of peeling-off effects in the thermically treated area. The value "0" has been assigned to the samples where the treated area does not show peeling-off effects. The value "−1" has been assigned to the samples where the PEEK coating surface was burned.

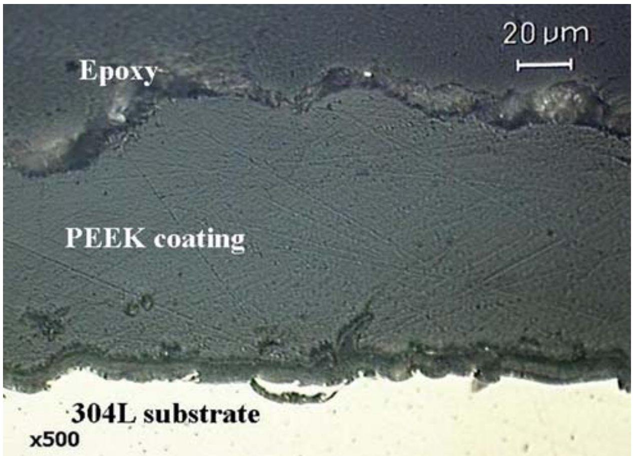

Fig. 5 Typical cross-sectional view of PEEK coating treated by Nd:YAG laser ($P = 54$ W; $s = 1$ mm s^{-1}; $z = +100$ mm)

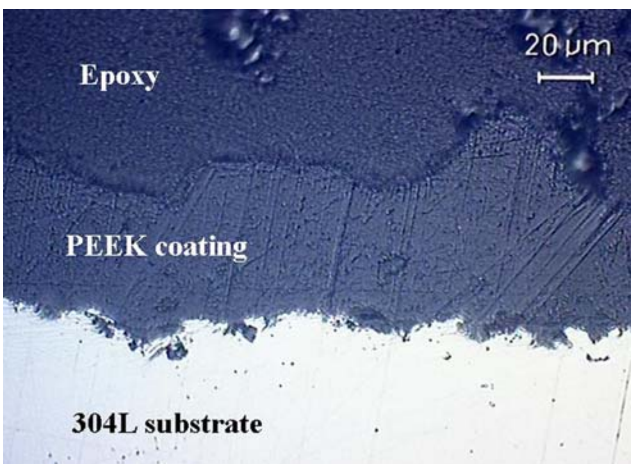

Fig. 6 Typical cross-sectional view of PEEK coating treated by diode laser ($P = 100$ W; $s = 2$ mm s^{-1}; $z = +40$ mm)

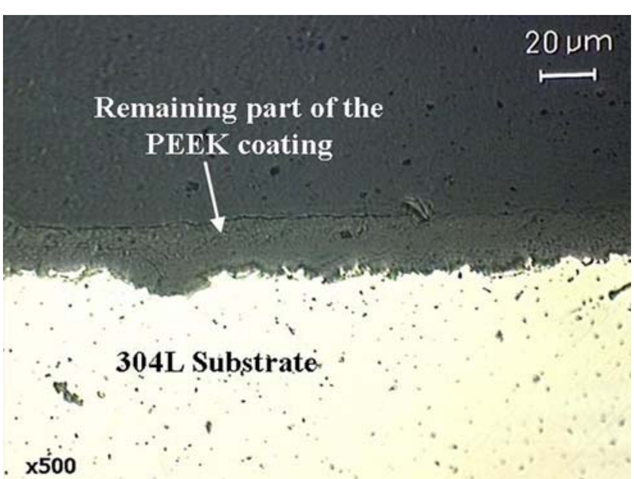

Fig. 7 Example of cross section observations of a burned PEEK surface coating

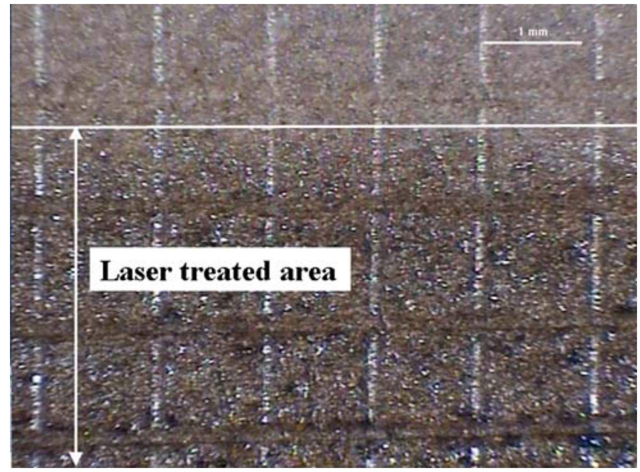

Fig. 8 Typical PEEK coating surface appearance after mechanical test application (CO_2 laser; $f = 1500$ Hz; $t_p = 110$ μs; $s = 3$ mm s^{-1}; $z = +110$ mm)

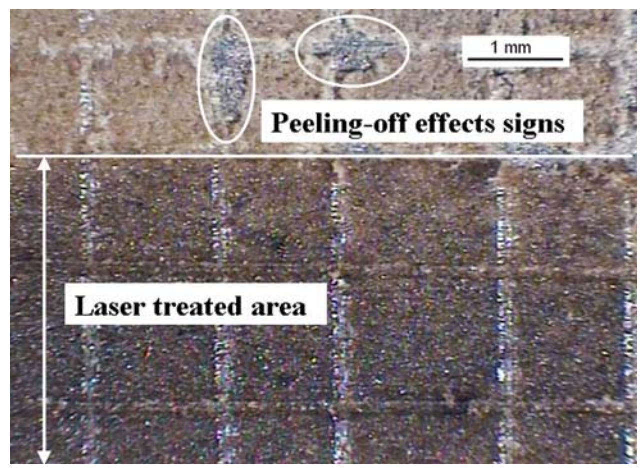

Fig. 9 Typical PEEK coating surface after mechanical test (Nd:YAG laser; $P = 50$ W; $s = 2$ mm s^{-1}; $z = +80$ mm)

Fig. 10 Typical PEEK coating surface after mechanical test (diode laser; $P = 100$ W; $s = 1$ mm s^{-1}; $z = +45$ mm)

3. Results and Discussion

Comparing the porous structure of reference surface (Fig. 1b) with the treated surface (Fig. 4-6), a good improvement of the coat compactness can be noticed for all used laser. The porosity was considerably reduced, which corresponds to a more homogenous structure.

During the cross-sectional observations, it has been noted that even though the PEEK was burned, a small vitreous part (Fig. 7) of the polymer remains at the substrate surface (about 20%). This result can be explained by the fact that the temperature at the PEEK-substrate interface does not exceed the melting temperature of the polymer. By consequent, the entire PEEK coating at the substrate surface was not eliminated.

Concerning the adherence, after implementing the mechanical test on the samples which were not burned, an improvement in adhesion of polymer coating to the substrate was noted. The laser-treated area does not presents peeling-off signs (Fig. 8-10). Nevertheless, in the case of Nd:YAG laser treatment, from the cross-sectional observations (Fig. 5), the existence of a small layer from the coating resin can be noticed between the PEEK coating and substrate. According to these results, the Nd:YAG laser treatment does not allow 100% improvement of the PEEK adhesion to the substrate.

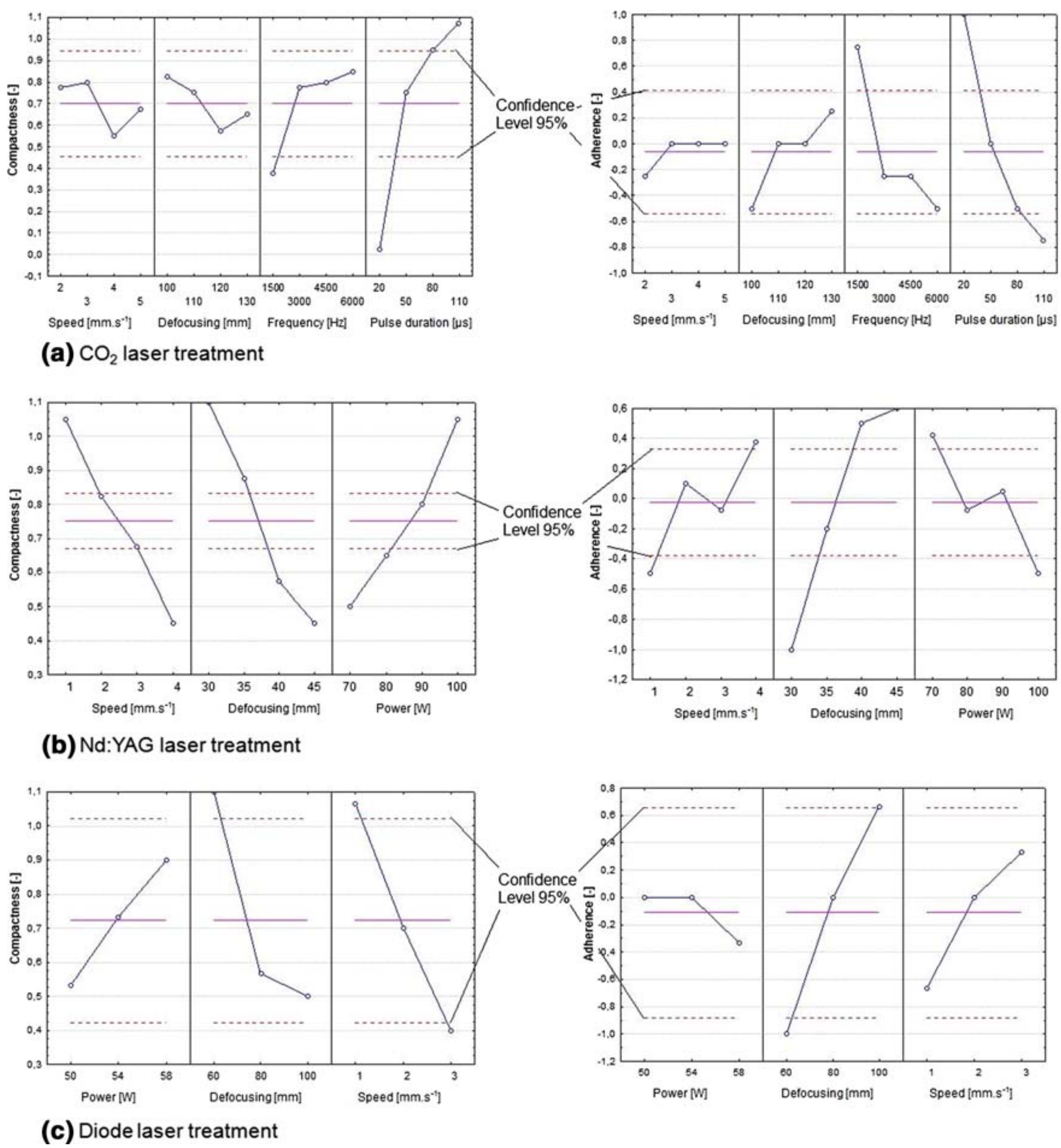

Fig. 11 Diagrams of laser parameters generated effects on objective functions

The application of an experimental design method (Ref 15, 16) allows us to statistically analyze the dependence of objective functions (compactness and adherence improvement of PEEK coating) on laser parameters. First, using the equations presented in Ref 15 and 16, the effects of laser parameters on objective functions have been determined. The graphical representations of effects for each laser parameter variation levels for all three wavelengths used in this study are presented in Fig. 11.

In the case of CO_2 laser and for a confidence level of 95%, only frequency and pulse duration have a significant influence on objective functions since they exceed the confidence interval. Using long pulse duration (50-110 μs) and a frequency of 1500-4500 Hz is appropriate in order to obtain a good compactness and adherence of PEEK coating in the same time (Fig. 11a).

For the same confidence level, the Nd:YAG laser treatment of PEEK is significantly influenced by defocusing and scanning speed. In order to obtain a good improvement of PEEK coating compactness, it is recommended to use a defocusing between 60 and 80 mm and a scanning speed of around 1.5 mm s^{-1} (Fig. 11b). A good improvement of PEEK coating adherence was observed for the same laser parameters, the remelted coating surfaces do not show any peeling-off effects, as observed on the as-sprayed area.

In the case of diode laser, all operating parameters have a significant influence on the compactness and the adherence of PEEK coating. Since only 20%, a good compactness of PEEK coating was obtained by using a small scanning speed and a significant defocusing. On the other hand, an increase of laser power is necessary to favor the melting process. In this case, the best results were obtained for a laser power of 100 W, a scanning speed of 1-2 mm s^{-1} and a defocusing of 40-45 mm (Fig. 11c).

These results are restricted to the used laser machine. So, in order to have a better understanding of the PEEK coating behavior during the laser treatment, a dependence of the melting processes on laser power density and interaction time was studied.

Analyzing the variation of PEEK compactness with the power density and the interaction time during a CO_2 laser treatment, it can be seen that the density power working zone (Fig. 12, filled zone) is very narrow. Depending on the interaction time, this zone ranges between 6 and 12 W mm^{-2}. The working zone becomes smaller in the case of adherence (Fig. 13). An inferior limit of the interaction time was observed. This leads to the reduction of the working range of power density. In this case to obtain both compactness and adherence, it is recommended to use an interaction time longer than 0.8 s and a power density between 6 and 8 W mm^{-2}.

In order to obtain an improvement of compactness and adherence of the PEEK coating by using a Nd:YAG laser, it is necessary to decrease the interaction time if the density power is increased (Fig. 14 and 15). A high-density power correlated with a long interaction time leads to an overheating of both PEEK coating and substrate. On the contrary, a low power density and a short interaction time do not enable the polymer to reach the fusion temperature. Short interaction time and high power density can be used, but in this case the working zone becomes very narrow and it is very easy to cross over from an unmelted to a burnt state. In this case, it is much better to work with a small power density (2-3 W mm^{-2}) and a longer interaction time (4 s), which offer the possibility that the polymer absorbs a sufficient quantity of energy in order to remelt the entire PEEK coating without burning it.

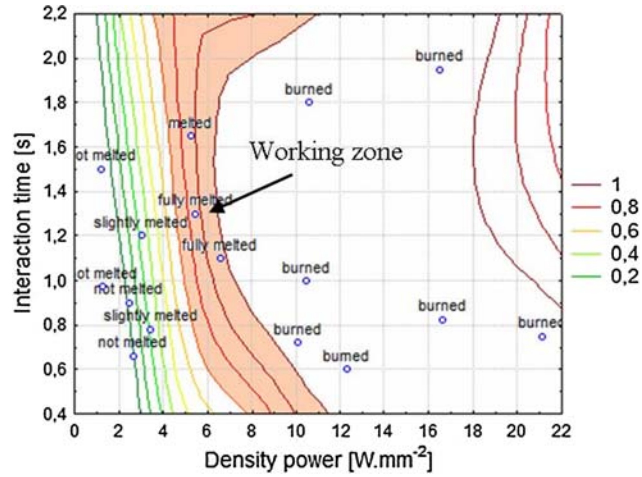

Fig. 12 PEEK coating compactness variation with power density and interaction time (CO_2 laser; $\lambda = 10.6$ μm)

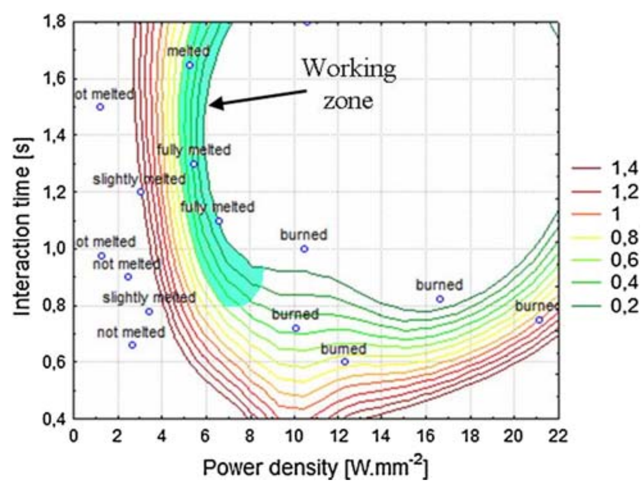

Fig. 13 PEEK coating adherence variation with power density and interaction time (CO_2 laser; $\lambda = 10.6$ μm)

In the case of diode laser treatment of PEEK coatings, an interaction time shorter than 3 s leads to a burning of PEEK coating, for any values of the power density. In this case, it is better to use a long interaction time (bigger then 4 s) and an appropriate density power (Fig. 16). In the case of adherence, the treated zone is larger than in the case of compactness (Fig. 17). Because 80% of the laser power irradiates the interface coating-substrate, the

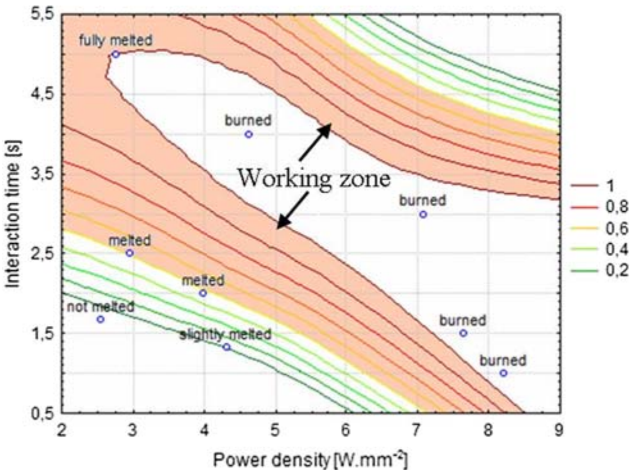

Fig. 14 PEEK coating compactness variation with power density and interaction time (Nd:YAG laser; λ = 1.06 μm)

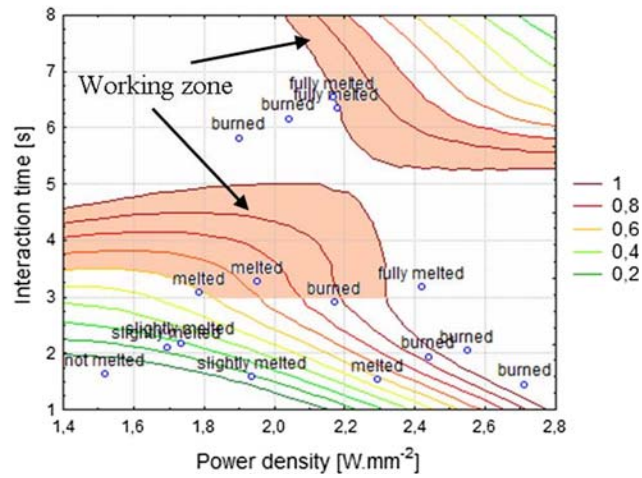

Fig. 16 PEEK coating compactness variation with power density and interaction time (diode laser; λ = 0.88 μm)

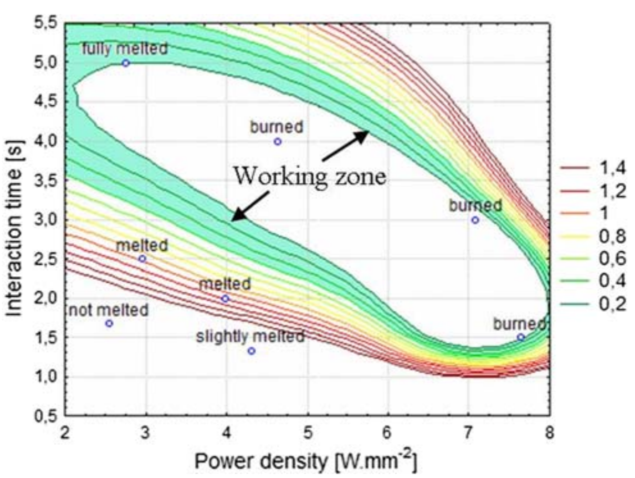

Fig. 15 PEEK coating adherence variation with power density and interaction time (Nd:YAG laser; λ = 1.06 μm)

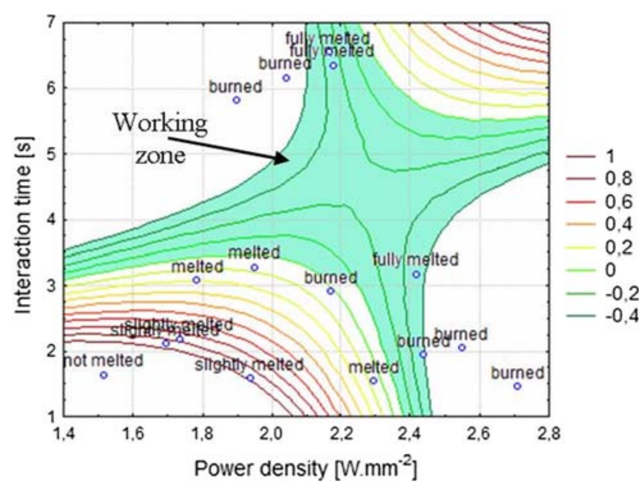

Fig. 17 PEEK coating adherence variation with power density and interaction time (diode laser; λ = 0.88 μm)

heating of the substrate is faster and the remelting of the polymer in this area is better.

This study allows to observe that the longer the applied wavelength, the better the compactness of the PEEK coating is. Unfortunately, an opposite behavior has been observed for the adherence. This phenomenon can be explained by the decrease of absorption coefficient of the PEEK coating with the wavelength. For example, for a wavelength of 10.6 μm (factor of absorption >90%), it is necessary to reduce the time of interaction in order to avoid a possible degradation of polymer on the surface, but without to be certain that a good adherence will be obtained at the same time. On the other hand, for a wavelength of 0.880 μm (factor of absorption ~20%), the substrate is more quickly heated and in this case a better fusion of polymer is obtained at this place, without to be certain that an entirely compact deposit will be obtained. So, to improve both PEEK coating compactness and adherence, it is necessary to increase the interaction time and decrease the power density, when decreasing the laser wavelength.

4. Conclusion

For all three wavelengths applied, the ranges of laser parameters for which both PEEK coating compactness and adherence can been obtained have been determined.

In conclusion, all laser wavelengths used in this work are appropriate for improving both compactness and adherence of PEEK coatings. Unfortunately, the interaction time necessary to remelt the PEEK coating is too long for production process. A solution would be to use two different lasers: CO_2 and diode laser. First, the diode laser (whose radiation is less absorbed by the polymer, ~20%) can be used to preheat the substrate and to improve the adherence of the PEEK coating. After that, the CO_2 laser (whose radiation is much more absorbed by polymer, >90%) can be used to remelt the PEEK coating. In this way, it is possible to reduce the interaction time and obtain good results.

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