Thermal Sprayed Coatings Adherence – Influencing Parameters

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Abstract: - Thermal spraying represents the process of obtaining new special multilayer structure materials, with good mechanical and chemical characteristics. These materials are often used in solving real important problems, like repairing worn parts working under severe wearing conditions or, ensuring efficient corrosion protection of parts used in sea, as platform, bridges, or obtaining high refractory surfaces. Most of the times, once obtained, these coatings need additional machining and, it is of interest to study how, and, if, any of the machining parameters do influence one of their very important characteristic, meaning, adherence to the basic substrate. The paper presents a study on the adherence of thermal sprayed coatings, obtained from some Romanian thermal sprayed materials and submitted to exterior cylindrical turning.

Key-Words: - thermal spraying, multilayer coatings, cylindrical turning, adherence, transducer, sample

1 Introduction

Thermal spraying or, metallizing is the process of spraying molten metal, metallic oxides or ceramics, onto a previously prepared substrate [4]. This process type can be carried out only if there is a *heating source* – to get the material into a (near to) molten state and, a *gas jet* (compressed air) – to propel the molten material's particles onto the target substrate.

So, the material is melted in a flame, or into an electric arc or in a plasma jet and atomized, by the blast of compressed air, into fine spray [7]. A schematic representation of the metallizing process is represented in figure 1.

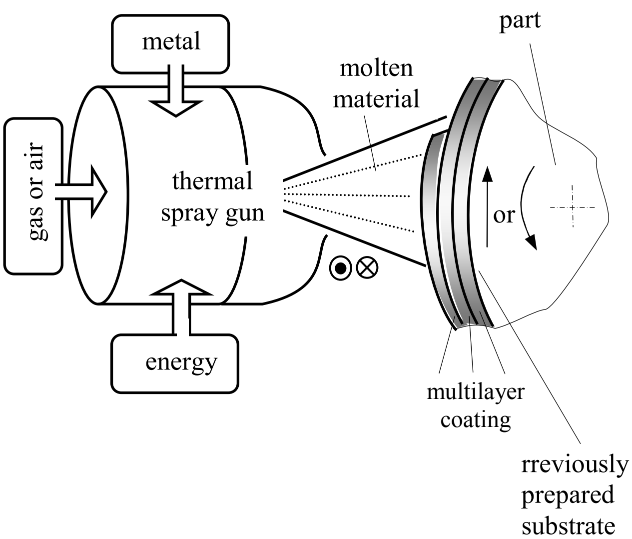


Fig. 1 Metallizing process scheme

When impacting the previously prepared part substrate or, the previously obtained thermal sprayed layer, the molten material particles flatten out, crack (specially the oxides surroundings) and anchor onto it.

By successive thermal sprayings, a lamellar, anisotropic structure is obtained. One can notice the flat melted and, then, solidified, sprayed material particles, surrounded by tough oxides or carbides, next to un-melted sprayed material particles and voids. So, the thermal sprayed coatings have multilayered lamellar porous structure, pointed out in figure 2.

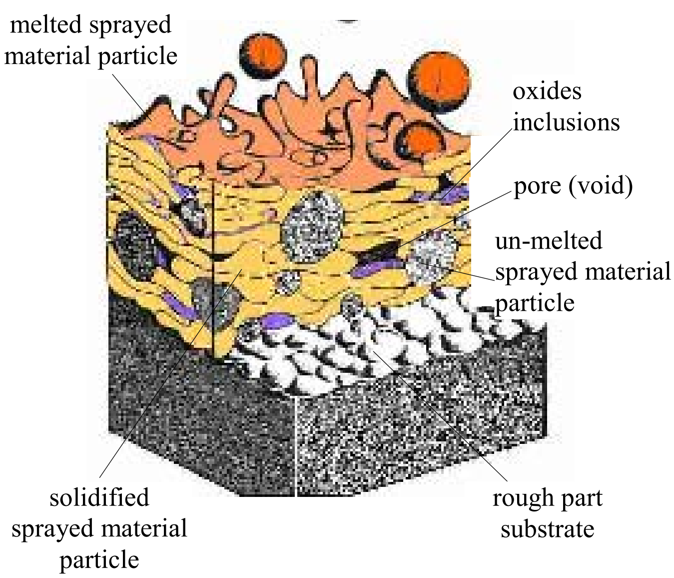


Fig. 2 Thermal sprayed coatings' structure [7]

The bonds between particles, as well as their bond to the basic substrate are obtained by: particles cementing of surroundings oxides, welding micro-points and mechanical anchoring onto the impact surface's roughness.

The sprayed molten particle that strikes the surface, flattens out, cools and solidifies so, residual stresses do appear. When the multilayer structure – meaning thermal coating – is obtained, these residual stresses do sum up and, thus, a permanent internal stresses field exists.

As result, the exterior coating is in tensile stress, while the basic material is in compressive stress – if considering exterior cylindrical thermal sprayed coatings. When spraying onto flat surfaces, the residual internal stresses, within the coating, may produce a curvature of part's the exterior sides while, if spraying onto interior cylindrical surface, there is the danger that multilayered structure falls off the basic substrate.

Thermal sprayed coatings have very important mechanical characteristics, such as:

- high compressive strength, and low tensile strength;
- remarkable hardness values (micro-hardness, HV 0.05);
- porosity – good when used in severe wear condition (for lubricant) or high temperature protection (refractory coatings) and bad when used for corrosion protection;
- high wear resistance - specially when metal / metal friction occurs;
- *adherence* to the basic substrate – due to mechanical (coupling), diffusion and chemical (oxides, carbides, Van der Waals forces) mechanisms.

When multilayer thermal sprayed coatings are generated to repair worn parts or, to obtain good wear resistance and high hardness characteristics, machining is often necessary. So, the prescribed geometrical precision conditions of the surfaces to be metallized can be obtained by turning, grinding and, sometimes, by drilling and milling.

One commonly used procedure (that, if carefully performed, does not harm the multilayered structure) is cylindrical turning – specially considering the shape of the metallized surfaces.

As the specific literature *does not mention*, if the machining parameters: cutting speed, cutting feed and cutting depth) have any influence on coatings' adherence, it has been considered useful a study on this topic [1]. In order to do this, a special transducers (resistive and inductive) system has been designed.

2 Experimental Research

The studies were carried out on specially prepared samples, whose thermal sprayed materials were Romanian produces ones: MET 4, Inox 18-8, S12Mn2Si and Al-Ol (alluminium steel alloy).

Chemical structure of the metallizing materials, as well as some mechanical properties of the thermal sprayed coatings are presented in Table 1.

The metallizing process used an electric arc for melting materials, whose initial shape was wired. Spraying melted material's particles towards the part's previously prepared substrate was done by compressed air. Specific parameters values of the metallizing process are mentioned by Table 2.

An image taken while thermal spraying is shown in figure 3.

Table 1

Thermal coatings characteristics

Material	Chemical Structure	HV 0.05	Porosity [% vol]	Steel Base Adherence [N/mm^2]
MET 4	(14 ÷ 15) % Cr, (0,4 ÷ 0,5) % C	370	7 ÷ 9	47
Inox 18-8	8,8 % Ni, 18,9 % Cr	340	7 ÷ 9	26
S12Mn2Si	max 0,12% C (1,8 ÷ 2,2) % Mn max 0,15% Si	290	7 ÷ 8	40
Al-Ol	99,5% Al + S10Mn1Ni2 [0,1% C (0,8 ÷ 1,2) % Mn (1,8 ÷ 2,2) % Ni]	150	7 ÷ 9	24

Table 2

Electric arc metallizing process parameters' values

Material	Process parameters values			
	U [V]	I [A]	h [mm]	p_a [bar]
MET 4	32	200	60	2,5
Inox 18-8	30	200	60	2,5
S12Mn2Si	30	200	60	2,5
Al-Ol	28	180	70	2,5

U – electric arc voltage
I - electric current intensity
h – spraying distance
p_a - pressure of compressed air

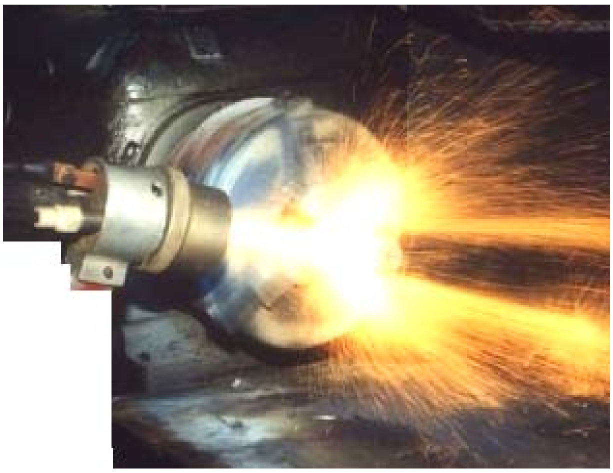
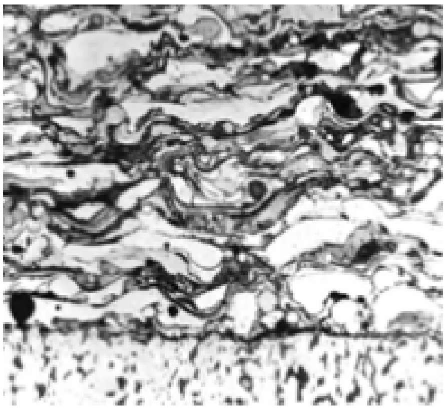


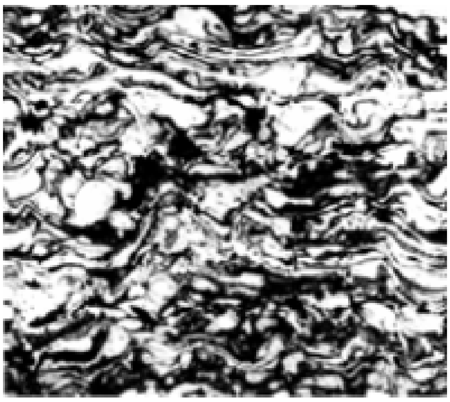
Fig. 3 Electric arc metallizing process

The metallographic structure of the multilayered thermal sprayed coatings is presented in figure 4, for each of the studied thermal sprayed materials [3]



a.

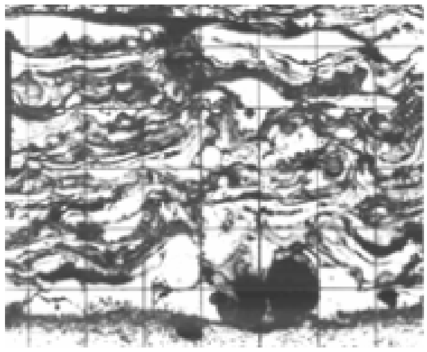
MET 4 – thermal sprayed coatings



b.

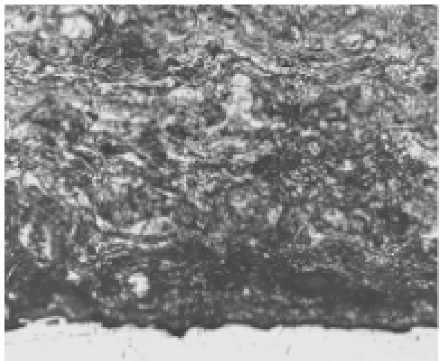
Inox 18-8 – thermal sprayed coatings

Fig. 4 Metallographic structure of metallized coatings



c.

S12Mn2Si – thermal sprayed coatings



d.

Al-Ol– thermal sprayed coatings

Fig. 4 Metallographic structure of metallized coatings - continued

There are two standardized methods of testing thermal sprayed coatings adherence to the basic material.

One is tensile testing, meaning measuring the force required to tear off the coating sprayed onto the frontal side of a ring shape sample. It has been carried out [9], as seen in figure 5 but, only for metallized coatings, not for metallized and machined ones.

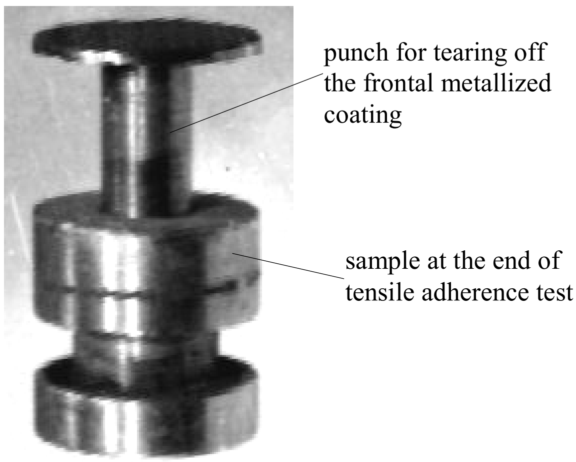


Fig. 5 Results of the adherence tensile testing

The main inconvenient is that it is very difficult to machine, by turning, the frontal side (face turning) of the sample, without damaging the whole multilayer structure.

The second method is that of shear testing, which is much more suitable for the mentioned goal of present research. The experiments samples are specially made ones, according to STAS 11684/4-83 specifications [8]. It means testing the adherence of thermal sprayed layers, by shearing failure of the coating.

A schematic representation of the test is shown in figure 6.

There have been obtained samples for each of the studied thermal sprayed materials. As the shape and dimensions of samples are specified by standard, there had to be machined the ring shape zones. The machining procedure used to do that was exterior cylindrical rough turning - very carefully performed.

Figure 7 presents a sample, before experimenting and figure 8 presents a damaged sample – where thermal sprayed coating is fallen off the basic substrate, because of bad turning.

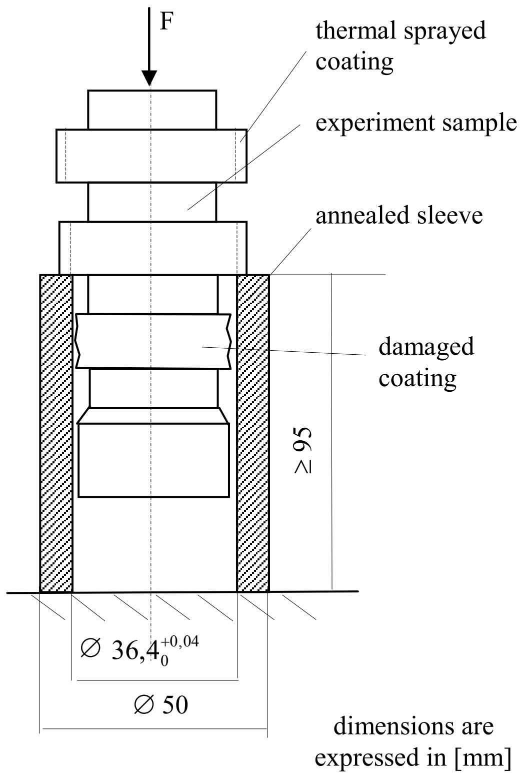


Fig. 6 Adherence test by shearing failure of the coating [STAS 11684/4 – 83]

Fig. 7 Experiments sample

Fig. 8 Experiments damaged sample

As for real experimenting stand, it is mentioned that it has been used a hydraulic press, WE-60 and, there have been designed and manufactured some special intermediate elements. These elements were really needed as, a well conducted experiment involves monitoring of, both applied compressive force's values and, of coating's displacement while failure occurs.

Fig. 9 Shear adherence test – for machined thermal sprayed coatings - detail

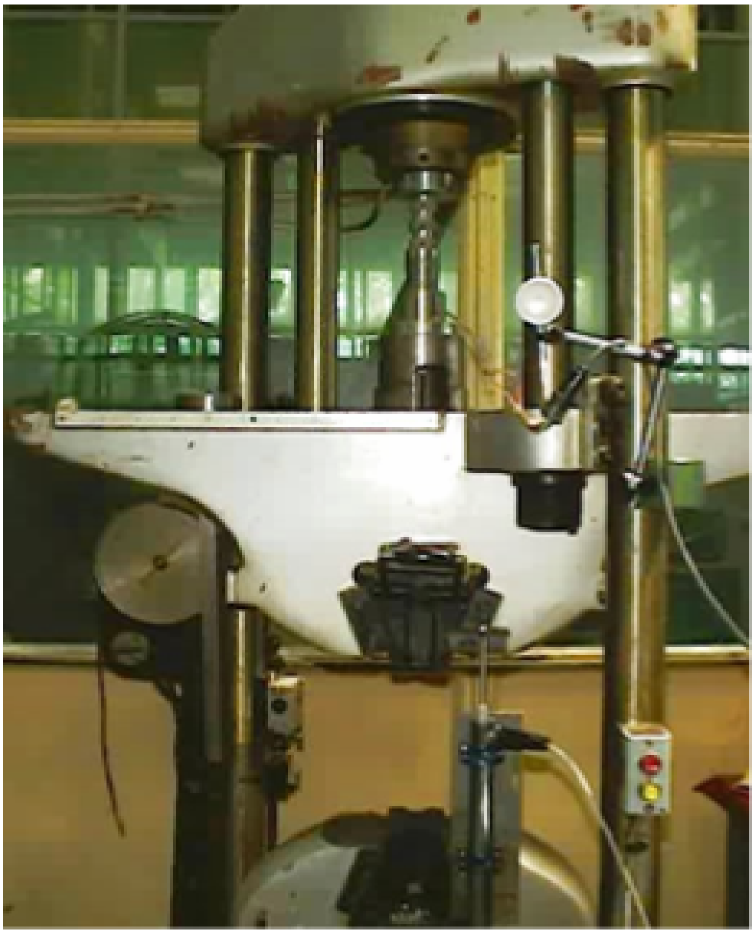


Fig. 9 Shear adherence test – for machined thermal sprayed coatings - continued

An image taken while experimenting is presented in figure 9.

In order to measure force's and displacements values , there were used *transducers – resistive* type, for force measuring and *inductive* type, for displacements measuring – see figure 9, both coupled to a voltage bridge.

Before starting experiments, the transducers were calibrated so:

- for the resistive ones, there have been known loadings and, the corresponding deformation, registered on bridge channel, noticed;
- for the inductive one, there have been known displacements (movements) of hydraulic press plateau and, the corresponding deformation, registered on other bridge channel, noticed.

Using a specific regression program [10], CurveExpert 1.3, there have been, thus, obtained the (linear) calibrating equations, as:

$$F = 58.8 \cdot \varepsilon \quad [\mathrm{N}] \qquad (1)$$

and, respectively,

$$L = 25 \cdot 10^{-3} \cdot \varepsilon \qquad (2)$$

Also, for complete, precise and more efficient measurements, a data acquisition system (AT-MIO-16L-9) was used, and LabVIEW soft-ware allowed the complete determination of studied values. Each experience involved 5,000 measurements/second, for 20 seconds each.

3 Experimental Results

As mentioned above, the target of this experimental research is to determine if the machining parameters do influence thermal sprayed coating adherence – to the basic substrate.

So, the machining procedure considered, was exterior cylindrical turning – based on the fact that most of metallized layers machining is done by turning and, also, that, even when checking adherence, the standard do mention exterior cylindrical thermal sprayed coatings.

The machining parameters values, meaning cutting speed, v; cutting feed, f and cutting depth, t are shown in table 3.

Table 3

Controllable inputs values

Material	v [m/min]		f [mm/rot]		t [mm.]	
Coded values						
	(-1)	(1)	(-1)	(1)	(-1)	(1)
Real values						
MET 4	10	30	0.1	0.2	0.3	0.6
Inox 18-8 / S12Mn2Si	23	67	0.2	0.315	0.25	0.5
Al-Ol	135	340	0.08	0.16	0.15	0.3
	Cutting tool nose radius, r = 0,4 mm Wear parameter of the cutting tool, VB = 0 mm					

To notice that, there are shown, both real and coded values of turning parameters, the coded ones being used in statistic regression calculus, if a dependence mathematical model of adherence variable, should be obtained [2].

When experimenting , on each sample there were three metallized coating zones (as seen in figure 7 and figure 9) but, turning was done only on two of the ring shape zones, the third being considered "witness" – for comparing adherence results.

Some of the experimentally obtained results are presented in Table 4, where:

$$p_a = \frac{F}{\pi D \cdot B} \quad [\text{N/mm}^2] \tag{3}$$

Table 4

Experimental results

Material	Specific elements	Experiments type - - machining parameters coded values combination -			
		reference	(-1; -1; -1)	(-1; -1; +1)	(+1; +1; +1)
MET 4	Force, when failing, F [× 10^4 N]	3.30	4.00	3.40	3.60
	Ring zone diameter, D [mm]	40.05	39.70	39.40	39.35
	Ring zone width, B [mm]	14.96	14.98	14.87	14.94
	Adherence, p_a [N/mm^2]	17.54	21.42	18.48	19.50
Inox 18-8	Force, when failing, F [× 10^4 N]	4.60	5.60	4.20	4.40
	Ring zone diameter, D [mm]	40.01	39.40	39.43	39.41
	Ring zone width, B [mm]	15.00	14.86	14.79	14.82
	Adherence, p_a [N/mm^2]	24.41	30.23	22.94	23.99
S12Mn2Si	Force, when failing, F [× 10^4 N]	4.20	5.20	4.50	4.80
	Ring zone diameter, D [mm]	4.000	39.68	39.40	39.42
	Ring zone width, B [mm]	14.99	14.83	14.76	14.98
	Adherence, p_a [N/mm^2]	22.31	28.14	24.64	25.89
Al-Ol	Force, when failing, F [× 10^4 N]	3.80	4.15	3.80	3.80
	Ring zone diameter, D [mm]	40.05	39.71	39.42	39.40
	Ring zone width, B [mm]	15.00	14.94	14.86	14.82
	Adherence, p_a [N/mm^2]	20.14	22.28	20.66	20.73

The LabVIEW data acquisition system allowed plotting the curves of, both, force and displacement time variation (for each of the studied thermal sprayed coatings).

Figure 10 shows the curves, when there is no cracking of the sprayed coating, only its failing after plastic yielding. The other possible situation, is that presented in figure 11, when, at some moment, the coating cracks and so, falls off the basic substrate.

One can notice the two failure types, depending on mechanical characteristics of sprayed material. But, for both situations, after passing over the part substrate, the failed coating's displacement speed increases.

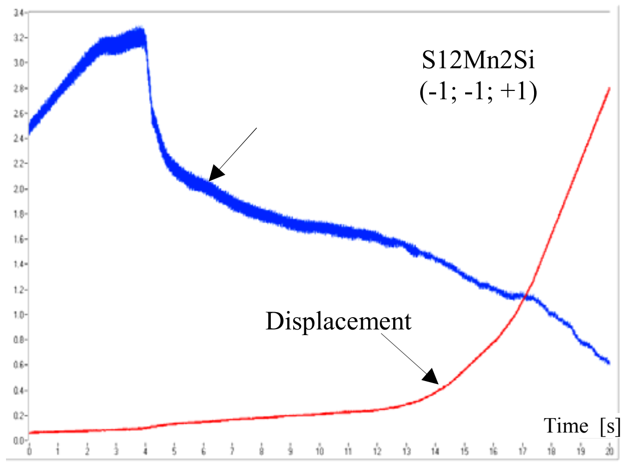


Fig. 10 Force and displacement graphs, as time variation – case 1

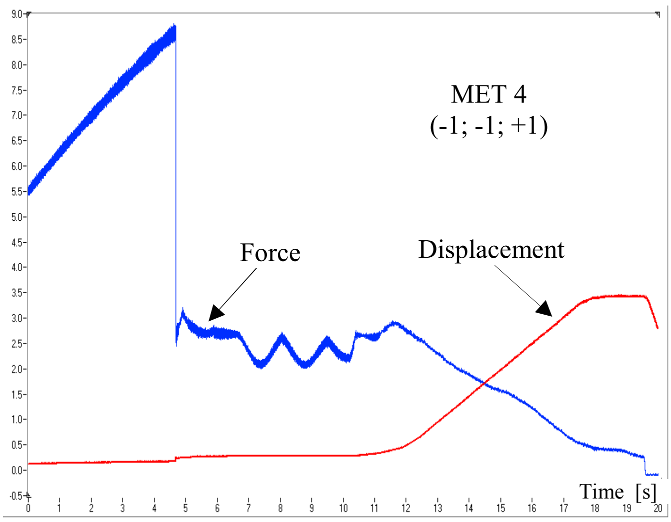


Fig. 11 Force and displacement graphs, as time variation – case 2

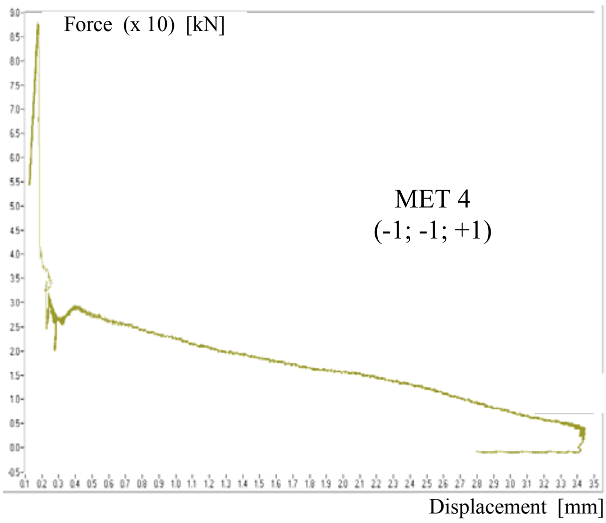


Fig. 12 Force variation, as dependence of coating's displacement

Also, by "combining" the signals (from the two transducers type), it was possible to obtain the force - displacement graph, that points out, the way compressive force varies, while the metallized coating is falling off the basic substrate. This graph is evidenced by figure 12.

As noticed, from the obtained results, the machining parameters do, hardly, affect metallized coating's adherence. Usually, its values are a little higher, than the initial ones (without machining) maybe, because of the internal stresses that do appear while turning.

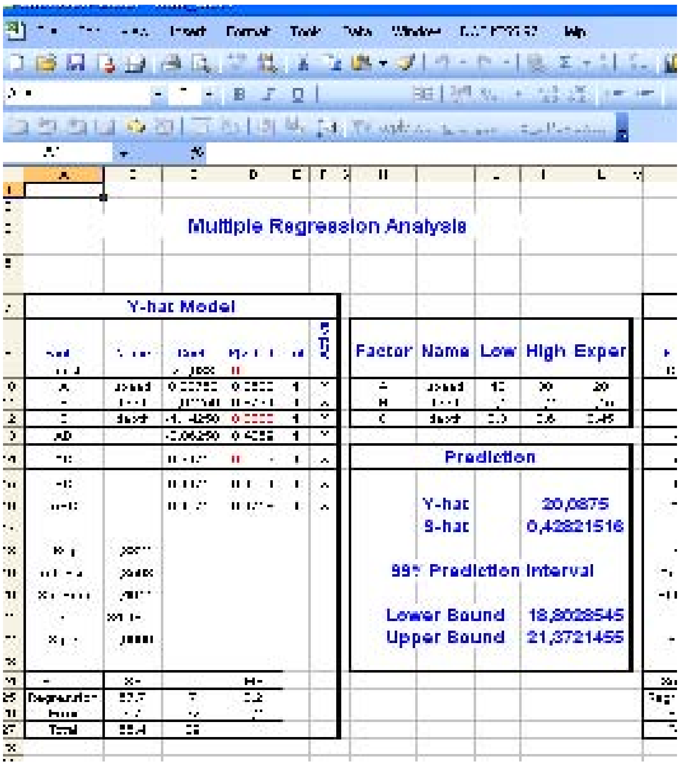
The experimental results of thermal sprayed coatings adherence study, induced the idea of finding a regression model, involving adherence values (as dependent variable) and machining parameters value (as independent variables) [3].

As, there have been noticed two types of force and displacement variation graphs, one for so called "mild materials" and the other for "hard materials", detailed study was carried on all the above considered materials – compared to their hardness values [10, 11].

The experiments designs were full factorial ones and all of the regression analyses were performed with a special software, called DOE KISS. This software enables the study of each factor's influence, as well as the factors interaction influence on the considered variable [6].

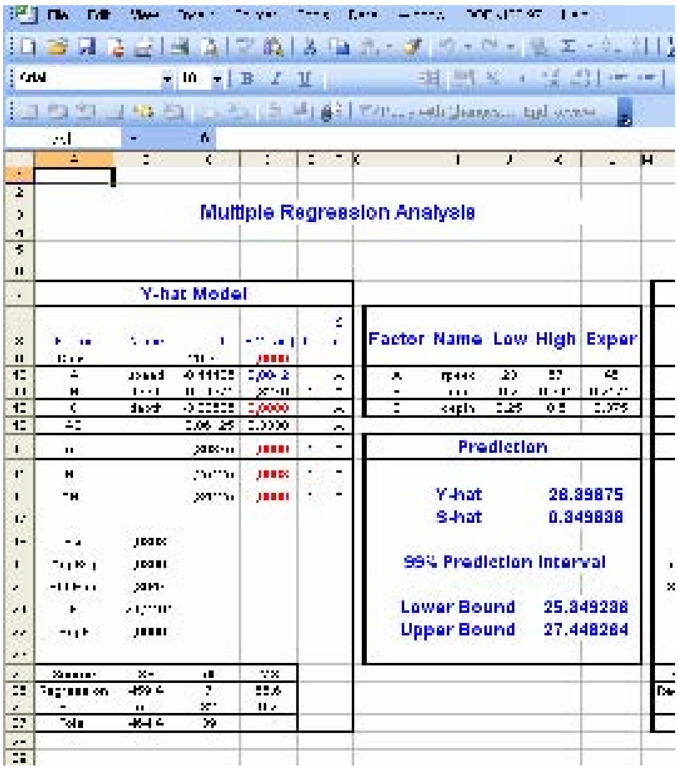
It's worth to be mentioned (for the obtained regression models) that one factor, or interaction, is considered to have significant influence on the "output" if, the corresponding value of P (2 tail) is smaller than 0.05.

So, DOE KISS regression analyses results are shown in figure 13.



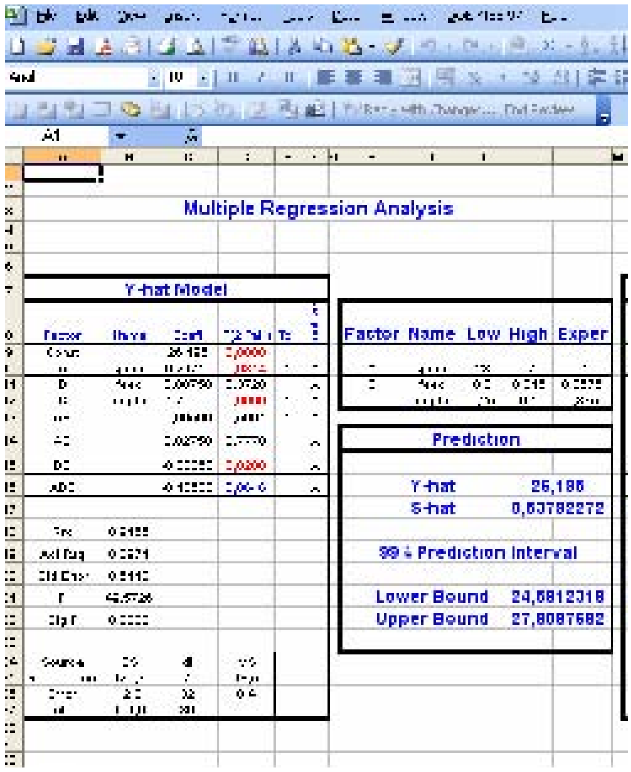
a.

DOE KISS – MET 4 thermal sprayed coating



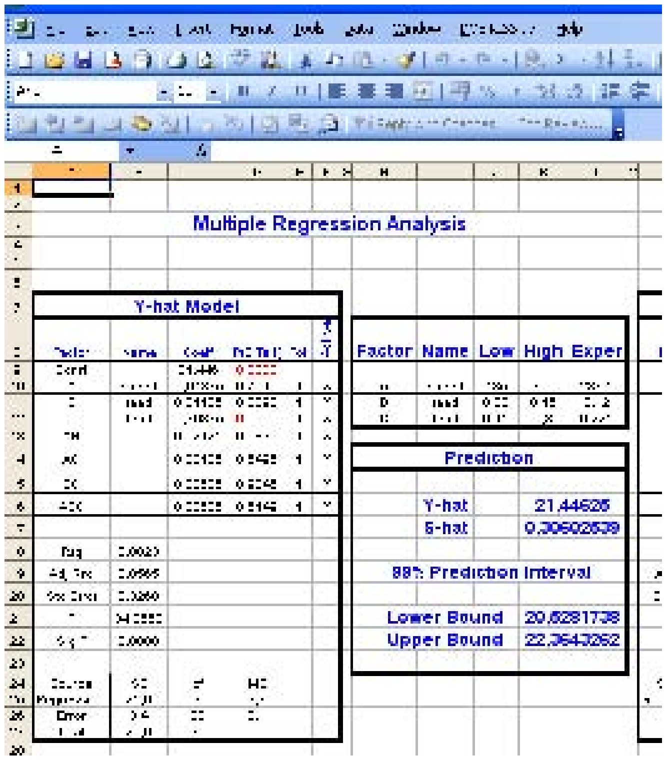
b.

DOE KISS – Inox 18-8 thermal sprayed coating



c.

DOE KISS – S12Mn2Si thermal sprayed coating



d.

DOE KISS – Al-Ol thermal sprayed coating

Fig. 13 Regression analysis results, with DOE KISS – software

So, considering only the significant factors, the obtained regression models were:

- for *MET4* metallized coating

$$Y = 20.088 - 1.1425x_3 + 0.3175x_1 \cdot x_3 \qquad (4)$$

where: Y is the adherence variable [N/mm^2];

$$x_1 = 2\frac{v - \frac{v_{min} + v_{max}}{2}}{v_{max} - v_{min}} = 2\frac{v - 20}{20};$$

$$x_3 = 2\frac{t - \frac{t_{min} + t_{max}}{2}}{t_{max} - t_{min}} = 2\frac{t - 0.45}{0.30}$$

.resulting:

$$Y \cong 25.421 - 3.383t - 0.096v + 0.218vt \qquad (5)$$

- for *Inox 18-8* metallized coating

$$Y = 26.399 - 3.336x_3 + 0.399x_1x_3 + \\ + 0.251x_2x_3 + 0.341x_1x_2x_3 \qquad (6)$$

where: Y is the adherence variable [N/mm^2];

$$x_1 = 2\frac{v - \frac{v_{min} + v_{max}}{2}}{v_{max} - v_{min}} = 2\frac{v - 45}{44};$$

$$x_2 = 2\frac{f - \frac{f_{min} + f_{max}}{2}}{f_{max} - f_{min}} = 2\frac{f - 0.2575}{0.115}$$

$$x_3 = 2\frac{t - \frac{t_{min} + t_{max}}{2}}{t_{max} - t_{min}} = 2\frac{t - 0.375}{0.25}$$

.resulting:

$$Y \cong 32.854 + 0.154v + 23.303f - \\ - 17.211t - 0.771vf - 62.143ft - \\ - 0.41vt + 2.157vft \qquad (7)$$

- for *S12Mn2Si* metallized coating

$$Y = 26.195 - 0.2175x_1 - 1.76x_3 - \\ - 0.2225x_2x_3 \qquad (8)$$

where: Y is the adherence variable [N/mm^2];

$$x_1 = 2\frac{v - \frac{v_{min} + v_{max}}{2}}{v_{max} - v_{min}} = 2\frac{v - 45}{44};$$

$$x_2 = 2\frac{f - \frac{f_{min} + f_{max}}{2}}{f_{max} - f_{min}} = 2\frac{f - 0.2575}{0.115}$$

$$x_3 = 2\frac{t - \frac{t_{min} + t_{max}}{2}}{t_{max} - t_{min}} = 2\frac{t - 0.375}{0.25}$$

.resulting:

$$Y \cong 28.936 - 0.01v + 11.609f - \\ - 6.109t - 30.957ft \qquad (9)$$

- for *Al-Ol* metallized coating

$$Y = 21.446 - 0.79875x_3 \qquad (10)$$

where: Y is the adherence variable [N/mm^2];

$$x_3 = 2\frac{t - \frac{t_{min} + t_{max}}{2}}{t_{max} - t_{min}} = 2\frac{t - 0.225}{0.15}$$

.resulting:

$$Y \cong 23.842 - 10.65t \qquad (11)$$

The DOE KISS software provides a Pareto Chart of coefficients – to points out how “strong” the influence of each independent variable, as well as its interactions, on the dependent variable is. So, it has been considered right to present this chart for two of the studied thermal sprayed materials, meaning for the ones with higher and, respectively, lower hardness values – see figure 14 and figure 15.

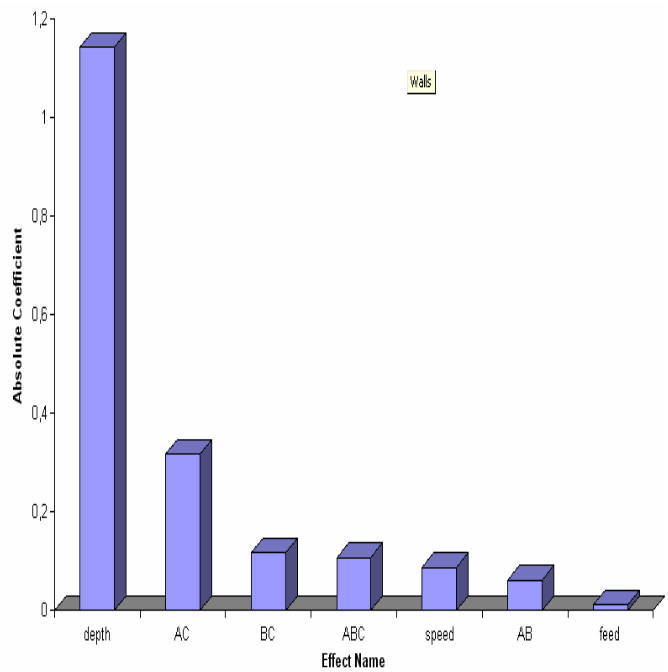


Fig. 14 Pareto Chart of regression coefficients – DOE KISS – software - MET 4 thermal sprayed coating

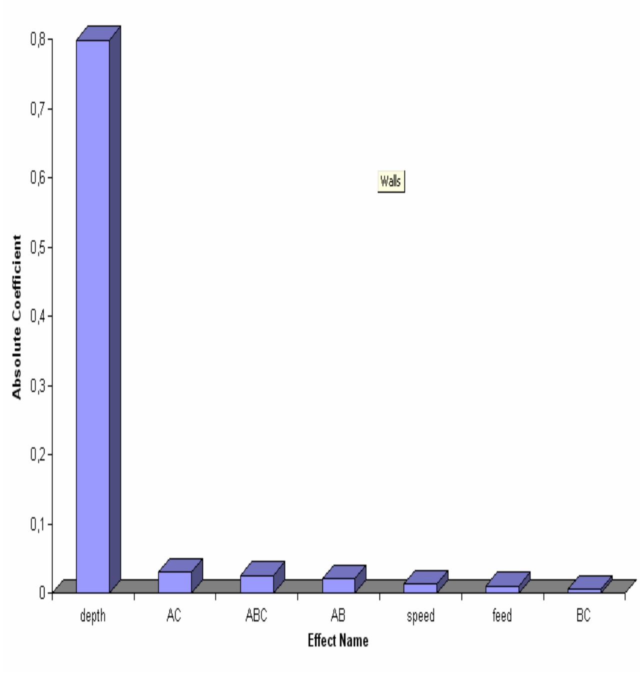


Fig. 15 Pareto Chart of regression coefficients – DOE KISS – software - Al-Ol thermal sprayed coating

4 Conclusion

Thermal sprayed coatings represent completely new materials (compared to the rough, initial ones) with very good mechanical characteristics – hardness, porosity, adherence, etc.

Many times, after metallizing, machining of the obtained coatings is necessary and, one important procedure is that of turning.

In order to study machining parameters influence on sprayed coating adherence there have been *designed and manufactured special elements*. All the experiments were carried out according to standardized conditions.

With two transducers type (fixed on the special elements) – a resistive and an inductive one, together with a data acquisition system, it was possible to plot the graphs of force (applied on the experimental samples) and displacement (of the tested metallized coating) variation, For "hard" materials, there was a "crack" of the coating while, for the "mild" materials, the coating just "slipped" down, with no sudden force variation.

Based on the experimentally obtained results, it has been considered right to find regression models, involving adherence values (as dependent variable) and machining parameters value (as independent variables).

The regression models determined, were adequate and, mainly, it was the cutting depth, t, parameter that did influence (but, not so high) the coating's adherence, when turning. Other factors with significant influence were cutting speed and cutting feed but, their influence was less than the one of cutting depth. Also, there is the interaction between studied variables that, sometimes, influence the coatings adherence.

Further research should be developed, in order to find if, there are any, more factors that could, possible, reduce or, even increase, thermal sprayed layers adherence to the basic substrate.

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