Optimization of Gritblasting Conditions by Supersonic Air Spraying

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Recent development of the HVAF-M3 system attracts a lot of interest in spraying Carbide-based and Iron-based materials as an economic, environmental and practical alternative to Electrolytic Hard Chrome (EHC) method, which is still widely utilized in the printing, automotive and off-shore industries. The use of compressed air instead of pure oxygen and the fact that gritblasting procedure can be operated with the HVAF gun itself offers economic advantages well perceived by the industry. A dedicated Design of Experiments on gritblasting parameters operated utilizing the HVAF-M3 system is proposed in the present work. Relationships between gritblasting media sizes – feed rate – offset – stand-off distance and substrate roughness profiles including grit residues level have been investigated. Additionally Carbide-based and Iron-based powder feedstock materials were HVAF-sprayed in order to study the influence of gritblasted conditions on their particular coating adhesion strength. Respective coating microstructure and adhesion strength are presented and discussed for further optimization of coating performances.

1 Introduction

In the field of wear, erosion and corrosion applications, recent restrictions in the use of carcinogenic hexavalent form of chrome element has driven the need of replacing Electrolyte Hard Chrome plating (EHC) by other material/process with equivalent tribological properties [1]. WC-based, Cr-based and Fe-based powders materials have been proposed as excellent candidates when processed with high kinetic spraying systems [2-4]. One of the latest low-temperature high-kinetic thermal spray processes, named as HVAF-M3 system, emerges as an interesting and promising alternative method to EHC and even to its predecessor HVOF systems [2]. The use of compressed air instead of pure oxygen and the fact that gritblasting procedure can be operated with the HVAF-M3 gun itself offers economic advantages well perceived by the industry. Learning from conventional spraying (APS and flame processes), substrate grit blasting is undertaken prior to spraying to generate an optimized roughness to promote mechanical anchoring of solidifying molten impacted particles [5, 6]. Similar to shot peening surface treatment, the grit blasting intensity is controlled by the nature and size of the grit media, its feed rate and the blasting exposure time [7, 8]. It is well described that an optimized resulting roughness is beneficial to APS coating adhesion strength, whereas it has not being systematically investigated for HVOF [7, 9] and even less for HVAF coatings. For those processes involving higher kinetic energies, semi-molten or fully solid particle impacts involve other bonding mechanisms, issued from solid state theories such as plastic deformation at high strain rates [10] and development of shear instabilities [11], and therefore the role of surface topography needs to be explored. Supersonic gritblasting processed by the HVAF-M3 system itself has been therefore investigated through a dedicated Design of Experiments. Influence of gritblasting conditions on surface topography and coating adhesion strength is given specific interests.

2 Experimental Procedure

2.1 Spray Process and Feedstock Materials

The novelty of utilizing the HVAF process itself to propel Alumina media for grit blasting procedure prior to spraying is an attractive economical argument for industries. Standard configuration of the HVAF-M3 system was utilized for gritblasting Domex355 substrates, i.e. operating the large combustion chamber, long nozzle and short axial powder injector. Three different sizes of mesh of Alumina media were used: 180 (-90+53) DURALUM White F180 (Washington Mills), 220 (-75+45) DURALUM White F220 (Washington Mills) and 260 (-45+22) AMDRY 6062 (Sulzer Metco). Mesh size, feed rate, offset angles and stand-off distance were selected as the main gritblasting parameters, also called factors, with designated levels (Tab.1). In order to study the influence of gritblasting parameters on several responses, either substrate surface properties or coating adhesion strength, two powder feedstock materials were selected to be HVAF-sprayed with identical hardware configuration: Cr_3C_2-NiCr 75/25 (H.C.Starck) and an iron-based powder (Höganäs AB).

2.2 Statistical Models

In this work, Design of Experiments (DoE) was used to establish relationships between gritblasting HVAF process parameters, grit residues, substrate roughness and coating adhesion strength. DoE is a standard statistical approach conventionally used to study relationships between process parameters and coating properties in thermal spray. The approach is usually a stepwise procedure starting with screening fractional or full factorial designs to response surface designs for optimization purposes. In this study, a full factorial design was selected since this design can gain valuable insight in how different process parameters interact on several responses such as substrate roughness and coating adhesion strength. It should be noted that quantification or discretization of all factors and responses is necessary when using DoE and that the results are dependent on the selected levels of the factors (Tab.1). The investigation was performed utilizing the statistical software MODDE ©, MKS Umetrics AB, Sweden. The full factorial screening design consisted of 19 experimental runs, performed in a random order to increase the model reliability, reproducibility and repeatability by including three centre points (runs 17-18-19), also called *replicates*. Multiple linear regression (MLR) was used to establish the relationships between the factors and the responses. The models were applied both for screening and prediction purposes. Separate MLR models were derived for each response variable to establish a best fit for the statistical representation of the significance of each factor and their interactions.

Tab.1. Design Matrix of Experiments

Run order	**Sample name**	**Mesh size**	**Feed rate (g/min)**	**SoD (mm)**	**Offset/ angle**
1	4	180	100	200	45
2	12	260	50	200	45
3	5	180	150	200	45
4	13	260	150	200	45
5	6	180	50	400	45
6	14	260	50	400	45
7	7	180	150	400	45
8	15	260	150	400	45
9	8	180	50	200	90
10	16	260	50	200	90
11	9	180	150	200	90
12	17	260	150	200	90
13	10	180	50	400	90
14	18	260	50	400	90
15	11	180	150	400	90
16	19	260	150	400	90
17	1	220	100	300	60
18	2	220	100	300	60
19	3	220	100	300	60

2.3 Characterization Methods

Grit residues: two different non-destructive spectroscopy techniques were utilized to quantify the level of grit residues present onto the substrate surfaces: (i) X-Ray Fluorescence (XRF) and (ii) Energy Dispersive X-ray (EDX). Both quantify elemental composition by respectively exciting the sample by high-energy x-ray with wide range of wavelengths (XRF), or using a direct electron beam (EDX). A third approach based on image thresholding algorithms utilizing the image analysis Aphelion ® software, was applied on 20 SEM pictures per samples, taken utilizing a TM3000-Tabletop Microscope (HITACHI).

Surface roughness: two different approaches were applied to evaluate gritblasted surface roughness: (i) 2D profilometry and (ii) 3D surface topography. For practical and economic issues, 2D profilometry was conventionally carried out to evaluate the arithmetical mean roughness (*Ra*) and Maximum peak height (*Ry*) over profile lines. 10 profile lines – 5 mm long each, were carried out per sample, utilizing Surftest-301 instrument (Mitutoyo). 3D surface topography was analysed by optical interference profiling method (Toponova AB, Sweden) to evaluate surface roughness parameters following ISO 25178 series. Scanning of three areas per sample – 2 x 2.5 mm2, was carried out to evaluate several 3D parameters (Tab.2). Motifs are here the key elements of the surface texture, and were discriminated by segmentation method using watershed algorithms, in order to evaluate the significant contours of pits and dales, and associated course and ridge lines (Fig.1). Two main parameters, peak density (*Spd*) and respective curvature (*Spc*) are given a particular interest.

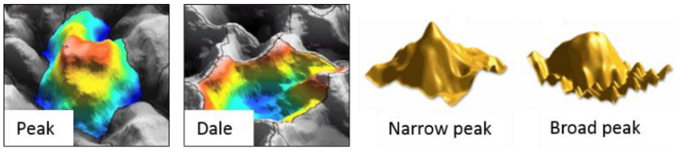

Fig.1 Definition of peak, dale and motifs curvature (Spc)

Micro Hardness Vickers: Micro-Vickers hardness measurements were carried out on the polished cross-section of the gritblasted substrates according to ASTM E384-10 with a Vickers indenter at a load of 10 g and dwell time of 15 seconds, using a Shimadzu Microhardness Tester. HV0.01 was calculated from averaging series of 20 impressions.

Coating adhesion strength: the standard polymer-based media FM1000 was substituted to a brazing media as an alternative method, earlier reported by the author [12] and optimized for Metallic-based alloys. Such induction brazing non-conventional method allows to evaluate adhesion strength above 90MPa, higher than ultimate strength than the standard FM1000 glue. Three samples per each gritblasting conditions were coated, brazed and tensile tested following ASTM C633-69 specifications, utilizing a Z100 universal tensile test machine (Zwick) up to 100 KN, at a load speed of 1.27 mm/min.

Tab.2. 2-D and 3-D roughness main parameters

Parameters		**Parameters description**
2D	**3D**	
Ra	*Sa*	Arithmetical mean height (μm)
Ry	*Sp*	Maximum peak height (μm)
Rz	*Sz*	Maximum peak-to-peak height (μm)
	Mn	Number of motifs
	My	Mean height of motifs (μm)
	Ms	Mean area of motifs (mm^2)
	Spd	Density of peaks ($1/mm^2$)
	Spc	Arithmetic mean peak curvature (1/mm)

3 Results and Discussions

3.1 Relationship gritblasting / grit residues

Limitations were encountered with image analysis and especially EDX quantifications to estimate the level of grit residues onto substrate surfaces. Relationships between gritblasting factors and grit residues has been here described by MLR models (Tab.3). The coefficients of linear regression R2 for the XRF method can be considered as very high compared to Image Analysis and especially EDX one with decreasing value respectively. Therefore the XRF method was selected in the following study. The effect of Offset, SoD and feedrate on grit residues level measured by XRF method is shown on contour plots (Fig.2). The grit residues level decreases with the highest amplitude of variation from 55% to 25%, with decreasing the spray angle from 90° to 45° (Fig.2-a). Lower amplitude of variation of grit residues level can be noticed for both other factors influences (Fig.2-b-c). Higher the Stand-off distance and lower the feed rate, lower the level of grit residues, whereas the mesh size of blasting media does not significantly affect its level. Offset and SoD factors show main significant influences on grit residues (Fig.2-d).

Tab.3. MLR models for grit residues responses

Coefficient	XRF	Image	EDX
Runs	19	19	19
R2	0,972	0,707	0.089
Q2	0,952	0,457	0.256
Residual	3,089	6,446	6.521
Confidence	95%	95%	95%

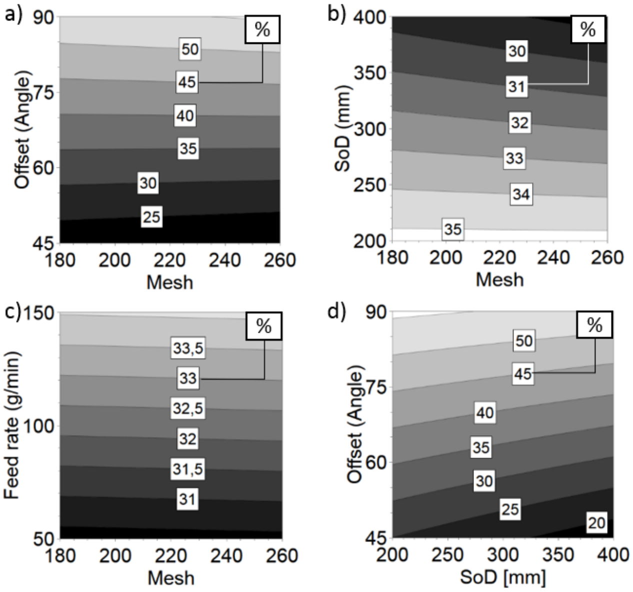

Fig.2 Contour plots of Grit Residues level measured by XRF method

3.2 Relationship gritblasting / surface roughness

When comparing conventional 2D parameters (*Ra, Ry*) to their 3D equivalent (*Sa, Sp*) to describe the surface roughness, both show similar influence on the mesh size. However the normal distributions of residuals show higher regression coefficients for the 3D parameters (*Sa, Sp*) than for the 2D ones (*Ra, Ry*), meaning that the 3D parameters are more significantly adapted than the 2D ones to describe the surface roughness response in this study. The Offset exhibit a lower significant influence on both selected responses, whereas SoD and Feed rate factors cannot weight any significant relations. Relationships between those factors and 3D motif features are here described by MLR models (Tab.4).

Tab.4. MLR models for grit residues responses

Coefficient	*Sa*	*Sp*	*My*	*Ms*	*Spd*	*Spc*
Runs	19	19	19	19	19	
R2	0,87	0,87	0,90	0,85	0,84	0,83
Q2	0,84	0,83	0,86	0,78	0,79	0,79
Residual	0,50	7,97	2,41	0,00	51,4	221,
Confidence	95%	95%	95%	95%	95%	95%

The main effect of Mesh size and Offset angle on respective 3D roughness responses is shown in on contour plots (Fig.3), displayed for respective medium values of Feed Rate (100 g/min) and SoD (300 mm) taken as the centre point values selected in this study. Increasing the Mesh size, meaning utilizing finer grit particles size, significantly reduces the roughness parameters *Sa* and *Sp*, as already learned performing conventional (*Ra, Ry*) 2D profilometry. However new precious information is here revealed thanks to the segmentation approach. Grit blasting with finer particles: (i) increase significantly the density of motifs, (ii) decreases significantly the motif height and area, and (iii) decreases significantly the motif curvature. Therefore finer grit media size introduces a more severe plastic deformation of the target surface, and generates significantly higher density of narrower peaks with reduced height. On the contrary, wider peaks, i.e. with larger radius of curvature, higher height and area, are generated by utilizing a coarser grit size (or finer mesh). It is quite interesting to notice that introducing the offset from 90° to a critical 45° angle has sensitively the same effect on those responses than coarsening the grit size, but necessarily for different reasons since the grit particle momentum transfer to the target surface is modified from fully normal (90°) toward half-tangential (45°), introducing shearing forces as well as elastic deformations, compared to pure peening actions in the normal direction. Those phenomena can therefore been investigated studying the next responses: micro hardness of respective targeted surfaces.

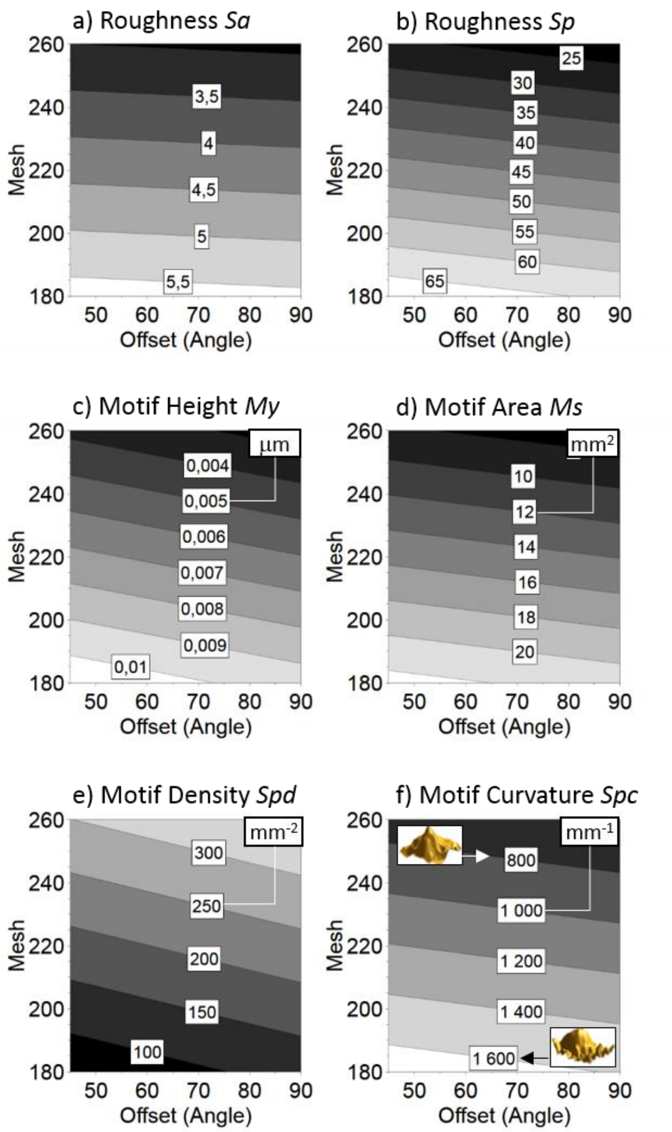

Fig.3 Contour plots of 3D roughness responses evaluated by segmentation method

3.3 Relationship gritblasting / micro hardness

Relationships between factors and the micro hardness response have been investigated by running a MLR model, which shows the significant and decreasing contribution of each factor, respectively Mesh, SoD, Offset and Feedrate, and their respective non-significant interactions. A general but strong trend shows that decreasing the grit media size and increasing the SoD, leads to a significant decrease of the micro hardness Vickers values of the targeted surface (Fig.4). An increase in grit media feed rate from 50 to 150 g/min significantly generates a 5% increase in hardness, whereas a decrease in offset angle from 90° toward critical 45° leads to a 5% decrease in hardness values.

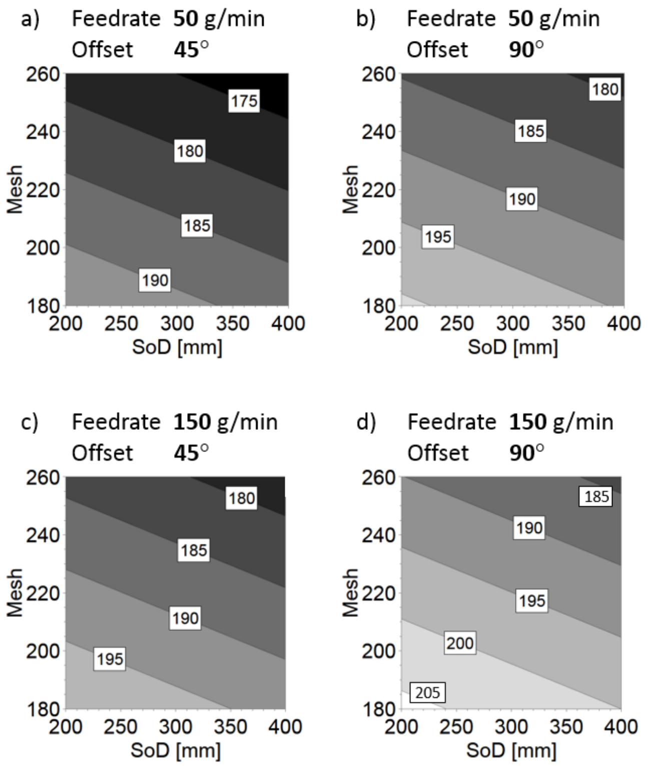

Fig.4 Contour plots of micro hardness values HV0.01

3.4 Relationship gritblasting / coating adhesion

Cr_3C_2-NiCr coatings: Independently of the gritblasting conditions, the Cr_3C_2-NiCr coatings exhibited adhesion strength higher than 200 MPa, exceeding the limit of the tensile test equipment. All samples were pulled up to 100 KN, without breaking. Therefore respective adhesion strength could not be measured even utilizing the specially developed alternative method to the standard ASTM C633-69, the latest utilizing a polymer-based media with limited strength to 85 MPa [12]. Even though no correlations to gritblasting conditions could be drawn in this specific case, the results that HVAF-sprayed Cr_3C_2-NiCr coatings exhibit extremely high adhesion strength up to 200 MPa, and thus independently of the gritblasting conditions, is a quite brilliant outcomes for wear and corrosion industrial applications.

Fe-based coatings: In order to study the effect of gritblasting conditions on coating adhesion strength, Fe-based materials were HVAF-sprayed with standard spray parameters. Their respective adhesion strength was successfully quantified between 110 MPa and 150 MPa, utilizing the specially developed brazing alternative method (Fig.5). First of all, those results highlight (i) the feasibility of the brazing alternative method to measure Fe-based thermal sprayed coatings, and (ii) that the Fe-based coatings sprayed with the HVAF-M3 system exhibits extremely high adhesion strength, up to 160 MPa. For such a response, MLR model shows significant coefficients for two main factors: Mesh size and SoD, and their interaction, whose non-linearity is highlighted in the contour plot (Fig.5). Decreasing the grit media size and increasing the stand-off distance has a positive effect in improving the Fe-based coating adhesion strength. This result is a valuable information if, for instance, gritblasting with fine grit presents economical restriction. Therefore coarsening the grit media size, which might work against strengthening the coating adhesion, can be compensated by increasing the SoD in order to get similar adhesion strength than the one obtained with a finer blasting media.

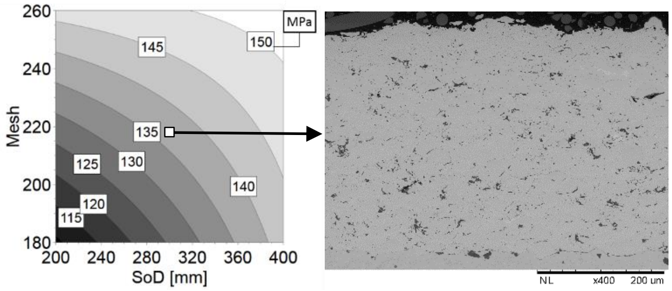

Fig.5 Contour plots of Fe-coating adhesion strength and SEM micrograph of coating cross section (centre point)

3.5 Discussion

Main relationships between the most relevant factors and most relevant responses, have been here described by Multi Linear Regression models through screening objectives. Several points can be here highlighted:

- The Grit Residues parameter shows a strong dependence on the offset angle (Fig. 2-d), as it could have been expected as well as on the SoD, but it is not significantly dependent on the Mesh size (Fig.6-a). Therefore it can be concluded that the adhesion strength of Fe-based coatings, evaluated at room temperature, is independent of the level of grit residues present at the coating/substrate interface. Presence of alumina at the interface could generate thermal stresses if high temperature applications are goaled, due to CTE mismatch. If it is the case for instance, the results show that grit residues level can be significantly reduced from 55% to 25% by decreasing the Offset angle from 90° to 45°, and this without influencing the coating adhesion strength.
- The Micro hardness response shows a strong dependence on both Mesh size and SoD, as the coating adhesion strength response. Therefore a direct cause/effect relationship can be concluded: lower the surface micro-hardness, higher the adhesion strength. Considering the semi-molten state of Fe-based particles impinging the targeted Fe-based surface at supersonic velocities, local plastic deformations might occur. Therefore softer surface with lower hardness might absorb those induced compressive stresses, resulting in a more favourable stress configuration at the interface, which has been studied earlier in the case of HVOF coatings [9]. Independently of the Feed rate, decreasing the Offset angle significantly reduces the surface hardness, which is favourable both to improve the adhesion strength and to decrease the grit residues level.
- The 3D roughness parameters show a strong and significant dependence of Mesh size and Offset angle, but neither on the SoD nor the Feed rate. This means that plotting each of them as Mesh vs. SoD (as for the adhesion strength response), instead of Mesh vs. Offset as previously displayed (Fig.3-a-e-f), will help our understanding. Each respective parameter show that when decreasing the Grit size (equivalent to increasing the Mesh size), (i) the roughness (*Sa)* decreases from 5 to 3 microns (Fig.6-b), (ii) the Motif density (*Spd*) increases from 100 to 300 peaks/mm^2(Fig.6-c, and (iii) the Motif curvature (*Spc*) decreases from 1600 to 800 mm^{-1} (Fig.6-d).

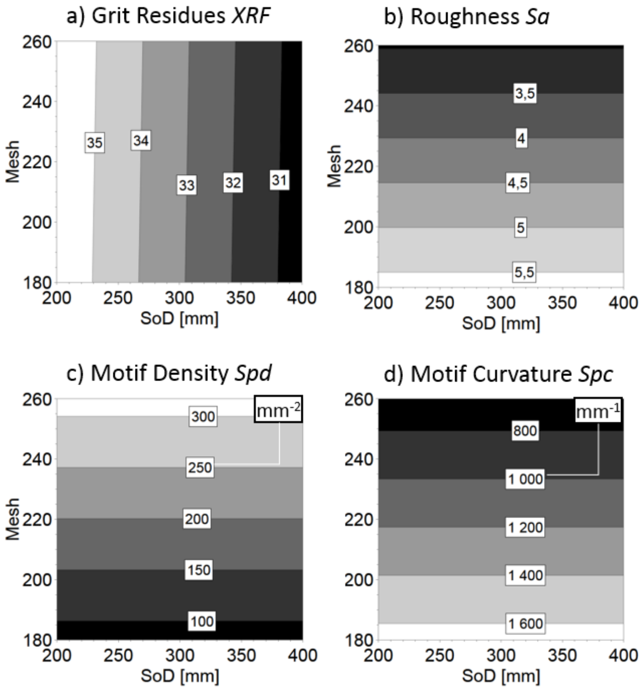

Fig.6 Mesh vs. SoD contour plots of Grit Residues and 3D roughness parameters – Summary

4 Conclusion

The practicability of utilizing the HVAF-M3 spraying system itself to operate fully automatized gritblasting surface preparation prior to spraying feedstock material, has been demonstrated in this work. Relationships between gritblasting parameters, surface hardness and topography, and resulting coating adhesion strength performances were developed in this study. The main results can be summarized as followed:

- Relationships between gritblasting conditions and grit residues level on targeted surfaces were derived: the lower the offset angle, and the higher the SoD, the lower the grit residues level.
- Clear relationships between gritblasting parameters and micro hardness of targeted surfaces were as well developed: the finer the grit particles, and the higher the SoD, the lower the surface micro hardness. Moreover the lower the feed rate, and the lower the offset angle, the lower the surface micro hardness.
- 3D-topography analysis show that decreasing the grit size significantly (i) increases the motif density, (ii) decreases the peak curvature and (iii) decreases the resulting surface roughness.
- Adhesion strength of Fe-based HVAFM3-sprayed coating was successfully evaluated by developing a specially designed brazing alternative to the standard ASTM C633-79. Multi-linear regression models show that the higher the density of narrower peaks of lower roughness, the higher the coating adhesion strength.
- Last but not least, such process map designed through a screening objectives integrating three centre points, highlights the repeatability, reproducibility and reliability of running a fully-automatized gritblasting operation using the HVAF-M3 system. All costs deriving from the use of an additional grit-blasting device such as installation, maintenance, operators, housing space and handling time are removed, besides increasing productivity and quality of the products.

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