

FUNCTIONAL AND FRACTAL PROPERTIES OF Fe-Al COATINGS AFTER GAS DETONATION SPRAYING (GDS)

The paper presents the results and provides an analyse of the geometric structure of Fe-Al protective coatings, gas-treated under specified GDS conditions. The analysis of the surface topography was conducted on the basis of the results obtained from the SEM data. Topographic images were converted to three-dimensional maps, scaling the registered amplitude coordinates of specific gray levels to the relative range of 0÷1. This allowed us to assess the degree of surface development by determining the fractal dimension. At the same time, the generated three-dimensional spectra of the autocorrelation function enabled the researchers to determine the autocorrelation length (S_{at}) and the degree of anisotropy (S_{tr}) of the surfaces, in accordance with ISO 25178. Furthermore, the reconstructed three-dimensional images of the topography allowed us to evaluate the functional properties of the studied surfaces based on the Abbott-Firestone curve (A-F), also known as the bearing area curve. The ordinate describing the height of the profile was replaced by the percentage of surface amplitude in this method, so in effect the shares of the height of the three-dimensional topographic map profiles of various load-bearing properties were determined. In this way, both the relative height of peaks, core and recesses as well as their percentages were subsequently established.

Keywords: Fe-Al, intermetallic alloys, GDS, SEM, fractal analysis

1. Introduction

The intermetallic alloys based on ordered intermetallic phases from Ti-Al, Ni-Al and Fe-Al equilibrium systems constitute the group of innovative and advanced construction materials, which have been introduced successively for industrial applications over the last thirty years. Until the 1970s, these alloys were considered to be useful functional materials displaying specific functional properties or seen as dispersed phases in conventional alloys designed for application at elevated temperatures [1]. The unique properties of this specific group of materials, especially their high temperature corrosion resistance (thermal resistance) in aggressive sulphide and chloride environments, potentially creates the possibility of using them as heat-resistant engineering materials. Unfortunately, through the prism of their application and common use, they exhibit high brittleness at ambient temperature and several difficulties associated with the process of producing homogeneous fine-textured alloys without structural defects [2,3].

The growing desire to improve the plastic properties of intermetallic alloys caused that many scientific studies and research centers began to focus on the thorough understanding of the mechanisms responsible for the susceptibility to plastic deformation and brittle cracking of the most interesting intermetallic alloys from the Ti-Al, Ni-Al, Fe-Al equilibrium systems, as well as Nb-Al and Mo-Si [4]. The result of this research, mainly carried out for the purposes of the aerospace, energy and

mining industries, was the commercialization of several alloys, mainly on the phase matrix from Ni-Al and Ti-Al equilibrium systems. The implementation-related processes are definitely less cumbersome when it comes to Fe-Al alloys, in which case the commercialization requires the solution of several problems, related to their low plasticity, tendency to brittle cracking at room temperature and low resistance to creep at elevated temperature. Despite these notable disadvantages, Fe-Al intermetals display a number of highly beneficial properties, especially during high-temperature oxidation of FeAl and Fe₃Al alloys in aggressive sulphide and chloride environments, which predisposes them to be particularly attractive construction materials in the thermal energy sector. The most important properties predestinating these alloys for industrial applications include inter alia [2,3]:

- excellent resistance to oxidation;
- structural and chemical stability at elevated temperatures;
- spontaneous formation of the Al₂O₃ passive layer on the Surface of the material, chemically resistant to arduous environments, hence their outstanding corrosion resistance at both ambient and elevated temperatures;
- low density (approx. 5,5 g/m³) – about 2/3 of the density of the Ni-based superalloys;
- high strength and stiffness;
- relatively high melting point;
- values of the coefficient of thermal expansion comparable to austenitic steels.

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Their relatively low price as compared to other groups of heat-resistant materials is also of paramount importance, with a lower relative density.

Favourable functional properties, especially these designated for working at elevated temperatures, paved the path for unconventional technological solutions used in surface engineering. Conventional manufacturing methods, such as melting or casting, in the case of Fe-Al intermetals are often deemed insufficient. The resulting „gap“ is filled with sinters using technologies based on powder metallurgy and in the form of thermally sprayed protective coatings, including the gas detonation method (GDS) [5]. Even though the method itself has been successfully practiced for many years, the technological conditions concerning the supersonic flow of two-phase (gas-powder) metallization stream and the creation of layered coating structures involving oxide phases formed in-situ during the gas detonation processes are still in the realm of extensive research [6]. This is particularly important in the case of brittle intermetallic phases from the Fe-Al equilibrium system, considering their limited susceptibility to plastic deformation, which manifests very high resistance to abrasive wear – the phenomena additionally increased by the participation of Al₂O₃ oxide ceramics and other oxide spinels.

The GDS technology reposes upon a number of complex physicochemical processes, dependent on many factors, the regulation of which allows to influence the spraying process course and thus the functional and fractal properties of cyclically formed coating and its structure at a frequency of approximately 6 Hz [7]. During the transport of the powder charge in the stream of hot products of gas detonation, the structure, morphology, chemical and phase composition of individual particles may undergo phase changes or be subject to processes such as softening, melting or premelting when impacted with hard ground, which causes a significant impact on the surface topography and the fractal properties of the coating. Some modifications in the particles morphology are already described in the paper [8]. According to the mentioned paper the particles are melted due to the gas detonation and subjected to partial solidification in the contact with air. The solidification of liquid particles occurs in two stages:

- the formation of the cellular structure of the particle coating when it is exposed to surrounding air and gas atmosphere; the inner surface of the solid coating is evened due to cutting of the cell tips by the rotating droplets and,
- rapid (amorphous) solidification of the droplets; jammed in the solid coating or released by the mother-particle through the punctures made in the solid coating; the droplets remaining inside the solid coating are subjected to slower solidification.

The aim of this manuscript is to analyze the results of the research on the geometric structure of Fe-Al protective coatings sprayed by means of the GDS technique under specific conditions in the context of their application in the thermal energy sector, underlying the significance of tribological and erosive-abrasive wear.

2. Materials and methods

The tests were applied to the detonation sprayed coatings obtained from the phase powder of the following composition: Fe40Al10.05Zr at.% + 50 ppm B, and granulation of 5-45 μm. The substrate material consisted of 15HM carbon steel plates with dimensions of 50×50×5 mm. The coatings were sprayed at the Paton Institute of Electric Welding using the „Perun S“ detonation system. Sprayed coatings in the form of a circular deposit with a diameter of approx. 25 mm were obtained at the stationary position of the ground and the barrel (without their mutual displacement in a plane perpendicular to the axis of the stream of hot detonation products. Two coatings, a and b, were tested (Fig. 1), and obtained respectively after: a – 100 and b – 400 shots of the detonation gun at a frequency of 6.66 Hz. The applied coating spray parameters are shown in Table 1.

TABLE 1

Applied parameters of the GDS process

Powder	Fe40Al10.05Zr at.%+50ppm B	
Size of the particles	5-45 μm	
Airflow	0.4 m ³ /h	
Composition of the detonation mixture	C ₃ H ₈ – 0.45 m ³ /h O ₂ – 1.52 m ³ /h Air – 0.65 m ³ /h	
Frequency of spraying	6.66 Hz	
Distance	110 mm	
Shots available	100	400
Thickness of the coatings obtained	0.79 mm	2.64 mm
Shots fired	100	400

3. Characteristics of the geometrical structure and the functional properties of coatings

The geometric surface structure (SGP) of the deposited FeAl (GDS) coatings was characterized based on the results of scanning electron microscopy SEM, using a Quanta 3D FEG (FEI) microscope for this purpose. The analysis was based on the equation describing the maps of the autocorrelation function $R(\tau)$. This procedure was successfully applied, among others, in the works [9-11].

$$R(\tau_x, \tau_y) = \frac{1}{S_g^2} \langle (z(x, y) - \langle z \rangle) \cdot (z(x + \tau_x, y + \tau_y) - \langle z \rangle) \rangle \quad (1)$$

where:

- S_g – root mean square (RMS) of gray level;
- $z(x, y)$ – pixel intensity value of (x, y) coordinates;
- τ_x, τ_y – distances between analyzed pixels;
- $\langle z \rangle$ – mean value of gray level;
- $\langle \rangle$ – mean value.

The spatial distribution of the spectrum $R(\tau)$ provides information on the surface directivity and its two-dimensional waveforms in the main directions of anisotropy allow us to

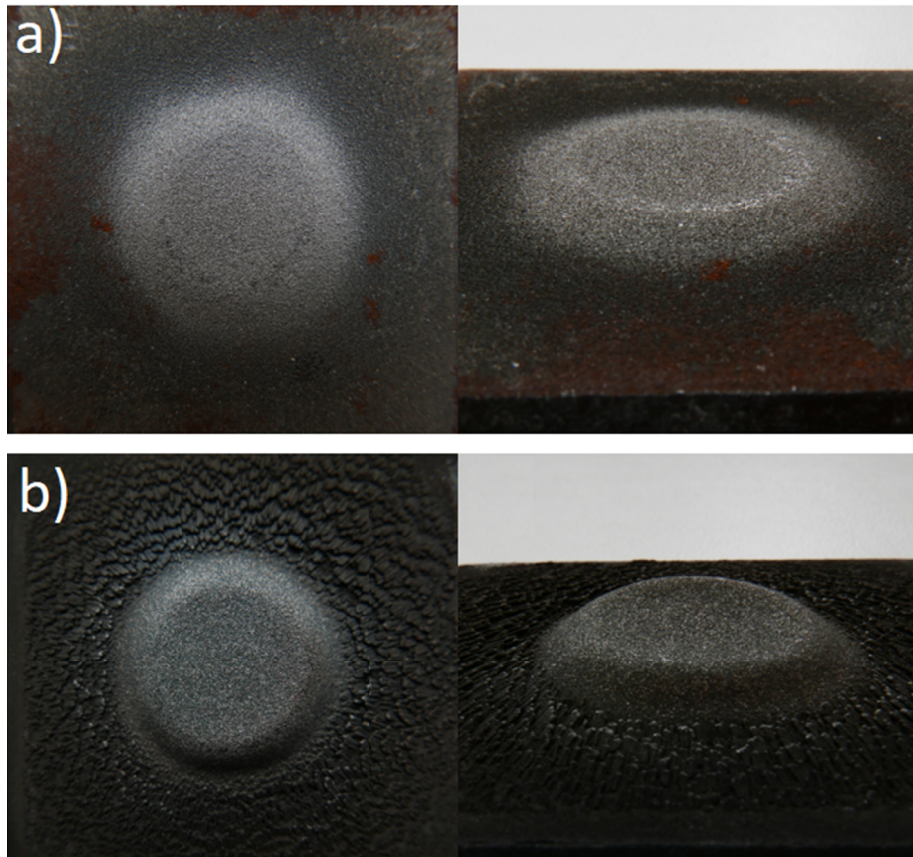


Fig. 1. FeAl sprayed coatings after: a) 100 and b) 400 – shots fired from the gun

determine the numerical value of the SGP shape factor [12-14], according to the equation (2).

$$S_{tr} = \frac{\text{Min} \left(\left[\frac{R(\tau)}{S_q^2} \rightarrow s \right] \right)}{\text{Max} \left(\left[\frac{R(\tau)}{S_q^2} \rightarrow s \right] \right)} \quad (2)$$

where s – numeric value within the range of 0-1 (in compliance with the recommendations of ISO 25178-3: 2012, the value set at $s = 0.2$ was assumed) [15].

The S_{tr} value is corresponding to the degree of SGP anisotropy and ranges from 0 to 1. The surfaces for which S_{tr} values greater than 0.5 are assumed tend to be considered as 'more or less' isotropic surfaces, whereas the surfaces characterized by indices lower than 0.5 are known as anisotropic. However, if the value of S_{tr} exceeds 0.3, extensive (high-frequency) surface directivity can be observed [13, 14].

The function $R(\tau)$ is related to the structure function $S(\tau)$ and the aforementioned dependence is determined by the equation (3). The structure function ($S(\tau)$) can be determined based on the AF function waveforms, according to the relation (3). The function $S(\tau)$ represents the vertical relations between the points on the surface, often referred to in the literature as a "height-height" function [16, 17].

$$S(\tau) = 2S_q^2(1 - R(\tau)) \quad (3)$$

The $S(\tau)$ curve, plotted in the logarithmic coordinate system (log-log plot), assumes a characteristic course, which displays two distinguished areas – the first one resembling a growing power function and the second one changing asymptotically to achieve values equal to the surface variance. This leads to finding the so-called "correlation length τ_c ", which defines the maximum distance between two points sharing the correlation of the height they are positioned at. The tangent of the slope angle of the curve $S(\tau)$ is directly related to the fractal dimension (D_{AF}), whereas the slope factor is connected with topothesia (Λ). The relation between fractal parameters and the structure function is described by equation (4).

$$S(\tau) = \Lambda^{2(D_{AF}-1)} \tau^{2(2-D_{AF})} \quad (4)$$

Topothesia (Λ) defines the distance between two points on the surface, between which the angular distance is 1 radian. The D_{AF} dimension ranges from 2 to 3, whereas 2 corresponds to the plane and 3 to surfaces deemed as extremely developed.

In order to characterize the functional properties of the surfaces, modified forms of the Abbott-Firestone curves were used. The modification applied consisted in replacing the actual surface amplitude of the A-F curves with the relative height in reference to gray levels. In this way, the brightest pixel assumed 0% and marked the largest height of the surface, while the darkest indicated the largest hollow, in which the material share was 100%. The modification of the methodology described in [12] allowed to determine the relative heights of: peaks (S_{pk}), core (S_k)

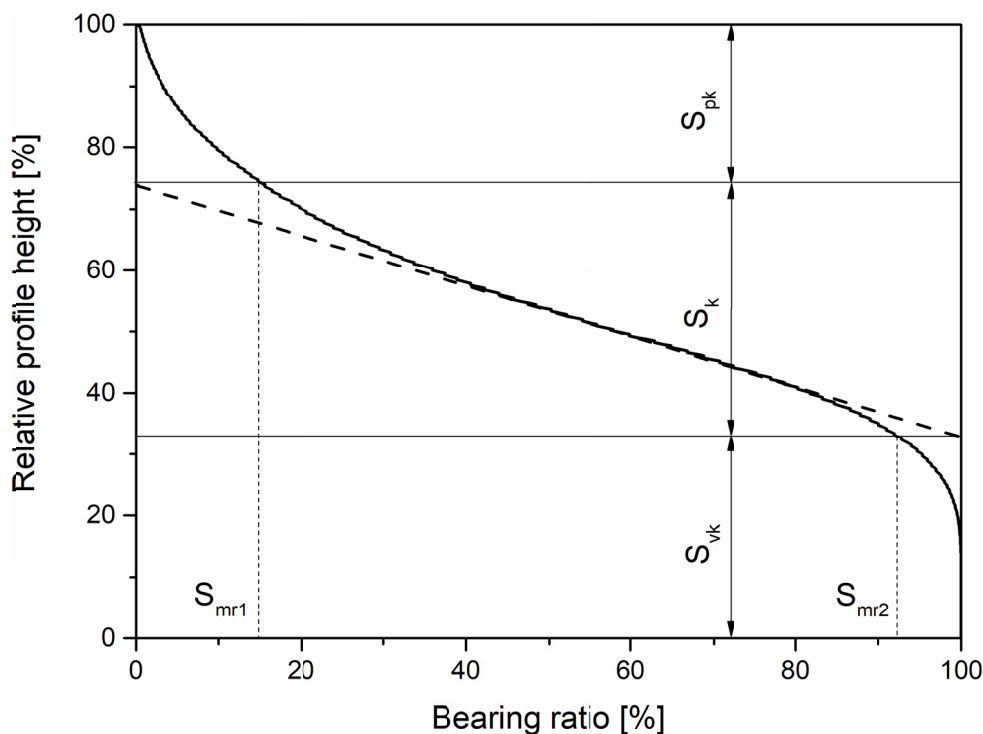


Fig. 2. Graphic representation of the method of determining functional parameters based on the Abbott-Firestone curves

and hollows (S_{vk}) and respectively their percentage shares (S_{mr1} and S_{mr2}). The method of determining the values of functional parameters is presented in Fig. 2.

4. Results and discussion

The set of factors influencing the geometric structure of FeAl (GDS) coatings described above was characterized on the basis of the results of the SEM/EDS research on morphology of grains displaying various shades of gray in the EDS detector. The tests were carried out in specific zones on the surface of the coating at a distance of approximately 12.5 mm from the center of the coating deposit with a diameter of approx. 25 mm. In the analysis of the obtained results, the SEM/EDS images reflected the signals emitted from the surface of the coating under the influence of the electron beam bombardment (Electron-beam physical vapor deposition) directed at the zonally distributed grains on the FeAl phase matrix. These grains, in the form of splats, are formed of very hot FeAl powder particles with various degrees of plastic deformation, which decreases on the periphery of the created coating deposit due to the dispersion of particles in the stream of gaseous detonation products along the length from the barrel's axis. Such an occurrence is the result of a slightly lower degree of heating of the particles on the periphery of the gas stream due to the radiation phenomenon in the barrel of the GDS water-cooled gun, as well as a lower velocity of the dispersed particles. In addition, the dispersed particles collide at a certain angle, which for a spray distance of 110 mm is set at approx. 4°. As a consequence, this causes a smaller impact area when the particles collide with the metallic substrate and affects

the tendency to anchor in the coating of larger size particles without their required plastic deformation, as evidenced by the results of own research (Fig. 3 c, g).

All the analyzes were carried out at a constant magnification of 500×, which enabled the reconstruction of rectangular areas with dimensions of 580×500 μm. Exemplary SEM surface topography images of the deposited coatings in specific zones are shown in Fig. 3.

The interpretation of topographic surface images based merely on the morphology and basic stereometric measurements is in fact an arduous task, often deemed impossible. According to Mainash, any interference with the material leaves a unique "fingerprint" on its surface [13]. Therefore, one should look for a correlation between production parameters, methods of shaping the surface layer, degradation processes, etc., and SGP [18-21].

Two characteristic and morphologically distinct types of areas were observed, as can be seen on the surface of each sample. They differ in such respect as the degree of non-dilatational strain deformation, which the powder charge is subjected to. Fig. 3c and Fig. 3g, obtained on the periphery of the coating deposited following either 100 shots (Fig. 3c) or 400 GDS shots (Fig. 3g), depict distinct spheroidal grains, which manifest a small degree of plastic deformation. The diameters of the grains, which were not subject to non-dilatational strain, oscillate around 45 μm and, hence, correspond to the thickest fraction of the charge. Fig. 3a and Fig. 3e show morphologically homogeneous surface topographies of the coatings in the central zone, within which the FeAl powder particles underwent a very extensive non-dilatational strain. In effect, this lead to the creation of distinct splats occurring in the form of clusters, involving local elevations conditioned by the heterogeneity of the plastic deformation of

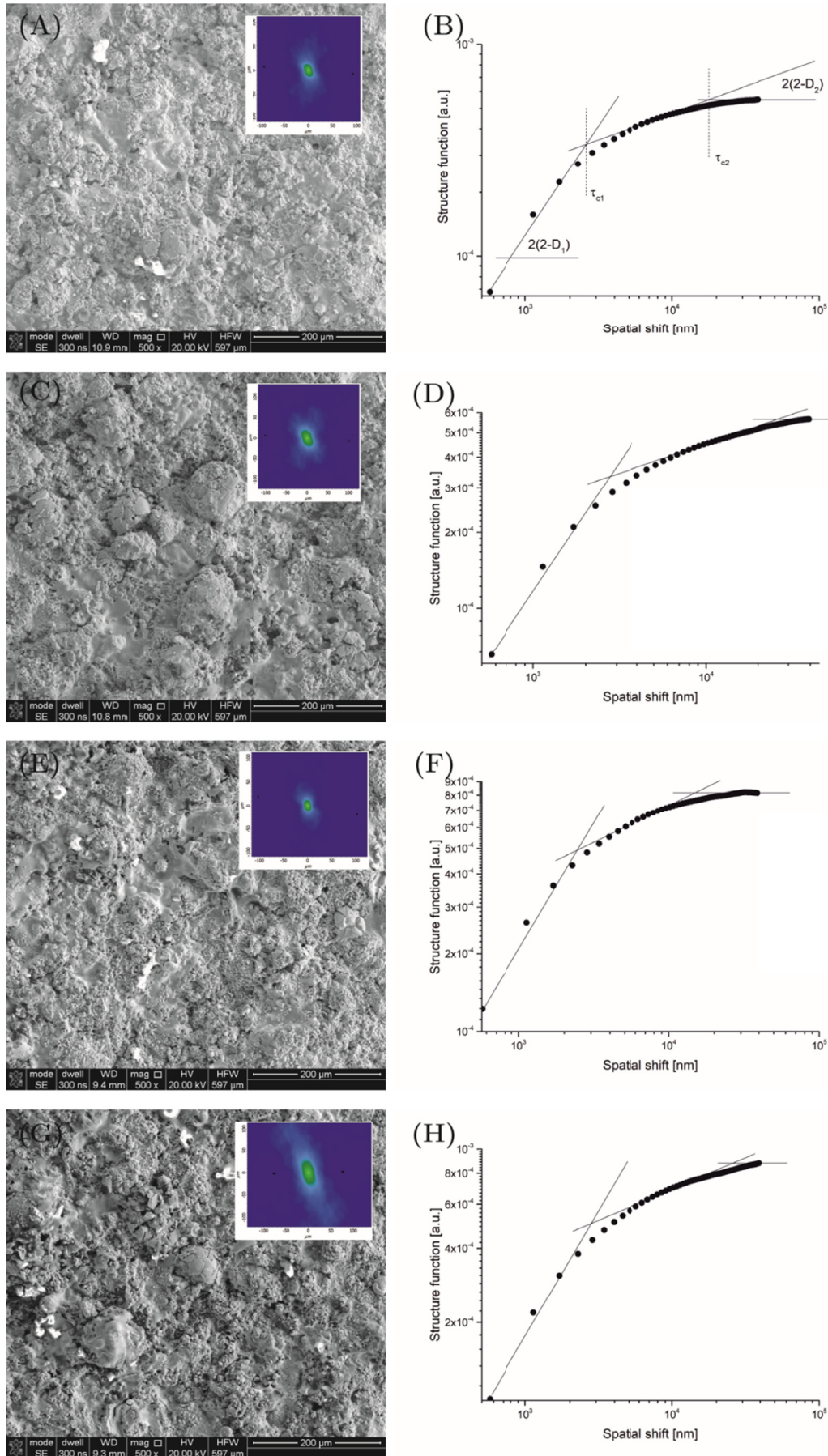


Fig. 3. SEM images of the topography of the layers (left column) along with their corresponding structure function (right column). (A–D) after 100 shots, (E–H) after 400 shots from the barrel. The corresponding spectra of the autocorrelation functions are shown in the upper right corner of the SEM images

the FeAl powder particles. The observed heterogeneity may be additionally compounded by in-situ forming of Al_2O_3 oxide phases under GDS spraying conditions on the surface of FeAl particles displaying lower hardness in comparison with the Al_2O_3 oxide ceramics [7].

Numerical analysis based on the spectra of the autocorrelation function showed the lack of directivity of the examined surfaces. In both cases, respectively after 100 and 400 shots, the surfaces are isotropic with the $S_{tr} \approx 0,5$ parameter exhibiting a slight tendency to anisotropy in areas with lower plastic deformation of FeAl particles – S_{tr} decreased its value to 0.46 on the peripheries following 400 detonation gun shots. In both cases, the increase in the degree of non-dilatational strain causes a subsequent decrease in the autocorrelation length (S_{dl}). It can therefore be applied with high probability as a qualitative parameter characterizing the degree of plastic deformation affecting the powder charge particles in the GDS technique. Detailed results of the fractal analysis are summarized in table 2.

TABLE 2

Fractal parameters on the surface of the GDS coatings subjected to tests

Number of GDS shots	Area of the coating deposit	Autocorrelation length	Surface anisotropy ratio	Fractal dimension	Fractal dimension	Corner frequency	Corner frequency	Mean grain size
		S_{dl} [μm]	S_{tr}	$D_{AF(1)}$	$D_{AF(2)}$	τ_{c1} [μm]	τ_{c2} [μm]	D_{ziaren} [μm]
100	Central zone	6.31	0.56	2.49	2.86	2.47	17.27	4.97
	Peripheral zone	8.83	0.53	2.50	2.85	2.50	20.42	5.99
400	Central zone	5.48	0.53	2.54	2.86	2.43	15.93	4.60
	Peripheral zone	9.19	0.46	2.51	2.83	2.51	20.28	6.86

The conducted fractal analysis clearly indicates the bifractal structure of the FeAl coating surfaces (GDS), depending on the degree of deformation of the particles (splats) – with the maximum value assumed in the central zone of the coating deposit (at a distance of 5-7 mm from the axis of the circular deposit). This type of morphology, proper to the construction of the cluster surface, is a result of a different degree of non-dilatational deformation affecting intensively heated and partially melted powder particles, depending on the degree of FeAl particle dispersion relative to the axis of the detonation gun barrel. Amongst the detailed results summarized in Table 2, the first $D_{AF(1)}$ dimension describes the obtained cluster structure. In each of the cases analyzed, its value is quite similar and amounts to 2.51 ± 0.02 on average, whilst the other fractal dimension, defined by the arrangement of clusters, amounts to 2.81 ± 0.01 . Similar values

of fractal parameters in specific zones of the coating indicate that they do not depend on the number of detonation gun shots fired and are adequate for the DGS method. The aforementioned parameters are conditioned by the dispersion of the two-phase metallization stream in relation to the axis of the detonation gun barrel. The reduction in the degree of deformation of the powder particles at the periphery of the metallization stream results in the presence of spheroidal powder particles on the surface of the FeAl (GDS) coatings. As a result, the corner frequency τ_{c2} increases, whilst the first index τ_{c1} is independent from the degree of non-dilatational deformation and remains constant. It can therefore be assumed that τ_{c2} is determined by the degree of non-dilatational deformation of the formulated grains on the FeAl phase matrix, analyzed on the surface of the coating formed under specific GDS conditions.

The full width at half maximum (FWHM) analysis of the spectrum of the $R(\tau)$ function, according to the methodology described in [22, 23], made it possible to determine the mean grain size (D_z) at half height. As can be seen in Table 2, the presence of plastically non-deformed powder particles causes an increase in the average grain size, which was determined on the basis of FWHM in accordance with a specific autocorrelation function.

The analysis of the obtained research results suggests that the increase in the number of shots in the GDS method (varying from 100 to 400 shots), partly affects the functional properties of the coating, determined by the parameters presented in Table 3.

TABLE 3

List of functional parameters of FeAl coatings formed under specific GDS conditions

Numer of GDS shots	Area of the coating deposit	S_k [%]	S_{pk} [%]	S_{vk} [%]	S_{mr1} [%]	S_{mr2} [%]
100	Central zone	44.01	24.20	31.80	13.51	93.65
	Peripheral zone	43.82	39.52	16.67	15.00	94.79
400	Central zone	55.51	28.87	15.61	13.34	95.37
	Peripheral zone	54.59	39.44	5.97	15.51	97.07

The increase in the number of shots (from 100 to 400) resulted in a 10% relative increase in load bearing properties of the surface profile, with unchanged percentage shares of elevations (S_{mr1}) and hollows (S_{mr2}). It should also be emphasized that the relative depth of hollows (S_{vk}) decreases by roughly 50% in the zones involving partially undeformed charge, whilst the percentage shares of these hollows remain unchanged.

5. Conclusions

The numerical analysis of the surface and the implementation of the autocorrelation function indicates the adequacy of the applied methodology used in order to describe SGP by means of SEM/EDS images of FeAl coatings formed and deposited under specific GDS spray conditions, following either 100 or 400 shots at a frequency of 6.66 Hz. The obtained coatings are

characterized by the lack of directivity of the structure, while the autocorrelation length (S_{al}) emerges as a promising parameter, enabling us to evaluate the degree of the non-dilatational deformation of the FeAl intermetallic powder charge.

The coatings exhibit a cluster structure of grains (splats) with isotropically arranged elevations, which display the characteristics of conglomerates with multiphase structure -developed as a result of in situ formation of Al_2O_3 oxide phases under GDS conditions in the form of thin films on the surface of plastically deformed FeAl particles.

The morphology of the deposited coatings, confirmed by the fractal analysis, clearly indicates the bifractal structure, characteristic for the layered structures with a cluster structure.

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