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Authors: Tomasz Chrostek, Mirosław Bramowicz, Kazimierz Rychlik, Adam Wojtkowiak, Cezary Senderowski

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Influence of gas detonation spraying parameters on the geometrical structure of Fe-Al intermetallic protective coatings

Tomasz Chrostek[1]***, Mirosław Bramowicz***[1]***, Kazimierz Rychlik***[2]***, Adam Wojtkowiak***[1]***, Cezary Senderowski***[1]

[1]***Departament of Materials and Machinery Technology, University of Warmia and Mazury, Oczapowskiego 11, 10-719 Olsztyn, Poland***

[2] ***Research Network ŁUKASIEWICZ - Institute of Mechanised Construction and Rock Mining, 6/8. Racjonalizacji str., 02-673 Warszawa, Poland***

Corresponding author: Tomasz Chrostek, Department of Materials and Machine Technology, University of Warmia and Mazury in Olsztyn, ul. Oczapowskiego 11/E27, 10-719 Olsztyn, phone: +48 89 523 38 55, e-mail: tomasz.chrostek@uwm.edu.pl

Abstract

The paper presents the results of an investigation and analysis of the geometrical structure of Fe-Al intermetallic protective coatings sprayed under specified gun detonation spraying (GDS) conditions. As GDS variable parameters there were applied two different barrel lengths and two powder injection position (PIP) at the moment of spark detonation as well as two different number of GDS shots with 6.66 Hz frequency.

The measurements of the surface's profile were carried out through means of contact profilometry, in which case TOPO-01 system and Mitutoyo SJ 210 profilometer were applied. On the basis of the measurements conducted the analysis of in two-dimensional (2D) and spatial (3D) systems was made possible. The authors assumed that roughness can be considered as a non-stationary parameter of variance of surface amplitude, which is highly dependent on the sampling rate and length of an elementary segments. Therefore, the changes in the amplitude parameters and functional properties of the surface at different lengths of measuring segments (ln), respectively: 1.25, 4 and 12.5 mm, were analyzed. In the analysis of the degree of development of the geometric structure of the surface, the RMS (Root Mean Square) fractal method was used, with an assessment of the geometric structure of the surface stretched over several size levels, taking into account the correlation between the roughness parameter Rq, the measuring length (ln) and the fractal dimension (D). The application of the RMS method with the determination of the fractal dimension (D) allowed for the characterization of the geometric structure of intermetallic Fe-Al protective coatings detonation sprayed under specific conditions of the GDS process - based on the surface roughness profiles of different measured length (ln).

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Keywords: GDS, Detonation Gas Spraying, Fe-Al, intermetallic alloys, fractal analysis, RMS

Introduction

Intermetallic alloys based on ordered intermetallic phases can be classified as innovative engineering materials used in industry. This group includes alloys from the Fe-Al equilibrium system, which as functional materials with unique performance properties, can be applied for operating at elevated temperatures as protective coatings (BYSTRZYCKI et al. 1996). Particularly high temperature corrosion resistance in aggressive sulphide and chloride environments potentially creates the possibility of their use as heat-resistant construction materials. However, their high brittleness at ambient temperature and difficulties associated with the process of producing solid alloys with a fine-grained structure, free of any structural defects (JASIONOWSKI et al. 2011, 2012, NIEWIELKI and JABŁOŃSKA 2007) constitute a major limitation of a more widespread application.

Irrespective of the aforementioned disadvantages, Fe-Al intermetallics display a number of very beneficial functional properties, including excellent oxidation resistance, structural and chemical stability at elevated temperatures, and what should be emphasized, the ability to form Al_2O_3 oxides on the surface. The tight, protective oxide layer strengthens the resistance to oxidation, carburizing and sulphation, which allows for the possible use of iron-aluminum intermetallics for elements working in an environment of aggressive gases. This was further confirmed by research results, where intermetallic alloys: Fe – 28% at. Al and Fe - 35% at. Al obtained incomparably better heat resistance in relation to the elements made of commercial chromium-nickel steel (Fe-25% according to Cr-20% according to Ni) and chromium-aluminum (Fe - 19% at Cr - 12% at Al). FeAl alloys are used for extrusion of aluminum processes, as well as grate bars for furnaces that have successfully undergone long-term operational tests at roughly 1000°C. Their practical application can also be found in various intermetallic (FeAl) pallets and stands in both heat and chemical treatment furnaces, as well as rails for furnaces and rollers for transporting hot-rolled steel sheets (BYSTRZYCKI et al. 1996, JASIONOWSKI et al. 2011, NIEWIELKI and JABŁOŃSKA 2007, SCHNEIBEL et al. 2005). Their relatively low price is also a significant factor compared to other groups of heat-resistant materials (JASIONOWSKI et al. 2011, NIEWIELKI and JABŁOŃSKA 2007).

Common methods for the production of Fe-Al intermetals, such as melting or casting, can cause many difficulties. Therefore, thermal spraying methods leading to the formation of a coating with potentially very promising properties became one of the more popular means of producing these alloys, characterized by adhesive bonding to a base material. In the area of thermal spraying technology, a number of methods are distinguished by which coatings with different properties and ranges of application are obtained, depending on the character of the spraying equipment and process parameters applied. Many years of research and practical industrial experience have led to the development of technologies that differ, especially through the prism of the generation and acceleration of the metallizing stream, into which the coating material particles are transferred. Generally, coatings are obtained by virtually all commonly available methods of spray metallization such as (ASSADI 2016, CINCA and GUILEMANY 2012, CHEN Y et al. 2009, HEJWOWSKI 2013, MUŠALEK et al. 2010, SENDEROWSKI et al. 2011, SENDEROWSKI 2015, SZULC 2013, XU et al. 2004, YAN et al. 2012, ŻÓRAWSKI 2010):

a) HVAS (High Velocity Arc Spraying),
b) APS (Atmospheric Plasma Spraying)
c) HVOF (High Velocity Oxygen Fuel),
d) Cold Spraying,
e) GDS (Gas Detonation Spraying)

Thermal spraying technologies are usually quite complex processes in which, irrespective of the application of a given method, a number of parameters affect the performance of the coating. A common feature of all thermal spraying methods is obtaining specific values of temperature and velocity of the metallizing stream, which determine the kinetic energy of the powder charge particles forming the coating. This allows us to form a compact structure of the coating within a short period of time and affect its performance properties such as porosity, hardness, adhesive strength, natural stress distribution and oxidation state (BOJAR et al. 1996).

One of the methods used for many years is the method of gun detonation spraying (GDS), but technological conditions still remain in the sphere of research with the supersonic flow of a two-phase (gas-powder) metallization stream and the formation of a layered structure of the coating with the participation of oxide phases, formed in-situ in the GDS process (SENDEROWSKI et al. 2011).

An important aspect of the GDS spraying process is obtaining geometrically uniform coatings with an axially symmetrical thickness distribution. Obtaining such an effect is possible by consciously controlling the entire spraying process and its repeatability in each working cycle. The geometry of the formed coatings generally depends on the speed of the powder particles at the moment of its collision with the ground and the value of thermal energy accompanying this event (SENDEROWSKI et al. 2011). The purpose of this work is to examine the effect of the length of the GDS gun barrel on the geometry of studied coatings, their performance and various features including roughness as well as functional and fractal properties.

Materials and Methods

Four coatings sprayed with the gun detonation spraying method of intermetallic powder material based on the FeAl phase matrix with 40% aluminum content were tested. The powder with size of granulation distribution set at 5 - 40 μm was manufactured using the VIGA (Vacuum Inert Gas Atomization) method. The base was 15HM boiler steel with dimensions of 50x50x5 mm (Fig. 1). Prior to spraying, the surface layer of the substrate material were blasted with alumina. Circular shells were obtained with the stationary position of the ground and the outlet of the barrel.

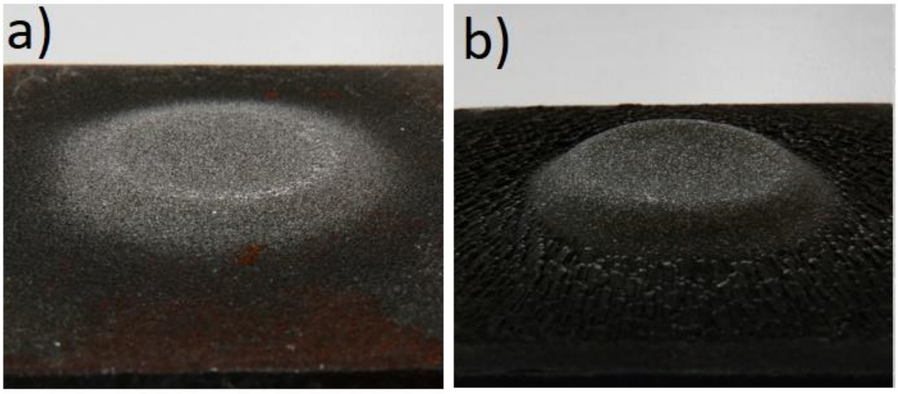

Fig. 1. FeAl coatings sprayed onto 15HM boiler steel under specified GDS conditions at: a) 100 and b) 400 shots, without changing the barrel position relative to the base material

Intermetallic materials based on FeAl phase are characterized by high resistance to high temperatures in chemically aggressive environments, a stable and ordered structure up to about 1100 ^{o}C, and impressive resistance to tribological wear – both abrasive and erosive (CHROSTEK et al. 2018). The GDS process was carried out using the Perun-S gun at the Paton Institute in Kiev with different spraying parameters, as presented in Table 1.

Parameters such as: PIP (powder injection position) changed, i.e. the location of the powder in the barrel at the moment of ignition initiation; the number of shots fired and the length of the barrel. The formation of the coating was cyclical with a frequency of 6.66 Hz, with a constant composition of the explosive mixture (propane) as a working gas. The distance of the barrel (inner diameter Ø23 mm) from the sprayed substrate was L = 110 mm.

Table 1.

GDS spraying parameters

Powder Fe40Al0,05Zr at.%+50ppm B granulation **5÷40 µm**					
Oxygen- fuel mixture			C_3H_8 – 0,45 m^3/h O_2 – 1,52 m^3/h Air – 0,65 m^3/h		
Powder-transporting air			0,4 m^3/h		
Spraying frequency			f=6.66 Hz		
Coating	**Spraying distance L [mm]**	**Barrel length [mm]**	**PIP* [mm]**	**Number of GDS shots**	**Coating thickness [mm]**
1	110	590	412,5	400	2,91
2			274,5	100	0,86
3		1090	412,5	100	0,56
4			274,5	400	1,13

** powder injection position - place of the introduction of the powder into the barrel at the time of detonation*

Surface profilometric measurements were carried out by the contact method, using the modular measuring system TOPO-01, which enables measurement of shape contours on flat, cylindrical external and internal surfaces. High accuracy and a wide measuring range enables full surface characterization by measuring its roughness, wave and shape in 2D. By using a table that allows moving the measured object in the Y axis, it is possible to perform stereometric 3D measurements. The measuring head has a diamond tip with a 2 µm radius and a 60^o cone angle (Fig. 2).

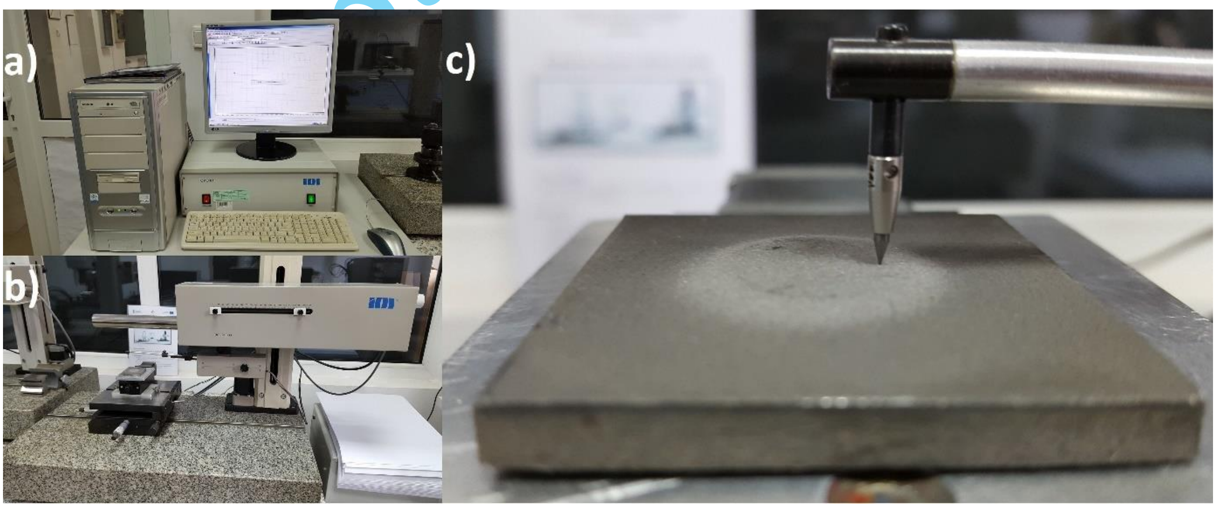

Fig. 2. System for measuring TOPO-01 surface topography: a) computer with TOPO-01 control module, b) shape-maker for 3D stereometric measurements PG 2/200, c) measuring head

To map the surface of the coatings, 26 passes were made at a speed of 1 mm/s in increments of 1 mm. Measurements were made in an area of 25x25 mm. The measurement parameters are given in Table 2.

Table 2.

Topography measurements on coating surface

X-axis feed	25 mm
Y-axis feed	25 mm
Feed speed	1 mm/s
Number of passes	26
Stroke in the Y-axis	co 1 mm
Straightness	0,3 µm / 25 mm
Resolution	0,1 µm
Maximum head load (auto)	15 N

Surface roughness tests were carried out using the Mitutoyo SJ-210 contact device. Its head is equipped with a cone-shaped diamond blade with an angle of 60° and a tip radius of 2 µm. This enables the surface to be mapped in the range of 360 µm (from -200 to +160 µm).

Taking into account roughness as a non-stationary parameter of variance of surface amplitude, depending on the sampling density and length of the elementary measuring section, an analysis of changes in amplitude parameters and functional properties of the surface was carried out at various lengths of measuring sections (ln), respectively: 1.25, 4 and 12.5 mm.

Roughness parameters express statistical distribution of points on tested surfaces on the measuring length (SAYLES and THOMAS 1978). According to the authors of the study, the variance (σ^2) of the height of points on the surface is proportional to the sampling length, which is described by the relationship (1).

$$\sigma^2 \propto L \tag{1}$$

The authors of the work (BERRY and HANNAY 1978) suggest that the relationship (2) describes the variance in the scale function in a more precise manner.

$$\sigma^2 \propto L^H \tag{2}$$

That is why the authors decided to apply the so-called RMS (Root Mean Square) method described inter alia in works (BHUSHAN 1999, MAINSAH et al. 2001, AUE 1997). The RMS method is one of the fractal methods that provides information about the degree of surface development. It also allows you to characterize the geometric structure of the surface stretched over several levels of size. In this case, determining the fractal dimension (D) will allow us to characterize the surface profile over the measuring length (ln) from 1.25 to 12.5 mm. As we know, the dimension D for a two-dimensional system, which is the surface profile, ranges from 1 to 2, where D = 1 corresponds to a straight line, while D = 2 to an extremely developed, i.e. consisting of an infinite number of sections. Referring to equation (2) to roughness Rq and taking into account the relationship between the fractal dimension and the Hurst parameter (H), the equation looks like this (3):

$$Rq \propto L^{\frac{2-D}{2}} \tag{3}$$

By plotting the relationship (3) in a logarithmic system, we can obtain three basic waveforms, as shown in Fig. 3.

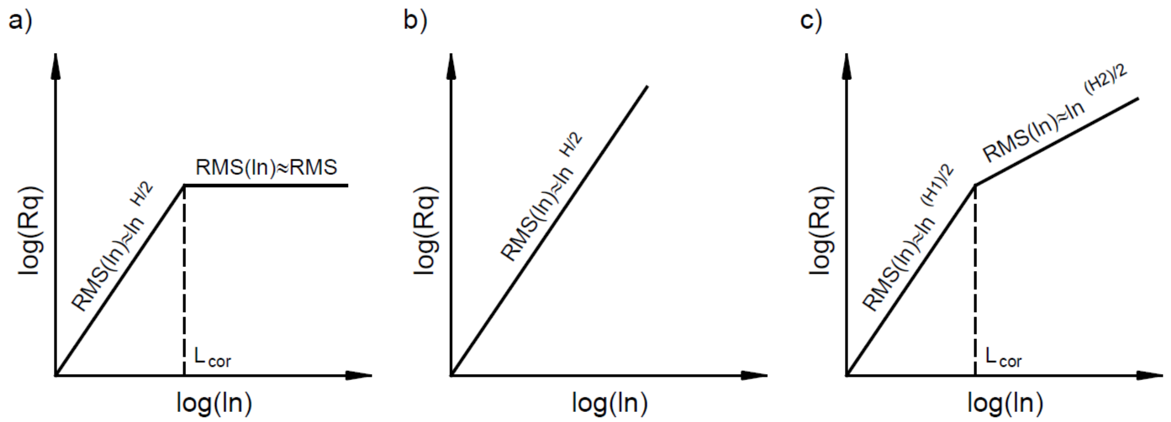

Fig. 3. Basic relationships of Rq = f (L) (AUE 1997)

In the case presented in Fig. 3a, the surface topography at the measuring length < L_{cor} is subject to the law of scaling, then after exceeding it the roughness becomes a stationary process and Rq no longer depends on the measuring length. We can characterize such a surface in the L < L_{cor} range by the fractal dimension D, which value is related to the exponent: $\frac{H}{2} = 2 - D$. In the case shown in Fig. 3b, the surface exhibits fractal properties over the entire scale range, while in the case shown in Fig. 3c, the surface exhibits bifractal properties. This type of structure corresponds to cluster structures, i.e. the cluster surface is characterized to a certain length of L_{cor}, then for L > L_{cor} the structure of cluster arrangement is characterized.

In this work, the RMS method described was used in order to carry out profilometric measurements. Then, in the log-log system, points with coordinates (ln, Rq) were plotted, the point relationship was approximated by the function $y = ax^b$, which logarithmic form is a linear function with the directional coefficient equal to: $1 - \frac{D}{2}$. An example, determined relationship log(Rq) = f (log (ln)) is shown in Fig. 4.

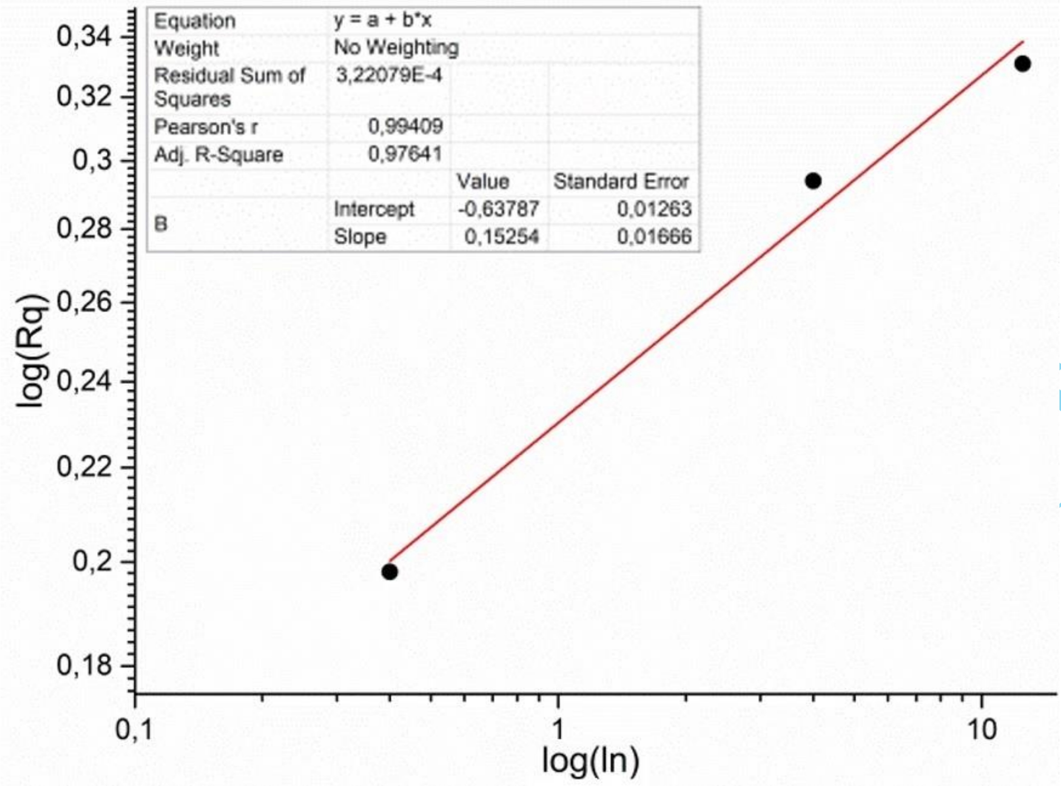

Fig. 4. Example of using the RMS method in profilometric measurements

The application of the above methodology allowed characterizing the surface over a measuring length of 1.25 to 12.5 mm and provided information on the degree of their development.

Results and Discussion

The result of the analyzes conducted is an isometric view and contour maps determining the surface and parameters, which describe the three-dimensional surface of the coating. As a result of 100 and 400 shots fired cyclically, with a constant barrel position relative to the sprayed substrate, geometrically homogeneous coatings are formed, with a diameter of approx. 25 mm and thickness dependent on the spraying parameters applied. The analysis of the obtained profilograph shows a significant impact of the spraying parameters used on the shape, flat dimensions and thickness distribution of the sprayed coatings (Fig. 5). The analyzes show that the thickness of the coating has the most significant impact on the length of the barrel used (ignoring the number of GDS shots fired). When using a shorter barrel (590 mm), much thicker coatings are formed compared to coatings sprayed with a 1090 mm barrel in length.

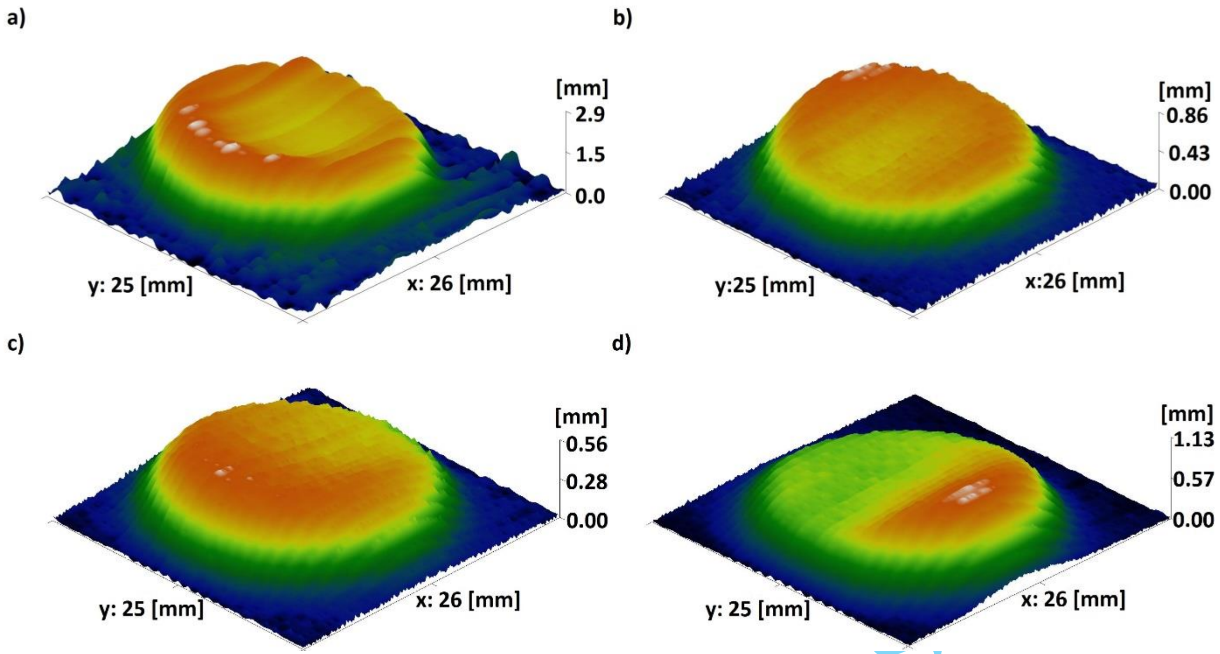

Fig. 5. Isometric view of Fe-Al coatings after GDS spraying - coating designation according to table 1: a) 1, b) 2, c) 3, d) 4

The zone of maximum thickness of such a "static" coating is shifted geometrically from approx. 6 to 8 mm, relative to the axis of the barrel of the detonation gun, depending on the spraying parameters (Fig. 6). The axially-asymmetrical distribution of the coating thickness indicates:

- a dynamic interaction of the detonation product stream with the substrate and powder particles transported in this stream,
- heterogeneity (unevenness) of the distribution of powder particles in the stream of detonation products, which at different flow rates translated to uneven heating of individual particles,
- more dispersed powder particles (shifted from the axis of the detonation stream) obtain a smaller degree of plastic deformation in collision with the ground.

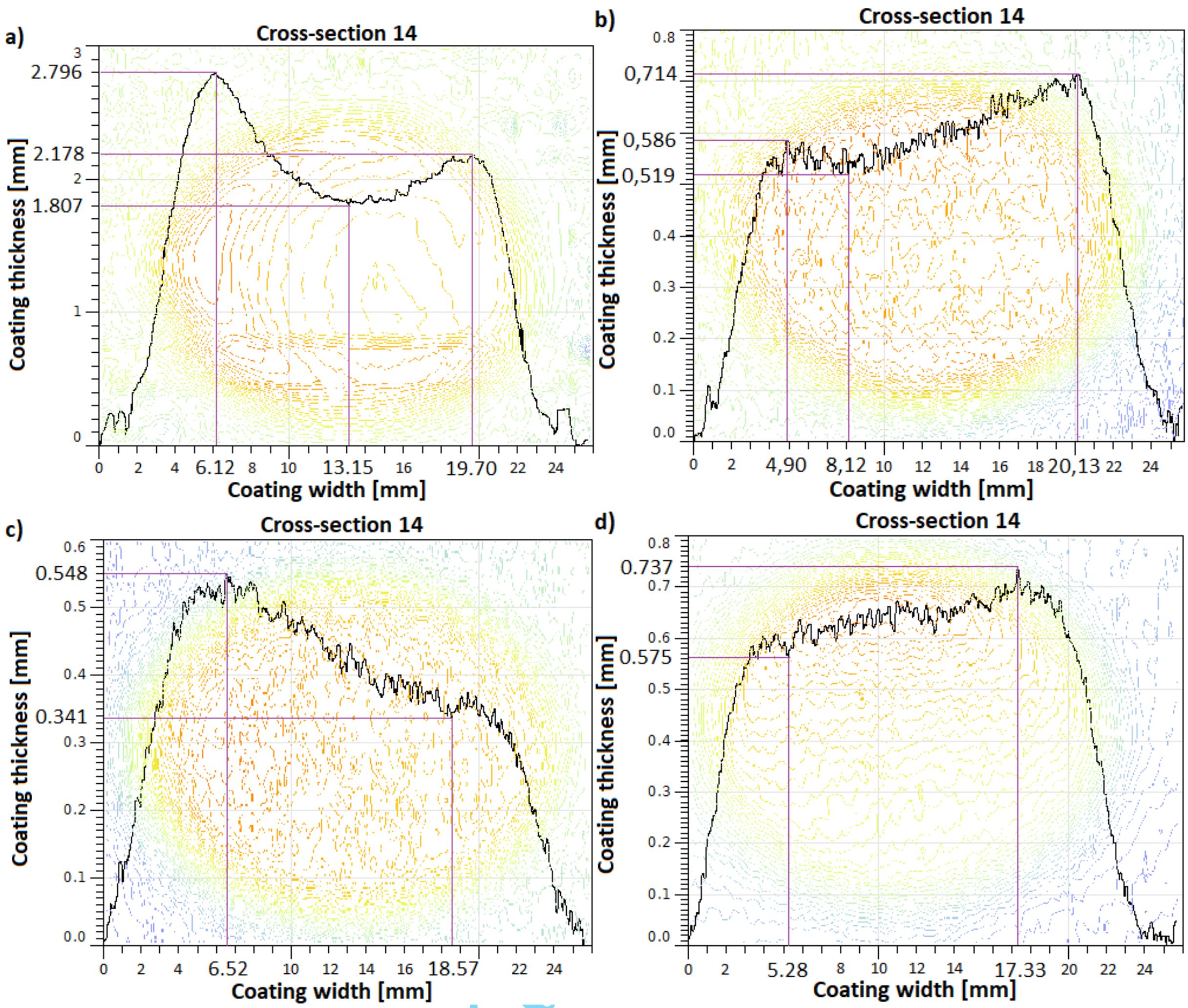

Fig. 6 The thickness distribution of Fe-Al coatings along the diameter of the cross-section along with the contour map - coating designation according to table 1: a) 1, b) 2, c) 3, d) 4

Surface roughness is a non-stationary process. The parameters describing it show the dependence on the sampling density and the length of the elementary measuring segment. Due to the irregular shape of the coatings, roughness measurements were carried out in the central area of the coating. Figure 7 presents the mean square roughness deviation as a function of the scanning area, plotted in a logarithmic coordinate system. Roughness parameters express static distribution of points on the tested surfaces. The analysis of the obtained results showed that the number of GDS shots had the biggest impact on the degree of surface development. Their increase reduces the degree of surface development (Table 3).

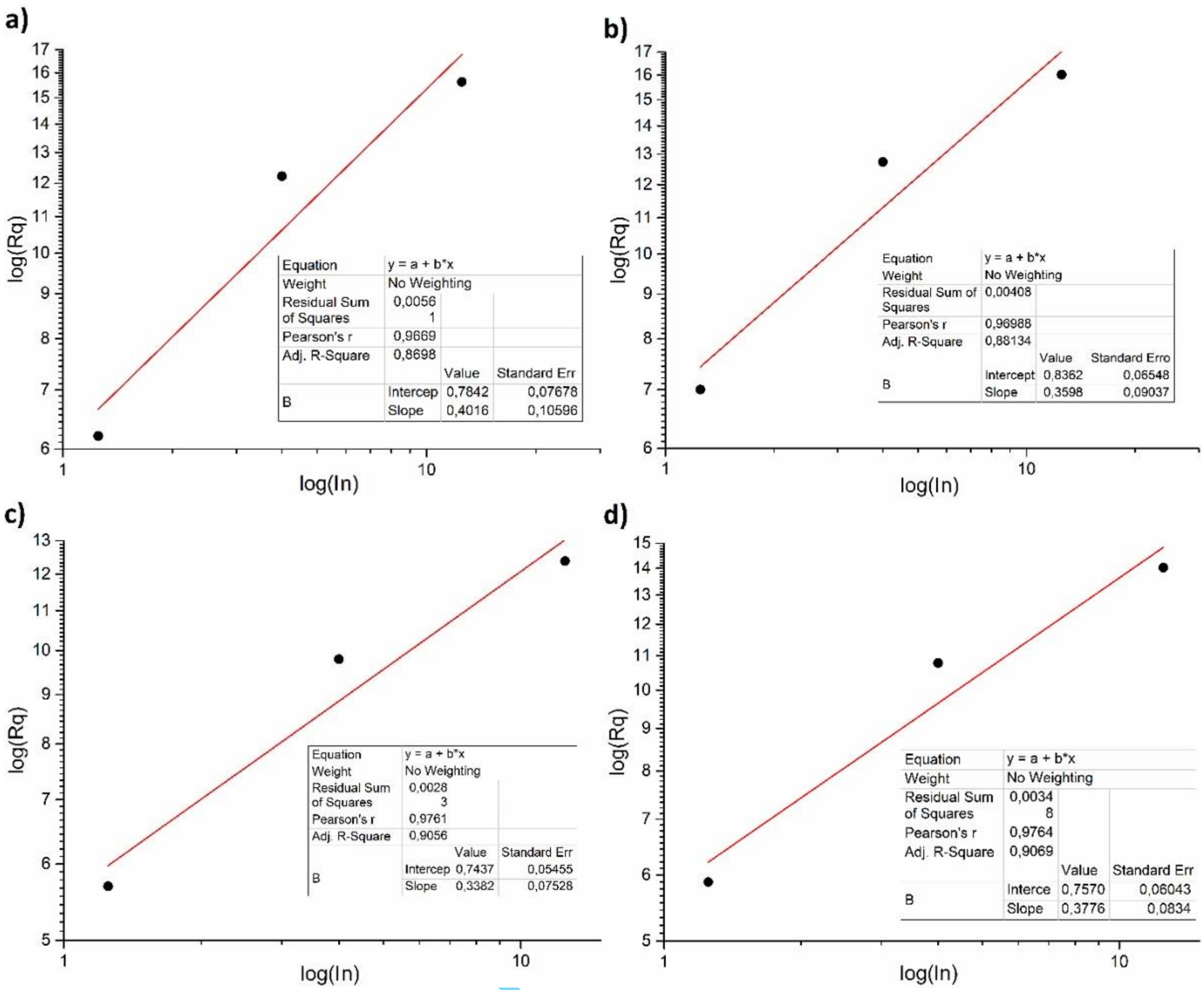

Fig. 7. Relationship of the mean square roughness deviation (RMS) as a function of the length of the scanned area (ln) - coating designation according to table 1: a) 1, b) 2, c) 3, d) 4

Table 3.

RMS test results

Coating	Measuring segment length *Ln* [mm]	Root-mean-square deviation *Rq* [um]	Slope *a*	Fractal dimension *D*
1[1]	1,25	6,2072	0,4016	1,1968
	4	12,2248		
	12,5	15,6286		
2[2]	1,25	7,0036	0,3598	1,2804
	4	12,7372		
	12,5	16,0178		
3[2]	1,25	5,6892	0,3382	1,3236
	4	9,7922		
	12,5	12,3834		
4[1]	1,25	5,8858	0,3776	1,2448
	4	10,7782		
	12,5	14,0264		

1)Coating 1 i 4 – 400 GDS shots fired
2)Coating 2 i 3 – 100 GDS shots fired

Conclusions

The conducted tests enabled the characterization of the geometrical structure of the intermetallic surfaces of protective coatings on the matrix of the FeAl phase sprayed in the detonation under certain conditions of the GDS process. Analysis of the obtained results showed a significant effect of changing the barrel length on the thickness of the formed coating. Regardless of the number of GDS shots fired, the coatings formed at a barrel length of 590 mm are much thicker.

RMS tests confirm the fractal surface properties. In the measured range from 1.25 to 12.5 mm, the surfaces of the coatings are subject to the law of scaling, showing self-similar properties (i.e. each fragment of the profile on a given scale is a reflection of the whole). The increase in the number of shots reduces the degree of surface development. This is probably the effect of compacting the structure with a shock wave during cyclic operation of the GDS gun, which is a precursor of gaseous detonation combustion products transporting part of the powder constituting the FeAl coating.

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