



# Influence of Detonation Gun Spraying Conditions on the Quality of Fe-Al Intermetallic Protective Coatings in the Presence of NiAl and NiCr Interlayers

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The paper presents results of detailed research of the application of detonation gun (D-gun) spraying process for deposition of Fe-Al intermetallic coatings in the presence of NiAl and NiCr interlayers. A number of D-gun experiments have been carried out with significant changes in spraying parameters which define the process energy levels (changes in volumes of the working and fuel gases, and the distance and frequency of spraying). These changes directly influenced the quality of the coatings. The initial results underlay the choice of the process parameters with the view to obtain the most advantageous of geometric and physical-mechanical properties of the coating material, interlayer and substrate. The metallurgical quality of the coatings was considered by taking into account grain morphology, the inhomogeneity of chemical content and phase structure, the cohesive porosity in the coating volume, and adhesive porosity in the substrate/interlayer/coating boundaries. The surface roughness level was also considered. It was found that the D-gun sprayed coatings are in all cases built with flat lamellar splats. The splats develop from powder particles which are D-gun transformed in their plasticity and geometry. A significant result of the optimization of D-gun spraying parameters is the lack of signs of melting of the material (even in microareas) while the geometry of the subsequently deposited grains is considerably changed and the adhesivity and cohesion of the layers proves to be high. This is considered as an undeniable proof of high plasticity of the D-gun formed Fe-Al intermetallic coating.

**Keywords** detonation gun spraying, intermediate layer, intermetallic (based on Fe-Al), protective coatings

## 1. Introduction

The analysis of the processes of intermetallic Fe-Al coating deposition from self-decomposing powders indicates that the range of detonation gun (D-gun) spraying, HVOF as well as plasma spraying techniques offer practically unlimited possibilities of control of spraying parameters and chemical compositions of coatings. This is very promising as the coatings obtained boast of unique properties (i.e. high abrasion, high erosion and corrosion resistance, resistance to high temperatures and thermal shocks, and good adhesion and low porosity) (Ref 1-35). For the purpose of this research the D-gun technique was employed.

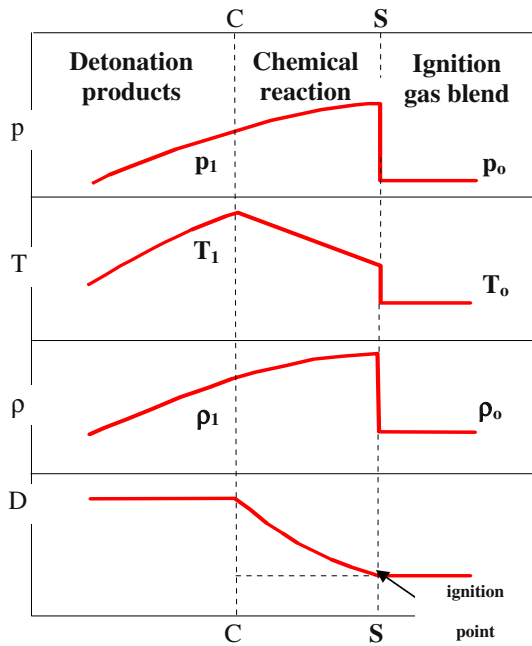
The essence of the D-gun spraying is the use of energy of the gas mixture detonation to heat and relay high kinetic energy to the particles of the powdered coating

material. The D-gun deposited coatings are formed through the collision with the substrate material and deformation of fine grains of the powder which, together with the detonation products, form metallic spray. High kinetic energy and low thermal energy accumulated in the two-phase metallic spray (Ref 36-42) are energy sources of the bonds between the deposited powder, substrate and coating layers. If the spray parameters are properly chosen, the powder particles temperature does not exceed the melting point temperature and the particles in-flight are slightly soft (Ref 36-40, 43, 44). During the D-gun process the characteristic lamellar structure of the coatings results from intensive, impulsive deformation of the intermetallic powders. This promotes formation of inhomogenous multiphase (composite) structure of the coatings.

All the analyses in the literature (Ref 36-40) can be reduced to the defining of parameters of the gas detonation wave, i.e. its velocity ( $D$ ), pressure ( $p_1$ ), density ( $\rho_1$ ), and temperature ( $t_1$ ), which can be considered, with high degree of approximation, within the frame of hydrodynamic theory and unidimensional stationary detonation (referred to as ZND, i.e. Zeldowicz-von Neumann-Doering theory).

According to the above-mentioned theory, the detonation wave (Fig. 1) is comprised of the shock wave, the chemical reaction zone and the detonation products zone. The shock wave is reduced to the (S-S) surface of high

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**Fig. 1** The distribution of parameters of the propagating detonation wave (Ref 37)

discontinuity of the pressure ( $p_0$ ), density ( $\rho_0$ ), and temperature ( $t_0$ ) of the gas mixture at the moment of detonation. The volume of the chemical reaction zone (hypothetically infinitesimal) is determined by the duration of the chemical reaction which follows the wave, and the velocity of detonation wave ( $D$ ). According to Kadyrov and Kharlamov (Ref 37, 39), the process of gas detonation can be presented as the fluctuations of pressure, density, temperature and acceleration in the propagation of detonation wave at the constant speed of the wave, which is reached only after covering some distance ( $l_{kr}$ ) from the ignition point. In case of gas detonation, after ignition, the detonation products propagate in all directions and once they have covered the distance ( $l_{kr}$ ) the system changes into the defined detonation wave characterized by the detonation products. The front of the detonation wave and the chemical reaction zone are a function of the product detonation parameters, i.e. the temperature, velocity, pressure and chemical composition, which depend on the type of the gas mixture. It is worth mentioning that in any given point of the D-gun barrel inner space, the velocity  $u_1(x, t)$ , temperature  $T_1(x, t)$ , pressure  $p_1(x, t)$  and density  $\rho_1(x, t)$  of the detonation products are functions which depend on the time and space coordinates.

The research (Ref 37) concerning the change in the parameters of the detonation wave indicates that the crossing of the (S-S) surface by the detonation wave is characterized by the pressure increase (up to several atmospheres) accompanied by temperature increase (to a couple of thousand degrees Kelvin above the critical temperature in which the gas enters the chemical reaction;

Fig. 1). Behind the front of the shock wave the temperature rises in the chemical reaction zone and then it is gradually reduced as the distance from the front of the wave increases. Directly behind the wave front the density and pressure are gradually reduced. When the detonation wave reaches the open end of the barrel, it has one defined detonation velocity  $D$  (Fig. 1) which is unique for any gas detonation mixture.

The above-mentioned analysis shows that the detonation wave parameters depend on the composition of the gas mixture and especially the contents of oxygen. From the practical point of view, other important control parameters of the D-gun spraying (apart from the proper percentage composition of the detonation mixture which is the decisive factor for the quality of the deposited coatings) are the powder particles size and the frequency and spraying distance. As the result of the experiments (Ref 38), it was found that the maximum acceleration of the powder particle occurs in a small span time (0.1-0.2) ms in the spraying distance ranging from 200 to 400 mm so both the maximum and final speed of the particles depend on the real gas detonation spraying distance.

The D-gun technology is based on complex physical-chemical processes which are governed by a great many factors. A proper understanding of the detonation spraying process depends on accepting the fact that the spraying depends on the perfection of the detonation gun design and the methods of the control of the detonation spraying process parameters (Ref 37-39). The prerequisite for control of the production conditions of D-gun spraying coating is the above-mentioned knowledge of the characteristics of the gas flow in the barrel during the burn-out of the combustible mixture and exit of gas after the detonation wave has reached the open end of the barrel.

In a more detailed approach, the velocity of the detonation wave is expressed as (Ref 37):

$$D = \sqrt{2(k^2 - 1)Q_0} \quad (\text{Eq 1})$$

where  $k = c_p/c_v$ , and  $c_p$  and  $c_v$  are the specific temperatures of the detonation products at constant pressure and volume, respectively.  $Q_0$  is the specific energy of chemical reaction during the detonation.

Alternatively, the velocity of the detonation wave can be found from (Ref 37):

$$D = \frac{k + 1}{k} \frac{\sqrt{8310k}}{\mu_1} T_1 \quad (\text{Eq 2})$$

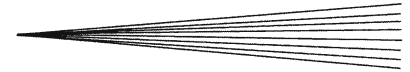
where  $\mu_1$  is the average molecular weight of the detonation product.

Temperature and pressure of the detonation products are given by (Ref 37):

$$T_1 = \frac{2k}{k + 1} \frac{Q_0}{\mu_1} T_1 \quad (\text{Eq 3})$$

$$p_1 = 2(k - 1)\rho_0 Q_0 \quad (\text{Eq 4})$$

A relation for an approximate estimation of the velocity  $v_p$  ( $\text{ms}^{-1}$ ) of particles was derived in Ref 39 from the



condition when the thermal energy and the kinetic energy of the particles are equal:

$$v_p = 100 \left( \frac{c_p T_p + L}{1} \cdot 195 \right)^{1/2} \quad (\text{Eq 5})$$

where  $c_p$  ( $\text{cal g}^{-1} \text{ } ^\circ\text{C}^{-1}$ ) is the specific heat capacity of the particle material,  $T_p$  ( $^\circ\text{C}$ ) is the temperature of the particles, and  $L$  ( $\text{cal g}^{-1}$ ) is the latent heat of melting of the particle material.

The evaluation of relative contribution of the thermal and kinetic energies to the coating formation can be done on the basis of calculation of velocity versus temperature relations for sprayed particles of various materials for particular D-gun spraying conditions. Then, appropriate spraying conditions for a calculated estimation of the contact processes leading to the formation of the bond between the materials can be selected. The optimum relations between the velocity and temperature of particles being sprayed exist for all types of materials. However, no theoretically or experimentally substantiated recommendations for a broad range of Fe-Al intermetallics phase are available at present.

Strong dependence of the thermodynamic properties of the materials on the temperature should be taken into account when analyzing coating formation mechanisms for defined D-gun spraying conditions. Kharlamov (Ref 39) believes that materials with a low heat capacity are more sensitive to the effect of the velocity of particles on their energy state. The required particle acceleration velocity at a predetermined substrate heating temperature  $T_s$  and a predetermined particle heating temperature  $T'_p$  may also be found from (Ref 39):

$$v_p = 91.48 \left[ T_k \left\{ \left( 1 + \frac{B_s}{B_p} \right) - \frac{T_s B_s}{B_p} - T'_p \right\} c_p \right]^{1/2} \quad (\text{Eq 6})$$

where  $T_k$  is the contact temperature required for the formation of a strong bond between a particle and the substrate, and  $B_s$  and  $B_p$  are the heat accumulation coefficients for the substrate material and the particle material, respectively. ( $B = (\lambda c_p)^{1/2}$ ,  $\lambda$  being the thermal conductivity coefficient.) The procedure for determining the optimum value of  $T_k$  is presented in studies on plasma spraying of coatings (Ref 39). The expression above allows for the estimation of required particle acceleration velocity for more moderate heating of particles to prevent undesirable physicochemical transformations.

The development of plasma spraying and supersonic flame spraying of coatings has been reported (Ref 11, 31-36, 39, 41-53), and the properties of the coatings produced by these methods are comparable with those of detonation coatings. The HVOF processes similar to the D-gun spraying are based on high-pressure combustion. As the particle velocities are a function of the chamber pressure (Ref 41), by using higher oxidant content and fuel pressure, impact velocities similar to those of D-gun could be achieved. Because the detonation phenomenon physically limits the pressure rise in the combustion chamber (and therefore the particle velocity), the continuously burning HVOF process can increase the chamber pressure over

the one achievable in the D-gun system and therefore can theoretically achieve higher particle velocity and coating quality (Ref 41).

In fact, the concept of HVOF spraying has evolved, along with a better understanding of the system, through modeling and measurements (Ref 28, 29, 45-47). Since the first HVOF torch (Jet Kote System) operating at 0.41 MPa, different torches have been designed with operating pressure up to 1.35 MPa (Terojet, Eutectic Castolin, CH) (Ref 44). Besides gas fuels such as propylene, propane, methane, and hydrogen, liquid fuels such as kerosene have been used in HVAF systems. The HVAF guns using air instead of oxygen have also been developed with kerosene as the fuel. However, comparing to the HVOF or HVAF methods, the D-gun spraying is strongly competitive due to the lower cost of the coatings and simplicity of the operation and life duration of the equipment (Ref 40). In the cyclic detonations the gases are fed under low pressure (70-210 kPa), there are no complex cooling systems (the flow of water coolant is 4 L/min), and there is no need to use supersonic nozzle (e.g. de Laval nozzle).

As far as plasma spraying is concerned, there are much data available that deal with the velocity and droplet temperature study as well as dynamics of structure splats evolution during plasma spraying (Ref 25, 31-35, 48-52). Plasma spraying is a high temperature and velocity version of thermal spraying technique, in which material is melted in a hot plasma jet and the melt is accelerated toward a substrate, where it rapidly solidifies. The plasma spraying processes are complex, involving rapid melting, high-velocity impact deposition of powder particles, their quenching and consolidation in a single operation.

Comparing the techniques (plasma versus D-gun or HVOF), the temperatures reached by the powder particles during the spraying have been much higher with the plasma spraying than with the D-gun or HVOF ones. The temperatures generated in the plasma jet are several thousand degrees, and the velocities can be sub- or supersonic. Heat and momentum transfer between a high-temperature flow and a single particle received a lot of attention in Ref 48-52. The value of the impulse pressure resulting from collision in the particle-substrate contact zone is determined from (Ref 39):

$$P_i = \rho_p C_{ep} v_p \left( 1 + \frac{\rho_p C_{ep}}{\rho_s C_{es}} \right) \quad (\text{Eq 7})$$

where  $C_{ep}$  and  $C_{es}$  are the velocities of sound in the particle and substrate material, respectively. For plasma spraying, it is customary to divide the collision of particles with the substrate into two stages (Ref 39):

1. the action of the impact pressure  $P_i$  over the time  $t = 2r/C_{er} \approx 10^{-7} - 10^{-9}$  s;
2. the action of the head pressure  $P_h = \rho_p v_p^2$  over the time  $t = (2r - h)v_p \approx 10^{-5} + 10^{-7}$  s, which coincides with the particle crystallization time.

In plasma spraying, the pressures  $P_i$  and  $P_h$  are generally in the ranges  $10^2 - 10^4$  MPa and  $10 - 10^2$  MPa,

respectively (Ref 39). As the amount of heating of the particles increases, the velocity of sound in the particle material and the impulse pressure decrease. The impulse pressure on impact of a melted particle is (22-27)% less than the one on impact of a solid particle at normal temperature (Ref 39). This diminishes the mechanical activation of the substrate surface rate and prevents the formation of a strong bond between the materials.

The high particle velocity in D-gun spraying practically promotes merging of both stages of particle collision with the substrate into a single stage and  $P_h \rightarrow P_i$ . High impulse pressures encourage mechanical activation of the contact surfaces concurrently with thermal activation of the contact area of the substrate; the interaction of the particle and substrate materials may be considered as a mechano-chemical reaction (Ref 39). The particles that spread over the substrate under high impulse pressure (as a result of contact friction) clean the contact surface by destroying surface films and forcing them to the periphery of the contact zone. At the same time particles form indentations in the metallic substrate (Ref 54). This gives rise to defects in the crystalline structure which, on their emergence to the contact surface, become active centers of chemical interaction between the bonded materials. The formation of defects in the contact zone is also promoted by contact friction. The rate of formation of active centers in the contact zone and the time of action of the high impulse pressure control the strength of the bond between particles and the substrate.

In case of thermal spraying, the substrate/coating bond strength is influenced by a number of other factors which have exhaustively been described in the literature (Ref 53-63). However, the most important factors are the residual stress generated in the conditions of high temperature and pressure and porosity which result from the splat oxidation during spraying (Ref 60-63).

One can expect (on the basis of the experience) the D-gun process to be a very promising technique which can be employed to produce high-quality Fe-Al intermetallic coatings. Research results in the Military University of Technology database prove that the D-gun process brings advantageous effects in case of both "in situ" coating deposition (spraying of pure elements powders) (Ref 5) and Fe-Al intermetallic powders spraying (Ref 1, 6, 16, 40, 54, 56). However, although the possibilities of control of spray parameters and chemical composition of the coatings to obtain best metallurgical results are practically unlimited, the choice of the parameters is complex.

The end result depends on many factors (Ref 1, 16, 36-40), out of which the most important are kinetic and thermal energies of the metallic spray formed in the barrel of the D-gun. One other important factor is the proper choice of powder size which prevents heating of the grains over their melting point temperature or even their evaporation. The evaporation was registered in case of <40  $\mu\text{m}$  self-decomposing powder grains reacting with high kinetic energy metallic spray.

In general, for the D-gun spraying process one employs powders which are bigger in size than the ones used in the plasma spraying or HVOF processes (Ref 4, 29, 34,

38-41, 48-52). This results from the character of the D-gun process (relatively low temperature and high kinetic energy of powder particles). Bigger particles which are close to the melting point but still in the solid state, as the result of their high velocity impact on the substrate, deposit as dense coating which has low porosity and high adhesive strength.

In the atmospheric plasma spraying method, however, the use of bigger powder particles is not desirable as there is a possibility of overheating and melting of the grain surfaces only, while the cores of the powder particles remain solid. This phenomenon results from the considerably higher temperature and lower velocity of the plasma spraying process. If bigger powder particles are used, the plasma sprayed coatings contain unmelted particles and oxides and are highly porous, which determines their lower adhesive strength.

This paper aims at presenting generalized analysis of interdependence between properly controlled technical parameters of the D-gun process and the quality of coatings produced with self-decomposing Fe-Al intermetallic powders in the presence of NiAl and NiCr interlayers. It also suggests the spraying parameters for which the most advantageous quality of the coatings was obtained. The quality was defined by a set of geometrical, morphological and physical-chemical properties of the coating grains forming both Fe-Al coating and NiAl and NiCr interlayers.

## 2. Experimental

The material selected for coating application was Fe-Al powder, the chemical composition of which was Fe-62.5at.%, Al-36at.%, C-1.5at.% (producer warranted), in three particle size ranges of <40  $\mu\text{m}$ , (38-75)  $\mu\text{m}$ , and (80-125)  $\mu\text{m}$ . The powder was produced by the Institute of Technology of Alloys and Composites, Silesian University of Technology, Katowice, Poland, in the self-decomposition process and was dried at the temperature of 100 °C for 15 min prior to spraying. As the interlayers were Ni-95at.%, Al-5at.% and Ni-80at.%, Cr-20at.% coatings produced from 38-53  $\mu\text{m}$  particle size powders. The powders were obtained in D-gun spraying. The depositing of all of the coatings was carried out in a number of experiments using different spraying parameters (discussed in the "Results" section) to influence detonation energy of the gas mixture and to influence metallurgical quality of the coatings. The substrate was 1045 steel plate hardened and tempered, ground, stress relieved and cleaned directly before spraying by abrasive blasting with 24  $\mu\text{m}$  aloxite.

The properties of the coatings were analyzed with Philips XL-30/LaB<sub>6</sub> scanning microscope integrated with DX4i-EDAX x-ray microanalysis. For the purpose of this paper, the following features were analyzed: the level of inhomogeneity in the phase and chemical composition, the grain morphology and discontinuities (nonmetallic defects (oxides), microcracks, cohesive porosities in the coating

volume as well as adhesive porosity in the coating/interlayer and interlayer/substrate joints). The porosity of the coatings was assessed by photomicrograph quantitative analysis carried out with (SEM) Philips XL30/Lab<sub>6</sub> programmed with SIS software. The Cavaleri-Hacquert (Ref 64) principle was applied, according to which the level of inner porosity of the D-gun coatings (i.e. cohesive porosity) is defined with planimetric method as the ratio of the sum of pore surfaces to the total surface of the specimen. The adhesive porosity (in the coating substrate joint) was defined as a ratio of the sum of the lengths of pore chords to the length of measuring length on the specimen surface. The coating surface roughness was measured with PGM-1C profilometer with G250BS head. The measurements were carried out on the measuring length of  $l=4$  mm at the head movement velocity of  $v=0.2$  mm/s and the static load of the blade of 3 mN.

### 3. Results

D-gun spraying as any other method of the coating thermal deposition shows close correlation between spraying conditions and physical-chemical and morphological properties of the powder. Therefore, it was considered necessary to carry out detailed research and analyses (Table 1) of the results of D-gun spraying application to produce coatings with different Fe-Al powders ranging from  $<40$   $\mu\text{m}$ , (38-75)  $\mu\text{m}$  to (80-125)  $\mu\text{m}$  in size. The powders were deposited directly onto 1045 steel substrates (without NiAl and NiCr interlayers).

In case of the powders, the particle size of which was lower than 40  $\mu\text{m}$ , the choice of proper D-gun spraying parameters was difficult. The coatings deposited in the same conditions had non-repeatable thickness (Fig. 2) and considerably differed in their surface roughness ( $R_a = 6.4$ -10.3  $\mu\text{m}$ ). The analysis of their chemical composition in the coating microareas confirmed the presence of  $\text{Al}_2\text{O}_3$  oxide films, particularly in the surface of the coatings. The presence of strongly oxidized (although the oxidation

degree varied) lamellar particles of coating material in the coating volume (Table 2) was also confirmed. The results suggest that during the D-gun spraying small particles of powders undergo intensive oxidation. This is disadvantageous in case of coatings because the oxides in the substrate-layer joint are responsible for the total loss of adhesion, the key feature for the coating applications (Fig. 3a). If the spraying parameters are not optimized, and the size of the powder particles used in the process is  $<40$   $\mu\text{m}$ , strong oxidation of the coating particles can result in delamination and decohesion of the coating (Fig. 3b).

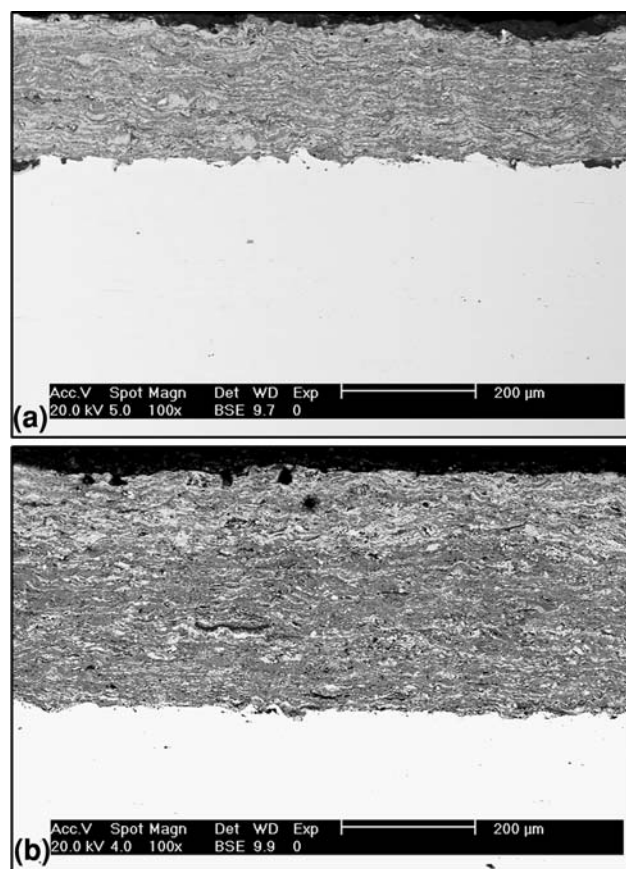


Fig. 2 Microstructure of two D-gun sprayed Fe-Al type coatings (a, b) deposited on 1045 steel with  $<40$   $\mu\text{m}$  powders in the same spraying conditions. The coatings differ in thickness

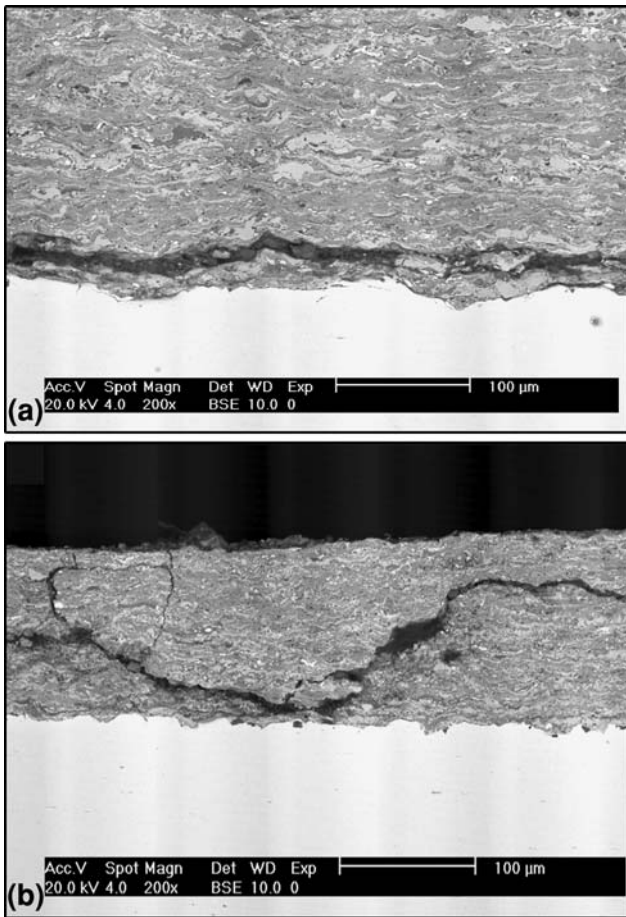
Table 1 Influence of the powder size on the quality of Fe-Al type coatings during “typical” D-gun operational conditions

Powder particle size, $\mu\text{m}$	Porosity, %		D-gun spraying process analysis
	Ra, $\mu\text{m}$	Ad Kh	
$<40$	6.4-10.3	To 19 2-5	Difficulty in the choice of the D-gun spraying parameters Thickness of the coatings differs Presence of $\text{Al}_2\text{O}_3$ films
80-125	25	To 20 $>6$	Difficulty in coating deposition
38-75	5.7	6-10 To 0.5	Repeatable thickness of coating deposited in the same spraying conditions Cohesive and lamellar structure of the coatings

Ra, roughness; Ad, adhesive porosity; Kh, cohesive porosity

Table 2 Microanalysis of the chemical composition of D-gun sprayed Fe-Al type coating deposited with  $<40$   $\mu\text{m}$  powder particles

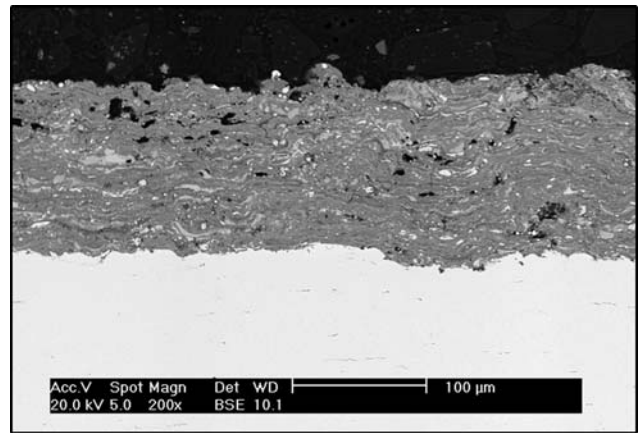
Grain area	Remarks	Content, at. %		
		Fe	Al	O
Grey	FeAl strongly oxidized	34	47	19
Light grey	$\text{Al}_2\text{O}_3$ oxide films in FeAl coating	33	32	35
Dark white	FeAl lightly oxidized	36	50	14



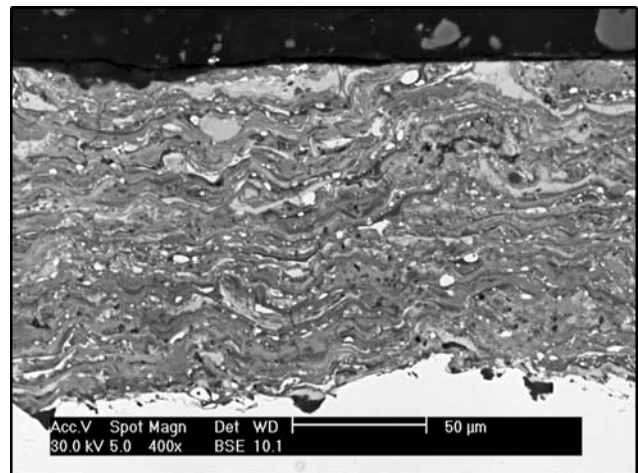
**Fig. 3** An example of D-gun Fe-Al type coating sprayed with  $<40\ \mu\text{m}$  powders. The delamination in the coating-substrate joint (a) and coating volume (b) is evident

When defining spraying parameters of Fe-Al coatings obtained from small size ( $<40\ \mu\text{m}$ ) powder particles, it was found that the application of powder carrier gas (nitrogen 2-5%) reduces the effect of oxygen activity in the metallic spray (less oxides in the coating volume). It also reduces the temperature and exit velocity of detonation products which in turn considerably influence kinetic energy of powder particles. This energy is a decisive factor which determines the quality of the structure of the coatings and particularly their cohesive porosity. The value of cohesive porosity was (2-5)% with a tendency to grow along with the volume increase of the carrier gas (nitrogen).

The subsequent D-gun spraying cycles were carried out with different parameters and various 80 to 125  $\mu\text{m}$  particle size powders were used. There were considerable difficulties in obtaining D-gun sprayed Fe-Al coating and the porosity of the deposited coating was high (over 6%) (Fig. 4). The surface roughness parameter  $R_a$  exceeded the value of 25  $\mu\text{m}$ . The difficulty in depositing Fe-Al coatings on the steel substrate with high particle size powders results most probably from the “abrasion effect.” This effect is a product of pulling out and removing



**Fig. 4** The microstructure of D-gun Fe-Al type coating deposited with 85-125  $\mu\text{m}$  particle size powder (porosity  $>6\%$ )



**Fig. 5** An example of structure of D-gun Fe-Al type coating deposited with 38-75  $\mu\text{m}$  particle size powder in the “typical” value of detonation energy (cohesive porosity  $<0.5\%$ , repeatable thickness)

deposited particles from the substrate in a cyclic interaction of the two-phase metallic spray (detonation products and the high particle sized powder).

Eventually it turned out that depositing a coating which exhibits lamellar structure, low cohesive porosity, and repeatable thickness was relatively easy with (38-75)  $\mu\text{m}$  particle size powders (Fig. 5).

Therefore, the (38-75)  $\mu\text{m}$  particle size powder was used for subsequent spraying cycles (both on 1045 steel substrate and NiAl as well as NiCr interlayers) and a number of D-gun spraying experiments with considerably different parameters controlling the detonation energy levels of the detonation mixture were carried out.

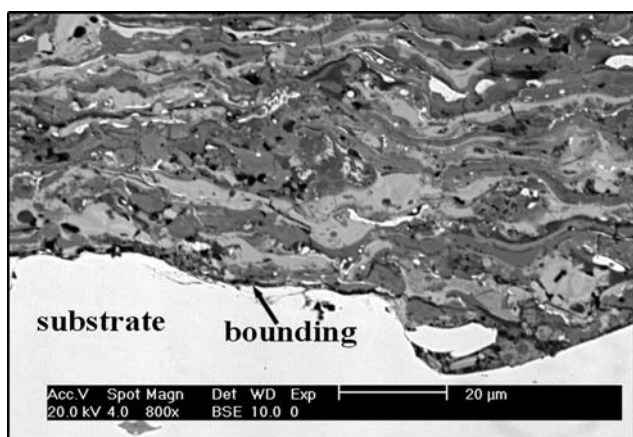
The research proved that even insignificant changes in one of the parameters of D-gun spraying (such as the volume of fuel, oxidant and carrier gases, the spraying distance or frequency) considerably influenced the

value of kinetic and thermal energies of the process which are decisive factors for the quality of D-gun sprayed coatings.

It was found that the change of the process parameters {pressure: propane-butane (0.01-0.028) MPa, oxygen (0.004-0.017) MPa, nitrogen (0.001-0.006) MPa, spraying distance (160-250) mm and the frequency of spraying (3-6) Hz} results in considerably different values of detonation energy, classified as “high,” “typical” and “low.” Limiting the parameters to the following range of pressures {propane-butane (0.019-0.028) MPa, oxygen (0.006-0.012) MPa, nitrogen (0.002-0.004) MPa and shortening spraying distance to (210-250) mm, and the choice of frequency of (4-6) Hz} results in the high value of detonation energy of the detonation blend. This is proved by high degree of deformation of the substrate surface (1045 steel and NiAl as well as NiCr interlayers) and the range of geometrical changes in lamellarly deposited coating grains which are flat and disintegrated (in microareas) (Fig. 6). High value of detonation energy of the mixture is also proved by high temperature of the sprayed powder particles. The temperature level is evaluated by signs of local overheating of the substrate material or even partial melting or evaporation of the finer powder particles. Particularly high level of detonation energy was obtained with the following parameters: {propane-butane (0.028) MPa, oxygen (0.004) MPa, nitrogen (0.001) MPa, spraying distance of 250 mm, and the frequency of 6 Hz}. When these parameters were employed it was difficult to obtain coatings even if 80-125  $\mu\text{m}$  powders were used.

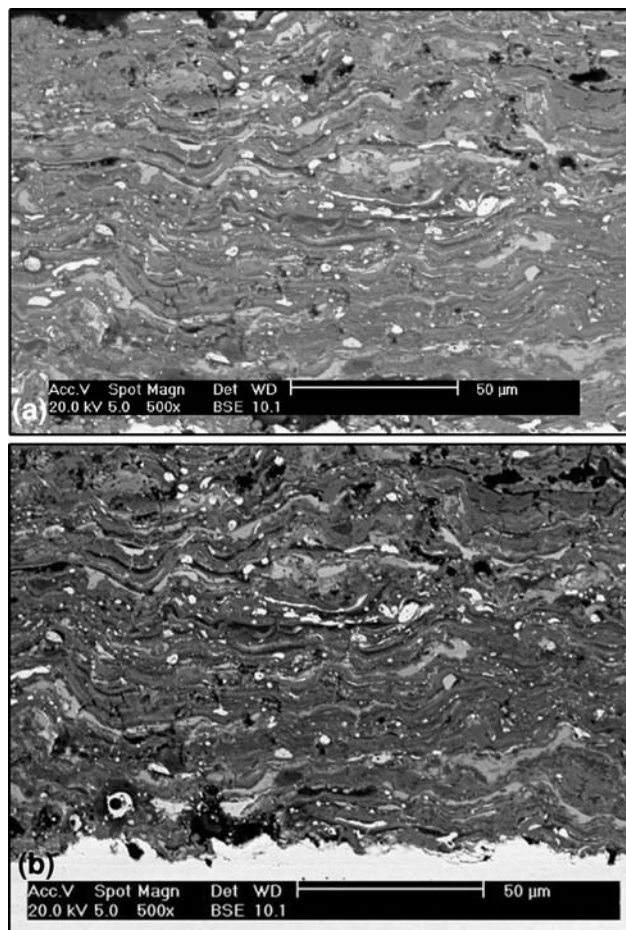
Controlling and changing individual above-mentioned parameters enabled gradual decrease in the value of detonation energy of the mixture to the values classified as “low.” The following regularities were recorded:

- The decrease in the D-gun operational frequency (from 6 to 3 Hz) results in the decrease of the temperature value of the detonation products. This is clearly seen if the spraying distance is diminished to 160 mm.



**Fig. 6** The structure of D-gun Fe-Al type coating deposited with 38-75  $\mu\text{m}$  particle size powder in the high value of detonation energy (deformation of the substrate surface)

- The reduction of pressure and, consequently, the reduction of the flow rate of propane-butane (from 0.028 to 0.019 MPa), and simultaneous increase of the oxygen volume (from 0.004 to 0.017 MPa) reduces kinetic energy of the metallic spray. As a result lower deformation level of the powder particles forming the coating is achieved. This is evident at the spraying distance reduced to (160 mm). The coatings produced in these conditions have higher cohesive porosity (even up to 5%; Fig. 7a), and in some cases (Fig. 7b) it can be assumed that the homogeneity of the flow of the two-phase metallic spray is disturbed.
- The increase in the pressure and in the flow rate of nitrogen (powder carrier and moderant of the process, preventing self-ignition in the mixing channel of the detonation gun) from (0.001 to 0.006) MPa, considerably reduces thermal and kinetic energies of the detonation products and powder particles exiting the gun in the detonation wave, regardless of the spraying distance.



**Fig. 7** The structure of D-gun Fe-Al type coating deposited at diminished value of detonation energy of the blast mixture (propane-butane 0.019 MPa; oxygen 0.017 MPa). Higher cohesive porosity of coating (a) and heterogeneous morphology of coating (b)

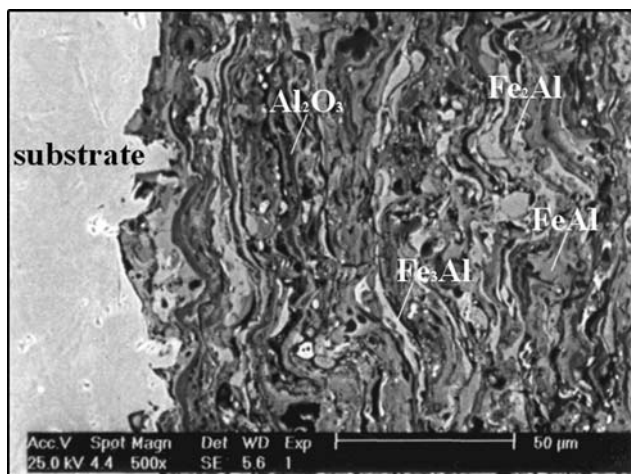
In the analysis of the results it was found that the best metallurgical quality of the Fe-Al coatings was achieved for D-gun spraying parameters which “assure” the “typical” value of the detonation energy of the mixture (Table 3). When these parameters are employed, the coatings deposited are built of lamellar splats formed by the powder particles which undergo strong plastic deformation and geometrical changes during their transformation into the coating material (Fig. 8).

Such effects of geometrical changes and simultaneous lack of signs of melting of the coating material (even in microareas) are a proof of high plasticity of intermetallic grains of the self-decomposing powder in the D-gun spraying conditions. The D-gun conditions referred to here are, in the first place, hydrodynamic interaction of the spray of detonation products, and prevailing influence of high kinetic energy, temperature and the speed of splats deformation. These changes are a proof of proper choice of spraying parameters.

Therefore, for the next stage of the research, steel 45 substrate was sprayed first with Ni-95at.%, Al-5at.% and Ni-80at.%, Cr-20at.%, 38-53  $\mu\text{m}$  particle size powders to obtain NiAl and NiCr interlayers and then it was sprayed with 38-75  $\mu\text{m}$  size Fe-62.5at.%, Al-36at.%, C-1.5at.% powders to obtain Fe-Al coating under identical conditions

**Table 3 D-gun spraying conditions for the “typical” value of detonation energy of the blast mixture for 38-75  $\mu\text{m}$  particle sized powders**

Detonation gun spraying conditions		
Fuel gas	Propane-butane	0.017 MPa
Oxidant	Oxygen	0.009 MPa
Carrier gas	Nitrogen	0.003 MPa
Spraying distance	...	240 mm
Detonation frequency	...	5 Hz



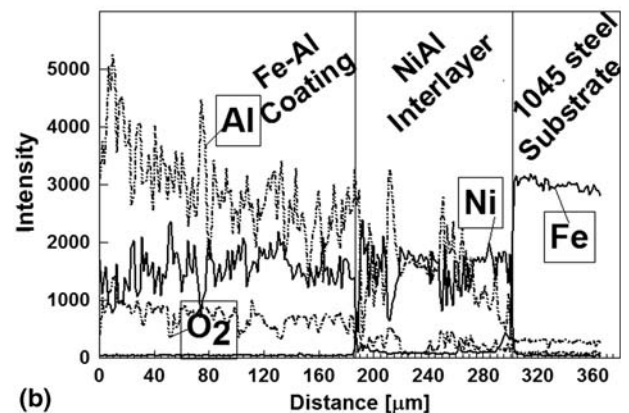
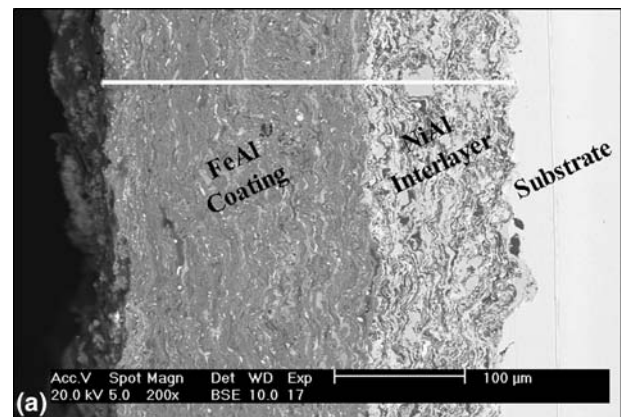
**Fig. 8** Morphology of strongly deformed, dispersive grains of the D-gun FeAl type coating, deposited with (38-75)  $\mu\text{m}$  particle sizes powder in “typical” detonation gun spraying parameters (propane-butane 0.017 MPa, oxygen 0.009 MPa, nitrogen 0.003 MPa, distance 240 mm, frequency 5 Hz)

of both D-gun spraying process (defined here as “typical”) and substrate pre-processing.

During the analysis of the metallographic examination results it was found that in case of both NiAl and NiCr intermediate layers their structure was not identical despite identical D-gun process conditions used (Fig. 9a and 10a). The aluminum-rich NiAl intermediate layer structure was similar to the basic FeAl coating, i.e. it had a layered structure and varied degree of oxidation of various phases from the Ni-Al system. The NiCr intermediate layer was also multiphase; however, less prominent due to lower affinity to oxygen. It was formed with the flattened Ni(Cr) solid solution grains containing spheroidal dispersions of the chromium enriched oxide.

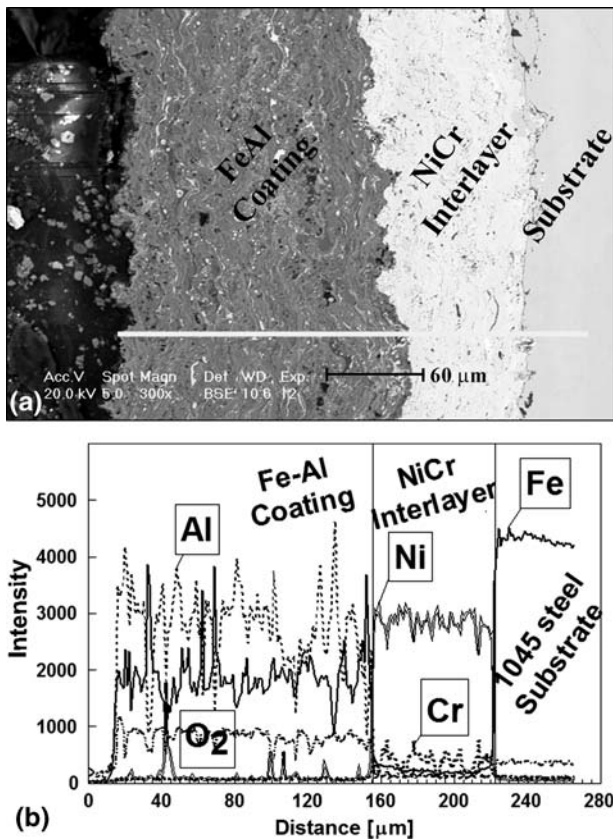
The observed differences in the morphology of the intermediate NiAl and NiCr layers were clearly reflected in the results of the linear microanalysis of the chemical composition (Fig. 9b and 10b).

In the plots (Fig. 9b) of the variations of the aluminum, oxygen and iron (for the FeAl coating) or nickel (for the intermediate NiAl layer), the peak aluminum oscillations (which are simultaneous to the minimum concentration level of iron or nickel) correspond to the passage of the analyzing beam of electrons through the oxide layers.



**Fig. 9** Surface layer of D-gun sprayed FeAl/NiAl type (a) and typical heterogeneity of chemical composition of coatings along the cross section (b)



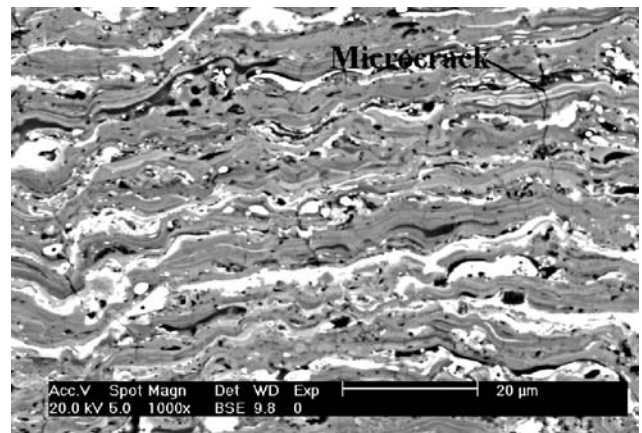


**Fig. 10** Surface layer of D-gun sprayed FeAl/NiCr type (a) and typical heterogeneity of chemical composition of coatings along the cross section (b)

The plots of the linear chemical analysis of the NiCr intermediate layer (Fig. 10b) exhibit completely different character. The NiCr intermediate layer is also multiphase; however, less prominent due to lower affinity to oxygen and the solubility of chromium in nickel. Therefore, the chromium concentration fluctuations (related to the formation of the oxide phase) are in this layer less pronounced than the fluctuations of aluminum in the FeAl coating or NiAl intermediate layer.

As a result of analysis of morphology and phase allocation of D-gun sprayed Fe-Al type coatings on the NiAl and NiCr interlayers, it was found that the coatings just as the coatings deposited onto 1045 steel have a microstructure typical for the D-gun spraying method. Their structure consists of the layered and flattened grains of the intermetallic phases from the Fe-Al diagram with predominant FeAl phase. The coatings, however, have varied chemical composition (according to the diagram the compositional range of the FeAl intermetallic is wide and ranges from 36 to 50 at.% Al).

On the basis of the point EDS analysis (Fig. 8), the low-aluminium  $Fe_3Al$  phases (the brightest areas) and the  $FeAl_2$  phases (dark grey) were identified where the aluminium content level exceeds the upper range of FeAl phase. It should be emphasized that the “composite” nature of D-gun sprayed Fe-Al type coatings is triggered



**Fig. 11** Microstructure of the D-gun sprayed Fe-Al type coating deposited with 38-75  $\mu\text{m}$  particle size powder at “typical” value of the detonation energy (characteristic microcracks running perpendicularly to the boundaries of the lamellar splats)

by the existence of dispersive intermetallic phases with different ordering (depending on the aluminium contents) and especially considerable participation of oxygen influencing the creation of stable and durable oxide layers (mainly  $Al_2O_3$ ).

The aluminum-rich Fe-Al powder is so chemically active in the D-gun spraying process that all powder particles are already oxidized and the deposited coatings always contain oxide films inside the coating and at the internal interfaces. It is the formation of the oxide films identified on the basis of the point EDS analysis (the darkest areas of the coating structure; Fig. 8) which brings about the lamellar structure of the FeAl intermetallic coatings and defines their composite character.

Most probably it is the presence of  $Al_2O_3$  scales and high ordering of intermetallic phases with increased amount of aluminium coupled with high kinetic energy of D-gun spraying which is the reason for the formation of visible microcracks (Fig. 11) running perpendicularly to the lamellar splats. This, in turn, proves that the splat boundaries are not prone to development of cracks and have high degree of cohesive strength.

One can also assume that the crack resistance of the identified earlier FeAl phases with different oxidation degree is similar, as the microcracks run through several layers of the coating splats (their usual length is up to 25  $\mu\text{m}$ ).

Perpendicular orientation of the microcracks to the coating surface allows one to infer that the cracking is caused by dominating thermal tensile stresses in the condition of high brittleness of the phases and is initiated during coating cooling down, as a result of difference in linear coefficients of thermal expansion which equal  $\alpha = (18 \times 10^{-6})$  for FeAl and  $\alpha = (12 \times 10^{-6})$  for steel.

The quantitative results of cohesive porosity (Fig. 5), which are considered important data for evaluation of the metallurgical quality of D-gun sprayed Fe-Al type coatings and NiAl and NiCr interlayers, prove that the coatings deposited are extremely cohesive in their volume (the porosity value is lower than 0.7%).

**Table 4** Porosity and surface roughness of D-gun sprayed Fe-Al type coating and D-gun sprayed interlayer coating (NiAl and NiCr) deposited during “typical” D-gun operational conditions

Powder particle size, $\mu\text{m}$	Kind of D-gun sprayed coating	Surface roughness Ra, $\mu\text{m}$	Porosity, %		
			Kh	Kind of joint	Ad
38-75	Fe-Al	5.7	To 0.5	1045 steel/Fe-Al	6-10
38-53	NiAl	4.6	To 0.5	1045 steel/NiAl	To 0.7
	NiCr	6.4	To 0.7	NiAl/Fe-Al	To 0.5
				1045 steel/NiCr	To 0.9
...	1045 steel after abrasive blasting	15.6	...	NiCr/Fe-Al	To 0.8

Ra, roughness; Ad, adhesive porosity; Kh, cohesive porosity

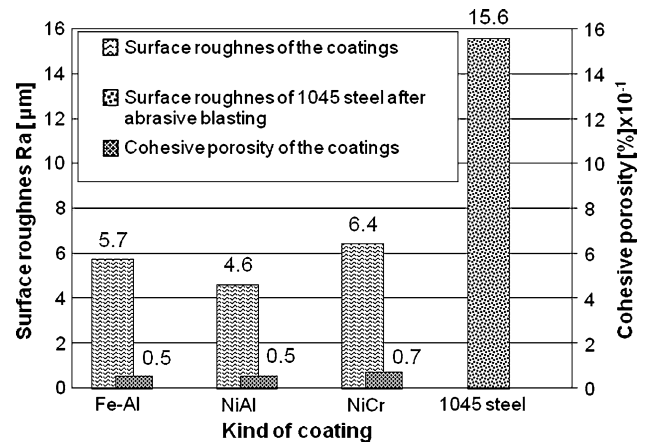
Equally low level of adhesive porosity (<1%) was found in the NiAl and NiCr-1045 steel bonds as well as in the NiAl and NiCr-FeAl joints. It equaled less than 0.5% for NiAl interlayer and approximately 0.8% for NiCr interlayer (Table 4). Although the process of chemisorption is especially intensive in the areas of increased surface energy (inclusions, vacancies, plasticization areas and stress concentrations), i.e. in the areas of surface layers of NiAl and NiCr interlayers, the analyzed areas did not reveal any tendency to accumulate impurities and the wavy line (Fig. 9a and 10a) proves a typical adhesive bond where plasticized powder particles are sprayed with high kinetic energy and get “mechanically clenched.”

It was found that the porosity level of Fe-Al coating-steel substrate joint (where the interlayers had not been deposited) was, unfortunately, one order of magnitude lower (6-10% on average) which would undoubtedly influence the adhesive strength of the coating.

This observation has a very important technological aspect—the results obtained prove that the application of interlayers is desirable to increase the level of substrate adhesivity of D-gun sprayed Fe-Al type coatings. The interlayers will also ensure independent control of functional features of the coating system and limit the influence of disadvantageous gradients (e.g. temperature and stresses). This will additionally influence adhesive strength of the coating.

The analysis of the origins of porosity in the D-gun process proves that both cohesive and adhesive porosities are enhanced by the presence of single, individual powder particles-coating grains which did not undergo sufficient deformation during their collision with the substrate or coating. This is most probably due to the partial distortion of the laminar flow or the change in the thermo-dynamic parameters of the detonation product.

Presented hypothesis concerning the process of porosity development is supported by the lamellar structure of the coating material, resulting from “shooting” plasticized powder particles at the substrate material. The particles undergo strong deformation and build up “composite” coating layers in the process of cooling down and reheating (until plasticized) during successive influxes of the product detonation waves.

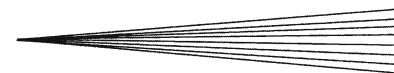


**Fig. 12** Surface roughness of D-gun sprayed (Fe-Al, NiAl, NiCr) coatings and substrate 1045 steel in comparison with cohesive porosity illustrated by photomicrograph quantitative analysis carried out with (SEM) Philips XL30/Lab6 programmed with SIS software

In the final phase of evaluation of metallurgic quality of the coatings, attention was paid to the geometry of their surface layer. The surface roughness is a very important parameter influencing the properties of the D-gun sprayed coating and depends on many physical-chemical factors which easily undergo changes in this high energy process.

The examination of the surface roughness of the D-gun sprayed Fe-Al type coatings deposited with (38-75)  $\mu\text{m}$  particle size powder in typical D-gun spraying conditions showed moderately similar value of roughness (average  $Ra \approx 5.7 \mu\text{m}$ ) in comparison to the roughness of the NiAl and NiCr coatings deposited with (38-53)  $\mu\text{m}$  particle size powder where the average roughness equaled correspondingly  $Ra \approx 4.6 \mu\text{m}$  and  $Ra \approx 6.4 \mu\text{m}$ . At the same time it was shown that the mean arithmetic deviation of the roughness profile of the surface of 1045 steel gritted before D-gun spraying was  $Ra \approx 15.6 \mu\text{m}$ .

On the basis of the claim stating that the coating surface roughness should correlate to the degree of cohesive porosity, an analysis of both parameters was carried out (Fig. 12). It showed that the Fe-Al and NiAl coatings have lowest surface porosity (Ra parameters



equal to 4.6 and 5.7  $\mu\text{m}$  respectively). These coatings, having undergone the D-gun spraying processing, show considerably lower porosity level (and the highest cohesiveness of the structure).

## 4. Conclusions

The paper presents evaluation of both the influence of particle size parameter of the FeAl self-decomposing powder and D-gun spraying parameters upon the metallurgical quality of the deposited coatings.

One of the most important research conclusions is the claim that the best metallurgical quality of the coatings is achieved by employing self-decomposing powder particles the sizes of which are (38-75)  $\mu\text{m}$ .

It was also determined that the quality of the coatings is equally influenced by the detonation energy of detonation mixture. The energy value is controlled by the volume of fuel gas, oxidant and carrier, spraying distance and spraying frequency.

It was found that the best metallurgical quality of the coatings was achieved for the parameters of D-gun spraying process which ensure the mixture detonation energy value defined as "typical." The deposited coatings are built of lamellar splats which result from powder particles transformed in the D-gun spraying process, i.e. undergoing plastic deformations and geometrical changes.

Geometrical changes and simultaneous lack of signs of melting of the Fe-Al coating material (even in microareas) are a proof of high plasticity of intermetallic grains of the self-decomposing powder (with prevailing FeAl phase) in the D-gun spraying conditions.

It was found that the grains of all coatings examined were (almost all over their surface) covered with complex oxide films (identified mainly as  $\text{Al}_2\text{O}_3$ ). The films could also be identified in the coating volumes and in grain boundaries.

The abundance of oxides and highly ordered high aluminium FeAl phases is the reason of the formation of a number of visible microcracks running perpendicularly to the surface (within individual lamellas) with characteristic faults in the lamellas identified as solid solution. The solid solution composition is highly similar to stoichiometric FeAl. This fact underlies the claim that the splat boundaries are not prone to the development of cracks and have high degree of cohesive strength.

It was proved that practically in all coatings which were examined the porosity in the FeAl/interlayer joint area was low (below 1%). This, together with the wavy line of the bond of the coatings, exerts positive influence on the adhesive strength.

High cohesive porosity (6-10)% was found in the Fe-Al joint which justifies the implementation of interlayers during depositing intermetallic Fe-Al coatings in the D-gun spraying process.

On the basis of the claim that the value of cohesive porosity of the D-gun sprayed coatings should correlate with the roughness value of the surface (characterized by

mean arithmetic deviation of the roughness parameter  $R_a$ ), the analysis of these parameters was carried out (Table 4). The lowest surface roughness value ( $R_a = 4.6$  and 5.7  $\mu\text{m}$ ) was found for the coatings Fe-Al and NiAl with the lowest cohesive porosity (below 0.5%).

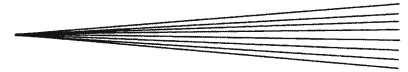
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