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ADHESION OF COLD-SPRAYED COATINGS AND THE WORKABILITY OF THE APPLIED SUBSTRATE

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Abstract: *Modern testing equipment for mechanical properties of materials allows for highly precise measurements of both hardness and the elasticity modulus. Advances in nanoindentation techniques, coupled with sophisticated software, make it possible to generate detailed load and unload curves. In particular, the area between the load and unload curves has been recognized as a new metric for evaluating the mechanical characteristics of a material. This area quantifies the energy absorbed during the loading process and released during unloading, which can be thought of as the 'workability' of the material. This term 'workability' is used because the value is calculated based on the area between the two curves, represented on a plane where the force (F) is plotted against the displacement of the indenter (d). The larger the area, the more work the material undergoes during deformation, thus giving an indication of its mechanical robustness and durability under stress.*

In the study, titanium coatings were applied using a cold gas spraying technique to several different metal substrates, including brass, steel, titanium, Al7075, copper, magnesium, and Al2024. Cold gas spraying was chosen because it is an effective method for depositing coatings without excessive heating, thus preserving the structural properties of both the coating and the substrate. In optimizing the spray parameters, the best conditions for coating titanium on the Al7075 alloy were identified: a spraying pressure of 40 bar, a gas temperature of 800°C, a gun traverse speed of 4 m/s, and a distance of 50 mm between the spray gun and the sample.

After the coatings were applied to the different substrates, several mechanical tests were conducted, focusing on hardness, elasticity modulus, and workability. A total of 36 nano-hardness and elasticity tests were performed on each substrate, allowing for the calculation of average values for each property. These results were summarized in a table, and a graph was plotted to illustrate the relationship between workability and elasticity. Interestingly, the experimental data points did not show a clear correlation between these two properties, indicating that the behavior of materials in terms of elasticity and workability may not be directly linked or may be influenced by other factors.

In addition to the mechanical property measurements, adhesion tests were conducted to determine how strongly the titanium coatings adhered to each substrate. The results did not show a strong correlation between adhesion strength and either workability or elasticity, suggesting that the adhesion process is likely governed by more complex factors beyond just the mechanical properties of the substrate. These discrepancies may be due to the intricate nature of the adhesion mechanisms or limitations in measurement accuracy, which could obscure any potential trends or relationships.

Keywords: *Cold Spray, hardness, elastic modulus, adhesion*

1. Introduction

Modern systems for transporting liquids or gases can interact with the materials used in their construction, such as pipelines, pumping stations, and pumps. When dealing with the transport of liquids or chemically active gases, the materials used in pipeline construction must demonstrate mechanical, chemical, and thermal resistance. To satisfy these requirements while simultaneously reducing transport system costs, thin coatings—measuring fractions of a millimetre—are applied to the substrate material. These coatings are made from materials that meet the necessary performance criteria.

Adhesion refers to the phenomenon where attractive forces between the substrate and the coating material create a durable bond. These forces arise from intermolecular interactions, including hydrogen bonds, Van der Waals forces, and electrostatic interactions. The value of adhesion forces is influenced by factors such as surface preparation and cleaning, surface roughness and topography, surface energy of the materials, and the polarity of the molecules [1]. Naturally, in addition to adhesion, maintaining the integrity of the coating itself is also crucial. The measure of a coating's integrity is the value of cohesion forces. Cohesion is the phenomenon of attraction between molecules of the same substance, which ensures that the material retains its structural integrity. In cold spray (CS) processes, numerous factors influence the value of adhesion. In their work, G. Prashar and H. Vasudev discussed methods for evaluating adhesion and identified the factors affecting its magnitude [2].These factors include gas temperature, gas type, the type of coating material, grain size and shape, and the degree of grain oxidation. Additionally, the properties of the substrate, such as hardness, temperature, and surface roughness, play a significant role.
An innovative coating application method is the thermal spray technique, specifically Cold Spray, in which the coating material is applied to the substrate without altering the properties of the deposited material layer.

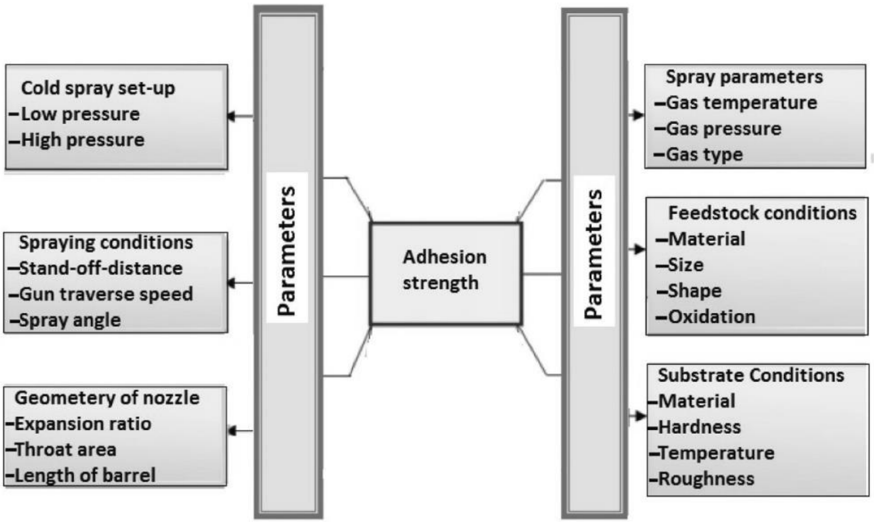

Fig. 1. Selected CS spraying parameters affecting adhesion

The essence of the Cold Spray process lies in imparting kinetic energy to the coating material particles, which, after passing through a converging-diverging nozzle, impact the substrate. When the particles reach the appropriate velocity, they adhere to the substrate, forming a coating. Depending on the coating material and the substrate, particle velocities must range from 300 to 1200 m/s. The successful deposition of the coating is determined by achieving the proper velocity—called the threshold velocity. This velocity is achieved by using a carrier gas with pressures in the range of 0.5–15 MPa. The temperature of the carrier gas is lower than the melting temperature of the feedstock material and typically ranges from 0 to 800°C. Common process gases include air, nitrogen, and helium. Figure 2 illustrates the Cold Spray process concept.
A powder of the coating material, with particle sizes ranging from 5 to 150 μm, is introduced into the gas stream. In the initial stage of the metal spray process, surface activation occurs, oxides are removed, and the substrate is cratered. In the subsequent phase of spraying, the actual coating is formed through mechanical interlocking, plugging, and the occurrence of adiabatic shear [3, 4].

Adiabatic shear instability refers to the loss of the material's shear strength, causing the deformation mechanism to shift from plastic to viscous. Therefore, the shape and size of the particles are of significant importance. In the spraying process, it is crucial for the particles to achieve sufficient kinetic energy. Particles with low mass and small size are preferred. Large particles have a greater surface area, which influences the amount of oxidized coating material. This process is undesirable from the perspective of coating formation and its adhesion to the substrate. From the perspective of the spraying process, it is preferable for the particles to have similar sizes. Such a particle size distribution ensures a precise determination of the critical velocity. As shown in Figure 2, the key component of the cold gas spraying system is the de Laval nozzle.

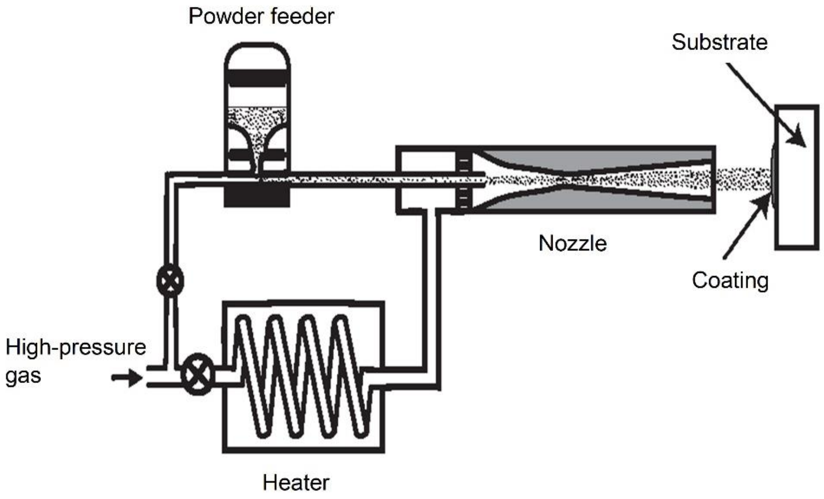

Fig. 2. The concept of cold gas spraying

The Cold Spray process is based on imparting kinetic energy to particles. To achieve this, particles are introduced into a gas stream (such as nitrogen, hydrogen, or air) before entering the nozzle, where they pass through a region of heated gas. As previously noted, the system used in the study is capable of heating the gas to 800°C. The heated gas increases the pressure at the inlet, which in turn elevates the velocity of the coating particles. After passing through the nozzle, the particle velocity can reach supersonic speeds. In the first, convergent section of the nozzle, the flow is accelerated to the speed of sound, while in the second, divergent section, the flow is further accelerated to supersonic speeds through an expansion process similar to that used in jet engines. Particles that attain high velocities, while maintaining relatively low temperatures, are directed in a solid state toward the substrate, typically at a right angle or near-right angle. The plastic deformation of the particles upon impact activates mechanisms that bind the forming coating to the substrate. These mechanisms involve jamming and plugging of the coating material with the substrate. The required particle velocity upon collision with the substrate is determined by the hardness (H) and Young's modulus (E) of both the coating material and the substrate. The Cold Spray process is fundamentally based on imparting kinetic energy to particles. In this process, particles are introduced into a gas stream—typically nitrogen, hydrogen, or air—before they enter the nozzle, where they are subjected to a region of heated gas. As previously noted, the system employed in the research is capable of heating the gas to temperatures as high as 800°C. The heating of the gas leads to an increase in pressure at the inlet, which, in turn, results in an increase in the velocity of the coating particles. After passing through the nozzle, the particles reach velocities that can exceed the speed of sound, entering the supersonic range. In the first, convergent section of the nozzle, the gas flow is accelerated to the speed of sound, where the velocity of the gas increases progressively. In the subsequent, divergent section of the nozzle, the gas is further accelerated to supersonic speeds through an expansion process, similar to the principle employed in jet propulsion systems. This acceleration of the gas stream imparts significant kinetic energy to the particles, which is crucial for the successful deposition of the coating.

Upon achieving high velocities while maintaining relatively low temperatures, the particles are directed toward the substrate in a solid state, typically at a right angle or close to a right angle. The impact of these high-velocity particles induces plastic deformation upon collision, which is a critical step in the formation of the coating. This deformation activates several key mechanisms that facilitate the bonding of the coating to the substrate. These mechanisms include jamming, where particles interlock upon impact, plugging, which refers to the filling of surface irregularities by the particles, and mechanical interlocking, where the particles become embedded within the microstructure of the substrate. Collectively, these processes contribute to the establishment of a strong adhesive bond between the coating and the substrate surface, ensuring effective coating adhesion.

The specific particle velocity required for successful deposition is influenced by the intrinsic material properties of both the coating and the substrate. These properties include hardness (H) and Young's modulus (E), both of which are vital in determining the extent of plastic deformation that can occur upon impact and, consequently, the quality of the bond that forms between the particles and the substrate. Hardness influences the resistance to deformation, while Young's modulus determines the material's ability to withstand elastic deformation. The interplay of these factors ensures that the coating adheres to the substrate with sufficient strength, thus enabling the coating to perform effectively in its intended application. Properly balancing these material properties with the correct particle velocity ensures that the coating meets the required performance characteristics, such as durability, wear resistance, and adhesion strength.

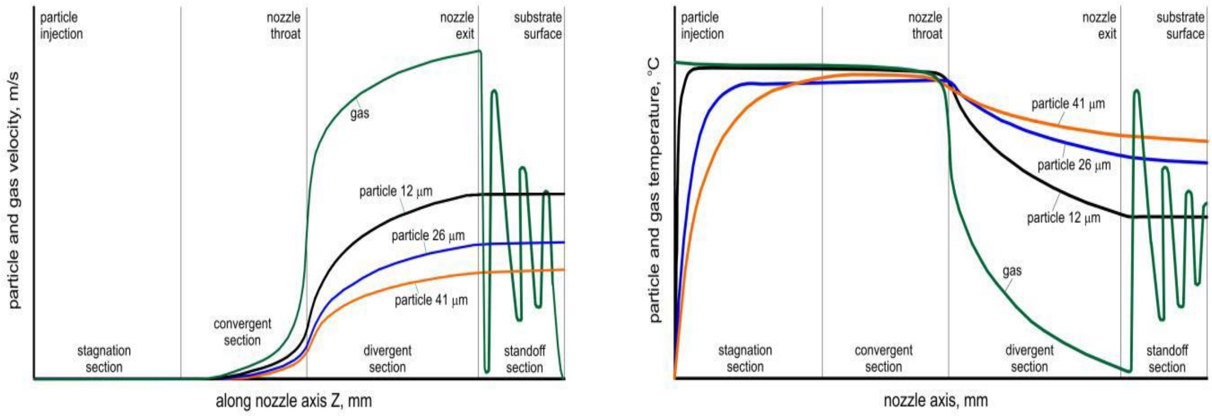

Fig. 3. The distribution of gas temperature and the velocity of the coating material particles [5]

The deposited coatings can serve a wide range of functions, each contributing to the enhancement of the substrate's properties. Functional coatings involve the deposition of a material, distinct from the substrate, onto the substrate surface to impart new functionalities, such as corrosion resistance, wear resistance, electrical conductivity, and viscosity reduction, among others. Part remanufacturing involves applying a material similar to the substrate onto a prepared surface to repair geometric defects in a component, which may have occurred due to wear, corrosion, or manufacturing imperfections. This process allows for the restoration of parts to their original shape and functionality. Additive manufacturing, enabled by cold spray technology, allows the creation of thick and very thick layers (several centimeters in thickness). This capability has opened new possibilities for producing parts with geometries close to their final form, reducing the need for post-processing and enhancing the efficiency of production.

Cold Spray, a technique introduced by Papiryn approximately 30 years ago, has become increasingly recognized, though it is still regarded as innovative in some industries [6]. Over recent years, the cold spray process has garnered significant attention due to its ability to produce dense, thick metal deposits while maintaining the purity of the sprayed powders. This is achieved without inducing phase transitions in the material—there is no formation of new phases or oxidation of the coating material during the process. As a result, the process preserves the integrity of the material properties, making it highly advantageous for various applications.

The benefits of Cold Spray technology have become widely acknowledged, particularly in fields such as aerospace, biomedicine, and energy. Its ability to deposit high-quality coatings without excessive thermal impact on both the coating and substrate materials has made it a valuable technique for producing durable, high-performance components.

2. Materials and Methods

In the experiment, the focus was placed on studying adhesion in relation to the mechanical properties of both the coating material and the substrate, while considering the values of the spray parameters. The shape and size of the coating material particles were consistent across all cases.

2.1 Research Equipment and Methodology

The equipment used in the cold gas spraying process is shown in Figure 4, highlighting the key components. As mentioned earlier, the central element of the spraying system is the de Laval nozzle (1), the gas preheating area (2), the powder material feeder, the electronic process control system for spray operation (4), and the robot that holds the de Laval nozzle and heaters, enabling precise control over the coating application process. This setup represents a typical industrial system, the Impact Innovations 5/8, in conjunction with the Fanuc M-20iA robot.

Fig. 4. The Cold Gas spraying coating system setup [7]

The adhesion value of the obtained coating was tested using a nanoindenter, applying an indentation force of 20 mN with an indenter loading and unloading rate of 40 mN/min. Based on the conducted tests, hardness (H) and Young's modulus (E) were determined. The research was carried out using a nanoindenter from NANOVEA. Measurements were performed at 36 points on cross-sectional samples of all coatings.

The adhesion of the coatings was evaluated by measuring the vacuum generated beneath a mushroom-shaped probe that was attached to the coating surface. This method involves placing the probe onto the coating, creating a sealed contact between the probe and the surface. A vacuum is then applied, and the resulting pressure difference is monitored. The degree of vacuum generated reflects the strength of the bond between the coating and the substrate. A higher vacuum indicates stronger adhesion, as it suggests that the coating is securely attached to the substrate, with minimal air gaps or separation. This technique provides valuable insights into the quality and effectiveness of the coating's adhesion properties.

Fig. 5. Vacuum pump and detachment mushroom holder

The detachment mushroom holder was adhered to the coating and subsequently detached by creating a vacuum ranging from 0 to 50 MPa. The diameter of the mushrooms used was 15 mm. Each coating was tested three times, and the result assigned to the coating was considered the average value. To characterize the morphology of the powders and their metallographic cross-sections, a scanning electron microscope (SEM, E-SEM FEI XL 30) was employed. This method allows for precise measurements of the coating's adhesion properties, with the vacuum-induced detachment force serving as a key indicator of the bond strength between the coating and the substrate. The use of SEM enabled a detailed analysis of the powder morphology and microstructure, providing valuable insights into the coating's characteristics and quality at the microscopic level.

2.2 Coatings and Substrate Types

Titanium powder with a particle size distribution shown in Figure 6 was selected as the coating material. For the spraying process, titanium particles with a granulometry range of 20–70 µm were chosen. The average particle size was approximately 31.5 µm. The Ti particles were approximately spherical in shape.

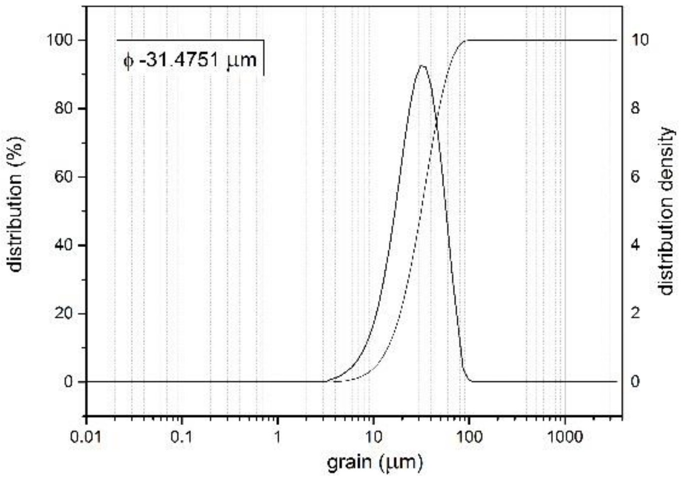

Fig. 6. The granulometric distribution of Ti particles

Seven metal plates were selected as substrates for the application of the titanium coating. These substrates were rectangular plates, each measuring 25 x 3 x 0.5 cm, which provided an adequate surface area for uniform coating deposition. To ensure optimal adhesion between the coating and the substrate, the metal plates underwent surface preparation processes before coating. Specifically, the substrates were sandblasted to increase surface roughness, which is crucial for enhancing the mechanical interlocking between the coating and substrate. Additionally, the plates were degreased to remove any oils, contaminants, or residues that could interfere with the coating process and adhesion quality.

Table 1 presents a detailed list of the materials used for the substrates, including their corresponding Young's modulus (E), which is a critical parameter in evaluating the mechanical properties of the substrate material. Young's modulus provides insights into the material's stiffness, which plays an important role in determining the behavior of the coating during the deposition process and under operational conditions.

Table 1: List of substrate materials

Substrate	E [GPa]
Al2024	175,0±11,0
Al7075	155,0±9,0
Brass	110,0±7,5
Copper	120,0±8,0
Magnesium	1,0±0,1
Steel	210,0±7,6
Titanium	150,0±8,0

To ensure the accuracy and reliability of the experimental results, all measurements were carried out using a single, consistent machine setup. This approach minimized variability in the testing conditions, contributing to the precision of the obtained data and making the results more comparable across different tests. The values of Young's modulus (E) were measured using the NANOVEA nanoindenter.

2.2 Selection of Experimental Parameters - G. Taguchi's Statistical Method

The Taguchi method is a structured approach to experimental design aimed at process optimization and the creation of high-quality systems. It reduces the number of experiments needed while maintaining precision and consistency. This approach, based on factorial design, utilizes an orthogonal array—a set of experiments performed under different conditions—to assess and optimize the selected factors (variables). Known for its simplicity, effectiveness, and reliability, the Taguchi method is widely used in optimization tasks [8, 9].

The Taguchi design utilizes a loss function, which is transformed into a signal-to-noise (S/N) ratio to assess the deviation between experimental outcomes and desired targets. The S/N ratio is calculated as the ratio of the mean response to its standard deviation, serving as a tool to pinpoint the optimal settings for each factor to improve performance. In S/N analysis, performance characteristics are classified into three categories: lower-the-better, higher-the-better, and nominal-the-better. Additionally, statistical analysis, often through analysis of variance (ANOVA), is employed to identify the most significant variables. By combining the Taguchi design with ANOVA, a powerful method for determining the optimal process conditions is achieved.

The S/N ratio represents the relationship between the mean (signal) and the standard deviation (noise) and is influenced by the quality characteristics of the product or process being optimized. Commonly used S/N ratios include nominal-is-best (NB), lower-the-better (LB), and higher-the-better (HB).

In the conducted experiment, the HB procedure was chosen due to the goal of achieving the highest possible adhesion value of the coating to the substrate.

Funkcha S/N przybiera postać

$$ {}^{S}\!/_{N} = -10 log_{10} \frac{1}{n} \sum_{i=1}^{1} \frac{1}{y_i^2} \tag{1} $$

where y is the measured quantity.
The selection of parameters was carried out for a coating made of titanium on a titanium substrate. In the experiment, controlled parameters and their value ranges were chosen, which are presented in Table 2.

Table 2: The set of cold gas spray parameters

T [^oC]			p [bar]			d [mm]			V [mm/s]		
700	750	800	30	37	45	30	40	50	300	400	500

The selection of parameters for the deposition of a titanium coating on a titanium substrate was carried out using industrial statistics based on the Genichi Taguchi method. One of the main advantages of this method is the significant reduction in the number of experiments required, along with the flexibility to adjust the parameter values in real-time. Four controlled parameters were chosen: temperature (T), pressure (p), velocity (V), and distance (d), with each parameter having three possible values. Without the application of the Taguchi method, the total number of combinations would be $3^4 = 81$, which is a substantial number of experiments. However, by applying the Taguchi statistical method, the number of experiments was reduced to just 9. The experimental design and its details are presented in Table 3. This approach not only saves resources but also streamlines the process of determining the optimal conditions for the coating deposition.

Table 3: Experimental plan according to Taguchi

Trial no.	Input parameters			
	T [^oC]	p [bar]	d [mm]	V [mm/s]
1.	700	30	30	300
2.	700	37	40	400
3.	700	45	50	500
4.	750	30	40	500
5.	750	37	50	300
6.	750	45	30	400
7.	800	30	50	400
8.	800	37	30	500
9.	800	45	40	300

After completing the parameter selection, a verification experiment was carried out to assess the effectiveness of the chosen parameter values in controlling the spray process. In this phase, the experiment aimed to confirm whether the selected conditions would produce the desired coating properties, such as optimal adhesion, uniformity, and thickness. This verification step helped ensure that the process was operating within the intended specifications and provided insights into any potential adjustments needed for further optimization.

3. Results and Discussion

According to formula (1), the calculated S/N (ETA) function and its solution are presented in Figure 7. Each input parameter was assigned specific values (Table 2). After completing the full set of experiments in accordance with Table 3, a statistical analysis was performed to determine the optimal values of temperature (T), pressure (p), velocity (V), and distance (l), and their influence on the S/N function was analyzed. The primary factor determining the quality of the experiment was the adhesion of the coating to the substrate.

For temperature, the value of the ETA function increases with rising temperature and reaches its maximum at a temperature of 800°C. A similar approach was used to determine the optimal pressure (p), which did not reach saturation within the designated range. The highest ETA value occurred at a pressure of 45 bar. An analogous procedure was followed for the spray distance (l) and velocity

(V). The ETA diagram for the distance l shows that the value for a distance of 50 mm is close to saturation. Similarly, in the analysis of ETA for the spray head velocity, the maximum value of the analyzed function was found at the extreme value of 500 mm/s, within the range of 300 to 500 mm/s.

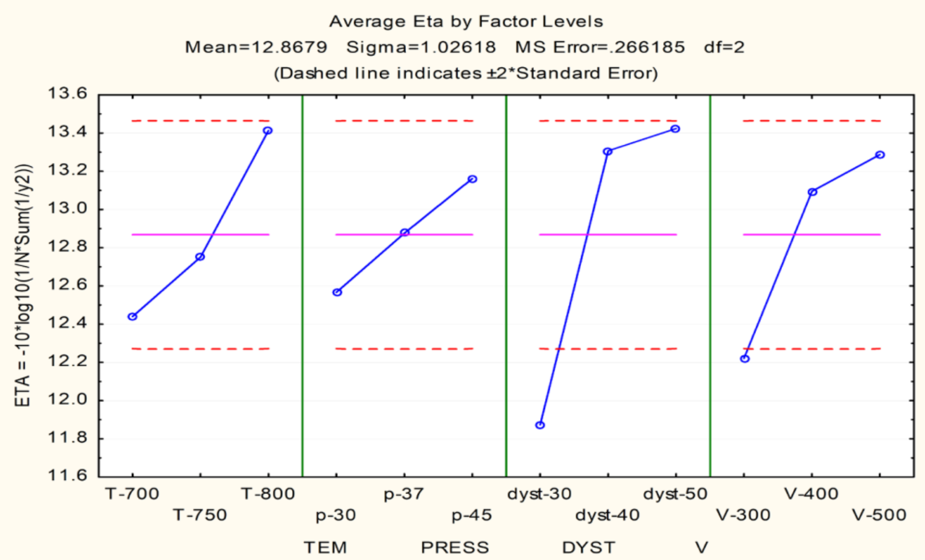

Fig. 7. Diagram of the values of the process control parameters for cold gas spraying

In optimizing the spray parameters, the best conditions for coating titanium onto titanium were identified: a gas temperature of 800°C, a spraying pressure of 45 bar, a distance of 50 mm between the spray gun and the sample, and a traverse speed of 500 mm/s. The selection of parameter ranges was mainly determined by the capabilities of the spraying equipment. Despite the identification of the optimal values within the specified range, the analysis suggests that higher values for temperature and pressure could potentially improve the process further.

The following Figure 8 presents selected cross-sections of the sprayed coating, obtained under the statistically chosen parameters that control the coating application process. The coating is uniform, showing no cracks or porosity. A clear interface between the coating and the substrate is visible, exhibiting the typical mechanical bonding features such as jamming and interlocking of the coating material with the substrate. To assess the quality of the deposited coating, control measurements of hardness (H) and Young's modulus (E) were carried out on cross-sectional specimens. These measurements are critical in evaluating the mechanical performance of the coating and its adhesion to the substrate.

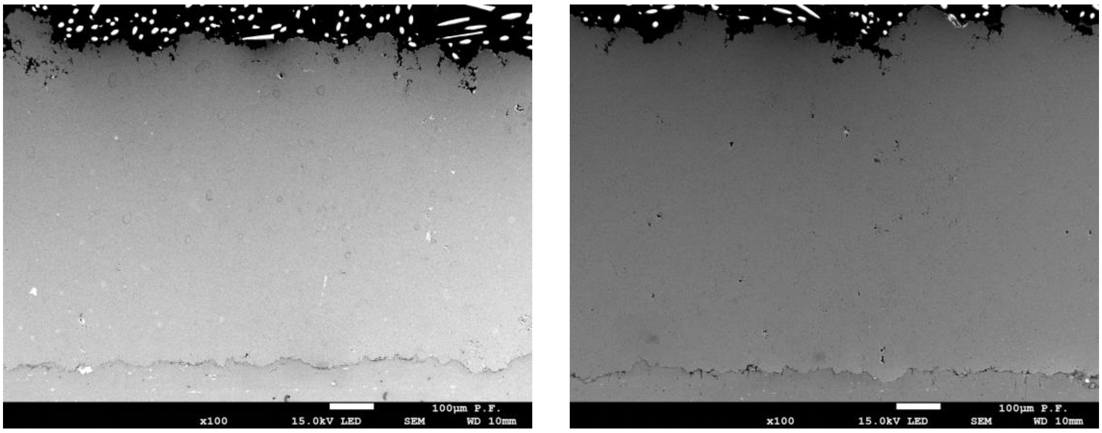

Fig. 8. Cross-section of the coating and substrate of the Ti coating on Ti.

The obtained results fluctuated around the mean with an error of less than 6%. This result was considered satisfactory.

The adhesion test for each coating was repeated three times, with the adhesion value taken as the average. The results were collected and graphically presented in Figures 9 and 10.

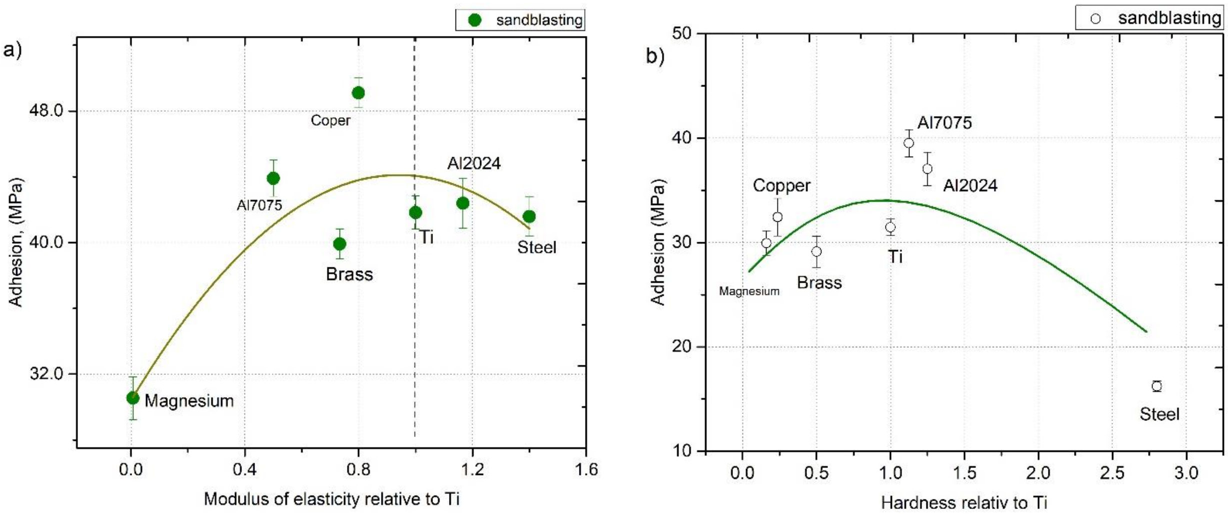

Fig. 9. a) Adhesion of the examined coatings as a function of Young's modulus (E), b) Adhesion as a function of hardness (H)

The horizontal axes of the graphs are scaled with respect to the values of Young's modulus (E) and hardness (H) for titanium, where both the hardness and the modulus of elasticity are assigned a value of one. In Figure 9a, the adhesion values are lowest for magnesium, highest for copper, and then decrease for steel. The relationship between hardness and adhesion is expected to follow a similar trend. However, in this case, the adhesion for steel deviates significantly from the values observed for other substrates. This is due to the considerably higher hardness and Young's modulus of the titanium coating compared to the steel substrate. This difference arises from the insufficient kinetic energy of the particles impacting the steel surface, which is unable to induce the surface deformation necessary for mechanical interlocking. Analyzing Figure 9, it can be concluded that the modulus of elasticity might be replaced by another material parameter that better characterizes the behavior of the material in the context of adhesion formation.

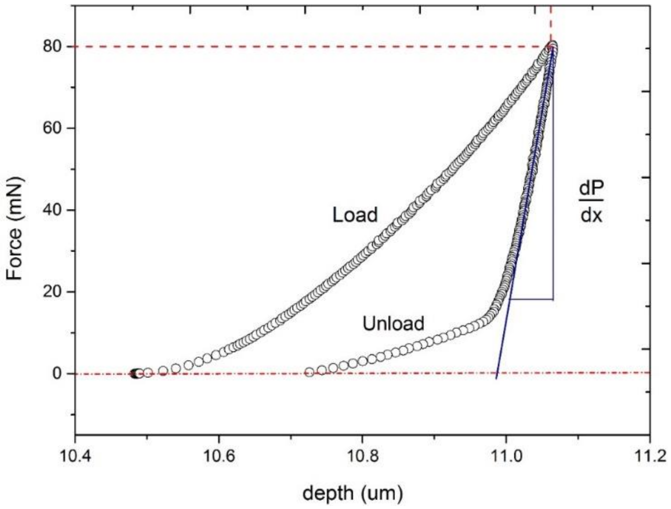

Fig. 10. The curves obtained in the hardness testing process using the indentation method

The elastic modulus is calculated from the initial slope of the unload curve, with the subsequent portion of the curve being disregarded. Some materials exhibit a very short unload curve, which reflects the almost unchanged indentation depth during indentation testing. In certain cases, the load curve reaches zero, indicating that the indentation in the material is significantly smaller than its maximum dimensions. This relationship suggests an alternative approach to plasticity, considering not only the shape of the curve but also the area enclosed by it.

Figure 10 presents the shape of the load and unload curves obtained during the indentation test. The area under the load curve represents the work done by the indenter in achieving the applied load force. The area under the unload curve reflects the work done by the material as it expels the indenter. The difference between the work performed by the indenter and the material is referred to as "workability" in this context. Integration under the curves was carried out to calculate the amount of work done, and the results are presented in Figure 11.

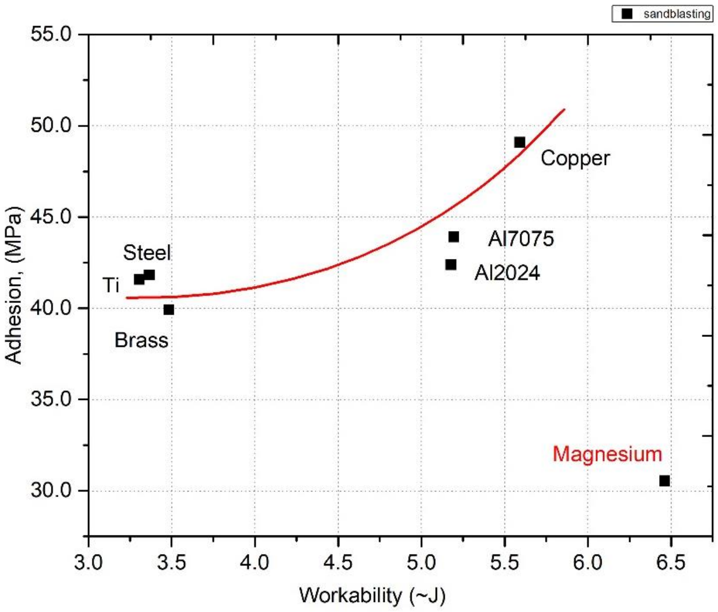

Fig. 11. Adhesion as a function of "workability"

It can be concluded that the relationship between adhesion and "workability" better reflects reality than the correlation between adhesion and Young's modulus. A correlation study was conducted between the Young's modulus and the "workability" values of the investigated coatings. The correlation coefficient was found to be -0.7±0.4, thus justifying the use of "workability" in further analysis.

4. Conclusion

A statistical optimization of the process of applying titanium coatings to various metal substrates was conducted. As a result of measuring the adhesion of titanium coatings on metal substrates with diverse mechanical properties, the highest adhesion value was observed in the case of copper. In contrast, the adhesion value for magnesium substrates was relatively low. There is a clear correlation between the adhesion results and the Young's modulus and "workability." Further analysis of adhesion in relation to Young's modulus and hardness leads to the conclusion that predicting the values of control parameters is effective for substrates whose Young's modulus and hardness values do not differ from those of the tested coating by more than 30%.
Analyzing the relationship between the examined variables (Fig. 9a and 12) confirms that the introduced parameter "workability" more accurately describes the material's behavior during the coating process using the cold spray technique.

When selecting the process parameters for cold gas spraying, it is important to choose the range of variability for the controlling parameters such that, in the statistical analysis (using the G. Taguchi method), the signal-to-noise (S/N) function reaches its extremum within the investigated range. This ensures that the process optimization results in the most favorable conditions for achieving the desired coating properties.

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