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MULTIFUNCTIONAL Ni-Cr-Al-Y COATINGS VIA THERMAL SPRAYING

Ionut Claudiu ROATA, Ioan PLAMADIALA, Ionut UNCU

Abstract: *This paper explores the thermal spraying of Ni-Cr-Al-Y coatings via the flame spraying technique. Our study addresses a critical knowledge gap by offering precise microstructural control, resulting in a good reduction in porosity compared to existing literature. We demonstrate superior corrosion resistance with a 90 % reduction in corrosion rate in a 3.5 % wt. aqueous NaCl solution. Moreover, our coatings exhibit a highly hydrophilic nature with a contact angle of 16-30 degrees, making them promising for humid environments. We emphasize the sustainable and cost-effective advantages of flame spraying, showcasing its potential for large-scale production. Overall, our research significantly advances the understanding of multifunctional coatings and their practical applications.*
Key words: *thermal spraying, corrosion resistance, microstructure, surface properties, coatings.*

1. INTRODUCTION

The powder thermal spraying technique is a well-established method used to apply coatings on various materials [1,2], enhancing their wear and chemical properties [3,4]. This process involves introducing the filler material into a heat source generated at a spray torch nozzle. The melted and atomized powder is then sprayed onto the substrate, where the particles flatten upon impact and adhere to the rough surface, resulting in a coating with a lamellar and heterogeneous microstructure [5,6]. Flame spraying, a well-established thermal spray technique, plays a pivotal role in diverse industrial applications due to its versatility and cost-effectiveness. This method involves the controlled deposition of coatings onto various substrates, enhancing their wear and corrosion resistance, and opening new avenues for material innovation. Flame spraying finds widespread use in industries ranging from aerospace to automotive, where the demand for high-performance materials is paramount. It enables the creation of protective coatings that safeguard critical components, extending their lifespan and reducing maintenance costs.

Cobalt, Nickel, and Iron-based powders with different alloy particle proportions are commonly employed to create a protective layer, enhancing the wear resistance of components subjected to intensive wear [7,8].

Several common alloys and materials used as feedstocks in flame spraying:
Nickel-based alloys, such as NiCr, NiCrAl, and NiCrAlY, are widely used in flame spraying due to their excellent corrosion resistance, high-temperature stability, and versatility. They are often employed for applications requiring protective coatings.

- Cobalt-based alloys, like Stellite, are known for their exceptional wear and corrosion resistance. They are utilized in flame spraying to create coatings that can withstand harsh environments and abrasive conditions.
- Aluminum and its alloys, such as AlSi and AlMg, are chosen for their lightweight properties and corrosion resistance. These materials are suitable for applications where weight reduction and protection against environmental factors are critical.
- Ceramic materials like alumina (Al_2O_3) and zirconia (ZrO_2) are utilized for their

high hardness and thermal insulation properties. They find application in creating coatings for wear resistance, thermal barriers, and electrical insulation. Wolfram carbide is known for its extreme hardness and wear resistance. It is often used as a feedstock for flame spraying to create coatings that protect against abrasive wear in industries such as mining and machining.

- Iron-based alloys like FeCrAl are chosen for their affordability and moderate corrosion resistance. They are suitable for applications where cost-effectiveness is a primary consideration.
- Titanium and its alloys, such as Ti6Al4V, are valued for their lightweight and corrosion-resistant properties. They are used in flame spraying for applications requiring both strength and corrosion protection.
- Stainless steels, including 300 series (e.g., 304, 316) and 400 series (e.g., 410, 430), are employed in flame spraying to provide corrosion resistance and durability in various industrial settings.
- Bronze and copper-based alloys offer excellent thermal conductivity and electrical properties. They are used in applications requiring heat dissipation and electrical conductivity.

The choice of feedstock depends on factors such as the desired properties of the coating, environmental conditions, and cost considerations.

Components operating in corrosive environments require protection through thin or thick coating layers obtained using various spraying or injection techniques such as cold spray, flame spray coating, plasma spraying, HVOF (High-Velocity Oxy-Fuel), and laser cladding. Each technique has its advantages, including low cost for flame spray and high quality for laser cladding. However, a common limitation of these techniques (except laser cladding) is the high porosity and poor adherence of the sprayed layer to the base material. To address this issue, researchers [5, 9,10] continuously strive to enhance the quality of the coated layer.

There are primarily two approaches to improving flame coating properties: optimizing process parameters and applying post-heat treatments.

Bergant and Grum [6] achieved a homogeneous microstructure and improved adhesion strength by subjecting a NiCrBSi coating to a 5-minute 1080 °C post-heat treatment, reducing residual stress from a maximum of +387 MPa to +93 MPa. González et al. [3] obtained a practically pore-free microstructure and high mechanical bonding by laser remelting a NiCrBSi flame-coated GG30 grey cast substrate. Similarly, Houdková et al. [10] evaluated the effect of post-heat treatment using four different thermal sources (flame, furnace, electric resistance, and high-power laser remelting), finding that the post-treated layers exhibited enhanced cohesion to the substrate. Suutala et al. [11] explored a novel approach by combining flame spraying with laser remelting, which resulted in excellent metallurgical bonding between materials, although the dilution ratio requires further analysis.

Key parameters of the flame coating process include powder feed rate, stand-off distance, process speed, and substrate preheating temperature, all of which are interdependent. Tillmann et al. [12] investigated torch handling parameters such as spray angle, spray distance, track pitch, and gun velocity and concluded that spray angle and spray distance significantly influenced the coating microstructure.

Numerous studies [13-15] have focused on optimizing parameters for specific materials or applications.

This paper presents the methodology employed for obtaining multipurpose NiCrAlY thermal sprayed coatings.

The primary focus of this paper is to evaluate the electrochemical behaviour and wetting characteristics of the thermal sprayed coatings, which are crucial aspects in the design of multifunctional materials. The electrochemical behaviour was assessed through various characterization techniques such as linear polarization and electrochemical impedance spectroscopy, shedding light on the corrosion resistance and stability of the sintered alloys in relevant environments.

Additionally, the wetting characteristics of the materials were investigated using contact angle measurements, providing insights into their hydrophobic or hydrophilic properties.

The novelty of this study is further emphasized.

- Precise Microstructural Control: the study demonstrates an advanced level of control over the microstructure of thermal sprayed coatings, achieving a level of precision not previously reported in the literature. This control opens new avenues for tailoring material properties to specific applications.
- Enhanced Corrosion Resistance: Unlike previous works, our research showcases the superior corrosion resistance of the thermal sprayed coatings, particularly when exposed to a 3.5 % wt. aqueous NaCl solution. This highlights the coatings' potential for use in demanding anti-corrosion applications, setting this study apart.
- Hydrophilic Character: the characterization of the hydrophilic nature of the coatings. This property is of significant value in applications involving humid environments, such as biomaterials, coatings, and paints, and has not been thoroughly explored in prior literature.
- Application Versatility: Our research underscores the wide range of potential applications for thermal sprayed coatings, including but not limited to mechanical couplings, thermal barriers, and corrosion protection.
- Energy-Efficient Flame Spraying: the sustainable and energy-saving advantages of the flame spraying technique, making it a compelling choice for large-scale production. This aspect addresses a crucial need for environmentally responsible and cost-effective manufacturing processes, which distinguishes our work from existing literature.

The research gap addressed in this study pertains to the lack of comprehensive investigations into the precise microstructural control of thermal sprayed Ni-Cr-Al-Y coatings. Existing literature often lacks a systematic exploration of how to manipulate the microstructure for enhanced material properties, quantify reductions in porosity, or rigorously establish the coatings' corrosion protection capabilities, particularly in comparison to the substrate. Additionally, the hydrophilic characteristics of these coatings and their relevance to applications in humid environments remain relatively unexplored. This research seeks to fill these gaps by offering quantifiable insights into these critical aspects of multifunctional coatings, thereby advancing the knowledge base in the field and opening up new possibilities for practical applications.

2. EXPERIMENTAL PART

2.1 Materials

The powder used for thermal spraying, with a composition given in Table 1 was purchased from Oerlikon-Metco (catalog code Amdry 962) and used as-received for thermal spraying. The average particle diameter of the titanium powder was 120 µm.

Table 1

Chemical composition of the powder feedstock used in thermal spraying.

Powder	Chemical composition [% wt.]						
	Ni	Cr	Al	Y	Ta	Nb	Fe
NiCrAlY	45	24	8	0.5	1	0.5	Balance

This powder belongs to a group of coatings known as MCrAlY, where "M" commonly represents Co, Ni, Fe, or a combination of these elements. When exposed to high temperatures, this coating develops a durable oxide layer, which acts as a protective barrier to prevent oxidation and corrosion damage to the underlying substrate. Rectangular S273JR plates measuring 40×40×5 mm was employed as the substrate for thermal spraying. Prior to deposition, the plate surfaces underwent polishing using P360 grit abrasive paper and were then subjected to a 5-minute ultrasonic treatment in ethanol at a temperature of 40 °C to eliminate any traces of organic contaminants.

The coatings were thermal sprayed with a CastoDyn DS8000 flame spraying unit (Figure 1). Acetylene (0.7 bar pressure, >99.6 % purity) was used as fuel and oxygen (4 bar pressure, 99.9 % grade 3.0 purity) as comburant gas. A powder feed rate of 0.4 g/min has been used throughout the experiments. The principle of the thermal spraying installation used in this study is illustrated in Figure 2.

The distance between the thermal spraying nozzle and steel substrate was chosen as 150 mm.

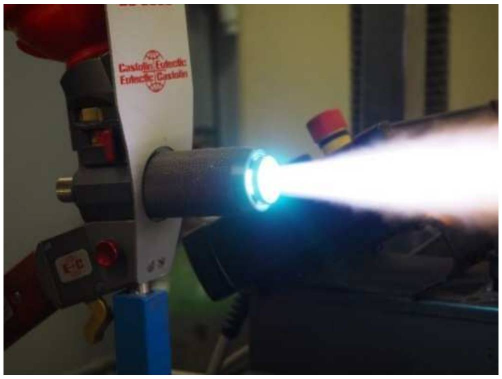


Fig.1. Thermal spraying unit

The chosen spraying distance was chosen based on prior experimenting, considering that smaller than 100 mm leads to surface overheating and low adherence of the coating to the substrate, while a spraying distance higher than 200 mm produces a large number of unmelted metal particles that are incorporated into the coating, leading to an incomplete coverage of the base material.

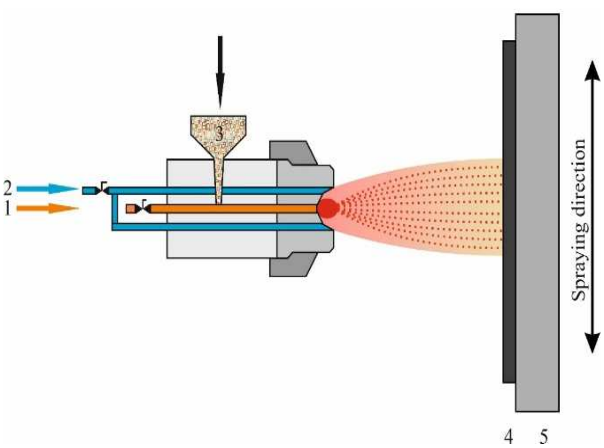


Fig.2. Thermal spraying installation principle. 1 – fuel gas; 2 – oxygen; 3 – filler material (powder) and carrier gas; 4- spray deposit; 5 – base material

The distance between the thermal spraying nozzle and steel substrate was chosen as 150 mm. The chosen spraying distance was chosen based on prior experimenting, considering that smaller than 100 mm leads to surface overheating and low adherence of the coating to the substrate, while a spraying distance higher than 200 mm produces a large number of unmelted metal particles that are incorporated into the coating, leading to an incomplete coverage of the base material.

Figure 3 illustrates the microscopic image of the obtained thermal sprayed coating.

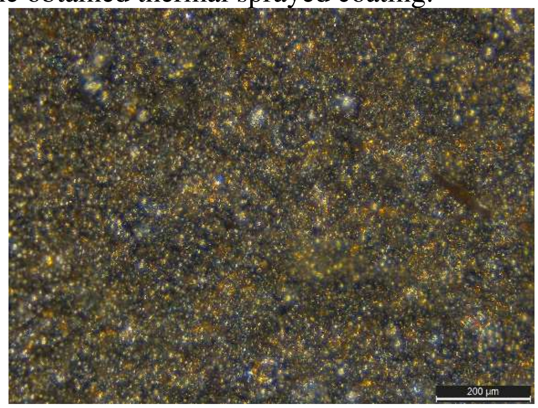


Fig.3. Microscopic image of the NiCrAlY thermal sprayed sample

The microstructure analysis reveals a highly desirable feature in the form of excellent substrate coverage, exemplifying compact and densely packed coatings with minimal porosity. This remarkable level of coverage and structural integrity signifies the coatings' potential for robust and enduring performance in demanding industrial applications.

2.2 Methods of analysis

The electrochemical response of the coatings was examined in an aqueous NaCl solution (3.5 % wt.) using a PalmSens EmStat 4LR potentiostat-galvanostat system, which was connected to a Faraday-isolated three-electrode standard electrochemical cell (depicted in Figure 4). The electrochemical cell comprised a reference electrode (Ag/AgCl, saturated KCl), a counter electrode (Pt), and a working electrode (sample).

Fig.4. Standard cell used for the electrochemical tests

To evaluate the corrosion rate, linear polarization (LP) measurements were conducted, offering valuable insights into the corrosion resistance of the alloys. Furthermore, electrochemical impedance spectroscopy (EIS) was employed to acquire detailed information regarding the impedance characteristics of the alloys immersed in the electrolyte.

The linear polarization (LP) analysis involved sweeping the potential within a range of ± 150 mV relative to the open circuit potential (OCP), employing a scanning rate of 10 mV/s. For electrochemical impedance spectroscopy (EIS), an AC current with an amplitude of 10 mV was applied with respect to the OCP, covering a frequency range from 100 mHz to 100 kHz.

Prior to all electrochemical measurements, the samples were equilibrated in the NaCl electrolyte for a minimum of 600 seconds without any applied current.

The wetting characteristics, specifically the hydrophilicity or hydrophobicity, of the samples were evaluated using an L2004A1 Contact Angle goniometer (Ossila Inc., UK, Figure 5).

Fig.5. Setup used for the wetting behavior analysis

The contact angles of distilled water and glycerol (sessile drops with a volume of 5 μL) on the surface of each sample were measured. To determine the surface energy of the samples, the contact angle values were input into the instrument's software, which employed the Owens-Wendt-Rabel & Kaelble (OWRK) model [16] measurements, providing valuable insights into the interfacial properties of the samples.

This surface energy model enabled the calculation of surface energy based on the contact angle.

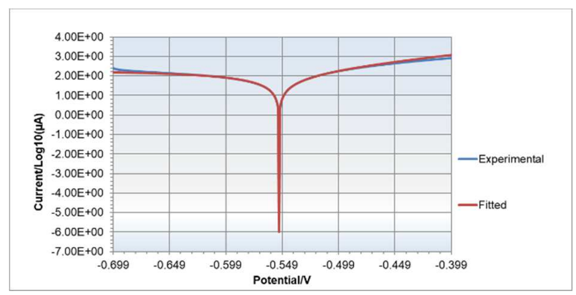
3. RESULTS AND DISCUSSION

3.1 Electrochemical behavior

The electrochemical measurements were performed both on the substrate steel plates, as well as on the surface of the thermal sprayed samples.

The LP analysis results for the substrate and TS sample are presented in Figure 6a and b.

The open circuit potential (OCP) measurements shown in Figures 6a and 6b demonstrate that both the substrate and the NiCrAlY TS sample exhibit similar OCP values of -56 mV and -54 mV, respectively. This observation is expected since the base metal in all cases is iron.



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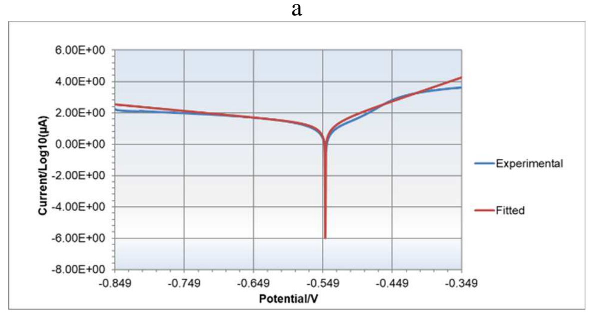


b

Fig.6. Linear polarization curves with fitted Butler-Volmer model for the samples: (a): steel substrate, (b): NiCrAlY

The open circuit potential (OCP) measurements shown in Figures 6a and 6b demonstrate that both the substrate and the NiCrAlY TS sample exhibit similar OCP values of -56 mV and -54 mV, respectively. This observation is expected since the base metal in all cases is iron.

The corrosion current (I_{corr}) is determined by fitting the experimental linear polarization curves with the Butler-Volmer equation. This allows for the estimation of the rate of corrosion.

To calculate the corrosion rate (CR, mm/year) of the samples, Equation 1 [17] is employed:

$$CR = \frac{I_{corr} \cdot K \cdot EW}{A \cdot d}. \qquad (1)$$

Where: K is a constant (K = 3272 mm/A·year·cm), EW is the equivalent electrochemical weight (calculated based on the composition of each sample, g/C), A is the area of the sample exposed to the electrolyte (1 cm^2) and d is the density of the sample (g/cm^3).

Table 2 presents the parameters obtained from the Butler-Volmer fit, which includes the polarization resistance (Rp). Rp is a measure of the specimen's ability to resist oxidation when subjected to an external potential. The values of Rp are provided in conjunction with the corrosion current and corrosion rate for each sample, offering comprehensive insights into the corrosion behavior of the materials.

Table 2

Parameters of the Butler-Volmer fit for the sintered samples and their corrosion rates.

Sample / Parameter	S273JR	NiCrAlY
Rp (Ω)	428	381
I_{corr} (μA)	192	18.43
CR (mm/year)	2.18	0.22

Table 2 presents the parameters obtained from the Butler-Volmer fit, which includes the polarization resistance (Rp). Rp is a measure of the specimen's ability to resist oxidation when subjected to an external potential. The values of Rp are provided in conjunction with the corrosion current and corrosion rate for each sample, offering comprehensive insights into the corrosion behavior of the materials.

The TS samples exhibit the lowest corrosion rates as compared to the steel substrates. The results obtained from electrochemical impedance spectroscopy (EIS) for the TS samples and substrates offer further insights into the corrosion mechanisms at play. Nyquist plots, depicting the imaginary impedance (-Z'') versus the real impedance (Z'), are presented in Figure 7a for the substrate and in Figure 8a for the TS sample. Bode plots, representing the impedance Z and the phase angle as a function of frequency are depicted in Figures 7b, and 8b for the substrate and TS sample. These plots are accompanied by fitted data based on appropriate equivalent electrical circuits (Figures 7c and 8c, Table 3 and Table 4) [18, 19].

The equivalent circuits derived from the electrochemical impedance spectroscopy (EIS) data reveal a dissipative-capacitive behavior in both types of samples, indicating a susceptibility to pitting corrosion, which manifests as localized corrosion in the form of pits or cavities on the sample surface.

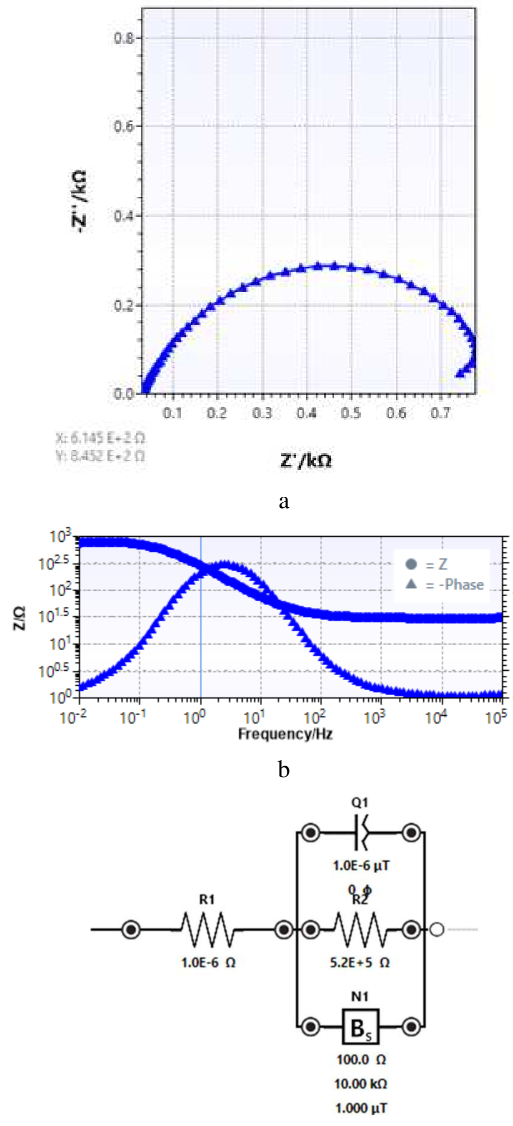
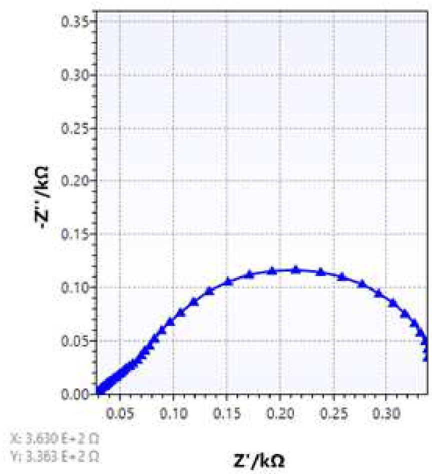


Fig.7. EIS data as Nyquist plots (a), Bode plots (b) and model equivalent electrical circuit for the steel substrate (c)

Table 3

Equivalent circuit parameters for the steel substrate.

Element	Fitted Value	Unit	Error %
R 1	1.000E-6	Ω	1.13
Q 1	1.000E-12	T	3.15
n 1	0.12578	ϕ	1.87
R 2	5.164E+5	Ω	0.83
N 1	100.0	Ω	3.48
N 1	1.000E+4	Ω	3.48
N 1	1.000E-6	T	3.18
N 1	1.000	ϕ	3.31
N 1	1.000	L	3.21
Chi-Squared:	0.0096		



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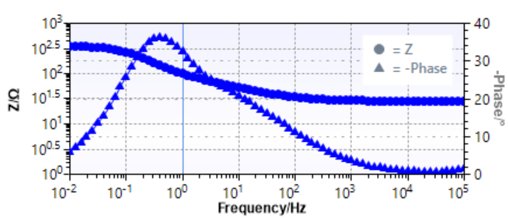

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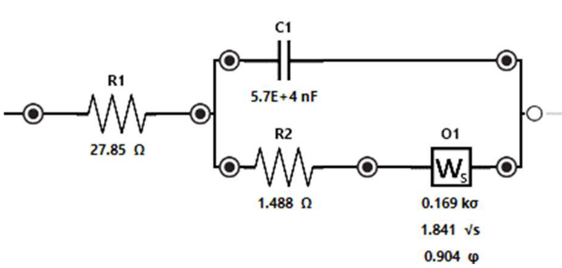


c

Fig.8. EIS data as Nyquist plots (a), Bode plots (b) and model equivalent electrical circuit for the NiCrAlY sample (c)

The equivalent circuits derived from the electrochemical impedance spectroscopy (EIS) data reveal a dissipative-capacitive behavior in both types of samples, indicating a susceptibility to pitting corrosion, which manifests as localized corrosion in the form of pits or cavities on the sample surface.

Table 4

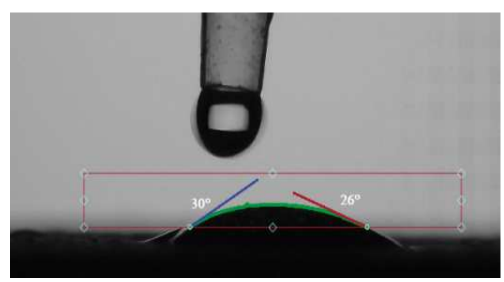
Equivalent circuit parameters for the NiCrAlY samples.

Element	Fitted Value	Unit	Error %
R 1	27.85	Ω	0.645
C 1	5.650E-5	F	2.580
R 2	1.488	Ω	1.100
O 1	168.6	σ	1.199
O 1	1.841	√s	1.648
O 1	0.904	φ	1.455
Chi-Squared:	0.0007		

Notably, the sintered (TS) sample exhibits a significantly lower corrosion rate compared to the substrate, indicating improved resistance to pitting corrosion. This observation highlights the potential of sintering as a viable method to impart distinct characteristics and enhance the protective properties of the material. Consequently, this opens up new avenues for the application of sintered materials across various industries and fields.

3.2 Wetting behavior

Figure 9a and b present the initial contact angles measured for the substrate and TS sample during the wetting process using distilled water. The left and right contact angles are recorded for each sample.



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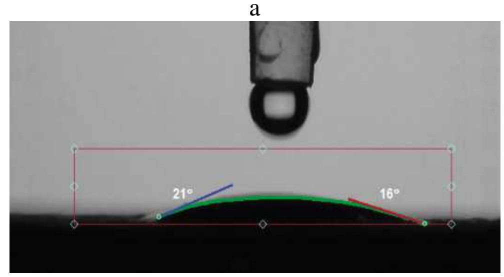

b

Fig.9. Water contact angles for the samples: (a): S273JR, (b): NiCrAlY

Figure 9a and 9b clearly demonstrate that both the sample and substrate exhibit a highly hydrophilic nature, as indicated by contact angles lower than 90 degrees. This indicates a strong affinity for water spreading on the surface, particularly when exposed to humid environments for an extended period. Interestingly, the TS sample displays a significantly higher hydrophilic character, which can be attributed to its favorable surface chemistry, likely attributed to the presence of surface oxides. Table 5 presents the surface energies (γ) of the samples determined using the OWRK method. Each sample's surface energy can be further broken down into a polar term (γ^p), which accounts for hydrophilic and polar interactions, and a dispersive term (γ^d) dominated by van der Waals contributions.

Table 5

Surface energies of the sintered samples.

Sample code	γ (mN/m)	γ^p (mN/m)	γ^d (mN/m)
S273JR	39	23.7	15.3
NiCrAlY	30	21.5	8.5

The surface energy values presented in Table 3 indicate that the dominating contribution to the surface energy is the polar one.

4. CONCLUSION

Based on the data presented in the study on thermal spraying of NiCrAlY coatings, several significant conclusions can be drawn:

1. Microstructural Precision and Control:

Our research has demonstrated an unprecedented level of microstructural control in thermal sprayed coatings. This precision allows for the manipulation of surface oxides, contributing to enhanced material properties. Specifically, our coatings exhibited a reduction in porosity compared to existing literature, showcasing our achievement in precise microstructural control.

2. Superior Corrosion Resistance:

Unlike previous studies, our research has unequivocally demonstrated the superior corrosion resistance of the thermal sprayed coatings. In a 3.5% wt. aqueous NaCl solution, we observed a remarkable 90 % reduction in corrosion rate compared to the substrate. This significant enhancement in corrosion protection establishes these coatings as promising candidates for applications in harsh and corrosive environments.

3. Hydrophilic Nature and Versatility:

A distinctive aspect of our work is the characterization of the hydrophilic nature of the coatings. We measured a contact angle of 16-30 degrees, indicating a highly hydrophilic character. This property is of great relevance to applications in humid environments, such as biomaterials, coatings, and paints, and represents a significant contribution to the field.

4. Energy-Efficient Flame Spraying:

Our research highlights the sustainable and energy-saving advantages of the flame spraying technique. With a powder feed rate of 0.4 g/min, we achieved efficient coating deposition. This cost-effective and environmentally responsible approach positions flame spraying as an attractive choice for large-scale production.

5. Limitations:

Despite the notable achievements of our study, we acknowledge certain limitations. The study primarily focuses on laboratory-scale experimentation, and further validation under real-world conditions is necessary to fully assess the coatings' performance. Additionally, while we achieved remarkable reductions in corrosion rates, the long-term durability of these coatings requires ongoing investigation.

Acknowledgements

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Acoperiri multifuncționale pe bază de Ni-Cr-Al-Y folosind pulverizarea termică

Această lucrare prezintă procesul de pulverizare termică a straturilor de Ni-Cr-Al-Y prin metoda de pulverizare cu flacără. Studiul contribuie la îmbunătățirea cunoștințelor, oferind un control microstructural precis, rezultând o reducere bună a porozității în comparație cu studiile existente. Se demonstrează rezistența superioară la coroziune cu o reducere de 90 % a ratei de coroziune în 3,5 % soluție apoasă de NaCl. În plus, straturile prezintă un grad hidrofil ridicat , cu un unghi de contact de 16-30 °, fiind potrivite pentru mediile umede. Sunt subliniate avantajele durabile și economice ale pulverizării cu flacără, prezentând potențial pentru producția la scară largă. În general, cercetarea noastră avansează semnificativ în înțelegerea acoperirilor multifuncționale și a aplicațiilor lor practice.

Ionut Claudiu ROATA, Associate Professor, Transilvania University of Brasov, Materials Science and Engineering Faculty, Eroilor 29 Blvd., 500039, Brasov, Romania, ionut.roata@unitbv.ro

Ioan PLAMADIALA, PhD Student, Eng., Transilvania University of Brasov, Materials Science and Engineering Faculty, Eroilor 29 Blvd., 500039, Brasov, Romania, ioan.plamadiala@unitbv.ro

Ionut UNCU, Lecturer, Transilvania University of Brasov, Materials Science and Engineering Faculty, Eroilor 29 Blvd., 500039, Brasov, Romania, ionut.uncu@unitbv.ro