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# STUDY OF THE MECHANICAL PROPERTIES OF NI+AL$_2$O$_3$+ZN COATINGS PRODUCED BY THE LOW PRESSURE COLD SPRAY TECHNOLOGY

*Low Pressure Cold Spraying (LPCS) is a thermal spraying technique for applying high-density coatings on almost any surface. The main features of the LPCS is that Air is used for the powder materials acceleration through a DeLaval Nozzle with a maximum operation temperature of 630 °C in a pressure range of 5 to 9 atm. The powders used in LPCS are usually metal matrix composite blends using Alumina very often as the Ceramic part of the composition. The aim of this study is to investigate the effect of the stagnation spraying temperature on the coating properties produced with the Dymet 405 Low Pressure Cold Spraying system and a Ni+Al2O3+Zn powder blend. The coatings are submitted to analyses using a SEM microscope and Metallographic techniques, Micro-Hardness testing and Adhesion-Cohesion measurements.*

*Key words: cold spray technology, Mach number, adhesive-cohesive strength, micro-harnesses.*

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# ВИВЧЕННЯ МЕХАНІЧНИХ ВЛАСТИВОСТЕЙ ПОКРИТТЯ NI+AL$_2$O$_3$+ZN ОТРИМАНОГО ТЕХНОЛОГІЄЮ ХОЛОДНОГО НАПИЛЕННЯ НИЗЬКОГО ТИСКУ

*Холодне напилення низького тиску (ХННТ) - це технологія для нанесення покриттів з високою щільністю на будь-які поверхні. Основними особливостями ХННТ є те, щоповітря для прискорення порошкових матеріалів через сопло Лаваля використовується з максимальною робочою температурою 630 ° С в діапазоні тисків від 5 до 9 атм. Порошки, використовувані в ХННТ, як правило, металеві матричні композитису міші з частим використанням глинозему в якості керамічної складової композиту. Метою даного дослідження є вивчення впливу стагнації температури розпилення на властивості покритті вотриманих на системі холодного напилення низького тиску ДИМЕТ 405 і порошкової суміші Ni + Al$_2$O$_3$ + Zn. Представлений аналіз покриття за допомогою мікроскопа SEM і металографічного методу, тестування мікро-твердості і виміру адгезионно-когезионної міцності.*

*Ключові слова: технологія холодного напилення, число Маха, адгезійно-когезійнаміцність, мікро-твердість.*

## 1. Introduction

The cold spraying technique was developed in the Soviet Union in the 80's. Mainly, the process is characterized from other Thermal Spraying coating techniques from the use of much more lower operative pressures and significantly lower process temperatures. The coatings are formed when powder materials are accelerated to supersonic velocities by the use of a convergent-divergent nozzle to consequently be impacted on a substrate; the bonding phenomena has been a controversial point of discussion, but there is an agreement on that bonding is due a high degree of plastic deformation on non melted particles during their impact [1].

The Low Pressure Cold Spraying Technology (LPCS) is characterized for using air as the accelerated fluid with inlet pressures from 5 to 9 atm and a top temperature that reaches the 630 Celsius. The acceleration of the particles through the diverging gas-stream is due the resultant drag force applied to their surfaces; this approach converge in the idea that not only the gas velocity is the responsible for particles acceleration, but the density of the gas as well which is dependent on the Mach number of the accelerated stream [2]. In LPCS a wide range of powder materials (i.e. Cu, Al, Ni, Zn) can be co-sprayed with ceramic particles as $Al_2O_3$ [3]. The main reasons for using ceramic particles are: to propagate the surface activation while spraying and to clean the nozzle while metallic particles are hammered and better deformed in the process in comparison of spraying them without ceramics; different studies [3-5] have shown that the addition of ceramic particles give results on better coatings adhesion and better density; for this reason it is acceptable to say, that the ceramics addition to the metallic matrix in the LPCS process acts as a reinforcement for the metal matrix composite (MMC) [6].

The aim of this study was to characterize the micro-structure and mechanical properties of Ni+$A_{l2}O_3$+Zn coatings in order to get more information about their adhesion strength and micro-hardness dependent on their spraying temperature.

## 2. State of the art (literature review)

The characterization of thermal spraying coatings requires tests in order to determine their mechanical properties as: micro-hardness, adhesion strength, corrosion, etc. The major emphasis between the coatings properties in surface science is for the tensile adhesion strength because it is usually a design requirement for most coating materials [7]. The coating properties are generally influenced by the process parameters as Temperature, Pressure, feedstock characteristics, etc. In order to determine the degree of adhesion (bonding strength) of coatings normal to a substrate an Adhesion or Cohesion test is used is performed. The adhesion testing methods are very controversial between the scientific community due the large variance between laboratory to laboratory [7-8]. The main reasons for this variances is attributed to: Bending moments during the effect of normal tensile forces, adhesive penetration into the coatings porosity, usually adhesion values depend on the coatings thickness, residual stresses could be distributed not evenly, machine vibration, difference in loading rates, non uniformity of coating surfaces, etc. Between the available methods for determining the adhesion strength in Thermal Spray coatings the ASTM C 633

standard is one of the most used. Different authors have reported various adhesion strength ranges and behaviors for Low Pressure Cold Sprayed coatings. A. Kuznetsov for example [9], have found a decrease in adhesion strength while incrementing the stagnation temperature of LPCS Al+Al2O3+Zn coatings. In [10-11] is discussed the adhesion strength as a function of the substrate material. In [10] adhesion strength measurements for coatings created with powders of Ni + Al2O3 with values of ~14 MPa for the coatings sprayed using air as carrier gas and stainless steel for the substrate and values of ~20 MPa for those coatings applied on Nickel alloy substrates. Hardness measurement of Thermal Sprayed Coatings is considered an easy and well standardized practice for this materials [7]. Other than adhesion strength characterization, hardness measurement is a measurement method that usually gives the same alike results within laboratories. The Vickers standard test is considered a standard micro-hardness measurement standard for thermal sprayed coatings. It is known that the Vickers hardness measurements vary depending on the metallographic preparation of the specimens. In order to get better readings, multiple measures are taken and averaged. As there is not a clear dependence between the coating properties and the low pressure cold spraying process parameters it is important to further develop scientific bases that address this aspect.

## 3. Objective of the research work

The objective of the present research work is to characterize coatings produced with the DYMET 405 Low pressure cold spraying equipment at different process stagnation temperatures (424 ˚C, 526 ˚C and 632 ˚C) for the powder N7-00-14 obtained from the Obnisk Center for Powder Spraying (OCPS) in order to better understand the effect of the process stagnation temperature on the coating properties. For the development of the proposed objective the following tasks were set:

1. N7-00-14 coatings deposition using the DYMET 405 low pressure cold spraying equipment at the stagnation temperatures 424 ˚C, 526 ˚C and 632 ˚C.
2. Metallographic preparation of the produced coatings.
3. Characterization of the produced coatings.

## 4. Experimental Procedures

In this investigation a N7-00-14 (65%wt.%Ni + 10wt.%$Al_2O_3$ + 25wt.% Zn) commercial powder (Fig. 1) from Obnisk Center for Powder Spraying (OCPS) was sprayed with a DYMET 405 Cold Spraying equipment available at the National Aerospace University of Ukraine. Aluminum 2024 substrates with a diameter of 24.7 mm for Adhesions tests and 50 x 30 mm substrates for metallographic studies were prepared for spraying with sanding paper P360. The SK-20 Nozzle with an Exit Diameter of 4.9 mm was used to accelerate the powder within three different stagnation temperatures - 424 ˚C, 526 ˚C and 632 ˚C. Five different specimens sets were prepared in order to spray them at different stagnation temperatures. The Coatings were characterized using a SELMI REM-106 Scanning Electron Microscope (SEM).

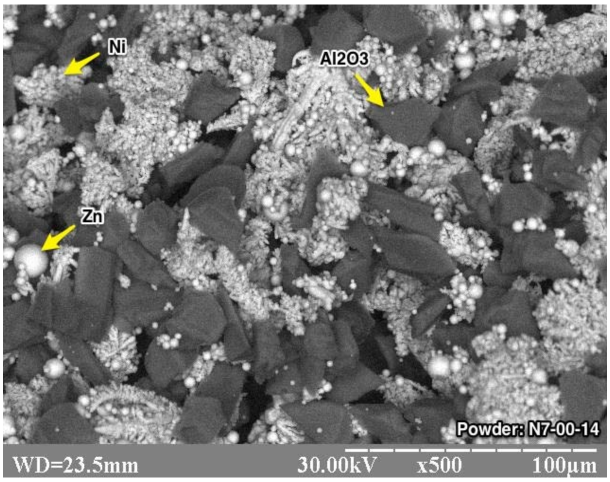


**Fig.1. Morphology of powder N7-00-14 Powder in a blend of 65%wt.%Ni+10wt.%$Al_2O_3$+25wt.%Zn provided by OCPS**

The ASTM E3-01 for Preparation of Metallographic Specimens was followed for the substrates of this study; the specimens were etched using dilute hydrochloric acid (HCl) in order to better distinguish the borders of the deformed particles. The Deposition Efficiency was measured for each Stagnation Temperature under the standard ISO 17836-2004.

Mechanical testing was performed on the coatings in order to determine their adhesive-cohesive strength and micro-harnesses. A tensile strength machine was used in order to determine the Adhesive-Cohesive strength of the coatings according to the ASTM 633 Standard; four tests per coating were performed to determine the average adhesive-cohesive strength at every stagnation temperature. Micro-Hardness tests were also performed under the ASTM E92 Standard (Standard Test Method for Vickers Hardness of Metallic Materials).

### 4.1 Metallographic analysis of Coatings

The LPCS coatings were prepared for metallographic analysis under the ASTM E3-01 standard to consequently be observed in a SEM Microscope. Fig. 2 Shows the SEM images at the 200x Amplification for specimens sprayed at 424 C (a), 526 C (b) and 632 C (c). In the microstructure the black particles are non-deformed Alumina particles that were impregnated into the coating.

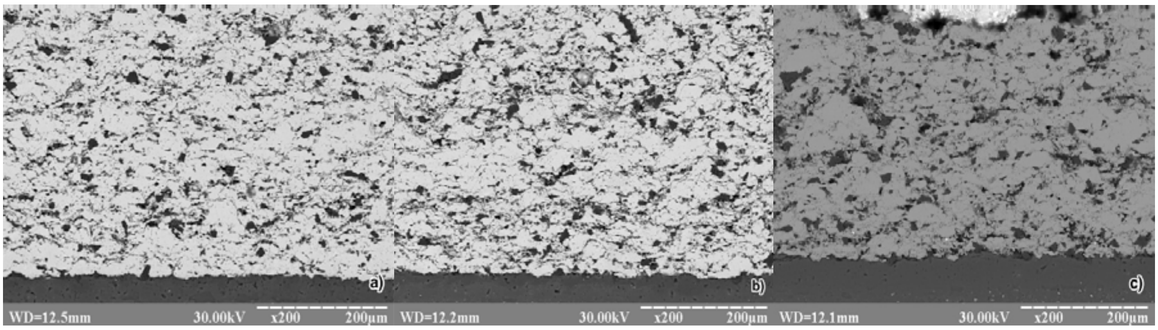


**Fig.2. SEM images at the 200x Amplification for specimens sprayed at 424 C (a), 526 C (b) and 632 C (c). In the microstructure the black particles are non-deformed Alumina particles that were impregnated into the coating**

The thickness of the N7-00-14 coatings was 200 microns. From visual inspection the coatings to be dense without any noticeable porosity. It is not possible to recognize the powder particle boundaries in the microstructure, for this reason it is etched with dilute hydrochloric acid (HCl). The flattened shaped particles, indicates a high degree of deformation. Despite the high density of the coatings, some open boundaries can be detected near the coating surface and some oxidized boundaries can be spotted within the coating structure. The oxidation layers can be attributed to the electrolytic feedstock powder production method; this is common for dendritic powders which because their morphology, have larger surface area than spherical particles produced by the atomized method. The Air used as operating gas can cause another oxidation source. Black particles within the coatings structure are the $Al_2O_3$ part of the powder blend.

### 4.2 Mechanical Properties of Coatings

Mechanical testing was performed on the coatings in order to determine their adhesive-cohesive strength and micro-harnesses. A tensile strength machine was used in order to determine the Adhesive-Cohesive strength of the coatings according to the ASTM 633 Standard; four tests per coating were performed to determine the average adhesive-cohesive strength at every stagnation temperature. Micro-Hardness tests were also performed under the ASTM E92 Standard (Standard Test Method for Vickers Hardness of Metallic Materials).

#### 4.2.1 Bond Strength

The ASTM C 633 standard for Adhesion or Cohesion Strength of Thermal Spray coatings studies the adhesion strength for coatings sprayed at different stagnation temperatures. The N7-00-14 powder blend was sprayed at different spraying stagnation temperatures - 424 ˚C, 526 ˚C and 632 ˚C - with a constant stagnation pressure of 0.8 MPa with the DYMET 405 Low Pressure Cold Spraying equipment.

Under the ASTM C 633 specifications 5 testing samples for each stagnation temperature were studied. Epoxy ED-20 was selected for this study and tested for its maximum adhesive strength presenting 22 MPa (SD 3) of adhesive bonding strength. Test samples were prepared for the spraying operation with sanding paper P360. Coatings of ~350μm were evenly applied on the test sample. Tensile specimens were assembled as specified in ASTM C 633 using the Epoxy ED-20, which was curated in 12 hours at ambient temperature.

Tensile strength was applied for each of the specimens with five runs for each stagnation temperature and material configuration. Table 1 shows the results of the adhesion strength study. The results show that the adhesion strength of N7-00-14 are above 22 MPa for all the stagnation temperatures.

Table 1

**Results of the adhesion strength study.**

| Stagnation Temperature | 424 ˚C | 536 ˚C | 632 ˚C |
|---|---|---|---|
| Bond Strength | > 22 MPa \| Epoxy | > 22 MPa \| Epoxy | > 22 MPa \| Epoxy |

#### 4.2.2 Micro-Hardness

The micro-hardness for coatings sprayed at different stagnation temperatures is studied under the ASTM E92 Standard (Standard Test Method for Vickers Hardness of Metallic Materials).

The N7-00-14 powder blend was sprayed at different spraying stagnation temperatures - 424 ˚C, 526 ˚C and 632 ˚C - with a constant stagnation pressure of 0.8 MPa with the DYMET 405 Low Pressure Cold Spraying equipment. The Vickers hardness was determined using a load of 50gr. for all the samples. Fig. 3 shows the results for micro-hardness readings.

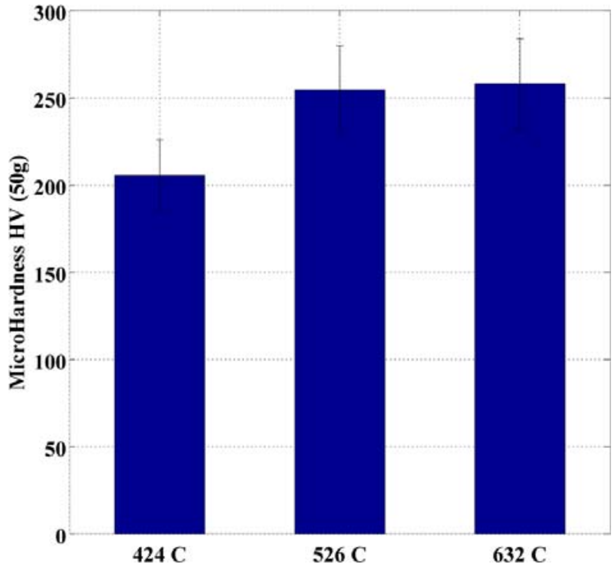


**Fig.3. Results of micro-hardness readings**

## 5. Results discussion

The results obtained after the characterization of coatings produced with the N7-00-14 Nickel based powder show a clear increase on micro-harness while increasing the process stagnation temperature. Since the maximal adhesion strength of the epoxy ED-20 used during the coatings adhesion strength characterization has a magnitude of 22MPa (SD 3) a clear effect of the stagnation temperature on the coatings adhesion to the substrate cannot be determined. The metallographic analysis have shown a good coatings density for every studied stagnation temperature. Further research is recommended in order to determine the yield adhesion strength of N7-00-14 coatings.

## 6. Conclusion

In this work, the characterization of Low Pressure Cold Sprayed coatings at several stagnation temperatures was presented. The N7-00-14 nickel based material powder MMC blend was sprayed at different stagnation temperatures - 424 ˚C, 526 ˚C and 632 ˚C - and a constant stagnation pressure of 0.8 MPa using the Dymet 405 Cold Spraying system. The coatings were applied onto Aluminum 2024 substrate specimens. The ASTM E3-01 standard was applied in order to prepare the specimens for metallographic study; the study revealed an increase on strain when the powder material was sprayed at higher stagnation temperatures. The standard ASTM C 633 was used in order to determine the bond strength of coatings sprayed at the different stagnation temperatures; the adhesion strength of the coatings was higher than the adhesion strength of the epoxy while presenting adhesive failure at the epoxy zone in all the cases. The ASTM E92 Standard was applied in order to determine the micro hardness of coatings; N7-00-14 coatings presented an increase in micro hardness when spraying it at higher stagnation temperatures. The increase on micro-hardness when spraying the material at higher stagnation temperatures can be attributed to the work hardening on the particles during deformation after impacting the substrate.

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# ОЦЕНКА КАЧЕСТВА СЕТОЧНЫХ ТРЕХМЕРНЫХ ОБЪЕКТОВ ПРИ РАЗЛИЧНЫХ СКОРОСТЯХ ЦИФРОВОГО ПОТОКА

*В работе рассматривается скорость передачи реальных трехмерных сеточных объектов. Анализируются скорости передачи для двух стандартов дискретизации в разных цифровых потоках. Было показано, как произвести проекцию трехмерного объекта на двумерное пространство. Получены зависимости канальной емкости от скорости передачи цифрового потока сцены из двух объектов для структур дискретизации 4:4:4 и 4:2:2. Найдены аппроксимирующие функции канальной зависимости в виде сплайнов.*

*Ключевые слова. Сеточные объекты, скорость передачи, цифровой поток, трехмерное пространство.*

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## QUALITY ASSESSMENT THREE-DIMENSIONAL MESH OBJECTS AT DIFFERENT SPEEDS OF DIGITAL STREAMS

*Abstract - The paper deals with the transmission rate of real three-dimensional mesh objects. We have aanalyzed the transmission rate for two sampled structures in different digital streams. It was shown how to make a projection of a three-dimensional object on a two-dimensional space. The paper deals with the transmission rate of real three-dimensional meshes. We have aanalyzed the transmission rate for sampling structures 4: 4: 4 or 4: 2: 2. and bit rate of the studied objects. It was shown how to make a projection of a three-dimensional object on a two-dimensional space. The dependences of channel capacity from a digital stream have been received and approximated by spleens for a scene of two objects*

*Keywords: Meshes, the transmission rate, the digital stream, the three-dimensional space.*

### Постановка задачи

Для исследования скорости передачи выбранных трехмерных объектов используем две структуры дискретизации 4:2:2 и 4:4:4. Скорость передачи для выбранных структур 4:2:2 (1) и 4:4:4 (2), и при разных параметрах исследуемых объектов, выглядят следующим образом [1, 2]:

$$V_p = R*2*W*H*F \tag{1}$$

$$V_p = R*3*W*H*F \tag{2}$$

где $V_p$ – скорость передача данных объекта, бит/с;

$W$ и $H$ – ширина и высота кадра в пикселях;

$R$ – разрядность для каждой компоненты, бит;

$F$ – кадровая частота, кадров/с.

### Проецирование трехмерных объектов на двумерный ТВ растр

Так как мы исследуем трехмерные объекты, то нам необходимо перейти из трехмерного пространства в двумерное. Координаты в двумерном пространстве имеют следующий вид:

$$X_c = x + k_x \times z\,, \tag{3}$$

$$Y_c = y + k_y \times z\,, \tag{4}$$

где $X_c$ и $Y_c$ – координаты на плоскости;

$x$, $y$, $z$ – координаты в трехмерном пространстве;

$k_x$, $k_y$ –коэффициенты проекции глубины, которые берутся по модулю и не превышают значения единицы. Коэффициенты принимаем равными 0,5.

При переходе из трехмерного пространства в двумерное надо учесть тот факт, что используется децимация координат и клиппинг вершин объекта. Это позволяет восстанавливать объект в двумерном пространстве с высокой четкостью [3]. Соответственно, ширину и высоту кадра исследуемого объекта рассчитаем по формулам (5) и (6) следующим образом:

$$W = X_{max} - X_{min}, \tag{5}$$

$$H = Y_{max} - Y_{min}, \tag{6}$$