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# Characterization of cermet coatings deposited by low-pressure cold spraying

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ABSTRACT

The paper presents the influence of substrate preparation on low pressure cold sprayed (LPCS) coatings adhesion strength and microhardness. The LPCS process parameters such as: (i) gas temperature; (ii) gas pressure; (iii) gun linear speed; (iv) powder feeding rate; and (v) spray distance were kept constant during deposition. The coatings were sprayed using metallic powders having different morphology, namely spherical (Al, Zn, Sn, Cu) and dendritic (Ni, Cu) with the grain size of about −50 + 10 μm. These powders were mixed with alumina before spraying in a weight ratio of 50:50. Copper and aluminium alloy were used as substrates. The substrate preparation included sand-blasting and grinding. The coating microstructures were characterized by scanning electron microscopy (SEM) and X-ray diffractometry (XRD). The adhesion strengths of coatings were determined by pull-off method and showed that coating adhesion reached 65 MPa. The measurements of coatings microhardness were carried out also. Finally, the values of adhesion strength and microhardness of coatings were correlated with the substrate preparation and the microstructure of coatings. The microhardness of LPCS coatings was in the range of 20 to 201 HV0.02 and depended mostly on sprayed powder material.



## 1. Introduction

In the cold spray method the powder particles get accelerated in the stream of compressed gas (air, nitrogen or helium) and subsequently impact a substrate. The gas is flowing in de Laval nozzle having special convergent–divergent shape. When the value of critical velocity is reached, the particles start to adhere to the substrate [1,2]. Two varieties of the method can be distinguished: (i) low-pressure and (ii) high-pressure cold spraying. In the low-pressure cold spraying (LPCS) method air or nitrogen is usually used as working gases with the pressure not exceeding 0.9 MPa and temperature up to 650 °C [2].

The adhesion strength and hardness are the most important mechanical properties in cold spraying. There is no metallurgical bonding between particles and substrate because powder particles are solid at deposition. The bonding is realized thanks to the local deformation of the material at the interface, as the powder particles strike the substrate. As a result of high strain-rate deformation of materials the adiabatic shear bands are formed. Subsequently oxides are removed from the surfaces of powders and substrate by the forming of the plastic metal jet and the metallically pure surfaces can come into contact [3,4]. Besides mechanical bonding mechanism, the other ones, such as e.g. the local metallurgical one was reported by Hussain [5] and by Guetta [6]. Such process parameters as the substrate preparation, operational spray parameters, and heat treatment of substrate prior to deposition and the deposited coatings may influence adhesion.

An important factor affecting the coatings' adhesion and hardness of cold sprayed coatings is the fraction of ceramic in a cermet powder. The addition of ceramics may act in a following way: (i) preventing of nozzle clogging; (ii), activating of the metallic surfaces by removing oxides; and, (iii) hardening of metal co-particles by tamping effect. Hence, an increase of the fraction of ceramics in cermet powder may, to a degree, improve the adhesion of the coating [2,7–15].

The adhesion values of low-pressure cold sprayed cermet coatings were reported in the literature to be as high as 60 MPa or even more [2,7–13]. The most commonly analysed cermet materials were: copper, aluminium and nickel with addition of $Al_2O_3$.

The presented research was carried out to check the influence of substrate preparation on mechanical properties of various types of cold sprayed coatings. The cold sprayed copper and nickel coatings can be used as electrical conductors. Aluminium, zinc and nickel can be deposited as the anticorrosive coatings. Moreover aluminium–alumina powder mixture is commonly used to regenerate defects in aluminium, steel or cast iron products. Tin coatings can be deposited on the contact surface of current connectors as an electrochemical corrosion protection coating.

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## 2. Materials and methods

### 2.1. Powders and substrates preparation

Commercially available powders of the following metals: tin (T2-00-05), zinc (K-00-11), aluminium (K-10-01), copper electrolytic (E-copperK-01-01), nickel (K-32) from Obninsk Center for Powder Spraying (Obninsk, Russia) and copper spherical (S-copper) from Sentes Bir, (Ankara, Turkey), were used in the spraying process. The granulometry tests (Analysette 22 MicroTec plus, Fritsch, Markt Einersheim) showed that the size of particles was in the range of $-50 + 10$ μm. Each powder was mixed with $Al_2O_3$ in weight ratio 50:50 before deposition (Obninsk Center for Powder Spraying, Obninsk, Russia). The morphology of the selected powders is shown in Fig. 1 (dark particles are $Al_2O_3$). The tin, zinc, S-copper and aluminium powders were produced by gas atomizing and were spherical. E-Copper and nickel powders were produced by electrolytic method and the particles were dendritic. The $Al_2O_3$ powder was prepared by crushing and its particles had an irregular shape.

The substrate materials were 7 mm thick discs having a diameter of 40 mm of copper M1E and aluminium alloy AA1350 with following chemical composition (wt.%): 0.12% Si, 0.24% Fe, 0.02% Cu, 0.01% Mn, 0.01% Cr, 0.07% Zn, 0.02% Ti and Al bal. The substrates surface was activated by sand blasting under a pressure of 0.6 MPa using alumina sand (mesh 45) or ground with SiC abrasive paper (mesh 1000). The aluminium alloy substrate AA1350 reached after grinding and sand-blasting surface roughness of $R_a = 0.36$ μm and $R_a = 6.45$ μm, respectively. The copper substrate the surface roughness were equal to $R_a = 0.33$ μm and $R_a = 5.27$ μm, respectively. The roughness was measured using Form Talysurf 120L profilometer (Taylor-Hobson, Leicester, UK).

### 2.2. Design of spray process experiments

The coatings were sprayed using a DYMET 413 (Obninsk Center for Powder Spraying, Obninsk, Russia) set up. The setup includes a heater and the de Laval nozzle with a circular outlet having an internal diameter of 5 mm. The process parameters such as: (i) gas temperature; (ii) gas pressure; (iii) gun linear speed; (iv) powder feeding rate; and (v) spray distance were kept constant during deposition (see Table 1). Air was used as the working gas and the distance between next spraying gun passes relative to the substrates was 3.7 mm. The process parameters collected in Table 1 were found to be optimal in respect of spraying efficiency for each powder.

### 2.3. Microstructure characterization

The metallographic examinations of powders and coatings were carried out using a SEM microscope with secondary electrons detector (Phenom G2 pro, Eindhoven, The Netherlands). The micrographs were made on the coating cross-section. The metallographic cross-section had been etched in accordance with Polish standard PN-75/H-04512 before examination, depending on the coating material: Sn coating with 1% NaCl; Zn with Nital; Al with 10% HF; Cu with $(NH_4)_2S_2O$ and Ni with 65% $HNO_3$. The phase analysis of coatings was made with X-ray diffractometer (D8 Advance, Bruker) using CuKα1 radiation in the range of 2$\theta$ angles from 20° to 100°. Diffrac + Eva software was used to identify phases in the coating microstructure. Furthermore semi-quantitative analysis was prepared to check the proportion of metallic powder and $Al_2O_3$ in the deposited coatings.

### 2.4. Adhesion strength

The adhesion tests were made following the standard PN-EN 582 [16]. The samples were hot bonded using epoxy resin Epidian 100 with an average strength of 70 MPa. In the case of tin coatings, cold-setting adhesive DISTAL with an average strength of 48 MPa was used. Three tests were carried out for each experimental run and the average adhesion value was determined. The tests were carried out for a constant coating thickness of 500 μm. The coatings deposited from the spherical Sn, Zn, S–Cu and Al powders were faced by turning. The coatings made of the dendritic E–Cu and Ni powders, were submitted to the tensile pull test as-sprayed. The following types of the failure at adhesion test were recognized: (i) adhesive—when the fracture occurred at the substrate/coating interface (A); (ii) adhesive–cohesive—the fracture occurred non-uniformly on the entire cross section (A/C); (iii) cohesive—when the fracture occurred within the coating (C); and (iv) failure in the epoxy resin (ER).

### 2.5. Microhardness measurements

Microhardness was measured following the standard PN-EN ISO 6507-3:2007P with the use of a Digital micro Hardness Tester MMT-X7 MATSUZAWA CO., LTD (Akita, Japan). Three different forces (1.961, 4.903, and 9.807 N) were chosen for each type of coating to eliminate the errors resulting from the heterogeneous composition of cermets. Microhardness was given as an average of five measurements in the middle of coating. However it is necessary to remember that the microhardness results cannot be compared directly because of different force values used in measurements.

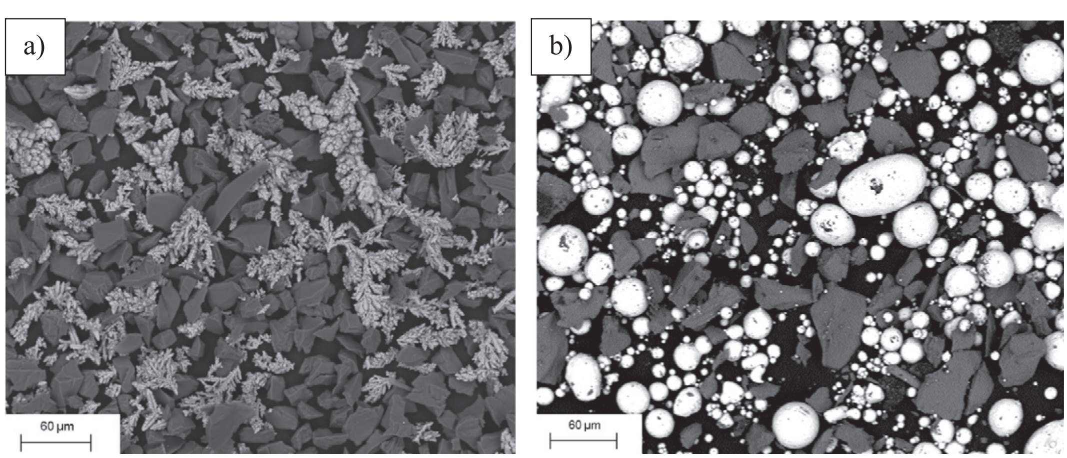


**Fig. 1.** SEM (secondary electrons) micrographs of example powders used to spray: E–Cu + $Al_2O_3$ (a) and S–Cu + $Al_2O_3$ (b).