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THE INFLUENCE OF COPPER POWDER MORPHOLOGY ON MECHANICAL PROPERTIES OF LOW-PRESSURE COLD SPRAYED COATINGS

Thermal spraying methods are commonly used to regenerate damaged surface or change materials surface properties. One of the newest methods is cold spraying, where coating is deposited of material in the solid state. Therefore shape and size of the powder particles are very important parameters. The article presents the influence of copper powder morphology on mechanical properties of the coatings (adhesion, hardness, Young's modulus) deposited with the Low Pressure Cold Spraying method on the AA1350 aluminium alloy substrate. The coatings were deposited using two commercially available copper powders with spherical and dendritic morphology and granulation of –40+10 μm. The bond strength of coatings was determined with the pull off method, the hardness with the Vickers method at load of 2.94 N, while the Young's modulus through measurement of nanoindentation. Microstructure of the coatings was analysed using the light and scanning electron microscopy (SEM). Shape of the powder influences mechanical properties of the coating significantly. The coatings deposited with dendritic powder had low mechanical properties, hardness of the 81 HV0.3 order and adhesion of about 4 MPa. However changing powder morphology to spherical increased hardness of the coating to 180 HV0.3 and adhesion to 38.5 MPa.

Keywords: cold spraying, copper coatings, coating bond strength, nanoindentation, AA 1350 aluminium alloy

1. Introduction

Coatings deposited with thermal spraying methods, because of the specificity of the process, as well as high efficiency, are widely used in the transportation, machine, electrotechnical, chemical and many other industries. The coatings are applied to improve the selected surface properties of material, protecting it against corrosion, abrasive wear, high temperature or increasing the electrical or heat conductivity. Thermal spraying is also applied for regeneration of damaged or worn parts [1-3].

In conventional thermal spraying process the melting of coating material occurs. Therefore the oxidation of metal particles occurs what negatively influences the structure of coating, and hence its properties [4-6]. The problem does not occur in cold spraying, because there is no melting of the powder used.

Powder particles introduced into the gas stream are only initially heated. The spraying gun equipped with the de Laval nozzle accelerates working gas to supersonic velocity. Thus particles introduced into the supersonic gas jet gain high velocity. High impact kinetic energy leads to plastic deformation of the particles which build the coating by mechanical interlocking and diffusion processes. Such a process enables obtaining homogeneous coating with very low porosity and oxidation [1,7-9].

Cold spraying can be divided into the low-pressure method and the high-pressure method. The low-pressure cold spraying (LPCS), because of the equipment mobility and low costs of the process is commonly used for the material regeneration [7-9].

Copper is the most frequently used material in the electronics, electrotechnical, refrigeration and installation industries. The cables, bus bars, terminals, coolers, heaters and many other components are produced of it which are frequently exposed to mechanical damage or corrosion losses. In order to bring back the correct functionality of a component the regeneration of the expensive parts is recommended. Various methods and materials can be used in regeneration process, e.g. arc spraying [10], flame spraying [11,12], plasma spraying [13], HVOF spraying [13]. However, high temperature processes cause defects in the coatings, e.g. oxidation and porosity. Therefore, many research were performed on cold sprayed copper coatings [14-19]. According to the research, the morphology of the applied powder is very

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important [20-25]. Particles with spherical shape compared to dendritic possess lower surface and concentrated mass. Therefore contact area of impacting particle is low and the kinetic energy is concentrated at smaller area. As a result intensive deformation of a spherical particle occurs, what converts to good mechanical properties of the coating. The dendritic particles have irregular shape, so their mass is not concentrated. In addition the contact area of dendritic particle with substrate is high which leads to distribution of energy. Therefore, the deformation of these particles runs non-uniformly [25,26].

Aluminium/copper contacts are the most prominently applied in power networks made of aluminium alloy busbars. However, bolted joints of Al and Cu cause galvanic corrosion of aluminium in the presence of electrolyte needing protective coatings or regeneration. The analysis of literature showed that the most frequently used material for the electrical conducting coatings is copper [27-29].

Copper coatings deposited by the low-pressure cold spraying (LPCS) are characterised by low porosity and high density, what provide high bond strength, as well as high electrical conductivity and resistance to corrosion [26,30-34]. In the relevant literature there is little publications concerning the influence of shape of the applied powder on properties of deposited coatings. Therefore in this article the mechanical properties of coatings deposited onto AA1350 aluminium alloy with powders of copper in the spherical and dendritic form was compared.

2. Materials and methods

In the tests coatings were sprayed using a DYMET 413 unit (Obninsk Center for Powder Spraying, Obninsk, Russia) equipped with spraying gun with the internal gas heater and the de Laval nozzle having the outlet diameter of 5 mm. Spraying gun was attached to the manipulator operating in 3 axes x, y, z. Spraying process parameters are presented in Table 1.

Two commercially available powders of copper: (i) dendritic (D-Cu) with granulation of –45+10 μm (average value of 31.6 μm), from Libra, Poland and (ii) spherical (S-Cu) and granulation of –55+10 μm (average value of 30.4 μm), from Sentes-Bir, Turkey (Fig. 1), were used in the tests. Before spraying the powders were subjected to separation using the vibration sieve with the mesh size of 40 μm. The spherical powder was produced with the gas atomisation method, whereas the dendritic powder with the electrochemical reduction method. The substrate was made of the AA1350 aluminium alloy (Table 2). For the coating pull-off test disc specimen of diamiter 40 mm were applied, whereas for the other tests specimen were rectangular of 20×30 mm. In both cases thickness of the substrate was 7 mm. Before spraying, the substrate surface was prepared by the grit blasting and obtained the roughness of $Ra = 9.62$ μm.

TABLE 1

Parameters of the spraying process

Working gas	Gas temperature T [°C]	Gas pressure p [MPa]	Stand-off distance l [mm]	Powder feed rate m [g/min]	Linear speed V [mm/min]
air	400	0.9	10	40	10
	600				

TABLE 2

Chemical composition of the AA1350 aluminium alloy (wt%, according to PN-EN 573-3:2010P standard)

Si	Fe	Cu	Mn	Cr	Zn	Ti	Al
0.12	0.24	0.02	0.01	0.01	0.07	0.02	balance

Measurements of surface roughness were performed using the stationary profilometer Form Talysurf 120L (Taylor Hobson, Leicester, U. K.), equipped with diamond cone probe with apex angle of 60° and rounding radius $r = 2$ μm. During the roughness measurements three parameters: Ra, Rz and Rt were determined.

The metallographic tests were performed with the use of SEM Phenom G2 pro microscope with secondary electrons detector (Eindhoven, The Netherlands), and the oxygen quantity at surface of the powder was determined using microanalyser of X-ray radiation LINK ISIS-300 (Oxford Instruments, Oxford, U.K.), coupled with SEM JSM 5800LV microscope (JEOL,
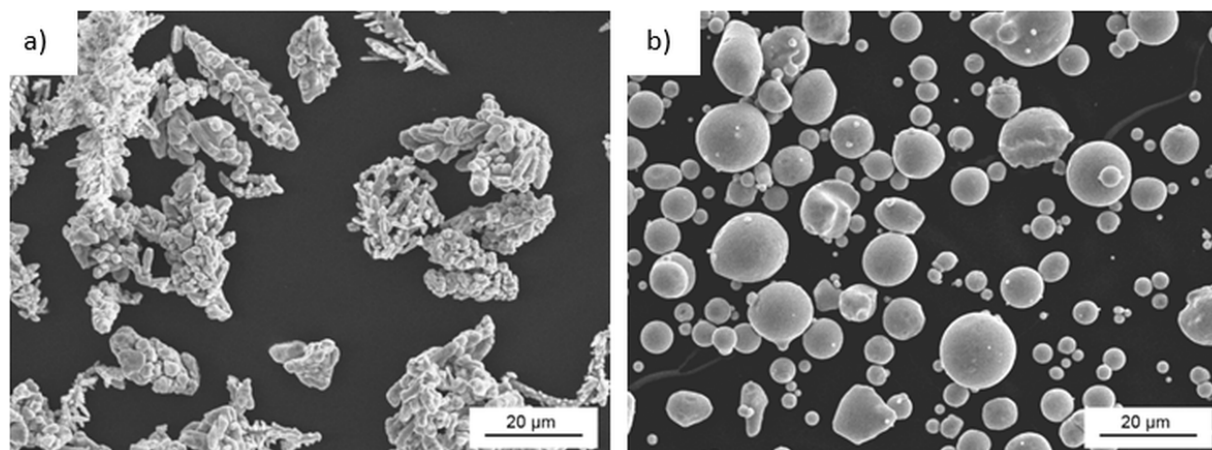

Fig. 1. SEM micrographs of morphology of copper powders used in the LPCS process: dendritic (D-Cu) (a) and spherical (S-Cu) (b)

Peabody, the USA). Porosity of coatings was determined by microstructure images performed at magnification of 100× using an image analysis software ImageJ. The metallographic cross-sections were etched in compliance with the Polish standard PN-75/H-04512 using $(NH_4)_2S_2O_8$. Microhardness measurement was performed with the Vickers method at the Digital Micro Hardness Tester MMT-X7 (MATSUZAWA CO. LTD, Akita, Japan), according to the European standard PN-EN ISO 6507-3:2007P.

The coating bond strength were analysed using the pull-off test according to the European standard PN-EN 582:1996 [35]. The value of bond strength R_H was determined as the ratio of the maximum applied load F_H to the specimen surface section S, according to the formula (1):

$$R_H = \frac{F_H}{S} \qquad (1)$$

For each specimen 3 tests were performed. The specimens were prepared using cold setting adhesive, DISTAL.

Measurements of nanoindentation were performed using the Indentation Release Candidate „SBO" (CSM Instruments, Peseux, Switzerland) and the Berkowicz indenter, which was pressed in with the maximum load of 250 mN in 15 s. As a result, an indent in the shape of a regular tetrahedron was obtained. The measurements allowed to define the microhardness HV_{it} and the indentation hardness H_{it}. What is more, the instrumental Young's modulus was determined by means of Olivier and Pharr method [36]. For each specimen 3 measurements were performed. The tests were conducted in the central part of the coating.

3. Results and discussion

Results of the coating surface roughness measurement are presented in Table 3. The roughness of the coatings deposited with the same powders at different temperature is similar. The highest value of roughness had the coating deposited with dendritic copper powder D-Cu. This results from irregular shape of the powder and low particle deformation. Dendritic particles deform non-uniformly, creating numerous craters on surface of the deposited coating [25,26]. Therefore the coating was characterised with high open porosity, which resulted in very high roughness. On the other hand the spherical powder after spraying showed higher deformation and provided low roughness of the S-Cu coating.

TABLE 3

Measurement results of roughness and waviness of the coating surface

Coating	Gas temperature T [°C]	Roughness [µm]					
		Ra	σ	Rz	σ	Rt	σ
D-Cu	400	32.4	1.5	61.6	7.8	190.3	6.2
	600	29.0	1.0	57.0	12.3	174.9	3.4
S-Cu	400	10.9	0.6	41.5	1.3	83.2	5.6
	600	11.4	0.8	44.2	1.0	90.3	6.3
σ – standard deviation							

The copper coatings deposited with dendritic powder and process temperature of 400°C and 600°C had thickness in the range of 420-640 µm and 460-760 µm, respectively. Similar thickness values indicate small dependence of powder deposition efficiency on the working gas temperature. In case of the spherical powder the coatings thicknesses of 260-590 µm and 460-780 µm were achieved, for process temperature of 400°C and 600°C, respectively. The influence of process temperature on deposition efficiency of spherical powder is significant and results from the increased softening of spherical particles.

The microstructure examination in the non-etched state showed that coatings deposited with dendritic powder at temperature of 400°C and 600°C had average porosity of 4.5% (Fig. 2a) and 3.5%, respectively. The spraying process temperature influences the degree of particles deformation and therefore the dendritic particles sprayed with higher temperature better filled the space in the coating. Coatings deposited with spherical powder at temperature of 400°C and 600°C obtained porosity of 1.1% (Fig. 2b) and 1.7%, respectively. The increase of process temperature allowed deposition of larger spherical powder
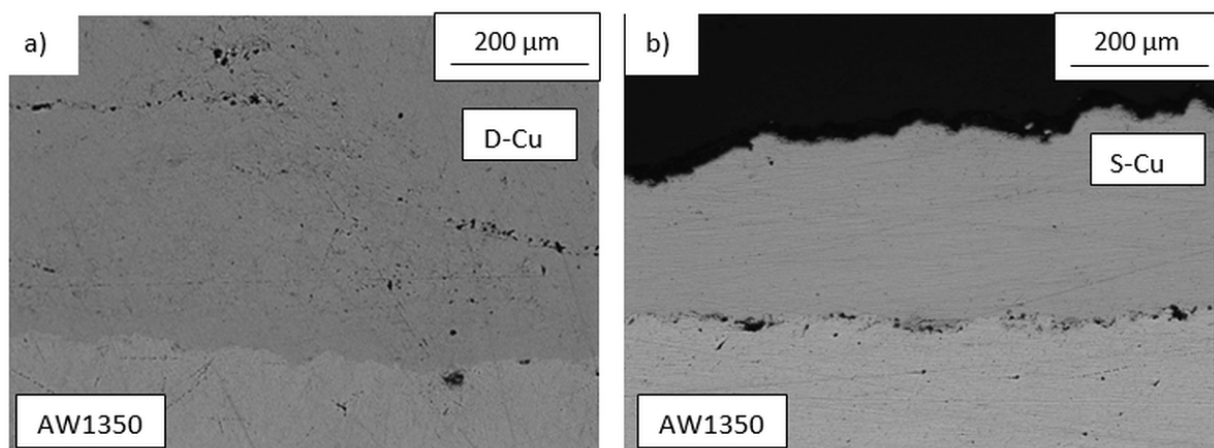

Fig. 2. Microstructure (light microscope) of coatings deposited with dendritic powder (a) and spherical powder (b) in temperature of 400°C

particles, which experienced smaller deformation. As a result increased porosity was obtained [7].

Microstructure of coatings deposited with both powders in the etched state revealed the shape of individual particles (Fig. 3, 4). The coating deposited with dendritic powder contained areas of intensive porosity where the particles experienced small plastic deformation (Fig. 3). In the coating deposited with spherical powder only local pores appeared, what provided high density of the coating (Fig. 4).

The obtained results of hardness measurement with Vickers method are presented in Fig. 5. Microhardness of particles of both powders before spraying was to 45.3 HV0.01 and 78.1 HV0.01 for D-Cu and S-Cu powder, respectively. For the coatings deposited with dendritic powder and process temperature of 400°C and 600°C the hardness reached the average values of 81 HV0.3 and 84 HV0.3, respectively. Obtained results are consistent with the literature [31]. Similar hardness values results from higher porosity of the coating deposited with temperature of 400°C (Fig. 6a), which caused high scatter of the results. At higher process temperature the density of the coating increased (Fig. 6b). In case of spherical powder the hardness of coatings was to 165 HV0.3 and 180 HV0.3 for process temperature of 400°C and 600°C, respectively. This results are comparable to Borchers et al. [20] as well as much higher than microhardness of 106 HV0.3 achieved by Koivuluoto et al. [31]. Microstructure of the coating deposited at 400°C showed high work hardening of the powder particles (Fig. 7a). Increase in process temperature led to further softening of the material, causing even higher deformation of particles, what is visible in the microstructure through significantly flattened and elongated grains (Fig. 7b). As a result the hardness
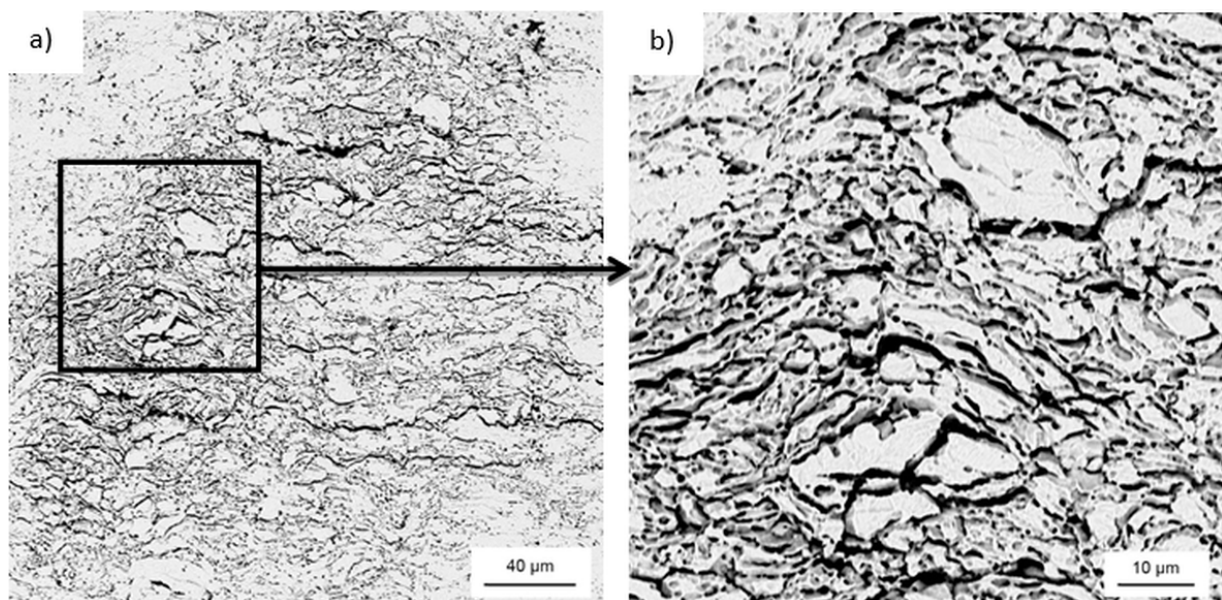

Fig. 3. Microstructure (SEM, BSE) of coating deposited with dendritic powder and temperature of 400°C (a,b)
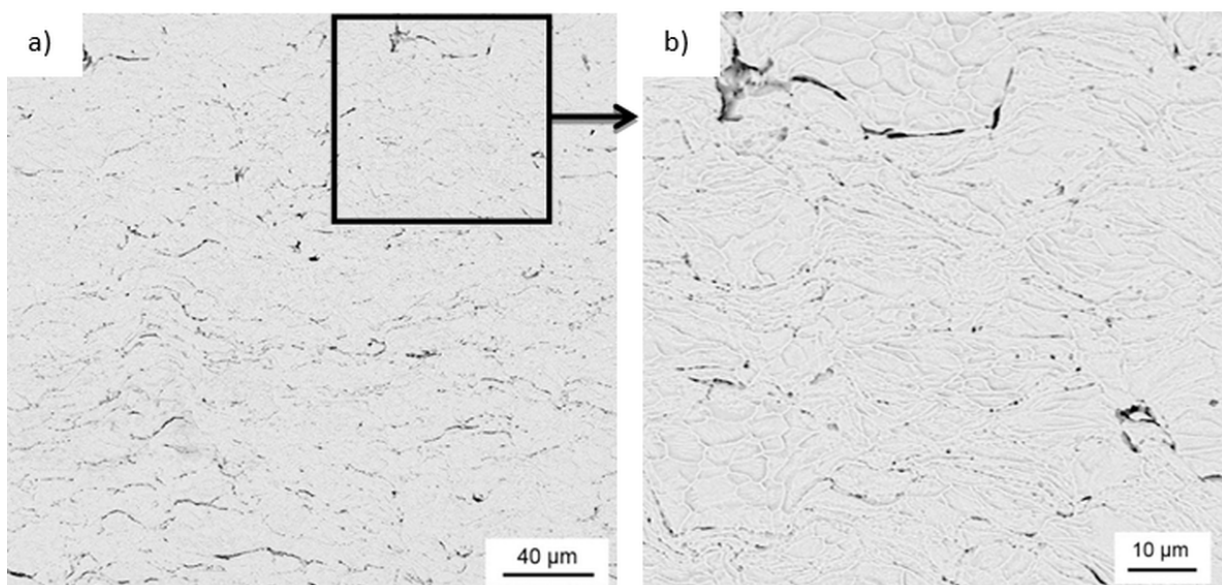

Fig. 4. Microstructure (SEM, BSE) of coating deposited with spherical powder and temperature of 400°C (a,b)
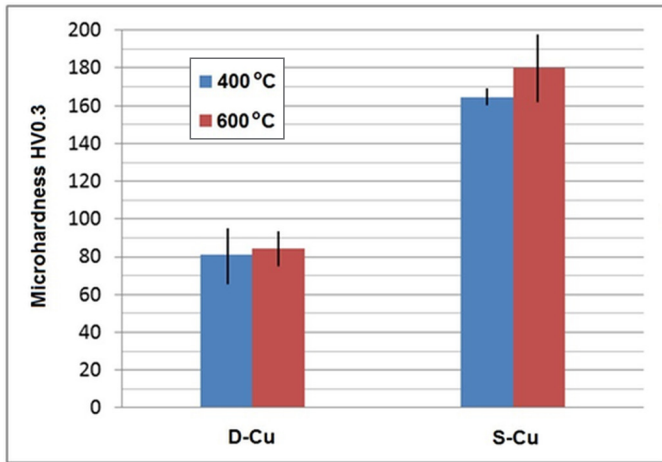
Fig. 5. Results of coatings hardness measurement

of the coating increased. Moreover, higher material hardness in the coating deposited with spherical powder is caused by several factors: (i) higher hardness of the applied powder, (ii) lower porosity of the coating, and (iii) higher degree of particle deformation during spraying process.

The results of the coating pull-off test are presented in Table 4. Significant difference between adhesion of coatings was obtained from dendritic and spherical powders, independently of the gas temperature applied in the process. The dendritic powder deformed very irregularly, what is visible in microstructure (Fig. 6). As a result of the porosity, the bond strength of coatings did not exceed 6 MPa. In case of spherical particles an intensive work hardening and decreased of porosity caused strengthening of the coating material (Fig. 7). As a result high bond strength of the coating up to 38.5 MPa was achieved. It is worth stressing
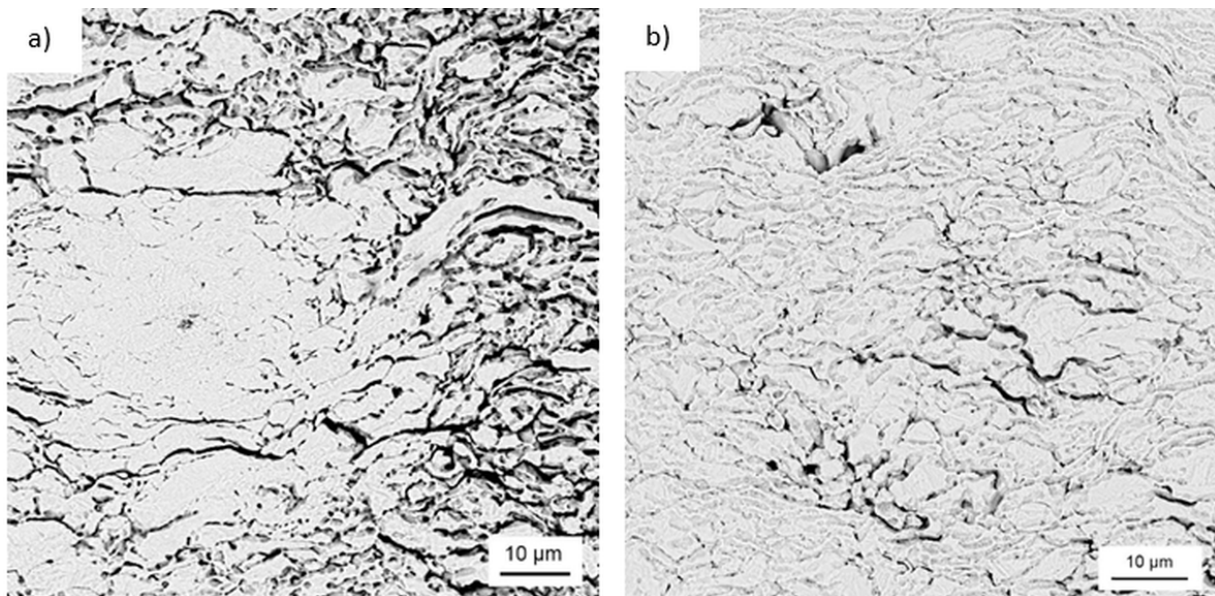

Fig. 6. Microstructure (SEM, BSE) of the coating deposited with dendritic powder in temperature of 400°C (a) and 600°C (b)
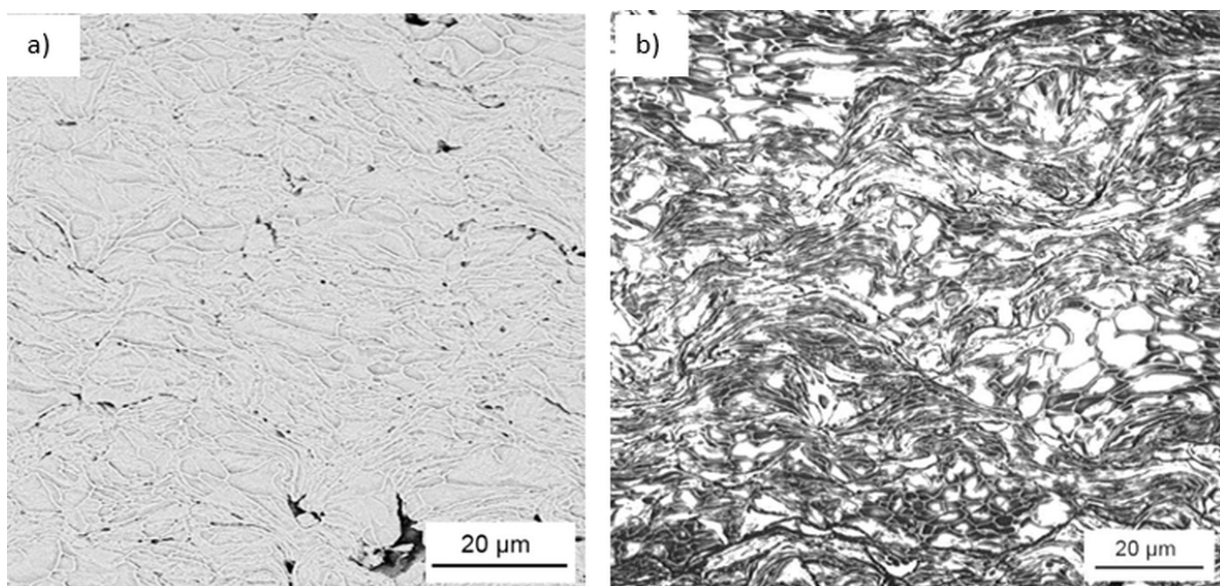

Fig. 7. Microstructure (SEM, BSE) of the coating deposited with spherical powder in temperature of 400°C (a) and 600°C (b)

that obtained results are consistent with the literature [20,31]. However, the bond strength achieved by Koivuluoto et al. [31] for spherical powder was 14 MPa, what arise probably from much lower gas pressure of 0.6 MPa.

Along with increase in the process temperature the bond strength of the coating deposited with dendritic powder increased, what results from the higher deformation of particles during spraying. In case of coatings deposited with spherical powder the bond strength was slightly lower. It is worth stressing, that the coatings obtained from spherical powder at temperature of 400°C had lower thickness, as well as smaller porosity. According to literature, along with increase in thickness of coatings their strength decreases [1,3].

For coatings deposited with dendritic powder the fracture was cohesive, as the failure occurred inside the coating. In order to improve the strength properties of dendritic coatings a deformation of particles during spraying should be increased, e.g. through introduction of admixture of ceramic powder to the copper powder [7-9]. In case of coatings deposited with spherical powder the adhesive fracture was achieved and the failure appeared on the coating/substrate boundary. Therefore satisfactory cohesion of the coatings was obtained.

The results of nanoindentation measurements are presented in Table 5. Analysis of the obtained results showed significant differences in mechanical properties of the tested coatings. It arise from different plastic deformation of powder particles sprayed of different morphology and with different working gas temperature. The coatings deposited with dendritic powder showed lower values of microhardness and Young's modulus in comparison with coatings deposited with spherical powder. The indentation hardness H_{it} for the coating deposited with dendritic powder at temperature of 400°C and 600°C was 0.869 H_{it} and 1.176 H_{it}, respectively, whereas for the coating deposited with spherical powder was 1.940 H_{it} and 2.055 H_{it}, respectively. The indentation hardness achieved by Sundararajan et al. [37] for coating depos-

ited with water atomised near spherical powder was 1.66 GPa. What is more, obtained in the research results are comparable with Vickers microhardness measurements. Higher microhardness of the coating deposited with spherical powder resulted from higher work hardening of particles and lower porosity. In case of both coatings an increase in microhardness was noticed along with the increase of working gas temperature. This was caused by more intensive deformation of powder particles in higher temperature.

The results of microhardness correlated with the value of Young's modulus, which is also much higher for the coating deposited with spherical powder and reached 114 GPa. The Young's modulus also increased with temperature for the coating deposited with dendritic powder. However, for the coating deposited with spherical powder a small decrease of Young's modulus was noticed, resulting from greater porosity of the coating. It is worth stressing, that obtained in the research highest Young's modulus of 114 GPa is comparable to copper bulk material elastic modulus of 124 GPa [38]. What is more, Sundararajan et al. [37] using water atomised non-spherical copper powder and similar cold spray process parameters achieved as-sprayed copper coating elastic modulus of 82.7 GPa and after 1 hour annealing in the temperature of 1073 K it increased to 115.0 GPa. Huang et al. [39] by annealing process in 700°C for 4 hours also decreased copper coating porosity and simultaneously increased coating elastic modulus from 84.3 to 111 GPa. Mechanical properties of material are determined by the porosity. However, elastic modulus decreases also due to occurrence of inter-splat boundaries [37,39]. Moreover, as conducted research showed, elastic modulus depend strongly on morphology of the used powder.

4. Conclusions

In the article the influence of copper powder morphology on mechanical properties of coatings deposited with the low

TABLE 4

Results of the coatings bond strength

Powder	Gas temperature T [°C]	Measurements					
		R_{H1} [MPa]	R_{H2} [MPa]	R_{H3} [MPa]	Average R_H [MPa]	σ [MPa]	Fracture type
D-Cu	400	3	4	4	3.8	0.9	K
	600	6	5	6	5.7	0.8	K
S-Cu	400	38	43	35	38.5	3.2	A
	600	39	36	32	35.7	2.9	A
R_H – coating bond strength, σ – standard deviation, K – cohesive fracture, A – adhesive fracture							

TABLE 5

Results of nanoindentation measurement

Powder	Gas temperature T [°C]	H_{it} [GPa]	σ	E_{it} [GPa]	σ
D-Cu	400	0.869	0.29	54	10.1
	600	1.176	0.05	89	4.1
S-Cu	400	1.940	0.13	114	6.1
	600	2.055	0.26	102	6.6
σ – standard deviation, H_{it} – indentation hardness, E_{it} – Young's modulus					

pressure cold gas spraying method was analysed. The conducted research showed that coatings deposited with powder of spherical shape were characterised with high work hardening and low porosity of 1.3%, which translated into high mechanical properties. What is more, the temperature of working gas showed a particular influence on properties of coatings deposited with dendritic powder.

The hardness of the coatings significantly exceeded hardness of the powder base material, which is the effect of intensive plastic deformation of particles during spraying. The coatings deposited with dendritic powder had lower hardness, what arise from high porosity. The coatings deposited with spherical powder achieved highest hardness of 180 HV0.3.

The pull off test showed significant difference in bond strength of the coatings. The coatings sprayed of dendritic powder, as a result of high porosity, had smallest bond strength, with cohesive fracture. In case of coatings deposited with spherical powder bond strength reached 38.5 MPa with adhesive fracture, which indicated for good internal strength of the coating. Increase in the process temperature increased significantly bond strength of the coating deposited with dendritic powder.

The results of nanoindentation measurement showed lower mechanical properties of coatings deposited with dendritic powder compared to the coatings deposited with spherical powder. Also, the influence of working gas temperature on properties of the deposited coatings was noted. The highest microhardness of 190 HV_{it} was shown by the coating deposited with spherical powder in temperature of 600°C, what arise from higher work hardening of particles. What is more Young's modulus of 114 GPa was also significantly higher for the coating deposited with spherical powder in comparison with 54 GPa obtained for the coating deposited from dendritic powder.

Summarizing the research LPCS coatings deposited with spherical copper powder showed highest mechanical properties and can be recommended to apply as copper coatings exposed to abrasion or other mechanical damage. However, further research is needed to analyse dedicated application.

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