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Possibility of spraying of copper coatings on polyamide 6 with low pressure cold spray method

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ABSTRACT

This paper discusses metallization of polymers using a low-pressure cold spray (Dymet 413). Three commercial copper powders: i) spherical and ii) dendritic were deposited on thermoplastic polymer - polyamide 6 (PA6). It was difficult to successfully apply a copper coating directly on the polymer substrate, therefore interlayers were applied. Additionally, the copper powder was pre-treated in hydrogen atmosphere to remove the oxide layer and reduce its critical velocity. Finally, the adhesion strength, electrical conductivity, oxygen content and microstructure of resulting coatings were determined. Coatings were characterized by one order of magnitude of lower conductivity than the bulk material and bond strength of 3.6 MPa. The powder shape turned out to have a decisive effect on the possibility on coatings formation.

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1. Introduction

Deposition of metal coatings onto polymers has been widely studied in order to improve thermal and electrical properties as well as to reduce surface degradation. Some well-established processes were used include physical vapor deposition (PVD) [1–4] or electroless deposition [5], but the thickness of the obtained layer was limited and deposition rates were low. Therefore, thermal spraying processes [6,7] are sometimes applied, which allow for the deposition of a thick coating on various substrate geometries, and the recoating of damaged elements. The main challenge is temperature-sensitivity of polymers. Recently, cold spray process was tested a potential solution. In this process, material is deposited in a solid state and therefore the temperature impact is lower in comparison to traditional thermal spraying methods. The powder particles are accelerated in stream of heated and pressurized gas and projected towards the substrate. Critical velocity is a key concept in the cold spray method [7]. It is defined as velocity that an individual particle of powder must attain in order to be deposited after impact with the substrate [8]. This definition is valid for ductile materials as brittle materials will cause erosion for any velocity at temperatures below their melting temperature [9]. Critical velocity depends mostly on the sprayed material's mechanical properties but varies also with particle size, particle morphology, particle impact temperature or powder oxidation [10–12]. The critical value may be calculated with the

equation proposed by Assadi et al. [11] and then developed by Schmidt et al. [10]. It takes the following form:

$$v_{cr} = \sqrt{\frac{F_1 \cdot 4 \cdot \sigma_{TS} \cdot \left(1 - \frac{T_i - T_R}{T_m - T_R}\right)}{\rho} + F_2 \cdot c_{pp} \cdot (T_m - T_i)} \quad (1)$$

where: ρ – density, σ_{TS} – tensile strength, T_m – melting point, T_i – impact temperature, T_R – reference temperature (293 K), c_p – specific heat of particle, F_1 – mechanical calibration (for cold spray 1.2), F_2 – thermal calibration (for cold spray 0.3).

The model takes into account the specific heat, tensile strength, mechanical and thermal calibration, but particles size is not included [10]: with decrease in particle size critical velocity increase. The possible reason for higher critical velocity of small particles may be the higher content of oxides or adsorbents hindering the bonding. Usually, powder contains a mixture of particles of varying diameters. In such cases, critical velocity is calculated for larger particles due to fact that smaller particles achieve a higher velocity [10].

Lupoi and O'Neill [13] pointed out that polymers might be coated when the particle impact energy calculated from the critical velocity of a given material and particle mass is sufficiently low. According to this formula, tin and lead will be easy to deposit, for aluminium and titanium deposition and erosion process will be coexistent, and for copper, the erosion process will be prevalent [13]. This assumption was confirmed for tin, which was deposited on various substrates PC/ABS, polypropylene, polystyrene and polyamide-6 [13]. The aluminium was deposited on PEEK [14] and Lexan [15], however in case of Lexan, the

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Table 1
Spray conditions for coatings deposition.

Powders	Gas temperature [°C]	Gas pressure [MPa]	Standoff distance [mm]	Gun traverse speed, [mm/min]	No. of spray gun passes	Powder feeding rate, [g/min]
Cu-S, Cu-D	200; 300	0.9	10	2000	1; 3	30

Cu-S – spherical copper powder; Cu-D – dendritic copper powder.

deposition process was possible only for high powder feed rates [15]. Additional spraying passes turned out to densify the aluminium coatings [16]. Thin copper coating was achieved on PA66 [17]. After deposition of the first layer, erosion occurred and further deposition was scanty. This was probably caused by a change of substrate surface to a metallic one and hence a change of parameters necessary for depositing a coating. Ganesan et al. [18] proved that the erosion process might be limited by taking advantage of different shapes of copper powders and using tuned interlayers. Thick copper coatings were achieved on PVC and epoxy [18]. They observed also that metal deposition on thermoset is more difficult because of their greater brittleness. The impact of powder particles causes erosion of the substrate instead of its plastic deformation.

The deposition efficiency on polyamide 6 depends probably also on the shape and oxidation level of the powder. It was already proved that some stresses remain in powder after the manufacturing process [19] which, together with a thin oxide layer, increases the critical velocity. These stresses might be reduced through heat treatment which results in lower critical velocity and hence better deposition properties. Therefore, in this paper the influence of heat treatment on copper deposition on polyamide 6 is studied.

2. Experimental procedure

2.1. Spraying conditions

The coatings were deposited using a low-pressure cold spray device Dymet 413 with a standard de Laval nozzle. Air was applied as working gas. The spraying parameters for coating are listed in Table 1 and for interlayers in Table 2. Two interlayers were selected based on literature: i) an interlayer from spherical copper particle and ii) tin with alumina admixture. Spherical copper particles have high inertia and therefore are stable in contact with subsequent sprayed particles [18]. Tin is an easy deformable material, with low critical velocity which might be deposited easily on various substrates [10,13]. These two interlayers should facilitate copper deposition. The final coatings were sprayed with dendritic copper powder on an Sn + Al₂O₃ interlayer in three spraying passes with gas temperature equal to 200 °C and gas pressure equal to 0.9 MPa.

2.2. Spraying materials

Three commercially available copper powders were used for spraying (Fig. 1): (i) spherical Cu (Libra, Poland), size of D10-5.94 μm, D50-13.98 μm, D90-25.41 μm (Fig. 1a), and (ii) dendritic Cu (Libra, Poland), size of D10-10.21 μm, D50-30.67 μm, D90-80.77 μm (Fig. 1c). Coatings were sprayed using Cu powders in delivery state and after heat treatment. Additionally, powder mixture of Sn + 50 wt% Al₂O₃ (Obninsk Centre for Powder Spraying, Russia), with particle size of

D10-11.3 μm, D50-22.3 μm, D90-40.6 μm (Fig. 1e), was used for interlayers.

Semicrystalline polyamide 6 (PA6) (Plastics Group, Poland) with glass transition temperature ~50 °C and melting temperature ~220 °C was used as a substrate material. The substrates were in shape of a plate with dimensions 3 × 50 × 100 mm for metallographic examination, in the shape of a disc with Ø 40 mm in diameter and a thickness of 10 mm for bond strength test, and in the shape of a rectangle with 5 mm width and 80 mm length for electrical resistivity measurements. Before spraying, the substrate was cleaned with acetone.

2.3. Temperature-programmed reduction measurement

The TPR measurements were done with a 15 mg sample placed into a quartz microreactor. It was made with a mixture of 5 vol% H₂ in Ar, and the temperature was increased linearly to 950 °C at a rate of 10 °C/min. The hydrogen consumption was monitored by TCD detector. The highest hydrogen consumption in case of a dendritic powder was ± 300 °C and in the case of spherical powder ± 450 °C (Fig. 2). The obtained values indicate that dendritic powders have higher oxygen content, which is caused by a developed surface. At the same time this oxide starts to reduce at a lower temperature.

2.4. Reduction of powder oxides

Data obtained from TPR measurements was used to reduce the oxides present in powders. The reduction was made with hydrogen (commercial purity 99.995%) using a self-made set-up consisting of a steel tube and furnace FCF7SM (Czylok, Poland), working in the temperature range 20–1150 °C with precision ± 3 °C. The powder was placed in the tube through which hydrogen was flowing. The temperature was measured using a thermocouple placed in the tube. The dendritic powder was reduced for 1 h in temperature ± 300 °C and the spherical one for 1 h at a temperature ± 450 °C. After heat treatment the powder was milled and sieved to get the particle size below 40 μm. The powders size after reduction of powder oxides amounted to: i) spherical Cu (Libra, Poland), D10-8.24 μm, D50-18.97 μm, D90-34.86 μm and (ii) dendritic Cu (Libra, Poland), D10-11.18 μm, D50-34.40 μm, D90-98.51 μm. The coatings were sprayed one day after preparing the powder.

2.5. Oxygen content measurements

The oxygen content in the used powder and resulted coatings was analysed using an O₂ and N₂ analyser - Leco TC436 (Leco, USA) with the inert gas fusion method.

Table 2
Spray conditions for interlayers' deposition.

Powders	Gas temperature [K]	Gas pressure [MPa]	Standoff distance [mm]	Gun traverse speed, [mm/min]	No. of spray gun passes	Powder feeding rate, [g/min]
Sn + Al ₂ O ₃	200	0.9	10	2000	1	30
Cu-S	200	0.9	10	2000	1	30

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