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Adhesion of functional layers based on epoxy and polyurethane resins for aluminum substrate

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ABSTRACT

The paper concerns pull-off strength test results of epoxy- and polyurethane-based coatings with three different powder fillers (carbonyl iron, ferrites, and graphite flakes). Polymer coatings were applied on an aluminum substrate that was degreased and/or pre-treated by means of abrasive blasting using electrocorundum. The roughness profile and the basic roughness parameters were determined, and the results showed a decisive effect of substrate preparation on the coating's adhesion. The highest values of pull-off strength were obtained from epoxy (3.88 MPa) and polyurethane (3.12 MPa) coatings containing MG192 graphite flakes. During the pull-off tests a cohesive detachment mechanism was observed only between the mentioned coatings and the substrate.

1. Introduction

Polymer coatings, due to their high durability, good adhesion to most substrates, ease of application, and low manufacturing costs, are commonly used for protection of various surfaces against the negative effects of the environment and external agents. Polymer coatings are most often applied for corrosion protection [1–4]. In addition to protective coatings, technical coatings are widely applied in order to endow the material with the desired mechanical, electrical, and thermal properties [5]. Technical coatings are used to ensure the material possesses the desired mechanical, electrical, and thermal properties, i.e. in coatings with improved hardness [6], abrasion resistance [7], or wear-resistance [8,9]. There are also a wide group of film-forming materials that have electromagnetic wave absorption properties [10,11]. In order to produce these electromagnetic wave absorption properties, various fillers are used for polymer coatings and materials, including carbonyl iron [12,13] or ferrites [14–16]. Graphite flake is also one of the fillers used in polymer coatings and composites to obtain electromagnetic radiation absorbing properties [17,18].

One of the crucial factors is good adhesion between the chosen coating and the substrate. The methods of quality and strength evaluation of such bonds include the following: knife, peel, hot water immersion, cathodic disbondment, salt spray, pull-off and bending test investigations [19–21].

Adhesion plays a crucial role in the bonding of many kinds of materials: composite materials, hybrid elements, lacquers, paints and coatings with metals or polymers, as well as on membranes and thin films. Adhesion, from the Latin "*adhesio*", is the process of attraction between molecules of touching substances. There have been many attempts to define this phenomenon, among others, adhesion is "a surface phenomenon involving the physical or chemical type of interaction between phases combined by their molecular contact, leading to the creation of new non-homogeneous system" [22] or "a phenomenon of surface binding to each other surface's layers of two different bodies, under the force of attraction between them" [23].

The key factor affecting the adhesion of a polymer coating to a metal or other substrate is its suitable preparation, for example: chemical modification of polymers by, for example [24,25], plasma, UV radiation, flaming, acid attack, application of intermediate layers [26], or mechanical treatments like sand blasting [27].

A properly prepared surface of a metal substrate (steel, aluminum) is a key factor affecting the good adhesion between the coating and the substrate. The preparation of the metal substrate prior to the application of the coating primarily consists of removing impurities and improving the surface roughness. The most common contaminants occurring on substrate surfaces are dust, dirt, oils, lubricants, water and moisture; in particular, metal oxides can prevent good adhesion of the coating to the substrate [28]. The necessity of an appropriate surface roughness for a substrate results from the procedure of the coating application process, because its adhesion is caused primarily by the mechanical anchoring of

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the first layer of the coating within the unevenness of the surface of the substrate. An improperly prepared surface of a metal substrate may cause many negative effects, i.e. reduction of adhesion of the coating to the substrate, development of corrosion at the interface between the coating and substrate, formation of bubbles, cracks, and peels on the coating, reduction of the smoothness and gloss of the coating, and changes in the colour of the coating [29].

This paper investigates the effect of the surface roughness produced by degreasing and abrasive blasting of an aluminum alloy on the adhesion of coatings with different functional fillers. Therefore, the main goal of this research is to determine the optimum surface preparation procedure from the point of view of the adhesion of certain types of coatings, and to study of the effect of fillers on the pull-off strength.

2. Materials and methods

2.1. Materials

2-mm-thick 6061 aluminum alloy sheet was chosen as the substrate. 6061 aluminum alloy (Werkstoff 3.3214, Polish designation PA45) has an average hardness of about 95 HB, has good corrosion resistance, and is characterised by the one of the highest thermal conductivities among aluminium alloys, which is above 160 W/m·K. The temper of 6061 aluminum alloy is fabricated (F).

Methods of modification of the 6061 aluminum alloy surface before coating treatment:

I – degreasing with acetone,
II – abrasive blasting (corundum F30, 30 s per sample, angle of abrasive blasting – 45°, ambient temperature, and 51% humidity) and degreasing with acetone. Corundum F30 (710–600 mm) is obtained from bauxite and contains 95% Al_2O_3, ~3% titanium oxide (TiO_2) and ~ 1–2% other impurities. The particles of corundum have a sharp-edged grain shape.

Two bi-component resins were selected to obtain polymer layers: epoxy (Epidian 652 + IDA hardener company Ciech Sarzyna) and polyurethane (F180 Axon company Sika).

As fillers the following were used:

- carbonyl iron powder Fe 99.5% min., APS <15 µm ($D_{10} \leq 2.5$–4.0 µm, $D_{50} \leq 6.0$–8.0 µm, $D_{90} \leq 12.0$–25.0 µm) (Kamb Import-Export, Poland) (Fig. 1a)
- ferrites powder FMS250; chemical composition: Fe_2O_3- 71%, MnO-20,7%, ZnO-8,3%, fraction; 0,25–0,30 mm: ≤ 5%; 0,20–0,25 mm: ≤ 15%; 0,15–0,20 mm: ≤ 25%; 0,10–0,15 mm: ≤ 25%; 0,05–0,10 mm: ≤ 25%; <0,05 mm:10%, (Ferroxcube, Poland) (Fig. 1b)
- flake graphite MG192, particles size <0,15 mm, (Sinograf, Poland) (Fig. 1c)

Coatings were prepared from mixtures of carbonyl iron powder, and ferrites were added to the amount of 30% by mass and flake graphite to the amount of 20% by mass with resins. Firstly, the resin component A was measured out, then the appropriate filler was added, mixed with a mechanical stirrer, and the resin component B was added, after which the whole was mixed until a uniform consistency was obtained. After mixing using a mechanical stirrer, the resin components and fillers, were manually applied to the 6061 aluminum substrates using a blade with cut-outs of a certain thickness, so that the obtained layers had an even thickness of about 500 µm.

Table 1 shows the compositions of samples produced and their descriptions.

2.2. Roughness measurements

The contact method was used to measure the roughness of the 6061 aluminum alloy substrates after treatments. This is based on the principle of moving a diamond blade with constant speed on the test surface. The signal can be registered or processed to determine the values of the roughness parameters Ra (average length between the peaks and valleys and the deviation from the mean line on the entire surface within the sample length) and Rz (vertical distance from the highest peak to the

Table 1
Obtained layers and their descriptions.

Name of samples	Resin	Fillers	Value of fillers [% mass]	Treatment of the substrate
D_PUR	polyurethane	–	–	degreasing
A_PUR	polyurethane	–	–	abrasive blasting
D_EP	epoxy	–	–	degreasing
A_EP	epoxy	–	–	abrasive blasting
D_PUR_iron_30%	polyurethane	carbonyl iron	30	degreasing
D_PUR_ferrite_30%	polyurethane	ferrites	30	degreasing
D_PUR_graphite_20%	polyurethane	flake graphite	20	degreasing
A_PUR_iron_30%	polyurethane	carbonyl iron	30	abrasive blasting
A_PUR_ferrite_30%	polyurethane	ferrites	30	abrasive blasting
A_PUR_graphite_20%	polyurethane	flake graphite	20	abrasive blasting
D_EP_iron_30%	epoxy	carbonyl iron	30	degreasing
D_EP_ferrite__30%	epoxy	ferrites	30	degreasing
D_EPgraphite_20%	epoxy	flake graphite	20	degreasing
A_EP_iron_30%	epoxy	carbonyl iron	30	abrasive blasting
A_EP_ferrite_30%	epoxy	ferrites	30	abrasive blasting
A_EP_graphite_20%	epoxy	flake graphite	20	abrasive blasting

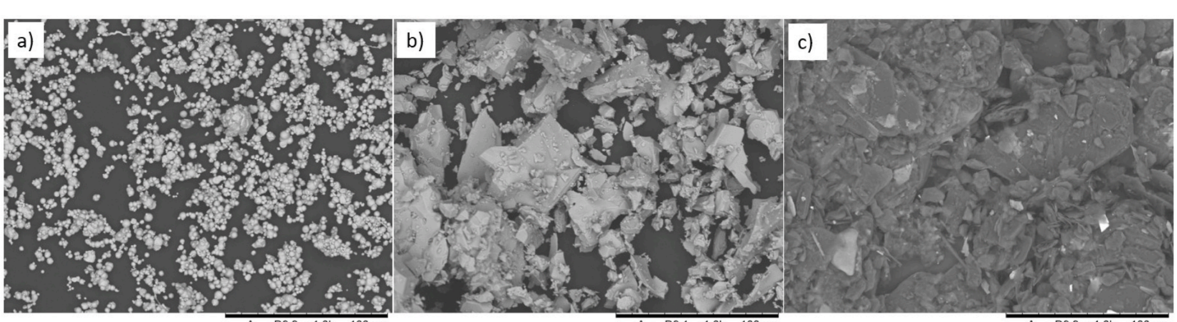

Fig. 1. SEM microscopic pictures of fillers: a) carbonyl iron powder, b) ferrite powder, c) flake graphite.

lowest valley within five sampling lengths and averaging the distances). Measurement of the surface roughness parameters was carried out using a MarSurf PS 10 contact-type surface roughness instrument manufactured by Marh GmbH, Germany.

2.3. Adhesion measurements

The pull-off adhesion tests of the epoxy and polyurethane coatings on differently prepared aluminum substrates were carried out according to the PN-EN ISO 4624:2016–05 standards using a Posi Test AT device, which allowed us to evaluate the adhesion (pull-off strength) of a coating by determining the greatest tensile strength that it can bear at the moment of detachment. Breaking points, demonstrated by fractured surfaces, occur along the weakest plane within the system consisting of a dolly, glue, coating layers, and substrate. Upon completion of the pull-off test, the dolly and coated surface were examined. On the surface of measured samples of 250 × 30 × 2 mm, measuring dolly of diameter 20 mm were attached by epoxy glue. After 24 h the circular notch around the stamp was cut and, subsequently, the pull-off test was carried out. The pull-off test classification according to the standard PN-EN ISO 4624:2016–05 [30] is presented in Table 2. The types of failures on the obtained samples are shown schematically in Fig. 2.

2.4. Scanning electron microscope (SEM)

A Hitachi TM-3000 SEM was used to examine the morphology of the pre-treated aluminum specimens. The samples of area 10 × 10 mm were graphite coated in order to produce a conductive surface before being placed in the vacuum chamber of the microscope. Cross-sections of the samples were prepared by immersing the samples in Epo-fix epoxy resin. The mounted samples were then polished with silicon carbide grinding papers (120, 400, 800, 1200, and 4000 grit size). Samples were subsequently fixed onto an aluminium stud using a double-sided sticky tape. The specimens were examined at chosen magnifications.

3. Results and discussion

3.1. Surface roughness

Fig. 3 (base units 0.5 μm) and Fig. 4 (base units 20 μm) present graphs of roughness profiles of aluminum alloy sheets after degreasing and after abrasive blasting, respectively. Additionally, Fig. 5 shows the cross-sections of the aluminum alloy sheet after degreasing (Fig. 5a) and after blasting (Fig. 6a). The presented microscopic photos as well as the roughness profiles show the development of the surface of the aluminum alloy sheet after abrasive blasting. The surface profile parameters (R_a and R_z) are given in Fig. 5, which shows that the largest values of the surface profile were measured for the surface prepared by abrasive blasting, i.e. after this pretreatment the aluminum sheet is characterised by a 25-fold higher R_a parameter and a 27-fold higher R_z parameter than observed for just degreasing. Considering the previously conducted studies, it was expected that the development of the specific aluminum surface would improve the mutual pull-off strength [19].

Table 2
Pull-off test classification of samples.

Designation	Description
A	Cohesive separation in the base
A/B	Adhesive separation between the base and the first layer
B	Cohesive separation in the first layer
B/C	Adhesive separation between the first and the second layer
N	Cohesive separation in the nth layer of the system
n/m	Adhesive separation between the nth and the mth layer of the system
-/Y	Adhesive separation between the last layer and the adhesive
Y	Cohesive separation in the adhesive
Y/z	Adhesive separation between the stamp and the adhesive

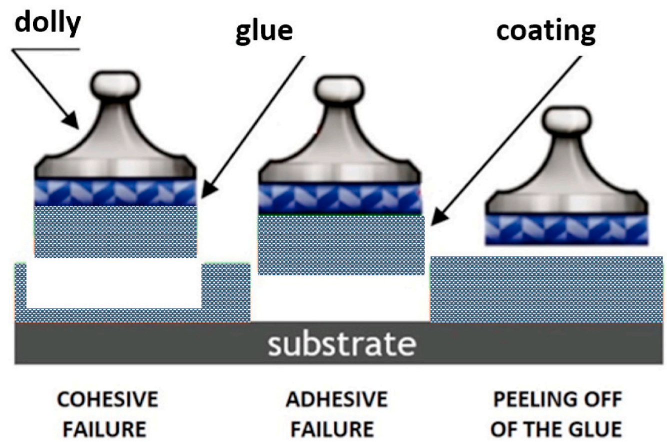

Fig. 2. The types of failures on the obtained samples.

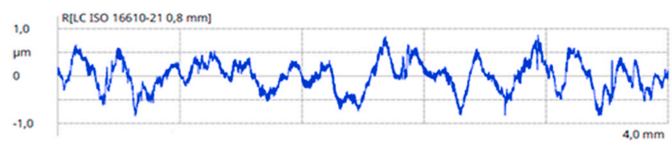

Fig. 3. Graph of roughness profile of aluminum alloy sheet after degreasing.

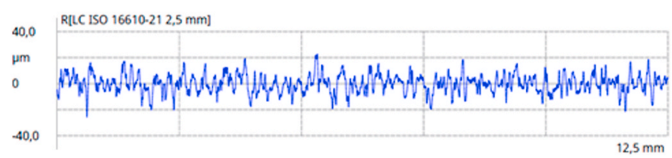

Fig. 4. Graph of roughness profile of aluminum alloy sheet after abrasive blasting.

3.2. Adhesion test

The average pull-off strength for the previously described specimens are depicted in Fig. 7. The average values were obtained from six pull-off tests, as in Ref. [1]. The pull off-strength was chosen as a reliable, simple and easy-to-use method for the evaluation of the adhesion between the coating systems and the substrates.

Epoxy resin-based coatings have greater adhesion on degreased 6061 sheet than do polyurethane resins. Epoxy-based coatings with ferrite powders exhibit higher pull-off strength than the same coatings reinforced with carbonyl iron. Various pull-off mechanisms were observed for samples after abrasive blasting (Figs. 8c and 9c). Detachment occurred inside the coating (cohesive detachment, classification according to the standard PN-EN ISO 4624:2016 [30]: B – cohesive separation in the first layer) only for coatings containing graphite; all other coatings detached from the substrate (adhesive detachment, classification according to the standard PN-EN ISO 4624:2016 [30]: A/B – adhesive separation between the substrate and the first layer) (Figs. 8a, 9a and 8b, 9b show the macroscopic view of samples). Adhesive fracture, being related to the exposed metal surface, leads to facilitated oxidation [1]. The increase in roughness as a result of abrasive blasting significantly affected the pull-off strength values. On the other hand, it has to be underlined that the substrate's roughness profile does not always correlate with the adhesion strength. There are usually some optimal values of roughness for a particular coating system, which are beneficial in terms of mutual adhesion. A further increase in the roughness parameters can even deteriorate the pull-off strength [19,28]. The highest pull-off strength values (3.88 MPa) were obtained for epoxy resin samples containing 20% mass flake graphite on a 6061 aluminum alloy

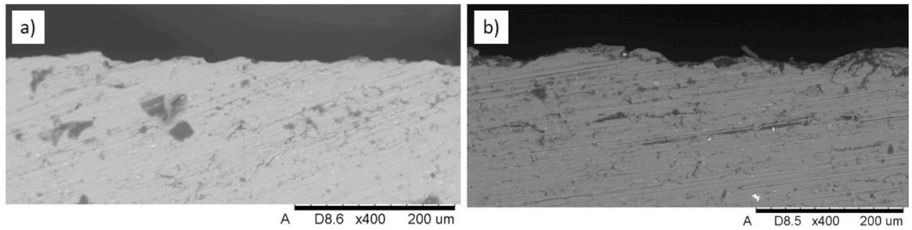

Fig. 5. SEM micrographs of the aluminum alloy sheet cross-sections a) after degreasing, b) after abrasive blasting.

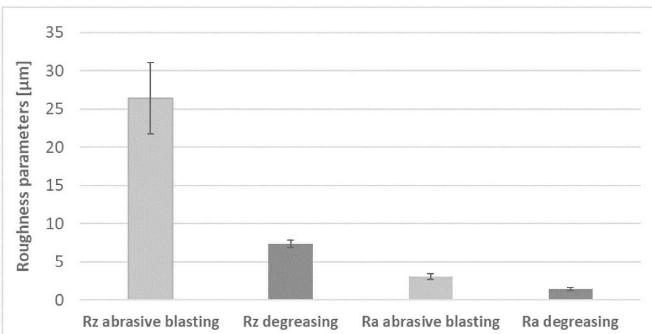

Fig. 6. Ra and Rz parameters for an aluminum substrate after abrasive blasting and degreasing.

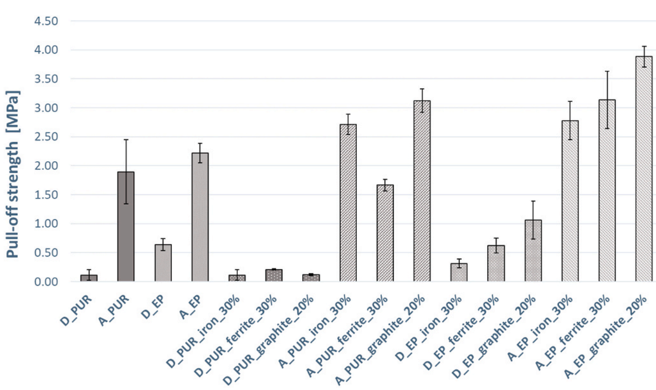

Fig. 7. Pull-off strength results of polymeric layers obtained on aluminum alloy substrates.

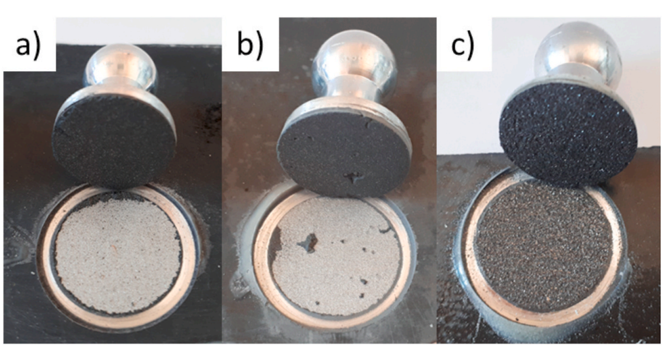

Fig. 8. Polyurethane coatings after pull-off tests a) with ferrites, b) with carbonyl iron, c) with flake graphite.

substrate after abrasive blasting. The same coatings applied only on the degreased aluminum alloy substrates had more than four times lower pull-off strength. Chemical treatment can be suitable to enhance the

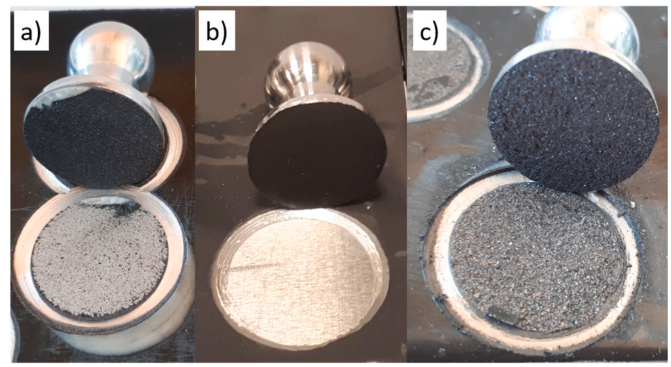

Fig. 9. Epoxy coatings after pull-off tests a) with ferrites, b) with carbonyl iron, c) with flake graphite.

adhesion of polymer coatings, which is connected with the surface roughening on a molecular scale [24], but in the studied case the mechanical methods were found to be more beneficial, both for epoxy and polyurethane matrices. Apart from abrasive blasting, other methods such as flaming [27] might also be considered in future tests.

Microfillers can lead to a distinct increase of the adhesion strength. It is also suspected that other kinds of reinforcement, e.g. nanoparticles, might be suitable for this purpose [31].

3.3. Microscopic analysis

Cross-sections of the samples confirm that the applied surface development methods were chosen properly. After abrasive blasting, in each of the observed examples, the surface of the aluminium substrate was significantly roughened, creating irregularities that promote mechanical anchoring with the adhesive. Fig. 10 shows the cross-sections of coating reinforced with 30% mass of ferrite filler. Due to the short pot life of the polyurethane resin (~3′25″), ferrite particles that are larger than carbonyl iron may not be completely wetted by the resin. Analysis of the micrographs and the results of pull-off tests lead to the conclusion that wetting of the aluminum surface by the reinforced epoxy resin (see Fig. 10a) was better than for the polyurethane one (see Fig. 10b), where the detachment is already visible, even before the pull-off tests. Such an area is described elsewhere as the debonding zone [26]. Thus, the pull-off strength of the A_PUR_ferrite_30% mass sample was severely deteriorated. Moreover, in both cases the gravitational sedimentation of the fillers in the polymer resins was ascertained, because of the high density difference. Fig. 11 presents the cross-sections of coatings composed of polyurethane resin and carbonyl iron (Fig. 11a) and flake graphite fillers (Fig. 11b). In both samples the observed wetting of the developed aluminum substrate's surface by the coating is correct, and the distribution of the filler within the matrix is beneficial, i.e. it is evenly dispersed with no sign of sedimentation.

4. Conclusions

The effect of filler content (carbonyl iron, ferrites, flake graphite) in a

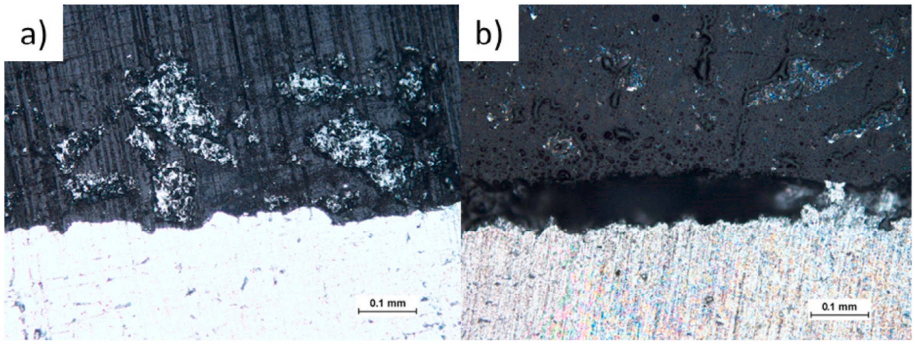

Fig. 10. SEM micrographs of the sample cross-sections: a) A_EP_ferrite_30%, b) A_PUR_ferrite_30%.

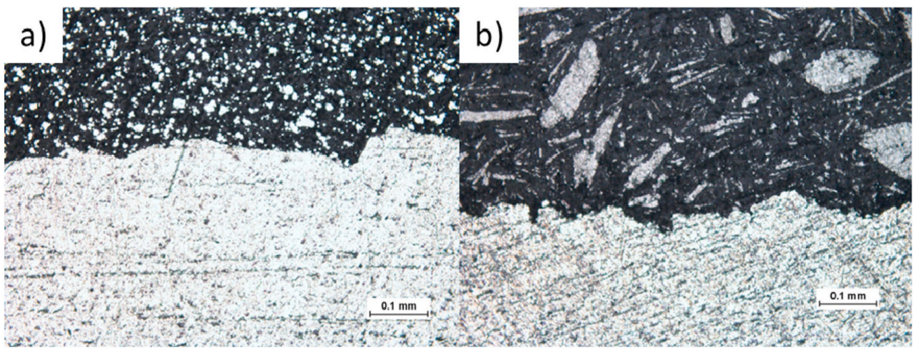

Fig. 11. SEM micrographs of the sample cross-sections: a) A_PUR_iron_30%, b) A_PUR_graphite_20%.

resin applied on a treated 6061 aluminum alloy surface was evaluated with the pull-off strength technique. Moreover, the identification of the bond failure at the coating-substrate interface was performed, which is crucial for the proper protection of the substrate during the life of the coating element.

The following main conclusions may be drawn:

1. Polyurethane coatings with or without fillers do not show good adhesion to an aluminum alloy substrate that has only been degreased with acetone.
2. An increase in 6061 aluminum alloy substrate roughness increases the adhesion of polyurethane and epoxy coatings.
3. Comparing the adhesion of pure polyurethane and epoxy coatings, the epoxy layers showed greater adhesion to the 6061 aluminum alloy substrate.
4. After abrasive blasting, the addition of fillers to epoxy-based layers caused an increase in pull-off strength, which can be explained by increased mechanical adhesion. An increase in the roughness of the substrate results in mechanical interlocking between the resin and the metal surface.
5. Various detachment mechanisms were observed during pull-off tests. For resin-based layers with carbonyl iron and ferrite, adhesive detachment from the substrate occurred. In contrast, cohesive detachments occurred for both polyurethane and epoxy coatings filled with graphite flakes. The highest pull-off strength values were obtained for these coatings (3.88 MPa for epoxy and 3.12 MPa for polyurethane).

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