Delft University of Technology

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Mariana Paz Martinez-Viademonte [a,b,*], Shoshan T. Abrahami [c], Theodor Hack [b], Malte Burchardt [d], Herman Terryn [a]

[a] Department of Materials and Chemistry, Research Group, Electrochemical and Surface Engineering (SURF), Vrije Universiteit Brussel, Pleinlaan 2, 1050, Brussels, Belgium
[b] Airbus Central Research and Technology, Willy-Messerschmitt-Straße 1, 82024, Taufkirchen, Germany
[c] Department of Materials Science and Engineering, Delft University of Technology, Mekelweg 2, 2628 CD, Delft, the Netherlands
[d] Airbus Operations GmbH, Airbus-Allee 1, 28199, Bremen, Germany

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ABSTRACT

The adhesion capabilities of sulfuric acid and tartaric-sulfuric-acid anodic oxide films on cladded AA2024-T3 aluminium substrates have been investigated in a quantitative manner. A relatively simple, versatile and industrially applicable test methodology based on DIN EN ISO 29862 has been used. In addition, the effect of anodising process parameters on the oxide film morphology has been studied and correlated with the adhesion results. The process parameters considered are the chemistry of the acidic pickling pre-treatment step, the addition of tartaric acid to the sulfuric acid electrolyte, the formation voltage, and the electrolyte temperature. The suggested tape peel adhesion test is able to differentiate among pre-treatments and anodising conditions showing a good correlation with the morphological features at the ultimate surface. High peel adhesion strengths are measured when topographies such as pore mouth widening and dissolution-driven roughness take place. These morphologies are typically found in anodic layers formed at elevated electrolyte temperatures. The proposed method can be beneficial for a fast assessment of anodising parameters for good adhesion, which is of special interest for the industrial optimisation of anodising processes.

1. Introduction

Aluminium alloys, in particular Al–Cu and Al–Cu–Zn–Mg alloys, are common in the aerospace industry due to their high strength to density ratio [1]. These alloys are susceptible to corrosion, therefore there is a high need for corrosion protection [2]. An aerospace corrosion protection arrangement typically consists of multiple layers: a porous anodic oxide layer, an inhibited organic coating and an organic top coat.

The anodic oxide layer has two purposes in this system. On the one hand it provides barrier protection against the ingress of moisture and salts, preventing direct contact with the aluminium substrate. On the other hand, its porous structure enhances the adhesion of the organic coating.

Determining the dry adhesion of organic coatings to metallic substrates is not an easy task. At an industrial level, the cross-cut test is typically used to assess the adhesion of organic coatings to anodised substrates [3]. This method consists of cuts in a lattice arrangement that penetrate into the corrosion protection system (e.g., organic coating and anodic layer) and into the underlying alloy. A pressure sensitive tape is applied over the cut area and rapidly peeled off. Adhesion is judged by optically assessing the area, where the coating is peeled off or detached. While this test is a simple and fast pass/fail test, it is a qualitative and an operator dependent test. Moreover, this method is not able to differentiate between the substrate and the coating adhesion capabilities. Instead, combined information of both components is obtained. Thus, bad adhesion results are difficult to attribute to the substrate, to the coating or to compatibility issues between them.

The aim of this investigation is to assess the suitability of a fast,

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simple and quantitative test method, based on DIN EN ISO 29862 [4], to characterize the dry adhesion properties of anodic oxide films as a coating-independent alternative to the industrial cross-cut test. The characterization of the adhesion properties of anodic oxide films is particularly relevant as not only the properties of organic coatings are relevant for adhesion but also the properties of the substrate on which these coatings are applied are of importance. In fact, the significance of the oxide surface chemistry (such as the amount of available surface hydroxyl groups) and of the oxide morphology (such as porosity, pore diameter and sub-surface roughness) in adhesion has been previously reported [5–8]. The oxide surface chemistry influences the molecular or atomic interaction at the oxide/coating interface, as proposed by the adsorption adhesion theory [9]. The oxide morphology also influences the mechanical interlocking of the coating at a macroscopic level, and therefore also contributes to adhesion from the perspective of the mechanical adhesion theory [9]. In fact, the ultimate oxide surface, or what is the same, the anodic oxide/organic coating interface region, has been shown to steer dry adhesion. That is the adhesion of organic coatings and adhesives in a dry condition, prior to the exposure to humidity or any aggressive media [5]. Bulk oxide morphology has been shown to have little effect on the dry adhesion behaviour [5].

The DIN EN ISO 29862 [4] standard describes a method to determine the peel adhesion strength of pressure sensitive tapes. The pressure sensitive tape is applied to a standardized substrate. Then, a tensile test machine peels the tape, recording the peel adhesion strength. In the approach proposed in this study, a well characterized pressure sensitive test is applied to substrates anodised under different conditions. This allows to characterize the peel adhesion properties of the tape on different anodic oxides and to derive the contribution of the ultimate oxide surface to adhesion. In comparison to the conventional cross-cut test, this method provides quantitative data (peel adhesion strength) and could be a useful first approach to optimise the anodic oxide layer characteristics, and consequently, to optimise the anodising process for good adhesion. In addition, this method could be applied in an industrial environment for quality control, minimising the human factor.

To assess the capabilities of the tape peel adhesion method within this paper, AA2024 cladded substrates have been pre-treated and anodised in different solutions. Also, the influence on adhesion and morphology of anodising process parameters, such as the formation voltage and electrolyte temperature, has been studied. Three anodising electrolytes have been considered: sulfuric acid (SAA), and two mixed tartaric-sulfuric acid electrolytes (TSA) with specific tartaric/sulfuric acid ratios. TSA has been introduced in the aerospace industry as a replacement for chromic acid anodising, in the frame of the European REACh regulations [10]. Despite the fact that there are already several studies dealing with the corrosion protection properties of TSA anodic oxide films ([11,12]), the adhesion properties of these oxide films, and the effect of anodising parameters on adhesion have received little attention so far.

2. Material and methods

2.1. Pre-treatment

The material used was an AA2024 alloy cladded with a commercially available pure aluminium grade (1050). The test panels (150 × 80 × 0.6 mm) have been degreased in an alkaline cleaner for 15 min and alkaline etched for 3 min. Both steps were carried out using commercially available solutions. Subsequently the samples have been immersed in an acidic pickling solution for 5 min. Two different chemistries have been used for the acidic pickling step: Bonderite C-IC SmutGo NC AERO and Socosurf A1858/A1806. Bonderite C-IC SmutGo NC AERO is a chromate free deoxidizer containing ferric sulfate, nitric acid and sodium hydrogen difluoride, while Socosurf A1858/A1806 is a nitric and sulfuric acid based fluoride free deoxidizer.

2.2. Anodising

After pre-treatment the samples were anodised to mimic a typical industrial profile (Fig. 1) according to the parameters listed in Table 1. Anodising was carried out in three different relevant electrolytes: SAA (40 g/l sulfuric acid), TSA (80 g/l tartaric acid and 40 g/l sulfuric acid) and T^+SA (150 g/l tartaric acid and 40 g/l sulfuric acid).

2.3. Adhesion testing

DIN EN ISO 29862 [4] describes a method to determine the peel adhesion strength of self-adhesive pressure sensitive tapes. In this method the single-sided pressure sensitive tape is applied on a clean reference stainless steel plate. The plate is then fixed vertically in one of the grips of a tensile testing machine. The other grip of the machine pulls the free end of the adhesive tape at an angle of 180° to the plate. In our case, instead of using a reference stainless steel plate, we have used the test panels prepared according to the description in sections 2.1 and in 2.2.

Two 25 mm wide pieces of a pressure sensitive tape (3 M XT-0034-0095) are applied per test panel. The tape consists of a polyethylene coated base material and a cross-linked rubber-resin adhesive. The tape is pressed by means of a silicone rubber roller. To avoid shear forces, the tape is positioned in such a way that the alignment to the panel edges is ensured. The tape is then peeled by a tensile test machine at a test speed of 300 mm/min within 24 h after application. The force needed to peel off the tape from the anodised aluminium substrate is measured and averaged over the test length (peel adhesion strength). Fig. 2 schematically depicts the test setup. The sample preparation and the mechanical tests were performed at 23 °C ± 1 °C and 50% ± 2% relative humidity. At peel adhesion strengths above 53 N/25 mm, the tape fabric would detach from the adhesive, while the adhesive continued to adhere to the metallic substrate or the tape would even break. Therefore, when this limit was reached the actual force needed to peel the adhesive from the substrate could not be measured. These cases are indicated as peel adhesion strength above 53 N/25 mm.

2.4. FE-SEM imaging

The morphology of the anodic oxide films has been characterized by a Zeiss Auriga field emission scanning electron microscope. The acceleration voltage was 15 kV. The anodic layers have been observed in top view and in cross-section. Cross-sections were prepared at room temperature by the fracture method.

3. Results

3.1. Peel-adhesion strength

In order to assess the sensitivity of the newly introduced method to differences in the anodising process, the formation voltage, the electrolyte temperature, and the electrolyte chemical composition have been varied. The peel adhesion strength of the adhesive tape on the anodised substrates has been measured for different parameter
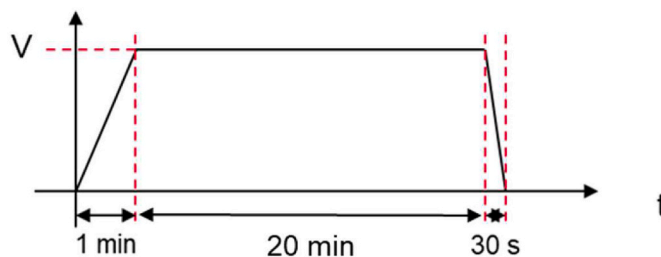

Fig. 1. Schematic view of the applied potential profile.

Table 1
Anodising parameters under study.

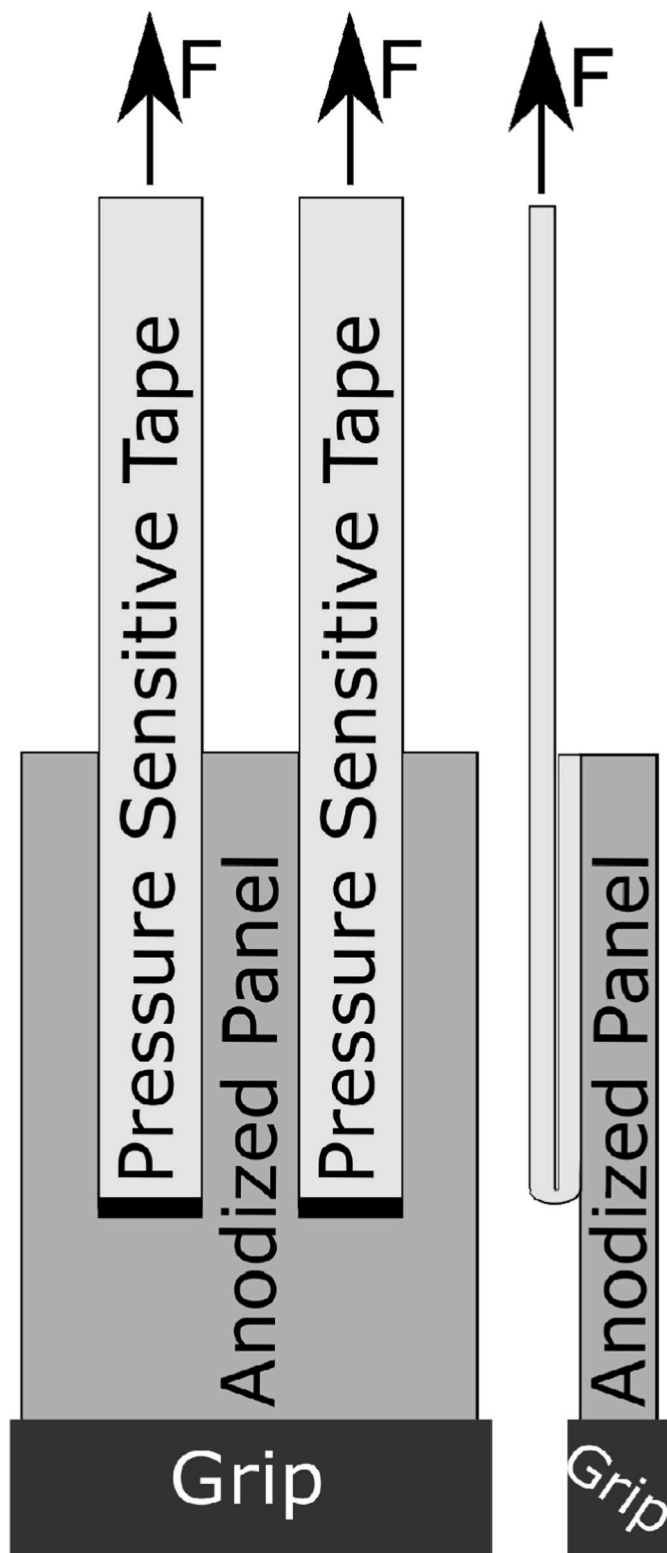| Temperature [°C]	Voltage [V]	[Tartaric acid] [g/l]
37	10	0
45	14	80
55	20	150

Fig. 2. Schematic representation of the adhesion test setup.

combinations. The average peel adhesion strength values for each anodising condition are summarized in Fig. 3, while the corresponding standard deviation can be found in the supplementary information. If during the measurement the adhesive film was de-bonding at the tape/ adhesive interface, instead of at the adhesive/aluminium interface or if the tape broke (see Fig. 4), this is indicated as a peel adhesion strength higher than 53 N/25 mm in Fig. 3. Therefore, the differences in adhesion capabilities among substrates with a peel adhesion strength higher than 53 N/25 mm cannot be assessed.

The three considered parameters (voltage, temperature, and electrolyte composition) have an influence on the obtained peel adhesion strength.

Analysing Fig. 3 deeper, it indicates that the formation voltage has a rather minimal effect on the peel adhesion strength measured. Yet, a trend towards a slightly higher peel adhesion strength with higher formation voltage can be observed. This is specially the case in combination with high electrolyte temperatures (e.g., SAA 45 °C, TSA 55 °C and T$^+$SA 55 °C in Fig. 3).

In contrast, increasing the electrolyte temperature leads to a marked increase in peel adhesion strength. However, from the results of the anodic oxides formed in SAA, it can be derived that there is an optimum temperature above which the peel adhesion strength drastically decreases.

While the addition of tartaric acid to the sulfuric acid electrolyte does not have a significant influence on the peel adhesion strength measured, at least for temperatures below 55 °C, it has an effect on the temperature at which the increase of the peel adhesion strength takes place. While the peel adhesion strength drastically increases for anodic films formed in SAA at 45 °C, this increase only takes place at 55 °C in the presence of tartaric acid.

If the dissolution of the oxide in the anodising electrolyte is low the pre-treatment steps prior to anodising also determine the ultimate-surface characteristics. Due to the fact that the anodic oxide growth takes place at the oxide/substrate interface, the modifications introduced by the pre-treatment at the substrate's surface could be retained after anodising at the ultimate oxide surface. Consequently, the surface features created during the pre-treatment would play an important role in the dry-adhesion properties of the oxide film. The peel adhesion strength of samples after pre-treatment in different acidic pickling solutions before and after anodising has been measured to assess the pre-treatment influence on the adhesion properties. In the case of the samples pre-treated in the Socosurf bath, the peel adhesion strength is slightly higher (35.7 ± 0.2 N/25 mm) than for the samples pre-treated in Bonderite C-IC Smut Go (30.9 ± 2.5 N/25 mm). After anodising there is no significant difference in terms of peel adhesion strength among pre-treatments (29.5 ± 0.4 and 30.7 ± 0.5 N/25 mm for Socosurf and Bonderite C-IC SmutGo respectively). Remarkably, as summarized Fig. 5 the peel adhesion strength measured for anodised substrates is similar to the results obtained on substrates that have been pre-treated only.

3.2. FE-SEM characterization

It can be expected that the set of anodising conditions discussed in the previous part will lead to important changes in the porous anodic oxide morphology and thus affect the adhesion properties as it has been suggested in prior research [5]. FE-SEM micrography has been conducted to investigate how the adhesion results correlate with the morphological features of the oxides under study.

The effect of the anodising voltage, the electrolyte temperature and the electrolyte chemical composition both in terms of total anodic oxide thickness and specific surface morphology has been assessed. The anodic oxide film thicknesses measured as a function of the anodising parameters are summarized in Fig. 6.

Increasing the voltage causes a drastic increase in the anodic oxide film thickness (Fig. 6). The cross-section images (Fig. 7) show increasing pore diameters with increasing voltage. In addition, there are no signs of
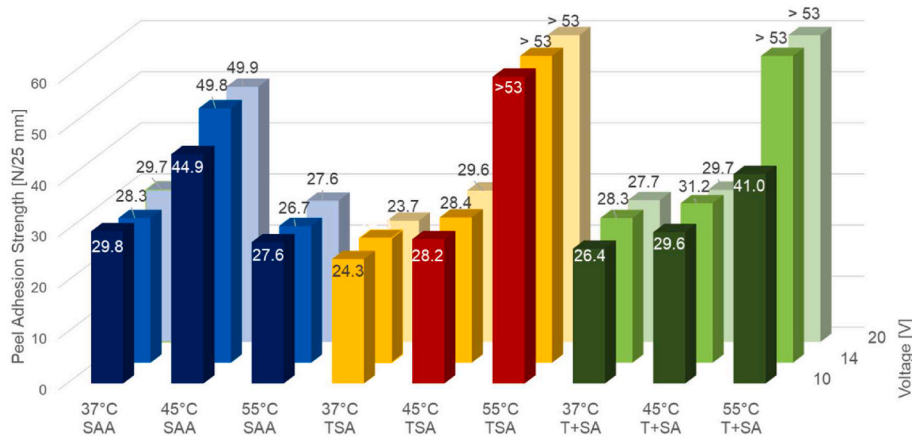
Fig. 3. Average peel adhesion strength results of substrates pre-treated in "Bonderite C-IC SmutGo AERO" and anodised in different electrolytes for 20 min under various temperature and voltage conditions.
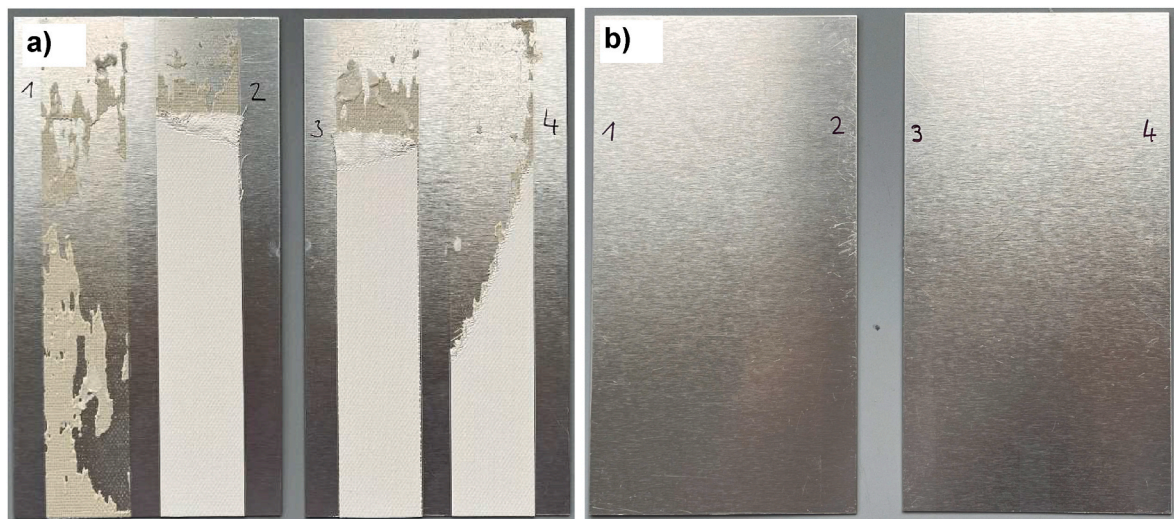

Fig. 4. Samples after tape adhesion test. a) Peel adhesion strength >53 N/25 mm. b) Peel adhesion strength <53 N/25 mm.
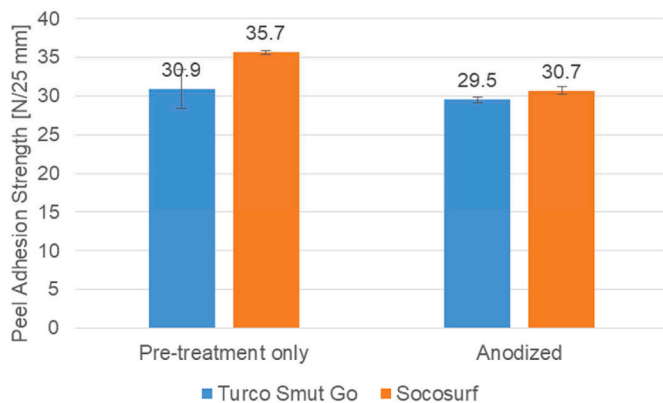

Fig. 5. Average peel adhesion strength results of substrates only pre-treated in acidic pickling solutions and subsequently TSA anodised.

changes in the degree of dissolution of the oxide layer during anodising, since the surface of the samples continues to be relatively flat and smooth regardless of the formation voltage applied.

A general trend towards thicker layers is observed when the anodising temperature is increased. However, in the case of layers which

have undergone major dissolution of the oxide layer during anodising, such as SAA layers formed at 55 °C, and T^+SA films at 55 °C and 20 V, the actual film thickness decreases. In addition, an increase of the electrolyte temperature leads to pore widening by pore wall thinning through oxide dissolution. This dissolution also leads to the creation of a rough ultimate surface as shown in Fig. 8b. However, for films formed in SAA at 55 °C the oxide dissolution is so extensive that the upper part of the porous structure loses its structural integrity and collapses (Fig. 8a).

Finally, the addition of tartaric acid to the SAA electrolyte yields, in general, slightly thinner anodic films if anodised at 37 °C or at 45 °C (Fig. 6). However, at 55 °C TSA anodic layers are thicker than SAA films. Further tartaric acid additions (150 g/l tartaric acid, 40 g/l sulfuric acid, T^+SA), lead again to slightly thinner anodic layers compared to TSA, regardless of the anodising temperature and the formation voltage. Also, the addition of tartaric acid to the sulfuric acid electrolyte leads to a smoother surface of the anodic oxide and to a denser porous structure with thinner pores. This is illustrated in Fig. 9. Furthermore, the micrographs in Fig. 9 show surfaces with curvature changes, related to pits formed during the alkaline etching pre-treatment.

The comparison of the morphology obtained from the two pre-treatment solutions under study (Socosurf and Bonderite C-IC SmutGo) reveals that a porous and rough pickling oxide remains on the surface (Fig. 10a) after acidic pickling in the Socosurf solution. On the contrary, after immersion in Bonderite C-IC SmutGo no porous and
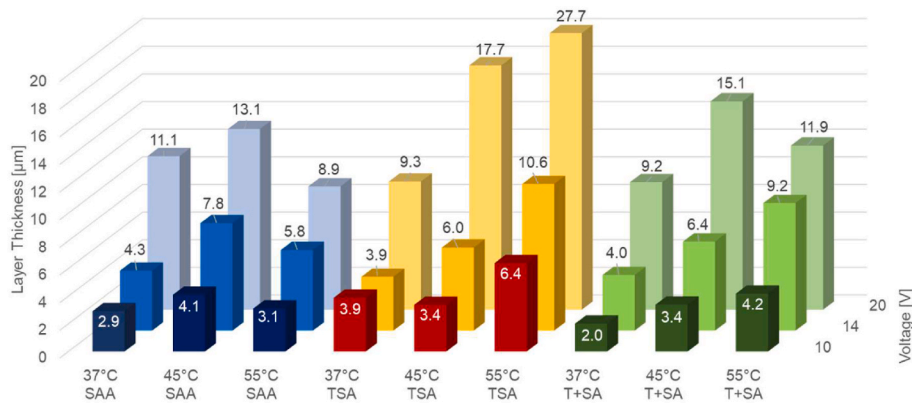
Fig. 6. Average anodic layer thickness formed on substrates pre-treated in Bonderite C-IC SmutGo AERO and anodised in different electrolytes for 20 min under various temperature and voltage conditions measured from FE-SEM cross-section images.
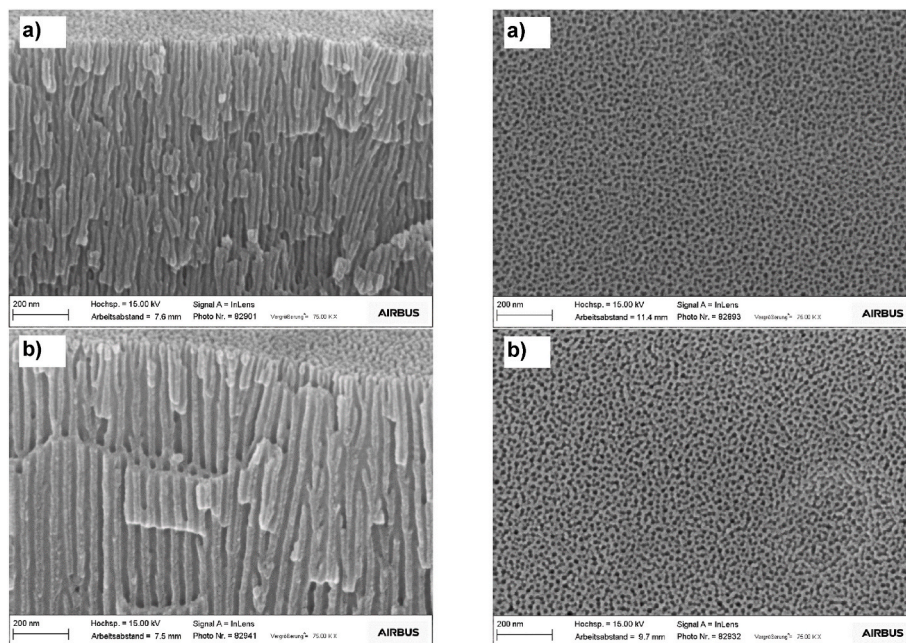

Fig. 7. High resolution FE-SEM cross-section images at the surface of the anodic oxide. Samples were pre-treated with Bonderite C-IC SmutGo NC AERO and anodised in SAA for 20 min at 37 °C and a) 10 V b) 20 V.

rough pickling oxide can be seen. Instead, a rather smooth surface with some cracking and scaling is observed (Fig. 10b). The morphology observed after acidic pickling is partially retained after TSA anodising (at 14 V, 37 °C). This can be seen in Fig. 10c and d. The porous and rough pickling oxide is very thin and it is not visible in the cross sections (not shown).

In addition, anodic layers are slightly thicker in the case of panels pre-treated with Bonderite C-IC SmutGo NC AERO (4.5 μm in average) compared to Socosurf pickled samples (4 μm in average) as measured from the cross-sections (not shown).

4. Discussion

4.1. Effect of anodising parameters on adhesion and morphology

The tape peel adhesion test used in this study shows very little influence of the formation voltage on peel adhesion strength. The formation voltage governs morphological characteristics of the bulk oxide such as the anodic oxide thickness, cell size and pore diameter [13]. Higher voltages lead to thicker oxide films (Fig. 6), bigger cell sizes and

wider pores, and have little effect on the ultimate surface morphology. (Fig. 7). Since the adhesive present on the tape is a pressure sensitive adhesive, which has not been cured and is not in a diluted form (as it would be the case of a primer in aerospace applications), it can be assumed that the adhesive does not fully penetrate into the several μm thick porous structure. As a consequence, the test methodology proposed is, by design, only able to capture the contribution of the oxide's ultimate surface contribution to adhesion. This explains the limited influence of the formation voltage on the peel adhesion strength results. While one could intuitively think that thicker layers and wider pores would have a positive effect on the adhesion properties, the work by Abrahami et al. [5] has shown that the dry-adhesion properties of anodic oxide films are mostly governed by the ultimate surface characteristics, with little to no influence of the bulk oxide morphology. Abrahami et al. [5] show that the dry adhesion peel strength is independent of the oxide thickness. Their work also shows that for oxides formed at lower electrolyte temperatures (20 °C and 35 °C), an increase in dry adhesion strength takes place as the average pore diameter at the surface increases up to a certain threshold (25 nm) [5]. They [5] report no further enhancement of the dry adhesion strength takes place for average pore

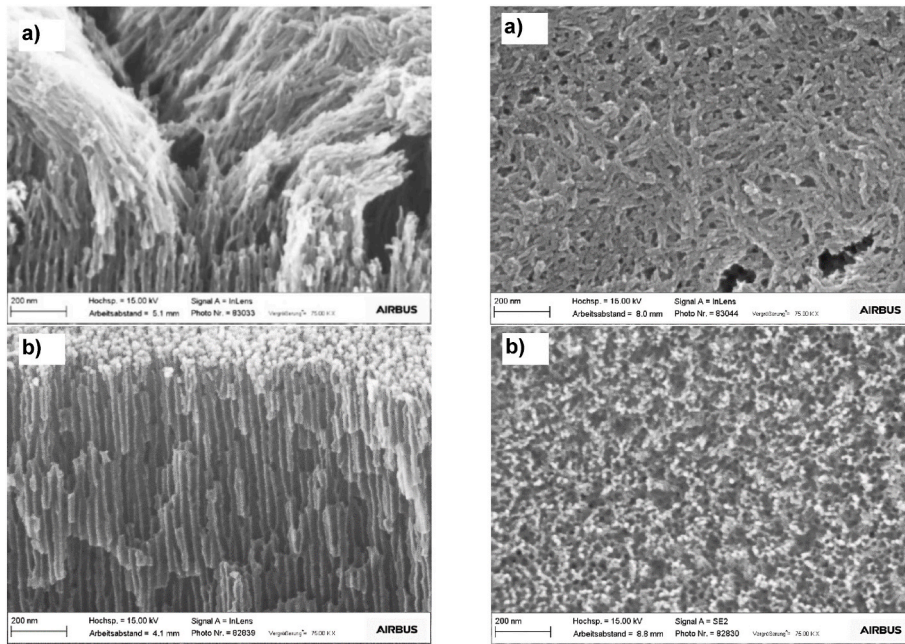**Fig. 8.** FE-SEM images (cross-section) at the surface of the anodic oxide: a) Collapsed anodic oxide layer (SAA 14 V 55 °C) and b) Anodic oxide layer (T + SA 14 V 55 °C). Pre-treatment with acidic pickling in Bonderite C-IC SmutGo NC AERO.
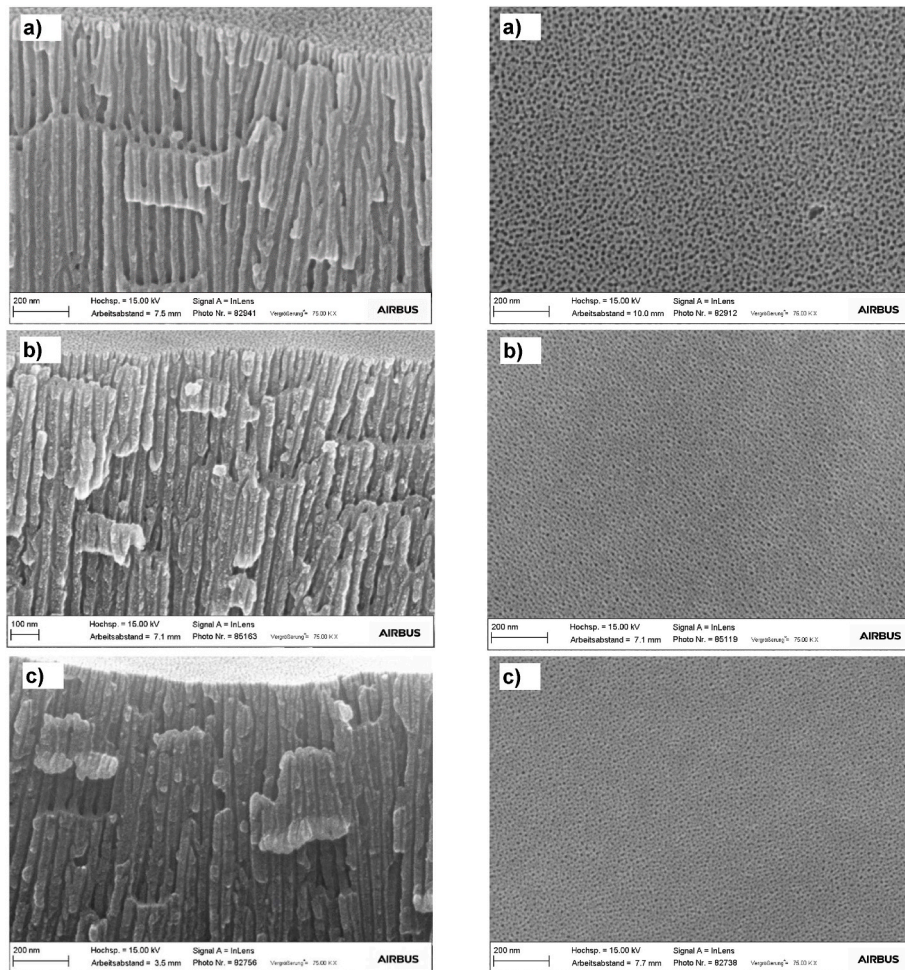

Fig. 9. FE-SEM images (cross sections) of anodic layers formed at 37 °C and 20 V in a) SAA b) TSA and c) T⁺SA (pre-treatment with acidic pickling in Bonderite C-IC SmutGo NC AERO).
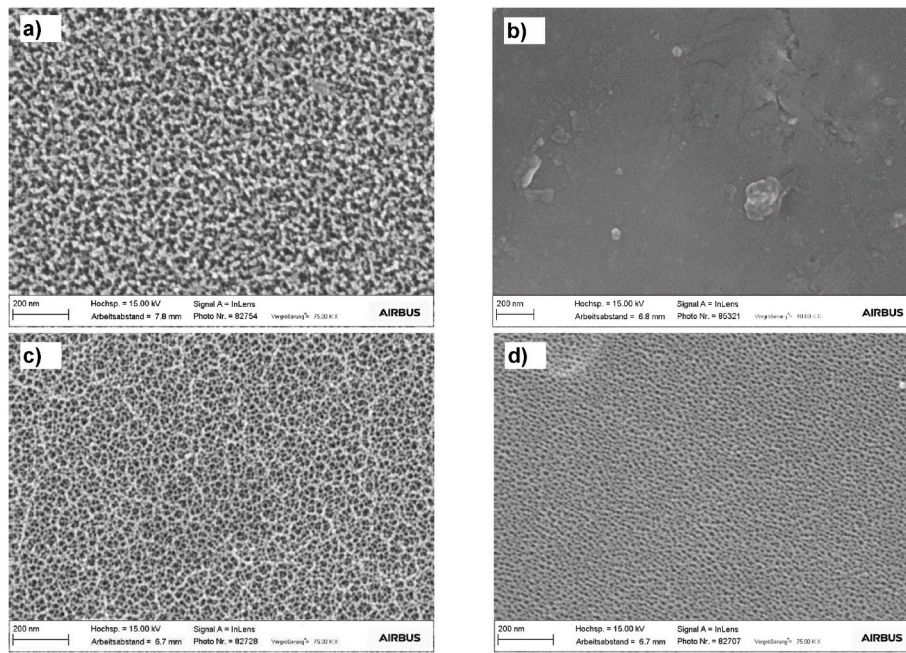
Fig. 10. FE-SEM images (top view) after a) pickling in Socosurf, b) pickling in Bonderite C-IC SmutGo NC AERO, c) pickling in Socosurf and TSA anodising and d) pickling in Bonderite C-IC SmutGo NC AERO and TSA anodising.

diameters larger than 25 nm. Remarkably, the dry peel adhesion strength results from Abrahami et al. [5] for oxides formed at elevated temperatures (50 °C), which show surface morphological features characteristic of extended dissolution processes, are independent of the pore diameter. The morphological features of the bulk oxide become of relevance in the case of wet-adhesion properties [14] and therefore, they are of importance to ensure good performance in humid and aggressive environments. Hence, based on the results by Abrahami et al. [5], it can be stated that the proposed methodology, which disregards the contribution of the bulk oxide and focuses on the ultimate surface morphological features (pore mouth diameter, surface roughness), is valid to characterize dry-adhesion.

While the increase in the average pore diameter driven by the formation voltage has little effect on the tape peel adhesion strength, the widening of pore mouths and the extra roughness at the oxide ultimate surface caused by the electrolyte dissolution at 45 °C for SAA layers, or at 55 C for TSA and T$^+$SA layers lead to a remarkable increase of the peel adhesion strength measured (Fig. 3). Fig. 8b shows and example of the morphological features that correlate with high peel adhesion strengths. However, if the temperature enhanced oxide dissolution becomes too aggressive, the excessive thinning of the pore walls causes the collapse of the porous structure. This is a phenomenon known as chalking. It receives its name from the fact that aluminium substrates acquire a white powdery appearance. FE-SEM characterization of SAA samples anodised at 55 °C shows that chalking has taken place (Fig. 8a). Chalking correlates with a drop of the adhesion peel strength (Fig. 3). Ultimate surface morphological features (dissolution driven roughness, pore-mouth widening and chalking) can be effectively characterized by the proposed tape peel adhesion test method.

The electrolyte temperature also has an effect on the anodic oxide layer thickness. The competition between oxide growth and dissolution rates explains the fact that, even though it is expected that increasing the electrolyte temperature would translate into faster growth rates, at very high temperatures the increase in the oxide dissolution rate offsets the increase in the oxide formation rate and, as previously introduced, can even lead to the collapse of the oxide's porous structure. As a consequence, the final anodic layer is thinner. This is the case for example of anodic films in SAA at 55 °C in comparison to SAA films formed at 45 °C

(see Fig. 6).

It can be stated that the tape peel strength is dominated by the electrolyte temperature. However, the electrolyte aggressiveness and hence its composition determine the temperature at which maximum adhesion performance takes place. The addition of tartaric acid to the electrolyte decreases the aggressiveness of the electrolyte, thereby reducing the dissolution rate of the layer [11].

Consequently, the temperature at which maximum tape peel adhesion strength is measured shifts towards higher temperatures when tartaric acid is added to the sulfuric acid electrolyte.

It has been reported that tartaric-sulfuric acid anodising processes (TSA, T$^+$SA) present lower anodising current densities and hence slower oxide growth rates compared to equivalent SAA anodising processes [11,15]. Consequently, as long as dissolution is not playing an important role, TSA-based anodic layers are thinner than equivalent SAA layers. However, at higher temperatures, the milder tartaric-sulfuric acid electrolytes diminish the rapid oxide dissolution, and hence thicker layers are formed.

As a general remark, the changes in surface curvature depicted in Fig. 7, caused by etching pits, are not regarded as relevant to explain the differences in the peel adhesion force measured. While they could locally have an effect on adhesion, taking into account their presence in all samples regardless of anodising parameters, their random distribution and the large test surfaces involved in the adhesion test, their contribution is considered to be similar for all tested conditions.

Among the limitations of the test methodology studied, it is important to keep in mind that the stability of chemical interactions in an aggressive or moist environment are not taken into account. It is possible that differences among electrolyte chemistries arise in such conditions, as suggested by the research conducted by Abrahami et al. [6,7]. It is also worth mentioning the importance of choosing an adequate tape. If the adhesion strength of the tape is too low, early failure at the tape-adhesive interface will take place. This early failure may conceal differences among adhesion properties of different anodic oxide films.

The ability of this method to characterize differences in dry adhesion properties depending on the anodising conditions used, opens the possibility to investigate intermediate points within or even beyond the

anodising process window considered for this study. This would be of special interesting due to the non-monotonic trend observed in the peel force results (e.g. SAA at 37, 45 and 55 °C).

4.2. Effect of pre-treatment on adhesion and morphology

An important observation is that the proposed tape peel adhesion test is capable of differentiating dry adhesion properties resulting from morphological differences among pre-treatments. A slightly higher peel adhesion strength is measured for substrates pre-treated in the Socosurf acidic pickling solution in comparison to Bonderite C-IC SmutGo NC. A thin residual pickling oxide is present after immersion in the Socosurf solution. This residual pickling oxide increases the surface roughness and therefore enables mechanical interlocking with the tape adhesive. The presence or absence of a residual oxide after pre-treatment depends on the nature of the acidic pickling solution used. The objective of acidic pickling is to remove the smut formed during previous pre-treatment steps (e.g., the alkaline etching step). During acidic pickling two competing reactions take place simultaneously: oxide formation and oxide dissolution. The rate at which these two reactions occur, and the morphology of the formed oxide depend on the chemistry of the pickling solution. As previously described, while a rough pickling oxide can be seen for the samples pre-treated in the Socosurf solution, this is not the case for the Bonderite C-IC SmutGo pickled substrates (see Fig. 10). The absence of a rough and porous pickling oxide after immersion in Bonderite C-IC SmutGo NC is most likely related to the chemistry of the bath as Bonderite C-IC SmutGo NC is a fluoride-containing pickling solution. Fluorides have a strong affinity to aluminium ions. This enhances the oxide dissolution reaction rate [16]. Consequently, fluorides are common additives that enable shorter processes and/or lower bath temperatures. This translates into a more powerful oxide dissolution, which offsets oxide formation. As a result, a smooth and featureless surface is obtained.

Even though a further enhancement of adhesion is expected after a subsequent anodising step, the proposed test methodology yields similar results for substrates that have been only pre-treated and pre-treated and subsequently anodised. The similarity in adhesion results is probably related to the relatively low anodising temperature, and consequently the smooth surface and narrow porous structure characteristic of anodic films formed at 37 °C. As mentioned in the previous section, the stability of chemical interactions in an aggressive or moist environment has not been taken into account in the present study. The different chemistries of the acidic pickling solutions, and consequently of the resulting pickling oxide, could lead to differences in wet-adhesion properties.

Finally, the choice of the pre-treatment solution also influences the thickness of the anodic oxide layer formed in the subsequent anodising step. The thinner anodic oxide films formed on substrates pre-treated in the Socosurf solution (4 μm compared to 4.5 μm for Bonderite C-IC SmutGo pre-treated samples) can be explained by the presence of a residual pickling oxide from the pre-treatment step. The residual oxide could delay the start of the oxide growth. Chemical thinning and field assisted dissolution of the pickling oxide layer must take place first, before oxide growth can resume [17,18]. This phenomenon is known as recovery effect [19].

5. Conclusions

A relatively simple test methodology, based on DIN EN ISO 29862, was proposed to discriminate the adhesion properties of anodic oxides formed using different anodising conditions and pre-treatments. This method could be useful for a fast assessment of anodising parameters to optimise for good adhesion. Furthermore, it could be applied in an industrial environment as a quality control test, to detect if deviations from the nominal manufacturing process have taken place. It would also lead to a reduction of the operator dependency and to an easier quantitative analysis compared to the industrial cross-cut testing standard.

The proposed methodology is able to capture differences of the ultimate-surface morphological features with a good correlation between the anodic oxide morphology and the measured peel adhesion strength. A significant increase of peel adhesion strength is observed for anodic layers with wide pore mouths and a rough oxide surface. This type of anodic oxide morphology forms when oxide dissolution is enhanced. This is the case for high electrolyte temperatures, as long as the integrity of the layer is retained.

Author contributions

Investigation: M.P.M.
Writing—original draft preparation: M.P.M.
Writing—review and editing: S.A., H.T., T.H., M.B.
Supervision: S.A., H.T., T.H., M.B.
All authors have read and agreed to the published version of the manuscript.

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Declaration of competing interest

The authors declare no conflict of interest.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ijadhadh.2022.103156.

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