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Effect of Roughness and Surface Topography on Adhesion of PVB to AA2024-T3 using the Blister Test

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ABSTRACT

Corrosion protection through protective coatings depends on the adhesion of the coating system to the base metal, which is controlled by surface treatments. Measuring adhesion strength is a challenging task. However, the Blister Test (BT) is a quantitative and reproducible technique not exploited in the corrosion field. In this investigation, the effect of roughness and surface topography is studied using this technique. Samples were abraded using SiC paper up to 120, 180, 600 and 1200 in a randomly or aligned manner to obtain different topographies and roughness levels in the range of 0.026 to 1.324 μm. The surfaces were characterized using Optical Profilometry before polyvinyl butyral (PVB) coating was applied. Then the adhesion strength was determined using the BT. The substrates abraded randomly presented lower average roughness than the ones abraded in an aligned manner due to continual cross abrasion of grooves. The adhesion strength results from the BT were reproducible and could rank different mechanical treatments. Roughness degree and surface topography were found to be very important factors for adhesion strength. Adhesion strength was found to increase with roughness for both abrasion methods; however the random samples exhibited the highest adhesion strength at similar roughness values. The groove peaks were found to be stronger barriers than the groove valleys as a result of a higher peeling angle needed for delamination to take place, increasing the energy used for plastic deformation and therefore decreasing the energy available for blister growth. An adhesion strength indicator (AS) was defined based on peeling rate and found to be effective in sensing adhesion strength.

1. Introduction

Coatings on metals are often designed to provide corrosion protection to the underlying metal [1], but a key parameter for coating performance is adhesion [2–4]. The coating can act as a barrier and also as a source of inhibitors to mitigate corrosion. However, even if a coating system contains the most efficient corrosion inhibitor species, limited protection is imparted if the primer is not well adhered to the substrate.

It is important to understand the difference between "fundamental adhesion" and "practical adhesion" [5]. Fundamental adhesion is related to the energy to break the combined bonding of all the interatomic forces that keep two surfaces together divided by area, while practical adhesion is what is obtained from adhesion tests regardless of the failure locus. The practical adhesion is the fundamental adhesion plus any other phenomena taking place during the test that consume energy, such as coating or substrate plastic deformation, stress stored in the coating, and test geometry. However, if coating rupture occurs during the test, the practical adhesion will underestimate fundamental adhesion since the adhesion strength is not reached. Therefore, details of the approach should be considered when selecting an adhesion test to use.

Measuring adhesion is a complex task [6]. Numerous techniques have been developed in the last decades to measure the adhesion strength of coatings to metals [5]. Nonetheless, these techniques are characterized by at least one of the following disadvantages: they are non-quantitative, non-reproducible, performed in dry conditions, or overestimate adhesion as a result of energy consumption by other phenomena, such as plastic deformation [7–9].

ASTM D3359 [10], known as "The Tape Test," comprises two different test methods depending on the test site (field or laboratory). In general, incisions are made into the coating to reach the substrate. The incisions can be performed on samples pre-exposed to water or a corrosive environment or without pre-exposure. After the incisions are made and the debris cleaned, a piece of tape is pressed against the coating. Then the tape is removed quickly at a 180° angle in one motion. Finally, the panel is classified depending on the percent of delaminated area. Even though this technique is widely used in industry, it only provides a fail/no-fail result and is thus extremely qualitative [8].

ASTM D4541 [11] is the most commonly used technique in industry for assessing adhesion because it is easy to perform. In this technique a stud or dolly is adhered to the coating and then pulled off at a specific rate while the force is measured. However, the failure mode in this test is usually characterized by a mixture of adhesive and cohesive failure or cohesive failure only [4,12], making the measurement non-

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representative of the adhesion at the coating/substrate interface. Also, the chemical components of the adhesive used to attach the stud could affect the coating/substrate interface [13]. Finally, the measurements are performed in dry conditions, which do not simulate reality. The presence of water in the coating/substrate interface is known to decrease adhesion, producing what is called "wet adhesion" [14].

As discussed in the last paragraphs, there are issues with the well-known and practical techniques, leaving a large gap between fundamental adhesion and practical adhesion. In this study, the BT is used to measure adhesion strength. The principal advantage of this technique is that it provides a measure of the normalized energy needed to initiate/propagate a crack at the substrate/polymer interface [15]. This is a quantitative measurement that is not given by any other technique. Also, the BT can be performed in the presence or absence of a wetting liquid, there is no system contamination from an adhesive, and it offers the ability to study the effects of a range of parameters [16]. Furthermore, the amount of plastic deformation during the test is minimized because the peeling angle is kept lower than 90° [17]. In addition, the BT simulates the actual stress situation in the coating/substrate interface [18,19], the need for which has been discussed [20]. Finally, the BT has been shown to be reproducible [16].

The BT was first reported in 1961 by Dannenberg [21] and then Williams [22] developed the analysis further using fracture mechanics. This test has been used for adhesion measurement of different interfaces such as silica/polystyrene [16], silica/polymethylmethacrylate, varnish/stainless steel [21], pressure-sensitive tape/plexiglass [23], and pressure-sensitive tape/carbon steel [24]. The test consists of pressurizing the interface between a rigid substrate and a flexible coating through a hole in the substrate to produce a blister in the coating [21]. Blister radius and pressure are recorded with time. It is known that a crack will not propagate until a critical energy release rate is achieved, the adhesion strength G_a. Different equations [22,23,25,26] have been derived from linear elastic fracture mechanics (LEFM), depending on the principal deformation modes that the flexible material, the coating, experiences during the blister test. Using an energy criterion where the energy supplied to the system is used in the delamination process, and taking into account the elastic energy changes in the coating, the following equation was derived [23],

$$G_a = \left[\frac{(Pr)^4}{17.4Et}\right]^{1/3} \quad (1)$$

where P is the blister pressure, r is the blister radius, E is the coating Young's Modulus, and t is the coating thickness. This equation was derived using LEFM since the coating behaves as an elastic membrane when the blister diameter is much larger than the coating thickness.

Surface preparation prior to coating application is critically important for controlling adhesion [27]. There are general types of surface preparation used to improve adhesion, mechanical and chemical treatments [8]. Mechanical treatment involves increasing the roughness of the surface to increase the interlocking of the primer with the substrate, while chemical treatment increases the bonding energy of the coating to the substrate. Increasing the roughness increases the surface area and also facilitates the flow of the coating around the substrate features [27]. Theoretical analyses [28] suggest that an increase in roughness improves adhesion strength not only by increasing surface area, but also by enhancing the processes that induce energy dissipation in the interface during delamination. In addition, an increase in roughness results in an increase in specific surface energy, which is believed to positively affect adhesion of organic polymers to metal by increasing the number of active sites and the effective area for bonding to occur [29]. For example, increasing the roughness of a carbon steel substrate was found to increase the adhesion of chlorinated rubber varnish coating [29]. Other authors [30] showed how the adhesion strength of thermally sprayed coatings on steel increased with roughness produced by sandblasting.

In the present investigation, the BT is used to study the effect of roughness and surface topography on the adhesion strength of polyvinyl butyral (PVB) to AA2024-T3 abraded using SiC paper.

2. Experimental

The BT uses a fluid to pressurize a coating covering a hole in a substrate and thereby create a blister at the substrate/coating interface. During this process, the diameter of the blister and its pressure are recorded until complete delamination of the coating occurs. A schematic of the setup is shown in Fig. 1. This setup is an improved version of the one used by Kappes et al. [24]. A programmable syringe pump (KD Scientific) in a vertical configuration, to facilitate purging of air bubbles from the system, was connected to the sample holder by a stainless steel tube. A T-valve was used between these two parts of the setup to feed the syringe with electrolyte when the BT needed to be conditioned for a new test. The pressure was recorded versus time by a pressure transducer (from Omegadyne). The polycarbonate sample holder had a cubic shape and connections for the syringe pump and the pressure transducer as well as reference and counter electrodes for electrochemical measurements. The largest hole was for attachment of the coated sample, which was tightened down with the help of a plastic coated C-shaped stainless steel plate and four bolts. All tubing and tubing accessories were made of stainless steel.

The setup also had a polycarbonate environmental chamber that can be attached to the top of the sample holder and sealed against the sample holder using an o-ring. The relative humidity of the air flowing through the chamber could be controlled and measured by a sensor in a downstream mini-chamber. Water-saturated air was pumped through the chamber using an aquarium pump and two washing bottles during the BT experiments.

The environmental chamber had a quartz window in the upper face to allow viewing and recording of the blister growth with a Charge-Coupled Device (CCD) camera attached to a face-down stereoscopic microscope (SZX12 OLYMPUS). The blister had a non-planar geometry so that incident light did not reflect back to the microscope, producing a high-contrast black circle in the recorded video image, which facilitated measurement of the blister radius. The syringe pump was controlled by a DasyLab software program. The BT could be operated in constant infusion (CI) rate mode or constant pressure (CP) mode. The infusion/withdrawal rates, target pressure, and other variables can be adjusted in the program. The experiments presented here were performed in CI mode at an infusion rate of 0.050 mL/h, using deionized (DI) water as the pressurizing fluid. This infusion rate was chosen to minimize coating rupture, which occurs more readily at higher infusion rates.

The substrates were AA2024-T3 with dimensions 30 mm × 30 mm × 1.3 mm. Different approaches were attempted [31] for preparation of a through-hole in samples for the BT. The goal was to have a high quality coating spanning a through-hole in the sample. The combination of milling and electrodissolution, described presently, was found to give the best results.

A 3 mm diameter hole was milled in the center of one side of the substrate to a depth of about 0.9 mm (leaving about 0.4 mm in thickness under the hole). The other side of the substrate was then prepared for coating. Some samples were abraded randomly or in an aligned manner using silicon carbide papers grit of 120, 180, 600, or 1200 grit, depending on the scope of the specific experiment. A MultiPrep™ System with DP-Lubricant Blue (Struers) was used for abrasion. The MultiPrep™ System has two rotatory parts, the abrasion wheel and the sample holder arm. The abrasion wheel was set to 100 rpm for all samples. For the randomly abraded samples, the sample holder arm was configured to continuously rotate and oscillate radially across the wheel for 5 min. For aligned samples, the process consisted of random abrasion for 1 min followed by 4 min with the arm kept in a fixed position for the final grit. After abrasion, samples were ultrasonically cleaned in acetone for 1 min and then stored in a desiccator.