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Torsion delamination test, a new method to quantify the adhesion of coating: Application to car coatings

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A B S T R A C T

A torsion delamination test was developed to characterize the interfacial cohesion of multilayer organic coatings. It consists in applying an increasing torque on a hexagonal plot directly glued on the coating until delamination occurs. The test is quantitative and allows the calculus of the shear stress that generates delamination. This torsion test was successfully used to test the adhesion of multilayer car coatings. The delaminating interfaces were directly observed via optical and electron microscopy. We have shown that the critical delaminating shear stress and the delaminating interface are dependent of both the primer and of the curing temperatures respectively. Furthermore, an interpretation of the delamination process is proposed.

Keywords:

Adhesion
Delamination
Coatings
Measurement

1. Introduction

In industry, coatings are used because they offer the advantage to drastically change the properties of surface while keeping the bulk properties of the substrate [1]. It provides functions of protecting, functionalizing and/or decorating the substrate surface. To warranty all these functions, the coating must adhere to its substrate for the expected use lifetime. In the case of automotive, the coating should maintain an attractive and shining appearance for at least ten years. Furthermore, the coating should resist and protect the metal sheet from numerous mechanical and physicochemical aggressions [2] such as stones shipping, marring, scratches, small impacts, UV, bird drops, acid rains, etc. Typical car coating is a complex assembly of five different layers deposited and cured onto relatively rough galvanized steel sheets (Fig. 1). It generally consists in (1) a Primer, generally a phosphate layer (thickness: $3 \pm 2 \mu\text{m}$), that allows adhesion of the coating and some anti-corrosion properties, (2) a Cataphoresis coat ($18 \pm 5 \mu\text{m}$) also called dip coat, that confers corrosion protection, (3) a Filler coat ($35 \pm 5 \mu\text{m}$) that flattens out the surface and plays a role as little shock absorber and of base color, (4) a Base coat ($15 \pm 5 \mu\text{m}$) that gives color and

sequin effects and (5) a Clear coat ($40 \pm 5 \mu\text{m}$) that gives shine and resistance to chemical aggression. This complex system has five different interfaces that should have a very good adherence to prevent any dramatic delamination whatever the kind of aggression. The different layers are mainly composed of thermoset resins. During the painting process, there are reticulated during three different steps of curing: After the deposition of the dip, filler, and clear coat. The two first curing should bring out a moderate reticulation of the coat to not consume all the reactive molecules that are required to create strong chemical bonding with the next layer. The last curing is realized after the successive deposition of the base and the clear-coat in order to cure these coats together. It results in a strong base-clear coat interface that never delaminates because of numerous strong chemical bonding generated at this interface. Other interfaces, especially the Metal/Primer/Cataphoresis (M/P/C), the Cataphoresis/Filler (C/F) and the Filler-Base (F/B) could be concerned by delamination. This is particularly the case for the Metal/Primer/Cataphoresis interface which is a double interface between a metal and a mineral layer and between a mineral layer and an organic layer. Clearly, car manufacturers shall prevent any delamination of the car coating that generate cost consuming car call-backs and depreciate its reputation. Paint manufacturers continuously offer new chemical formulations for the different layers. Thus, the car manufacturer needs to make evolve its painting process to conform to technical progress and drastic ecological

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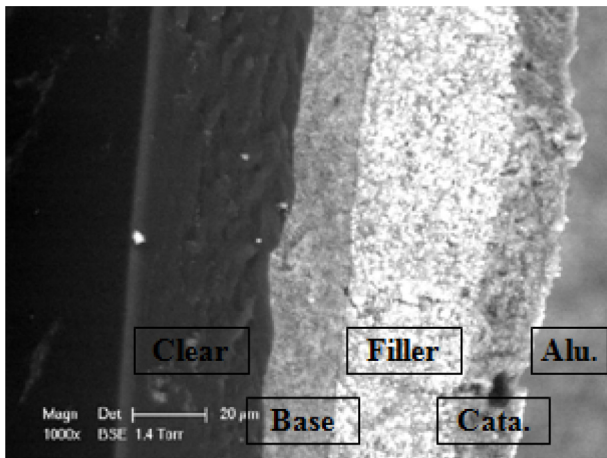


Fig. 1. ESEM image of the trans-section of the multilayer car coating. From right to left, aluminum substrate, Cathaphoresis coat ($\approx 19 \mu\text{m}$), Filler coat ($\approx 39 \mu\text{m}$), Base coat ($\approx 17 \mu\text{m}$), and Clear coat ($\approx 42 \mu\text{m}$).

legislation, to reduce the complexity and the financial cost of the painting process. Nevertheless, the change of one layer leads to the change of at least one interface that should conserve or increase its adhesive quality. Thus, for any change in the chemical composition of one layer or in the parameter of painting process, the risk of delamination should be considered, analyzed and estimated. For example, the replacement of the phosphate primer which present a bad environmental impact (high consumption of water, generation of mud with heavy metals as nickel, manganese, etc.) by a new generation of free phosphate primers, called green primers, Tectalis[®] from Henkel [3] or Oxsilan[®] from Chemetal [4–7], shall be respond to all car specifications. These new treatments known to have long-term corrosion protection and significantly reduces costs and environmental impact.

If adhesion is the most important property of coating, its measurement is a difficult task in surface engineering due to the huge number of parameters implicated in this phenomena (roughness, waviness, residual stress, etc. [8]) and the specific conditions of the different delamination tests (plastic deformation [9], sample rigidity [10]). Thus, simple, quick and reliable tests to evaluate coating adhesion are of considerable usefulness in the engineering field. ‘Basic adhesion’ could be defined by the sum of all the chemical and physical interactions at the coating/substrate interface that take parts in the work necessary to separate coating and substrate along the interface. Nevertheless, the material properties of the substrate, the preparation of surfaces, the chemical and physical interactions at the interface, the residual stresses, etc., have drastic effects on coatings adhesion. The notion of ‘practical adhesion’, based on practical tests to quantify the quality of coating, has been developed to distinguish from the ‘basic adhesion’. The ‘practical adhesion’ takes into account the operating conditions of the test, i.e. the mechanical properties of the materials and the interface, the defects of the interface, the mechanical stress and strain and their direction applied to the sample during the test. Obviously, the ‘practical adhesion’ is more interesting for surface engineering as it gives a good prediction of the adhesion quality of the thin coatings in real cases. Therefore, numerous mechanical methods [9,11] including normalized tests have been developed to measure adhesion between substrate and coating like scratch [12–15], bending, chipping [14,15], cavitations, tape [16], pull-off [10], scrape [17] and cupping test [18,19] also known as Erichsen test.

In the car coatings science, the quality of the adhesion is estimated through normalized tests presented above that simulate physical aggression of the coating. The multi-impact stone chipping test [14,15] consists in a bombardment of small angular cast iron

particles (4–5 mm in diameter) with a normalized angle impact. It is design to reproduce the impact of gravel on road. The tape test [16] consists in applying and removing a tape over cross hatch cuts made in the coating. It allows determining the quality of adhesion after severe damage of the coating. The cupping test [18,19], consists in stamping the metal sheet by a steel ball (radius = 1 inch) at a fixed velocity (12 mm/min) and to measure the critical punch depth that generate the first change of aspect of the coating (crack or delamination). It gives the critical adhesion quality of the coating needed to recover the metal sheet by car body repairers without delamination. Sometimes, it happens that these tests provide contradictory results for same sample: good results could be found for one test, whereas poor results are obtained for another one. The reason is that delamination is governed by several parameters as the mechanical stress and strain applied on the interfaces, the strain rates, the coating and substrate properties (thickness, mechanical properties), their elastic and plastic deformations. Thus, these tests are still difficult to interpret. It seems impossible to get quantitative data which characterizes the adhesion between the different layers by these methods. For example, in the tape test, the coating is first scratched. These scratches could initiate delamination at the different interfaces by brittle fracture that will propagate during the removing of the tape. As the opposite, it could have severe plastic deformation of the substrate and of the coating during cupping test, before delamination occurs. Thus, the test measures a combination of adhesion between layer and of cohesion inside each layer. Definitely, classical delamination tests do not measure a magnitude which characterizes delamination resistance, by means of delamination stress or energy, but a result, sometimes quantitative, which characterizes the delamination resistance in specific conditions linked to the test, i.e. mixed of tensile/shear stress, with or without plastic deformation, mixed delamination mode.

Consequently, it is thus necessary to develop a test that could measure a magnitude and which could be related to an adhesive property in simple experimental condition without ambiguities on the mechanical stress, and/or the delamination mode. Pull-off test could be considered as respecting these conditions but in the case of coating on thin plates, the increasing pull-off force leads to an increasing bulge of the substrate. If the stress is now applied by a torsion force, there is no more bulge of the substrate and the stress can be considered as a pure shear stress (Fig. 2a). The maximum shear stress is located at the periphery of the plot, and delamination is initiated at the periphery and propagated to the center of the plot leading to a pure delamination mode III (Fig. 2b). In experimental case, delamination would be initiated at a given default interphase near the periphery and propagates toward the center of the plot in a mixed II–III delamination behavior (Fig. 2c). According to these remarks, we have developed and characterized a new test, called torsion delamination test, to measure coating adhesion. It consists in measuring the shear torsion stress which initiates coating delamination. The shear stress is applied by a dial torque wrench via hexagonal plots glued on the coating. The test was employed to measure the adhesion of different car coating with different substrates, different primers, and nine sets of curing temperatures. Furthermore, observation of the delaminating interface and values of the shear stress which generate delamination were used to propose delamination mechanisms.

2. Materials and methods

2.1. Samples preparation

All samples were prepared in a paint shop line to get coating as close as those obtained in the industrial process. Two substrates were used: back hardening aluminum alloy (6016 XDR) and gal-

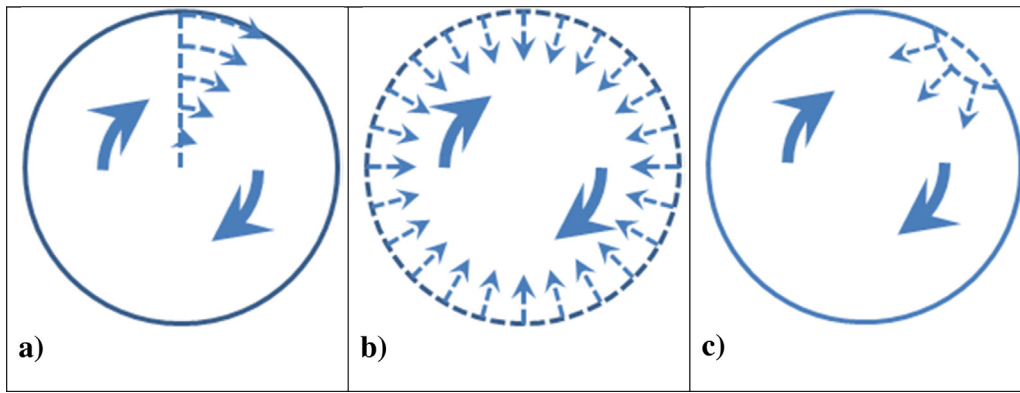


Fig. 2. Shear stress and delamination in torsion delamination test, circles represent the glued area between the plot and the coating where stress is applied. (a) In mode II, the shear stress (solid arrows) is parallel to the propagation of delamination (dashed arrows). (b) In mode III, delamination propagates perpendicularly to the stress. (c) In the torsion shear test, delamination should initiate at the periphery where shear stress is maximum. Thus, the delamination process is a mix of mode II and mode III.

vanized steel (ES G10/10) which are commonly employed in car industry. The thickness of the two sheets is 0.85 and 0.77 mm respectively. The sheets were degreased by an alkaline treatment. For each substrate, three sets of sample were prepared: The first set was treated by a classical phosphate primer; the second set by the Oxsilan® primer (Chemetal) whereas the third set did not receive any primer treatment. Cataphoresis, filler, base coat and clear coat were deposited on the sample following the parameters of a classic industrial process. Curing steps were realized after the deposition of the cataphoresis, filler and clear coat as in the industrial process. The temperature of the curing was maintained constant for the clear coat (150 °C) but were fixed to three different values for the cataphoresis ($T_1 = 130, 180, 250$ °C) and the filler ($T_2 = 120, 145, 200$ °C) leading to nine set of curing temperature ($T_1 - T_2$). All curing times were fixed to 30 mn according to paint purchaser specifications. Thus, 54 samples (two substrates, three primer treatments and nine set of curing temperature) were prepared and tested to seek the influence of the substrate, the primer and the curing temperature on the behavior of car coating.

2.2. Torsion delamination experiments

The torsion delamination test consists in applying directly a shear strain to the coating by the intermediate of a plot. The plots are in aluminum and have a hexagonal shape (17 mm large, 15 mm high) in order to be twisted by a dial torque wrench. They are directly glued on the coating surface. In a first step, the coating surface was dipped in ultrasonic bath of pure ethanol for two minutes and then dries. The surfaces of the coating and of the plot were

then lightly abraded with an abrasive paper (grade 1000) to promote adhesion of the glue and prevent any delamination at the coating/glue/plot interface. The surface was then again ultrasonic rinsed with ethanol for two minutes and dry. A small amount of glue (Araldite 2011) was manually deposited on the plot, and it was then manually pressed on the coating. The sample was rested for one night and cured four hours in an oven at a temperature of 40 °C. This temperature is too low to modify their reticulation or cross linking. The sample was then fixed on a sample holder (Fig. 3a) and an increasing torque was manually applied on the plot with the help of dial torque wrench (RS Components Ltd., Model 815-521) (Fig. 3b) until the plot was brutally snatched (Fig. 3c). The critical torque which leads to the delamination of the coating is measured by the dial torque wrench (Fig. 3d). The reproducibility of the critical torque generating delamination during the experiment was measured. For eighteen experiments conducted on the same sample (Steel substrate, phosphate primer, $T_1 = 180$ °C and $T_2 = 145$ °C) a standard deviation of 4% was observed on the value of the critical torque This standard deviation is similar to those observed for the most reproducible pull-off method [10].

For the 54 samples, three experiments were conducted and the maximum value of the shear delamination test τ_{Max} was recorded. This choice is justified by the presence of a few abnormal low values of the critical shear stress encountered for the sample without primer. This sample presents an erratic behavior which affects the meaning of the average value for a given set of temperatures and substrate. Finally, the delaminating interfaces were observed using optical and electron microscopy (Philips XL30 ESEM-FEG) to conclude about the delaminating process.

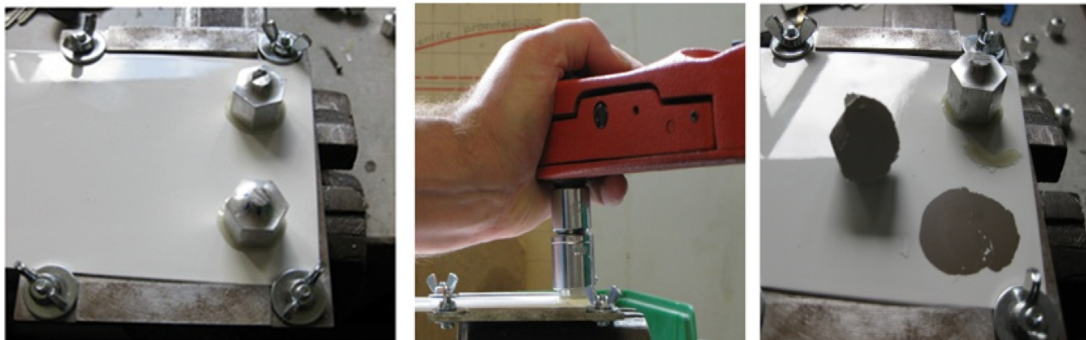


Fig. 3. Optical images of the conduct of the experiments. The hexagonal plots are glued on the sample and the sample is fixed to a sample holder (left). An increasing torque is manually applied on the plot with the help of dial torque wrench (center) until the plot is snatched (right).

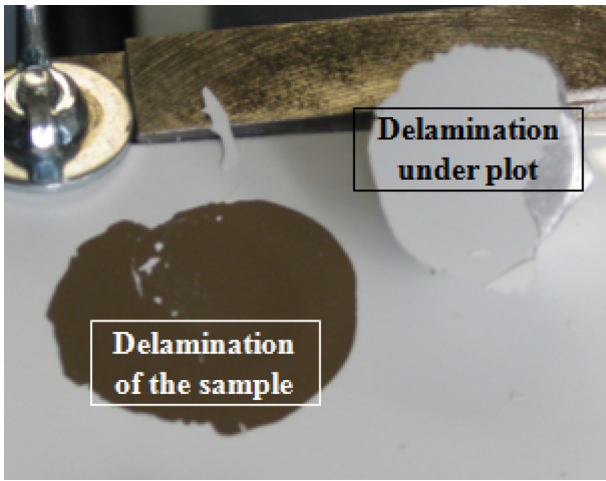


Fig. 4. Sample and hexagonal plot after delamination. A brittle delamination at the Cataphoresis/Filler interface is easily identified (aluminum substrate, phosphate primer, $T_1 = 250^\circ\text{C}$, $T_2 = 120^\circ\text{C}$).

The advantages of the present test are cheap, quick, reproducible and easy to implement. It can be realized on-site or in laboratory whatever the substrate or the coating. It leads to a quantitative test which measures the shear stress that initiates the delamination of the layer. The test could be easily automatized to increase the reproducibility of the operating conditions especially the normal load and the velocity of the increasing torque both applied on the plot. Indeed, if the test is realized manually, these two factors could slightly depend of the operator and could have an influence on results even if a good reproducibility has been observed with manual application of the torque.

To obtain the most reliable adhesion test, it is necessary to minimize plastic deformation of the coating/substrate system [10]. In torsion delamination test, a high strain can be applied on coatings without important plastic deformation of the substrate or the coating that occurs in some other tests. Indeed, in cupping test, a huge plastic stress on the metal sheet and coating could initiate cracks or fractures which will propagate through the coating and modify the mechanism of delamination. Therefore, the test is both influenced by the cohesion of the layer and the adhesion at the interfaces. However, for pull-off test, the pull-off pressure generates a bulge of the substrate. Consequently, the pull-off pressure depends on the rigidity of the substrate, i.e. its thickness, especially in the case of thin substrate, like metal sheet [10]. Indeed, the pull-off strength is usually lower for thinner substrate.

2.3. Calculus of the delamination shear stress

The shear stress τ which generates coating delamination is estimated from the maximum torque applied during the test using the following approximation. The shear stress on the coating is considered equal to the shear stress that occurs in the hexagonal plot due to the torque T applied on it. The stress applied on the plot is equal to:

$$\tau = \frac{T \cdot e}{I_0} \quad (1)$$

where e is the distance from the center of the plot and I_0 its quadratic moment of inertia. The plot presents a manufactured surface of circular shape (8 mm in radius). The quadratic moment of a circular shape is equal to:

$$I_0 = \frac{\pi \cdot R^4}{2} \quad (2)$$

where R is the radius. The torque T applied on the plot generates shear stress τ which is maximum at the periphery of the plot ($e = R$) [20], thus:

$$\tau_{\text{Max}} = \frac{2 \cdot T \cdot e}{\pi \cdot R^4} = \frac{2 \cdot T}{\pi \cdot R^3} \quad (3)$$

3. Results and discussion

3.1. Delaminating shear stress

The torque generating delamination allows computing the maximum shear stress applied when delamination initiates. The maximum values of the delaminating shear stress τ_{Max} are presented in Tables 1–3 for the different primers. The average value and the standard deviation for the six sets of samples (2 substrates by 3 primers) for a given set of curing temperatures (T_1 – T_2) varies from 66 ± 17 to 74 ± 9 MPa. ANalysis Of VAriance test (ANOVA) shows no significant influence ($p > 0.05$) of the set of curing temperatures on the value of the delaminating shear stress. Similarly, the average value of τ_{Max} for aluminum alloy and steel substrates are 69 ± 14 and 70 ± 12 MPa respectively with no significant difference between the two substrates according to ANOVA test ($p > 0.05$). At the opposite, a statistical significant difference for the three primers was observed ($p < 0.05$). An average value with a standard deviation of τ_{Max} that vary from 57 ± 11 for no primer, 71 ± 6 for Oxsilan® and 81 ± 3 MPa for phosphate primer was observed. It appears clearly that only the nature of the primer has a statistical significant influence on the delaminating shear stress for the substrates and the sets of curing temperatures tested. Phosphate is the most efficient primer with a significant difference with Oxilan®.

3.2. Analysis of delaminating interfaces

Optical microscopy has been used to observe the delaminating surface. Due to the different colors of the different coating layers (brown for cataphoresis, gray for filler and white for base coat) the delaminating interface could be easily determined (Fig. 4). The visible delaminating interface is generally not identical for the entire delaminating surface but presents a clear predominance. The main visible surface is called principal delaminating interface. Whatever, the substrate or the primer, the delaminating interface depends only of the set of the curing temperature. Table 4 presents the principal delaminating interfaces in function of temperature. Three different delaminating interfaces could be observed corresponding to three different scenarios: (1) If the cataphoresis dip coat curing temperature T_1 is equal to 130°C and the filler curing temperature T_2 is inferior or equal to 145°C , the delaminating interface is the Metal/Primer/Cataphoresis interface. The delaminating interface shows crazing behavior indicating a ductile deformation of the cataphoresis. We can conclude that a temperature superior to 145°C is needed to reticulate correctly the cataphoresis coat. Above this temperature, it is too ductile to obtain a good adhesion (Scenario 1). (2) For sets of curing temperature with $T_1 > 180^\circ\text{C}$ and $T_2 < 200^\circ\text{C}$, the delaminating interface is the Cataphoresis/Filler interface. For these temperatures, the first curing consumes too many reticulating agents to obtain a good adhesion between cataphoresis and filler layer (Scenario 2). (3) For filler curing temperature T_2 superior to 200°C , the delaminating interface is the Filler/Base coat. It suggests that the curing at 200°C consume too many reticulating functions and the cured surface is not enough reactive to get enough covalent bonds with the base coat deposited on it (Scenario 3). As expected, the Base coat/Clear coat interface never delaminates because there are cured together and covalent bonds are always formed. Between these three delaminating interfaces the most acceptable one is the Filler/Base coat interface as it main-

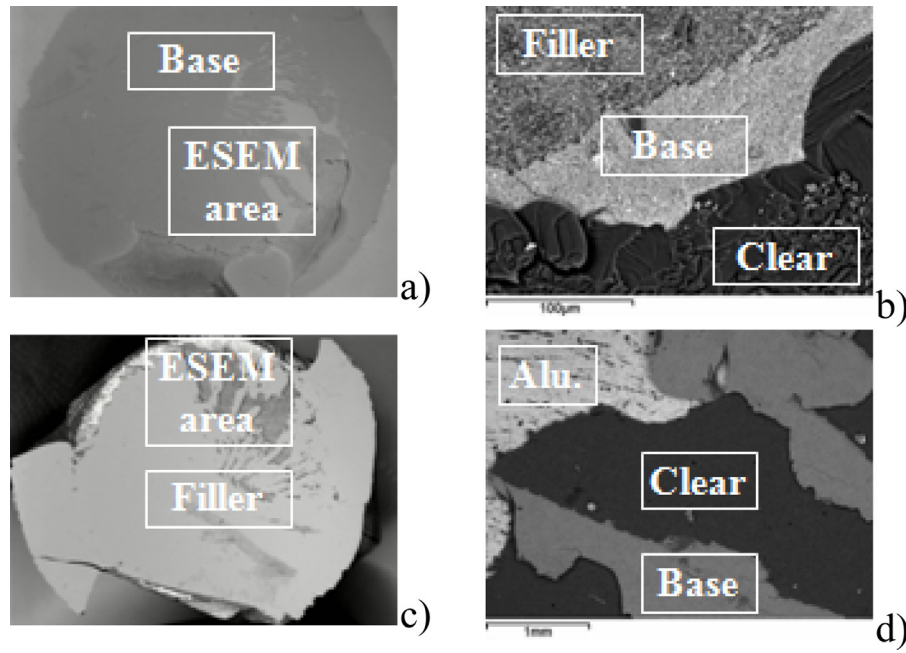


Fig. 5. Optical (a, c) and ESEM (b, d) images of the delaminating surface of plot (c, d) and sample (a, b) (steel substrate, phosphate primer, $T_1 = 180^\circ\text{C}$, $T_2 = 200^\circ\text{C}$). Delamination has occurred at the Filler/Base interface in brittle fashion. ESEM images show that delamination migrates to different interfaces.

Table 1
Delaminating shear stress (MPa) for phosphate primer for the aluminum and steel substrates as a function of the curing temperatures.

		T_2 : Filler curing temperature ($^\circ\text{C}$)					
		120		145		200	
		Alu.	Steel	Alu.	Steel	Alu.	Steel
T_1 : Cataphoresis curing temperature ($^\circ\text{C}$)	130	80	76	80	75	81	83
	180	82	76	83	82	82	85
	250	80	85	86	83	83	81

Table 2
Delaminating shear stress (MPa) for Oxilan primer for the aluminum and steel substrates as a function of the curing temperatures.

		T_2 : Filler curing temperature ($^\circ\text{C}$)					
		120		145		200	
		Alu.	Steel	Alu.	Steel	Alu.	Steel
T_1 : Cataphoresis curing temperature ($^\circ\text{C}$)	130	68	70	65	71	68	73
	180	73	81	72	73	58	80
	250	72	61	73	72	77	65

Table 3
Delaminating shear stress (MPa) without primer for the aluminum and steel substrates as a function of the curing temperatures.

		T_2 : Filler curing temperature ($^\circ\text{C}$)					
		120		145		200	
		Alu.	Steel	Alu.	Steel	Alu.	Steel
T_1 : Cataphoresis curing temperature ($^\circ\text{C}$)	130	70	42	32	75	57	68
	180	52	50	65	50	60	58
	250	62	53	65	65	36	58

Table 4
Delaminating interface and delamination behavior (into brackets) as a function of the curing temperatures. The delaminating interface does not depend of the substrate and of the primer.

		T_2 : Filler curing temperature ($^\circ\text{C}$)					
		120		145		200	
		Alu.	Steel	Alu.	Steel	Alu.	Steel
T_1 : Cataphoresis curing temperature ($^\circ\text{C}$)	130	Metal/Cataphoresis (Ductile)	Metal/Cataphoresis (Ductile)	Metal/Cataphoresis (Ductile)	Filler/Base-coat (Ductile)	Filler/Base-coat (Ductile)	Filler/Base-coat (Ductile)
	180	Cataphoresis/Filler (Brittle)	Cataphoresis/Filler (Brittle)	Cataphoresis/Filler (Ductile)	Filler/Base-coat (Ductile)	Filler/Base-coat (Ductile)	Filler/Base-coat (Ductile)
	250	Cataphoresis/Filler (Brittle)	Cataphoresis/Filler (Brittle)	Cataphoresis/Filler (Brittle)	Filler/Base-coat (Brittle)	Filler/Base-coat (Brittle)	Filler/Base-coat (Brittle)

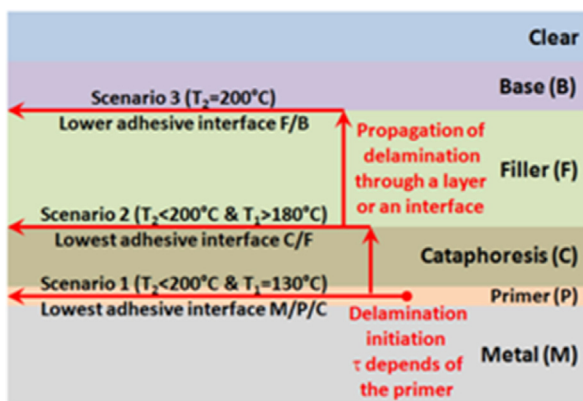


Fig. 6. Different scenarios of the delamination process according to the curing temperatures. Delamination is initiated at the Metal/Primer/Cataphoresis interface. Competition between adhesion at the different interfaces leads to different scenarios.

tains a maximum of coatings especially the anticorrosive layers. Nevertheless, the fact that the delamination shear stress depends mainly of the primer and the delaminating interface depends of the curing temperatures is somewhat contradictory. One can expect that delamination appears for a given stress at a given interface. However delamination appears for a given stress according to a given primer and leads to a given delaminating interface for a given set of curing temperatures. It suggests the delamination process is both governed by the primer and the delaminating interfaces. A closer observation of the delaminating surface by Environmental Scanning Electron Microscope (ESEM) shows abrupt steps from an interface to another one (Fig. 5). Thus it is clear that delamination is initiated at an interface and can migrate and propagate to another less adhesive interface thanks to propagation through a low cohesive layer. Then, the principle delaminating interface can be considered as the less adhesive and not as the interface where delamination is initiated.

The fact the nature of the primer has the main influence of the delaminating shear stress suggests that delamination is initiated at the metal/primer/filler interface. Without any primer, the critical shear stress is equal to 57 ± 11 MPa showing that the process is erratic probably because it is governed by surface heterogeneity as roughness, residual stress, surface defects at the Metal/Primer/Cataphoresis interface. The use of Oxilan® allows an increase of the delaminating shear stress and a decrease of the standard deviation (71 ± 6 MPa, +25% compares without primer) showing that the adhesion becomes more homogeneous. Nevertheless, the adherence performance of Oxilan® is lower and less homogeneous than the classical phosphate treatment (81 ± 3 MPa, +43%). In the case of a set of low curing temperatures ($T_1 = 130^\circ\text{C}$, $T_2 \leq 145^\circ\text{C}$), the delamination initiated at the Metal/Primer/Cataphoresis interface propagates at this interface because the cohesion of the cataphoresis layer is too high or more probably because the adhesion of the other interfaces are better than the Metal/Primer/Cataphoresis interface (Scenario 1). For higher cataphoresis curing temperature ($T_1 > 130^\circ\text{C}$, $T_2 \leq 145^\circ\text{C}$), the cohesion of the cataphoresis layer becomes lower than the Metal/Primer/Cataphoresis interface adhesion, that allows delamination to migrate to the lower adhesive interface that is the Cataphoresis/Filler interface (Scenario 2). Finally, for the highest filler curing temperature ($T_2 > 200^\circ\text{C}$), the lowest adhesive interface is the Filler/Base interface, involving the migration of the delamination through the different layers till this interface. The different scenarios are summarized in Fig. 6.

4. Conclusion

In conclusion, we have developed a simple torsion delaminating test that measures the shear stress generating delamination of multilayers coating. The test is cheap, quick, reproducible and easy to implement and could be used for a large categories of coatings. The test could be easily automatized from classical torsion test to increase the reproducibility of the operating conditions. The shear delamination test has been successfully used to characterize the adherence of car coatings using different substrates, primers and curing temperatures. The test coupled with optical and ESEM observations allowed to evidence that delamination is initiated at the metal/primer/filler interface and migrates to the less adhesive interface that depends of the curing temperatures.

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