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Adherence of coatings: determination - problems - examples

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1 Abstract

There exist more and more multi-layer coatings worked out to meet multipurpose needs. The reliability of each interface between the layers must be studied. So that the coatings can fulfil their roles (corrosion, esthetics, wear, optics...). The optimization of adherence is thus a crucial stake for the industry of the surface treatments. However, the experimental determination of the layer's adhesion is still badly controlled. Indeed, many questions remain. First, all the authors do not grant the same direction under the terms "adhesion" and "adherence". Moreover, a multitude of experimental tests make their choice difficult. Some are qualitative, other are quantitative ones... Moreover, each test comprises experimental parameters which can influence the results. It seems difficult to compare the results for the same coating. Finally, the results of adherence are significant only if the interface between two layers or a layer and the substrate are requested. Consequently, a fractographic and physico-chemical study postmortem prove to be necessary in many cases to check the localization of the rupture. The presented work concerns the study of the adherence of "thick" electrolytic coatings of Zn-Ni and NiP on aluminium and steel substrates. These coatings are developed to replace coatings implementing carcinogenic baths containing of hexavalent chromium. Those seem moreover representative of the electrolytic coatings in general. Because their thicknesses, their mechanical properties and their adherence vary in a broad range. The influential parameters of the three tests used (scratch test, bending test and tensile test) are presented. The limits and the potentialities of each test will be highlighted.

2 Introduction

There is a multitude of coatings worked out by different techniques for different functions. To play a role, the coatings must adhere to their substrate. First, it has already been pointed out by earlier writers that the terms adherence and adhesion for coating substrate systems have many semantic difficulties. Mittal [1] deals with the term adhesion by dividing it into three categories: basic or fundamental adhesion, thermodynamic or reversible adhesion and practical adhesion. Basic or fundamental adhesion was "related to the nature and strength of the binding forces between two materials in contact with each other". Thermodynamic or reversible adhesion was defined in terms of the reversible work of adhesion. Experimental or practical adhesion was the maximum force per unit area required to separate a coating from its substrate with any method. The relationship between the practical adhesion and the fundamental adhesion is expressed as follows:

$$\text{Practical adhesion} = \text{function}(\text{fundamental adhesion}, \text{others factors}) \text{ Equation 1}$$

Others factors can be: residual stress in the coating, mechanical properties of the coating or the substrate, work consumed by plastic deformation, mode of failure, mode and rate of applying the force to detach the film depending on the choice of the test... Furthermore, there is a multitude of adherence tests: more or less simple or sophisticated, more or less qualitative or quantitative. More than 355 different tests have been compiled by Mittal [1]. The choice of a test must be in relationship with its application. It must be simplest to implement while being pertinent. It is necessary to know and control all the parameters of the tests. Finally, a very important point when the adherence of coating is studied is the localization of the rupture. Is it located in the coating, in the substrate, at the interface, in an interphase? We try to answer these various problems. Two types of coatings (ZnNi on aluminium EN2024T3 alloy and NiP on steel 36NiCrMo16) are investigated. These coatings were studied in order to replace surface treatments of the chromatization or hard chromium plating free from hexavalent chromium which is carcinogenic [2][3].

3 Experimental Procedure

3.1 Adherence tests

3.1.1 Scratch test

This test consists in striping the surface coated using a indenter (figure 1). A constant or progressive load is exerted on the indenter in translation. The adherence of the deposit is defined by the critical load (L_c : critical load). It is the load for which the deposit is separated from the substrate [4]. We will see that its determination can vary according to the means of characterization implemented. Various studies showed that this test could be used to compare the adherence of several coatings on the same substrate or the adherence of a coating on several

substrates [5][6]. The critical load is very largely influenced by extrinsic test parameters and by intrinsic test parameters which it is necessary to know in order to control them. For example, all the authors confirm the increase in the critical load when the hardness of the substrate [4][7] or the thickness of the coating [1][8][9][10] grows. It decreases on the other hand when the roughness of the deposits or substrates [11], the coefficient of friction [12][13] or the coating's internal stresses [7] increase. Furthermore, L_c decreases with the loading rate, the translation speed and increases when the indenter ray increases [5] [14].

The scratch tests were performed with a micro-hardness tester instrumented with a translation table. A Vickers stylus is drawn across the sample at different loads between 15-500g. Several testing conditions parameters are fixed to not influence the results. Scratch speed is equal to 10 μ m/s; the indenter doesn't change; all the samples are cleaned with alcohol before test.

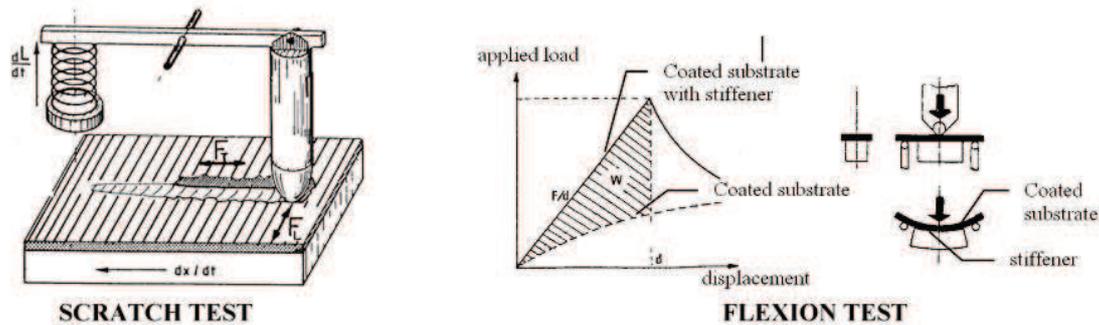


Figure 1: principle of the scratch test and the three-point flexion test.

3.1.2 Three point bending test

This test was in particular developed for the characterization of the adherence of organic coatings on titanium by Roche [15] (figure 1). It provides the informations on the rigidity of the system (slope of the curve forces/displacement in the linear part), on the force and maximum displacement before rupture, on the type of rupture (abrupt or by jump) and finally on the localization and the propagation of the rupture. Gaillard and al. [16] are studied the influence of the various test parameters. The rigidity of the beam increases with the thickness of the substrate, the volume of adhesive and the length of the adhesive. The maximum force of adherence increases with the thickness of the substrate and the length of the adhesive.

The three-point flexion test is performed on a tensile test apparatus with a specific assembly. The sample dimensions and the test conditions are defined by the AFNOR NFT 30010 standard. The stiffener, which was curing at ambient temperature, is a solvent free epoxy adhesive (CIBA AW 134/HM994). The average of the ultimate load (F) and its standard deviations were determined for the different surface treatments.

3.1.3 Tensile test

The micro tensile test is used on systems made up of a ductile coated substrate in order to analyze the mechanical stability and the damage of the deposits [17][18]. These tests require various experimental measurements like the tensile strength and spacing inter-cracks characteristic of film split up. According to the Agrawal and Raj model [19], the maximum shearing force interfacial, τ , developed with the interface between the deposit and metal, is related to the tensile strength of the coating, σ , by the equation 2. In wich $\sigma = E\epsilon$, with ϵ the stress corresponding to the beginning of cracking of the deposit. E and δ are respectively the Young modulus and the thickness of the coating. Finally λ is the wavelength of the function sine which defines the maximum interfacial shearing force. It is equivalent to a virtual spacing inter-cracks characteristic of saturation in transverse cracks in spite of the increase in the tensile stress. This equation is valid for a system film/coating free from residual stresses. This model was then modified to extend these validity for a thicker and harder deposit with residual stresses (equation 3) [20].

$$\tau = \frac{\pi\delta\sigma}{\lambda}, \text{ Equation 2}$$

$$\sigma_e = \sigma_f + \sigma_R, \text{ Equation 3}$$

$$\tau = \frac{\pi\delta\sigma_e}{\lambda}, \text{ Equation 4}$$

These various studies have nevertheless a weakness related to the observation of the mode of failure. Indeed the majority of the observations are carried out ex situ by electronic scanning microscopy. This type of observations presents disadvantages: first, the tensile test is not continuously. Then the effects induced by the first stages of elastic strain of the system do not occur continuously. The stress state of the deposit is not constant; the cracks can be closed again and become undetectable. In situ inspections under optical microscopy or scanning electronic microscopy make it possible to follow the damage of the deposit uninterrupted and thus to eliminate these disadvantages [21][22][23].

3.2 Elaboration of the coatings

3.2.1 Zinc-Nickel coatings on aluminium alloy

Zn-Ni coatings were electrodeposited onto an aluminium alloy 2024 T3 sheets in a laboratory flow cell [24]. To obtain several Ni percentages (from 4% to 16 wt. %) in the coatings, different Ni²⁺ ion concentrations were used (Table 1). It is known [25] that a good corrosion behaviour of the coatings is obtained for 4% to 16% of nickel. Moreover, the percentage of nickel makes it possible to modify the hardness of the deposit and thus to modify the behaviour of the substrate-coating system. The different steps of the normal pre-treatment were: acetone cleaning and rinsing in distilled water, NaOH cleaning and rinsing in distilled water, HF/HNO₃ cleaning, rinsing in distilled water and air-drying. The influence of specific pre-treatments like zincate immersion (100g/l ZnO, 525g/l NaOH) or phosphoric anodisation (36 wt. % H₃PO₄, 20°C) prior to electrodeposition were investigated to promote adherence.

3.2.2 Nickel phosphorus – Talc coatings on steel

The chemical platings Ni-P/talc were deposited on steel 36NiCrMo16 substrates [26]. The substrates are polished mirror and then pickled in a hydrochloric bath of acid diluted and rinsed with the water distilled before the development of the deposits. The coatings were carried out on disc starting from a commercial bath Europlate Ni216 (low phosphorus) in which talc particles were put in suspension. Thermal pre-treatments (420°C and 600°C under atmosphere controlled during 1 hour) were carried out (Table 1).

Nickel coatings (Europlate Ni216 commercial bath)			ZnNi coatings			
	NiP [g/l]	NiP-talc [g/l]		Zn-4%Ni	Zn-8%Ni	Zn-16%Ni
Nickel metal	6 (±1)	6 (±1)	NiCl ₂	13	39	142.5
Nickel hypophosphite	23 (±3)	23 (±3)	ZnCl ₂	334	334	334
Sodium orthophosphate	0 - 180	0 - 180	KCl	340	340	340
Talc	-----	20 - 120	current density	50A/dm ²	50A/dm ²	50A/dm ²
Temperature	88°C (86-92)	88°C (86-92)	Temperature	60°C	60°C	60°C
pH	5.0 (4.8-5.4)	5.0 (4.8-5.4)	pH	2	2	2

Table 1: elaboration parameters of electroless NiP and of ZnNi coatings

4 Results and Discussion

4.1 Scratch test

The scratch tests were carried out on ZnNi coatings. The results show that the width of the scratch tracks increases with the load applied and decreases with the nickel percentage of the deposits. It is due to the increase of the deposits' hardness with the percentage of Ni [24]. The variations of the various sizes measured during the scratch test, such as the friction coefficient, the width of the scratch tracks according to the various test parameters, lead us to think that the deposits have different mechanical behaviours. These evolutions result from conservative phenomena to the weak loads and dissipative with cracking with the strong loads as soon as the Ni content of the coatings is high. The Zn-4%Ni coatings do not seem to dissipate energy at the interface. The scanning electron microscopy observations of the scratches confirm it. The deposits with small percentage of nickel are characterized by a ductile behaviour (figure 2). For the very weak loads, the scratch is discontinuous. Only the most important clusters are deformed and compacted below the indenter. This explains the variations of the friction coefficient for the weak loads. The more important the load applied is and the perfectly more defined contours of the scratches are. The chippings cohesive, i.e. limited to the deposit, are observed at the edge of the stripe. No cracking is noted in bottom and at the edge of the stripe whatever the even strong load applied. The critical load is defined in this case by the load which allows the setting naked substrate in bottom of stripe (figure 2). The cross sectional observations show that the deformation is limited in the deposit for loads lower than the critical load. The deposit is thinned when the load increases. The substrate deformation appears only when the indenter is in contact with this one.

The behaviour of the deposits having percentages out of nickel higher than 8% is completely different from that previously described (figure 2). The scratches have the contours defined better as of the weak loads because of their low roughness. This explains the weak variations of the friction coefficient for the weak loads. As of the weak loads applied, a transverse cracking is observed in the behind curved stripe. Spacing between the cracks decreases when the load is increased. Moreover, a cracking which occurs on the sides of the scratches extend in the deposit. With more raised loads, transverse cracking always appears; combined with an increased longitudinal cracking. It leads to a fragmentation of the deposit. The critical load is then reached with the appearance of side chippings. It increases as function of the thickness of the deposits but remains lower than that obtained for deposits with 4% Ni. The cross sectional observations of the scratches give additional information on the mechanical behaviour of the deposits rich in Ni (figure 2). Indeed, we observe a combined deformation of

the deposit and substrate. Thus, under the depression of the substrate, the deposit is put in tension on the sides what involves its loss of adherence. These two types of behaviour can be explained by the difference in the deposits hardness and more particularly by the effect of the ratio hardness deposit/hardness substrate ($H_{Zn-4\%Ni}/H_{Aluminium} = 0.6$, $H_{Zn-16\%Ni}/H_{Aluminium} = 3.25$). Consequently, the scratch test makes it possible to show that a continuity of the mechanical properties to the interface is necessary to obtain a sufficient adherence. In conclusion, if we reason on the loss of the coating's function; the scratch test gives a critical load according to the studied coatings. However, this critical load is a function not only of the properties of the interfaces but also of the properties of the deposits. Which is then the contribution of mechanical dissipation on the critical load for the Zn-4%Ni coating less hard than the substrate?

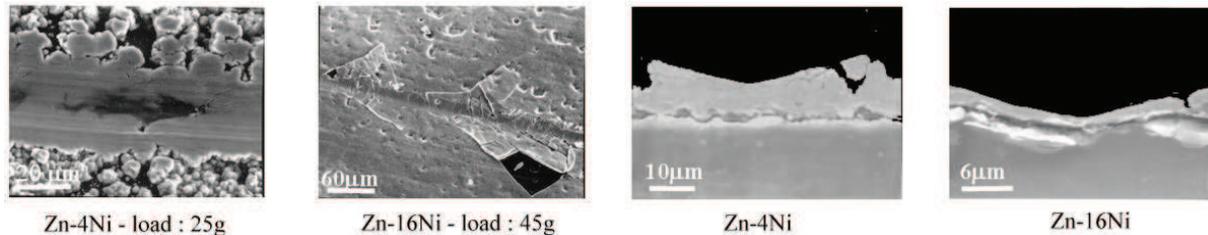


Figure 2: scratches on Zn-Ni coatings function to the load applied.

4.2 Three point bending test

The three bending tests were carried out on ZnNi deposits coated on aluminium pickled only or treated by zincate or anodization. The tests allow highlighting variations of adherence according to the Ni percentage and pre-treatments (figure 3). Without pre-treatments, the deposits with 4% Ni seem more adherent than the other deposits. The SEM fractographies show the rupture of the deposits. Analysis EDS allows to locate the rupture zone and thus its type (adhesive, cohesive, mixed) (figure 3). The rupture of the deposits containing up to 10 % nickel is mixed (cohesive in the deposit and adhesive). On the other hand, the rupture of the deposits containing 16% nickel seems adhesive. The deposit having a content of 4% Ni has a mode of rupture divided into two stages. Moreover, we observe a light inflection of the curves before rupture representative of a weak plastic deformation. The samples having a percentage out of nickel equal to or higher than 10%, are characterized by a brittle fracture.

It is interesting to note, first of all, the increase in the maximum force reached before rupture for all the samples pre-treated by immersion in a zinc bath compared to the untreated. The maximum force and displacement reached for Zn-4%Ni deposit are slightly higher than that of the two other deposits. The fractographies provide us more information on the mode of rupture. Two zones of rupture are distinguished on Zn-4%Ni coatings. The first zone corresponds to a rupture located in the deposit and the stiffer. certain clusters seem to have broken under the applied load. The second zone corresponds to a rupture between the deposit and the substrate. Spectrometry X analyses indicate the presence of a zinc film on alloy 2024 T3. The edges of the rupture are badly defined. The rupture of high nickel coatings mainly occurred at the interface deposit-stiffener. Consequently, the maximum force obtained during various tests for these two deposits does not represent the maximum force of adherence. However, a mixed rupture with a Zn-16%Ni coating is observed. The fractographies are different from those observed on the sample coated of the same deposit but no pre-treated. It remains always present by place.

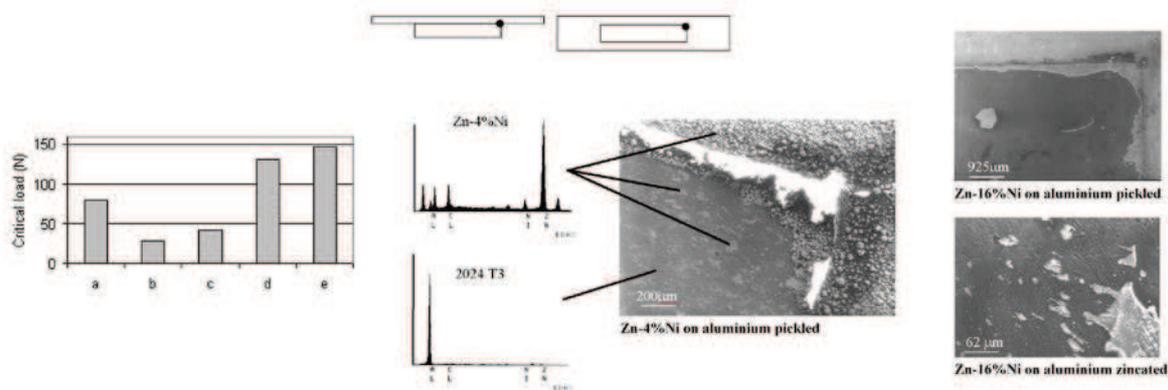


Figure 3: critical load for coatings directly electrodeposited onto 2024 aluminium alloy (a) 4%Ni, (b) 8%Ni, (c) 16%Ni, (d) 16%Ni on EN AW2024 zincate pre-treated, (e) 16%Ni on EN AW2024 anodised.

An increase of the maximum load of adherence is observed thanks to the phosphoric anodization pre-processing. For the Zn 4%Ni deposits, the rupture is mixed since it is localised at the same time in the deposit and at the

interface deposit / anodic substrate. The fractographies of the deposits Zn-16%Ni after anodization are different from that is previously described. The fracture zones are more distinct and testify to the fragile character from the rupture. Many clusters imprisoned in the cavities of the substrate's surface are sheared with the interface. The spectrometry X analyses do not enable us to conclude on the precise localization from the rupture. It would seem that the rupture is also localised in the layer anodized considering the small percentages of oxygen raised in the rupture zone. Consequently, the difficulty of the determination of the rupture zone arises ? SIMS analysis made it possible to highlight an interphase made by the deposit anchored in the pores of anodization. A finite element model was made in order to determine the stress at the interface. The model was checked from the maximum displacement measured and calculated for equal load applied. The flexion test is a simple and relevant test. But it does not allow to determine the adherence of very adherent deposits. In order to free itself from this last problem and the ratio from hardness deposit-substrate, another test was implemented: the tensile test.

4.3 Tensile test

The crack density due to the strain rate was measured during the test video acquisition. Talc seems to modify the adherence of the coatings. It was shown that the rate of cracking to saturation increases with talc for untreated coatings. Moreover, the cracks are deviated on coatings containing of talc. Observations post-mortem by SEM supplemented by EDS analyses confirm this result and attest of a brittle fracture of the coatings. The rupture is adhesive for untreated deposits without talc and becomes mixed with the insertion of talc. Cohesion particles / matrix seems less strong than that deposit / substrate.

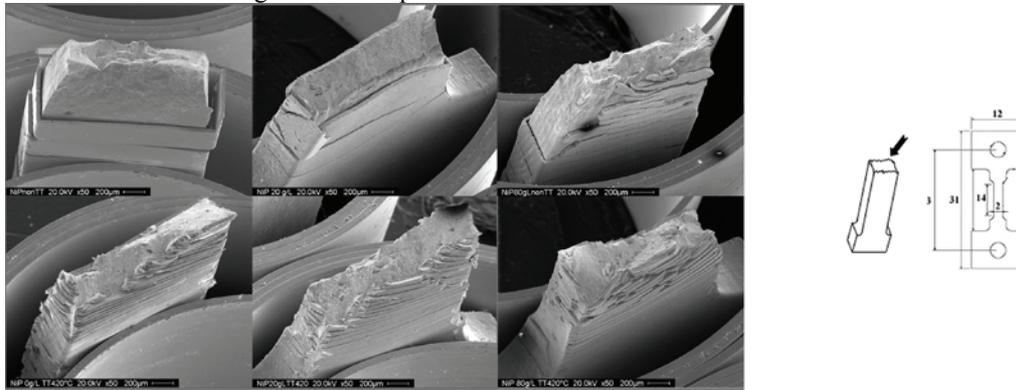


Figure 4: SEM fractographies after tensile tests.

Treatments	coatings	ϵ_f (μm)	λ_0 (μm)	E_f (GPa)	ϵ_f (%)	σ_R (GPa)	τ_{A-R} (GPa)	τ_{res} (GPa)	Rupture mode
untreated	NiP	35.4	1562	164	1.37		0.08		adhesive
	NiP-20g/l	38.6	200	140	3.75		0.28		mixed
	NiP-40g/l	35.4	451	134	1.94		0.32		mixed
	NiP-80g/l	39.3	100	130	3.51		3.03		cohesive
	NiP-120g/l	47.8	44	119	4.34		9.96		cohesive
Treated at 420°C	NiP	34.3	27	201	0.4	0.039	1.10	1.14	cohesive
	NiP-20g/l	37.5	10	180	1.67		3.89		cohesive
	NiP-40g/l	36.8	31	181	2.06	0.143	4.45	4.59	cohesive
	NiP-80g/l	39.4	15	160	1.24		2.45		cohesive
	NiP-120g/l	47.7	34	172	2	0.186	6.35	6.54	cohesive
Treated at 600°C	NiP	36.8	148	202	1.54	0.009	1.21	1.22	adhesive
	NiP-40g/l	42.2	299	152	2	0.211	0.80	1.01	cohesive
	NiP-120g/l	29.2	139	142	2.06	0.247	0.96	1.21	cohesive

Table 2: various values of τ for each type of coating

The cracks density due to the rate of deformation strongly increases after a heat treatment. The observations in situ by optical microscope and post mortem by SEM confirm the increase in adherence of the deposits heat treated. The coatings remain adherent close to the rupture zone of the substrate (figure 4). The influence of talc on the deviation of the cracks is less obvious because of a better cohesion particle/matrix but more especially of a better rigidity of the deposits. The ruptures are respectively adhesive and cohesive in the deposit for deposits without talc and with talc. The interfacial shear stress was thus given with the model of Agrawal and Raj without holding account (τ_{A-R}) or by holding account (τ_{res}) residual stresses of the coatings (table 2). The Young modulus of the various deposits was given thanks to nanoindentation tests [26]. The thicknesses were measured in cross section with SEM, the residual stresses by the $\sin^2 \psi$ method by X-rays diffraction. Note that for the studied

coatings τ_{A-R} and $\tau_{residual}$ are almost identical since the values of $\sigma_{residual}$ (a few hundreds of MPa) are negligible in front of $\sigma_{applied}$ (about GPa).

The heat treatment increases the adherence of the deposit considerably. It also seems that the talc addition improves the behaviour of the coating. These results confirm preceding experimental results and testify to the relevance of the model for a quantitative approach of the adherence of the studied coatings.

5 Conclusion

The determination of the adherence of a deposit is very complex. There is not a single test. It is desirable to perform more tests in order to cross the results to obtain tendencies. It is in this objective, for example, that three types of test (scratch test, bending test, tensile test) were implemented in order to characterize the adherence of ZnNi and NiP coatings on aluminium or steel substrates.

The scratch tests made it possible to highlight two types of damage resulting in a loss of adherence: the different scratching behaviours are due to the variation of the intrinsic properties of the deposits according to the percentages of nickel compared to the constant properties of the aluminium alloy substrate. However, the stress field generated by the indenter in the deposit is very complex. It is difficult to separate the contribution from the intrinsic properties of the deposits or the substrate and from those of the interface in the critical load of adherence. Consequently, conscious of the interest but also of the limits of the scratch test, the study of the adherence was continued using an other mechanical test: the bending test. Indeed, this test enabled us to quantify a maximum force of adherence for each type of coatings according to the pre treatments and the electroplating conditions. Moreover, a numerical modelling in support of the experimental tests allowed us to determine a rate of strain in a zone close to the rupture. This one enabled us to confirm the brittle fracture observed during the tests. The loss of adherence of the electroplating coatings directly on aluminium alloy appears whereas the deposits did not reach their yield stress. The given maximum force during the bending thus is influenced little by the deformation of the deposit. This test gives good results only when the deposits are not very adherent on the substrate. The stiffeners are detached from the coatings without being able to test their adherence with the substrate. For the very adherent deposits, such as for example the NiP deposits on steel, a fragmentation tensile test gives very good results. However, in certain cases, it is necessary to deform the substrate plastically in order to load the coating. One can wonder consequently about the contribution of mechanical dissipation on the final result of adherence. Lastly, post mortem observations often supplemented by physico-chemical analyses are necessary to determine the type of rupture of the coatings.

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