Revista de metalurgia

Accelerated tests of coil coatings

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Abstract Accelerated laboratory tests on 12 materials in study in the Subgroup 6 of the PATINA Network (CYTED), are discused for different exposition periods in salt spray, SO_2 and Prohesion chambers. International standards used to evaluate failures caused by the different aggressive agents of these laboratory tests are the same as those applied for outdoor expositions. The results exposed contribute to a better understanding of the mechanisms occurred in the diverse natural environments, being mentioned the main analogies and differences respect to factors affecting natural tests.They also allowed to evidence the advantages and limitations in the application of these tests during several days, as compared to the years required to attain similar failure magnitudes through outdoor tests.

Keywords Accelerated test. Normalised test. Coil-coating.

Ensayos acelerados de recubrimientos para banda continua

Resumen En este trabajo se discuten los ensayos de laboratorio acelerados, realizados sobre 12 materiales de estudio en el Subgrupo 6 de la Red PATINA (CYTED), a diferentes periodos de exposición en cámaras de niebla salina, SO_2 y Prohesion. Se utilizaron las normas internacionales para evaluar los fallos causados por los diferentes agentes agresivos de estos ensayos de laboratorio, las cuales se aplican también para los ensayos de exposición a la intemperie. Los resultados expuestos contribuyen a una mejor comprensión de los mecanismos ocurridos en los diversos ambientes naturales, mencionándose las principales analogías y diferencias respecto de los factores que afectan los ensayos naturales. También permitieron evidenciar las ventajas y limitaciones en la aplicación de estos ensayos durante varios días, en comparación con los años requeridos para alcanzar magnitudes de fallos similares por medio de ensayos a intemperie.

Palabras clave Ensayos acelerados. Ensayos normalizados. Recubrimiento para banda continua.

1. INTRODUCTION

The long duration of the outdoor tests, necessary to select on a performance basis among different corrosion protection schemas (paints, primers, oils, etc.) was the origin of the accelerated laboratory test.

The need to reproduce the effect of natural environments was a consequence of the distinct behaviour observed for a given system, according to the site where the material has been tested or has to spent its service life. However, the acceleration in the failure nucleation and propagation, with respect to that occurred in highly aggressive natural atmospheres, must consider the need to maintain the same degradation mechanism as in the natural environment. Thus, salt spray[1 and 2] and SO_2[3 and 4] chambers, were developed and normalised to test protective systems in marine and industrial environments respectively. Prohesion[5] is another test during which samples are submitted to alternate salt fog and drying periods in a laboratory chamber.

Each of these test provides the possibility of analising the separate effect of variables simultaneously acting on materials during natural exposures. They are usefull tools contributing to understand the mechanism of materials degradation in the atmosphere. However, there is not a single laboratory test able to cause the same

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type and relative magnitude of damage, on determined set of protection schemas, as that produced by an aggressive natural atmosphere. On the contrary, this normalized set of test allows to reproduce them when desired, in well defined conditions. Different researchers from various laboratories can get results with such a low scatering, as cannot be done at any given time in natural atmospheres.

The experience developed in order to obtain more relyable resuts led to a series of normalissed equipment to simulate different atmospheric conditions, which cannot be extrapolated to the real situation it tries to simulate and accelerate, nor to give a time correlation, unless the performance comparison concerned a single type of materials.

Evaluation of the failures caused by the mentioned different simulated atmospheres was done by application of ISO 4628/82, 1 to 6[6-11] standards, also followed to quantify type and magnitude of failures after outdoor tests.

2. EXPERIMENTAL

Coil-coatings of 12 different formulations, shown in table I, have been tested in 3 accelerated test chambers to determine the type and magnitude of failures observed for different exposure times, as compared to those found in 9 MICAT Test Stations of distinct natural atmospheric conditons, up to 42 month exposure.

The results obtained after various exposure times for the distinct accelerated tests are discussed to intend a sequence, time or order necessary to get most reliable results to those obtained during outdoor exposures. Also a relationship among the type of damage nucleated is intended when compared to the simulated natural environment. Similarities and differences, as much as time required initiating the different deffects are discussed.

Among the characteristics changing during accelerated tests of the 12 schemas of this type of

Table I. Characteristics of the tested materials

Tabla I. Características de los materiales ensayados

Code	Metallic coating	Paint								
		Thickn.	Base			Finishing				Total thick.
		(μm)	Vehicle	Pigm.(**)	E (μm)	Vehicle	Pigment	Colour	E (μm)	E (μm)
A1	Galvalume	20	Epoxy	Ti, Cr, Co, Al	4	PVDF	Al, Ti, Fe, Si	Light Blue	20	44
A2	Zn	20	Epoxy	Fe, Cr, Zn, Ti	6	PVDF	Fe, Zn, Si	Blue	17	43
A3	Zn	20	Epoxy Acrilic	Si, Fe, Ti	6	Poly. Silic.	Fe, Si, Pb	Red	17	43
A4	Galvalume	20	Polyester	Fe, Si, Ti	5	Poly. Silic.	Fe, Si, Al, Ti	Light Blue	17	42
A5	Galvalume	20	Epoxy	Fe, Si, Ti	4	PVDF	Al, Si, Ti	Red	19	43
A6	Galvalume	20	Polyester	Ti, Si, Al	5	Polyester	Ti, Si, Al	White	18	43
CH1	–	–	Epoxy	Fe, Si	15	Polyester	Fi, Fe, Al, Si	Beige	15	30
CH2	–	–	Epoxy	Fe, Si	15	Polyester	Fe, Si	Brown	15	30
E1	Zn	20	Polyester	Ti, Al, Si	20	Polyester	Ti, Al, Si	White	15	55
E2	Zn	25	Polyester	Ti, Cr, Si, Al	20	Poly. Silic.	Fe, Si, Ti, Ca	Red	10	55
E3	Zn	20	Epoxy Aminopl.	Fe, Ti, Si	20	PVDF	Fe, Al, Si	Green	16	56
(*)E4	Zn	25	–	–	–	Alcid. Melam.	Ti, Si, Al	White	25	50

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(**) Elements detected through EDX, in decreasing order of relative abundance.

materials, blistering and corrosion, both at the incision, are the most sensitive effects in all natural as also in simulated environments. Next, corrosion in the sample body away from the incision gives an idea of the porosity of the paint schema and reactivity of the respective metallic sustratum to the distinct accelerated ambients.

Representative single data, involving blistering size and density, were estimated on the basis of the proposal made by Keane et al.[12] and shown in table II. Value 0 was assigned for the absence of failure and 5 for complete damage, according to the ISO scale of 4628/82-1 standard[6].

Table II. Deterioration grade established as a function of blistering size and density

Tabla II. Deterioro establecido en función del tamaño y la densidad de ampollado

SIZE	DENSITY				
	1	**2**	**3**	**4**	**5**
S1	**1**	1	1	1	2
S2	1	**2**	2	2	3
S3	2	2	**3**	3	4
S4	3	3	4	**4**	5
S5	4	4	4	5	**5**

2.1. Prohesion test

Prohesion is an accelerated test in which the samples are exposed to alternate cycles of saline fog (NaCl 0.05 % and $(NH_4)_2SO_4$ 0.40 %) and drying in a close laboratory chamber. For this test, one-hour time were fixed for each cycle.

Samples of 10 × 15 cm were tested, one of each material with a 2 cm incision from the bottom and 2 cm from each side, according to ISO 2409[13]. Materials characteristics are shown in table III.

Periodically, the deterioration magnitude of the samples was evaluated, observing that the incision region was the most sensitive to corrosion and blistering, as can be seen in table IV. Samples showing the best and worst performances against the Prohesion test aggressive atmosphere were identified.

The evaluation of the corrosion grade was made according to ISO 4628/3 standard[8]. For visualising the percentage of corrosion in the incision, bars diagrams showing the corrosion grade of the samples for different test times were elaborated. At the incision, a visual estimation of the area percentage covered by corrosion products was carried out for different test times.

The performance observed for the 12 materials was discused according to the schema total

Table III. Tested samples. Those labelled with "s" have no incision

Tabla III. Muestras ensayadas. Las designadas con "s" no tienen incisión

MATERIAL ORIGIN	CODE	MEDIUM THICKNESS (μm)	CHARACTERISTICS
	A1	46	Galvalume + epoxy/PVDF
	A2	48	Zn + epoxy/PVDF
Argentina	A3	47	Zn + epoxy acrylic/PVDF
	A4	46	Galvalume + polyester / siliconized polyester
	A5	51	Galvalume + epoxy/siliconized polyester
	A6	59	Galvalume + polyester/polyester
Chile	CH1	32	Steel (epoxy/polyester)
	CH2	24	Steel (epoxy/polyester)
	E1	37	Zn (polyester/polyester)
	E1s	38	Zn (polyester/polyester)
	E2	53	Zn (polyester/siliconized polyester)
Spain	E2s	57	Zn (polyester/siliconized polyester)
	E3	37	Zn (epoxy aminoplaste/PVDF)
	E3s	35	Zn (epoxy aminoplaste/PVDF)
	E4	48	Zn (Alcidic melamine)
	E4s	53	Zn (Alcidic melamine)

Table IV. Salt Spray Chamber – Time for failures initiation, in hours

Tabla IV. Cámara de Niebla Salina – Tiempo de inicio de falla, en horas

SAMPLE CODE	BLISTERING		CORROSION		OBSERVATIONS
	Body	Incision	Body	Incision	
A1		168		White	
A2				White	Test finished at 3500 hs.
A3		1128		White	Best behaviour A2
A4		3500		White	Worst behaviour A1 also in protected
A5		3500		White	borders in A4 and A5
A6		3500		White	
CH1	624			24	Test finished at 1680 h
CH2	624			24	Similar behaviour in CH1 and CH2
E1	624	168		24	Test finished at 3500 hs.
E2	624	168		24	Best behaviour E3. Worst behaviour E2
E3	3500	168		24	Blistering and white corrosion also in non-
E4	1128	168		24	protected borders

thickness, metallic coating presence and composition, base and finishing paint coatings.

2.1.1. Results of Prohesion test

A1 to A6, samples

- Blistering on the body: A1 and A5 samples showed no blistering up to 1500 test hours, as can be seen in figure 1 a.
- Blistering at the incision: A4 sample showed the best behavior as can be seen in figure 1 c.
- Corrosion: Only sample A6 presented corrosion before 1500 test hours, showing the worst behavior.
- Corrosion % at the incision: Best behavior was observed for samples A6, A4, A3 and A1, in decreasing order.
- Observations: At the end of the test similar aspects for all the samples, with little blisters in incisions and on the body of some of them was observed. At all the incisions white corrosion products were observed.
- Conclusion: Samples A1 and A4 showed the best behavior. In every sample the protector character of the Galvalume coating was appreciated.

CH1 and CH2 samples

- Blistering on the body: Similar for both (Fig. 1 a).
- Blistering at the incision: CH1 sample presented a better behavior than CH2 (Fig. 1 a).
- Corrosion: CH1 sample presented better behavior than CH2.
- Corrosion % at the incision: Similar behavior for both.
- Observations: The most severely damaged on the whole body, with abundant corrosion products in incision and borders, was CH2.
- Conclusion: Best behavior is shown by CH1 sample.

E1 to E4 samples

- Blistering on the body: Best behavior in E2s, E3s and E4s samples. Better behavior was shown by samples without incision as shown figure 1 b.
- Blistering at the incision: Samples E2 and E3 presented the best behavior as shown figure 1 d.
- Corrosion: None presented corrosion.
- Corrosion % at the incision: Sample E3 presented the best behavior.
- Observations: All samples presented incision deterioration, such as blistering, white corrosion products and disbonding. In samples E1 and E4 brown corrosion products were also observed. Samples E2s, E3s and E4s showed a better behavior than the respective with incisions.
- Conclusion: In general the best behavior was shown by sample E3.

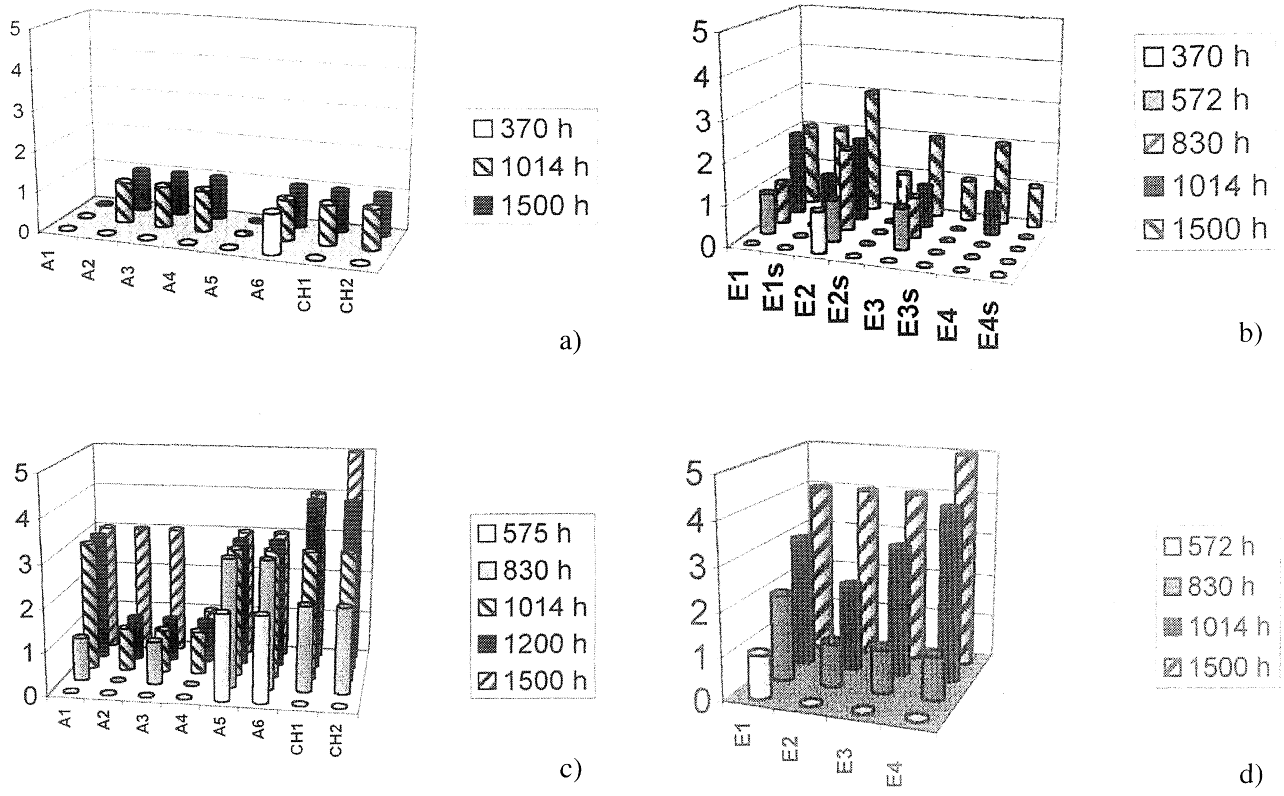

Figure 1. Blistering on the body for different exposure times of samples A1 to A6, CH1 and CH2 (a) and E1 to E4 (b); Blistering at incision for different exposure times of samples A1 to A6, CH1 and CH2 (c) and E1 to E4 (d).

Figura 1. Ampollado en el cuerpo para distintos tiempos de exposición de las muestras A1 a A6, CH1 y CH2 (a) y E1 a E4 (b); Ampollado en la incisión para distintos tiempos de exposición de las muestras A1 a A6, CH1 y CH2 (c) y E1 a E4 (d).

In the body zone of "samples"

- Deterioration such as small blistering (grade (1) was observed in most paint systems with the exception of A5, (epoxy acrylic/polyester siliconized finishing), which resisted 1500 hours test without failing and in the E2, E3 and E4 (melamine alcidic) that resisted more than 1000h in the prohesion test without presenting this type of failure.
- Against corrosion the behaviour is best (grade (2). Only in some systems, such as in A6, which means in polyester/polyester paint systems and CH1 epoxy/polyester without metallic coating, Zn base, alquidic coatings and not over the grade 1 of the norm.

On the "incision"

- At 500 h all samples presented blistering near the incision, which increased with exposure time.
- Also, in very few hours all the samples evidence corrosion in the incision of the steel base schemas while in the galvanised ones more than 700 h were necessary for oxide points nucleation.

2.1.2. Conclusions of Prohesion test

- In the 12 coil-coating schemas tested, deterioration in the form of blistering and corrosion at incision occurred for all samples. Blistering increased in areas next or influenced by the incision.
- Cathodic protection by the Zn base coating occurred in areas with paint defects and incisions.
- Coil coatings present a good performance in spite of their low thickness (35-50 μm) as steel protection schema.

2.2. Salt spray test

International standards most generally applied to follow this test, evaluate samples and express results for different exposure times, are ASTM B 117 [1] and ISO 7253 [2]. Results are shown in table IV.

2.3. Moist SO_2 test

The ASTM G 87[4] and ISO 3231[3] are the international standards. Results are shown in table V.

4. CONCLUSIONS

From the results exposed for each accelerated test the following general conclusions can be obtained:

- Experimental results obtained show that each material appears as the best or the worst, depending on the type of failure evaluated (blistering, corrosion, gloss, color, etc.), even for the same accelerated test applied.also in natural environments, are corrosion and after blistering, both at incision.
- Metalic Zn based coatings increases time for red corrosion initiation in borders and incision.
- Thicknes of the overall protective schema decreases the sensitivity to blistering in the sample body.
- Metal Zn base coatings promote pore blockage in organic coatings, but when Zn corrosion is intense organic coating disbonding is produced. This also occurs in natural environments.

Differences in factors controlling coil coating failures in these laboratory tests as compared to outdoor tests, are:

- The strong effect of the sun radiation during outdoor test is not involved in the organic coating laboratory weathering processes.
- 100 % TOW for salt spray tests form Zn corrosion products with lower protectiveness than during alternated dry and wet periods during outdoor exposures.
- SO_2 chamber produces such acceleration in Zn corrosion that body blistering on all samples is highly promoted as compared to industrial or urban natural atmospheres.

Acknowledgements

The authors gratefully acknowledge to CYTED for financing the National Coordinators Meetings and to Bettina Marchisio for her assistance during the preparation of this paper.

Table V. Moist SO_2 Test – Time for failures initiation, in h

Tabla V. Ensayo de cámara húmeda de SO_2 – Tiempo de inicio de fallo, en h

SAMPLE CODE	BLISTERING		CORROSION		OBSERVATIONS
	Body	Incision	Body	Incision	
A1	96	480		24 red	Best behavior A2
A2	1680	168		24 grey	Worst behavior A6
					480 h corr. attenuated in A3 and at 1440
A3	168	168		24 grey	360 disbonding in A4
A4	96	360		24 grey	624 cracking in A5
A5	624	624		24 white	Disbonding in incision in A6
A6	480	360	1680	24 grey	Test finished at 1680 h
CH1	168			24	Similar behavior in CH1 and CH2
CH2	96			24	Test finished at 360 h
E1	440	168		24 white	Best behavior in E3
E2	168	360	1680	24 white	Worst behavior E1
					480 disbonding in borders in E2
E3				24 white	240 disbonding in E3
E4				24 white	Test finished at 1680 h

REFERENCES

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[2] ISO 7253, Paints and vernishes- Determination of resitance to neutral salt spray.

[3] ISO 3231, Paints and vernishes. Determination of resistance to humide atmospheres containing sulphur dioxide.

[4] ASTM G 87, Practice for conducting moist SO_2 test.

[5] ASTM 87, Modified salt spray test.

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