



Correlations between standard accelerated tests for protective organic coatings and field performance

O.Ø. Knudsen^{a,*}, A.W.B. Skilbred^b, A. Løken^b, B. Daneshian^c, D. Höche^c

^a SINTEF, Richard Birkelandsvei 2B, 7465 Trondheim, Norway

^b Jotun, Hystadveien 167, 3209 Sandefjord, Norway

^c Hereon, Max-Planck-Straße 1, 21502 Geesthacht, Germany

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ABSTRACT

Accelerated testing is widely used in development and pre-qualification of protective organic coatings. In this work 26 coating systems have been investigated in a 2-year C5 atmospheric exposure field test, ISO 9227 salt spray test, ISO 12944-9 cyclic ageing and ISO 16773 electrochemical impedance spectroscopy measurement of coating resistance. Of the 26 coating systems, 16 have epoxy mastic primers and 10 have zinc rich epoxy primers. In the field test, the zinc rich primer improved corrosion creep resistance from scribe by a factor of about 10. However, this is not reflected in any of the accelerated lab tests. The lab tests all show rather poor correlation to the field test with respect to corrosion creep. All the coatings had little corrosion creep from scribe in the field test, even the coating systems with epoxy mastic primers. The large focus on this parameter in coating pre-qualification testing, e.g. in ISO 12944-6 and 12944-9, may therefore not be justified.

1. Introduction

Corrosion of steel constructions under atmospheric conditions is usually controlled by application of protective paint systems. In 2013, the cost of corrosion in USA was estimated to 3.4% of GDP, and for the industries and public sectors that were analysed, application and maintenance of protective paint coatings constituted a significant proportion of this. Of the total costs for corrosion protection, about 90% was estimated to be protective paint coatings. The market volume for protective paint coatings was estimated to be 6.7 billion USD, which is only 4–20% of the total application costs [1]. Similar results have been found for other countries. Given the magnitude of resources spent on paint, application and coating maintenance, optimizing coating properties and coating selection may give huge savings for the owners of painted steel. Protective paint coatings are the only cost effective solution to limit and prevent corrosion of steel structures. Performance testing is essential throughout the development and qualification of protective paint, and the basis for such testing is accelerated laboratory testing.

Numerous accelerated test methods for protective organic coatings have been issued by standardisation organizations like ISO and ASTM, and in addition many companies have developed their own accelerated test methods. An accelerated coating test method should test the

properties that limits coating lifetime and correlate well with field performance.

Historically the continuous salt spray test [2,3] has been used for evaluating coating performance, and the method is still used in ISO 12944-6 [4] as an aid for selecting coating systems for carbon steel structures in environments with corrosivity C2 – C5. However, salt spray testing of organic coatings has been heavily criticized for poor correlation to field performance [5–9], and for producing other degradation mechanisms than observed in the field [8,10]. Cyclic ageing tests on the other hand, have better reputation regarding correlation to field performance. LeBozec et al. found good correlation between ISO 16701 [11] and 2 years field performance in a test program with 15 coating systems. They also concluded that increasing the acceleration factor in a test reduced the correlation to field performance. Knudsen et al. investigated 36 combinations of coating systems and surface cleaning methods in various accelerated laboratory tests and 5 years field exposure [12]. They found that the test described in NORSOK M-501, revision 1 (1994) [13], that later became ISO 20340, had a correlation factor of 0.6 – 0.7 to the field test. A closer look at the results indicates that correlation was best for coating systems with poor performance. As LeBozec et al., Knudsen et al. also concluded that increasing the acceleration factor decreased correlation to field performance. Another

Abbreviations: DFT, Dry film thickness; NDFT, Nominal dry film thickness; PU, Polyurethane; EIS, Electrochemical impedance spectroscopy.

* Corresponding author.

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conclusion from the investigation was that the salt spray test had poor correlation to the field test. Reuterswård and Tidblad investigated 25 coating systems in a 105 months field test, ISO 16701 and ISO 20340 (later ISO 12944-9) [14], and concluded that neither of the two accelerated tests gave a satisfactory ranking of the performance of the coating systems in the field test. Spearman's rank correlation coefficient was 0.41 and 0.56 respectively for ISO 16701 and ISO 20340. However, they found a strong correlation between performance after 19 months and 105 months in the field test, with a Spearman's rank correlation coefficient of 0.86. Hence, a field test of less than 2 years seems to predict performance after 10 years. Common for all these studies is that very few coating systems showed any significant visual degradation, hence the only discriminator was the measured corrosion creep from an artificial damage in the coating.

In a Round Robin test, investigating variation between labs performing the ISO 12944-9 cyclic ageing test [15], considerable variation was found between the investigated laboratories [16]. Both the variation between laboratories and the moderate correlation between lab testing and field performance indicates that results from accelerated lab testing of organic coatings must be carefully considered during product development and qualification of coating systems. The testing can neither be used for estimating field lifetime of the coatings, nor accurate ranking the performance of coating systems in the field. Rather, the best we can hope for is that accelerated tests can reveal coatings with inferior properties and quite poor performance. Both the variation between laboratories and the moderate correlation between lab testing and field performance indicates that, when focusing on the corrosion creep from an artificial scribe, the results from accelerated laboratory testing of organic coatings must be carefully considered during product development and qualification testing.

The objective with this study was to perform a systematic investigation between accelerated lab tests and field exposure, focusing on accelerated test methods that are used for pre-qualification of protective paint.

2. Experimental

2.1. Sample preparation

All samples were prepared from 5 mm thick S355JR structural steel. Panels, 150 × 75 mm, were grit blasted to Sa 2½ medium roughness before the coatings were applied by airless spray [17].

The 26 different coating systems tested are described in Table 1. All coating systems were composed from commercially available paint products. The two zinc epoxy primers used conform to the compositional requirements in ISO 12944-5 [18], i.e. with a zinc dust content of more than 80% by weight in the dry film. The backside and edges of the panels were protected with one coat epoxy mastic. Three parallels of each coating system were tested in the lab tests. For field testing, sets of three parallels, comprising two samples with scribe and one without scribe, were evaluated.

2.2. Field exposure

The samples were exposed at Kjerringvik in the Oslo fjord in Norway, at a dedicated marine test area about 20 m from the sea. The corrosivity at the site was measured according to ISO 9226 [19] and found to be within category C5, with an average corrosion of $137 \pm 9 \mu\text{m}$ during first year exposure. The samples received a horizontal coating scribe down to bare steel with dimensions 2 × 50 mm before exposure. The samples were exposed facing towards the sea (south direction), tilted about 45° from vertical position. Samples were retrieved after 1 and 2 years exposure.

2.3. Accelerated testing

2.3.1. ISO 9227 salt spray test

The test was performed according to the standard [2] with a duration of 1440 h (60 days). Test electrolyte was 50 g/l NaCl solution at pH between 6.5 and 7.2.

2.3.2. ISO 12944-9 Annex B cyclic ageing test

The test was performed according to the standard [15] with a

Table 1

Coating systems (NDFT = nominal dry film thickness). All film thicknesses in μm . Expected lifetime in years according to ISO 12944-5 [18].

System	1st coat	2nd coat	3rd coat	Total NDFT	Expected lifetime
1	Epoxy mastic A	250		250	7–15
2	Epoxy mastic A	125	Epoxy mastic A	125	7–15
3	Epoxy mastic A	190		190	7–15
4	Epoxy mastic A	155	Epoxy mastic A	155	> 25
5	Epoxy mastic B	250		250	7–15
6	Epoxy mastic B	125	Epoxy mastic B	125	7–15
7	Epoxy mastic B	190		190	7–15
8	Epoxy mastic B	155	Epoxy mastic B	155	> 25
9	Epoxy mastic C	250		250	7–15
10	Epoxy mastic C	125	Epoxy mastic C	125	7–15
11	Epoxy mastic C	190		190	7–15
12	Epoxy mastic C	155	Epoxy mastic C	155	> 25
13	Epoxy mastic D	250		250	7–15
14	Epoxy mastic D	125	Epoxy mastic D	125	7–15
15	Epoxy mastic D	190		190	7–15
16	Epoxy mastic D	155	Epoxy mastic D	155	> 25
17	Zn-Epoxy A	60	Epoxy A	200	> 25
18	Zn-Epoxy A	60	Epoxy B	200	> 25
19	Zn-Epoxy A	60	Epoxy C	200	> 25
20	Zn-Epoxy A	60	Epoxy mastic A	200	> 25
21	Zn-Epoxy A	60	Epoxy mastic D	200	> 25
22	Zn-Epoxy B	60	Epoxy A	200	> 25
23	Zn-Epoxy B	60	Epoxy B	200	> 25
24	Zn-Epoxy B	60	Epoxy C	200	> 25
25	Zn-Epoxy B	60	Epoxy mastic A	200	> 25
26	Zn-Epoxy B	60	Epoxy mastic D	200	> 25

Table 2
Results from the various tests for each coating system.

	NDFT µm	ISO 9227 mm		ISO 12944-9 mm		EIS MΩcm ²	1 year in C5 mm	2 years in C5 mm	
1	250	*0.2	± 0.04	*1.5	± 0.19	0,04	*1.8	*3.4	± 0.07
2	250	0.4	± 0.90	2.5	± 0.41	640	1.7	2.4	± 0.21
3	240	1.0	± 0.37	2.7	± 0.25	41	1.2	1.6	± 0.21
4	360	1.1	± 0.18	3.6	± 1.14	2800	1.4	2.1	± 0.07
5	250	*1.3	± 0.16	*1.5	± 0.76	8	1.1	1.5	± 0.07
6	250	0.5	± 0.16	1.7	± 0.21	390	1.1	1.5	± 0.21
7	240	1.2	± 0.06	4.2	± 0.45	1400	0.9	1.0	± 0.21
8	360	1.4	± 0.03	3.0	± 1.08	210	0.7	0.8	± 0.57
9	250	0.6	± 0.30	4.0	± 0.50	220	1.4	2.1	± 0.35
10	250	0.2	± 0.14	4.9	± 0.31	680	2.6	4.0	± 1.27
11	240	1.0	± 0.20	4.2	± 0.50	2900	0.7	1.5	± 1.06
12	360	1.0	± 0.10	4.4	± 0.12	5600	0.9	2.3	± 0.07
13	250	0.5	± 0.22	4.5	± 0.35	11,000	0.6	1.1	± 0.07
14	250	0.6	± 0.18	4.4	± 0.15	15,000	0.8	1.3	± 0.21
15	240	1.4	± 0.14	3.2	± 0.40	25,000	0.6	0.9	± 0.57
16	360	1.3	± 0.06	4.6	± 0.36	23,000	0.9	1.5	± 0.64
17	320	1.0	± 0.32	3.6	± 0.10	9,6		0.1	
18	320	0.5	± 0.30	3.3	± 0.33	7000		0.3	
19	320	0.6	± 0.12	2.6	± 0.35			0.2	
20	320	0.4	± 0.21	2.5	± 0.36	11,000		0.1	
21	320	0.5	± 0.31	3.0	± 0.33	8900		0.4	
22	320	0.6	± 0.26	2.8	± 0.42	3900		0.1	
23	320	0.9	± 0.20	2.7	± 0.14	5600		0.3	
24	320	1.0	± 0.15	2.4	± 0.15			0.1	
25	320	0.5	± 0.26	2.3	± 0.30	8600		0.5	
26	320	0.7	± 0.31	2.3	± 0.10	38,000		0.4	

* The coating was degraded by blistering and rusting independent of the scribe

duration of 4200 h, i.e. 25 weeks. Test electrolyte was 50 g/l NaCl solution at pH between 6.5 and 7.2.

2.3.3. ISO 16773-2 electrochemical impedance spectroscopy on high impedance coated specimens

EIS was obtained on one sample of each system from the 2 years field test. An electrochemical cell with area 14.6 cm² was attached to the specimens and filled with a 50 g/l NaCl solution. The spectra were obtained between 100 kHz and 10 mHz 24 h after the cell was filled with electrolyte. The spectra were fitted to a Randle's circuit and pore resistances are reported [20].

2.4. Evaluation of corrosion creep

Corrosion creep was evaluated according to ISO 4628-8 [21] after gentle removal of delaminated coating with a knife. Corroded distance was measured with a stereo microscope. Average corrosion creep was calculated from nine equidistant measurements on each side of the scribe.

2.5. Visual assessment and pull-off adhesion

All coating systems were evaluated after exposure by visual assessment as per ISO 4628-2, ISO 4628-3, ISO 4628-4, and ISO 4628-5. Pull-off adhesion testing was performed as per ISO 4624.

3. Results

3.1. C5 field test results

Only one coating system (system 1) showed any visual degradation. Further, there was no significant variation in pull-off adhesion results for all systems tested. Hence, for all systems tested (except system 1) corrosion creep was the only significant discriminator. Fig. 1 shows pictures of coating systems no. 1 and 16 after 2 years in the field test, exemplifying the appearance of the coatings after the test.

Corrosion creep measured on the 26 coating systems in the field test

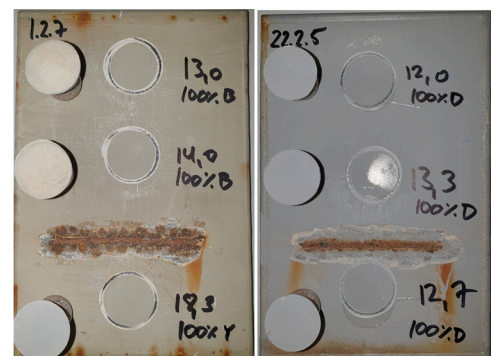


Fig. 1. Appearance of Coating systems no. 1 and 16 after 2 years C5 field test.

after 2 years exposure is shown in Fig. 2. The figure shows a very strong beneficial effect of using zinc rich primers. After 2 years exposure in a C5 environment, the ten coating systems with zinc rich primer have only

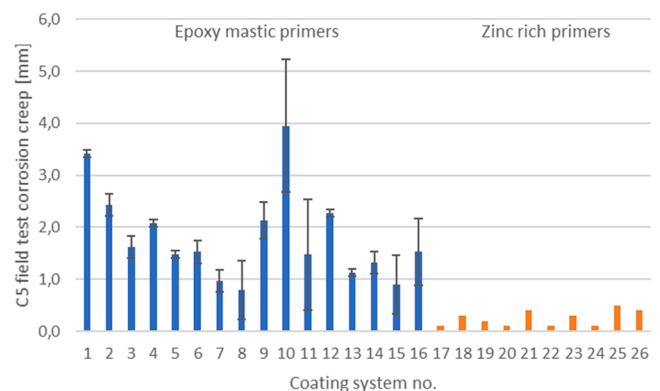


Fig. 2. Corrosion creep after 2 years for the various coating systems in the C5 field exposure test.

between 0.1 and 0.4 mm average corrosion creep along the scribe. There was no statistically significant difference between the two zinc rich primers. The samples with epoxy mastic primers had 1–4 mm corrosion creep, i.e. about ten times more corrosion creep than the systems with zinc rich primers.

For the samples without zinc rich primers, there were differences both between the four products and the applied systems. The coating systems based on Epoxy mastic B and D both had average corrosion creep of 1.2 mm, while the systems based on Epoxy mastic A and C both had average corrosion creep of 2.4 mm. For the systems with zinc rich primers, the epoxy barrier coat showed no effect on the result.

The samples were only exposed for 2 years. This is short time compared to the expected lifetime of the coating systems in a C5 environment. In ISO 12944-5 [18], comparable systems with a zinc rich primer (zinc rich primer, 3–4 coats, 320 μm NDFT) are expected to have "very high" durability, i.e. give more than 25 years lifetime. The same standard estimates that coating systems without zinc rich primer and NDFT 240–300 μm will have "medium lifetime", i.e. 7 – 15 years. Systems no. 4, 8, 12 and 16 had NDFT of 360 μm , which according to the standard also should give "very high" lifetime. Judging from the corrosion creep results in this test, these systems are not comparable to the systems with zinc rich primers with respect to corrosion creep. However, corrosion creep was rather slow, even for the coating systems without zinc rich primers, so this property may not be the parameter that determines coating lifetime. A precondition for corrosion creep is initiation of corrosion on the painted steel. This can be from a cut edge, a mechanical damage, or coating degradation by blistering, rust penetration, or cracking. The latter three degradation mechanisms depend on the ability of the coating to act as a barrier against ions [22], and may be independent of the presence of a zinc rich primer. Coating system 1 was degraded by blistering and rusting independent of the scribe, which affected a much larger area of the painted surface than the corrosion creep, and therefore must be regarded as much more severe.

In Fig. 3, corrosion creep results in the C5 field test after 2 years are plotted against results after 1 year. Only the samples without zinc rich primers were evaluated after 1 year. The figure shows strong correlation between corrosion creep after 1 and 2 years, with R^2 of 0.84. This corresponds well with the investigation of Reuterswård and Tidblad, showing good correlation between 19 and 105 months field performance. The slope of the linear regression line in Fig. 3 is 1.5, which means that corrosion creep the second year was 50% lower than the first year. Hence, corrosion creep seems to decrease with time. This is reasonable, since the degraded coating still may provide some protection until it physically falls off the sample. Rust is also known to provide corrosion protection, as described in ISO 9224 [23], which may reduce corrosion creep.

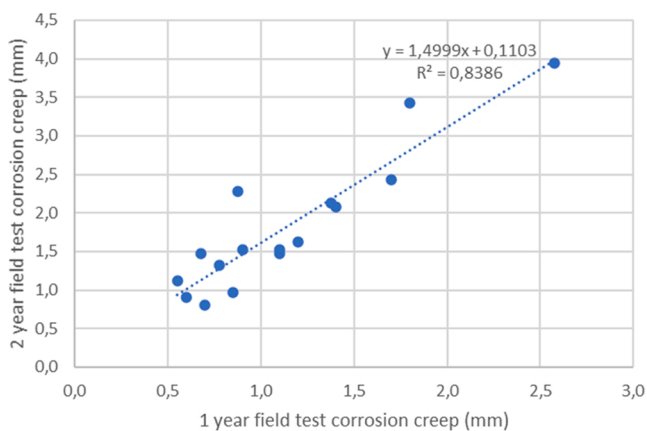


Fig. 3. Corrosion creep results in the C5 field exposure test. Results after 2 years plotted as function of results after 1 year.

3.2. Correlation between C5 field test and salt spray test

Since the zinc rich primers had a large effect on the field test result, correlation between field test result and accelerated testing is calculated separately for systems with and without zinc rich primers.

Correlation between corrosion creep in the field test and the salt spray test is shown in Fig. 4. The samples with epoxy mastic primers show a negative correlation between salt spray test and field performance, i.e. coating systems with the lowest corrosion creep after laboratory testing showed the highest corrosion creep after field testing. The Pearson correlation coefficient (Table 3) showed a strong correlation of -0.7 . For coating systems with zinc rich primers, there is also a negative correlation, but the linear regression only explains a small fraction of the variation ($R^2 = 0,13$) and the Pearson correlation coefficient is only -0.36 . When treating all the coating systems as one single data set, the Pearson correlation coefficient is reduced to -0.23 .

Corrosion creep in the 1440 h salt spray test was generally low, and all samples had less than 1.5 mm creep, compared to up to 4 mm in the field test. This may be too little to distinguish between the various coatings. On the other hand, Systems 1 and 5 both blistered in the salt spray test, while System 1 blistered in the field test, so the salt spray test at least partly predicted this behaviour.

3.3. Correlation between C5 field test and cyclic ageing test

When considering the corrosion creep from the artificially induced damage, the cyclic ageing test showed no correlation to the field test, neither for the systems with epoxy mastic primers nor the systems with zinc rich primers, see Fig. 5. The Pearson correlation coefficients were below 0.24 (Table 3), which is considered as no correlation. The linear regressions also indicate that there are no correlations. In the cyclic ageing test, corrosion creep values from 1.5 mm to 5 mm were found, which is in the same range as in the field test. In the field test, the zinc rich primers almost eliminated corrosion creep, but this was not the case in the cyclic ageing test where creep results varied between 2.3 and 3.6 mm, i.e. about the same range as the systems with epoxy primers. The average acceleration factor (lab test result divided by field test result) for coating systems with epoxy primers was 2.3, while for systems with zinc rich primers it was 16. Hence, the accelerated test was unable to replicate the beneficial effect of the zinc rich primers in the field test.

3.4. Correlation between C5 field test and coating impedance

Correlation between EIS results and the corrosion creep after field test is shown in Fig. 6. Fig. 7 shows the impedance spectrum and the fitted Randal's circuit for coating system 1, 3 and 4 after the 2 years field

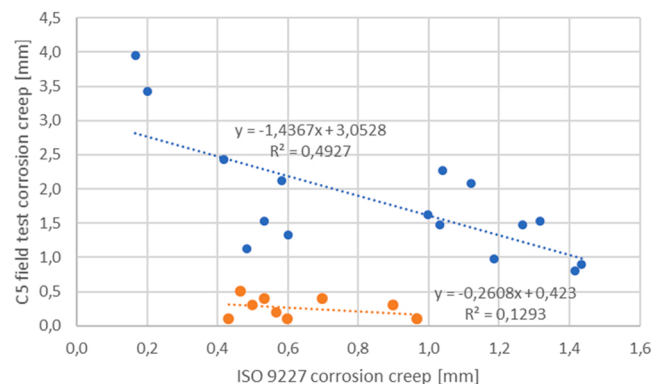


Fig. 4. Corrosion creep in the C5 field test as function of corrosion creep in the ISO 9227 salt spray test. The blue points are the epoxy mastic based coating systems (system 1–16), while the orange dots are the coating systems with zinc rich primers.

Table 3

Pearson correlation coefficients between laboratory tests and 2 years C5 field test.

	2 years C5		
	Epoxy mastic primers	Zinc rich primers	All systems
1 year C5	0,92		
ISO 9227	-0,70	-0,36	-0,23
ISO 12944-9	-0,02	-0,24	0,24
ISO 16773	-0,37	0,42	-0,33

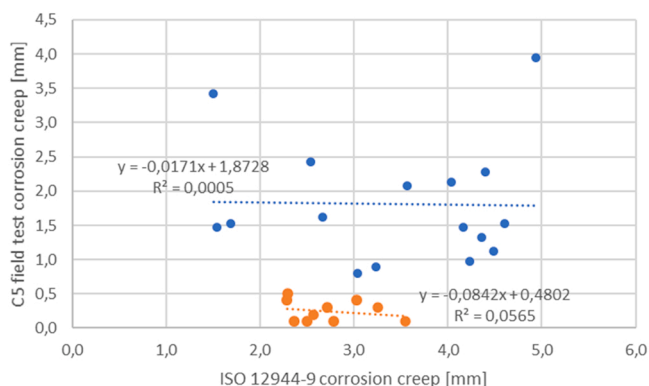


Fig. 5. Corrosion creep in the C5 field test as function of corrosion creep in the ISO 12944-9 cyclic ageing test. Orange dots are the coating systems with zinc rich primers.

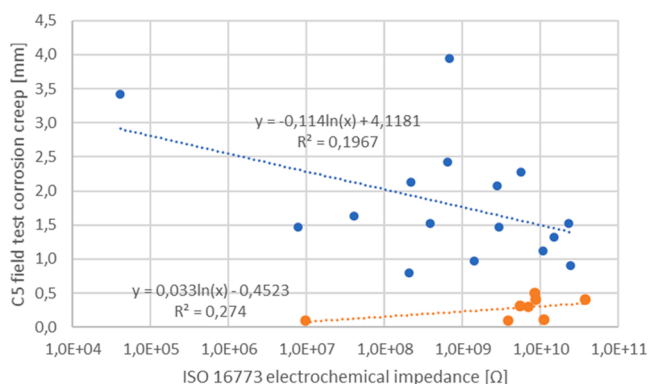


Fig. 6. Corrosion creep in the C5 field test as function of pore resistances (Randle's circuit) according to ISO 16773-2. Orange dots are the coating systems with zinc rich primers.

test, as an example. The figure indicates a weak negative correlation between corrosion creep and impedance for the systems with epoxy mastic primers, i.e. that higher impedance gives less corrosion creep. The figure indicates the opposite trend for systems with zinc rich primers. The Pearson correlation coefficients are in the order of 0.4, which means that the correlation is weak and may be incidental. This is as expected since the impedance is a measure of barrier properties, and corrosion creep is a measure of in plane transport of ions.

However, there is correlation between impedance and blistering in the test. The blistering is due to ions penetrating the paint film, initiating electrochemical reactions at the steel-paint interface [22]. System 1 had only $4 \cdot 10^4$ Ohm cm^2 impedance after the two years field test, which is due to the blistering and rusting that had opened direct electrolytic contact to the steel substrate. System 5 also had rather low impedance, $5 \cdot 10^6$ Ohm cm^2 , and blistered in the salt spray test but not in the field test. Impedance in this range is however associated with early coating failure [24]. Hence, impedance measurements may be useful to predict

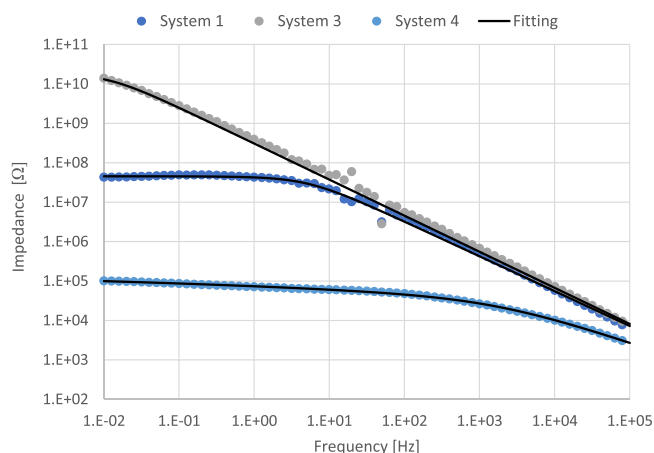


Fig. 7. EIS data of systems 1, 3 and 4 after 2 years in field, exemplifying representative impedance data, along with an equivalent circuit (Randle's circuit) fitted to the data.

coating failure by blistering or rusting, i.e. degradation that is caused by ions penetrating the film.

4. Discussion

The field test results show that epoxy based coating systems, and systems with zinc rich primers in particular, have good resistance against corrosion creep from an artificial damage. A precondition is that the steel preparation, blast cleaning and paint application are all according to specification. Considering the corrosion creep rate after 2 years, even the systems with the most corrosion creep in the test will have an annual corrosion creep rate up to 1 mm/year. For the systems with zinc rich primers, corrosion creep rates will be up to 0.2 mm/year. For a steel construction with 20–30 years design life, corrosion creep from a damage in the coating therefore seems to have a limited effect on the degradation of epoxy based protective coatings. Other degradation mechanisms will be determining coating lifetime in corrosive atmospheres, as also indicated in ISO 12944-1 [25].

Negative correlation was found between corrosion creep in the salt spray test and the C5 field test. The salt spray test has previously been heavily criticized for having little correlation to field performance, which is supported by the results in this test program. The driving mechanism for coating failure around the scribe in the test is cathodic disbonding, which differs from the field where both cathodic disbonding and anodic undermining seems to contribute [8,10]. However, some correlation was found between the salt spray test and the field test with respect to blistering. Samples that blistered in the salt spray test also blistered in the field test. Thus, the test seems to be able to evaluate barrier properties to some degree.

In this study there was no significant correlation between corrosion creep after the cyclic ageing test and the C5 field test, but certain correlation has been reported before [8,12]. The difference between this study and the previous work is probably that a wider range of coatings and surface cleaning methods were applied, so that coating systems with rather poor performance were included. In this study, all samples were prepared as described in ISO 12944-6, ISO 12944-9 and NORSOK M-501 ed. 6 [26] and had quite good resistance against corrosion creep.

In the C5 field test there was a notable difference in corrosion creep between systems with and without zinc rich primer, but this was not reflected in the cyclic ageing test. As stated above, the acceleration factor in the ISO 12944-9 test for coating systems with zinc rich primers was 16, but only 2.3 for systems without zinc rich primers. The ageing test seems to overload the protective capacity of the zinc rich primer, which indicates that the test is too aggressive and too accelerated. Given that the test already takes 6 months to perform, reducing the

aggressivity in the test and extending the test duration is probably not acceptable to the users of the test, though. Proposing a new test is beyond the scope of this work.

Both the salt spray test and the cyclic ageing test show poor correlation to C5 field performance with respect to corrosion creep. In addition, there was little corrosion creep in the field test for all the coating systems investigated, and for the systems with zinc rich primers in particular. Little scribe creep for systems with zinc rich primers was also found by Reuterswård and Tidblad in their 105 months field test, indicating that this result will not change with prolonged exposure. It is therefore reasonable to ask why corrosion creep is so much in focus in accelerated testing of paint, e.g. in ISO 12944 parts 6 and 9. A review study of coating failure incidents in the Norwegian offshore oil and gas industry showed that corrosion creep on steel did not contribute significantly to the failures [27].

Correlation between impedance and C5 field test corrosion creep was weak or incidental. However, the two systems with the lowest impedance had rust penetrating the film in some or all the other tests. EIS measures barrier against ions and will therefore correlate well with all degradation that is caused by ionic penetration, i.e. rust penetration and blistering [22,24,28], which is regarded to pose a greater threat to the general condition of the corrosion protective coating system than corrosion creep from a damage.

5. Conclusions

- In the C5 field test, long term corrosion creep rates for systems with zinc rich primers were in the order of 0.2 mm/year, while for the epoxy mastics creep rates of about 1 mm/year were found. Hence, the focus on this property in pre-qualification testing in NORSOK M-501 and ISO 12944 is not justified by the results presented here. Corrosion creep does not seem to limit coating lifetime, given that surface profile, cleaning, and coating application are properly performed and a suitable coating system is selected.
- The poor correlation between the laboratory test methods and field testing with respect to corrosion creep further weakens the relevance of the accelerated tests. Also, the accelerated tests were unable to replicate the difference between systems with and without zinc rich primers in the field test.
- There was a strong correlation between results after 1 and 2 years in the field test. Hence, a relatively short field test seems to predict performance in the longer run.

CRedit authorship contribution statement

O.Ø. Knudsen: Funding acquisition, Data curation, Visualization, Writing – original draft. **A.W.B. Skilbred:** Supervision, Project administration, Conceptualization, Methodology, Investigation, Writing – review & editing. **A. Løken:** Methodology, Investigation, Writing – review & editing. **B. Daneshian:** Formal analysis, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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