LONG-TERM TESTING OF HIGH PERFORMANCE CORROSION INHIBITING SPRAYABLE THERMOPLASTICS (CIST)

ABSTRACT

CIST employs an active corrosion-inhibiting barrier to arrest corrosion within an entire assembly by isolating it from oxygen and moisture. Its unique methodology has different testing requirements from those which apply to typical coating systems, which are mainly designed for passive coatings that bond directly to the substrate.

This paper brings together a range of tests relating to the performance of CIST applications during prolonged exposure to elevated temperatures and high salt concentrations in atmospheric and laboratory conditions. The tests are designed to replicate the most extreme natural conditions as well as measuring results against internationally recognised standards. The paper includes data and results from tests conducted between 2012 and 2014 and examines the technical background to the success of this type of coating in preventing and arresting corrosion in marine environments on complex substrates that may not respond to more traditional anti-corrosion solutions.

INTRODUCTION

Using Coatings to Prevent and Arrest Corrosion in Complex Substrates

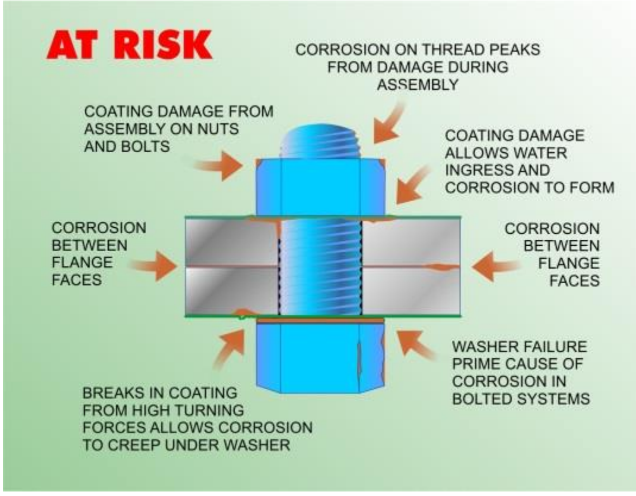
In previous papers we have discussed the problems associated with ageing infrastructure and the effects of salt, water and oxygen on steel in marine environments. Corrosion is a problem wherever steel is exposed to the atmosphere, but it is in marine environments that the problems are most acute and where effective remedies are hard to find. So a solution which can be applied successfully in marine and offshore conditions is a worthwhile objective for any coatings company.

There are two aspects to the problem: 1) prevention and 2) cure. How can corrosion be prevented in new substrates and what can be done once corrosion is present.

Corrosion in steel is an electrochemical process that oxidises the steel in the atmosphere - and most coatings are simply designed to protect the steel by providing an impervious barrier between it and the effects of the moisture and oxygen. Additional protection may be provided by metallic coatings such as zinc, which also provide a measure of galvanic protection but, essentially, as long as there is a firmly adherent impervious coating on all surfaces, there will be no corrosion.

Unfortunately, even for new equipment, coating problems arise in many ways. Coating choice is often inappropriate with single-pack light-duty coatings on components failing in weeks or, even if the coating is well-chosen and well-applied, damage during assembly can leave bolt threads and nut edges devoid of protection and over-compressed coatings cracked and compromised.

Fig 1 - coating damage during assembly



So, even for new build, a bolted assembly can be vulnerable and, when these vulnerabilities lead to corrosion, how can an effective remedy be applied?

Without complete disassembly, some corrosion will remain between bolts, washers and substrate and, in the case of flanges, between the flange faces. No amount of blasting and cleaning will remove corrosion from these areas. So, painting will always be over-coating some rust, which will soon reappear.

Fig 2 - Over-painting covering rust

Other factors also have an impact on corrosion in bolted assemblies, particularly galvanic effects where, for example, a carbon-steel bolt in a stainless steel flange will become a sacrificial anode and may completely fail within 18 months - even if protected with zinc and PTFE.

Fig 3 - PTFE/zinc coated bolt failure after 18 months in the splash zone

Fig 4 - rusting carbon bolts in stainless substrate

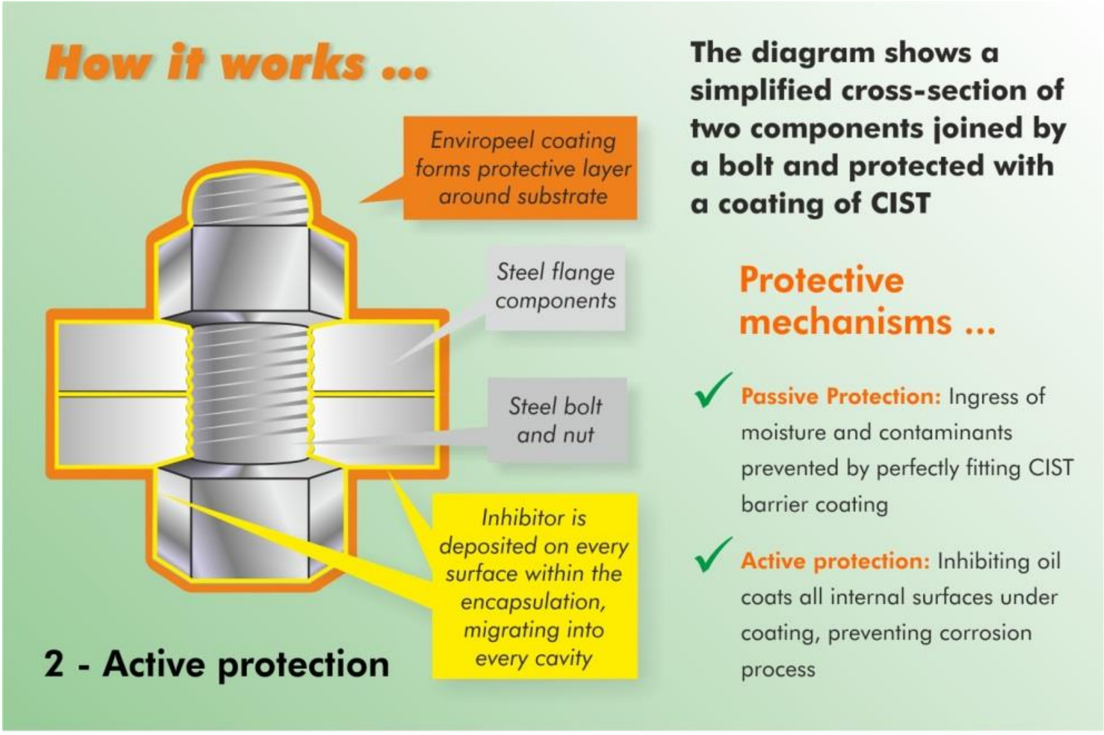
PROTECTING THE ENTIRE ASSEMBLY

Rusting platforms and marine infrastructures across the globe are a witness to the failure of protective and remedial systems in bolted structures. Individually addressing all the problems inherent to bolted assemblies has led to the introduction of many partial solutions such as bolt caps, flange protectors, tapes and caulking compounds - which may work individually but still allow unprotected areas to fail.

A more suitable approach is to deal with the assembly in its entirety. By protecting the whole, each individual component is also protected. We have all heard the expression 'divided we fall, united we stand' and this as true for bolted assemblies as it is for labour movements, nation builders and the military.

CIST is designed to address each individual problem within a bolted system by protecting the whole assembly - not only with an impervious barrier but also by actively supressing galvanic processes and crevice effects - to prevent the start and arrest all existing corrosion within its protection zone.

Fig 5 - How CIST works



Although fourteen years of case studies attest to the success of this strategy ...

Fig 6 - Offshore platform and mixed metal substrate after 7 years

Fig 8 - BHP Billiton data

DATA FROM BHP BILLITON & RIO TINTO IN AUSTRALIA

On stored conveyor pulleys

• Return for replacement without Enviropeel	**44.5%**
• Failure rate with full Enviropeel protection	**0%**

On operational pulleys

No bearing has failed since Enviropeel applied, previous average life only 9 months!

• Average bearing life in original location:	**9 months**
• Current bearing life in original location with Enviropeel applied:	**48+ months**
• Resulting component life increase:	**500+%**
• Resulting saving in pulley changeout costs:	**500+ %**
• Reduction in maintenance costs:	**95 %**
• Percentage of Enviropeel costs to rebuild costs:	**10-15 %**
• Percentage of Enviropeel costs to pulley change out costs:	**5-7 %**
• Resulting percentage reduction in risk exposure:	**90+ %**
• Anticipated increase in component lifetime:	**500 %**

... we live in a world of bean counting and box ticking, so we also have to provide verifiable test data for those who have a more vicarious view of the world.

TICKING ALL THE BOXES

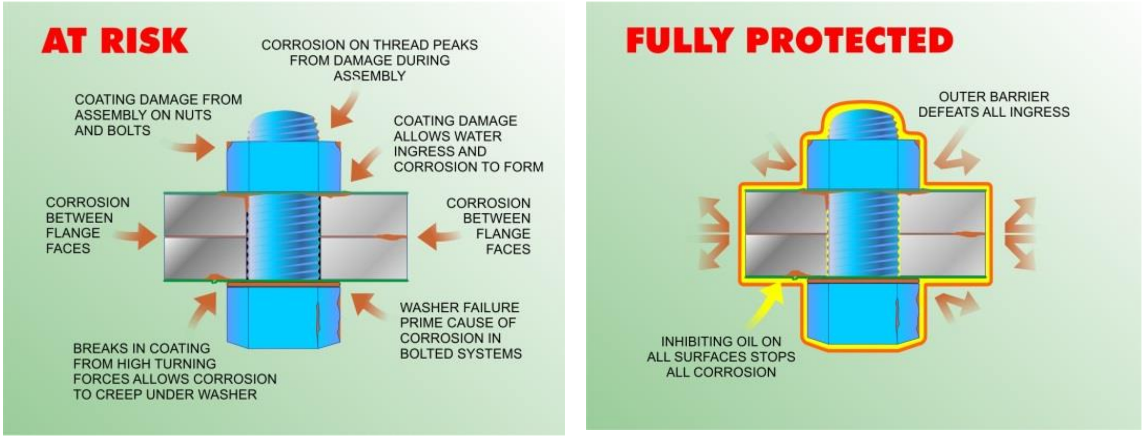
Before looking at testing an anti-corrosion system, let's review the problems that occur in bolted systems in marine and offshore environments:

Figure 9 - Causes and symptoms of bolting failure

	Cause	Symptom
1	Damage to nuts and bolt heads during assembly	Moisture penetration of coating causing corrosion and eventual coating failure
2	Damage to bolt thread coating during assembly	Moisture in bolt cavity corrodes threads leading to bolt seizure and/or loss of tension
3	Coating damage due to compression and/or rotation	If coating is cracked or adhesion to substrate is compromised, corrosion between components, particularly in washers and around bolt site in substrate leads to rapid coating failure
4	Crevice Corrosion	An assembly is fill of edges, surfaces and cavities, crevice corrosion in any of these areas is common, compromising the substrate
5	Galvanic effects #1	Once a coating is compromised, galvanic effects between dissimilar metals accelerates corrosion
6	Galvanic effects #2	Small components in any large structure, but particularly carbon steel bolts in stainless steel substrates will act as sacrificial anodes, rapidly deteriorating and failing

If we examine these symptoms in the context of the diagram in figure 5, it is easy to see how a CIST application resolves all these problems.

Figure 10 - CIST resolves all bolted assembly corrosion problems



We have already established that CIST has an enviable track record with more than 14 years of successful protection and documented examples of failure rates reduced to zero and greatly increased component lifetimes - but how does the product measure up in the lab?
Tests were applied to the CIST product that is featured in the case study examples mentioned above. This particular product, Enviropeel, is the only product of its type that has a sufficiently long performance record to effectively compare real-world examples with laboratory simulations and the key tests listed below are in response to the most frequently asked questions in relation to the material's performance:

1. How does the product perform in marine and high UV environments?
2. How long will it last?
3. How long before the inhibitor runs out?
4. How impervious to water is the material?

The list below itemises some key tests and results before we go on to look at how the testing was accomplished.

Fig 11 - Key Tests

	Test Standard	Description	Result
1	ASTM B117 Hot salt fog	3000 hr exposure	No corrosion
2	ASTM B117 Hot salt fog	1000 hr on partially protected substrate	Heavy corrosion in exposed areas No corrosion in protected areas
3	ASTM B117 Hot salt fog	4500 hr exposure	No corrosion
4	ASTM B117 Hot salt fog	7500 hr exposure	No corrosion
5	Modified ASTM G154	4000 hr accelerated weathering/UVA exposure	Minor colour changes No surface degradation
6	Long-term deluge test	Continuous salt-water deluge test - 8600 hour interim results	No corrosion
7	Long-term inhibitor loss	Weight loss after 12 month cycling temperature exposure	Less than 1% weight loss

Q1: How does the product perform in marine and high UV environments?

ACCELERATED WEATHERING

The most common weathering test is ASTM G154, in which samples are exposed to 42 cycles of 8 hours of UVA-340 ultra-violet light at a temperature of 60°C followed by 4 hours of condensation at 50°C for a total of 21 days of exposure. Like many other standard tests, this may provide a benchmark for comparison but does it has very little to say about what will happen in the real world. Other tests such as ASTM 4587 and G 155 provide for a similar combination of UV irradiation, heat and water while ASTM B117 focuses on a combination of water salt and heat. Both tests are widely accepted in assessing suitability of materials for outdoor use but, for example, a 1000 hrs of UVA exposure is only the equivalent of 1 year in South Florida and a bolt that has passed a 1000 hrs hot salt fog test may be completely unserviceable within 18 months in some environments.

Fig 12 - Bolt corrosion after 18 months offshore North Sea

So, in order to provide some real-world equivalency, the scale of the tests for CIST was revised to be significantly longer and under more extreme conditions. For the weathering test, it was decided that the rapid half-daily cycles of wet and dry specified under ASTM G154 were not representative of natural weather cycles and a regime was implemented that allowed much longer periods of exposure to each element of the test. The cycles were between UV exposure and hot salt fog (HSF) exposure and the test was to run for 7 months.

The UV exposure is via 4 x 15w 450nm UVA tubes arranged to give continuous 360° exposure at while the HSF element employed standard ASTM B117 parameters with 5% salinity, fog temperature 47°C and chamber temperature 35°C.

Fig 13 - Exposure schedule

HOUR COUNTER	MONTH	WEEK 1	WEEK 2	WEEK 3	WEEK 4
672	1	UV	UV	HSF	UV
1344	2	UV	HSF	UV	UV
2016	3	HSF	UV	UV	HSF
2688	4	UV	UV	HSF	UV
3360	5	UV	HSF	UV	UV
4032	6	HSF	UV	UV	HSF
4704	7	UV	UV	HSF	UV

Indicates the samples in UV Cabinet

Indicates the samples in HSF Cabinet

Fig 14 - Exposure parameters

HSF PARAMETER			
Salinity, IN	5%	Salinity, OUT	5%
pH		Fog Collectors Data	
pH in	7.2	Fog Collector 1	2.0 ml/hr/80cm²
pH out	6.5	Fog Collector 2	1.8 ml/hr/80cm²
Temperature		Pressure	
Saturated Air Temp	47°C	Chamber Pressure	1 bar
Chamber Temp	35°C	Air Intake Pressure	0.9 MPa
Hygrometer		Downtime	
Wet Bulb	35°C	From	-
Dry Bulb	35°C	To	-
UV PARAMETER			
UV LAMP NO	4	PLACEMENT	360 DEGREE
WAVELENGTH		15W/ 450nm	
HEATER NO	2	AVERAGE TEMPERATIRE	45 DEGREE C

The samples were prepared using carbon steel test pieces, blasted and coated with a single coat of epoxy and fastened using standard nickel-plated B7 bolts. They were then coated with CIST using 2 coats at 2mm per coat, with edges sealed using standard cable ties and the pipe ends left unprotected.

Fig 15 - sample preparation

The test pieces were then entered into the UV cabinet to initiate their exposure. Two samples were exposed to the weathering process and in different orientations to allow interim removal and expose all areas, testing for ingress as well as weathering. A number of small buff-coloured CIST coated bolts were included to allow interim removals as required.

Fig 16 - Samples in UV Cabinet at start of test

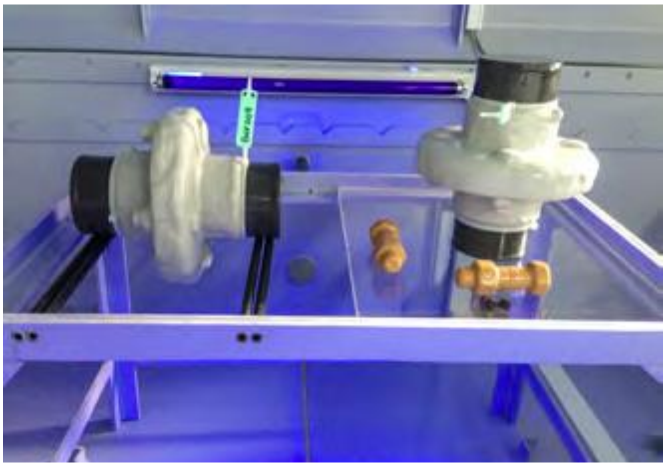


Fig 17 - Samples at 336 hours in hot salt fog cabinet

Fig 18 - Sample at 2016 hours in UV cabinet

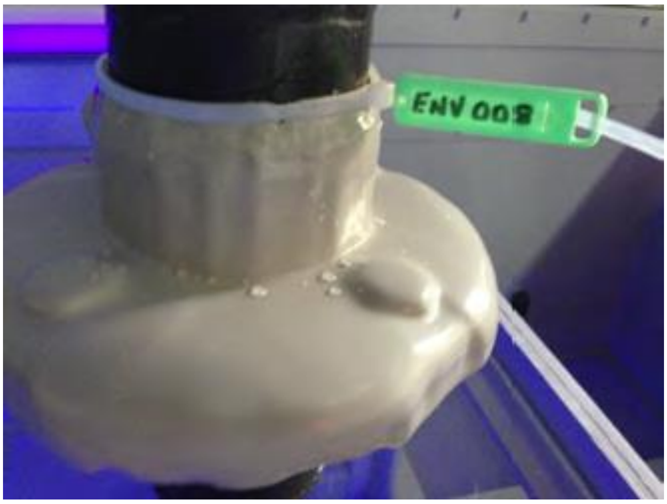


Fig 19 - Sample at 2520 hours back in hot salt fog

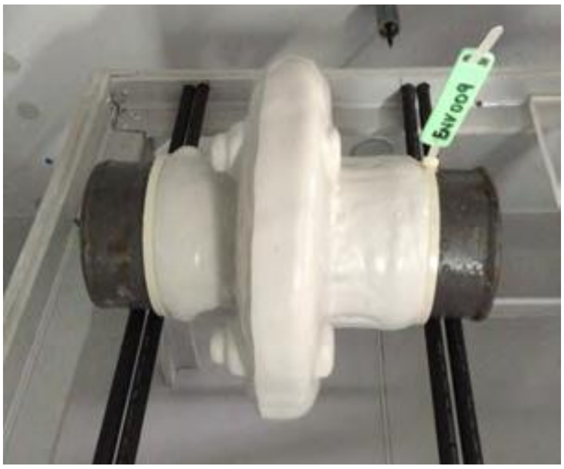
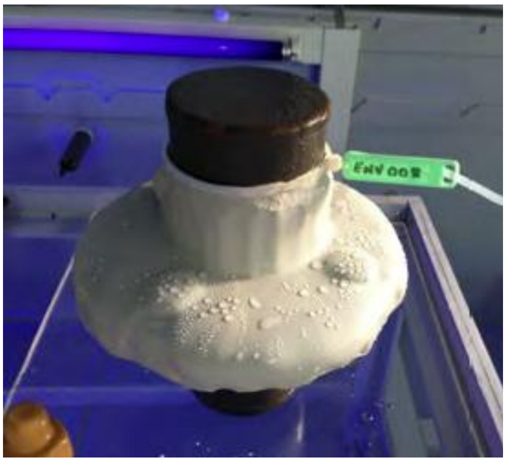


Fig 20 - sample at 4500 hours



Initially the test was set to last 4500 hours (6 months) but, as the number of weeks increased, it became increasingly obvious that a longer period was needed as there was no discernible effect on the CIST.
It was decided to leave the samples in for another six months and just remove one of the small bolts to assess the changes so far. The material was cut from the bolt to see if there had been any changes either to the bolt or the CIST coating.

The bolt itself displayed no signs of deterioration, and although the exterior of the CIST coating was found to be relatively free of inhibiting oil, inside the encapsulation inhibitors were evident on all surfaces.

Visual examination of the surface showed slight fading, about 5% compared with a reference sample and microscopic analysis of the surface of the coating showed no deterioration of the surface structure. Analysis of a section through the coating confirmed a slight oil loss at the surface but showed that oil was uniformly present throughout the coating.

Fig 21 - part of the removed coating showing the inside and outside of the sample after 4500 hours exposure

Fig 22 - comparison with a reference sample shows slight colour loss after 4500 hours

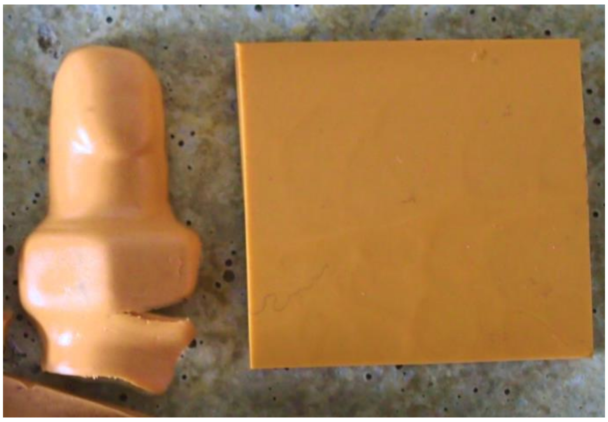


Fig 23 - close examination showed no crazing or cracking of the surface

Fig 24 - Cross section of cut CIST shows slight drying at outer edge

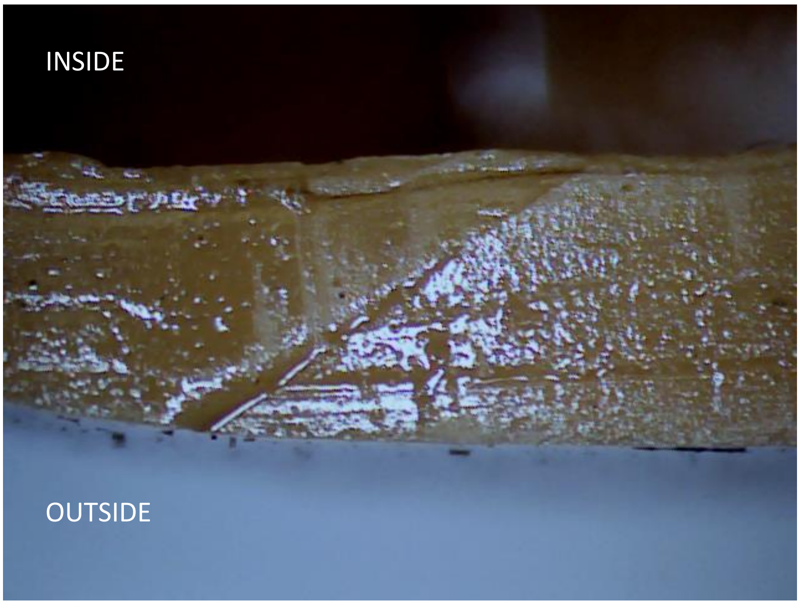
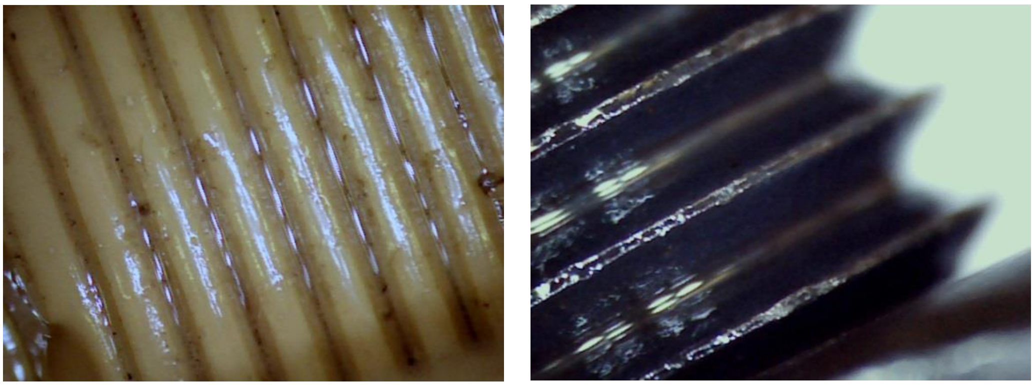


Fig 25 - Bolt thread and removed coating show good levels of inhibiting oil



It was concluded that the accelerated weathering cycle over 6 months had no detrimental effect on the CIST encapsulation and that all performance criteria remained at 100%. Further tests will be carried out after 12 months exposure.

Q2: How long will it last?

ASTM B117 HOT SALT FOG TESTING

This test is not applicable for all coating types. You will see comparison tests by bolt manufacturers, for example, favourably contrasting the longevity of their proprietary coating in a hot salt fog (HSF) with that of galvanised bolts. Since everybody knows that galvanised steel bolts perform very well, we are supposed to infer that this manufacturer's bolt coating must be superior. However, what these comparisons fail to mention is that the protective mechanism for galvanising requires a wet/dry cycle for it to work. As the HSF test is continuously wet, the galvanising fails much more quickly than it would in a normal weather cycle where wet and dry periods are the norm. Hence the cyclic test described above. Nevertheless, survival for long periods in a HSF is a good indication that a coating is providing good protection for the substrate - but how long should the test be - and how long should it last? 1000, 2000 and 3000 hour tests are quite commonly quoted in bolting specifications but, if a 1000-hour bolt is lasting 18 months, does this mean a 3000-hour bolt is good for 4 or 5 years?

Most authorities agree that there can be no extrapolation of an HSF test to correlate with a defined number of years - but they will agree that the longer the test, the more likely it is that a successful

outcome will be achieved in the real world. So it was decided that an unprecedented 9000 hours (over 12 months) of continuous HFS testing would go a long way to showing what CIST could achieve.
The most recent milestone in this test was the removal after 7500 hours of a second test piece after the successful examination of a sample removed at 4500 hours. In both cases, while the uncoated control piece displayed increasing levels of deterioration, the CIST samples were in excellent condition. No corrosion or deterioration of any kind could be detected within the CIST encapsulation, bolts, nuts and flange were in perfect condition.

Fig 26 - Side-by-side comparison of control (left) with 4500-hour sample (middle) and 7500-hour sample (right). The corrosion at the top and base of the samples is on areas outside the CIST encapsulation

Figure 27 - Comparison of bolts from below and above

Every indication is that the same result will be found after 1000 hours.

Q3: How long before the inhibitor runs out?

CIST is an active coating, the deposition of the inhibiting oil on to the substrate and the ability of this inhibition process to prevent corrosion are key elements in the overall corrosion protection package so, if this deposition is a continuous process, how long can the coating maintain its performance?
In fact, the process by which inhibiting oil is maintained on the encapsulated substrate is more akin to the water cycle that governs the world's rainfall than it is to a constantly pouring tap. On application, the hot material deposits a thin film of oil between the coating of CIST and the substrate. On cooling, some of this is reabsorbed into the CIST and, as the substrate surface and ambient temperatures go through their natural cycle, a continuous cycle of deposition and absorption is maintained. Loss through the surface of the CIST encapsulation is negligible, it soon becomes almost dry to touch but a 'dew point' effect is maintained between the substrate and the CIST producing sufficient inhibitors to fill the pits and crevices within the bolted structure, coating all internal surfaces without escaping into the environment.

Prolonged high temperatures will produce more volatility in the process but even at elevated temperatures weight reduction by inhibitor loss only 4%.

CYCLIC INHIBITOR LOSS TEST

For this test, an encapsulated substrate was subjected to a continuous cycle of temperature variations from 45°C to 4°C on a 24-hour cycle for six months. The weight of the substrate and the applied CIST material was recorded before and after encapsulation. The sample was suspended over a tray to collect any potentially escaping liquids.

A laboratory oven was used to raise the substrate to 45°C during the day, overnight the substrate was transferred to a refrigerator monitored at 4°C. The substrate was removed from cooling at 9 am and kept at ambient temperature until 11.00 when it was replaced in the oven. The oven was set to 45°C and switched on for 3 hours. After 3 hours the oven was switched off and the substrate allowed to cool in ambient temperatures for 2 hours before repeating the cycle, five days on, two days off. At weekends the substrate was left alternately at 45°C or ambient temperatures.

Over the six-month period there was no significant change in weight nor any escape of oil on to the tray. Initially weekly measurements were taken but, with no changes found, the interval was increased to monthly. At the end of six months, the material was removed and examined. Good oil deposits were found within the encapsulation, on bolt threads, metal and CIST interior surfaces. Other than some soiling from handling, no surface effects were found on the CIST exterior.

Fig 28 - Test piece before & after coating, close up of bolt thread and removed CIST after test completion

Figure 29 - Test results

Date	Weight uncoated	Weight coated
17-8-12	1427.5 g	1565.0g
21-9-12	N/A	1565.0
18-10-12	N/A	1565.1
23-11-12	N/A	1565.0
2-1-13	N/A	1565.0
31-1-13	N/A	1564.9
28-2-13	1427.6	1565.0

SUSTAINED HIGH TEMPERATURE TEST

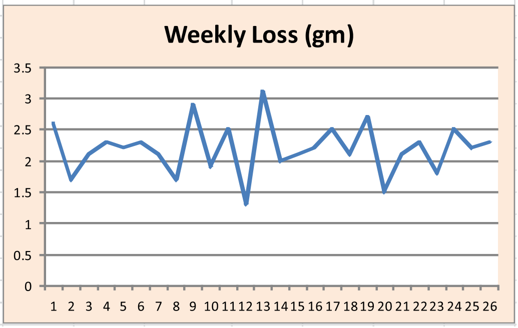
A measured and weighed sample of CIST material was placed in a laboratory oven for 3 months at 80°C in order to establish the rate of inhibitor loss at elevated temperatures. At the end of each week the material was removed from the oven, measured, weighed and immediately returned to the oven to maintain its temperature. Weight loss was consistent over the period with an average loss of 0.08% per week. Weekly measurements showed no measurable shrinkage over the period.

Fig 30 - Data from High Temperature Test

	Date	Measured Weight	Weekly Loss (gm)
Mar	11	2778.5	
	18	2775.9	2.6
	26	2774.2	1.7
Apr	2	2772.1	2.1
	8	2769.8	2.3
	15	2767.6	2.2
	22	2765.3	2.3
	29	2763.2	2.1
May	8	2761.5	1.7
	14	2758.6	2.9
	20	2756.7	1.9
	28	2754.2	2.5
Jun	4	2752.9	1.3
	11	2749.8	3.1
	18	2747.8	2
	25	2745.7	2.1
July	2	2743.5	2.2
	8	2741	2.5
	16	2738.9	2.1
	23	2736.2	2.7
	30	2734.7	1.5
Aug	6	2732.6	2.1
	13	2730.3	2.3
	21	2728.5	1.8
	28	2726	2.5
Sep	4	2723.8	2.2
	12	2721.5	2.3
Loss over 26 wks		57	57

ELEVATED TEMP WEIGHT LOSS TEST

Total Loss Over 26 Weeks (gm)	57
Percentage loss over 26 weeks	2.05%
Average loss per week	0.08%
Annual loss	4.10%



Q4: How impervious to water is the material?

Not satisfied that standard tests were sufficiently demanding, a deluge test was devised to run on a CIST-coated flange and pipe.

A 20% saline solution was cycled at approximately 400 litres an hour for 11 hours a day, 5 days out of 7 for 6 weeks. Ambient temperatures were between 24°C and 38°C. The steel pipe substrate and flange were new and uncoated low-carbon steel, a section along the length of the pipe was abraded prior to coating with CIST to provide the best potential conditions for corrosion. The CIST encapsulation was sealed using a standard tie-wrap technique.

At the end of the test the CIST was cut away to reveal the substrate. No corrosion of any kind was found within the CIST encapsulation, whereas significant corrosion occurred along the length of the exposed, unprotected pipe.

Fig 31 - Test substrate at commencement of test

Fig 32 - Test substrate at 7 days, 28 days and 38 days with increasing levels of corrosion on the unprotected substrate.

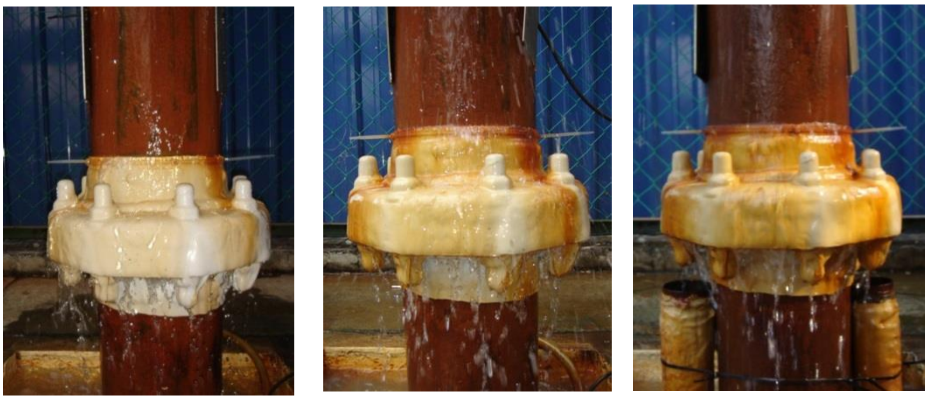


Fig 33 - Cutting away the material reveals bright steel, no blemishes or corrosion

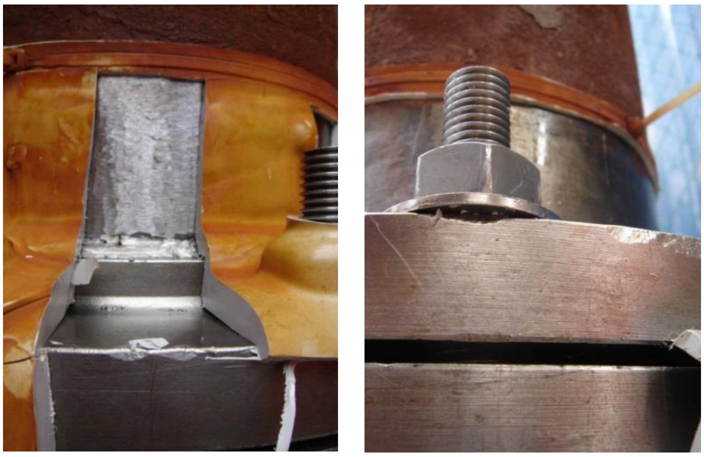
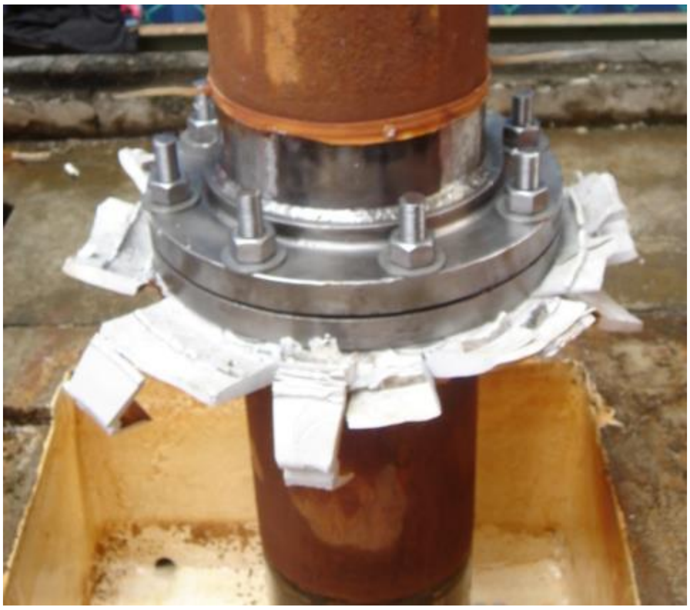


Fig 34 - Complete removal of the material shows no corrosion on substrate



Conclusion

Fifteen years ago, the first test carried out on a CIST application was the ASTM B117 3000-hour test - months before any commercial applications had taken place - as part of a testing regime to make sure that the product would perform to expectations.

As the material was cut away from the first test piece, the levels of corrosion outside the CIST encapsulation led to some scepticism that the protected areas could have survived intact. Yet, as the material was removed, inside the protective cocoon the steel substrate was found to be in perfect condition.

Fig 35 - Video of CIST removal

Since that time, many thousands of CIST applications have vindicated the results of that first testing regime, yet a wide range of testing continues to play an important role in understanding and explaining the technical background to CIST's unique protection system. And, with new opportunities and application areas being suggested all the time, CIST and the testing process has to adapt to meet these new challenges.

I'll tell you about them next year!

Thank you for your attention.