

ON PREDICTING LONG-TERM CORROSION BEHAVIOUR FROM SHORT-TERM TESTS

Robert E Melchers and Robert J Jeffrey
Centre for Infrastructure Performance and Reliability
The University of Newcastle, Australia

SUMMARY: It is common in corrosion practice to use relatively short-term observations to make inferences about the longer term reliability of infrastructure components or mechanical equipment. Short-term accelerated tests are widely used, even though they are known to produce only indicative results, not necessarily related to long-term field experience. A significant challenge remains in extrapolating short-term results to the longer term. It has been common, in corrosion science to use a simple model to extrapolate on the assumption that the underlying corrosion process does not change with time. However, as has now been shown for a number of realistic scenarios such as immersion, splash, atmospheric marine environments, this appears not to be the case and that the underlying mechanics are more complex. Some aspects of this were investigated by exposing identical steel coupons to natural seawater and to identical seawater but sterilized so as to remove all living material. This produced differing mass losses as a function of time. The results from this are used in comparison with other data, reported in the literature, to indicate that the controlling, underlying reactions change as the corrosion process proceeds, even without the influence of microbiological activity. It is proposed that these findings have major implications for estimating the long-term reliability of infrastructure.

Keywords: Steel, Seawater, Sterile seawater, Tests, Long-term.

1. INTRODUCTION

This paper is concerned with the prediction of the amount of corrosion loss (such as measured by mass loss), usually assumed to be equivalent to 'uniform' corrosion, and the prediction of maximum pit depth for long-term exposures of steel in marine environments. Long-term in this context means years and decades and is of interest mainly for infrastructure situations for which either protective measures are not feasible or have been neglected. It is of interest also in the context of very long-term storage of low and medium level nuclear wastes and other hazardous materials since steel containers repeatedly have been proposed for this purpose owing to cost considerations (King 2007, Pena et al. 2008). Evidently, it is not feasible to perform long-term trials to prove one material against another or to test protective systems. Instead, reliance commonly is placed on extrapolation from short-term tests in the actual or related exposure environment or on tests accelerated either by creating a more severe environment (e.g. salt-spray chambers) or speeding-up the corrosion process artificially, such as through anodic polarization (application of a direct electric current to accelerate the ionisation of ferric iron in aqueous solutions). One assumption made for obtaining results by these techniques is that they do not change the corrosion processes relative to those that act 'in the field'.

More realistic estimates of field corrosion behaviour can be obtained using coupons of the material to be used for the eventual structure or system and exposing these for some period in the realistic exposure environment. However, a significant challenge remains in extrapolating these results to the longer term. It has been common, in corrosion science, to make the assumption that the corrosion process does not change with time. However, as has now been shown for a number of realistic scenarios such as immersion, splash, atmospheric marine environments, this appears not to be the case.

Of course, for some applications the onset of corrosion is sufficient to render the system ineffective or unserviceable, but this is only seldom the case for major infrastructure applications. Usually these can undergo a degree of corrosion without significant loss of function and little, if any, safety implications, even though there may be visual issues.

Because long-term corrosion testing often is costly and usually inconvenient for decision-making, various attempts have been made to produce models of the progression of the corrosion process with time (Melchers 2008). The model most commonly

adopted, for atmospheric corrosion (Morcillo et al. 1995), for pitting depth (Englehardt and MacDonald 1998) and also for nuclear containments (King 2007) is the power law model:

$$c(t) = A t^B \quad (1)$$

where $c(t)$ is the corrosion loss or pit depth as a function of time t and A and B are constants obtained by fitting the function to data. It was derived originally from the oxidation reaction of copper as used in roofs in Europe (Tamman 1923) based on the notion that oxygen diffusion to the corroding metal surface becomes increasingly difficult as the corrosion product layers build-up on the metal surface. Refinements have been made to the model and its basic mathematical assumptions (e.g. Booth 1948, Evans 1960). It has been shown that for the corrosion of steel in marine environments the model (1) is consistent with field observations provided allowance is made for the fact that on first exposure there are no rusts present on the metal and that during this period oxygen diffusion from the surrounding medium most likely is the rate-controlling step (Melchers 2003).

It is important to distinguish what is expressed by (1) from what is possible chemically as determined through the chemical thermodynamics of a reaction, typically expressed by free energy considerations, or through a Pourbaix diagram. For example, for corrosion in seawater at the usual pH of around 8.2 the oxidation reaction can be written as



or



which shows, incidentally, that overall the chloride ions (in the NaCl) act only as 'spectator' ions - they are not involved in the oxidation reaction once it occurs, although they are likely to be involved in initiating the corrosion process - a matter that is still receiving active research attention (Waseda and Suzuki 2006). Note that in (2) oxygen is the electron acceptor and the cathodic reduction of oxygen is the rate-limiting step.

Oxygen as the electron acceptor is not the only possibility, however. Under limited oxygen conditions ferrous chloride FeCl_2 may form as part of the rusts, usually quickly converted on exposure to air to FeCl_3 and then Fe_3O_4 . When oxygen is even more limited (anoxic conditions) the most likely reaction involves the cathodic reduction of water (hydrogen reduction):



or



In seawater conditions the pH is sufficiently high for the reduction to occur of Fe^{3+} to Fe^{2+} and thus for the $\text{Fe}(\text{OH})_2$ to slowly change to hydrated magnetite $\text{Fe}_3\text{O}_4 \cdot x\text{H}_2\text{O}$ (Misawa et al. 1974) which gives the total reaction as (cf. Szklarska-Smialowska 1986)



Evidence for the evolution of hydrogen gas has been noted through bubbling from pits in rusts (Szklarska-Smialowska 1986). The magnetite Fe_3O_4 is hard and black, and difficult to remove from the metal. It also is often observed under the usual red and brown rusts on steel exposed for a long time to seawater (Waseda and Suzuki 2006).

A different corrosion product altogether usually found under older rusts is iron sulfide (FeS). It is a soft, black material often with the smell of hydrogen sulfide. When wiped from the steel typically bright steel is revealed. For this reaction sulfur acts as the electron acceptor (oxidizer). This can occur only in the presence of sulfate reducing bacteria (SRB), as shown by the following partial equations:



The above has shown that the electron acceptor (oxidizer) can change from oxygen to the hydrogen ion (from the dissociation of water) or to sulfur, depending on the level of availability of oxygen and whether or not bacterial activity can occur. It might be expected, therefore, that there will be a change in the availability of oxygen as the rust layers build-up on a corroding surface and hence a change in the oxidiser. In turn this means that there would be a change in the rate-controlling process. This means immediately that (1) cannot be valid without qualification. The evidence for this will now be considered in the light of four different sets of observations.

2. LONG-TERM MARINE IMMERSION CORROSION

2.1 Long-term marine immersion corrosion

Among the first to recognise explicitly that long-term corrosion was not necessarily simply related to short-term corrosion were Southwell et al. (1979) who proposed, on the basis of their extensive, observations of Panama Canal Zone (PCZ) immersion corrosion data that long-term corrosion losses were better represented by linear function that did not pass through the origin, as shown in Figure 1.

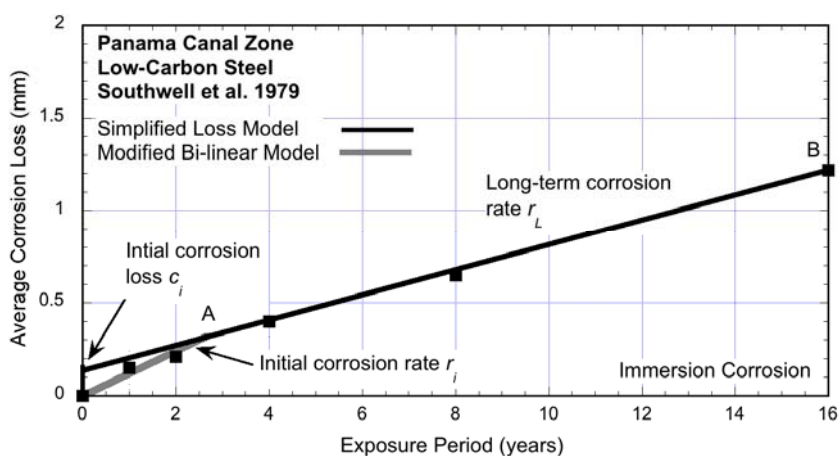


Figure 1. Data for long-term immersion corrosion in the Panama Canal Zone showing apparent linear long-term corrosion loss trend and intercept c_i in the model proposed by Southwell et al. (1979). Also shown is the possibility that the early corrosion data can be represented by a separate linear function (the 'bi-linear' model).

They attributed the long-term corrosion rate to the effect of sulfate reducing bacteria, based on the observation that the corrosion losses were substantially different after biocide had (inadvertently) been added to the local seawater. Since the average seawater temperature in the PCZ is relatively high (around 27°C), the influence of the early part 0A of the bi-linear function in Figure 1 is not significant for practical design of infrastructure. It has been shown (Melchers 2003) that this early branch lasts less than about 1 year in the PCZ. However, for more temperate waters the approximation adopted by Southwell et al. (1979) is more severe and the early branch 0A might last 3-5 years. Nevertheless, Figure 1 clearly recognises that early corrosion is unrelated to the longer term corrosion rate. Whether the long-term process is linear as proposed by Southwell et al. (1979) is an interesting question.

2.2 Long-term corrosion of nuclear containments in rock

The current practice in parts of Europe for the disposal of spent nuclear fuel is to store it initially (for about 30-40 years) in a central intermediate storage facility and then transport it to a geological repository where it is stored at depths up to 700 metres after first being encapsulated in corrosion-resistant disposal containers. Mechanical strength issues dictate that the containers must be cast iron or mild steel. Because of the presence of largely oxygen-free near neutral groundwaters, long-term corrosion of these containers is of considerable interest (Smart et al. 2002). The only reaction being considered for this environment is (3), noting that with time and temperatures above about 50°C reaction (3c) may produce $\text{Fe}_3\text{O}_4 + 2\text{H}_2\text{O} + \text{H}_2$. Laboratory tests showed that for containers in granite rock the formation of the corrosion product film is the rate-limiting step, although it has been suggested that in sedimentary rock the rate limiting step may actually be the diffusion of hydrogen to the atmosphere (King 2007). This is clearly a case where *hydrogen reduction* governs the long-term corrosion of steel as a result of low oxygen availability.

For determining or estimating the long-term corrosion losses and the evolution of maximum pit depths, the nuclear waste literature has taken two approaches. In one, theoretical consideration is given to limitations created on the various processes involved in corrosion of steel to magnetite, the principal corrosion product under anaerobic high temperature conditions. Most attention has been given to the diffusion of Fe ions and water and its ions (Pena et al. 2008). This suggests a long-term *non-linear* function. This also appears to be the consensus of empirical studies, the second approach to estimating long-term corrosion losses and pitting. These have taken the conventional power law (1) as a starting point, both for general corrosion and for pitting. This is based on other, parallel studies, such as the observations of the long-term burial of cast iron artefacts (e.g. Neff et al. 2005). As in many other corrosion studies, it was noted that the exponent in some cases appears to be close to unity (i.e. a constant corrosion rate).

The possibility of SRB and other bacterial involvement has been recognised (King 2007). However, it has not been considered a factor owing to the assessment that 'the near-field environment will not support microbial activity or biofilm formation on the container surface'.

2.3 Long-term atmospheric corrosion

Atmospheric corrosion studies have a long history of application of (1) (e.g. Morcillo et al. 1995), with almost all studies claiming it provides a good fit to the data, for exposure environments ranging from marine coastal to inland rural and industrial polluted atmospheres. However, as noted recently (Melchers 2009), there are many cases, even in atmospheric corrosion and including those for which a good fit is claimed where close examination shows that (1) does not fit the data particularly well. It has long been the norm to state that the poor fit is due to data errors and poor experimentation. However, a more useful approach is to consider whether (1) is valid. Many data for atmospheric corrosion are complicated by the presence of chlorides, SO_x or NO_x gasses or other pollutants. These could account for departures from (1). However, there are some examples, for exposures in rural and alpine regions, essentially unpolluted, that are of particular interest. Figure 2(a) shows one such example, using the data reported by Morcillo et al. (1995). If the data was consistent with (1) it should plot as a straight line on a log-log plot. Evidently this is not the case. There are two very distinct and very different trends. The second trend, for long-term corrosion, is also a reasonable fit and this implies that corrosion loss as a function of exposure time is not a linear function (i.e. does not have a constant corrosion rate). Instead, working back to estimate the fitted line on the log-log plot gives the function shown. It has the form of (1) but displaced relative to the origin. Figure 2(b) shows both functions and the respective data on the original corrosion loss - exposure time axes. The longer term trend is not linear as expected and does not pass through the origin.

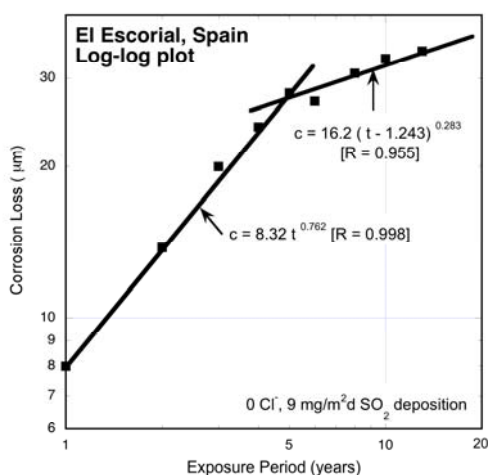


Figure 2(a) Log-log plot showing data from Morcillo et al. (1995). Data that follows (1) should plot as a linear function. Here there are two distinctly different trends.

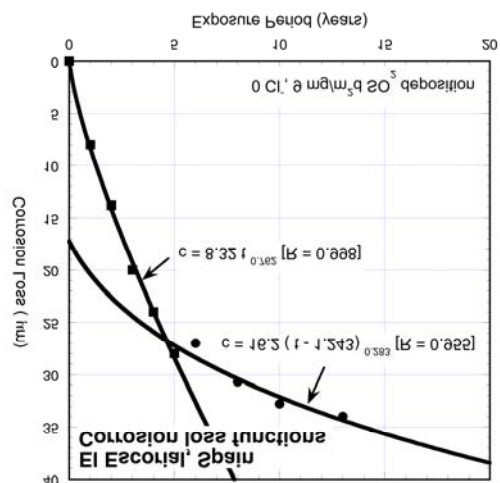


Figure 2(b) Data and extended (non-linear) trends corresponding to those shown in Figure 2(a) on a natural axes plot. The long-term data is distinctly different from the short-term trend.

Because the corrosion of the steel occurred in rural and alpine areas with presumably negligible pollution and hence nutrient availability, there is a very low probability that microbiological influenced corrosion (MIC) was involved.

2.4 Abiotic immersion corrosion

A recent 2.5 year study aimed at understanding the contribution of bacterial activity to pitting corrosion (Jeffrey and Melchers 2009) also contributes to the above observations, and puts the contribution of MIC into perspective. In brief, the experiment consisted of exposing identical coupons of steel to slow-moving coastal seawater, separated into two streams, one of which was filtered and subjected to UV radiation to render the sea water biologically inactive (sterile). This was confirmed regularly throughout the test period with bacterial culturing testing. The other seawater stream was not treated in any way. Details have been given elsewhere (Jeffrey and Melchers 2009). As a result of the way the test was set-up, the waters in each stream were identical in composition and in temperature fluctuations. However, the UV tubes increased the water temperature of the sterile stream by just under 2°C throughout the exposure period.

The steel specimens exposed in each stream were recovered at various times after first exposure, cleaned immediately to normal corrosion testing standards, dried, labelled and weighed and then examined in detail (Jeffrey & Melchers 2009). The mass losses were converted to corrosion losses. The results are shown in Figure 3 for the whole exposure period. There are a number of observations to be made. Firstly, Figure 3 shows a clear difference between short-term corrosion losses (OA), as might be measured by various electrochemical techniques and the 'one-year' observations (OB) such as typically obtained in field trials. Neither of these corrosion loss trends connects well with later data, shown here as 'long-term' trends, assumed to start at about 1.5 years of exposure. Clearly the situation would be worse if the 2-year data was assumed as the start of long-term corrosion. Figure 3 also shows that the longer term corrosion losses for the natural seawater are greater than those obtained in sterile seawater and that this is the case irrespective of whether a linear trend or some form of curvilinear trend is assumed to pass through the data points. The available data is insufficient to make that distinction. What is clear, above all else, is that a simple curve of the type (1) would be unable to capture, adequately, the trend implicit in all the data.

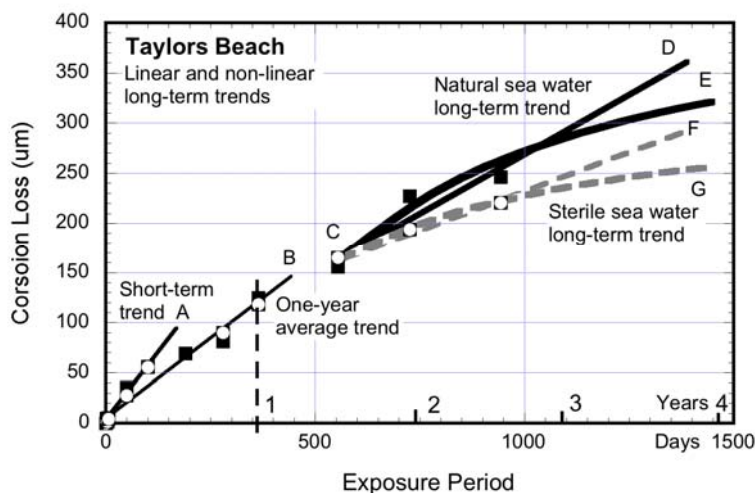


Figure 3. Corrosion loss data obtained in natural seawater and in sterilised seawater showing that short-term trends (OA and OB) are distinctly different from longer-term linear data trends (CD and CF) and also that the longer-term data may be represented perhaps better by the non-linear trends (CE and CG), respectively for natural and sterilised seawater. Data from Jeffrey and Melchers (2009).

3. DISCUSSION

All of the results shown above have the common feature that there is no direct link between corrosion observed over the initial period, lasting perhaps a few years but sometime much less, and that for much longer-term exposures. As noted, this was considered already by Southwell et al. (1979). They proposed the straight-line representation shown in Figure 1, based on data extending over 16 years in the corrosive tropical Panama Canal Zone. This interpretation was based on the then available data, consisting of mass loss (and pit depths) at about 4, 8 and 16 years of exposure, the earlier data being more difficult to interpret (Figure 1). There are very few other observations over longer periods and with sufficient *intermediate* observations to define the long-term trend unambiguously. As has been argued previously, there is likely to be a transition zone (phase 3) immediately prior to the establishment of the longer-term trend, as shown schematically in Figure 4 (Melchers 2003, Melchers and Jeffrey 2008). This phase 3 phenomenon may be all that can be discerned, for example, in Figures 2 and 3, despite the observations extending, in the case of Figure 2, over some 13 years. For atmospheric corrosion the development of corrosion products, and hence the change in corrosion pattern is much slower than for immersion corrosion, particularly for higher water temperature. This will tend to delay any change in corrosion loss trend.

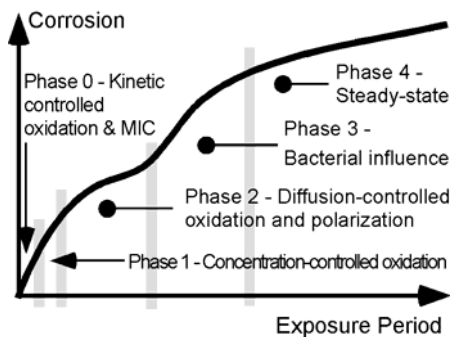


Figure 4. Schematic representation of the model proposed for corrosion loss and pit depth showing the different phases corresponding to different rate-controlling mechanisms for corrosion (Melchers 2003, Melchers and Jeffrey 2008).

The idealised corrosion loss pattern and the different phases in corrosion loss trend shown in Figure 4 are based on the concept that during phase 2 the diffusion of oxygen to the corroding surface becomes increasingly less owing to the build-up of corrosion products rendering oxygen diffusion from the exterior environment to the corroding surface more difficult (Melchers 2003). In principle this will render regions of the interface, and eventually the whole of the interface, anoxic. Thus corrosion that follows must occur under anoxic conditions. It has been argued that subsequent corrosion, initially in phase 3, but also extending into phase 4, will involve bacterial influences (MIC). Diffusion analysis showed that phase 3 at least, was non-linear (Melchers and Wells 2006). Of course, corrosion under hydrogen reduction (equations 3) also may occur. This is the interpretation adopted for the corrosion of steel cannisters in rock formations (King 2007). Under these conditions analysis of the diffusion of the various species involved in the corrosion process suggests that such diffusion will become increasingly difficult owing to the containment effect of the rock (and any bentonite used to fill spaces between the rock and the cannisters). The conclusion from this must be that the corrosion loss trend will not be a linear function (although this does not preclude the possibility that it might be closely approximated by one).

It is doubtful, however, that the conclusion about the long-term corrosion loss being non-linear can be transferred directly to, say, atmospheric or immersion corrosion since that external environment is distinctly different from that for sealed cannisters in bentonite-sealed rock formations. More likely these results have relevance to the corrosion of reinforcement bars in concrete (e.g. Chitty et al. 2005).

On the basis of the various observations above, and the progression of corrosion as modelled by Figure 4, it is likely that for steel exposed to the environment and for which corrosion products are permitted to build-up without interference, the long-term corrosion processes will involve both hydrogen reduction and microbial corrosion. For such exposed steel it is likely also that erosion of the external weaker and more porous rusts will occur, leaving an increasingly denser remaining rust layer. Under these conditions, too, a (perhaps only) mildly non-linear long-term corrosion loss function would be expected. But it clearly has no affinity, in concept, in processes involved and in mathematics, with those for short-term corrosion. This disconnect is illustrated, for example, in Figure 4, even though it is unlikely that the corrosion represented in this data set has reached a long-term stability in the corrosion loss function. Of course, where corrosion products are not permitted to build-up, or where they are continually removed by wear or erosion, the short term trends are highly relevant, even for long-term exposures.

The practical implications of the foregoing is that short term observations, particularly those taken in the laboratory, but also the typical one-year field observations, must be used with care. Usually they provide a very conservative (over-estimate) of the long-term corrosion rate under conditions where the corrosion products can build-up. If field exposure tests are contemplated careful planning of the experiment is essential. Simply using one or more arbitrary points in time to take observations is not optimal. Thus, it is immediately clear (Figure 5(a)) that any estimate of corrosion rate based on using the origin 0 and any one of the observations at A,B or C will seriously over-estimate the long-term rate. However, a more considered use of the data can give increasingly better estimates of the likely long-term corrosion rate. This is illustrated in Figure 5(b). It shows that observations of corrosion loss such as B and C, obtained after phase 3 of the model in Figure 4 has been reached will permit a reasonable estimate of the long-term corrosion rate provided the origin 0 is ignored. Earlier corrosion loss measurements, such as A, together with B, will provide only an approximation. Evidently, if it is feasible to make corrosion loss measurements over some years, the long-term corrosion rate can be estimated with a reasonable degree of accuracy in this manner (even allowing for the likelihood that there is likely to be a very slowly declining rate). Such an estimate will improve with a systematic approach to the use of corrosion loss observations, as indicated in Figure 5(b).

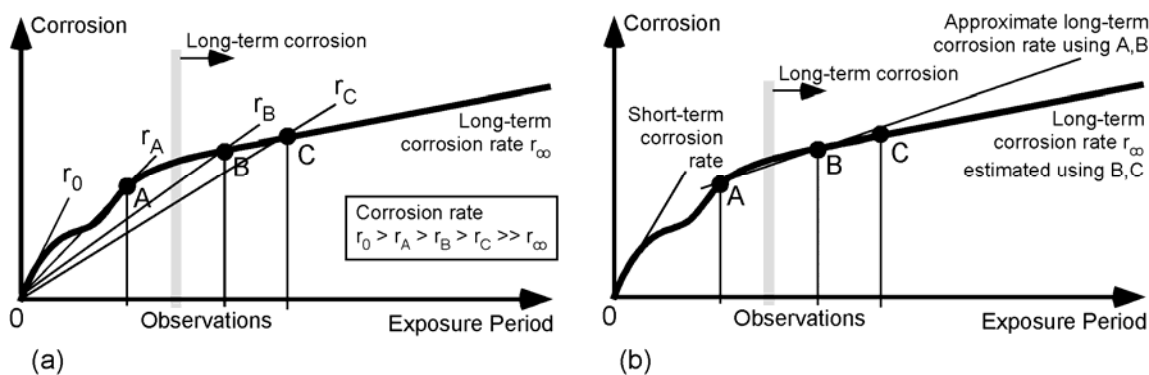


Figure 5. (a) Conventional use of corrosion loss observations, showing poor estimation of longer term corrosion rate. (b) Use of corrosion loss data to estimate the longer term corrosion rate.

If medium-term field testing is not feasible, estimates for the longer term corrosion rates might be obtained from data such as provided by the Panama Canal Zone observations, perhaps reduced for the effect of average water or atmospheric temperature and with judicious interpretation of the effect of precipitation and exposure if relevant.

4. CONCLUSION

The following conclusions may be drawn from this study:

1. There is now considerable evidence that for steels in marine environments or in atmospheric exposure conditions the trend for long-term corrosion loss is distinctly different from short-term observations as obtained, typically from laboratory observations or standard one-year field tests. This makes short-term tests of doubtful validity for estimating longer-term corrosion losses.
2. Although long-term corrosion loss trends may be non-linear, the available evidence for atmospheric and marine exposures suggests a long-term linear trend. The data, however, supports there being a non-linear transition between short- and long-term corrosion losses.
3. Provided field experiments to obtain longer term data are properly planned and set in a model context, they can be used to estimate longer-term corrosion loss rates without actually performing very long-term experiments. Two techniques for doing this are given.

5. ACKNOWLEDGMENTS

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7. AUTHOR DETAILS



Robert E Melchers is Professor of Civil Engineering and Australian Research Council Professorial Fellow at The University of Newcastle, Australia. He has a BE and MEngSc from Monash University and a PhD from the University of Cambridge, UK. He was awarded the 2004 TP Hoar Prize (Institute of Corrosion, UK) (with Robert Jeffrey), the 2007 Guy Bengough Award (Institute of Materials, Minerals and Mining, UK), the Marshall Fordham prize (Australasian Corrosion Association) in 1999, 2002 and 2007 and the ACA Corrosion Medal in 2009. His research interests include structural reliability and marine corrosion.



Robert Jeffrey is Research Fellow at The University of Newcastle, Australia where for the last ten years he has been investigating corrosion in marine, tidal and atmospheric conditions. Robert is also principal consultant for Pacific Testing Pty Ltd, a company specializing in corrosion problems. He is a past president of the ACA and has been on the committee of the Newcastle branch for twenty years. He has been awarded the prestigious TP Hoar Prize (Institute of Corrosion, UK) and has twice been presented with the ACA's Marshal Fordham award for corrosion research. He holds a PhD from The University of Newcastle.