LPR TESTING ON RESLO – FINAL SUMMARY REPORT

Report prepared for:

Lightning Protection International Pty. Ltd. (“LPI”)

Prepared by:

Mr. Ruiliang Liu, Mr. Ruifeng Zhang and Prof. Nick Birbilis

Department of Materials Science and Engineering,

Monash University

Clayton, VIC, 3800

Australia

Phone: +61399054919

4th April 2016

Signed:

Contents

Project brief:

In this project, the linear polarisation resistance (LPR) of stainless steel rods and copper-plated steel rods in RESLO earth enhancing compound (EEC) supplied by LPI was determined over a suitable period of time. The method and results of the LPR testing are included herein. All testing and results are referenced to IEC 62561-7, which is used internationally to assess EECs.

Experimental methods

Sample preparation

Stainless steel rods and Cu-plated steel rods (sourced from LPI) were sectioned, and a quality fitting was tapped into the top of each sample to achieve robust electrical connection. The setup of the samples at varying degrees of preparedness is shown in Fig. 1. A multi-layered self-adhesive heat-shrink was applied over both ends of the samples, including the connections, to seal them and protect them from possible damage. Additionally, the heat shrink also defined the exposed surface area of each ground rod sample (see Appendix 1), which allowed for the polarisation resistance to be determined relative to area (as required).

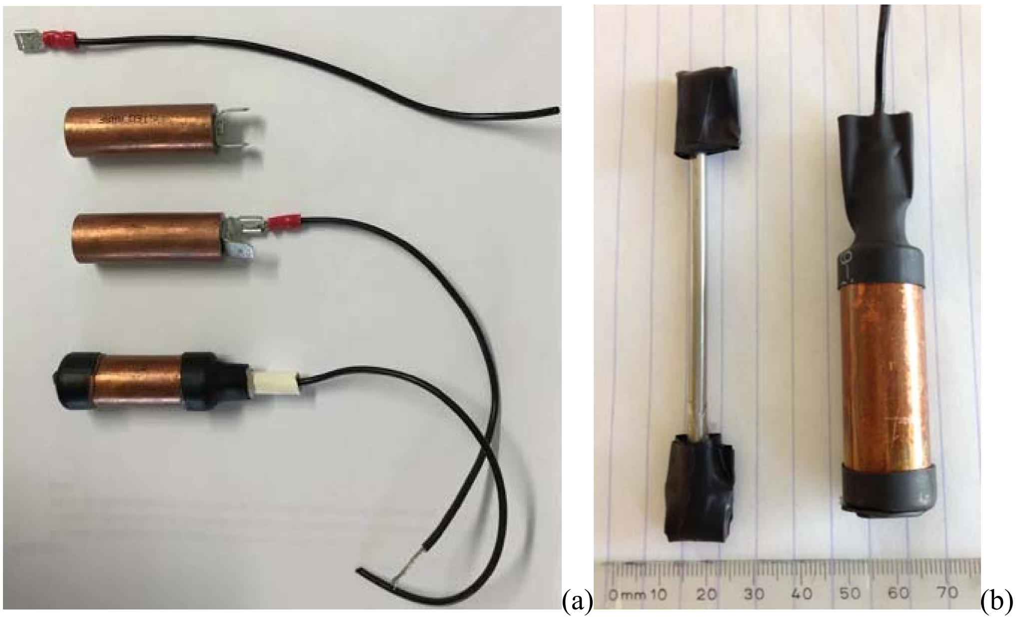


Figure 1: (a) Copper-plated rod samples in various stages of preparation; (b) Stainless steel rod (left) and copper-plated steel rod (right) sample.

Setup of test cell

As shown in Fig. 2, each sample was assembled in a three-electrode cell consisting of a working electrode, a reference electrode (in this case a saturated calomel electrode) and a counter electrode (in this case, mixed-metal-oxide coated titanium mesh which is an industrially available counter electrode (known as the Lida® mesh electrode).

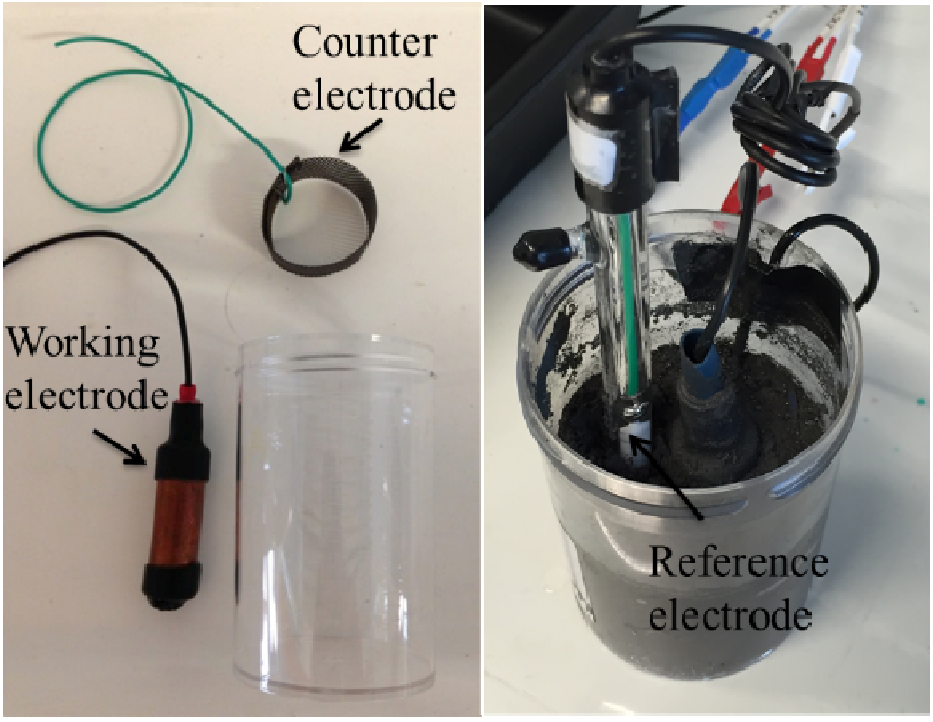


Figure 2: Setup of test cell.

In this test the working electrode is defined by the exposed area of stainless steel or Cu-plated rod. The aim of the test regime herein is to probe electrochemical corrosion reactions on the surface of the test sample (working electrode). The reference electrode is a standard half-cell, and its only role is to serve as a reference in measuring and controlling the working electrode potential; at no point does the reference electrode pass any current. The saturated calomel reference electrode consists of a combination of mercury and mercurous chloride in contact with a saturated potassium chloride (KCl) solution. The potential of this reference electrode is +0.241 V vs. the standard hydrogen electrode[1]. We note that for testing herein, the reference electrode was inserted for each test, and not permanently housed in the test cell. The reason for this was that any cementituous material in sample formulations may attack and etch the glass casing of the reference electrode. Any leakage of saturated KCl solution from the reference electrode would be detrimental to the test sample. The intermittent insertion of the reference electrode is also beneficial as it allows the electrode to remain well maintained when not in use. For this study, a dedicated rotating pool of 10 calibrated reference electrodes was used. In the laboratory, the saturated calomel reference electrodes used for electrochemical testing are compared to a master/calibration reference electrode (which is not used for any electrochemical testing) prior to each use, assuring that electrode stability exists.

Preparation of sample electrolytes

For the purpose of this work, a chloride-containing environment of 0.1M NaCl (5.8 g of NaCl / 1 L of H_2O) was selected to simulate an "aggressive" environment. Three RESLO samples were mixed with 0.1M NaCl solution according to the mixing ratio provided by LPI. Each cell was prepared individually to ensure the minimum recommended ratio of water and RESLO was met, per the requirements of IEC 62561-7.

Test environment

All the tests reported herein were conducted in a controlled laboratory environment in Building 82 at Monash University (Clayton, VIC, Australia), at the constant room temperature of 25 °C.

Testing parameters for the linear polarisation resistance (LPR) test

The linear polarisation resistance (LPR) method was used to determine the "polarisation resistance" (R_P) of test specimens. R_P is inversely proportional to the corrosion current density expressed by the Stern-Geary equation [1, 2] (described further in Appendix 2). The LPR test is non-destructive in nature, as the applied polarisation results in small alteration of current. Therefore, a non-destructive electrochemical polarisation in the range of -10 mV to +10 mV from the rest potential (also known as the open-circuit potential) was executed at a rate of 10 mV/min. The test duration was therefore 2 minutes.

Multiple samples of the electrodes were tested, all in individual cells, each in the same RESLO EEC supplied. Since copper or copper-coated electrodes represent the most common earthing electrode by far, copper rods were tested in an aggressive and non-aggressive ("normal") environment in triplicate, whilst for stainless steel electrodes, tests were carried out in duplicate under normal conditions, since the latter electrode type is far more corrosion-resistant than copper.

Prior to each test, the reference electrode was calibrated.

A Bio-Logic SP-150 potentiostat (Knoxville, TN) was used for the LPR testing (as shown in Fig 4 below).

Figure 4. The Bio-Logic SP-150 potentiostat for LPR testing

Results and parameters recorded

Independent measured R_p values for each cell and post-test observations (copper-coated steel electrodes)

The "aggressive" and "non-aggressive" environment corrosion testing was commenced on 4 September 2015. The measured value of R_P as a function of time for each sample is presented in Table 1 in the units required by IEC 62561-7, i.e., Ω.m^2.

Table 1: LPR results for copper-coated steel electrodes embedded in RESLO EEC for both "aggressive" and "non-aggressive" environments.

Copper-coated steel rod		**R_p (Ω.m^2)**					
	Sample	Day 0 04/09/2015	Day 1 05/09/2015	Day 2 06/09/2015	Day 3 07/09/2015	Day 7 11/09/2015	Day 14 18/09/2015
Aggressive	1	18.8	17.7	18.3	18.3	20.6	18.4
	2	9.85	14.3	16.8	17.5	20.3	20.5
	3	27.9	21.6	20.3	3.03	8.42	13.5
Non-aggressive	1	385.3	161.9	358.5	392.7	347.8	159.4
	2	130.8	161.5	188.3	168.5	133.4	136.4
	3	138.7	153.8	160.5	161.2	154.9	97.2

Some variation of the measured R_P values between samples can be seen. Such variations are normal statistical fluctuations. Importantly, the LPR results for RESLO-embedded coppr-coated steel electrodes are consistently high with time. The LPR values for both non-aggressive and aggressive environments are well in excess of what is required to meet the requirements of the IEC standard.

In summary, the average values of R_P for copper-coated steel electrodes embedded in RESLO easily pass the criteria specified by IEC 62561-7, i.e., they are higher than 8 Ω.m^2.

Independent measured R_p values for each cell and post-test observations (stainless steel electrodes)

Stainless steel electrodes are also used for earthing, even though they are not as common as copper-coated steel electrodes. Hence, some additional corrosion testing was also carried out on stainless steel electrodes embedded in RESLO.

"Non-aggressive" environment (normal condition) corrosion testing was commenced on 11 November 2015 on duplicate samples. The measured value of R_P as a function of time for each sample is presented in Table 2 in the units required by IEC 62561-7, i.e., $\Omega.m^2$.

Table 2: LPR results for stainless steel electrodes embedded in RESLO EEC under normal ("non-aggressive") conditions.

Stainless steel rod	Sample	**R_p ($\Omega.m^2$)**					
		Day 0 15/11/2015	Day 3 18/11/2015	Day 11 26/11/2015	Day 13 28/11/2015	Day 17 02/12/2015	Day 23 08/12/2015
Non-aggressive	1	14.35	19.65	54.44	73.69	78.24	68.68
	2	12.85	15.06	95.71	95.12	90.12	83.50

Some variation of the measured R_P values between samples can be seen. Such variations are normal statistical fluctuations. Importantly, the LPR results for RESLO-embedded stainless steel electrodes are consistently high and actually increase with time. These LPR values, for a non-aggressive environment, are well in excess of what is required to meet the requirements of the IEC standard.

In summary, the average values of R_P for stainless steel electrodes embedded in RESLO exceed by a large margin the pass the criteria specified by IEC 62561-7 for galvanised steel electrodes, namely > 3 $\Omega.m^2$.

Summary

1. LPR testing was successfully carried out on multiple samples of copper-coatred steel and stainless steel electrodes embedded in RESLO EEC.
2. The LPR data for copper-coated steel electrodes indicate that, in the RESLO EEC environment, they show good corrosion resistance under both normal (“non-aggressive”) and extreme (“aggressive”) conditions. In both cases, the polarisation resistance exceeds the minimum requirements in IEC 62561-7.
3. The LPR data indicate that for stainless steel electrodes in the RESLO EEC environment, they show good corrosion resistance in the “non-aggressive” conditions under which they were tested. The polarisation resistance exceeds the minimum requirements in IEC 62561-7.
4. Aside from the requirements and criteria in IEC 62561-7, the work herein aimed to provide an objective characterisation of RESLO EEC. As a result, calculated penetration depths, which provide a physical basis for damage measured from LPR tests, have been included in Appendix 2 in order to allow for a practical engineering assessment.

References:

1. D. Jones: 'Principles and prevention of corrosion', 1996, Prentice Hall.
2. J. Scully: 'The Polarization Resistance Method for Determination of Instantaneous Corrosion Rates', in 'Electrochemical Techniques in Corrosion Science and Engineering', 2002, CRC Press.New York.
3. IEC, 2011, IEC 62561-7: “Lightning protection system components (LPSC) – Part 7: Requirements for earthing enhancing compounds”, Geneva, Switzerland.

Appendix 1: Exposed surface area (cm^2) of each sample.

Sample	Stainless steel rods	Cu-plated steel rods
A	3.17	19.99
B	3.77	20.72

Appendix 2: Projected penetration

Determination of corrosion current density, i_{corr}, was based on the Stern-Geary Equation. Such calculations are not influenced by the thickness of the Cu-plating, and only require the surface area of the electrode.

$$R_P = \frac{B}{i_{corr}}$$

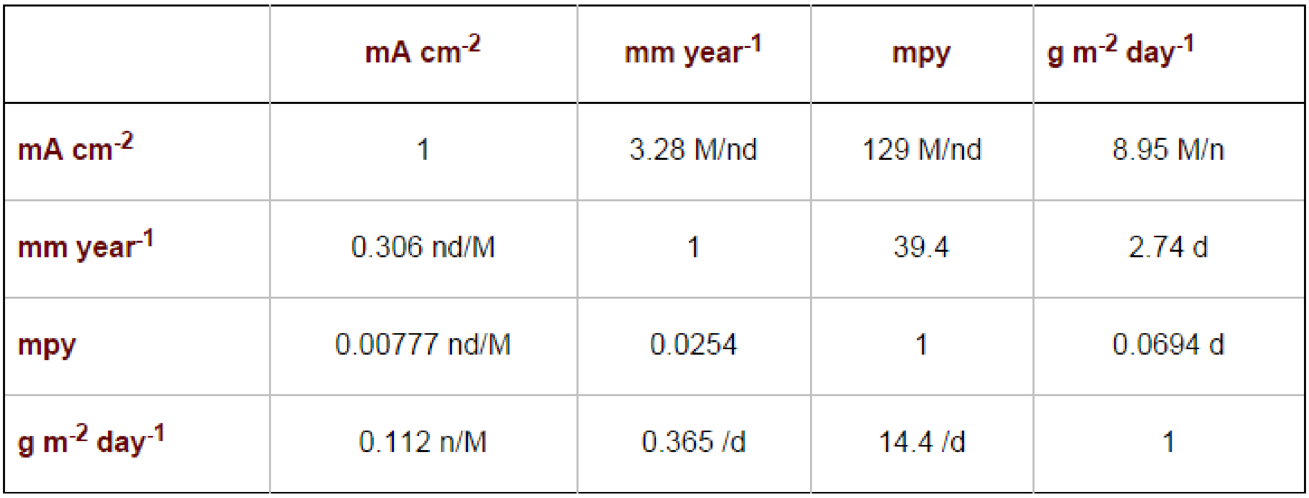
where R_p is the polarisation resistance, i_{corr} is the corrosion current density, and B is the proportionality constant. In this case, B was taken as being equal to 25 mV.

The origin of the value “B” arises from the Tafel slopes of the anodic and cathodic partial corrosion reactions. The true value of B is expressed as:

$$B = \beta_a.\beta_c / 2.3(\beta_a + \beta_c)$$

Where, β_a is the anodic Tafel slope, and β_c is the cathodic Tafel slope. Note: neither β_a or β_c are measured in an LPR test. The determination of B requires destructive testing. As a result, a rational value of B is used as a constant. The value of B nominally varies between ~25 and 52 mV, the range covering active to passive systems. B = 25 mV was selected because of (i) the low R_P values determined in this work indicating non-passive conditions, and (ii) the use of a low B value is conservative, such that quoted corrosion rates are the upper bound (i.e. worst case).

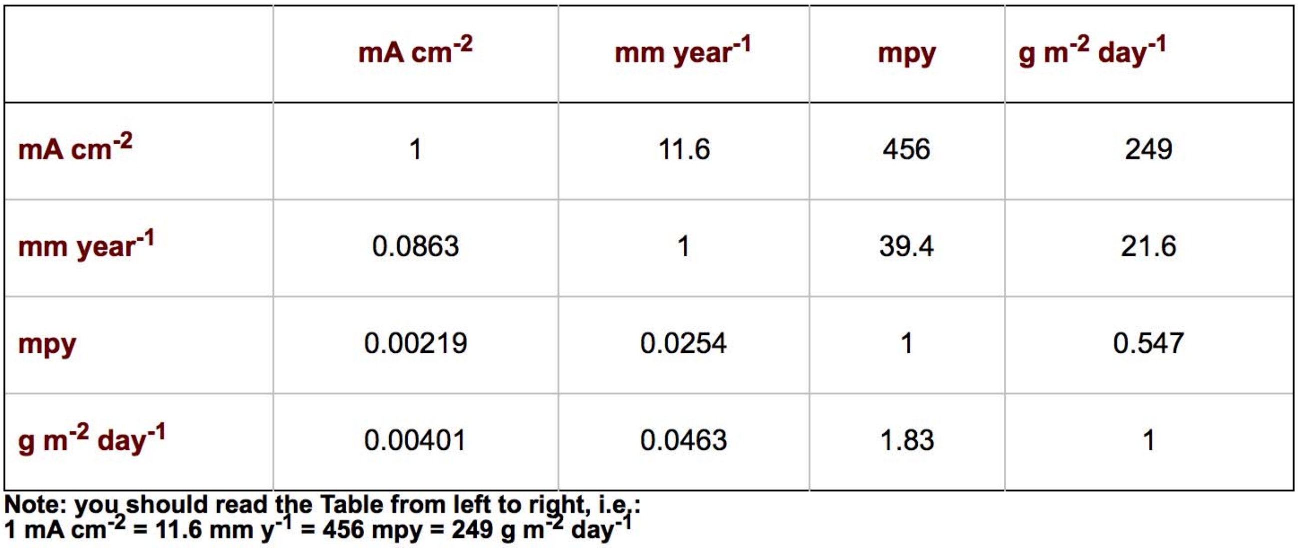
The calculated corrosion currents densities can be converted into penetration rates using Faraday’s law according to the following table:



	mA cm^{-2}	**mm year^{-1}**	**mpy**	**g m^{-2} day^{-1}**
mA cm^{-2}	1	3.28 M/nd	129 M/nd	8.95 M/n
mm year^{-1}	0.306 nd/M	1	39.4	2.74 d
mpy	0.00777 nd/M	0.0254	1	0.0694 d
g m^{-2} day^{-1}	0.112 n/M	0.365 /d	14.4 /d	1

where mpy = milli-inch per year, n = number of electrons freed by the corrosion reaction, M = atomic mass, d = metal density

If the metal is steel, n = 2, M = 55.85 g and d = 7.88 g/cm^3 and the corrosion rate conversion is:



	mA cm^{-2}	mm year^{-1}	mpy	g m^{-2} day^{-1}
mA cm^{-2}	1	11.6	456	249
mm year^{-1}	0.0863	1	39.4	21.6
mpy	0.00219	0.0254	1	0.547
g m^{-2} day^{-1}	0.00401	0.0463	1.83	1

Note: you should read the Table from left to right, i.e.:
1 mA cm^{-2} = 11.6 mm y^{-1} = 456 mpy = 249 g m^{-2} day^{-1}

In the case where the plating material is copper, n = 2, M = 63.5 and d = 8.96 g/cm^3 and the corrosion rate conversion is 1 mA/cm^2 = 11.6 mm/yr.

Projected penetration rate for 30 years exposure based on the average R_P of the final three data points collected (covering the final 10 days of exposure). The values calculated are also the average of the expected penetration from the multiple samples (two or three).

Sample	Estimated penetration for 30 years of exposure (µm)
Stainless steel rods **(Non-aggressive conditions)**	~14
Cu-plated steel rods **(Non-aggressive conditions)**	~7
Cu-plated steel rods **(Aggressive conditions)**	~45