

CHAPTER 2: EXPERIMENTAL

2.1 Materials

The high strength AISI 4340 steel was used as testing material in the mechanical testing (SSRTs) of this work, whilst low-carbon steel shims were applied in hydrogen permeation measurements. Moreover, the aluminium-based coatings, examined in the programme as possible alternatives to Cd, were SermeTel CR984-LT and Alcotec Galvano-Aluminium.

The metallographic examination of the previously referred materials was performed by the following procedure. Small pieces of AISI 4340 steel in the quenched and tempered condition, SermeTel CR984-LT and Alcotec Galvano-Aluminium coated steel panels were cut with a Buhler Isomet 2000 precision saw. Afterwards, these sections were mounted in bakelite in a Simplimet 2000 automatic mounting press.

Following mounting, the specimens were polished on grinding wheels, using initially 120-grit carborundum paper, then 220-grit and finally 1200-grit. Subsequent to this, the sections were polished on Struers diamond polishing wheels, initially with a 6-micron diamond paste and then with 1-micron. The mounted specimens were then etched using 2% Nital solution to reveal the microstructure of the test material and the coatings, examined in an Olympus optical microscope. Moreover, the coatings thicknesses were measured under the optical microscope by using the eyepiece graticule, and on the Scanning Electron Microscope (SEM) from the image on screen and from photographs taken in both cases.

2.1.1 Testing Materials

The material used in the hydrogen embrittlement tests in this work was the high strength steel AISI 4340, the chemical composition of which is given in Table 1, while Figure 34 shows its microstructure in the quenched and tempered condition. Quenching and tempering of this material produced the acicular appearance of the

tempered martensite structure. For the hydrogen permeation measurements, the composition of the utilised mild low-carbon steel shim is displayed in Table 2.

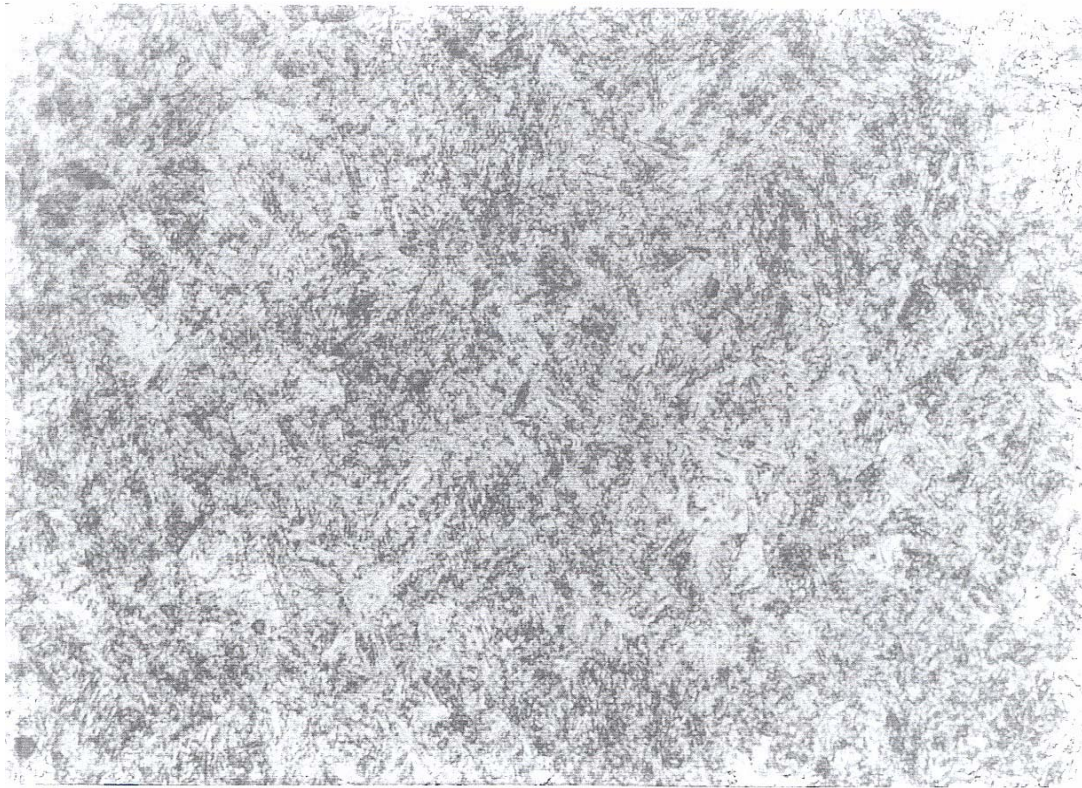


Figure 34. Optical micrograph showing the martensite phase of the quenched and tempered AISI 4340 steel

% C	% Mn	% P	% S	% Si	% Cu	% Ni	% Cr	% Mo	% Al	% V	Fe
0.41	0.79	0.009	0.003	0.23	0.13	1.67	0.88	0.22	0.024	0.004	balance

Table 1. Analysis of AISI 4340 steel, used in embrittlement tests.

% C	% Mn	% P	% S	% Si	% Cu	% Ni	% Cr	% Mo	% Al	Fe
0.04	0.24	0.008	0.012	<0.02	0.04	0.02	<0.01	<0.03	0.05	balance

Table 2. Analysis of the mild steel shim, used in permeation tests.

2.1.2 Coatings

SermeTel CR984–LT

The SermeTel CR984–LT coating was examined by optical and scanning electron microscopy, and shown to consist of Al particles in an inorganic chromate and phosphate binder. The optical micrograph in Figure 35 shows the section of a SermeTel coated steel panel. The surface of the steel had been roughened by abrasion prior to coating, and the coating is approximately 30 μm thick. As viewed from the electron back scattered image in Figure 36, the Al particles range in size up to 8 μm in diameter. EDAX (Energy Dispersive X–Ray) analysis showed that the aluminium particles of SermeTel did not contain any other alloying elements, whilst the binder was rich in P and Cr. (Figure 37) Environmental concerns about chromium led to a quantitative analysis with the help of the SEM, according to which the content in Cr is 1.5% approximately. Al is present in 93% and P in almost 5%. (Table 3)

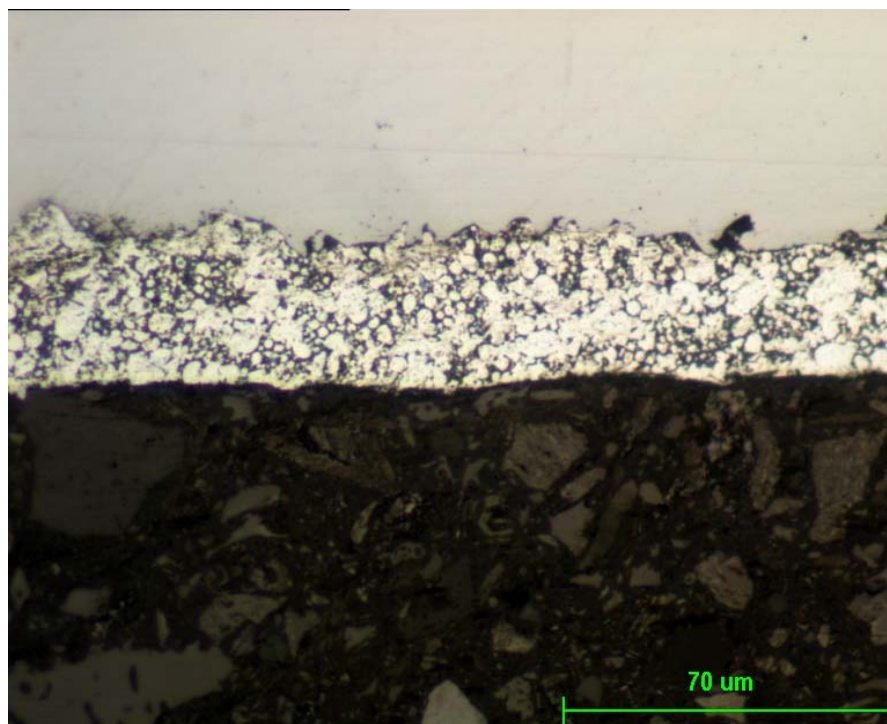


Figure 35. Optical micrograph of SermeTel coating

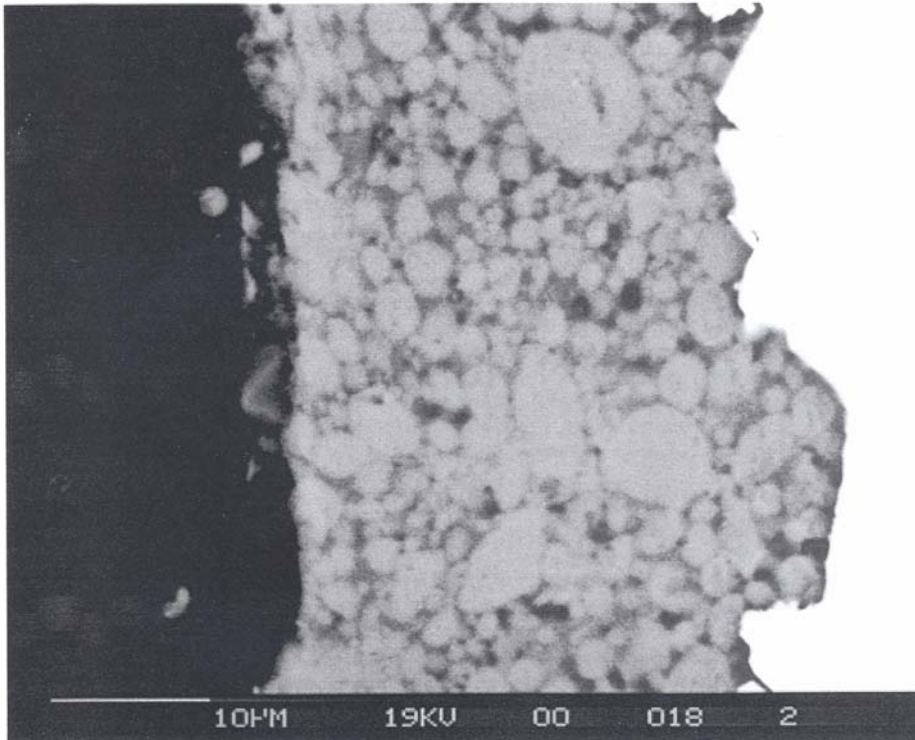


Figure 36. Electron back-scattered image of SermeTel coating

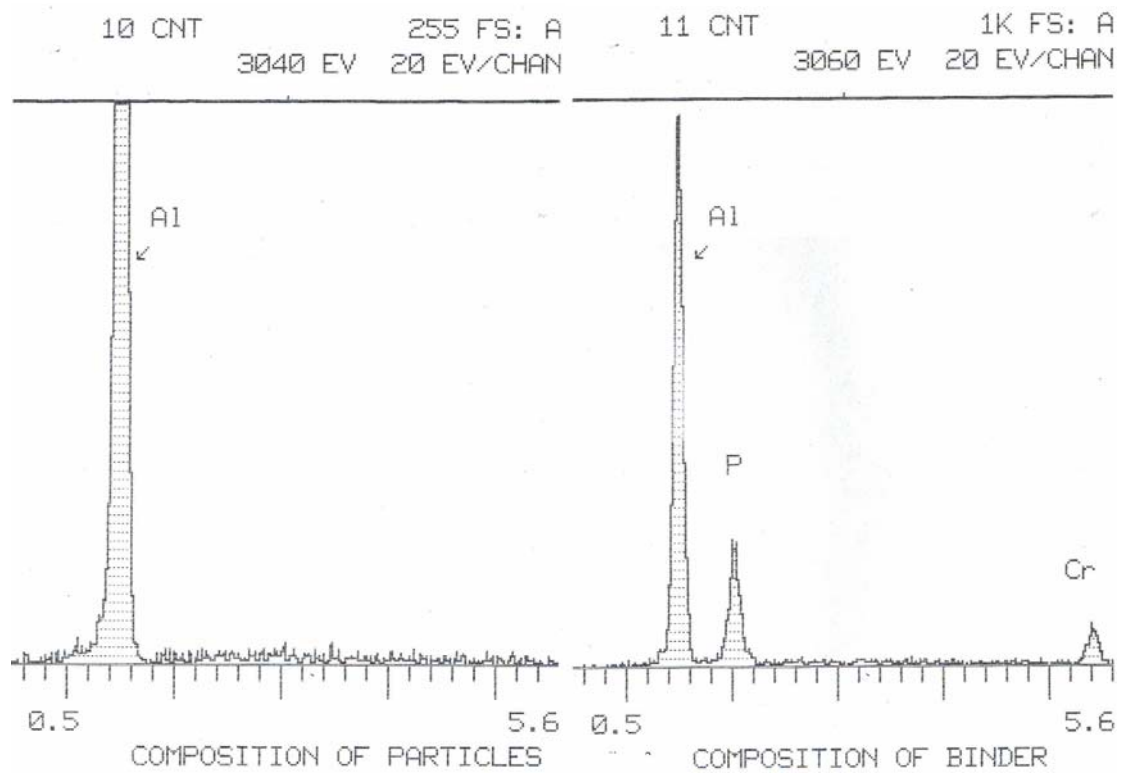


Figure 37. EDAX analyses for the SermeTel particles and binder.

ELMT	ZAF	%ELMT	ATOM.%
AlK: 4	.968	93.157	94.702
P K: 2	.491	4.738	4.196
CrK: 4	.837	1.562	.824
FeK: 4	.845	.567	.279
TOTAL		100.024	100.000

Table 3. Quantitative analysis for the SermeTel coating

Alcotec Galvano–Aluminium

The optical micrograph in Figure 38 shows the section of an Alcotec coated steel panel. The coating is approximately 20 µm thick Alcotec contains only Al and is electrodeposited from an organic electrolyte. EDAX analysis showed the composition of Alcotec to be Al and Cr. (Figure 39) The presence of chromium is due to the subsequent passivation treatment which is based on Cr VI, whilst the presence of iron can be attributed to the steel substrate of the panel.

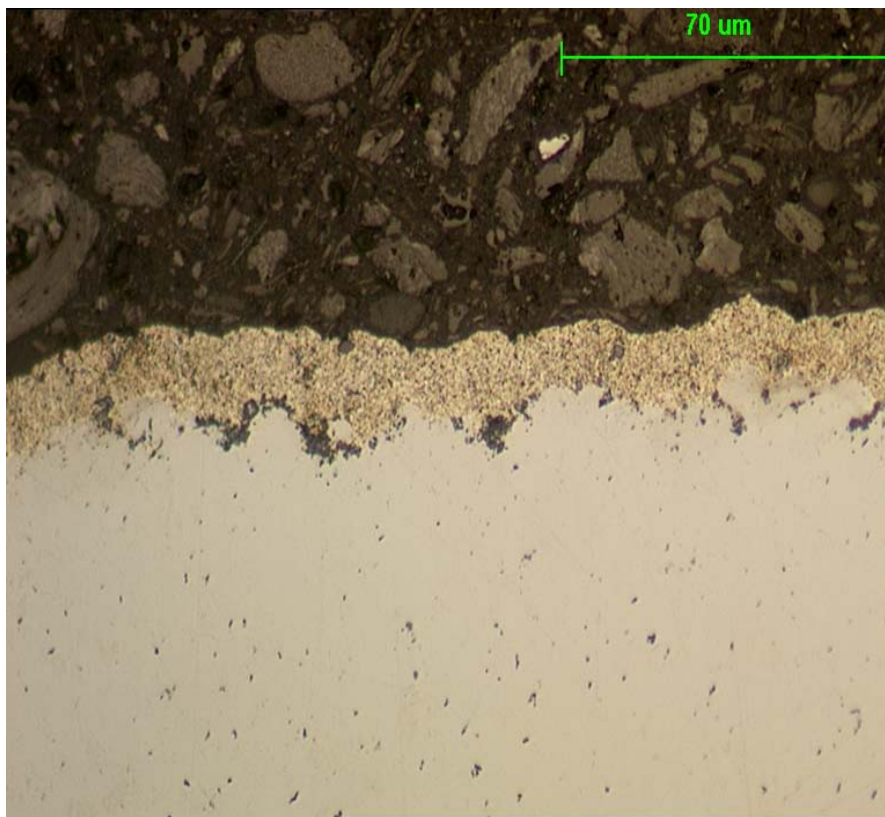


Figure 38. Optical micrograph of the Alcotec Galvano–Aluminium coating

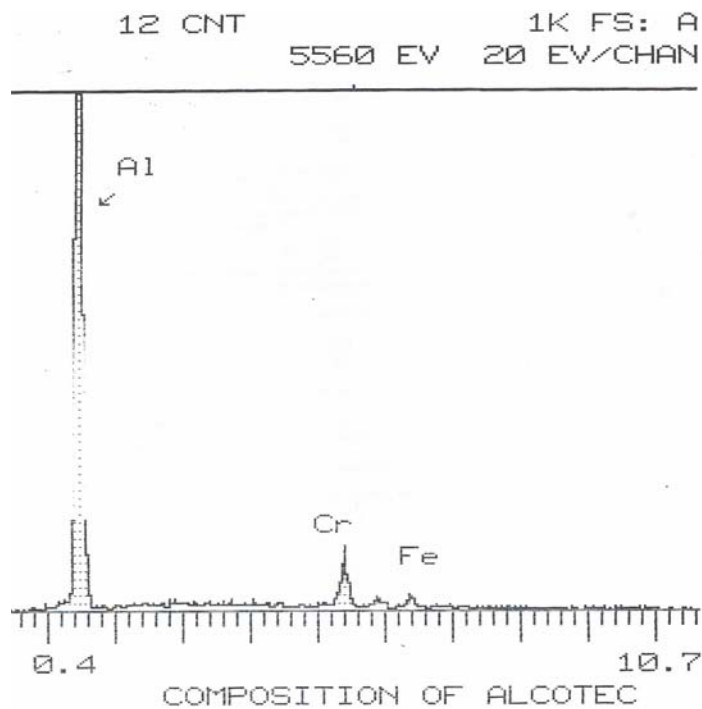


Figure 39. EDAX analysis for the composition of Alcotec

2.2 Mechanical Testing

The slow strain rate method of testing is used in this research work for the quantification of the degree of hydrogen re-embrittlement of the AISI 4340 steel, used in undercarriage components of commercial and military aircraft. A slow strain rate test allows time for hydrogen to diffuse in the sample structure, and hence, the embrittling effect of hydrogen can be detected. Ideal strain rates for observing the effects of hydrogen embrittlement are in the range of 10^{-6} and 10^{-5} s^{-1} . [101] In this work the strain rate was set to $1.1 \times 10^{-6} \text{ s}^{-1}$.

2.2.1 Specimen Design

The tensile specimens were prepared from high strength AISI 4340 steel bar by CNC machining, so as to control the dimensions within close tolerances. The specimens were prepared according to the specifications shown in Figure 40, to give a tensile load bearing circular area of approximately 4 mm^2 . This cross sectional area in the

2.2.3 Quality Control

The quality control of the tensile specimens was carried out as follows. Optical inspection before cleaning determined any evident defects in the machining of the specimens. Moreover, once cleaning had occurred, further inspection was performed with the measurement of the diameters along the specimens' gauge length. If any defects like machining marks or an uneven diameter were found, the specimen was discarded.

In order to check if the original heat treatment was consistent with that of the other specimens, hardness measurements with an applied load of 10 kg were performed on a clean, flat section of all the tensile specimens providing Vickers pyramid numbers, from which UTS values were deduced and compared. The applied principle for the correlation between the hardness measurements and the estimated UTS is that during hardness testing, the strain rate resulting from the indentation is large, thus not allowing hydrogen sufficient time to diffuse to high stress fields before failure occurs, and as a result hydrogen embrittlement is not observed. Therefore, the size of the indentation can be related to the UTS of the non-embrittled specimen. [120]

The reproducibility of hardness measurements for each of the two heat treatment batches of the tensile specimens was acceptable, as the mean UTS of the specimens of the first batch was 1824 ± 24 MPa (10 specimens), and for the second batch the mean UTS was 1800 ± 42 MPa (11 specimens). By applying a Student's *t*-test in these two populations with a percentage of confidence of 99%, it cannot be assumed that these two sets of results belong to different populations.

2.2.4 Tensile Specimen Preparation

Before the tensile specimens were tested, the oil on the surface of the gauge length, present as a result of heat treatment, had to be carefully removed. Firstly, the gauge length of each specimen was polished with a 1200-grit carborundum paper, so as not to alter the diameter of the specimens by removing any of the metal. Afterwards, degreasing in an ultrasonic bath containing propan-2-ol was performed for 15 minutes. Subsequent to degreasing, the diameter of each specimen was measured with

a Mitutoyo micrometer or on a Mitutoyo Profile Projector Type PJ-300. Several diameter measurements were completed over the length of each specimen's gauge, with the average being calculated and the reduction in area being possibly used later as the criterion and the basis for the determination of the embrittlement index. In general, the gauge dimensions of the specimens affect the observed embrittlement, as specimens of larger diameter would necessitate higher absorption of hydrogen over a longer period of time before the formation of a critical hydrogen concentration leading to failure.

After this preparation, the uncoated steel tensile specimens were coated with various coatings. Some specimens were electroplated with cadmium, which is the reference material, with which the alternative aluminium-based coatings are compared. The other test specimens were coated with SermeTel CR984-LT or plated with Alcotec Galvano-Aluminium. If it was required by the specification of each experiment, baking of the coated specimens could be performed. Hence, control tests were carried out on unplated specimens to examine the response of these specimens in the unbaked, as well as baked condition, and investigate if any microstructural change had occurred. Repeating each set of conditions produced a set of results that could be examined statistically.

2.2.5 Cadmium Electroplating Process

Cadmium electroplating was performed by Stotfold Plating Limited, Stotfold, Hertfordshire. Tensile specimens were initially cleaned by abrasion followed by degreasing in propan-2-ol, as described in the tensile specimen preparation section. Pickling, anodic and caustic cleaning were avoided, in order to avoid hydrogen from being taken up by any other means apart from plating. The plating process used a rack method of plating from an alkaline cadmium cyanide bath and the specimens were plated to a thickness of 8 microns. No further passivation treatment was given to the specimens after plating, as seen from an EDAX analysis which proved that the Cd electrodeposition performed by Stotfold Plating contained only Cd. (Figure 41) Subsequent to plating the specimens were immediately placed in liquid nitrogen until mechanical testing began. In this way, mobile hydrogen was virtually immobilised in the tensile specimens until they were reheated in room temperature before testing.

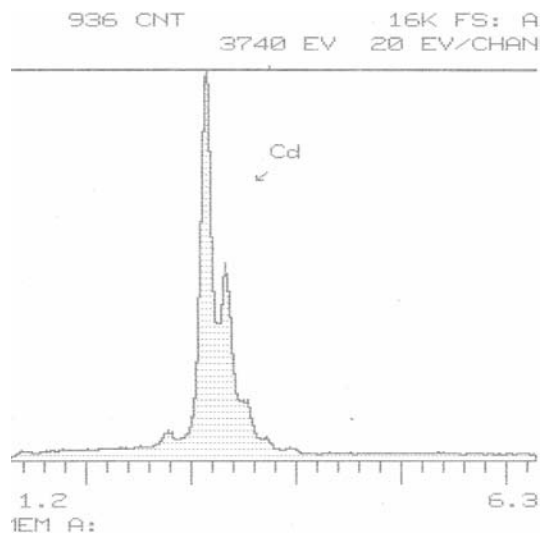


Figure 41. EDAX analysis for Cd plated on steel panel

2.2.6 Application of SermeTel CR984–LT Coating

SermeTel CR984–LT was one of the two aluminium–based coatings selected for study in the programme as possible alternatives to cadmium. SermeTel CR984–LT is the most suitable of the products of Sermatech International Inc. for use with AISI 4340 steel, as it is cured between 163–191°C, thus not affecting the tempering treatment of the steel. The application process of metallic–ceramic aluminium pigmented coatings, under which SermeTel CR984–LT is classified, is performed by spray painting. [5] In this work steel specimens were coated with SermeTel CR984–LT by Sermatech International Inc, based at Ripley in Derbyshire. The surface of the steel was roughened by abrasion prior to coating and the thickness of the coating was approximately 30 µm.

2.2.7 Application of Alcotec Galvano–Aluminium Coating

Alcotec Galvano–Aluminium was the other aluminium–based coating investigated. Steel specimens were coated with Alcotec Galvano–Aluminium by Alcotec GmbH, located in Vilkenrath near Cologne in Germany. The thickness of the coating was approximately 20 µm. The coating contained 99.99% Al in a highly reactive state and was electrodeposited from an organic electrolyte operating at 100°C, thus not affecting the tempering temperature of steel, and utilising a toluene electrolyte with

additions of Al alkyl complexes, like Triethylaluminium. The organic nature of the process necessitated that oxygen be excluded from any contact with processing chemicals. Therefore, the processing plant was sealed into a gas tight structure. [14, 15]

There were four steps involved in the electroplating process including activation (cleaning) followed by aluminium deposition, with a subsequent re-activation and finally a passivation post treatment. The first activation and the Al deposition were non-aqueous processes, carried out under nitrogen, whilst the second activation and the passivation were conventional atmospheric aqueous processes. The passivation post treatment of the Alcotec plated steel specimens in this project was based on Chromium VI. [14, 15]

2.2.8 Baking Treatment

Coated tensile specimens, which were proven to cause embrittlement as a result of their application process, were subjected to post-application baking treatment. A typical example, applied as well in this work, are the cadmium plated AISI 4340 steel tensile specimens. Cadmium electroplated specimens were immediately placed in liquid nitrogen at Stotfold, and baking was performed at Cranfield after they had reached room temperature. De-embrittlement baking treatment was carried out in a vacuum oven for 24 hours at 200°C complying with the specification of BAE Systems ABP1-4041 [3] and the Draft Dowty specification PS 131 about ultra high tensile steels [4]. Similar baking treatment was implemented in the case of any other coated steel tensile specimens that were showing embrittlement, i.e. their times to failure were shorter than the mean time to failure for the unplated control specimens.

2.2.9 Re-Embrittlement of AISI 4340 Steel

A range of corrosive conditions was selected for the investigation of the severity of hydrogen re-embrittlement on AISI 4340 steel specimens, coated with Cadmium, SermeTel and Alcotec. The exposure to the corrosive environment was either preceding the SSRT or occurring during the test. Immersion in 3.5% NaCl solution and neutral salt spray testing, complying with ASTM B117-94 standard [121], were

used in the laboratory. Moreover, other tensile specimens were exposed to a coastal marine atmosphere for two years at a QinetiQ test site in Weymouth, Dorset, as marine atmosphere exposure is considered to represent most closely the service conditions on an aircraft.

Additional uncoated steel tensile specimens were exposed to 3.5% NaCl solution and held at the electrochemical potential of a corroding coating to investigate the effects of electrolytic hydrogen charging when a coating corrodes in service. In these SSRTs, the tensile specimens were either exposed to the salt solution before the test or exposed during the test only. The following Table 4 summarises the full range of the corrosive conditions applied for the study of the re-embrittlement of coated steel specimens.

Specimen	Test Condition	Exposure
Coated with Cadmium, SermeTel and Alcotec	3.5% NaCl	Simultaneous
	Salt Spray	Pre-exposed
	Marine Atmosphere	Pre-exposed
Uncoated	Applied Potential in 3.5% NaCl Solution	Pre-charged
		Simultaneous

Table 4. Summary of re-embrittlement SSRTs.

2.2.10 Slow Strain Rate Test (SSRT) Procedure

After the preparation stage, each tensile specimen was mounted into the Cortest Incorporated, C.E.R.T. system sensotec D/3971-03 SSRT rig. A constant pre-load of 300 N was established, in order to align the specimen, remove any slack in the equipment and standardise the testing procedure with a consistent starting point for all the tested specimens. The information obtained initially from these tests was load versus time graphs, recorded on a chart recorder during testing. Bearing in mind the cross sectional area and the set strain rate of $1.1 \times 10^{-6} \text{ s}^{-1}$, the load-time measurements were converted into stress-strain diagrams for all the strained specimens.

Once the SSRT specimens were broken, the fractured ends of the specimens were measured with the help of the profile projector, in order to determine if a reduction in area had occurred. Afterwards, the fractured ends were cut to a suitable length and mounted on stubs, so as to examine the fracture surfaces on the SEM. These observations were very useful to determine the fracture mode of the tensile specimens combined with the first indication given by the reduction in area and the time to failure, either as plain numbers or as the bases for the definition of an index for the degree of embrittlement. Two principal modes of fracture were expected; ductile type for non-embrittled specimens and brittle type for embrittled ones.

2.2.11 Embrittlement Index

There are various ways for the investigator to represent the degree of embrittlement measured in the SSRT, as there are no generally accepted standards for the method. It is usual to define the embrittlement index either based on the time to failure or the reduction in cross sectional area at the point of fracture. In this work the time to failure criterion was chosen and the reference value was taken to be the mean time to failure of all uncoated, i.e. non-embrittled, specimens. Hence, the embrittlement index (EI) is defined as

Equation 28:

$$EI = 1 - \frac{t_f}{t_o}$$

where t_o is the mean value of the times to failure of all the uncoated control specimens and t_f is the time to failure for every test specimen individually.

The reason for the selection of time to failure as the basis for the definition of the EI is justified on the account of a more reliable and easier quantification of the degree of re-embrittlement or embrittlement in general. If reduction in area (RA) were selected as an EI criterion, there would have been EI figures in the range from 0 to 1 for the specimens failing after necking. On the other hand, all specimens failing before necking would have had EI values equal to zero no matter whether they had failed at different times to failures. Consequently, there would not be any distinction in the

degree of embrittlement for these specimens. However, this distinction is feasible when using the time to failure as the basis for the EI definition, as there are dissimilar EI values for specimens failing at different times. This renders the choice of the time to failure criterion a reliable and linearly proportional way to quantify with a single number the extent of embrittlement.

2.2.12 Weibull Model of Failure Times

It is common for replicate hydrogen embrittlement tests, carried out under apparently identical experimental conditions, to result in a range of times to failure due to the variation in the number, size and distribution of defects in the microstructure of the specimens. Weibull statistical analysis was used in this work, in order to distinguish between the effects of each of the experimental variables. More information about the Weibull model of failure times in terms of its mathematical implications, its evolution through the previous five decades, as well as its adaptation to the failure of metals are available in Chapter 1 and the Appendices. However, a brief explanation of the implementation of the Weibull model to the results of SSRTs in this project is presented below.

Equation 29 demonstrates how the probability of survival P_s for an ordered specimen can be calculated from a total set of n specimens,

Equation 29:

$$P_s = 1 - P_f = 1 - \frac{i}{n + 1}$$

where i is the ordered specimen number, n the population of specimens and P_f the probability of failure. Should the probability of survival be set up, the Weibull probability could be applied using Equation 30.

Equation 30:

$$P_s = e^{-x(t-t_i)}$$

The shape parameter x , termed the Weibull slope, represents the probability per unit time that during time, t , a crack will appear in the specimen of sufficient size to cause failure. Otherwise expressed, the slope x gives the scatter of failure times and is an indication of the range of defect sizes within the specimens. The value of x can be obtained from the negative gradient of a graph of $\ln P_s$ against time t . On the other hand, this is the mode that Weibull plots are calculated and presented in this work. Concerning t_i , this is the minimum crack incubation time, below which there are no failures, i.e. when $P_s = 1$ or $\ln P_s = 0$.

2.3 Permeation Measurements

The hydrogen permeation technique was implemented in the research programme as an alternative method for the assessment of the risk of re-embrittlement of the steel substrate. In particular, permeation studies were concerned with determining the extent of hydrogen uptake resulting from the corrosion of a sacrificial aluminium-based coating. The amount of hydrogen absorbed by the metal is measured quantitatively using a modification of the double cell permeation technique of Devanathan and Stachurski [65], details of which were presented in Chapter 1.

The general arrangement of this method is shown in Figure 42, where a thin mild steel shim electrode was the lone material isolating two different electrochemical cells. The mild steel shim, supplied by Knight Strip Metals, Potters Bar, was cut to shape and abraded with 1200-grit carborundum paper on both sides of the membrane before being degreased using propan-2-ol. One surface of the membrane acted as the cathode in the left hand side cell (cathodic cell), and the other surface acted as the anode in the right hand side cell (anodic cell). Consequently, the shim electrode was held in place between the rubber washers of the anodic and cathodic cell adapters by metal bulldog clips. Vacuum grease was applied between surfaces to ensure a watertight seal.

The anodic (exit or detecting) cell was firstly used to deplete hydrogen from the anodic surface of the mild steel shim, as the shim was maintained at -94 mV (SCE) by a Sycopel Scientific AEW-2 potentiostat connected to a laptop, and using a Sycopel electrochemical software, in order to allow the oxidation of the hydrogen

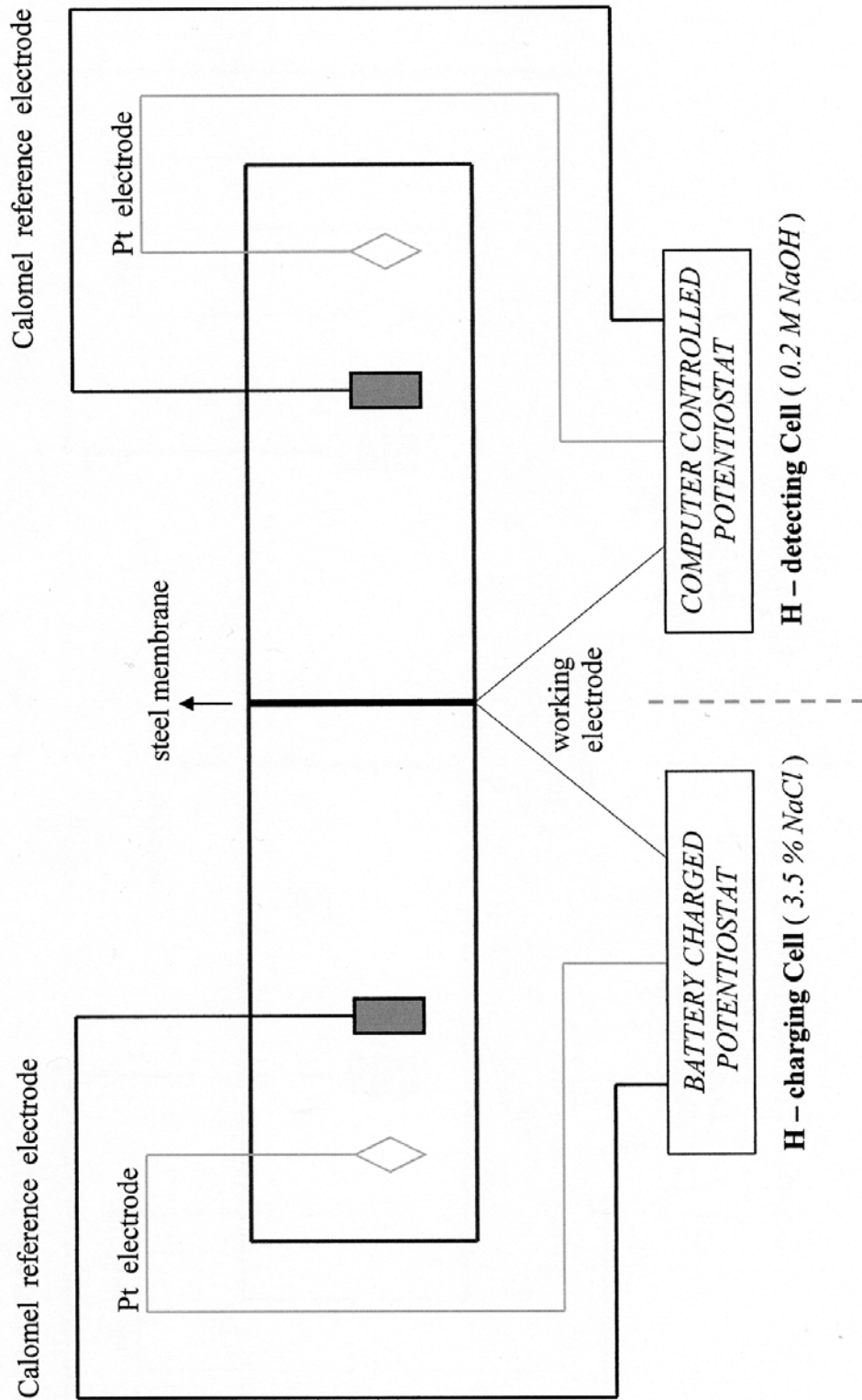


Figure 42. Double permeation cell

reaching its surface. The used electrolyte in the exit cell was 0.2 M NaOH solution and the removal of hydrogen ions at the previously referred potential was maintaining the anodic surface of the membrane at a zero hydrogen concentration. The above described arrangement in the anodic cell was used in all the performed permeation measurements.

When a satisfactory background level had been reached, the cathodic cell was filled with 3.5% NaCl and the unplated steel membrane was potentiostatically charged at the electrochemical potential of a corroding coating with the help of a battery charged Sycopel Scientific Multistat 2 potentiostat, in order to reduce electrical noise. This produced a current that was measured in the detecting cell after the elapse of a period of time, termed as the breakthrough time. This current corresponded to the hydrogen uptake of the membrane, as from the hydrogen oxidation reaction there is one electron corresponding to a hydrogen atom. The hydrogen uptake measured by this approach is equivalent to that in the case of a coated steel membrane surface on the charging side with a scratch introduced into it, and with the advantage of the elimination of the need to measure the area of the exposed steel.

2.4 Corrosion Testing

A wide range of corrosion tests can evaluate the sacrificial corrosion protection on steel specimens provided by the investigated metallic coatings. The aluminium-based coatings were tested both as detached coatings to measure the inherent corrosion behaviour of the materials, as well as coupled to steel, in order to simulate the corrosion behaviour of a coated fastener. In the case of detached coatings the linear polarisation resistance (LPR) technique was used, together with the recording of the free corrosion potential of coatings. In the case of coatings coupled to steel, galvanic coupling measurements were performed using a zero resistance ammeter (ZRA), including the recording of both galvanic current and coupled potential of coatings to steel. The galvanic coupling measurements were complemented by the determination of the electrochemical polarisation curves for the various couples of coatings to steel. Moreover, coated steel panels and tensile specimens were exposed at a coastal test site, as marine atmosphere exposure conditions were considered as most closely representing the service condition on an aircraft. Finally, salt spray testing conditions

were implemented for the study of the corrosion behaviour of coated steel panels and the assessment of the risk of re-embrittlement of coated steel tensile specimens subjected to this aggressive corrosive environment.

2.4.1 Linear Polarisation Resistance (LPR) Measurements

The corrosion rate of the coatings alone was measured using the LPR technique. A piece of coated steel was masked using Lacomit so that only an area of 40mm x 25 mm of the coating (SermeTel, Alcotec or Cadmium) was exposed. It was then suspended in a 3.5% NaCl solution as the working electrode (WE) of an electro-chemical cell, together with a platinum auxiliary or counter electrode (CE) and an external calomel reference electrode (RE). Moreover, a luggin capillary was placed in the vicinity of the test specimen to provide solution contact with an external saturated calomel RE.

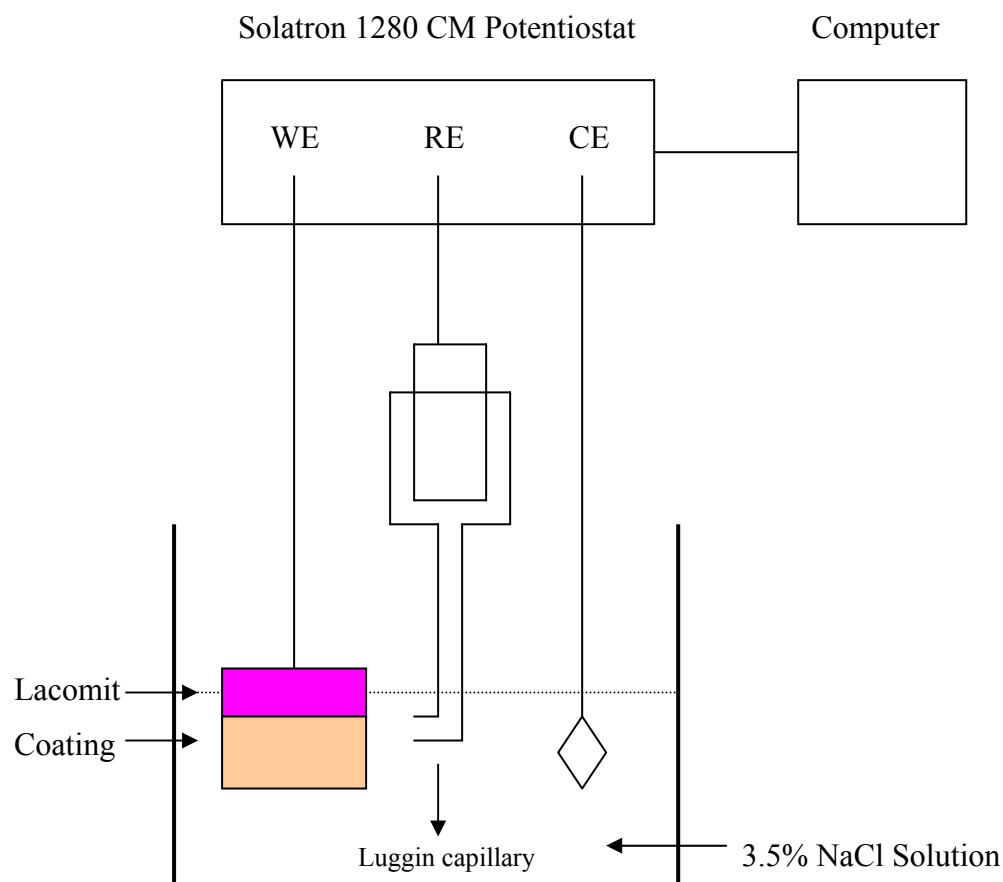


Figure 43. Experimental arrangement for LPR measurements

LPR scans were performed using a Solatron 1280 CM computer controlled potentiostat, as viewed on the experimental setup depicted in Figure 43. Before the beginning of every polarisation scan the open-circuit potential of the coating was recorded. Afterwards, the potential E of the coated specimen was anodically polarised by 10 mV, then polarised cathodically by 20 mV (i.e. returned to its rest potential and a further 10 mV below it), and finally polarised anodically by 10 mV to the rest potential again. The applied scan rate was 10 mV/min and the resulting current that passed (ΔI), following to the change in potential (ΔE), was recorded 25 times per minute at equal intervals. Further LPR tests were performed for each coated steel panel over longer immersion times of more than 1000 hours, where sweeps and potential measurements were conducted typically once every 24 to 96 hours. Finally, the calculation of the polarisation resistance (R_p) is enabled by Equation 31.

Equation 31:

$$R_p = \frac{\Delta E}{\Delta I}$$

The polarisation resistance is the resistance to the flow of corrosion current and it can be directly converted to give the corrosion rate of the coating. Finally, the data recorded from these tests are the trends in the free corrosion potential and the polarisation resistance for each coating during the experimentation time.

2.4.2 Galvanic Coupling Measurements

The general arrangement of this experimental method is shown in Figure 44. A coated steel panel was coupled with a steel panel of the same composition (BSEN 10130.1991). Both panels were masked with Lacomit so that only 10 cm² of each material were exposed in 3.5% NaCl solution. These two panels are the working electrodes, which with a reference one, were connected to a ZRA. As there were more than one set-ups being monitored, a 12-channel system with a ZRA per channel was used. Moreover, each channel had the ability to monitor the couple potential. This system was an instrument, called GalvoGill 12, and manufactured by ACM Instruments in Cumbria.

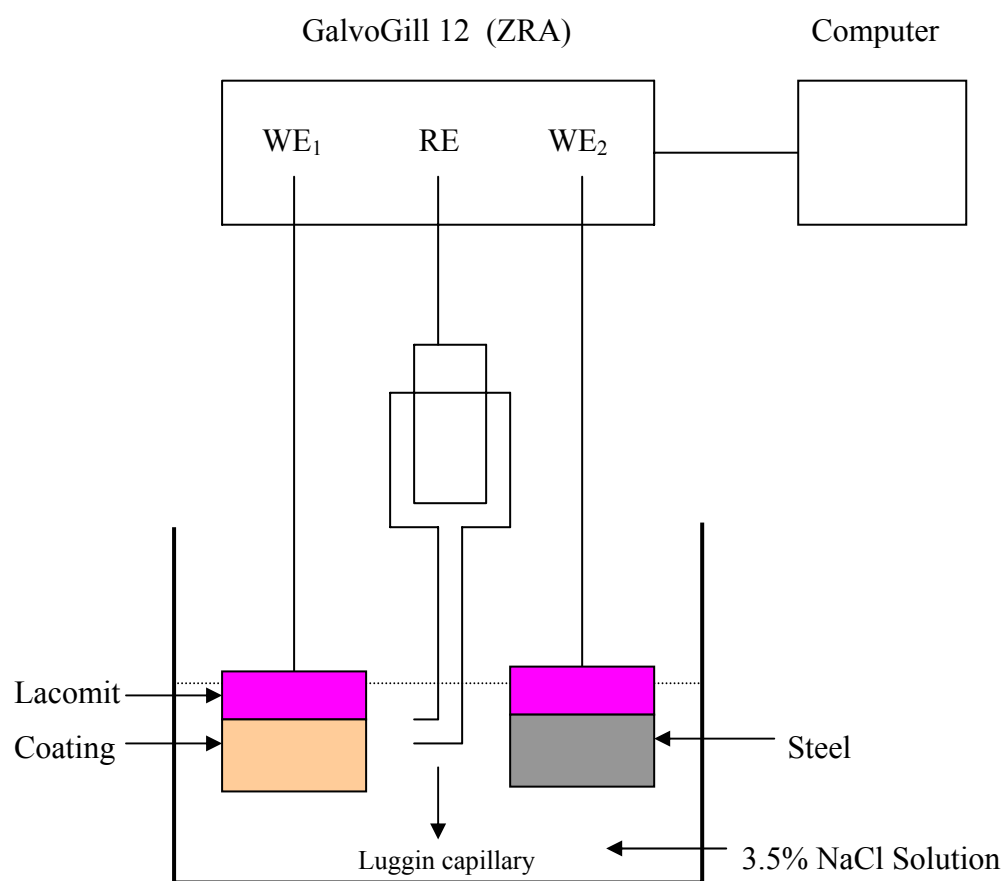


Figure 44. Experimental arrangement for galvanic coupling measurements

2.4.3 Determination of Polarisation Curves

The galvanic coupling measurements were accompanied by the determination of the electrochemical polarisation curves for the various couples of coatings to steel. Each of these curves depicted potential versus current for both electrodes of the coating/steel couple on the same plot, and were a useful diagnostic tool to understand if the electrochemical phenomenon was under anodic, cathodic or mixed control, and hence, it was easier to attempt an interpretation of the galvanic coupling results.

To achieve this determination, the experimental procedure consisted of the measurement of both electrode (steel and coating) potentials, with respect to a calomel reference electrode. They were first disconnected from the ZRA and a resistance box was connected between the two electrodes. A large resistance value, like 1 M Ω , was initially chosen that was then lowered in steps, resulting in 10 or 20 ohms eventually. For every resistance value R, there were two measured potential

values, E_{steel} and E_{coating} , and hence, the current allowed to flow between the two electrodes was given by the ratio $I = |E_{\text{steel}} - E_{\text{coating}}| / R$. The curves were easily plotted by correlating the logarithm of each current value with the correspondent E_{steel} and E_{coating} values. Whenever this experiment was performed, the galvanic coupling experiment was just interrupted for no more than half an hour, in order to deter any dramatic change in the characteristics of the various cells.

2.4.4 Marine Atmosphere Exposure Testing

This type of testing was required for the evaluation of the corrosion behaviour of the aluminium-based coatings, as well as the assessment of the risk of re-embrittlement of the coated steel fasteners through the performing of SSRTs.

In the first case, coated steel panels were exposed at a coastal test site, as this environment is considered to represent most closely the service condition on an aircraft. The effectiveness of the coatings was assessed by recording the time for the first appearance, as well as the extent, of 'red rust' corrosion products on the steel.

In the second case, tensile specimens machined from the high strength steel were coated with one of the aluminium-based materials and then exposed to a corrosive environment. The extent of hydrogen re-embrittlement of the steel was then assessed by measuring the times to failure of the specimens in SSRTs and comparing them with those for uncoated specimens. In general, the coated tensile specimens were firstly exposed at a coastal test site and then subjected to the SSRT.

In the beginning of November 2000 six unscribed coated steel panels and 59 coated tensile specimens were prepared and sent for marine atmosphere exposure trials to QinetiQ Bin Cleaves, in Weymouth, Dorset. These consisted of 2 steel panels and 20 tensile specimens coated with SermeTel CR984-LT, 2 panels and 20 tensile specimens coated with Alcotec Galvano-Aluminium and finally 2 steel panels and 19 tensile specimens were Cadmium plated and baked, as described in a previous section.

The tensile specimens and the panels were mounted in three plastic holders, the general arrangement of which is shown in Figure 45. Nylon lock nuts were used on

the underside of the plastic holders to secure each of the tensile specimens. The panels were held by nylon nuts, bolts and spacers so that they are clear of the holder. Finally, the holders were mounted on frames, at the test site, a view of which is portrayed in Figure 46.

The commencement date of the tests was the 17th November 2000, whilst almost exactly a year afterwards (15th November 2001) another nine coated panels began to be exposed at the marine atmosphere of Weymouth, but this time the coated panels were scribed, so as to expose some steel surface to this corrosive environment, and simultaneously challenge the sacrificial corrosion protection ability of each of the coatings. All the coated specimens and panels were brought back to Cranfield on the 20th of November 2002 and SSRTs immediately started with some of the tensile specimens, whilst photographs were taken for both panels and tensile specimens, showing the effects of the coastal site environment on them.



Figure 45. Arrangement of specimens and panels exposed to marine atmosphere.



Figure 46. Overview of the coastal test site

2.4.5 Salt Spray Testing

Salt spray conditions were another corrosive environment selected for the evaluation of the corrosion behaviour of the SermeTel and Alcotec coatings and the assessment of the risk of re-embrittlement of coated steel tensile specimens, as a result of the corrosion of the previously referred coatings. The salt spray test is considered to be as a more severe corrosion condition, compared to other tests, such as the marine atmosphere exposure. However, it is a case of aggressive environment useful to be studied, especially, when comparing the sacrificial corrosion protection effectiveness of various coatings.

Salt spray tests were performed twice for the objectives of this work. In the first case nine tensile specimens (3 coated with Cadmium, 3 with Alcotec and 3 with SermeTel) and three panels, each one of these coated with one of the previously referred coatings, were tested. Concerning the second test, another nine tensile specimens,

coated with the same coatings and the same number of specimens per coating, were subjected to the salt fog conditions.

All these specimens were tested in an ASCOTT S450/S salt fog cabinet for 1000 hours. The solution used in the test was a 5% w/w NaCl solution with a pH value of 6.5 complying with the ASTM B117–94 [121] standard of the range between 6.5 and 7.2. The pressure to produce the fog was 17.5 psi on average. The temperature of the humid air was 45°C and the temperature of the cabinet was 35°C. Moreover, the positions of the specimens in the cabinet were changed at regular intervals to allow for any differences in the distribution of salt fog. Measurements showed that the fall rate at different points ranged between 1.0 and 2.0 ml/h for a surface of 80 cm², which, on the other hand, is the range permitted by ASTM B117–94 standard. After the end of the tests all the specimens were photographed and afterwards, the tensile specimens were placed in liquid nitrogen, in order to deter any hydrogen, entrapped during the tests, from diffusing out of the specimens prior to slow strain rate testing.