

- a) Rate of general corrosion.
- b) Mechanism, by using the effect of flow as a diagnostic criterion.
- c) The characteristic hydrodynamic parameters.
- d) The composition of the solution by electro-analytically monitoring compositions, or by measuring redox potentials, pH's etc.

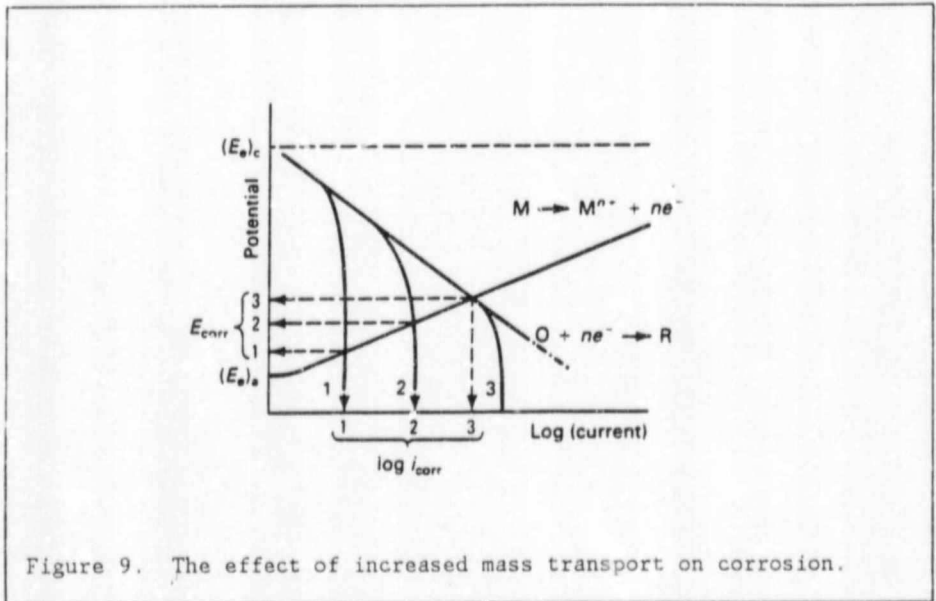
2.7.1 HYDRODYNAMIC CONCEPTS

At fairly low relative velocities between the wall of a pipe line and a fluid, the flow is laminar, i.e. streamlines in the flow remain parallel. Above a critical velocity dependent on the temperature, fluid and geometry of the pipe, the flow becomes turbulent. To define whether a particular flow regime is turbulent or laminar, the dimensionless Reynolds Number is used where:

$$Re = \frac{v \cdot d \cdot \rho}{\mu} = \frac{v \cdot d}{\nu} \quad (2.16)$$

For pipe flow the critical Reynolds Number is approximately 2300.

Even under turbulent flow conditions, there will always be a thin laminar sublayer at the flow boundary, caused by the viscous drag on the fluid by the wall. Within this laminar sublayer is a diffusional (or mass transfer) boundary layer, in which the relative velocity is small, or even zero. In this boundary layer the rate controlling phenomena with respect to corrosion is usually ionic diffusion. Outside this layer, increased velocity and turbulence cause convective diffusion to be the main mass transport phenomena. Figure 9 shows the effect of increasing transport rate to the corroding surface (6).



The thickness of the boundary layer is denoted δ_d and that of the laminar layer denoted δ_h . The relationship between the thickness of these layers is governed by the dimensionless Schmidt Number (Sc) defined by:

$$S_c = \frac{U}{D} = \frac{\mu}{D} \quad (2.17)$$

The relationship is:

$$\frac{\delta_h}{\delta_d} = \frac{1}{3} \quad (2.18)$$

This relationship holds if the two layers develop from the same point, which is a reasonable assumption in most cases. For a flat plate the thickness of the diffusional boundary layer is given by (27):-

$$\delta_d = 0,38 \times Re^{0,2} \quad (2.19)$$

It may also be assumed that the rate of mass transfer across this boundary layer is inversely proportional to the thickness of the layer (δ_d).

Associated with the velocity gradient which exists across these boundary layers is a frictional force which may be expressed in terms of an average surface shear stress (τ_s). It has been suggested that the mechanical stability of surface films is controlled by the shear stress. This shear stress varies with velocity, viscosity and other hydrodynamic parameters, but is typically less than 6.9×10^{-3} Pa for nominal velocities up to 30 ms^{-1} (18).

The overall transport to the corroding surface consists of bulk convection and turbulent convection. With the former, there is no transport of fluid along the pipe, and with the latter there is an exchange of fluid between the boundary layer and the bulk. Both depend on the concentration driving force, bulk convection due to D and turbulent convection which is also a function of the eddy diffusivity ϵ (units $\text{m}^2 \text{ s}^{-1}$). The overall resistance to diffusion is seen as the sum of the resistance of the laminar and turbulent regions. Empirically a mass transfer coefficient (K) has been defined as:

$$K = \frac{\text{rate of reaction}}{\text{concentration driving force}} \quad (2.20)$$

Mass transfer rates may be expressed in a non-dimensionless group using the Stanton number (St) or the Sherwood number (Sh). The Sherwood number is then defined as:

$$Sh = \frac{Kd}{D} \quad (2.21)$$

and the Stanton number as:

$$St = \frac{K}{V} \quad (2.22)$$

and thus:

$$St = \frac{Sh}{ReSc} \quad (2.23)$$

Empirical correlations have given:-

$$Sh = \text{constant } Re^x Sc^y \quad (2.24)$$

where $x \approx (0,3;1)$ and $y \approx 0,33$. For a reaction that is diffusion controlled

$$\text{rate} = K \cdot \Delta C \quad (2.25)$$

In electrochemical terms, when the rate equals the limiting current density, LCD, this gives:

$$LCD = Knf\Delta C \quad (2.26)$$

If this approach can be applied to a corrosion process in which both an anodic and cathodic reaction occur then the corrosion current is given by (assuming neither reaction is controlled by diffusion i.e. concentration polarisation):

$$\text{corrosion current} = nF\Delta C K \quad (2.27)$$

The value of ΔC will be the bulk concentration of cathodic reactant for cathodically controlled systems. It will equal the solubility limit of the relevant product when the anodic reaction is diffusion controlled. Then substituting for K:

$$\text{Corrosion current } i_{\text{corr}} = nF \Delta C D \frac{D}{d} \left(\frac{vd}{D}\right)^x \left(\frac{D}{\delta}\right)^y x \quad (2.28)$$

i.e. if the relevant parameters were known then the corrosion rates could be predicted without tests.

2.7.1.1 Fully developed tube flow

The Chilton-Colburn analogy (27,28,17) gives the values of x and y in equation 2.24 as 0,8 and 0,33 respectively and the constant as 0,023. This then gives:

$$Sh = 0,023.Re^{0,8}Sc^{0,33} \quad (2.29)$$

From all these and rearranging (2.26)

$$K = \frac{LCD}{nFAC} \quad (2.20)$$

This allows the mass transfer coefficient to be obtained by electrochemical techniques and allows a basic correlation to be made with different pipe sizes and flow conditions.

2.7.2 HYDRODYNAMIC EFFECTS ON PITTING

It is only fairly recently that properly characterised test systems have been used to perform corrosion testing under flow conditions, (characterised as shown in the previous section).

The literature is fairly extensive on the behaviour of various alloys, especially those used in heat exchanges under flow conditions. However, the hydrodynamic variables are often not quoted. This creates problems in making comparisons between different piping, sizes and geometries. There is a dearth of information in the literature on flow loop testing under controlled conditions.

Beck and Chan (22) conducted work on the hydrodynamic effects on the growth of small pits in a flow loop. They created artificial pits on a

specimen surface, and then using a potentiostat, monitored the pitting current. They found an increase in the pitting current with electrolyte velocity past the pits, which agrees with the predictions from a mass transport current density. Figure 10 shows the effect of flow and no flow on pitting current.

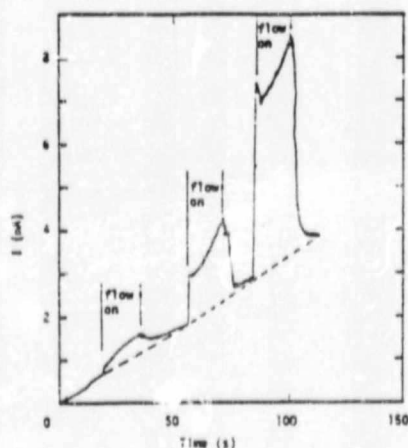


Figure 10. The effect of flow on pitting current.

Karaminezhad-Ranjbar et al (34) conducted tests on the pitting of Inconel 600 in high temperature chloride solutions in a well characterised annular flow channel. Their premise was based on the literature stating that localised attack might be expected to occur at points on the surface at which the passive film exhibits instability resulting in high local shear forces. Thus hydrodynamic factors could influence the distribution and morphology of pitting attack. The breakdown potential could also be affected if the chemical and electrochemical properties of the film are modified by fluid flow. For this particular alloy-corrodent combination there was little effect of the flow rate on the breakdown potential. The major effect of flow rate was on pit morphology with a smaller number of pits forming at high flow rates than at low flow rates, but these pits

were larger in size. There was also a tendency for the pits to elongate in the direction of flow. However, flow velocity may be beneficial especially for active/passive materials. For example type 316 stainless steel has been shown to pit in static sea water, but not in moving seawater (6). This is because when the water is moving, the mass transfer rate of oxygen to the metal surface is sufficient to maintain the passive layer, but with no flow the transfer rate is too slow and the surface cannot maintain its passivity.

2.8 MINING CONDITIONS

The mining environment is frequently characterised by extremes. Waters may have very high levels of dissolved and suspended solids, and temperatures varying from 4°C up to 55°C. Piping may be carrying relatively "clean" water internally, but be wotted on the outside by very aggressive waters. Some mines have high sulphate, and chloride levels in their water, and nitrogen/oxygen compounds will be generated during blasting. These will then be oxidised to nitric acid. Most of these mine waters are contaminated with microbial organisms, and control of these is often not economically viable as long as the damage they cause is within acceptable limits.

The largest requirements for water in a mine is for general service, use such as dust suppression, water spraying after blasting, as a coolant in rock drills, for washing down the working face and numerous other uses. Between 0,5 and 5T of water are required per ton of rock removed (38). Service water is usually recirculated, but typically 10-20% of make-up water is required to make up for losses. These losses may be made up from fissure water or by water from the various water supply authorities. In the Rand Water Board area 75 000M³ of water was supplied by this authority to the 22 mines in this area during 1983. Some mines have access to river water and use this. Due to the various sources and uses of mine water, its composition varies considerably.

2.8.1 WATER COMPOSITION

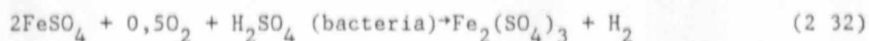
In South African gold mines there are three general groupings of water type.

- a) The Orange Free State mines which have waters that contain high levels of dissolved salts, mostly chlorides and sulphate.
- b) Far West Rand, with medium chlorides and high sulphate ion concentrations.
- c) Rand, with low chlorides and high sulphates.

The highest chloride values noted in the Orange Free State were 2950 parts per million (ppm) at Unisel mine, which also has the extreme sulphate value of 2200 ppm. The average chloride values for this area are 1450 ppm and ± 1000 ppm of sulphate. The Far West Rand averages ± 190 ppm chlorides and ± 630 ppm sulphate.

Many of the mines attempt to reduce the suspended solids level to decrease erosion and erosion-corrosion.

Mine waters near the stope are frequently acidic. This mainly due to iron pyrites being commonly found in gold reef. On exposure to air and moisture, the pyrite is oxidised to ferrous/ferric sulphate and sulphuric acid, the basic reactions are as follows:-



At a pH of around 4,5, the abiotic oxidation of Fe^{2+} slows and bacterial oxidation becomes primarily responsible for producing acidity and ferric hydroxide. This reaction takes place due to the acidophilic bacteria, *Thiobacillus Ferroxidans*. Inhibition of bacterial activity would thus greatly reduce acid formation as the rate of acid production would be determined by the slow abiotic oxidation of FeS_2 in air, rather than by the rapid oxidation by Fe^{3+} .

The acidic water formed as above accelerates the breakdown of clay, other silicates and carbonate minerals, thus increasing the Total Dissolved Solids (TDS) content, the conductivity and the corrosive properties of underground water. Recirculation of mine water increases the TDS concentration and the water may need to be diluted with fresh low TDS water to decrease the dissolved solids concentration.

Treatment of mine water to reduce the suspended solids concentration and produce a more neutral pH is frequently performed. Coagulation/flocculation and settling are used to remove suspended solids usually to a concentration of around 20mg/l. This would be especially important for aluminium which has low resistance to abrasive wear. Abrasion will also continually remove the protective oxide film leading to corrosion.

The pH of mine water is usually controlled within the range 6 - 9 by the addition of lime. However, pH values of 3 and 4 do occur either in ground water or due to a lack of pH control.

Dissolved oxygen levels are generally at the saturation level of 7mg/l depending on the particular temperature and pressure. Levels of dissolved oxygen of less than 4mg/l have been measured, chiefly in fissure water with a high concentrations of sulphides.

2.8.2 WATER TEMPERATURES

Water temperatures vary greatly between a minimum of 4°C and maximum of 55°C in South African mines. The virgin rock temperature increases with depth, in some areas reaching 60°C at a depth of 3000m. There is also a heat gain of ventilating air due to auto compression, giving a wet bulb temperature rise of approximately 4°C per 1000m of depth (38).

In order to maintain a maximum wet bulb temperature of 28°C, the upper limit for safe and productive work, cooling is required in the mines. This is usually done using water chilled in a refrigeration plant and pumped down the mines. This water starts at a minimum of 4°C. Water in the stopes may reach the rock temperature of 55°C or even 60°C. Protheroe (39) notes that in mines an increase of 15°C in the corrosion rate has been found to occur for each 10°C rise in temperature.

2.8.3 FLOW VELOCITIES

These are generally less than 2ms^{-1} and as such there is a negligible erosive effect due to the water alone.

2.8.4 GALVANIC CORROSION

In the mining environment there are bound to be junctions containing dissimilar metals. The majority of presently used materials such as cast iron, mild steel, stainless steel and copper are all cathodic to aluminium alloys. Copper is especially deleterious as it tends to plate out of

solution onto the aluminium surface and promote a very severe pitting attack of surrounding material.

Mansfeld (35) in his paper on the effects of solution velocity on an aluminium alloy/copper galvanic couple found the current vs velocity relationship could be expressed in the form

$$i_g = i_g^0 + k(v)^{0,5} \quad (2.34)$$

2.8.5 ALUMINIUM IN THE MINING INDUSTRY

The inherent corrosion resistance of aluminium and its high strength/mass ratio are attractive to the mining industry. Indeed, aluminium alloys have been used in American and Canadian coal mines since the 1930's to obtain mass savings in equipment that is regularly transported. In South Africa considerable work is being done on the redesign of mine skips using aluminium alloys to allow greater payloads to be carried, for the same maximum winder load. In the 1940's in Canada, aluminium pipes were used in mines to carry steam, clean water, drainage water and air. Where dissimilar metal joints occurred, the pipes were electrically grounded as a whole and disposable sections were inserted at these joints.

As was seen in Figure 6, aluminium is generally regarded as being passive in the pH range of 4 - 8,5, the pH values of most mine water.

Cruise (41) reported the results of tests conducted on various coated steel, aluminium, stainless steel, 3CR12 and plastic pipes. One of his more general, but extremely important findings, was that the degree of corrosivity and mechanical damage increases substantially from a mine shaft towards the stope. This testing involved the use of 100mm pipes, 1000mm long set within a test loop. The flow rate was $7,5 \text{ l s}^{-1}$ which gives a bulk flow velocity of $0,93 \text{ m s}^{-1}$. TDS were 3000 mg l^{-1} of which chlorides

made up 500mg l^{-1} with a pH of 7. The aluminium generally performed "fairly well" under these conditions.

McEwan (42) carried out tests using both the mass losses of coupons and a linear polarisation resistance technique. The aluminium alloys tested were types 1200, Alclad 3003, 3004, 5251, 6063 and 6261.

The corrosion rates obtained were low being of the order of 0,1 - 0,2mm/year from the mass loss coupons and around 0,02 - 0,06mm/year from the LPR method.

Even though general corrosion rates were low, pit depths of up to 3,0mm were measured. The Alclad performed the best in this respect, with a maximum pit depth of 0,82mm. Alloy 5251 (used in marine applications) had the next lowest pit depths, with a maximum depth of 1,85mm. The possibility of microbial attack was noted, but no definite conclusions were made.

3.0 DESIGN AND CONSTRUCTION OF FLOW LOOP AND CONTROLLED TEMPERATURE WATER BATH

3.1 INTRODUCTION

This section is a brief summary of the design of the flow loop and other equipment. The final designs are specified in section 3.3 and the concepts from which the designs were developed are shown in 3.4.

The design procedure taken was to define the problem and its parameters, formulate concepts with reference to existing designs, and then to develop the design to the point of producing working drawings.

3.2 DEFINITION OF PROBLEM

3.2.1 PROBLEM STATEMENT

The effect of mine waters under both static and flow conditions on the corrosion of aluminium alloys is poorly understood.

3.2.2 AIM

The aim was to design and construct a system capable of producing corrosion test data under flow conditions using both electrochemical and immersion testing techniques.

3.2.3 REQUIREMENTS

- The system must not contaminate the water i.e. it must be constructed of corrosion resistant materials.

- The flow rate must be controllable to give a bulk velocity of 0,5 to 3,0ms⁻¹ over the surfaces of the test specimens.

- Control of the water temperature to any set point within the range 5 to 60°C must be available.

- The controlled temperature bath must be capable of containing total immersion specimens in the test water at a fixed temperature in the range of ambient to 60°C.

3.3 SOLUTION SPECIFICATION - FLOW LOOP

3.3.1 TOTAL IMMERSION TEST SECTION

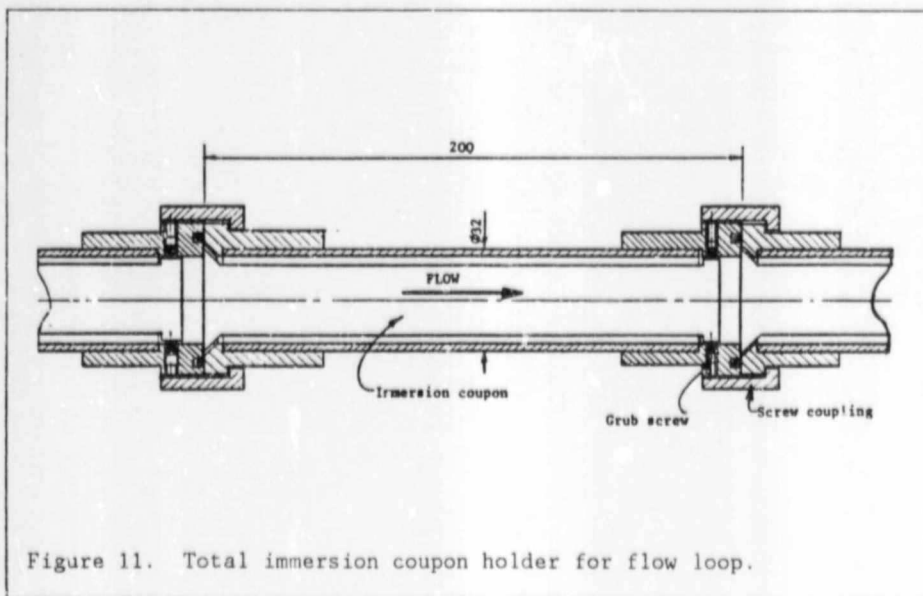
-Capable of holding 10 immersion specimens.

-Constructed from $\varnothing 32$ class 9 unplasticised polyvinylchloride (uPVC) pipe with screw couplings, having machined slots and grub screws in the couplings to hold the specimens - see Figure 11.

-The complete section may be removed and replaced with another, for different length specimens or to hold tubular specimens.

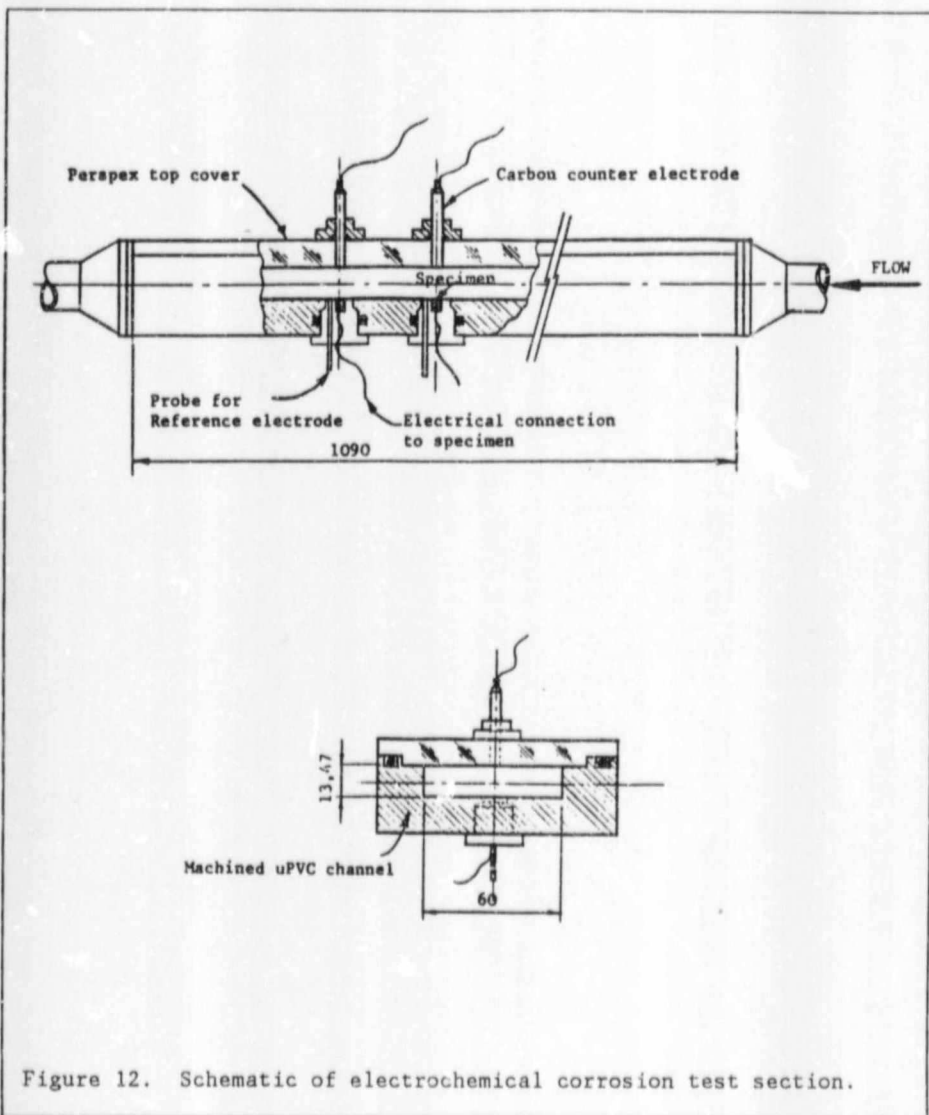
-Length of individual specimen holders - 200mm.

-Overall length of section - 2000mm.



3.3.2 ELECTROCHEMICAL TEST SECTION

- A rectangular flow channel of internal dimensions 60 x 13,47mm capable of accommodating 7 flush mounted electrochemical test specimens (Figure 12).
- Main body of flow channel machined from a 120 x 50mm section uPVC block.
- Cover of flow channel machined from 20mm Perspex sheet.
- Overall length of flow channel 1090mm.
- Reference electrode ports built into specimen mounts.
- $\phi 10$ carbon counter electrodes clamped in position in aluminium holders in perspex cover.
- All joints and entry ports sealed with "O"-ring seals.
- Entry and exit connections to circular pipes are made with glass reinforced plastic transition pieces.
- All screws and bolts type 316 stainless steel.



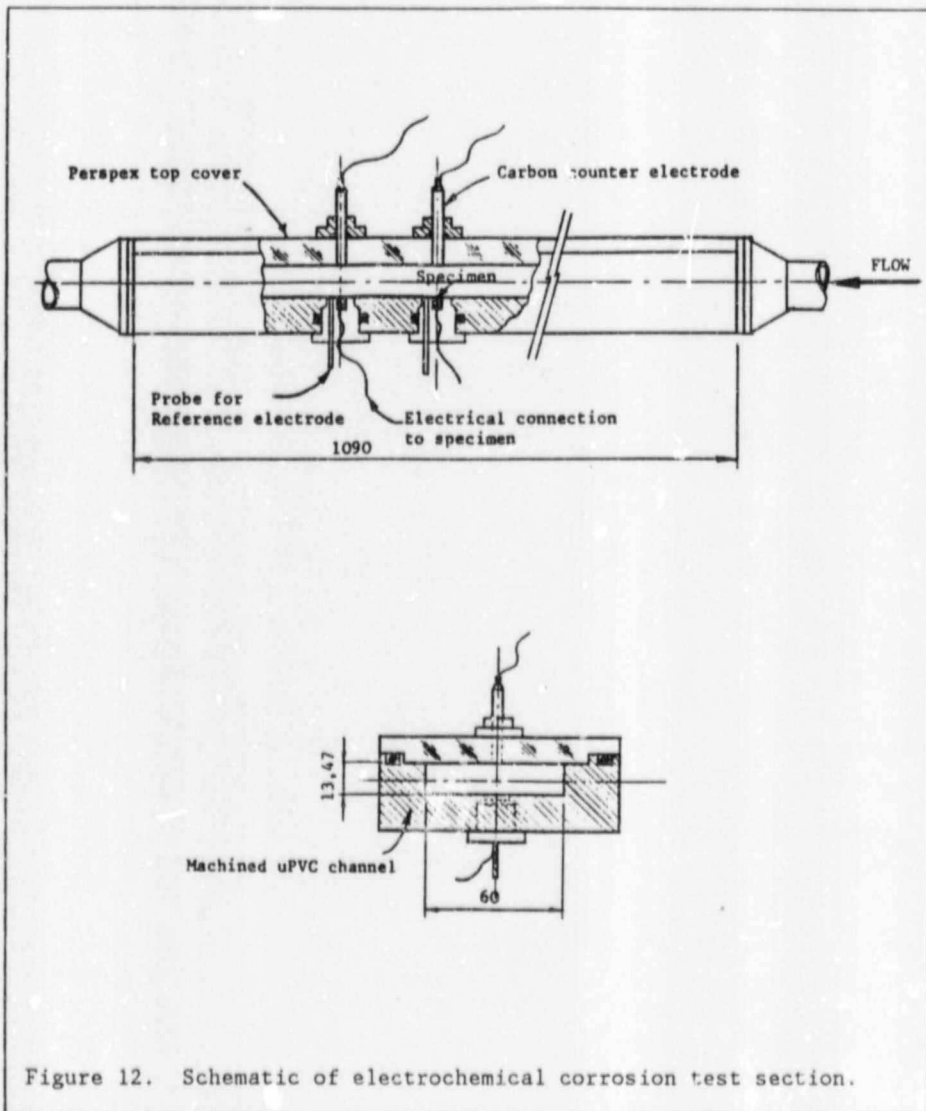


Figure 12. Schematic of electrochemical corrosion test section.

3.3.3 PIPING

-All piping bar flexible hose at pump inlet and outlet, is unplasticised polyvinylchloride of diameters 32mm, 40mm or 63mm.

-Flexible reinforced I.D.40mm polyethylene tubing used at pump inlet and outlet connections.

-All bends, elbows, Y-pieces, T-pieces, flanges, valves and couplings made from uPVC.

-Flange backing plates, cadmium plated mild steel.

3.3.4 PUMP

-One Pumptec PV101, polypropylene, centrifugal pump, fitted with a ceramic mechanical shaft seal, driven by a 0,55kW 220VAC motor. Flow range 1000 to 10 000ℓ/hr.

3.3.5 FLOW CONTROL AND METERING

-Flow rates may be varied by either or both of the following methods:

a) Bypass of the system using a 40mm diaphragm valve to vary the volume of water returning directly to the tank.

b) Increasing the system resistance by partially closing any of the valves in the pipeline.

-Flow metering; Krohne™ Measurements type GA24/R3/6 50mm flow meter, calibrated to read from 1000 to 10 000l/hr. Float, springs, inlet and outlet - Type 316 stainless steel. Metering cone - glass.

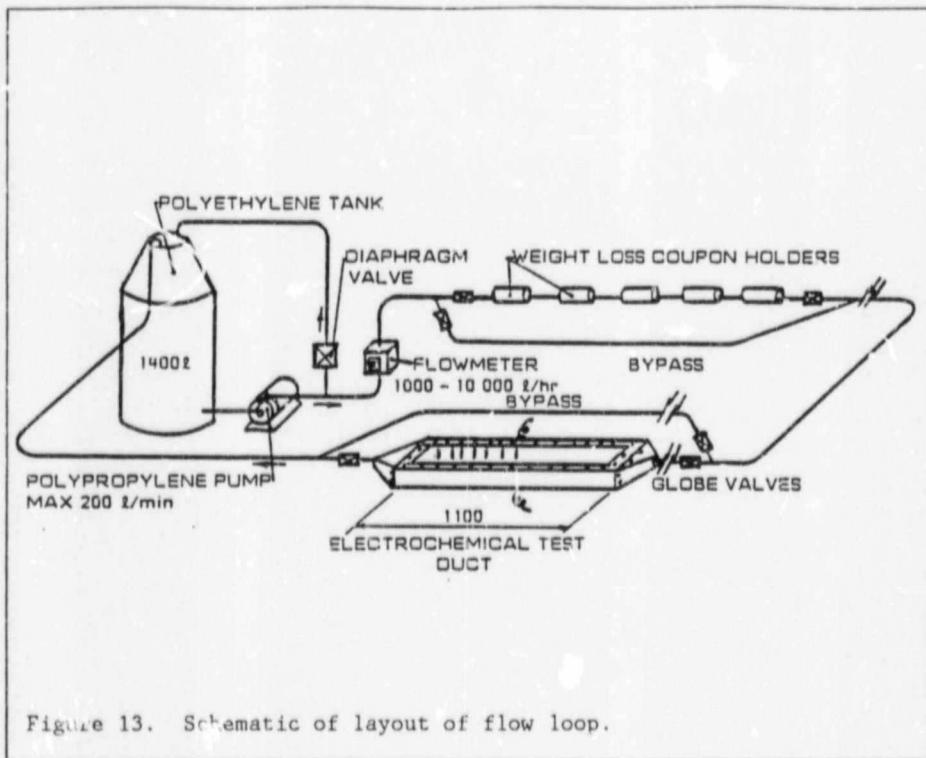
3.3.6 HEATING

-Heating is provided by a 3000W, 220 VAC, Vitreosil glass immersion element.

-Temperature control is provided by a temperature controller with a PT100 thermocouple. The controller operates a relay that switches the heating element on and off.

3.3.7 LAYOUT OF THE FLOW LOOP

The general layout is shown in Figure 13. The pump draws water from the tank, pumps it through the flowmeter and then through Test Section 1 (Immersion Test Section) or bypass No.1. The water then flows through Test Section 2 (Electrochemical Test Section) or bypass No.2 and then it returns to the tank.



-The piping in the horizontal plane is tilted $0,5^\circ$ to the horizontal for drainage. A drain plug is fitted at the lowest point in the return loop.

-The system bypass draws from a Y - piece just above the pump, through the diaphragm valve and back into the tank.

3.3.8 TANK

-One 1400l polyethylene tank.

3.3.9 ELECTRICAL CIRCUITS

- All supplies 220 VAC with controls mounted in PVC box on bench top.
- Pump. 10A overload trip and Telemecanique on/off switch with integral contactor. Cable 22A Norse.
- Heating element. 15A overload trip, connected to temperature controller, 30A relay and then to element.

3.3.10 CONSTRUCTION

- All permanent pipe joints made using Mircoweld™ PVC solvent cement according to manufacturers instructions.
- Specimen holders for test Section 1 made from 186mm lengths of 32mm pipe with couplings solvent welded on ends.
- Test Section 2 machined from perspex and uPVC and assembled using stainless steel M5 threaded rod and nuts.
- All piping supported at a maximum of 1000mm intervals using pipe support clamps on varnished pine blocks, sized to give a slope to the system for drainage.
- Tap off from tank made using a 1,25" tank connector.
- Connection to pump with I.D.40mm braided polyethylene hose clamped with jubilee clamps.
- Pump and motor mounted on welded steel angle base bolted to floor, with 3mm rubber gasket (between base and floor) to damp vibrations.

- Outlet of pump connected to PVC piping with polyethylene pipe and jubilee clamps as for inlet.
- Flowmeter bolted to bench using aluminium mounting plate.
- Stub flanges with backing plates and flat rubber gaskets connect/seal flowmeter to PVC pipes.
- Test Section 1 connected with screw couplings and suspended from 20mm square aluminium tube with pipe clips.
- Connection to Test Section 2 made with 40mm stub flanges, backing plates and flat rubber gaskets.
- Drip trays under Test Section 1 and 2 fabricated from 120mm wide PVC guttering.

3.3.11 OVERALL DIMENSIONS

- Maximum length - 4300mm.
- Maximum width - 1160mm between pipe centres, 2440mm including tank.
- Maximum height above floor level 2000mm.
- Average height above floor level 1060mm.

- Outlet of pump connected to PVC piping with polyethylene pipe and jubilee clamps as for inlet.
- Flowmeter bolted to bench using aluminium mounting plate.
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3.3.11 OVERALL DIMENSIONS

- Maximum length - 4300mm.
- Maximum width - 1160mm between pipe centres, 2440mm including tank.
- Maximum height above floor level 2000mm.
- Average height above floor level 1060mm.

3.3.12 PERFORMANCE

-Flow range 0,5 to 3,25 $\pm 0,03$ m/s (1455-9456 l/hr).

-Temperature range 36 - 60 $\pm 0,5^{\circ}\text{C}$, without a refrigeration system.

3.4 SOLUTION SPECIFICATION - CONTROLLED TEMPERATURE BATH

3.4.1 TANK

-0,5mm stainless steel tank with inside dimensions 885 x 510 x 510 mm. i.e. volume of 230 l.

-Drain plug at bottom.

-Tank mounted on 40 x 40mm equal angle stand.

-Two 25 l tanks polyethylene for corrodent 265 x 265 x 440 mm.

3.4.2 HEATING

-3000W, 220 VAC, 2000mm, long, heating element, laid out in base of tank.

-Controlled with PT 102 thermocouple, and Brainchild BTC102 temperature controller switching a 30A contactor.

-Supply through 20A overload trip switch.

3.4.3 SPECIMENS

-Rectangular 200 x 20 x 1,2mm with $\varnothing 5$ hole 10mm from end.

3.4.4 SPECIMEN MOUNTING

-Suspended from glass hooks hanging from glass cross bars across plastic tanks.

3.4.5 INSULATION

-50mm thick expanded polystyrene foam bonded to tank sides.

3.5 CONCEPT FORMATION

3.5.1 OVERALL DESIGN

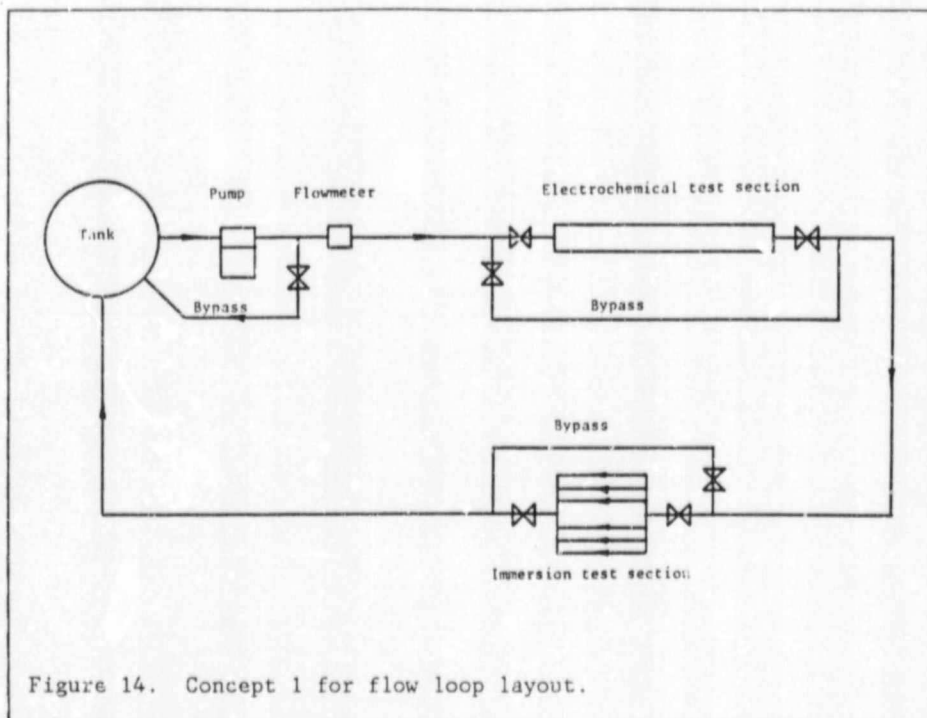
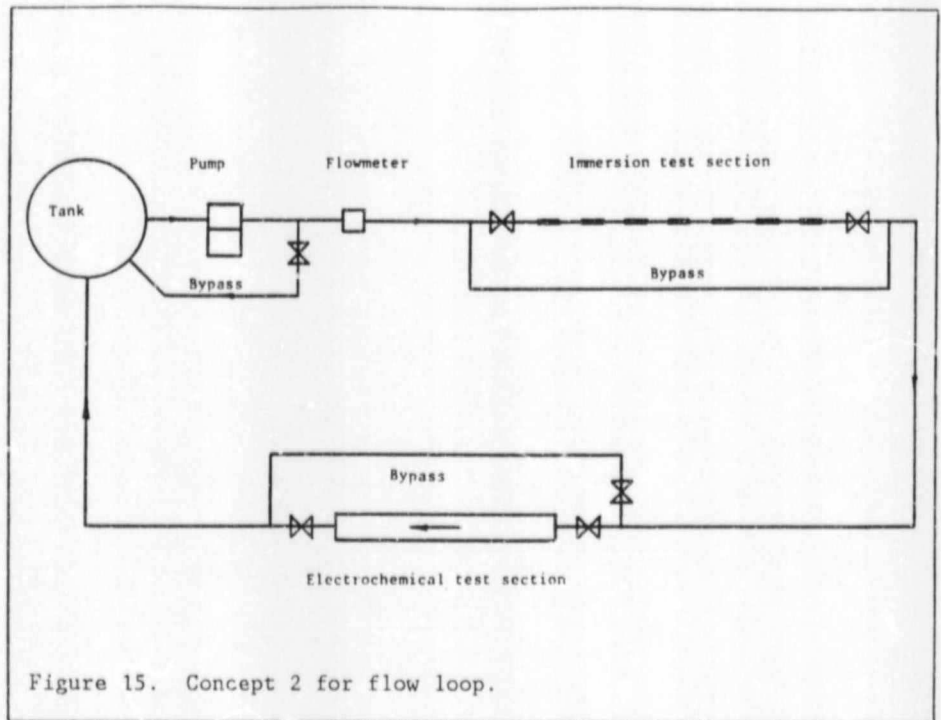


Figure 14. Concept 1 for flow loop layout.

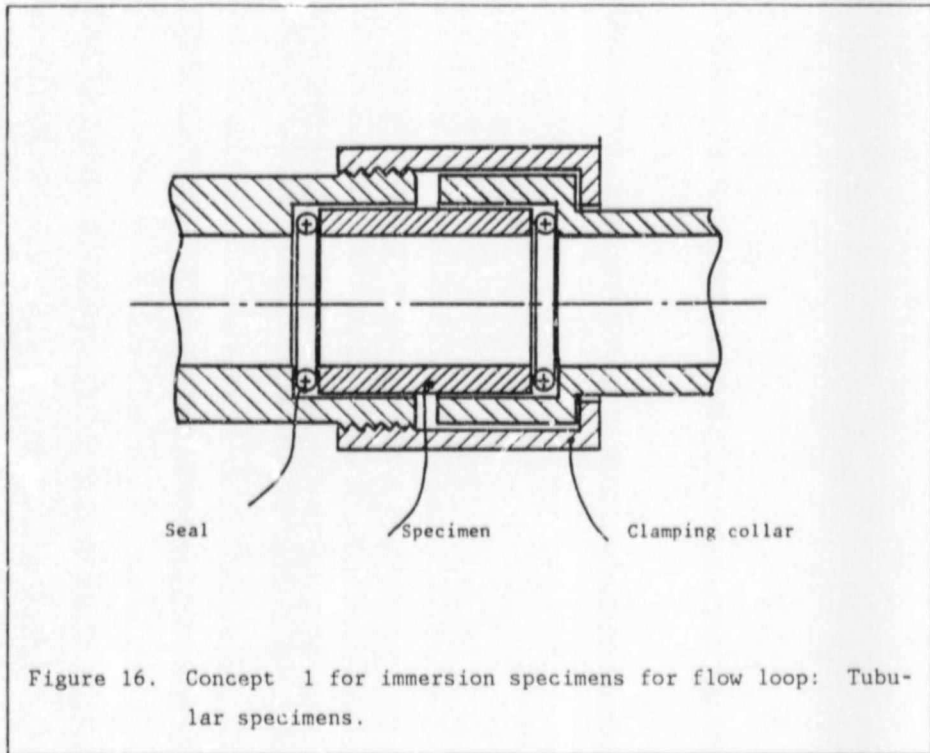


Concept 2 was chosen as all immersion test coupons will get exactly the same flow rate passing over them.

3.5.2 TANK

Selected as this volume would give low contamination levels, the material was relatively inert (polyethylene) and a tank manufacturer was prepared to donate it.

3.5.3 IMMERSION TEST SECTION



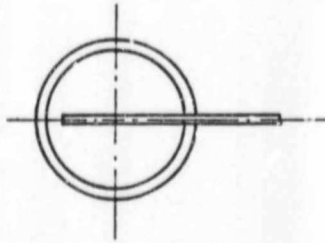


Figure 17. Concept 2 for immersion specimens for flow loop: Flat specimens inserted through side of pipe.

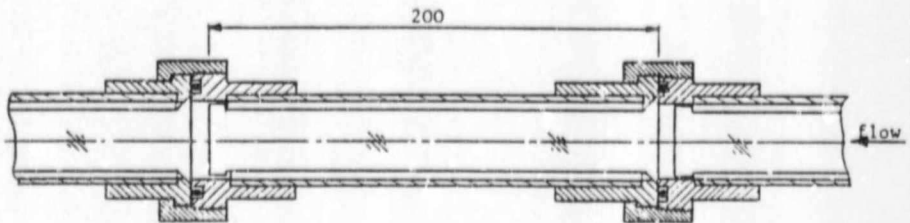


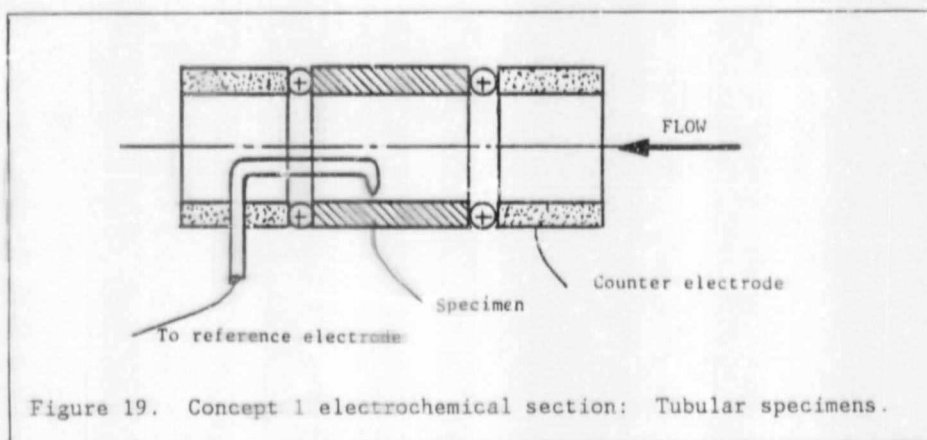
Figure 18. Concept 3 for immersion specimens for flow loop: Flat specimens fixed within pipe.

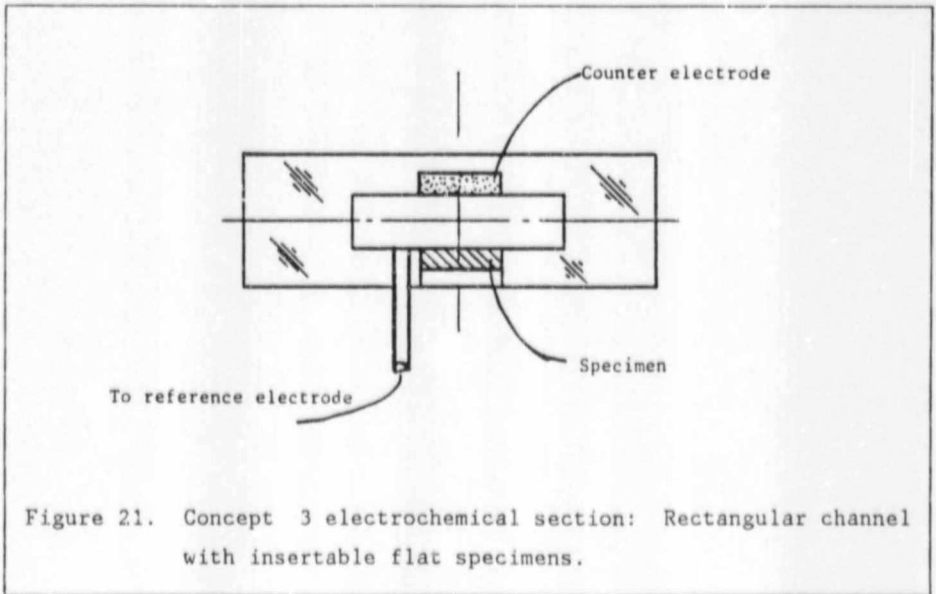
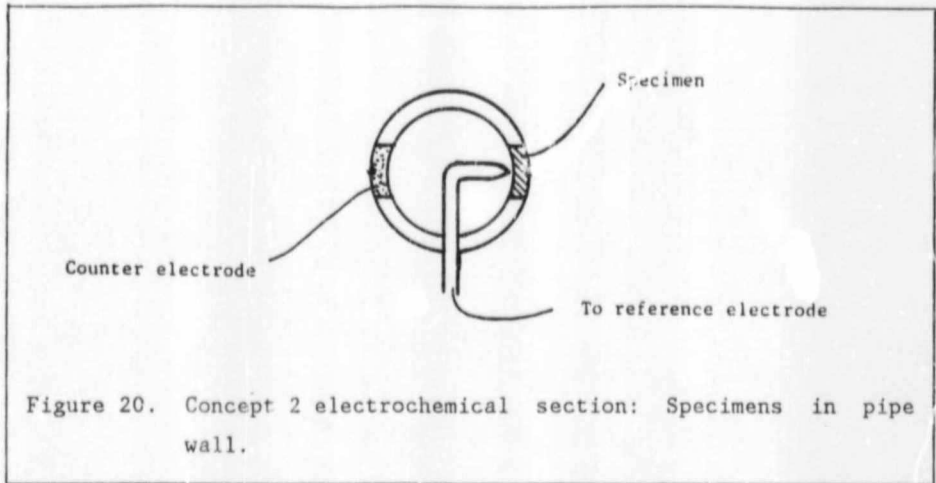
Concept 3 was chosen since not all alloys/materials are available in tubular form and sealing was simpler than concept 2. A specimen length of

200mm was chosen so that tensile test specimens may be machined from corroded specimens.

3.5.4 ELECTROCHEMICAL TEST SECTION

The most important criterion here was to define a system where flow was equivalent to that in a pipe of circular cross section.





Concept 3 was chosen as it allows the use of small flat specimens rather than tubular specimens. The dimensions of the channel were chosen to be 60 x 13,47mm. This gives similar flow conditions over the specimen as for an ID 29mm pipe.

The final specimen design places the reference electrode connection in the specimen mount to bring it as near to the specimen as possible.

3.5.5 PIPING

Unplasticised PVC, is used to manufacture a comprehensive range of pipes and pipe fittings. Joints may be either screw couplings, press fit or solvent welded. Maximum temperature of use is 60°C. The range of sizes and fittings available and the cheap simple joining provided by the solvent welding method made uPVC the choice for the flow loop. It is also the least expensive of all the choices when the solvent joining method is used.

3.5.6 PUMP SELECTION

The pump was sized by calculating the head loss through the flow loop and determining the range of required flow rates at these head losses.

3.5.7 FLOW CONTROL AND METERING

Three methods of controlling the flow rate through the flow loop were considered:-

- a) Using a variable speed drive to the pump.
- b) Throttling the flow through the loop to increase head losses.
- c) Providing a bypass of the flow loop so only a controlled quantity of water would pass through the loop.

A combination of ideas (b) and (c) were chosen as this would give the full range of required flow rates, within the pump's capabilities at a lower cost than if idea (a) was used.

Various types of flow metering devices were considered such as:-

- positive displacement meters
- orifice plate meters
- rotameters

The rotameter is a simple, robust and easy to read meter. It has the advantage that it can easily be recalibrated for different fluids.

It was decided that for the time periods the meter would be in use, type 316 stainless steel would be both capable of resisting corrosion effects and not contaminating the water system. So a meter using this material was chosen as it was cheaper than other versions utilising PTFE components, while still performing the required functions.

3.5.8 HEATING

3.5.8.1 Flow loop

Various methods for heating the water in the flow loop were considered. Electrical resistance heating was considered the simplest and easiest to control accurately. Most electrical heating elements are metal sheathed and this could contaminate the solution, especially if one of the copper alloy type sheaths was used. A glass sheathed immersion element was chosen as being non - contaminating. A power output of 3kW was selected. Control of the element was chosen to be using a thermocouple, with a controller unit, which activates a contactor to switch the current to the element. It is important to note that although the heating system is capable of bringing the water to boiling point, the maximum temperature for the system must not exceed 60°C .

3.5.8.2 Water bath

As the element would only be in contact with tap water, not the corrosive test water, a standard Incoloy sheathed electrical resistance heating element was chosen. This was sized at 3kW and the same type of thermocouple, controller and contractor as for the flow loop were chosen. To spread the energy input more evenly to the water, the element was bent into a waveform.

3.5.9 COOLING

To run corrosion tests below ambient temperature and to remove the heat input from friction losses in the pump and pipes, a cooling system is necessary. Calculations were performed to show the heating effect of the pump and these gave the approximate equilibrium temperature of the flow loop, without cooling, to be 35°C. So for a controllable temperature, tests would have to be conducted at temperatures a few degrees higher than this i.e. around 40°C.

Rough calculations to size a cooling system gave a cooling power requirement of 3kW.

Possible methods for cooling the water include:-

- An immersion cooling coil placed in the tank, connected to a refrigeration unit.
- A heat exchanger with one side being the test water and the other being a liquid cooled by a refrigeration system.

The first method appears to be the simpler and cheaper as it eliminates the need for the extra heat exchanger.

Due to cost constraints no cooling system was added to the flow loop at this stage.

3.5.10 SUPPORTS FOR PIPING AND TEST SECTIONS

Pipe support clamps for uPVC piping are commercially available and these were chosen as being simple and practical. For 40mm Class piping, supports

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are required every 1000mm at a temperature of 50°C. To ensure drainage of the system, it was decided to place all piping at an angle to the horizontal. For ease of access, at least 90mm was required between the bench top and the bottom of the test section two. This gave a maximum height of 120mm above the bench for piping. The simplest method of supporting the system was chosen, and this was wooden blocks screwed to the bench with pipe clamps screwed to the blocks.

Test sections 1 and 2 were supported with aluminium brackets fabricated from 25mm equal angle aluminium sections.

3.5.11 DRIP TRAYS

As leaks would occur at the test sections while changing specimens, drip trays were regarded as necessary. The simplest solution found was to fabricate drip trays to go under test sections 1 and 2 from PVC guttering with polyethylene hose draining the water to the laboratory drains.

4.0 APPARATUS SPECIFICATIONS

4.1 FLOW LOOP

Used for electrochemical and total immersion tests with provision for 7 electrochemical test specimens and 10 immersion specimens. It has the following capabilities:

Flow rate: 1000 - 10 000 ± 100 lhr⁻¹

Flow velocity: 0,50 - 3,25 $\pm 0,05$ ms⁻¹

Temperature: 36 -60 $\pm 0,5^{\circ}\text{C}$ (without cooling)

Water volume: Max. 1400 l

Recommended 500l (for floor loading)

Pump power: 0,55kW (220V AC)

Heating element power: 3,0kW (220V AC)

Figures 22 and 23 illustrate the general layout of the flow loop. Figure 24 shows part of the electrochemical test section with all the connections.

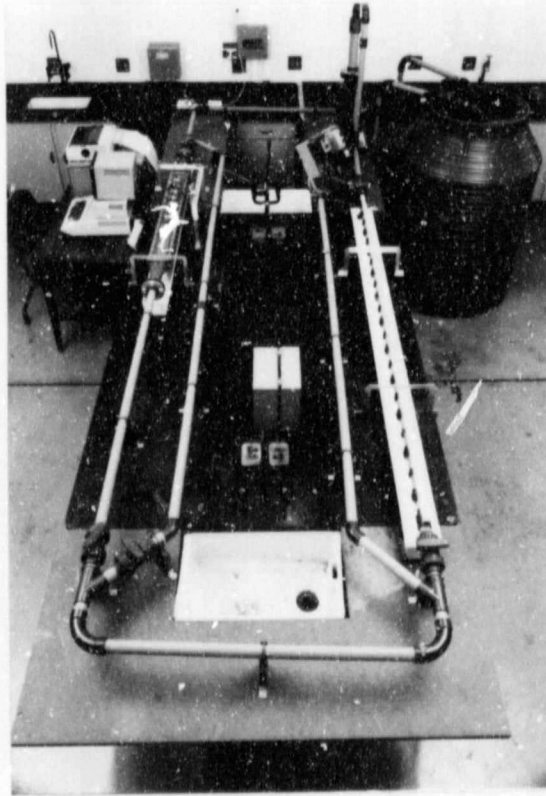


Figure 22. General view of flow loop.

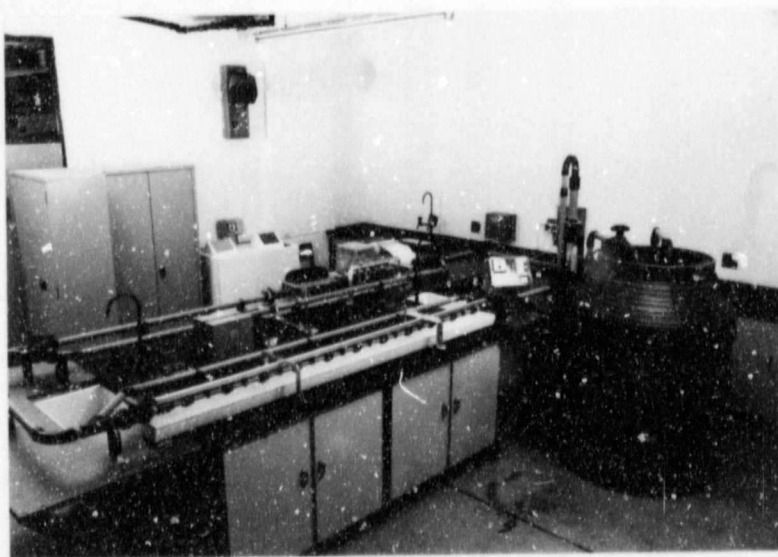


Figure 23. General view of flow loop.

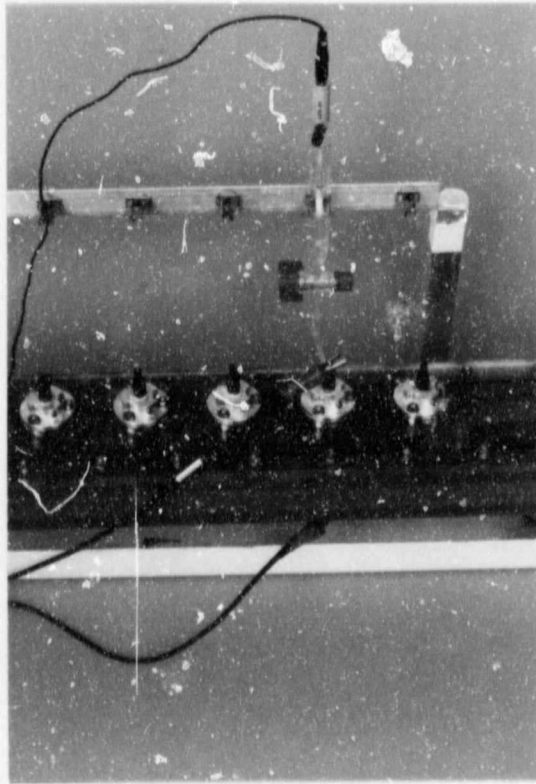


Figure 24. A portion of the electrochemical test section.

4.2 CONTROLLED TEMPERATURE BATH - IMMERSION TESTING

Shown in Figure 25.

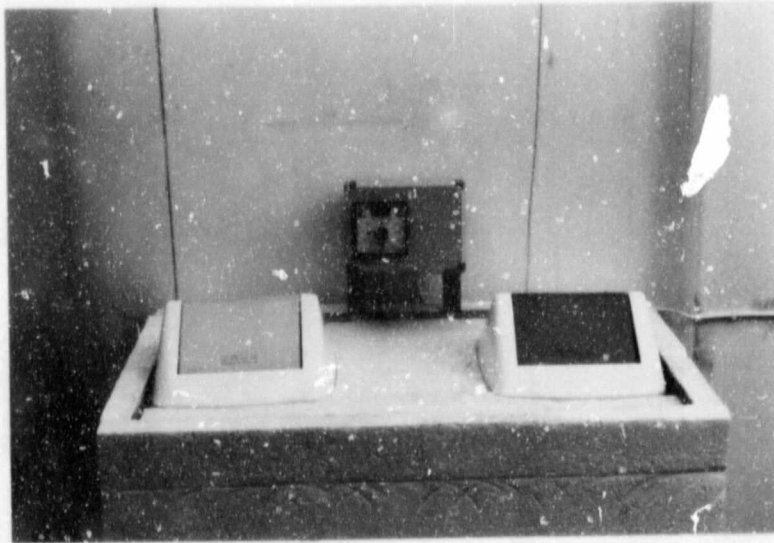


Figure 25. Control temperature water bath.

- Dimensions: height - 510mm, length - 885mm, width - 510mm.
- Contains two 25ℓ immersion tanks.
- May contain up to 48 specimens per tank viz. a total of 96 specimens.
- Heating element power: 3,0kW (220V AC).
- Temperature range: ambient to $96 \pm 0,5^{\circ}\text{C}$.

4.3 CONTROLLED TEMPERATURE BATH - ELECTROCHEMICAL TESTING

Shown in Figure 27. This consists of an insulated stainless steel bath with:

Dimensions: 550 x 405 x 150mm.

Volume: 33ℓ.

Heating: 2,35kW Grant Instruments type 1605, thermostatically controlled element with an electrically driven stirrer.

Temperature range: Ambient to $96 \pm 0,5^{\circ}\text{C}$ with heater
: $4 \pm 0,5^{\circ}\text{C}$ to ambient with additions of ice to the water in the bath.

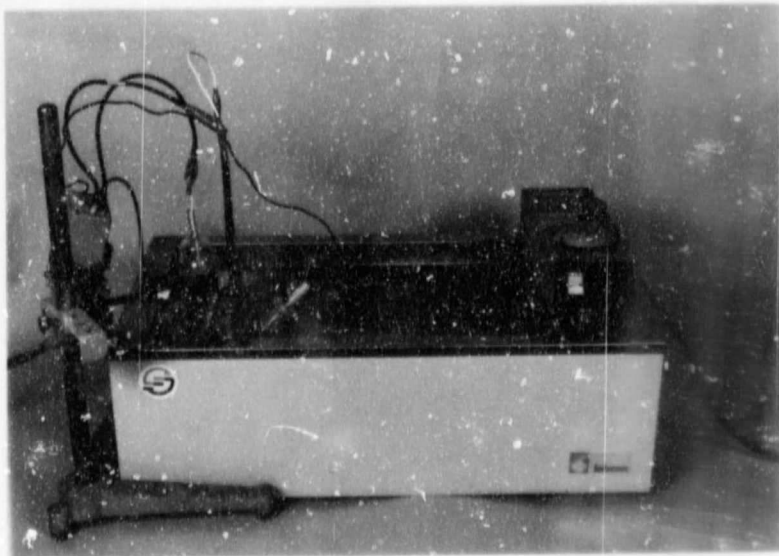


Figure 26. Controlled temperature bath for electrochemical testing.

4.4 CORROSION CELL

This consists of a glass flask of the ASTM G5 type with two graphite counter electrodes and a Standard Calomel Reference Electrode (S.C.E.). Compressed air for aeration or argon for de-aeration is bubbled into the liquid in the flask through 5mm fritted glass tube. The complete cell as set up for testing is shown in Figure 27. Figure 28 shows the specimens for the flow loop and static immersion testing, mounted in the resin blocks.

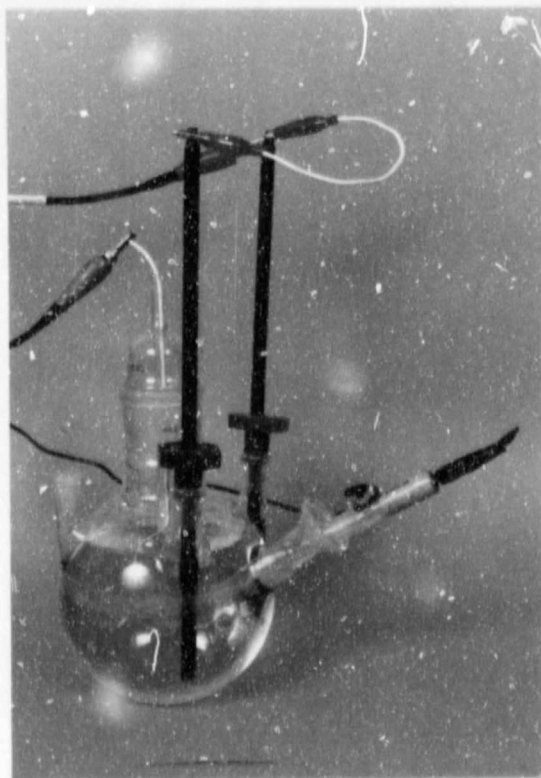


Figure 27. Corrosion cell as set up for testing.

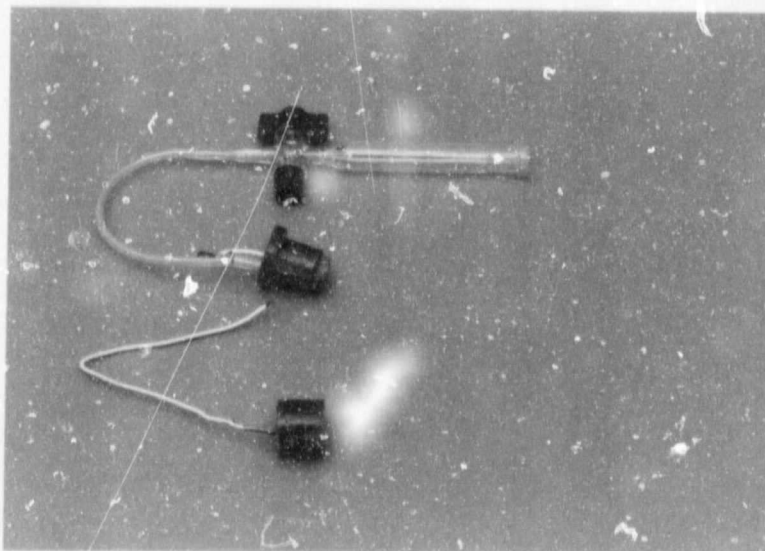


Figure 28. Specimens for flow and static tests mounted in resin.

4.5 POTENTIOSTAT

The potentiostat, computer and printer are shown in Figure 29. This is a Princeton Applied Research (PARC™) Potentiostat/Galvanostat Model 273. Its basic specifications are:-

- 100V compliance with 1A output capability.
- Slew rate 10V/μs.
- Voltage temperature stability <50μV/°C.
- Input impedance $10^{10}\Omega$.

-Input bias current $<20\text{pA}$ at 20°C .

-Maximum input voltage $\pm 10\text{V}$.

-Current accuracy: 0,2% of range for $10\mu\text{A}$ - 1A .

: 0,5% of range for 100nA - 1μ .

The potentiostat may be set up from the front panel or may be controlled by proprietary software from a microcomputer.

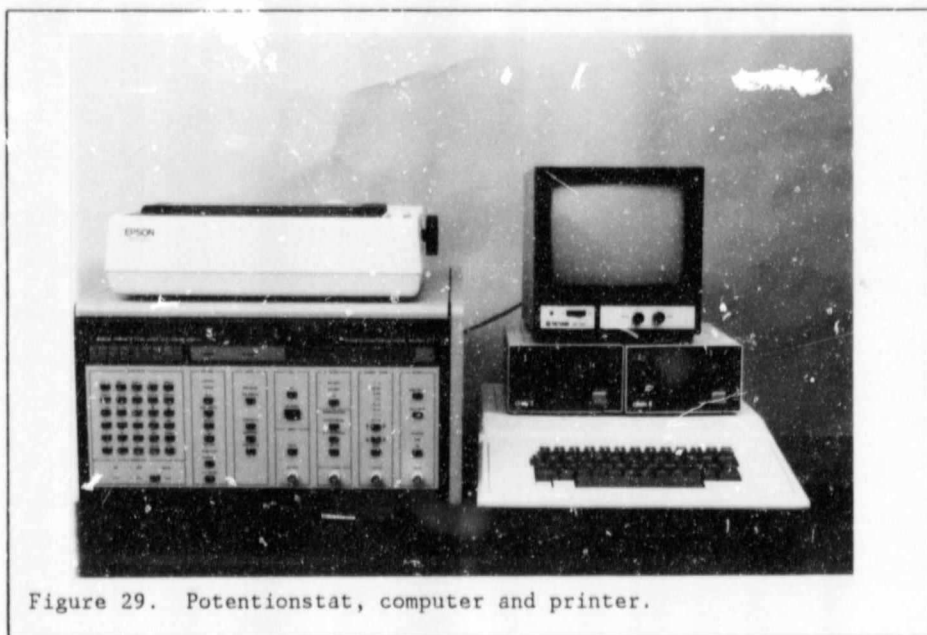


Figure 29. Potentionstat, computer and printer.

4.6 COMPUTER

This was used for running software to control electrochemical tests, processing test data and plotting the results.

It was an Apple II™ fitted with an IEEE 488 card to interface with the potentiostat.

4.7 PRINTER

An Epson RX-80 dot matrix printer was used to print the results and the curves produced during the tests.

4.8 SOFTWARE

This is the Princeton Applied Research Model 273 corrosion software, enabling various types of corrosion scans to be performed. Calculations of corrosion rates may be done by either a Tafel Slope method or a polarisation resistance method.

4.9 REFERENCE ELECTRODES

A standard Calomel Electrode (S.C.E.) was used both for the static and the flow condition electrochemical tests. The model used was a Metrohm 1077.

4.10 TENSILE TEST MACHINE

A Clockhouse Instrument Company model A12 machine was used for the tensile tests. It was fitted with taper grip jaws and has a maximum capacity of 1kN.

4.11 CHART RECORDER

This was used to plot the load/time curves during the tensile tests. It was a Hewlett-Packard model 862/A.

4.12 SCANNING ELECTRON MICROSCOPE

A Jeol GSM-840 scanning electron microscope was used in examining the specimens and corrosion products. This SEM was fitted with a Link™ EDAX system for element analysis. It is capable of analysing for elements from sodium onwards in the periodic table.

5.0 EXPERIMENTAL PROCEDURE

5.1 OVERALL APPROACH

This was to use standardised test procedures to ensure repeatability of results. Where applicable, ASTM standards were applied to both specimen preparation and the testing procedures. Both total immersion and electrochemical corrosion testing techniques were used under static and flow conditions. This was to provide a basis for comparison between mass loss coupon and electrochemical testing, and to evaluate the effects of flow conditions on corrosion rates. This latter point is especially important as piping systems may operate under different conditions, such as flow during normal service followed by static during a shutdown.

5.2 TEST MATERIALS

The range of test materials included seven aluminium alloys as well as three ferrous materials for a comparison purposes.

For identification purposes each alloy was assigned a one digit number. These were as follows:-

Alclad 3004 - 1, alloy 1200 - 2, alloy 3004 - 3, alloy 5251 - 4, alloy 6063TB - 5, alloy 6063TF - 6, alloy 7017 - 7.

The aluminium alloys and their nominal compositions were :-

Table 5.1 Aluminium Alloy Compositions (%).

ALLOY	CLAD	1200	3004	5251	6063	6261	7017
ELEMENT							
Cu	0,03	0,05	0,25	0,15	0,10	0,15- 0,40	0,20
Mg	-	-	0,8- 1,3	1,7- 2,4	0,45- 0,09	0,70- 1,0	2,0- 3,0
Si	0,12	Fe+Si =1,0	0,30	0,4	0,20- 0,60	0,40- 0,70	0,35
Fe	0,20	Fe+Si =1,0	0,70	0,50	0,35	0,40	0,45
Mn	-	0,05	1,0- 1,5	0,1- 0,5	0,1	0,2- 0,35	0,05- 0,5
Zn	1,3- 1,7	0,10	0,25	0,15	0,10	0,20	4,0- 5,2
Ti	-	0,05	-	0,15	0,10	0,10	0,1
OTHERS							
EACH	0,05	0,50	0,05	0,05	0,05	0,05	0,05
TOTAL	0,15	0,15	0,15	0,15	0,15	0,10	0,15
Al	Rem	99,0 min	Rem	Rem	Rem	Rem	Rem 0,35Cr
DENSITY (g/cc)	2,71	2,71	2,71	2,69	2,71	2,71	2,77

Rem - remainder. Single figures show maximum content.

Alclad was not used in electrochemical tests. Mild steel (M.S.), and alloys 3CR12 and 316L were not used in immersion testing as M.S. and possibly 3CR12 would have contaminated the solutions with Fe^{2+} ions.

5.3 ELECTROCHEMICAL CORROSION TESTING

5.3.1 SPECIMEN PREPARATION

All specimens were prepared from extruded aluminium rod which was machined to final dimensions of 10mm diameter and 10mm long. By turning the specimens from larger diameter rod, any surface inclusions or other surface inhomogeneities which may effect results were removed. A 3mm diameter hole was then drilled into one end of each specimen, 0.6mm² Copper wire was inserted into the hole and a screw put in to retain the wire and ensure electrical connection. The specimens were then cast into catalyst hardened polyester resin holders of the forms shown in Figure 28.

For the specimens in the flow loop, a 4mm diameter perspex tube was placed against and parallel to the specimen in the mould. In this way the reference electrode was as close as possible to the corroding surface

The specimens were then prepared for testing according to ASTM Standards G59 and G61 (46). The procedure was:-

- Wet grind surface with 240 grit Si-C paper.
- Wet polish surface with 600 grit Si-C paper.

- Rinse with tap water.
- Rinse with distilled water.
- De-grease with acetone.
- Mask alloy/resin interface with a masking compound to prevent crevice effects.
- Immerse specimen within 1 hour of preparation.

5.3.2 POTENTIODYNAMIC SCANS

Initially a one hour immersion time was allowed before commencement of a scan. This was found to be satisfactory for all the alloys, aluminium and iron based, under static conditions but not for the aluminium alloys under flow conditions. Under these latter conditions the free corrosion potential was monitored every 10 minutes and once it stopped changing by more than 0,2mV/min the scan was begun.

The scans were commenced at -200mV vs E_{corr} and the potential was increased at a rate of 1mV/s up to the final potential of 1200mV vs E_{corr} . Some problems were encountered due to electrical noise with scans conducted in the flow loop. When this occurred, the initial and final values of the scan were referred to the reference electrode rather than to E_{corr} . Effectively this gave the same potential range of -200mV vs E_{corr} to 1200mV vs E_{corr} , but the potentiostat appeared less sensitive to extraneous influences.

The scan results were then plotted and the software used to calculate corrosion rates by both the "Parcalc Tafel Analysis" programme and "Linear Polarisation" technique. All scans were duplicated as a check for repeatability of the tests, and where this was not found to be the case,

further tests were conducted. Potentiodynamic scans were conducted under the following conditions:-

- Detailed tests, under flow conditions at 40°C and $1,75\text{ms}^{-1}$.
- Limited tests, under flow conditions at 40°C and $1,0\text{ms}^{-1}$, $2,50\text{ms}^{-1}$ and $3,25\text{ms}^{-1}$.
- Static, aerated at 40°C.
- Static, de-aerated at 40°C.
- Static, aerated at temperatures between 5°C and 78°C.

These tests were conducted to both obtain data on the behaviour of the individual alloys under flow and static conditions at 40°C, and then to provide an indication of the effects of flow velocity and temperature on corrosion rates.

5.3.3 CYCLIC POLARISATION SCANS

The details concerning specimens and stabilisation times are the same as for the potentiodynamic scans.

The potential was increased at 1mV/s from 1mV vs E to some point and then decreased at the same rate back to 1mV vs E . The potential at which the scan was reversed was determined from the shape of the potentiodynamic scan curve. Typically the scans were reversed at a current density of $100\mu\text{A/cm}^2$.

Author Buchan Andrew John

Name of thesis Corrosion Of Aluminium Alloys In Static And Recirculating Mine Waters. 1988

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