ELECTROCHEMICAL CORROSION TESTING IN A HOT CELL ENVIRONMENT

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EXECUTIVE SUMMARY

An electrochemical corrosion testing rig was set up in a hot cell in order to investigate the corrosion properties of irradiated steel reactor materials. The objectives were to test pre-fabricated, inactive IGA (inter-granular attack) sensors alongside irradiated stainless steel, and also to observe the effect of electrolytes on the steel. Solution chemistry variants were chosen to demonstrate inhibition or initiation of corrosion in the materials during testing. To perform these tests, a series of experimental rigs were designed to allow fabrication of ~375mm^2 samples of a reproducible size and quality. A spot welding rig was developed for assembly of the active electrodes. The electrochemical test tanks were designed to run at temperatures up to 60°C, for prolonged cycles of 60 days, over a 2 year period. Tanks were coupled with a system capable of measuring electrical signals from inside the hot cell. Electrodes that were 'friendly' to remote handling and the hot working environment were also necessary. The considerations and implications for the test and the design materials are considered and discussed.

1. Introduction

The post irradiation examination (PIE) facilities at the Windscale Laboratory, Sellafield, have been developed over 40 years of nuclear operations, with capabilities ranging from examination of full fuel elements to microscopic examination of surface coatings.

A recent requirement for hot cell electrochemical corrosion testing led to the National Nuclear Laboratory being contracted by Sellafield Limited to undertake a work package that has been funded by the Nuclear Decommissioning Authority. This work is being carried out in support of safety case development for the long term wet storage of AGR fuel. The work included the in-house design of electrochemical corrosion testing equipment optimised for remote operation

The following paper presents the experience gained from the design and commissioning of a small-scale, hot cell rig for electrochemical corrosion and the subsequent corrosion testing of irradiated material.

2. Test Programme

The test programme was chosen to test irradiated reactor materials for sensitivity to inter-granular corrosion alongside previously developed sensors and to investigate the effect of changing solution chemistry. The solution chemistry was varied to initiate or inhibit corrosion in order to prove the inhibiting agents behaved as expected, and that the sensor was functioning correctly. A series of four tests are planned to run under differing chemical conditions, with a preliminary test, already completed, designed to identify suitably sensitised steels for later tests. Tests are also planned to run at elevated temperatures of 40°C, 50°C and 60°C for 60 day periods. Open circuit corrosion currents will be monitored throughout the test duration with a zero resistance ammeter to confirm correct operation of the equipment and to measure corrosion of the materials.

3. Design

3.1 Design Requirements

The experiment required that reactor irradiated samples of a uniform size and surface area were to be cut from an irradiated component, then assembled into an electrode and placed in a water tank with a pre-determined solution chemistry. In-house design of equipment ensured that it would be amenable to remote operation by master-slave manipulator (MSM). The main items of equipment required for the experiment were cutters, spot welding equipment, corrosion tanks and some specially designed tools including tweezers and “fingers” for the MSMs.
Standard measures such as weighted equipment bases to minimise vibration and movement were used. The drill stand, welder and tanks were all manufactured, assembled and inactively commissioned to ensure operability outside the hot cells. After successful commissioning the items were partly disassembled before posting into the hot cell and reassembly.

3.2 Cutting Equipment

Inactive trials showed that a diamond-tipped coring bit would allow the extraction of suitably-sized samples from the irradiated component. Previous good experience led to the selection of an air-driven tool in preference to an electric drill. The resulting rig (Figure 10) included a camera in order to allow remote and accurate positioning of the material within the vice. The vice was manufactured of nylon to protect the surface of the steel from damage and cross-contamination. The eventual dimensions of the usable sample were 25 x 15 x 2mm. To facilitate handling, a specially designed pair of small “finger” grips was attached to the MSMs in-cell. Rubber pads were attached to the grips using rivets, as adhesive would have degraded over time, and rivets minimised the number of possible replacements.

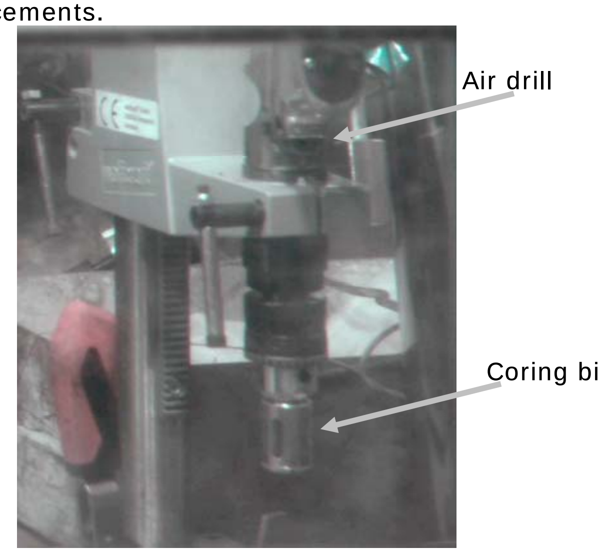

Figure 10: Air-driven pillar drill with coring bit

3.3 Pre-assembly of electrodes

Electrode holders (Figure 11), unirradiated cathode sections and anode holders (Figure 12 and 4) were all manufactured and part-assembled outside the hot cells. The electrode heads had electrical connectors and reference electrodes inserted, and guides were drilled into the holders to give easier access to the cathode and anode connections that would have to be made inside the hot cell. Lifting rods were also fastened to the blocks as there was no room to place MSM grips between the blocks once in place in the tank. The anode holders were made using an electrically insulating polyetheretherketone (PEEK) pad with a hole, through which was inserted the cut end of the electrical connection. This connection was insulated with tubing to isolate it during the tests, and connected to a gold plated fitting. The electrode design was chosen to be robust, to isolate the spot weld from the solution and to present a constant surface area for corrosion. Gold plated pins were also used as the contact points fitted into the upper part of the electrode holder, to make contact with a printed circuit board (PCB) above the tank. This allowed for minimal corrosion and increased pressure on contact, giving better conductivity.

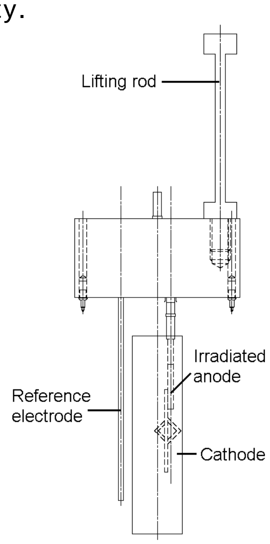

Figure 11: PEEK electrode holder with lifting rod

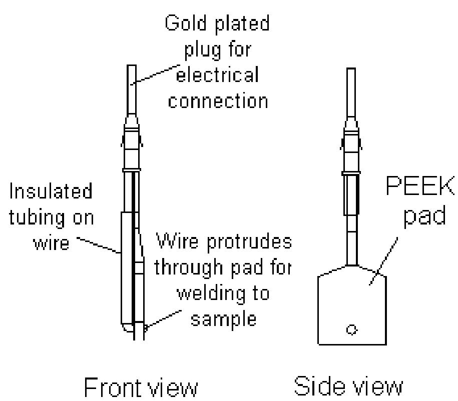

Figure 12: Drawing of pre-assembled anode holder

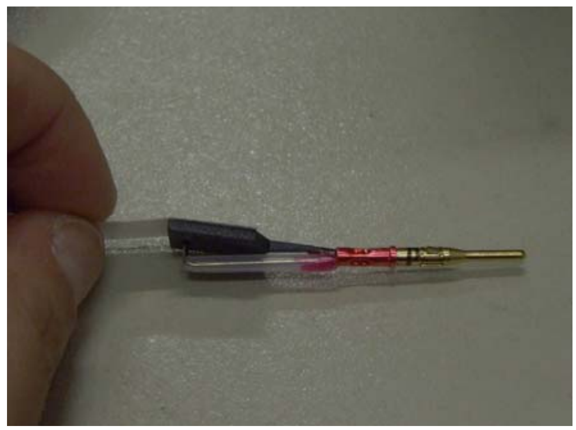

Figure 13: Pre-assembled anode holder

3.4 Spot Welding

Assembly of the cored sample and the pre-assembled anode holder was achieved by spot welding. This required the surface of the sample to be cleaned. A rig was designed to clean a minimal area of the sample with an air driven pencil grinder (Figure 14).

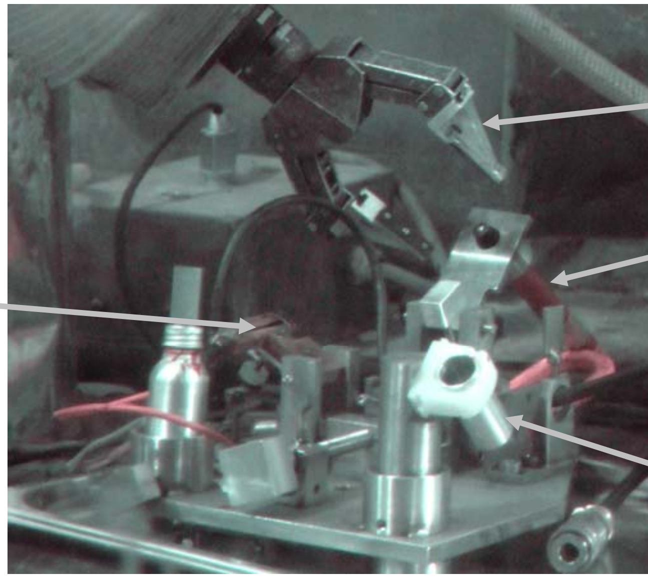

Figure 14: Welding rig with pencil grinder

The grinder was fixed in position about a pivot point, with a replaceable grinding tip. A jig and spanner were made to allow the grinding tip (~20mm length, 4mm diam) to be changed remotely, whilst leaving the grinder in position. After cleaning, the sample was clamped firmly in place, and adapted tweezers used to attach a pre-assembled welding rod and electrical connection to the welder. The sample was welded, using a camera for accurate positioning, and the weld tested for mechanical strength using a gentle twisting motion. Insulation of the weld was ensured by painting "lacomit" varnish onto the weld with a 5mm paintbrush, and allowing capillary action to draw it up the insulation tube of the pre-assembled welding rod, thus electrically sealing it. Finally, the assembled electrode was mounted into the electrode holder

3.5 Corrosion Tanks

The in-cell tanks (Figure 15 to Figure 17) were designed to hold a maximum of 1.5 litres of test solution and were made from stainless steel. The tanks also had a stainless steel frame with adjustable legs (for levelling), and a mica heating pad to provide the required temperatures. A ceramic calcium silicate plate was used to insulate the tank. The entire construction was limited to 10kgs, for easier in-cell handling. The PCB lids were topped with Perspex in order to protect them from degradation and damage (see Figure 17). The PCB connectors were gold coated, to reduce the risk of corrosion, and electrical connections were made via LEMO plugs. The LEMO plugs gave unique identities to each connection, and had coded keyways. The plugs were also insulated with PEEK and either soldered or crimped to the electrical wires that passed the signal back through the wall of the hot cell. In order to have easy MSM access to connect/disconnect the plugs, a space of 30mm was left in between the sockets.

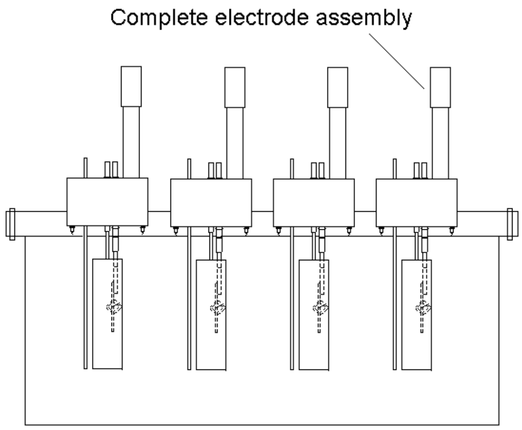

Figure 15: Side view of tanks containing assembled electrodes

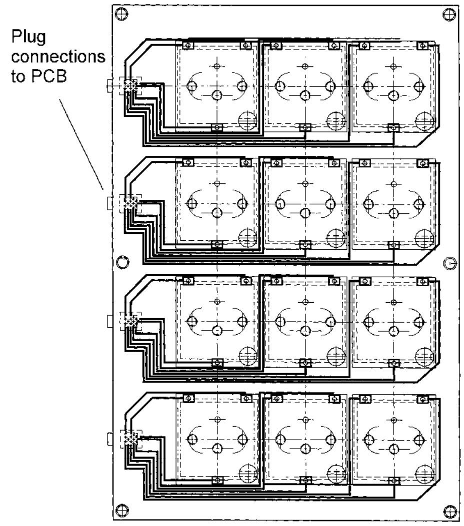

Figure 16: Overview of PCB tank lid and connections

The corrosion test solution was pumped from external tanks into the cell by a peristaltic pump using Norprene tubing feeding into an "L" shaped inlet on the side of the tank to prevent backflow of solution out of the hot cell. The corrosion tanks were operated with a slow but constant feed of solution at ~100ml/hour. The test tanks were designed with a "weir" overflow system which allowed the solution to run into evaporator tanks heated by a ceramic pad. An evaporator system was chosen in order to eliminate the problem of either pumping active liquor out of cell or of storing the spent liquor in-cell during the lengthy test runs. Each heater pad was accompanied by a second pad, in case of failure of the first heater. Appropriate specification of the heating pads allowed the evaporator tanks to run dry without causing failures of the pads, and so could be left on at all times if necessary.

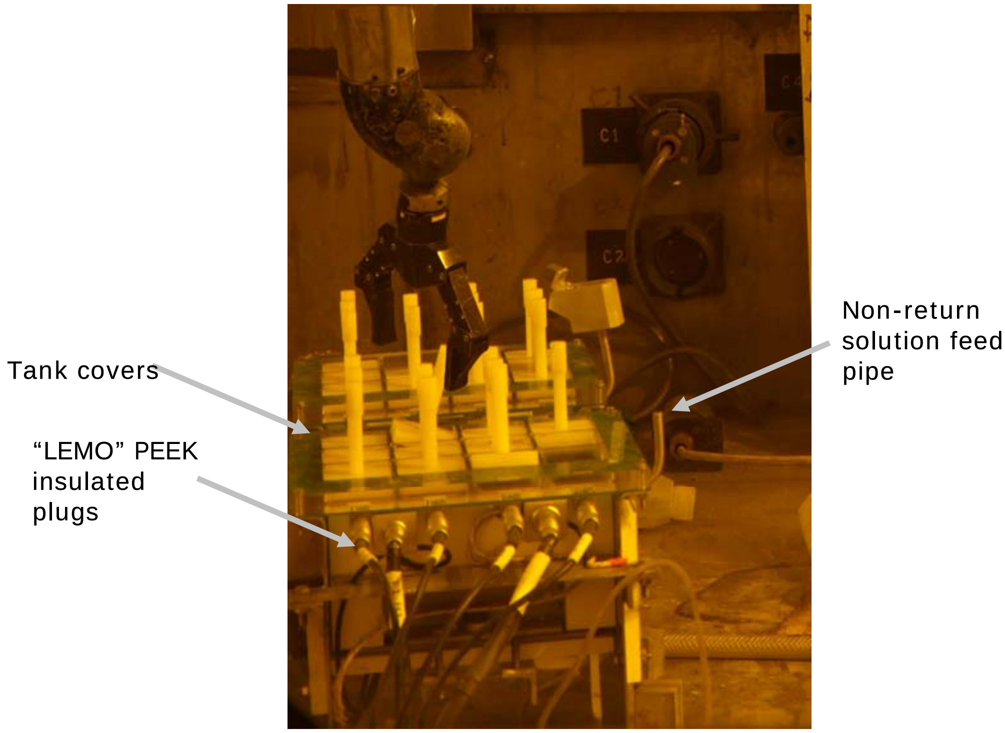

Figure 17: Corrosion tanks in hot cell

Layout of equipment in the hot cell (shown schematically in Figure 18) was arranged to give minimum interference with the constantly running tanks, and to allow easy access between the assembly equipment and the test tanks.

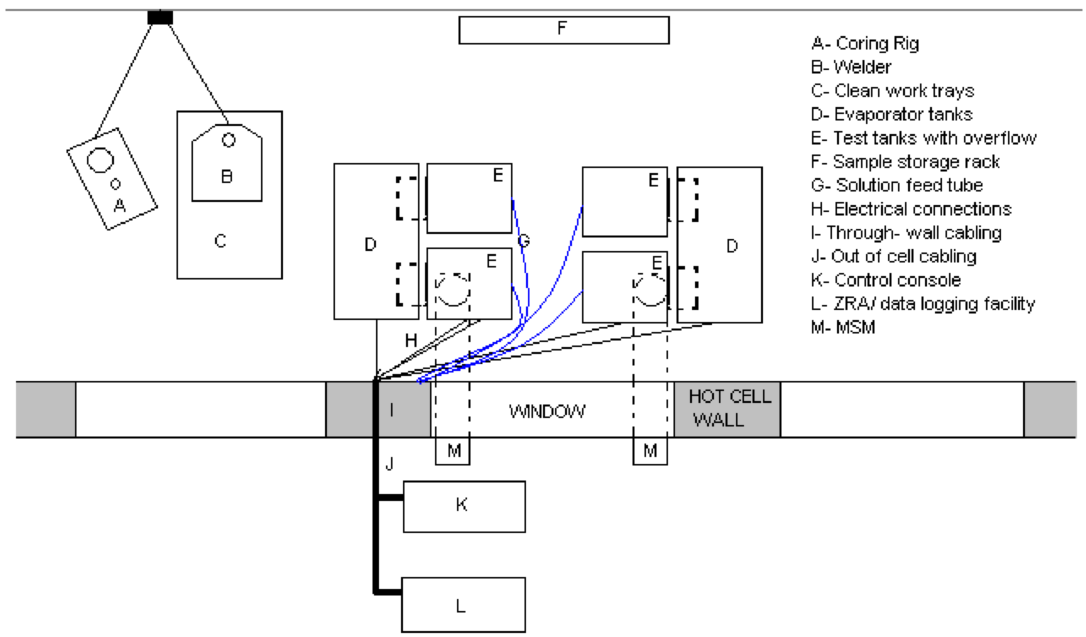

Figure 18: Schematic drawing of in-cell equipment layout

4 Experimental Progress

Experiments at several temperatures and chemical conditions are planned. The first stage of the experiment has been successfully completed. The solution reservoirs outside the hot cell allowed the tanks inside to function, and the overall solution chemistry to be changed, or maintained, on a regular basis. De-ionised water flushing was used successfully to minimise in-cell chemical contamination, and the unused tanks or cells were kept covered to minimise ingress of contaminants.
In-cell temperatures, solution flow rates and solution chemistry were all controlled from a console on the face of the hot cell to allow easy access and management of the experimental parameters.
Corrosion currents occurred and were successfully measured, demonstrating good working practice in getting contact within the welded electrode assemblies. Initiation and inhibition of corrosion was observed during the experiment when the solution chemistries were altered, and the inhibitors behaved as expected.

5 Summary

The successful design, set up and operation of an electrochemical testing rig within a hot cell has been described in this paper. Carefully designed equipment and inactive testing allowed the first stage of the tests to proceed successfully, maximising lifetime in the hot cell, and minimising repair and maintenance issues. The effect of corrosion inhibitors and initiators on the irradiated stainless steel has been observed under elevated temperatures of 40°C.
The status for future planned tests is positive, and the implications for reliable production, assembly and testing of small irradiated components on a large scale are excellent.