The Installation of an IASCC Autoclave Test System at the SCK•CEN Hot Laboratory

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Abstract

Irradiation assisted stress corrosion cracking (IASCC) is becoming a major concern for ageing power reactors worldwide. In order to assess the influence of irradiation damage on the stress corrosion cracking resistance of materials, it is imperative that one has the facility to subject irradiated material to a relevant environment in a well-defined and controlled stress state. In order to simulate the primary reactor environment in an LWR, an autoclave system with recirculation loop is used, able to operate up to 350 °C and 25 MPa. The loop is equipped with a parallel system of water purification and on-line chemistry monitoring system, so the sample is exposed to a constant and controlled environmental condition. For the stress corrosion testing, an active loading system is available, with a pull rod penetrating the autoclave cover. According to the test type, different types of samples (tensile, precracked fracture mechanics specimens such as compact tension or notched round bars) can be subject to various loading schemes (constant load, slow cycling load or slow strain rate tensile loading).

Concepts, which are known from testing non-radioactive materials, have been adapted to fit the practice of hot-cell testing of irradiated samples. The autoclave systems have been adapted to facilitate remote handling and reduce the risk of failure and the consequences thereof. The system has been designed in such way that the activity is confined to well defined parts of the system, so that hands-on manipulation of the loop part of the system is possible, while the autoclaves are placed inside a hot-cell. The cell itself has been refurbished for accepting the autoclave system inside and the loop part in the basement room below. In addition, special manipulation devices and storage capacity for the samples have been provided in the cell.

1. Introduction

For many ageing nuclear power plants, the phenomenon of stress corrosion cracking of reactor core internals is becoming a major concern in the lifetime management optimisation. The processes involved in IASCC, depend on a large number of variables, such as temperature, water chemistry, mechanical stresses and material condition, which includes the level of irradiation-induced microstructural damage. The evaluation of the influence of all these parameters calls for a systematic study of the phenomenon. This study can involve tests on model materials in controlled environment, irradiated materials from commercial or test reactors in simulated environment (pre- and post-irradiation tests) and in-pile tests. As the complexity of the tests increases, the possibility of variation of conditions and the number of tests usually decrease both for technical and economical reasons.

The effect of neutron irradiation on the stress corrosion cracking of materials is twofold, namely the effects of radiation induced material degradation and radiation induced environmental changes. In the first category of phenomena, irradiation hardening and radiation-induced segregation are considered to have the greatest influence on IASCC. In the second category, radiolytic formation of oxidising species is considered to have an influence on the stress corrosion behaviour of stainless steels, due to an upward shift in electrochemical potential. In many nuclear reactors, the effect of radiolysis is (partially) counteracted by the addition of hydrogen to the water, which promotes the recombination of the radiolysis products.

The experimental techniques for assessment of the IASCC susceptibility of irradiated stainless steels are numerous and in many case debatable. In general 3 classes of stress corrosion tests can be discerned:

- ✓ Constant load or constant deflection tests (samples with smooth surface): these tests are a good simulation of the real component's situation, but require a high number of samples and long experiment times. The test technique is very simple and requires only that a sufficient space is available, in which a representative environment can be sustained for a sufficient period of time. In order to ensure constant environmental conditions during the tests, water chemistry and temperature monitoring are required. For accurate determination of the failure time, on-line monitoring or addition of failure recorders is necessary, in order to avoid frequent inspections. The experimental results are qualitative in nature and require statistical analysis. Usually, these tests are used for initiation studies or time to failure determination of component-like samples. For irradiated material, these tests are less suitable, due to the number of specimens needed for a significant analysis. In material test reactors, rigs with a large number of specimens can be irradiated to obtain statistically significant data, but this method is very time consuming for studying phenomena, which occur at high irradiation dose (such as IASCC).

- ✓ Rising load tests on samples with smooth surfaces: the slow strain rate tensile tests (SSRT) is a typical example of this type of testing. These tests are of short duration and yield semi-quantitative results; they are very suitable for comparing different material - environment combinations in order to rank them in order of (IA)SCC susceptibility. Technically, these tests are relatively simple, but they require an active loading mechanism to be present in the desired environment. The test results are usually reported based on the fracture surface morphology. However, this practice may overlook a part of the information in the test and also the mechanical properties of the material, measured during the SSRT test should be reported and compared to the properties in inert environment. Due to the fact that the method of loading is not very well defined and does not correspond to real, in-service, situations, the results of this can hardly be used for extrapolation to estimate the risk on IASCC in real components.

- ✓ Constant or slow cycling load on precracked samples (compact tension, notched 3 point bend specimens...): in these tests, the mechanical loading at the crack tip is well determined by fracture mechanics and in many cases, the crack length evolution is monitored during the test. These tests then yield a quantitative relationship between the mechanical loading and the crack propagation rate by stress corrosion of the material in the environment under consideration. However, the technical complexity of these tests is high and they require a longer testing time than the rising load tests.

In the Reactor Materials Research department of SCK•CEN, expertise on SCC testing in simulated LWR environment has been built during the past five years. Experiments have been carried out on unirradiated specimens in autoclave environment and in-pile in the BR2 materials test reactor. Currently, the testing capabilities include electrochemical measurements (potential monitoring, potentiodynamic measurements, SCC detection by electrochemical noise and acoustic emission and electrochemical impedance spectroscopy), constant load tests for initiation with on-line failure detection for in-pile use (pressure tube specimen type) and mechanical tests in autoclave environment. The active mechanical loading for SCC testing in autoclave environment is capable of temperatures and pressures up to 350°C and 20 MPa. The environment can be stagnant or circulating, with continuous monitoring of pH, conductivity and oxygen, hydrogen and hydrogen peroxide levels in the water. Typical tests that are carried out in these installations are slow strain rate tensile (SSRT) tests and crack growth rate tests on precracked specimens, with on-line crack length recording by potential drop techniques (AC or DC can be used).

2. The hot-cell characteristics

For the installation of the SCC test equipment, a concrete hot-cell was available with dimensions of $6x2.1x3.5m^3$. The cell is equipped with two lead glass windows and two pairs of master-slave manipulators. The cell has a stainless steel liner, formerly intended as alpha tight box. As there was a need for many additional penetrations and no alpha contaminated specimens are expected, the alpha tight qualification was not sought for the refurbished cell.

The working table in the hot cell is designed around the autoclaves, so there is no risk of dropping of tools or samples on the ground, where they would be difficult to recuperate. Behind the autoclave set up, there is a free space for maintenance of the electrical connection boxes in case of interventions.

The cell is equipped with an internal crane and a storage facility for radioactive samples in the bottom of the cell. The storage facility consists of two independent columns with 6 levels of storage cans (6 on each level). The columns can be automatically lifted from the basement into the hot-cell by a motor-driven spindle. When closed, the storage facility is lead shielded in order to minimise the background radiation in the hot-cell; this lead shielding can be opened and closed automatically. For transfer of samples and tools, there is a transfer door for connection of standard containers of the La Calhène type.

3. The SCC test installation inside the hot-cell

Recently, a set of three autoclaves was acquired and installed inside a hot-cell at the SCK•CEN hot laboratory, for testing of irradiated samples. Two of the autoclaves are constructed out of stainless steel 316L, the third one of Hastelloy C. The stainless steel autoclaves are used connected to a loop with simulated LWR primary water, while the Hastelloy autoclave can be used connected to the same loop, or in stagnant conditions, with more aggressive environments.

The autoclaves are equipped with a servo-electric tensile loading device, penetrating the autoclave lids (see figure 1). The loading devices are operated in SSRT mode, constant load/displacement mode or cyclic loading. Each autoclave can be equipped with an internal thermocouple, reference electrode and sample instrumentation wires, so electrochemical measurements and potential drop crack length monitoring are possible. The opening and closing of the autoclaves is fully automatic, with fixed pneumatic wrenches on each closing bolt. The number of closing bolts has been reduced to 4 instead of 8. Replacement of samples is facilitated by using special grips, adapted for manipulator use (figure 2).

The autoclave characteristics are the following:

- Volume: 2 litres each.
- Maximum Pressure and Temperature: 20 MPa & 350 °C.
- Maximum load: 20 kN.
- Displacement rate range: 2.10^{-7}mm/sec to 4.10^{-1} mm/s.

The circulation loop is constructed in the basement below the hot-cell. A flow sheet of the loop and the ancillary systems is represented in figure 3. The loop consists of a high-pressure and low-pressure part. The high-pressure part is penetrating the hot-cell shielding with two thermally insulated water pipes through a lead block. The circulation can pass through the autoclaves in a serial or parallel mode. The high-pressure pump provides a maximum circulation rate of the water at high pressure is 50 litres per hour. The high-pressure part further contains a heat exchanger, electrical preheater, cooler and pressure regulator.

In the low-pressure part of the loop, the water is preconditioned, monitored and cleaned during the

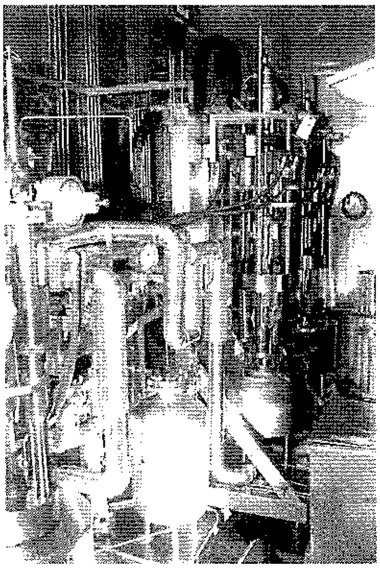

Figure 1 View of the autoclaves inside the hot cell, with the blow down tank in the middle.

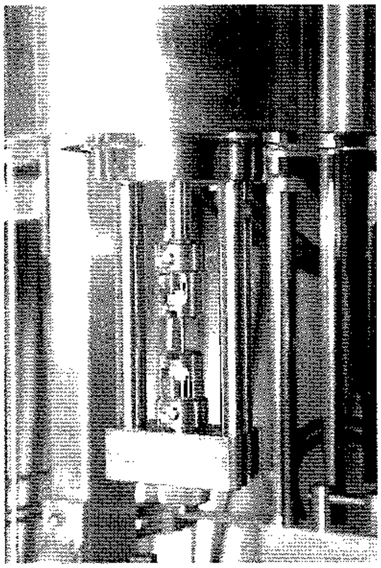

Figure 2 The mounting of the SSRT sample.

entire test. A surge tank of 200 litres, with continuous gas purification, feeds the high-pressure loop, while a purification and monitoring loop is installed in bypass flow on this tank. The tank water is circulated (300l/h) over a mixed resin bed and water inlet chemistry monitoring devices. The returning water from the autoclaves passes another resin bed and the chemistry is monitored also. The on-line chemistry monitoring system records the electrical conductivity, pH dissolved oxygen and hydrogen in the water. The water chemistry can be changed from BWR (both normal and hydrogen water chemistry can be simulated) to PWR chemistry conditions. The changing of the water chemistry typical takes 3-4 days of cleaning and replacing resins. Typical chemistry conditions are:

- ✓ BWR: conductivity < 0.1µS/cm; DO: up to 200ppb (NWC), DH: 10-30CC/kg (HWC).
- ✓ PWR: typical Li (2-3ppm) + B (400ppm); pH 7.5 (at room temperature), conductivity 20-30 µS/cm; dH 20 – 40 CC/kg.

4. Special features for hot-cell use

Some of the special features that were installed in the system were mentioned already above. Other changes that were made to the system, compared to the non-nuclear version are:

- ✓ Installation of a blow-down tank: this tank is added to the system in order to be able to discharge the hot water from an autoclave in case of an emergency (large leak, overpressure...). The tank contains a sufficient quantity of cold water in order to be able to cool down the content of the autoclaves. It can also be used as wastewater collector.
- ✓ The crud filter is integrated in the high temperature / high pressure part of the loop and located inside the hot cell. The size is taken considerably larger than in the non-radioactive case, in order to ensure a high life time. In case of saturation, it can be removed from the circuit with the manipulators and disposed of a s waste. Furthermore, there are two ion exchange beds (instead of one in a non-radioactive loop), in order to have minimum contamination of the low-

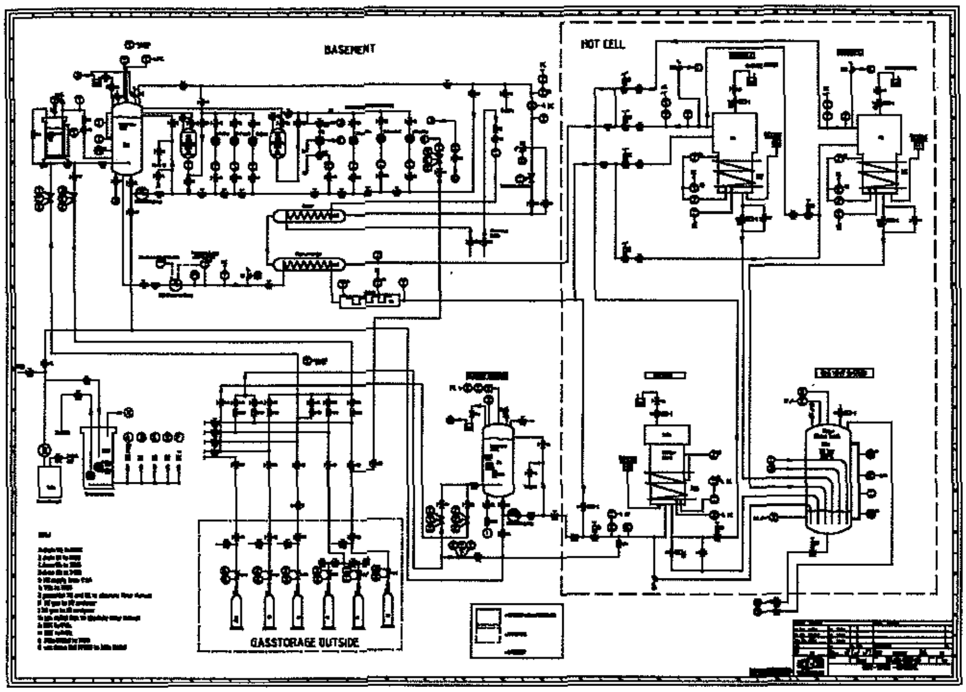

Figure 3 Flow sheet of the test system: the low pressure part (top left corner), containing the water feed and stirage tanks, the water chemistry monitoring and the the cleaning system; the high pressure section, contaning the pump, heat exchangers and the autoclaves (left, inside the hot-cell); the conditioning and filling tank for the Hastelloy autoclave (lower center); the gas supply system (lower left) and the blow down tank, also inside the hot-cell (lower right).

pressure part of the loop. The water coming back from the autoclaves is 100% filtered in the first ion exchanger, while the second one acts as conditioning exchanger for the 200 litre storage tank (and thus in principle only receives fresh water or water that has been cleaned before).

✓ All hydrogen gas used is collected in a closed circuit and released into the ventilation of the hot cell. As the release rate and inventory of hydrogen in the system is very small compared to the ventilation flow rate, there is no risk of creating an explosive gas mixture. Hydrogen sensors are installed both inside the hot cell and the basement, with automatic action to close the hydrogen supply in case of leak detection or ventilation failure.

✓ All high-pressure valves inside the hot-cell are redundant; there is an automatic pneumatic valve, with a manual back up. The flow through the three autoclaves can be in parallel or in series. In normal operation, only the automatic valves are used. The manual valves are used for isolating part of the loop in case of anomalies.

✓ The use of a small number of closing bolts with torque wrenches reduces the failure probability (compared to 8 bolts). In case of failure of a wrench, it can be replaced with another one by use of the crane and manipulators. In case of blocking of the nut on the bolt, the bolt can be removed by unlocking it at the bottom, so the autoclave can still be opened. The use of a small number of bolts implies the use of a very thick lid; this is beneficial for reducing the chance of leaks through the sealing, because the possible distortion of the lid is much smaller than in the case of a thinner lid with many bolts.

- ✓ The feed through of instrumentation wires will be adapted in order to require a minimum of operations inside the hot-cell. The principle is to assemble instrumented tests outside the cell (except for the insertion of the irradiated sample) and then insert this in the autoclave system. Special closure systems are developed for use with manipulators or special tools.
- ✓ A particular design has been made for the use of reference electrodes in the hot-cell autoclaves. The major problems with a high temperature reference electrode in a hot cell are:
  - Sufficient long lifetime
  - Reference electrode should survive under irradiation
  - The design should allow manipulation with the hot cell manipulators

The concept of the external pressure balanced reference electrode has been used as a staring point. This reference electrode has a sufficient long lifetime as it operates at room temperature. Also it wouldn't suffer by radiation as the reference electrode is positioned outside the autoclave and so the radioactive sample is shielded by the autoclave and the test solution in the autoclave. Special attention has been given to the design, regarding control by manipulators.

The pressure balanced reference electrode consists of a movable piston to maintain a pressure balance between the reference solution and the test solution. The inner part of the reference electrode is a Ag/AgCl reference electrode i.e. a silver wire with a silver chloride coating in a 0.1 M KCl electrolyte solution. This will give a reference potential of 288 mV versus the Standard Hydrogen Electrode (SHE) at 25 °C. A porous plug made from magnesium stabilised zirconium oxide, maintains a conducting connection with the test solution. The reference electrode is fixed in a stainless steel tube, which is connected to the autoclave. All inner parts of the stainless steel tube are covered with Teflon to avoid contact of the conducting walls with the test solution. The reference electrode chamber of the stainless steel tube is kept at room temperature by circulation of cooling water. For temperatures higher then 300°C the lower end of the Teflon tube ("salt bridge") can be replaced by a zirconium oxide tube.

5. Conclusions

A test system for IASCC research has been built up in the hot laboratory. It is designed for performing slow strain rate tests and crack propagation tests on irradiated materials in simulated primary water environment. Both PWR and BWR chemistry can be simulated and also a Hastelloy autoclave is available for simulating crevice or faulted water chemistry conditions.

For the operation in the hot cell environment, the system has been automated to an important extend. Instrumentation components inside the cell have been redesigned in order to facilitate replacement and operation. The system is conceived to limit the radioactive contamination by activated corrosion products from the samples to a minimum. Therefore, the filtering system has been enhanced in order to confine the contamination to high-pressure part of the loop.

The mechanical design and lay out of the system are to enhance the fail-safety by redundancy of systems (manual and pneumatic valves) and special measures to limit the consequences of failures (adaptation of closing and opening mechanism and introduction of a blow down tank).

The system has been integrated in the refurbishment of the hot cell by modification of the storage, manipulators, sample transfer door and lifting crane. The total concept of the cell and its equipment will allow an optimum efficiency and safety of the corrosion test system.