Design and Construction of New Hot Cells and Micro-Machining/EDM Facilities at ANSTO

K J Thorogood<sup>1</sup>, E Bamberg<sup>2</sup>, E G Obbard<sup>1</sup>, K Toppler<sup>1</sup> and R P Harrison<sup>1</sup>

#### **ABSTRACT**

The Australian Nuclear Science and Technology Organisation (ANSTO) has operated research reactors since the late 1950s. The OPAL reactor was commissioned in 2006 and is used as a source of neutrons for medicine, provision of beams for research, silicon irradiation, NAA/DNAA and the effects of neutron irradiation on structural materials. While ANSTO has extensive hot cell facilities on site these are mainly used for the production of medical isotopes or reactor maintenance activities. Additional capability was required for the examination and testing of material from the OPAL materials surveillance program and from research programs aimed at monitoring changes in structural materials for advanced nuclear power generation systems. The purpose of the surveillance program is to monitor changes in the properties of OPAL's core materials; principally zirconium and aluminium alloys, and to ensure that the changes are as expected.

ANSTO undertook an extensive review of hot cell facilities internationally and put out a tender for construction. This was awarded to Robatel Industries of France. An additional and essential part of the facility was the capability to undertake machining of active materials, in order to convert them into suitable test specimens. Several options were explored with the final contract being awarded to Viteris Technologies for the development of their micro-machining/electro discharge machining (EDM) device for hot cell operation.

This paper presents the overall design of the facility and a detailed examination of the micromachining/EDM centre and describes the actions undertaken to convert a selection of retired reactor components into test samples such as "dog-bone" tensile specimens, compact tension fracture specimens and small punch and TEM discs. The activity of the samples will be very high, with samples of zirconium alloy irradiated to a fluence of up to  $4 \times 10^{22}$  n/cm² (E > 1 MeV) being prepared (the cells are designed to contain up to ~200 GBq of Co60 each).

### 1. INTRODUCTION

Research reactors have been used to irradiate structural materials for power and research reactor (RR) applications in many countries for several decades; indeed, this was one of the original purposes of such devices. Along with the capability for irradiating materials, additional facilities are required in order to handle the radioactive material when removed from the reactor. These post-irradiation examination (PIE) facilities are, however, large capital items that may not be affordable to all RR operators. At ANSTO the original use of the HIFAR reactor was for training and materials research for a planned nuclear power program. Hot cell facilities were constructed and materials irradiation and extensive PIE studies were undertaken from the early 1960s to the late 1970s. Since the cancellation of the nuclear power program in the early 1970s, the use of these cells has changed and is now principally directed towards medical isotope production and reactor maintenance.

<sup>&</sup>lt;sup>1</sup> Institute of Materials Engineering, Australian Nuclear Science and Technology Organisation (ANSTO), New Illawarra Road, Lucas Heights, NSW, 2232, Australia

<sup>&</sup>lt;sup>2</sup> Viteris Technologies, 1883 South 5070 West, Salt Lake City, Utah, 84104, USA.

The renewed interest in nuclear power internationally in the early part of the current century has resulted in a reinvigorated interest in the use of RRs for materials research. Several new facilities are under construction and the need for irradiation facilities has increased. ANSTO also renewed its focus on future nuclear power systems in the last 5 years and has recently commenced irradiating materials in OPAL for research applications. Several materials including Fe-based ODS, Zr alloys, titanium/aluminum alloys, Ni-based alloys and aluminum alloys are being irradiated to study the effect of neutron irradiation on structural materials. Other programs are also due to commence in the near future. This renewed interest in materials research and the needs of the OPAL materials surveillance program prompted a full review of current facilities and concluded that new facilities would be required. This has resulted in the opportunity to obtain state-of-the-art PIE facilities, while still being cognisant of funding limitations.

ANSTO's new hot cells consist of three individual but linked cells with different facilities. The first cell incorporates cut-off and polishing equipment plus a machining centre (including EDM) capable of reducing the volume of samples and converting them into test pieces. The second cell is the main interface with the outside world and includes a docking facility to transfer samples from a compact DU flask or from a Padirac flask interface at the rear (all cells have this facility). The third cell is essentially a shielded test facility. Various instruments, including mechanical test machine and hardness testers, will be installed for testing purposes, decontaminated after testing and then be removed. The facility also includes an enclosed area behind the cells where flask activities and decontamination may be undertaken.

The first samples to be tested in the new facility include tensile and small punch disc samples from the OPAL materials surveillance program. These are to be removed from OPAL in early 2014 and will be tested when the cells are fully commissioned. The irradiation and PIE facilities are available for third party projects.

# 2. OPAL RESEARCH REACTOR

The OPAL research reactor was commissioned in 2006 and commenced routine operation in 2007. It operates on a 30 – 35 day cycle with a short refueling period and operated for 293 days in 2013 (with a target of 300 days in 2014). The reactor is a multipurpose device with the main objectives being the generation of neutrons for medical isotope production and research using neutron scattering instruments (Bragg Institute). In addition, facilities exist for the irradiation of materials for a variety of purposes. Along with the dedicated materials surveillance program for OPAL many irradiation positions are provided for NAA, DNAA, silicon, minerals and radiation damage studies.

Further information on OPAL can be found in the literature [1, 2, 3] or the ANSTO web page [4].

#### 3. OLD POST-IRRADIATION FACILITIES AT ANSTO

ANSTO has extensive hot cell facilities on site and these have been heavily utilized mainly for isotope production since the mid-1970s. Prior to this time extensive irradiations were undertaken in the HIFAR reactor for radiation damage studies. With the construction of the OPAL reactor the need for facilities for the testing and characterization of active materials became apparent. These were for two purposes; the first to provide the means of handling radioactive materials that form part of

research into the effects of radiation on material, and the second to provide the capability of satisfying the requirements of the OPAL Materials Surveillance Program.

The existing hot cells include facilities for handling highly radioactive medical and industrial isotopes and processing facilities for Mo99 production. However, facilities for undertaking sample preparation and mechanical testing were no longer available, necessitating the need to develop this capability.

#### 4. NEW PIE FACILITIES

## Layout and Functionality

Three hot cells are connected by airlock transfer shutters. Cell-1 contains cutting and machining equipment that is expected to become contaminated during use. Cell-3 contains clean test equipment that will be maintained non-contaminated during normal operations. For this reason the handling of alpha emitters and other highly radiotoxic nuclides is currently beyond operational scope. Cell-2 acts as the barrier to passing contamination into Cell-3, sample decontamination area and main docking interface to other facilities. This design is intended to maximize utilization of the available space in Cell-3 by enabling the possibility of changing equipment, via the rear door into the hot cell maintenance area. It is intended that future requirements for new experimental programs may be met by reconfiguring equipment in Cell-3 (Table 1).

Table 1: Internal dimensions and general features of the containment boxes

	Dimensions (W-D-H, m)	Windows	Manipulators	Function	Operating pressure (Pa)
Cell-1	2.8 x 1.5 x 2.0	2	4	Milling/EDM, Metallographic polishing	-300
Cell-2	1.8 x 1.5 x 2.0	1	2	Vertical flask docking, Source storage, Decontamination	-250
Cell-3	1.8 x 1.5 x 2.0	1	2	Mechanical testing, Optical microscopy, External posting port	-200

## Project Management and Procurement Strategy

The Engineering-Procurement-Construction project was managed within the ANSTO Institute of Materials Engineering by a combined Facility Scientist/Project Manager Role (EGO). This was chosen in order to embed the project team in the client organization and ensure constant alignment of the project business case, functional requirements and project deliverables. Nuclear engineering scope, including hot cells, ventilation system and confinement enclosure design was delivered by Robatel Industries; the confinement enclosure construction, civil works, fit-out and building upgrades were delivered by local consultants and building contractor, Kane Constructions; the combined CNC milling/EMD machine is provided by Viteris Technologies.

A requirements-based specification was developed to assist procurement. This specified the source term for which to design shielding and allowable external dose rates ( $<10\mu Sv/hr$  at the cell face). Source term and dose rate specification, as opposed to shielding material and thickness, has the advantage that performance can be verified by using a source for acceptance testing, while the vendor retains a high degree of design flexibility for shielding design and its associated risks. The vendor must design in their own engineering safety factors. Docking and doorway frequencies and expected hot cell source duty cycles were specified which, along with issuing ANSTO's standard organizational quantitative risk assessment matrix, enabled the vendor to carry out quantitative risk assessment and therefore infer PLC reliability and hot cell interlock performance as per IEC 61511. ANSTO verified the vendor's calculations and equipment choices through a design review process.

#### Shielding

The resultant shielding is Pb-4%Sb, 205 mm at the cell face with reduced thickness in other, less frequently occupied areas. Robatel Industries' proprietary method of soldered-in steel inserts for fasteners and low-tolerance, CNC-milled stepped edges for lead shielding slabs means the shielding forms the only mechanical structure of the hot cell, without the need for additional steel bracing to meet seismic requirements. Shielding was tested at Factory Acceptance Testing (FAT) with a 700 GBq Co-60 source. This was intense enough to give easily quantifiable dose rates outside the shielding to verify design compliance, casting defects, and post-assembly shine-paths. Based on favorable test results, the allowable hot cell inventory for licensing purposes was able to be revised upwards from the time of writing the procurement specification to make full use of conservatisms introduced during the design process. Shielding was re-tested at Site Acceptance Testing (SAT) with a much smaller source, more for the purpose of verifying correct site assembly, than as a full repeat of the FAT.

### Containment

To ensure forward compatibility with regulatory requirements the containment design was specified to meet ISO 17873. While cutting or grinding irradiated metallic materials is not likely to produce great amounts of airborne contamination, the requirement to open, cut and core ceramic wasteform sintering cans could potentially do so, depending on the waste formulation. This scenario was therefore taken as a worst case airborne contamination for the purpose of defining containment classes. Thus the primary containment glove-boxes provide containment class C4 corresponding to ventilation system design at least Type IIIA under ISO 17873 guidelines.

Static primary containment is provided by hot cell containment boxes that meet leak tightness ISO 10648-2 class two (<0.25% per hour) at operating pressure, with electrical connectors and manipulators fitted. Leak testing was performed at both FAT and SAT. The primary ventilation system is composed of two parallel fan/filter lines running in duty/standby configuration to achieve a high reliability. Reliability performance was specified during procurement using a Mean Time Between Failure (MTBF) value and compliance was ensured by requesting a vendor's calculation note for the ventilation systems according to IEC 61078. The hot cell containment glove-boxes incorporate an automatically activated inert gas fire-suppression system to guarantee integrity of primary containment in case of hot cell fire.

Secondary containment is provided by a confinement enclosure, constructed from polyurethane foam/stainless steel sandwich panels and structural steel frame, which encloses the hot cell line, the primary ventilation system and the rear-of-cells maintenance area. The confinement enclosure achieves dynamic containment by virtue of an independent extract ventilation system, and is split into a cell face side that remains non-contaminated in normal operation and the rear-of-cells maintenance zone with controlled contamination expected during hot cell maintenance procedures, nominally corresponding to respectively containment classes C1 and C2, depending on operations. The secondary containment ventilation system was also procured by specification and design review to a MTBF value according to IEC 61078.

The facility stands in an existing building that provides a level of tertiary containment, and which could be upgraded to include controlled ventilation if required in the future. A mock-up facility with a volume the same as a single cell has been constructed at ANSTO to enable in-cell sample preparation and testing activities to be practiced in a non-active environment. Spare manipulators have been procured with the cells and these will be used in the mock-up.

Assembly has recently commenced of the cells at ANSTO. Figure 1 shows the state of assembly in mid-October 2013 (Figure 1a) and recently (Figure 1b). It is expected that the cells will be completed later this year with testing of the first active samples in early 2015.

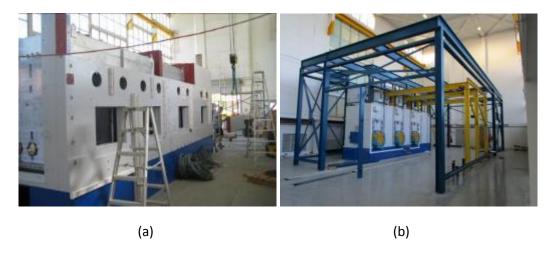


Figure 1 Assembly of the new ANSTO PIE hot cells as at (a) mid-October 2013 and (b) August 2014. The stainless steel confinement boxes are visible behind the lead shielding.

## 4.1. In-cell machining centre

A novel feature of the new PIE facilities at ANSTO is the capability to undertake conventional and electro-discharge machining (EDM) of active materials. The requirement for this capability came from both the OPAL materials surveillance program and the need to reduce the volume of active materials so that they can be examined in conventional equipment such as an SEM or TEM. ANSTO does not have these facilities in a hot cell but is able to examine small volumes of active materials under controlled conditions. As part of the hot-cell project a request for tender was issued for such a capability. The task of providing an EDM device to fit into the hot cell is difficult and although Viteris Technologies LLC had not done this previously for an active facility, they were contracted on the basis of their extensive experience in micro-EDM and their willingness to provide customized solutions. A schematic of the machining centre is given in Figure 2. All tool changes and wire

threading can be undertaken remotely. The system was trialed at the hot cell FAT in France and is currently being used in the mock-up in order to develop experience prior to final in-cell commissioning later in 2014.

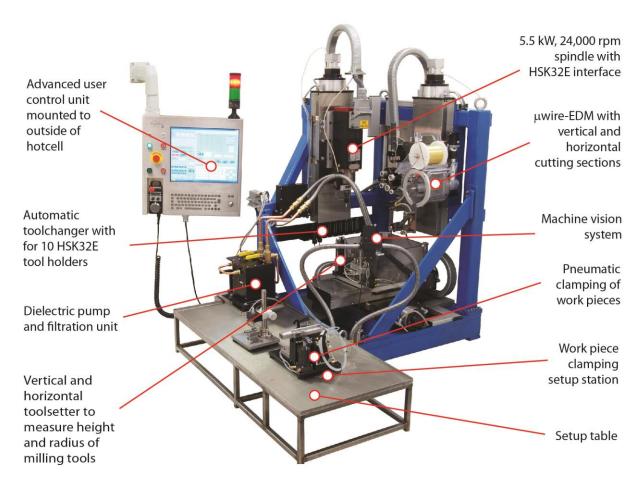


Figure 2. Overview of the machining centre to be installed in the ANSTO hot cells.

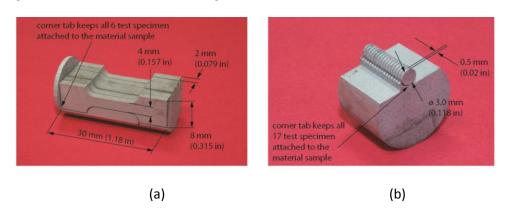


Figure 3. Test samples to be machined from an OPAL control rod (16.2 mm diameter). Tensile test specimen (a) machined in a single setup using the vertical and horizontal WEDM of the VHMC01. Six samples can be machined together from a rod section of 32 mm (1.26 in) length. TEM (transmission electron microscopy) samples (b) are machined as disks with 3 mm (0.118 in) diameter and 0.5 mm (0.02 in) thickness. 17 disks are machined together from a 12 mm (0.47 in) section in a single setup using the vertical and horizontal WEDM. Both sample types are held by small tabs that can be snapped off later using the manipulator.

It is possible to section active materials into the variety of sample forms using the machining centre. Examples are shown in Figure 3; (a) being the nearly finished miniature tensile test pieces (30 mm long) and (b) showing a section of OPAL control rod (16.2 mm diameter) sections to provide small punch discs (6 mm dia) and TEM discs (3 mm dia). This sample had already been sliced into 0.6 mm thick slices prior to the circular wire cut.

Sectioning of a pre-tested non-active tensile "dog-bone" sample was undertaken on the mock-up (Figure 4). The end tabs were sliced into small punch discs; 6 mm diameter and 0.6 mm thick. Two such samples can be obtained from a 2 mm-thick tensile sample using EDM. The samples will be surface ground (both sides) down to a thickness of 0.5 mm prior to testing.



Figure 4. EDM machining of tested mini-tensile sample. Two small punch discs can be obtained from each end-tab (the third is too thin for testing).

Because the samples are very small, pneumatic tweezers will be used to pick the samples up and transfer them to transport holders while a small USB camera is used to assist. The camera is a low-cost unit and will be replaced when radiation damage has caused a loss of image quality or other failure. This process will be developed in the mock-up and is acknowledged as being the most difficult in developing skills and experience of the operators.

# 4.2. Mechanical property measurement

A dedicated in-cell mechanical testing machine (made by TestResources Inc.) will be installed in cell 3 and will be used to provide both tension tests – mainly for the miniature tensile specimens shown in Figure 3 – and small punch tests. All controls are external to the cell and appropriate grips, jigs and extensometers and being developed to suit a range of samples. A micro-hardness tester will also be used to measure radiation induced hardness.

## 5. SUPPORT FACILITIES

Along with the hot cell facility used for initial sample handing and preparation, several other items are available at ANSTO to undertake examination and characterization of active materials. Where the samples are small enough or have low activity the opportunity is available to examine them in an SEM in a controlled area. Focused ion beam (FIB) equipment is also available for sectioning samples to reduce volume and provide cross sectional grain boundary samples for TEM examination. Although the FIB is not located in a designated radioactive area it is possible, with appropriate health physics support and a carefully controlled method, to section small radioactive samples. The Zeiss

FIB is fully operational and a large number of transverse TEM samples have been prepared for various research projects. Figure 5 shows a selection of test pieces that have been prepared using the FIB.

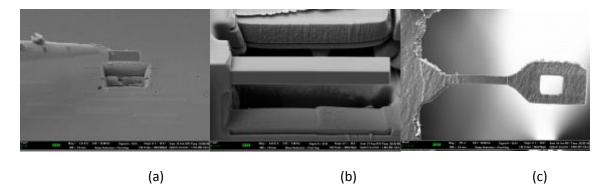


Figure 5. Samples produced using the FIB; (a) in-situ lift-out of a cross-sectional TEM foil, (b) cantilever with dimensions of 20 x 3 x 2  $\mu$ m for bending test, and (c) tensile specimen with gauge length of 130  $\mu$ m

An in-situ micro-mechanical testing device is available for performing compression and tensile testing. Additional testing modes (such as high temperature testing, fatigue testing, etc.) will be implemented in the near future. Figure 6 shows results for (a) a tension test and (b) a compression test, the gauge length of each sample being  $^{\sim}100~\mu m$ .

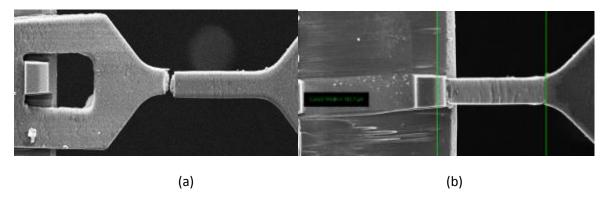


Figure 6. Tested samples from the in-situ micro-mechanical test facility; (a) a fractured tensile sample and (b) a compression sample showing deformation bands.

### 6. POST-TESTING SAMPLE MANAGEMENT

ANSTO has the facilities to prepare and store radioactive materials. Storage facilities exist that are either retrievable (mortuary holes in other hot cells on the ANSTO site) or more semi-permanent. All samples are currently retained on-site. Although ANSTO is not permitted to import radioactive waste, import of radioactive material associated with research or other applications is possible in many instances.

#### 7. SUMMARY AND CONCLUSIONS

ANSTO has the capability to irradiate a variety of materials in the OPAL reactor either in water-cooled or nitrogen-cooled irradiation facilities. While the reactor is not a dedicated MTR it can provide useful flux levels for materials irradiations. ANSTO has also invested heavily in the development of its PIE facilities so that materials that have been irradiated in OPAL and elsewhere can be examined and characterized without need to send them to facilities overseas. ANSTO is now in the position to not only irradiate materials of interest to GenIV reactor developments but also assist in the assessment of materials for research reactors. Medium and higher power research reactors built in the last 20 years and into the future will have, or will be required to possess, a materials surveillance program aimed at assuring that radiation-induced materials property changes are as predicted. There will be a need to either have PIE facilities at the reactor site or to provide the ability to ship samples to facilities elsewhere to undertake the required measurements. ANSTO has this capability and is prepared to accommodate material from other research reactor and power reactor operators.

#### 8. ACKNOWLEDGEMENTS

The development of the new PIE facilities at ANSTO has been the product of a number of people and organisations. Within IME, Kevin Thorogood and Karl Toppler have made significant contributions to the methods and instrumentation to be used in-cell. Extensive work was undertaken by the Contracts section of ANSTO Finance and Procurement and also Engineering and Capital Programs. Robatel Industries have contributed to the design and now construction of the hot cells, while Viteris Technologies LLC has contributed significantly in the development of the micro-machining capability.

## 9. REFERENCES

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