The new Shielded Metallography Box in Paul Scherrer Institut Hot Laboratory

Didier GAVILLET[1], Andreas URECH Paul Scherrer Institute Laboratory for Materials Behaviour CH-5232 Villigen – PSI Switzerland

Abstract

The new metallography box has been finally completed and installed in PSI hot laboratory. The definitive set-up of the heavy shielded box is presented including the infrastructure used for the specimen preparation and observation.

The Box was commissioned at the beginning of the year 2004. The paper presents the experiences gained during the first months of operation and shows the capabilities of the infrastructure for the preparation and observation of highly radioactive materials using chosen examples.

Keywords: *PIE, Instrumentation, Metallography*

A short overview of a long story

After about 25 years of service, the old PSI metallography box was strongly corroded with outdated infrastructure. In 1998 it was decided to start the conception and construction of a new modern box.

The project formally started at the beginning of 1999 with the presentation of the box concept and financial plan. The international call for offer and evaluation of these offers were realised mid 2000 and the formal ordering of the boxes and of the major infrastructure (polishing machines, saw, manipulators, etc) were made at the end of the same year. At the same time the decommissioning of the old metallography box started. During 2001, the dismantling of the old box was completed and the preparation of the laboratory, which included a re-enforcement of the floor due to the larger weight of the new box, was completed. The inner steel boxes for the alpha-containment and the 25 cm thick, 80 tons steel shielding constructed by a local Swiss firm was delivered and mounted during year 2002.

At that point the installation and test of the infrastructure started. This phase took much more time than expected and the final commissioning of the box was completed only in beginning of 2004.

Finally, after 4 years of hard work, the first radioactive specimens have been prepared and analysed in the new PSI metallography box during summer 2004.

The paper gives a general description of the new infrastructure available in PSI. Two metallographic / ceramographic investigations of radioactive materials are used to demonstrate the capabilities and qualities of the new and versatile infrastructure.

Main features of the new metallography box

The concept and basic design of the shielded box were realized entirely in PSI using the available competences. A sketch presenting the most important features of the box is presented in Fig. 1.

The concept is based on one large box for the specimen preparation with a large floor space and one small box for the optical microscopy investigation of the specimens. Both boxes are entirely and independently shielded (250 mm steel for the preparation box and 200 mm for the microscopy box). Both inner boxes are in stainless steel and alpha-tight. The transfer of the specimens between the two boxes is made with a rotating lock.

The construction was realized by a Swiss Firm. The inner alpha-containment is entirely made of stainless steel with an electro-polished inner surface to insure an easy cleaning / decontamination of the working zones. The shielding walls are entirely made of steel. The rotating locks are from own design. O-Rings

[1] Phone (+ 41) 563 10 22 82, Didier.gavillet@psi.ch

insure the tightness of the lock during operation. Pictures of the inner box and the shielding during fabrication are presented in Fig. 2.

The working pressure of the boxes is set to -2 to -4 mbar with a larger under-pressure in the preparation box than in the microscope box and in the inactive lock. The under pressure is regulated by mechanical valves that insure a safe operation of the box (Fig. 3). The box can bee filled with laboratory air, with pressurised air (dry and clean) or with nitrogen. With nitrogen operation, an oxygen level under 5%, insuring that alcohol cannot be inflamed is obtained in less than 30 minutes. A safety device has been installed to insure that no pressurisation of the box occurs if the exhaust system falls out.

In order to insure an easy transfer of consumables in the preparation box, a so called inactive lock equipped with a large rotating lock has been installed on one side wall (Fig. 3). This allows an easy and quick transfer of the polishing paper, cloths or embedding material in the box and greatly reduces the amount of material that must be stored inside the containment.

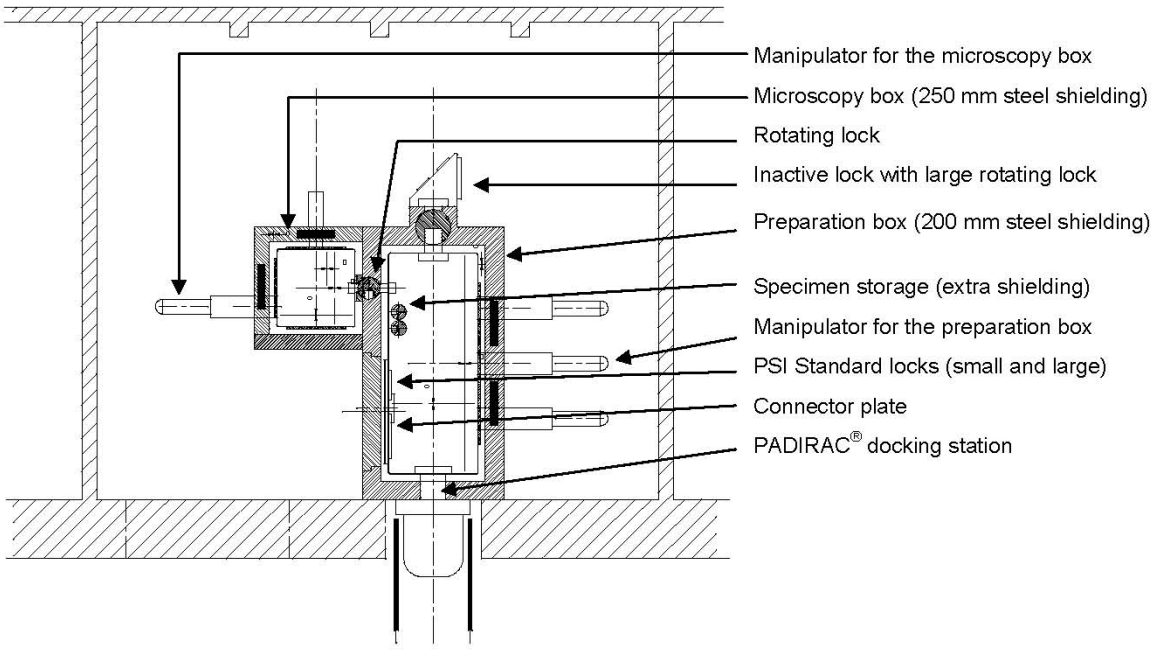

Fig. 1 Sketch of the new PSI metallography box

On the other side wall, a PADIRAC® docking station is available for the transfer of highly radioactive material or fuel specimens in and out of PSI using this transport cask.

In addition, two "PSI standard lock systems" (small and large) are available for the internal specimen and waste transfer (Fig. 4). The standard waste-box in PSI has an internal diameter of 209 mm and a length of 500 mm. All contaminated materials must be removed from the box using such a waste-box. This has put large constraints on the infrastructure of the box.

A shielded internal storage room for specimens is available in the box (Fig. 4). This allows the storage of about 30 specimens inside the containment without unacceptable dose rate elevation in it.

Fig. 2 Picture of the inner alpha containment, of the steel shielding and of the large rotating lock during the construction of the box

Fig. 3 Left: under-pressure regulation and feeding systems for the boxes (upper part of the picture) and inactive lock for the transfer of consumables (bottom part of the picture); Right: PADIRAC cask docked on the box

Fig. 4 Left: shielded cask in front of the PSI lock for waste disposal; Right: internal shielded specimen storage

The specimen preparation in the large box is made with three master/slave manipulators (Fig. 5). Two large windows and two small ones on the side insure a good vision of the work area. A large steel mirror installed on the back wall allows having a full view of the box floor.

Fig. 5 Left: front face of the preparation box; right: microscopy box

The specimen manipulation and positioning on the optical microscope in the microscopy box is made with one master/slave manipulator (Fig. 5).

The entire infrastructure installed in the preparation box has been specially designed or modified for our specific use. The major modifications were needed in order to insure a possible transfer of the machines inside the standard PSI waste container. The standard infrastructure includes one cutting machine, two polishing machines, one ultra-sonic cleaner, one embedding system and one hot plate and one specially designed etching box that should reduce the long term corrosion in the box (Fig. 6).

All electrical, gas and liquid feedings are made through special connectors all positioned on one connector plate that could be removed or modified in case of need (Fig. 6). All electrical cable are kept out of the floor in order to reduce the risk of damage (Fig. 6).

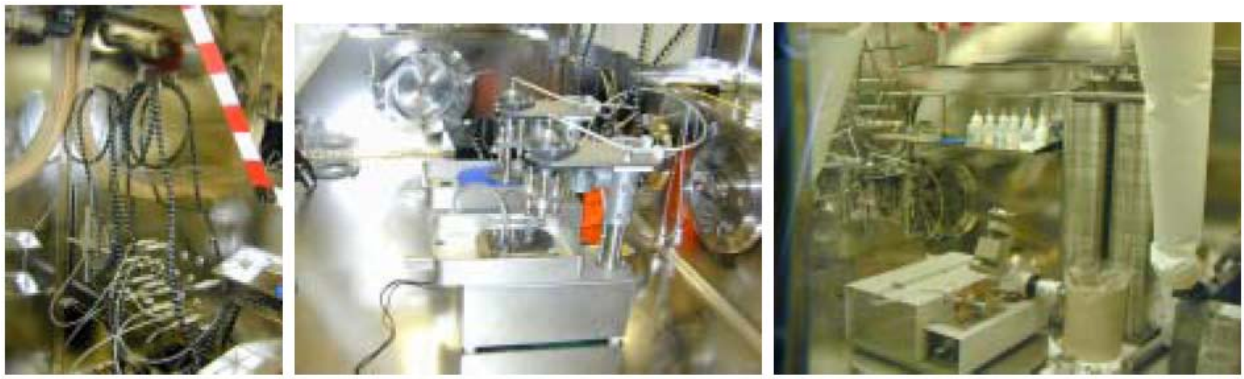

Fig. 6 Left: connector plate and cable holders; middle: polishing machines; right: cutting machine

The optical microscope installed in the microscopy box is a Leica® TELATOM4® inversed metallographic microscope (Fig. 7). It is fully remote controlled and allows the observation of the specimen at magnification between 8 to 2500x. The image acquisition is realised with a high resolution digital camera and dedicated software running on PC. The software allows a quantitative analysis of the image, it's archiving and the automatic preparation of documentation files. A Plexiglas plate has been mounted on top of the microscope to protect it and prevent a fall of a specimen at the bottom of the box.

The microscopy box includes a sliding door that allows an easy access to the microscope for cleaning and service operation (Fig. 7). Four gloves allow the routine service of the instrument without opening of the alpha containment. Large servicing and complete cleaning can only be done after a decontamination of the containment and removal of the front Plexiglas plate.

Fig. 7 Up-left and centre: microscopy box with sliding door closed and open; Up-right: external part of the microscope; down-left: internal part of the microscope; down-right: upper part of the internal part of the microscope with its Plexiglas shield

First analysis of radioactive materials

The first metallography and ceramography analyses of radioactive material have demonstrated the versatility of the specimen preparation box and the very high quality of the microscope.

Two examples are given here: an observation of a hydrided Zircaloy cladding specimen and the observation of an irradiated fuel pellet cross section. The goal here is just to show the quality of the observation realised and not to give information of the specimen themselves.

In Fig. 8, the hydride structure in the Zircaloy specimen has been observed at different magnification. This reveals the lenses formation as well as the local structure of hydride. The capability to stitch numerous pictures together in order to get a very high resolution image of a large area demonstrates the very good quality of the optics.

The observation of an UO_2 irradiated pellet cross section is presented in Fig. 9. The specimen preparation was focused on the gap region and the observation of the interaction layer between the fuel pellet and the cladding. The presented pictures demonstrate the capability of the microscope to get high contrast, high resolution pictures of the fuel pellet at magnification between 10 to 2500 times.

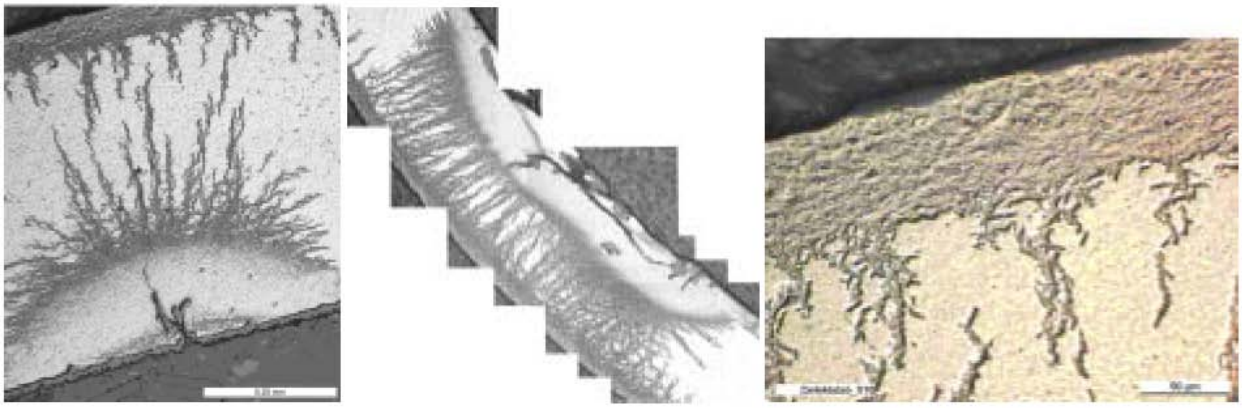

Fig. 8 Observation of the hydride structure in an irradiated Zircaloy cladding cross section at different magnifications. The two left pictures have been realised by stitching two and 7 pictures together respectively

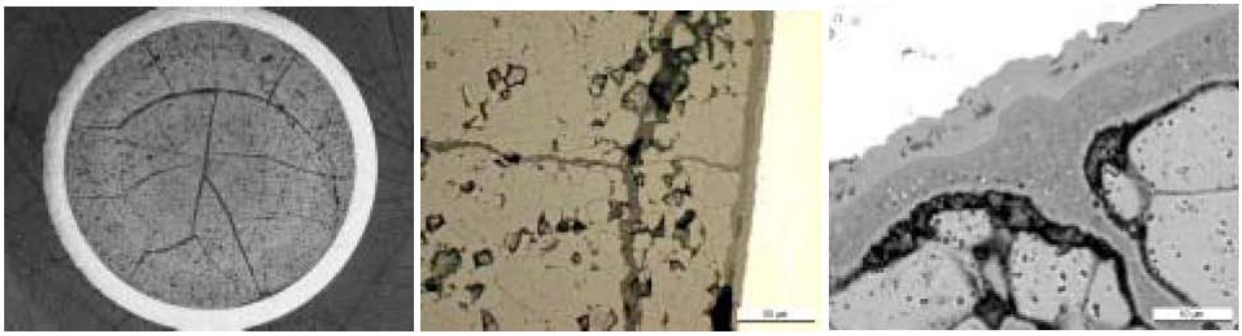

Fig. 9 Cross section of a pellet observed at different magnification between 10x to 2500x

Summary

A new modern and versatile shielded box has been developed in PSI for the optical microscopy of highly radioactive materials. The box has been commissioned in April 2004 and the first analyses of irradiated cladding materials and nuclear fuel have already been realised.

Thank to the modern and powerful infrastructure (optical microscope, digital camera) very good analysis can be realized on irradiated materials, including fuel. The first analyses have already demonstrated a clear improvement of the PSI capabilities for optical observation of nuclear material.