The new Isidore microscope

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Abstract

In the frame of the refurbishment of LECI hot laboratory in Saclay, it was decided to renew one of the two metallography lines of the building. This line is located at one end of the "Isidore" line of lead-shielded hot cells.

The work started by the cleaning of 5 out of 9 cells in Isidore line. Two were 2 m x 1.5 m cells, whereas the 3 others were smaller. Decontamination was difficult in both larger cells, because a lot of metallographic preparation had been performed there and because the cleaning of the lower parts of the cell, below the working area, was uneasy by remote manipulators.

The refurbishment of the cells included:

- Changing the windows, because old windows were made of glass panels separated by oil, which is now prohibited by safety requirements,
- Putting of a new pair of manipulators on one large cell, and adding bootings on manipulators on both large cells,
- Changing all the ventilation systems in these cells (new types of filters, new air-ducts),
- Modifying and changing metallic pieces constituting the working area inside the cell,
- Increasing the height of the small cells in order to add a manipulator for charging the sample on microscope or on hardness machine,
- Simplifying the electrical wiring in order to decrease the fire risk in the hot cell line,
- Add a better fire protection between the working area and the transfer area, i.e. between the front and the rear part of the cells.

The scientific equipments for these cells are:

- An Olympus microscope, modified by Optique Peter (company based in Lyons), equipped with a motorised sample holder (100 x 200 mm), maximum size of sample: O.D.=100 mm, 6 magnifications: x12.5, x50, x100, x200, x500 & x1000, two microhardness positions: Vickers and Knoop, Polaroid image and digital camera with SIS image analysis system.A new periscope manufactured by Optique Peter, magnification x2 and x9, digital image and SIS system, and an old periscope,

- A Testwell hardness machine: Vickers, Brinell and Rockwell from 29.4 to 981 N.
- Preparation system for TEM thin foils,
- An embedding system for EPMA sample designed for Sn-Bi metallic embedding,
- Several polishing equipments and epoxy embedding system for metallography.

After completing installation of the equipments, tests have been performed to check the radioactive shielding of the cells, as well as a control of air tightness. The work is now nearly finished and the cells will soon be in operation. The main difficulties have been encountered when cleaning the larger cells, and also to perform the refurbishment with other works going on in neighboring cells (electrical wire modifications, changing windows).

Next step will be to perform the same type of work on the other metallography line, which includes 3 cells. If funding is available, this can start in 2002 or 2003.

KEYWORDS: Hot laboratory, hot cell, refurbishment, metallographic examination, microscope, hardness

1. Introduction

The LECI (French acronym for laboratory for irradiated fuel study) is a hot laboratory built in Saclay in the early sixties for examinations on fuel rods. It includes a main line (K) of 11 concrete-shielded cells + 2 for metallographic examinations, a line (Isidore) of 9 lead shielded cells and 2 separate cells for SEM and XRD.

In the early nineties, some strategic decisions were taken: most of fuel examinations should be performed in Cadarache, at the LECA-STAR facility, whereas Saclay should concentrate on material studies, although some examinations on ramp tested fuel are still conducted in LECI.

Around 1995, a refurbishment programme up to 2004 was decided and started. It includes the

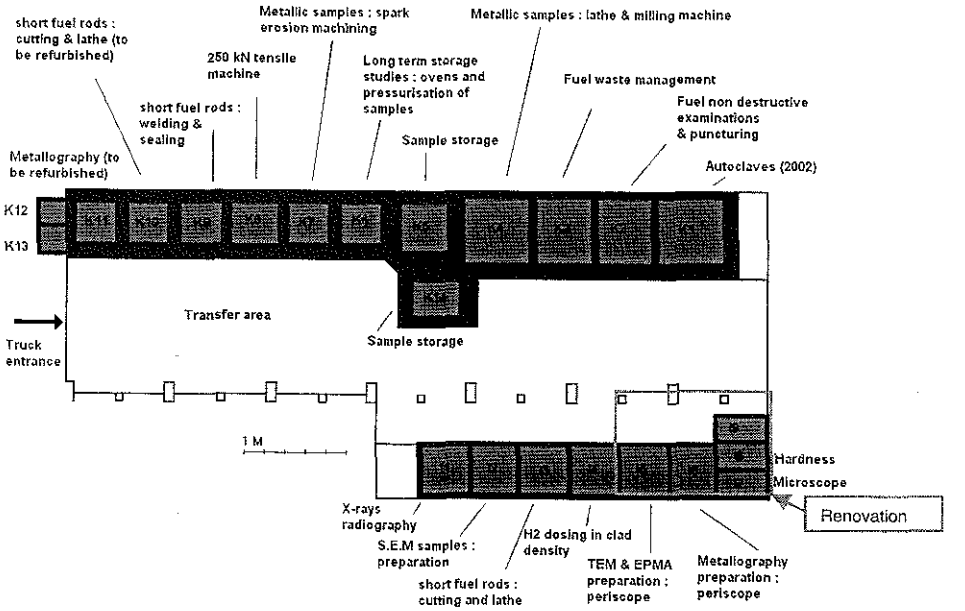


Figure 1 Present status of the two lines of hot cell in the LECI main building

renovation of about half of the cells of the existing building and the construction of a new building with about twenty lead-shielded hot cells for mechanical testing. This paper presents the renovation of 5 out of 9 cells in Isidore line extremity.

Hot cell n° I5 is dedicated to:

- The preparation of samples for EPMA examination (outer diameter: 25 mm),
- The mechanical preparation of thin foils for Transmission Electron Microscopy,
- The macroscopic observation of samples through a periscope.

Hot cell n° I6 is dedicated to:

- The preparation of metallographic samples (maximum diameter: 100 mm),
- The preparation of samples for hardness measurement,
- All chemical or electrochemical etchings.

Hot cell n° I7 is equipped with an inverse stage microscope for metallography. This microscope will be used for:

- Macrographic and micro-structural analysis of metallographic samples,
- Control and observation of EPMA samples,
- Microhardness measurements.

Hot cell n°I8 is equipped with an hardness machine for Brinell, Rockwell and Vickers-type testing. The samples could be either polished for tests under small load (29.4 to 98.1 N), or tested as received under a higher loading (up to 981 N).

Hot cell n° I9 has been cleaned and is waiting for an equipment which is not yet defined.

Hot cells from I1 to I6 are connected by a trolley system. Devices for transferring samples with remote manipulators (table on a roll-bearing carriage) have been installed between cells I5/I6, I6/I7, I6/I8. The maximum dimension of transferable samples is 100 mm. Hot cells I5 and I6 are equipped with hatches for cask which are put on the top of the cell for evacuation of wastes. I5 is the only hot cell with a hatch on the rear door for connecting a horizontal cask in the transfer area.

2. Renovation of the hot cells

All five hot cells underwent a complete cleaning. All internal equipments were dismantled and sent to waste disposal, including the working plane, the plugs on the front and rear walls, ventilation devices, cranes and electrical panels.

2.1. Preparation cells i5 and i6

To limit fire hazard, safety authorities required us to change our windows with oil for dry windows of the same dimensions (manufactured by SOVIS). Holes in front walls for remote manipulators were enlarged to enable installation of leak-proof booting. A pair of new La Calhène GHD-type manipulators were bought.

Ventilation ducts were entirely rebuilt using stainless steel. Pre-filters installed inside the cell are designed to be dismantled and compacted to gain some volume when they will be put to waste disposal. In hot cell n° I6 a hood equipped with two levels of filtration (active coal and very high efficiency filter) is designed for the storage of chemical products and chemical or electro-chemical etching of samples.

Working plane is constituted with a stainless steel casing designed to avoid that highly

contaminated fluids, such as polishing or cuttings liquid wastes, cannot flow down to the bottom part of the cell. There are no ducts to evacuate liquid wastes: the fluid quantity is optimised, evaporation is important and residual liquid wastes can be solidified inside plaster or cement. Inside the working plane, a storage compartment for the waste canister and another storage well for samples, shielded with 5 cm of lead have been arranged.

Artificial lightings are put inside protective glasses in order to change neon lamps from the working area without any contamination risk. Cold lightings were selected to avoid to high temperature inside protective glasses.

Electrical feeding networks are completely new. Inside each hot cell, an electrical panel gathers all connecting plugs (provided by Framatome Connectors International) for inside equipments. In the working area, control panels were installed inside electrical boards on the front part of the cells. Cable lengths were reduced to the maximum to limit the calorific load in the working area. All electronic driving systems for the equipments are integrated in these electrical boards inside the working area.

Each cell is equipped with a lifting unit of 1471 N capacity. This unit is motor driven by a joystick in the X and Y axes. Lifting can be performed up to 300 mm from the cell walls. The part including the winch can be extracted through the roof of the cell if repairing is needed.

Through wall plugs situated on the front and rear walls were all replaced. Most of them are equipped of helical tubes designed for the following appliances :

- Fire extinguishing feeding systems for gas and powder CO_2 (from the working area),
- Ducts for compressed air and fluids (from the working area), for high pressure argon and vacuum (from rear zone),
- Cell depression measurement (from the working area),
- Specific electrical feedings systems for the weighing scale and the motor driven table of the periscope (from the working area),.

3. Equipments

We describe hereafter the equipments installed in the hot cells together with the main technical specifications and constraints due to the nuclear environment.

3.1. Hot cell n° 15

3.1.1. Preparation equipments

- 1 pneumatic vice,
- 1 precise cutting device with diamond blade BUHLER Isomet with its control panel transferred in the working area and all controls modified to facilitate remote handling,.
- 1 embedding system for epoxy (bell under primary vacuum),
- 1 embedding system for Sn-Bi eutectic alloy, designed by CEA, with a heater up to 250 °C, under primary vacuum, then under argon pressure up to 30 bars,

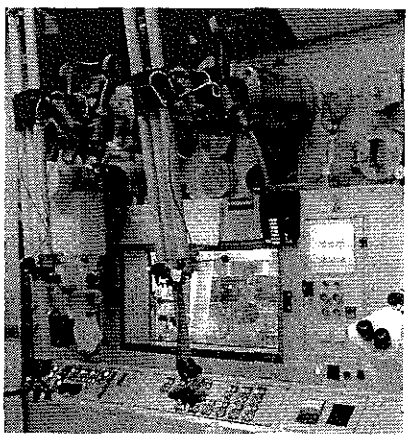


Figure 2 Hot cell n° I5

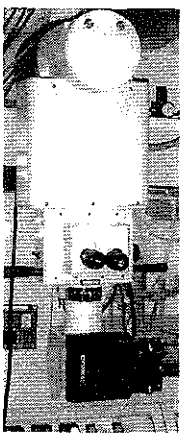
- 2 polishing machines, one sample at a time, for EPMA samples ∅ 25 mm, designed by CEA, whose speed can be controlled from the working area, and which can easily be taken to pieces by remote handling and sufficiently small to be removed in a high level waste canister,
- 1 ultrasonic cleaner,
- 1 polishing machine, 4 samples at a time for preparing TEM thin foils, designed by CEA, speed controlled from the working area, and which can easily be taken to pieces by remote handling, final grinding level of the foil is controlled by diamond standards.
- 1 heater for gluing TEM thin foils,
- 1 pneumatic punch for TEM thin foils(∅ 3 mm)

3.1.2. Periscope

This periscope was manufactured by Optique Peter company in Lyons and is designed for a high quality macrographic observation of various samples. This periscope possesses a very high resolution (200 lines / mm). It also enables to observe in true colours thanks to a catadioptric, although irradiation resistant, optical system.

Technical characteristics:

- 2 magnifications : X2 , X9
- 1 binocular head equipped with :
- A photographic Polaroid output 4"x5"
- A video output with tri-CCD colour camera,
- Rotation of vertical arm of +/- 45 °
- Protection sleeve with tight window,
- Complete biological shielding,
- Sample holder with XYZ movements
- Motor driven XY range : 100 x 100mm,



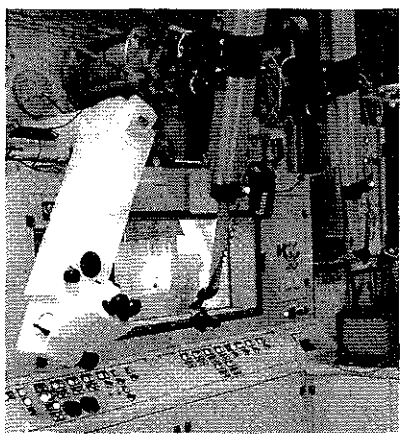


Figure 3 Periscope in I5 **Figure 4** Hot cell n° I6

- Motor driven Z range with focus : 200 mm,
- Lighting appliance by 6 halogen bulbs,
- Computer system:
- SIS image acquisition, storage and analysis,
- Driving mechanism for XYZ sample holder.

3.2. Hot cell n° 16

3.2.1. Metallographic preparation equipments

- 1 pneumatic vice,
- 1 precise cutting device with diamond blade Buhler Isomet with its control panel transferred in the working area and all controls modified to facilitate remote handling,.
- 1 embedding system for epoxy (bell under primary vacuum),
- 2 polishing machines, one sample at a time, for metallographic samples ∅ 100 mm, designed by CEA, whose speed can be controlled from the working area, and which can easily be taken to pieces by remote handling and sufficiently small to be removed in a high level waste canister,
- 1 vibrating polishing machine BUHLER Vibromet 2, control panel transferred in the working area and all controls modified to facilitate remote handling,
- 1 ultrasonic cleaner,
- 1 electronic weighting machine manufactured by Mettler.

3.3. Metallographic microscope – hot cell n° 17

This microscope was developed by Optique Peter. It was designed from an inverse stage OLYMPUS base microscope. It is equipped for observation up to a magnification of x1000 and with two dents for Vickers and Knoop microhardness. It also possesses an objective x1,25 associated with a module of image reconstruction which enables to obtain good quality macrographs and avoid buying a macroscope. Lighting appliances are transferred in the working area. Image and light transfer systems were adapted to the hot cell. Sample holder was designed for large samples up to 100 mm. All internal mechanisms are motor driven. There are two ways to handle this microscope, either completely "manually" (Polaroid pictures + Joystick), or by computer assisted handling.

Transferring and putting the sample on the microscope is ensured by the manipulator. A stainless steel working plane separates the upper part of the microscope where the sample can be, from the bottom part reserved to the microscope systems.

All optical or electrical passages are gathered on a rectangular plug situated at the bottom part of the biological shielding. The microscope is fixed to the lower biological shielding of the cell. The whole thing is put on a carriage with rack and pinion which enables to open the hot cell and gives access to the microscope for maintenance or repairing, using some particular precautions.

A contamination probe and a dose rate probe are installed inside the hot cell to facilitate sample movements and cell openings.

Technical characteristics:

- All mechanisms are motor driven.

- Observation mode (universal lighting system using reflected light) : bright background, black background, polarization : rotating polarizer / detector of polarization angle,
- Interferential contrast Nomarski (DIC)
- Control of field and aperture diaphragms
- Control of the 8 positions tower:
- 6 objectives / 2 microhardness dents from 0.005 N to 3.9 N
- Control of indentation: Vickers and Knoop microhardness
- Focus and sample displacement:
- - Stage with 3 axes for large samples,
- - Samples up to Ø 100 mm,
- - Displacement range : 100 x 200 mm
- - Rotating sample holder 360° with angular detector with a precision of 0.1°,
- Halogen lamp lighting and arc-lamp situated outside the hot cell.
- Protection of optics against radiations: when no observation is underway, the sample holder is removed to a so-called charging zone, far from the objectives and situated above a lead shield.
- Software and control systems:
- Control panel for all microscope systems,
- Controls for axes,
- Autofocus,
- SIS image acquisition, storage and analysis,
- Modules for grain analysis and hardness measurement,
- Alignment of several images enabling a large field and a high resolution : performance is equivalent to a macroscope.
- Augmentation of field depth by vertical scanning..

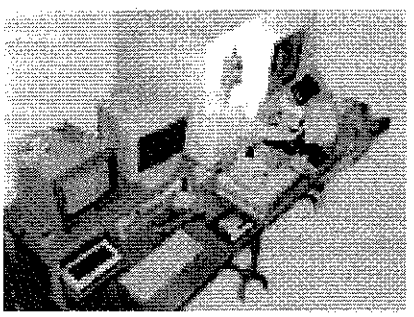


Figure 5 Control desk

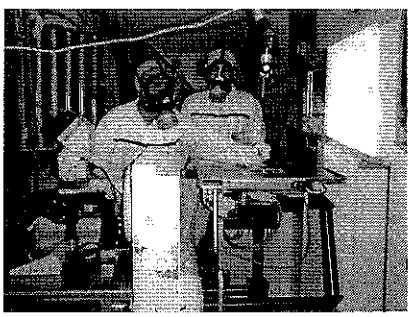


Figure 6 Microscope in maintenance

3.4. Hardness machine – hot cell n° 18

This machine was bought to Testwell Company. It is a "EMCO M4U-250" machine, modified to answer our need. This equipment enables to realize Brinell, Rockwell and Vickers hardness tests under an adjustable load ranging from 29.4 to 981 N. Maximum dimension of samples that can be introduced in hot cell n° I8 is define by the size of the manipulator-handled conveyor i.e. a cube of 100 x 100 x 100 mm.

Adaptation of the machine to its nuclear environment consisted in removing all electric or electronic controls inside the machine body to put them in the working area inside an electrical board. This adjunction of about 5 m of cables between the machine and its control systems was performed during manufacturing. Furthermore, the machine is fully computer controlled. Visualization of indentation is ensured by a video camera connected to the driving system. A XY motor-driven stage of about 50 x 100 mm connected to the software was installed on the machine table. No hand manipulation is necessary because dents and objectives are originally installed on a motor driven tower.

The master slave manipulator is only required for transferring samples and putting them on the stage. The measurement is then fully automatic: displacement of sample to perform a line of indentations or on pre-defined locations, focusing on the tested area, indentation, finally measurement by image analysis of the indentation. Our main difficulty was to find the best way to arrange such a large equipment compared to the size of the small hot cell, taking into account the possibilities of vision through the lead glass, the movements of the master slave manipulator and the introduction of the sample. We were even led to forbid a part of the cell (stainless steel casing) along the machine, to avoid that a sample could fall in a place that cannot be reached by the manipulator.

A contamination probe and a dose rate probe are installed inside the hot cell to facilitate sample movements and cell openings.

Two levels of intervention are possible on the machine:

- Regular maintenance: as on cell n° I7 the lower part of the hot cell can be open on the working area. The machine stays inside the hot cell, but an access is cleared which permits to change objectives, dents, light bulb, etc...
- If fixing is needed, the roof of the cell should be opened, and the machine removed by an overhead crane. This is an exceptional operation, which must be accompanied by specific precautions.

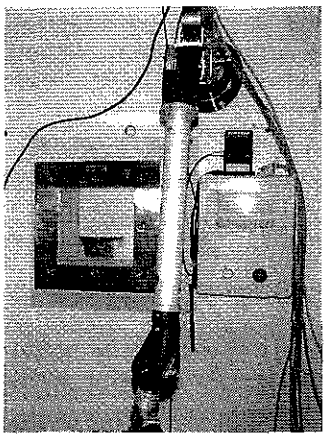


Figure 7 Front part of cell n° 18

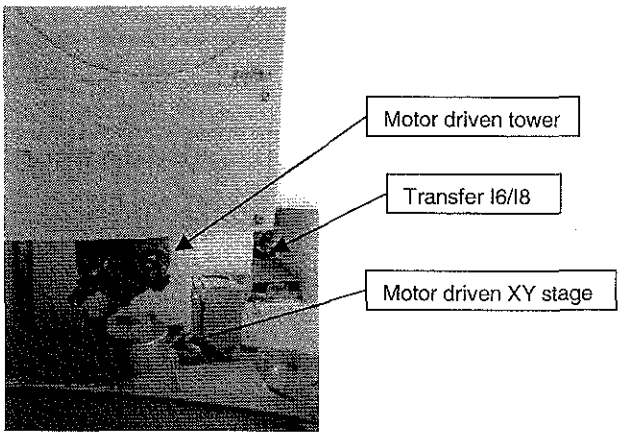


Figure 8 Machine in the cell

4. Conclusion

The refurbishment of this group of 5 hot cells lasted about 4 years. This group of cells now presents homogeneous and complementary equipments. Concerning preparation equipments, we preferred strength and robustness to sophistication. On the other hand, we chose modern and really performing characterization equipments.

Starting these installations will be followed very soon by stopping the metallography hot cells of line K (K11 to K13) for refurbishment (see figure 1). Due the decrease of such kind of equipments in France and the loading factor on this type of examinations, the need for a second metallographic zone in our installation is fully justified.