Experiences from Refurbishment of Metallography Hot Cells & Application of a New Preparation Concept for Materialography Samples

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Abstract

After more than 30 years of operation the lead shielded metallography hot cells needed a basic renewal and modernisation not least of the specimen preparation equipment. Preparation in hot cells of radioactive samples for metallography and ceramography is challenging and time consuming. It demands a special design and quality of all in-cell equipment and skill and patience from the operator. Essentials in the preparation process are: simplicity and reliability of the machines, and a good quality, reproducibility and efficiency in performance. Desirable is process automation, flexibility and an "alara" amount of radioactive waste produced per sample prepared. State of the art preparation equipment for materialography seems to meet most of the demands, however, it cannot be used in hot cells without modifications. Therefore, IFE and Struers in Copenhagen modified a standard model of a Struers precision cutting machine and a microprocessor controlled grinding & polishing machine for Hot Cell application. Hot cell utilisation of the microcomputer controlled grinding & polishing machine and the existing automatic dosing equipment made the task of preparing radioactive samples more attractive. The new grinding & polishing system for hot cells provides good sample preparation quality and reproducibility at reduced preparation time and reduced amount of contaminated waste produced per sample prepared. The sample materials examined were irradiated cladding materials and fuels.

Keywords: metallography, ceramography, materialography, grinding, polishing, cutting, Zircaloy, uranium dioxide, cell windows

1. Introduction

Norway has one Hot Laboratory for doing remote handling and examination of irradiated nuclear materials and fuels by means of non-destructive and destructive examination methods. Metallography and ceramography are important examinations. The IFE hot laboratory near Oslo with its well equipped concrete and lead shielded laboratory cells is operative and is supporting the international OECD Halden Reactor Project and its presently 20 participating member countries since more than 30 years in topics concerning lifetime and safety of nuclear fuel and structural materials in nuclear installations. However, after over 30 years of continuous operation the lead shielded metallography hot cells needed renewal, not least of the original PMMA (polymethylmetacrylate) containment box windows, up-dating of machines for cutting and grinding & polishing, and up-dating of the preparation techniques in direction higher efficiency and less waste produced per sample prepared.

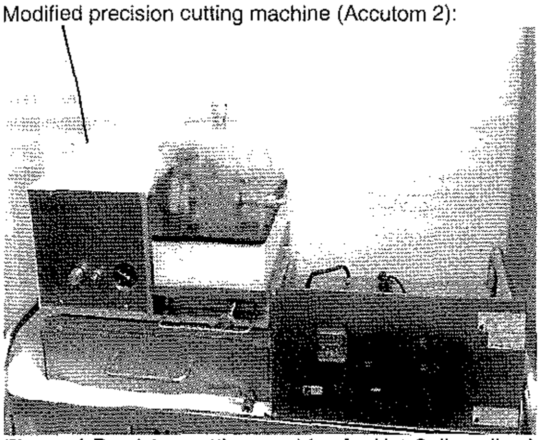
2. Experimental

Remote handling of the specimens and preparation equipment in hot cells makes the preparation work of metallography and ceramography samples challenging and costly. It demands a high degree of skill and patience from the operator and a special design and quality of all in-cell equipment used. Simplicity and reliability of all equipment, good quality and reproducibility in its performance and efficiency are essentials in the preparation process. State of the art preparation equipment for materialography seems to meet most of the demands, however, it cannot be used in Hot cells and be remotely handled without modifications. Refurbishment of existing cells and implementation of new equipment is time consuming. The schedule for refurbishment of the cells was as follows:

- Planning and preparation of cell refurbishment (January 00 – October 00)
- Design & construction of new remotely handled preparation equipment in cooperation with the producer (Struers, Copenhagen) (January00 – September00)
- Functionality test of the equipments at IFE (October 00-February01)
- Cleaning & decontamination of three cells (each 1 m^3) and old equipment,
    - emptying & dismantling of cells,
    - coating of cell interior surfaces,
    - installation of infra structure improvements and new equipment in cells,
    - installation of new cell windows,
    - lead shielding of cells and testing of cells (Nov00-March01)
- Inactive testing of cells and equipment (April 01)

- Cell and equipment in active operation (since May01)With the goal in mind to make the sample preparation process in hot cells not only more efficient and reproducible but also more efficient with respect to a reduction in the amount of radioactive waste produced per sample prepared, IFE and Struers modified a standard model of a precision cutting machine (Accutom 2) and a standard model of a microprocessor controlled grinding & polishing machine (RotoCom with RotoPol-15 & RotoForce 1 specimen mover) for remote handled application in a hot cell. Modifications done to the cutting machine are specified in Figure 1.

Modified precision cutting machine (Accutom 2):



Modifications:

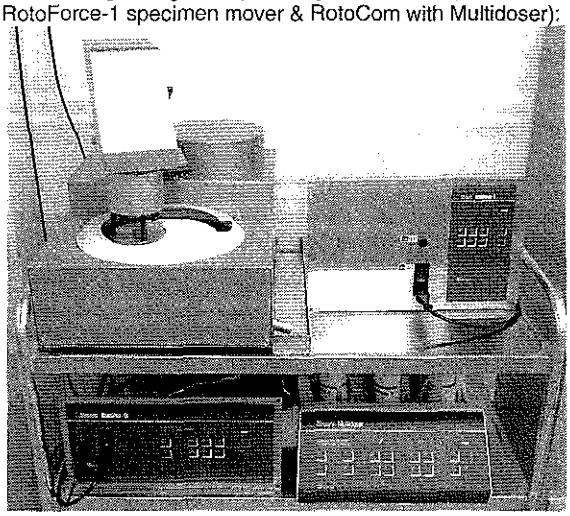
- Size reduction (40cm x 35cm x35 cm)
- Screw heads were modified to be operated with manipulator tongs
- The control unit with all electronics was separated from the machine to be installed on the outside of the cell
- Modification of parts to resist the radioactive environment and to facilitate cleaning and decontamination
- Installation on the water basin of a tap
- A filter system for recycling cutting fluid

Figure 1 Precision cutting machine for Hot Cell application

The grinding & polishing machine (RotoPol-15 & RotoForce 1 specimen mover) supports a state of the art grinding and polishing system using magnetic discs (MD-System). Design modifications necessary to the machine are specified in Fig. 2. The grinding & polishing system includes a memory and out of cell multidoser and control unit (RotoCom) to control every single parameter in each step. Parameters such as preparation time, force, rotation (rpm), type of abrasive suspension, lubricant, dosing level and preparation disc used can be saved, recalled and run again at any time.

After the equipment arrived at IFE, first functionality test of the equipments and training of operational routines and new preparation routines were done outside the cells. Before the equipment was installed in the hot cells the three cells (each 1 m^3) and the old equipment were cleaned and decontaminated. The cells were made empty and the lead shielding was dismantled. Then, the cells interior

Modified grinding & polishing machine (RotoPol-15 & RotoForce-1 specimen mover & RotoCom with Multidoser):

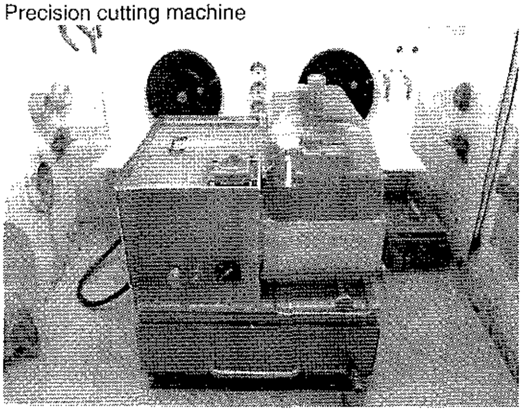


Modifications:

- Size reduction of RotoPol-15 & RotoForce-1 to 50cm x 41cm x 45cm
- Adapting of parts to allow remote handling
- Modification of parts to resist the radioactive environment and to facilitate cleaning and decontamination
- The control unit with all electronics was separated from the machine to be installed on the outside of the cell.
- All plastic components were changed to metal

Figure 2 Grinding & polishing machine supporting a MD-system for Hot Cell application

Precision cutting machine



Grinding & polishing machine

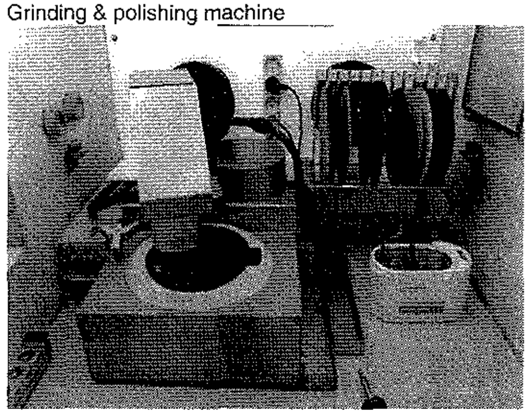
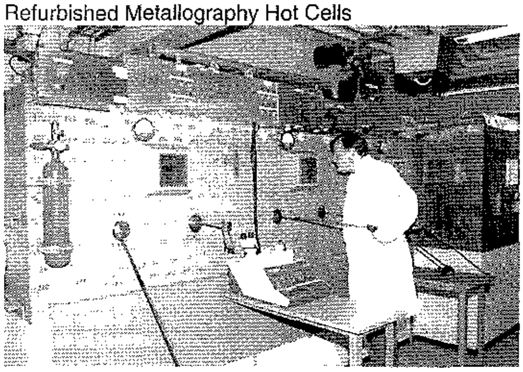


Figure 3 Precision cutting machine and grinding & polishing machine installed in Metallography Hot Cells

Refurbished Metallography Hot Cells



RotoCom + Multidoser

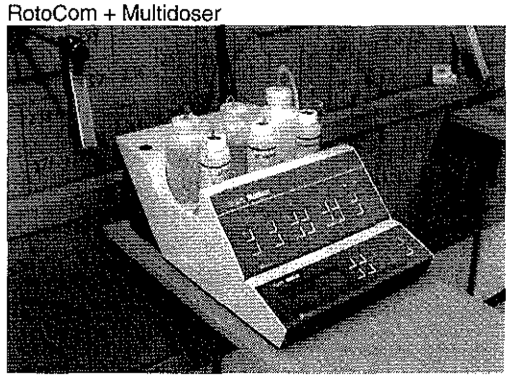


Figure 4 Refurbished Metallography Hot Cells with new preparation equipment in operation (RotoCom + Multidoser)

walls were re-coated with epoxy paint before infra structure improvements and the new equipment was installed in the cells (see Figures 3) and the new cell windows put into place. Following the ASTM C 852-93, June 1993 recommendation the PMMA (polymethylmetacrylate) windows were substituted with PC (polycarbonate) windows smaller in size. Subsequently, the cells were lead shielded again and the functionality tested prior to inactive testing of the new equipment in the cells, see Figure 4.

3. Result

Test runs were done to check on the ***functionality of the equipment*** in the hot cell and to establish ***operational routines*** for remote handled processes such as

- Sample mounting and changing
- Operating of the machines

- Changing of consumable material between working steps such as cut-off wheel or grinding and polishing discs
- Handling of cut-off wheels and grinding and polishing discs
- Cleaning of disc between working steps
- Cleaning of samples and holder between working steps
- Treatment of contaminated liquids from grinding and polishing
- Cleaning and decontamination of the machines
- Service/repair of the machines
- Dismantling of the machine

Cutting of various types of irradiated materials with the modified precision cutting machine proved that all operations could be satisfactorily operated in the hot cell with manipulator tongs. Planar parallel slices from Zircaloy, steels and ceramic can be cut with the machine as thin as 100 µm with good precision. The test runs with the modified grinding and polishing machine designed for manipulator operation showed that the operations can be done remotely handled and safely in the hot cell by using manipulators.

To establish new ***preparation routines*** for metallography and ceramography of irradiated material test runs were done. The sample materials prepared with the new hot cell equipment were in epoxy resin (Specifix) mounted irradiated Zircaloy tubes and sintered uranium-dioxide in Zircaloy tubes. As result of this study a preparation plan for the material combinations with the MD-System was put up and is presented in Table 1. The preparation plan gives the preparation parameters used in each working step, namely time, force, rotation (rpm) and the consumable materials used, such as type of abrasive suspension, lubricant and preparation disc. To allow a direct comparison of the working steps used in MD-System technique and in two more traditional techniques the preparation plan shows the necessary consumables and time consumption for all three techniques.

Table 1 Preparation of UO_2 and Zr

Traditional technique I:

Grinding disc	Time	rpm	Force [N]	DP-Paste	Lubricant	Comments
SiC paper #320	[min]		2			
SiC paper #600	30	200	2	-	Water	1 paper for 2-3 samples
Polishing cloth	30	200		-	Water	1 paper for 2-3 samples
DP-Dur						
DP-Mol			2			
DP-Nap	60	120	2	3 µm	Blue	Added some paste after 30 min
	30	120	2	1 µm	Blue	
	5	120		¼ µm	Blue	

Traditional technique II:

Grinding disc	Time	rpm	Force [N]	DP-Paste	Lubricant	Comments
	[min]		15			
Diamond Pad 20µm	5	220		-	Water	
			20			
Polishing cloth	25	150	20	9 µm	Blue	Added some paste after 30 min
DP-Plan	20	150	15	6 µm	Blue	
DP-Pan	15	150	10	3 µm	Blue	
DP-Dur	5	150		1 µm	Blue	
DP-Nap						

New MD-System technique:

Grinding disc	Time [min]	rpm	Force [N]	DP-Susp. P	Lubricant	Comments
Diamond Pad 20 µm	2	300	20	-	Water	
MD-Allegro	3	150	20	9 µm	Blue	
MD-Largo	10	150	35	6 µm	Blue	
Polishing cloth						
MD-Dac	6	150	30	3 µm	Blue	
MD-Chem	2	150	15	OP-S	-	

For the material combinations examined, namely epoxy resin/zirconium alloy and epoxy resin/zirconium alloy/UO_2 tests were done to establish the ***quality of samples*** with respect to polishing quality and surface smoothness.

In comparison to fresh Zircaloy cladding the irradiated Zircaloy cladding of fuel rods shows surface oxidation and bulk hydriding with formation of (ZrH_4) hydride-platelets. Hydriding leads to hardening. The challenge with such a material is to obtain sufficiently high grinding and polishing rates and maintain a low surface deformation.

Sintered UO_2 ceramic changes its microstructure depending on burn-up. Depending on the burn-up fission products form in the UO_2. Fission products are responsible for the formation of pores within grains and on grain-boundaries. In high burn-up UO_2 the break out of grains during preparation is a problem. Impregnation of the specimen surface with Specifix between grinding steps proves beneficial to reduce or avoid breakouts from the microstructure.

The grinding & polishing machine is designed for one to three samples to be prepared simultaneously. A ***reproducibility*** study of the sample preparation quality was done by checking on preparation induced artefacts such as unevenly prepared sample surface, scratches, break outs, rounded edges at material interfaces, and deformation in the materials surface. First, the same sample was prepared several times by using the same preparation parameters, then one, two and three similar samples were prepared in one run. The test showed a good reproducibility of the results. Critical parameters seem to be the quality of the individual ultrasonic cleaning of the specimen surfaces between the preparation steps and the cleaning of the sample holder.

4. Discussion

The grinding & polishing machine supporting a MD-preparation concept needs more space in the hot cell, in return the machine is stronger and works precise and makes it possible that up to three samples can be prepared simultaneously with controlled preparation parameters, such as rotation speed, sense of rotation of sample holder and grinding/polishing disc, pressure of sample versus disc. The MD-System applies different consumable materials than the traditional preparation technique. In traditional preparation SiC grinding paper glued onto plastic or metal holder plates (discs) is used, while diamond particle suspension on more permanent steel and plastic discs with a magnetic attachment to the holder are used in the MD-technique, resulting in an improved sample quality in terms of an improved sharpness of edges between different materials, and not at least reduction of preparation time. Grinding of irradiated UO_2 ceramic with SiC paper in hot cells requires that the paper is glued on holders. For each renewal of a self-adhesive grinding paper or self-adhesive polishing cloth the holder has to be disposed. Since the magnetic discs for grinding and polishing are not only efficient but also long lasting with the application of the MD-System a significant ***reduction of contaminated waste*** is achieved as shown in Table 2.

Table 2 Volume of waste per sample from preparation disc

Volume of waste per sample from preparation discs	Grinding	Polishing	Equipment
200 cm^3 Traditional Technique I	Grinding on self-adhesive SiC-paper glued on plastic or metal discs	Polishing on self-adhesive polishing cloths glued on plastic discs	Old equipment Preparation of one sample
100 cm^3 Traditional Technique II	Grinding on Diamond Pad disc glued on plastic or metal discs	Polishing on self-adhesive polishing cloths glued on plastic discs	Old equipment Preparation of one sample
50 cm^3 New Technique using Magnetic Disc-System	Grinding on MD-Diamond Pad disc	Polishing using the MD-system	New equipment Preparation of three samples simultaneously.

5. Conclusion

Refurbishment of the lead shielded metallography hot cells and upgraded with new preparation equipment involved a stop in operation of 6 months. The refurbished metallography cells are in operation again since 6 months. In the mean time more than 30 precision cuts of both fuel rod and steel materials were performed, a full preparation programme for metallography and ceramography examination of some 20 different fuel rod samples was completed successfully, and the machines were cleaned. The experience showed that the new strong cutting machine supports a fast cutting operation at high precision (planar cuts). The powerful grinding and polishing machine supporting a magnetic disc system (MD-system) modernised the preparation technique. This resulted in a good and reproducible sample preparation quality, significantly reduced preparation time and reduced amount of contaminated waste produced per sample prepared. Further, the memory and control unit of the system made the preparation process more attractive, reproducible and less operator dependent.