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RECENT DEVELOPMENTS IN ELECTRON-OPTICAL TECHNIQUES FOR STUDYING IRRADIATED FUEL AND COMPONENTS IN THE UK

by

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RECENT DEVELOPMENTS IN ELECTRON-OPTICAL TECHNIQUES FOR STUDYING IRRADIATED FUEL AND COMPONENTS IN THE U.K.

**PART 1:** Complementary techniques for studying irradiated UO₂, using miniature radial sections of fuel pellets, used at Windscale Nuclear Laboratories, UKAEA. By J.H. Pearce, R. Sumerling (WNL).

**PART 2:** New facilities at Harwell for the preparation and examination by SEM and TEM of small samples of highly irradiated material. By J.E. Bainbridge and G.J. Small (Harwell, UKAEA).

**PART 3:** Proposed new shielded facility for an analytical scanning electron microscope at Berkeley Nuclear Laboratories (CEGB). By A. Jones and G.K. Rickards (BNL).

**ABSTRACT**

The small radial sections of irradiated fuel pellets which are prepared for electron probe microanalysis (EPMA) to determine the distribution of Pu and fission products, are being studied by a variety of techniques at WNL in order to obtain the maximum useful information from one specimen. These techniques include scanning electron microscopy, microfractography and X-ray diffraction measurements of lattice parameter across the fuel radius.

A gamma spectrometer has now been fitted to one of the five spectrometer ports on the EPMA (a JEOL 733) so that a radial isotopic gamma scan can be used to complement the fission product analysis; initial results on a sample of highly-irradiated UO₂ fuel are reported.

Facilities have recently been installed at Harwell for the preparation and examination, by SEM and TEM, of small samples of highly irradiated materials. The facilities are located in adjacent rooms with the preparation equipment in one section of a large Active Handling Laboratory and the microscopes in a separate 'clean' area. The Active Handling Laboratory houses a shielded cell for the preparation of thin foils and replicas, a reception cell for cleaning, mounting and coating SEM specimens and a transfer box linked to the SEM. Specimens are posted from this box into the SEM via a tube passing through the wall separating the two laboratories. A similar arrangement is planned for posting specimens to the TEM.

It is planned to install at BNL a fully shielded scanning electron microscope, incorporating X-ray spectrometry for elemental analysis. Operating experience at BNL with similar, but older, machines is summarised and used to draw up a list of criteria which the proposed instrument must satisfy, covering normal operation, maintenance and repair. The layout of an overall facility (including cells for sample preparation) to meet these requirements is described.
PART I: COMPLEMENTARY TECHNIQUES FOR STUDYING IRRADIATED UO,
USING MINIATURE RADIAL SECTIONS OF FUEL PELLETS.
by J H Pearce, R Sumerling (WNL)

1.1 INTRODUCTION
A paper\(^{(1)}\) presented last year to the Hot Laboratories meeting described the methods which are used at WNL to prepare miniature radial fuel samples from irradiated fuel rods, for electron probe microanalysis of plutonium and fission products. The samples include the cladding and are typically 5 to 7mm long and 0.6mm square, held in a conducting plastic mount. These specimens are metallographically prepared using non-aqueous solvents to avoid loss of caesium. The activity of each sample is usually low enough (<10 mSv y\(^{-1}\) at 10cm) to be examined in both the EPMA and X-ray diffraction (XRD) equipment using only local shielding. When such a specimen is prepared, it is advantageous to examine it by a number of techniques, in order to obtain the maximum useful information from the sample with little extra effort.

1.2 TECHNIQUES USED
The radial distribution of plutonium and fission products in the fuel are determined using an EPMA (a JEOL 733). The element concentration profiles and maps of selected areas are then correlated with the pore structure in the fuel, using the EPMA in the scanning electron mode. However, the activity of the sample prevents the ultimate resolution of the instrument being achieved, so that pores less than 50nm cannot be resolved by this technique. Lattice parameter measurements are made on the fuel using an X-ray beam about 1.0mm wide at various positions along the fuel radius. A Siemens D500 diffractometer is used with fuel sample radius at 90\(^{\circ}\) to the collimated X-ray beam. A removable lead glass block (30 x 25 x 5cm) thick is used in front of the specimen within the X-ray cabinet to shield the operator from \(\beta\gamma\)-radiation. Exposure times of 24h are used at each location.

When microanalysis is complete, further information on the UO\(_2\) microstructure is being obtained by a new technique\(^{(2)}\) which WNL have termed 'scratch microfractography'; it reveals the fracture mode of the brittle material and allows use of a transmission electron microscope (TEM) to obtain higher resolution images of the fuel structures.

\(\times\) Scratch microfractography involves scratching the polished UO\(_2\) surface at several radial positions, using a diamond knife, that operation being carried out in a fume hood, with the specimen surface flooded with methanol to reduce the spread of contamination. Cellulose acetate film softened in acetone is then used to prepare successive replicas of the surface. The first replica removes much of the fracture debris and is regarded as a cleaning replica; it emits typically ~20 \(\mu\)Sv y\(^{-1}\) and ~200 \(\mu\)Sv \(\beta\gamma\)/h at 10cm. The second and third replicas are shadowed with Pt-C before carbon coating to obtain TEM images of the fractured surface.

It has recently been possible to obtain gamma spectra from the same radial fuel section by fitting a heavy metal collimator, having a slit width of 0.25mm, into one of the spectrometer ports in the EPMA (Fig 1.1), with an external gamma
detector. The sample is positioned normal to the collimating slit so that automated radial traverses can be made across the fuel pellet. The γ-signal is processed and the stage controlled by the analyser system (Link AN 10000) fitted to the EPMA. The electron-excited X-ray data and the γ spectrum can be displayed and the data stored on disc. Ultimately it should be possible to obtain EPMA and micro-gamma scan results simultaneously.

1.3 APPLICATION OF TECHNIQUES TO HIGHLY IRRADIATED FUEL

These techniques have been used to characterise a sample of UO₂ fuel irradiated to high burn-up (36.5 GWd/tU) in the SGRWR.

An automated gamma scan for Cs134 and Cs137 along the radius of this sample is shown in Fig 1.2. In this case 31 analysis points each with a dwell time of 1000 seconds were made at intervals of 257 μm across the pellet radius. The Cs134 profile shows a displacement towards the cooler rim compared to Cs137 ie the Cs134 apparently shows greater mobility in moving from the hotter to cooler positions in the fuel. However, this effect could be due to the decrease in thermal neutron flux from fuel surface to centre, which determines the rate of Cs134 production from stable Cs133 by an n, γ reaction. If the rates of Xe and Cs are similar in the fuel then the longer half-life of the Xe precursors in Cs133 compared to Cs137 (5.3d compared with 3.3m) would have little effect on the distribution of the Cs isotopes.

These curves can be compared with those of Cs and Xe obtained by electron-excited wavelength-dispersive X-ray analysis in the EPMA (Figs 1.3,1.4). The gamma-data for Cs has less scatter due to superior peak-to-background signal levels and distinguishes between different isotopes. The EPMA results, however, give much better spatial resolution (down to 1μm) eg in detecting the high local concentration of Cs at the pellet rim.

Scanning electron microscopy (using back-scattered electrons) in the EPMA had shown sintering of micropores near the fuel rim and equixed growth (up to 20μm) towards the fuel centre, together with the development of coarse intergranular porosity associated with gas release. At about 0.6 of the pellet radius, intragranular pores became visible and coarsened towards the fuel centre. After making the transverse scratches and removing of the debris, it was seen that the scratches made within 0.7mm of the rim showed mainly intergranular fracture, whereas the other scratches made towards the fuel centre revealed mainly transgranular fracture. Near the fuel centre voids 100-200nm in size could be seen in the cleavage faces (Fig 1.4).

The previous EPMA work had shown that Cs was concentrated at the fuel surface and the grain boundaries within 0.2mm of the rim of this specimen. This is thought to explain the intergranular weakness revealed by microfractography: it seems likely that a grain boundary phase containing Cs is present - an important deduction as it helps to throw fresh light on the state of the fuel near to the clad/fuel interface. The lattice parameter measurements showed a larger UO₂ cell size near the rim compared with the mid-radius and fuel centre (547.4pm compared with 547.2pm), possibly due to the increased Cs content or a decrease in the O:U ratio.
Cleavage fragments extracted from near the fuel rim showed numerous bubbles 2 to 8nm in diameter, usually associated with finer particles. Towards the fuel centre there were fewer gas bubbles, covering a larger size range; the largest (10-20nm) tended to be crystallographic and were often associated with larger precipitates. The smaller gas bubbles present (3-5nm) were frequently associated with dislocations, not with fine particles (Fig 1.5).

These initial results show that additional information to improve the understanding of fuel behaviour can be obtained from small radial samples of fuel by employing several complementary techniques, involving little extra effort. The techniques are now standard in the electron-optical examination laboratory at WNL.

PART 2: NEW FACILITIES AT HARWELL FOR THE PREPARATION AND EXAMINATION BY SEM AND TEM OF SMALL SAMPLES OF HIGHLY IRRADIATED MATERIAL

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2.1 INTRODUCTION

Facilities have recently been installed at Harwell for the preparation and examination, by SEM and TEM, of small samples of highly irradiated materials. The facilities are located in adjacent rooms with the preparation equipment in one section of a large Active Handling Laboratory and the microscopes in a separate 'clean' area. Specimens are transferred from the Active Handling Laboratory into the SEM via a tube passing through the wall separating the two laboratories. A similar arrangement is planned for posting specimens to the TEM.

The microscopes are operated as a service for examination of both active and non-active specimens arising from nuclear and non-nuclear research programmes. However the main requirement for shielded transfer facilities is for a detailed study of the release of fission gases from highly irradiated UO₂ under simulated reactor fault conditions. Equipment used for these studies is also located in the Active Small Sample Laboratory which is described in the next section.

2.2 ACTIVE SMALL SAMPLE LABORATORY

An overall view of the laboratory is shown in Fig 2.1. To the left, inbetween the two fume cupboards, stand a rapid annealing furnace and a gas analysis panel used for the transient fission gas release programme. Small samples (~50mg) of high burn-up (2-4%) UO₂ are heated in the furnace to <2500°C at ramp rates of <50°C s⁻¹. Fission gases released from the fuel are swept from the furnace to the gas panel by a continuous flow of helium. The activity of the gas as it is released is measured by a β-counter.

After annealing, the UO₂ samples are polished to optical flatness and placed in the SEM reception cell, which consists of a steel box surrounded by 50 mm lead
shielding, which is sufficient to reduce the radiation level per specimen from $1 \text{R} \ (0.01 \text{ Sv}) \ hr^{-1} (\beta)$ at 30 cm to $-100\mu\text{R} \ (1\mu \text{ Sv}) \ hr^{-1} (\gamma)$ at the cell wall. In this cell the specimens are decontaminated and mounted on specimen stubs in preparation for examination on the SEM.

Following SEM examination, the specimens are sent to another cell, in the centre of the laboratory, for replication of the polished surfaces and electro-chemical thinning for examination in the TEM. This cell also comprises a steel a box shielded with 50mm lead. Tongs are used for handling specimens. The specimens can be placed within secondary shielding in the cell, allowing the use of glove ports to manipulate equipment within the cell. Since acetone is used in the replication technique, the cell atmosphere is purged with nitrogen and monitored continuously to ensure that the oxygen content is kept below 0.05%, thus avoiding any risk of explosion. Other items in the Small Sample Laboratory include the SEM transfer box, a vacuum deposition unit for coating the replicas and a second gas loop, in which the retained gas content of the specimens is analysed by crushing and dissolving. These items are all against the wall to the rear of Fig 2.1.

2.3 SEM FACILITIES

The first instrument used for examining radioactive materials, a Cambridge Instruments SEM 2A, was installed in 1968. This was later fitted with a hot stage and variable gas environment in which dynamic carburisation/oxidation studies were carried out. Initially no permanent radiobiological shielding was fitted - protection was afforded by light shielding and speed of transfer of the specimens into the chamber. All specimens were coated with hydrocarbon aerosol and the microscope was operated on this basis for five years, without mishap from either radiation or contamination. However in 1973 it was recommended that permanent shielding be fitted. Figure 2.2 shows the size and volume of this containment. The lead cell was manufactured from 50mm lead bricks; it was cumbersome, heavy, and difficult to seal to the SEM whilst maintaining the 'floating column' stage necessary for the operation of the microscope. The biggest problem was that it drastically slowed down the loading procedure, from 1-2 seconds to several minutes, and introduced unnecessary handling problems.

In 1979 a Phillips PSEM 500X was installed for the examination of both active and non-active materials. This instrument is equipped with Energy Dispersive and Crystal Wavelength Spectrometers (Link Systems Limited, and Microspec (USA) respectively) together with backscattered specimen current and cathodoluminescence detectors. It was decided to return to the original method of specimen preparation and loading since a consistent handling time of 2 seconds from container to stage could be maintained. As previously, no problems were encountered.

In 1983, with the proposed introduction of more stringent Health Physics requirements, the technique was reviewed and a plan devised by which the advantages of rapid transfer could be incorporated into a shielded facility. It was vital to maintain the flexibility of active/non-active specimen examination with minimal delay in changing from one type of work to the other. The resultant design is illustrated in Figs 2.3, 2.4.
The emphasis is on the provision of an α-tight system providing containment with a degree of β shielding. This, combined with strict control of the activity levels allowed in the instrument, provides protection against both contamination and radiation.

The maximum specimen activity that can be handled is 1R (0.01 Sv) hr\(^{-1}\) (β) at 30 cm. For high burn-up UO\(_2\) this means a specimen volume of <5mm\(^3\). Few problems have been encountered in removing, by trepanning, specimens of this size from fuel pellets. For the gas release experiments specimens of 3 mm diameter are used, thinned to 0.5 - 1.0mm to reduce the radiation to an acceptable level. This is necessary both to protect the operator and to maintain the efficiency of the SEM. High levels of β radiation affect both the picture quality and the accuracy of the analysis, particularly that obtained by energy dispersive spectroscopy. The wave-length (crystal) spectrometers can be shielded and the signal collimated. Heavy alloy (tungsten or depleted uranium) is used to shield the spectrometers but an increase in thickness gives a sharp rise in cost. To give maximum visibility and ease of handling the boxes through which the specimens pass have been made from lead-loaded perspex (Trade Name Premac). One box surrounds the airlock entry port of the PSEM and incorporates a smaller box through which unirradiated specimens can be introduced into the system, via a door activated by an α monitor. No entry is possible if levels in excess of 5cps α maximum are detected in the larger box. If this is the case a radiation lock is activated.

A stainless steel tube plus transfer unit connects the two Premac cells either side of the wall(Figs 2.3, 2.4). Specimen movement is carried out by a motorised endless chain drive and the whole system is purged and maintained at a slightly negative pressure of high purity nitrogen.

The original aim was to use the energy-dispersive spectrometer (EDS) for non-active specimens and the wavelength spectrometer (WDS) for active specimens and elements of low atomic number (z < 11). Both detectors have the same take-off angle (beam to specimen surface) and both are used with the specimen at the eucentric height position of the stage. In practice the EDS has been used for the majority of specimens, active and non-active, since the high count-rate acceptance detector can handle specimens with the preset activity levels.

The WDS unit is slower to operate but has the advantage of detecting small concentrations, ie 5 ppm as compared with >500 ppm for the EDS. An updated Microspec is to be fitted in early 1986; this will analyse as quickly as the EDS and both units, together with the specimen stage, will be controlled by the Link Systems computer. The computer will carry out the ZAF correction programme on all data irrespective of the spectrometer used. In this manner comprehensive analysis should be possible on all specimens.

The PSEM 500X is interfaced with a Kontron 1 megabyte Ibas I and II image analysing system. Images can be transferred from the PSEM 500X to the Ibas either for immediate analysis or storage on 8 inch (203mm) dia floppy discs. At present the Ibas can control the stage of the microscope but with the fitting of the new Microspec unit, control of the complete complex will be via the Link.
Systems computer. Image analysis of the X-ray generated maps and secondary electron images will be done in parallel to evaluate the interaction, if any, of topographical and chemical data.

2.4 TEM FACILITIES

In 1984 a Phillips 301 transmission electron microscope was installed as part of the electron beam instrument examination facility for radioactive materials. A similar transfer system has been designed for this microscope but although the Premac reception box has been delivered, the transfer tube has yet to be installed. There is no box adjacent to the microscope but the transfer tube is attached to the specimen holder entry port by a light-weight bellows unit. A glove port is incorporated into the transfer tube to allow the operator to load the specimen holder in the standard manner.

The specimen is loaded into a Phillips holder in the thinning cell situated in the active laboratory. The holder plus specimen are transferred to the Premac cell, loaded into the carriage in the transfer tube and carried to a position opposite the glove entry port. The operator lifts the holder from the carriage and loads it into the microscope.

As with the SEM system the complete circuit is maintained at a slightly negative pressure of high purity nitrogen. The nitrogen enters the system adjacent to the microscopes and flows to an extract in the preparation cell, this helps to ensure that any airborne particulates are moved away from the microscopes towards the reception cells, which are in the active handling laboratory.

Provision has been made, should the need arise, for the transfer tunnels to be shielded in the event of a specimen transfer breakdown. In the event of an operator problem, sudden illness, etc. the specimen and its holder are returned automatically to the safety of the reception cell in the active laboratory.

PART 3: PROPOSED NEW SHIELDING FACILITY FOR AN ANALYTICAL SCANNING ELECTRON MICROSCOPE AT BERKELEY NUCLEAR LABORATORIES (CEGB).

By A Jones and G K Rickards (BNL)

3.1 PREVIOUS EXPERIENCE WITH AN EPMA AND AN SEM INSTALLED IN HOT CELLS

Two electron beam instruments are currently installed at BNL for the examination of active material. The oldest of these is a Cambridge Microscan 5, originally purchased in 1969. It is an electron micro-probe optimised for elemental analysis (using wavelength dispersive X-ray spectrometers) rather than its image performance. This instrument is shielded to some extent but is not fully contained and specimens have to be loaded manually with some risk of radiation dose to the operator. Although its X-ray performance is still adequate, even with active samples, the shielding is no longer considered adequate for the more active samples now available and the lack of containment causes problems with airborne activity. The age of the instrument and its low image resolution determine that it should now be replaced.
The second instrument is a fully contained and shielded ISI super IIIA bench top SEM which is now about 5 years old. It is fitted with a fixed position ORTEC energy-dispersive X-ray detector and analyser. The instrument was purchased because it would just fit into a vacant cell of limited size in the Shielded Area. After a protracted period of installation in which many problems had to be overcome in order to fit the instrument into the cell, it is now in operation, but without, as yet, satisfactory X-ray analyser performance on active samples.

Although both of these instruments are proving valuable in obtaining visual information in a variety of studies, neither of them can be regarded as adequate for present or future needs. It would be better to have a single, fully-shielded, instrument combining the SEM's high image resolution, depth of focus and imaging capability with the X-ray analysis facility offered by the probe.

3.2 PRINCIPLES ADOPTED IN DESIGNING THE NEW FACILITY

Based mainly on the experience with installing the ISI SEM into a hot cell, the following principles are being followed in the new SEM installation:

a. The cell size must be sufficient to accommodate the instrument as-received.

b. As far as possible, a standard instrument should be used to ensure ease of installation, servicing and repair. Any modifications must be carried out by the manufacturer prior to delivery.

c. Maintenance requirements must be considered at the design stage. Easy access for servicing is essential.

d. The design of the installation should minimise the effect of vibration.

e. The column of the instrument should be sited away from the shielding to avoid problems with stray a.c. magnetic fields.

f. Both wavelength and energy-dispersive X-ray spectrometers are desirable and their specifications are considered in more detail in the following section. It should be noted that a reliable and convenient supply of liquid nitrogen is required for the energy-dispersive system.

g. For the X-ray analysis system to be fully quantitative, wavelength dispersive spectrometers must be used and, if simultaneous analyses of light and heavier elements are required, then two units would normally be preferable. But, because imaging is to be the main purpose of the new instrument, it is intended to fit only one spectrometer to it. Since a remote instrument must be as simple as possible, a horizontal and inclined spectrometer is preferable, because this type is more tolerant of specimen height and so does not need optical back-up. This choice also enables samples having rough or semi-rough surfaces to be handled more easily. It should be noted that this spectrometer must incorporate four analysing crystals to allow analysis of the complete element spectrum.
h. Assuming that an energy-dispersive system will be fitted in addition to the
wavelength type it is imperative that a detector proven in high radiation
fields is used. The ORTEC detector fitted to the ISI shielded SEM has not
proved satisfactory, whereas experience with KEVEX detectors has shown that
they will function with moderately active specimens. In addition to this,
a retractable type of detector must be chosen as this permits optimum
positioning of the detector and also allows different types of shielding
collimators to be used.

3.3 DESCRIPTION OF THE PROPOSED INSTALLATION OF AN SEM IN A HOT CELL FACILITY

An SEM must be supported by a number of ancillary operations (eg sample
preparation) and is, therefore, only part of the overall facility. It is
efficient to carry out the supporting operations in inter-connected cells, as
shown in figure 3.1, with the SEM at one end of the line.

In figure 3.1 it can be seen that the largest of the ancillary cells is cell 2.
It will house various items of equipment for sample preparation, such as
cutting, grinding and polishing, together with an ultrasonic cleaning bath which
would be used prior to transfer of samples to cell 3. This cell will contain a
coating unit and a second ultrasonic bath for use before passing the samples on
to the SEM itself. The third cell is therefore acting as a buffer between the
relatively dirty operations of sample preparation and the SEM cell, which must be
kept as clean as possible to facilitate maintenance. Cell 2 would have
provision in its rear door for posting to and from sample transfer flasks. In
addition, cell 1 provides the possibility of connecting the facility to the
Shielded Area pneumatic sample transfer system.

All of these cells will be constructed from steel plates of 300mm total
thickness and, apart from cell 1, each will be fitted with a pair of
manipulators. These must operate through the front wall of the cells because
headroom is limited at the site of installation. In line with current practice,
apparatus in cells 1-3 will be enclosed within containment boxes.

Because of the special requirements of the SEM, the box in cell 4 will be
non-standard. Figures 3.1 and 3.2 show that only the stage door of the SEM will
be exposed to the interior of the containment box in cell 4. Samples from the
preparation cells will be passed into the containment box and then loaded into
the SEM. This feature is designed to keep the surface of the instrument as
clean as possible. The SEM will stand on a concrete block which is isolated
from the rest of the floor and the coupling between the SEM and the containment
box will not be rigid. The objective is to eliminate vibration; other steps in
this process include isolation of the containment box by mounting it on
antivibration pads and also by paying special attention to its service
penetrations, manipulator gaiters and sample transfer port. The microscope's
inherent antivibration mountings will also help, consequently structural
vibrations are expected to have minimal effect on image quality.

One mishap that could occur during normal operation is that a sample could fall
from the stage. Various design features will be included to minimise the
possibility of it falling into the position that is beyond the reach of the
manipulators. For example, a wire catch-pot will be positioned at the bottom of
the chamber to prevent loss of a sample into the vacuum line. The chances of a
sample falling, however, depend on the method of sample loading. The swinging
door type of stage is preferred (rather than an air-lock) as there is less
chance of dropping a sample into an inaccessible position.

The need for access to the chamber and the range of operations to be carried
out, including the maintenance requirements, imply that this cell must be fitted
with two manipulators rather than a tong.

All stage motions, together with as many other functions as possible will be
motorised for operation from a remote control desk situated in an area adjacent
to the cell, alongside the normal microscope controls.

Access to the microscope for maintenance will be achieved by removing various
parts of the shielding, as shown in figure 3.1, to leave as much space as
possible around the machine. The outside of the instrument is expected to be
free of contamination. The internal parts of the vacuum system will, however,
be contaminated to some extent and so a special fitting will be provided on the
side of the microscope column, via which a temporary connection to the Cell
Extract System can be made. Thus, when it is necessary to break into the vacuum
system, the airflow will be into the instrument and away from the maintenance
personnel.

Since the stage and its associated motors protrude into the containment box,
maintenance of these items will be undertaken remotely. To this end as many
components as possible will be replaceable by manipulator, although a set of
glove-ports will be provided in the side of the box (point G in figure 3.1) for
tasks where this is not feasible. A special trolley will be provided in the box
to enable the weight of the open door to be supported. The door hinge will be
designed to be disassembled (probably by manipulator) and the door removed, on
its trolley, to the glove station. If necessary, the whole door can then be
posted out via the large diameter port shown at P in the figure.

The coupling between the microscope and the containment minimises the
transmission of vibration, because it is in the form of a convoluted gaiter.
The gaiter has a second function during maintenance operations where the
instrument has to be separated from the containment, as it will extend (as shown
in figure 3.3) to the point where it can be severed, thereby bagging the
contaminated surfaces of the instrument. After servicing it will be possible to
re-install the microscope, with a new gaiter, whilst maintaining containment at
all stages.

Although the microscope has been segregated from the dirty operations of sample
preparation to facilitate maintenance, it will be necessary for operators to
limit the spread of contamination by, for example, cleaning of manipulator jaws
prior to passing prepared samples along the line.

3.4 CONCLUSIONS

i. Experience at BNL with electron beam devices used on active materials has
enabled a specification to be prepared for an SEM facility which includes
sample preparation and ensures ease of operation, maintenance and repair.
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FIG 1.1: Schematic diagram of micro-gamma scan facility incorporated into an electron probe micro-analyser.
FIG 1.2: Cs137 and Cs134 concentration profiles along the radius of SGHWR fuel irradiated to 36.5 Gwd tU, obtained in the EPMA fitted with an integrated gamma spectrometer. The effective spatial resolution is ~0.5mm, leading to some apparent loss near the edges of the specimen.
FIG 1.3: Concentration profiles for Cs and Xe across sample used in Fig 1.2. Note high concentration of Cs at pellet edge (10 x the mean Cs level)
(a) Part of radial UO₂ strip (6 mm long) showing scratches 1 - 3

(b) SEM views of scratch 1 near edge (left) and scratch 7 at centre (right)

(c) Detail of edge fracture (intergranular) and centre (transgranular)

FIG 1.4: SEM views of diamond scratch marks on irradiated UO₂ sample
FIG 1.5: Plastic-carbon replica of scratched area showing mainly intergranular fracture near fuel pellet edge (left), with cleavage fragment showing bubbles 3-8 nm dia associated with precipitates (right).
FIG 2.1: Overall view of the active small sample laboratory at Harwell, showing furnace in fume cupboard with gas supply panel (A), EM specimen preparation cell (B) and posting facility to SEM (C)
FIG 2.3: Rapid specimen transfer facility for Philips PSEM 500x. SEM reception cell at left. Premac transfer cell (middle) with steel transfer tube to Premac SEM specimen entry cell (right)
FIG 2.4: Premac SEM specimen entry cell (rear view, left and operating face, right) connected via the transfer tube to the active preparation facilities.
FIG 3.1: A plan view of the overall facility, (schematic only), BNL
FIG 3.2: Section through SEM Cell, (schematic only)

FIG 3.3: The SEM during separation from the containment box
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