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NEW CELLS FOR THE PREPARATION OF HIGHLY IRRADIATED SAMPLES FOR ELECTRON MICROSCOPY AND DENSITY MEASUREMENT

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INTRODUCTION

The development of the Fast Breeder Reactor system requires a detailed knowledge of void swelling characteristics in pin cladding and core structural materials. Furthermore, changes in fuel structure and the development of secondary phases have important operational consequences. Much of the required information can only be obtained utilizing specialist examination methods such as transmission electron microscopy, scanning electron microscopy, x-ray microprobe analysis and precision density measurement. All of these methods involve difficult specimen preparation techniques which in the case of nuclear material is complicated by high activity levels and an attendant contamination hazard.

Suitable routes have been evolved at Dounreay over the past years utilizing existing resources and as a result the various preparation stages are scattered throughout several laboratories within the group. This necessitates frequent specimen movement within the area. In general, the preparation techniques have relied on partial shielding to effect some protection to the operators and this together with the need to use a limited amount of hand operation has given rise to some radiation exposure to the laboratory personnel. This has been maintained at an acceptably low level by virtue of the following:-

1. low sample activity arising from long "cooling" times
2. a limited amount of material available for destructive examination
3. use of high grade staff exercising constant vigilance
4. personnel radiation exposure limits set sufficiently high to enable handling to be achieved.

The philosophy adopted at Dounreay for the preparation of such samples is similar to that of other UKAEA establishments and has been presented previously (1).

The future experimental programme associated with PFR calls for the detailed examination of an increased number of samples with shorter periods of cooling than previously given. Consequently, plans are being made to erect a suite of cells specifically designed to prepare specialist samples from highly-irradiated plutonium-contaminated material. This paper presents the background to and the salient features incorporated in the design.

EXPERIMENTAL PROGRAMME

The required programme can be basically considered as two separate parts. Firstly, the study of void production (2) in core structural and cladding materials and secondly, the chemical changes which occur under irradiation in the fuel and fuel/can gap. Void formation in most samples is quantified by the precision measurement of material density (3). Here measurements are made on clean contamination free samples which weigh in the order of 0.3 gm. A selected number of the density samples are then taken a stage further and examined in the transmission electron microscope (TEM). For this examination the samples have to be in the form of thinned 3 mm diameter discs for insertion into the TEM. At this stage the samples are fragile and easily damaged. However, most of the preparation route from bulk irradiated material to final sample is common with that for samples which terminate examination at the density measurement stage.

Chemical changes in the fuel and can gap are investigated by the use of an electron probe microanalyser (EPMA). The sensitivity of the EPMA detectors to radioactivity means that only thin slices of material can be examined. For accurate measurements the surface of this material must be given a diamond polish and preferably an attack polish. Because of the relatively higher specific activity of the fuel samples and the presence of plutonium, samples for this work are prepared by a completely separate route to that for the void study samples.

The fracture faces from pieces of failed fuel pin cladding are also examined in the scanning electron microscope (SEM). These are of necessity relatively large samples with the possibility of adherent fuel. The samples for SEM examination essentially follow the EPMA route.

CURRENT SAMPLE PREPARATION ROUTES

The steps involved in the preparation of samples for void studies have been extensively reviewed in the literature (4) and are only briefly outlined here. They involve:-

1. rig breakdown
2. sectioning of fuel pins or pieces of structural material
3. removal of gross amounts of fuel material
4. decontamination to remove traces of plutonium fuel residue.

If TEM work is required, the following additional steps are necessary:-

5. electrospark trepanning of 3 mm discs
6. hand grinding using a steel block to pre-thin the disc to $\sim$ 0.13 mm
7. electropolishing using a jet technique to perforate the disc.

The preparation route for EPMA samples has also been fully documented (5, 6) and the current practice has not been changed significantly.

During routine ceramographic examination of fuel pin sections in a heavily shielded alpha beta gamma facility, interesting features requiring EPMA examination are identified. A thin slice containing the fuel section is cut from the metallographic sample, using a high speed cutting wheel. The remnants of the original mount are removed and the fuel slice placed in a pre-prepared mount and impregnated with cold-setting resin. This pre-prepared mount contains definable features which permit accurate grinding to known depths. The ground sample is then conveniently polished with diamond abrasives. Following ultrasonic cleaning, the specimen is transferred in an alpha beta gamma transfer flask to a partially shielded glovebox where the outer part of the mount is broken away from the inner ring to give a small diameter mount containing the polished thin slice of fuel pin. This small mount is further cleaned ultrasonically and loaded into the EPMA specimen holder where conducting paint is applied to give electrical contact. The assembly is then ready for transfer to the instrument using a specially designed alpha sealed flask. The use of the sealed loading flask together with the installation of comprehensive local shielding around the EPMA has virtually eliminated contamination and radiation hazards during the examination stage.

The final part of the preparation in the partially shielded glovebox involves specimen handling with Ledex gloves and tongs.

The preparation of SEM samples involves similar handling.

PROPOSED PREPARATION ROUTES

As indicated previously the projected experimental programme for PFR material involves an increase in the annual throughput of specimens with reduced cooling times. The target is to process 300 samples per year for density measurement and 200 for TEM work. In most, but not all cases, one examination would logically follow the other, and this would require an increase by a factor of about 3 in the present rate of processing. It is estimated that the reduced cooling time would enhance the specific activity of the material by a factor of 3.

The existing specimen preparation routes involve a number of facility-to-facility flask movements and cannot be considered as efficient in terms of manpower or in the time required to process a particular specimen. In order to overcome this deficiency and also keep operator exposure to a low level, a new fully shielded suite of cells has been designed for specimen preparation. The facility will contain the preparation route for void study samples from stage 3 onwards and the later stages in the route for EPMA and SEM samples. All existing steps are retained but full operator protection is maintained throughout. Figure 1 shows the flow diagram for material movement through the facility.

All samples enter the facility from remote alpha beta gamma cells by the use of a Padirac flask. The void study samples then progress along the facility, all traces of Pu being removed before they enter cell 4. This involves the interesting principle of one facility embracing both alpha and non-alpha areas. From cell 4 the plutonium free samples can be sent to and from an external density measuring cell by a pneumatic transfer system. The samples continue along the facility, pre-thinned TEM discs passing to a shielded blister cell and bulk reject material to waste. It is intended to electropolish the pre-thinned discs in the blister cell. Handling will be through glovebox gloves using tweezers so that some hand dose will be involved. The shielding will provide adequate body protection. The technology for fully remote electropolishing and handling of perforated TEM discs is not sufficiently developed for its adoption in the current design; however, the design is sufficiently flexible to permit future installation. The prepared fragile discs for examination in the TEM emerge from total enclosure into a fume cupboard via a small diameter La Calhene hatch. Here they are loaded into the TEM sample holder and transferred to the instrument.

The EPMA and SEM samples pass from the reception cell and progress by a route separate to that for the void study samples. All operations previously done by glovebox gloves and partial shielding will be done with remote handling and full shielding. The facility is compatible with the custom-built sealed flask used previously to transfer samples to the EPMA and this method will be retained for the final transfer.

NEW CELL DESIGN

A plan view of the proposed facility is shown in Figure 2. This clearly shows the individual cells, their purpose and the numbering given to each. A more detailed isometric drawing of the facility is shown in Figure 3.

The shielding is designed to give a maximum exterior surface dose of 2.5 μ Gy (0.25 mR)/hr during the processing of the most active specimens likely to be handled. The shielding on the working faces is constructed from steel plates bolted together in suitable sections. On most of the cells, part of the shielding is removable to provide gloved access for decontamination to low levels and to allow equipment maintenance. This feature is illustrated in Figure 3; the shielding will be removed by the use of a forklift device. Permanent shielding only is provided on the reception cell. High density concrete is used for shielding non-working faces.

Alpha sealing is maintained by providing a separate internal alpha box with gaiters as appropriate. Individual glovebox modules can be replaced after dismantling the shielding at that point. The ventilation for the facility is controlled by a Vortex amplifier, a fluidics device capable of providing a high degree of safety (7). With this system, an inward airflow of ~ 1 m/sec is established at the port opening in the event of a complete loss of a glove or gaiter.

The specification for each individual cell is given below:-

Cell 1a (Reception Cell)

A shielded alpha tight enclosure which houses the active filter change unit containing three 25 litres/sec HEPA filters. In-cell equipment includes three ultrasonic cleaning baths. Incorporated in this cell is a non-active posting port. A padirac transfer system is provided for posting contaminated materials.

Remote handling is provided by three sets of conventional ball and tong units with alpha sealed gaiters.

This cell will hold the alpha beta gamma material, including fuel samples for SEM routing. Initial cleaning of the specimens will be undertaken in this cell prior to transfer to cells 1b, 2 or 4.

Ventilation extract for all the cells in the facility will be routed through the active filter bank and ducted away to a 125 litres/sec Vortex amplifier.

Cell 1b and 1c (SEM Sample Preparation Cells)

These two cells are combined in one shielded alpha tight enclosure, divided by a partition to give clean and dirty areas, the side adjacent to the reception cell being the dirty area. A 200 mm gas tight posting port is to be fitted in the gamma shielding between cells 1a and 1b.

In-cell equipment in the dirty cell (1b) is to include a vice capable of 90° rotation, a metal saw and an ultrasonic cleaning bath which will be shared between both contaminated and clean sections.

Remote handling for cell 1b is provided by two conventional ball and tong units with alpha sealed gaiters. Cell 1c equipment is to include specimen magnified viewing by closed circuit TV. Two "Tru-motion mini-manip" master/slave manipulators provide the remote handling for this cell.

A posting port for the SEM transfer flask is provided on the front working face of the cell.

Note: Both cells 1b and 1c will be run with an inert gas atmosphere.

Cell 2 (Electropolishing Cell)

This is a shielded alpha tight enclosure with remote handling provided by two conventional ball and tong units with alpha sealed gaiters. In-cell equipment includes an ultrasonic cleaning bath and specimen electropolishing facilities.

Initially cleaned bulk samples will be transferred into this cell from cell 1a for further ultrasonic cleaning and electropolishing to produce a mirror bright finish on the bulk specimens.

Cell 3 (Monitoring Cell)

Cell 3 is a shielded alpha tight enclosure fitted with monitoring equipment for alpha beta gamma activity, and an ultrasonic cleaning bath. Two conventional ball and tong units with alpha sealed gaiters serve this cell. A close viewing binocular system is also provided.

In this cell inspection of the bores of the fuel pin cladding will be undertaken. As well as further ultrasonic cleaning of the specimens this cell also caters for gamma and beta/gamma activity level measurements of the specimens.

Cell 4 (Transfer/Storage Cell)

A shielded cell containing a pneumatic transfer terminal and specimen carrier loading equipment. The flight lines will be linked via a diverter to the shielded density measuring facilities and an adjacent beta gamma shielded specimen preparation cell. Incorporated in the floor is a shielded area for the storage of bulk samples. Two "Tru-motion mini-manip" master/slave manipulators will be used for remote handling.

A small gas tight port between this cell and cell 3 ensures that only the smallest of specimens can be transferred, thereby reducing the risk of alpha contamination in the rest of the facility.

Cell 5 (Machining Cell)

This is a shielded enclosure served by two "Tru-motion mini-manip" master/slave manipulators for remote handling. The equipment in the cell will include a modified spark erosion head, small drill stand and trepanning tool and ultrasonic cleaning baths. A gas tight posting port incorporated in the partition wall between cells 4 and 5 will be used for sample through transfer.

The 3 mm diameter TEM discs will be cut from the bulk material in this cell. It will also be possible to section large samples using slow speed cutting wheels.

Cell 6 (Grinding Cell)

This is a shielded cell served by two "Tru-motion mini-manip" master/slave manipulators. In-cell equipment includes an ultrasonic cleaning bath, a custom built grinding machine and a micrometer measuring system.

The 3 mm diameter TEM discs will be ground down in this cell to a thickness of approximately 0.13 mm, measured and cleaned before passing through the intercell posting port to the blister cell for final preparation.

The end wall of cell 6 will incorporate a posting port for flask posting of unwanted beta gamma active specimens and beta gamma waste material.

Blister Cell

This is a shielded glove box containing specialist electropolishing equipment for the final preparation of the TEM discs. A 110 mm La Calhene type posting port is incorporated in the side wall for the transfer of material.

Fume Hood

A small fume hood will be provided adjacent to the cell having a posting port compatible with the 110 mm posting port on the blister cell. This is to permit TEM discs and consumable materials to be posted in and out of the facility without contamination hazard.

Common Items

The following items serve several cells:-

1. A closed circuit television system using cameras with macro zoom lenses will be installed. This system will mainly serve cells 1c, 5 & 6.

2. A forklift truck will be supplied for lowering and removal of the cell shielded doors.

3. Lighting for each cell is to be provided from 400 watt high pressure sodium lamps (GEC type SON/p 400 watt 220-250 volt with associated control gear).

Concluding Remarks

The principle has been adopted throughout to design a facility which is extremely flexible and capable of housing specialist specimen preparation routes for many years to come. The modular design concept will permit radical but relatively cheap modification of individual cells whilst more minor modifications can be tackled easily by gloved access after removing the retractable shielding. In addition, the shielding is adequate to cope with increased activity and the ventilation is to the best of modern standards.

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SPECIMEN FLOW DIAGRAM

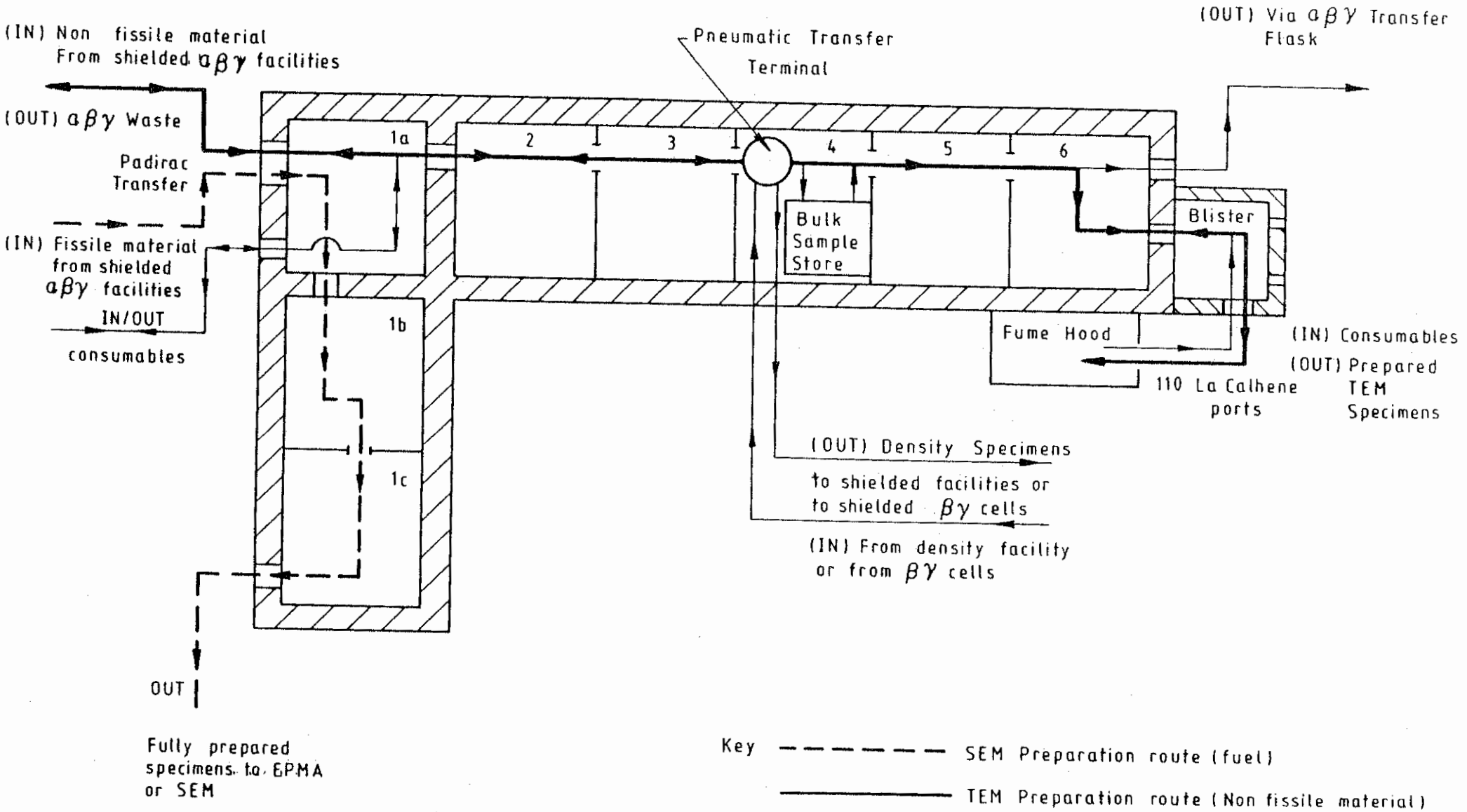
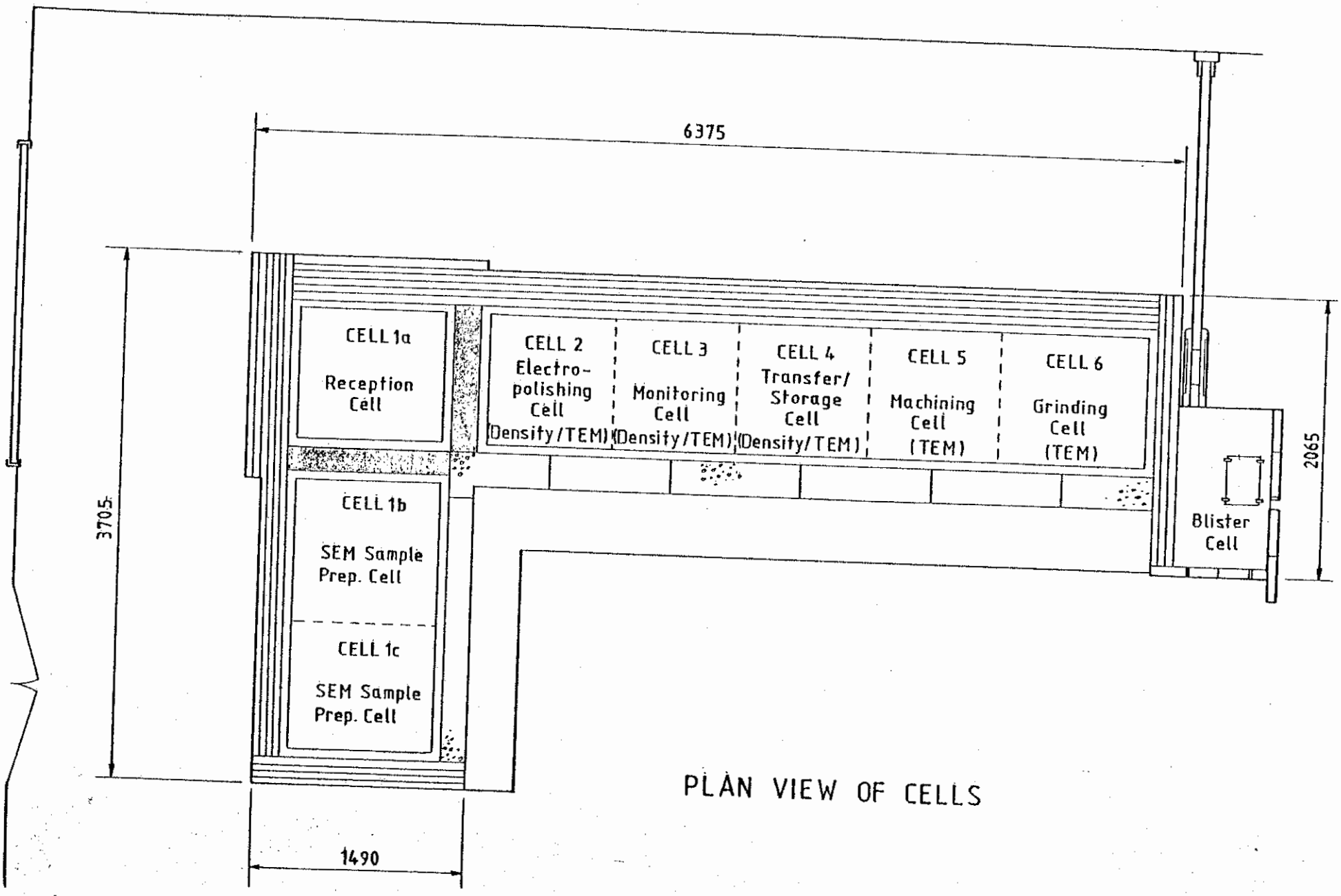


FIG. 1



PLAN VIEW OF CELLS

FIG. 2

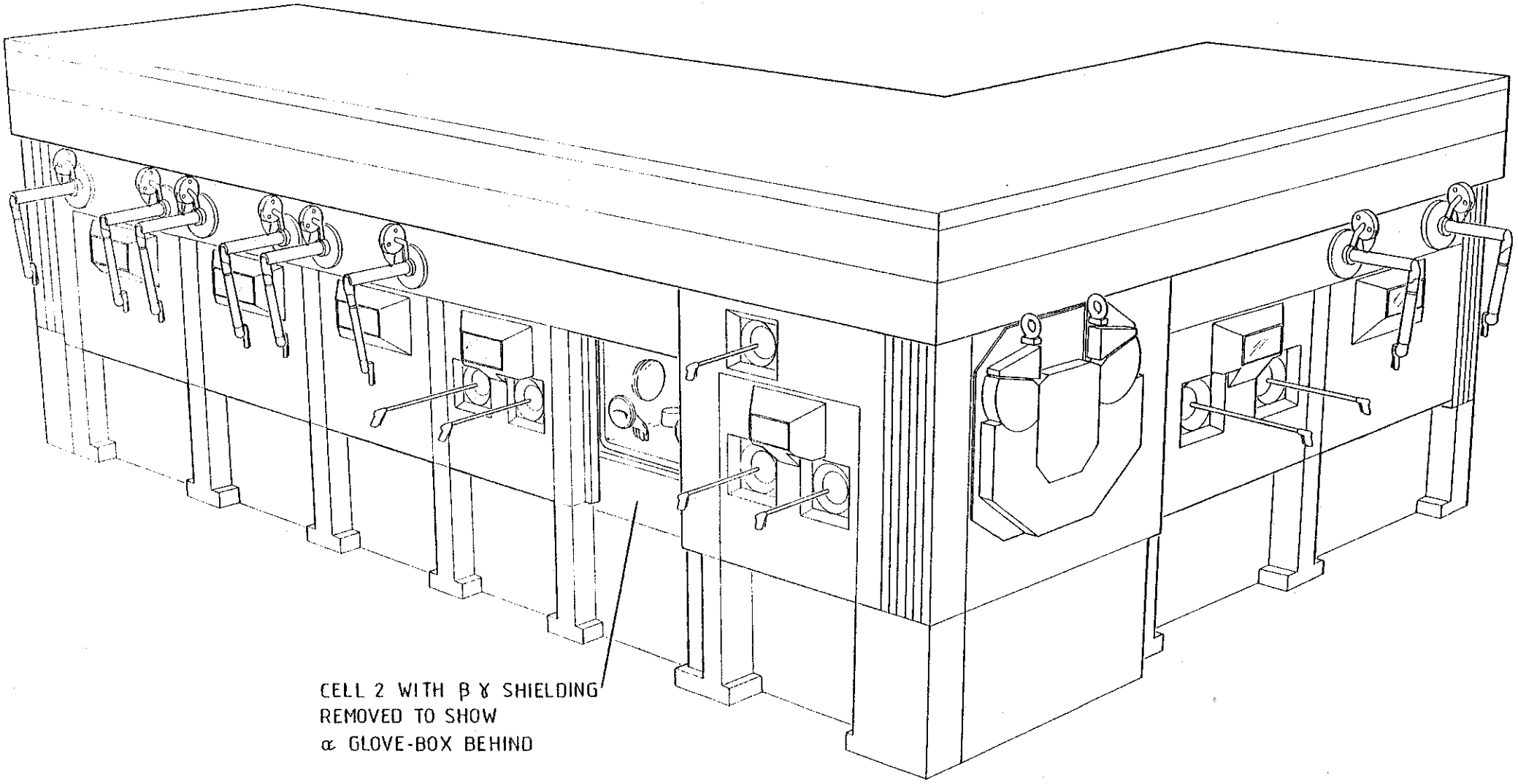


ILLUSTRATION OF OPERATING FACES.

FIG. 3