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The Examination of Nuclear Fuel Samples by Scanning Transmission Electron Microscopy

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The Institute for Transuranium Elements has recently taken delivery of a Hitachi H700 HST Scanning Transmission Electron Microscope (STEM), which has been adapted for the examination of α-contaminated samples. The instrument is being used in programmes of research into the swelling and transient behaviour of advanced nuclear fuels and in the examination of Pu-bearing aerosols. This has involved the development of an α-tight transfer system for loading samples into the microscope, and some modifications to the specimen stage of the instrument itself.

Up to the present time, the activity of the samples examined has been relatively low, so that comprehensive biological shielding has not been required. When larger fuel samples are examined the preparation steps will be performed remotely inside a lead cell, and the time spent in specimen transfer into the microscope column will be relatively short. A moveable lead screen can be positioned between the microscope column and the operator should this be necessary.

The Microscope

The Hitachi STEM operates at a maximum accelerating voltage of 200 kV, and combines the facilities of a high resolution transmission microscope with those of a normal scanning electron microscope. A detector mounted below the specimen enables a scanning transmission image to be obtained, and an energy dispersive X-ray analyser is fitted.

Thus the system can provide on the same sample area:

1. High resolution transmission data (down to 2 Å resolution) of the internal microstructure

2. Electron diffraction data, giving information about the crystal structure, from areas down to 200 Å diameter

X 3. Secondary electron imaging of the surface structure, down to a resolution of around 30 Å

4. Semi-quantitative chemical information from sample areas as small as 200 Å diameter.

The STEM instrument is unique in being able to provide all these modes of information from one sample area without altering the conditions. The only sacrifice which must be made compared with the normal SEM is a limitation of sample size - in this case to 5 mm x 15 mm. But for the types of sample examined this does not prove to be a handicap because even smaller size limits are imposed by the sample activity.

Specimen Transfer System

A three stage glovebox system was chosen and developed for the transfer of samples to the microscope column. This is shown in figure 1. This has the advantage of simplicity over a specific transfer device, such as the chamber used on the JEM 200 A in the Institute.

The glovebox system is connected to the microscope column via a flexible bellows and double door arrangement, as seen in figure 2. The gloveboxes are coupled to the microscope and the door opened for specimen loading, but decoupled while the microscope is in operation. This is essential, because the vibration transmitted from the underpressure system of the gloveboxes is detrimental to the resolution of the microscope.

The three stage system is designed to minimise the transfer of contamination to the microscope column.

Samples for examination are introduced into glovebox 1 through a transfer chamber A, which is also compatible with the gloveboxes used for specimen preparation.

Specimen holders and microscope accessories are stored in glovebox 2. For loading the samples only the very tip of the specimen holder, where the sample is clamped, projects into the loading box 1. This can be seen at B. This minimises the pick-up and transfer of contamination to the microscope column, and in the first two months of operation no detectable contamination was found on any part of the microscope specimen chamber or objective aperture system. The tip of the specimen holder can be observed using a binocular microscope during loading operations.

The loaded holder is then tranfered carefully through boxes 2 and 3, which are separated by a door, into the side entry goniometer stage of the microscope, and the bellows system is decoupled from glovebox 3.

The side entry goniometer stage has been slightly modified internally to make it α-tight, but this involved no changes in the design or principle of operation. No additional restriction is imposed on the specimen movement or specimen tilting capability.

The rotary pumps used for prepumping the column and for backing the diffusion pumps are arranged to pump through filters, but this has not significantly increased the pumping times required.

Specimen Preparation

Normal electron microscope preparation techniques of electropolishing, ion-beam thinning or powder crushing are used, except that all steps are performed inside gloveboxes or shielded cells. Powder or fragile specimens are mounted between carbon films supported on copper grids as an additional safeguard.

Full details of the preparation techniques used are given in a separate report which will be available on request.

Fig. 1

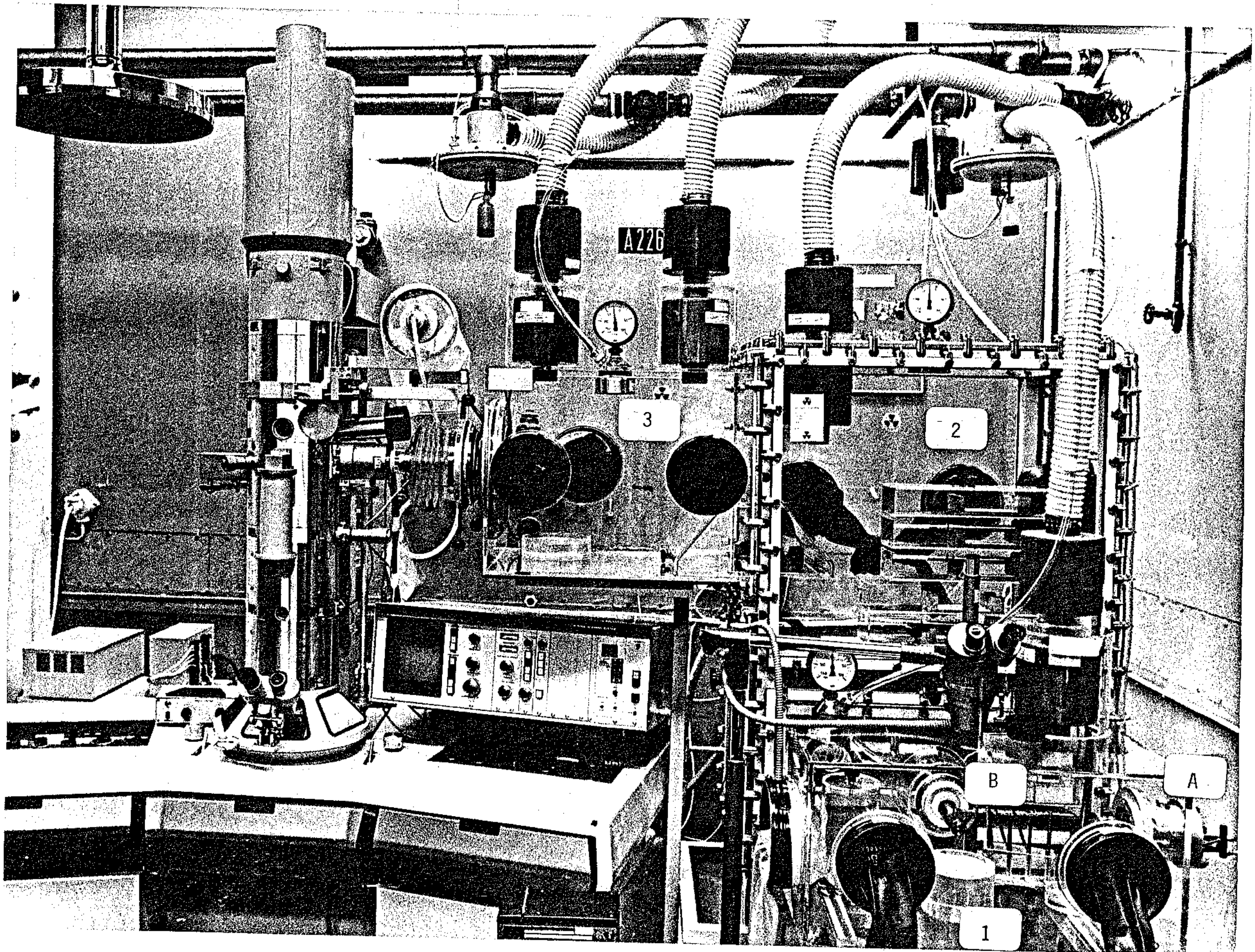
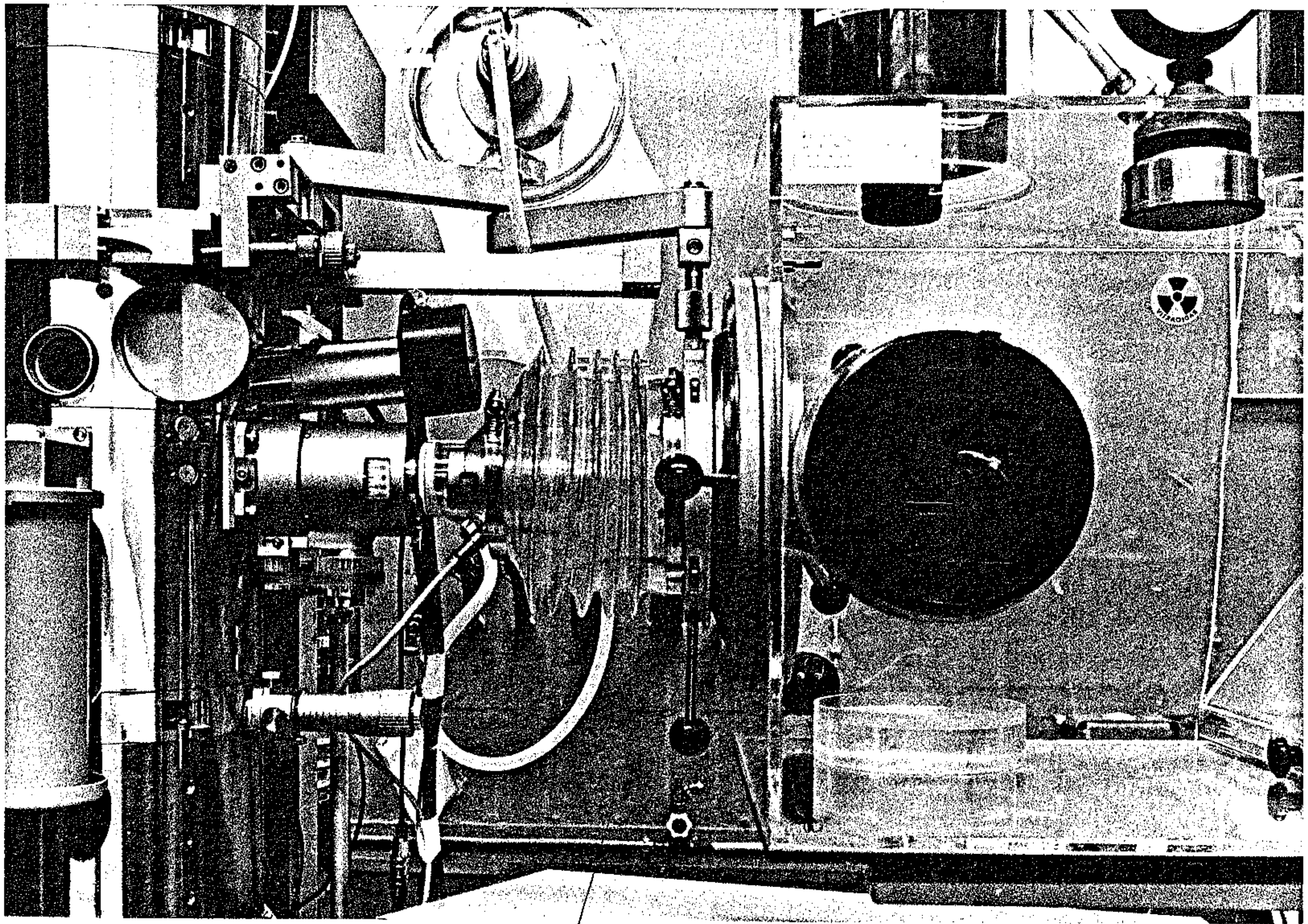


Fig. 2



door can be disconnected / separation from α box.