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THE EXAMINATION OF NUCLEAR FUEL SAMPLES BY SCANNING TRANSMISSION ELECTRON MICROSCOPY

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Abstract - A modified electron microscope for examining highly radioactive samples is described. This is being used in programmes of research into the swelling and transient behaviour of advanced nuclear fuels and the examination of plutonium-bearing aerosols. Some examples are illustrated.

At the Institute for Transuranium Elements a Hitachi H700 HST Scanning Transmission Electron Microscope has been adapted for the examination of α-emitting and highly irradiated samples of nuclear fuels. 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.

A three stage glovebox system was built for the transfer of samples to the microscope column. This is connected to the microscope column via a flexible bellows and double door arrangement for specimen loading, but decoupled while the microscope is in operation to prevent vibration transfer to the microscope column. The system is shown in figure 1.

The Hitachi STEM operates at a maximum accelerating voltage of 200 kV, and combines the facilities of a high resolution transmission microscope with more of a normal scanning microscope. A detector mounted below the specimen enables a scanning transmission image to be obtained. A PGT energy dispersive X-ray analyser is lifted. The detector can be screened for protection when very active samples are in the column. Samples with activities up to 5 Rad/hr at 10 cm have been examined. Thus the system can provide on the same specimen area:

1) High resolution transmission data of the internal microstructure (down to better than 10 Å resolution)
2) Electron diffraction data giving information about the crystal structure, from areas down to 200 Å diameter
3) Secondary electron imaging of the surface structure, down to a resolution of around 30 Å
4) When the sample activity is not too high (∼10 m REM/10 cm) semi-quantitative chemical information from 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 2 mm x 5 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.
The main application of the energy dispersive X-ray analyser has been to the characterisation of aerosol particles, where the background radiation level is not too high to mask the spectrum. Fig. 2 shows a secondary electron image of small Upu0₂ particles on a filter, figure 3 shows the corresponding image in a Pu X-ray window. Examples of applications will be shown.

Particularly impressive is the performance on active samples in the SEM mode, where relatively noise free images can be obtained from samples with an activity up to 5 Rad/hr. This is because there is no optical path between the sample and detector, which is mounted above the objective lens. Fig. 4 shows an example of a fractured surface at an irradiated (PuU)C₅ sample showing small fission gas bubbles, with a resolution better than 50 Å.

The samples used for transmission work are electropolished pieces of fuel extracted from optically examined fuel cross sections. No special preparation techniques are required for the SEM specimens.

So far the specimen handling system has functioned satisfactorily and no detectable contamination has been introduced into the microscope column during two years of operation.
Figure 2

Figure 3 (Inset)

Figure 4