MLM-2275(Ed)

CONF-750989
VOLUME IV
Distribution category UC-20

PROCEEDINGS OF THE INTERNATIONAL CONFERENCE HELD AT GATLINBURG TENNESSEE, OCTOBER 1-3, 1975

Sponsored By

OAK RIDGE NATIONAL LABORATORY
UNITED STATES ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
AMERICAN NUCLEAR SOCIETY
NUCLEAR METALLURGY COMMITTEE OF AIME

RADIATION EFFECTS *and* TRITIUM TECHNOLOGY *for* FUSION REACTORS

Editors

J.S. WATSON
F.W. WIFFEN

Compiling Editors

JULIA L. BISHOP
BRENDA K. BREEDEN
OAK RIDGE NATIONAL LABORATORY

MARCH, 1976

A SCANNING ELECTRON MICROSCOPE FACILITY FOR CHARACTERIZATION OF TRITIUM CONTAINING MATERIALS

G. L. Downs
P. A. Tucker

Monsanto Research Corporation, Mound Laboratory

ABSTRACT

A scanning electron microscope (SEM) facility for the examination of tritium-containing materials is operational at Mound Laboratory. The SEM is installed with the sample chamber incorporated as an integral part of an inert gas glovebox facility to enable easy handling of radioactive and pyrophoric materials. A standard SEM (ETEC Model B-1) was modified to meet dimensional, operational, and safety-related requirements. A glovebox was designed and fabricated which permitted access with the gloves to all parts of the SEM sample chamber to facilitate detector and accessory replacement and repairs. A separate console combining the electron optical column and specimen chamber was interfaced to the glovebox by a custom-made, neoprene bellows so that the vibrations normally associated with the blowers and pumps were damped. Photomicrographs of tritiated pyrophoric materials show the usefulness of this facility. Some of the difficulties involved in the investigation of these materials are also discussed.

A new facility for the investigation of radioactive pyrophoric materials by scanning electron microscopy (SEM) techniques was installed at Mound Laboratory. This SEM installation was designed so that the specimen chamber was incorporated as an integral part of a glovebox to enable easy handling of these materials. The modifications required to accomplish this with a standard SEM involved considerable design changes and installation innovations.

The enclosed specimen chamber concept required that the design of the SEM be such that it could be interfaced with a glovebox by vibration damping techniques and that it not be easily contaminated. In addition, all parts of the chamber needed to be accessible for cleaning or sealing off for removal of detectors for maintenance and repair.

The SEM's of only two manufacturers, AMR and ETEC, were considered as they were the only ones equipped with column liner tubes. The ETEC instrument was chosen because its column liner tube was continuous and the chamber was more accessible for cleaning and decontamination.

A rubber boot interface concept was designed for coupling the front side of the specimen chamber to the back side of a glovebox, and a specially shortened glovebox was purchased to enable a person of average size to reach to the back of the microscope chamber through the glove ports. The box was 12 in. deep and the chamber was 9 in. deep. ETEC changed its standard instrument to meet our mating needs and designed special isolation valves for the column and gun. Port covers also were designed by ETEC. These adaptations now allow every entrance to the chamber to be blocked from the glovebox atmosphere for repair and maintenance without opening the glovebox atmosphere to the room.

The appearance of the boxline installation is shown in Figure 1. A fumehood for introducing samples into a vacuum passbox is shown in the back of the room. The passbox opens into the glovebox which contains a circulating dry argon atmosphere purified by an individual Dri-train system. In addition the atmosphere is circulated through a separate catalytic deoxo unit which is used to remove tritium gas and keep the contamination level to a minimum. A Pedatrol pressure control system can also be seen. This system maintains the desired glovebox pressure by admitting pure argon or exhausting to the

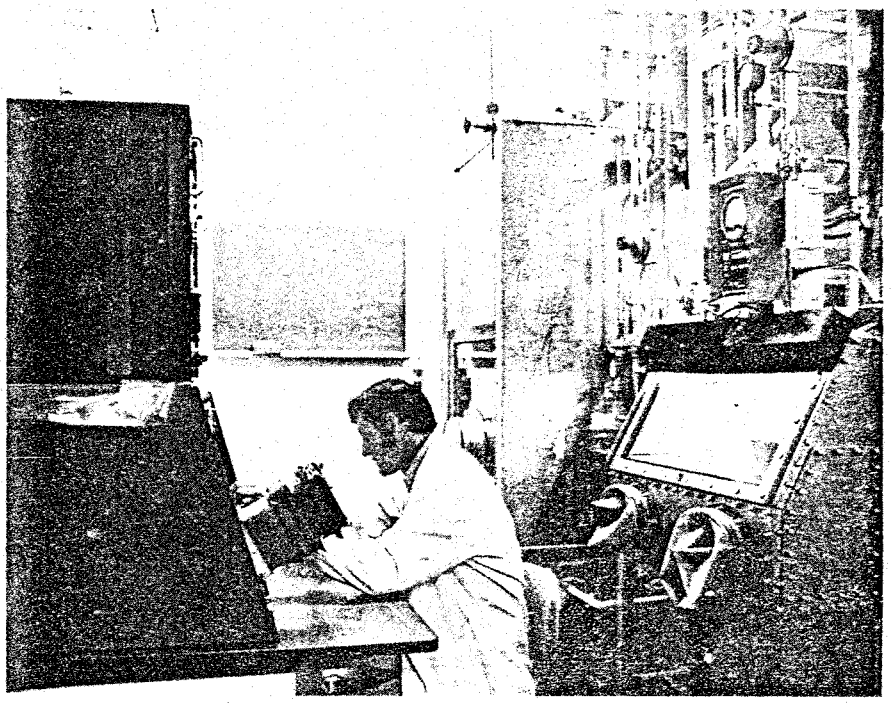


Fig. 1. Appearance of the SEM Glovebox Installation from the Front.

Effluent Recovery System as required. The overhead pipes lead to the Dri-train system in the adjacent room and to the house argon, chill water, and Effluent Recovery System lines. A Kanné monitor is located above the hood opening.

Figure 2 shows the back of the glovebox with the electron optical column and the rubber boot interface. The system is monitored by a Panametrics moisture analyzer and a Delphi oxygen analyzer. An ORTEC energy dispersive x-ray analyzer is shown in the foreground. The microscope chamber is mounted on a table top supported by hydraulic vibration dampers. The operating consoles for the SEM and the ORTEC are shown in Figure 3.

The specimen chamber door is shown in Figure 4 through the glovebox window. The stage controls shown can all be operated remotely at the main console. These five different motions are X, Y, Z, tile, and rotate. The specimen stage is

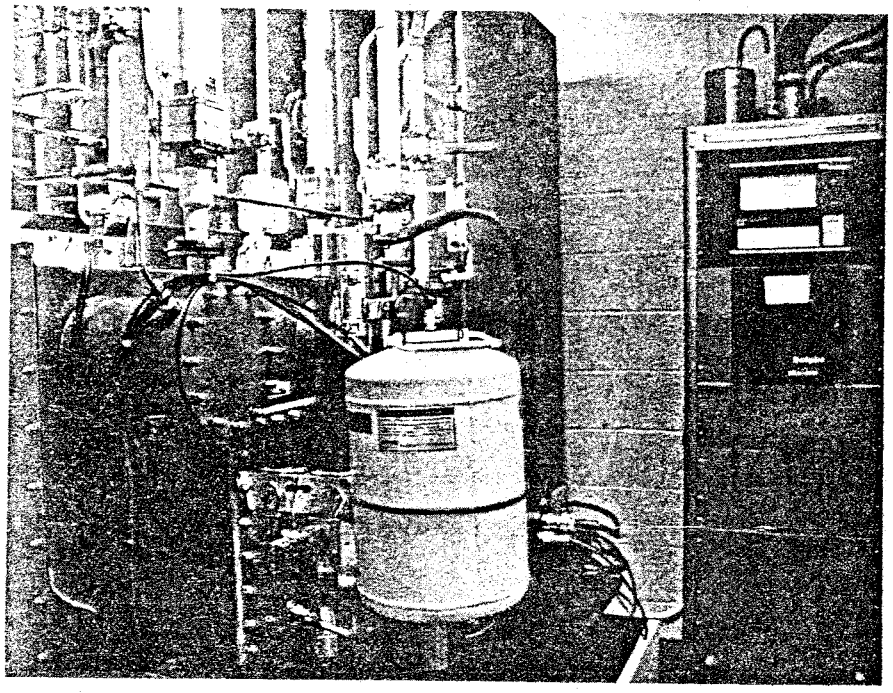


Fig. 2. View from Behind the Glovebox Showing the Electron Optical Column, Chamber to Glovebox Interface and Energy Dispersive X-ray Analyzer.

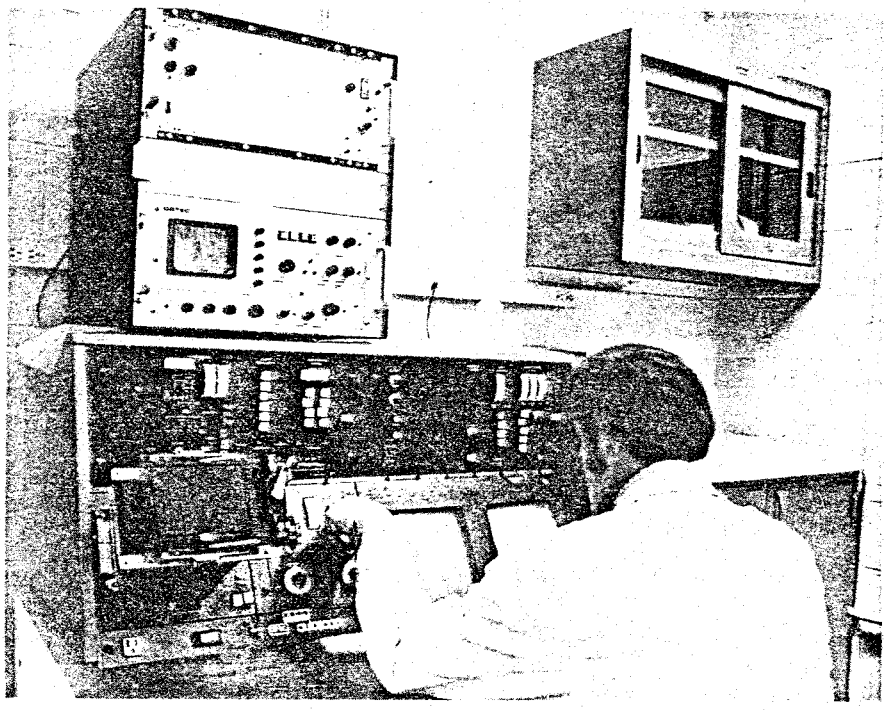


Fig. 3. The Operating Console for the SEM and the ORTEC Energy Dispersive X-ray Analyzer.

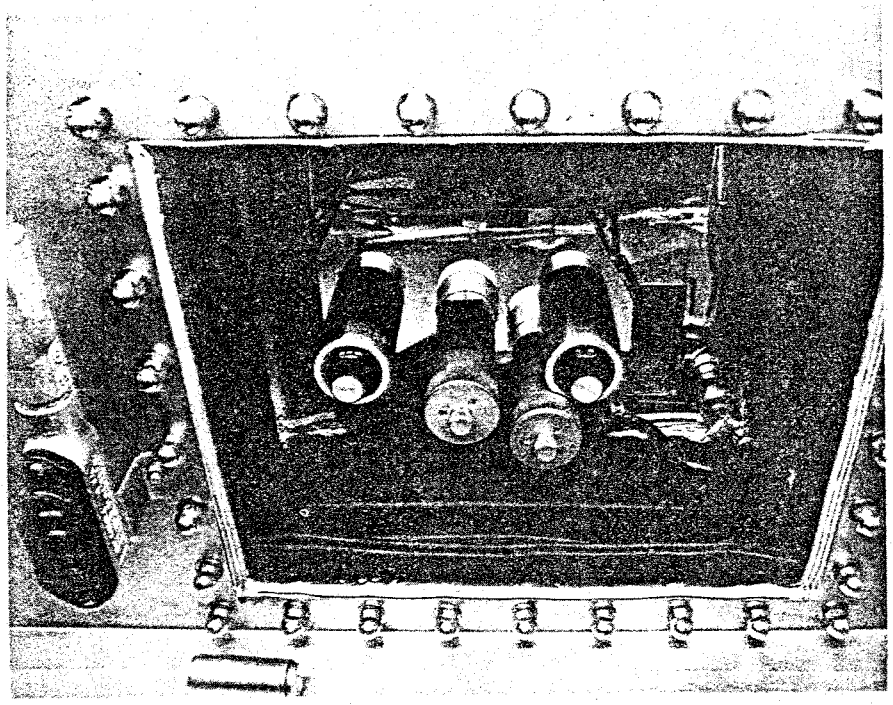


Fig. 4. View of the Specimen Chamber Door; the Stage Controls Shown Have All Been Remotized.

affixed to the door as shown in Figure 5. With the door open the chamber is empty except for analyzers and can be readily cleaned or decontaminated. The stage door is removable for changing types of stages or to bag out if decontamination or repair is necessary.

In a nearby laboratory a vacuum coating unit is also enclosed in a glovebox. A rapid access passbox is available at the rear of the glovebox for small samples only. Entry for larger samples or supplies is three boxes removed.

Samples from the evaporator or sample preparation box are transferred using the container shown in Figure 6. This is a common calorimeter can to which a flat Lucite plate is attached with epoxy so that it sits upright. Pin-type SEM sample stubs are placed in the holes of a half-cylinder cut from aluminum rod. This assembly fits inside the can which

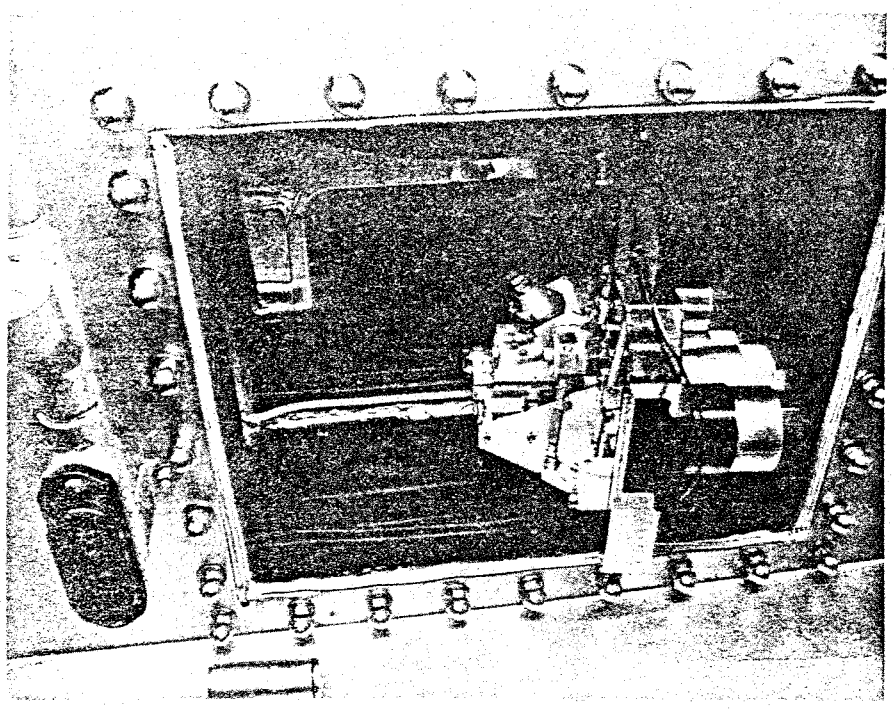


Fig. 5. View of the Specimen Stage and Chamber.

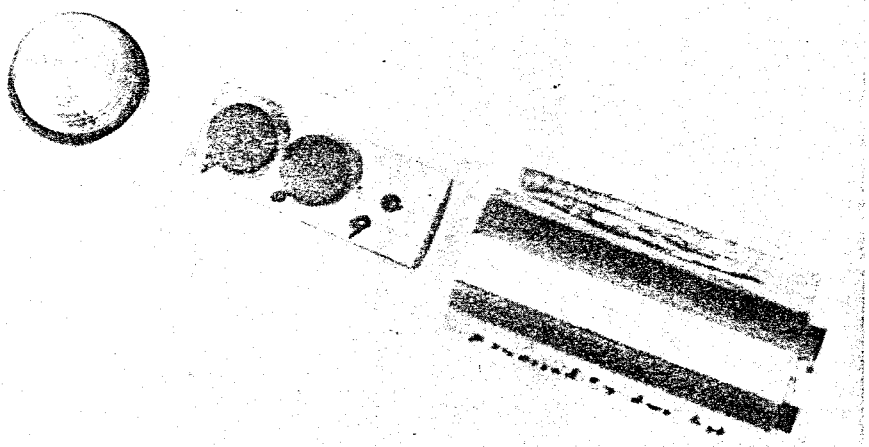
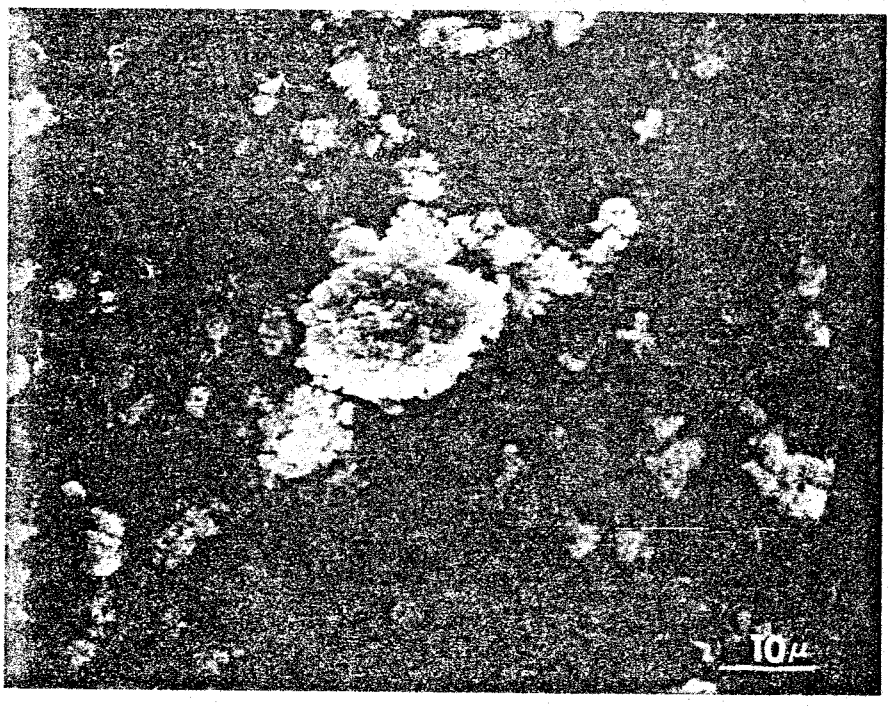


Fig. 6. A Sample Container for Moving Samples from the Sample Preparation Box to the SEM Glovebox.

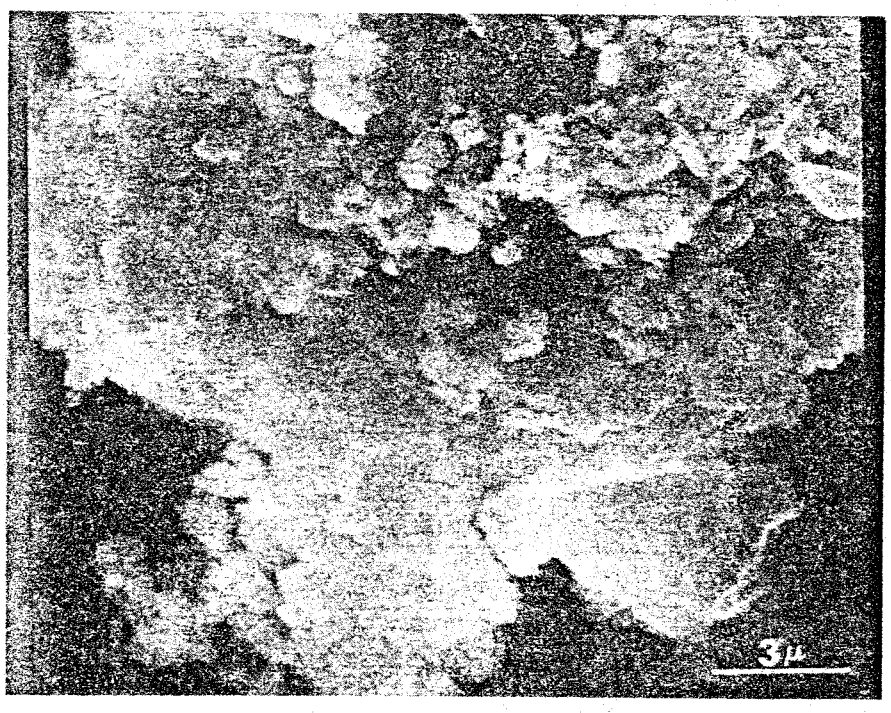
is closed with an O-ring sealed cap. The sample container is double-bagged out for transfer to the SEM passbox.

The operational procedures and glovebox atmospheres were first checked on pyrophoric nonradioactive samples, finely divided LiH. The powder was mounted using a technique obtained from T. Gregory (formerly at LASL) on similar material. The powder was sprinkled on a sample stub which previously had been covered with double-faced tape and heavily coated by vapor deposition of gold. The appearance of the particle distribution is shown in Figure 7A. The appearance at higher magnification (5000X) is shown in Figure 7B. This material was investigated at magnifications up to 20,000X, uncoated, with no apparent charging in a 20 kV beam and no apparent reaction with the atmosphere.

Initial investigations of small quantities of radioactive material were then initiated by examining lithium hydride containing approximately 1 mol % tritium. This was examined with only a carbon coating, and no differences were observed between this and the cold material. Typical photomicrographs are shown in Figures 8A and 8B. Samples of lithium and uranium hydrides containing approximately 30-50 mol % tritium were then examined. These materials were then removed from the SEM glovebox, and the equipment was surveyed for radioactivity. Wipes of 100 counts/min were obtained from the gun anode; however, no counts were obtained on the grid cap or the column liner tube. A single wipe covering 1/3 to 1/2 of the surface area of the glovebox interior counted 10,000 counts/min, and a cold sample, left open during the examination of the hot material, wiped 2000 counts/min. A cold sample examined after removal of all hot material was found to wipe less than 50 counts/min after examination.

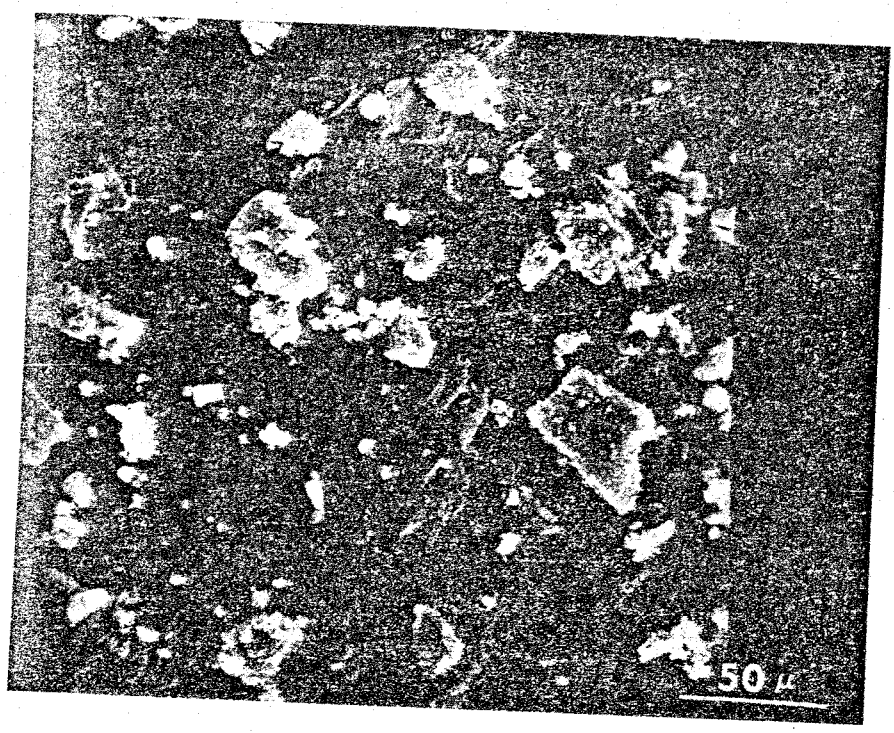


(A)

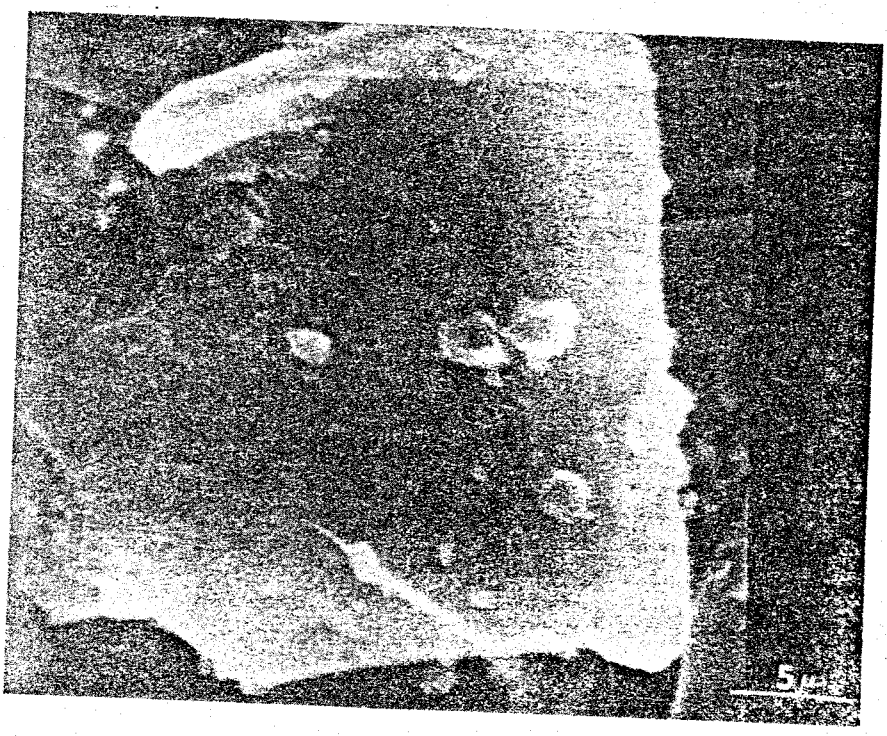


(B)

Fig. 7. Ultrafine LiH Powder Showing Sample Distribution (A) and an Agglomerate at Higher Magnification (B).



(A)



(B)

Fig. 8. LiH Containing Approximately 1 mol % Tritium, 60 Mesh Material at Low Magnification (A) and High Magnification (B).

Samples of uranium tritide were then prepared and a photomicrograph of the reaction product is shown in Figure 9. This material appears to have formed in layers due to columnar growth of the tritide grains from the metal with subsequent spalling and cracking of the layers due to stresses arising from volume change. After spalling the reaction proceeds with the freshly exposed metal.

In summary, an SEM facility for investigating radioactive, pyrophoric materials is currently in operation at Mound Laboratory. This facility has many applications in the study of tritium-contaminated materials. There is a limitation in the amount of tritiated material that can be examined because of the high background of secondary electrons from the β-radiation of the sample itself. This limit has not yet been determined quantitatively; however in practice, when this phenomenon is understood, compensations in techniques can be made. An ion

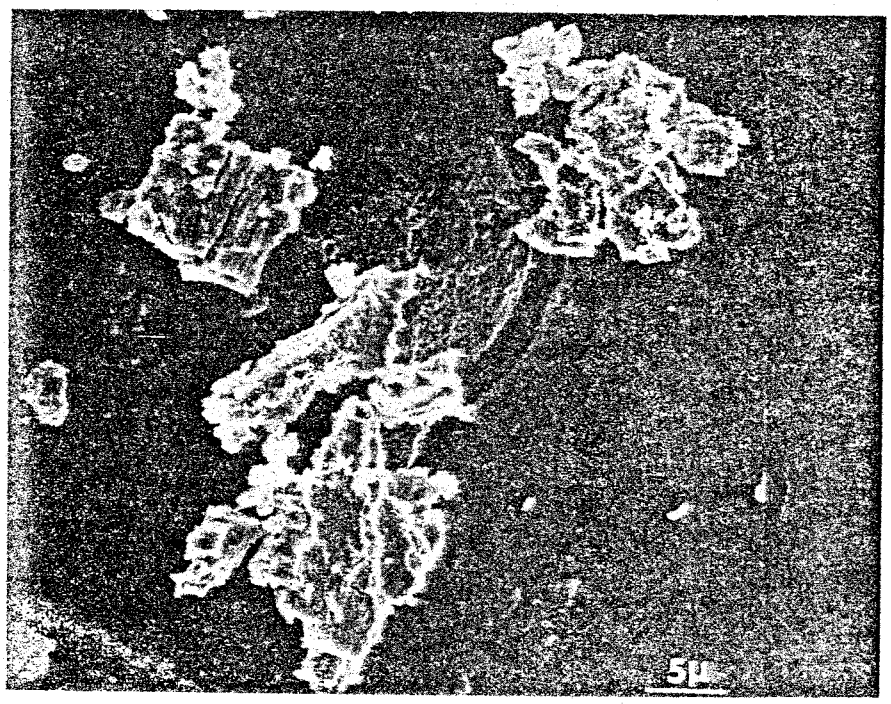


Fig. 9. Uranium Tritide.

mass analyzer attachment for this instrument has been purchased and is scheduled for installation in the near future. This will make possible analysis for masses from 1 to 300 amu.