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Sherwood F. Duliere

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Sandia National Laboratories

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Sherwood F. Duliere

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ILLUSTRATIONS

EQUIPMENT AND PROCEDURES USED FOR PREPARING SPECIMENS FROM PROMPT BURST ENERGETICS EXPERIMENTS*

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ABSTRACT

Post-test examinations are required to aid in the evaluation of results from Prompt Burst Energetics Study experiments. In order to perform these examinations the test packages must be disassembled in a radiation shielded, non-oxidizing environment. This report describes the equipment and procedures used to accomplish the disassembly and to prepare specimens suitable for optical and scanning electron microscopic examinations.

*Prepared for Division of Reactor Safety Research Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission, Washington, DC 20555, under Interagency Agreement DOE 40-550-75 NRC FIN No. A1016
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INTRODUCTION

The Prompt Burst Energetics (PBE) experiments being conducted at Sandia Laboratories, Albuquerque, New Mexico in support of the Advanced Reactor Safety Research Program involve irradiation of instrumented packages containing enriched fuel pins with sodium coolant in the Annular Core Pulse Reactor (ACPR). The experiments are being conducted to identify and examine the pressure sources and phenomena resulting from conversions of thermal energy to work during a simulated core disruptive accident. Additional information can be obtained from the post-test disassembly and examination of the experiment debris. Post-test information of interest includes micro and macrostructure of the debris, elemental distribution within the debris as well as size, geometry, and spatial distribution of the loose particles of fuel and cladding.

A shielded and inert containment of the material was necessary for disassembly of the fuel pins due to the radioactivity of the fuel and the reactivity of the sodium coolant in an oxidizing environment. Shielding requirements were minimal since the beta-gamma and alpha radiation were low as a result of the long elapsed time since the irradiation of the material. A glove box was found to provide sufficient beta-gamma shielding and prevent contamination due to alpha particles during sample preparation. The glove box also provides for inert containment of the sodium in an environment of argon containing less than one part per million of oxygen. Standard sample preparation equipment, modified according to needs identified during the successful disassembly of two experiments, was used in the box.

The experiments were disassembled and segments cut from predetermined areas of interest for the optical and scanning electron microscopic examination. Each segment was given an identifying number as were segment subdivisions. All specimens were controlled and stored as radioactive materials according to this identifying system.

PROCEDURE AND EQUIPMENT DESCRIPTION

A typical instrumented experimental package is shown in Figure 1. After irradiation, the test package was stored for over one year awaiting disassembly This allowed for the decay of the short lived radionuclides. The outer package was then disassembled and the pressure vessel containing the fuel pin was removed. Figure 2 shows a typical cross-section of the fuel and vessel. The radioactivity of the vessel was measured and the highest radiation level was found to be 35 milliroentgen beta-gamma at contact. Alpha activity was only slightly above background. These levels were low enough to allow for brief unshielded handling.

Two x-ray radiographs were then taken of the vessel at 90° orientations. A typical radiograph, Figure 3, shows the distribution of material in the pressure vessel. Areas of interest for the optical and scanning electron microscopic examination were selected from the radiographs. Their positions were marked on the vessel exterior and entered in the log book along with sufficient information for future identification. Figure 4 shows segments selected for cutting from experiment PBE-7S. After marking these areas of interest, the vessel was placed in the glove box.

The glove box assembly shown in Figure 5 consists of a 43-inch long VAC Atmosphere Company Dri-Lab (HE-43-6) which includes a 24 x 15 inch anti-chamber, Homolite front, side and skylight panels, two glove ports in the front panel and one in the side. An in-line filter to the anti-chamber prevents radioactive particulates from entering the chamber from the box during back-filling. In addition, the box contains electrical feed throughs and plumbing for gas recirculation. Filters are present in the input and output to prevent circulation of radioactive particles through the system. Stock argon gas used to create the inert atmosphere is purified by passing through catalyst beds in the Dri-Train, where water is removed and oxygen is gettered. The resulting atmosphere contains less than one-tenth part-per-million oxygen and less than twenty part-per-million water. Two catalyst beds are present to allow for the regeneration of one bed while the other is being used. The regeneration process uses stock nitrogen or argon gases containing hydrogen. An evacuation-pressurization system automatically provides either a positive or negative pressure. For this operation the box is kept at a slightly positive pressure. The evacuation system is vented to a filtered radioactive exhaust.

After an experimental assembly was placed inside the box, a flat was filed on the side of the vessel to preserve rotational orientation. The box was evacuated and backfilled with argon four times in order to remove as much residual air and moisture as possible. The recirculating system and Dri-Train were then turned on to remove the remain ing moisture and oxygen. With repeated regeneration of the catalysts the oxygen concentration was lowered to a level of 0.5 to 1.0 parts-per-million. Atmospheric monitoring was performed with a Panametrics Model 1000 hygrometer to determine water content. A Thermox Probe was used to obtain the oxygen concentration. The oxygen concentration was also checked using a bare tungsten wire filament.

A Leco Corporation Model VC-50 saw was used to cut the segments from the vessel. The saw includes a micrometer adjustable vise specimen holder, a variable speed motor and wet or dry cutting options. Several saw blades were evaluated for these procedures. The best blade was found to be a five inch diameter metal wheel with 220 grit diamond bonded to the cutting edge. The first two saw cuts were made to remove the end flanges. Since the end flanges contained both fuel and sodium, they were sealed in plastic bags to await future sodium dissolution and fuel removal. A Poly Sealer Model 210 was used to seal the polyethylene plastic bags. Individual segments, as marked on the radiographs, were then cut and a notch was filed on the bottom for orientation. Cutting times varied from thirty to one hundred twenty minutes depending upon the type and amount of material in the cross-section. During cutting the saw speed was kept at less than three hundred feet per minute to minimize the heat buildup, prevent sodium from melting, and minimize smearing. Periodic dressing of the saw blade with a silicon carbide dressing block was required. Cutting was performed both dry and with kerosene as a lubricant. Kerosene offered no obvious advantages and resulted in oxidation of the sodium and cleaning difficulties, therefore, all subsequent cutting was performed dry.

After several cuts, the saw malfunctioned. An investigation showed the motor brushes to be worn. The lack of lubricating moisture and the poor heat conducting properties of the atmosphere were determined to be the causes of failure. New brushes were fabricated using a lower wear rate Stackpole graphite. This minimized future brush wear. With continuous cutting, the heat buildup due to the low heat transfer characteristics of the atmosphere caused the saw control electronics components to fail. Replacing the components and installing a heat-sink provided longer operation times, but failures still occurred. This problem was finally eliminated by removing the electronic controls from the box and controlling the saw externally utilizing the electronic feedthroughs.

The cutting operation required personnel to have their hands in the gloves for one hour or less per day. Radiation monitoring showed that the personnel extremity radiation dose level was much less than the limit of 25 rem per quarter. The radiation level at an operating observer position outside the box was found to be less than one milliroentgen per hour, thus, the whole body dose limit of 2.5 milliroentgen per hour was also not exceeded.

After cutting, the segments were de-burred with a file. The surfaces were then lightly ground with 600 grit silicon carbide and swabbed lightly with an alcohol dampened swab to remove any sodium smearing caused by the grinding. The location of the fuel, sodium and metal could be observed on both surfaces of the segments. These surfaces were optically photographed and compared to the radiographs.

In order to obtain these optical photographs containers had to be designed and built so that the segments could be removed from the glove box and examined by optical microscopy while being confined in a non oxidizing environment. The containers, Figure 6, have thin quartz windows in each end and an external screw adjustment to move the segment ends into position in a focal plane next to the windows. O-rings are used in the ends and in the screw adjustment to seal the container. The sealed container maintained the segments in the nonoxidizing atmosphere for times of one to two hours, allowing sufficient time for examination. Figure 7 is a typical optical photograph taken through a container window.

After the microscopic examination the containers were placed in a hood vented to the radioactive exhaust system. The containers were opened and the segments removed. The inert container was then decontaminated for reuse. The segments were placed in a ten to twenty percent water in methanol solution in order to dissolve the sodium and release the loose fuel and stainless steel particulate. After dissolution was complete the residue was washed repeatedly in methanol and then in distilled water. The material previously hidden by the sodium was then available for examination by optical as well as scanning electron microscopy and the loose particulate could be characterized as to size, geometry, and location in the pressure vessel. The size and location of the particles were determined by screening the particulate from representative segments through a series of sieves ranging in size from 3350 microns to 38 microns. Sizes were also determined from scanning electron micrographs. The geometry and elemental composition of the particles were determined by using optical and scanning electron microscopy as well as x-ray energy dispersive analysis. Figure 8 shows a typical scanning electron micrograph composite from the lower end of the assembly after the sodium and particulate were removed from the center area. This segment was cut and polished in a separate alpha facility. Finally, each specimen was labeled and logged for future reference and for radioactive material inventory.

CONCLUSIONS

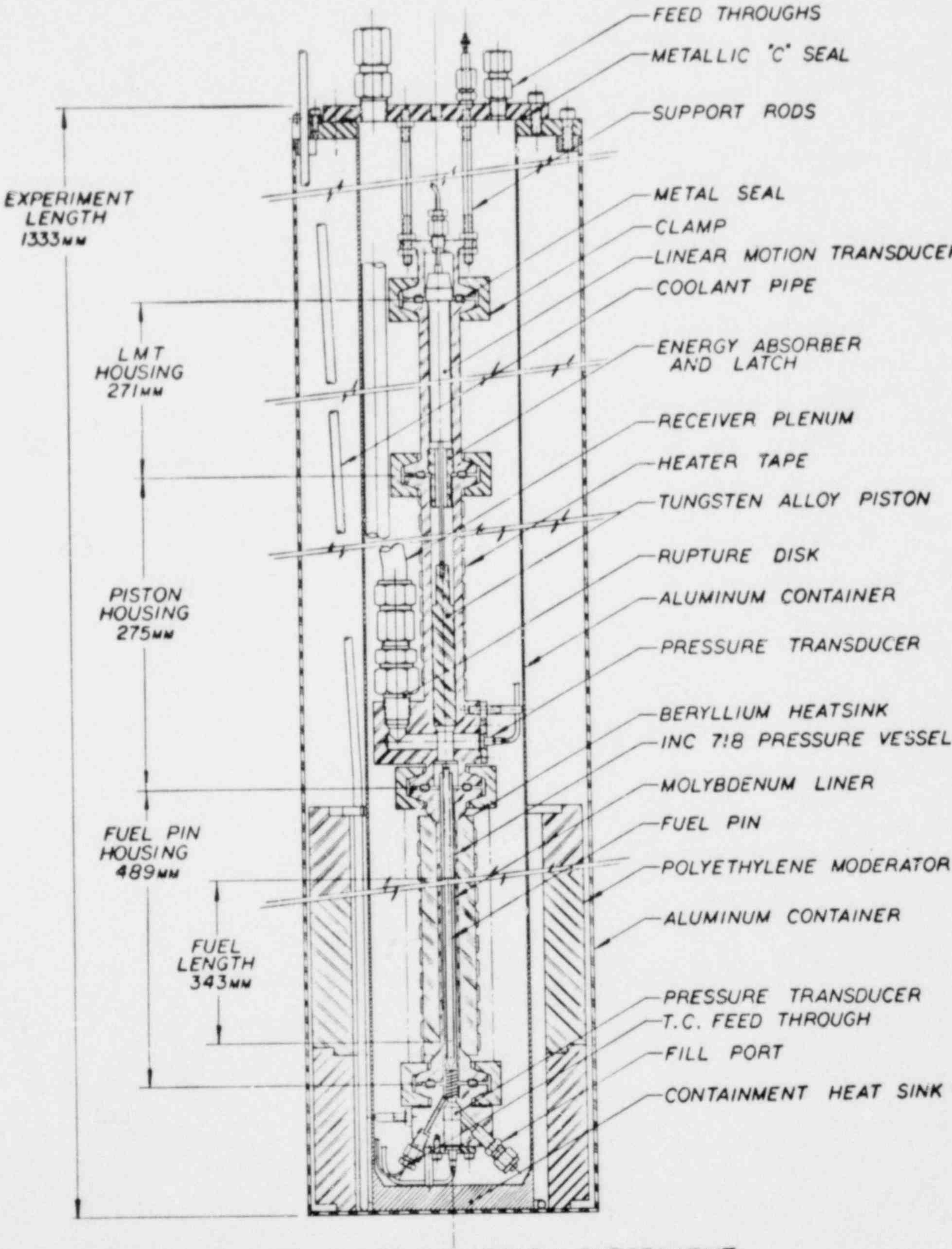
Installing and using this facility served several purposes:

1. It provided a means of obtaining specimens for post-test analysis of the PBE experiments.
2. Inert transfer containers were designed which allow the removal of the sodium containing segments from the glove box. Glass windows in the end of the containers permit optical microscopy examination of the segments.

3. Techniques were developed to remove the sodium from the fuel containment vessel.

4. Methods of characterizing the particulate debris were established.

5. The experience gained in evaluating the equipment and developing procedures can be utilized in applications of Sandia's hot-cell operations, and can add insight into use of equipment in glove box operations.



PROMPT BURST EXCURSION EXPERIMENT

Figure 1. Instrumented Experimental Package

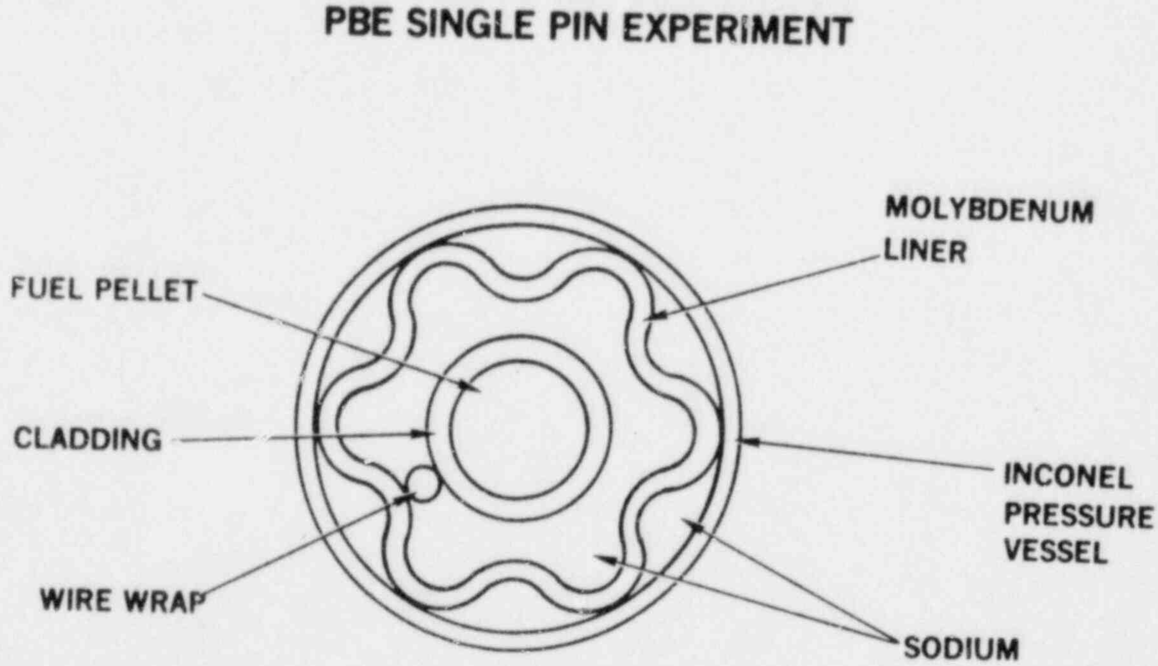


Figure 2. Schematic of cross section of fuel pin and containment vessel.

Figure 3. X-Ray Radiograph Showing Distribution of Material in Experiment PBE-7S Pressure Vessel

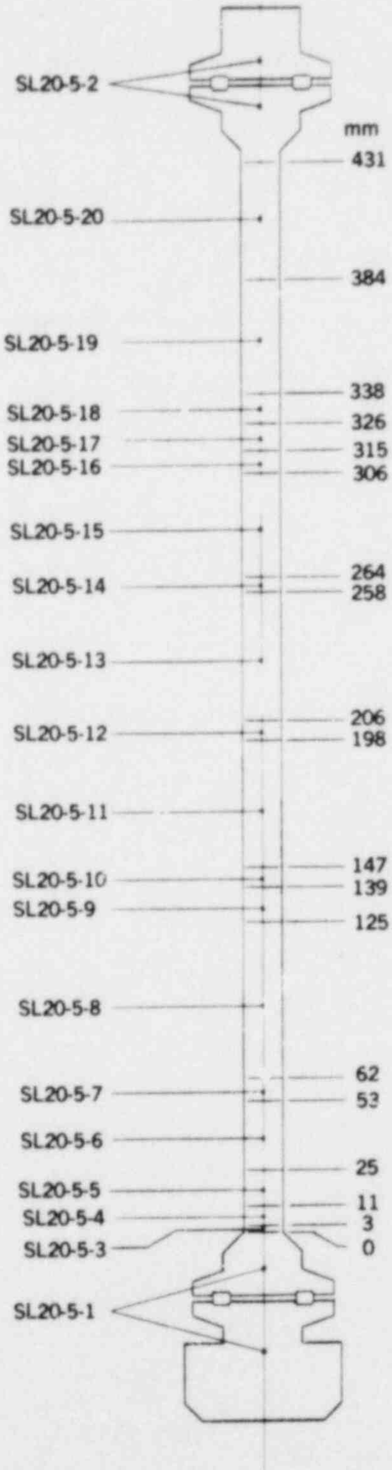


Figure 4. Experiment PBE-75 Specimen Location and Number

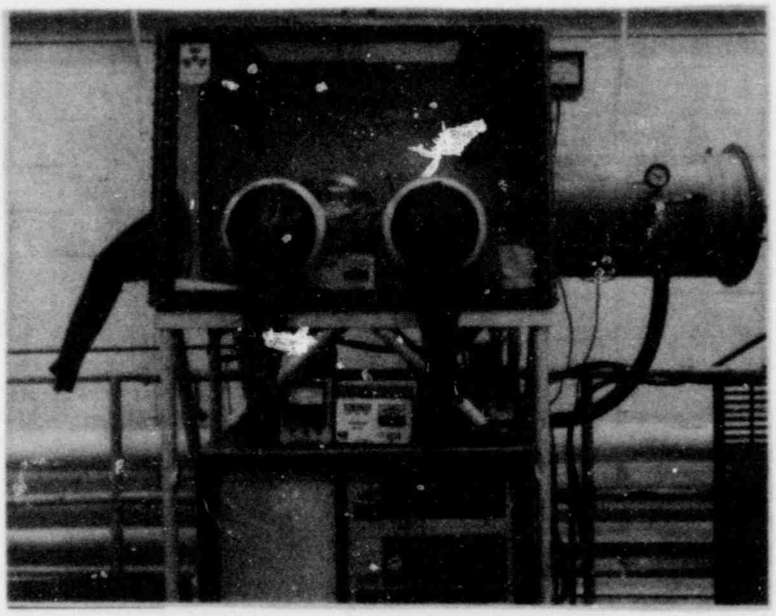


Figure 5. Glove box and equipment used for disassembly of the fuel pins

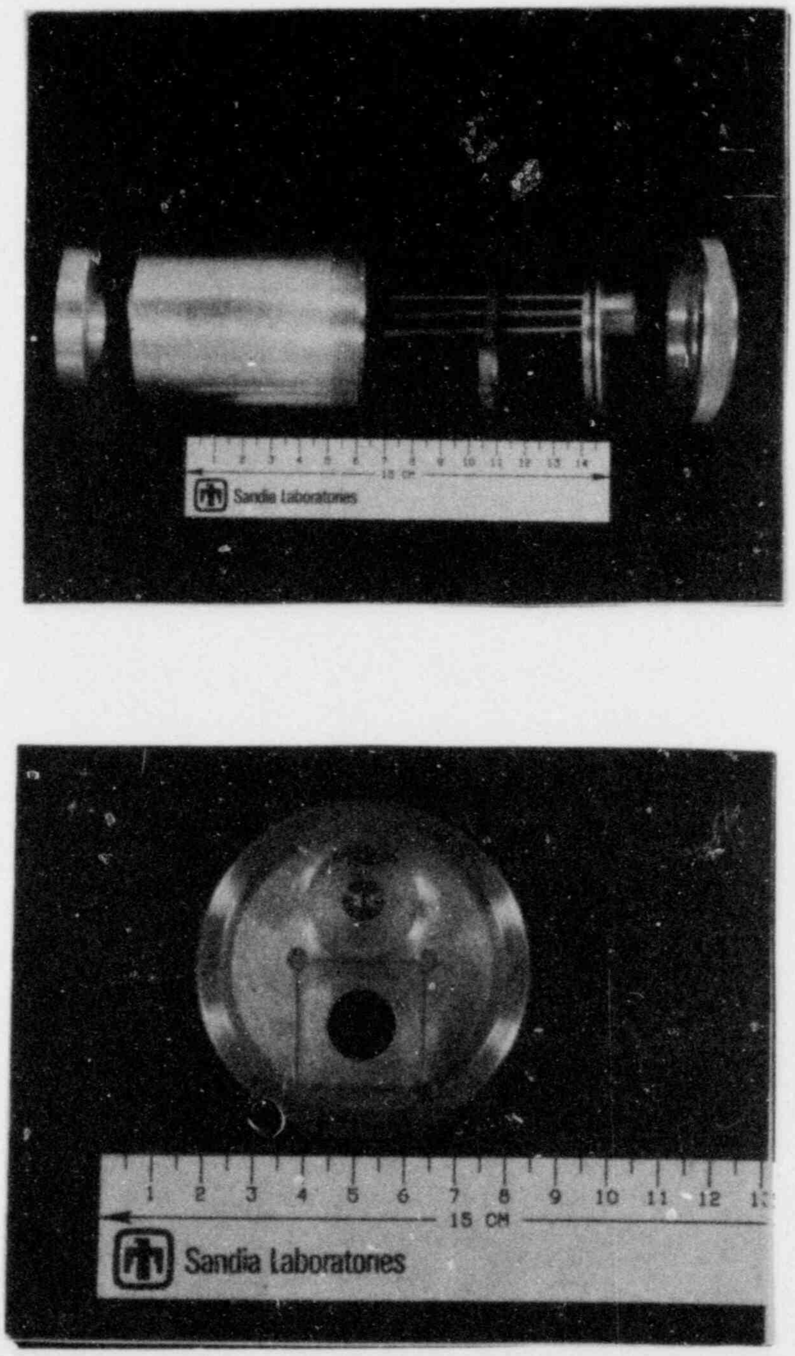
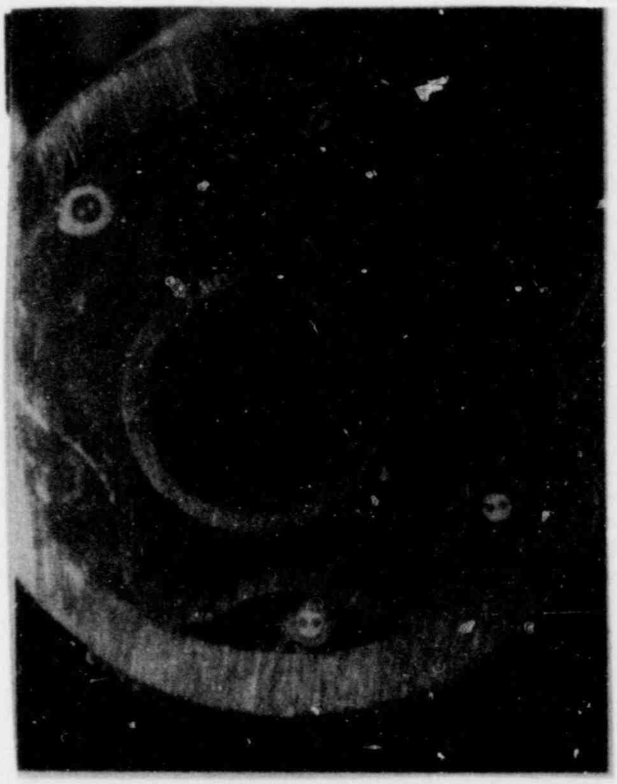


Figure 6. Container designed to allow optical examination of sodium containing specimens in an inert atmosphere



7X

Figure 7. Typical cross-section of experimental assembly as photographed through the inert container window

15X

Figure 8. A composite of scanning electron micrographs showing a cut and polished section from segment SL20-5-5

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