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DEVELOPMENT OF REMOTE METALLOGRAPHIC TECHNIQUES FOR IRRADIATED MATERIALS

by

R. Carlander

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DEVELOPMENT OF REMOTE METALLOGRAPHIC TECHNIQUES FOR IRRADIATED MATERIALS

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R. Carlander

Metallurgy Program 6.1.3

September 1961

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ABSTRACT

A remote metallographic facility has been in operation at Argonne National Laboratory since 1954. During that period of time, many new techniques relative to better contamination control and equipment operation have been developed. Further improvements will continue to be made in the normal evolvement of the operational procedures. The techniques used for microscopic examination of irradiated materials have been standardized with variations only in the final polishing steps, and detailed procedures are given for several alloys. The procedures used for macroscopy vary from sample to sample, and new procedures are developed as required to suit each particular problem.

INTRODUCTION

The remote metallographic facility at Argonne National Laboratory, which became operational in 1954, was specifically designed to accommodate not only the experimental fuels of the present, but, also, any conceivable fuel materials of the future. The basic construction and layout of the equipment was arranged in such manner as to attain good contamination control and simplicity of operation while retaining all the concepts which govern good metallographic procedures. The normal evolvement of improved remote-handling procedures has permitted the redesign and substitution of some equipment. It is believed that further modifications will continue to be made as more experience is gained in remote metallography.

The metallographic facility is a three-cell cave (see Figures 1 and 2) capable of shielding 100 curies of 1-Mev gamma activity. The basis for the design and layout of the installation and the equipment has been given in previous reports.(1,2,3) There have been slight changes in the methods of radioactivity containment and equipment operational procedures in order to achieve continuity in the metallographic processes.

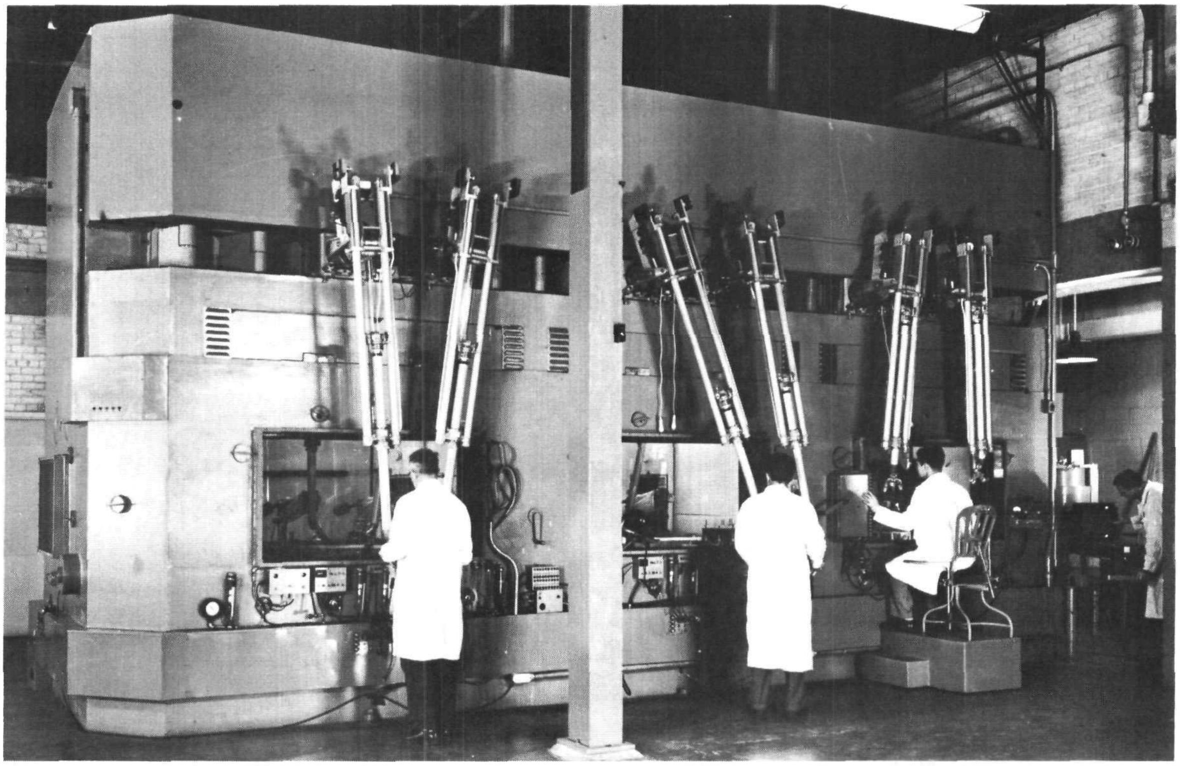

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Fig. 1. View of Metallography Cave. (Shielded Metallograph Is at Far Right.)

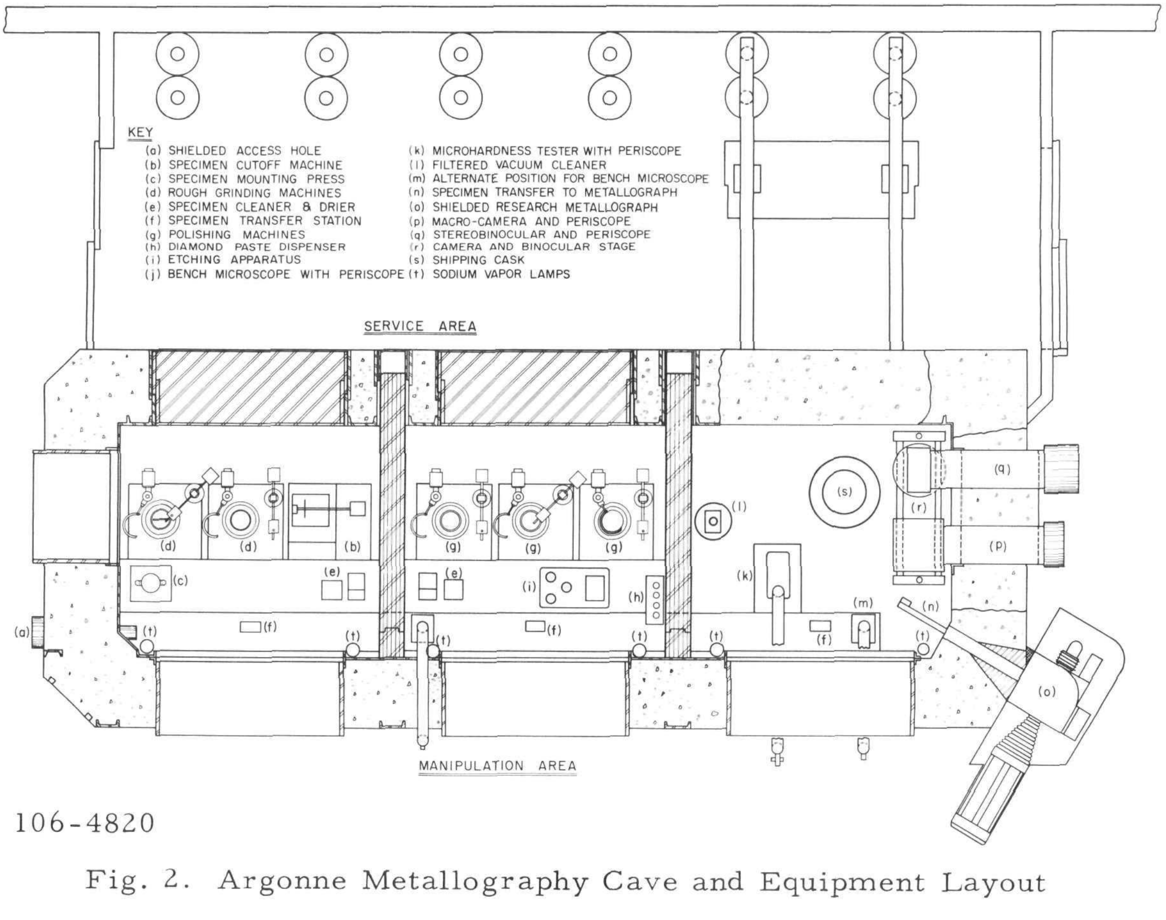

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Fig. 2. Argonne Metallography Cave and Equipment Layout

The techniques employed in preparing specimens for metallographic analysis is contingent upon whether microscopic or macroscopic examination is desired. The methods utilized in preparation for microscopy are standardized, with variation only in the polishing procedure and etchant employed in the final step. Preparation for macroscopic investigation depends upon the physical dimensions and condition of the specimen, and different techniques are generally used for each group of specimens submitted for examination.

SECTIONING

Samples are cut with either an electrical discharge machine,(4) an abrasive cutoff machine, or a small hand saw. The latter has been essentially discarded as a method of sectioning and is employed as a last resort on slightly irradiated specimens in order to expedite their examination.

The advantages of the electrical discharge process over that of the abrasive cutoff are: the control of surface finish, the abolishment of heating effects, and the virtual elimination of a deformed surface layer. Disadvantages are: the inability to cut nonconducting materials, and the selective corrosion of one component of a multicomponent sample due to differences in electrical properties.

The principal alterations to the commercial unit were directed at containment of the radioactive waste produced during the cutting operation, protection of certain irreplaceable components from contamination, and maintenance of uniform cutting conditions. The containment of radioactive waste is accomplished by cutting the specimen while completely submerged in water. Enclosure of the cutting unit in plastic and maintenance of a negative pressure over the unit by means of a vacuum cleaner with accompanying filter prevent spread of airborne activity into the cave proper. Separating the cutting unit from the electrical control and motor-drive unit prevents these difficult-to-clean parts from becoming heavily contaminated. Substitution of the method of cutting by driving the work piece into a stationary tool is made by clamping the specimen to the work table and sectioning it with a revolving circular brass disc. This disc possesses the advantages of exposing the cut surface to fresh water and of maintaining uniform wear on the tool circumference. If this uniform wear feature were not incorporated into the machine, a new tool would have to be substituted each time a different size or configuration of the work piece were encountered.

The operation of the machine (see Figures 3 and 4) consists of applying a difference of potential between the suitably aligned specimen and the revolving brass disc. The specimen is placed in a special fixture and made the anode in the cutting process. The rotating brass disc is made the cathode and is automatically driven into the specimen by means of a feed control. The specimen is eroded by the spark discharge occurring between the cutting disc and the specimen. The feed rate is determined by the gap existing between the cutting disc and the sample. The gap is automatically controlled at 0.01 in. at a potential of approximately 30 volts. The disc is slowly fed into the specimen while maintaining the proper gap until the section has been completed. The specimen is cut while submerged in water; the small deformed layer formed is easily removed during grinding.

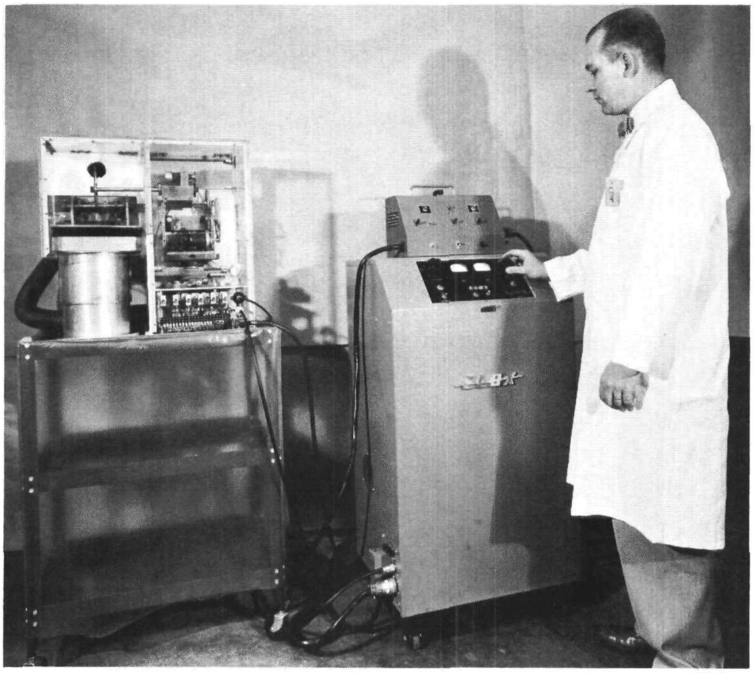

Fig. 3

Electrical Discharge Machine for Sectioning Metallographic Specimens

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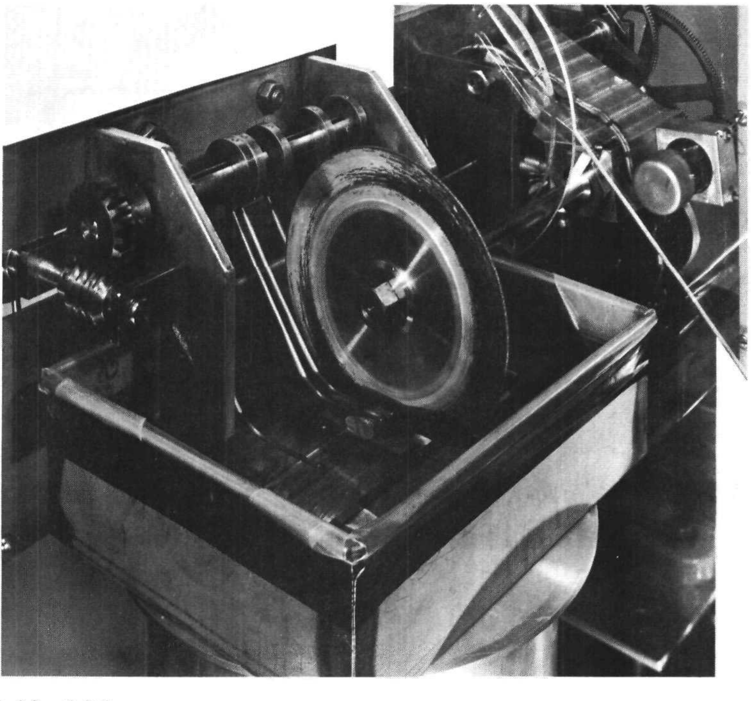

Fig. 4

Cutting Unit of Electrical Discharge Machine in Operation

141-921

The spread of contamination from the electrical discharge operation is minimized by enclosing the cutting unit in a plastic box and maintaining the enclosure at a slightly negative pressure. The liquid waste, when necessary, is pumped out of the water pan into an absorbent such as vermiculite. The pan is then collapsed remotely and discarded into a suitable container. Several parts of the machine become highly radioactive and are quite difficult to clean remotely. These parts are principally those which provide the electrical path and are necessarily made of brass, which does not lend itself as readily to decontamination as stainless steel. These parts are cleaned as much as possible remotely and then detached from the machine in the adjoining isolation room and, if required, replaced with duplicate parts.

An abrasive cutoff machine (see Figures 5 and 6) has been recently developed for remote operation by the Remote Control Division from a basic Buehler No. 1015 unit and has been used for the sectioning of some ceramic and metallic fuel specimens. This machine has been operated in a higher-level cave because of the high activity level of the specimens for which sectioning has been necessary. The performance of the machine is not novel relative to commercial units, except that the rate at which the specimen is fed into the abrasive disc can be varied down to one inch in 8 min. The abrasive disc revolves at 3450 rpm and is powered by a one-hp motor. The specimen is cooled by a jet spray of filtered water to which detergent has been added. The important modifications to the machine were concerned with radioactivity containment and disposal, and are still in a process of development as the machine continues to be operated. The water from the sectioning tank is circulated by a centrifugal sump pump through a disposable filter. The water from this filter flows into a plastic container. When decontamination is necessary, the water is absorbed by the addition of vermiculite and the plastic container is discarded. All rubber tubing can be disconnected remotely and replaced. The pump impeller and housing from the sump pump and the entire feed pump are discarded rather than decontaminated.

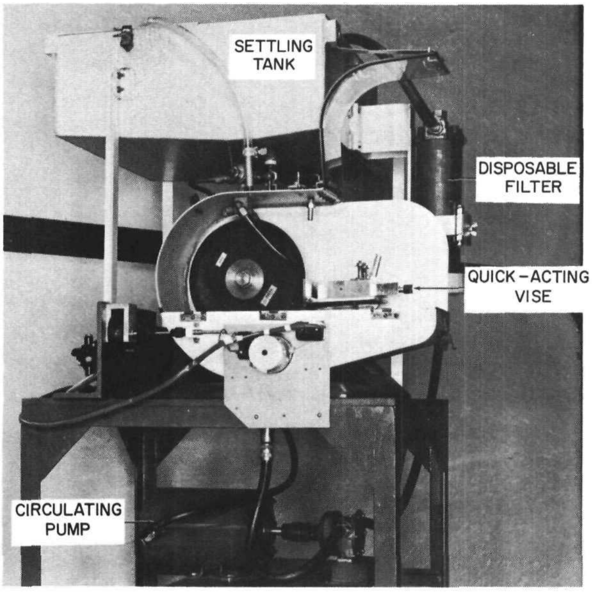

Fig. 5

Abrasive Cutoff Machine Modified for Remote Operation

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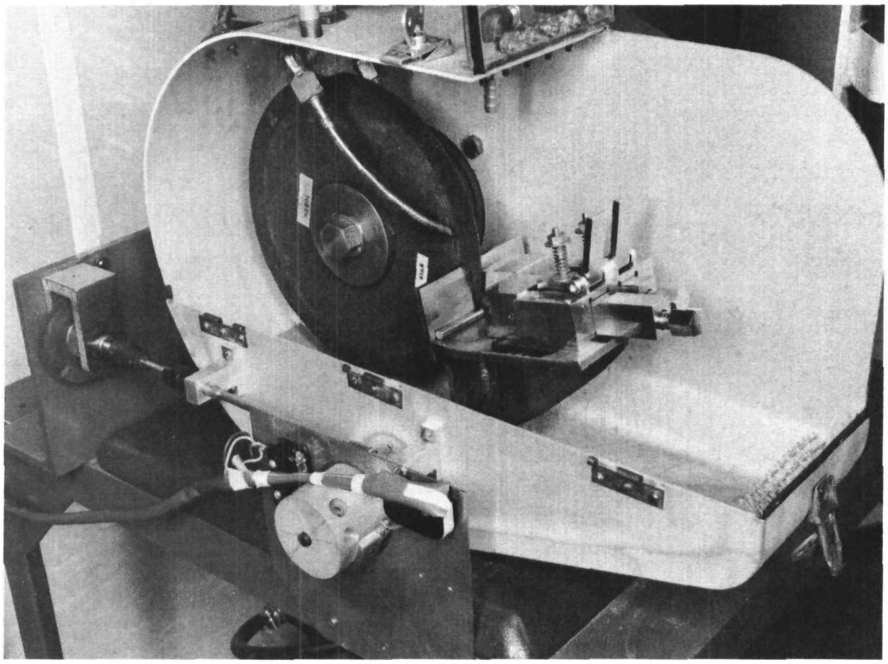

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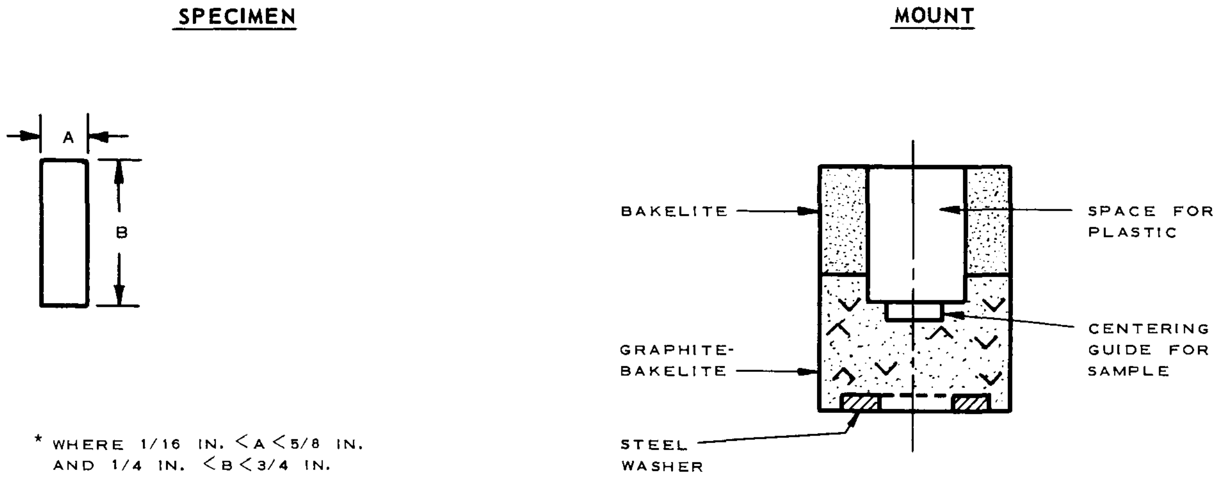
Fig. 6. Remotely Operated Abrasive Cutoff Machine in Operation

MOUNTING

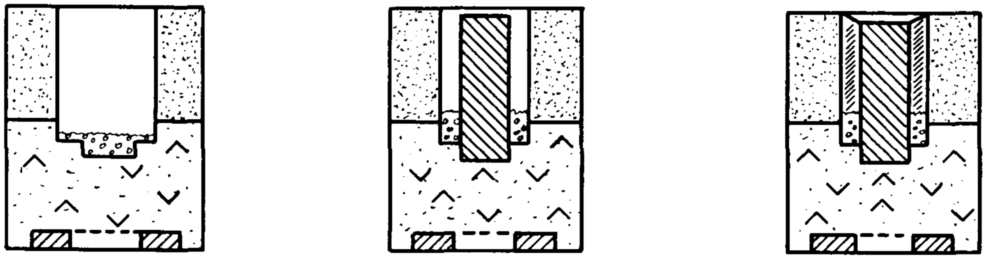
Specimens are mounted by either of two techniques, depending upon the physical characteristics and irradiation history. If the samples are slightly irradiated, are solid rather than porous, and are not embrittled, they are mounted in green Bakelite at 135°C to 145°C and at 5000 psi pressure in a modified Buehler Ltd., speed press.(1) A premarked steel washer is inserted in the back of the mount to facilitate identification and handling with magnetic devices. The necessary modifications to the speed press in order that its operation may be remotely controlled were slight. In fact, all that was necessary was to bring the thermocouple and heater leads out through a utility plug into a controller. The pressure is controlled by bringing the hydraulic leads through the same plug into a Blackhawk Bantam "Porta Power" hydraulic jack. The closing of the press and manipulation of the heating and cooling sinks are easily performed by the Argonne Model 8 manipulators.

The use of this machine, however, has been limited recently due to the large amounts of highly embrittled and porous specimens which are being encountered. Samples of this type are mounted in Hysol-6040 cold-setting plastic.(5) The techniques employed in cold mounting are illustrated in Figure 7. As may be seen from this figure, the time spent in mounting a large number of specimens would be considerably less than that encountered in using the speed press. This is realized because premolds may be made during the worker's free periods, and when a sufficient number of specimens are available, they may be all mounted in one operation during the latter part of the day, allowing the plastic to harden overnight. The mounting of specimens with the speed press would require the operator's constant attention.



1) DETERMINE DIMENSIONS (± 1/32 IN.) OF SPECIMEN.

2) PREPARE BAKELITE-GRAPHITE/BAKELITE (2/1) MOUNT INCORPORATING INDENTIFYING STEEL WASHER IN BOTTOM.



3) FILL MOLD WITH SMALL AMOUNT OF WOOD'S METAL AND INSERT IN CAVE

4) MELT WOOD'S METAL ON HOT PLATE...INSERT SPECIMEN...COOL.

5) FILL ANNULUS WITH HYSOL-6040 COLD-SETTING PLASTIC AND CURE AT ROOM TEMPERATURE FOR 24 HOURS.

106-5607

Fig. 7. Metallographic Mounting of Irradiated Materials

GRINDING

The mounted specimens are ground with a combination of the grinding equipment developed at Argonne(2) and new commercial equipment which has recently become available. The grinding machines previously used (Figure 8) gave excellent edge retention and preserved the flatness of the specimen. The main drawbacks of the operation were the requirement for processing one specimen at a time and the accessibility of many parts to contamination. These parts were not readily detached by remote means and presented difficulties because of radioactivity level whenever repairs were necessary. "S.M." automatic polishers (Figure 9) were substituted for the older machines because they possessed the following advantages: simplicity of design, accessibility of parts to remote maintenance, and simultaneous grinding of three specimens. In the operation of this machine, the grinding head sweeps the specimens across the surface of the lap in a motion approximating the arc of a circle. The passing of the holder over the center of the lap imparts a circulatory motion to the samples which eliminates unidirectional grinding and preserves the flatness of the mount.

106-2504

Fig. 8. Argonne Metallographic Polishing Machine

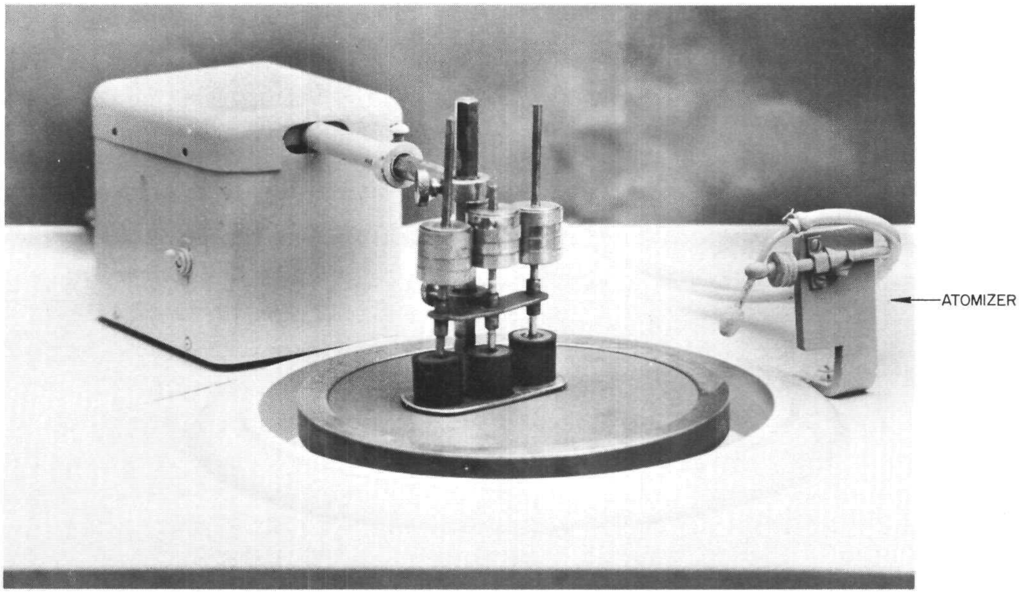


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Fig. 9. "S.M." Automatic Polishing Machine

Recently, however, the number of specimens have been cut from three to two. This is necessary because of an innovation in the application of weight to the mounted sample. In the present method, weight is applied to the mount by enclosing it in a brass holder in a manner similar to that described for the vibratory polishing technique.(6) The weight and specimen are then inserted into the sample holder of the machine and allowed to grind in the conventional manner. The specimens revolve as before; the method possesses the added advantage of maintaining the flatness of the specimen, even if the back of the mount should possess some irregularity which would cause tipping by the point of the weight applicator in the load attachment (see Figure 9). In addition to the above advantage, the machine is further simplified by eliminating additional contamination-prone parts and by speeding the grinding operation through the increase in applied weight. The amount of weight applied to the specimen may be varied by altering the size of the weight applicator. The weight is made of brass instead of stainless steel, because of ease of machinability. Moreover, the cost of cleaning such an accessory does not warrant making an expensive item, and therefore the entire piece is discarded after a certain period of time instead of decontaminated. The method of changing grinding papers remains unchanged from that originally devised,(2) except that the center pin which facilitated the alignment of papers on the turntable has been eliminated because of the necessity of passing the specimens over the center of the lap.

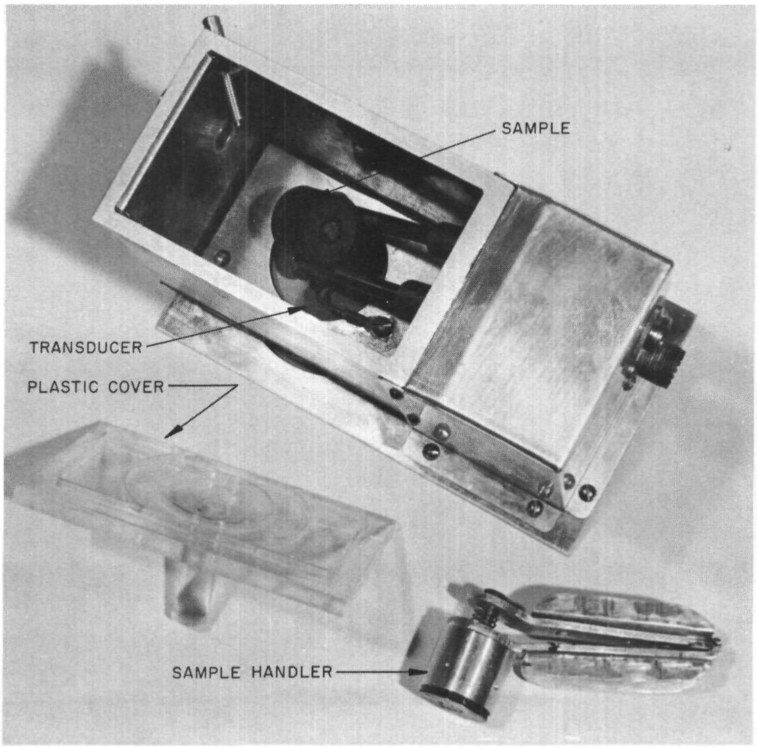
The grinding operation consists of positioning the mounted specimens in the holder of the "S.M." automatic polishing machine and grinding successively through 80-, 120-, 320-, and 600-grit SiC papers which are spray-lubricated with "OS" Hyprez fluid.(7) The time which the specimen remains on the 80-grit paper depends upon the depth of the deformed layer produced during the cutting operation, the hardness of the material, and the cross-sectional area. Therefore, the depth of the deformed layer is usually predetermined with an unirradiated specimen of the same material. The amount of material removed per unit time for particular groups of specimens is usually known from past operation; thus, it is easy to gauge when the necessary surface has been attained on the 80-grit grinding step. The remaining grinding operations are performed on each paper for a period of time dependent upon the amount of deformed layer produced during the preceding operation. The grinding waste adhering to the specimens is removed ultrasonically in carbon tetrachloride.

The grinding and polishing machines are protected from contamination by painting and enclosing them in plastic. This plastic, when necessary, is removed from the machines manually rather than remotely. The sample holder and load attachments can be removed remotely by means of the screws shown in Figure 9. Since these parts are of stainless steel, they are easily decontaminated. During this period, clean holders and load attachments may be substituted for the original parts. The grinding and polishing waste is concentrated in a plastic bowl beneath the grinding lap, thus preventing radioactivity from reaching the turntable motor. When decontamination is required, the liquid waste in the bowl (mostly "OS" Hyprez) is absorbed in vermiculite, and the bowl and contents are then discarded into a solid-active-waste container.

POLISHING

The polishing operations are performed with the same equipment described in the grinding section. The polishing cloths are changed remotely by inserting them into the cave in plastic bags and placing them on the polishing laps. The cloths are also lubricated with "OS" Hyprez fluid by means of an atomizer operated by compressed air, and the specimens are cleaned between succeeding steps in an ultrasonic washer containing carbon tetrachloride. Carbon tetrachloride was chosen as the cleaning solution because it is nonflammable, evaporates quite readily, and does not stain the specimen. The ultrasonic washer which was originally used for cleaning specimens was developed during a time when the technique was considered a novel approach to the cleaning of metallographic specimen surfaces. In order to attain the best possible cleaning conditions, many features were incorporated into the design which now, due to better transducers and associated equipment, are no longer necessary.

The operation of the instrument (Figure 10) originally consisted of placing the specimen on a holder facing the concave surface of a barium titanate crystal. The carbon tetrachloride solution was injected into the tank through a stainless steel solenoid valve. The necessary amount of bath to wash any specimen in the tank was gauged by running solution into a standpipe of a specific volume from which the solution was discharged into the cleaning tank. A small motor rotated the specimen to insure uniform cleaning action. When the necessary cleaning time was complete, another solenoid valve was opened and the bath drained out of the tank. As the tank discharged the cleaning solution, a jet of carbon tetrachloride was sprayed against the specimen to rid the surface of old solution. The spent carbon tetrachloride was allowed to drain into a settling tank from which it evaporated.

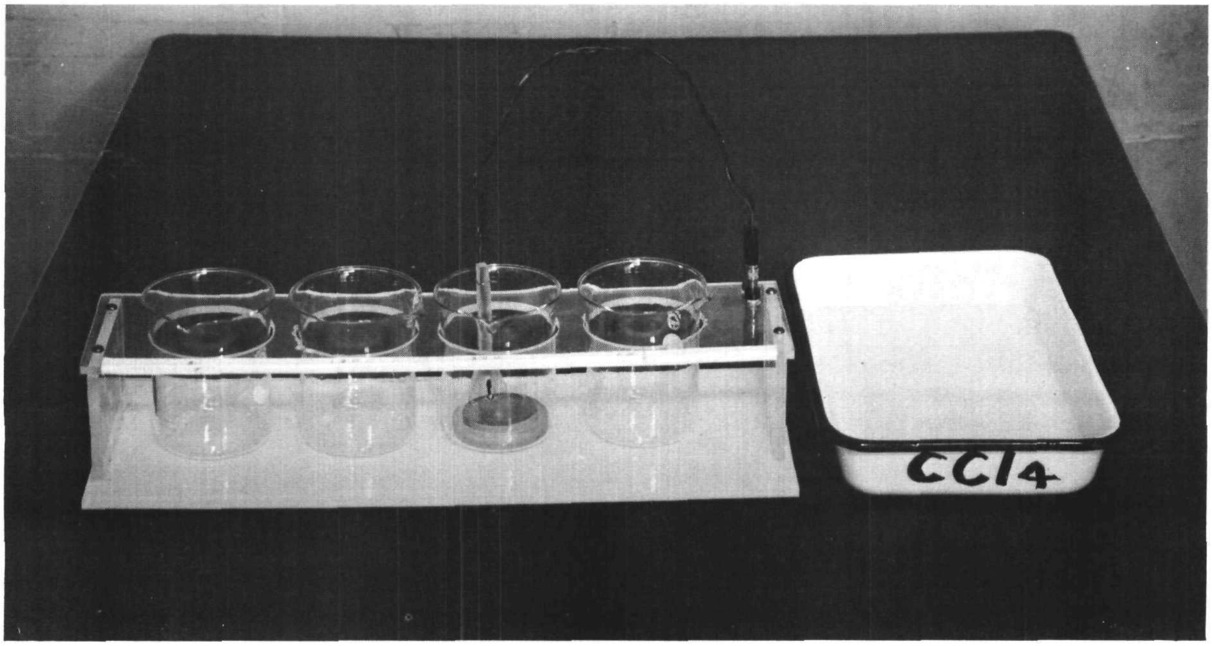


106-2008

Fig. 10. Ultrasonic Specimen Washer Adapted for Remote Operation

Although the specimens were cleaned quite thoroughly of polishing and grinding wastes, many difficulties were encountered because of failure of many of the components of the instrument. For instance, the solenoid valves became clogged, and the gasket maintaining a seal between the crystal and the stainless steel tank eventually leaked. In addition, the settling tank for the solution became highly radioactive, which made its disposal almost impossible. Repairs to the instrument were unduly prolonged owing to the buildup of activity on the crystal, valves, gasket, and tank.

In order to eliminate some of the bad features of the instrument, a new setup was devised in order to satisfy the following conditions: the same power supply and crystal would be used, components which tend to fail in service would be eliminated, the washer would be put above table level to facilitate repairs, and the settling tanks would be readily disposed of by remote means. The solution was relatively simple once it was determined that the cleaning power of the present barium titanate crystal was at a satisfactory level. Glass beakers, which clean rather easily compared with stainless steel, were chosen as the cleaning tanks. The same piezoelectric crystals are used except that they were made remotely removable from the solution (see Figure 11). The waste carbon tetrachloride is dumped by hand into porcelain trays from which the cleaning solution is allowed to evaporate. These trays are of such size that they can be readily discarded into conventional waste containers. The present system, although simple, works effectively because of the high cleaning power of the piezoelectric crystals, which were used in the previous system, and does not indicate that the many commercial units presently on the market might not satisfy the cleaning requirements more cheaply and conveniently.

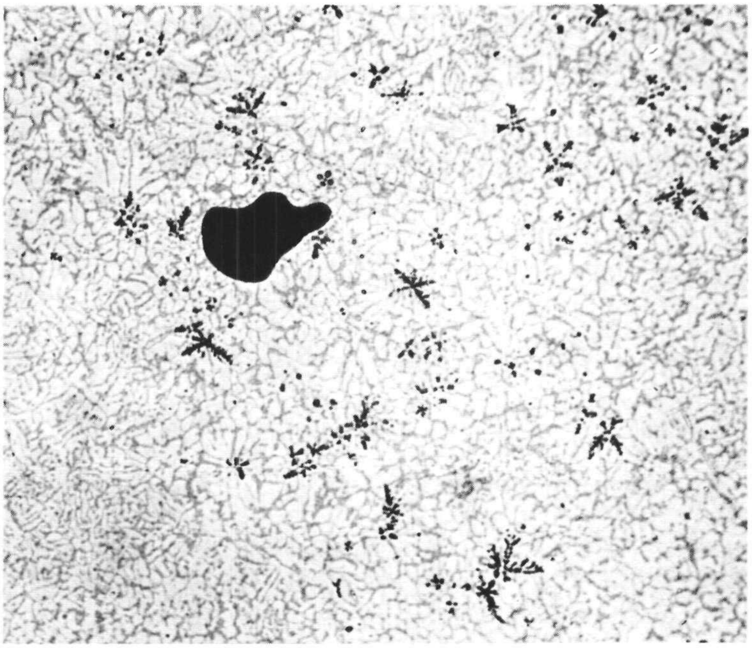


29734

Fig. 11. Modified Ultrasonic Washing Apparatus

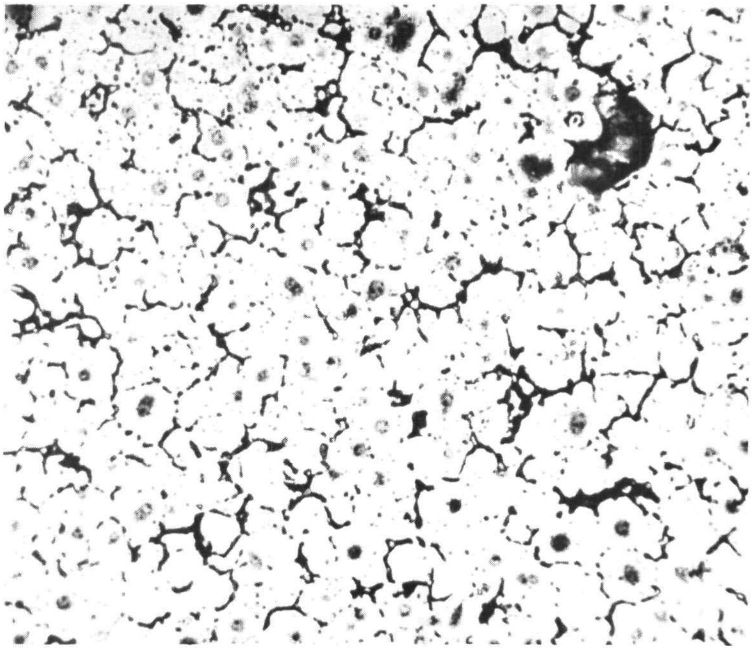
The same type of machines and brass laps which are used in grinding are used in the polishing operations. The polishing procedure is standardized for most of the materials, with variations in the final polishing step dependent upon the condition of the matrix or the physical properties of the alloy. Generally, the ground samples are polished, successively, with 3- and 1-micron diamond compound on silk cloth and with $\frac{1}{4}$-micron diamond abrasive on microcloth. If necessary, deformed metal is removed during the 1-micron polishing step by etching slightly and repolishing with 1-micron diamond compound on silk cloth. The final polishing steps, which have been worked out in advance of the examination, are then in accordance with the type of specimen being processed. The final procedures used for representative materials are given in the following paragraphs.

Thorium-Uranium Alloys: The thorium-uranium alloys processed to date have varied in composition from 10 to 28 w/o uranium and from 0.8 to 3.7 a/o burnup. Due to the large number of impurity particles in these alloys, it was necessary to prolong the final polishing step. The specimens were polished for one hour during the 1-micron step, and from three to four hours with $\frac{1}{4}$-micron diamond abrasive on microcloth. No etch-polishing steps were employed. The results of polishing several of these alloys are shown in Figures 12 and 13.



29207 250X

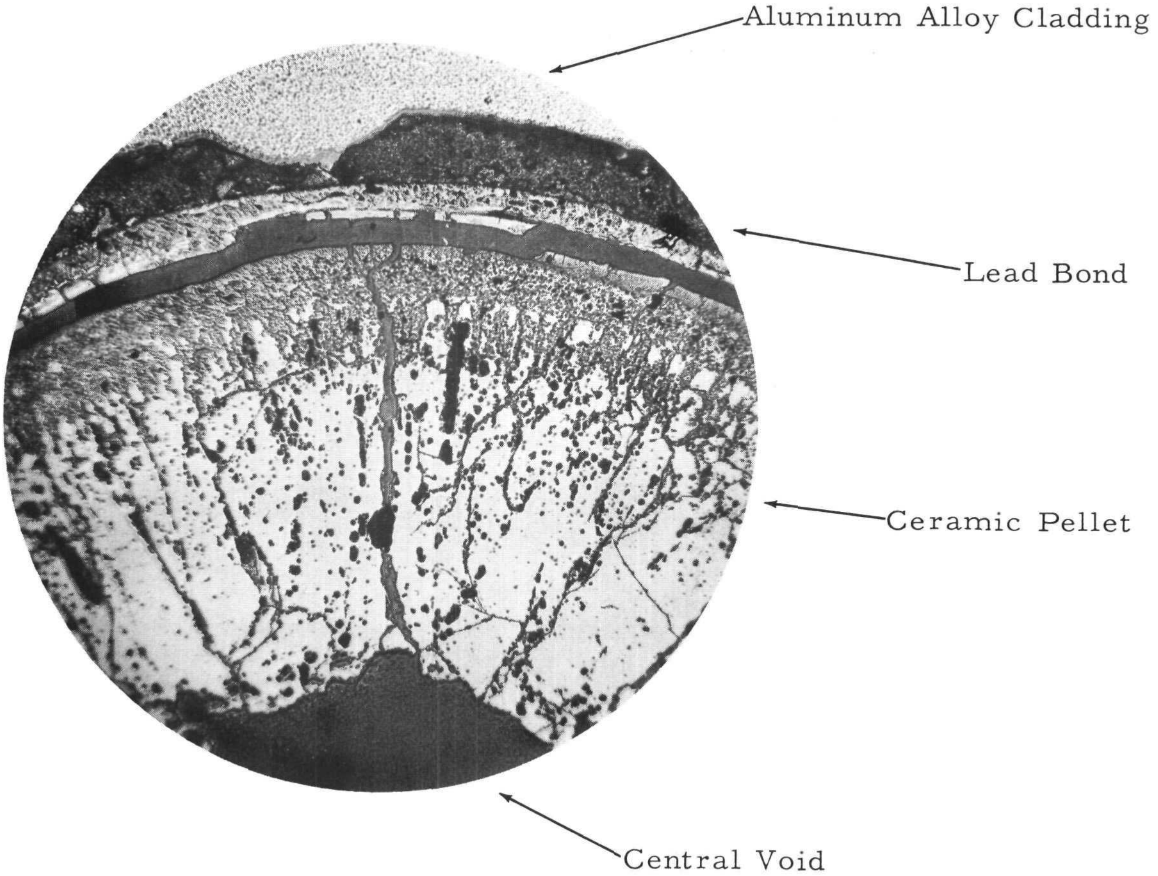
Fig. 12. Microstructure of Irradiated Thorium-25 w/o Uranium Alloy. As Polished



29213 250X

Fig. 13. Microstructure of Irradiated Thorium-15 w/o Uranium Alloy. As Polished

Thoria-Urania Pellets: The thoria-urania ceramics treated have been from the Borax-IV reactor program. They consist of sintered pellets of ThO_2-6.35 w/o UO_2 enclosed in aluminum tubes with lead as the bonding medium.(8,9) The mounting procedure outlined in Figure 7 was not used for this material. Instead, a 1-in.-diameter cylindrical mount of aluminum was prepared and laid on a glass slide. A small amount of cold-setting plastic was put into the cylinder and the ceramic specimen was inserted. The remainder of the mount was then filled with plastic. This procedure forced the plastic to flow into the cracks and central voids. If a void area appeared during the grinding operations into which plastic had not completely flowed, a small amount of plastic was put on the surface and forced into the void with a straight edge. The grinding operations were then completed. The specimens presented no difficulties during the polishing steps, even though the section consisted of a composite of three materials with wide variations in hardness. After one hour on the 1-micron cloth, the specimens were final polished with $\frac{1}{4}$-micron diamond abrasive on microcloth for one hour. The results of using these procedures are shown in Figures 14 and 15.



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35X

Fig. 14. Cross Section of Irradiated Aluminum Alloy-clad Lead-bonded ThO_2-6.35 w/o UO_2 Pellet. As Polished

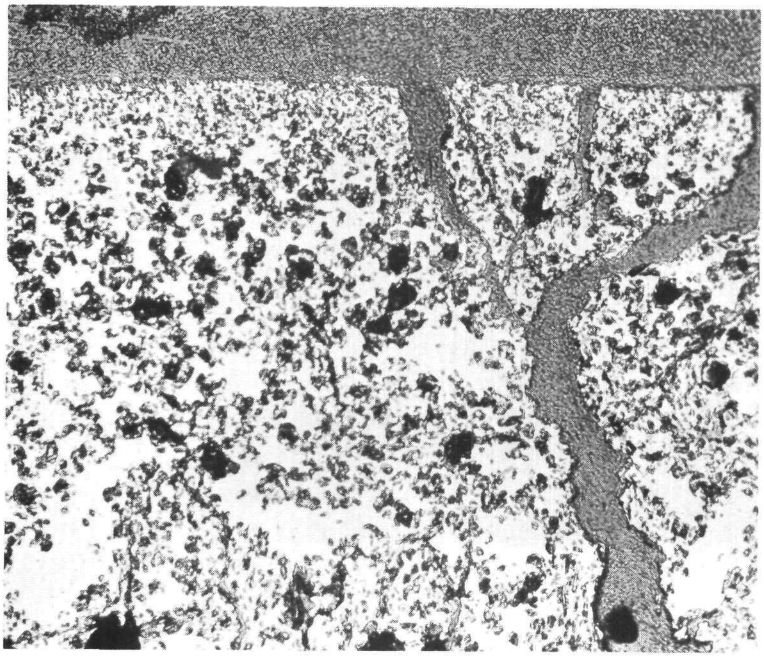


Fig. 15

Voids Appearing in Irradiated ThO_2-6.35 w/o UO_2 Pellet. Note retention of ceramic by mounting material. As Polished

29294

250X

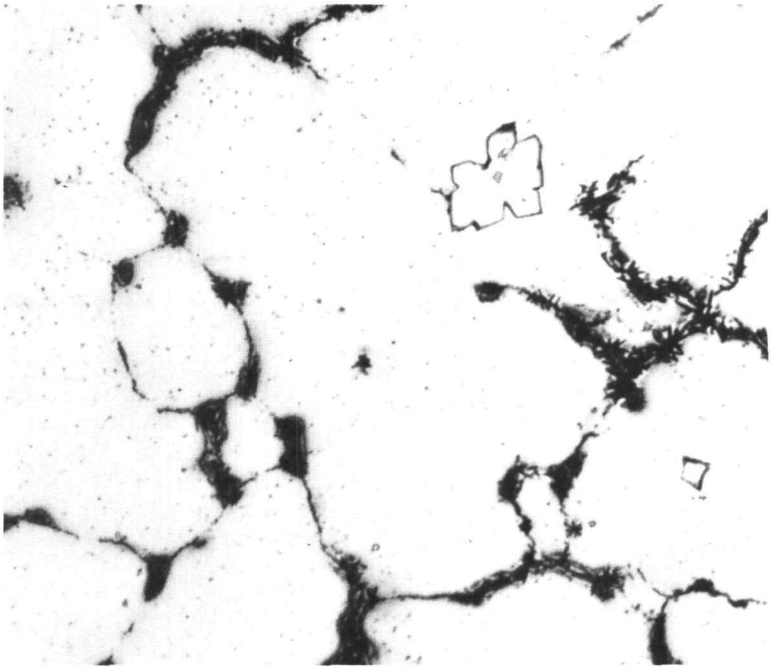
Uranium-Fissium Alloy: The uranium-5 w/o fissium alloy is the EBR-II Core I reference alloy. This material is injection cast and consists of particles of U_2Ru in a matrix of alpha and gamma uranium. Samples of this material are polished for one-half hour on the 1-micron cloth, etched 10 sec in the solution indicated in Table I, and repolished on 1-micron for one-half hour. The specimens are final polished with $\frac{1}{4}$-micron diamond abrasive on microcloth from one to two hours. Results of this procedure are shown in Figure 16.

Table I

ETCHANTS EMPLOYED IN THE METALLOGRAPHIC EXAMINATION OF IRRADIATED ALLOYS

Etchant Composition	Alloys	Time	Volts*
1 part (100 ml chromic acid in 100 ml of water) - 18 parts acetic acid	U-Mo	1-2 min	10
4 parts phosphoric acid - 5 parts ethyl alcohol - 4 parts ethylene glycol	U-Fs, Th-U, U-Zr	1-2 min	10
2 parts sulfuric acid - 1 part phosphoric acid - 2 parts water	Th-U	2 sec	10
10 parts nitric acid - 10 parts lactic acid - 1 part hydrofluoric acid	U-Zr	5-15 sec	Swab Etch
10 parts citric acid - 1 part nitric acid	U-Mo, U-Zr-Nb	1/2-1 min	15-20
100 parts water - 10 parts sodium hydroxide	Al-U	10-15 sec	Swab Etch
1 part (100 ml potassium dichromate, 2 ml sodium chloride, 10 ml sulfuric acid) - 9 parts water	Ag-In-Cd	10-15 sec	Swab Etch

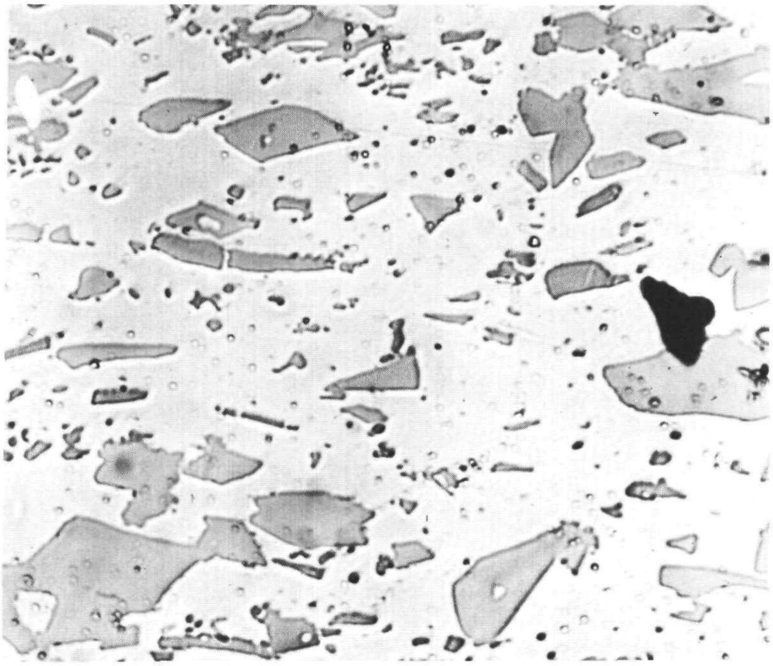
*Platinum anode and stainless steel cathode



27555 500X

Fig. 16. Uranium-5 w/o Fissium Alloy after Approximately Two-sec Irradiation at 1000°C. Etchant: 5 parts ethyl alcohol, 4 parts ethylene glycol, and 4 parts phosphoric acid.

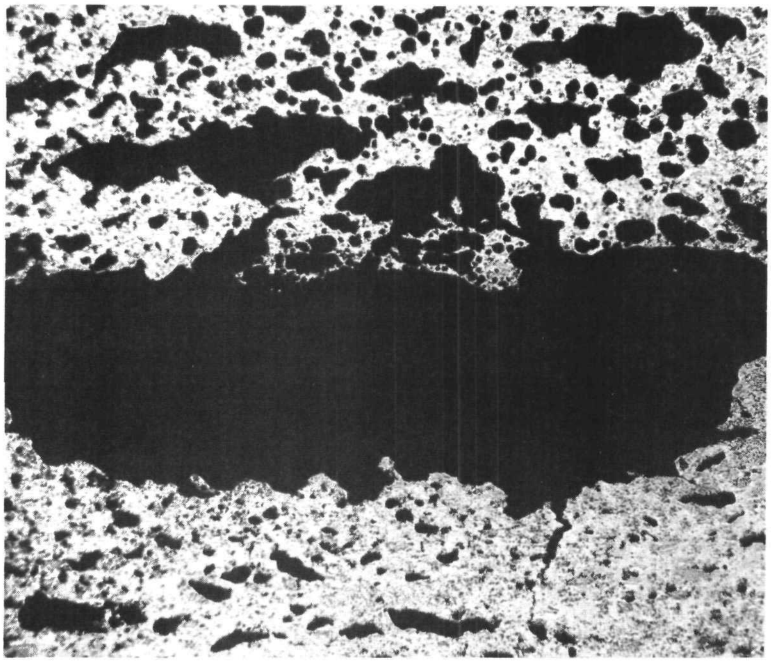
Aluminum-Uranium Alloy: An aluminum-17.4 w/o uranium-1 w/o nickel, clad with X-8001 aluminum alloy, was used as the fuel for the SL-1 reactor. The matrix contains various aluminum compounds and, hence, is difficult to remotely polish and obtain a scratch-free surface. Samples of this material are polished for one-half hour with 1-micron diamond abrasive on silk cloth, swab-etched with 10% NaOH for 10 sec, and repolished on the 1-micron cloth for one-half hour. The above procedure is once more repeated. The specimens are final polished from one to two hours on Selvyt cloth using AB Magomet compound. A disc of plexiglass is inserted between the cloth and the polishing lap to prevent corrosion of the aluminum. The results of using these polishing steps are illustrated in Figures 17 and 18.



28514 50X

Fig. 17. Microstructure of Irradiated Aluminum-17.4 w/o Uranium-1 w/o Nickel Alloy. As Polished

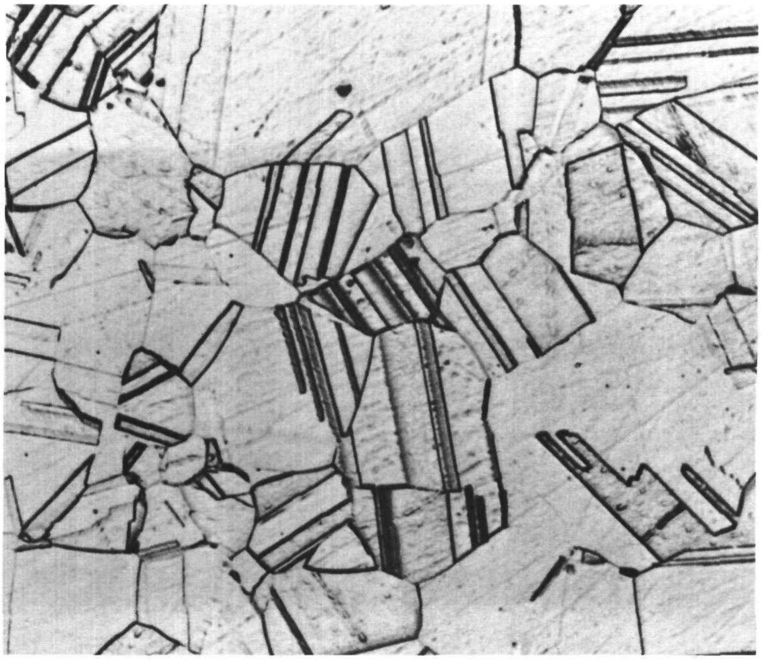
Silver-Indium-Cadmium Alloy: An 80 w/o silver-15 w/o indium-5 w/o cadmium alloy was used as an experimental control rod material. The alloy as prepared is a solid-solution alloy.(10) Irradiation, however, causes the precipitation of tin by the transmutation of indium, and thereby necessitates careful polishing procedures in order to retain these inclusions for identification. The procedure adopted consisted of several etch-polish steps to remove the large amount of flowed metal resulting from the extreme softness of the alloy. The samples are polished for one-half hour with 1-micron diamond compound on silk cloth, swab-etched for 2 sec with potassium dichromate solution (see Table I), and repolished on the 1-micron cloth. The above procedure is repeated. The samples are final polished for one-half hour with Linde A on silk cloth and for one-half hour with Linde B on silk velvet. The result of using this procedure is shown in Figure 19.



26535 50X

Fig. 18

Voids in Aluminum-18 w/o Uranium-1 w/o Nickel Alloy. As Polished.



28520 500X

Fig. 19

Microstructure of Irradiated 80 w/o Silver-15 w/o Indium-5 w/o Cadmium. Etchant: 1 part (100 cc $K_2Cr_2O_7$-2 cc NaCl-10 cc H_2SO_4) plus 9 parts water.

Uranium-Zirconium-Niobium Alloy: The majority of specimens examined in the metallographic facility have been from EBWR. The fuel in this reactor is uranium-5 w/o zirconium-1.5 w/o niobium alloy clad with Zircaloy-2. The preirradiation heat treatment for this material consists of a gamma soak at 825°C, an isothermal quench and holding at 640°C for 23 hr, followed by air cooling.(11) This material is polished in the conventional manner: 1 hr on silk cloth with 1-micron diamond abrasive, and 1 hr on microcloth using $\frac{1}{4}$-micron diamond compound. Results of an examination of this alloy with increasing burnup are shown in Figure 20.

ETCHING

Specimens are etched by the same methods used in conventional metallography although the electrochemical etch is most commonly employed. Electrolytic etching is performed with either a platinum touch wire or a back connection. The touch wire is used whenever the size of the specimen prohibits the obtaining of a back connection with the present mounting technique (see Figure 7). In either case, the specimen is etched in the required solution in a Pyrex dish containing a stainless steel cathode. The length of the etching period is predetermined with similar samples of unirradiated material. The etchants which are used for a variety of alloys are given in Table I.

The etching solutions, when exhausted, are put into a large plastic jar for storage. When the total solution reaches a specified level, the contents of the jar are neutralized with sodium hydroxide. This neutral solution is absorbed with vermiculite, and the jar and contents are then placed in a waste container.

EXAMINATION

The etched or unetched specimen is transferred and inserted into the metallograph transfer system shown in Figures 21 and 22. The original sequence of operations consisted of the following steps:

1. The stage was raised electrically to the top of its travel. (None of the transfer mechanism motions could be initiated until this point was reached.)

2. The sample was placed in the specimen holder and the "in" switch was activated.

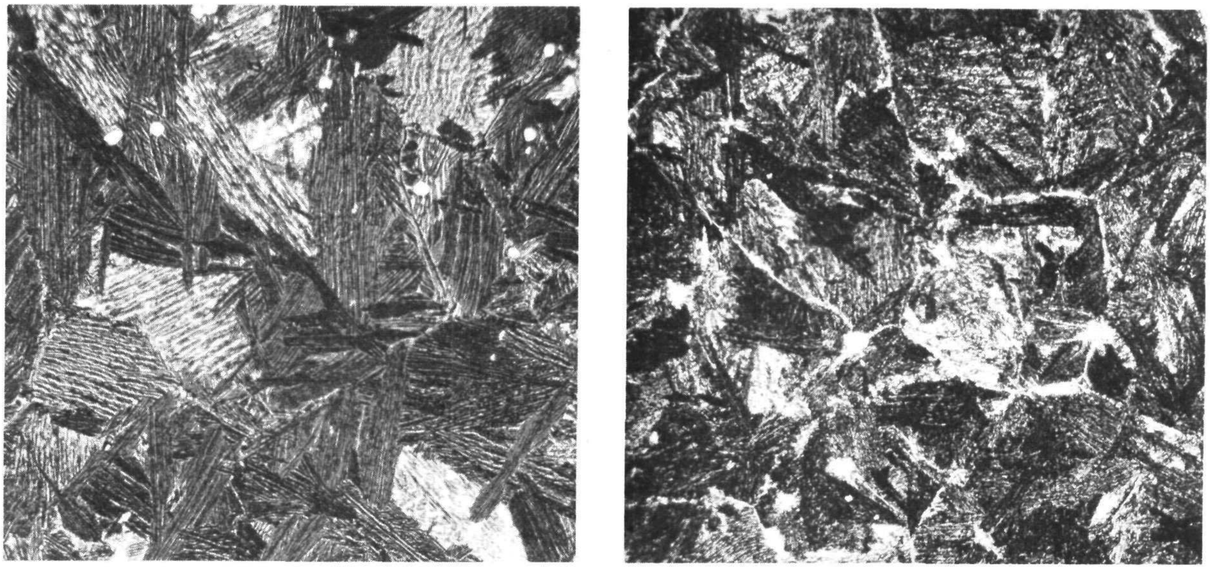
3. The specimen was shuttled through the cave wall into the shielded metallograph(3) outside the cell by means of a chain drive.

4. The specimen was inverted as the entire transfer system turned over and positioned the specimen into a hole in the microscopic stage.

5. A microswitch was activated automatically at this point to lower the stage so that the specimen dropped free of the cup.

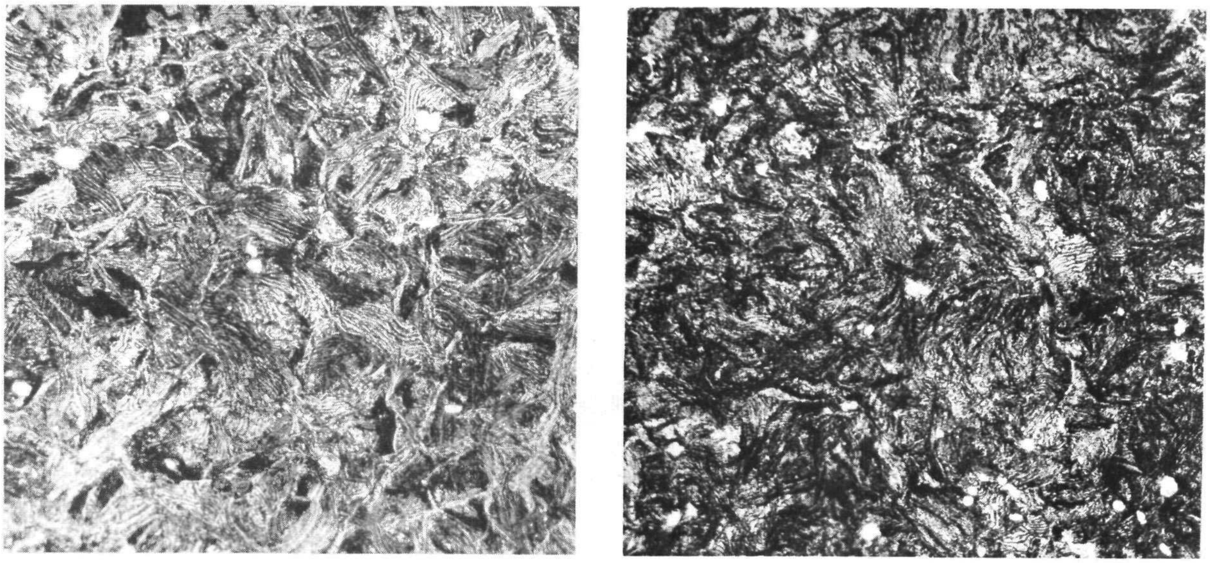
6. Another microswitch was activated by the stage, causing the transfer system to retreat to a point midway in the cave wall. (No new specimens could be inserted while the system was in this position.)

7. The specimen was removed by the reverse of this procedure.



25269 Unirradiated 250X
(a)

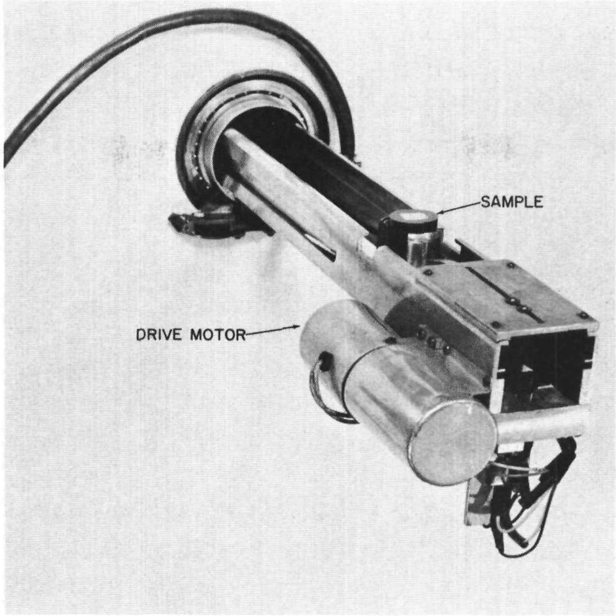
25003 0.017 a/o Burnup 250X
(b)



24883 0.088 a/o Burnup 250X
(c)

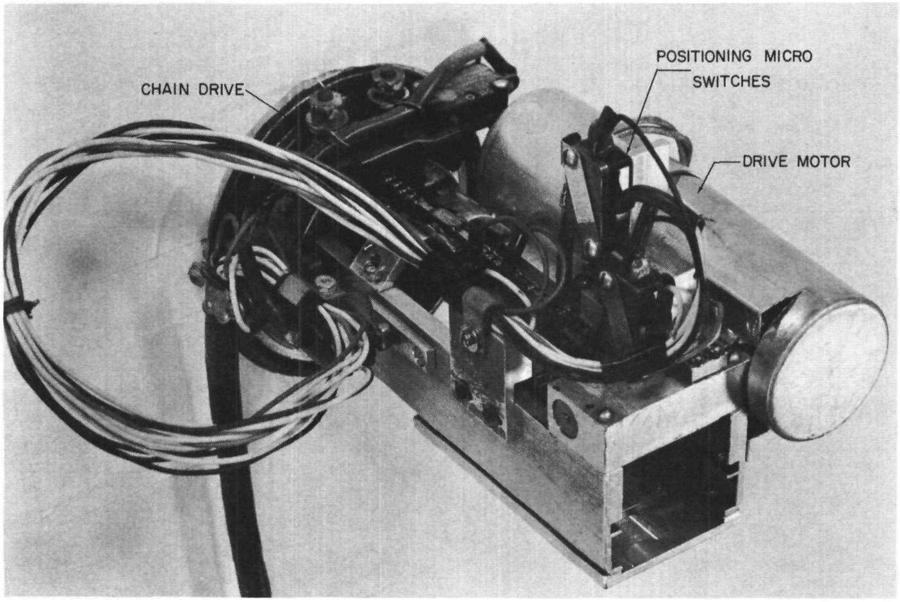
24292 0.11 a/o Burnup 250X
(d)

Fig. 20. Effect of Irradiation on Microstructure of Uranium-5 w/o Zirconium-1.5 w/o Niobium Alloy. Etchant: 5% Citric Acid, 0.5% Nitric Acid.



141-1001

Fig. 21. Specimen Transfer System



141-1003

Fig. 22. Transfer Mechanism in Inverted Position

The above system worked quite conveniently but, when one item failed, the determination of the cause for the malfunction usually involved lengthy periods of investigation, resulting in a breakdown of the most important portion of the metallographic investigation. In addition, the limit switches which controlled the operational sequences did not function adequately, and the readjustment of these switches relative to their original positions incurred many lengthy delays.

This transfer system was altered, therefore, to place more reliance on manual operation and to effect more positive insertion and removal of specimens. The present method illustrates only what has been done with the existing setup to date, and does not indicate that some more refined technique is not feasible. In fact, it may be considered as an intermediate development stage.

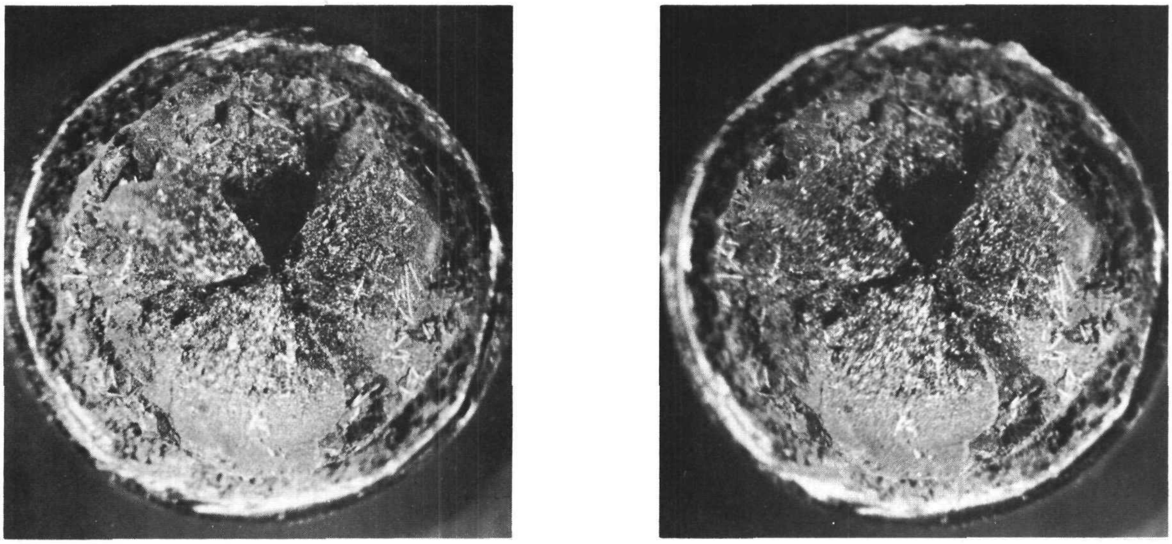
In the latest alteration, all electrical controls and limiting switches have been eliminated. The basic operational sequences have been retained except that the specimen is loaded onto a permanent magnet and cranked by hand through the cave wall into the shielded metallograph. The system is inverted and the specimen is positioned into the hole in the stage as before. The transfer system is withdrawn and the sample is easily detached from the magnet. The stage up-and-down motions are controlled by a two-way switch which is activated manually. The specimen is removed by the reverse of the procedure.

The period of time allowed for examination varies with each specimen because of the differences in oxidation rates of the polished surfaces. A particular specimen may oxidize while the examination is in progress. If this is the case, the examination must be completed before proceeding to the succeeding specimen. The microphotograph negatives of the desired fields are developed immediately before removing the specimen from the stage. When such a situation arises, the microscope objective is removed in order that browning of the lens does not occur. The specimens can be examined at magnifications ranging from 50 to 1500X.

MACROSCOPY

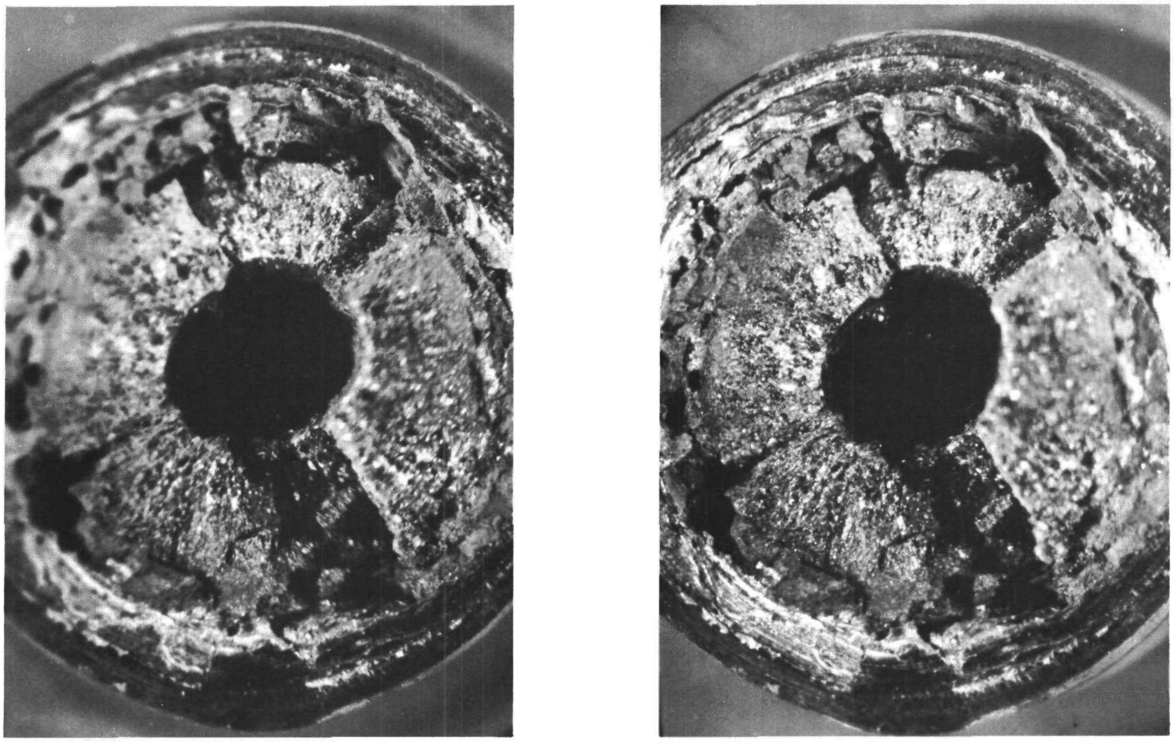
The basic objective of macroscopic examination is to detect possible defect areas, and, hence, to determine whether further microscopic investigation is warranted. A commercial stereobinocular microscope,(1) available for remote use and capable of magnifications from 4 to 38X, is generally used for scanning the samples, while an Argonne-developed macrocamera(1,12) with a magnification range of $\frac{1}{2}$ to 4X is available to obtain composite photographs of larger objects.

The variety of steps involved in preparing a group of specimens for examination is typified by a recent examination of some wire-reinforced ThO_2-UO_2 pellets enclosed in Zircaloy-2 tubing. Since the exact position of the pellets in the tubing could not be accurately determined by external appearance, autoradiographs were made by placing the assembly against a piece of X-ray film enclosed in protective plastic sheathing. The exposed films were then developed, and the position of each pellet in the tube was ascertained from the autoradiographs. The tubing was cut near the center of each pellet with a tubing cutter, with the pellet fracturing at the cut surface. The fractured pellets were loaded in a special holder designed to maintain the surface of the pellet level relative to the microscopic stage and examined. The results of the stereobinocular microscope examination are illustrated in Figures 23-25.



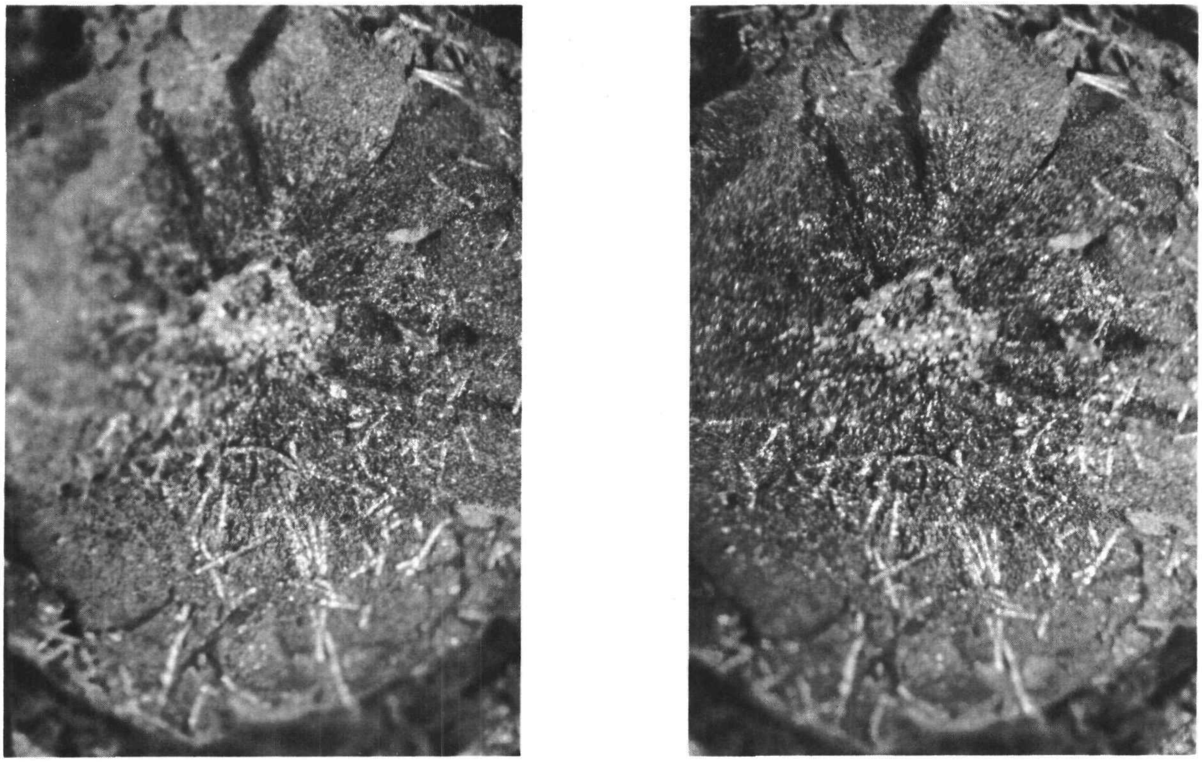
25730 4X

Fig. 23. Stereophotograph Showing Effect of Irradiation on Wire-reinforced ThO_2-UO_2 Pellet



25726 6X

Fig. 24. Stereophotograph Showing Effect of Irradiation on ThO_2-UO_2 Pellet



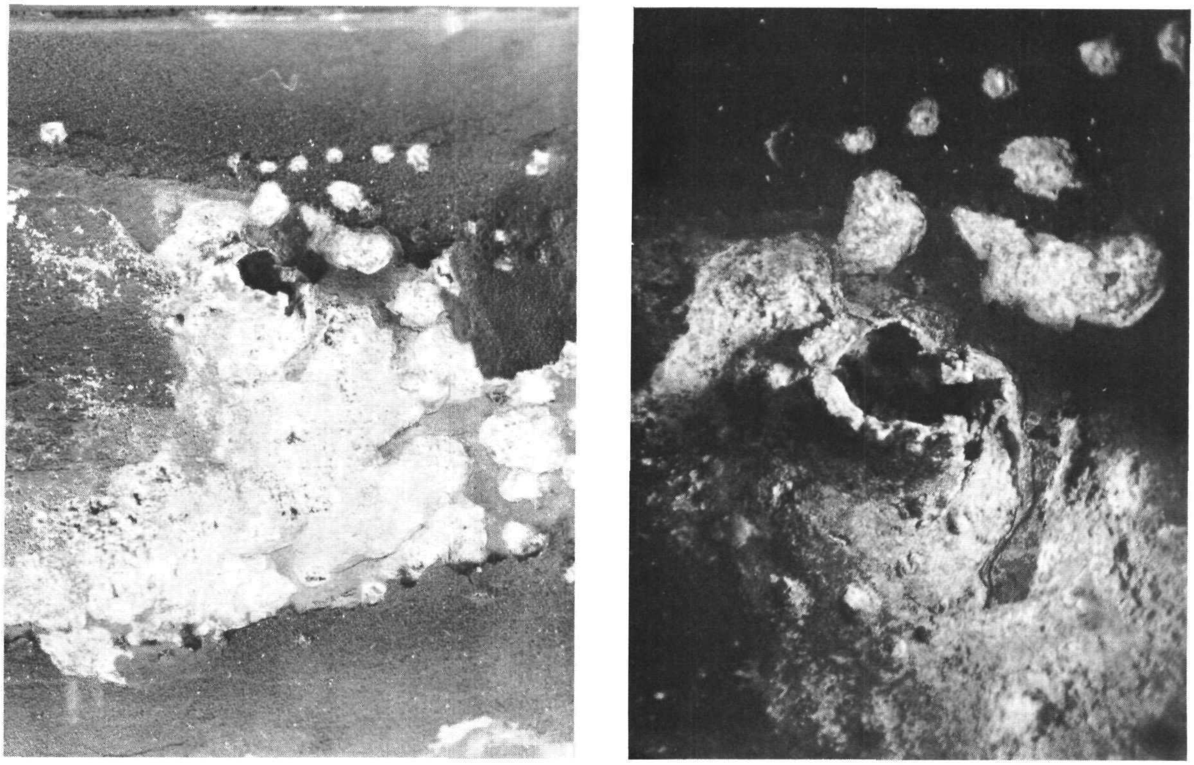
25728 13X

Fig. 25. Stereophotograph Showing Effect of Irradiation on Wire-reinforced ThO_2-UO_2 Pellet

The amount of preparation preliminary to using the macrocamera is not as involved as that requiring the utilization of the stereobinocular microscope. The samples are generally larger fuel plates or fuel rods which are loaded directly on the microscope stage and photographed. The results of the photography of an irradiated aluminum alloy-clad aluminum-uranium alloy fuel plate, utilizing both the stereobinocular microscope and the macrocamera, are shown in Figures 26 and 27.

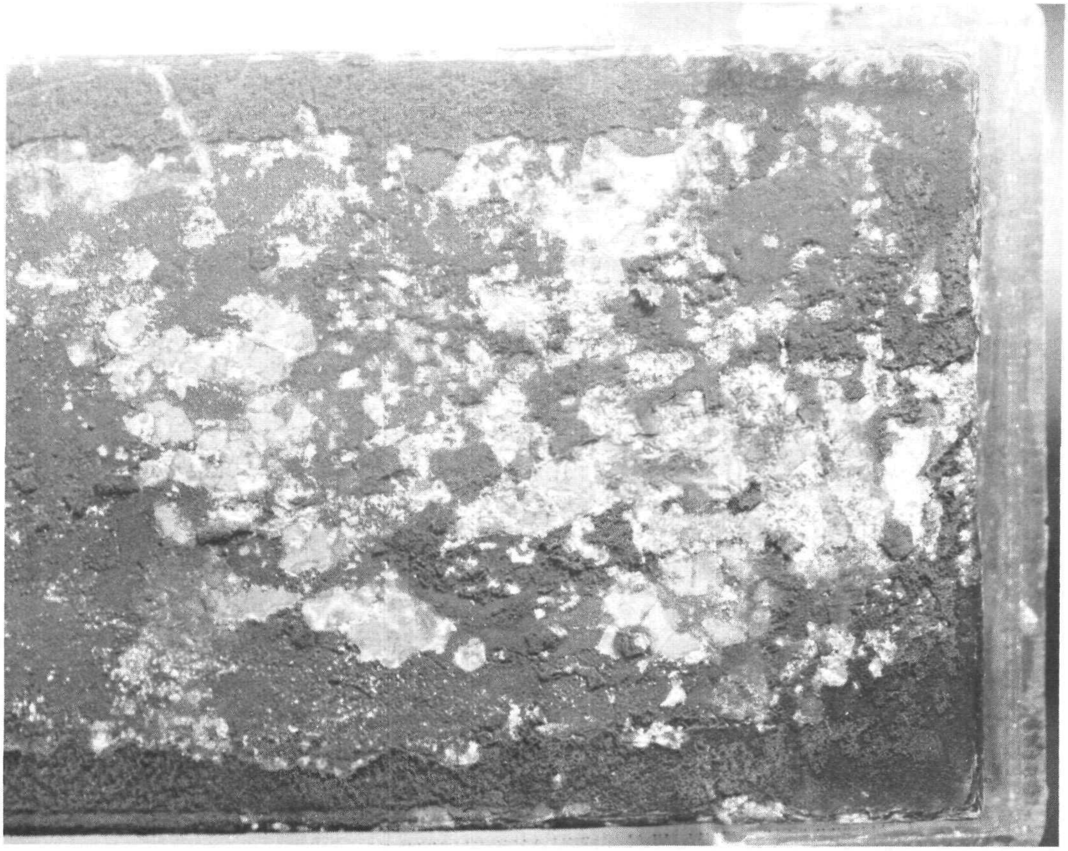
CONTAMINATION CONTROL AND MAINTENANCE

It was originally thought that decontamination and maintenance of the facility and associated equipment would permit operation of the facility for only about half the available time. This estimate was not far from wrong. Frequent repairs to equipment were necessary in accordance with the normal evolvement of new techniques. In addition, the control and disposal of radioactive waste presented various difficulties and exerted greater influence over the continuous operation of the facility than the equipment.



26111 A 2X 26122 B 4X

Fig. 26. Cladding Defect in Irradiated Aluminum Alloy-clad Aluminum-Uranium Alloy Fuel Plate as Pictured by Macrocamera (A) and by Stereobinocular Microscope (B).



26110 2X

Fig. 27. Macrophotograph Showing Spalling of Scale at Hot End (Right) of Irradiated Aluminum Alloy-clad Aluminum-Uranium Alloy Fuel Plate.

The original layout of the cave (see Figure 2) allowed for the segregation of equipment according to the relative release of radioactive waste. In agreement with this philosophy, the grinding and sectioning operations were confined to the left cell, the polishing and etching operations to the middle cell, and the optical instruments were placed in the right cell. Flow of air in the cave was directed from the optical cell to the grinding cell in order to protect the optical equipment and to prevent cross contamination in the polishing and grinding operations.

The grinding cell has presented the most difficulties regarding decontamination and repair in comparison to the other two cells. Although the containment of radioactive waste has been effective, the buildup of activity on parts of the grinding and sectioning machines has been of such extent as to prohibit the entry of personnel for maintenance. Whenever repairs were necessary, the entire cell operation had to be suspended, and decontamination procedures were initiated before restoration could be effected. In fact, the radiation background was so high that periods of from four to six weeks of inactivity were required for minor repairs to an instrument. These repairs were usually in the electrical circuits, which were not readily accessible to remote maintenance. In order, therefore, to allow continuous operation of the cave, the sectioning equipment has been moved to higher-level caves which have better accessibility for high-level decontamination. The sectioning operations are not intended to be excluded indefinitely from the metallography cave, but will be resumed once a system is developed to which alterations can be made remotely and which can be economically scrapped if required.

Probably the most important factor in providing sufficient contamination control is "good housekeeping." This can be made more effective by using white paint for the protection of equipment and handling devices. Not only does the white background create the impression of cleanliness, but it also facilitates the location of radioactive waste and serves as a light reflector for better viewing conditions. Items of equipment which have been protected from contamination by paint are cleaned before removal from the cell by remotely stripping the paint with a suitable solvent. Small handling devices which are not conveniently painted because of their frequent contact with solvents or acids are decontaminated ultrasonically in the same manner used for removing polishing and grinding waste from the metallographic specimens.

The manner in which the work benches are protected from contamination has undergone a considerable number of changes. Originally, bare tables of stainless steel were used for the handling of radioactive material, the decontamination of the surface being an easy matter through the use of rust remover. The cleaning of these surfaces, however, spread activity in the cell due to the transportation of fine particulate matter by the vapors from the acids and solvents.

The best combination for surface protection proved to be that of cheesecloth and Pyroxcote.(13) In this method, a thin layer of Pyroxcote is applied to the surface of the table with a paint brush, a sheet of cheesecloth is laid over this wet surface, and a second layer is applied to the top of the cheesecloth. Approximately $\frac{1}{2}$-hr drying time is allowed, after which a final layer of Pyroxcote is applied to the cheesecloth in order to make it impervious to liquids or radioactive waste. When it becomes necessary to clean the table, the cheesecloth-Pyroxcote combination is painted over remotely with a paint brush in order to fix any loose contamination. This protective surface is then easily stripped from the table and discarded into a solid-active-waste container.

ACKNOWLEDGMENTS

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