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MASTER

METALLOGRAPHY OF URANIUM:
PROCEDURES AND STANDARDS

BY

M. H. CORNETT

AEC RESEARCH AND DEVELOPMENT REPORT

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

FEED MATERIALS PRODUCTION CENTER
NATIONAL LEAD COMPANY OF OHIO

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METALLOGRAPHY OF URANIUM: PROCEDURES AND STANDARDS

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NOTICE

Readers wishing macrographs and micrographs of the highest resolution may obtain glossy photographs corresponding to the halftone reproductions in this manual. Five sets are available:

1. All photomacrographs, Figures 4 through 16.
2. All photomicrographs, Figures 17 through 26 and 35, 36, 38, 39, 40.
3. Color prints of the photomicrographs, Figures 27 through 34.
4. All standards and examples presented in Section 6 (grain-size, recrystallization, and inclusions), Figures 41 through 60.
5. All FEDC grain-size standards, Figures 61 through 64.

These photographs are available only in the sets listed above. All requests should be directed to the technical Librarian, National Lead Company of Ohio, Box 39158, Cincinnati, Ohio 45239. Prices will be furnished on request.

PREFACE

This manual presents techniques for the preparation of uranium specimens so that their macro- and microstructures may be studied. Included are procedures for sample preparation, etching, and photography. Also included are metallographic standards for the evaluation of the prepared samples with respect to grain size, recrystallization, and inclusion content. It is assumed that the reader is familiar with basic metallographic techniques. In general, these are not discussed.

The procedures and standards in this manual have, to a large extent, been developed empirically through the years to meet specific needs at the National Lead Company of Ohio. Some of them are based on earlier work at other AEC sites, and throughout this development there has been an exchange of information with metallographers at other sites. Their assistance is gratefully acknowledged. The metallographic standards presented in Section 6 have proved especially valuable here, since they provide a means for making uniform evaluations of grain structures and inclusion content by metallographers, even when working independently of one another.

Because the field of uranium metallography is comparatively new, little information has been published. It is hoped that this manual will serve as a practical, industry-wide guide for the preparation of uranium samples for metallography and for the evaluation of the metal structures revealed.

While this manual was in preparation, the AEC Fuel Element Development Committee (FEDC) adopted grain-size standards for the evaluation of beta heat-treated fuel cores. They are presented in the Appendix together with the procedures used for the preparation of their samples. A comparison of the two sets of standards (those of the FEDC and those developed by the National Lead Company of Ohio) will disclose the very close agreement between them — generally within one half a grain size. It will also be seen that the standards developed at this site cover a somewhat wider range of grain sizes.

CONTENTS

	Page No.
LIST OF FIGURES	vi
1. SAMPLE PREPARATION	1
1.1 Cutting	1
1.2 Grinding	1
1.3 Polishing	3
1.4 Etching and Pickling	3
1.5 Etching and Heat Tinting	3
2. PHOTOGRAPHY	5
2.1 Photomacrography	5
2.1 Photomicrography	6
3. MACROGRAPHIC PROCEDURES	9
3.1 Macrostructures of As - Cast, Beta Heat - Treated, and Extruded Uranium Ingot Slices	11
3.2 Macrostructures of Beta Heat - Treated, Water - Quenched; Beta Heat - Treated, Oil - Quenched; and Beta Heat - Treated, Oil - Quenched, and Extruded Uranium Fuel Cores	15
3.3 Macrostructure of Alpha - Extruded Uranium Fuel Cores Alloyed with Various Amounts of Iron, Aluminum, and Silicon Not Exceeding 250 ppm (Total)	17
3.4 Macrostructure of Alpha - Extruded Uranium Fuel Cores Alloyed with Various Amounts of Iron, Aluminum, and Silicon Exceeding 265 ppm	19
3.5 Metal Cleanliness: Overall Inclusion Content and Inclusion Segregation in Uranium Metal	21
3.6 Macrostructure of Uranium Metal Alloyed with 2 Weight Percent Zirconium	23
3.7 Macrostructure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum	25
3.8 Pickling Uranium Ingot Slices and Billet Slices	27
4. MICROGRAPHIC PROCEDURES	29
4.1 Inclusion Contents of As - Cast or Cast and Formed Uranium Metal	31
4.2 Grain Structures of As - Cast, Beta Heat - Treated, and Formed Uranium Metal	33
4.3 Grain Structure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum	35
4.4 Grain Structure of Uranium Metal Alloyed with 2 Weight Percent Zirconium	37
4.5 Grain Structure of Uranium Metal Alloyed with 0.18 Weight Percent Silicon	39
4.6 Precipitates in Alloyed Uranium Metal	41
4.7 Color Technique to Identify Inclusions in Uranium Metal	43
4.8 Grain Structures and Inclusion Contents of Uranium Metal (Alternate Method)	47
5. MISCELLANEOUS PROCEDURES FOR THE TREATMENT OF URANIUM AND THORIUM METALS.	49
5.1 Cleaning Uranium Metal for Inspection or Display	51
5.2 Primary Inclusions, Secondary Inclusions, Precipitates, and Grain Structures of Thorium Metal	53
6. METALLOGRAPHIC STANDARDS USED AT THE NATIONAL LEAD COMPANY OF OHIO	57
6.1 Grain - Size Standards for Beta Heat - Treated Uranium Fuel Cores	59
6.2 Recrystallization Standards for Formed Uranium Metal	65
6.3 Standards to Evaluate Random and Clustered Inclusions in As - Cast Uranium Metal	67

CONTENTS (Continued)

	Page No.
6.4 Standards to Evaluate Random Inclusions in Formed Uranium Metal	75
6.5 Standards to Evaluate Stringers of Inclusions in Formed Uranium Metal	77
7. APPENDIX	83
7.1 Uranium Grain Size Standards of the Fuel Element Development Committee (FEDC) .	83
7.2 FEDC Procedures for Sample Preparation	85

LIST OF FIGURES

Figure No.		Page No.
1	Hudorkut Cutting Machine	2
2	Deardorff 8 × 10 View Camera	6
3	Bausch and Lomb 5 × 7 Macro Camera	7
4	Macrostructure of As-Cast Ingot Slice	12
5	Macrostructure of Cast, Beta Heat-Treated, and Water-Quenched Ingot Slice	13
6	Macrostructure of Cast, Beta Heat-Treated, and Alpha-Extruded Ingot Slice	14
7	Macrostructure of Beta Heat-Treated, Water-Quenched, Uranium Fuel Core	15
8	Macrostructure of Beta Heat-Treated, Oil-Quenched, Tubular Uranium Fuel Core	16
9	Macrostructure of Beta Heat-Treated, Oil-Quenched, and Extruded Tubular Uranium Fuel Core	16
10	Macrostructure of Alpha-Extruded Uranium Fuel Core Alloyed with Fe, Al, and Si in Amounts Not Exceeding 250 ppm, Total	17
11	Macrostructure of Alpha-Extruded Uranium Fuel Core Containing 900 ppm (Total) of Fe, Al, and Si	19
12	Overall Inclusion Content in Cast Uranium Metal	22
13	Inclusion Segregation in Cast Uranium Metal	22
14	Macrostructure of Uranium Metal Alloyed with 2 Weight Percent Zirconium	23
15	Macrostructure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum	25
16	Pickled Uranium Ingot Slice	27
17	Inclusion Content of As-Cast Uranium Metal	32
18	Inclusion Content of Formed Uranium Metal	32
19	Grain Structure of As-Cast Uranium Metal	33
20	Grain Structure of Wrought and Beta Heat-Treated Uranium Metal	34
21	Grain Structure of Formed Uranium Metal	34
22	Grain Structure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum	35
23	Grain Structure of Uranium Metal Alloyed with 2 Weight Percent Zirconium	37
24	Grain Structure of Uranium Alloyed with 0.18 Weight Percent Silicon	39
25	Precipitate Dispersion in Uranium Ingot Containing 125 ppm Fe and 80 ppm Si	42
26	Precipitate Dispersion in Uranium Ingot Containing 1000 ppm Fe and 1000 ppm Al	42
27	(1) Uranium Carbide (UC), Orange-Red; (2) Uranium Carbonitride (UCN), Yellow	44
28	(1) Uranium Carbide System, Orange-Red; (2) Uranium Phosphide (UP), White-Yellow	45
29	(1) Uranium Carbide, Orange-Red; (2) Uranium Phosphide, White-Yellow. Note Precipitate in Grain Boundary.	45
30	(1) Stringer of Uranium Carbide, Orange-Red; (2) Uranium Phosphide, White-Yellow	45
31	(1) Uranium Carbide with Center Etched Out, Orange-Red; (2) Chain-Like Precipitate of U_6Fe , Blue; (3) These Inclusions are Thought to be UC.	45
32	Uranium Hydride (UH_3). Note Silver Halo Effect. Inside Section is Faintly Multicolored.	46
33	Uranium Carbide, Orange-Red. Note that Center of Carbide is Etched Out.	46
34	Uranium Carbide, Orange-Red	46

LIST OF FIGURES (Continued)

Figure No.		Page No.
35	Grain Structure of Beta Heat - Treated Uranium Metal	48
36	Inclusion Content of As - Cast Uranium Metal	48
37	Uranium Metal Cleaned for Display	51
38	Primary Inclusions in As - Cast Thorium Metal	54
39	Secondary Inclusions and Precipitates in Wrought Thorium Metal	54
40	Grain Structure of Wrought Thorium Metal	55
41	Grain - Size Standards for Rolled, Beta Heat - Treated, Water - Quenched, Uranium Fuel Cores	60
42	Grain - Size Standards for Rolled, Beta Heat - Treated, Oil - Quenched, Uranium Fuel Cores	61
43	Grain - Size Standards for Extruded, Beta Heat - Treated, Oil - Quenched, Uranium Fuel Cores	62
44	Grain - Size Standards for Rolled, Beta Heat - Treated, Water - Quenched, Uranium Fuel Cores	63
45	Recrystallization Standards for Formed Uranium Metal	66
46	Standards for Random Inclusions in As - Cast Uranium Metal	69
47	Standards for Clustered Inclusions in As - Cast Uranium Metal	70
48	Magnaviewer with Grid (4 - Inch Square Divided into ½ - Inch Squares	71
49	Random and Clustered Inclusion Rating of As - Cast Uranium	72
50	Random and Clustered Inclusion Rating of As - Cast Uranium	72
51	Random and Clustered Inclusion Rating of As - Cast Uranium	73
52	Random and Clustered Inclusion Rating of As - Cast Uranium	73
53	Random and Clustered Inclusion Rating of As - Cast Uranium	74
54	Standards for Random Inclusions in Formed Uranium Metal	76
55	Standards for Stringers of Inclusions in Formed Uranium Metal	78
56	Inclusion and Stringer Rating of Formed Uranium	79
57	Inclusion and Stringer Rating of Formed Uranium	79
58	Inclusion and Stringer Rating of Formed Uranium	80
59	Inclusion and Stringer Rating of Formed Uranium	80
60	Inclusion and Stringer Rating of Formed Uranium	81
61	Grain - Size Standards for Rolled, Salt - Heated, Water - Quenched, Uranium Fuel Cores	86
62	Grain - Size Standards for Rolled, Bronze - Heated, Tin - Quenched, Uranium Fuel Cores	87
63	Grain - Size Standards for Rolled, Salt - Heated, Oil - Quenched, Uranium Fuel Cores	88
64	Grain - Size Standards for Extruded, Salt - Heated, Oil - Quenched, Uranium Fuel Cores	89

1. SAMPLE PREPARATION

The first step in any metallographic technique, after the sample has been selected, is surface preparation. This first step is most important, for any information obtained thereafter depends upon the condition of the sample surface before it is etched. At the National Lead Company of Ohio, samples are taken from all phases of our uranium production, that is, from ingots, billets, rods, and cores. The sections that follow contain procedures for the preparation of all types of samples commonly examined.

1.1 CUTTING

1.1.1 For examination at 1X magnification, the samples (usually ingot slices) are cut by hacksaw or circular saw and are usually machined to obtain a flat surface. The resulting specimen is approximately 1 inch thick and 5½ to 13 inches in diameter. Both transverse and longitudinal sections are taken.

1.1.2 For metallographic work at magnifications of 3X or higher, uranium, usually in the form of rods or cores, is cut on a Campbell Hudorkut machine (Figure 1) using an Allison abrasive wheel (No. C120-PRAG6). The samples for macrometallography, both longitudinal and transverse, are cut from 1 to 2 inches long. Micrometallographic samples, both longitudinal and transverse, are cut at various lengths up to 4 inches. (The maximum length is limited by the capacity of the cutting machine.)

NOTE: It is important during all cutting operations that the work-piece be submerged in coolant to avoid overheating at the cutting point. This could cause changes in grain structure that would give a misleading picture of the condition of the metal.

1.2 GRINDING

1.2.1 Macrography

Large ingot slices to be photographed at 1X magnification are too large to grind and therefore are etched in the as-sawed or as-machined conditions. Machined surfaces of course, give better results, but, if etched deeply enough, as-sawed surfaces are adequate.

Samples for 3X magnification are rough-ground unmounted on a standard grinding wheel operating at 1150 rpm. Sixty-grit silicon carbide paper is used; running water is the coolant. The samples are ground until all saw marks and metal distortions are removed. They are then washed successively in water and absolute alcohol and dried in air.

1.2.2. Micrography

Samples are ground unmounted on a standard two-speed grinding wheel running at 1150 rpm. Sixty-grit silicon carbide paper is used in the rough-grinding step to remove saw marks and metal distortions. The sample is washed successively in water and absolute alcohol and dried in air. To remove the grinding marks of the 60-grit paper, the sample is then ground with 400-grit silicon carbide paper. Again the sample is washed in water and absolute alcohol

FIGURE 1 Hudorkut Cutting Machine

and dried in air. For the best photographic results, it is important that the sample be perfectly flat from edge to edge.

1.3 POLISHING

The polishing techniques for uranium vary considerably, depending on the type of metallographic examination planned; therefore, these techniques are described with the specific metallographic procedures to which they pertain.

1.4 ETCHING AND PICKLING

1.4.1 Concentrated HCl (Reagent Grade) has been found to be the most suitable etching reagent for uranium because of its rapid reaction. However, it is important to note that all etching times depend on the strength of the acid bath, the number of pieces being etched, the temperature of the bath, and the sample surface. Uranium reacts less rapidly with HCl which has been in use for some time because the acid deteriorates. But, on the other hand, acid in use is heated by the exothermic reaction and this tends to increase the rate of reaction. For these reasons, it is not possible to state definite, unvarying etching times. *In the procedures that follow, the etching times given will be those recommended for a fresh acid bath and for the treatment of only one sample.* The metallographer will have to adjust the etching times thereafter according to his own circumstances.

1.4.2 After the sample has been immersed in HCl and washed in water, it is then washed with concentrated HNO₃ (Reagent Grade) to remove the black oxide film and show the grains in contrast. Again, it is difficult to state how long the sample should be in contact with the acid. If the sample is removed too soon, not enough of the black oxide film will be removed, and the sample will be too dark. Conversely, if the sample is not removed from the acid bath soon enough, it will be so light that the grains will not be visible. Therefore, the metallographer is obliged to determine by trial and error when the sample has the best contrast for its intended purpose.

1.4.3 Pickling is used for evaluating macrocracks and macroporosity. It merely involves cleaning the sample surface with concentrated HNO₃ (Reagent Grade) and inspecting it for irregularities.

1.5 ETCHING AND HEAT TINTING

By using an aqueous-organic acid etchant on uranium metal samples, it was observed that a specific color could be produced on uranium carbide inclusions. Other inclusions present were not affected by the etchant; however, after heat tinting the samples, specific colors did appear on other inclusions. The etchants found most effective for producing colors are the following:

<u>Etchant</u>	<u>Chemical Composition</u>	<u>Remarks</u>
A	Equal volumes of: (1) Concentrated HNO ₃ (2) Glacial Acetic Acid (3) H ₂ O	Solution becomes exhausted after ½ hour. Must be made fresh for each use.

- B Equal volumes of:
(1) Concentrated HNO_3	Solution is unstable above 25°C . Small quantities
(2) 88% Formic Acid	must be used and then discarded.
(3) H_2O	

CAUTION: Because of the instability of Etchant B, the following precautions should be taken:

- (a) Use eye protection.
- (b) Do not use more than 100 ml of solution at one time.
- (c) Mix the H_2O and the HNO_3 , cool the solution to 25°C , and then add the formic acid.
- (d) After using the solution, discard it immediately and flush the sink with large quantities of water.

2. PHOTOGRAPHY

Photography associated with metallography is not complex; however, here, as in the etching step, it is difficult to establish rigid procedures. Evaluating photographs is at least partly subjective. The metallographer is the best judge of the acceptability of a photograph, and he can determine the photographs that are best suited for revealing grains or inclusions. Since opinions vary markedly in this area, and especially in the matter of print contrast, the following photographic procedures should be regarded only as guides. Included are those most frequently used at the National Lead Company of Ohio.

2.1 PHOTOMACROGRAPHY

2.1.1 1X Magnification

Camera:	Deardorff 8 × 10 view (Figure 2) or Bausch and Lomb, Model L, 5 × 7 macro (Figure 3)
Lens:	14-inch Commercial Ektar or 158-mm Macro Tessar
Lights:	Four 300-watt floods. Use white background
Film:	Kodak Royal Pan
Film Exposure Time:	Approximately 2 seconds at f22
Film Developer:	DK 50
Film Developing Time:	5½ minutes in tank at 68 °F
Printer:	Morse contact
Print Paper:	Kodak Illustrators' AZO F-3
Paper Exposure Time:	Approximately 0.7 second
Paper Developer:	Kodak Dektal
Paper Developing Time:	60 to 90 seconds

NOTE: All samples are photographed wet.

2.1.2 3X to 20X Magnification

*Camera:	Bausch and Lomb, Model L, 5 × 7 Macro (Figure 3)
*Lens:	158-mm Macro Tessar
Lights:	500-watt flood lights at 45° angle (permanent setup), Powerstat controlled for Brockway meter reading of 30,000 foot candles at sample
*Film:	Kodak Royal Pan
*Film Exposure Time:	1 second at f16
*Film Developer:	DK 50
Film Developing Time:	5½ minutes in tank at 68 °F
Printer:	Morse Contact
*Print Paper:	Kodak Illustrators' AZO F-3
Paper Exposure Time:	Approximately 0.7 second
Paper Developer:	Kodak Dektal
Paper Developing Time:	60 to 90 seconds

NOTE: All samples are photographed wet.

* These items are now recommended by the FEDC (AEC Fuel Element Development Committee) for sample rating.

FIGURE 2 Deardorff 8 × 10 View Camera

2.2 PHOTOMICROGRAPHY

Three types of illumination are used for making photomicrographs:

- (a) Polarized light for grain structures.
- (b) Bright field illumination for inclusions.
- (c) Dark field illumination for precipitates.

Each form of illumination should be obtained as described in the owner's manual for the particular instrument.

2.2.1 Black-and-White Photomicrography

Photographs should be made with the metallograph using the appropriate lenses to obtain the desired magnification according to the instructions furnished with the instrument. The remaining procedures are as follows:

Film:	Kodak Royal Pan, 5 × 7 inches
Film Exposure Time:	
Polarized Light	30 seconds
Bright Field	$\frac{1}{50}$ second
Dark Field	1 second
Film Developer:	DK 50
Film Developing Time:	5½ minutes at 68°F
Printer:	Morse Contact

Print Paper: Kodak Illustrators' AZO F2, 3, 4, or 5*
Paper Exposure Time: Approximately 1 second
Paper Developer: Kodak Dektal
Paper Developing Time: 60 to 90 seconds

2.2.2 Color Photomicrography

Successful photomicrography in color requires close control of the color temperature of the light source in addition to correct exposure. Color temperature is expressed in degrees Kelvin.

When using the white-flame carbon arc illuminator and daylight-type color film, a color correction must be made. The approximate color temperature of the white-flame arc, using a current of 10 amperes, is 3800 °K, whereas most daylight color film and Polacolor are rated at approximately 6000 °K. Since the white-flame arc contains less blue than sunlight, a Wratten 82C filter must be inserted in the optical system of the metallograph to add blue and thus balance the color. When Ektachrome Type B film (3200 °K) is used, a yellow filter, Wratten 81C, must be used.

It is also recommended that a 1% sodium nitrate solution be used in the water cell of the metallograph to filter out ultraviolet rays.

The exposure times for color photography will vary with the type of film used, but it has been found that exposures in excess of 30 seconds should be avoided because of the risk of reciprocity failure.

* Paper grade depends upon the contrast of the negative.

FIGURE 3 Bausch and Lomb 5 × 7 Macro Camera

3. MACROGRAPHIC PROCEDURES

The macrometallographic procedures that follow are those most frequently employed in the evaluation of uranium at this site. They include 1X, 3X, and 10X magnifications.

Procedure 3.1 Macrostructures of As-Cast, Beta Heat-Treated, and Extruded Uranium Ingot Slices

Sample Preparation

See Sections 1.1.1 and 1.2.1.

Etching

1. Etch by immersion in HCl for approximately 1 minute (see Section 1.4.1).
2. Rinse in cold water until sample is cool.
3. Rinse in HNO_3 until the desired contrast is obtained, approximately 1 to 5 seconds (see Section 1.4.2).
4. Rinse in cold water until the sample is cool.

Photography

Photograph the sample wet at 1X magnification, using either the macro camera or the 8×10 camera, depending on the size of the ingot slice (see Section 2.1.1).

Figures 4, 5, and 6 are typical macrographs.

FIGURE 4 Macrostructure of As-Cast Ingot Slice (1X)

FIGURE 5 Macrostructure of Cast, Beta Heat-Treated, and Water-Quenched Ingot Slice (1X)

FIGURE 6 Macrostructure of Cast, Beta Heat-Treated, and Alpha-Extruded Ingot Slice (IX)

Procedure 3.2 Macrostructures of Beta Heat-Treated, Water-Quenched; Beta Heat-Treated, Oil-Quenched; and Beta Heat-Treated, Oil-Quenched, and Extruded Uranium Fuel Cores

Sample Preparation

See Sections 1.1.2 and 1.2.1.

Etching

1. Etch by immersion in HCl for approximately 30 seconds (see Section 1.4.1).
2. Rinse in cold water until the sample is cool.
3. Rinse in HNO_3 until the desired contrast is obtained, approximately 1 to 5 seconds (see Section 1.4.2).
4. Rinse in cold water until the sample is cool.

Photography

Photograph the sample wet at 3X magnification with the macro camera (see Section 2.1.2).

Figures 7, 8, and 9 are typical macrographs.

FIGURE 7 Macrostructure of Beta Heat-Treated, Water-Quenched, Uranium Fuel Core (3X)

FIGURE 8 Macrostructure of Beta Heat-Treated, Oil-Quenched, Tubular Uranium Fuel Core (3X)

FIGURE 9 Macrostructure of Beta Heat-Treated, Oil-Quenched, and Extruded Tubular Uranium Fuel Core (3X)

Procedure 3.3 Macrostructure of Alpha-Extruded Uranium Fuel Cores Alloyed with Various Amounts of Iron, Aluminum, and Silicon Not Exceeding 250 ppm (Total)

Sample Preparation

See Sections 1.1.2 and 1.2.1

Etching

1. Etch the sample by immersion in HCl for approximately 30 seconds (see Section 1.4.1).
2. Remove the sample from the acid bath and *let the acid dry on the sample*. This usually takes from 5 to 15 seconds.
3. Rinse the sample in HNO_3 until the desired contrast is obtained (approximately 1 to 5 seconds) (see Section 1.4.2).
4. Rinse in cold water until the sample is cool.

Photography

Photograph the sample wet at 3X magnification with the macro camera (see Section 2.1.2).

Figure 10 is a typical macrograph.

FIGURE 10 Macrostructure of Alpha-Extruded Uranium Fuel Core Alloyed with Fe, Al, and Si in Amounts Not Exceeding 250 ppm, Total (3X)

Procedure 3.4 Macrostructure of Alpha-Extruded Uranium Fuel Cores Alloyed with Various Amounts of Iron, Aluminum, and Silicon Exceeding 250 ppm

Sample Preparation

See Sections 1.1.2 and 1.2.1.

Etching

1. Etch the sample by immersion in HCl for approximately 30 seconds (see Section 1.4.1).
2. Submerge the sample in cold water until it is cool.
3. Rinse the sample in HNO_3 until the desired contrast is obtained (approximately 1 to 5 seconds) (see Section 1.4.2).
4. Rinse the sample in cold water until it is cool.

Photography

Photograph the sample wet at 3X magnification with the macro camera (see Section 2.1.2).

Figure 11 is a typical macrograph.

NOTE: Alpha-extruded fuel cores with higher additive levels must be treated differently from those with lower additive levels because if etched according to Procedure 3.3 the sample would pit, and these pits would fill with uranyl chloride. When rinsed with HNO_3 , the uranyl chloride remains in the pits and is often mistaken for inclusions.

FIGURE 11 Macrostructure of Alpha-Extruded Uranium Fuel Core Containing 900 ppm (Total) of Fe, Al, and Si (3X)

Procedure 3.5 Metal Cleanliness: Overall Inclusion Content and Inclusion Segregation in Uranium Metal

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts of acetic acid. Polish the sample for 45 seconds at 20 volts, using a stainless cathode and the sample as the anode.
3. Wash the sample in water, then in absolute alcohol; dry with air.

Etching

1. Electroetch the sample in a bath consisting of one part of perchloric acid to eight parts of acetic acid. Etch for 2 minutes at 20 volts, using a stainless steel cathode and the sample as the anode.
2. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

Photograph the sample at 3X magnification using the macro camera (see Section 2.1.2).

Figures 12 and 13 illustrate the overall inclusion content and inclusion segregation in cast uranium metal.

FIGURE 12 Overall Inclusion Content in Cast Uranium Metal (3X)

FIGURE 13 Inclusion Segregation in Cast Uranium Metal (3X)

Procedure 3.6 Macrostructure of Uranium Metal Alloyed with 2 Weight Percent Zirconium

Sample Preparation

1. See Sections 1.1.1 and 1.2.2.
2. Mechanically polish the sample (when sample size permits) on a lap covered with a good grade of polishing cloth (such as, A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.

Etching

1. Etch the sample by immersion in HCl for approximately 30 to 45 seconds (see Section 1.4.1).
2. Wash the sample thoroughly in water using cotton to remove the black oxide deposited on the surface.
3. Rinse the sample in a bath consisting of 10 parts HNO_3 and one part potassium or sodium perchlorate for 3 to 5 seconds. (*Note: The perchlorate is added to eliminate the explosion hazard that would exist if HNO_3 alone were used to rinse a uranium-zirconium sample.*)
4. Wash the sample thoroughly in water.

Photography

Photograph the sample wet at 1X magnification on the macro camera or the 8 × 10 camera (see Section 2.1.1).

See Figure 14.

FIGURE 14 Macrostructure of Uranium Metal Alloyed with 2 Weight Percent Zirconium (1X)

Procedure 3.7 Macrostructure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample with water and absolute alcohol; dry with air.

Etching

1. Etch the sample by immersion for 5 to 20 seconds in a bath consisting of 20 parts of concentrated HCl and two parts of concentrated HNO_3 .
2. Wash the sample in cold water for 10 to 15 seconds.
3. Rinse the sample for 3 to 5 seconds in concentrated HNO_3 .
4. Wash the sample thoroughly in cold water.

Photography

Photograph the sample wet on the macro camera at 10X magnification (see Section 2.1.2).

See Figure 15.

FIGURE 15 Macrostructure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum (10X)

Procedure 3.8 Pickling Uranium Ingot Slices and Billet Slices to Determine Macroporosity and Macrocracking

Sample Preparation

1. See Section 1.1.1.
2. Machine the sample surface to a 125-microinch finish.

Pickling

1. Immerse the sample for 5 minutes in a bath of HNO_3 (see Section 1.4.3).

NOTE: After several slices have been pickled, the acid will become hot and the pickling time can be cut to 3 minutes.

2. Wash the sample thoroughly in water, then in absolute alcohol and dry with air.

Inspection and Photography

1. Inspect the slices visually or use a low powered microscope; 10X magnification is usually sufficient.
2. Photograph the sample at 1X magnification, using either the macro camera or the 8 × 10 camera, depending on the size of the ingot slice (see Section 2.1.1).

See Figure 16.

FIGURE 16 Pickled Uranium Ingot Slice (1X)

4. MICROGRAPHIC PROCEDURES

The micrographic procedures that follow are those most frequently used in the evaluation of uranium at this site. The magnifications employed range from 50X to 2400X.

Procedure 4.1 Inclusion Contents of As-Cast or Cast and Formed Uranium Metal

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Polish the sample again on the same type lap using 1-micron diamond compound as the abrasive.
5. Wash the sample in water, then in absolute alcohol; dry with air.
6. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts of acetic acid. Polish the sample for 15 seconds at 20 volts using a stainless steel cathode and the sample as the anode.
7. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

Evaluate or photograph the inclusion content at 100X magnification under bright field illumination (see Section 2.2).

Figures 17 and 18 are typical micrographs.

FIGURE 17 Inclusion Content of As -Cast Uranium Metal (100X)

FIGURE 18 Inclusion Content of Formed Uranium Metal (100X)

Procedure 4.2 Grain Structures of As-Cast, Beta Heat-Treated, and Formed Uranium Metal

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Polish the sample again on the same type of lap using 1-micron diamond compound as the abrasive.
5. Wash the sample in water, then in absolute alcohol; dry with air.
6. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts of acetic acid. Polish the sample for 30 seconds at 20 volts using a stainless steel cathode and the sample as the anode.
7. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

Evaluate or photograph the micro grain structure at 100X magnification under polarized light (see Section 2.2).

Figures 19, 20, and 21 are typical micrographs.

FIGURE 19 Grain Structure of As-Cast Uranium Metal (100X)

FIGURE 20 Grain Structure of Wrought and Beta Heat-Treated Uranium Metal (100X)

FIGURE 21 Grain Structure of Formed Uranium Metal (100X)

Procedure 4.3 Grain Structure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts acetic acid. Polish the sample for 5 to 10 seconds at 6 amperes using a strainless steel cathode and the sample as the anode.
5. Wash the sample in water, then in absolute alcohol; dry with air.
6. Etch the sample by swabbing with a solution consisting of one part lactic acid, one part HNO_3 , and $\frac{1}{20}$ part hydrofluoric acid for 5 to 10 seconds.
7. Wash the sample thoroughly in water, then in absolute alcohol; dry with air.

Photography

Photograph the grain structure under polarized light at 250X magnification on a research metallograph (see Section 2.2).

Figure 22 is a typical micrograph.

FIGURE 22 Grain Structure of Uranium Metal Alloyed with 10 Weight Percent Molybdenum (250X)

Procedure 4.4 Grain Structure of Uranium Metal Alloyed with 2 Weight Percent Zirconium

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Polish the sample again on the same type lap using 1-micron diamond compound as the abrasive.
5. Wash the sample in water then in absolute alcohol; dry with air.
6. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts of acetic acid. Polish the sample for 1 to 2 seconds at 6 amperes using a stainless steel cathode and the sample as the anode.
7. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

Photograph the grain structure under polarized light at 100X magnification on the research metallograph (see Section 2.2).

Figure 23 is a typical micrograph.

FIGURE 23 Grain Structure of Uranium Metal Alloyed with 2 Weight Percent Zirconium (100X)

Procedure 4.5 Grain Structure of Uranium Metal Alloyed with 0.18 Weight Percent Silicon

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Polish the sample again on the same type lap using 1-micron diamond compound as the abrasive.
5. Wash the sample in water, then in absolute alcohol; dry with air.
6. Electropolish the sample in a bath consisting of one part perchloric acid to eight parts acetic acid. Polish the sample for 2 minutes at 6 amperes using a stainless steel cathode and the sample as the anode.
7. Wash the sample in water, then in absolute alcohol; dry with air.
8. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts acetic acid. Polish the sample for 2 minutes at 6 amperes using a stainless steel cathode and the sample as the anode.
9. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

Photograph the grain structure under polarized light at 100X magnification on the research metallograph (see Section 2.2).

Figure 24 illustrates the grain structure revealed.

FIGURE 24 Grain Structure of Uranium Alloyed with 0.18 Weight Percent Silicon (100X)

Procedure 4.6 Precipitates in Alloyed Uranium Metal

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Polish the sample again on the same type of lap using 1-micron diamond compound as the abrasive.
5. Wash the sample in water, then in absolute alcohol; dry with air.
6. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts acetic acid. Polish the sample for 15 seconds at 20 volts using a stainless steel cathode and the sample as the anode.
7. Wash the sample in water, then in absolute alcohol; dry with air.
8. Immerse the sample in a nitric acid bath (one part concentrated HNO_3 to one part water) for 10 seconds.
9. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

Evaluate or photograph the precipitates under dark field illumination at 100X magnification on a research metallograph (see Section 2.2).

NOTE: When using dark field illumination, care should be taken to keep the sample stain-free. If stains are present they will appear as bluish areas on the sample and may cause an incorrect evaluation.

Figures 25 and 26 illustrate the precipitate dispersions in uranium metal.

FIGURE 25 Precipitate Dispersion in Uranium Ingot Containing 125 ppm Fe and 80 ppm Si (100X)

FIGURE 26 Precipitate Dispersion in Uranium Ingot Containing 1000 ppm Fe and 1000 ppm Al (100X)

Procedure 4.7 Color Technique to Identify Inclusions in Uranium Metal

Sample Preparation

1. Cut the sample $\frac{3}{4}$ inch long, $\frac{1}{2}$ inch wide, and $\frac{1}{2}$ inch thick (see Section 1.1.2).
2. Grind the sample successively with 60-grit, 180-grit, and 400-grit silicon carbide papers. During each grinding rotate the sample 45° three times.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Mechanically polish the sample on a lap covered with nylon polishing cloth, using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
5. Wash the sample thoroughly in water, then in absolute alcohol; dry with air.
6. Polish the sample again on a lap covered with microcloth, using 1-micron diamond compound as the abrasive.
7. Wash the sample in water, then in absolute alcohol; dry with air.
8. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts acetic acid. Polish the sample for 10 seconds at 10 volts using a stainless steel cathode and the sample as the anode.
9. Again, mechanically polish the sample on a lap covered with microcloth using 1-micron diamond compound as the abrasive.
10. Wash the sample in water, then in absolute alcohol; dry with air.
11. Electropolish the sample again in the same solution as above for 6 seconds at 10 volts.

Etching and Heat Tinting

1. Etch the sample in either aqueous-organic acid solution listed in Section 1.5. *The sample should be etched within 30 seconds after preparation to avoid oxidation.*
The etching time varies from 10 seconds for as-cast metal to 45 seconds for formed metal (25°C).
2. Wash the sample in water, then in absolute alcohol; dry with air.
3. Heat tint the sample as follows:
 - (a) To make the heating chamber, fill a small crystallization dish $\frac{1}{3}$ full of standard Ottawa sand and cover this dish with a larger one. The use of glass dishes permits observation of the sample during the heat-tinting process.
 - (b) Preheat this chamber to 300°C on a variable-temperature hotplate.
 - (c) Place the sample in the heating chamber for 3 to 5 minutes. During this time the sample will change in color from light tan to brown to deep blue.
 - (d) Remove the sample from the chamber and allow it to cool in air.

Photography and Evaluation

Photograph the inclusions on the metallograph under bright field illumination at a magnification suitable for seeing the desired inclusion clearly (See Section 2.2.2).

Several types of common inclusions have been identified by this color technique:

- (a) Uranium carbide (UC) — Orange-red
- (b) Uranium dioxide (UO₂) — Dark Gray
- (c) Uranium hydride (UH₃) — Silver Halo
- (d) Uranium monoxide (UO)* — Dark Gray
- (e) Uranium phosphide (UP) — White yellow
- (f) Uranium Carbonitride U(CN) — Yellow
- (g) Uranium iron phases (U₆Fe) — Blue

As can be noted above, uranium dioxide and uranium monoxide are not affected by the color producing technique. The uranium phosphide and the intermetallic phase (U₆Fe) have been identified by microprobe analysis. The other inclusions have been identified by the 1:1 aqueous nitric acid treatment and the electrodeposition technique.

Figures 27 through 34 are black-and-white reproductions of color prints showing various inclusions that were colored by this heat-tinting procedure. (See note at the beginning of this report regarding the availability of corresponding color prints.)

* *This material was identified by electron microprobe analysis as being near UO in composition. This material is often referred to as U(C,N,O).*

FIGURE 27 (1) Uranium Carbide (UC), Orange-Red; (2) Uranium Carbonitride (U(CN)), Yellow (1000X)

FIGURE 28 (1) Uranium Carbide System, Orange-Red; (2) Uranium Phosphide (UP), White-Yellow (1000X)

FIGURE 29 (1) Uranium Carbide, Orange-Red; (2) Uranium Phosphide, White-Yellow. Note Precipitate in Grain Boundary. (1000X)

FIGURE 30 (1) Stringer of Uranium Carbide, Orange-Red; (2) Uranium Phosphide, White-Yellow (1000X)

FIGURE 31 (1) Uranium Carbide with Center Etched Out, Orange-Red; (2) Chain-like Precipitate of U_6Fe , Blue; (3) These Inclusions are Thought to be UC (1000X)

FIGURE 32 Uranium Hydride (UH_3). Note Silver Halo Effect. Inside Section is Faintly Multicolored. (1000X)

FIGURE 33 Uranium Carbide, Orange-Red. Note That Center of Carbide is Etched Out. (1000X)

FIGURE 34 Uranium Carbide, Orange-Red (1000X)

Procedure 4.8 Grain Structures and Inclusion Contents of Uranium Metal (Alternate Method)

Sample Preparation

1. See Sections 1.1.2 and 1.2.2.
2. Polish the sample on a lap covered with Beuhler Ltd. No. 40 -7208AB Microcloth using the following mixture as the abrasive:
 - (a) 10% of a solution of 118 g CrO_3 in 100 ml H_2O
 - (b) 10% of a saturated solution of $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ in water
 - (c) 5% of a solution of Linde A Compound in water (mixed to a paste consistency)
 - (d) 75% of distilled water

Polish the sample until the desired finish is obtained.

3. Wash the sample in water, then in absolute alcohol; dry with air.

Photography

1. Photograph the grain structure on the metallograph under polarized light at 100X magnification (see Section 2.2).
2. Rinse the sample in 1:1 HNO_3 for 5 to 15 seconds.
3. Wash the sample in water, then in absolute alcohol; dry with air.
4. Photograph the inclusion content on the metallograph under bright field illumination at 100X magnification.

Figures 35 and 36 illustrate the grain structures and inclusions revealed by this method.

FIGURE 35 Grain Structure of Beta Heat-Treated Uranium Metal (100X)

FIGURE 36 Inclusion Content of As-Cast Uranium Metal (100X)

5. MISCELLANEOUS PROCEDURES FOR THE TREATMENT OF
URANIUM AND THORIUM METALS

Procedure 5.1 Cleaning Uranium Metal for Inspection or Display

1. Remove any oil or grease by washing the piece with alcohol or acetone.
2. Wash the piece in water, then in absolute alcohol; dry with air.
3. Electropolish the piece in a bath consisting of one part perchloric acid to eight parts acetic acid. Polish the piece for 2 minutes at 20 volts using a stainless steel cathode and the sample as the anode. Keep the bath cool by circulating cold water around the container holding the bath.
4. Wash the piece in water, then in absolute alcohol; dry with air.
5. If the piece is to be used for display purposes, spray it with clear plastic to preserve the finish. Uranium treated in this manner will remain bright and lustrous for 3 or 4 days.

Uranium metal cleaned for display is shown in Figure 37.

FIGURE 37 Uranium Metal Cleaned for Display

Procedure 5.2 Primary Inclusions, Secondary Inclusions, Precipitates, and Grain Structures of Thorium Metal

1. Grind the sample successively with 60-grit, 120-grit, and 400-grit silicon carbide. Twice during each grinding rotate the sample 45°.
2. Wash the sample in water, then in absolute alcohol; dry with air.
3. Mechanically polish the sample on a lap covered with a good grade of polishing cloth (such as A.B. Metcloth) using 6-micron diamond compound as the abrasive. Polish until all grinding marks are removed.
4. Wash the sample in water, then in absolute alcohol; dry with air.
5. Polish the sample again on the same type lap using 1-micron diamond compound as the abrasive.
6. Wash the sample in water, then in absolute alcohol; dry with air.
7. Evaluate or photograph the primary inclusions at 100X magnification using bright field illumination (see Section 2.2). Figure 38 is a typical micrograph.
8. Electropolish the sample in a bath consisting of one part perchloric acid to ten parts acetic acid. Polish the sample for 3 to 5 seconds at 20 volts using a stainless steel cathode and the sample as the anode. *For good results, it is very important to keep the bath cold.* This can be accomplished by circulating cold water around the container holding the bath.
9. Wash the sample in water, then in absolute alcohol; dry with air.
10. Evaluate or photograph the secondary inclusions and precipitates at 100X magnification under bright field illumination (see Section 2.2). Figure 39 is a typical micrograph.
11. Electropolish the sample in a bath consisting of one part of a base solution of 118 g CrO_3 to 100 ml water and four parts acetic acid. Polish the sample for 15 seconds at 20 volts using a stainless steel cathode and the sample as the anode.
12. Wash the sample in water, then in absolute alcohol; dry with air.
13. Evaluate or photograph the grain structure at 100X magnification under polarized light (see Section 2.2). Figure 40 is a typical micrograph of thorium grain structure.

FIGURE 38 Primary Inclusions in Thorium Metal (100X)

FIGURE 39 Secondary Inclusions and Precipitates in Wrought Thorium Metal(100X)

FIGURE 40 Grain Structure of Wrought Thorium Metal (100X)

6. METALLOGRAPHIC STANDARDS USED AT THE NATIONAL LEAD COMPANY OF OHIO

Several types of metallographic standards have been developed at the National Lead Company of Ohio to aid in determining the effects of various processing conditions on uranium metal structures. These standards include:

- (a) Grain-size standards for uranium fuel cores (most at 3X) following the processes of casting, alpha forming and beta heat treating.
- (b) Recrystallization standards for uranium metal (at 100X) after casting and alpha forming.
- (c) Standards for rating inclusions in as-cast uranium metal (at 100X).
- (d) Standards for rating inclusions in formed uranium metal (at 100X).

Illustrations of these standards, instructions for their application, and examples of metal samples rated by the standards are presented in this section.

6.1 GRAIN-SIZE STANDARDS FOR BETA HEAT-TREATED URANIUM FUEL CORES

Grain-size standards are presented for uranium subjected to the following processing conditions:

- (a) Cast, rolled in the high alpha phase, heat treated by quenching in water from the beta phase (Figure 41 at 3X and Figure 44 at 100X).
- (b) Same as (a) but quenched in oil (Figure 42).
- (c) Cast, extruded in the high alpha phase, heat treated by quenching in oil from beta phase (Figure 43).

To use these standards, view the prepared sample in the metallograph at the proper magnification (3X or 100X). Compare the enlarged image of the sample directly with the appropriate grain-size chart, or photograph the sample, make a print, and compare this print with the chart.

FIGURE 41 Grain-Size Standards for Rolled, Beta Heat-Treated, Water-Quenched, Uranium Fuel Cores (3X)

1

2

3

4

5

6

7

8

9

FIGURE 42 Grain-Size Standards for Rolled, Beta Heat-Treated, Oil-Quenched, Uranium Fuel Cores (3X)

FIGURE 43 Grain-Size Standards for Extruded, Beta Heat-Treated, Oil-Quenched, Uranium Fuel Cores (3X)

FIGURE 44 Grain-Size Standards for Rolled, Beta Heat-Treated, Water-Quenched, Uranium Fuel Cores (100X)

6.2 RECRYSTALLIZATION STANDARDS FOR FORMED URANIUM METAL

To determine the degree to which uranium metal is recrystallized by forming operations such as rolling or extrusion, view the sample at 100X magnification in the metallograph. Compare the enlarged image directly with the standards on the recrystallization chart (Figure 45); or photograph the sample, make a print, and compare this print with the standards.

FIGURE 45 Recrystallization Standards for Formed Uranium Metal (Numbers Express Percent Recrystallization)(100X)

6.3 STANDARDS TO EVALUATE RANDOM AND CLUSTERED INCLUSIONS IN AS-CAST URANIUM METAL

Inclusions in as-cast uranium metal are evaluated for type, concentration, and size by use of the standards presented in Figure 46, for random inclusions, and Figure 47, for clustered inclusions. The sample is viewed under bright field illumination with the magnaviewer on the metallograph, the magnaviewer being marked off with grid lines as shown in Figure 48.

First, determine the inclusion type (random, clustered, or both) by comparing the sample with the two sets of standards. If the inclusions are random, determine the concentration by comparing the sample with the standards in Figure 46. The numbers given under "Concentration" in the figure are the average number of inclusions per $\frac{1}{2}$ -inch square on the magnaviewer. Determine the average sizes of random inclusions by comparing the inclusions of the sample with the standards (Figure 46). At 100X magnification the sizes are as follows:

<u>Letter</u>	<u>Size (in.)*</u>
A	0.010
B	0.020
C	0.040
D	0.080
E	0.100

If the inclusions are clustered rather than random, use Figure 47 to determine the concentration. The numbers given under "Concentration" in the chart are the average number of inclusions per cluster. Note that this is *not* the average number of clusters per $\frac{1}{2}$ -inch square. To determine the average size of *the inclusions making up each cluster*, compare the sample with the standards. At 100X magnification the sizes are as follows:

<u>Letter</u>	<u>Size (in.)*</u>
A	0.015
B	0.020
C	0.030
D	0.060
E	0.070

A typical inclusion evaluation might appear as follows:

<u>Concentration</u>	<u>Size</u>	<u>Type</u>
6	A	R

* Sizes taken from chart prepared by the Cooperative Metallography Group.

Here R denotes "random," the type of inclusions present, 6 is the average number of inclusions per $\frac{1}{2}$ -inch square, and A denotes the average size of the inclusions, obtained by comparison with the standards. Another sample might result in this evaluation:

<u>Concentration</u>	<u>Size</u>	<u>Type</u>
2	C	R
6	B	C

In this case, both random and clustered inclusions were present (C denoting clustered); hence two evaluations were necessary:

- (a) 2 is the average number of random inclusions per $\frac{1}{2}$ -inch square; R denotes the type of inclusion and C denotes the average size of the random inclusions, obtained by comparison with the standards.
- (b) 6 is the average number of inclusions per cluster, C denotes the type of inclusion, and B denotes the average size of the individual inclusions which make up the clusters.

Five samples have been evaluated to illustrate the use of the standards. They are presented in Figures 49 through 53.

LETTERS BELOW DESIGNATE THE AVERAGE SIZE OF INCLUSIONS (IN INCHES AT 100X)

A
(0.010)

B
(0.020)

C
(0.040)

D
(0.080)

E
(0.100)

NUMBERS BELOW DESIGNATE CONCENTRATION: THE AVERAGE NUMBER OF INCLUSIONS PER HALF-INCH SQUARE (AT 100X)

2

6

10

16

20

FIGURE 46 Standards for Random Inclusions in As-Cast Uranium Metal

LETTERS BELOW DESIGNATE THE AVERAGE SIZE OF INCLUSIONS (IN INCHES AT 100X)

A
(0.015)

B
(0.020)

C
(0.030)

D
(0.060)

E
(0.070)

NUMBERS BELOW DESIGNATE CONCENTRATION: THE AVERAGE NUMBER OF INCLUSIONS PER CLUSTER (100X)

2

6

10

16

20

FIGURE 47 Standards for Clustered Inclusions in As-Cast Uranium Metal

FIGURE 48 Magnaviewer with Grid (4 -Inch Square Divided Into $\frac{1}{2}$ -Inch Squares)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
<1	E	R

FIGURE 49 Random and Clustered Inclusion Rating of As -Cast Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
25	A	R
4	A	C

FIGURE 50 Random and Clustered Inclusions Rating of As -Cast Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
2	C	R
8	C	C

FIGURE 51 Random and Clustered Inclusion Rating of As-Cast Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
7	B	R
16	D	C

FIGURE 52 Random and Clustered Inclusion Rating of As-Cast Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
< 1	C	R
9	E	C

FIGURE 53 Random and Clustered Inclusion Rating of As-Cast Uranium (100X)

6.4 STANDARDS TO EVALUATE RANDOM INCLUSIONS IN FORMED URANIUM METAL

The standards for evaluating random inclusions in formed uranium metal are shown in Figure 54. These standards are employed in the same manner as those for evaluating random inclusions in as-cast uranium metal (Section 6.3). The sizes of the inclusions at 100X magnification are as follows:

<u>Letter</u>	<u>Size (in.)*</u>
A	0.015
B	0.020
C	0.040
D	0.080
E	0.100

* *Sizes of inclusions taken from chart prepared by the Cooperative Metallography Group.*

LETTERS BELOW DESIGNATE THE AVERAGE SIZE OF INCLUSIONS (IN INCHES AT 100X)

A
(0.015)

B
(0.020)

C
(0.040)

D
(0.080)

E
(0.100)

NUMBERS BELOW DESIGNATE CONCENTRATION: THE AVERAGE NUMBER OF INCLUSIONS PER HALF-INCH SQUARE (AT 100X)

2

6

10

16

20

FIGURE 54 Standards for Random Inclusions in Formed Uranium Metal

6.5 STANDARDS TO EVALUATE STRINGERS OF INCLUSIONS IN FORMED URANIUM METAL

Stringers of inclusions in formed uranium metal are evaluated as to number per field, length, and width. The standards used for the evaluation are shown in Figure 55. The sample is viewed under bright field illumination with the magnaviewer on the metallograph, the magnaviewer being marked off with grid lines as shown in Figure 48.

Determine the number of stringers by comparing the sample with the standards. The value obtained is the actual number of stringers that appear on the field being rated.

Determine the length and width of the stringers by comparing the sample with the standards. The values given are the length and width in inches at 100X magnification. The $\frac{1}{2}$ -inch squares of the magnaviewer assist in making these determinations.

It has been found in practice that any given field in formed uranium metal will probably contain both random inclusions and stringers of inclusions (see Figure 55). Two evaluations are therefore necessary. Evaluate random inclusions by the standards presented in Figure 54 and the stringers according to the method above. If the sample surface also shows clusters of inclusions, as happens occasionally, they are rated according to the standards presented in Figure 47.

A typical evaluation might appear as follows:

<u>Concentration</u>	<u>Size</u>	<u>Type</u>	<u>No. Per Field</u>	<u>Length (in.)</u>	<u>Width (in.)</u>
6	A	R	2	4	$\frac{1}{16}$

This is interpreted as follows:

- There are 6 random inclusions per $\frac{1}{2}$ -inch square.
- The random inclusions are of size A.
- There are 2 stringers in the field under observation.
- The length of the stringers is 4 inches; that is, the stringers covered eight $\frac{1}{2}$ -inch squares.
- The stringers are $\frac{1}{16}$ -inch wide.

Figures 56 through 60 are evaluations of typical formed uranium samples that have been rated by the standards given.

NUMBER OF STRINGERS PER FIELD (AT 100X)

2

3

6

9

LENGTH AND WIDTH OF STRINGERS (AT 100X)

1" $\times$ $\frac{1}{32}$ "

2" $\times$ $\frac{1}{16}$ "

3" $\times$ $\frac{1}{16}$ "

4" $\times$ $\frac{1}{8}$ "

FIGURE 55 Standards for Stringers of Inclusions in Formed Uranium Metal

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
6	A	R

<u>NO. STRINGERS PER FIELD</u>	<u>LENGTH (IN.)</u>	<u>WIDTH (IN.)</u>
4	1	$\frac{1}{32}$
1	$1\frac{1}{2}$	$\frac{1}{16}$
1	3	$\frac{1}{32}$

FIGURE 56 Inclusion and Stringer Rating of Formed Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
10	B	R

<u>NO. STRINGERS PER FIELD</u>	<u>LENGTH (IN.)</u>	<u>WIDTH (IN.)</u>
3	$2\frac{1}{2}$	$\frac{1}{16}$
6	1	$\frac{1}{16}$

FIGURE 57 Inclusion and Stringer Rating of Formed Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
1	C	R

<u>NO. STRINGERS PER FIELD</u>	<u>LENGTH (IN.)</u>	<u>WIDTH (IN.)</u>
4	3	$\frac{1}{16}$
3	4	$\frac{1}{8}$
2	1	$\frac{1}{16}$

FIGURE 58 Inclusion and Stringer Rating of Formed Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
1	D	R

<u>NO. STRINGERS PER FIELD</u>	<u>LENGTH (IN.)</u>	<u>WIDTH (IN.)</u>
2	3	$\frac{1}{8}$
2	1	$\frac{1}{8}$

FIGURE 59 Inclusion and Stringer Rating of Formed Uranium (100X)

<u>CONCENTRATION</u>	<u>SIZE</u>	<u>TYPE</u>
<1	E	R

<u>NO. STRINGERS PER FIELD</u>	<u>LENGTH (IN.)</u>	<u>WIDTH (IN.)</u>
1	1½	1/16
4	1	1/16

FIGURE 60 Inclusion and Stringer Rating of Formed Uranium (100X)

7. APPENDIX

7.1 URANIUM GRAIN SIZE STANDARDS OF THE FUEL ELEMENT DEVELOPMENT COMMITTEE, REVISION 2*

The Working Committee of the Fuel Element Development Committee (FEDC) selected the grain size standards presented in Figures 61 through 64 on April 29, 1965, as the preferred manner of reporting the grain size of beta heat treated uranium fuel cores. The grain size standards were prepared from 3X photomicrographs of transverse sections of uranium fuel elements. The recommended procedure for sample preparation to be used with these standards is attached. If other procedures are followed, these procedures should be tested against the recommended one to assure that bias in the rating has not been introduced.

Grain size standards A-1 through A-7 are for fuel cores made from rolled uranium and beta heat treated in a molten chloride salt bath and water quenched in solid core blank form.

Grain standards B-1 through B-5 are for fuel cores made from rolled stock and beta heat treated in a bronze bath and quenched in a tin bath in hollow core blank form.

Grain standards C-1 through C-8 are for fuel cores made from rolled stock and beta heat treated in salt and oil quenched in hollow core blank form.

Grain standards D-1 through D-9 are for fuel cores made from extruded stock, beta heat treated in salt and oil quenched in hollow core blank form.

The following grain standards are to be used for making your own chart or book to fit your particular needs.

The uranium Grain Size Standards will be revised in the future if better standards are found or new methods of beta heat treating uranium cores are developed.

* Published by the FEDC, September 1, 1965. Members include representatives from AEC contractor sites at Hanford (General Electric); Savannah River (du Pont); Weldon Springs (Mallinckrodt) and Fernald (National Lead Co. of Ohio).

7.2 FEDC PROCEDURES FOR SAMPLE PREPARATION

1. Wafers are sectioned from uranium cores with an abrasive cut-off machine using a carborundum wheel.
2. Samples are rough ground on a belt grinder using an 80-grit silicon carbide belt with a water or water-soluble oil lubricant and rinsed in cold flowing water.
3. The ground surface is etched in 38% HCl at room temperature for 30 seconds, rinsed in cold flowing tap water and then etched in 71% HNO₃ at room temperature until the grains are distinct.
4. Following etching, the samples are rinsed in cold flowing water and kept submerged in water until photographed.
5. At the time of photographing, the excess water on the sample is swabbed off and the picture taken while the sample is still wet.
6. 3X photomacrographs are made using a Bausch and Lomb camera, Model L, with a 158 mm Photomacrographic Lens. The samples are illuminated with one 32-watt Westinghouse Circline fluorescent lamp. White background paper is used.
7. Kodak Royal Pan film is used. The developer is Kodak DK-50.
8. The photographic paper is Kodak Single Weight Illustrator's Azo F-2, 3 or 4, depending upon the contrast of the sample. The developer is Kodak Dektal.

FIGURE 61 Grain-Size Standards for Rolled, Salt-Heated, Water-Quenched, Uranium Fuel Cores (3X)

FIGURE 62 Grain-Size Standards for Rolled, Bronze-Heated, Tin-Quenched, Uranium Fuel Cores (3X)

FIGURE 63 Grain-Size Standards for Rolled, Salt-Heated, Oil-Quenched, Uranium Fuel Cores (3X)

FIGURE 64 Grain-Size Standards for Extruded Salt-Heated, Oil-Quenched, Uranium Fuel Cores (3X)