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Chicago, Illinois

Compiled by

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Concord, Massachusetts

THE
UNITED STATES
DEPARTMENT OF
THE ARMY
WASHINGTON, D. C.

TO THE SECRETARY OF THE ARMY
FROM THE CHIEF OF STAFF
SUBJECT: [Illegible]

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LIST OF ATTENDEES
 13th METALLOGRAPHIC GROUP MEETING
 AT ARMOUR RESEARCH FOUNDATION
 ILLINOIS INSTITUTE OF TECHNOLOGY
 CHICAGO, ILLINOIS
 JANUARY 14, 15, 1959

Peterson, David	Ames Laboratory, Iowa State College
Benson, Eugene	Argonne National Laboratory
Carlander, Richard	Argonne National Laboratory
Dwight, Austin E.	Argonne National Laboratory
Dunworth, Richard J.	Argonne National Laboratory
Kehl, George	Argonne National Laboratory
Yario, William R.	Argonne National Laboratory
Domagala, Robert F.	Armour Research Foundation
Dvorak, James R.	Armour Research Foundation
Grala, Edward M.	Armour Research Foundation
Holtz, Frederick C.	Armour Research Foundation
Nelson, Alan D.	Armour Research Foundation
Rajala, Benjamin R.	Armour Research Foundation
Van Thyne, Ray J.	Armour Research Foundation
Baumrucker, John E.	Atomics International
Bauer, Arthur A.	Battelle Memorial Institute
Cheney, Donald M.	Battelle Memorial Institute
Dickerson, Ronald F.	Battelle Memorial Institute
Music, Gene A.	Bridgeport Brass Company
Paul, Robert N.	Bridgeport Brass Company
Schuetz, Arnold	Bridgeport Brass Company
Leith, W. Harold	Du Pont Company, Savannah River Laboratory
Posey, Wilbert N.	Du Pont Company, Savannah River Laboratory
Draghic, J. D.	General Electric Company, Cincinnati
Filer, E. Willard	General Electric Company, Cincinnati
Rau, Robert C.	General Electric Company, Cincinnati
Brandt, Robert L.	General Electric Company, Hanford Operations
Guay, Arthur E.	General Electric Company, Hanford Operations
Hartcorn, L. A.	General Electric Company, Hanford Operations
Tews, W. G.	General Electric Company, Hanford Operations
Allio, R. J.	General Electric Company, Knolls Atomic Power Lab.
Baldwin, Elmer E.	General Electric Company, Knolls Atomic Power Lab.
Johnston, Wm. V.	General Electric Company, Knolls Atomic Power Lab.
Leipziger, Fred D.	General Electric Company, Knolls Atomic Power Lab.
Wemple, Bruce D.	General Electric Company, Knolls Atomic Power Lab.
Anderson, Robert W.	Los Alamos Scientific Laboratory
Jones, Thomas I.	Los Alamos Scientific Laboratory
Hall, Charles F.	National Lead of Ohio
Wise, Walter N.	National Lead of Ohio
Roth, Harriet P.	Nuclear Metals, Inc.
Crouse, Robert S.	UCNC, Oak Ridge National Laboratory
Gray, Robert J.	UCNC, Oak Ridge National Laboratory
Long, Ernest L., Jr.	UCNC, Oak Ridge National Laboratory
Van Cleve, John E.	UCNC, Oak Ridge National Laboratory
Willmarth, Theodore E.	UCNC, Oak Ridge National Laboratory
Cotter, Francis	Watertown Arsenal
Gamber, Charles P.	Westinghouse Electric, Bettis Plant

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REMOTE SPECIMEN MOUNTING*

W. H. Leith

Savannah River Laboratory
E. I. du Pont de Nemours and Company

"Eastman 910" adhesive is used to mount metallographic specimens remotely and in a manner that does not interfere with subsequent cathodic etching. The specimen is glued to a metal mount for grinding and polishing, and is easily removed when this specimen is to be etched.

"Eastman 910" adhesive is used at the Savannah River Laboratory for remotely mounting irradiated metallographic specimens in a manner that will not interfere with subsequent cathodic etching. Conventional methods of mounting by embedding specimens in plastics or low melting alloys do not permit good results to be obtained in subsequent cathodic etching. Such plastic mounts char, degas, do not conduct an electrical current, and prevent adequate cooling of the specimen. Low melting alloys evaporate and plate out on the specimen, ruining the etch. Specimens that are not radiation hazards can be ground and polished by hand or can be held in a simple clamp, but such handling is not possible in remote operations, where manipulation is difficult.

An extensive search was made to find a material that would hold the specimen for grinding and polishing and that could be removed easily before the specimen was cathodically etched. "Eastman 910" adhesive was found to be satisfactory in both respects. It is described as "a cyanoacrylate monomer modified with thickening agent and plasticizer". When the monomer is pressed into a thin film between two surfaces, a polymerization reaction occurs which results ultimately in steel-steel bond strengths of 5,800 psi. If the catalyst provided with the adhesive is used, this strength is reached in a few minutes. Bonds formed with this adhesive have been irradiated to 10^8 F without adverse effects.

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* The information contained in this report was developed during the course of work under contract AT(07-2)-1 with the Atomic Energy Commission, whose permission to publish is gratefully acknowledged.

The specimen is remotely glued to a steel mount with the adhesive, and the specimen is then ground and polished (Fig. 1). After the specimen is polished, the mount can be removed from it by immersing the joint in iso-octyl alcohol at 150°C for five minutes. The specimen can then be cathodically etched without complication.

The remote mounting operation requires less than five minutes and utilizes small, inexpensive equipment that can be operated by a master slave manipulator. The surface of a specimen cut on an abrasive wheel is satisfactory for gluing after the cut surface is cleaned. The items needed to mount a specimen are shown in Fig. 2. To begin the mounting procedure the specimen is wiped with the catalyst and allowed to dry. A drop of adhesive from a dropping bottle is then placed on a clean mount, which is normally larger than the specimen. The ring is placed over the mount to retain the specimen which is then placed on the mount and moved around to spread the adhesive. The specimen is then clamped to apply a slight pressure on the glue, which sets in less than 60 seconds. Although the maximum strength of the bond is not reached for thirty minutes, the bond is sufficiently strong to permit the specimen to be ground almost immediately. After the specimen is ground and polished, it is removed by immersing the joint in an iso-octyl alcohol bath at 150°C for five minutes.

Specimens 1 inch in diameter or 3/4 inch square are most desirable for the 1-1/4-inch diameter mounts used at SRL. However, 60° segments of a 1-inch diameter rod have been polished successfully. Smaller specimens or slivers can be glued, but methods of grinding such specimens have not yet been fully explored.

Fig. 1 - Mounted specimen.

Fig. 2 - Glue mount equipment.

THE METALLOGRAPHY OF NIOBIUM-OXYGEN
AND NIOBIUM-CARBON ALLOYS

Rodney P. Elliott

Armour Research Foundation
Illinois Institute of Technology

Previous experience has shown that a comprehensive understanding of the phase relationships of the interstitial elements with a base metal will minimize many problems in the development of alloys of the base metal. Concurrent with the emphasis of development of high-temperature niobium-base alloys, the phase diagrams niobium-oxygen and niobium-carbon have been determined. Microstructures in these systems are distinctive. This paper is intended to illustrate the typical structures that occur in three systems so that the practicing metallographer may have a reference for identification of these structures.

The principal tool in the determination of these diagrams has been a metallographic investigation of arc-cast alloys. Electron-gun refined niobium was used as melting stock with the exception of the very high carbon alloys where it was necessary to resort to sintering powder compacts which were subsequently arc melted.

Metallographic laboratory techniques used in these studies are essentially the time-honored techniques that have been developed for titanium and zirconium base alloys. The polishing technique consists of the following steps:

- 1) Mount in Bakelite
- 2) Rough grind on 120-grit belt
- 3) Rough polish on silicon carbide papers of 240, 320, 400, and 600 mesh grit
- 4) Polish on metallographic wheel with progressively finer diamond abrasive of 30, 9, and 1 micron size. A 1573 AB Metcloth is used on the polishing wheel
- 5) Final polish with Linde "B" alumina suspended in a vehicle of 1% hydrofluoric acid in water. A 1576 AB Microcloth is used on the polishing wheel.
- 6) Etch with a solution containing 20cc nitric acid, 20cc hydrofluoric acid, 10cc 30% hydrogen peroxide and 50cc glycerine

To remove the worked layer resulting from the polishing, steps 5 and 6 are repeated several times. If the hydrofluoric acid solution vehicle in step 5 is ineffective, a saturated solution of potassium ferricyanide is substituted.

No special optical techniques were necessary to identify microconstituents.

The niobium-oxygen phase diagram is presented in Fig. 1. It is characterized by three intermetallic compounds: NbO , NbO_2 , and Nb_2O_5 . There are two eutectic reactions: one between niobium and NbO at 1910°C ; the second between NbO and NbO_2 at 1815°C . Metallographic evidence indicates that there is peritectic reaction between NbO_2 and Nb_2O_5 . The solid solubility of oxygen in niobium decreases from 0.72 w/o at the eutectic temperature to 0.3 at 500°C .

A microstructure typical of alloys in which NbO is precipitated from solid solution of niobium is illustrated in Fig. 2. NbO preferentially precipitates at grain boundaries. There is no tendency for the NbO to envelope the grains, but rather it agglomerates at the grain boundaries. The Vickers hardness of the solid solution varies from approximately 200 to 500 depending on the oxygen content. No difficulty is experienced in polishing such specimens because of the relative hardness of the niobium solid solution.

As the oxygen content increases, a eutectic matrix begins to surround the pro-eutectic dendrites of niobium solid solution, as Fig. 3 typifies. This alloy contains 5 percent oxygen by weight.

The Nb-NbO eutectic structure has been determined to occur at 10.5 w/o oxygen. This consists of a very fine aggregate of niobium solid solution with NbO in a granular configuration, as shown in Fig. 4.

Pro-eutectic NbO appears as needles in the metallographic structure. This is in contrast to the dendritic nature of pro-eutectic niobium solid solution. No difficulty is experienced in determining metallographically whether an alloy lies to the oxygen- or niobium-rich side of the eutectic composition. Such a typical structure is the 12 percent oxygen alloy as shown in Fig. 5.

Solidification of compositions near that of NbO results in the typical structure as shown in Fig. 6. Converging crystals entrap a small quantity of NbO-NbO₂ eutectic between the irregularly shaped grains.

Structures in the composition range between NbO and NbO_2 are very interesting. As the system approaches higher oxygen-rich compositions, the alloys are more of a glass or slag than they are of metallic nature. Such microconstituents are relatively inactive chemically. Alloys in this range

require no etching to reveal the metallographic structure. Pro-eutectic NbO appears white (Fig. 7) while pro-eutectic NbO₂ appears gray (Fig. 9) in a eutectic matrix of gray and white. The eutectic structure itself (Fig. 8) is geometrical in its morphology with spines of NbO radiating at mutual angles of 120° from nucleation centers.

In the composition range between the compounds NbO₂ and Nb₂O₅ the arc-cast alloys are exceedingly fluid and slag-like. In this composition range it was necessary to alter the final polishing procedures because of the chemical inactivity of the alloy. Instead of repeated etching and final polishing treatment to remove fine scratches, a fine silk lap cloth was substituted. Careful polishing techniques alone were relied upon to produce a scratch-free surface.

Arc-cast Nb₂O₅ can vary widely in color depending on minute variation in its oxygen content. Normally, powdered Nb₂O₅ is creamy-white in color. However, if this is heated in a temperature range about 1000°C in a vacuum, the powder becomes blue due to infinitesimal losses of oxygen. Arc-cast Nb₂O₅ appears mottled on microexamination.

A typical arc-cast structure of an alloy in the composition range between the two oxygen-rich compounds is given by Fig. 10. No eutectic microstructure is evident. It was not possible to develop any peritectic type of microstructure either, although other experimental evidence indicates that this is more probably the freezing reaction.

The niobium-carbon diagram is presented in Fig. 11. Two intermediate phases are known to exist, Nb₂C and NbC. Nb₂C does not exist at the stoichiometric composition (6.07 w/o carbon)² but at slightly lower carbon contents. NbC has a wide range of composition. A eutectic freezing reaction at 2230°C exists between the niobium solid solution and Nb₂C.

Alloys containing 0.5 percent carbon and less give metallographic evidence of being single phase at elevated temperatures. On subsequent cooling to room temperature the Nb₂C precipitates. This is illustrated by Figs. 12 and 13. The fields of these photos have been selected to show the as-cast grain structure. In contrast to the niobium-oxygen system, precipitating Nb₂C does not preferentially agglomerate at boundaries. Grain boundaries are delineated by a relatively minor portion of the precipitating Nb₂C; the bulk of the Nb₂C precipitates on preferential planes within the grain. More dilute alloys result in a very fine precipitate (Fig. 12), while richer carbon compositions produce larger platelets of Nb₂C (Fig. 13).

The matrix of the niobium-carbon solid solution is relatively soft with a Vickers hardness of about 130. The more dilute niobium-carbon alloys that do not have a large amount of intragranular precipitate are subject to scratching during polishing and so must be carefully prepared.

A micrograph of a Nb-0.95%C alloy (Fig. 14) contains pro-eutectic niobium solid solution surrounded by a matrix of Nb-Nb₂C eutectic. The pro-eutectic solid solution on cooling has rejected Nb₂C.

The Nb-Nb₂C eutectic at 1.5% carbon is shown in Fig. 15. In contrast to the niobium-rich eutectic in the niobium-oxygen system, this structure is well defined and consists of plates of Nb₂C in an almost lamellar configuration.

A typical micrograph of pro-eutectic Nb₂C in a matrix of Nb-Nb₂C eutectic is presented in Fig. 16.

Arc-cast alloys in the range 6-12 percent carbon segregate on casting. A typical micrograph of a mixture of NbC and Nb₂C is shown in Fig. 17. The Nb₂C has developed the typical Widmanstätten pattern. These striations have been rationalized as a precipitation of NbC on preferential planes of Nb₂C on cooling. It will be remembered that Nb₂C does not exist at the stoichiometric composition at lower temperatures. At elevated temperatures the composition range of Nb₂C probably extends to the stoichiometric composition. On cooling, NbC is rejected on preferential planes; this produces characteristic striations.

Equilibrium structures between NbC and Nb₂C give no evidence of eutectic reaction. The equilibrium structures suggest a high-temperature peritectic reaction. Indeed, alloys in the range 4 to 6 percent carbon suggest that the pro-eutectic Nb₂C had nucleated on prior NbC crystals.

Both Nb₂C and NbC are very hard, with Vickers hardness in excess of 1200. Metallographic samples of alloys containing these carbides in a matrix of the softer eutectic are difficult to polish maintaining a flat field.

Single-phase NbC does not etch very rapidly. Etching times up to one hour have been required to bring out grain boundaries.

Carbon-niobium alloys containing more carbon than stoichiometric NbC are typified by Fig. 18. Pro-eutectic NbC exists in a matrix of NbC-graphite eutectic. No etching is required to produce this contrast of microconstituents.

Acknowledgment

The author wishes to express his appreciation to the Atomic Energy Commission for sponsorship of this investigation under Contract No. AT(11-1)-515 and for permission to publish. The metallographic preparation of samples was performed by Mr. James Dvorak of the Armour Research Foundation Metallographic Laboratory and his able assistants.

Fig. 1 - Niobium-oxygen equilibrium diagram.

250X

etched

Fig. 2 - Nb-0.8%. NbO precipitated at grain boundaries of niobium solid solution. Annealed 1200°C.

250X

etched

Fig. 3 - Nb-5%O. Pro-eutectic niobium in matrix of Nb-NbO eutectic.

250X

etched

Fig. 4 - Nb-10%O. Nb-NbO eutectic. A few dendrites of pro-eutectic Nb are evident.

250X

etched

Fig. 5 - Nb-12%O. Needles of NbO in matrix of Nb-NbO eutectic.

250X

unetched

Fig. 6 - Nb-15%O. NbO with NbO-NbO₂ eutectic in grain boundaries.

250X

unetched

Fig. 7 - Nb-20%O. Pro-eutectic NbO in matrix of NbO-NbO₂ eutectic.

250X

unetched

Fig. 8 - Nb-21%O. NbO-NbO₂ eutectic.

250X

unetched

Fig. 9 - Nb-22%O. Pro-eutectic NbO₂ in matrix of NbO-NbO₂ eutectic.

250X

unetched

Fig. 10 - Nb-28%O. Cast equilibrium structure between NbO₂ (white) and Nb₂O₅ (gray).

Fig. 11 - Niobium-carbon equilibrium diagram.

250X

etched

Fig. 12 - Nb-0.11%C. Nb_2C precipitate from niobium solid solution.

250X

etched

Fig. 13 - Nb-0.52%C. Nb_2C precipitate from niobium solid solution.

250X

etched

Fig. 14 - Nb-0.95%C. Pro-eutectic niobium in matrix of Nb-Nb₂C eutectic.

250X

etched

Fig. 15 - Nb-1.50%C. Nb-Nb₂C eutectic.

250X

etched

Fig. 16 - Nb-3.38%C. Pro-eutectic Nb₂C in matrix of Nb-Nb₂C eutectic.

Fig. 17 - Nb-6.80%C. NbC
(irregular shapes) with
striated Nb₂C.

Fig. 18 - Nb-12.7%C. Pro-eutectic
NbC in matrix of NbC-
graphite eutectic.

THE REMOTE PREPARATION OF BORON CARBIDE, ZIRCALOY-2 SAMPLES

Bruce D. Wemple

General Electric Company
Knolls Atomic Power Laboratory

This paper describes the equipment and techniques used to remotely process irradiated boron carbide, Zircaloy-2 metallographic specimens. The results obtained using these procedures are also illustrated.

INTRODUCTION

With the possible use of boron carbide particles, dispersed in a Zircaloy-2 matrix, as a reactor control material, it was necessary to develop a satisfactory metallographic procedure for investigating the effects of radiation on the material.

Standard metallographic procedures proved to be unsatisfactory for obtaining the desired results. This was due largely to the extreme variance in hardness of the boron carbide and Zircaloy-2 matrix. Grinding with silicon carbide abrasive papers often resulted in the loss of the majority of the boron carbide particles and also lowered the Zircaloy-2 matrix below the level of the boron carbide particles. Standard polishing techniques resulted in the further loss of particles and lowering of the matrix. The matrix was also often extremely cold worked, resulting in the loss of the true structure of the Zircaloy-2 when examined with polarized or tinted illumination.

To overcome these difficulties, and obtain satisfactory metallographic results, the following procedures and equipment were used at the Knolls Atomic Power Laboratory Remote Metallography Cell.

EQUIPMENT

The samples were processed in a cell with 9-inch steel walls having an internal working space of 4 by 14 feet. The cell interior is divided into three modules, each being separated from the other with 1/4-inch plexiglass walls. The first module is used for the grinding operations, the second for polishing and mounting, and the third for the etching, replicating and cleaning of samples. Each module is equipped with two model No. 4 Argonne type master slaves, for the remote operation of the in-cell equipment.

Buehler Automet polishers, modified slightly for remote use, are used for all the grinding operations. These units enable the processing of five specimens at one time, with polishing and grinding pressures of up to 60 lbs. There are three of these units located in the first module for grinding, and two in the second module for polishing operations. Figure 1 shows two of the Automet polishers, specimen mounting press and specimen loading assembly.

The samples were examined with the use of a Bausch and Lomb Research Metallograph located at one end of the cell. The stage of the metallograph is shielded with 4 inches of lead. The carbon light source and camera are positioned on the outside of the shielding for convenient use and maintenance. Several controls were extended through the front of the shielding to enable the remote operation of the various stage movements such as translation, aperture decentering, coarse and fine focus, illumination turret, and objective lens turret. Figure 2 is a general view of the shielded metallograph and controls.

MOUNTING

The samples to be processed are mounted in 1-1/4 inch mounts with the use of the Precision-Jarrett mounting press shown in Fig. 1. Melmac powder is used to make the mounts. A stamped metal washer is also placed in the back of the mounts to identify the specimen. The usual size of the specimens to be processed is approximately 3/8 by 1 inch.

COARSE GRINDING

For the coarse grinding of the specimens the standard bronze wheel, used with the Automet polishers, was replaced with a cast iron lapping wheel. This wheel is commercially available for use with the Automet polishers and requires no modification for remote use. The samples are then lapped on the cast iron wheel, with 170-mesh diamond paste, at approximately 15 lbs. pressure. The length of grinding time depends on the amount of stock removal that is desired. The amount of stock removed is approximately 1 mil per minute, at 15 lbs. pressure, when five specimens are being processed. A water-soluble diamond paste thinner is applied directly onto the wheel, during lapping, for lubrication of the samples. The sample and sample holder are cleaned with cotton swabs and water after the coarse grinding operation is completed. This grinding is used for large amount of stock removal and to keep the boron carbide particles and Zircaloy-2 at the same surface level.

FINE GRINDING

For fine grinding the cast iron lapping wheel is replaced with a spiral-grooved lead lapping wheel. This wheel is also commercially available for use with the Buehler Automet polishers with no modification for re-

mote use. The samples are ground on this wheel for approximately 5 minutes at 10 lbs. pressure with the use of 30-micron diamond paste. Water-soluble diamond paste thinner is again used as the lubricant during grinding. After fine grinding the samples and holder are cleaned with cotton swabs and water. This operation keeps the boron carbide particles and Zircaloy-2 matrix at the same surface level and also keeps the boron carbide particle tear-out at a minimum. It also removes the majority of the deep scratches left on the sample from the coarse grinding operation.

COARSE POLISHING

The samples are coarse polished on a suede type polishing cloth for approximately 2 to 3 minutes, at 5 lbs. pressure, with the use of 6-micron diamond paste. A 10% oxalic acid and water solution is applied directly onto the wheel during polishing. This tends to prevent the formation of cold work in the Zircaloy-2 matrix and also acts as a lubricant during polishing. It is believed that the light pressure used prevents the lowering of the matrix from the boron carbide particles, which in turn prevents the tearing out of the particles since they remain at the same level as the Zircaloy-2 matrix and do not catch on the cloth. It has also been found that long periods of coarse polishing will eventually lower the Zircaloy-2 matrix and result in the loss of boron carbide particles. This coarse polishing operation also removes the majority of the scratches left in the sample from the fine grinding operations.

FINE POLISHING

The samples are then fine polished on a suede type polishing cloth with the use of 1/2-micron diamond paste and 0.1 micron aluminum oxide polishing powder, at 5 lbs. pressure, for approximately 1 to 2 minutes. A 10% oxalic acid and water solution is again applied directly on to the polishing cloth to act as a lubricant, and to prevent the formation of cold work in the Zircaloy-2 matrix. The light polishing pressure again prevents the tearing out of the boron carbide particles, and the lowering of the Zircaloy-2 matrix. This final polish also removes the majority of the scratches that may have been left from the coarse polishing operation. Also, the Zircaloy-2 matrix is left quite free of cold work and is easily examined with polarized or tinted illumination.

In the final polishing procedure, the 1/2-micron diamond paste may be replaced with 3-micron paste for faster polishing. However, the results obtained are not quite as fine as those when the 1/2-micron paste is used.

ETCHING

As a rule, the specimens are not etched since this results in a definite lowering of the Zircaloy-2 matrix from the boron carbide particles, and also a loss of many of the particles. However, if it is desired to etch the samples, a 1% HF etch-polish technique is used. The specimen is held on the fine polishing wheel with one manipulator. The wheel is then rotated and the 1% HF and water solution is applied onto the wheel with the second manipulator in the module. The sample is normally etched in 10 to 20 seconds. This etch has no apparent effect on the boron carbide particles but enables the general structure of the Zircaloy-2 matrix to be examined with bright field illumination.

RESULTS

With the use of the polishing procedure described, excellent retention of the boron carbide particles has been obtained.

Fig. 1 - Automet polishers, specimen mounting press and specimen loading assembly.

Fig. 2 - General view of the shielded metallograph and controls.

100X - Bright field

Fig. 3 - General view of specimen
showing particle retention.

100X - Bright field

Fig. 4 - General view of specimen
showing particle retention.

50X - Pd. Lt.

Fig. 5 - General view of sample with polarized light.

500X - Bright field

Fig. 6 - Particle and matrix interface.

250X - Bright field

Fig. 7 - Particles and matrix.

250X - Tinted light

Fig. 8 - Particles and matrix structure.

250X - Pd. Lt.

Fig. 9 - Particles and matrix structure.

STRUCTURES IN THE ZIRCONIUM-IRON-TIN SYSTEM

Lee E. Tanner and David W. Levinson

Armour Research Foundation
Illinois Institute of TechnologyINTRODUCTION

The zirconium-iron-tin system was investigated as a part of an Atomic Energy Commission program on the heat treatment of zirconium-base alloys. The area of interest was the zirconium-rich corner bounded by the binary compounds $ZrFe_2$ and Zr_4Sn between 200° and $1100^\circ C$. The important microstructures observed in this system are presented in this paper.

EXPERIMENTAL PROCEDURESAlloy Preparation and Heat Treatment

Alloys in the form of 10-, 15-, and 25-gram button ingots were prepared by nonconsumable electrode arc melting techniques in an inert atmosphere. (1) Materials used were of the highest purity including "Grade 1" (hafnium-free) iodide zirconium*, National Research Corporation gas-free iron (99.95% pure), and Vulcan Detinning Corporation tin (99.998% pure). Prior to equilibration anneals at temperatures between 200° and $1100^\circ C$ the ingots were homogenized at 800^{**} and $850^\circ C$. Cold work was employed where alloys were sufficiently ductile (i.e. those having <10% Fe and <10% Sn.) Annealing treatments were carried out with specimens sealed in Vycor bulbs which were either evacuated or under a partial pressure of argon. At temperatures where melting was expected, samples were wrapped in molybdenum sheet for protection when annealed in groups. Otherwise, they were annealed in separate bulbs. Alloys were quenched by breaking the bulbs under cold water.

(1) M. Hansen, H. D. Kessler and D. J. McPherson,
Trans. ASM 44 (1952) 518

* The iodide zirconium used for dilute alloys was initially electron bombardment melted for further purification.

** dilute alloys

METALLOGRAPHIC PROCEDURES

Heat-treated samples were mounted in Bakelite and rough polished on a belt sander employing 120-grit, silicon carbide abrasive to obtain a preliminary flat surface. The deep scratches were removed by lapping on a Lapmaster unit using Lapmaster 1900 compound followed by several passes on No. 600 paper. Final polishing involved two steps: polishing on airplane cloth impregnated with diamond paste in a kerosene vehicle, then on silk impregnated with "Linde B" synthetic sapphire, distilled water being the vehicle. The etchant used for most of the alloys was a solution of 20% HF and 20% HNO_3 in glycerine which was swabbed on the polished surface. Etching times ranged between 1 and 30 seconds. The dilute alloys required careful etching with the above solution (or one made more dilute by the addition of glycerine) for periods of 1 to 10 seconds followed by a water rinse. Subsequently, the specimens were swabbed rapidly by a solution of 50% H_2O_2 , 45% HNO_3 , and 5% HF. All etching was followed by a water and then alcohol rinse with drying accomplished in a warm air flow.

DISCUSSION

Three partial isothermal sections for 1000, and 900, and 700°C are presented in Figs. 1, 2 and 3, respectively, and show all the phase relations of the system. The important features are as follows:

- (1) A ternary phase, θ , was found having the approximate composition of 8% Fe and 24.5% Sn.
- (2) A four-phase ternary eutectic ($L \rightleftharpoons \beta + \theta + \text{ZrFe}_2$) occurs between 930° and 935°C.
- (3) The binary peritectoid reaction ($\beta + \text{Zr}_4\text{Sn} \rightleftharpoons \alpha$) produces α at 980±20°C which leads to the four-phase ternary peritectoid $\beta + \text{Zr}_4\text{Sn} \rightleftharpoons \alpha + \text{Zr}_5\text{Sn}_3$ followed by another four-phase ternary peritectoid $\beta + \text{Zr}_5\text{Sn}_3 \rightleftharpoons \alpha + \theta$, at temperatures above 900°C.
- (4) It is likely that a four-phase ternary peritectoid ($\beta + \theta \rightleftharpoons \alpha + \text{ZrFe}_2$) occurs at a temperature just above the binary zirconium-iron eutectoid (which takes place between 790° and 800°C).

Evidence of the ternary eutectic is graphically displayed in the first two microstructures. An alloy of 13% Fe and 7.5% Sn quenched from 935°C shows the primary crystals of θ (the dark phase) and ZrFe_2 (the twinned, white phase) in a frozen liquid matrix (Fig. 4). In Fig. 5 a typical

eutectic of $\beta + \theta + \text{ZrFe}_2$ is seen in the same alloy quenched from 930°C .^{*} The color of θ depends on the length of etching time and varies from light beige for a rapid swab to a deep brown for longer etches. ZrFe_2 is always white and, if the particles are large enough, twins^{**} are observed. A narrow two-phase field exists between θ and ZrFe_2 at all temperatures. This structure is seen in Fig. 6 of a 25% Fe, 15.5% Sn alloy quenched from 1100°C . Nearly single-phase θ is shown in Fig. 7 of a 7.5% Fe, 24.5% Sn alloy annealed at the same temperature.

The θ phase is metallographically identical to Zr_4Sn , and it was necessary to use X-ray diffraction techniques to establish that they were separate phases. Zr_4Sn has little or no solubility for iron and is seen as the major component of the three-phase structure ($\text{Zr}_4\text{Sn} + \alpha + \text{Zr}_5\text{Sn}_3$) in Fig. 8 of a 0.5% Fe, 24.5% Sn alloy quenched from 900°C . The darker phase, Zr_5Sn_3 , has a tendency to be eaten out if the etching is too long as is evidenced in this photomicrograph. An alloy of 2% Fe, 24.5% Sn annealed at this temperature shows $\theta + \alpha + \text{Zr}_5\text{Sn}_3$ (Fig. 9) where a variation of shading is observed in the θ peritectoid walls, as well as the θ precipitate rejected from α . This may be due to non-equilibrium distribution of the alloying elements in the phase. Such is not the case for the θ in a 5.1% Fe, 24.5% Sn alloy (Fig. 10) also quenched from 900°C .

At 900°C an alloy of 2% Fe, 9% Sn has an $\alpha + \beta + \theta$ structure, as seen in Fig. 11, while at 800°C it is $\alpha + \theta$ (Fig. 12). Most of the θ in the latter has precipitated in a Widmanstätten manner and is continuous with equiaxed θ which was probably formed during the 850°C homogenization treatment.

Typical β , which has transformed martensitically to α' upon quenching, is seen in Fig. 13 of a 1% Fe, 1% Sn alloy annealed at 1100°C . The β is retained in the $\alpha + \beta$ structure of a 2% Fe, 1% Sn alloy (Fig. 14) but transformed in the $\beta + \text{ZrFe}_2$ structure of a 3% Fe, 0.5% Sn alloy (Fig. 15), both quenched from 800°C .

In an effort to determine the solubility of iron and tin in α at low temperatures, extremely dilute alloys were prepared. A special etching technique was necessary to prevent the precipitate particles from being etched out while also bringing out the α grain structure. It appears that the maximum solubility of iron in α is lower than the minimum iron addition made ($<0.01\%$) at temperatures of 600°C and lower.

^{*} The lowest eutectic in both binary systems is between 945° and 950°C in the zirconium-iron system.

^{**} ZrFe_2 is f.c.c.

Tin solubility is somewhat higher, as is apparent from the binary system.⁽²⁾ Therefore, α exists as a narrow field close to the zirconium-tin system. Figure 16 shows a structure of 100% α (0.76% Sn at 700°C). ZrFe_2 particles and hydride platelets are evident in Fig. 17 (0.052% Fe at 700°C). The particles seen in Fig. 18 could be θ and/or Zr_5Sn_3 (0.028% Fe, 0.36% Sn at 700°C). Hydride platelets are prominent once again.

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ORAL DISCUSSION

Question: What was the concentration of the H_2O_2 used in the etch for alloys?

Answer: 30%

Question: Was there any reaction between the molybdenum sheet and the alloys when the latter melted and, if so, how did you account for this when interpreting the structures?

Answer: An extremely hard compound was produced from the reaction of the molybdenum sheet with the molten zirconium-iron-tin alloys. To eliminate contamination and thus be able to make valid interpretation of the structures quenched from these temperatures, duplicate samples were annealed in separate Vycor bulbs. These were quenched as soon as melting was observed.

Question: How would you interpret the structures found in Zircaloy based on the results of this phase diagram study?

Answer: Pure zirconium-iron-tin alloys approximating the various compositions of Zircaloy in the range of room temperature to 400°C would be expected to exhibit structures of $\theta + \text{ZrFe}_2$, θ , or $\theta + \text{Zr}_5\text{Sn}_3$ in equilibrium with α . Hydride particles would also be observed. However, Zircaloy also contains some nickel and chromium, as well as a larger amount of oxygen than in these alloys, and it is unknown as to what extent these elements are soluble in the existing phases or whether additional compounds are formed.

Fig. 1 - Partial isothermal section at 1000°C, Zr-Fe-Sn system.

Fig. 2 - Partial isothermal section at 900°C, Zr-Fe-Sn system.

Fig. 3 - Partial isothermal section at 700°C, Zr-Fe-Sn system.

250X

Fig. 4 - 13% Fe, 7.5% Sn quenched from 935°C, θ + ZrFe_2 + solidified liquid.

500X

Fig. 5 - 13% Fe, 7.5% Sn quenched from 930°C, θ + ZrFe_2 + β .

500X

Fig. 6 - 25% Fe, 15.5% Sn quenched from 1100°C, θ + ZrFe_2 .

500X

Fig. 7 - 7.5% Fe, 24.5% Sn quenched from 1100°C, nearly 100% θ .

500X

Fig. 8 - 0.5% Fe, 24.5% Sn quenched from 900°C, $Zr_4Sn + Zr_5Sn_3 + \alpha$.

500X

Fig. 9 - 2% Fe, 24.5% Sn quenched from 900°C, $\theta + Zr_5Sn_3 + \alpha$.

500X

Fig. 10 - 5.1% Fe, 24.5% Sn quenched from 900°C, $\theta + Zr_5Sn_3 + \alpha$.

500X

Fig. 11 - 2% Fe, 9% Sn quenched from 900°C, $\alpha + \beta + \theta$.

1000X

Fig. 12 - 2% Fe, 9% Sn quenched from 800°C, $\alpha + \theta$.

250X

Fig. 13 - 1% Fe, 1% Sn quenched from 1100°C. α' (transformed β).

500X

Fig. 14 - 2% Fe, 1% Sn quenched from 800°C, $\alpha + \beta$.

500X

Fig. 15 - 3% Fe, 0.5% Sn quenched from 800°C, α' (transformed β) + ZrFe_2 .

1000X

Fig. 16 - 0.76% Sn quenched from 700°C, α .

1000X

Fig. 17 - 0.052% Fe quenched from 700°C. α + ZrFe_2 + hydride.

1000X

Fig. 18 - 0.028% Fe, 0.36% Sn quenched from 700°C. α + (θ and/or Zr_5Sn_3) + hydride.

CONTRIBUTION OF THE JOMINY-TYPE END QUENCH TO METALLOGRAPHIC
PHASE IDENTIFICATION IN URANIUM*

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ABSTRACT

Jominy-type end quench tests provide a convenient means of exploring uranium transformation characteristics. Metallographic examinations indicate that macrostructural data provide the best means of phase identification and interpretation. (See Figs. 1 and 2 on next page.)

*Report BRB-44, Bridgeport Brass Company

2X

Fig. 1 - Typical gamma-cooled macrostructure.
Specimen 6. Gamma-treated at 1000°C -
15 min. and end quenched. HCl and
 HNO_3 Etch.

2X

Fig. 2 - Typical beta-cooled macrostructure. Specimen 2. Double beta-treated at 760°C - 15 min. and end-quenched. HCl and HNO_3 Etch.

IMPURITY CONTENT DETERMINATION OF URANIUM AND HIGH-URANIUM ALLOYS BY VISUAL INSPECTION

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INTRODUCTION

During a metallographic examination of uranium and its alloys, the question of the impurity content often arises. Normally, and for accuracy, this question is answered by one of the conventional methods of chemical, spectrographic, or vacuum fusion analysis. These procedures, however, can result in an appreciable time delay which is undesirable in many cases. A quick and relatively accurate estimate would suffice in most instances in order to determine whether or not new material or changes in melting and fabrication procedures are required. This can be accomplished by microscopic inspection of an "as-polished" surface.

METHODS

The most prominent nonmetallic inclusions to be found in uranium are: UC, UO_2 , UN, and UH_3 , with the carbon and hydrogen contents usually of greatest interest. Up to the present time, the most effort, with the greatest success at this laboratory, has been devoted to determining the amount of hydrogen present. Less time has been spent on carbon analysis but the accuracy has been as high as ± 50 ppm in samples containing 250 to 700 ppm. Only a token effort has been expended on the analysis of nitrogen and oxygen but visual analysis probably could be expanded to include them also.

The technique of visual impurity analysis is quite similar to the determination of grain size in that the observer compares the sample in question with photomicrographs of known impurity content. Micrographs shown here are those used for comparison at Battelle. Some of these were taken of samples which were purposely impregnated with an impurity while others are of routine metallographic specimens. In order to simplify reporting, the different impurities will be discussed separately. The technique appears to be generally applicable to either as-cast material or material fabricated by normal methods where involved heat treatments are not encountered. While no attempt has been made to analyze heat-treated material, it is probable that heat treatment will affect both the amount and distribution of the precipitating impurity phases.

Hydrogen. As mentioned above, the most effort and success has involved analysis of hydrogen content. Published information⁽¹⁾ has established that the solubility of hydrogen in uranium at room temperature is extremely low, 0.6 to 0.7 ppm. Using this level as a starting point, it is comparatively easy to establish as many standard micrographs as is felt to be necessary. At Battelle only two are commonly used. Figure 1 shows a sample containing an analyzed 0.8 ppm hydrogen. Notice that there is only one very small UH_3 inclusion showing. Figure 2 shows a sample containing 3.2 ppm hydrogen.

Samples have come into our laboratory containing hydrogen in amounts varying from approximately 1 to about 5.5 ppm. Accuracy of the metallographic analysis has been ± 0.2 ppm in many cases in the 1 to 3.5 ppm range and ± 0.5 ppm on the samples of higher content. This is usually sufficiently accurate for an initial analysis.

It should be noted that separate micrographs for different magnifications should not be necessary if the observer has a good grasp of the relative sizes of microscopic fields and inclusions. However, for UH_3 inclusions, size and shape are very important. If there are large inclusions either of the usual "feather" shape or of the circular type, a mental estimate is made to reduce these larger shapes to a series of small ones similar to those shown in Fig. 2. At first, this may seem to involve quite a bit of time but an operator can do this balancing after some practice with very little conscious thought and arrive at a reasonably accurate estimate.

Carbon. No absolute standards have been set up for carbon analysis; however, Figs. 3, 4, and 5 show fields from samples that analyzed 300, 700, and 1300 ppm carbon, respectively. Interpolations can be made between these amounts without undue difficulty. Concentrations of carbon above 1000 ppm and below 200 ppm are rather difficult to estimate. Usually, though, samples of metal which are of interest contain 300 to 600 ppm carbon.

At this time the accuracy, as indicated previously, is about ± 50 ppm. The accuracy undoubtedly could be greatly improved with more study.

Size and shape of the inclusions themselves is again important as with UH_3 . In arc-melted or chill-cast ingots the carbides can assume a dendritic shape similar to these shown in Fig. 6. No reliable estimates of the amount of carbon present have been made on metal in this condition.

(1) F. A. Rough and A. A. Bauer, "Constitution of Uranium and Thorium Alloys", BMI-1300, June 2, 1958.

When dealing with large UC inclusions of regular geometric shape, a mental subdivision to the sizes pictured in the standard micrographs is possible.

Nitrogen. Inclusions of UN are usually either of two general shapes. They can assume the "Chinese script" form as seen in Fig. 2 or appear as angular inclusions similar to the carbides appearing in Fig. 1. If they are present in the second shape, a reliable method to differentiate between the carbide and nitride is to immerse the sample in a solution of 50 percent nitric acid in water. After 5 to 20 seconds in this solution, the UC or high-carbon inclusions are darkened as shown in Figs. 3, 4, 5, and 6 while the nitrides are unaffected.

Figures 7 and 8 show uranium specimens in which Kjeldahl analyses of 20 and 50 ppm nitrogen were obtained. Since the solubility of nitrogen in uranium is very low⁽¹⁾ and the upper limit encountered in most routine samples in our laboratory is approximately 100 ppm, these micrographs serve as standards. No attempt has been made to visually analyze for nitrogen except to place it in "high" (50 to 100 ppm) or "low" (50 ppm or less) categories. The actual nitrogen analyses are usually quite low and the inclusions do not seem to affect the fabricability of the metal so that the question of how much is present rarely arises.

Since UN and UC seem to be mutually soluble and can form complex U(C-N) inclusions, the visual analysis can sometimes be of no help. When the U(C-N) combinations are present the nitric acid etchant will affect them according to the compositions of the inclusions themselves; that is, high-carbon inclusions will darken while those with high nitrogen content will not. When either carbon or nitrogen is in combination in small amounts the over-all visual analysis is not appreciably affected. However, if both are present in relatively high quantities, estimations of content will be poor.

Oxygen. No attempts have been made to analyze visually for oxygen content. Limited efforts to add oxygen to uranium in order to provide standards have been unsuccessful due to the tendency of the UO_2 inclusions to segregate in clusters. Oxygen in ingot or fabricated material also is usually either clustered or is not present in sufficient quantities to appear as UO_2 . As a result of this tendency toward segregation into clusters, widely varying oxygen contents between different areas of a sample result, rendering visual analysis difficult if not impossible.

CONCLUSIONS

On the basis of work performed it has been found that with suitable standards and with some practice a metallographer can make reasonably accurate estimates of the amounts of hydrogen and carbon present in uranium samples by visual metallographic analysis. It is indicated that nitrogen analyses may be similarly performed.

Routine metallographic specimens of uranium with alloy additions of up to 10 w/o niobium and molybdenum have also been successfully analyzed by visual examination with verification being obtained by subsequent conventional analyses. The method has not been applied to other alloy systems but the method can in all probability be applied, particularly for hydrogen, to material containing up to about 5 w/o alloy additions.

500X

N-7440

Fig. 1 - High purity uranium impregnated with hydrogen.
Vacuum fusion analysis 0.8 ppm H_2 .

500X

N-7443

Fig. 2 - High purity uranium impregnated with hydrogen.
Vacuum fusion analysis 3.2 ppm H_2 . Feather
like inclusions are UH_3 , others are UN.

250X

N-10507

Fig. 3 - Uranium containing an analyzed 300 ppm carbon etched with 1:1 HNO_3 : H_2O .

100X

RM-9706

Fig. 4 - Reactor grade uranium containing 700 ppm carbon 1:1 HNO_3 : H_2O etchant.

250X

N-14728

Fig. 5 - uranium containing 1300 ppm carbon. As polished.

250X

N-11976

Fig. 6 - Demonstrating dendritic shapes which uranium carbides, nitrides, and oxides can assume when chill cast etched with 1:1 HNO_3 : H_2O .

500X

N-3148

Fig. 7 - Uranium analyzed to
contain 20 ppm N_2 .

500X

N-3147

Fig. 8 - Appearance of 50 ppm N_2
in uranium.

CINTEL FLYING SPOT MICROSCOPY

A New Laboratory Tool for Metallographers

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Metallographers frequently face the problem of furnishing particle size distribution data on a wide variety of materials including powdered metals, metallic compounds, ceramics, abrasives, discrete inclusions, of material in which the structure itself is fundamentally disperse and heterogeneous. It is generally agreed, of all the tasks encountered in the laboratory, the size characterization of small particles, of any sort, by direct observation in the microscope is the most onerous. Making measurements of projected, or photographically recorded images of the particles affords little, if any, real relief. If the sample volume for this type work is great, the job becomes tedious to the extent that human error can, and often does, becloud the results. Over the years, it has been the fervent hope of anyone of necessity engaged in this sorry business, that someday, someone would develop a mechanical or electronic means of doing the job. Today there exists such a machine, capable of furnishing accurate, reproducible results in a short period of time, available as a commercial instrument, under the trade name -- Cintel Flying Spot Particle Resolver (Fig. 1).

The principle of operation embodied in this instrument can be most simply summarized as follows (Fig. 2): A scanning spot on a cathode ray tube is imaged through a suitable optical system so as to scan a very small field of the subject. The light passing through the subject will be modulated in accordance with the density and configuration of the subject material and is collected by a photo multiplier cell. Thus the density/time relationship has been changed to a voltage/time relationship. The electrical signal, so formed, is amplified, and can be used to modulate a TV type monitor cathode ray tube, or can be fed to a computing system, using a magnetic drum memory, from which particle count and size distribution data is read off directly on decastrons, as units, tens, and hundreds.

In the Cintel instrument the size increments are set up electronically by a "quantizer" (Fig. 3). Here the values of the increments in terms of microns are established by the optical lens combinations used in the microscope, or the magnification of photographic plates placed at the face of the scan tube. It was found that if one placed a photographic plate or film at the face of the cathode ray scanning tube, an image of

the field shown on the plate would appear on the monitor tube at a 4X magnification, and would be accepted into the counting circuit in the same manner as dispersed particles at the normal optical stage position. This makes possible the use of reversals of electron micrographs on 2 x 2 lantern slides, and a considerable extension of the working range of the instrument from 160 microns down to whatever particle size can be resolved by the electron microscope in use.

The imperative need for a method, other than physical counting, which would embrace such a range, arose at Oak Ridge National Laboratory, when, during the course of the thorium slurry program, it was learned that slight differences in particle size and shape would critically affect the rheological properties of the material, and that a great number of samples would have to be sized. An investigation made by the Optical and Electron Microscopy Section of the Analytical Chemistry Division to determine the possibilities existing for doing such counts mechanically or electronically, disclosed that two American companies were developing such equipment, and a third, the Cinema Television Co., Ltd., of England was currently producing an instrument available through the Marconi Instrument Co., of New Jersey. Arrangement was made for a demonstration of the latter instrument, during which a wide variety of powder samples were submitted for size analysis. Comparison was made between these results, and data collected from physical measurements made of the same samples. The results from two such trials are shown in Figs. 4 and 5. On the basis of such tests, equipment design, electronic stability, and other factors, including price, the Cintel Flying Spot Resolver was purchased.

Since its installation a year ago, almost daily use of the instrument has been made for all types of material, including hundreds of thorium samples. It has proved invaluable in those size regions where other techniques, such as sedimentation, cannot provide suitable data, namely material below the 0.5 micron size. For example, in a study of the relationship existing between line broadening in electron diffraction patterns and measured particle size, many electron micrographs of particles of vaporized metals were made and the size range established by the Cintel. A representative field from such a sample with the size range plot is shown in Fig. 6. The particles illustrated are ideal for machine count - they are nearly spherical, and a reversal would have excellent contrast. Certain types of particles are troublesome to the extent that they are not suitable for direct presentation to the instrument because of poor contrast in the body or edge of the particles (Fig. 7), geometrical shape, or irregular shape (Fig. 8). In the first, the difficulty can usually be overcome by photographic means, or fine adjustment of the contrast controls on the instrument - much like the contrast controls on a TV set. Elongated or acicular particles can be done with two scans, the second being made after rotating the sample or photographic plate 90°. Irregulars, because they provide reentrant points for the scan spot, will cause multiple counts for certain particles. However, it has been found both in practice and statistically that errors arising from particles of uniform irregular shape will tend to even out over a large field.

Possible other uses of the instrument, aside from counting free particles, include counting, and checking the uniformity of, openings in wire cloth, wire cloth, wire screens, and electromesh, and measuring pore sizes in metal filters and similar materials (Fig. 9). Providing sufficient contrast can be obtained, counting and measuring inclusions can be done by using an intermediate photographic step. A reflection type instrument to directly do the latter is now under development. With the Cintel, the time needed to establish particle size distributions is so short, roughly fifteen minutes per prepared sample as against a possible several hours with other methods, that it is expected many other applications will be found where previously time and expense had made size control impractical.

Fig. 1 - Cintel Flying Spot Particle Resolver.

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Fig. 2 - Schematic showing principal electronic components of Cintel Flying Spot Particle Resolver.

Fig. 3 - Quantizer, or particle sizing unit of Cintel Flying Spot Particle Resolver.

Fig. 4 - Plot of typical results of Cintel versus physical count.

Fig. 5 - Plot of typical results of Cintel versus physical count.

Fig. 6 - Ideal field for Cintel particle sizing.

40,000X

Fig. 7 - Vaporized indium particles having low body and edge contrast.

40,000X

Fig. 8 - Vaporized indium particles of irregular shape.

Fig. 9 - Cintel evaluation of pore size in SS filter.