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*Development of Plutonium  
Transmission Electron Microscopy*

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# Development of Plutonium Transmission Electron Microscopy

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# DEVELOPMENT OF PLUTONIUM TRANSMISSION ELECTRON MICROSCOPY

by

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## ABSTRACT

Efforts to develop routinely successful methods to prepare samples for transmission electron microscopy are described. Since this work has been performed intermittently for nearly 20 yr, the background, the technical need for the work, and the future plans all are also discussed.

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## I. INTRODUCTION

The six allotropic forms of plutonium and their unusual transformations are of metallurgical interest, and understanding the basic mechanisms that govern the physical behavior of plutonium is of particular interest. Transmission electron microscopy (TEM), a standard tool for such metallurgical studies, requires quality thin foils for successful investigations.

The TEM of metallic plutonium and plutonium alloys has had a long and frustrating history. Aside from the difficulties of developing thinning techniques for such an easily oxidized material, the safety problems of handling this toxic material compound the experimental problems.<sup>1-2</sup> Several attempts to solve these problems for plutonium TEM have been made at Los Alamos, originally in 1963 through 1967 and now in the current work of 1981 through 1983. The several efforts made on plutonium from 1967 to the present concentrated mostly on films made directly by vapor-deposition<sup>3</sup> or splat cooling.<sup>4</sup> During this period reasonable progress was made on TEM of plutonium oxide and other actinide oxides.<sup>5-8</sup>

As early as October 1963, we attempted at Los Alamos to thin <sup>239</sup>Pu delta-phase alloys.<sup>9</sup> Delta-phase plutonium (alloyed with aluminum or gallium) was the

material of choice because of the ease of cold rolling and fabricating 5-mil-thick sheets suitable for subsequent electrochemical thinning. The mechanical behavior of delta plutonium and aluminum is similar, and delta plutonium has a large temperature/composition stability range. The original work was initiated primarily because high-purity electrorefined alpha plutonium was available for alloying. These early unsuccessful efforts produced foils with heavy surface layers of oxide in which only the metal grain boundaries could be discerned with confidence. In 1963, even metallographic preparation of delta plutonium was just beginning to approach acceptable quality.<sup>10-12</sup> Further, the foils deteriorated in the microscope; the electron-beam or hot-stage heating of these foils caused the metal to react with the residual oxygen in the microscope and rapidly transform into films of well-defined grains of PuO<sub>2</sub>. Additional problems were encountered: the window technique,<sup>13</sup> commonly employed at that time to produce TEM foils, generated an excessive amount of contaminated electrolytes and rinse solutions. Each foil preparation took many hours and then rarely produced viewable foils. Therefore, the first TEM studies of metallic plutonium were abandoned by 1967.<sup>14-15</sup> However, this early work established that specimen oxidation was the fundamental problem in plutonium TEM preparation. Techniques and equipment

of that era were incapable of solving the problems in any practical way, that is, without a large commitment of funds and personnel.

Several factors prompted the present work: (1) the availability of twin-jet electropolishers; (2) an additional 20 yr of experience in electron microscopy technology and equipment; (3) advances in plutonium metallography; (4) improved inert-gas quality and inert-system capabilities; (5) significant changes in TEM capability and TEM column vacuum technology; (6) availability of computer techniques for diffraction analysis;\* and (7) a renewed programmatic interest within our Laboratory.

## II. EXPERIMENTAL DETAILS

### A. Material

The five lots of high-purity  $^{239}\text{Pu}$ -1.5 wt% gallium delta alloy used in this study had been stored in vacuum since 1965. These lots and their experimental histories are listed in Table I. Spectrochemical results for Lots B and D are typical and are given in Table II.

### B. Equipment: Use and Development

1. The work reported here was performed on an argon-inerted Siemens Elmiskop I microscope operated at 100 kV. This instrument, designated for use with  $^{239}\text{Pu}$  since it was installed in 1957, is a bit of an antique and has limited dark-field (DF) and tilt capabilities. However, in the Siemens a foil is inserted into a recessed cup and then firmly held in place by a threaded collar; the whole assembly is then threaded into the sample chamber through an open port. It is a secure specimen-holding arrangement.

2. We also have available a JEOL\*\* 200B microscope with twice the accelerating voltage and modern tilting and DF accessories, which make it most attractive for use with plutonium. We have considered using the 200B for contaminated specimens since its purchase. However, in this microscope the foils are placed in the double-tilt or rotation holder, which is a relatively delicate tip of the whole specimen wand assembly. A 3-mm-diam bronze snap ring holds the foil in place, and then the wand is inserted through a small (1/2-in.-diam) gasketed port for preliminary pumpdown and final entry into the microscope column through an O-ring-sealed gate valve.

\*This information provided by Martin J. Carr, Sandia National Laboratories (1983).

\*\*JEOL, USA, Inc., 11 Dearborn Rd., Peabody, MA 01960.

TABLE I. Experimental Alloy History

Pu-1.5 wt% Ga	
Lot	Prior Treatment
A (11452)	5-mil-thick rolled coupons, 1/2 × 1 in., stored in glass vacuum capsules since 1965
B (11454)	5-mil-thick rolled coupons, 1/2 × 1 in., stored in glass vacuum capsules since 1965
C (11478)	Arc-melted button, stored in glass vacuum capsule since 1965
D (11481)	Arc-melted button, stored in glass vacuum capsule since 1965
E (11482)	Arc-melted button, stored in glass vacuum capsule since 1965
A-1	Coupons annealed 1 h at 400°C, 5/1981
C-1	Button, remelted to remove 17-yr irradiation damage, 6/1982
D-1	As-rolled from button cold rolled 98%, 12/1981
D-2	Coupons again annealed 1 h at 400°C, 1/1982
D-3	Coupons again annealed 1 h at 400°C, 10/1982
E-1	Button remelted to remove 17-yr irradiation damage, 6/1982

Before using this higher voltage instrument, we had to consider several factors: (1) possible contamination of the entry port and the problems that such contamination could cause with regular noncontaminated samples; (2) more important, the design of the specimen wand, which if the snap ring should dislodge or break, could lead to loss of the specimen; and (3) a means of transporting the wand and sample in open air from a hood to the microscope.

After some trial handling of the components involved, we decided to use the 200B essentially as designed and

TABLE II. Spectrochemical Results for Lots B and D

Element Analyzed	Lot B (ppm) <sup>a</sup>	Lot D (ppm) <sup>a</sup>
Li	<1	<1
Al	<5	<5
V	<3	<3
Ni	<5	7
Y	<25	<25
Cd	<10	<10
Ta	<100	<100
Be	<1	<1
Si	15	8
Cr	<5	<5
Cu	3	1
Zr	<100	<100
Sn	<5	<5
W	<10	<25
B	7	4
K	<5	<5
Mn	<1	<1
Zn	100	40
Hf	<25	<25
Bi	<1	<1
Nb	<10	<10
Ba	<2	<2
Re	<25	<25
Na	4	3
Ca	<3	<3
Fe	<5	<5
Rb	<10	<10
Mo	<3	<3
La	<25	<25
Pb	<5	<5
Mg	8	10
Ti	<5	<5
Co	<3	<3
Sr	<5	<5
Ag	<1	<1

<sup>a</sup>< represents limit of detection.

designated a spare specimen wand for this work. A simple, effective plastic loading tube was constructed for transporting and handling the specimen wand. Using this tube, one person could take the loaded wand from an open-faced hood, insert it into the column of the 200B in a controlled manner, and later remove the wand and return it to the hood. The handle of the wand could be kept contamination free. The loading tube was easy to make and therefore disposable if contaminated.

We used the snap-ring holding arrangement as designed with the added precaution of testing that it was firmly seated before each insertion of the specimen wand into the microscope.

The slight contamination of the entry port was easily removed.

3. A commercially available electrolytic twin-jet thinning unit manufactured by E. A. Fischione Instruments\* was used throughout this work.

4. An inert glove box with less than 5 ppm combined O<sub>2</sub> and H<sub>2</sub>O was used to investigate the role of atmospheric air on the electrothinning process. Its use is discussed in more detail below.

### C. Procedures

Because of the high atomic number and high density of plutonium, a sample to be examined by TEM must be only a few hundred angstroms thick. Such thicknesses are usually attained by electrochemically thinning sheets from a starting thickness of a few mils.

1. The commercial jet thinner was set up in an open-faced hood to explore systematically several polishing/etching solutions used in plutonium metallography. We used 4- to 5-mil-thick, 3-mm-diam specimen disks to determine whether the far better controlled thinning conditions and much shorter thinning times would improve foil quality over that of the window technique<sup>13</sup> used earlier. A family of phosphoric acid solutions was evaluated at ambient temperature in atmospheric air (Table III). These attempts produced only oxidized foils.

2. In an attempt to minimize oxidation by the atmosphere, a polyethylene glove bag was installed in an open-faced hood. The glove bag was equipped with silicon rubber feedthroughs for ac and dc power and was hooked up to a forepump vacuum system and to an argon tank. The jet thinner together with accessories and tools was set up within the glove bag. The bag was evacuated and flushed three times with argon before each thinning attempt, and during thinning the solution was

\*E. A. Fischione Instruments, 216 Red Oak Dr., Pittsburgh, PA 15239.

TABLE III. Electropolish Solutions Used

Solution Composition	Voltage (V)	Current (ma) for 3-mm-diam foil	Temperature °C	Results/Comments
<u>Successful Solutions</u>				
133 ml HAc <sup>b</sup> 25 ml H <sub>2</sub> O 25 g CrO <sub>3</sub>	15	5	10-13	IV inflection at 15 V, shiny long-lasting (passivated) surface, cold CH <sub>3</sub> OH rinse; ac current gave black surfaces. <sup>16</sup>
7 parts HAc <sup>b</sup> 1 part HClO <sub>4</sub>	11-12	1-2	12 (slush near 0)	I/V plateau at 11-12 V, very shiny foil upon perforation (5-10 min thinning time) which deteriorated rapidly in air. Area around perforation looked crumpled or ruffled. Edge badly cracked with only oxide detected by diffraction. In inert box surface quite durable.
<u>Unsuccessful Solutions</u>				
8 parts H <sub>3</sub> PO <sub>4</sub> 5 parts CH <sub>3</sub> OH 5 points glycol (8-5-5)	20-25	3-10	RT <sup>a</sup>	I/V inflection at 12 V, 20-30 min thinning time. Foil edge blunt, diffraction shows primarily oxide, CH <sub>3</sub> OH rinse, foils visually shiny, perforation near edge of foil (not centered).
2 parts tetraphosphoric 3 parts H <sub>2</sub> O 5 parts ethoxy ethanol (2-3-5)	20	25	RT <sup>a</sup>	I/V plateau at 10 V, but foil heavily etched. H <sub>2</sub> O rinse best, ethoxy ethanol next. Times varied unpredictably. Foils heavily wedged, holes well centered, foils visually shiny.
3 parts tetraphosphoric 2 parts H <sub>2</sub> O 5 parts ethoxy ethanol (3-2-5)	20	25	RT <sup>a</sup>	Same as for 2-3-5.

TABLE III. Electropolish Solutions Used (cont)

Solution Composition	Voltage (V)	Current (ma) for 3-mm-diam foil	Temperature °C	Results/Comments
53 parts H <sub>3</sub> PO <sub>4</sub> 100 parts H <sub>2</sub> O	4-5	50	RT <sup>a</sup>	No I/V plateau found, 2-min thinning time. Holes always lacy, even hole bridges thick. Foils deteriorated visually after rinsing.
10 parts HNO <sub>3</sub> 90 parts H <sub>2</sub> O	---	---		All foils black upon perforation.

<sup>a</sup>RT is room temperature.  
<sup>b</sup>HAc is CH<sub>3</sub>COOH, acetic acid.

bubbled with argon and the bag kept under constant argon flushing. No attempt was made to measure the oxygen content in the glove bag. Foils were washed in the argon-atmosphere bag after thinning and transported to the microscope in an argon-filled container. The foils saw air for approximately 30 s during loading into the microscope. The microscope column was retrofitted with an inert-backfill capability and was argon flushed three times just before foil insertion. However, minimizing ambient air contact made no difference in the appearance of the foils compared with full in-air operation. All foils examined were oxidized, and the thinned edges were deeply cracked back into the body of the foils (Fig. 1).

3. Next the chromic-acetic acid solution used in the 1960s was tried in the glove bag. Foils produced with this solution at room temperature gave the poor results shown in Figs. 2 (a) and 2 (b). However, the foils did look quite shiny after thinning and transferring in and out of the microscope; they appeared to have a passivated foil surface.

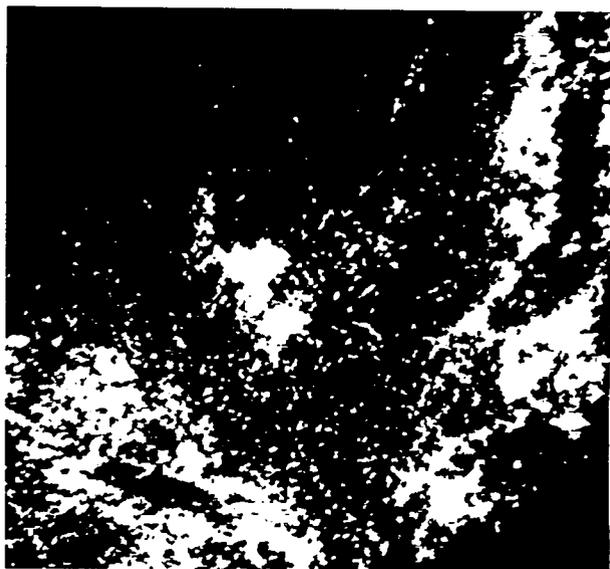
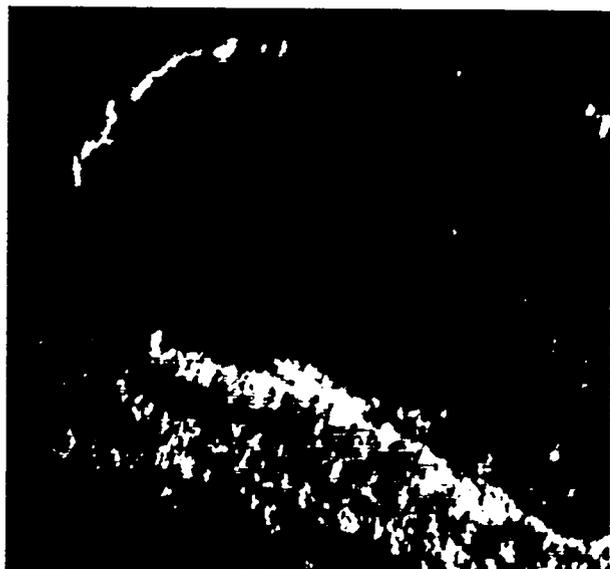
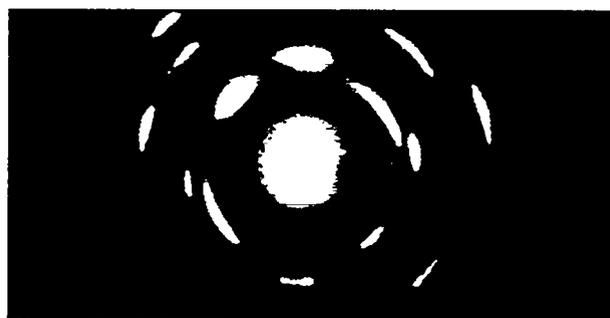
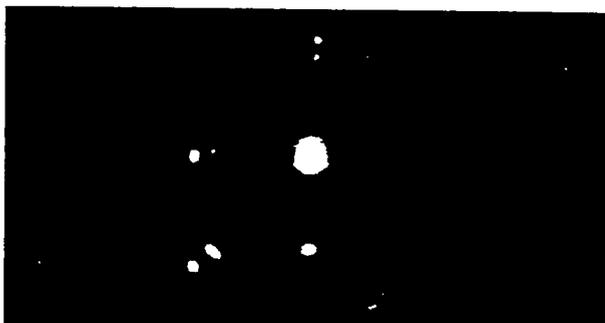
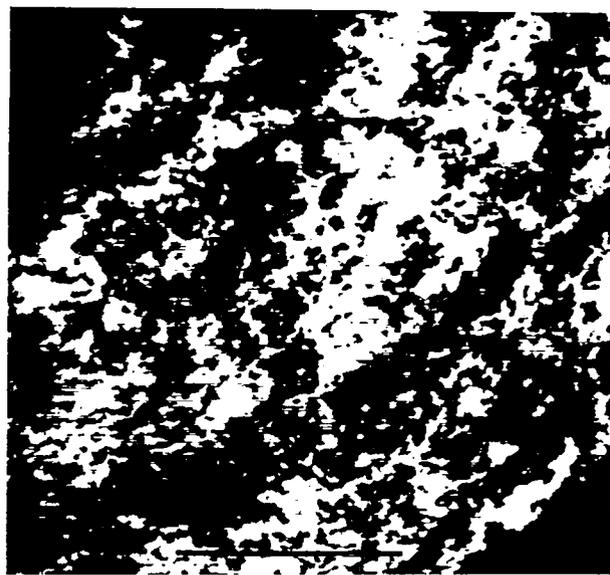
In another effort we cooled the thinning solutions to diminish chemical oxidation. We discarded the glove bag but retained the microscope column inerting. A marked improvement occurred when the chromic-acetic solution was chilled to 10-15°C. At less than 5°C, the solution froze into a thick slurry. Experimentation showed the current/voltage (I/V) inflection at 15V to be the best voltage. Perforations were produced at 12°C in 5-7 min. As important as cooling the thinning solution was prompt removal of the foil from the electrolyte upon perforation



Fig. 1. Example of oxidized, deeply edge-cracked foil produced in the argon-flush inert glove bag. Bar = 0.5 μm.

and immediately submerging it in chilled CH<sub>3</sub>OH followed by rinsing it with cold CH<sub>3</sub>OH. The alcohol containers were packed in dry ice before use.

Prior treatments (such as heating or cold working) of the metal and lot composition varied the quality of the foils produced and required slightly different thinning parameters, especially slight variations in bath temperature.



(a)

(b)

Fig. 2. Examples of oxidized TEM foils of Pu-1.5 wt% Ga thinned at room temperature in a chromic-acetic acid solution. Bar = 0.5  $\mu\text{m}$ .

4. Realizing the need for a better working atmosphere, that is, minimal oxygen and water, we attempted to thin foils in a temporarily available high-quality inert glove box (less than 5 ppm combined  $O_2$  and  $H_2O$ ). We installed a jet thinner in the recirculating argon glove box and used the 7:1 acetic-perchloric solution to thin several foils. These bright, shiny foils did not degrade in appearance after several hours in the inert atmosphere compared with the air-exposed foils, which when thinned in this solution, had turned black within 5-10 s. However, the shiny foils oxidized when transported, and when viewed in the 200B, only oxide was detected. Although perchloric acid is an oxidant, if on perforation the foil is removed and well rinsed in a quality inert atmosphere, the surface can be maintained. An I/V curve for the thinning conditions showed the plateau to be between 10 and 12 V at about  $9^\circ C$ .

This partial success and strong indication that a good inerting system is important have led us to design, fabricate, and test a system whereby plutonium foils can be thinned, transported, and examined in an inert environment. The next series of experiments will be carried out in this facility.

### III. RESULTS

The perforations in most thinned foils, even those with considerable viewable area, had a highly edge-cracked appearance (Fig. 3). The edges of the perforations often had a rim that the diffraction patterns showed to be totally oxide. The surface layers of oxide converging at the foil edge with no metal "core" remaining [Fig. 4 (a)] probably form this oxide rim. The bright-field (BF) micrograph [Fig. 4 (b)] shows a portion of a foil taken from type location 1 of the sketch [Fig. 4 (a)]. This portion is primarily metal together with its typical double-spot selected area diffraction (SAD) pattern [Fig. 4 (c)]. The DF micrograph [Fig. 4 (d)] taken from type location 2 of the sketch shows the oxide rim imaged by using the first ring (111) from the SAD pattern [Fig. 4 (e)].

Figures 2 (a) and 2 (b), produced by room-temperature thinning in chromic-acetic acid solution, show two sets of BF/DF micrographs and associated SADs. The BF micrograph in Fig. 2 (a) shows a foil in which only a small amount of metal is present, and the SAD shows double spots that will be discussed later in this section. The DF micrograph [Fig. 2 (a)] exhibits bend contours associated with the metal. The BF micrograph in Fig. 2 (b) shows a heavily oxidized foil in which the SAD exhibits oxide rings, and the DF micrograph [Fig. 2 (b)]

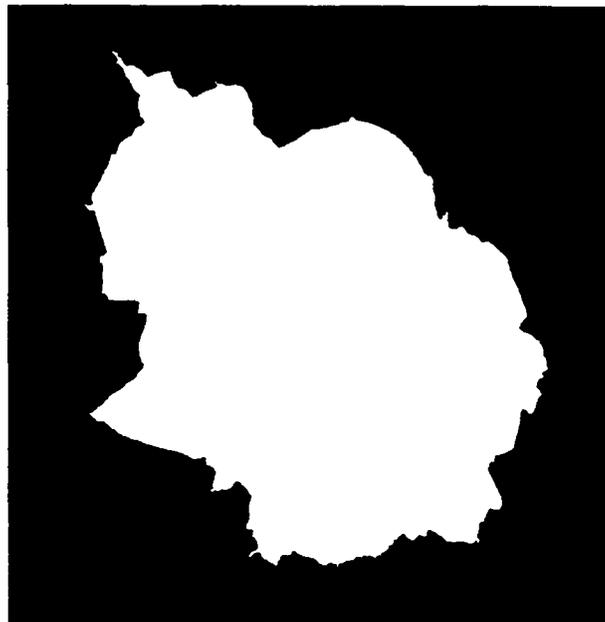
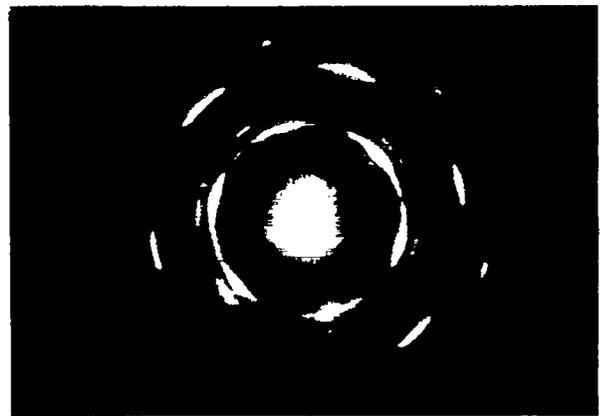
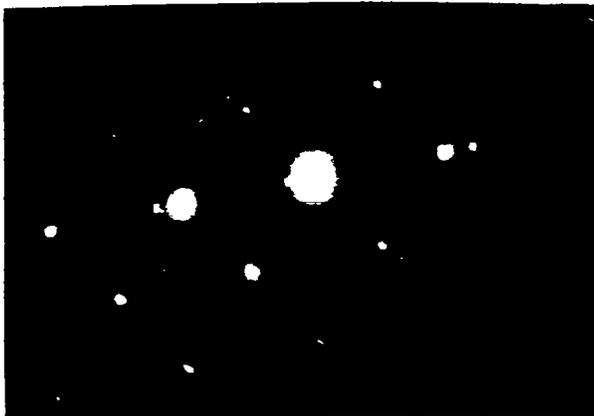
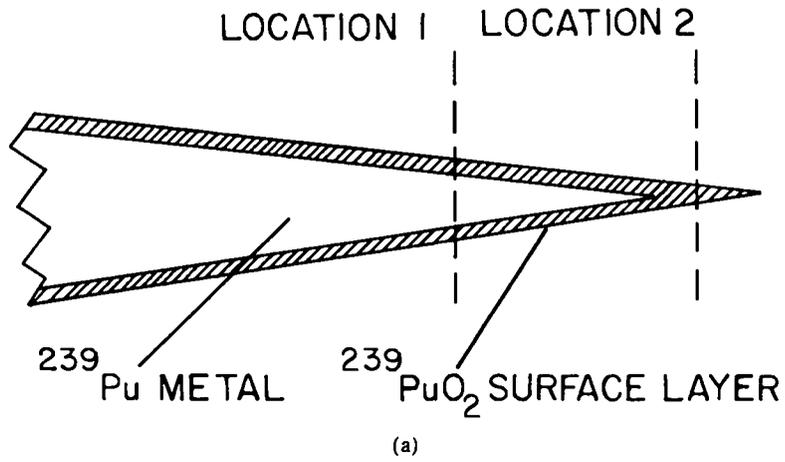


Fig. 3. A TEM perforation showing edge cracking.

shows oxide crystallites corresponding to the mottled BF texture.

The best of the prepared metal foils were electron transparent and revealed bend contours, boundaries of typical equiaxed grains, and occasional dislocations [Figs. 5 (a) and 5 (b)]. Severe bend contours were always present in the metal portion, particularly in the thin edges of the foils. We think that the strains in the foils are due to the epitaxial oxide layer exerting considerable stress on the extremely thin edge of the perforation (estimated to be about  $250\text{\AA}$  near the edge). This strain also contributes to the cracking of the metal/oxide sandwich. The difference in thermal conductivities of the two materials making up the sandwich may through differential expansion be an additional reason for the contours. We do not believe that these contours are handling artifacts caused by foil flexing because experience with other "dead soft" metals does not result in such structures. The thermal shock of the cold rinse could aggravate cracking and cause bend contours, but room-temperature-rinsed foils also were cracked and contained contours.

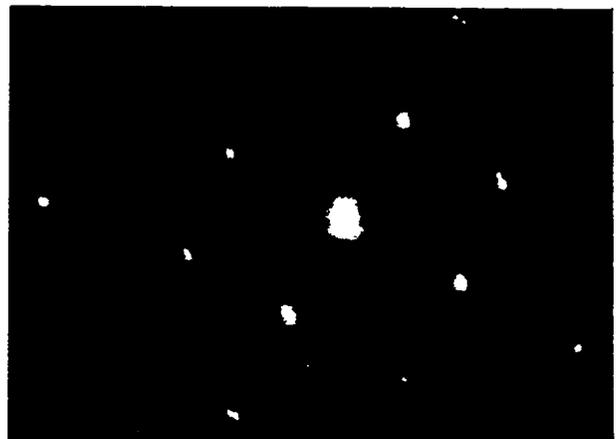
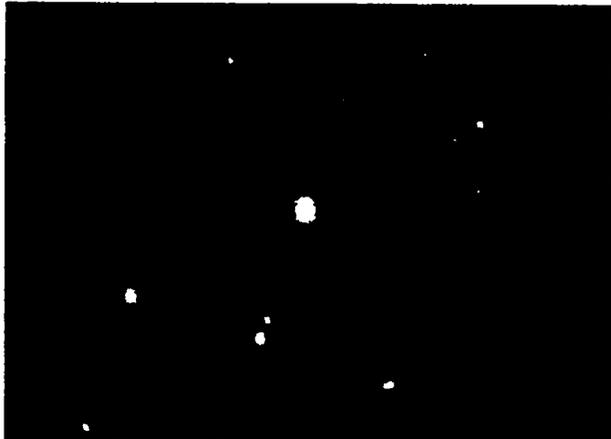
The diffraction patterns showed the best foils to be predominantly plutonium metal with a thin epitaxial passivating oxide layer. The double spots indicate the presence of  $PuO_2$ , which also is face-centered cubic (fcc), with a unit cell of  $5.396\text{\AA}$  compared with a unit cell of  $4.595\text{\AA}$  for 1.5 wt% gallium delta plutonium [Figs. 5 (a)



(d)

(e)

Fig. 4. (a) Schematic of oxidized TEM foils shown with typical (b) and (c) BF/SAD from location 1 and (d) and (e) DF/SAD from location 2. Bar = 0.5  $\mu$ m.



(a)

(b)

Fig. 5. Examples of TEM foils of Pu-1.5 wt% Ga with epitaxial oxide surfaces. Bar = 0.5  $\mu\text{m}$ .

and 5 (b)]. Indexing of the diffraction spots is shown schematically in Fig. 6 for the delta-plutonium alloy and the epitaxial  $\text{PuO}_2$  for the (110) orientations.

Recently annealed foils of the 17-yr-old material showed an interesting feature, bubbles in the grain boundaries [Figs. 7 (a) and 7 (b)]. These structures probably are helium bubbles produced by alpha generation in the plutonium, but they could be voids resulting from alpha-irradiation-damage build-up in the material during the long storage period.

#### IV. SUMMARY AND DISCUSSION

Our investigation shows that TEM can be applied to delta-phase plutonium foils. The remaining technical and engineering problems are to develop techniques, polishing solutions, and equipment that will make TEM routine and practical.

These recent experiments reconfirmed that the ongoing problem of plutonium TEM is making and preserving a thin foil free enough of oxidation to permit examination

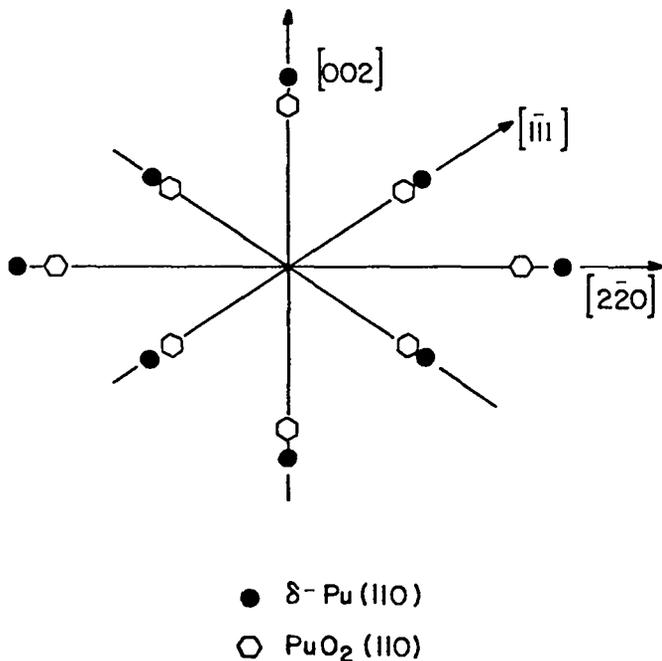


Fig. 6. Schematic (110) SAD showing indexing of diffraction spots for Pu-1.5 wt% Ga and the epitaxial PuO<sub>2</sub>.

of the microstructure. The main components of the new inert system, now undergoing tests with nonradioactive materials, are a specially designed recirculating inert-atmosphere glove box, a transport system with an oxygen-free TEM specimen holder, and an electron microscope with an integral inert-backfill system. The jet thinner has been modified for digital voltage and amperage data acquisition; also, it has been modified to operate with a reduced amount of electrolyte.

Assuming that practical methods can be developed for TEM of plutonium and its alloys, a number of pertinent experiments are planned. The following experiments could answer specific questions concerning plutonium metallurgy within the ability of our current equipment.

- Investigate the pressure transformation of alloyed plutonium from delta to alpha.
- Characterize the cold-rolling response of delta plutonium in relation to dislocations, cells, and stacking-fault energy as a function of alloy content.
- Characterize the delta-to-alpha transformation.
- Ascertain if delta quenched from epsilon is possibly a lath martensite or a massive transformation.
- Characterize doped crack-free alpha plutonium.



(b)

Fig. 7. Grain boundary bubbles (note arrows) in Pu-1.5 wt% Ga annealed after 17-yr *in vacuo* storage. Bar = 0.5 μm.

- Investigate the microstructural nature of as-cast high-purity alpha plutonium. It is colonies, grains, twinned, lath martensite, etc.
- Support weapons experiments on plutonium and plutonium alloys.

The following experiments are also of interest given the appropriate equipment, which is not available for plutonium work at this time.

- Investigate at temperature, *in situ*, phase transformations of plutonium and plutonium alloys.
- Investigate nucleation and growth of various phases by hot-stage electron microscopy.
- Study *in situ* fractures of alpha plutonium.<sup>17</sup>

## V. CONCLUSIONS

Contemporary techniques can produce acceptable delta-phase plutonium foils. Additional work is under way to develop these techniques for more routine applications.

## ACKNOWLEDGMENTS

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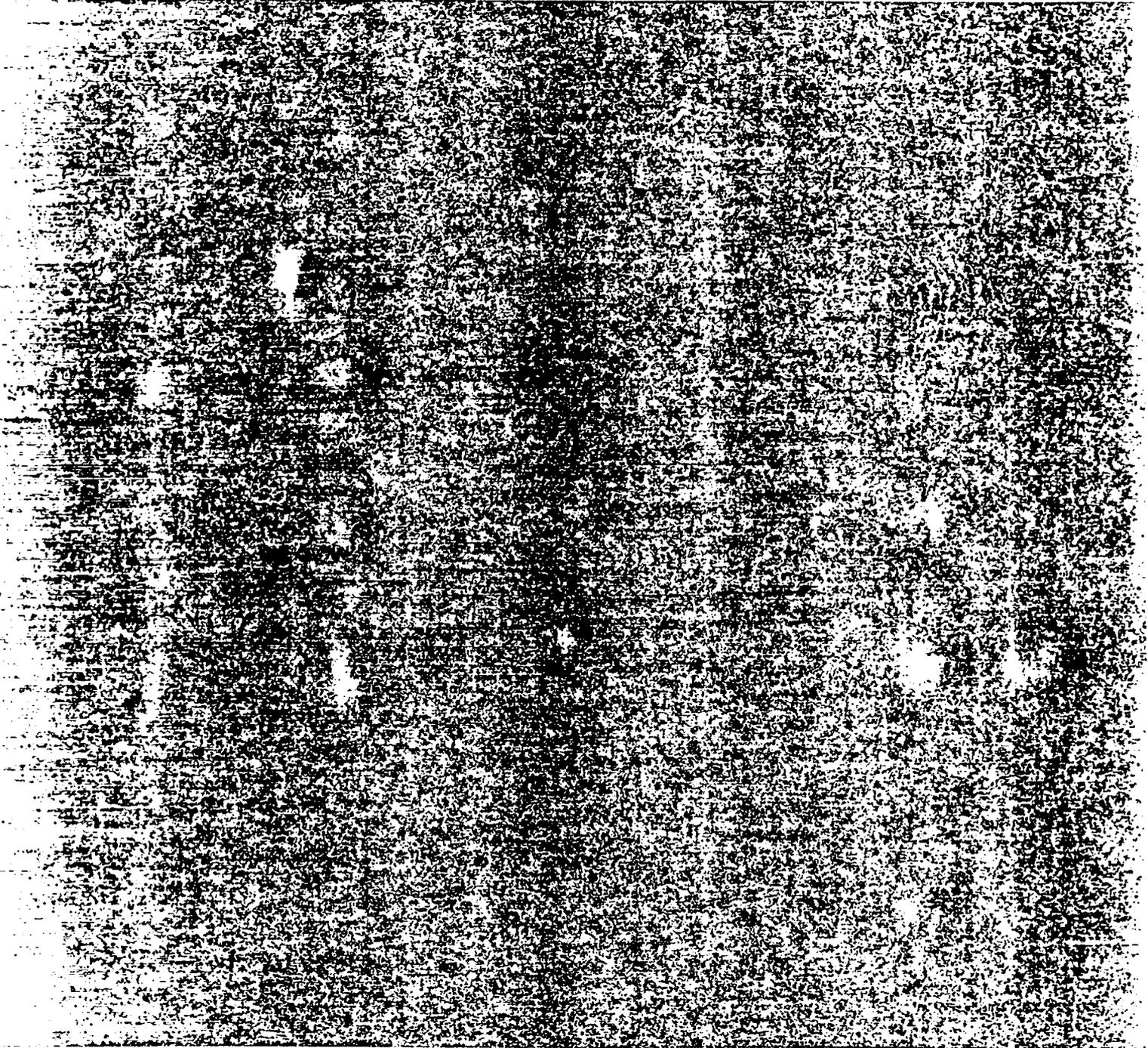
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051-075	A04	201-225	A10	351-375	A16	501-525	A22
076-100	A05	226-250	A11	376-400	A17	526-550	A23
101-125	A06	251-275	A12	401-425	A18	551-575	A24
126-150	A07	276-300	A13	426-450	A19	576-600	A25
						601-up*	A99

\*Contact NTIS for a price quote.



Los Alamos