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METALLOGRAPHY OF PLUTONIUM

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Technology - Plutonium

ABSTRACT

Three samples of plutonium have been examined under the microscope, two as reduced from the fluoride and one vacuum remelted, rolled, and annealed.

All samples have shown at least two phases, but probably not the same two phases in every case.

It has been found that the phosphoric acid uranium electropolishing solution can both polish and etch plutonium.

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METALLOGRAPHY OF PLUTONIUM

Three impure plutonium samples have been examined for microstructure. The first sample (micro 2396) was reduction sample no. 1355 b, the first successful 50 mg reduction made by Magel by reduction of PuF_4 with lithium in BeO in the centrifuge. The second sample (micro 2439, reduction no. 1466a) resulted from a 930 mg reduction under the same conditions as above which gave 56 per cent yield of material assaying 97.6 per cent plutonium and of density, 17.7. Spectrographic analysis (Nachtrieb) showed considerable segregation. The better parts contained 1000 ppm of iron, 100 ppm Li, 200 ppm Be, 100 ppm Mg, and the less pure portions (soluble in HNO_3) had 8000 ppm iron, 11,000 ppm Li, 2400 ppm Be, 2000 ppm Mg and 300 ppm Mn. The 30 mg sample taken for micro examination was probably representative of the purer material. The third sample was a 100 mg portion of the 930 mg reduction, but was remelted by Weissman, Lipkin and Perlman in vacuo on a uranium nitride block. This was reduced to 0.009 in. thickness by annealing and rolling with two intermediate anneals, and given a final anneal at 600° C. This is micro sample no. 2485. The hardness (Eberbach) varied from about 123 in the annealed condition to 226-264 after cold working.

All three of the samples appeared to show at least two phases, with the second phase distributed in the grain boundaries of the parent metal.

Figs. 1 and 2 show the structure of the two "as-reduced" samples. The structure of the second sample differed from that of the first in that

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the grain-boundary phase was sharper and more well defined than in the first sample. It is doubtful if it is the same composition and may be associated with the high iron content.

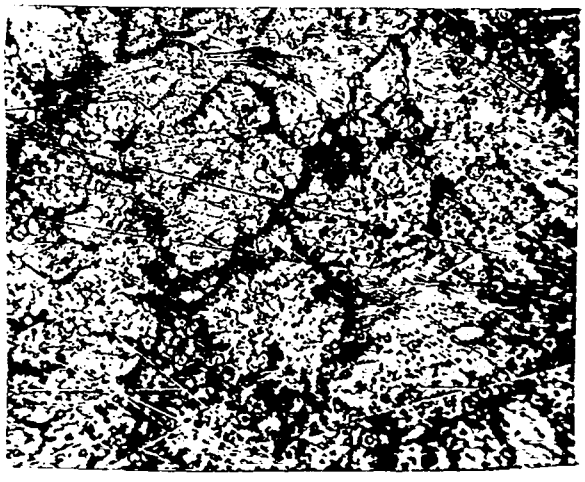

Fig. 1. First 50 mg Plutonium Sample as Reduced.

2396-1-1 Not etched x 250

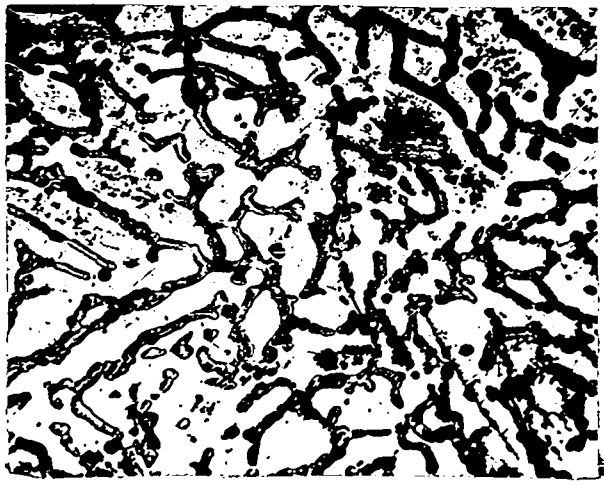

Fig. 2. First 1 gram Reduction, as Reduced.

2439-1-2 Not etched x 250

The polishing in all cases was mechanical, ending with levigated alumina on a silk wheel. The structures shown in Figs. 1 and 2 are after mechanical polishing, without etching. All the etching reagents listed in Table I were tried, but failed to do more than to increase the contrast slightly or to cause pitting.

Table I. Etching Reagents Used on Plutonium Sample No. 2

Reagent	Results
1% nital	Little effect. Possible darkening of main (light) constituent.
5% nital	Same.
0.5% HF in water	Attacks dark network constituent.
Saturated I_2 in alcohol	Darkens main constituent. General pitting. (Works well on thorium.)
1:1 above solution and alcohol	Action less drastic.
Picral	No effect
5 $FeCl_3$ 10 HCl 100 H_2O	Straight and very dilute in alcohol caused bad pitting.
Dichromate solution 100 cc H_2O 2 g $K_2Cr_2O_7$ 4 cc sat. NaCl solution 8 cc H_2SO_4	Bad pitting.
10% oxalic acid	Stains major constituent. Causes blackening if left more than 30 sec.
2% oxalic (electrolytic)	Bad pitting.
5% oxalic (electrolytic)	Bad pitting.

The third sample was examined after annealing in vacuo at 600° C. and after rolling to 0.009 in. thick, the last reduction being about 59 per

cent. The various structures observed are shown in Figs. 3-10. The micro hardness (Eberbach) was about 123.

Unlike the samples of Figs. 1 and 2, very little structure was visible in the remelted and rolled sample (2485) after mechanical polishing alone. Various etching reagents were used which had given some hope of success with the earlier samples, but which could not be accurately evaluated because of the tendency for earlier samples to polish-etch. These etching reagents included the following in various concentrations; nital, iodine in alcohol, and oxalic acid. However, it was found that the usual solution used for electrolytic polishing of uranium (45 per cent ethyl alcohol, 25.7 per cent each of ethylene glycol and orthophosphoric acid) did bring out a structure, Figs. 3-10.

This etching solution appeared to etch differently depending upon the time that it was allowed to act. Shorter times (about 1-2 min.) disclosed a continuous dark etching phase surrounding a discontinous light etching phase. Longer times appeared to lighten the dark phase and darken the light phase; still longer times resulted in some electrolytic polishing.

Another thing which made some difference in the appearance of the structure was actual composition differences along the length of the specimen. The rolled specimen was mounted on edge so that a minimum of material would be polished away. As the specimen was repeatedly polished and repolished, the appearance of the microstructure varied. In one portion, there appeared to be many small oxide islands in the metal. Here and there clumps of oxide could be seen on the outside of the specimen (Fig. 5). This

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oxide was caused, at least in part, by a small leak in the evacuated quartz tube during one of the 600° C. anneals. The distribution of the oxide particles is not what one would expect had they had no chance for rearrangement other than by the rolling since the material was first remelted, hence it is not beyond the realm of possibility that the material had been melted during the 600° C. anneals. The distribution of the other phases is not inconsistent with this, although it certainly does not immediately suggest a cast structure. Very light etching (Fig. 3) discloses a fine network of a dark etching phase which is quite similar in appearance to that visible more abundantly in the same sample before remelting (Fig. 2). Deeper etching discloses a two phase structure which certainly indicates some transformation either during heating or cooling, and is not a structure that would result from mere recrystallization and grain growth of a pure metal or two phase alloy not undergoing transformation or wide change of solubility. On deeper etching, the appearance reverses, the continuous phase now appearing lighter than the discontinuous, somewhat acicular phase. The boundaries of the acicular cores are not clearly defined, and it is possible that they are actually the same phase with more copious precipitation of some other constituent, distributed in this manner because of coring.

The electrolytic polishing solution used for uranium can probably be used for polishing plutonium, since the sample, after a prolonged treatment in this solution, is practically scratch-free (Fig. 10) and the metal areas between the oxide chunks show unmistakable evidence of having been electropolished.

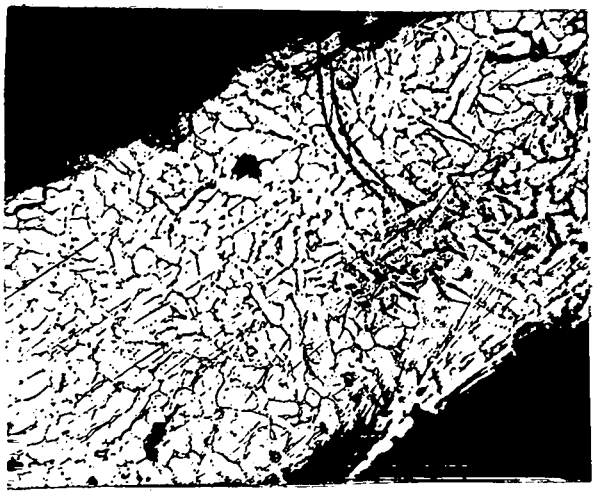

Fig. 3. Rolled and Annealed Plutonium. Etched electrolytically in phosphoric acid solution.

2485-1-1 x 250

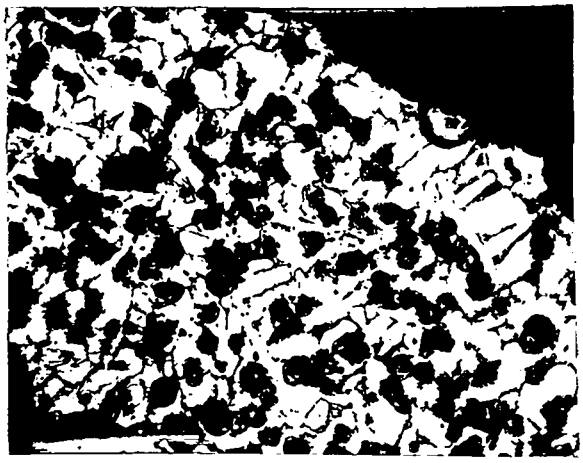

Fig. 4. Same as Fig. 3, but shows dark etching patches (oxide?).

2485-1-9 x 250

Fig. 5. Same as Fig. 3. Etched about 2 min. Continuous phase etched darker.

2485-1-5 x 500

Fig. 6. Same as Fig. 3. Etched about 5 min. Continuous phase light. Center of former light grains now dark.

2485-1-4 x 500

Fig. 7. Same as Fig. 3. Etched about 2 min. Shows dark continuous phase.

2485-1-3 x 500

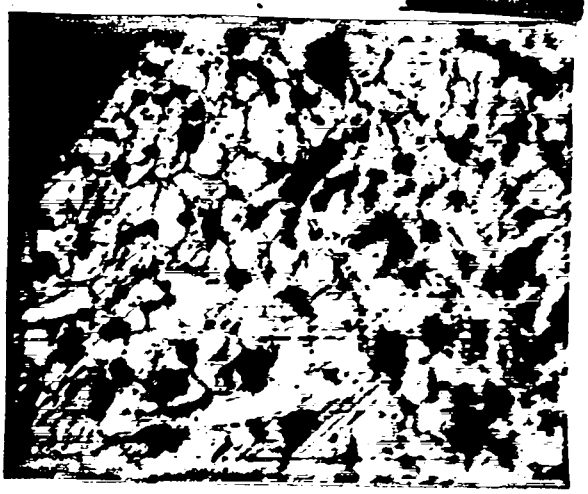

Fig. 8. Same as Fig. 3. Shows dark etching (oxide?) patches and grain boundary constituent.

2485-1-2 x 500

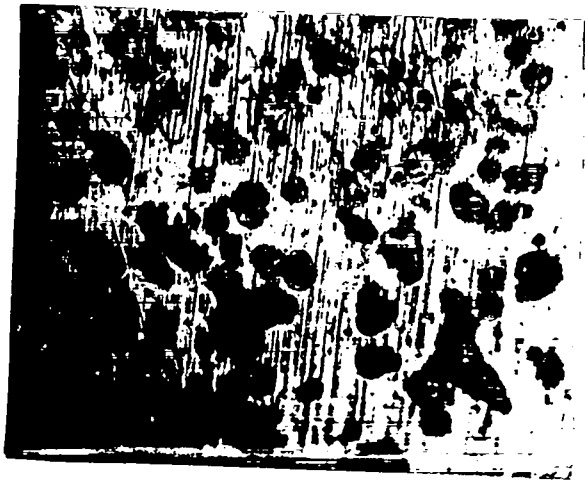

Fig. 9. Same as Fig. 3. Shows oxide? patches.

2485-1-11 x 500

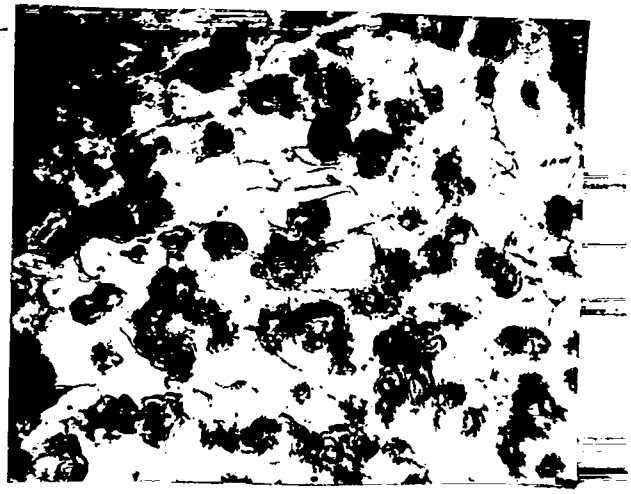

Fig. 10. Same as Fig. 1. Shows dark etching patches, possibly oxide. Etched for 25 min.

2485-1-8 x 500