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ELECTRON MICROSCOPY OF ALPHA-PLUTONIUM

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During the past several decades metallurgists world-wide have sought for a reliable and reasonable method to characterize plutonium and its alloys via transmission electron microscopy. Metallurgically, plutonium (Pu) represents one of the most fascinating metals; it has six solid phases and contracts when melted.

A number of workers over the years have tried to produce thin foils of plutonium, but the foils were later shown to have been completely converted to oxide.[1,2] The primary problem has been in the preparation of electron transparent thin foils of a material having one of the highest atomic numbers that is highly oxidation prone, in addition to being radioactive. Advances in Pu metallography in the late 70's and early 80's produced a new series of electro-chemical thinning solutions modified for plutonium.[3-5] The δ (FCC) phase was first prepared using the techniques employed for this work and has been reported previously.[6] This investigation has focused on the higher density, lower symmetry structure (monoclinic) α phase and is part of a larger study on plutonium and its alloys, and is the first reported TEM success with the alpha phase of Pu. Optical microscopy of the alpha phase has been very frustrating for years with many workers in the field speculating on the nature of the transformation to alpha from the higher temperature phases. Support for these theories has been delayed pending confirmation by TEM. The alpha crystal structure determined by Zachariasen and Ellinger[7,8] from x-ray powder patterns in 1957 has not been confirmed by any single crystal diffraction work, to date. We have obtained for the first time TEM and SAD of the individual α phase crystallites of Pu. These are shown in Figs. 1 and 2. Computer aided electron diffraction analysis[9] identified the SAD of Fig. 2 to be [010] and is shown in Fig. 3. Comparisons with the calculated pattern clearly substantiates the Zachariasen-Ellinger monoclinic structure and lattice parameter to within the accuracy of the TEM technique. All the TF' samples observed have traces of PuO_2 (FCC) on the surfaces. This is evident in Fig. 4 showing both the [001] α-Pu and the FCC PuO_2. These oxide layers appear to grow with a preferred or epitaxial orientation.

The preparation, environment, thinning solutions and transfer mechanisms used in this investigation are reported elsewhere.[6]

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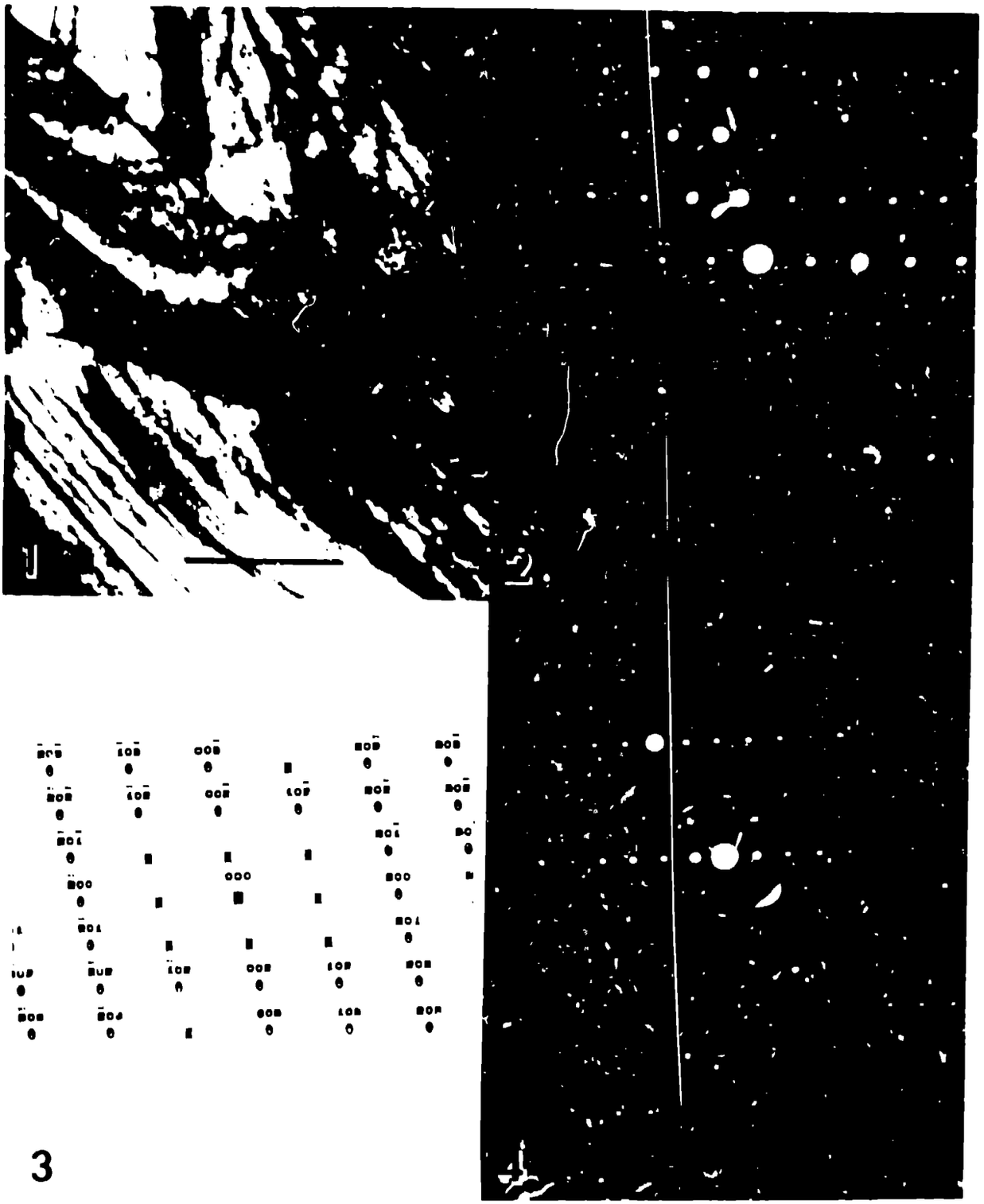

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Fig 1.--Grain boundaries in α-Pu.Bar = 0.25μm.
Fig 2.--SAD of α-Pu, (020).
Fig 3.--Calculated diffraction pattern (020).
Fig 4.--SAD of α-Pu (001) showing PuO_2 oxide trace.