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Studies of einsteinium metal (*)

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Résumé. — Nous avons préparé des films minces d'einsteinium métallique par réduction de Es_2O_3 par du lanthane, puis par distillation de l'einsteinium métallique et condensation sur une grille de microscope électronique sur laquelle on a préalablement effectué un dépôt de carbone. Les dépôts de métal ont été analysés par diffraction d'électrons. Les résultats montrent que l'einsteinium déposé à partir de la vapeur est divalent et présente la phase fcc ($a_0 = 5,75(1) \text{ \AA}$) très semblable à la phase *dilatée* du californium métallique [1, 2, 3].

Abstract. — Thin films of einsteinium metal were prepared by reducing Es_2O_3 with lanthanum, distilling the einsteinium metal, and condensing it on carbon substrates supported by electron microscopy grids. The metal deposits were then analysed by electron diffraction. The results show that vapour-deposited einsteinium metal is divalent and exhibits a fcc phase ($a_0 = 5.75(1) \text{ \AA}$) very similar to that reported for the expanded form of californium metal [1, 2, 3].

1. Introduction. — The scarcity of einsteinium isotopes and the high specific radioactivity of the most available isotope (^{253}Es , $5.6 \times 10^{10} \alpha \cdot \text{min}^{-1} \mu\text{g}^{-1}$) require that microtechniques be used for preparing and studying the metallic state of einsteinium. Further, since einsteinium is available only twice a year, it is necessary to carry out these studies over a long period of time.

The use of electron diffraction for studying einsteinium metal minimizes some of the experimental difficulties encountered when X-ray diffraction is used for analysis. The larger sample sizes and longer film exposures required for X-ray analysis are not compatible with the rapid degradation of the metal's crystallinity and the severe blacking of the film in powder cameras from the sample's radioactivity.

2. Experimental. — The einsteinium in this work was obtained through the Transuranium Elements Program of the U.S. Department of Energy. Both pure ^{253}Es and ^{253}Es - ^{254}Es mixtures ($< 1\% \text{ } ^{254}\text{Es}$) from several different purification runs were used in making the metal samples. Final purifications of the einsteinium were done by ion-exchange techniques [4]. Single pieces ($\sim 20 \mu\text{g}$) of Es_2O_3 were prepared by microprecipitation of einsteinium oxalate followed by calcination of the latter at $1\,000^\circ\text{C}$ in air [5].

The einsteinium metal samples were prepared by reducing Es_2O_3 with lanthanum metal at $\sim 1\,050^\circ\text{C}$ in a tantalum effusion crucible and allowing the einsteinium vapour to condense on a 3-mm O.D.,

electron microscopy grid which was covered with a carbon film. The techniques and conditions for einsteinium were similar to those previously used for preparing and analysing californium metal [1].

3. Results and discussion. — The data reported here were obtained from eleven Es_2O_3 -lanthanum preparations; approximately three successive distillations and deposits were obtained from each preparation. Since 10-200 ng of einsteinium were desired on a microscopy grid, experimental conditions were chosen so that the major portion of the volatilized metal condensed in the tantalum chimney below the grid. It was expected that this material would serve as a *getter* for any residual impurities before the metal started to deposit on the grids. The amount of metal deposited on the grids was a function of the amount of Es_2O_3 in the crucible, the diffusion time and the temperature of both the crucible and the chimney. A rough estimate of the quantity of einsteinium that had collected on a grid could be made by observing the grid in the helium atmosphere of the glove-box in the dark, and noting the illumination that resulted from the interaction of the einsteinium's radiation with the helium.

About one-third of the grids had einsteinium deposits that initially yielded good electron diffraction patterns. The remaining samples either contained an insufficient amount of einsteinium or had amorphous-like deposits. Deposits which were initially crystalline, decreased in crystallinity with time (several hours) depending on the amount of einsteinium present. The best diffraction patterns were obtained from the heavier deposits, but the crystallinity of these degraded rapidly and data taken from these samples were probably affected by the self-irradiation of einsteinium.

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The initially crystalline deposits were indexed on the basis of a fcc structure with $a_0 = 5.75(1) \text{ \AA}$ (average of ten samples). The variation in parameter reflects the radiation damage not only to the einsteinium deposits but probably also to the internal standard (gold) used to calibrate the wavelength of the electron beam. A line list from one of the samples is given in table I, where a least-squares refinement yielded a lattice parameter of $a_0 = 5.746(5) \text{ \AA}$.

Table I. — *Electron diffraction data for einsteinium metal.*

$d, \text{ \AA}$			
hkl	obs.	calc.	Intensity
—	—	—	—
111	3.317	3.315	10
200	2.858	2.871	6
220	2.033	2.030	8
311	1.736	1.731	8
222	1.661	1.658	4
400	1.439	1.436	2
331	1.318	1.317	4
420	1.295	1.284	2
422	1.178	1.172	2
511	1.110	1.105	4
440	1.012	1.015	2
531	0.969	0.971	4
620	0.909	0.908	2
533	0.875	0.876	2

When the initial metal deposits were heated in the electron microscope, it was possible to observe the particles coagulate to form *puddles*. This process was believed to be due to melting of the metal, and the temperature at this point was taken to be the melting point of einsteinium metal ($860 \pm 50^\circ \text{C}$). As a consequence of the greater reactivity of the molten metal, the carbon substrates had a very limited lifetime under these conditions. No evidence for carbide formation was noted; it was assumed that the reaction to form carbides was responsible for breakage of the carbon substrate, which then prevented further diffraction analysis. In some instances, diffraction patterns attributed to einsteinium oxides [6] were observed after extended heating of the deposits, but oxide formation in the einsteinium samples was less prevalent than found in similar work with rare earth metals.

The fcc form of einsteinium metal is very similar to the divalent form of californium metal prepared either by vapour deposit or by high-temperature trifluoride reduction [1, 2]. Due to the scarcity of einsteinium and its high specific activity, studies of its metallic state will be limited, and another metallic valence state of einsteinium, if it exists, may not be observed. Further, it is necessary to understand fully the californium metal system to extrapolate its behaviour to aid in interpreting the einsteinium system. At this point in time, the experimental data for einsteinium metal is consistent with that reported for californium metal, and also with theory, which predicts the increased stability of the divalent state in going across the actinide series [7].

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