

Electrorefining Depleted Uranium (dU)

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Abstract

The intent of this project was threefold. First, was the growth of single crystal dU via electrorefining in a KCl-LiCl eutectic salt solvent at vacuum pressure. Second was to improve upon the safety measures surrounding this growth process. Third was to educate and train the newest member of the Tozer undergraduate group: Elisabet Wackes.

Introduction

dU is uranium whose main component is U-238 rather than U-235 as is typically found in nature. Due to its high density, dU has several military and civilian applications including radiation shielding, aircraft counterweighting, and military vehicle armor plating. Similar to many actinide elements Uranium has a complex pressure-temperature-magnetic field phase diagram. The study of ultra-pure Uranium which is free from lattice defects in extreme conditions is the key to understanding this complex behavior as well as things like the electrical, magnetic and optical properties of the material. However, Uranium is highly susceptible to twinning and slip defects making in-house growth the best option.

Our growth processes is based on a technique for electrorefining plutonium developed during the Manhattan project that was refined for Uranium during the 1980's^[1]. A bulk piece of dU (Fig.3), mounted to a stainless steel (SS) anode, is placed into a crucible containing: a KCl-LiCl eutectic salt, a thermocouple, and a blank SS cathode. The crucible is then heated via induction until a molten salt bath forms. The dU dissolves in the mixture temporarily entering a UCl₃ state. A potential is then applied across the two electrodes causing pure dU to be deposited on the cathode.

Previous Work

In my previous work with the Tozer group I was able to successfully grow high quality dU. The aim of this project was to explore the effects of varying growth parameters such as applied voltage, growth duration, and temperature, on crystal yield. However, it was discovered early on that the system was no longer heating properly making the new aim of my project to resolve this system defect.

Safety Improvements & Heating Solutions

The first goal of this project was to improve the safety of the growth procedure. To do this I designed and installed a plexiglass shield mounted to a G10 base (Fig.2) Because the system is kept under vacuum any defects in the quartz tubing could lead to shattering. The shield will protect the user from any possible debris.

After conferring with synthesis experts and through a large amount of trial and error I was able to correct the system faults by: constructing a new induction coil, decreasing the transformer taps, and drying the KCl and LiCl salts to remove excess water (Fig. 4) Upon making these changes the system was once again able to heat properly.

Experimental Procedure

Prior to the growth care was taken to ensure that all materials were sterilized and that the system could hold a vacuum. The KCl-LiCl eutectic mixture is housed inside a SS crucible that is placed inside a quartz tube. The top of the tube has inserts which allow for the electrodes and K-type thermocouple to be inserted. The eutectic used was a 44.3 mol% KCl – 55.7 mol% LiCl with a melting point of approximately 353 °C^[2]. The salts used were J. T. Baker LiCl (>99.0%) and G-Biosciences KCl (>99.0%). The bulk dU was vacuum induction melted U-238 Using RF induction to heat the system the temperature was brought to 450±10 °C and a voltage of 1.00±0.01 V was applied for 6 hours and then due to a short circuit in the system (see discussion section), a voltage of 0.66±0.01V was applied for 30 hours for a total growth duration of 36 hours. The system was purged with Argon before removing the electrodes. The entire process was done with the help of Elisabet as part of her training in growing dU.

Results & Discussion

The growth resulted in “mossy” dense dU which spread from the anode to cathode. There was also some dU deposit in the SS crucible. The crystals were etched in HNO₃ and electropolished at 1.27±0.01 V in a H₂O/H₃PO₄/H₂SO₄ mixture. As seen in Fig.5 the dU grew across electrodes short circuiting the system. This caused the voltage drop and what possibly led to the growth of dense dU rather than long crystals as expected.



Fig.5 – Mossy dU on electrode before removal



Fig.6 – Mossy dU before electropolishing

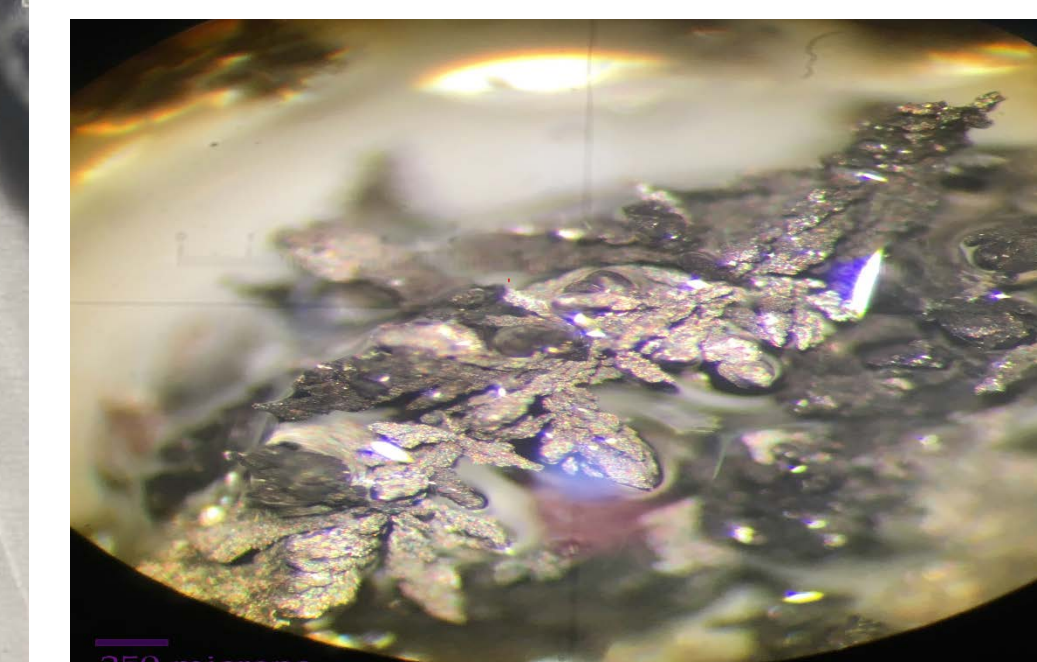


Fig.7 – dU crystals after chemical etch and electropolishing

Future Work

Moving forward we will begin to explore the effects of growth parameters on crystal quality and yield. Elisabet will continue to work with me before doing a growth of her own. We will also be sorting and preparing single crystals for experimentation through a process of individual selection, electropolishing, and gold plating to prevent oxidization.

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References

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Fig.1 - Full growth set up



Fig. 2 - Full growth set up with safety shield.



Fig.3 - Bulk dU being electropolished prior to growth.



Fig.4 - LiCl before drying (L) and after drying (R). Not the decrease in volume and clumping.