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# Some metallographic observations on thorium metal

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## Some metallographic observations on thorium metal

### **Abstract**

For several years an investigation of the physical properties of thorium and methods of preparing higher purity thorium metal has been conducted at the Ames Laboratory of the U. S. Atomic Energy Commission. In the metallographic examination of high purity thorium, an electropolishing procedure which did not obliterate the inclusions was desired. The mechanical polishing of thorium to reveal small inclusions is difficult and slow, and becomes more difficult as the hardness of the metal decreases with higher purity. In addition, a number of tensile specimens were to be examined to determine the grain size, and a convenient method of revealing the grain structure was needed. A previous study of the electropolishing of thorium had shown that a solution of hydrochloric and hydroiodic acids in formic acid had some promise as an electropolishing solution. A more extensive study of electropolishing solutions which used formic acid as the solvent was undertaken to see if a satisfactory solution and procedure could be evolved. The results are incomplete and the solutions used are not recommended for general use but some very interesting observations have been made on thorium metal.

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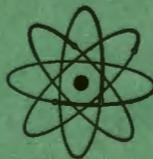
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**AMES LABORATORY  
RESEARCH AND DEVELOPMENT REPORT  
U.S.A.E.C.**



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SOME METALLOGRAPHIC OBSERVATIONS  
ON THORIUM METAL

by

David T. Peterson

June 1960

Ames Laboratory  
at  
Iowa State University of Science and Technology  
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## SOME METALLOGRAPHIC OBSERVATIONS ON THORIUM METAL

David T. Peterson

## INTRODUCTION

For several years an investigation of the physical properties of thorium and methods of preparing higher purity thorium metal has been conducted at the Ames Laboratory of the U. S. Atomic Energy Commission. In the metallographic examination of high purity thorium, an electropolishing procedure which did not obliterate the inclusions was desired. The mechanical polishing of thorium to reveal small inclusions is difficult and slow, and becomes more difficult as the hardness of the metal decreases with higher purity. In addition, a number of tensile specimens were to be examined to determine the grain size, and a convenient method of revealing the grain structure was needed. A previous study of the electropolishing of thorium had shown that a solution of hydrochloric and hydroiodic acids in formic acid had some promise as an electropolishing solution. A more extensive study of electropolishing solutions which used formic acid as the solvent was undertaken to see if a satisfactory solution and procedure could be evolved. The results are incomplete and the solutions used are not recommended for general use but some very interesting observations have been made on thorium metal.

## RESULTS AND DISCUSSION

Variation in Electrolyte Composition - The composition of the electrolytic solution could be varied considerably without losing the electropolishing action on thorium metal. The solution containing 2% hydrochloric acid in formic acid had a high resistance which required high voltages to obtain the necessary current density. Ammonium chloride was added to the solution to increase the conductivity and enabled polishing currents to be reached at approximately 20 volts. However, the hydrochloric acid in this solution reacted chemically with the thorium surface and pitted the samples badly. A solution containing two grams of ammonium chloride per 100 ml of formic, but no hydrochloric, acid had approximately the same conductivity as a similar solution containing 2% hydrochloric acid. Thorium went into solution at the anode but no polishing of the metal surface was observed. However, the addition of 6% by volume of water resulted in excellent polishing and smoothing action by this electrolyte. Apparently a small amount of water was necessary to obtain polishing and this had been supplied by the hydrochloric acid.

This solution (2 grams ammonium chloride, 100 ml formic acid and 6 ml water) at 20 volts produced a smooth scratch free surface in 60 seconds on thorium samples which had been ground through 600 mesh silicon carbide paper. The principal disadvantages of this solution were that a thin soft blue film was left on the specimen and the grain boundaries were not revealed. This film was epitoxic and under polarized light often gave contrast between grains. The surface film could be easily removed by swabbing with a dilute nitric acid solution but the swab dislodged particles of thorium oxide and scratched the surface. A number of other organic and inorganic compounds containing ionizable chloride were substituted for ammonium chloride. This included methylamine hydrochloride, aniline hydrochloride,

guanidine hydrochloride and tetramethylamine hydrochloride. The polishing action of all of these solutions was nearly the same as that of the ammonium chloride solution. The last two compounds are the salts of strong organic bases and consequently sodium chloride was used and found to work quite well. Apparently the necessary constituents of a thorium polishing solution based on formic acid are a compound which ionizes to provide chloride ions and a small amount of water or glycerine. The successful use of salts of an acid rather than the acid in electropolishing has also been reported for perchloric acid by Tegart.\*

Observations on Thorium Microstructure - The major portion of this investigation was made with specimens of arc-melted thorium which had been prepared by the magnesium reduction of thorium tetrachloride. This thorium was comparable to crystal bar thorium in purity and had microstructures similar to crystal bar thorium. The most prominent feature of the microstructure of thorium in the arc-melted condition is very extensive twinning. The twin boundaries were revealed by almost all electropolishing or etching procedures before other details of the microstructure became evident. A photomicrograph of a specimen of crystal bar thorium heated at 1200°C and furnace cooled which has been repeatedly electropolished is shown in Fig. 1. The twin boundary is deeply etched but the grain boundaries are imperfectly delineated although the severe polishing conditions have produced pitting and surface undulations. A heavily twinned area in an arc-melted specimen is shown in Fig. 2. A portion of the structure apparently consists of straight parallel twinned lamellae. Although the

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\*W. J. McG. Tegart, *The Electrolytic and Chemical Polishing of Metals* (1959) Pergamon Press Inc., New York, N. Y.

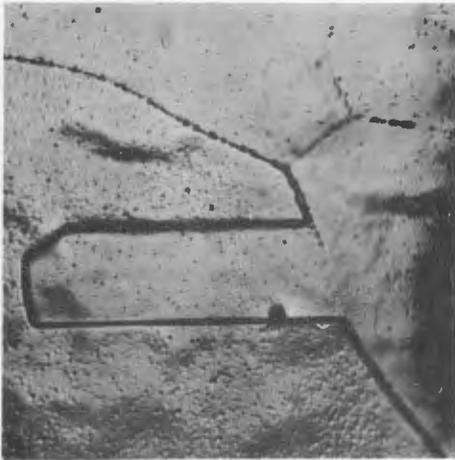


Fig. 1. Crystal bar thorium heated to 1200°C and furnace cooled. Bright field. 50X.



Fig. 2. Mg-reduced thorium as arc-melted. Polarized light. 200X.

twinning areas are clearly visible, the grain boundaries are not revealed. The microstructure of arc-melted thorium resembles that of other metals which have undergone an allotropic transformation. Large grains formed by crystallization of the high temperature phase transform to areas which are extensively twinned and fragmented by subgraining. A fairly typical microstructure photographed under polarized light is shown in Figs. 3 and 4. The same area is shown in both figures but was rotated slightly for Fig. 4. The twinning and subgrain formation probably result from the transformation from the body-centered-cubic form to the face-centered modification but this has not been verified by quenching experiments.

Thorium oxide is often found in the microstructure of thorium metal. If present in amounts greater than about 200 ppm it occurs as dendrites or large angular inclusions which may be readily seen in mechanically polished specimens. In the arc-melted magnesium-reduced thorium a much smaller thorium oxide inclusion was found in addition to the dendritic form. These particles were not observed in the mechanically polished specimens, probably because of the tendency of thorium to smear during polishing. These particles ranged in size from 1 micron to 6 microns. In Fig. 5 are shown these particles in crystal bar thorium under bright field illumination. The size of these particles is about 4 microns and the shape was typical of this kind of inclusion. The particles are slightly larger and more numerous in the specimen of magnesium-reduced thorium shown under dark field illumination in Fig. 6. These particles may be responsible for the extensive pitting of thorium by perchloric acid electropolishing solutions. Perchloric acid electropolishing solutions produce pitting around the larger oxide particles and would likewise be expected to cause small pits at these very small inclusions.



**Fig. 3. Mg-reduced thorium as arc-melted. Polarized light. 200X.**



**Fig. 4. Mg-reduced thorium as arc-melted. Polarized light. 200X. Rotated several degrees.**



Fig. 5. Crystal bar thorium.  
Bright field. 500X.



Fig. 6. Mg-reduced thorium.  
Dark field. 900X.