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CONSUMABLE ELECTRODE ARC-MELTING
OF COLD-COMPACTED THORIUM POWDER

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ABSTRACT

The consumable electrode arc melting of cold-compacted thorium powder has been developed to produce dense, clean, and soft ingots which can be hot rolled without cracking. By using a water-cooled copper crucible, contamination has been prevented and resultant ingots cleaner than the original powder have been produced.

The relationship of specific variables such as power at the arc, crucible size, electrode size, and argon pressure have been determined relative to minimum requirements to obtain sound ingots. The data has been found to be applicable to other metals when reasonable corrections are applied.

I. INTRODUCTION

Thorium metal requires special care in melting operations because it has a relatively high melting point and is chemically reactive above room temperature. Thorium has been melted by the vacuum induction technique with zirconia and beryllia crucibles, but contamination by zirconium occurs up to amounts of 1 per cent by weight. This high chemical affinity of the metal for crucible materials has necessitated investigation of other melting methods. The most successful method to date has been consumable electrode arc-melting in a water-cooled copper crucible⁽¹⁾.

Melting thorium in a water-cooled crucible eliminates many of the disadvantages of other processes. Volatile constituents which can form slags are condensed at the cold crucible walls and can be eliminated by machining the ingots. Crucible contamination of the ingot is prevented because the metal never gets hot enough at the cold crucible wall to cause alloying or pick-up.

This investigation is basically concerned with the technique established for the consumable arc-melting of thorium electrodes made by cold-compacting thorium powder. The powder was made by calcium reduction of thorium oxide. The ultimate object of the investigation was to produce a dense, clean, and soft ingot which could be subsequently extruded into rods for applications such as fuel elements.

II. EQUIPMENT AND PROCEDURE

A. Arc-Melting Equipment

A schematic diagram of the basic furnace used for the arc-melting experiments is depicted in Fig. 1. This furnace was originally designed for melting ingots of 1.0 to 1.5 pounds capacity, but this has been increased to 10 to 20 pounds by increasing the sizes of the crucible and electrode. The electrodes were cold-pressed from thorium powder and ranged from 3/4" to 1.35" diameter and 12" to

38" long. Crucibles used had 1-1/2", 2", and 3" diameters and ranged from 6" to 32" long. The larger electrode and crucible sizes were used to make ingots up to 10 pound capacity.

The consumable thorium electrodes were attached to the water-cooled copper electrode by means of a threaded adapter welded on the thorium electrode. The entire furnace was then sealed and pumped down to 50 microns with a roughing pump. The gas pressure was then adjusted by bleeding argon into the system at pressures of 3 mm to 210 mm of mercury. The pressure was balanced continuously by the action of the roughing pump during the melting cycle. When the argon pressure was balanced at the desired level, the power was turned on, and the electrode struck against the thorium button placed centrally in the bottom of the water-cooled copper crucible. The arc was struck, the starting button melted, and a pool of metal started to form. Then the current was raised to the value necessary to obtain the desired melting rate.

Power for melting was obtained from two 880-ampere welding rectifiers in parallel. The maximum current obtainable was 1600 amperes at 30 volts for short time intervals up to ten minutes. These rectifiers can be operated continuously at 60 per cent of their rated output on a three-phase 440 volt main power line. It was found that this equipment provided good arc stability during the entire melting cycle when the proper conditions for melting were established.

B. Properties of Powder and Fabrication of Electrodes

Briefly, the calcium reduction process by which the thorium powder was made consists of three basic steps, namely, reduction, leaching, and drying. The reduction charge is composed of a blended mixture of thorium oxide, calcium chloride, and calcium metal which is fired in a retort to yield a reduction product or "cake." The "cake" is disintegrated by leaching with water, and the resultant powder is washed with water to remove residual calcium hydroxide. Oxidation is limited during leaching by careful temperature control. The wet powder is filtered, washed, and dried. The resultant thorium powder is spongy in nature and has nonpyrophoric characteristics, facilitating cold compaction in air. Fig. 2 is a photomicrograph showing the spongy character of the powder having apparent densities in the range of 1.8 to 2.2 g/cc. A typical analysis of the powder is presented in Table I, indicating high calcium and hydrogen contents. A comprehensive

description of powder made by this process has been published by the U. S. S. R.⁽²⁾

Figs. 3 and 4 illustrate the effect of cold compacting pressure on density and strength characteristics of the electrodes. A compacting pressure of 50 tsi yields a density approximately 94 per cent of the theoretical density of thorium and a modulus of rupture near 7000 psi. Specimens weighing 50 gms. each had electrical resistivities around 5.0×10^3 microhm-cm.

Electrodes of two- to eight-inches were cold compacted at 50 tsi with diameters of $3/4"$ to 1.35". Die lubricants used were either aquadag or vacuum grease. The aquadag was wire-brushed from the surface prior to welding and melting operations. Melting of larger ingots requires that two or more compacts be welded into one continuous electrode. Welding was accomplished in an argon atmosphere with 70-90 amperes d.c. welding current for small diameter electrodes and 150-180 amperes for larger diameters. Electrodes lubricated with vacuum grease in compacting are melted as ejected, and the quality of the melted ingot is not affected thereby.

III. EXPERIMENTAL PROCEDURE

A. Operational Results

The most important problem is the determination of the proper arc-melting conditions for high quality ingots. A study of the literature on consumable electrode arc-melting of thorium, molybdenum, and zirconium indicated the importance of power input^(1, 3, 4). It was found that the rate of melting depends directly on the power applied. During initial melting experiments, several problems arose which are summarized below:

It was found that absolute values of power across the arc or current densities were unsatisfactory guides to sound ingots in all cases. This was established by plotting curves of current density input vs. current at the arc for data found in the literature for molybdenum and zirconium.

A plot of current density versus current at the arc indicated that an increase in current density could be obtained by using small diameter electrodes, but information on zirconium showed that current density is an improper guide to good in-

got quality. Because several cases of relatively low current density were nevertheless high in power at the arc and resultant sound ingots were obtained current density was eliminated. Accordingly, a new approach was tried using relative power rather than an absolute value of power. The power is defined by the equation $P = I^2 R$, where R is the resistance in ohms obtained from $R = \rho l/A$, ρ is the resistivity of the metal close to its melting point in ohm-cm, A is the area (inches²), and l is the length (1/10"). This length was assumed to consist of material that was in a state of being melted off the electrode. The following values of resistivity were obtained for the metals concerned near the melting points as indicated⁽⁵⁾:

<u>Metal</u>	<u>Temperature</u>	<u>ρ, ohm-cm x 10⁶</u>
Th	1900°C	30
Zr	1850°C	59
Mo	2660°C	81.4

The power number obtained by this method was used as a relative value and a guiding parameter only. By taking values for power obtained from data for molybdenum and zirconium in the literature and some of the values obtained for thorium, Fig. 5 was plotted for power versus crucible diameter. On the basis of the power available, a 2" diameter crucible was used in early work, and electrode diameters of 1/2" to 1" were experimented with in this crucible. Empirical data obtained showed that the largest diameter that could be used without arcing through the crucible was 3/4". Fig. 6 is a plot of relative quality of the resultant thorium ingots as a function of power at the arc. It is seen that the curve rises sharply and levels off near approximately one watt. This curve aided considerably in defining the minimum power required to obtain sound ingots with a 3/4" diameter thorium electrode. Fig. 7 is a composite group of curves plotted to show the effects of power, electrode diameter and currents required to obtain sound ingots when melting thorium in varying diameter crucibles. It is possible, by using these curves, to determine the proper electrode diameter for a given size of ingot of acceptable quality.

Five runs were made with power at the arc ranging from 0.16 to 1.6 watts. The quality of ingots made at higher arc

power inputs had the best visual appearance and were more sound (see Fig. 6).

Modification of the original equipment was found necessary to provide better control of manual feed of the electrode and better cooling of the copper crucible when using higher power inputs for the following reasons:

1. Sticking of the electrode to the starting thorium button was encountered when trying to feed the electrode due to the short arc length required at low amperages.
2. When the current was increased to more than 700 amperes, arcing through the crucible walls resulted.
3. It was also observed that above 400 amperes, enough heat was generated at the arc to cause reaction at the crucible walls, resulting in difficulty when ejecting ingots from the crucible after cooling.

The first modification instituted was to suspend the water-cooled copper electrode from an overhead spring attachment to provide better manual feed control. Another improvement was to modify the water cooling chamber surrounding the copper crucible to increase the velocity of water passing over the outer surface of the crucible per unit time. To accomplish this, a 3/4" water line was installed in place of the 1/4" water line originally used. The volume of the water jacket was also reduced to attain the required rate of flow.

As a result of the above modifications, arcing through the crucible wall was eliminated for high amperages and sticking of the ingots no longer occurred when ejecting them from the crucible. Ingots were ejected easily after reaching arc currents as high as 800 amperes. In later work, amperages as high as 1600 amperes showed no effect.

Another difficulty was encountered when making an ingot using 700 amperes arc current. A 3/16-inch pipe resulted at the top of the ingot. It was found that this could be eliminated by reducing the current at the end of the cycle and "puddling" the melt.

As a result of this work, it was possible to establish a tentative melting procedure using an argon atmosphere at 210mm mercury and 600 to 800 amperes current input at the arc. Cold-pressed thorium electrodes 12" long and 3/4" diameter were used to make these ingots.

Some experiments were then performed to determine the feasibility of melting in vacuo. The first run tried was with a vacuum of 40 microns. The result was a glow discharge when the current at the arc exceeded 250 amperes, and the run had to be stopped. It was felt that this was due to the outgassing of the electrode, and another run was tried using an electrode that was preheated in the arc-melting equipment by its own resistance with the current on. Outgassing of the electrode was noted on the vacuum gauge, and when the outgassing was completed, the electrode was cooled down. The final vacuum before melting was 40 microns. However, as soon as the arc was struck, the vacuum was lost, and a glow discharge again resulted. Finally, one experiment was made with an electrode that had been presintered and outgassed in vacuo at 1300°C. A vacuum of 0.3 microns was obtained in the sintering operation before the electrode was allowed to cool down. The arc-melting was started with a vacuum of 50 microns, but once again a glow discharge occurred, and the run had to be stopped.

Further investigation showed that a ratio of 80 - 20 argon - helium atmosphere was better than the vacuum, but not as good as the argon atmosphere. The vacuum gauge indicated a maximum of 100mm of mercury during the run. The arc was erratic and the molten metal pool was not as uniform as when an argon atmosphere was used. However, the resultant cooled ingot was relatively sound after ejection from the mold.

In addition to the foregoing, some modifications were made to increase the capacity of metal that could be melted in one run. A new crucible and a new water-cooled copper electrode were made, increasing the length of thorium electrodes up to 38". The crucible diameter was increased to 3". So far, ingots up to 10 pounds capacity have been made with the modified furnace, but it is possible to melt 1.5 to 2 times that amount of thorium when necessary.

Another important factor to consider is the ratio of the crucible diameter to the electrode diameter. Early investigations indicated that arcing through the Cu crucible or excessive sticking of

cooled ingot in the mold occurred when this ratio was less than $2\frac{1}{2}$ to 1. However, it was later found that when the argon pressure is lowered to 10mm of mercury, lower ratios of the crucible diameter to thorium electrode diameter were feasible. For instance, a 1" diameter electrode was successfully melted into a 2" diameter crucible without any of the above deleterious effects occurring. It is believed that this was possible because a superheating effect occurred at this argon pressure even though only 600 amperes were used for melting-down. To obtain an equivalent quality ingot in an argon pressure of 210mm, it would have been necessary to employ currents of 800 to 900 amperes, and as stated heretofore, these high currents cause arcing through with 1" thorium electrodes in a 2" diameter crucible. This effect is due to the short path to the crucible wall when the spacing is so small, resulting in the arc jumping to the side wall.

The amount of metal required to fill the entire mold depends upon the rate at which the melting is done, and therefore consideration was given to methods of improving the rate of melting. Experiments on deep pool melting of uranium derby material⁽⁶⁾ showed that the deepest pool depth is attained by melting in a low pressure atmosphere only slightly above a pressure of 30mm of mercury where glow discharge occurs. Fig. 8 shows that the maximum melting rate for thorium occurs near 10mm of mercury for $\frac{3}{4}$ " diameter electrodes melted in a 2" diameter crucible. This shows that by using the proper argon pressure, it is possible to attain a superheating effect equivalent to an increased amperage at the arc. It is estimated that by doing this, an effective increase of approximately 400 to 600 amperes resulted.

Returning to Figs. 7 and 8, we see it is significant that these curves can be adapted to obtain reasonable starting conditions for melting other metals. For instance, recent work on arc-melting of uranium by the U. S. Bureau of Mines⁽⁶⁾ indicates the value of such curves. A $2\frac{1}{2}$ " diameter thorium electrode was used to melt a 4" diameter sound ingot with 2300 amperes current at the arc and 25 to 30 volts d.c. The curve shows that approximately 3300 amperes are required for melting, which is not unreasonable if corrections are made for relative melting points of uranium and thorium, as well as for argon atmosphere and type of melting stock.

Taking into account the relative melting points, we find that a minimum of 1950 amperes are required for arc-melting a sound ingot. The difference between this figure and 2300 amperes can be accounted for by the fact that Derby uranium was used for melting stock mainly, and also by the fact that an argon pressure of 10mm of mercury will cause the superheating affect previously mentioned. Derby thorium was found to give inferior results as compared with the powder made from the oxide reduced with calcium. It is therefore reasonable to conclude that these curves are adaptable to establishing minimum conditions required for consumable electrode arc-melting of other metals.

B. Properties of Ingots

Table I contains composite chemical and physical data for the thorium powder used, buttons melted with nonconsumable tungsten electrode and thorium ingots produced by consumable electrode arc-melting. The data show a decided improvement in the hydrogen and calcium contents for the arc-melted buttons and ingots. The HCl insolubles show a pick-up however, indicating a higher final oxygen content for the arc-melted materials. Densities for arc-melted thorium are very close to the theoretical density of 11.71 g/cc, and the hardness of 57 BHN is relatively soft for this material. All of this data is based on single arc-melted ingots. It is possible to improve the analysis by remelting the ingot in a larger diameter crucible. As yet, this has not been tried, since the ingots singly melted are relatively clean and can be hot rolled satisfactorily. One ingot made from Derby thorium had a density of 11.60 g/cc and a hardness of 85 R_p, indicating the effects of impurities in the raw material used for electrodes.

A typical ingot was made from calcium-reduced powder electrodes and was rolled down from 1-1/2" diameter to 3/4" diameter after preheating to 800°C in argon. The metal flowed nicely and showed no evidence of surface cracking. Further work is in progress on deformation characteristics. Cold rolling experiments have shown that this material can be reduced 70 to 90 per cent, or more, by flat rolling with no intermediate annealing. The amounts of reduction permissible depends upon the percentage reduction per pass.

Figs. 9 and 10 show representative microstructures of the consumable electrode arc-melted thorium ingots at 250X and 100X. They show a dispersion of a second phase in the thor-

ium matrix, which is believed to be thorium oxide, on the basis of chemical analyses. Close inspection of Fig. 10 shows that the grain boundaries are partly outlined by another phase which has not been positively identified.

The top of the ingots, as cooled from the melt, have a bright and lustrous appearance on the surface. "Ripples" are apparent at the periphery where superheated metal has frozen. Fig. 11 shows machined ingots of various sizes and the resultant satin-type finish after machining approximately 1/8" to 1/4" from the diameter.

IV. CONCLUSION

The present investigation on the consumable electrode arc-melting of cold-compacted thorium powder showed that it is possible to obtain high-purity ingots that can be successfully rolled, hot or cold. The powder used for this work was made by calcium reduction of the oxide and all ingots were melted in one operation. Conditions necessary to obtain the most favorable ingots are summarized in the following relative to powder requirements, fabrication of electrodes, melting conditions, and purity and properties of resultant ingots:

A. Powder Requirements

The best results were obtained from powders having a hydrogen content about 250 ppm, calcium content near 500 ppm, and acid insoluble impurities below 0.03 per cent. Apparent densities of this powder ranged from 1.8 to 2.2 g/cc.

B. Fabrication of Electrodes

Electrodes were made by cold-compaction in air of powders to 85 to 94 per cent of theoretical density. The resultant moduli of rupture up to 7000 psi insured sufficient strength for handling and fabrication by welding into long, continuous electrodes. All welding was accomplished in an argon atmosphere.

C. Melting Conditions

The best results were obtained using an argon atmosphere at a pressure of 10mm of mercury. Pressure of the argon was

balanced continuously by the action of a roughing pump during the melting cycle. Specific melting rates obtained for various argon pressures by melting a 3/4" diameter electrode in a 2" diameter crucible at 600 amps are summarized below:

Melting Rate (gms/sec.)	Argon Pressure (mm Mercury)
10.2	4
10.4	10
7.0	30
4.5	210

Further work indicated that the current at the arc necessary for melting the sizes of electrodes used were as follows:

Electrode Diameter (inches)	Ingot Diameter (inches)	Arc Current (amperes)
0.75	2	600- 800
1.35	3	1400-1600

To prevent the formation of a "pipe" at the top of the ingot, it was necessary to decrease the current at the end of each melting cycle and "puddle" the melt. For 2" diameter ingots, the current was lowered to about 400 amperes, and for the 3" diameter ingots, the current was 1000 to 1200 amperes at the end of the cycle.

Experimental work on vacuum arc-melting resulted in "glow discharge" and the cycles had to be stopped. Outgassing of the electrode during melting evidently was excessive for the existing pumping system to handle and maintain low pressures during the cycle. Preheating and presintering electrodes in vacuum prior to melting was not helpful in preventing "glow discharge." Future work is contemplated on vacuum arc-melting using a faster pumping system.

Experiments with melting in a 80-20 helium-argon atmosphere were more successful than the vacuum arc-melting. However, it was found that the arc stability was not as good as in the case of an argon atmosphere.

D. Purity Workability and Properties of Ingots

It was observed that the quality of the single-arc-melted ingot depends on the purity of the powder used for making electrodes. An ingot melted from Derby thorium powder had a lower density and a higher hardness than ingots made from calcium-reduced thorium powder. It is conceivable that better results could have been obtained with Derby thorium powder by using a double-arc-melting technique, but this was beyond the scope of this work.

It has been demonstrated in this investigation that the chemical purity of the ingots produced are comparable in quality with ingots produced by the double arc-melting of Derby thorium⁽⁷⁾. It is also significant that the ingot analysis relative to the original powder shows little or no pickup of impurities. The following briefly summarizes the results for various contaminants:

1. Acid insolubles (oxides)

Double arc-melted Derby thorium contains <1 per cent oxide impurities in comparison to the 0.45 per cent oxide obtained for single arc-melted, calcium reduced thorium. The calcium-reduced thorium powder contained < 0.03 per cent oxide, but the higher results obtained for the ingot are explainable on the basis of air picked up and trapped while making the powder and fabricating it into electrodes.

2. Hydrogen

The original powder contained approximately 200 ppm hydrogen, which was reduced to below 50 ppm by arc melting.

3. Calcium

This element can be reduced to below 50 ppm by the method used herein. The original powder usually contains 500 ppm to 900 ppm of calcium.

4. Carbon

A small reduction in the amount of carbon occurs after melting. If the original powder is low in car-

bon, an ingot can be made with below 100 ppm carbon.

5. Magnesium

With original powder containing about 15 ppm, an ingot can be made with below 10 ppm. Double-arc-melted Derby thorium contains about ten times this amount of magnesium.

Ingots produced have very good densities near the theoretical for thorium, which indicates **sound** castings. This is apparent when one considers that reguline thorium made by casting, rolling, and extruding has densities in the range of 11.57 to 11.63 g/cc in comparison with 11.69 g/cc obtained in this investigation. This ingot density compares favorably with the 11.67 to 11.70 g/cc reported for double-arc-melted Derby thorium⁽⁷⁾. Hardness below 60 BHN **is also** comparable with the Derby thorium results.

Double-arc-melted Derby thorium has been extruded with less than 10 per cent rejections due to internal flaws. This is considerably better than extrusion results reported for thorium cast in various types of refractory crucibles and graphite molds⁽⁷⁾. It is reasonable to expect that the high-purity material reported on herein can be extruded as well as the Derby material. Preliminary results on cold- and hot-rolling of ingot material indicate that the workability is exceptional. No deleterious effects were observed on calcium-reduced thorium metal hot rolled to 75 per cent reduction in area. Cold rolling experiments have indicated that reductions of 70 to 90 per cent and over are obtainable by flat rolling with no intermediate anneals. The percentage reduction before annealing depends on the percentage reduction per pass.

Further work is now in progress on the deformation characteristics and physical properties of ingots made from high purity calcium-reduced thorium powder. Some work is also contemplated on vacuum arc melting using a faster pumping system than the existing facilities.

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Project 1890

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TABLE I

Arc Melted Thorium Comparison Chart

Material	Chemical Analysis (ppm)							Hardness (BHN)	Archimedes Density (g/cc)
	N ₂	C	H ₂	Ca	Zn	Mg	HCl insol.		
SYLVANIA powder	60-70	140- 150	~250	~500	-	~15	~0.03%	-	-
Typical Arc Melted Button	40	200	29	~100	-	-	1 - 1.2%	-	11.67-11.70
Typical Arc Melted Powder Ingot (SYLVANIA)	55	~40	45	~30	-	8	0.45%	57	11.69 (geome- tric)
Double Arc Melted Derby Ingot ⁽⁷⁾	150	-	-	200	50	100	<1%	50-55	11.65-11.70

Fig. 1 - ARC CASTING EQUIPMENT

Fig. 2 Photomicrograph of thorium powder
made by calcium reduction of thor-
ium oxide.

Plate No. 12730 Unetched 100X

Fig. 3 - Relationship of compacting pressure to green density for thorium powder made by reduction of the oxide with calcium.

Fig. 4 - Relationship of compacting pressure to modulus of transverse rupture for thorium powder made by reduction of the oxide with calcium.

Fig. 5 - Relation of power and ingot diameter for arc melting molybdenum, zirconium and thorium.

Fig. 6 - Relation between ingot quality and power at the arc in watts.

Fig. 7 - Electrode current for various electrode diameters to produce sound ingots.

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Fig. 8

Fig. 9 Photomicrograph of oxide inclusions in thorium
matrix
Plate No. 14382 Unetched 250X

Fig. 10 Same specimen as in Fig. 9 showing dispersion of
oxide inclusions. These inclusions appear to out-
line grain boundaries.
Plate No. 14381 Unetched 100X

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Fig. 11 Various sizes of machined ingots made from calcium-reduced thorium powder.

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