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The Contamination of Molten
Thorium by Zirconia and
Beryllia Crucibles

Office
Department
Washington .

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CLASSIFICATION CANCELLED

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1. Introduction

A project is currently active in the Metallurgical Department of National Research Corporation to determine the necessary data for the design of a furnace to dezinc, melt and cast thorium metal. One of the problems in this operation is the decision as to the refractory material which should be used for the crucible. In current operation at the Ames Laboratory, a beryllia crucible is being used. There has been some evidence from operations at the Oak Ridge National Laboratories that a stabilized zirconia would be as satisfactory from the standpoint of contamination. Such a substitution is a very attractive one because of the lower cost of zirconia, and the elimination of the health hazard involved in handling beryllia.

Since small scale melting facilities were available at National Research Corporation, it was proposed by the New York Operations Office that a series of melts be made to evaluate the relative amounts of contamination introduced by beryllia and zirconia crucibles.

2. Experimental Technique

2.1 General Method

A series of four melts in each type crucible was planned with the goal of determining the degree of contamination obtained over the temperature interval of 1750°C to 1900°C, with a constant time of exposure at temperature of 30 minutes.

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The lower temperature limit was selected as being the lowest pouring temperature compatible with the melting point of 1625°C reported by Oak Ridge National Laboratories. The upper one is that which has been given as the point beyond which contamination increases rapidly by reason of the reduction of the oxide.

2.2 Materials

2.21 Beryllia crucibles were obtained from Brush Beryllium Corporation, and stabilized zirconia crucibles were from Titanium Alloy Manufacturing Division of National Lead Company.

2.22 Thorium was obtained as a dezincd biscuit from the Ames Laboratory.

2.3 Melting Procedure

The melting unit used was a metal tank type vacuum furnace containing an induction coil assembly for a crucible approximately 2-3/4 inches diameter by 5 inches height. It was capable of operating at a pressure of .001 mm. A 10,000 cycle, 200 volt, 30 KVA motor generator provided ample power to heat very rapidly to 2000°C or higher.

The general procedure used was one of first determining the cold rate of leak of the unit to be satisfactory, and then charging the crucible and pumping down to operating pressure.

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Power was then applied to heat at as rapid a rate as possible without risking loss of the crucible by thermal shock. The melting was accomplished and temperature raised to the soaking temperature as rapidly as possible. Upon completion of the 30-minute treatment, the melt was allowed to solidify in the crucible.

2.4 Instrumentation

2.41 Pressure was measured by a mercury manometer in the high pressure range, and by an Alphatron vacuum gage in the range from .001 mm. to 10 mm.

2.42 Temperature was measured by two methods: The first was by a Leeds and Northrup optical pyrometer with three scales. This was sighted through a sight glass which could be cleaned by a wiper. The second method utilized a wolfram-molybdenum thermocouple, the E.M.F. of which was measured by a Leeds and Northrup potentiometer. Since this thermocouple was subject to frequent failure, it was the secondary method, used primarily to check the first.

2.5 Analytical Technique

2.5 Sampling was accomplished by drilling the solidified casting. Only one sample was taken from each melt because a pronounced stirring effect is obtained in the melt, and the very low heat of fusion makes solidification very rapid. These factors would indicate a minimum of trouble from segregation.

2.52 Analyses were performed by the Battelle Project under the direction of Dr. Arthur E. Bearse. The beryllium determination was made by a direct spectrographic method. The zirconium was first concentrated by a cupferron extraction procedure attributed to Dr. Rodden of the New Brunswick Laboratory, and then determined spectrographically.

3. Experimental Results

3.1 Specific Treatment Data

3.11 Zirconia melts were made under the following conditions:

<u>Melt</u>	<u>Soak Temp. °C</u>	<u>Time (Min.) From M.P. to Soak Temp.</u>
1	1235 - 1750	12
2	1812	10
4	1900	15
5	1838 - 1858	15

All melts were held at the soak temperature for 30 minutes. The first two melts were made at low pressure (.001 mm.) entirely. The violent activity of the melt, presumably from the vaporization of residual zinc, made it necessary to melt under 100 mm. of argon in subsequent melts, pumping down to .001 mm. after melting had been accomplished.

3.12 Beryllia melts were made under the following conditions:

<u>Melt</u>	<u>Soak Temp. °C</u>	<u>Time (Min.) From M.P. to Soak Temp.</u>
6	1740 - 1750	35
7	1800 - 1815	13
8	1840 - 1850	19
9	1900 - 1915	21

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All melts were held 30 minutes at the soak temperature. In melt 6, there was an unusually long time to come to the soaking temperature because of melt activity, probably due to zinc segregation in the piece.

3.2 Analytical Data

The data obtained for both beryllia and zirconia melts are summarized in the following table. It should be noted that a control sample was taken from the dezincd biscuit which had been obtained from the Ames Laboratory and cut up for the melts.

<u>Series</u>	<u>Nominal Temp. °C</u>	<u>Wt.% Zr</u>	<u>Wt.% Be</u>
Zirconia	1745	0.040	
	1810	0.060	
	1850	0.035	
	1900	0.043	
Beryllia	1745		0.054
	1810		0.025
	1845		0.075
	1910		0.075
(Control)	----	0.015	0.001

4. Conclusions

4.1 It appears that the following conclusions may be drawn from these experiments.

4.11 Within the range of 1745°C to 1900°C, with a constant exposure time, there seems to be no correlation between temperature and degree of contamination.

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4.12⁷A comparison of the contamination data for ~~beryllia~~^{beryllium} ~~conia~~^{fine} reveals an average increase in ~~beryllium~~^{fine} level

5. Recommendations

This comparison of beryllia and zirconia as crucible material for containing molten thorium indicates that there would seem to be no reason for preferring beryllia over zirconia, and in fact indicates a possible slight advantage of zirconia from the contamination standpoint. It is accordingly recommended that zirconia be tested on a larger scale.

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