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Preparation of Zirconium from Zirconium Tetrafluoride

by

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Preparation of Zirconium from Zirconium Tetrafluoride*

C. J. Baroch and G. H. Beyer

Abstract

Increased interest in zirconium as a material of construction has resulted in numerous attempts to develop more economical processes for its production. Currently the Kroll Process, which reduces zirconium tetrachloride with magnesium, is used. A process developed at the Ames Laboratory of the Atomic Energy Commission involves the bomb reduction of zirconium tetrafluoride with calcium. By substituting magnesium for calcium, the cost of producing zirconium by this process might be considerably reduced.

This report is concerned with experimental bomb reductions of zirconium tetrafluoride with magnesium. A charge of 350 grams of zirconium tetrafluoride, 105 grams of zinc fluoride, and 142 grams of magnesium gave good metal recovery and good metal-slag separation. Reaction of the zinc fluoride with magnesium furnished the additional heat needed to melt the slag. The zinc-zirconium alloy formed melted at about 1000°C, while the magnesium fluoride slag melted at about 1250°C. After dezincing, the zirconium sponge was melted and its hardness measured. Hardness values of 62 on the Rockwell A Scale indicated the probable presence of impurities. With more stringent control of the purity of the reactants and by the use of larger bombs, it should be possible to produce higher purity zirconium by this method.

Introduction

Zirconium, often called the "female metal" because of its unpredictable characteristics, is element number 40 in the periodic table and has an atomic weight of 91.22. In nature zirconium is always associated with about two per cent hafnium, which greatly increases the neutron cross section of zirconium. However, the hafnium has little effect on the chemical and physical properties of zirconium.

Until recent years, industrial use of zirconium and zirconium compounds was limited primarily to the production of ceramics and refractories. With the advent of nuclear power, interest in zirconium has increased rapidly. Low neutron absorption and good corrosion resistance at high temperatures make zirconium a

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desirable material of construction for nuclear reactors. The United States Atomic Energy Commission has fostered interest in the development of new and better processes for the production of ductile zirconium. Currently the Kroll Process is producing ductile zirconium by the reduction of zirconium tetrachloride with magnesium at a cost of about \$10 per pound.

In the Kroll Process zirconium tetrachloride is sublimed into a bath of molten magnesium under a protective atmosphere of helium. During the ensuing reaction zirconium sponge and magnesium chloride are formed. The excess magnesium and the magnesium chloride are then drained and distilled away from the zirconium sponge.

Several other processes have been developed for opening up the zircon ore and reducing zirconium compounds to metal (1,5,6). One of these processes, developed at the Ames Laboratory, reduces zirconium tetrafluoride with calcium in a bomb reduction (2,8). Zinc fluoride and iodine boosters were used to lower the melting points of the zirconium metal and the calcium fluoride slag, and to furnish additional chemical heat.

A large portion of the cost of producing ductile zirconium is attributable to the reduction step. There are many compounds of zirconium which can be reduced with either alkali or alkaline earth metals. However, many zirconium compounds contain oxygen which is virtually impossible to eliminate from the metal.

Zirconium tetrachloride is very hygroscopic, and must be kept in a dry atmosphere. When zirconium tetrachloride hydrolyzes, corrosive fumes of hydrogen chloride are evolved. In sharp contrast, zirconium tetrafluoride is stable even in rather moist atmospheres.

The ease of handling the tetrafluoride lends itself to reduction with calcium in closed bombs. Metal of hardness 45 or less on the Rockwell A Scale has been made by calcium reduction. By substituting magnesium for calcium, the cost of producing zirconium from the tetrafluoride might be considerably reduced.

The following report is concerned with experimental bomb reductions of zirconium tetrafluoride with magnesium.

Thermodynamic Considerations

The bomb reduction of zirconium tetrafluoride with magnesium requires that sufficient heat be liberated by chemical reaction to melt the products and allow separation into two phases. Because of the high melting point of zirconium (about 1850°C), it is advantageous to produce a lower-melting alloy, such as the zinc-zirconium eutectic containing 30 weight per cent zinc, which melts at about 1050°C. The zinc can later be removed by heating the alloy in a vacuum.

The thermodynamic properties of some of the materials of interest are given in Table 1. These data were compiled from Lange (4) and Quill (7). The standard heats of reaction for some metallothermic reductions are shown in Table 2.

It is well known that a temperature of 3000°C may be produced almost instantaneously by igniting a powdered mixture of ferric oxide and aluminum. This heat of reaction, shown in Table 2, is seen to be abnormally high. Reactions of zirconium compounds with magnesium and calcium produce considerably less heat. Calcium reacts with tetrafluoride more exothermically than does magnesium. Substituting magnesium for calcium as a reductant decreases the heat liberated. Other reactants such as zinc fluoride may be added to supply additional heat, as well as to form a low-melting alloy with zirconium. Substituting magnesium for calcium also decreases the heat obtained from such "booster" reactions.

The heat needed to reach a sufficiently high temperature for good metal-slag separation depends also on the heat capacities and heats of fusion of the substances involved. Enthalpy data are presented graphically in Figure 1, compiled from Quill (7) and Kelley (3). Data are incomplete and in some cases the straight-line extrapolations shown may be questionable.

Using the data presented in Tables 1 and 2 and in Figure 1, it is possible to estimate the adiabatic reaction temperature for any bomb charge, assuming complete reaction occurs without heat loss. Such calculations can be compared with successful and unsuccessful experimental bomb results to estimate the heat required for satisfactory phase separation.

It is, of course, possible to supplement the heat supplied by chemical reaction by preheating the reactants. Such preheating is limited by the temperature at which reaction spontaneously occurs, but does permit the attainment of appreciably higher maximum temperatures. Thus, a gas-fired bomb which does not react until preheated to a temperature of 900°K may contain sensible heat representing 25 to 35% of the reaction heat liberated. Preheating may make possible the elimination of boosters, such as iodine, the primary purpose of which is to generate additional heat.

Experimental Bomb Reductions

The metallothermic reduction of thorium and uranium halides has been successfully developed at the Ames Laboratory of the Atomic Energy Commission. The experience gained with these metals led to the development of a process in which zirconium tetrafluoride was reduced in a closed bomb using purified calcium as a reductant. Substitution of magnesium for calcium required changes in the composition of the charge to the bomb and in the method of firing, to supply sufficient heat for phase separation.

Table 1
Thermodynamic Properties

Material	Melting point deg C	Boiling point deg C	Specific gravity	Heat of fusion cal per g mole	Heat of formation Kcal per g mole
Aluminum	660	2057	2.70	2,550	0.0
Aluminum oxide	2040	-	3.99	2,600	-399.1
Calcium	851	1440	1.55	2,230	0.0
Calcium fluoride	1420	2507	3.18	7,100	-290.2
Calcium iodide	740	1227	3.96	5,000 ^a	-143.4
Ferric oxide	1560	-	5.12	-	-196.5
Iron	1535	3000	7.86	3,860	0.0
Magnesium	651	1110	1.74	2,160	0.0
Magnesium fluoride	1263	2270	3.00	13,900	-263.0
Magnesium chloride	712	1412	2.33	10,300	-153.2
Magnesium iodide	650	900	1.46	5,300 ^a	-101.7
Zinc	419	907	7.14	1,600	0.0
Zinc fluoride	872	1502	4.84	7,000 ^a	-176.0
Zirconium	1860	-	6.40	5,500 ^a	0.0
Zirconium tetrafluoride	902 ^a	902 ^a	3.25	18,000 ^a	-445.0
Zirconium tetrachloride	437	-	2.80	10,500 ^a	-230.0

^aEstimated values

Table 2

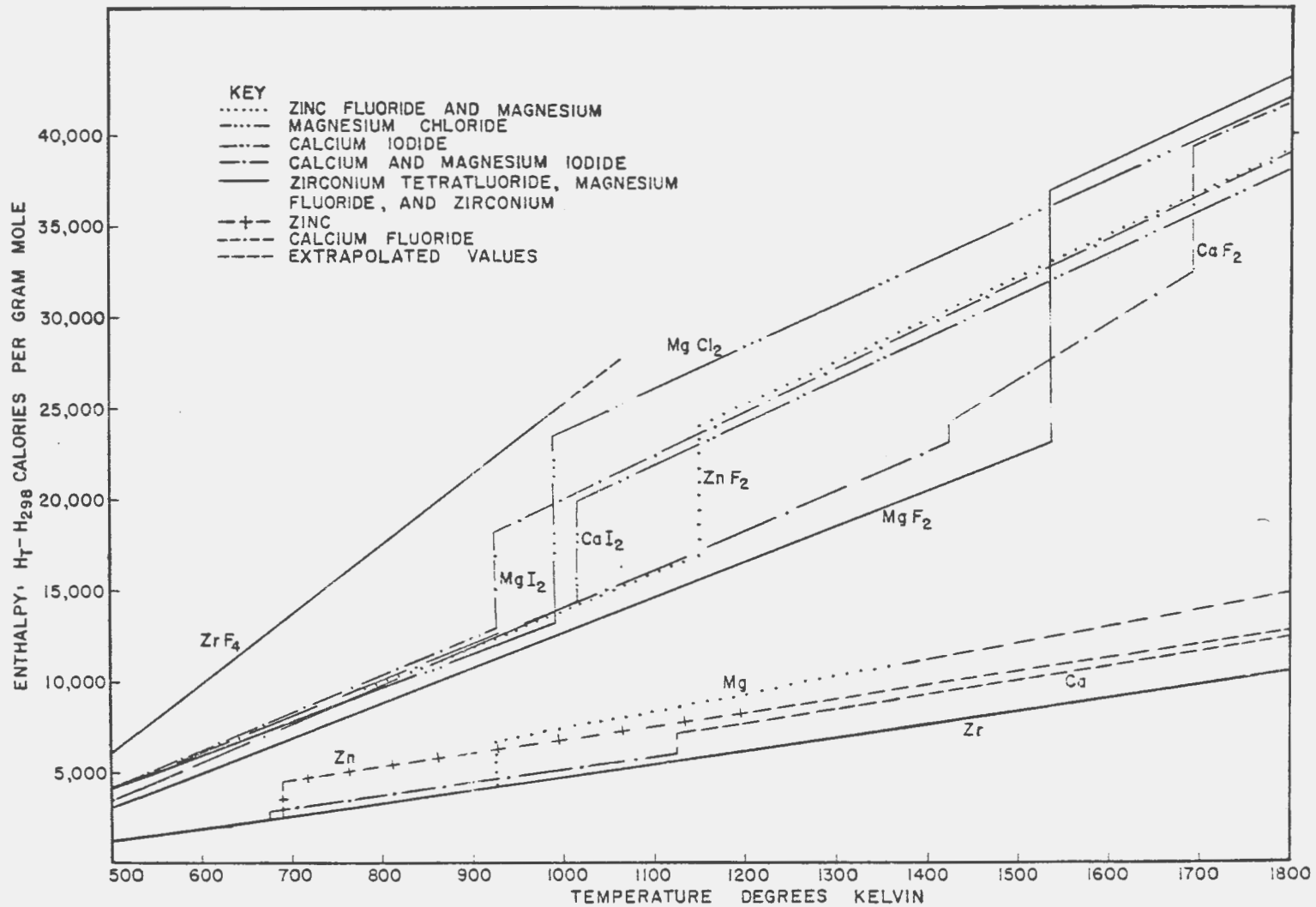
Standard Heats of Reaction for Some Metallothermic Reductions

Reaction	Heat of Reaction Kcal
$\text{Fe}_2\text{O}_3 + 2\text{Al} \longrightarrow \text{Al}_2\text{O}_3 + 2\text{Fe}$	-202.6
$\text{ZrCl}_4 + 2\text{Mg} \longrightarrow 2\text{MgCl}_2 + \text{Zr}$	- 76.4
$\text{ZrF}_4 + 2\text{Mg} \longrightarrow 2\text{MgF}_2 + \text{Zr}$	- 81.0
$\text{ZrF}_4 + 2\text{Ca} \longrightarrow 2\text{CaF}_2 + \text{Zr}$	-135.4
$\text{ZnF}_2 + \text{Mg} \longrightarrow \text{MgF}_2 + \text{Zn}$	- 87.0
$\text{ZnF}_2 + \text{Ca} \longrightarrow \text{CaF}_2 + \text{Zn}$	-114.0

A cross section of a typical experimental bomb is shown in Figure 2. The bomb was constructed of ordinary steel pipe, welded shut at the bottom and closed by a cap at the top. A refractory liner was necessary to prevent interaction of the molten products with the walls. Liners were usually jolt-packed around a mandrel to insure firm support by the steel wall. When presintered liners were used, they were secured in the bomb with granular "magnorite" to prevent mechanical failure. Additional refractory material was packed on top of the charge. Jolt-packed liners of calcium fluoride or magnesium oxide were used in most of the experiments.

Two different methods of firing were used. Spark-fired bombs were ignited by passing a current through a niobium wire attached to a spark plug in the wall of the bomb. This current heated a small portion of the charge to ignition temperature. Gas-fired bombs, such as shown in Figure 2, were ignited by placing in a furnace maintained at about 850°C. The sensible heat provided by

Figure 1 - Enthalpy Data



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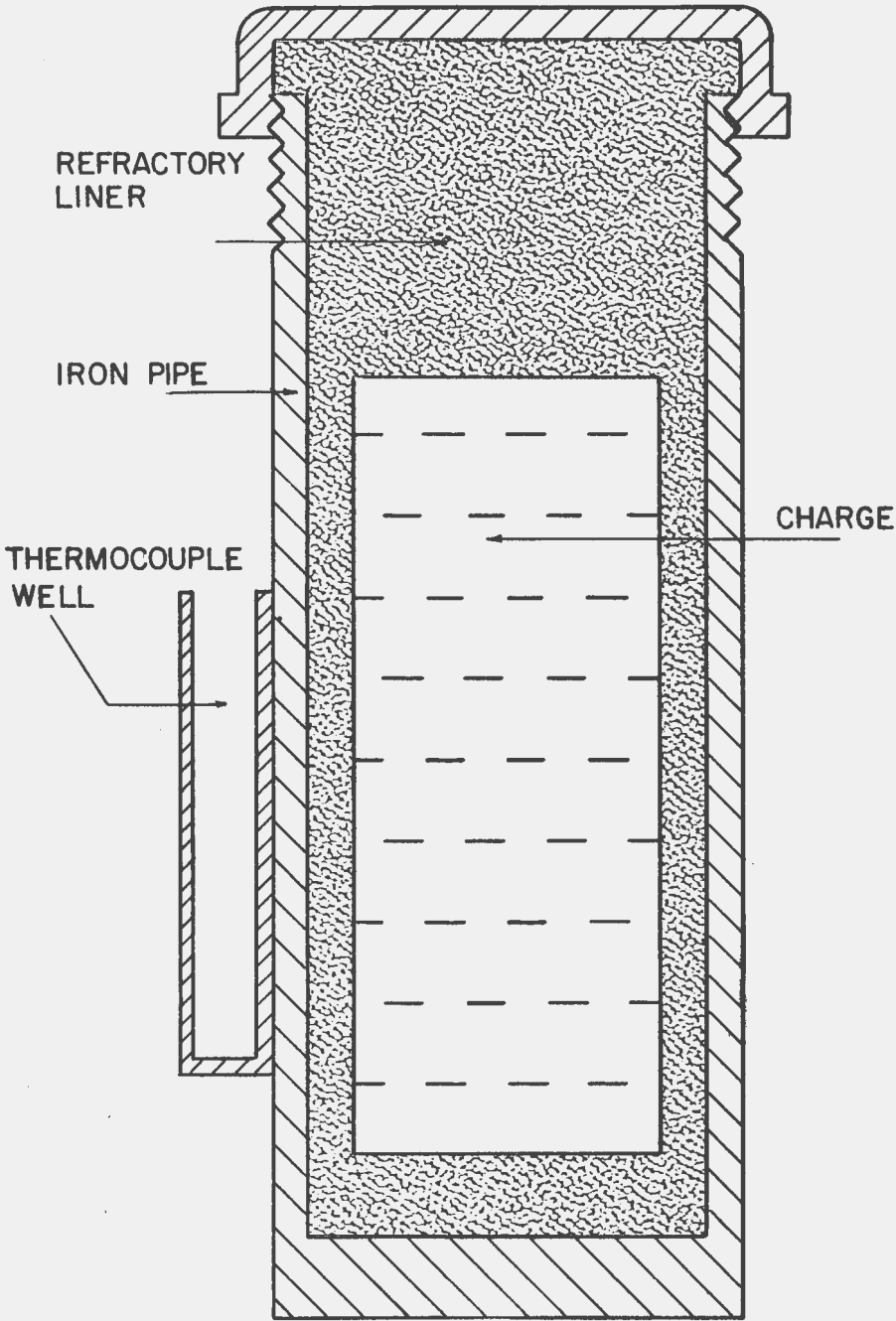


Figure 2 - Gas-Fired Reduction Bomb

gas-firing produced higher temperatures with the same charge. Boosters, such as iodine, might be eliminated by using gas-firing. On the other hand, gas-firing degassed the liner and increased metal hardness. In the case of calcium reductions the spark-fired reduction bombs generally yielded softer zirconium than the gas-fired bombs.

A comparison of the bomb reduction charges used is presented in Table 3. With a heat of reaction of 315 Kcal, it was possible to obtain good separation of phases when calcium was used. Table 3 shows a heat of reaction of only 297 Kcal for the spark-fired magnesium bomb. In most cases this proved insufficient to allow the molten alloy formed to drain completely to the bottom and separate from the slag. Gas-firing added sufficient sensible heat to accomplish good metal-slag separation even when the iodine booster was omitted from the charge. Figure 3 indicates the excellent metal-slag separation obtained when sufficient heat was provided.

Table 3
Comparison of Bomb Reduction Charges

	Spark-fired Calcium	Spark-fired Magnesium	Gas-fired* Magnesium
Composition	350 g ZrF_4 70 g Zn 221 g Ca 56 g I_2	350 g ZrF_4 105 g ZnF_2 150 g Mg 100 g I_2	350 g ZrF_4 105 g ZnF_2 142 g Mg
Heat of Reaction	315 Kcal	297 Kcal	257 Kcal
Sensible Heat	0 Kcal	0 Kcal	83 Kcal
Total heat	315 Kcal	297 Kcal	340 Kcal
Heat per g mole ZrF_4	151 Kcal	142 Kcal	163 Kcal
Estimated Temperature	2033 deg C	1641 deg C	1909 deg C

* Preheat temperature of 900°K



Figure 3 - Metal-Slag Separation

Because the quality of the materials charged to the bomb largely determines the quality of the metal, a brief description of the source of each of these materials follows:

Calcium: Commercial grade calcium, produced by the aluminum reduction of calcium oxide, was purified by distillation at the Ames Laboratory.

Calcium Fluoride: The calcium fluoride used for liners contained about 97 per cent calcium fluoride. By drying at 500 to 600°C in a hydrogen fluoride atmosphere, the volatile constituents such as water and carbonaceous material were eliminated.

Iodine: Chemically pure iodine was used.

Magnesium Oxide: Magnesium oxide or "Magnorite" for refractory liners was dried at about 600°C to remove water and carbonaceous material.

Magnesium: A commercial grade of magnesium was used in some reductions, distilled magnesium was used in others. Analyses indicated that the distilled magnesium contained less than 100 parts per million of oxygen and very small amounts of metallic impurities.

Zinc: Lathe turnings were obtained from slab zinc (99.99% pure).

Zinc Fluoride: Zinc fluoride was prepared by reacting zinc oxide with 70 per cent hydrofluoric acid. The wet zinc fluoride was either vacuum-dried at 200 to 300°C or dried in an anhydrous hydrogen fluoride atmosphere at 400 to 500°C.

Zirconium Tetrafluoride: Zirconium tetrachloride was dissolved in water and the solution evaporated until crystals of zirconyl chloride octahydrate formed. After filtering and washing, these crystals were treated with 48 per cent hydrofluoric acid, forming zirconium tetrafluoride monohydrate crystals. Anhydrous zirconium tetrafluoride was obtained by first air-drying the crystals at 100°C and then drying in an anhydrous hydrogen fluoride atmosphere at 400 to 500°C. Difficulty was encountered in the hydrogen fluoride drying, and some zirconium oxide or zirconium oxyfluoride was formed. Instead of yielding zirconium with a Rockwell "A" hardness of 40 to 42 in a standard calcium bomb reduction, the zirconium tetrafluoride used in the following reductions yielded zirconium with a Rockwell "A" hardness of 48 to 50.

In packing the bombs precautions were taken to eliminate oxide contamination. The ingredients were weighed and placed in a container connected to a vacuum system. The pressure was reduced to less than 50 microns, the system flushed with argon, sealed, and the ingredients were then thoroughly mixed. After mixing, small amounts

of the charge (about 100 grams) were poured into the refractory-lined bomb and tamped with a rod. While packing the bomb, a protective atmosphere of argon was maintained over the charge. When the entire charge was in place, a refractory material was packed on top of the charge. The bomb was closed with either a flanged cap or a refractory-lined screw cap.

After the bomb fired, it was removed from the furnace and allowed to cool to room temperature. The bomb was then opened and the ingredients were removed by tapping its sides. The zinc-zirconium alloy was cleaned of loosely adhering slag and leached in dilute hydrochloric acid to remove any unalloyed zinc or magnesium. After the biscuit was washed and dried, it was ready for dezincing and casting.

The dezincing and casting operations were performed in an ATZ graphite crucible mounted in a split-graphite resistance furnace capable of obtaining temperatures as high as 2000°C. During a satisfactory dezincing and casting operation, a vacuum of less than 100 microns was maintained. After a vacuum of one to ten microns had been maintained for about one-half hour, the current was turned on and the sample melted. The melting operation generally required 20 to 30 minutes. The sample was allowed to remain molten for two to ten minutes before the furnace was turned off. After the system cooled to room temperature, the vacuum was released and the graphite crucible removed from the furnace. The graphite was chipped from the zirconium biscuit, the biscuit weighed, sectioned with a carborundum wheel, and a Rockwell "A" hardness test taken.

Table 4 presents the results of twelve experimental bomb reductions using the charges given in Table 3. Yields for the spark-fired calcium bombs varied from 65 to 80%. The hardness values obtained were some 6 to 8 points higher than the values possible with high-quality tetrafluoride (40-42). Differences between replicates are indicative of the poor reproducibility of results in small-scale reductions. On a larger scale both yield and quality would ordinarily be expected to improve.

Lower yields (50-65%) were obtained in the spark-fired magnesium reductions. Zirconium hardness was high, although use of high-purity magnesium lowered hardness somewhat. Gas-firing the magnesium bombs improved the yield, but hardness values of 61-65 indicated that more stringent control of the quality of the reactants is necessary to produce higher purity zirconium by this method.

Adiabatic reaction temperatures are reported in Table 3 for each of the charges used. Several assumptions were necessary in arriving at "estimated temperatures." It was assumed that heat capacities could be extrapolated linearly, that reactants were

Table 4. Experimental Bomb Reductions

	Wt. alloy recovered (grams)	Wt. after dezincing (grams)	Alloy composition (Wt.% Zn)	Zirconium yield %	Rockwell A Hardness	Comments
<u>Spark-fired calcium</u>	1. 207.6	125.6	39.5	66.3	49.3	
(CaF ₂ liner)	2. 241.0	148.5	38.3	78.3	48.3	
	3. 226.1	141.7	37.4	74.5	49.2	17 grams of magnesium reductant added
<u>Spark-fired magnesium</u>	1. 147.3	104.8	28.6	55.6	72.8	
(CaF ₂ liner)	2. 166.6	122.0	26.6	64.5	72.5	
	3. 152.7	100.0	34.2	52.7	61.2	High purity magnesium
<u>Gas-fired magnesium</u>	1. 210.7	131.8	37.6	69.4	62.6	Excellent metal-slag separation. (Cf Fig. 3)
(MgO liner)	2. 184.6	126.4	31.5	66.6	65.3	High purity magnesium
	3. 193.6	138.0	28.8	72.5	64.0	High purity magnesium
(Graphite liner)	4. 165.4	116.3	29.4	68.7	62.8	High purity magnesium, sublimed ZrF ₄
	5. 207.5	127.9	28.4	67.4	61.2	High purity magnesium, sublimed ZrF ₄
	6. 158.5	107.0	33.7	58.9	62.9	High purity magnesium, sublimed ZrF ₄

completely converted into products, that no thermal effects accompanied the formation of the zinc-zirconium alloy, and that the reaction was adiabatic.

Attempts were made to measure the maximum temperature attained during reduction. The thermocouple well attached to the side of the bomb was of use only in indicating when the bomb had fired. Maximum temperatures from 1130 to 1175°C were measured by inserting a thermocouple well into the center of spark-fired bombs. Measurements on gas-fired bombs were unsuccessful because the thermocouple wells melted and the contents of the bomb were discharged. Experimental verification of the melting point of the magnesium fluoride slag (about 1250°C) indicated that temperatures at least that high were actually attained in spark-fired bombs. Temperatures perhaps 200-300°C higher were probably attained in gas-fired bombs.

Since pressure as well as temperature was of interest, attempts were also made to measure the pressure developed inside a spark-fired bomb. A pressure gauge connected to the bomb indicated an instantaneous pressure of 100-150 psi upon firing, followed by an intermediate pressure of 60 psi for 1 - 2 minutes before dropping to zero.

Several important conclusions followed from the experimental work. One important conclusion was that magnesium can be used to reduce zirconium tetrafluoride in gas-fired bombs with the aid of zinc fluoride booster to provide additional chemical heat. Yields of 60 to 75% were obtained in small bombs, with good prospects for even better yields in larger bombs.

A second important conclusion was that magnesium reduction results in an appreciably harder metal than calcium reduction. Magnesium may be satisfactory, however, if sufficiently pure reactants are used. Oxygen and nitrogen are thought to be the chief impurities. Since the Kroll process uses magnesium successfully, it should be possible to produce ductile zirconium by bomb reduction of the tetrafluoride if the impurities are properly controlled.

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APPENDIX: SAMPLE CALCULATIONS

Minimum Heat Requirements: In calculating the minimum heat requirements and maximum temperature attained in the reductions, the following assumptions were made: (1) that the reaction was adiabatic, (2) that the heats of formation of the zinc-zirconium alloy and the slag were negligible, (3) that the enthalpy data of Figure 1 can be extrapolated linearly, and (4) that at temperatures greater than 1100 degrees centigrade the zirconium is present as a liquid in the zinc-zirconium alloy. The total enthalpy of the zirconium, therefore, equals the value obtained from Figure 1 plus the heat of fusion of zirconium (5.5 kilocalories per gram mole).

The minimum heat requirements for a gas-fired reduction bomb using magnesium as a reductant are presented in Table 5. The charge consisted of 350 grams of zirconium tetrafluoride, 105 grams of zinc fluoride, and 142 grams of magnesium. To obtain a good metal-slag separation, the magnesium fluoride as well as the zinc-zirconium alloy formed in the reaction must be molten. To melt the magnesium fluoride, the inside temperature of the bomb must be greater than 1263 degrees centigrade or approximately 1550 degrees Kelvin. The enthalpy of the reaction products (magnesium fluoride, zinc, zirconium, and excess magnesium) at a temperature of 1550 degrees Kelvin determines the minimum heat requirements. This heat must be supplied by the heat of reaction and the sensible heat provided by preheating the bomb.

Table 5

Minimum Heat Required to Obtain Molten Products in a Gas-Fired Magnesium Reduction Bomb

Material	Enthalpy 1550 Kelvin Kcal per g Mole	Moles in Bomb	Total Enthalpy Kcal	Per cent of Total Enthalpy
MgF ₂	-37.3	5.18	-193.5	79.6
Zn	-10.8	1.00	- 10.8	4.4
Zr	-14.3	2.09	- 29.9	12.3
Mg	-12.6	0.71	<u>- 8.9</u>	<u>3.7</u>
		Total	-243.1	100.0

Estimated Temperature: By knowing the total heat input and the enthalpy versus temperature data for the reactants and the reaction products, one can estimate the maximum temperature achieved during reaction. If it is assumed that the reaction is adiabatic, then the enthalpy of the reaction products must equal the enthalpy of the reactants plus the heat of reaction. One can calculate the enthalpy of the products at various temperatures. The maximum temperature obtained is the temperature at which the enthalpy of the reactants plus the heat of reaction equals the enthalpy of the products. The maximum temperature determination for the gas-fired magnesium reductions, with the aid of zinc fluoride booster, is presented in Table 6.

Table 6. Estimated Maximum Temperature in a Gas-fired Magnesium Reduction Bomb

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Input								
Mat.	Wt. grams	Moles	Heat of Reaction Kcal per g mole	Total Heat of Reaction Kcal	Sensible Heat at 900 Kelvin		Total Heat Sensible Plus Heat Reduction	Per cent of Total
					Kcal/g mole	Total Kcal		
ZrF ₄	350	2.09	-81.0	-170.0	-21.5	-45	-215	63.8
ZnF ₂	105	1.00	-87.0	- 77.0	-12.0	-12	- 99	29.1
Mg	142	5.94	0.0	0.0	- 4.0	-24	- 24	7.1
			Total	-257.0		-81	-338	100.0

Output									
Mat.	Wt. grams	Moles	Sensible Heat at 1700 Kelvin		Sensible Heat at 1800 Kelvin		Sensible Heat at Estimated Temperature		% Total at Est. Temp.
			Kcal/g mole	Total Kcal	Kcal/g mole	Total Kcal	Kcal/g mole	Total Kcal	
MgF ₂	323	5.18	-40.8	-211	-43.2	-223	-52.2	-269	78.9
Zn	65	1.00	-12.0	- 12	-12.8	- 13	-15.8	- 16	4.7
Zr	190	2.09	-15.3	- 32	-16.1	- 34	-19.1	- 40	12.3
Mg	18	0.76	-14.0	- 11	-14.9	- 11	-18.3	- 14	4.1
			Total	-266		-281		-339	100.0

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Change in enthalpy per 100 degrees Kelvin = $-281 - (-266) = -15$ Kcal.

Estimated temperature = $1800 + 100 \frac{[-338 - (-281)]}{-15} = 2182$ Kelvin or 1909 degrees centigrade.