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METALLURGY AND CERAMICS

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UNITED STATES ATOMIC ENERGY COMMISSION

QUARTERLY SUMMARY RESEARCH REPORT IN METALLURGY

For January, February, March, 1955

by

Ames Laboratory Staff

October 21, 1955

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For The Atomic Energy Commission

H. F. Cancele
Chief, Declassification Branch

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QUARTERLY SUMMARY RESEARCH REPORT IN METALLURGY
For the period January, February, and March, 1955

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submitted by the group leaders of
the Laboratory

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METALLURGY

Under the direction of F. H. Spedding, H. A. Wilhelm,
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D. Peterson, and J. F. Smith

1. Purification and Separation Studies

1.1 Some Extractions with Primene-JMT Solutions (1)*

As reported earlier an aqueous solution containing aluminum, bismuth, cadmium, copper, chromium, iron, lanthanum, magnesium, manganese, neodymium, nickel, tin, thorium, uranium, vanadium, yttrium and zinc in the presence of sulfate was made for test purposes. It has been found that if an oxidizing agent is added to this complex sulfate solution, the bismuth, iron, thorium and uranium are preferentially extracted into an organic phase containing Primene-JMT. When a reducing agent was added to the aqueous phase, only bismuth, thorium and uranium were preferentially extracted by the organic phase containing the Primene-JMT.

An application of this type of separation has been made on a sulfuric acid extract of monazite. The thorium was preferentially extracted into the organic phase. The most selective extraction of thorium has been observed with combination of the monazite sulfate extract and Primene-JMT with additions of hexone and HCl.

1.2 The Separation of Niobium from Titanium (1)

A method of analysis for titanium in the presence of niobium in the range from 2.5% to 34 ppm, was set up by the spectrographic group using the 80 grams of high purity Cb_2O_5 prepared by a method given in a previous report. The method of analysis showed that niobium containing only 34 ppm of titanium could be obtained by a single extraction of niobium from a hydrofluoric acid solution containing 2.0% titanium with respect to the niobium. Hydrofluoric acid solutions containing 6.25, 13.5, and 25% of titanium in niobium were also prepared and the niobium extracted with isophorone, tributyl phosphate, and a mixture of 95% diethyl ketone with 5% primene-JMT.

These experiments showed that 67% of the niobium containing as much as 25 per cent titanium could be recovered with a purity

*Numbers indicate group leaders in charge of work. See Appendix III.

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time. The reduced transfer of thiocyanic acid with time is possibly due to polymerization of the acid.

In rechecking weight data on some separation extractions of hafnium and zirconium with tributyl phosphate, it was found that some weights were much greater than should be possible as the oxides. It was suspected that in the particular analytical procedure used, some phosphate was being formed instead of the assumed plain HfO_2 and ZrO_2 . A conversion of the residues to phosphate by H_3PO_4 and calcining gave consistent results which indicated the earlier method was at fault.

1.4 Separation of Magnesium from Calcium by Sublimation (2)

About two pounds of high purity calcium was prepared for use in studies of the physical properties of calcium metal. Production grade calcium containing 0.3 w/o magnesium was heated at 625°C for 8 hours in a vacuum of 10^{-3} mm of Hg to sublime off the magnesium. The residue was then vacuum distilled at 900°C and the product collected on an air cooled condenser. Chemical analysis of calcium thus prepared showed it to be metal of 99.95% purity. The reported analytical values of the principal impurities in this product were as follows: 0.01 w/o magnesium, 0.01 w/o nitrogen, 0.01 w/o iron, 0.001 w/o aluminum and 0.005 w/o manganese.

1.5 Separation of Mo, Zr, Nd, and Ru from U-5.18 w/o Cr Eutectic by Liquid Metal Extraction (6)

A series of experiments has been conducted in an attempt to remove Mo, Zr, Nd and Ru from U-5.18 w/o Cr eutectic by extraction with magnesium and some magnesium eutectics. The amount of Mo, Zr, Nd and Ru added as impurities to the U-Cr eutectic was approximately 728, 1130, 3080 and 650 ppm, respectively. These concentrations correspond roughly to the concentrations which would exist in a U-5.18 w/o Cr fuel after two per cent "burn-up" of the uranium.

The procedure employed and the apparatus used in carrying out these extractions are described in some detail in the preceding quarterly report. In general, the U-Cr eutectic and impurities along with an equal volume of extractant are enclosed in a tantalum crucible which in turn is enclosed inside a stainless steel jacket. This assembly is then heated in a rotating resistance furnace at 925 to 1000°C for approximately two to three hours. The furnace swings through an arc of 360 degrees in one minute, a toggle switch actuated by an arm fastened to the drive shaft to the furnace then reverses the direction of rotation. This rotation provides thorough mixing of the charge. After mixing for two to three hours at temperature the rotation of the furnace is stopped and the charge permitted to settle at temperature for one hour.

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The crucible is then removed and quenched in water. The extractant and U-Cr layers are then sectioned and analyzed.

Several procedures were employed in adding the Mo, Zr, Nd and Ru impurities to the charge. One procedure involved arc melting the impurities and the U-Cr eutectic together prior to extraction. A second procedure involved mixing the impurities with the U-Cr eutectic in a sealed tantalum crucible at 1000°C as described above prior to the addition of the extractant. A third procedure involved placing all the components including the extractant in a tantalum crucible and carrying out the mixing and extraction all in one step. All these procedures gave the same values, within experimental error, for the distribution coefficients of the impurities between the U-Cr eutectic and the extractant layer. The results obtained are given in Table I. The values for the distribution coefficients were calculated from results of chemical analyses except as otherwise noted. The results for the distribution coefficients for zirconium as determined from spectrographic analyses are somewhat higher than those determined from chemical analyses. Several extractions were made using radioactive molybdenum. The distribution coefficients for molybdenum as determined by radiochemical analyses are in general agreement with the values obtained by chemical analyses and show about the same amount of scatter. Attempts to obtain a material balance for the impurities added were unsuccessful. Apparently part of the impurities are lost to the tantalum crucible, or as oxide films or scale during the processing of the samples.

It may be concluded from the results obtained that the extractants used are effective in removing neodymium and ineffective in removing molybdenum or ruthenium. The distribution coefficient for neodymium was estimated from spectrographic results which showed in each case that less than 1000 ppm of neodymium remained in the uranium-chromium eutectic. None of the analytical methods tried for the determination of neodymium in the uranium-chromium eutectic were satisfactory. The chemical and spectrographic analyses for neodymium in the extractant layer were in good agreement and showed that most of the neodymium added had concentrated in the extractant layer. However, the material balance in most cases was rather poor. The distribution coefficients for zirconium showed a much greater scatter than the distribution coefficients obtained for the other elements. Although values as high as two were obtained, it is difficult to evaluate the significance of this distribution coefficient since the material balance was particularly poor in this case. Apparently part of the zirconium alloyed with the tantalum crucible or may have collected as a thin slag layer between the uranium-chromium eutectic and the extractant. The net result being that 25 to 50 per cent of the initial amount of the zirconium added to the fuel was removed.

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Table I

Distribution Coefficients for the Extraction of Zr, Ru, Mo, and Nd from U-5.18 w/o Cr Eutectic with Magnesium and Some Magnesium Eutectics

Grams of U-Cr Eutectic	Extractant and Grams Used	Distribution Coefficient $*K_D$			
		Zr	Ru	Mo	Nd
103.3	Mg 13.15	0.16 0.32**	0.03	0.05	20**
109.6	Mg 16.79	0.48 0.83**	0.01	0.01	20**
125.8	Mg 14.10	1.42**	0.08	0.01	20**
130.2	(Mg-16.3 w/o Ca) 13.45	1.17**	0.01	0.004	20**
90.8	(Mg-16.3 Ca) 7.22	2.20**	0.01	0.31	20**
79.0	(Mg-16.3 w/o Ca) 11.84	1.28**	0.001	0.000	20**
82.2	(Mg-16.3 w/o Ca)	0.89**	0.01	0.000	20**
69.6	(Mg-52 w/o Zn) 9.09	0.86	0.10	-----	20**
102.7	(Mg-36 w/o Sn) 13.37	0.30	0.02	0.15	20**
73.3	(Mg-66.8 w/o Pb) 9.56	0.49	0.22	-----	20**
63.2	(Mg-59.5 w/o Bi) 8.18	0.50	0.09	-----	20**
201.1	Mg 18.42	----	----	0.14***	---
411.7	(Mg-36.4 w/o Sn) 24.58	----	----	0.023***	---

Table I (Continued)

Grams of U-Cr Eutectic	Extractant and Grams Used	Distribution Coefficient *K _D			
		Zr	Ru	Mo	Nd
484.9	(Mg-15 w/o Al) 15.04	----	----	0.003***	---
411.7	(Mg-52 w/o Zn) 31.51	----	----	0.001***	---

$$*K_D = \frac{\frac{\text{grams of impurity in extractant layer}}{\text{grams of extractant}}}{\frac{\text{grams of impurity in U-Cr layer}}{\text{grams of U-Cr eutectic}}}$$

** Value of K_D as determined from spectrographic analysis.

***In these samples only radioactive molybdenum (800 ppm) was added to the U-Cr eutectic. The distribution coefficient was determined from radiochemical analyses using the relation

$$K_D = \frac{\text{counts/min./gram in extractant layer}}{\text{counts/min./gram in U-Cr layer}}$$

It was also interesting to note that magnesium eutectics containing Ca, Zn, Sn, Pb and Bi showed no evidence upon microscopic observations for interaction between these extractants and the U-Cr eutectic. Spectrographic analyses show no detectable amounts of Ca, Zn, Sn, Pb, and Bi in the U-Cr eutectic. Consequently, magnesium would very effectively separate any of these elements from the U-Cr eutectic. Little or no chromium is extracted by the magnesium eutectic.

2. Preparation of Pure Compounds

2.1 Preparation of Thorium Tetrachloride (5)

A mixture of thorium oxide and carbon was formed into pellets using a sucrose solution as a binder. Chlorination of this mixture

at 550°C resulted in 75 to 95 per cent conversion to thorium tetrachloride. One mole of carbon per mole of thorium oxide was used in this mixture. The heat evolved in this reaction is a serious problem and has limited the rate at which the reaction can be run.

Reaction of thorium oxalate with chlorine was investigated as a possible reaction for the preparation of thorium tetrachloride. Thorium oxalate initially seemed to be a desirable starting material since it is a convenient form in which to precipitate thorium from solution and it contains the proper amount of carbon to react with chlorine to form thorium tetrachloride and carbon dioxide. However, thorium oxalate decomposes below the temperature at which chlorination proceeds at an appreciable rate. Thorium oxalate heated under non-oxidizing conditions was found to decompose into a mixture containing 0.4 moles of carbon per mole of thorium oxide. This amount of carbon is not sufficient for the chlorination reaction. As a result of these observations, thorium oxalate alone does not appear to be a satisfactory starting material for the chlorination reaction.

The rate of sublimation of thorium tetrachloride at various temperatures was determined. The purity of the thorium tetrachloride purified by vacuum sublimation has been satisfactory except for the iron content. Most of the purified product has contained more than 500 ppm of iron. This was a higher iron content than that of the crude thorium tetrachloride. Thus at least some of the iron contamination appeared to be caused by corrosion of the sublimation retort. Treatment of the crude thorium tetrachloride with hydrogen at 550°C did not decrease the iron content of the sublimed product. Apparently the iron was already present as ferrous chloride, which was volatile at the temperatures necessary to sublime thorium tetrachloride. Replacing the stainless steel liner and baffle by a Hastellaoy liner and baffle reduced the corrosion and thorium tetrachloride was sublimed without an increase in the iron content.

2.2 Preparation of Yttrium Trifluoride (2)

Yttrium oxide was dissolved in 6N hydrochloric acid and the solution filtered in order to remove insoluble impurities. The solution was then concentrated by evaporation and crystals of the hydrated chloride were formed upon cooling. To these crystals 70% hydrofluoric acid was added, and the mixture stirred during the acid addition. After an excess of the acid was added (determined by the thorium nitrate tetrahydrate test), the mixture was allowed to cool. It was then filtered, and the precipitate of yttrium fluoride, which is believed to be a half hydrate, was washed with acetone, and dried at approximately 100°C in platinum trays. The fluoride was then dried in an anhydrous hydrogen fluoride atmosphere for eight hours at 300°C in order to remove the last amount of

water present. Since no analytical method of determining fluoride content in yttrium fluoride has been developed, it is assumed that complete conversion takes place. This is substantiated by weight changes. X-ray patterns of the fluoride have been taken but no positive identification was obtained since a reference card has not yet been established.

Yttrium oxide was placed in a platinum tray and anhydrous hydrogen fluoride passed over it at 600°C for eight hours in a stationary bed type furnace. Weight change of the salt indicates nearly complete conversion to yttrium fluoride. X-ray patterns of the fluoride prepared in this manner are identical to the patterns obtained from the fluoride prepared by the wet process.

3. Metal Preparation Studies

3.1 Preparation of Vanadium Metal (5)

The source of the oxygen contamination in the magnesium reduced vanadium has been difficult to find. A possible source is the presence of vanadyl trichloride in the vanadium tetrachloride. The boiling point of vanadyl trichloride is 127°C, which is close enough to the boiling point of vanadium tetrachloride so that distillation might not separate the two compounds. A reduction was carried out using vanadium tetrachloride which had been contaminated with water and then purified by distillation. The vanadium metal from this reduction contained 0.50 per cent oxygen. This high oxygen content indicates that vanadyl trichloride can not be removed by distillation and that its introduction into vanadium tetrachloride must be prevented if vanadium metal of low oxygen content is to be prepared.

3.2 Preparation of Tantalum and Niobium Metals (2)

Van Arkel (1) has reported preparing ductile niobium metal by the thermal decomposition of niobium pentachloride depositing it on a heated filament. Niobium pentachloride, prepared by the chlorination of niobium carbide, was passed over a niobium filament at approximately 1800°C. The entire system was under a dynamic vacuum. The most successful attempt to date has resulted in an increase in the diameter of the filament from 0.023 inches to 0.062 inches and an increase in weight from 0.77 grams to 2.38 grams. This weight increase represented approximately 40 per cent of the theoretical yield. The resulting deposit had a bright metallic lustre but was extremely brittle. Efforts are under way to improve the ductility of the final product and to increase the scale of the experiment.

(1) Van Arkel, A. E., Metallwirtschaft 13, pp. 405-8 (1934).

3.3 Preparation of Thorium Metal (3,5)

Preliminary experiments on the production of thorium by the hot wire method have led to considerable alteration of the apparatus. This project is active and will be continued.

Thorium metal is also being prepared by heating thorium tetrachloride and magnesium in tantalum and titanium crucibles. These metal crucibles have several advantages over magnesium oxide crucibles. They are non-porous and so do not absorb any thorium tetrachloride, can be reused several times, and do not contaminate the thorium with carbon or oxygen. Some of the thorium prepared in these crucibles has contained less than 100 ppm of carbon and had a hardness of 18RE.

3.4 Preparation of Yttrium Metal (2)

Yttrium metal was prepared by the reduction of anhydrous yttrium fluoride, with calcium in a bomb process. Zinc fluoride added to the charge supplies additional heat and forms a lower melting Y-20 w/o Zn alloy. Iodine was added to the charge as an additional booster forming calcium iodide which lowers the melting point of the slag and thus aids metal-slag separation. A sintered calcium fluoride liner was used in the 2 1/2 inch bomb. The bomb was heated slowly in a gas fired furnace with the reaction beginning at an outside bomb temperature of 575°C. Well shaped massive yttrium-zinc alloy biscuits were obtained in yields of 80 to 90 per cent.

Cooling curves run on the Y-Zn alloy revealed that the alloy melts at approximately 860°C. Removal of the zinc and calcium from the alloys by vacuum distillation has been investigated. Several refractories were tested as crucibles for containing the alloy during heating. These materials include graphite, magnesia, beryllia, CaF_2 and a mixture of MgO -16 w/o CaF_2 . Of these tested the CaF_2 crucible was found to be most suitable for the "dezincing" operation. There appears to be little, if any, interaction between the metal and the crucible up to a temperature of 1200°C. The yttrium sponge thus heated contains approximately 0.5 w/o of residual zinc and a similar amount of calcium.

Various oxide refractories have been tested as crucibles for melting yttrium metal. To date all the refractories tested have exhibited extensive crucible metal interactions or considerable crucible-penetration by the molten yttrium metal. Currently, arc-melting in an inert gas atmosphere appears to be the most satisfactory melting method. On small scale, the remaining zinc and calcium have been successfully removed by this procedure.

4. Alloy Studies

4.1 Uranium-Zinc System (6)

A critical review has been made of data obtained during this report period and all previous thermal, metallographic, and x-ray data obtained on the uranium-zinc system. The following features, including the mean values for the horizontal temperatures, are proposed for compositions above 28.8 w/o U on the basis of these data:

- (1) An intermetallic compound, UZn_9 , at 28.8 w/o U which either melts or decomposes into zinc vapor and liquid, at $1070 \pm 20^\circ\text{C}$ at one atmosphere pressure,
- (2) A eutectic between UZn_9 and essentially pure uranium at 42.4 w/o U with the eutectic horizontal at $944 \pm 5^\circ\text{C}$,
- (3) A monotectic at about 93 w/o U with the monotectic horizontal at $1050 \pm 20^\circ\text{C}$ extending from 47 w/o U to essentially pure uranium,
- (4) The uranium α to β and β to γ transformations occur at $663 \pm 10^\circ\text{C}$ and $772 \pm 10^\circ\text{C}$, respectively, and are apparently unaffected by alloying,
- (5) No significant solubility of zinc in solid uranium up to the melting point of uranium.

Attempts to check the liquidus and the phase regions for the 0 to 30 w/o U alloys from vapor pressure measurements employing a dew point method have continued. The apparatus and technique is still undergoing modification. Present tentative results indicate a large negative deviation from Raoult's Law for the vapor pressure of 10 w/o U and 20 w/o U alloys.

4.2 Thorium-Zirconium System (2)

Work was continued during this report period on the study of the thorium-zirconium phase diagram using high temperature x-ray techniques. A body-centered cubic phase has been identified in three alloys at 1000°C which is interpreted as evidence of solid solubility between the high temperature forms of thorium and zirconium at that temperature. The lattice constant for each of the three alloys was calculated and was as follows:

Th-25 w/o Zr	3.90 Å
Th-40 w/o Zr	3.79 Å
Th-60 w/o Zr	3.70 Å

4.3 Thorium-Hafnium System (2)

Additional information has been obtained on the thorium-hafnium constitutional diagram. Data were obtained on several alloys by differential cooling curves and melting point determinations.

Cooling curves were run on several alloys using a differential thermocouple with a zirconium metal "neutral body". A tungsten-niobium thermocouple was employed and temperatures were recorded on an X-X Brown Recorder. The data obtained are shown in the accompanying table. The solidus temperature was determined for these alloys using a melting bar method in which first signs of melting are observed by means of an optical pyrometer sighted on a small hole in the electrically heated specimen. The solidus data thus obtained are also listed in Table II.

Table II

Cooling Curve and Melting Data of Thorium-Hafnium Alloys

Composition w/o Hf	Cooling Curve Breaks °C	Observed Melting °C
5.2	no data	1650
7.7	1312	1595
9.9	no data	1540
22.1	1274	1450
39.4	1300	1435
47.8	1280	1440
65.4	1281	1465
73.2	1272	1635
75.2	no data	1760
77.6	1306	no data
94.7	no data	2010
100.	no data	2153

These data are in good agreement with those obtained by electrical resistivity vs temperature measurements, and indicate the possibility of a eutectoid horizontal at about 1280°C and a minimum in the solidus at about 1440°C. These have not as yet been confirmed by microscopic evidence, however.

4.4 Thorium-Carbon System (6)

Further study of the x-ray diffraction patterns of thorium-carbon alloys taken at high temperatures shows that the thorium phase for low carbon content breaks up into two face-centered cubic phases of slightly different lattice constants at temperatures above 1300°C. Alloys containing 1.6 to 2.2 w/o carbon show a similar splitting of the carbide diffraction peaks. Attempts are being made to fix the phase boundaries of the phases observed from the x-ray data obtained.

4.5 Uranium-Niobium System (3)

This section will be devoted mainly to a description of the present state of the investigation. Figure 1 is helpful for this purpose.

The solidus line is about as shown but a few additional melting determinations are yet to be made. Alloys of intermediate composition are inclined to be inhomogeneous; hence, melting points in this region frequently are uncertain.

The loop near the center is established with reasonable accuracy but the maximum may need to be raised slightly above its present position at 950°C. The position of the curved boundary of the loop has been established from x-ray patterns of alloys quenched from a series of temperatures below 950°C. Each of the patterns had a double set of lines corresponding to the two phases formed from the decomposition of the high-temperature gamma solid solution. The corresponding compositions could be determined from the parameter-composition curve (Fig. 2) of alloys quenched from the gamma region. Dilatometric measurements furnished the data for plotting the horizontal line at 645°C.

Aside from the boundary that separates the beta-plus-gamma from the gamma field, the arrangement of lines at the extreme left is uncertain. Addition of niobium to uranium has little effect on the temperature of the alpha-beta change. Hence, distinction between the possible eutectoid and peritectoid arrangements is not easy.

4.6 Zirconium-Tantalum System (3)

As a result of new melting point determinations, the zirconium end of the eutectic horizontal at 1830°C is now estimated to lie

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Fig. 1 - Preliminary constitutional diagram of uranium-niobium alloy system.

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Fig. 2 - Lattice parameter-composition curve of uranium-niobium alloys.

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between 1 and 2 per cent tantalum. The end of this horizontal must also be a point on the boundary line of the beta-zirconium-rich solid solution area. As its lower end, this boundary line must terminate at the eutectoid point which lies at 800°C and at more than 3 per cent tantalum. Accordingly, the boundary must have a negative slope, a situation that is unusual but not unknown.

The terminus of the eutectoid horizontal on the tantalum side of the diagram lies between 90 and 98 per cent tantalum. At present the evidence does not agree as to whether the end lies below or above 95 per cent. The resistance-temperature curve of a 95 per cent tantalum alloy showed no anomaly when heated to 1000°C. On the other hand, a polished and etched section of a slowly cooled 95 per cent alloy showed evidence of two phases. Furthermore, x-ray data indicate that the eutectoid line terminates between 90 and 95 per cent tantalum.

Attempts to use resistance-temperature measurements to locate points on the boundary of the tantalum-rich solid solution have been abandoned. Curves for the 60, 70 and 75 per cent tantalum alloys all showed irregularities in the vicinity of 1500° to 1700°C but the variability of results was unreasonably bad.

4.7 Zirconium-Zinc System (6)

Due to the high vapor pressure of zinc at the melting point of the Zr-Zn alloys progress on this system has been slow. The general outline of the system is known but further work is necessary to establish the phase regions. Vapor pressure measurements, employing a dew point method, have thus far given incoherent results. As indicated in an above section, modifications and improvements in the vapor pressure apparatus are being made.

4.8 Tantalum-Vanadium System (2)

A series of tantalum-vanadium alloy specimens has been annealed at 900°C, 1000°C, and 1100°C for 423 hours in evacuated quartz tubes. A plot of composition vs Knoop hardness values of the annealed specimens shows a maximum hardness at approximately 65 w/o Ta. This maximum coincides with the composition of the brittle intermetallic phase, believed to be sigma, as estimated from microstructures and x-ray evidence. These studies called attention to the need for a higher purity vanadium metal than that obtained by the usual reduction of V₂O₅ with calcium. Commercial V₂O₅ was purified by dissolving in H₂SO₄ solution and then reprecipitating it as ammonium metavanadate. This material was thermally decomposed to V₂O₅ and reduced to metal. The metal obtained was quite hard (Rockwell 55A) and contained a large amount of inclusions or impurities as revealed by microscopic inspection. Attempts to purify further this vanadium by heating in a ThO₂ crucible and

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cooling in a temperature gradient resulted in slight improvement of the microscopic purity.

Attempts to isolate a single crystal of "sigma phase" so far have been unsuccessful. Annealed alloys containing the intermetallic phase were crushed in a diamond mortar and the fragments were examined for possible single crystals, but without success. Another method tried was the dissolution of the alloy matrix with the possible isolation of compound crystals. At present no chemical has been found that will dissolve the matrix and not the intermediate phase. Another method which is being tried is to melt some uranium-5 w/o eutectic alloy in a tantalum crucible. Tantalum is slightly soluble in uranium below 1200°C, and may react with the vanadium present forming dendrites of the vanadium-tantalum compound. The first attempt failed to produce significant results.

4.9 Niobium-Zinc System (2)

The possible use of a niobium-zinc alloy as an intermediate step in the preparation of niobium metal has been contemplated. A series of experiments have been run to determine the solubility of niobium in molten zinc in the temperature range of 600 to 825°C. Zinc metal is heated in a niobium crucible sealed inside an evacuated vycor tube. Chemical analyses of drillings taken from the zinc-rich region after cooling show that the solubility of Nb increases up to about one-half per cent at 700°C. Erratic data at higher temperatures for this portion of the liquidus leaves some doubt as to the degree of solubility above 700°C.

5. Metal Coatings and Corrosion

5.1 Corrosion of Refractory Metals by Liquid Metals (5)

The corrosion of niobium, molybdenum, zirconium, and thorium by the eutectics of uranium with iron, with manganese, and with nickel at 800°C was studied. Crucibles, made from thin sheets of the metal to be tested, were filled with the eutectic alloy, sealed in stainless steel outer crucibles and held in a muffle furnace at 800°C for definite periods of time. They were then opened, sectioned and examined. All four metals were excessively attacked by the three eutectics within seven days.

The corrosion of tantalum and other refractory metals at 500°C by the aluminum-magnesium eutectic alloy containing 66 weight per cent magnesium was studied using the method described above. Low carbon steel, type 430 and type 302 stainless steel, and Inconel were excessively attacked by the alloy. Results of tests on tantalum, niobium and molybdenum are given in Table III.

The corrosion of tantalum by silver and calcium at 1200°C was studied using the same experimental method. Results of these tests are given in Table IV.

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Table III

Corrosion of Refractory Metals by Aluminum-Magnesium Eutectic at 500°C

Time	Remaining Wall Thickness (in mils)		
	Ta	Nb	Mo
0 days	5.8	5.4	3.5
7 days		5.5	3.2
14 days	5.3		
28 days	4.9	5.3	3.2
56 days	5.1	4.9	
95 days	5.2	5.2	

Table IV

Corrosion of Tantalum by Silver and Calcium at 1200°C

Time	Wall Thickness (in mils)	
	Silver	Calcium
0 days	5.8	5.8
2 days	5.2	5.2
7 days	5.2	
12 days	5.4	5.3
35 days	5.0	

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5.2 Corrosion of Coated Metals by Liquid Materials (3)

Tests of 1000 hours or more of the ability of carbide-coated tantalum crucibles to contain molten uranium-chromium eutectic at 1100°C were planned but have not succeeded because of power and apparatus failures. In one instance the test had run for 864 hours and in another for 912 hours. The crucibles showed more resistance to the oxidizing conditions that prevailed after the failure of the equipment than had been anticipated.

A test run at 1100°C under approximately atmospheric pressure of CS₂ was stopped after 360 hours. The crucible was found to be extremely brittle. Some deposits on the container walls, were presumed to be carbon and sulfur and were undoubtedly the result of a gradual decomposition of the CS₂. A decrease in pressure within the container agreed with this hypothesis.

Thin carbide coatings were put on zirconium and titanium crucibles. Both crucibles were penetrated by the uranium-chromium eutectic during a test lasting 144 hours at 1100°C.

Apparatus for producing silicide coatings on tantalum and other metals was completed. A heavy coating was formed on the outside of a tantalum crucible but the inside remained bare. Alterations in procedure are being considered. The tantalum silicide layer appears to be relatively ductile. Formation of a silicide coating on a titanium crucible was undertaken but the crucible was found to be brittle; microscopic examination indicated that the silicon had diffused completely through the wall. If this view is correct, silicon must diffuse into titanium very rapidly.

5.3 Corrosion of Carbides by Molten Uranium (2)

Continuing the investigation of the resistance of carbide refractories to corrosion by liquid uranium, crucibles of WC, TiC and 4TaC-ZrC were charged with uranium metal and heated in vacuo to 1200°C for 2 hours. All three of the materials tested showed excessive penetration of the crucible by the molten uranium, although in the case of 4TaC-ZrC, penetration appeared to result from porosity rather than interaction.

A ZrC crucible which had exhibited good corrosion resistance at 1200°C was tested at 1300°C for 3 hours. Complete alloying or interaction was observed between uranium and the zirconium carbide crucible in the 1300°C test. An attempt to deposit a coating of ZrC on a 10 mil tantalum sheet by means of a shellac-zirconium powder-carbon mixture proved unsuccessful.

5.4 Hot-Press Bonding of Aluminum to Other Metals (6)

A number of hot-press bonded cylindrical tensile test specimens of Al/Zn couples (prepared by pressing at 380°C at approximately 30,000 psi for 10 to 15 minutes) showed a maximum ultimate tensile strength of 7900 psi which is in the range of the ultimate tensile strength of pure annealed zinc. The tensile strength of five Al/Cu-9.7 w/o Mg couples (prepared by pressing at 480°C and 16,000 to 30,000 psi for 10 minutes) ranged from 4600 to 7500 psi. All except one of these specimens failed at the bond area. One specimen failed at 7450 psi in the Cu-Mg eutectic.

5.5 Corrosion of U-Zr-Nb Alloys (2)

The present investigation of the alloys of uranium-niobium-zirconium has been terminated during this report period. Electrical resistivity measurements were made on three ternary alloys in both the as-cast and gamma-quenched condition. The resistivities are much greater in the quenched condition. The effect of aging temperatures of 425°, 400°, 375° and 325°C upon the corrosion resistance of the quenched alloys has also been determined. One alloy, containing 5 w/o Nb-1 w/o Zr-U, withstood 1760 hours of testing in 325°C water. These data are reported in detail in ISC-609.

5.6 Casting Uranium into Zirconium Cans (2)

Zirconium cans have been filled with uranium by direct casting and end closures have been successfully arc welded on them. Fourteen closed cans were shown to withstand 120 hours in an autoclave at 178°C. Attempts at eliminating the weld lip by spinning it over after welding appear promising. Studies are also under way on the effect of the surface preparation of zirconium cans before casting upon the strength of the bond formed. These data have been reported in detail in ISC-609.

5.7 Hot-Press Canning of Zinc-Coated Uranium Slugs (6)

Attempts were made to hot-press can zinc-coated four-inch Hanford fuel elements in a specially constructed three piece die. Complete bonding between the aluminum can and the zinc-coated slug was not obtained after pressing for 15 minutes at 365°C and at pressures as high as 22,500 psi. Likewise only partial bonding was obtained between the aluminum cap and can. The use of higher temperatures resulted in the formation of aluminum-zinc eutectic and penetration of the can wall. More careful preparation of the surface of the components might result in successful bonding at 365°C.

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Some slugs are being coated with Zn-5 w/o Al eutectic in the hope that such a coat will permit the use of temperature above 365°C without excessive penetration of the can wall. Thinner coats of the Zn-5 w/o Al eutectic 0.5 to 1 mil in thickness as compared to 1 to 2 mil coats of zinc, can be applied to uranium slugs by the hot-dip procedure. Rates of interpenetration between the Zn-5 w/o Al eutectic and uranium, as well as between the eutectic and aluminum at various temperatures are being measured in order to evaluate the feasibility of using such a coat in the hot-press canning procedure.

6. Solid State Investigations

6.1 Physical Constants of Metals from Ultrasonic Measurements (4)

Measurement of the elastic constants of zinc as a function of temperature is in progress. The results obtained during this period are shown in Tables V, VI.

In both cases plots of the data show that within experimental error the temperature dependence is linear over the range measured. Extrapolation to 0°K gives an intercept of 6.87×10^{11} dyne/cm² for C₃₃ and 4.92×10^{11} dyne/cm² for C₄₄. Plans have been made for an apparatus to make measurements at liquid hydrogen temperatures to see if the linear extrapolation is justified.

6.2 Thermal-Cycling of Metals through their Transformation Temperatures (6)

It has been pointed out by many authors that under pile irradiation, high energy particles and particularly fission processes produce localized thermal pulses (thermal spikes) which may exceed the melting point of the metal. Metals which exist in more than one crystalline form may be consequently cycled through their transformation temperature in the region of the thermal spike. The opinion has also been expressed that the operation of fuel elements at high temperatures might result in improved dimensional stability since annealing effects would tend to limit the effects of radiation damage. It was therefore considered worthwhile to observe what happens to uranium and some of its alloys, as well as some other metals as they are cycled through their transformation temperatures, and as they are cycled over temperature intervals both above and below their transformation temperatures.

Cast rods of uranium, uranium-chromium eutectic and uranium containing 650 ppm Ru, 728 ppm Mo, 3080 ppm Nd and 1129 ppm Zr were thermal cycled between 500 to 800°C. The latter composition corresponds roughly to the concentration of fission products after two per cent burn-up of the fuel element.

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Table V
Temperature Dependence of C_{33}

Temperature °K	C_{33} (10^{11} dyne/cm ²)
79	6.73
81	6.71
87	6.77
99	6.71
102	6.65
108	6.61
115	6.52
121	6.66
124	6.59
128	6.51
137	6.44
144	6.66
148	6.45
151	6.45
161	6.42
170	6.46
174	6.46
182	6.54
205	6.40
216	6.31
221	6.33
231	6.16
234	6.47
245	6.34
259	6.34
260	6.25
260	6.19
271	6.27
282	6.26
292	6.29
297	6.32

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Table VI
Temperature Dependence of C_{44}

Temperature °K	C_{44} (10^{11} dyne/cm ²)
86	4.40
92	4.70
93	4.61
98	4.67
117	4.48
160	4.34
201	4.25
212	4.16
215	4.38
216	4.13
218	4.16
243	3.91
255	3.96
256	4.02
269	3.97
277	4.01
293	3.92
298	3.95
299	3.96
299	3.84

The samples cycled were in rod form, 1/4 inch in diameter and four inches long. The rods were clamped between water-cooled copper electrodes, one end being free to move over a sliding contact. The bars were heated from 500 to 800°C in 3 to 5 seconds by suddenly increasing the electric current passing through them. When the temperature reached 800°C, the current was automatically reduced to the initial value. The cooling cycle required approximately one minute. Two bars of each compositions were cycled in this manner. After 450 cycles, all the bars except one showed considerable distortion and surface roughening with pits and small open fissures on the surface. One bar containing simulated fission products showed little distortion and the surface

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remained relatively smooth. Why this sample behaved differently is not known. A second bar of the same composition showed typical distortion and surface roughening as shown in Figure 3. The cooling cycle for this specimen was 90 seconds, approximately 30 seconds longer than the cooling cycle for the other specimens tested.

Some preliminary testing of thorium shows that it also distorts on cycling through its 1400°C transformation. However, in the case of thorium, no appreciable distortion was observed on cycling below the transformation temperature.

These results show that thermal cycling uranium through its transformations results in considerable distortion even though the minimum temperature is maintained at 500°C . Distortion that occurs on cycling thorium through its transformation at 1400°C indicates that such distortion is not limited to non-cubic metals.

6.3 Structure of Zr-Ge Compounds (2)

Previous work on this system has been reviewed and assembled for publication. The x-ray powder diffraction data on the supposed compound, Zr_2Ge , was re-examined to see if it could be better explained by the formula, Zr_5Ge_3 with a structure isomorphous with that reported for Zr_5Sn_3 , Ti_5Sn_3 , Ti_5Ge_3 , and Ti_5Si_3 .

An x-ray pattern from the 31.2 weight per cent Ge alloy was indexed satisfactorily, as hexagonal with $a = 7.99 \text{ \AA}$ and $c = 5.54 \text{ \AA}$ being very similar to the diffraction pattern of Zr_5Sn_3 . The formula Zr_5Ge_3 with 2 molecules/unit cell is in agreement with atomic volume and density considerations. The x-ray density of 7.29 g/cc calculated from atomic weights and cell dimensions compares favorably with the measured density of 7.12 g/cc of the 31.2 a/o alloy. The volume of the unit cell, $306 \text{ \AA}^3$, is only slightly smaller than the calculated cell volume of $326 \text{ \AA}^3$, based on atom volumes in the respective metal lattices. Microscopic evidence in this region of the diagram also indicates a one-phase region close to the theoretical composition for Zr_5Ge_3 at 32.2 w/o Ge as well as another at about 44 w/o Ge. The intermediate phase occurring at the latter region is proposed to have the formula ZnGe having the theoretical composition 44.2 w/o Ge.

7. Other Investigations

7.1 Reclamation of Zirconium Chips (2)

In order to determine what effect pickling of zirconium and zircaloy scrap in 33% hot sulfuric acid has on the corrosion resistance of the final metal, triplicate samples were prepared from both arc-melted and graphite-melted zirconium and zircaloy #2 scrap.

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Figure 3 - Photograph Showing Distortion of a Uranium Alloy Bar Containing 650 ppm Ru, 728 ppm Mo, 3080 ppm Nd and 1129 ppm Zr, after 450 Cycles between 500 and 800°C, Heating Time 4 Sec., Cooling Time 90 Sec.

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These were corrosion tested in water-filled stainless steel bombs at a temperature of 300°C. Specimens approximately one centimeter square and two millimeters thick were used in this test. The data secured is tabulated in Table VII and represent average rates for the three samples of each designated treatment. A very marked

Table VII

Corrosion of H₂SO₄ Treated Zirconium and Zircaloy in 300°C Water

Specimen	Rate after 12 1/2 Days mg/dm ² /day	Rate after 56 Days mg/dm ² /day
1. Original Stock Crystal Bar Zr	+0.57	+29.4
2. Arc Melted Specimens Prepared from Treated Scrap:		
a. Zirconium Chips	+0.68	+38.7
b. Zirconium Turnings	+0.80	+37.2
c. Zircaloy #2 Chips	+0.33	+0.45
d. Zircaloy #2 Turnings	0.00	+0.43
3. Graphite Melted Samples of Treated Scrap		
a. Zirconium Chips	+2.87	-136.3
b. Zirconium Turnings	+9.81	- 21.7
c. Zircaloy #2 Chips	+0.58	+ 0.80
d. Zircaloy #2 Turnings	+0.52	+ 0.63

increase in corrosion rates of the zirconium samples took place between the 12 1/2 and 56 day tests. Since a corresponding increase was also observed for pieces of the original stock crystal bar zirconium that accompanied the chips, there is some question remaining as to the interpretation of these data. The zircaloy that was recovered showed good resistance to corrosion.

A recovery of zirconium and zircaloy scrap was attempted forming a Zr-Zn alloy by bomb reducing a mixture of zirconium and zinc fluoride with calcium in the presence of considerable quantities of zirconium scrap. The zinc fluoride acted as a thermal booster for the reaction and also providing the zinc for a Zr-Zn alloy. This was subsequently dezincing and the resulting sponge was arc-melted.

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Samples of metal treated in this manner were corrosion tested for a period of 14 days at 300°C. The data secured are given in Table VIII. It is readily apparent from the data presented in the

Table VIII

Corrosion of Zirconium Scrap Treated with Zinc Rate in mg/dm²/day

Metal	Bomb C	Bomb L	Bomb G	Average
Zirconium (untreated)	+ 1.496	+ 1.242	+ 2.735	+ 1.824
Zircaloy #2	-3090.	- 80.0	-1990.*	
Zircaloy #2	- 107.	- 205.0	- 101.0	-138.0
Zirconium	-1775.0*	-1510.0*	- 492.0	

*Samples completely gone.

table that this reclaiming treatment definitely reduces the corrosion resistance for the scrap.

7.2 Vacuum Fusion Analysis of Metals (5)

Lanthanum samples, containing 1000 ppm of nitrogen, were analyzed using an iron bath and a temperature of 1850°C. The recovery of nitrogen was incomplete and the evolution of gas during the analysis was slow. An iron bath is apparently unsatisfactory for use in the vacuum fusion analysis of lanthanum.

A survey of properties of metallic elements, which are readily available in a pure form, indicated that nickel might be a useful vacuum fusion bath material. The volatility of the pure metal is slightly less than that of iron. The solubility of carbon in liquid nickel is sufficient to supply carbon for the reaction with oxides and nickel oxide is readily reduced by carbon. The rate of gas evolution from a nickel bath was measured at temperatures up to 1800°C. The blank from the nickel bath was as low as that from an iron bath at the corresponding temperatures. The rate of vaporization of the nickel bath was slightly greater than that of an iron bath. Several samples of an Armco iron bar which had been analyzed previously were analyzed using the nickel bath. The results using the nickel bath agreed with the previous results within the precision of this analytical method. The use of a nickel bath in vacuum fusion analysis of rare earth metals will be investigated.

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Vanadium metal samples were analyzed for oxygen, nitrogen, and hydrogen. An iron bath was used at an operating temperature of 1550°C. The precision of the results was very good and the method seemed to be quite accurate.

7.3 Low Melting Fuel Element Studies (1)

More quantitative data on the solubility of uranium and thorium in aluminum-magnesium alloys are being obtained. Two cylindrical uranium slugs were drilled along their long axes. Into the cavities were placed portions of aluminum-magnesium eutectics. The uranium slugs were sealed in stainless steel and heated to 500°C. After four days at this temperature the alloys were cooled and sampled. Analysis indicates about 55 w/o uranium in the 68 w/o magnesium-aluminum eutectic and about 65 w/o uranium in the 33 w/o magnesium-aluminum eutectic. However, the melting points were raised in both cases.

7.4 High Temperature Furnace for Growing Single Crystals (2)

The furnace described in the preceding quarterly report was used in several crystal growth attempts, with high purity (C.P. grade) nickel shot as a starting material. The first run produced a sample consisting of several large crystals, while the sample from the second run was apparently a single crystal. The size of this crystal was about 1.5 inches long with a diameter of about 0.6 inches.

8. Rare Earth Metallurgy

This material reported in ISC-606.

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APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-535 R. D. Cooper and D. J. Zaffarano. Photodisintegration of Light in Nuclear Emulsions. UNCLASSIFIED.
- ISC-541 J. S. Wiley and M. Smutz. Separation of Thorium and Radium-228 by Solvent Extraction. SECRET.
- ISC-565 J. D. Corbett and S. von Winbush. The Solubility of some Metals in their Molten Halides. UNCLASSIFIED.
- ISC-566 J. A. Happe and D. S. Martin, Jr. Some Isotopic Exchange Reactions of Manganese. UNCLASSIFIED.
- ISC-567 R. F. Mitchell and D. S. Martin, Jr. High Specific Radio-activities of Osmium from Photonuclear Reactions. UNCLASSIFIED.
- ISC-568 D. E. Griffith and M. Smutz. Purification of Uranium by Solvent Extraction. SECRET.
- ISC-571 J. S. Fritz and M. Johnson. Volumetric Determination of Zirconium. UNCLASSIFIED.
- ISC-572 B. A. Rogers, L. Ried, Jr. and P. E. Palmer. The Rachinger Conversion Rule. UNCLASSIFIED.
- ISC-573 Ames Laboratory Staff. Engineering. Quarterly Summary Research Report. October, November, and December, 1954. SECRET.
- ISC-578 W. A. Rhinehart, D. J. Zaffarano and R. C. Prior. An Accurate Digital Count-to-Analog Voltage Converter Employing a Reliable Decade Scaler. UNCLASSIFIED.
- ISC-581 J. D. Corbett and R. K. McMullan. The Lower Oxidation States of Gallium. I. The $\text{GaI}_3\text{-GaI}_2\text{-GaI}$ System. UNCLASSIFIED.
- ISC-583 L. S. Ring, Jr. The Photodeuteron/Photoproton Yield from Sulfur. UNCLASSIFIED.
- ISC-529 Ames Laboratory Staff. Engineering. Quarterly Summary Research Report. July, August, September, 1954. SECRET.
- ISC-530 Ames Laboratory Staff. Chemistry. Quarterly Summary Research Report. July, August, September, 1954. CONFIDENTIAL.

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- ISC-506 Ames Laboratory Staff. Metallurgy. Quarterly Summary Research Report. April, May, June, 1954. SECRET.
- ISC-532 Compiled by P. Chiotti and O. N. Carlson. Hanford Slug Program. Quarterly Summary Research Report. July, August, September, 1954. SECRET. *del*
- ISC-533 Ames Laboratory Staff. Physics. Quarterly Summary Research Report. July, August, September, 1954. UNCLASSIFIED.
- ISC-549 F. H. Spedding, J. E. Powell and D. M. Provow. Determination of the Equilibrium Constant for the Exchange of Nitrogen Isotopes Between Ammonium Hydroxide Solution and Ammonium Ion Adsorbed on Dowex-50. SECRET.
- ISC-553 G. H. Beyer and R. B. Edwards. Flooding Characteristics of a Pulse Extraction Column. UNCLASSIFIED.
- ISC-558 C. L. Hammer and A. J. Bureau. A Method for the Prompt Destruction of the Electron Beam in a Conventional Synchrotron. UNCLASSIFIED.
- ISC-559 H. J. Hettel and V. A. Fassel. Determination of Fractional Part Per Million Amounts of Certain Rare Earths in Zirconium Metal. UNCLASSIFIED.
- ISC-561 C. L. Hammer and A. J. Bureau. A Method for the Prompt Destruction of the Electron Beam in a Conventional Synchrotron. II. UNCLASSIFIED.

2. Papers Published in Scientific Journals

- Goodman, L. and H. Shull.
Molecular Calculations. III. A Modification of the Naive Semi-empirical M O Method. J. Chem. Phys. 23, 33-43, (January, 1955)
- Johansson, S. A. E.
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Margoshes, M. and V. A. Fassel
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97, 434-443, (January, 1955).

Pohm, A. V., R. C. Waddell, J. P. Powers and E. N. Jensen
The Beta Spectrum of Cl⁴. Phys. Rev. 97, 432-433,
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Vander Haar, R., C. V. Banks and R. Vander Wal
Magnetic Studies of Nickel(II) and Palladium(II) Complexes
with Some of Its Alloys. J. Am. Chem. Sci. 77, 324-325.
(January, 1955)

Wilder, D. R. and E. S. Fitzsimmons
Further Study of Sintering Phenomena. J. Am. Cerm. Soc.
38, 66-71, (February, 1955).

Williams, J. T.
The Vanadium-Zirconium Alloy System. J. of Metals. 7,
345-350, (February, 1955).

APPENDIX II: LIST OF SHIPMENTS

<u>Destination</u>	<u>Item</u>
Dow Chemical Company Midland, Michigan	229 g uranium alloys
Astrophysical Lab, Vatican Observ. Attn: The Rev. Joseph Junkes S. J. Castel Gondolfo, Rome, Italy	2 g terbium oxide

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APPENDIX III

- (1) H. A. Wilhelm
- (2) O. N. Carlson
- (3) B. A. Rogers
- (4) J. F. Smith
- (5) D. Peterson
- (6) P. Chiotti

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