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AEC RESEARCH AND DEVELOPMENT REPORT

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PROGRESS REPORT FOR MONTH OF JANUARY, 1945

February 8, 1945

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Progress Report
Month of January, 1945

CONTRACT NO. W 7405 ENG-175

SUMMARY
(Morris Cohen)

Production of large magnesia crucibles is going smoothly, and similar methods will be applied to the smaller sizes as soon as dies are available. The orders for high density magnesia crucibles have been filled, and efforts are being directed toward the attainment of high densities in beryllia and thoria.

The expansion of sulphide facilities is making good progress. In the nitride field, higher firing temperatures and higher molding pressures are being tried as a means of reducing porosity.

A apparatus has been constructed for the creep testing of X-metal wires at temperatures up to 500°C in high vacuum. Detailed heat treatment, X-ray and microscopic studies are being applied to a series of high-purity, well-homogenized X-Zr alloys in order to establish the phase relationships. X-Pd alloys are not attractive from a corrosion standpoint. *what for?*

In specially prepared carbon-free alloys, the epsilon phase of the X-Si system forms at 940°C as a result of a peritectoid reaction between gamma X and X_5Si_3 . Carbon seems to have a material effect on the formation of epsilon. The corrosion rate of epsilon alloys in boiling distilled water approaches zero after several hundred hours of exposure whether the water is saturated with hydrogen, air or oxygen. Epsilon has a tensile strength of 40-50,000 psi with an elongation at fracture of .2%.

Extruded beryllium tubing has a ductility of .17% elongation in the longitudinal direction compared to .36% in the circumferential direction. The relative merits of graphite and water-cooled steel molds for beryllium casting are discussed. Apparatus is being assembled for the distillation of beryllium in order to obtain oxygen-free metal.

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Progress Report
Month of January, 1945

Division T-1

A. ABSTRACT - F.H. Norton

Production of the large magnesia crucibles is under good control and as soon as dies are received other sizes will go into production with the automatic tamping machine. The production of small non-porous magnesia crucibles has gone on rapidly to fill all current orders and thoria and beryllia crucibles are now being made up. The production of sulfides is expanding due to our new laboratory for the production of high sulfides and the new furnaces for reduction and firing crucibles. Research on nitride crucibles is continuing especially along the lines of higher pressures in molding and higher firing temperatures. Some mixtures with the nitrides have given fairly promising results.

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Progress Report
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Division T-1

B. PRODUCTION OF CRUCIBLES - T. T. Magel

During the past month the production group has been making A-6 crucibles fired in the catenary kiln in order to have a backlog for future shipments. The production of A crucibles is now well systemized and they are being made by machine tamping with reasonably good yields.

Experiments have been carried out on the production of R type crucibles and a number of satisfactory ones have been produced, although it is much more difficult to produce a good bottom on this size of crucibles and more research must be undertaken.

Molds are being made up for the S type crucible and as soon as these come in work will be started on the production of this item.

A number of small orders of cast beryllia ware have been made up during the month for other parts of the project.

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Progress Report
Month of January, 1945

Division T-1

C. SULFIDE PRODUCTION - A. L. Johnson

Production of High Sulfide ($CeS_{1.5}$)

The production of cerium sulfide (Ce_2S_3) of sufficiently low carbon contents has been successfully brought about during this month and all orders met. This was realized by following the plan outlined previously, i.e., using CS_2 and H_2 on the first run and H_2S for re-runs. This method has yielded a product analyzing between $CeS_{1.50}$ - $CeS_{1.53}$ and under 0.15% carbon in two runs, and increased production. The problem of high carbon is still under investigation however, and several theories are in the process of being explored. The results on this work should be available within a few weeks. It is hoped that Ce_2S_3 of the proper composition will be made available in one treatment as a result of these investigations.

The enlargement of the facilities for the production of high sulfide is almost completed. Units will be put into operation as quickly as preliminary runs indicate that production is satisfactory.

Production of Cerium Hydride (CeH_2)

This phase of the work is proceeding at a satisfactory rate. Plans are being developed to build a new unit for production of the hydride.

Production of Low Cerium Sulfide (CeS)

The production of CeS has been proceeding very satisfactorily. The following table gives results on the last five ingots.

Table C-1

Reduction no.	Temp $^{\circ}C$	mm Hg* Pressure	Ingot analyses %S	Oxid. no.	Comp.	Oxid. no. on Powdered sample
R-27	2230	9×10^{-5}	19.4	2.10	$CeS_{1.050}$	0.00
R-30	2000	3×10^{-4}	18.7	2.03	$CeS_{1.075}$	0.010 2.11
R-31	2030	8×10^{-4}	18.7	2.02	$CeS_{1.005}$	0.005 2.08
R-33	2000	5×10^{-4}	18.7	2.03	$CeS_{1.008}$	0.007 2.05
R-34	2035	1×10^{-3}	18.7	2.01	$CeS_{1.005}$	0.00 2.06
R-35	2035	5×10^{-4}	18.7	2.04	$CeS_{1.005}$	0.015 2.07

*Pressures measured on ionization gauge calibrated for air.

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Division T-I

C. SULFIDE PRODUCTION (Cont)

This table indicates the range in composition of the cerium sulfide being produced. Reduction no. R-27 was included because of the higher temperature at which it was prepared. The oxidation number of 2.10 should not be misconstrued in this case. Because of the higher temperature of preparation, the loss of cerium was greater than allowed for consequently the S/Ce ratio in the product is greater than unity and would manifest itself in a higher oxidation number. The purity of the ingot with respect to oxygen is good, there being no oxygen in the sample.

However, the reductions made at the usual operating temperature of 2000°C show uniform results of sufficient purity. The principle objection to the preparations is that the ball milled samples are inclined to be pyrophoric. At present, the pyrophoric nature is evaluated in the following manner in order to explain the occasional burning of the powdered material. The grinding operation is accompanied by a pick-up of oxygen as evidenced by the increase in oxidation number. It is assumed that the S/Ce ratio remains appreciably constant during ball milling and any increase in the oxidation number could be attributed to an oxygen pick-up. For example, Lot S used for pressing crucibles which consisted of Reductions R-25, R-26, R-29 and R-30 combined in one sample showed that each sample after ball milling had analyses respectively as shown in Table C-II.

Table C-II

<u>Lot S</u>		<u>Lot T</u>	
<u>Reduction No.</u>	<u>Composition*</u>	<u>Reduction No.</u>	<u>Composition*</u>
R-25	CeS _{1.007} 0.053	R-13	CeS _{0.965} 0.055
R-26	CeS _{1.015} 0.040	R-31	CeS _{1.005} 0.035
R-29	CeS _{1.010} 0.035	R-34	CeS _{1.005} 0.025
Average - Lot S: CeS _{1.012} 0.045		Lot T: CeS _{0.92} 0.038	

*On ball milled sample

The material from this lot was used in a manner which could not be employed for Lot T. The dies were loaded in air using Lot S but in the case of Lot T the old method of using a dry box had to be resorted to. Good crucibles were made from Lot S even though certain precautions usually taken were relaxed. It appears then, that in the ball milling operation oxygen pick-up is possible and probably the particles of brass are slightly oxidized on the surface which offers a protective coating against further oxidation if some precautions are exercised. Probably the reductions incorporated into Lot S were protected by some such film as the values of the oxygen content would indicate. Whereas the batches

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C. SULFIDE PRODUCTION (Cont)

which made up Lot T were not oxidized to the same extent (see Table C-II). The difference in oxygen pick-up was attributed to a faulty gasket on the ball mill.

A portion of ball milled powder obtained from R-27, the high fired ingot, was exposed to the air together with a sample of R-29 which was fired to the usual temperature of 2025°C. Neither sample burned although each showed a slight oxygen pick-up. The point is, however, that the ball milled sample of R-29 showed 0.035 mols of oxygen per mol of cerium (See Table C-III) and this amount may be sufficient to prevent further oxidation upon exposure. The sample of R-27 (high fired) which had 0.00 mols of oxygen picked up 0.01 mols on exposure but in any event no burning took place and in the exposed condition the material was quite satisfactory when compared with the oxygen content of the usual run of preparations (see Table C-I).

Definite conclusions are not possible at this time and higher temperatures for reductions will be aimed at in the future to test the validity of these remarks.

TABLE C-III

	<u>Ball milled sample</u>		<u>Exposed sample</u>	
R-27 (high fired)	CeS	1.05 0.00	CeS	1.05 0.010
R-29 (normal)	CeS	1.01 0.035	CeS	1.01 0.035

Fabrication of Crucibles

In spite of the tedious procedure required for the pressing of crucibles, production is slowly increasing so that outstanding orders can be readily filled. At present dies are available for L-5, A-20⁴, A-20³ and the stopper rods. On order are dies for L-8, P-4, and T-2.

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Progress Report
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Division T-I

D. NITRIDE RESEARCH - L. S. Foster

Work has been carried out principally on the production of dense N type crucibles of titanium nitride. Towards this end new equipment has been procured such as a 100 ton hydraulic press and a hydrostatic pressing chamber for the use of rubber molds. A tungsten carbide ball mill has also been received for fine grinding without contamination.

Additions to the titanium nitride have been made, especially titanium dioxide. This has been added in various percentages and finely ground in with the nitride. These mixtures have then been fired in ammonia at various temperatures. With 30% titanium oxide, porosities as low as 3% have been obtained at temperatures of 1800°C. With lower amounts of titania the porosity increases. It is felt that in order to solve this problem we shall have to obtain every advantage such as the proper grain sizings, the proper lubricant and the maximum forming pressure as well as sufficiently high firing temperature in order to achieve minimum porosities.

Additions of Ce_2S_3 have been made to titanium nitride and a 10% addition fired at 1750°C showed a porosity of 3.7%. Metallic cerium was melted in this crucible at 1500°C and no attack or penetration was noted.

Some boron nitride of inferior quality has been received and successfully pressed. In firing, however, fumes were evolved and a poor crucible resulted. We expect to obtain some high grade boron nitride soon and will repeat the work. It is hoped that boron nitride will be satisfactory for radiation shields and electrical insulators at high temperatures.

Considerable experimentation has been carried out on high frequency techniques to obtain higher temperatures. Tungsten heaters and thoria shields are being studied as a combination for the higher temperature work.

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Division T-I

E. FURNACE LABORATORY - I. Amdur

Most of the efforts of the furnace laboratory this month have been confined to reductions or firing of sulfide crucibles. The work has been carried out on a single tube unit in molybdenum heaters. During the month a considerable number of reductions were made and a large number of crucibles fired successfully.

The multiple unit with 7" tubes is now installed and only awaiting the arrival of the motor generator set up for completion. This set should be in this week.

Some experimental firings have been carried out on nitride crucibles in vacuum. Temperatures in excess of 2400°C have been obtained in a test fire and this temperature can be reached even in nitrogen. As soon as thorium radiation shields are completed further work will be carried out at higher temperatures. It is felt that the problem of obtaining really dense nitride crucibles depends on the reliable production of somewhat higher temperatures than we have been obtaining and it is hoped that this work will be an answer to this problem.

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Division T-I

F. OXIDE RESEARCH - H. B. Barlett

Magnesia Crucibles

All the type D1a magnesia crucibles on this order have been or are ready to be shipped. They were made from 50% G.E. fused magnesia, 50% calcined magnesia, bonded with 12% Carbowax 4000, and pressed at 50,000 lbs. per sq. in. in a steel die. Improvements in die design, processing of the raw materials, oxidation of the organic lubricant, and firing technique made it possible to produce these with very little loss. They were fired in an oxy-propane furnace at 1760°C. The density was 3.3, the shrinkage 11%, and the porosity less than 0.1%.

A few experimental crucibles of larger sizes made from this composition will be fabricated to determine if there is any further need for magnesia crucibles of this density.

Beryllium Oxide Crucibles

Investigations are going forward to determine the conditions necessary to produce non-porous BeO crucibles. Numerous mixtures were tried, but the most promising results seem to come from the very pure calcined BeO without additions. The best material is too fine for molding with Carbowax or paraffin emulsion, but success has been had with a thermal setting resin of the phenol-formaldehyde type. This is less satisfactory with present equipment than a lubricant that could be molded at low temperatures, so a search for others is being made. Cetyl alcohol shows some promise.

A few non-porous pure BeO crucibles of the A-203 type are being tested to determine if the high density imparts any particularly desirable property. It is hoped that somewhat better surfaces than these crucibles had can be produced, which ought to enhance materially the benefit derived from the non-absorbing surface.

Thorium Oxide Products

Since thoria with 5% added ZrO₂ seems to have a surface with desirable properties, as shown by tests on Type N crucibles, larger sizes are to be made from this composition. Considerable difficulty has been encountered in the matter of raw material, for there is considerable variation in the thoria from one shipment to another. In addition to this difficulty, the Carbowax 4000 also varies, with the result that to

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Progress Report
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Division T-I

F. OXIDE RESEARCH (Cont)

None but badly cracked crucibles have been produced. Lots of various grades of thoria are being obtained from Lindsay Light & Chemical Company, and before anything of value can be produced they will have to be tested and a selection made for future use.

Type Q crucibles for radiation shields are being made by casting. At present shrinkage data are being obtained preparatory to making a mold of the proper size.

Pressed 4" thoria bars are in process also. These were pressed at the A C Spark Plug Division while on a recent visit at that plant. They were fabricated with 7% thermal setting resin, and brought back here for firing. This has not yet been done successfully owing to the considerable thickness (0.9").

Metallographic Work on Sulfides and Nitrides

While all samples of cerium sulfide to date examined have shown varying percentages of two blue phases (one called light blue, one red-blue) no correlation can be found with either the oxidation number (range 2.01 to 2.30) or with the amount of oxygen in the formula (0.00 to 0.083). Apparently it will be necessary to determine more accurately than is known at present the compositions of these blue phases as well as of the "brass" before the microscope can be used in lieu of chemical analysis as control means. A program of research along this line is to be started.

As additions are made to the nitrides to bring down the sintering temperature and reduce the porosity of crucibles made from them, it is expected that the examination of polished sections will become more fruitful. The very high porosity of the pure nitride crucibles in the past has made them quite difficult to study.

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Progress Report
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DIVISION T-II

N. ABSTRACT
(A. R. Kaufmann)

Beryllium billets of 4" diameter contain internal cracks when cast in steel molds. Electrical resistivity measurements indicate that oxygen may be soluble in Be, but the evidence is not complete. Extruded Be tubing has an elongation at fracture of 0.17% and 0.06% in the longitudinal and transverse directions respectively. Apparatus for the distillation of Be should be in operation shortly.

Further evidence has been accumulated to show that carbon influences the formation of epsilon in the X-Si alloys. The epsilon phase has an elongation at fracture of about 0.2% in tension and 15% in compression.

The X-Zr phase diagram still is not understood. An alloy containing 5 atomic per cent Pd has no remarkable corrosion resistance to boiling water, either in the as-cast or the quenched condition.

Apparatus for the creep testing of X-metal wire is about ready for operation.

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DIVISION T-II

O. CREEP-TESTING OF X-METAL
(R. Palmer, A. R. Kaufmann)

1. Purpose of Work

To determine the rate of creep of X-metal at temperatures up to 500°C.

2. Status Last Month

No previous report has been made. All work to date will be discussed here.

3. Progress This Month

a. Type of Creep Apparatus

The test specimen will consist of X-metal wire which is .035" in diameter. In order to prevent contamination of the specimen, the test will be made in a high vacuum creep furnace. The plan of the furnace and its cooperating units is shown in Figure O-1.

A two-stage mercury diffusion pump (A) backed by a Cenco Hyvac mechanical pump is used to evacuate the creep furnace (D). A McLeod gage (B) measures the vacuum attained and a cold trap (C) prevents the mercury vapor from diffusing to the creep furnace.

Heating is achieved by the condensation of such vapors as mercury or high boiling organic liquids. The heating liquid is vaporized in the boiler (G) by an electric furnace, and the vapor conducted into the annular space in section (E) of the furnace, where it surrounds the inside tube of that section. By maintaining the boiling rate so that condensation takes place in the heating zone, a constant temperature is obtained. The reflux condenser (I) returns the remainder of the vapor to the boiler. The chamber (E) allows the heating system to be closed in order to eliminate changes in the boiling point of the liquid caused by variations in atmospheric pressure. With a closed system, a neutral atmosphere can also be maintained in the heating system.

The creep specimen is threaded through the narrow inside tube of section (H), and suspended from the top of the vacuum chamber. The load is applied by means of a dead weight attached to the bottom end of the wire. The top support includes a plug that closes the open end of the furnace and holds a dial gage to measure elongation. A quartz tube around the wire specimen forms an extension of the dial

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DIVISION T-II

O. CREEP-TESTING OF X-METAL (Cont'd)

gage to the bottom end of the wire. Since this arrangement would measure the elongation of the whole length of the wire, both inside and outside of the hot zone, it was decided to reduce the cross section of that part of the wire that is in the hot zone. This reduction is being attempted by grinding away some of the material to form a symmetrical flat strip.

b. Temperature Distribution in the Hot Zone

A survey was made using water vapor as the heating medium, and it was found that the temperature varied from the end to the center by about 18°C. To minimize this variation, a length of copper tube was inserted in the heated section, and a survey then made showed the variation from end to center to be about 10°C. The temperature is constant within $\pm 1^\circ\text{C}$ over a length of 6" at the center of the 10" heated zone. The reduced section of the wire will be made to coincide with the region of the uniform temperature.

4. Future Work

Testing will be started as soon as the wire specimens with a reduced section are ready.

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DIVISION T-II

P. METALLURGY OF BERYLLIUM

(P. Gordon, E. Gordon, E. Vivian, A. R. Kaufmann)

1. Object of Work

The purpose of this work is to learn how to make reasonably ductile beryllium and to develop satisfactory methods of melting and casting the metal on a commercial scale.

2. Status Last Month

Fairly satisfactory castings of 4" diameter billet size were made in graphite molds. Data on the recrystallization of cold worked beryllium as a function of temperature and time was given. Extruded beryllium tubing was found to have the same preferred orientation as extruded rod.

3. Progress This Month

a. Tensile and Elongation Experiments

An extruded tube of beryllium which was found to have a degree of preferred orientation with the basal plane tending to become parallel to the tube axis was used for making tensile and elongation tests. The tube had been extruded to the dimensions 1 3/8" O.D. and 1" I.D. The purpose of these tests was to compare the ductility of the tube in the circumferential direction, i.e. perpendicular to the fibre axis, with that in the longitudinal direction, i.e. parallel to the fibre axis. The longitudinal specimens were flat bars with a gage length of 1 1/2" ground to a cross section 0.110" by 0.130". The rings were machined to a wall section of 1/8" by 1/2" making sure to get below surface imperfection on the tube.

The tests made on the longitudinal specimens were made according to ordinary tensile testing procedure using electrical resistivity strain gages to measure the elongation. In testing the rings, an attempt was made to simulate hydraulic pressure by driving a wedge through a split steel ring placed inside the test rings. However, the stress applied to the ring was far from uniform, and the rings tended to break over the split in the steel ring, where the stress was a maximum. Here again the strain was measured with electrical strain gages; the break was confined to the gage length by placing the test rings in such a position that the gage lay directly over the split in the steel ring.

The test results are listed in Tables P-I and P-II, and the stress-strain curves for the longitudinal specimens are shown in Figure P-I. The per cent elongation for both types of specimens was small, but the average value for the longitudinal specimen, 0.17%, is about three times that for the rings, 0.062%. In other words, it

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TABLE P-I

Test Results for Longitudinal Specimens from Extruded Tube

<u>Specimen</u>	<u>Proportional Limit, lbs. per sq. inch</u>	<u>Stress at Rupture, lbs. per sq. inch</u>	<u>% Elongation at Rupture</u>	<u>Modulus of Elasticity lbs. per sq. inch</u>
1	17,700	30,600	0.14	34 x 10 ⁶
2	17,600	30,100	0.11	38 "
3	21,300	32,800	0.13	37 "
4	20,100	34,500	0.18	34 "
5	19,000	32,400	0.14	39 "
6	18,400	38,200	0.30	38 "
7	18,000	34,600	0.20	35 "
Ave.	18,900	33,300	0.17	36 "

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TABLE P-IITest Results for Ring Specimens from Extruded Tube

<u>Specimen</u>	<u>% Elongation at Rupture</u>
1	0.033
2	0.04
3	0.064
4	0.063
5	0.112
6	0.057
7	0.022
8	0.102
Ave.	0.062

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P. METALLURGY OF BERYLLIUM (Cont)

appears that with the presence of preferred orientation the ductility in a direction parallel to the fibre axis is appreciably greater than that perpendicular to the fibre axis. It seems likely also that most of the strain in the rings was elastic, while in the longitudinal specimens the strain was partially elastic and partially plastic.

b. Electrical Resistivity Measurements

Table P-III lists the results of resistivity measurements made on several extruded specimens received from Creutz and two extruded specimens from metal melted at M.I.T. The metal received from Creutz had been melted and cast by the Brush Company. In general, the Creutz specimens have lower resistivities than the M.I.T. specimens. The only persistent difference in composition lies in the magnesium content, the Creutz specimens containing considerably more magnesium.

It may be noted that the quenched resistivities of the Creutz specimens are all higher than the corresponding annealed values. This indicates that there is some solution of the impurities effected by quenching. However, there does not seem to be any obvious correlation between the amount of the various impurities and the absolute resistivities.

In an attempt to determine whether oxygen dissolves in beryllium to any appreciable extent, specimen M.I.T. #2 was heated in air for two hours at 1150°C and then soaked in vacuum for six days at 1000°C. Measurements of the resistivity after this treatment were not very reproducible, but the values obtained were good enough to indicate that the resistivity of the specimen had increased considerably. Four measurements were made, three measured over a 1" length giving for the resistivity of the specimen after the oxidation treatment 5.01, 5.03, 4.97, and 5.29 $\times 10^{-6}$ respectively. The increase in resistivity (over 4.30×10^{-6} before oxidation) cannot be definitely attributed to dissolved oxygen, since there is some rather inconclusive microscopic evidence for minute precipitated particles in the beryllium. Further work will be done on this problem.

c. Melting and Casting

The problem of casting beryllium in 4" billet sizes is not yet satisfactorily solved due to reaction with the mold material or to cracks in the casting. It is probable that good castings could be made in BeO molds, but the use of this material has been avoided in the hope of finding something more convenient and less expensive. Efforts so far have been directed toward the use of graphite and of water-cooled metal molds.

There is a definite reaction between beryllium and graphite which leads to an evolution of gas (particularly if the mold has been used before) which throws quite a lot of metal out of the mold. This

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TABLE P-III

Electrical Resistivities and Composition of Some Be Specimens

<u>Specimen</u>	<u>As Extruded</u>	<u>Annealed 1000 C</u>	<u>Quenched 1000 C</u>	<u>% Al</u>	<u>% Fe</u>	<u>% Mg</u>	<u>% Si</u>
Creutz #1 ¹	3.93×10^{-6}	3.90×10^{-6}	4.07×10^{-6}	.14	.056	.69	.083
Creutz #2	4.11 "	3.97 "	4.04 "	.16	.042	.58	.20
Creutz #4 ¹	4.06 "	3.91 "	4.08 "	.15	.028	.81	.12
Creutz #6 ¹	4.03 "	4.02 "	4.19 "	.23	.042	.81	.14
Creutz #7 ¹	4.01 "	3.99 "	4.11 "	.12	.27	.39	.17
Creutz #8 ¹	4.09 "	3.96 "	4.01 "	.17	.055	1.00	.45
Creutz #9 ¹	3.88 "	3.93 "	4.07 "	.14	.055	.89	.19
Creutz Ave.	4.02 "	3.95 "	4.08 "				
M.I.T. #2		4.31		.18	.15	.056	.12
M.I.T. #4		4.28		"	"	"	"
Ave.		4.30					

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P. METALLURGY OF BERYLLIUM (Cont'd)

leads to a poor recovery of metal and an exaggerated pipe, even when the top of the graphite mold is hot. In general, about 75% of the metal charged into the crucible ends up in the mold, while about 10 to 12% is thrown out into the furnace. Of the remaining metal, about 5% remains as a button in the crucible, another 5% collects on the condensing plates, and the rest is unaccountably lost. About a 5" length of solid metal is obtained when enough Be for a 9" length is cast. The solid metal usually has no defects other than surface blemishes. A transverse and longitudinal radiograph of a typical graphite-cast billet is shown in Figure P-III. Most of the pipe has been machined away.

When a water-cooled iron mold with a BeO hot top is used, there is only occasionally a superficial reaction with the iron. The molten Be remains in the mold, and this leads to about 65% recovery in the casting. In general, the pipe is not as large as when graphite molds are used, and thus a 6" length of solid metal is usually obtained. These castings were considered to be very satisfactory until it was discovered that they always contain internal cracks as revealed by the radiographs in Figure P-II. Most of these defects run in a radial direction, and sometimes extend the length of the ingot. The transverse view in Figure P-II shows one long crack and several transverse cracks towards the right end of the picture. These transverse cracks occur where the crystals growing up from the bottom of the ingot encounter those growing out from the side.

The cracks just mentioned presumably are caused by thermal stresses due to the rapid cooling in the steel mold. They are almost always absent in the billets cast in graphite, and hence these must be considered to be better than the steel mold castings even though there is more wastage of metal in making them.

d. High Purity Beryllium

A program to study the possibility of improving the ductility of commercial beryllium metal has been initiated. The research phases of the program have been divided into two sections: first, the production of very pure beryllium with a view to determining its properties as an engineering material; and second, the investigation of the effect on ductility of low alloying and deoxidizing additions to beryllium. In the first section of the research, the elimination of oxygen will receive special attention. The lines along which the research may be conducted can be grouped as follows: (1) The development of very pure materials from which beryllium metal can be produced; (2) development of special reduction processes which will yield pure beryllium; and (3) development of special refining methods

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P. METALLURGY OF BERYLLIUM (Cont'd)

for producing pure beryllium from commercially available metal. The phase of the research under investigation at this time comes under the third heading, viz. the production of pure beryllium by refinement of commercial metal.

The method of refinement being used consists of a vacuum fusion in which the low boiling metal impurities are distilled from the beryllium. This is followed by a molecular distillation of the beryllium leaving the high boiling impurities and oxides in the residue. The vacuum fusion furnace is in operation at present. No oxygen analyses are available at this time, but since the metal is handled in beryllia, it may be assumed to be saturated with oxygen.

The molecular distillation furnace is in the process of design and construction. Of the metal impurities remaining after removal of the low boiling metals, calculations indicate that aluminum and manganese will appear in the distillate from a simple molecular distillation, as was found to be the case by Kroll. Hence, in order to remove these metals, special care will have to be taken to achieve their elimination either in the reduction reaction or by a sharp fractional distillation. Calculations also indicate that it should be possible to keep the silicon as well as the iron in the residue, providing their concentrations in the residue are kept low.

4. Future Work.

Research on the casting of Be for billets and other large shapes will continue. Laboratory studies of the solid solubility of elements, particularly oxygen will be carried out. The apparatus for distillation of Be should be in operation soon.

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C. X-Si ALLOYS - Epsilon Phase

(G. B. Searles, E. G. Gilly, R. D. Bastian, A. R. Kaufmann)

1. Purpose of Work

To determine the equilibrium diagram in the region of the epsilon phase of the X-Si system and to study the technical properties of the epsilon alloys.

2. Status Last Month

There were indications that carbon influenced the formation of the epsilon phase. Initial tests indicated that the epsilon alloys were much superior to pure X-metal in corrosion-resistance to boiling distilled water.

3. Progress This Month

a. Equilibrium Diagram

In order to study the effect of carbon on the epsilon phase, a series of alloys was made in which the carbon content was held down to about .01 weight %, while the silicon was increased from 4 to 23 atomic per cent. A second group of specimens was prepared containing about 20% Si and increasing amounts of carbon up to about 0.14% by weight. Microexamination indicated that substantially all of this carbon was in solution or in some combination other than free carbide.

The plan of investigation was to quench specimens of each alloy of these two groups from a series of temperatures between 750 and 960°C and then to examine them microscopically to determine the phases present. Some of the specimens were to be heated first to 960°C and then quenched from successively lower temperatures as the temperature was decreased slowly, while others were to be converted first to epsilon and then quenched from successively higher temperatures. In this way, the first formation and the first decomposition of epsilon on each of the specimens could be determined.

Results so far obtained on the carbon-free alloys (.01% C) indicate that epsilon forms at a definite temperature (940°C) in alloys ranging from 4 to 23% Si. This establishes fairly well that carbon-free epsilon forms by a peritectoid reaction between gamma solid solution and X_5Si_3 . It is hoped that the completion of this work will definitely prove this point and will further demonstrate

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X-Si ALLOYS - EPSILON PHASE (Cont'd)

the influence of carbon on the reaction.

X-ray determinations of the phases present in the quenched alloys have not been very successful due to difficulty in obtaining satisfactory diffraction patterns.

b. Corrosion Tests

The corrosion behavior of alloys containing the epsilon phase is being determined in distilled water at 100°C and at 70°C. The water is continuously replenished at a rate of about 20 cc. per minute and it is saturated with either hydrogen, oxygen, or air. Loss of weight without any treatment of the specimen other than drying is taken as a measure of the corrosion. Insufficient results have been obtained to date to warrant a detailed presentation. It may be said, however, that under these conditions pure X-metal corrodes at a rate of about 2 milligrams per square cm. per hour at 100°C and at about one tenth that rate at 70°C. The corrosion rate is very little affected by any of the gases previously mentioned. At 100°C the epsilon alloys containing 15 to 23 atomic % Si show a decreasing corrosion rate which approaches zero after several hundred hours. This is true regardless of which gas is added to the water, although the total corrosion is about twice as great in hydrogen as in oxygen saturated water. At 70°C, all of the alloys show very little corrosion for the first 200 hours, and after that is a slow loss of weight.

Attempts have been made to evaluate the corrosion rate after the removal of any adhering film. This is accomplished by placing the specimen after test into 25% nitric acid for 5 minutes and then weighing. There does seem to be a definite weight of film on the specimen, but the extent to which this increased with time has not yet been determined.

c. Tensile Test

Tensile tests on specimens containing substantially 100% epsilon indicate a tensile strength of about 40,000 to 50,000 lbs. per sq. inch and an elongation at the time of fracture of about 0.2%. This is to be compared with a strength of 300,000 lbs. per sq. inch and a deformation of 15% in compression.

There can be no doubt that epsilon is a brittle material and yet it should be emphasized that there is sufficient ductility to

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X-Si ALLOYS - EPSILON PHASE (Cont'd)

allow for machining and rough handling without fear of breakage.

4. Future Work

Attempts to establish the phase diagram will continue. The influence of carbon on the corrosion behavior of epsilon alloys will be determined, and other ternary combinations such as with columium will be examined.

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R. X-Zr ALLOYS

(A.R. Kaufmann, R. D. Cullity, H. Feher)

1. Purpose of Work

To establish the X-Zr phase diagram and to determine any useful properties of the alloys.

2. Status of Work at End of Last Month

Thermal analysis had shown that the addition of Zr raises the melting point of X and lowers the temperature of both solid transformations. The gamma phase was not retained by quenching, either in X-Zr or in X-Xr-Cb alloys.

3. Progress During Current Month

An alloy analyzing 11.3 atomic per cent Zr has been subjected to various quenching experiments. In the slowly cooled condition, this alloy contains free zirconium, or a Zr-rich solid solution, in a matrix having a complex structure, which is probably due to a precipitated phase that cannot be resolved under the microscope. (This structure was called martensitic in a previous report, but it is felt that this term was misused and should be avoided.) When this alloy is quenched from temperatures above 700°C, and as high as 1100°C, the matrix becomes practically homogeneous in appearance, although careful examination reveals a sort of acicular structure standing in relief. X-ray diffraction of the quenched alloy gives a diffuse pattern of alpha X.

Heat treatment of the same alloy at 675°C results in partial spheroidization of the precipitate believed to occur in the slowly cooled alloy, and the structure after a 700°C treatment indicates that gamma may be starting to form at this temperature. These results are in agreement with those obtained by thermal analysis which showed the beta-to-gamma transformation occurring at about 700°C.

Similar quenching experiments on a 20 atomic per cent Zr alloy give similar results except that no trace of the acicular structure is found in the alloys quenched from above 700°C, i.e. the matrix appears perfectly homogeneous. Nevertheless, X-ray diffraction shows that the gamma phase is not retained.

Since it was felt that the alloys made in the initial stages of this investigation were not sufficiently pure and homogeneous, a new series of alloys has been made for future work.

4. Program for Future Work

A systematic series of quenching experiments will be made using the new series of alloys in an attempt to work out the correct phase relationships in this system.

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S. X-Pd ALLOYS

(R. P. Postian, E. D. Gullity)

1. Purpose of Work

Investigation of the corrosion resistance of the X-rich alloys is the present object of this work.

2. Status Last Month

The general features of the X-Pd diagram had been established and the solid solubility of Pd in X was shown to be about 4 atomic per cent Pd at 930°C.

3. Progress This Month

The poor corrosion resistance of a 5 atomic per cent Pd alloy in boiling distilled water is shown by the curves of Figure S-1. One piece of this alloy had been used for thermal analysis; its structure was thus characteristic of a slowly cooled specimen. The corrosion resistance of this material is of the same order of magnitude as that of X.

When the same alloy is water-quenched from 950°C, its corrosion resistance is improved, as shown by the lower curve of Figure S-1, but not to any great degree. The actual corrosion rate is still large and continues to increase with time.

4. Future Work

As much additional work on the clarification of the X-Pd diagram will be done as time permits.

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FIGURE 2

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Figure P 1. Stress-Strain Curves for Longitudinal Tensile Specimens from Extruded L-Metal Tube

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P-2 Radiograph of Beryllium Cast into
Water-cooled Steel Mold

Top End

Transverse Direction

Bottom End

Parallel
to Axis
of Ingot

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FIGURE 4

P-3 Radiograph of Beryllium Cast into Graphite.

Top End

Transverse Direction

Bottom End

Parallel
to Axis
of Ingot

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FIGURE 5

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CORROSION TESTING OF X-Pd ALLOYS

Corroding Medium: Distilled Water At 100°C.

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