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NUCLEAR METALLURGY LECTURES -- CHAPTER 18

AUTHOR
I. D. Thomas

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NUCLEAR METALLURGY LECTURES

Metallurgy of Plutonium - I. D. Thomas

Plutonium metal has been found to be rather unique in many ways, and this lecture will attempt to summarize the more important metallurgical knowledge available at this site. The greatest portion of our knowledge of plutonium was obtained at the Los Alamos Scientific Laboratory during the early phases of the Manhattan Project with additional contributions from the few other sites engaged in this work.

For all practical purposes, the "natural abundance" of plutonium in nature is insignificant (one part in 10^{11} in pitchblende). It is therefore regarded as a purely synthetic element, and its "synthetic abundance" is limited to the "natural abundance" of U-238, the only isotope from which plutonium has been practically derived.

A. Physics and Health Considerations

Plutonium metal is an alpha particle emitter with a half-life for the isotope 239 of 24,300 years. The alpha particles have an energy of 5.15 mev and a range in air of 3.68 cm. In body tissue, their range is only about 45 microns so that the health hazard involved in handling plutonium occurs when it is taken into the body. Damage to body tissue is confined by the low penetrating power of the large alpha particles to a small area immediately surrounding the point of plutonium deposition.

When plutonium is taken into the body, it is primarily deposited in the bones and excreted very slowly. The current maximum permissible body burden (in bone) has been set at 0.04 microcurie or 0.5 microgram. The time required for the body to excrete one-half the body burden is estimated at 100 years. The body burden of people working with plutonium must be periodically checked by determination of the Pu content of the urine based on an average urinary excretion rate of 0.01 percent of the body content per day.

Plutonium can enter the body in three different ways:

1. Inhalation and absorption through the lungs.
2. Ingestion and absorption from the gastrointestinal tract.
3. Absorption through the skin.

The most important of these is inhalation since for soluble plutonium compounds about 10% are retained. Insoluble compounds are retained on the average only about 200 days. Since this hazard is so great, continuous air samples are taken at this site in all areas potentially contaminated by plutonium and are counted daily.

Ingestion can be easily controlled so that this is only a minor hazard. Ingress of plutonium taken into the gastrointestinal tract can be reduced to about 0.01% by proper treatment.

Plutonium compounds can otherwise enter through the skin from organic solvents or through breaks. Personnel handling plutonium must always wear rubber gloves, and those having cuts on the hands or wrists must not work with plutonium or its compounds.

Since plutonium metal is a mixture of isotopes, there is also a low energy gamma and x-ray radiation which requires that handling times be kept to a minimum in order to prevent undue hand exposures. With the shielding provided by the ordinary rubber gloves (0.025 inch thick), the dosage rate is approximately 0.9 rep/hour, but this may vary considerably due to variation in purity and in shielding.

Time will not permit a description of the many techniques developed for the handling of this metal, but it should be pointed out that nearly all work is carried out inside sealed and ventilated gloved boxes. The handling is done through long gauntlet gloves sealed to the front of the box. Frequent monitoring is required to prevent the spread of contamination, and materials removed from the gloved boxes must be sealed in containers free from external contamination.

The problem of critical mass must also be considered in the handling of plutonium. Very conservative safety limits are defined in Table I.

TABLE I

Form	Maximum density in 3-dimensional array of units kg/ft	Maximum density in 2-dimensional array of units kg/ft	Macimum mass in a unit package kg
Massive metal	1.5	2.0	2.0
Aq. Solutions	0.15	0.15	0.25

The values listed for pure plutonium metal can also be applied to plutonium alloys except alloys of beryllium and lithium where the moderating effect of the light element atoms may not, in the calculations of critical mass, be fully compensated for by the diluting effect. Also, the solution values should be used if powders or shot is stored where there is the least chance that the gloved box could be accidentally flooded by water. Under certain conditions with proper consideration given to shapes, larger quantities of metal can be safely handled. Thus, untamped alpha plutonium has the following critical sizes: a slab 1.07" thick, a sphere 3.14" diameter, and a cylinder 2.18" in diameter.

B. Reduction

The winning of plutonium from a pure plutonium compound cannot be accomplished by reduction with hydrogen or carbon, as is done with the less reactive metals, as stable hydrides and carbides are formed. The reduction can be effected by the Goldschmidt process using aluminum, calcium, or magnesium. While plutonium oxide can be reduced by this method, the resulting alkaline earth oxide causes the slag to have a high melting point and a poor separation of metal results. Thus, plutonium halides are ordinarily used according to one of the reactions:

$$3PuF_4 + 4Al \longrightarrow 3Pu + 4AlF_3 + 47\ \text{Kcal}$$
$$2PuF_3 + 3Mg \longrightarrow 2Pu + 3MgF_2 + 40\ \text{Kcal}$$
$$PuF_4 + 2Mg \longrightarrow Pu + 2MgF_2 + 103\ \text{Kcal}$$
$$2PuF_3 + 3Ca \longrightarrow 2Pu + 3CaF_2 + 121.6\ \text{Kcal}$$
$$PuF_4 + 2Ca \longrightarrow Pu + 2CaF_2 + 157.6\ \text{Kcal}$$

The exothermic reduction reaction is started by heating the reactants in an enclosed bomb. After firing, the generated heat should be sufficient to maintain all the reaction products in the molten state so that the metal can coalesce in the bottom of the crucible. This can be better assured by additional heat gained from a "booster" reaction between iodine and some excess calcium, magnesium, or aluminum.

$$Ca + I_2 \longrightarrow CaI_2 + 128.5 \text{ Kcal}$$
$$Mg + I_2 \longrightarrow MgI_2 + 101.7 \text{ Kcal}$$
$$2Al + 3I \longrightarrow 2AlI_3 + 198 \text{ Kcal}$$

In addition, the calcium, magnesium, or aluminum iodide formed often decreases the melting point of the slag which improves the slag-metal segregation. Very high recoveries can be obtained by this method under proper conditions.

C. Allotropy and Structure

Pure plutonium metal has been found to exist in six different modifications, and in this respect it is very unique among pure metals. Table II shows the transformation temperatures and volume changes for these phases.

TABLE II (UNCLASSIFIED)
(Los Alamos Scientific Laboratory)

Transformation	Dilatometer	Thermal Analysis	$V = \frac{V_2 - V_1}{V_1}$
$\alpha - \beta$	122 ± 2	122	8.9%
$\beta - \gamma$	206 ± 3	203	2.4%
$\gamma - \delta$	319 ± 5	317	6.7%
$\delta - \delta'$	451 ± 4	453	-0.4%
$\delta' - \epsilon$	476 ± 5	477	-3.0%
ϵ - liq.		639.5 ± 2	

Tables III and IV give the structures and interatomic distances for the gamma, delta, delta prime, and epsilon phases in high purity metal. At the present time, the x-ray powder data for the alpha and beta phases have not yielded their unit cell dimensions or their crystal systems. Attempts to obtain small single crystals for structure determinations have also been unsuccessful due primarily to the low atom mobility at the temperatures involved.

TABLE III
(Los Alamos Scientific Laboratory)

CRYSTAL STRUCTURE DATA ON HIGH PURITY PLUTONIUM METAL

				Density
Gamma: Face-Centered Orthorhombic				
At 210 C	a_o = 3.1603Å	b_o = 5.7624	c_o = 10.141	17.19
Delta: Face-Centered Cubic				
At 320 C	a_o = 4.6370			15.92
Delta-Prime: Face-Centered Tetragonal				
At 465 C	a_o = 4.701	c_o = 4.489	c/a = 0.955	15.99
Epsilon: Body-Centered Cubic				
At 500 C	a_o = 3.638			16.48

TABLE IV
(Los Alamos Scientific Laboratory)

INTER-ATOMIC DISTANCES IN PLUTONIUM METAL STRUCTURES

Phase	Structure	Coordination	Distances	Temp. °C
Gamma	Orthorhombic	10	4 Pu 3.021Å 2 Pu 3.160Å 4 Pu 3.286Å Ave. 3.155Å	210 " " "
Delta	Cubic F.C.	12	3.279Å	320
Epsilon	Cubic B.C.	8	3.150Å	500

Since the structures of the majority of the compounds of plutonium have been found to be isomorphous with the corresponding uranium compounds, it was thought to be reasonable that the structures of α-plutonium and α-uranium might be isomorphous. However, powder data definitely establish the fact that this is not the case.

D. Physical Properties

The most unusual physical property of plutonium is its very large thermal expansion. Table I shows the volume expansion between alpha and delta plutonium to be about 18 percent. While the large majority of this expansion is due to phase changes, the linear expansion coefficients listed in Tables V and VI for the lower temperature phases are two to three times as large as those for most metals.

TABLE V
(Los Alamos Scientific Laboratory)

PHASE DATA

Phase	Density	Linear Expansion Coefficient(1) x 10^6	Resistivity $\rho \times 10^6$	$1/\rho \cdot d\rho/dt \times 10^5$
α	19.737 (25°)	50.8 (25°)(2)	145 (28°)	-21
β	17.65 (150°)	38.0 (164°)(2)	110.5 (132°)	- 6
γ	17.19 (210°)	34.7 (ave)(2)	110 (230°)	- 5
δ	15.92 (320°)	-10.0 ± 0.5	103 (353°)	+ 7
δ'	15.99 (465°)	-120	105 (462°)	+45
ϵ	16.48 (500°)	25.7 ± 2.0	114 (490°)	- 7
Liquid	16.50 (665°) ±0.08	50(3)		

(1) $\frac{1}{L} \cdot \frac{L}{t} = \alpha$

(2) See Table VI
(3) Volume coefficient
Note: t = °C

TABLE VI
(Los Alamos Scientific Laboratory)

EXPANSION COEFFICIENTS OF α, β, AND γ PHASES OF PLUTONIUM METAL

(t = °C)

Alpha (-180° to +122°C)

$$\alpha \times 10^6 = (48.39 \pm 0.01) + (0.0959 \pm 0.0012)t$$

Beta (82° to 206°C)

$$\alpha \times 10^6 = (2604 \pm 0.01) + (0.0740 \pm 0.0015)t$$

Gamma ($\alpha \times 10^6$ in each case)(149 to 319°C)

$$\alpha_{[100]} = -19.7 \pm 1.7 ; \ \alpha_{[010]} = 39.5 \pm 0.9 ; \ \alpha_{[001]} = 84.3 \pm 2.3$$

$$\alpha_{\text{Ave.}} = 34.7 \pm 3.0$$

This is illustrated graphically by the dialation curve on heating shown in Figure 1. It is especially interesting to note that the more efficiently packed face-centered cubic structure of delta plutonium (coordination 12) is more expanded at a lower temperature than the less efficient body-centered cubic structure of the epsilon phase (coordination 8) at a higher temperature.

The phase densities are listed in Table V. One interesting fact that is to be noted is that the delta and delta prime phases are even less dense than the molten metal and that the epsilon phase has a density very close to that of the liquid. The wide variation in density between alpha (19.737) and delta (15.92) gives a very convenient method of distinguishing between the phases.

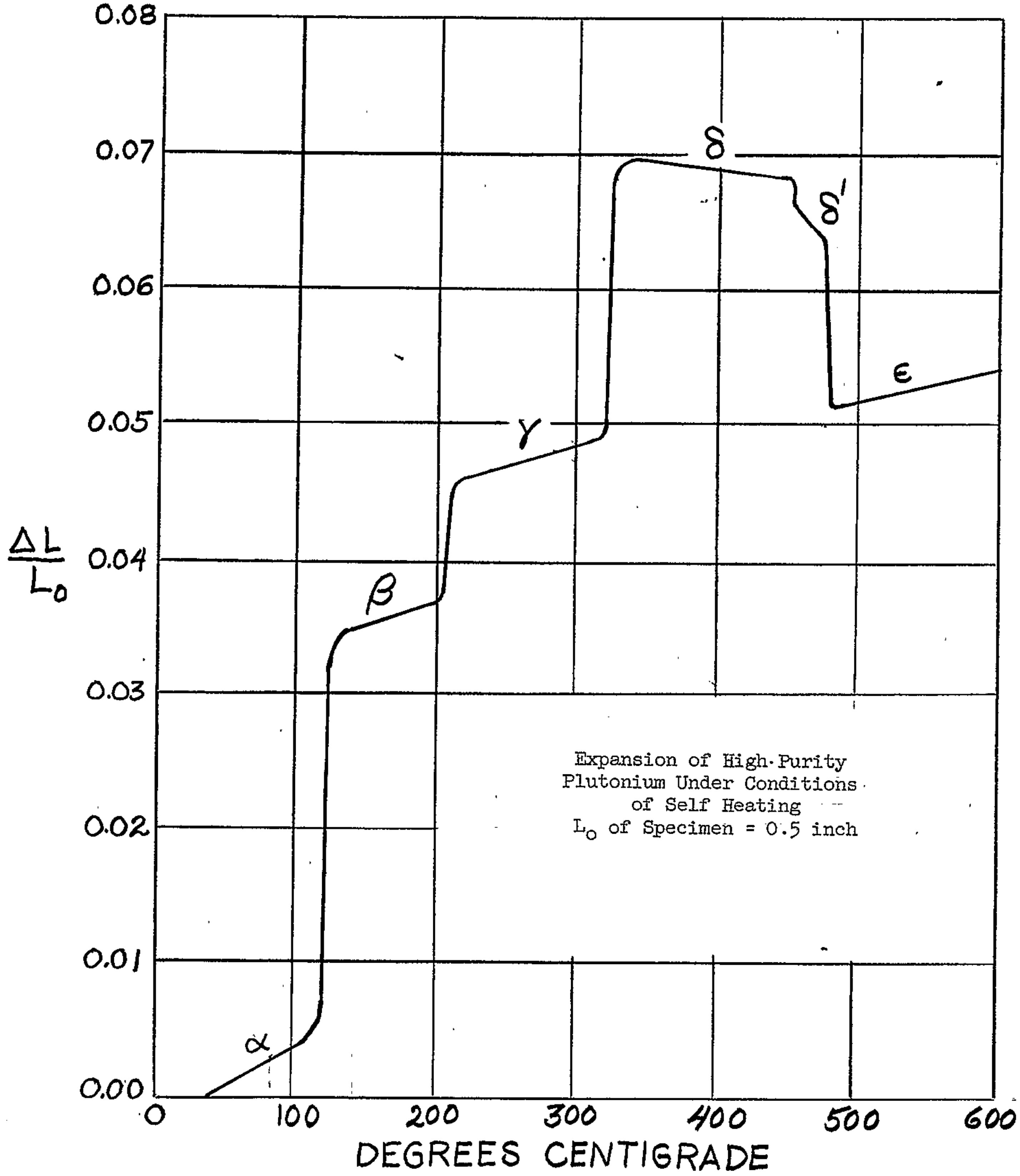
The resistivity of the various phases is also listed in Table V. These values are to be compared with those for uranium (α 59 x 10^{-6}, β 56 x 10^{-6}, and γ 55 x 10^{-6}). While no data are available on thermal conductivity of plutonium, it is expected it would be somewhat lower than that for uranium since thermal and electrical conductivity are usually closely related.

Very little work has been done on transformation rates between the various phases. However, the early dialation curve shown in Figure 2 indicates the sluggishness of the transformation on cooling. These rates are very dependent upon purity, and the curve shown was obtained on material of 99.87 wt % (99.01 atomic %) pure. Small amounts of some alloying elements cause stabilization of the delta structure as will be shown later. Impure metal has hampered plutonium work for some time, but high purity material (99.97 wt %) is now available. The typical analysis of cast metal is given in Table VII.

TABLE VII

Element	Content ppm	Element	Content ppm
Aluminum	<30	Manganese	50
Beryllium	< 0.08	Sodium	5
Calcium	<10	Nickel	100
Chromium	50	Silicon	20
Iron	200	Boron	0.8
Lanthanum	<80	Fluorine	2.8
Lithium	< 1	Carbon	350
Magnesium	40	Oxygen	-

Σ ppm = 988.9



Expansion of High-Purity
Plutonium Under Conditions
of Self Heating
L_o of Specimen = 0.5 inch

Los Alamos Scientific Laboratory
Reference (3)

FIGURE 1

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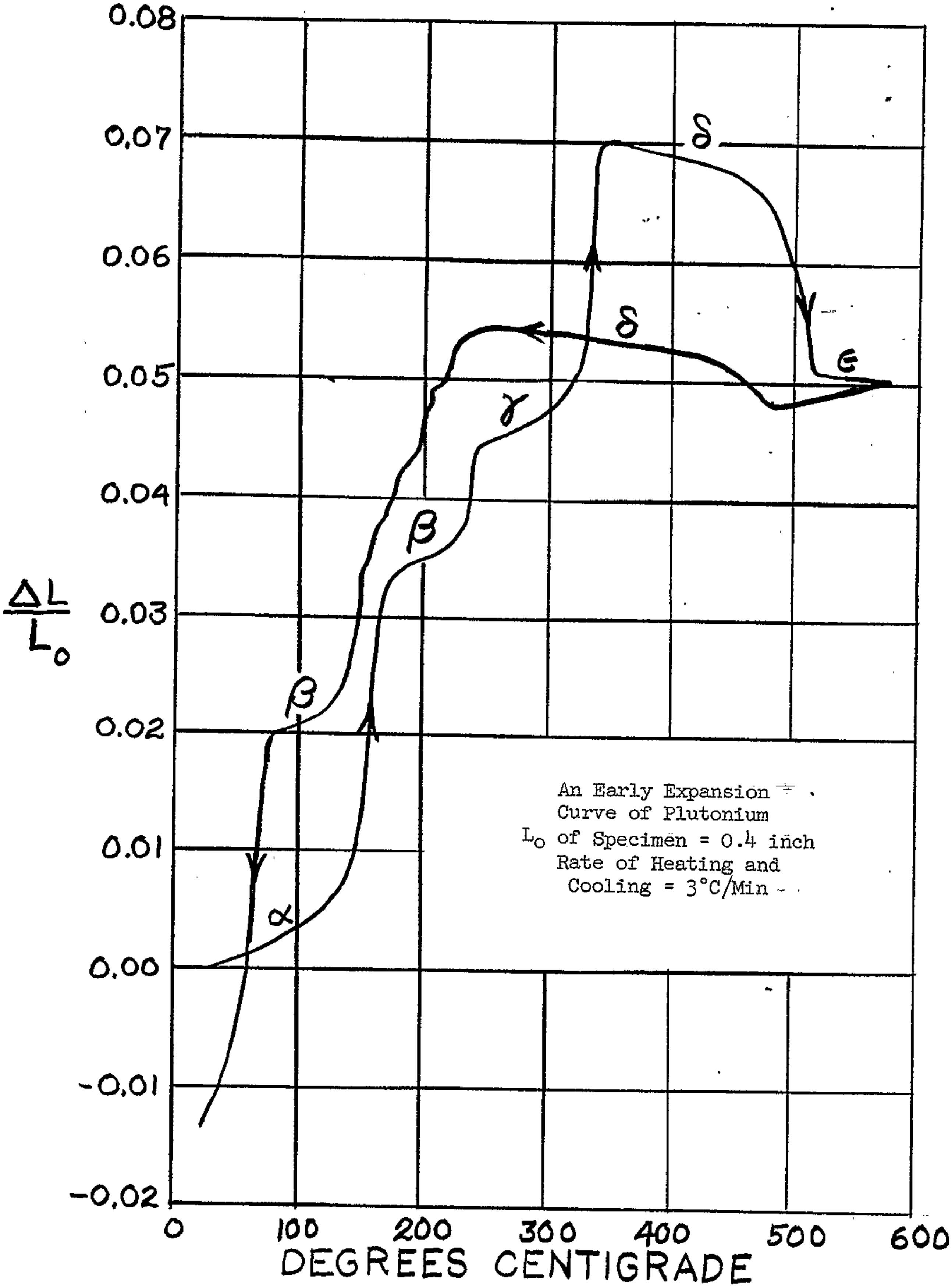

An Early Expansion
Curve of Plutonium
L_0 of Specimen = 0.4 inch
Rate of Heating and
Cooling = 3°C/Min

Los Alamos Scientific Laboratory
Reference (3)

FIGURE 2

E. Mechanical Properties

The available mechanical properties of the various phases of plutonium have not as yet been completely established. Table VIII lists some of the data for the alpha and delta phases

TABLE VIII

	α Pu	δ Stabilized	Uranium
Diamond Pyramid Hardness (2 Kg Load)	250	40	225
Modulus of elasticity (psi)	14.7 x 10	6.9×10^6	23×10^6
Yield strength 0.2% offset (psi)	45,000	8,900	33,500
Ultimate tensile strength	51,800	15,300	91,500
Reduction in area	< 1%	71%	9.5%
Elongation (1-inch)	< 1%	77%	12.5%
Type of Fracture	Brittle	Ductile	--
Sample	Cast-annealed	As-cast	Beta heat treated

These values for plutonium were obtained here in the Product Metallurgy Unit using tensile specimens. Higher strengths than these have been found at Los Alamos in compressive tests on alpha plutonium (ultimate strength 125,000 psi). By comparison with uranium, alpha plutonium is relatively weak and brittle while the delta material is more ductile and can be compared with 2S-0 aluminum.

F. Alloys

Small amounts of aluminum or gallium in plutonium stabilize the delta structure down to room temperature, as can be seen from the constitution diagrams, Figures 3 and 4. The solid solubility of plutonium in the following metals has been found to be too low to be detected by precision x-ray diffraction measurements of unit cell size: aluminum, beryllium, bismuth, copper, gold, lead, tin, and vanadium. This is illustrated for aluminum and vanadium in Figures 3 and 5.

The solid solubility of plutonium has been found to be appreciable in magnesium, nickel, titanium, uranium, and zirconium. This is shown in Figures 6 and 7 for nickel and uranium. With uranium, plutonium forms a nearly complete series of solid solutions upon solidification. From 580 C to room temperature approximately 10 atomic percent plutonium is soluble in uranium. An isomorphous series between the two compounds PuU_3 and Pu_3U exists in the composition range between 25 and 75 atomic percent plutonium. Plutonium-rich alloys dissolve at most only one or two percent uranium below 450 C.

G. Fabrication

Alpha plutonium can be fabricated only by casting and machining. Fabrication in the delta range with subsequent transformation to alpha material could conceivably be useful in some cases, but the 18% volume change between these phases usually causes warping of the final shape. Hot pressing with transformation in the die under pressure (pressure helps the thermodynamics of the transformation) has some definite possibilities.

[redacted]

Delta stabilized material can be fabricated by casting, machining, rolling, press forming, and extruding much the same as pure aluminum. These mechanical working operations can be conducted at room temperatures, although intermediate annealing in the delta range may be required for extensive deformations. The major difficulty encountered is the reactivity of the metal. Plutonium oxide doesn't form an adherent protective film as is the case with aluminum. Corrosion rates in damp air are excessive, especially if the metal is above room temperature as in annealing or hot working. A dry purified inert gas, or preferably a high vacuum is required in annealing and soaking furnaces. Storage at room temperature is best carried out in dry air as the oxygen helps to passivate the surfaces.

Melting of plutonium is done in vacuum furnaces. Vacuums of 10^{-5} to 10^{-6} mm Hg are required for good purity and recovery. Both resistance and induction (high or low frequency) melting furnaces have been successfully used, and the metal is poured under vacuum. Since the melting point of Pu is 627 C and the boiling point is 3240 C (higher than that of nickel or iron), many volatile impurities are distilled off without too great a loss of plutonium. This is especially useful in decreasing the hydrogen content. Some trouble is encountered in starting the pour ~~in~~ and in obtaining coalescence of the molten charge without mechanical agitation due to the tough oxide skin formed about each piece of charge or over the surface of the melt. For this reason it is difficult to remelt turnings at least without using some sort of flux.

Since plutonium is so very reactive, extreme care must be taken during fabrication to avoid impurities if high purity metal is desired. MgO crucibles and molds have been almost universally accepted for melting and casting operations. Molybdenum sulfide is used as a die lubricant for pressing and extruding. Carbides, nitrides, and hydrides are easily formed especially when the material is hot. Massive pieces of metal are self-heating due to the radioactivity of Pu-240 which is present to about 4.5% in material from pile exposures of 600 MWD/T. This self-heating obviously increases corrosion rates.

Another difficulty often encountered during the fabrication of plutonium is due to its pyrophoric nature. Turnings, filings, melting skulls, etc., will spontaneously catch fire with the possibility of igniting more massive pieces. This danger can be decreased by using carbon tetrachloride or trichlorethylene as a coolant and filling the gloved box with inert gas. All such operations should be followed by immediate clean up of chips and powder, with storage in sealed containers.

H. Irradiation Behavior

The crystal structure of alpha plutonium is described as being of the "uranium type". It is to be expected therefore that plutonium will behave similarly to uranium under irradiation. However, the little experience that has been had with the irradiation of plutonium has been with delta stabilized material. Delta stabilized plutonium rods 5 1/2" long by 0.647" diameter were used to operate the Los Alamos Fast Reactor (Clementine). The plutonium rods were coated with 0.003" nickel and inserted in mild steel cans having a wall thickness of 0.020". The cans were then drawn through a die to insure mechanical contact and closed by welding. During the first year of operation the plutonium operated at temperatures up to 150 C with a total irradiation of approximately 0.2 MWD/T. At the end of this period the fuel elements were unchanged in external appearance, but radiographs showed that they had decreased in length inside the jackets. The rod showing the greatest contraction was decanned for examination. The plutonium slug contained a large number of small cracks as well as a large round spot having a "shriveled and cracked" appearance. The material had increased in density about 2%.

During subsequent operation of the reactor the temperature of the central plutonium slug was about 170 C and irradiation continued up to approximately 250 MWD/T for the 14.4 Kg of plutonium. At this exposure thirty-one of the thirty-two slugs involved were intact, but one slug was badly ruptured by a longitudinal slit.

Delta stabilized plutonium pins 1/16" diameter x 3/16" long, clad with nickel and mounted in zirconium blocks have been irradiated in CP-3 by Argonne National Laboratory. Upon examination after about 2700 and 8100 MWD/T, "changes in length are negligible and all pins have retained their shape remarkably well."

Ivor D Thomas
Product Metallurgy Unit
Pile Technology Section
ENGINEERING DEPARTMENT

ID Thomas:kb

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TENTATIVE PHASE DIAGRAM OF THE PLUTONIUM-ALUMINUM SYSTEM

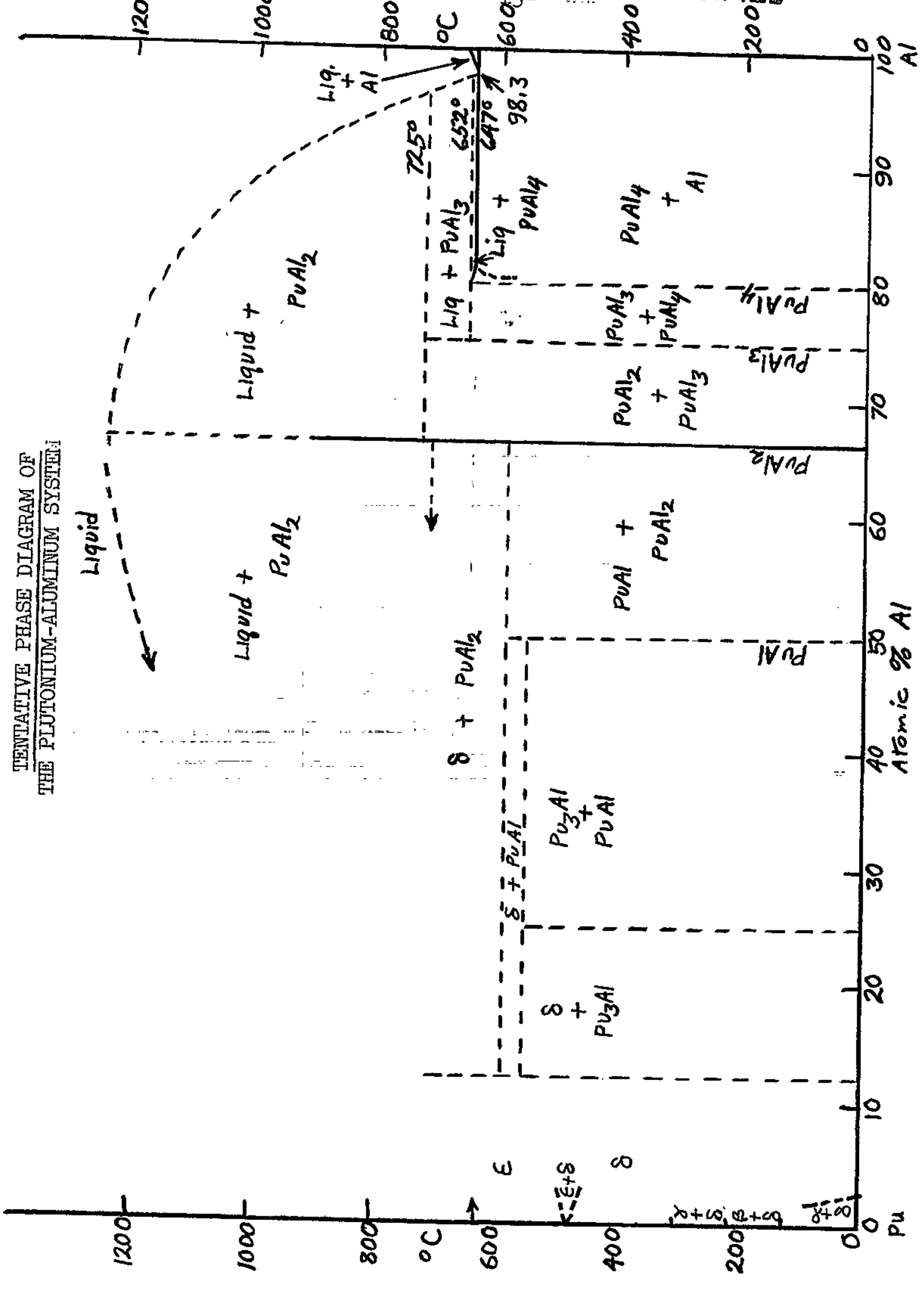

FIGURE 3

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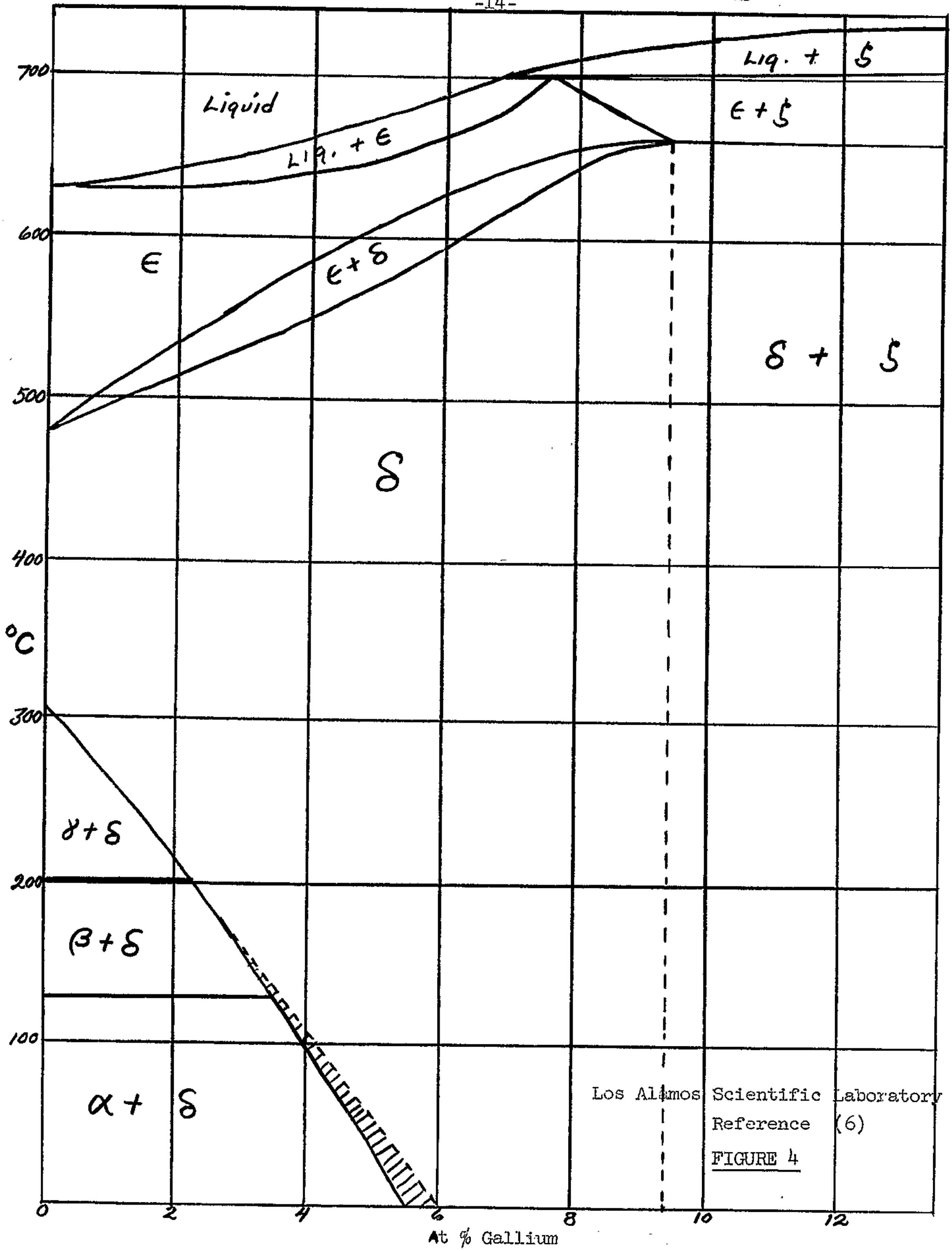

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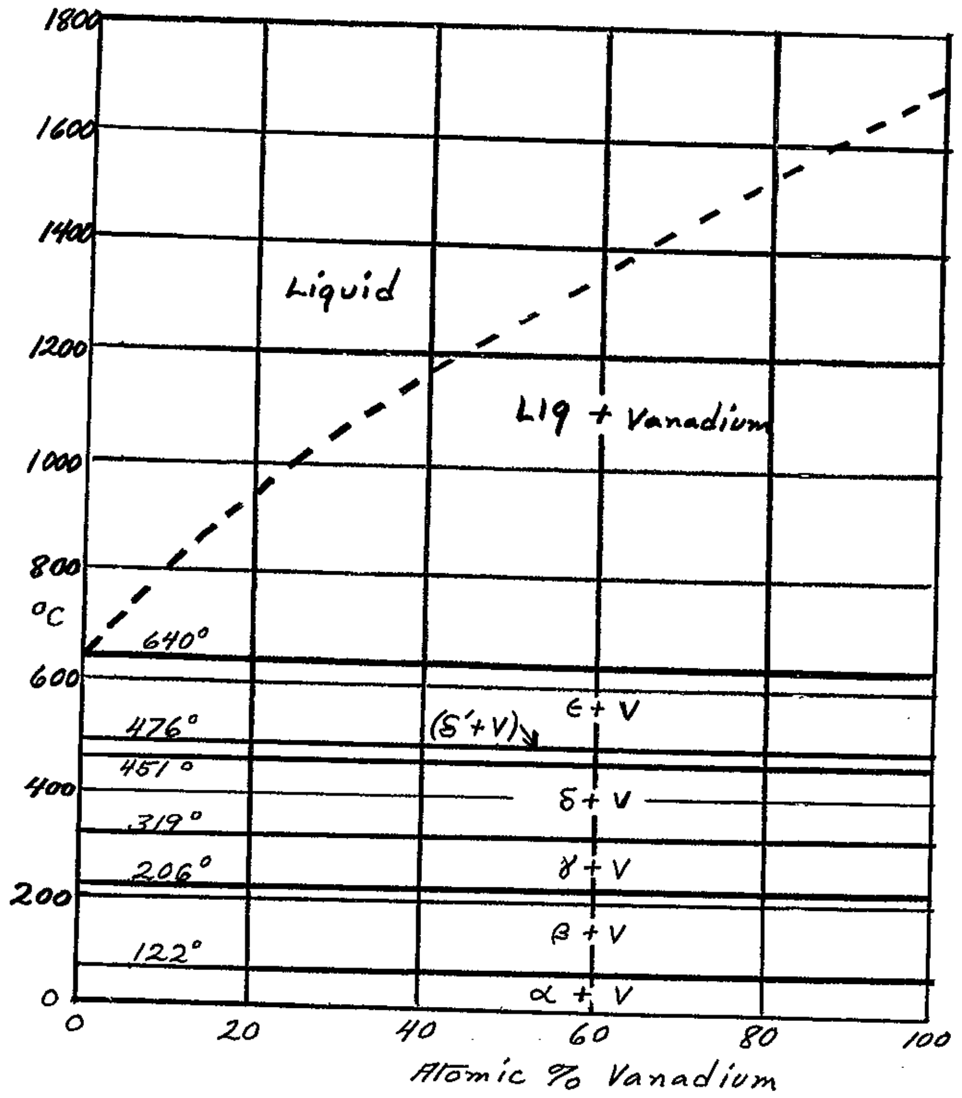
Reference (6)

FIGURE 4



Reference (1)

FIGURE 5

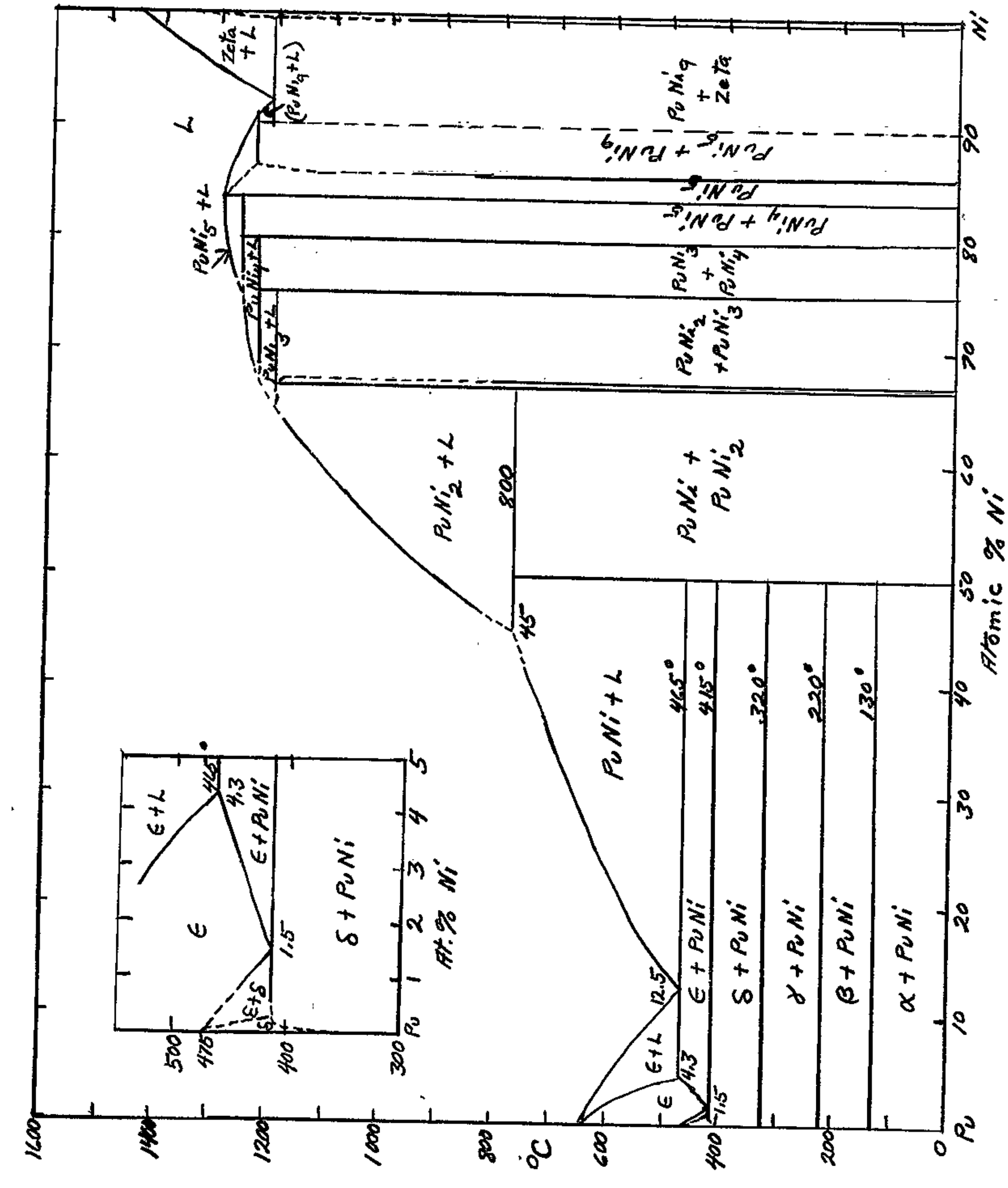


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FIGURE 6

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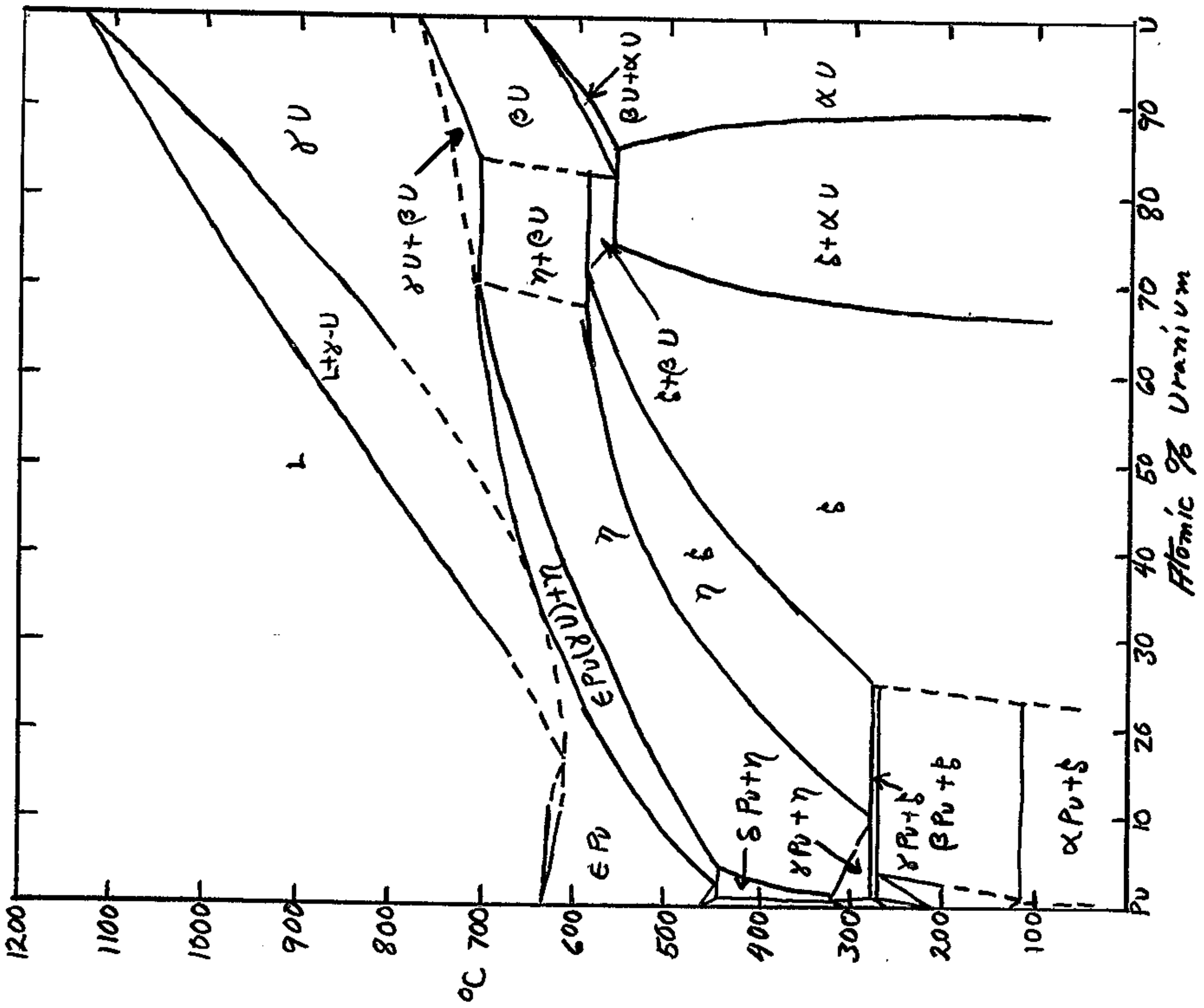

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Reference (8)

FIGURE 7

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