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A STUDY OF THE KINETIC AND MECHANICAL PROPERTIES OF A STABILIZED BETA PHASE PLUTONIUM ALLOY

A Thesis

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in the

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by

John Maurice Taylor

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John Maurice Taylor was born in Kellogg, Idaho on April 21, 1932. He attended the Kellogg High School and was graduated in 1950. In the fall of 1950 he entered the Brigham Young University and in 1955 was graduated with a Bachelor of Science degree in Chemistry. In the summer of 1955 he was employed by the General Electric Company at Hanford, Washington. In 1957 he entered the General Electric Nuclear Engineering Program affiliating with the University of Idaho. In June of 1962 he completed the requirements for the Master of Science degree, of which this thesis is a part.

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ABSTRACT

The elements Sn, Mg, Ti, Zr, Hf, Si, Cd, and U were alloyed with plutonium to determine their effect on stabilization of the beta phase. Zirconium was found to be the most effective. Kinetic studies were conducted on Pu-Zr alloys having 0.24, 0.65, and 1.7 a/o Zr contents. The beta to alpha transformation in the Pu-Zr alloys was somewhat similar to that in unalloyed plutonium. The main difference being that Time, Temperature, Transformation curves of the alloys were shifted to the right as the zirconium concentration increased.

It was determined that the amount of beta phase retained at room temperature increased as the zirconium concentration increased. The minimum amount of Zr needed to stabilize the beta phase of plutonium at room temperature was determined to be 2.0 a/o. A 2.4 a/o Zr addition stabilized the beta phase of plutonium at atmospheric pressure from room temperature to 265 C.

During mechanical property testing it was determined that the stabilized beta Pu-2.4 a/o Zr alloy was brittle from room temperature to 230 C. Both tensile and compression strengths of the beta stabilized alloy were greater than the unalloyed beta phase of plutonium.

A brittle-ductile transition was noted between 130 and 180 C on the Pu-0.65 a/o Zr alloy.

Age hardening studies were conducted at room temperature with a Pu-2.4 a/o Zr alloy. A slight aging effect was noted due to the precipitation of an intermetallic compound.

Compression tests on the Pu-2.4 a/o Zr alloy indicated that below 80 C the stabilized beta phase started to transform to alpha phase during the application of compressive loads.

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CHAPTER I

INTRODUCTION

Plutonium metal is unique in that it is characterized by six solid allotropic forms. In accordance with conventional practice these solid allotropes are called α, β, γ, δ, δ', and ϵ. As shown in Table I the stable temperature ranges of any of the phases are not extensive.

TABLE I

CRYSTAL STRUCTURE OF THE PLUTONIUM ALLOTROPES

Allotrope	Range of Stability (°C)	Crystal Lattice	No. of Atoms per Unit Cell	X-ray Density (g/cc)
Alpha	Below 122	Monoclinic	16	19.82
Beta	122-206	Monoclinic	34	17.70
Gamma	206-319	Orthorhombic Face Centered	8	17.14
Delta	319-452	Cubic Face Centered	4	15.92
Delta Prime	452-480	Tetragonal Body Centered	2	16.00
Epsilon	480-640	Cubic Body Centered	2	16.51

As expected, a metal with these characteristics has rather limited engineering application. Consequently it would be very desirable to stabilize a high temperature allotrope of plutonium so that a single phase would exist over a wide temperature range.

Since the factors which influence the stability of one phase with respect to another are poorly understood even for such common metals as iron and zirconium which possess only two solid allotropes, it is difficult to predict which elements are likely to cause stabilization of a particular allotrope of plutonium. Some idea, however, as to what elements might stabilize a high temperature phase of plutonium could be obtained from a study of the existing equilibrium diagrams.[1,2] These indicate that both the beta and delta phases of plutonium can be stabilized at room temperature by proper alloy additions. In unalloyed plutonium the delta phase is characterized by a cubic structure, high ductility and very low strength.[3] The beta phase has a monoclinic structure and is considerably stronger and more ductile than the delta phase.[4]

A good deal of effort has been expended in studying the mechanical properties and the stability of several of the delta stabilized alloy systems.[5,6] Although the delta phase is stabilized up to 450 C, it is limited in application because of its low strength. Since no beta information was found an investigation of the stabilized phase of beta plutonium relative to its range of stability and mechanical

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[1]F. W. Schonfeld, E. M. Cramer, W. N. Miner, F. H. Ellinger and A. S. Coffinberry, "Plutonium Constitutional Diagrams," Progress in Nuclear Engineering, Vol. 2, Ser. 5, Pergamon Press, New York, 1959, pp 579-99.

[2]A. A. Bochvar, S. T. Konobeevskii, V. I. Kutaitsev, T. S. Menshikova and N. T. Chebotarev, "Interaction Between Plutonium and other Metals in Connection With Their Arrangement in Mendeleev's Periodic Table," Second United Nations International Conference on Peaceful Uses of Atomic Energy, Geneva, 1958, Paper 2197.

properties was considered necessary because of its superior strength, ductility and greater density.

I. PURPOSE OF STUDY

This study was initiated to determine what elements will stabilize the beta phase of plutonium. In addition studies of beta phase stability as a function of alloy addition and mechanical property determination on a stabilized beta phase were to be conducted.

II. LITERATURE SURVEY

Early work at Los Alamos[7] indicated that when zirconium in amounts of 5 a/o was added to plutonium the x-ray powder patterns showed the alloy to be beta plutonium. Later that year[8] they reported that additions of 3 a/o Zr gave an x-ray pattern of beta plutonium at room temperature. Russian investigators[9] also reported that the beta phase of plutonium could be retained* at room temperature by adding 3 to 17 w/o of uranium to plutonium and quenching.

Zachariasen[10] in his work on determining the unit cell of beta plutonium used 0.5 a/o zirconium to stabilize the beta phase.

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*A beta retainer is an element which when alloyed with plutonium will retain beta phase plutonium upon quenching down to room temperature.

[3] H. R. Gardner and I. B. Mann, "Mechanical Property and Formability Studies of Unalloyed Plutonium," Plutonium 1960, 513-570 and Proceedings of the Second International Conference on Plutonium, (Grenoble, France) Memoire 36. April 1960.

Preliminary phase diagram[11] reported by the British on the Pu-Ti alloy system indicate that β Pu and α Ti exists from 0 to 270 C when 0.5 to 98 a/o Ti is added to plutonium.

With this information these elements plus others having similar crystal structures, valencies, metallic radii and electronegativities could be added to plutonium and their effect on beta stabilization evaluated.

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[4] Ibid., p. 524.

[5] LAB-CM-B-11-3094, p. 33, First Annual Plutonium Metallurgy Information Meeting, February 1957.

[6] R. O. Elliot and K. A. Gschneidner, Jr., "Behavior of Some Delta-Stabilized Plutonium Alloys at High Pressures," Extractive and Physical Metallurgy of Plutonium and Its Alloys, pp. 243-262. New York: Interscience Publishers, 1960.

[7] Materials and Information Branch, Division of Research, Washington D.C., Excerpts from the Progress Reports, Los Alamos Scientific Laboratory, p. 7, WASH-59, (Confidential) April 20, 1951.

[8] Materials and Information Branch, Division of Research, Washington D.C., Excerpts from the Progress Reports, Los Alamos Scientific Laboratory, p. 7, WASH-60, (Declassified) May 20, 1951.

[9] Bochvar, op. cit., p. 192

[10] W. H. Zachariasen and F. H. Ellinger, Acta Crystallograph, Vol. 12, 175, March 1959.

[11] D. M. Poole, et al. "Phase Diagrams of Some Plutonium Alloy Systems," Plutonium 1960, p. 278 and Proceedings of the Second International Conference on Plutonium (Grenoble, France), Memoire 6 April, 1960.

CHAPTER II

EXPERIMENTAL WORK

Experimental Material and Alloy Makeup. Special precautions must be used in the handling of plutonium and its alloys. Plutonium is a fissionable material and an alpha emitter. Because it is fissionable care must be exercised to prevent the assembly of a critical mass. Due to its radioactivity extreme caution must be used in making certain that the operator is adequately protected from exposure and possible ingestion of plutonium. The alpha particles of plutonium-239 only have a penetrating power of 3.68 cm in air and can be absorbed completely by a piece of paper. Thus performing work with the metal can be done safely within a glove box enclosure maintained at a negative pressure. All work with plutonium at Hanford meets the safety requirements established by the Atomic Energy Commission.

The plutonium used in this study was produced by the bomb reduction of plutonium tetrafluoride with calcium. High purity buttons were produced by this metallothermic reaction. Prior to alloying the plutonium buttons were cast into 1/2-inch diameter rods to improve the metal quality and to produce a more convenient form from which to make up alloy stock. See Appendix for chemical analysis data.

When molten, plutonium is highly reactive with most elements. For this reason great care must be taken in selecting the proper crucible and mold materials. All melting must be done under vacuum or in an inert atmosphere because of the high oxidation rate of plutonium.

Tantalum crucibles heated in air to about 500 C until a hard black oxide coat is established were found to be nonreactive with the plutonium alloys studied. All charges were poured into heated graphite molds coated with MgO and slowly cooled to room temperature. No reaction was evident between the alloys and graphite or MgO. A typical mold is illustrated in Figure 1.

Several different procedures were used to make up the various alloys. The first alloy made was Pu-U. No special casting technique was required to make up this alloy. The Pu-U alloy was made in a vacuum resistance furnace. The charge was loaded in a tantalum crucible and held molten at 1200 C (this exceeded the melting points of both metals) for 20 minutes and then poured into a graphite mold. Because of the high melting points of Ti, Zr, Hf, and Si it was necessary to first make master alloy buttons by arc fusion. The master alloy buttons could then be charged with the proper amount of plutonium in the vacuum resistance furnace and cast as before. Since the boiling points of Cd and Mg are low, substantial amounts are lost by vaporization when charges containing them are heated in vacuo to the 640 C melting point of Pu. To reduce evaporation the charges were melted in helium at 2 atmospheres of pressure.

To insure good homogeneity in the make up of all but Cd and Mg alloys each alloy charge was melted and cast at least four times.

The Pu-Zr alloys were the only alloys chemically analyzed. The reason for this will be explained below. The method of analysis for zirconium is described by Bricker and Waterbury.[1]

FIGURE 1

GRAPHITE MOLD AND AS CAST 0.250
INCH DIAMETER TENSILE SPECIMEN

I. ALLOY EVALUATION

To avoid altering the nuclear properties of pure plutonium appreciably, the introduction of foreign atoms was held to a maximum of 3 a/o except with uranium, U^{235} being fissionable. The beta stabilizing effects of the alloying elements were evaluated by density and x-ray diffraction measurements, Table II. The data indicate that Ti, Zr, and Hf retain varying amounts of beta phase depending on the amount added. The other alloys produced mixtures of alpha phase plutonium and intermetallic compounds in which even beta heat treatments and quenching to room temperature would not produce retained beta.

From the above studies it was found that a zirconium addition was the most effective in retaining the beta phase at room temperature. Consequently, the kinetics and mechanical properties of the Pu-Zr alloy system will be the only one discussed in this thesis.

II. KINETIC DETERMINATION TECHNIQUE

The transformation kinetics of Pu-Zr alloys were studied using a fluid displacement technique where the volume change of a plutonium specimen was determined as it transformed. Wafers 1.45 inches in diameter and 0.030 to 0.070 inch thick were prepared. A hole was

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[1] C. E. Bricker, and G. R. Waterbury, "Separation and Determination of Microgram Quantities of Zirconium," Analytical Chemistry, Vol. 29, 1957, pp. 558-561.

TABLE II

A COMPARISON OF VARIOUS BINARY ALLOYS AS TO THEIR ABILITY TO RETAIN BETA PHASE PLUTONIUM AT ROOM TEMPERATURE

Alloy Composition	As Cast Density (g/cc)	Plutonium Phases Present
Pu - 2 a/o Sn	18.92	α
Pu - 3 a/o Mg	18.79	α
Pu - 3 a/o Cd	18.85	$\alpha + \delta$
Pu - 1 a/o Si	16.35	$\alpha + \delta$
Pu - 1 a/o Ti	19.25	$\alpha + \beta$
Pu - 2 a/o Ti	18.59	$\alpha + \beta$
Pu - 3 a/o Ti	17.49	$\alpha + \beta$
Pu - 0.24 a/o Zr	19.56	α
Pu - 0.65 a/o Zr	19.33	$\alpha + \beta$
Pu - 1.7 a/o Zr	17.56	$\alpha + \beta$ ($\alpha < 2\%$)
Pu - 2.4 a/o Zr	17.38	β
Pu - 1 a/o Hf	19.11	$\alpha + \beta$
Pu - 2 a/o Hf	17.25	$\alpha + \beta + \delta$
Pu - 3 w/o U	19.02	α
Pu - 7 w/o U	18.72	α
Pu - 15 w/o U	18.53	α

drilled near the edge the wafer so that a wire could be attached. The wafer plus wire could then be suspended in solution from the beam of an analytical balance, Figure 2. To avoid the effect of composition variations kinetic studies were conducted on samples from the same casting heat.

Because of the oxidation of Pu-Zr alloys in air at elevated temperatures special liquid baths and glove box atmospheres had to be used. Degassed Dow Corning 760 silicone oil did not react with the alloys at temperatures up to 200 C, when used in an inert atmosphere. An argon atmosphere was provided by evacuating a glove box, Figure 3, and back filling with argon. It was necessary to use carbon tetrachloride and dry ice at -22 C as the oil was too viscous. Acetone and dry ice was used as the quench medium at -40 C and -80 C.

Weight change versus temperature curves were obtained from 26 C to 300 C for each alloy composition.

Isothermal reaction curves for the beta to alpha transformation were obtained by solution heat treating at 175 C for one-half hour, quenching to temperatures between -80 C and +75 C and holding until no further apparent transformation as indicated by weight change. The fraction transformed was plotted as a function of the time of transformation and was determined from the following relationship.

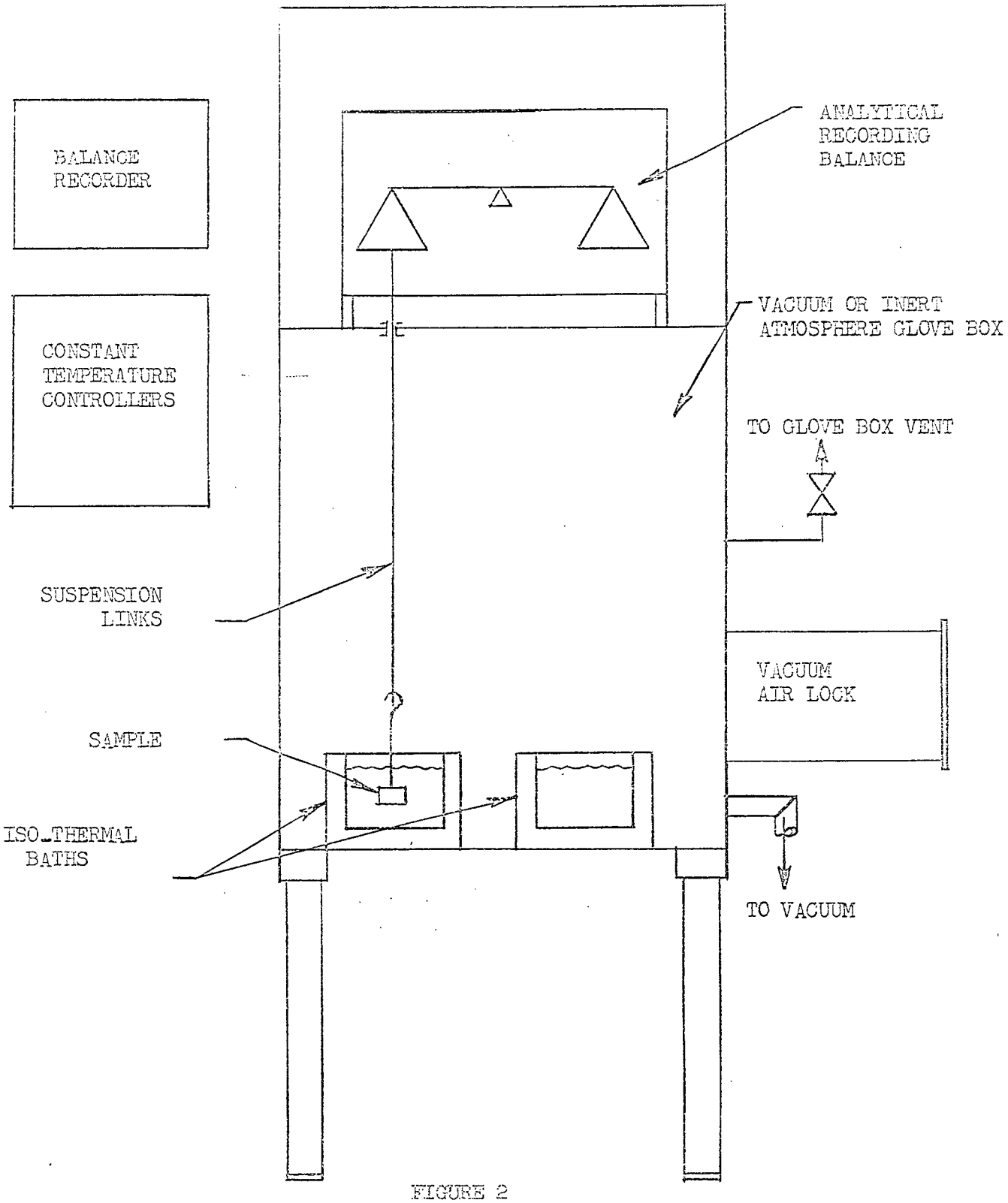

FIGURE 2

SCHEMATIC DIAGRAM OF GLOVED BOX AUTOMATIC
RECORDING BALANCE USED IN KINETIC STUDIES

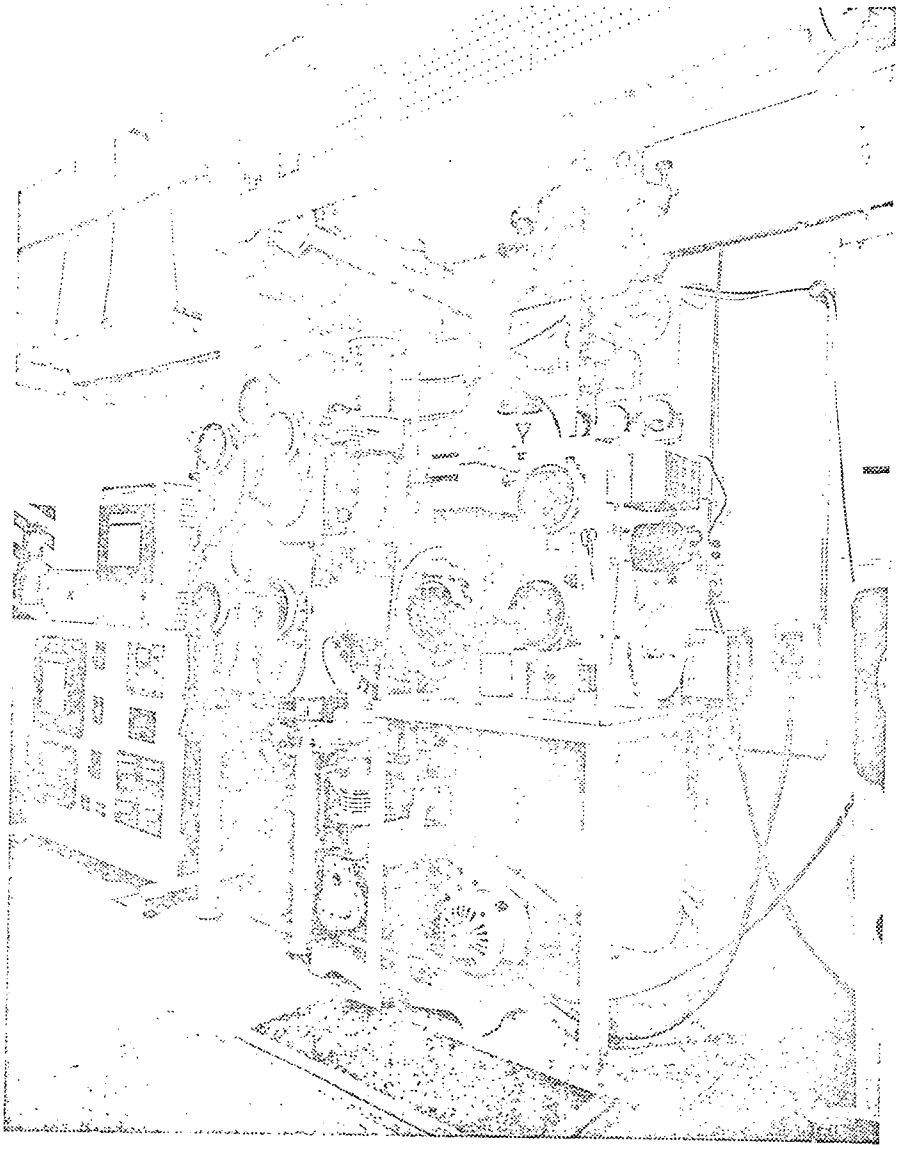

FIGURE 3

EVACUABLE GLOVE BOX WITH ATTENDING EQUIPMENT
USED IN KINETICS WORK WITH Pu-Zr ALLOYS

$$f(t) = \frac{W_t - W_e}{W_f - W_e}$$

W_t = weight in solution at any time

W_e = weight after specimen has come to temperature in isothermal bath.

W_f = weight of specimen at holding temperature after being cold treated at -20 C.

It should be pointed out that W_f does not necessarily indicate that the transformation was complete. There could still have been beta phase present. However, after the cold treatment at -20 C and reweighing at the holding temperature no weight change or density change was observed after many hours. Therefore, the transformation was considered complete. The time elapsed while transferring the sample from the heat treating bath to the quench bath and then allowing the recording balance to stabilize was approximately ten seconds.

III. MECHANICAL TESTING TECHNIQUES

Mechanical property tests were conducted on 0.250 inch diameter tensile specimens, Figure 4 and 0.500 inch diameter, 0.750 inch long compression specimens, Figure 5. The test specimen designs had been previously evaluated on unalloyed plutonium and found to be satisfactory.[2] All specimens were tested on a 440,000 pound Baldwin-Lima-Hamilton Universal Testing Machine equipped with a glove box, Figure 6.

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[2]Gardner, op. cit., pp. 519, 534.

At room temperature a Baldwin Separable Averaging Microformer Extensometer was used with the tensile specimens to obtain a stress-strain curve. From the curve, Figure 7, 0.01 per cent yield strength modulus of elasticity and elongation data could be determined. A one-inch gage length was used as a basis for the elongation data. At elevated temperatures an extensometer was not used since the type available either gouged the specimen or slipped on the gage diameter at elevated temperatures. However, by using a Baldwin Platen Deflectometer a load-platen displacement curve could be obtained, Figure 8.

Since it was not possible to convert from platen displacement to strain in the tensile specimen a stress-strain relationship could not be obtained. Therefore offset yield strength values could not be determined. However, an arbitrary yield strength could be determined from the load-platen displacement curve. The initial portion of the curve is straight, thus the point of departure from the straight line was taken as an indication of the yield strength. This type of yield strength data was also obtained for the compression tests.

Peanut oil was used as a heating medium for the elevated temperature work. A Brown Pyro-Vane Controller was used to automatically control bath temperatures within $\pm$ 2 C during testing. Through vigorous agitation a uniform temperature was assured throughout the bath.

Testing speed, the rate of platen motion, was controlled by a Baldwin Platen Motion Pacer. In all studies on the effect of temperature on tensile properties, the testing speed was 0.015 inch/minute.

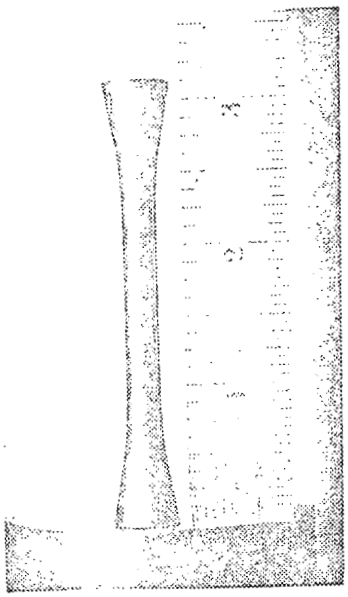

FIGURE 4

MACHINED 0.250 INCH DIAMETER TENSILE SPECIMEN

FIGURE 5

ACTUAL SIZE MACHINED COMPRESSION SPECIMEN

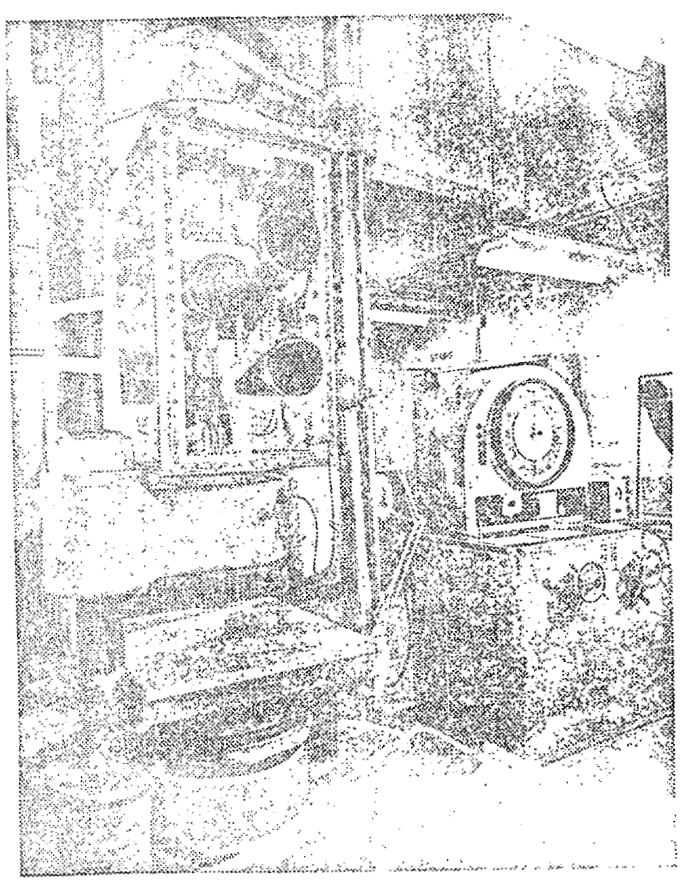

FIGURE 6

REMOVABLE HOOD ARRANGEMENT FOR MECHANICAL PROPERTY TESTING

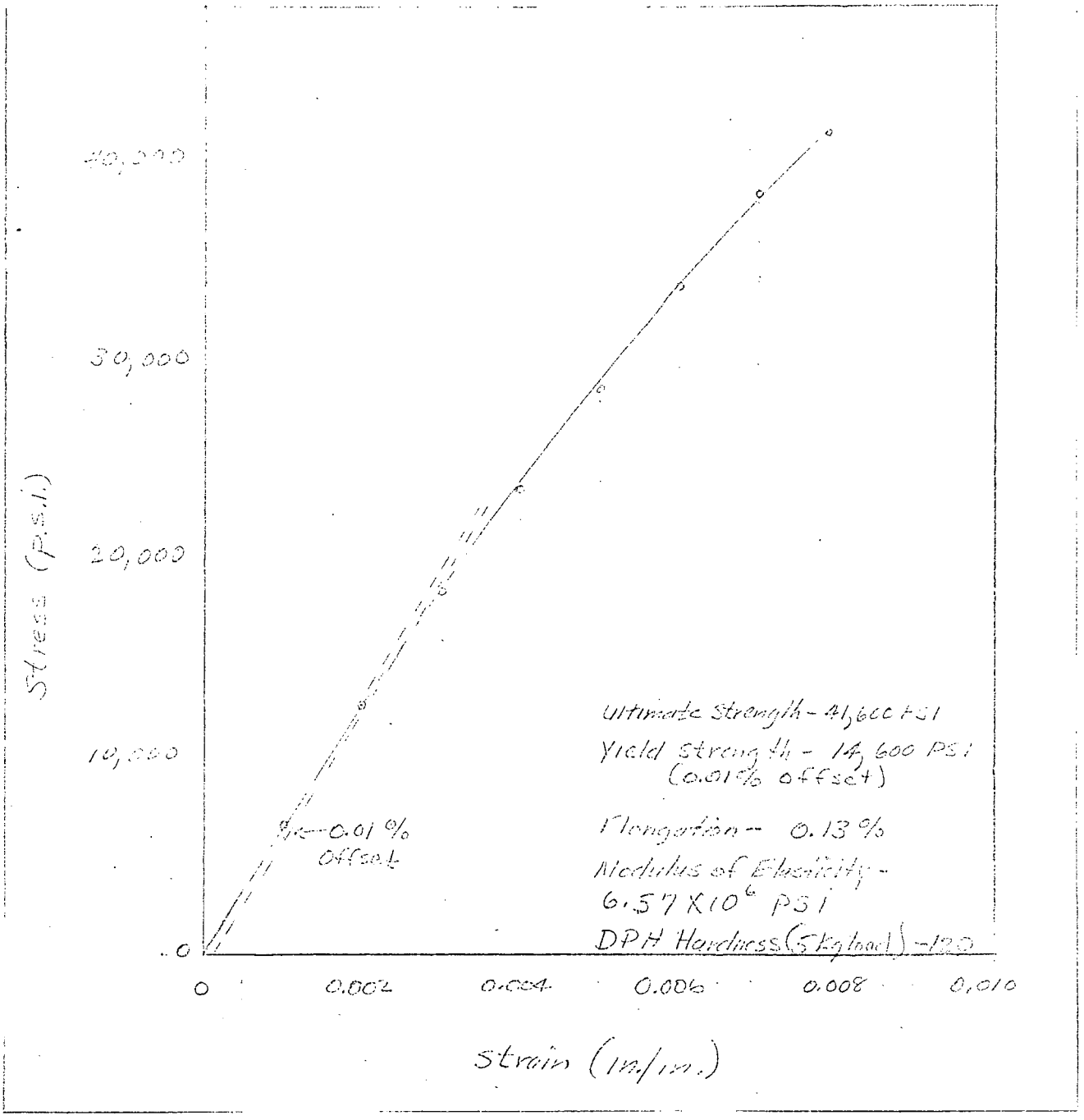

FIGURE 7

ENGINEERING STRESS-STRAIN RELATIONSHIP FOR
BETA STABILIZED Pu-2.4 a/o Zr ALLOY
AT ROOM TEMPERATURE

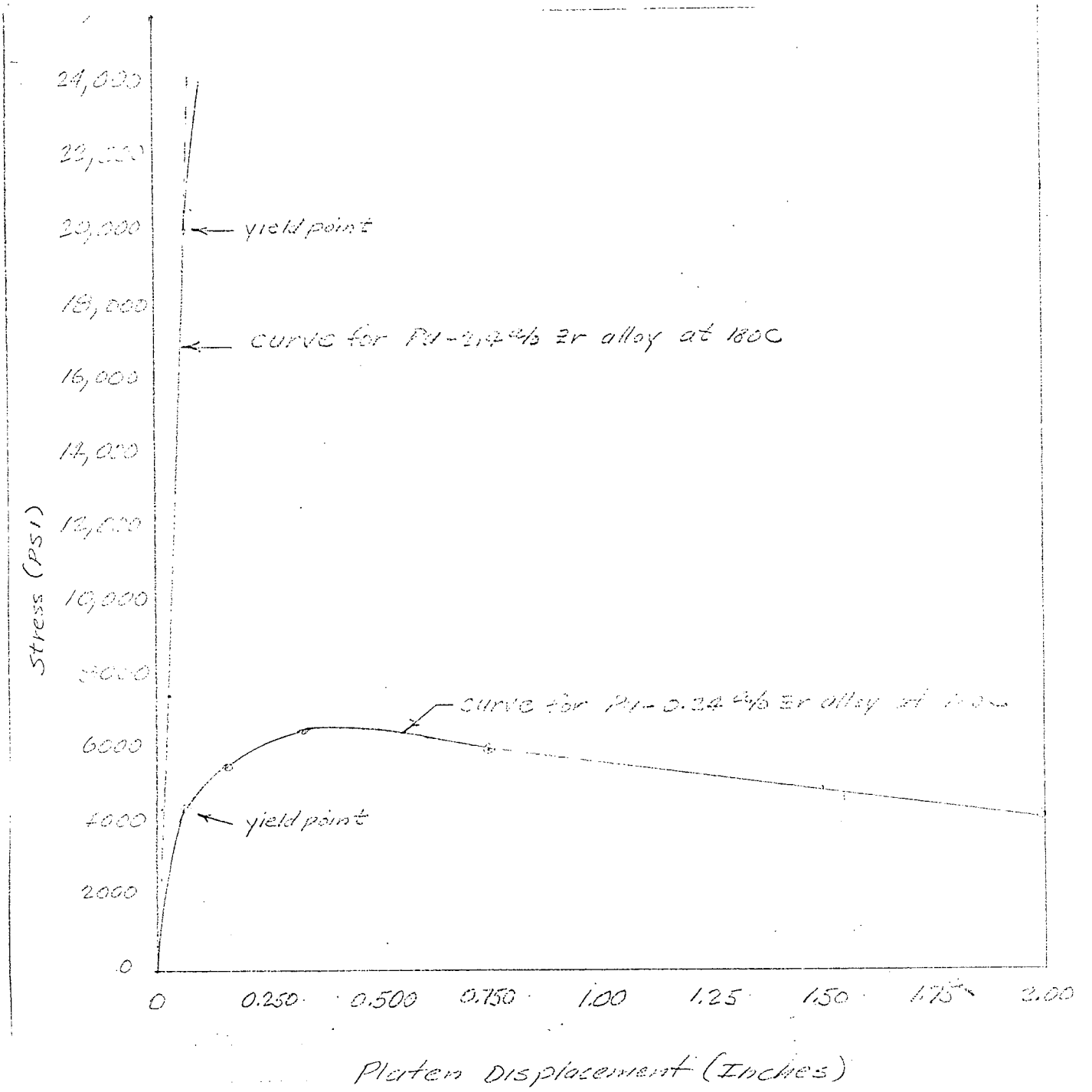

FIGURE 8

STRESS-PLATEN DISPLACEMENT CURVES

IV. METALLOGRAPHIC PROCEDURE

The metallographic specimens were mounted in catalyzed Thalco polyester resin which completely polymerizes below 70 C. Rough grinding was accomplished on wet silicon carbide paper from 180 through 600 grit. The lubricant used was carbon tetrachloride.

Fine polishing was done on Syntron vibratory polishers. A two hour polish on billiard cloth with Linde A emulsion in carbon tetrachloride followed by a five to fifteen minute polishing period on Buehler micro-cloth with Linde A and carbon tetrachloride gave an acceptable as-polished surface finish.

Electropolishing for grain structure was done in a solution of 20 parts tetraphosphoric acid, 30 parts water and 50 parts 2-ethoxyethanol. In the study of grain structure the specimens were electropolished for sixty seconds.

Microhardness data were taken on some of the metallographic specimens. A Tukon Microhardness Tester with a 136 degree diamond pyramid indentor and a 5 kg load were used.

CHAPTER III

RESULTS AND DISCUSSION

Kinetic Studies with the Pu-Zr Alloy. The kinetic studies on the Pu-Zr alloys were initiated to evaluate the stabilizing effect of zirconium and to determine the range of stability of the beta phase. The plutonium-zirconium equilibrium diagram, Figure 9, shows two of the solid state reactions studied. One is the $\beta \rightarrow \alpha$ allotropic transformation at 115 C which occurs below 2 a/o zirconium. The other, also at 115 C, a $\beta \rightarrow \alpha + \theta$ eutectoid reaction occurs between 2 and 3.2 a/o zirconium. The $\beta \rightarrow \alpha$ allotropic transformation was studied with Pu-Zr alloys containing 0.24 and 0.65 a/o zirconium. The eutectoid reaction was investigated using a Pu-1.7 a/o Zr alloy.

Weight changes versus temperature curves were determined for the above alloys, Figures 10, 11, and 12. The curves for the Pu-0.24 Zr and the Pu-0.65 a/o Zr alloys indicate that beta stabilization is incomplete. Since the per cent weight change associated with the alpha to beta transformation is smaller for the Pu-0.65 a/o Zr alloy than for Pu-0.24 a/o Zr it is seen that more beta phase is retained below the beta to alpha transformation as zirconium concentration is increased. The Pu-1.7 a/o Zr alloy retains the beta phase from room temperature to 265 C where transformation to the gamma phase occurs. Thus addition of 1.7 a/o Zr extends the stable range of beta approximately 60 C above the normal beta to gamma transformation at 206 C. Isothermal reaction curves of the characteristic sigmoid shape were

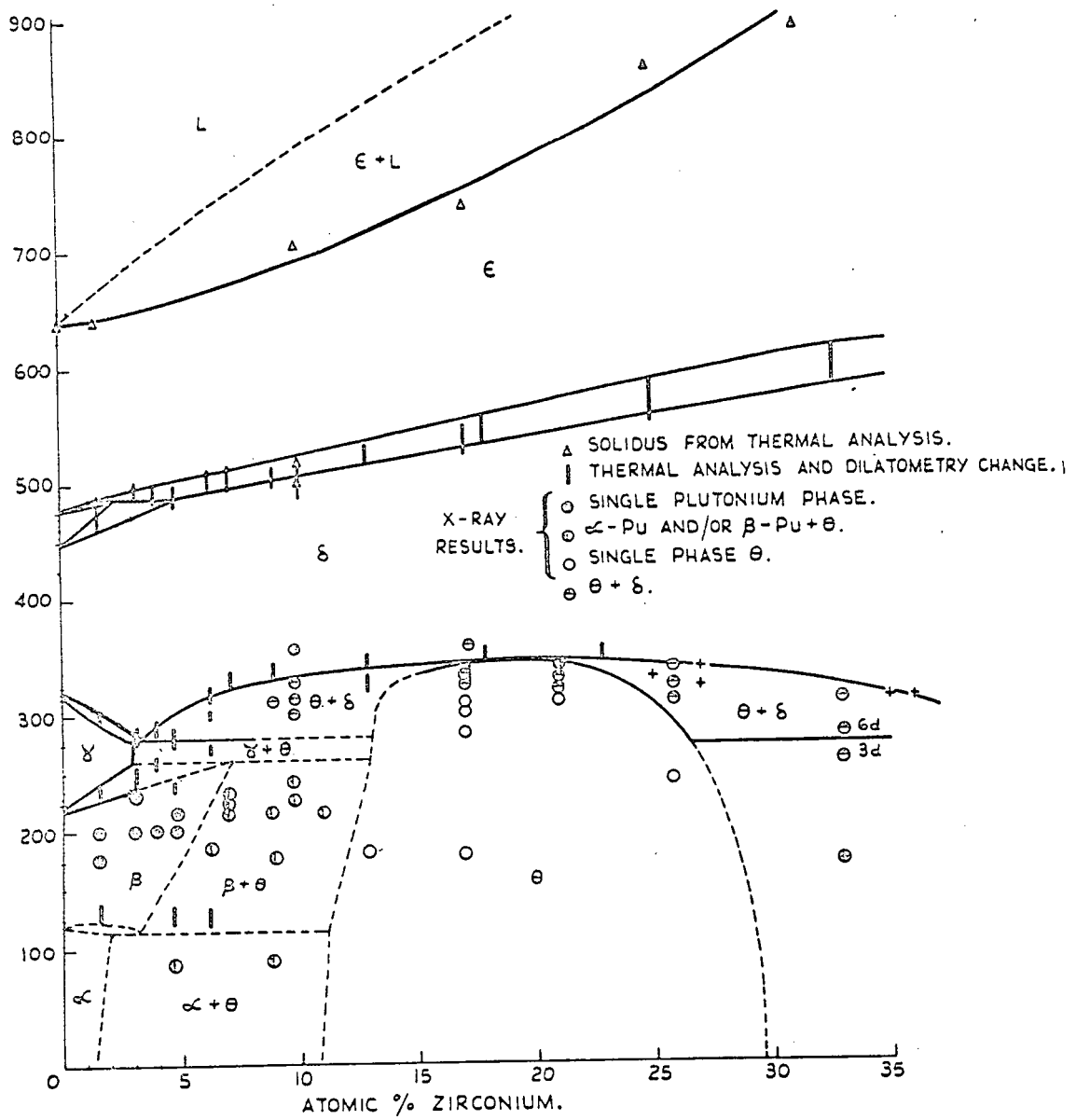

FIGURE 9

PLUTONIUM-ZIRCONIUM EQUILIBRIUM DIAGRAM
0-35 ATOMIC % ZIRCONIUM

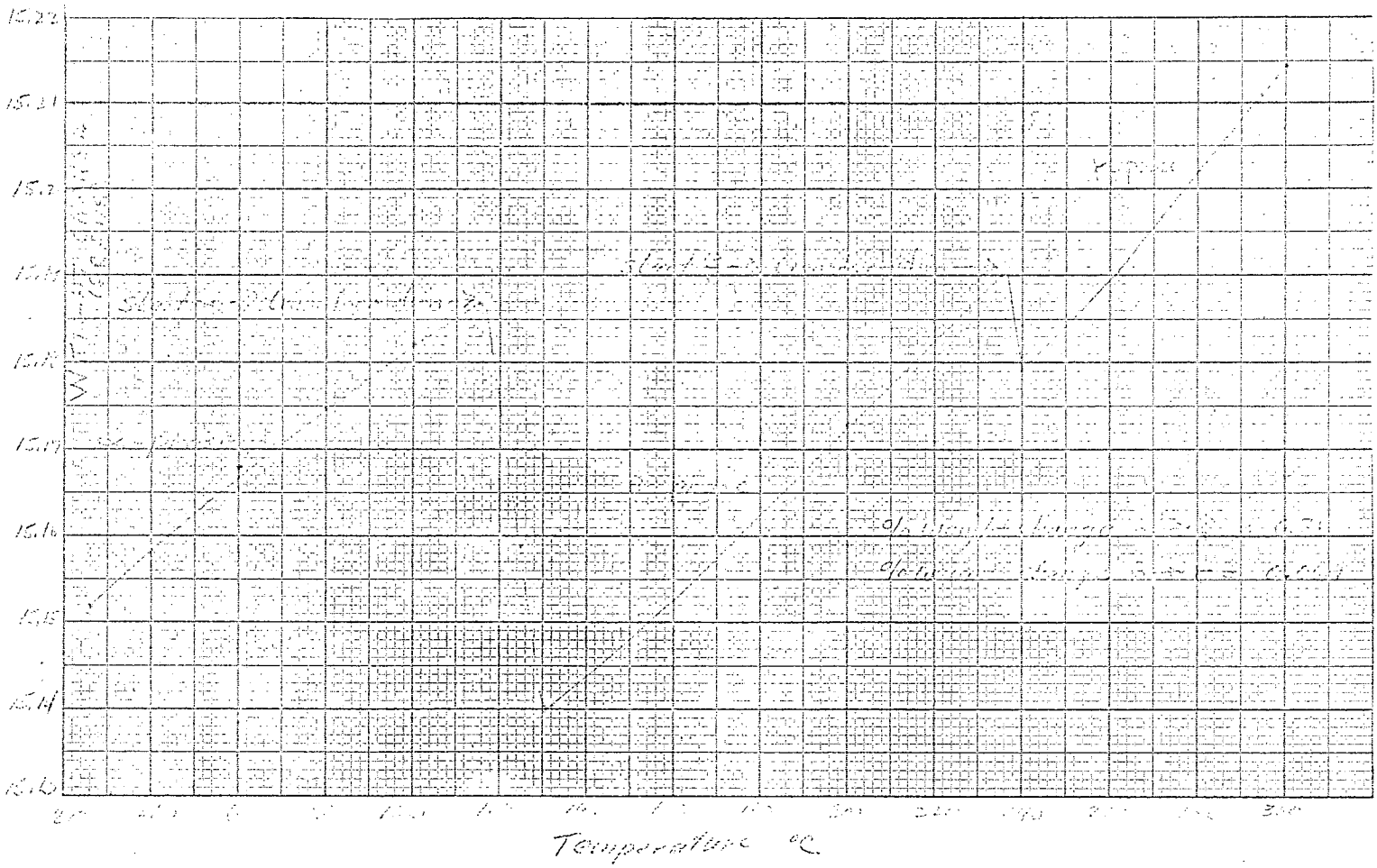

FIGURE 10

WEIGHT CHANGE VERSUS TEMPERATURE CURVE
FOR A Pu-0.24 a/o Zr ALLOY

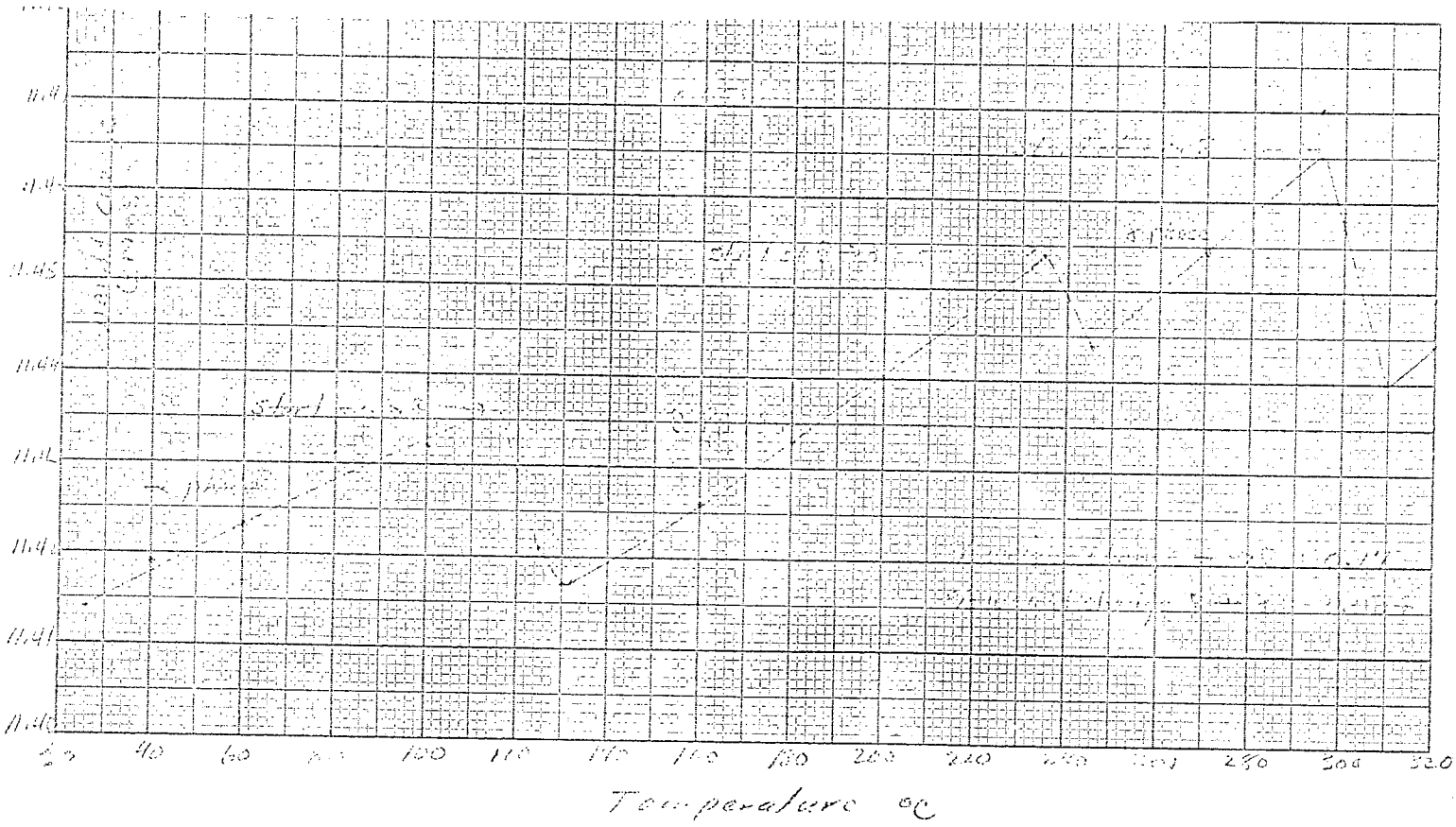

FIGURE 11

WEIGHT CHANGE VERSUS TEMPERATURE CURVE
FOR A Pu-0.65 a/o Zr ALLOY

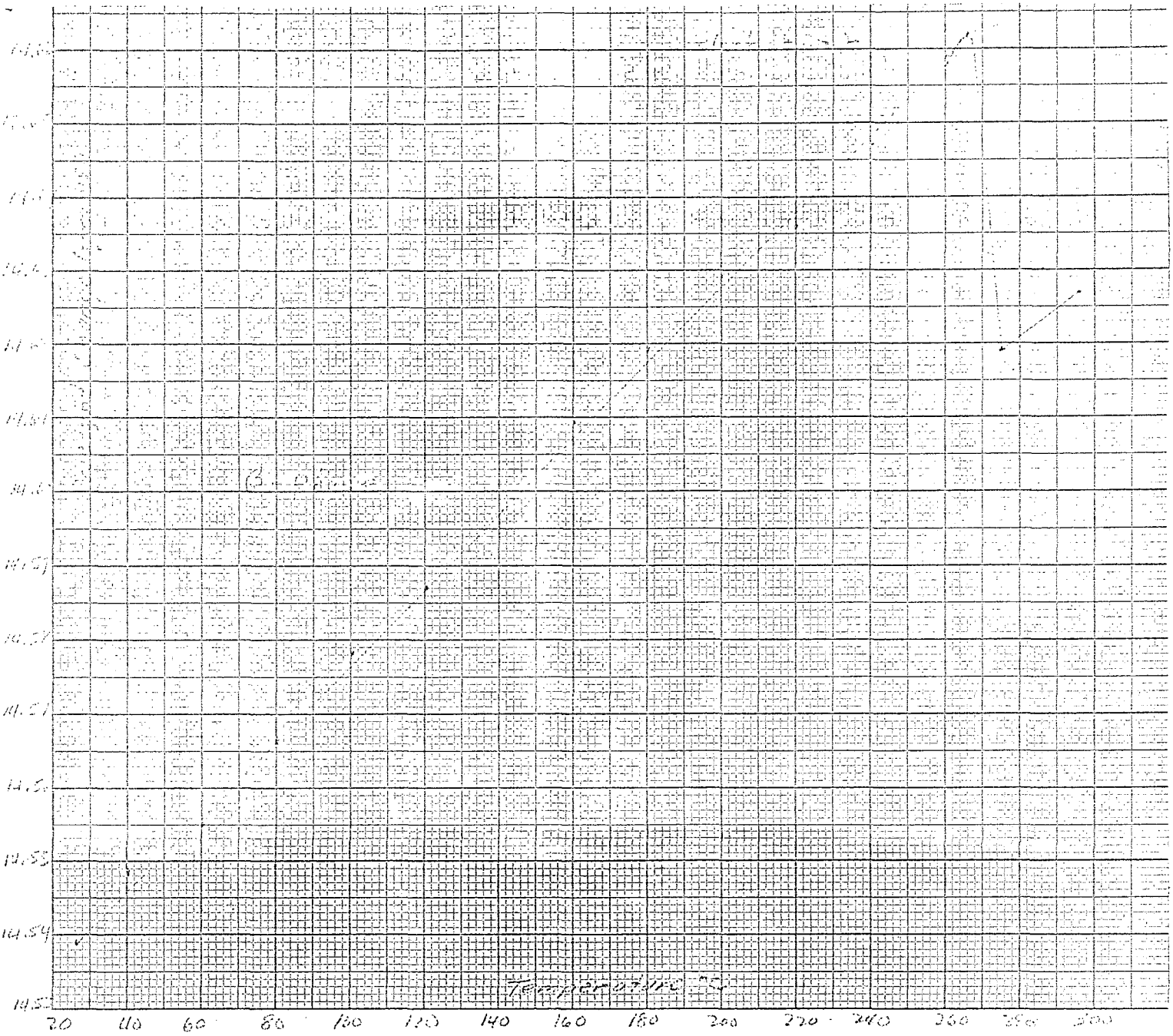

FIGURE 12

WEIGHT CHANGE VERSUS TEMPERATURE CURVE
FOR A Pu-1.7 a/o Zr ALLOY

obtained for the Pu-0.24 a/o Zr and Pu-0.65 a/o Zr alloy, Figures 13, 14, 15, and 16. The reaction curves for the alloys show that the maximum rate of transformation occurs at about -20 C. Above and below this temperature the reaction rate is slower. The degree of transformation in the alloys is influenced by zirconium concentration and isothermal holding temperature. As zirconium concentration increases lower holding temperatures are required to obtain complete transformation. The athermal nature of the transformation is demonstrated by the fact that the degree of transformation at a particular holding temperature can be increased by lowering the holding temperature. In the Pu-0.24 a/o Zr alloy holding at 71 C results in 40% alpha transformation. Lowering the holding temperature causes further beta to alpha transformation.

The retention of beta phase below the normal $\beta \rightarrow \alpha$ transformation temperature has been observed in kinetic studies on the beta to alpha transformation in unalloyed plutonium.[1,2] The British suggest that while a normal nucleation and growth mechanism is indicated certain athermal characteristics are present.[3]

From the isothermal reaction data typical C-shaped Time-

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[1] R. D. Nelson and I. D. Thomas, "Transformation Kinetics of Plutonium," Proceedings of Second United Nation International Conference on Peaceful Uses of Atomic Energy (Geneva), 6: 170-173 New York: United Nations, 1958.

[2] R. G. Loasby and J. N. Lowe, "Kinetics of the Beta-Alpha Transformation in Plutonium," Plutonium 1960, pp. 3-16, Cleaver-Hume Press Ltd., London, 1961.

[3] Nelson, op. cit., pp. 170-173.

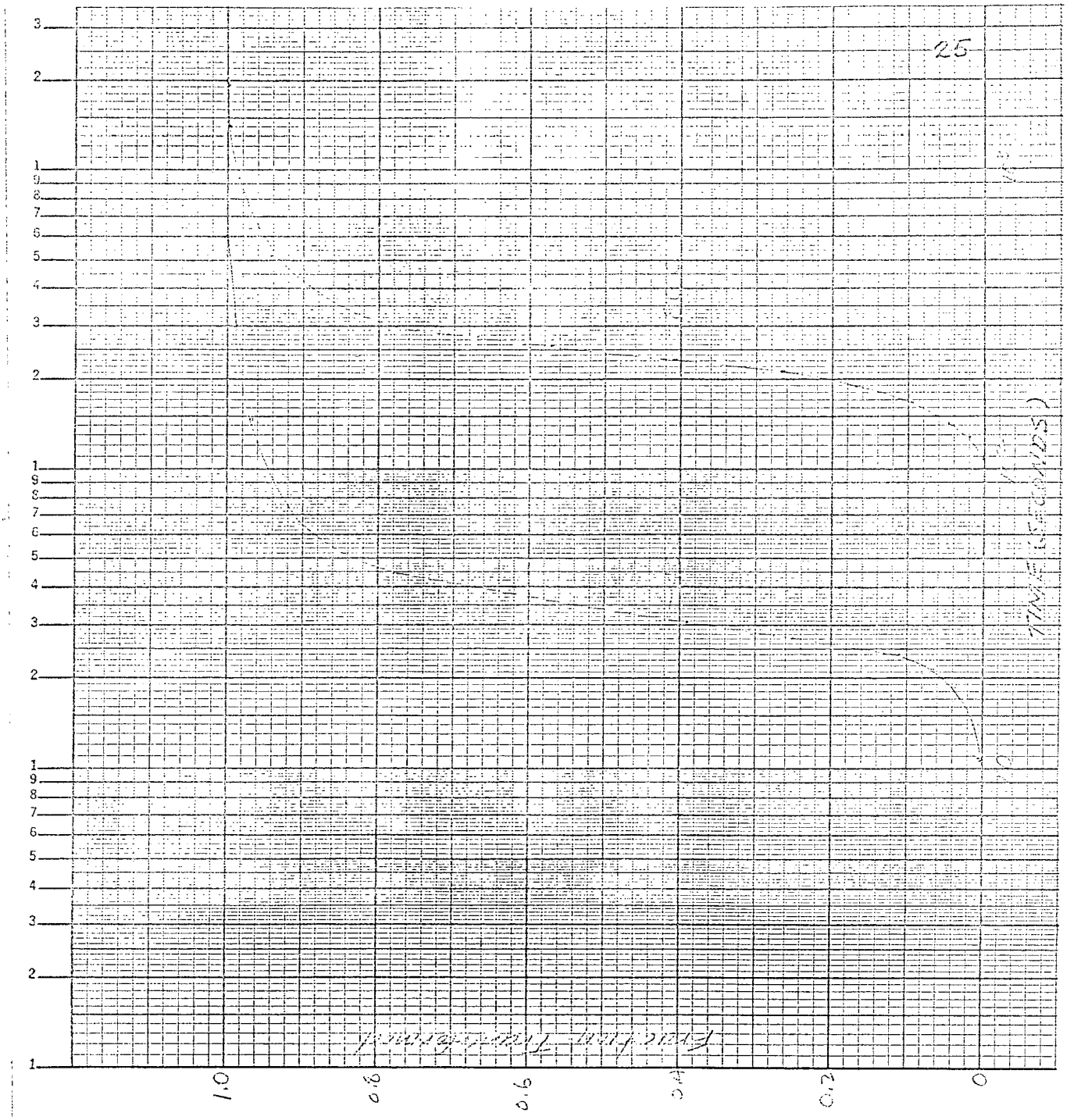


FIGURE 13

ISOTHERMAL REACTION CURVES
FOR Pu-0.24 a/o Zr ALLOY

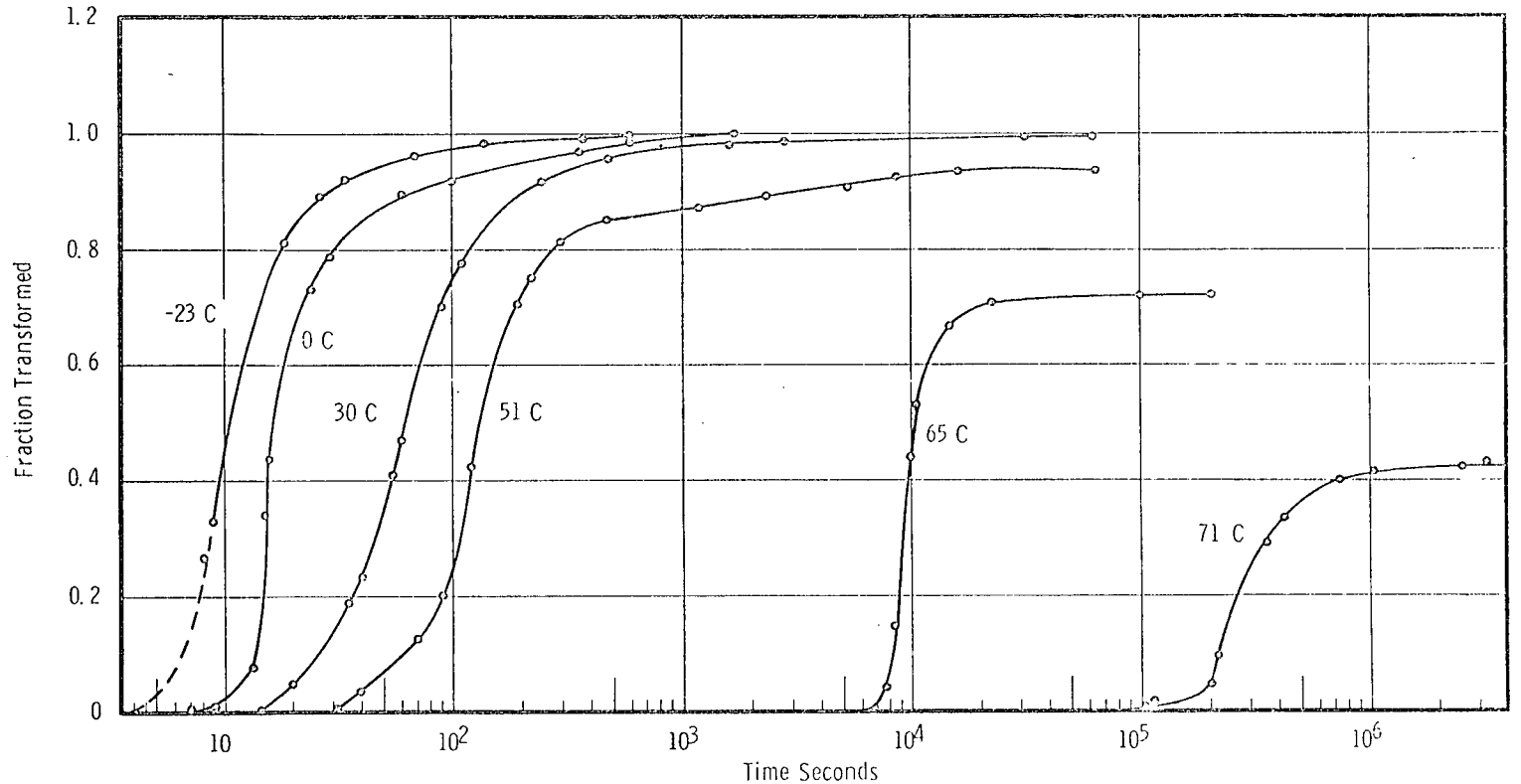


FIGURE 14

ISOTHERMAL REACTION CURVES
FOR Pu-0.24 a/o Zr ALLOY

CLI 037

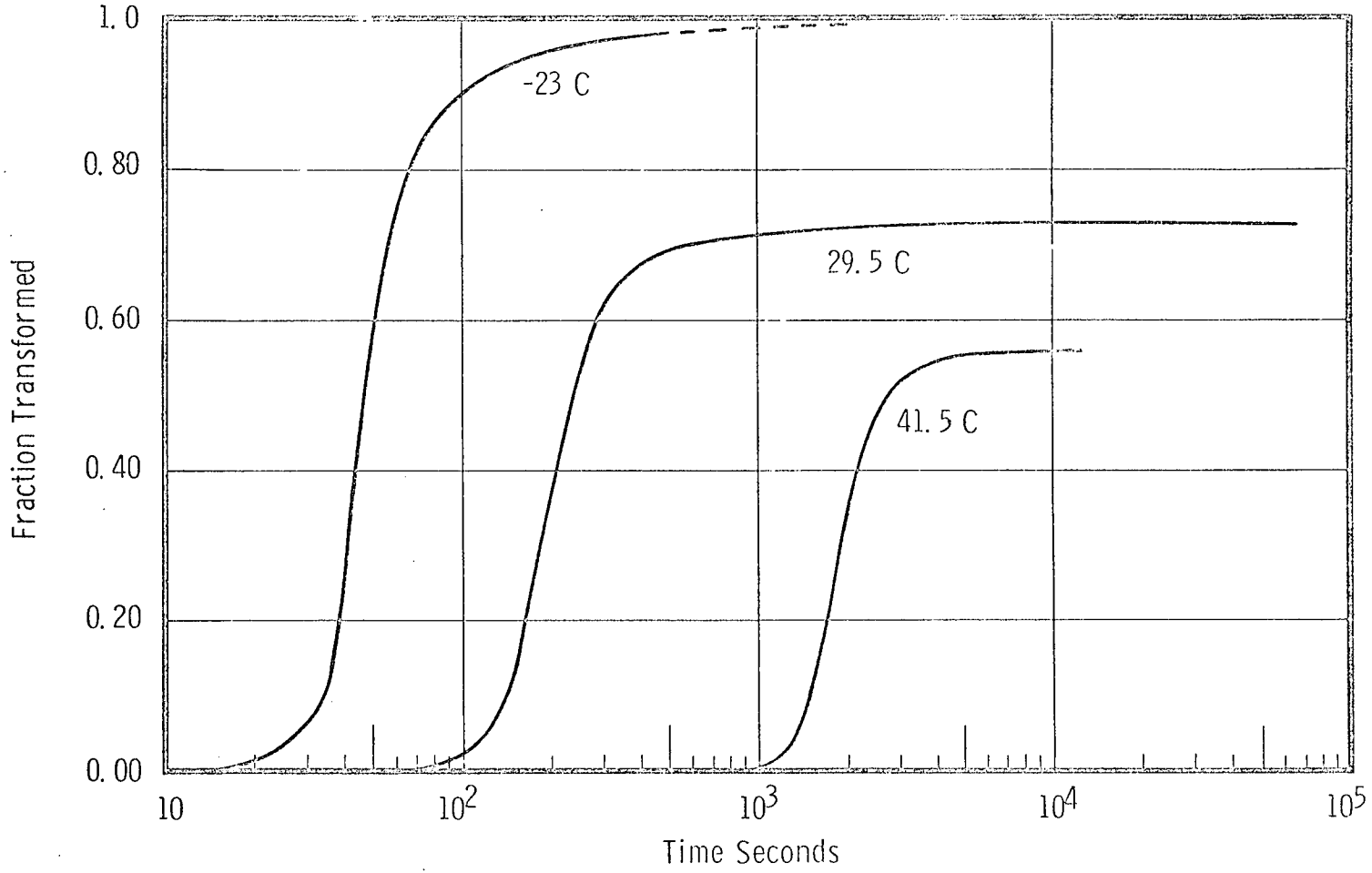


FIGURE 15

ISOTHERMAL REACTION CURVES
FOR Pu-0.65 a/o Zr ALLOY

517 038

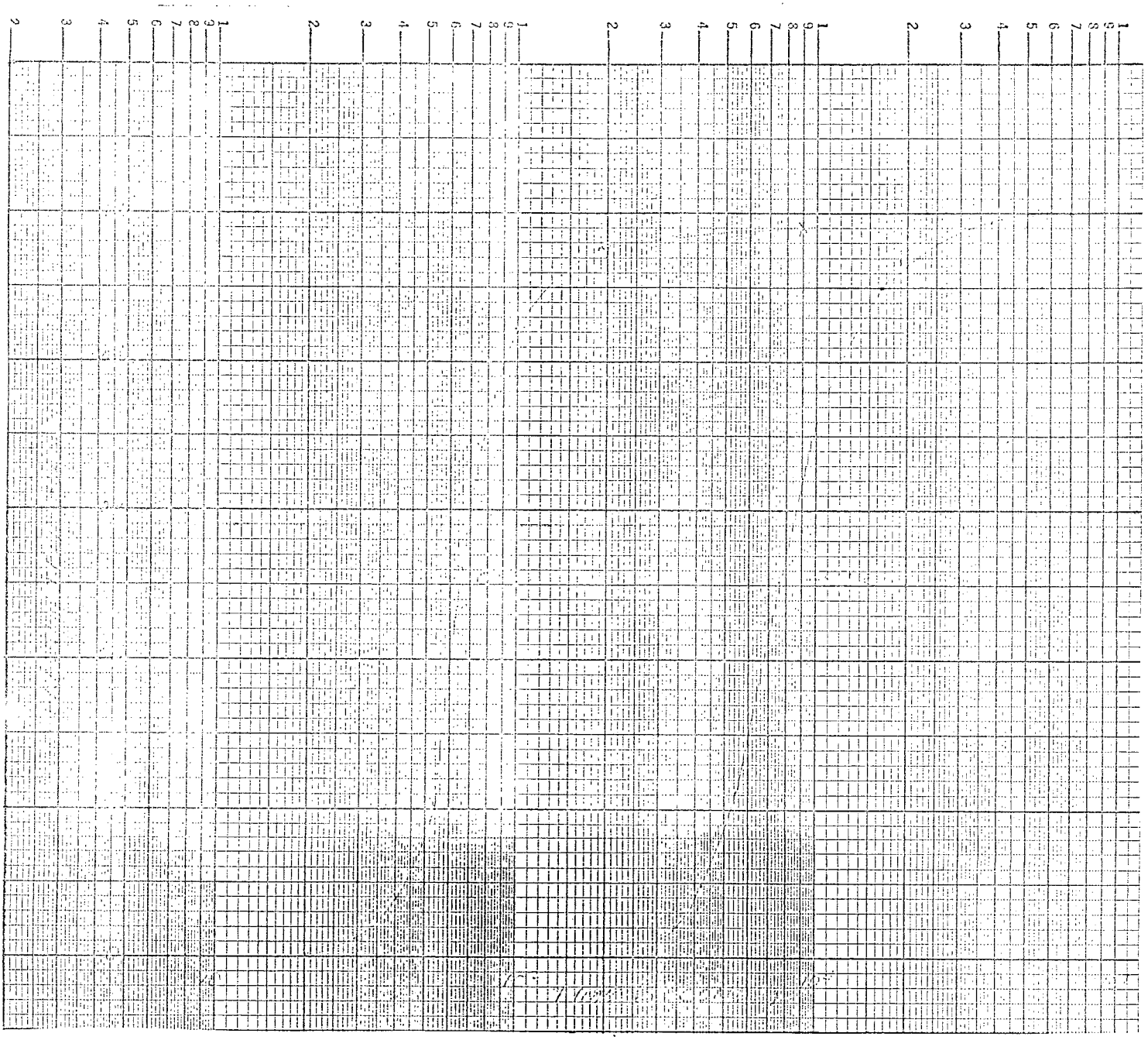


FIGURE 16

ISOTHERMAL REACTION CURVES
FOR Pu-0.65 a/o Zr ALLOY

Temperature-Transformation curves were obtained, Figures 17 and 18. Note that as zirconium concentration is increased the T-T-T curves are shifted to the right indicating a slower transformation rate. In addition, the Pu-0.24 and Pu-0.65 a/o Zr alloys have critical holding temperatures of 80 and 60 C respectively. Above these temperatures the transformation will not occur for long periods of time.

The T-T-T curves for the beta to alpha transformation in the Pu-0.24 and Pu-0.65 a/o Zr alloys are similar to those for unalloyed plutonium. The main difference being the rate of transformation. For comparison T-T-T curves for 50 per cent transformation in unalloyed plutonium and in the Pu-0.24 a/o Zr and Pu-0.65 a/o Zr alloys are presented in Figure 19. The beta to alpha transformation is much more sluggish in the alloys than it is in unalloyed plutonium. At -20 C the 0.24 a/o Zr addition increases the time for 50% transformation by a factor of 2 while 0.65 a/o Zr increases transformation time by a factor of 9.

Because of the sluggishness of the beta to alpha transformation as the zirconium content is increased, only one isothermal reaction curve was obtained on the Pu-1.7 a/o Zr alloy, Figure 20. Since the reaction rate was quite slow densities changes could be determined as a function of time. The reaction is only 17 per cent complete after 70 hours. At complete beta to alpha transformation this alloy would have a density of 19.51 g/cc. After 70 hours a slight increase in density was observed which is attributed to the precipitation of the intermetallic compound θ. The stoichiometric proportions of the

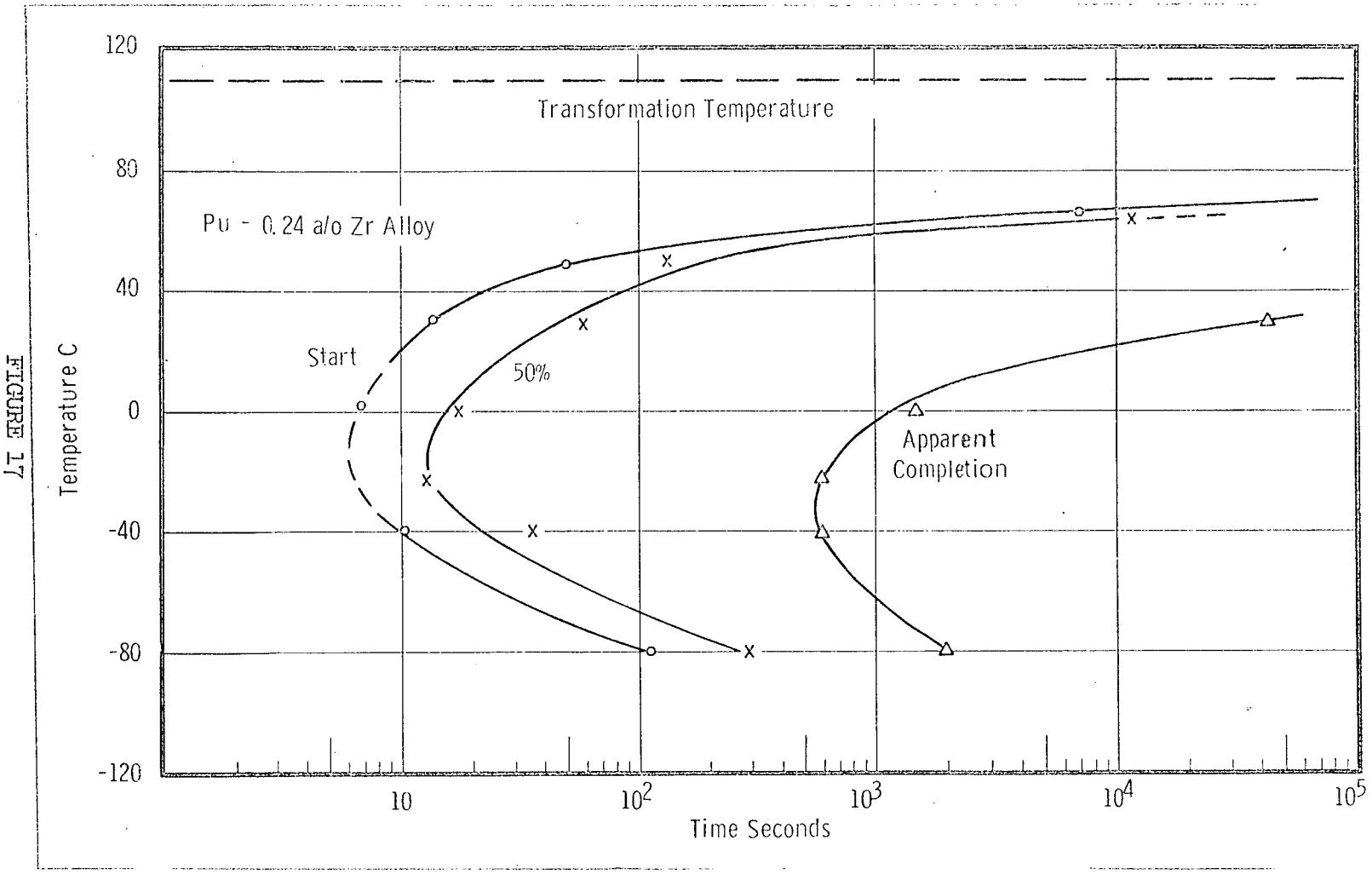


FIGURE 17

T-T-T CURVES OF Pu-0.24 a/o Zr ALLOY

017 041

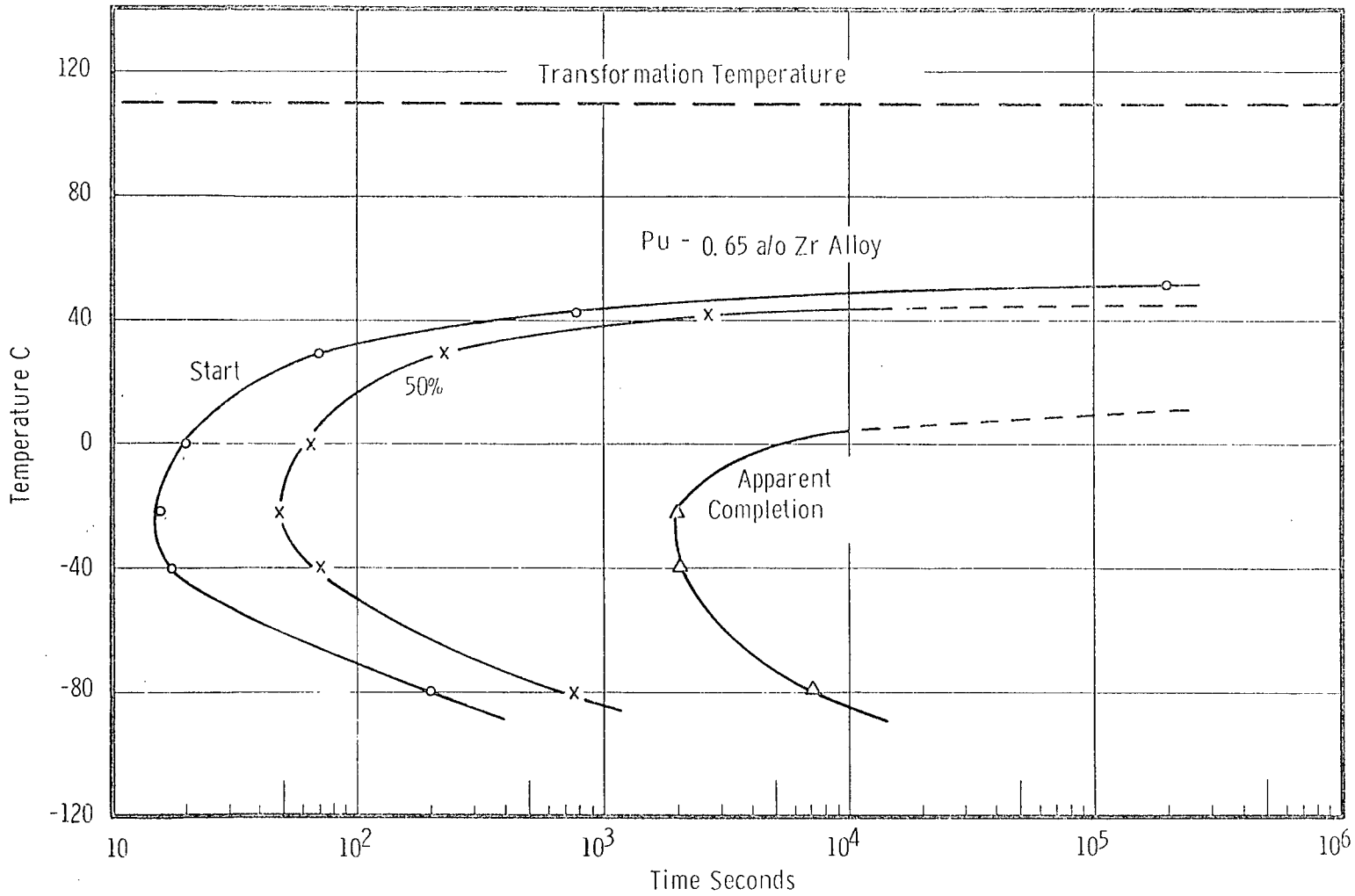


FIGURE 18

T-T-T CURVES FOR Pu-0.65 a/o Zr ALLOY

517 042

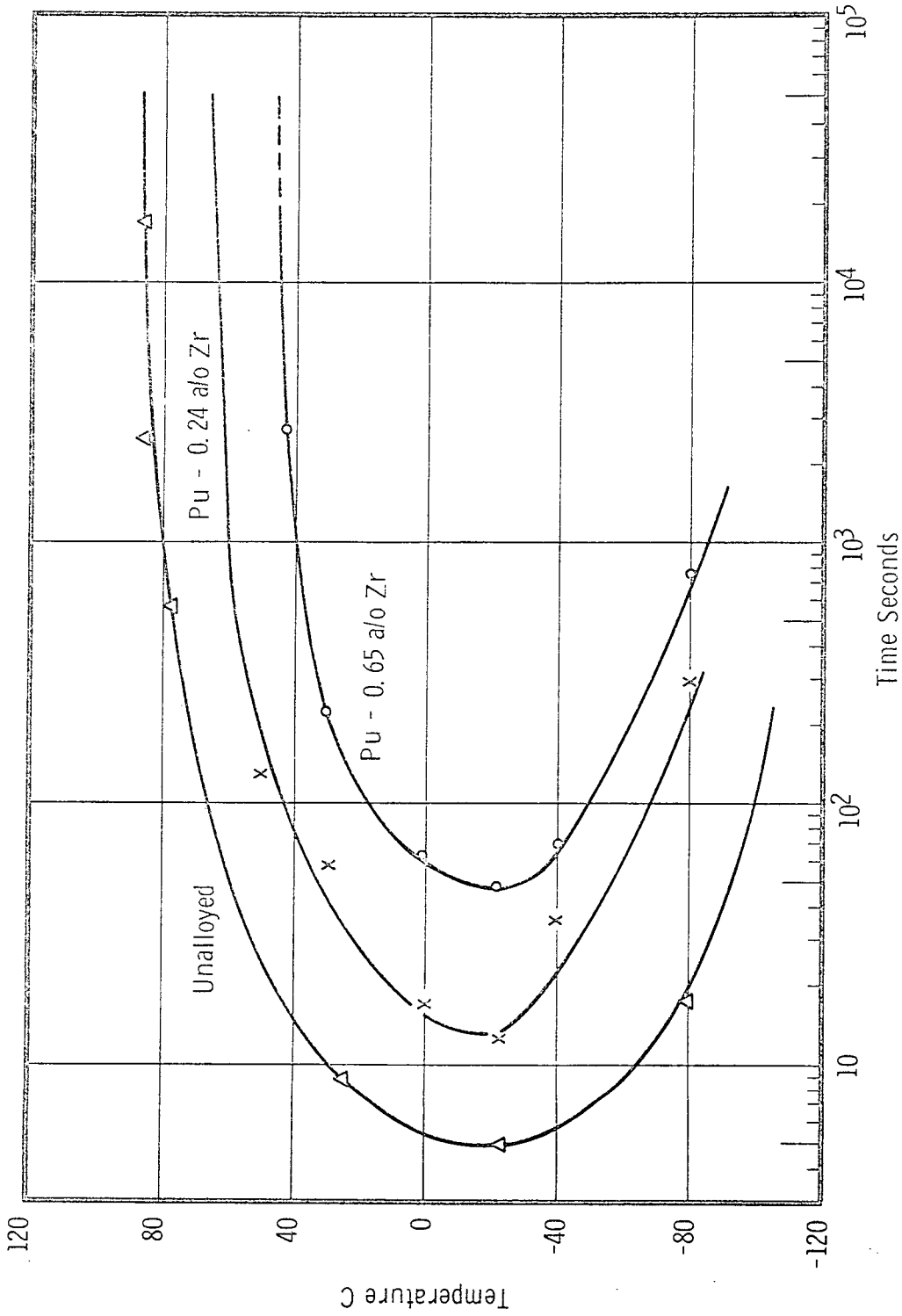


FIGURE 19

T-T-T CURVES SHOWING 50 PER CENT TRANSFORMATION OF UNALLOYED PLUTONIUM. A Pu-0.24 a/o Zr ALLOY AND A Pu-0.65 a/o Zr ALLOY

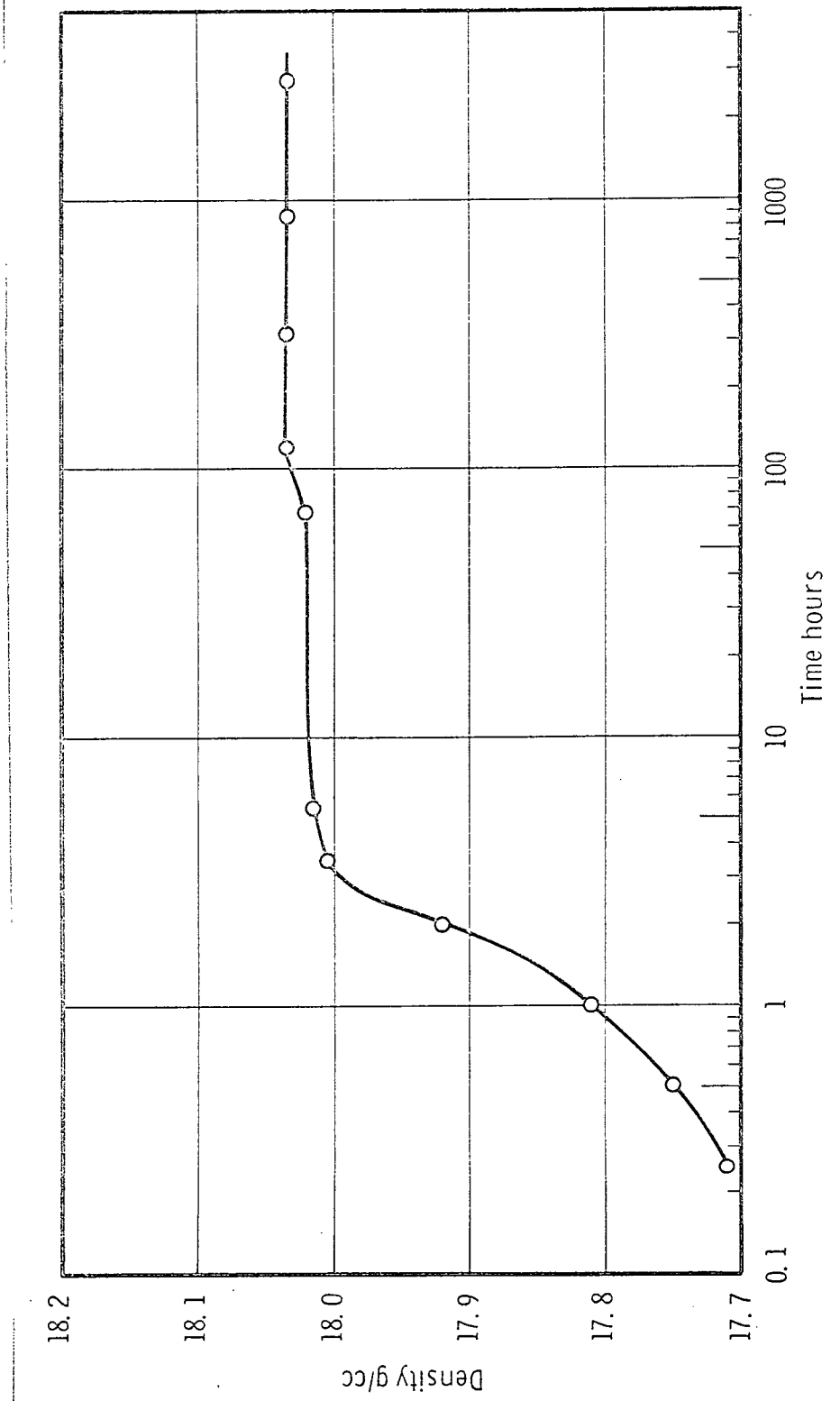


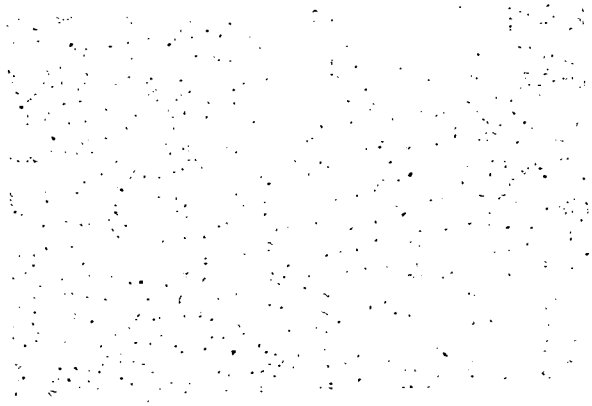
FIGURE 20

ISOTHERMAL REACTION CURVE OF
Pu-1.7 a/o Zr ALLOY AT -23 C

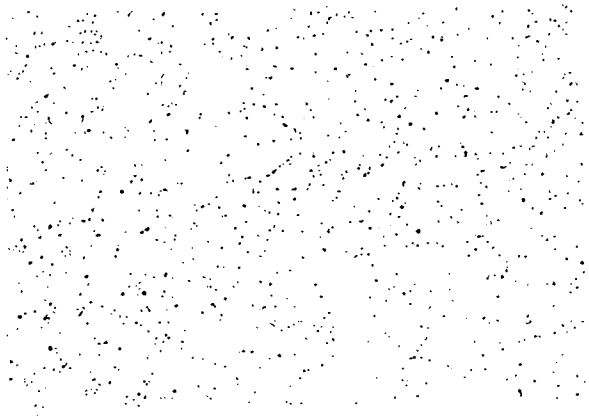
compound have not been determined. Precipitation of the compound was studied at room temperature by metallographic techniques. Disks one-quarter inch thick were cut from a half-inch diameter Pu-1.7 a/o Zr cylinder. The disks were solution heat treated at 220 C for 24 hours and quenched in silicon oil at room temperature. The photomicrographs in Figure 21 illustrate the microstructure at time intervals of 5 and 48 hours after quenching. Note that after 5 hours the grain boundaries contain no precipitate, however after 48 hours a precipitate is present at the grain boundaries. The appearance of precipitate after 48 hours at room temperature was not unexpected since diffusion would occur more rapidly at room temperature than at -20 C. The grain structure of the alloys remained quite uniform. Figure 22 is a photomicrograph illustrating the equiaxed grain structure of a Pu-2.4 a/o Zr alloy. The average grain diameter of the alloys tested was 0.028 mm.

From the above transformation kinetics information the stabilizing effect of zirconium can be determined. When the zirconium concentration is graphed against time for the start of the beta to alpha transformation at -20 C, a curve is obtained which asymtotically approaches 1.9 a/o Zr, Figure 23. Thus under ambient conditions a Pu-2.0 a/o Zr alloy would stabilize the beta phase of plutonium.

Mechanical Property Studies With The Pu-Zr Alloy. The bulk of the mechanical property studies were conducted with a Pu-2.4 a/o Zr alloy composition. A 2.4 a/o Zr addition insured stabilization of the beta phase at room temperature. Tensile tests were also conducted with alloy compositions that had less zirconium.



PHOTOMICROGRAPH OF A Pu-1.7 a/o Zr ALLOY HELD AT
220 C FOR 24 HOURS AND THEN QUENCHED
TO 30 C 5 HOURS AFTER QUENCH. 100X



PHOTOMICROGRAPH OF THE SAME ALLOY AS ABOVE
48 HOURS AFTER QUENCH. 100X

FIGURE 21

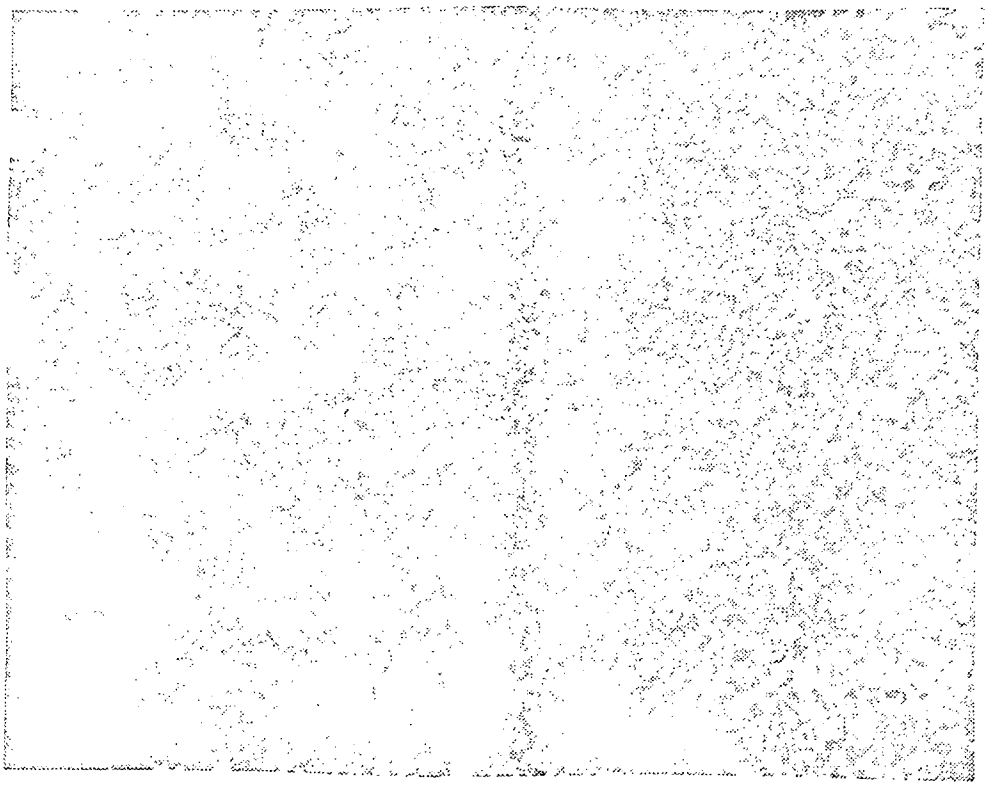


FIGURE 22

ELECTROPOLISHED Pu-2.4 a/o Zr ALLOY
POLARIZED ILLUMINATION, ~~[illegible]~~ 170X
GRAIN SIZE 0.028 mm

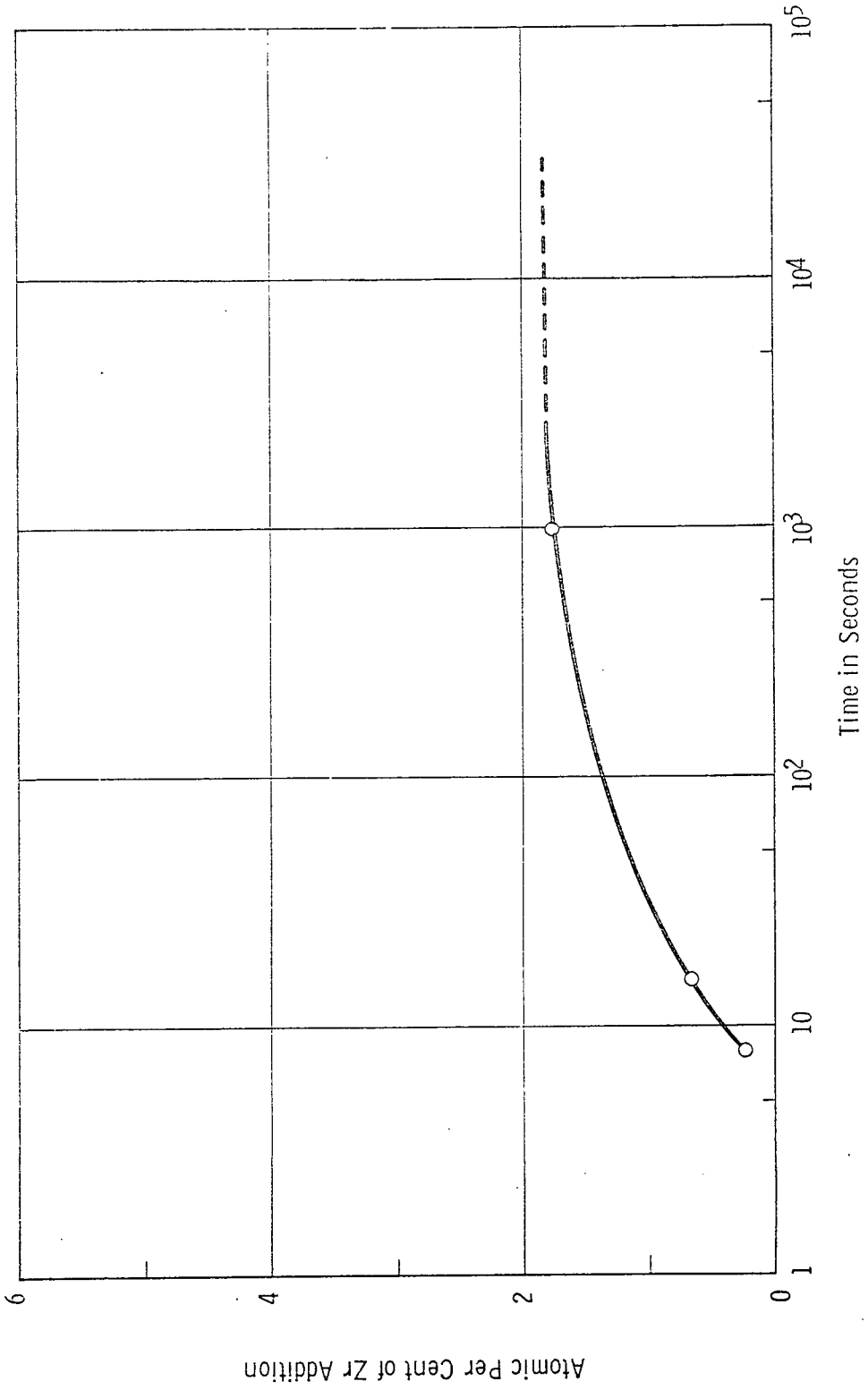


FIGURE 23

START OF TRANSFORMATION AT
-20 C vs. Zr CONCENTRATION

Tensile Property Studies. The effect of increasing temperature on ultimate strength of as cast Pu-0.24 a/o Zr alloy is illustrated in Figure 24. Table III contains the data obtained.

As the temperature increases the strength decreases. The ultimate strength of the alloy is greater than unalloyed plutonium for all test temperatures. The alloy fractures in a brittle manner up to 210 C. However, it has some ductility at 260 as seen from elongation and reduction in area data. A comparison is made of the effect of temperature on the ductility of unalloyed plutonium and a Pu-2.4 a/o Zr alloy, Figure 25. The type of deformation and fracture at various test temperatures is illustrated in Figure 26. Note the necking in the specimens tested at 250 and 260 C.

Because of the marked embrittling effect that a 2.4 a/o zirconium addition has upon the beta phase of plutonium it was decided to add smaller amounts of zirconium to determine the effect of alloying content on ductility. Since a stable beta phase below 2 a/o Zr could not be retained at room temperature the tests were conducted in the beta phase at 130 C and 180 C. Table IV lists the strength and elongation data as a function of zirconium addition. At 130 C the elongation falls very rapidly decreasing by a factor of 2 with a 0.5 a/o Zr addition, Figure 27. At 180 C the elongation decreases at a slower rate with increasing Zr additions. At 1.7 a/o Zr a brittle failure is obtained. Obviously very small additions of zirconium markedly affect the ductility of the beta phase of plutonium. Ultimate strength is similarly affected by small additions of zirconium,

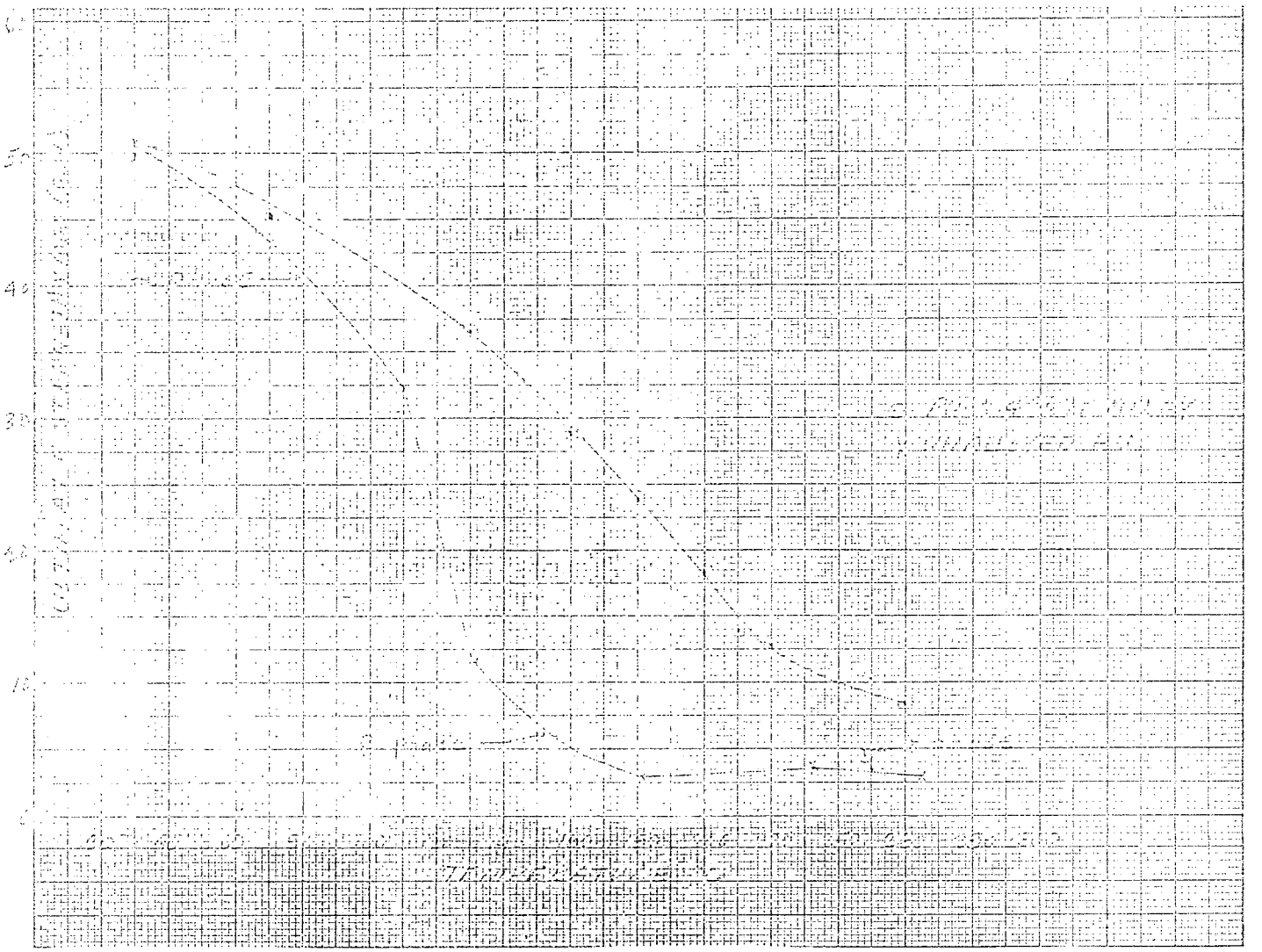


FIGURE 24

EFFECT OF TEMPERATURE ON TENSILE
STRENGTH OF Pu-2.4 a/o Zr
ALLOY AND UNALLOYED
PLUTONIUM

TABLE III

EFFECT OF TEMPERATURE ON TENSILE PROPERTIES OF A BETA STABILIZED Pu-2.4 a/o Zr ALLOY(1)

Test Temp. (°C)	Ultimate Strength (psi)	Yield Strength (psi)	Elongation(2) (%)	Reduction In Area (%)	Heat Treatment And Remarks
30	51,000	28,400(3)	.076	--	Specimen held at 220 C for 24 hours quenched to room temperature, tested 7 hours after quench.
70	45,546	38,500	--	--	Held at 220 C for 5 hours. Cooled to 70 C and held for 20 minutes and tested.
130	36,800	33,400	--	--	Held at 220 C for 5 hours. Cooled to 130 C and held for 20 minutes and tested.
160	28,700	23,300	--	--	Held at 220 C for 5 hours. Cooled to 160 C and held for 20 minutes and tested.
180	23,835	20,000	--	--	Held at 220 C for 5 hours. Cooled to 180 C and held for 20 minutes and tested.
200	18,367	14,300	--	--	Held at 220 C for 24 hours quenched to room temperature. Heated to 200 C and held for 20 minutes then tested.
210	13,700	11,600	--	--	Held at 220 C for 24 hours quenched to room temperature. Heated to 210 C and held for 20 minutes then tested.
230	12,240	8,300	--	2	Held at 220 C for 24 hours quenched and heated to 230 C and held for 20 minutes then tested.
250	10,290	7,300	5.0	28	Held at 220 C for 24 hours quenched and heated to 250 C for 20 minutes then tested.
260	7,800	6,900	6.3	40	Held at 220 C for 24 hours quenched and heated to 260 C for 20 minutes then tested.

(1) Testing Speed: 0.015 inches/ minute (2) Based on one inch gage length (3) A 0.01% offset yield strength.

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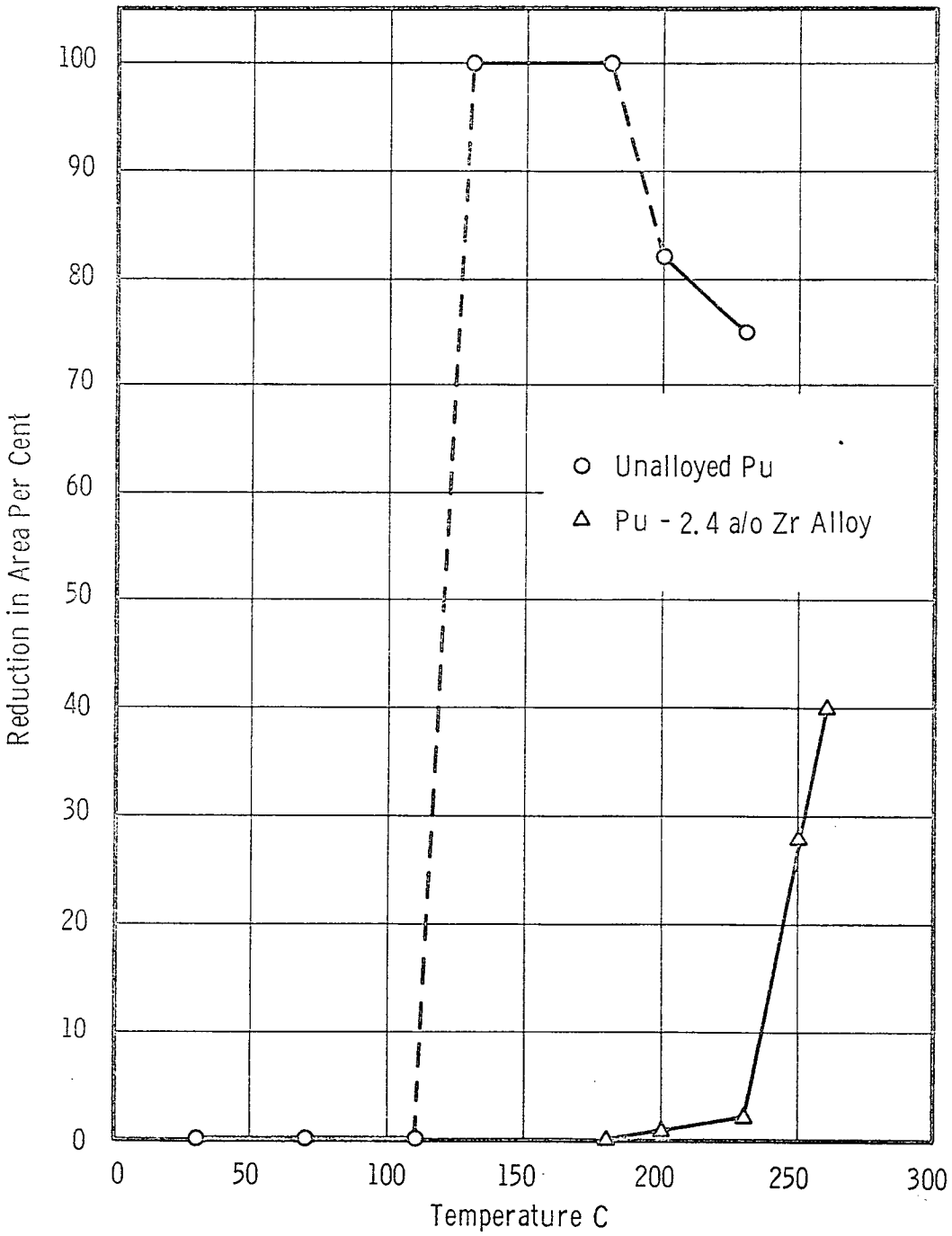

FIGURE 25

COMPARISON OF THE EFFECT OF TEMPERATURE
ON THE DUCTILITY OF UNALLOYED PU
AND A Pu-2.4 a/o Zr ALLOY

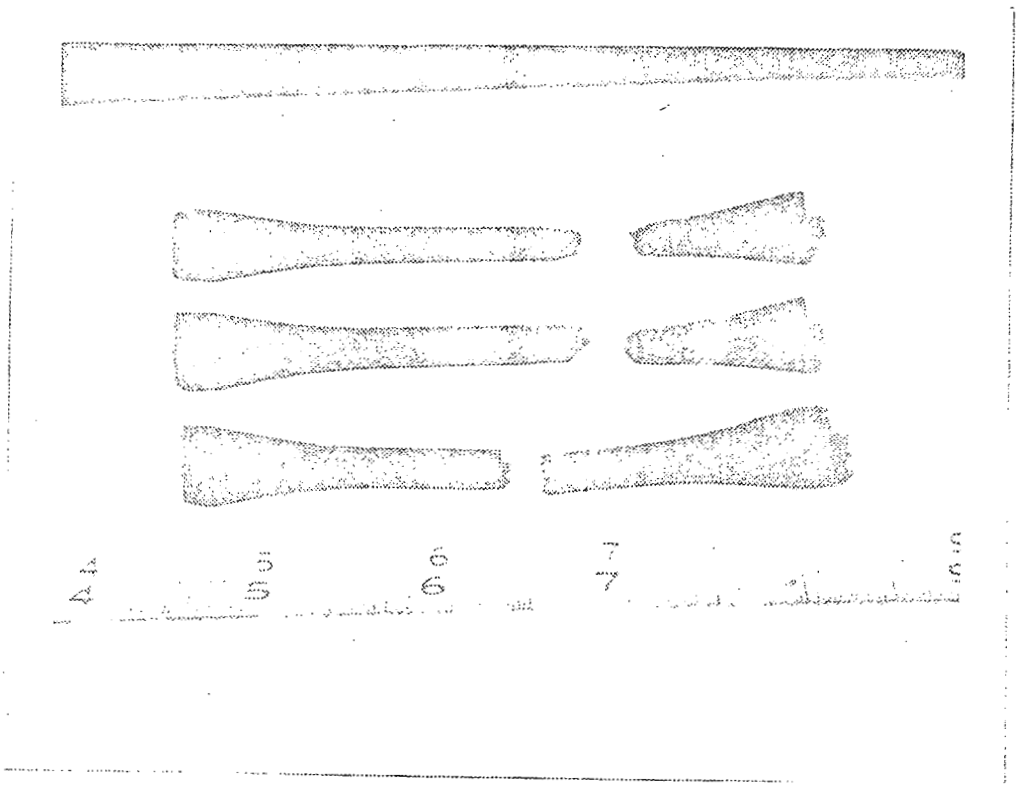

FIGURE 26

DEFORMATION AND FRACTURE OF Pu-2.4 a/o Zr ALLOY SPECIMENS. TOP SPECIMEN TESTED AT 260 C. MIDDLE AT 250 C. BOTTOM AT 30 C.

TABLE IV

COMPARISON OF MECHANICAL PROPERTIES[1] OF VARIOUS COMPOSITIONS OF PLUTONIUM-ZIRCONIUM ALLOY AND UNALLOYED PLUTONIUM AT ELEVATED TEMPERATURES

Composition	Test Temperature (°C)	Ultimate Strength (psi)	Elongation[2] (%)	Reduction In Area (%)	Average Density at Room Temperature, (g/cc)	Remarks
Pu-2.4 a/o Zr	180	18,000	---	---	17.41	Held at 180 C 20 min then tested
Pu-2.4 a/o Zr	130	28,940	---	---		Held at 130 C 30 min then tested
Pu-1.7 a/o Zr	180	16,105	---	---	17.55	Held at 180 C 20 min then tested
Pu-1.7 a/o Zr	130	26,000	---	---		Held at 130 C 30 min then tested
Pu-0.65 a/o Zr	180	12,000	100	100	19.21	Held at 180 C 20 min then tested
Pu-0.65 a/o Zr	130	24,224	---	---		Held at 130 C 30 min then tested
Pu-0.24 a/o Zr	180	6,448	450	75	19.45	Held at 180 C 20 min then tested
Pu-0.24 a/o Zr	130	13,308	160	30		Held at 130 C 30 min then tested
Unalloyed Pu	180	3,580	503.2	100	19.59	Beta Phase Plutonium
Unalloyed Pu	130	12,100	294	100		Beta Phase Plutonium

(1) All tensile specimens 0.250-inch diameter. Testing speed 0.015 inch per minute.

(2) Gage length one inch.

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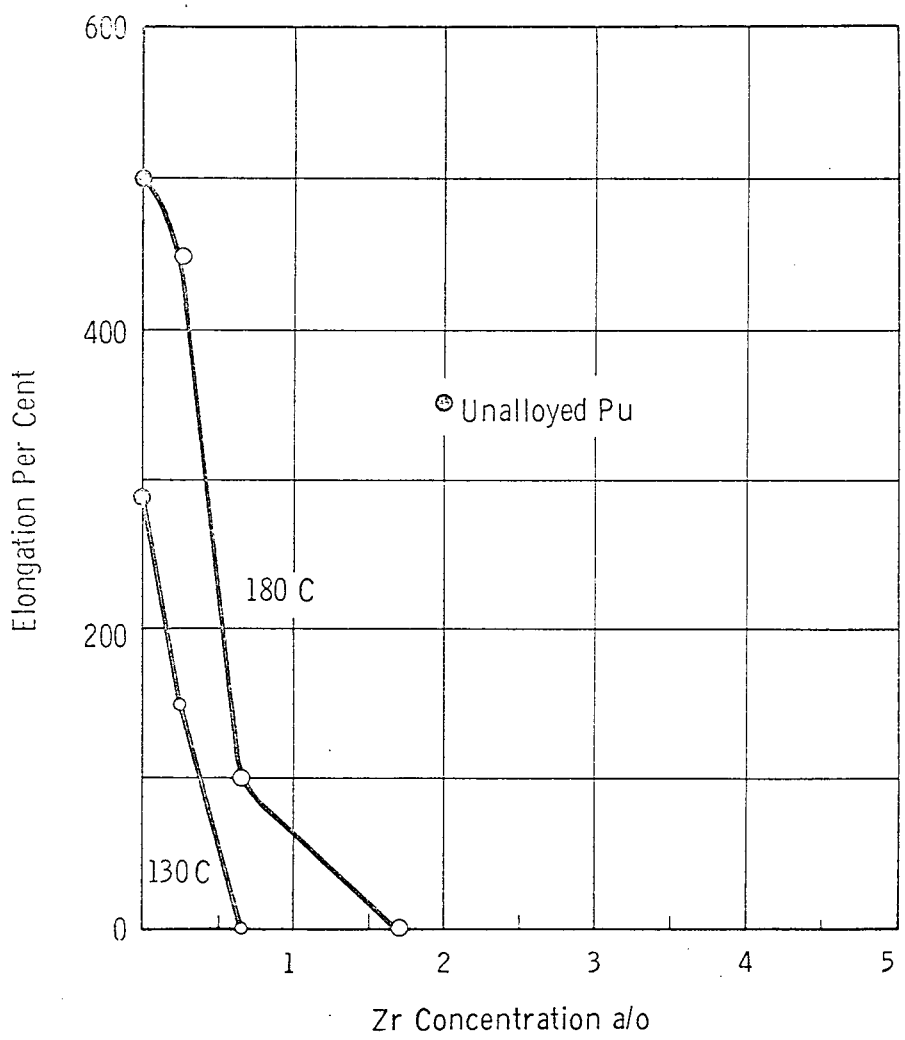

FIGURE 27

EFFECT OF Zr ADDITION ON THE BETA PHASE ELONGATION OF Pu AT 130 AND 180 C

Figure 28. A rapid increase in strength occurs up to 0.65 a/o Zr, however the rate of increase is much lower as Zr additions are increased beyond this point.

Figure 29 illustrates the type of deformation and fracture for the different Pu-Zr alloys. From the shape of the curves and the fracture of the Pu-0.65 a/o Zr alloy it appears a possible brittle ductile transition takes place between 130 C and 180 C. A possible explanation exists for this marked change in mechanical properties. At 130 C a two-phase structure could exist where at 180 C a single phase is present. According to work done by Ellinger[4] with plutonium rich zirconium alloys two phases do exist at 130 C for a 0.65 a/o Zr concentration. However, the published Pu-Zr phase diagram of the British (see Figure 9) shows a single phase. At 180 C both diagrams show only one phase. The mechanical property reported here seems to support Ellinger's data that two phases exist. Consequently, different mechanical properties result for the same Pu-0.65 a/o Zr alloy at the two different temperatures.

Compression Property Studies. The compression properties of a Pu-2.4 a/o Zr alloy were determined at temperatures from 30 to 220 C. Table V contains the data. A comparison between the compressive strength of the alloy and unalloyed plutonium is made, Figure 30.

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[4] F. H. Ellinger, "Discussion - The Plutonium-Zirconium System," Plutonium 1960, (London: Cleaver-Hume Press Ltd., 1961) , pp. 318-319.

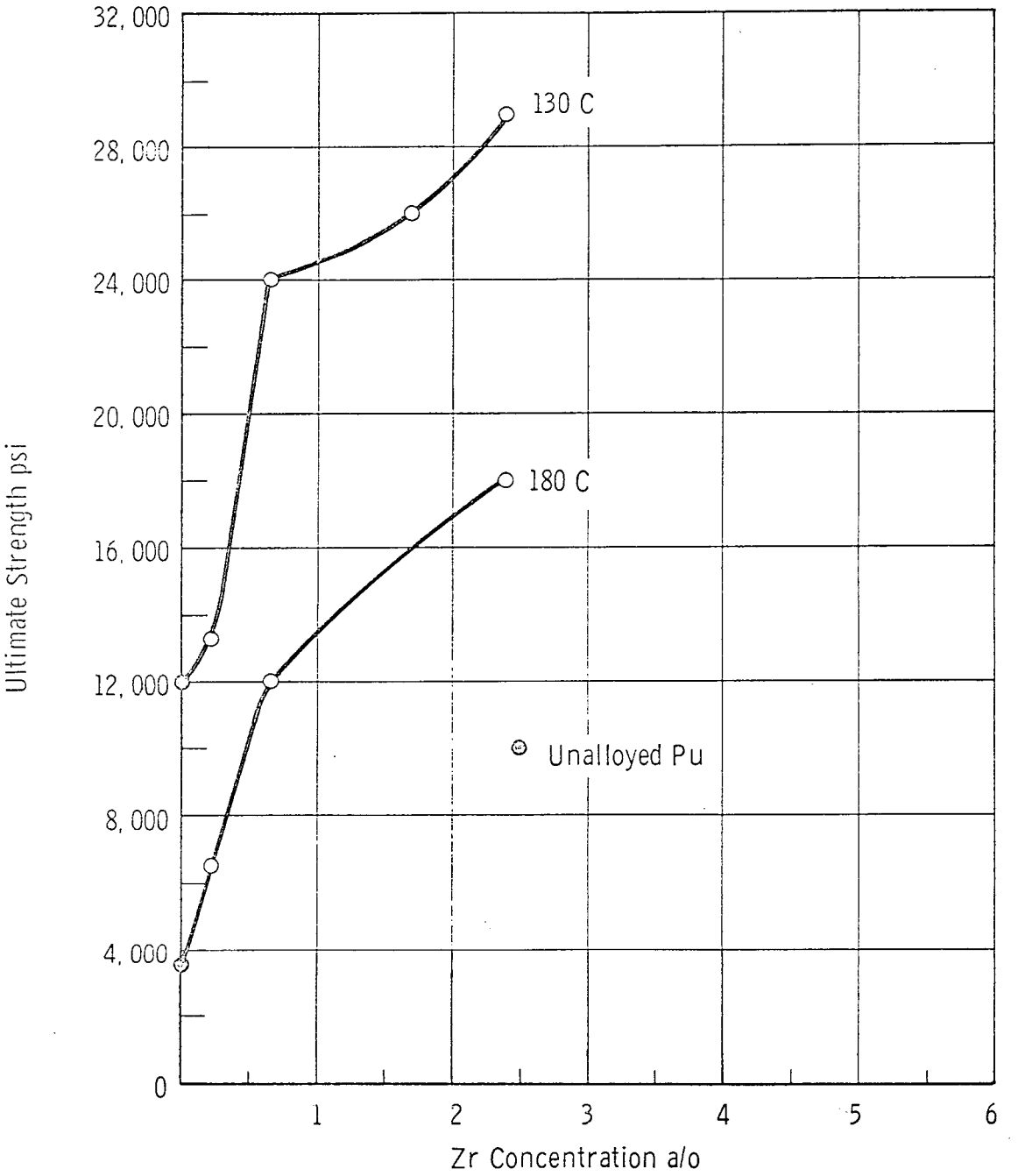
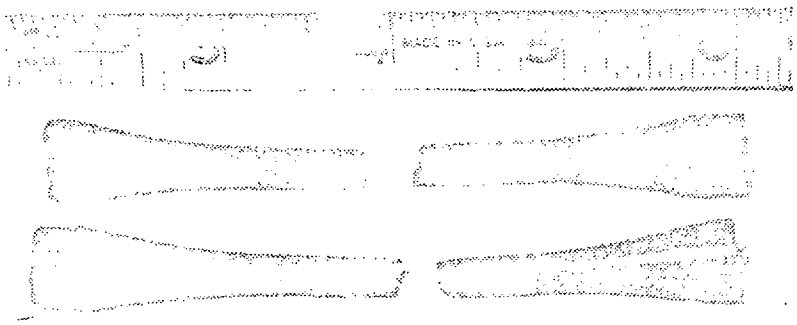
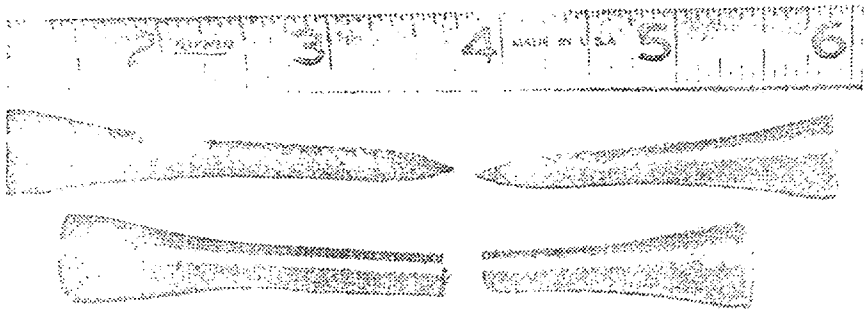


FIGURE 28

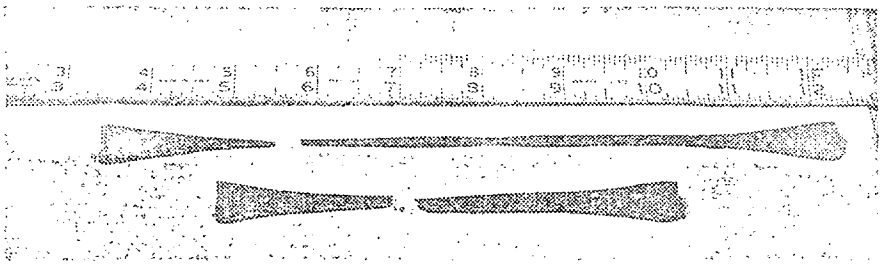
EFFECT OF Zr ADDITION ON THE ULTIMATE TENSILE STRENGTH OF BETA PHASE PLUTONIUM AT 130 AND 180 C



Pu - 1.7 a/o Zr Alloy. Top specimen tested at 180 C bottom specimen at 130 C. Testing speed 0.015 in./min.



Pu - 0.65 a/o Zr Alloy. Top specimen tested at 180 C bottom at 130 C.



Pu - 0.24 a/o Zr Alloy. Top specimen tested at 180 C bottom at 130 C.

FIGURE 29

COMPARISON OF DEFORMATION AND FRACTURE OF A Pu-1.7 a/o Zr ALLOY, A Pu-0.65 a/o Zr ALLOY AND A Pu-0.24 a/o Zr ALLOY TESTED AT 130 AND 180 C

TABLE V

EFFECT OF TEMPERATURE ON THE COMPRESSION PROPERTIES (1)
OF BETA STABILIZED Pu-2.4 a/o Zr ALLOY

Test Temperature (°C)	Ultimate Strength (psi)	Yield Strength (psi)	Plastic Decrease In Length(2) (%)	Heat Treatment (°C)
60	67,000	59,800	4.6	30 minutes at 60
80	64,600	57,900	5.3	30 minutes at 80
130	53,500	48,700	5.3	30 minutes at 130
160	45,600	41,000	6.0	30 minutes at 160
180	46,800	43,000	5.0	30 minutes at 180
200	32,800	27,700	5.0	30 minutes at 200
220	28,500	26,900	5.4	30 minutes at 220

(1) Testing speed 0.015 inches per minute

(2) Total plastic decrease was determined from load-platen displacement curve at point of failure.

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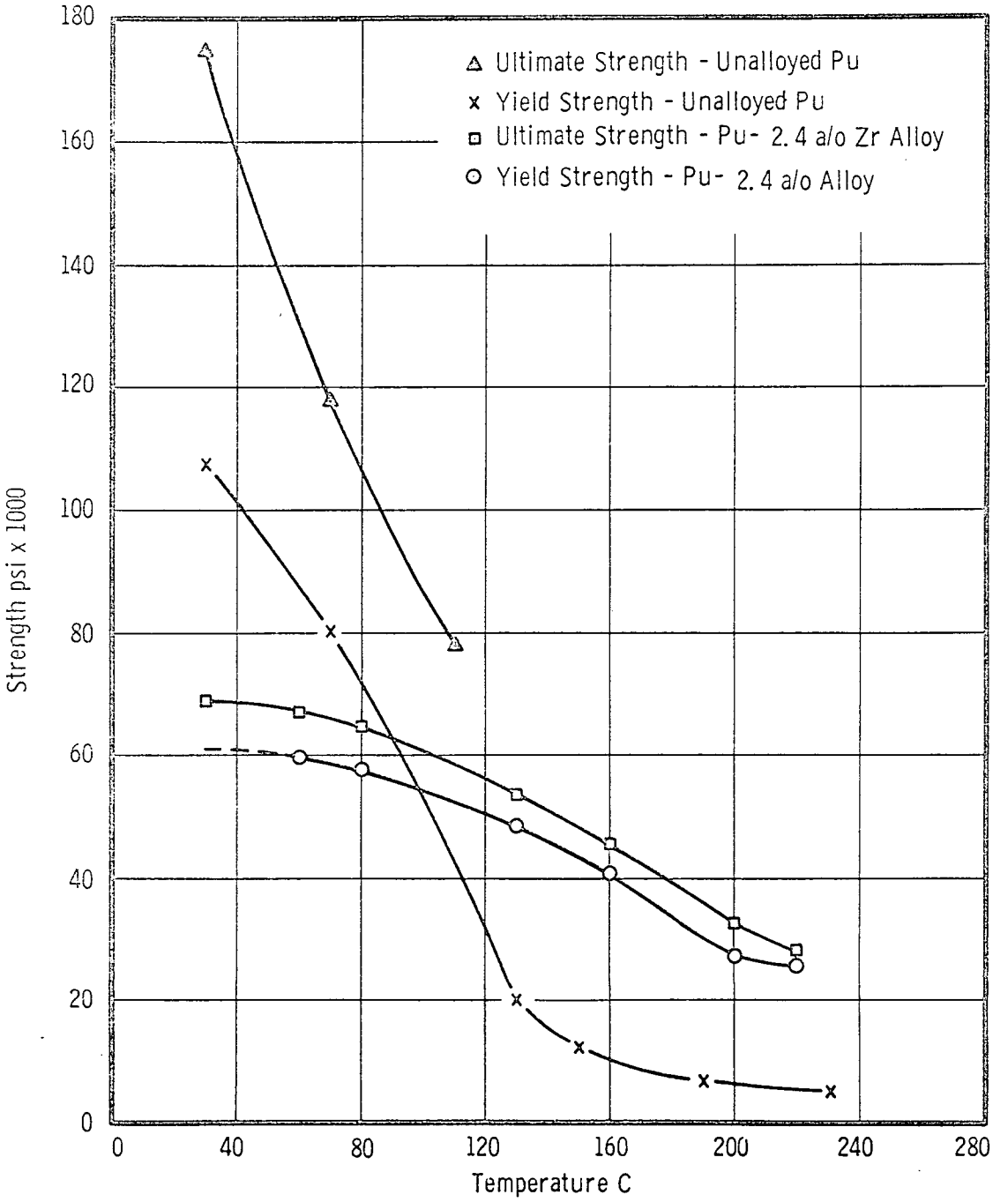

FIGURE 30

COMPARISON OF COMPRESSIVE STRENGTHS OF
Pu-2.4 a/o Zr ALLOY AND
UNALLOYED PLUTONIUM

The compressive strength of alpha phase plutonium is greater than the alloy. The stabilized beta alloy however, is considerably stronger than the beta phase of plutonium. Due to the tremendous plasticity of the beta phase ultimate strength data could not be obtained on unalloyed plutonium.

The alloy was found to be essentially brittle over the range of temperatures studied. The total plastic decrease in length averaged 5.2 per cent for all temperatures.

During compression testing densities indicated that the transformation took place below 80 C, at temperatures above this the densities remained essentially constant. Consequently, when the pressure exceeds atmospheric pressure below 80 C zirconium does not stabilize the beta phase.

Age Hardening Studies. Since a compound precipitates above 1.5 a/o Zr after quenching to room temperature from a beta solution heat treatment it was decided to determine whether age hardening occurs in alloys of a Pu-2.4 a/o Zr composition. A series of eight tensile specimens were heat treated at 220 C for 24 hours and then quenched in oil at room temperature. The specimens were tested in duplicate at selected time intervals after quenching. The results are presented in Table VI. A slight aging effect is observed with the alloy, Figure 31. The maximum strength was not accurately determined since tests were not made between 7 and 24 hours after quenching. The yield strength data indicate that overaging occurs after 48 hours. The age hardening effect has not been investigated at

TABLE VI

STUDY OF THE EFFECT OF AGING TIME ON THE TENSILE PROPERTIES[1] OF A BETA STABILIZED Pu-2.4 a/o Zr ALLOY

Aging Time After Quench[2]	Ultimate Strength (psi)	0.01 Per Cent Yield Strength (psi)	Elongation (%)	Reduction In Area (%)	Modulus of Elasticity x 10^{-6}	Temperature Of Test (°C)
40 minutes	40,000	22,100	0.076	--	6.43	30
7 hours	51,000	28,400	0.28	--	5.73	30
24 hours	42,700	24,800	0.081	--	6.09	30
48 hours	43,000	16,200	0.102	--	6.59	30

(1) Testing speed 0.015 inches per minute

(2) All tensile specimens were heat treated at 220 C for 24 hours and then quenched in oil at room temperature.

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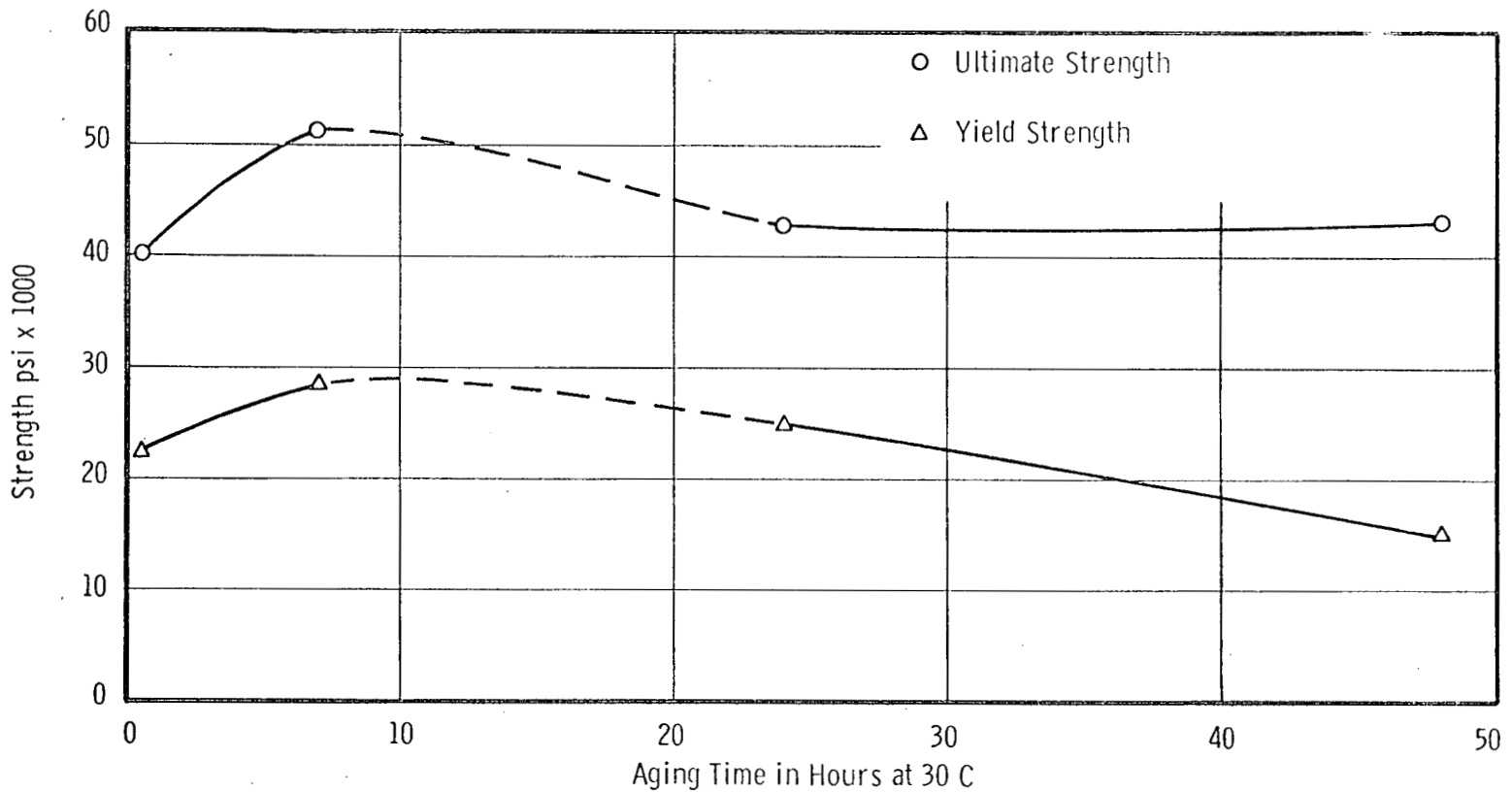

FIGURE 31

AGING CURVES FOR Pu-2.4 a/o Zr ALLOY AT 30 C

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elevated temperatures. It is reasonable to assume that above 130 C the intermetallic compound won't precipitate. The aging phenomena is only expected to be in evidence below 130 C.

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CHAPTER IV

SUMMARY AND CONCLUSIONS

The beta phase of plutonium can be retained at room temperature in varying amounts by additions of Ti, Zr and Hf. Zirconium was, however, the only element which would stabilize the beta phase of plutonium with additions less than 3.0 a/o.

Kinetic studies with the Pu-Zr alloys indicate that amounts of zirconium as little as 0.24 a/o slow the beta to alpha transformation by a factor of 2 compared to unalloyed plutonium. The transformation rate was a maximum at -20 C for this alloy.

Time, Temperature, Transformation curves for the Pu-0.24 a/o Zr and the Pu-0.65 a/o Zr alloys were C shaped and similar to the T-T-T curves for unalloyed plutonium. Critical holding temperatures for beta to alpha transformation were observed for the 0.24 and 0.65 a/o Zr alloys. Above 80 C for the 0.24 a/o alloy and above 60 C for the 0.65 a/o alloy beta to alpha transformation will not start for many months.

As the zirconium concentration increases more beta phase plutonium is retained below the normal beta to alpha transformation temperature. A 2.4 a/o Zr addition stabilized the beta phase of plutonium from room temperature up to 265 C.

Precipitation of an intermetallic compound was observed when 1.7 a/o of Zr was added to plutonium. Precipitation of the compound did not occur until 70 hours after the start of the beta to alpha

transformation at -20 C. At room temperature the compound precipitated after about 48 hours.

Mechanical property studies indicate that the Pu-2.4 a/o Zr alloy has greater compressive and tensile strength and much lower ductility than unalloyed beta phase plutonium. As test temperature is increased to 260 C strength decreases in a continuous manner. The alloy fractures brittley up to 230 C while above this temperature some ductility is observed.

It was noted that small additions of zirconium affect the mechanical properties of the beta phase of plutonium. Additions of as small as 0.24 a/o increase the strength of the beta phase by a factor of about 1.5 and decrease the elongation from 500 per cent to 450 per cent at 180 C.

Mechanical property data on the Pu-0.65 a/o Zr alloy show that a brittle - ductile transition takes place between 130 and 180 C. This is thought to result from the presence of two phases at 130 and not at 180 C.

During compression testing of the Pu-2.4 a/o Zr alloy at temperatures below 80 C it was determined that transformation to alpha phase occurs.

Although the ductility of beta phase plutonium was greatly reduced by the addition of zirconium the Pu-2.4 a/o Zr alloy had a number of desirable features. It is a factor of 3 stronger than unalloyed beta phase plutonium. The beta phase is stabilized from room temperature up to 265 extending the working range of the metal

some 140 degrees. Even though the alloy transforms at room temperature when subjected to compressive forces, by conducting forming operations above 80 C no transformation would occur. Also at 250 C reasonable ductility is present for forming. Void formation due to the large volume change accompanying the beta to alpha transformations in unalloyed plutonium are eliminated. Finally, the beta phase of plutonium can be stabilized by a relatively small addition of foreign atoms, thus maintaining very nearly the same nuclear properties as unalloyed plutonium.

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APPENDIX

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TABLE VII

CHEMICAL ANALYSIS OF PLUTONIUM AND OTHER METALS USED IN ALLOY STUDIES

Metal Typical Purity	Impurities (ppm)																									Total
	Al	B	Be	Ca	Cd	Cr	Cu	Fe	Ge	K	La	Mg	Mn	Na	Ni	Pb	Si	Sn	V	Zr	C	O_2	N_2	H_2	Other	
Pu	14	-	-	50	-	5	5	75	-	-	-	100	10	5	<5	-	<1	<5	-	-	230	50	20	5	-	570 ppm
Ti	50	-	-	30	-	20	3	20	-	-	-	30	30	-	1	-	50	10	-	-	10	20	20	-	500 Zr 490 Cl .095 %	.794 ppm
Zr	<20	<.2	-	<10	<.2	-	<20	380	-	-	<20	-	<20	330	<20	<20	31	-	-	-	120	1180	46	-	H_2O	-
Mg	<10	-	<10	<10	-	-	<10	100	-	10	-	-	100	10	-	-	40	-	-	-	ND	ND	ND	ND	-	270 ppm
Si	100	-	-	10	100	-	100	1000	ND	-	-	100	100	<10	10	-	-	-	<100	-	ND	ND	ND	ND	100 Ti	1730 ppm
U	<10	<10	-	<5	-	10	5	100	-	1	-	5	10	<1	20	5	50	<1	-	<10	ND	ND	ND	ND	-	243 ppm
Hf	20	15	-	50	-	30	40	50	-	-	-	10	10	-	400	30	70	-	20	-	ND	ND	ND	ND	3-4% Zr	3.07 to 4.07 %

ND = Not determined

- = Not detectable

Cadmium, 99.9999% Pure

Lead	0.1 to 0.2 ppm
Copper	0.1 to 0.2 ppm
Thallium	0.1 to 0.2 ppm
Silver	0.1 to 0.2 ppm

No other impurities detected spectrographically

Tin, 99.999% Pure

Ca	0.1 ppm
Pb	1.0 ppm
Mg	0.1 ppm
Si	0.1 ppm

No other impurities