6 Compositional and pressure effects in the plutonium-gallium system:

H. R. Gardner

Lattice parameters, density, and hardness were determined on plutonium-gallium alloys containing 0.37 to 1.26 wt.-% gallium. Lattice parameter and density decrease with increasing gallium content while hardness increases because of solid solution strengthening. The author's data are compared with those of other investigators.

The effect of a 150,000 lb/in^2 pressure on alpha phase formation in both cored and homogenized alloy is presented. In addition the stability of alpha-delta phase mixtures with respect to annealing and room temperature storage is discussed. An explanation of the observed behaviour of alpha-delta phase mixtures is presented.

1 Introduction

1.1 In unalloyed plutonium, the delta phase region extends from 319 to 451°C. As indicated in the plutonium-gallium phase diagram, Fig 6-A,[1] the addition of appropriate quantities of gallium stabilizes the delta phase over a much wider range of temperatures including room temperature.

1.2 The metallurgy of delta stabilized plutonium-gallium alloys is complicated by three phenomena. During nonequilibrium cooling through the liquid plus epsilon and epsilon plus delta regions, coring, which is a type of segregation, occurs. This results in a variation in gallium content within each grain, the central portion having a much higher gallium concentration than that near the grain boundary. Using microprobe analysis, Johnson[2] has determined that the cored grains in a cast Pu 1.0 wt.-% Ga alloy range from 0.1 wt.-% Ga near grain boundaries to 1.5 wt.-% Ga at the centre. Coring can be eliminated by an appropriate anneal in the 400 - 500°C temperature range. Diffusion occurs and a uniform

distribution of gallium results. Since cooling rate during and after solidification affects the size of the cored grains, the rate of diffusion or homogenization depends on the diameter of the cored grains; as diameter decreases, the rate of homogenization increases[3].

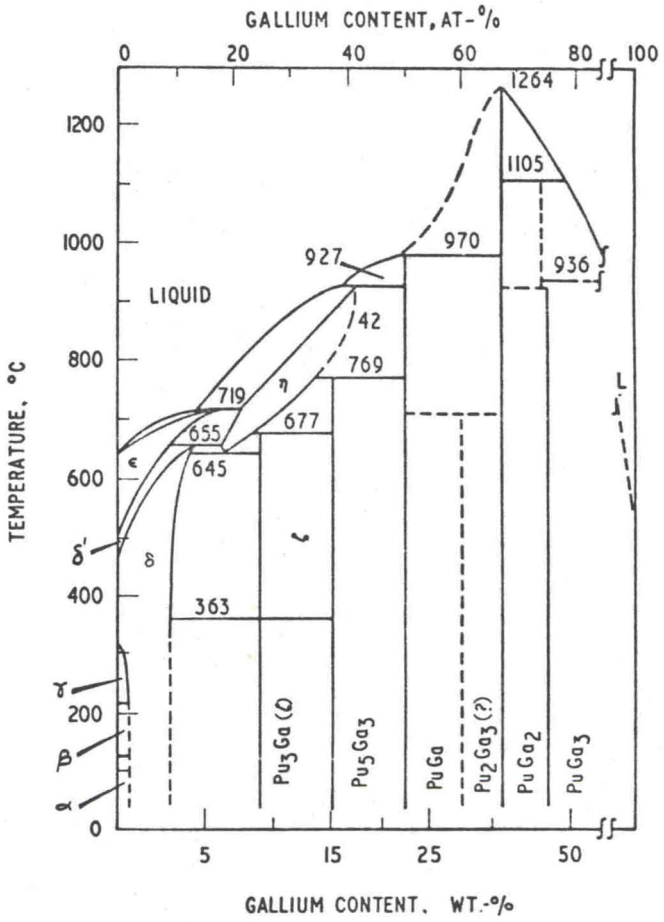

Fig 6-A Pu-Ga Phase Diagram after Ellinger[1].

1.3 The second phenomenological feature concerns metastability with respect to the application of pressure[4]. In alloys containing less than 1.2 wt.-% Ga, the stabilized delta phase irreversibly transforms to alpha phase in proportion to the pressure applied. Above 1.2 wt.-% Ga, the formation of alpha

phase under pressure was believed to be reversible and is undoubtedly controlled by reaction kinetics.

1.4 The Pu 1.0 wt.-% Ga alloy, therefore, even when completely homogenized, will be metastable with respect to the application of pressure. Because of the 0.1 to 1.5 wt.-% Ga gradient resulting from coring, the degree of pressure metastability or alpha forming tendency will be greater. A test for proximity to an equilibrium or homogenized condition would then be the determination of density before and after the application of pressure. The greater the density increase because of alpha formation, the farther the alloy is from the equilibrium state. As the equilibrium or homogenized condition is approached, the density change should decrease and level off at a constant value.

1.5 A third feature concerns the tendency to form intermetallic compounds during preparation of the alloy. For alloy compositions up to 3.0 wt.-% Ga the formation of Pu_xGa_y compounds reduces the gallium available for delta stabilization. Thus, while the total gallium content is as specified the amount of gallium available for stabilization may be substantially lower. To prepare successfully the alloy using induction or resistance heating facilities, it is felt that melt temperatures in the range of 1100 - 1300°C must be achieved. Because of the inherently high metal temperatures achieved during arc melting, this method of alloy preparation prevents the formation of Pu_xGa_y compounds.

1.6 In most of the compositional studies performed by other investigators, neither of the above-mentioned methods was used to prepare the alloy. Therefore it was decided to redetermine the compositional effects on several properties using properly prepared alloys. In addition, it was decided to study the phenomenon of pressure metastability in both cored and homogenized alloys.

2 Experimental Procedure

2.1 Experimental Material

2.1.1 The plutonium-gallium alloys used in this work were

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used in this work were

prepared by first arc melting a Pu 11.1 wt.-% Ga master alloy. The master alloy was diluted with unalloyed plutonium to the desired composition in either an induction or resistance heated vacuum furnace where it was also cast to shape. The initial arc melting prevented compound formation so that subsequent dilution in resistance or induction melting facilities could be successfully accomplished at melt temperatures of approximately 900°C. When reference is made to the "homogenized condition" in subsequent text, a 150 h anneal in vacuo at 450°C is implied. Since the grain size was of the order of 0.005 - 0.007 mm, the anneal was more than sufficient to achieve complete homogenization. The average impurity content of the alloys is presented in Table 6-I.

Element	Analysis (ppm)	Element	Analysis (ppm)
Al	27	Mg	5
Ca	<5	Mn	11
Cr	2	Ni	5
Cu	9	Si	<15
Fe	<50	C	160
			<289

Table 6-I Average Chemical Analysis of Pu-Ga Alloys

2.2 Experimental Techniques

2.2.1 X-Ray Diffraction - Specimens were mounted in polyester resin in 3/4 in. ID mounts. The mounted specimen was faced flat on a small lathe in increments of 0.010, 0.005, 0.002, 0.002, 0.001, and 0.001 in., in that order, to minimize alpha phase formation during machining. Rough polishing was accomplished on the 180, 260, 600 grit cloths on rotating laps.

2.2.2 A Syntron vibratory polisher using silk cloth and Linde A alumina abrasive for 2 h served as the intermediate polish. Carbon tetrachloride was used as a lubricant. The final polish consisted of an initial 100 second electropolish at 20 V and 0.8 - 1.0 amp in an electrolyte composed of 20 parts

tetraphosphoric acid, 30 parts water, and 50 parts 2-ethoxy-ethanol. A Norelco diffractometer using CuK_α radiation and a scanning speed of 0.25°/min was used to obtain the (331) peak on a rotating specimen. After the half-height width was determined, the specimen was electropolished for an additional 20 sec. Again the (331) peak was obtained, half-height width determined, etc. The cycle was repeated until the minimum line width for the (331) peak was obtained. Scans of the (331), (420), (422), (511/333), (440), and (531) peaks were then made at 0.25°/min. Lattice parameters were calculated and plotted against the Nelson-Riley function. It was found that the best estimates of a_o could be obtained either from the Nelson-Riley extrapolation or from the lattice parameter of the (531) plane.

2.2.3 In the second method, developed by Hays[5], powder was filed from the specimens in a nitrogen atmosphere glovebox and stress relieved in vacuo at 300°C for 3 h. After mounting the powder in a recessed perspex holder covered with a cellophane tape, a diffractometer scan of the (511/333), (440), and (531) peaks was made using the fixed count method. The time necessary to register 25,600 counts on a diffractometer scaler panel was recorded at 0.05° increments of 2θ for each diffraction peak. Using the method of Koisteinen and Marburger[6], the vertex of a parabola was fitted to each of the diffraction peaks, the vertex being taken as the diffraction angle. The data points used in fitting the parabola were at least 90% of the maximum peak intensity. The intensity readings were initially corrected for Lorentz-polarization and background. This correction was found to cause little or no change in the final value of lattice parameter and was therefore discontinued. Lattice parameters were obtained from the Nelson-Riley extrapolation.

2.2.4 Density, Hardness, and Pressure Testing - Density determinations were made at room temperature using a fluid displacement technique with FC-75* as the displacement medium.

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*FC-75 is an inert fluorochemical consisting principally of isomers of perfluoro cyclic ether: $C_8F_{16}O$.

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Hardness data were obtained on a Wilson "Tukon" Microhardness Tester using a 2 kg load. To achieve a nearly isostatic pressure, specimens approximately 1/8 in thick with an initial area of one square inch were compressed on a 200 ton hydraulic press.

3 Results and Discussion

3.1 Lattice parameter - As illustrated in Fig 6-B, Hay's lattice parameter versus composition data are in good agreement with Ellinger's[1]. This is not surprising considering that homogenized and annealed powders were used in both cases. The data of Gardner, however, are displaced by a constant amount from Hay's and Ellinger's. The presence of a nonrepresentative specimen surface in the solid specimens used by Gardner was felt to be the main source of error since, for compositions less than 1.2 wt.-% Ga, alpha phase formation occurs during the mechanical polish. The effectiveness of removal of the alpha phase during electropolishing is now considered to be an important factor in the validity of the lattice parameter determination. Other than a possible difference in impurity content, no explanation can be given for the large difference between the data of Hays and Ellinger and those of Lee[7].

3.2 Density

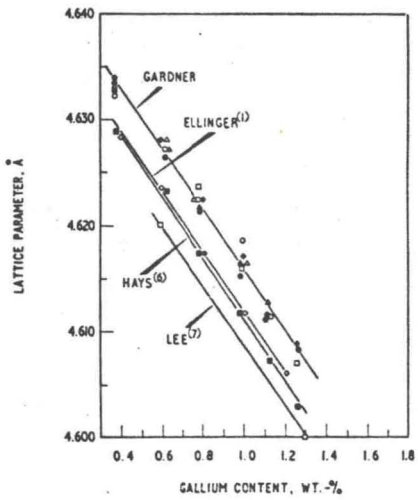
3.2.1 Density versus composition data were obtained on alloy in both the as-cast cored condition and homogenized condition, Table 6-II. The linear relationship for the homogenized alloy indicates that the higher densities in the cored alloy for compositions below 0.99 wt.-% Ga were caused by the presence of alpha phase.

3.2.2 In Fig 6-C the above data are compared to those of Miller and White[7] and Elliott and Gschneidner[4]. The data from the three investigations are in reasonably good agreement.

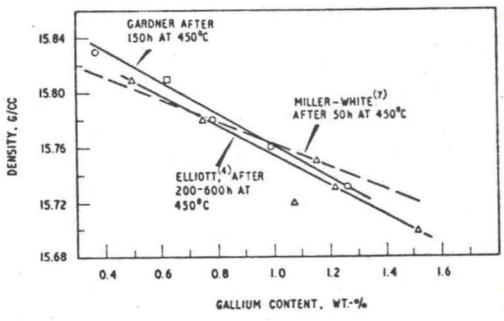
3.3 Hardness

3.3.1 Hardness versus composition data were obtained on alloys in both the as-cast cored condition and the homogenized

condition as illustrated in Table 6-II and Fig 6-D. In the homogenized alloys, the linear increase in hardness with gallium content was caused by solid solution strengthening.



6-B



6-C

Fig 6-B Effect of Gallium Content on Lattice Parameter

Fig 6-C Effect of Gallium Content on Density

Wt.-% Ga(1)	As Cast		After Anneal(2)		After Anneal(2) & Compression(3)	
	Density g/c.c.	DPH(4)	Density g/c.c.	DPH(4)	Density g/c.c.	% Alpha
0.37	16.12	53	15.83	32	18.11	62.0
0.62	15.95	42	15.81	36	17.08	35.0
0.78	15.83	48	15.78	38	16.40	17.0
0.99	15.79	46	15.76	40	15.92	4.5
1.12	-	49	-	44	15.75	-
1.26	15.71	49	15.73	46	15.78	1.0

(1) By analysis (3) 150,000 lb/in^2

(2) 150h at 450°C (4) 2 kg load

Table 6-II Effect of Gallium Content on Density, Hardness and Pressure Metastability

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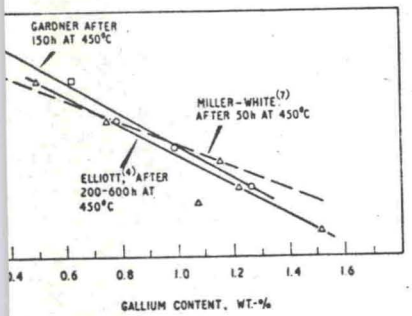


6-C

nt on Lattice Parameter

nt on Density

eal[2]	After Anneal[2] & Compression[3]	
DPH[4]	Density g/c.c.	% Alpha
32	18.11	62.0
36	17.08	35.0
38	16.40	17.0
40	15.92	4.5
44	15.75	-
46	15.78	1.0

150,000 lb/in^2

2 kg load

nt on Density, Hardness

In the as-cast cored condition, however, the lower gallium content alloys contained alpha phase which resulted in higher hardnesses. In addition, alpha phase formation occurred during hardness testing and further increased the apparent hardness over that of the homogenized alloy. The plausibility of alpha phase formation can be examined from the standpoint of the pressures produced during hardness testing and the pressures required for alpha phase formation. King[8] has reported that in cored Pu-1.0 wt.-% Ga alloy, measurable alpha phase formation occurred when the isostatic pressure applied exceeded 25,000 lb/in^2. The 32 kg/mm^2 DPH number for the homogenized Pu 0.37 wt.-% Ga alloy corresponds to a value of 45,000 lb/in^2. Realizing that an exact comparison cannot be made between this value and the 25,000 lb/in^2 isostatic pressure, it was nevertheless felt that the pressure imposed during a hardness test was of sufficient magnitude to form alpha phase.

3.3.2 Interestingly then, the following four factors influence a hardness versus gallium composition curve in cored alloys. As gallium content increases.

a. hardness increases, because of solid solution strengthening,

b. the amount of alpha phase present in the as-cast condition decreases,

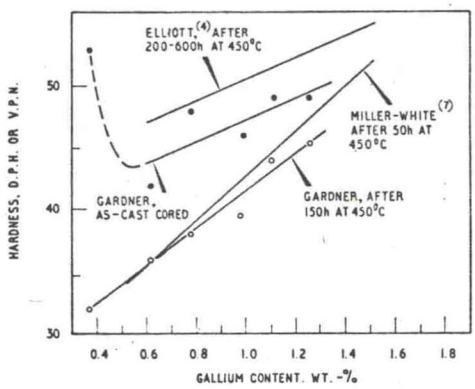
c. the tendency to form alpha phase upon application of pressure decreases,

d. the pressure applied during a hardness test increases.

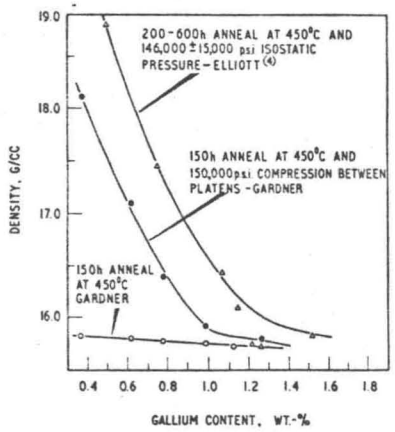
3.3.3 The observed hardness versus composition curve in cored alloys is the resultant of the combined influence of the above factors. It is expected that the hardness curves for as-cast, cored and the homogenized specimens will eventually coincide at the point where alpha formation no longer occurs in the cored area of minimum gallium content.

3.3.4 A comparison of the above data for homogenized alloys with those of Miller and White[7], and Elliott and Gschneidner[4] is illustrated in Fig 6-D. The Miller-White and Gardner data

compare very well; however, for some unknown reasons the Elliott-Gschneidner data are considerably higher.



6-D



6-E

Fig 6-D Effect of Gallium Content on Hardness

Fig 6-E Effect of Composition and 150,000 lb/in^2 Pressure on Density in Plutonium-Gallium Alloys.

3.4 Metastability

3.4.1 The effect of the application of a pressure of 150,000 lb/in^2 to homogenized specimens on their density-composition relationship is illustrated in Fig 6-E. As gallium content decreases, the effect of pressure on density increases at a rapid rate because of increased alpha phase formation. Similar data obtained earlier by Elliott and Gschneidner[4] generally shows the same result except for a substantial vertical displacement, Fig 6-E. Since their pressures were nearly the same as that in the present work, the difference between the two curves may be the result of the difference between isostatic compression and that between platens.

3.4.2 The fact that alpha phase forms as a consequence of pressure in alloys containing less than 1.2 wt.-% Ga, raises the question of the stability of alpha-delta phase mixtures at room temperature and their response to annealing temperature and time, followed by storage at room temperature. Since a wide range of gallium content exists in cored alloys, its

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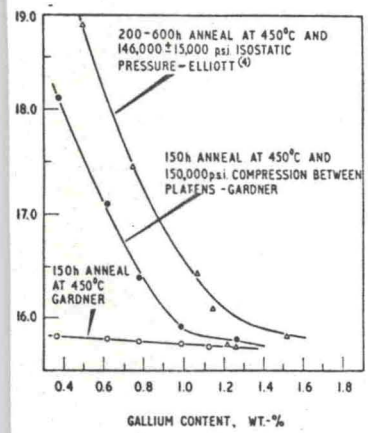

6-E

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degree of stability was expected to be lower than homogenized alloys. Therefore the stability of alpha-delta phase mixtures was studied in both alloy conditions.

Anneal Time, h	Density, g/c.c.				
	As-Rolled	150°C	200°C	250°C	300°C
0.167	16.08	16.05	15.92	15.86	15.75
6.0	16.08	16.05	15.91	15.86	15.75
24	16.19	16.12	16.02	15.84	15.75

Table 6-III Effect of Anneal Time and Temperature on Density at Room Temperature in 94% Cold Rolled Plutonium-0.94 wt.-% Gallium Alloy

3.4.3 Cored alloy - Cored Pu-0.94 wt.-% Ga alloy having a density of 15.75 g/c.c., was cold rolled to 94% reduction forming approximately 10% alpha phase. It was then annealed for times up to 24 h at temperatures up to 300°C, Table 6-III. The density changes indicate that a 10 min anneal was sufficient to produce the maximum phase transformation that can occur at any given temperature. In addition, densities after the 150, 200 and 250°C anneals indicate that either beta and gamma phases were being retained to room temperature or the amount of transformation of alpha phase to delta phase was proportional to anneal temperature. To test the hypothesis of retention of beta and gamma, cored 0.37 wt.-% Ga alloy was compressed with 150,000 lb/in^2 at room temperature. A density of 18.20 g/c.c. was obtained indicating that approximately 60% alpha phase was present. During a 10 min anneal at 200°C in the beta phase region, the density decreased to 16.27 g/c.c. which corresponds to the presence of approximately 24% beta phase after the anneal. During phase identification studies using x-ray diffraction, no beta phase could be detected. A similar study after a 250°C anneal to form gamma phase indicated that gamma phase was not being retained to room temperature. Since beta and gamma phases were not retained to room temperature after appropriate anneals of alpha-delta phase mixtures, it was decided to perform additional annealing

experiments designed to provide data leading to an explanation of the phenomena.

3.4.4 Portions of the cored Pu-0.94 wt.-% Ga alloy, cold rolled 94%, which was used earlier in this study and contained alpha phase, were given 10 min anneals at temperatures from 200 to 300°C. The density data, obtained at room temperature after the anneal, decrease with increasing annealing temperature up to approximately 280°C, where the normal delta phase density is achieved, Fig 6-F. The following mechanism is proposed to account for the observed relationship. At temperatures up to 280°C, the alpha phase originally present transforms to beta or gamma phase plus delta phase. The amount of delta phase formed in this way is related to temperature. As temperature increases the proportion of beta or gamma transforming to delta phase increases until at approximately 280°C the transformation is complete. During cooling to room temperature from temperatures below 280°C, the remaining beta or gamma phases transform back to alpha phase while the delta phase which formed from beta or gamma remains delta phase, at least during the interval of density determination. The immediate net result is a decrease in density of the alpha plus delta phase mixture, the higher the anneal temperature the greater the decrease in density.

3.4.5 The question of interest, as indicated earlier, is the degree of stability of the annealed and cooled alloy during room temperature storage. Accordingly, the specimens given the above annealing treatment were stored at room temperature and, in the case of the 200 and 250°C anneals, specimens were subjected to up to three cycles of cold treatment from room temperature to -54°C, Fig 6-G. The initial density decrease, from 16.30 or 16.31 g/c.c. to 15.94 or 16.95 g/c.c., indicates that approximately 5% alpha phase was present at room temperature after the 200°C anneal. After the 250°C anneal, however, less than 1% alpha phase was present. The rate of density change during storage was much higher after the 200°C anneal than after the 250°C anneal, indicating that a larger amount of the delta phase formed from the beta phase at 200°C was exhibiting metastable behaviour. As annealing temperature is

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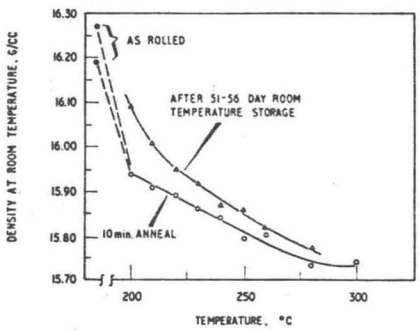
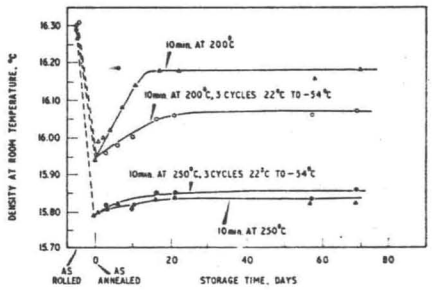
increased from 200 to 250°C, sufficient gallium diffusion to the gallium poor areas apparently occurs to substantially decrease the amount of delta phase capable of exhibiting metastable behaviour during room temperature storage. This was considered reasonable since the areas low in gallium in the cored alloy have the greatest tendency to exhibit metastable delta to alpha transformation. The three cold treatment cycles improved stability after the 200°C anneal, but did not measurably affect stability after the 250°C anneal.



6-F



6-G

Fig 6-F Effect of Anneal Temperature and Storage Time on Density of Plutonium-0.94 wt.-%, Cold Rolled, Cored Alloy

Fig 6-G Effect of Anneal Temperature, Cold Treatment and Storage Time on Delta to Alpha Transformation in Plutonium-0.94 wt.-% Ga Alloy.

3.4.6 Looking again at Fig 6-F, the densities obtained after 51-56 days of room temperature storage are expressed as a function of anneal temperature. The decrease in density after storage with increasing anneal temperature up to 280°C is further evidence for the proposed effect of diffusion.

3.4.7 Homogenized alloy - Since the instability of annealed alpha-delta phase mixtures is related to the amount of low gallium area in the cored grains, it was decided to study the stability of annealed alpha-delta mixtures in homogenized systems. To produce large and varying amounts of alpha phase in homogenized alloy during the application of pressure, the series of plutonium-gallium alloys ranging from 0.37 to

1.26 wt.-% Ga used in the compositional studies were employed. Prior to compressing with 150,000 lb/in^2, the alloys were annealed for 150 h at 450°C to ensure complete homogenization. The density measurements after the anneal and after the compression are presented in Table 6-IV with the approximate percentages of alpha phase produced. For compositions greater than 0.99 wt.-% Ga the amount of alpha phase produced was 1% or less.

Wt.-% Ga	Density, g/c.c.		% Alpha after Compression	T_f (3) °C
	After Anneal (1)	Annealed (1) & Compressed (2)		
0.37	15.83	18.11	62.0	250
0.62	15.81	17.08	35.0	260
0.78	15.78	16.40	17.0	230
0.99	15.76	15.92	4.5	240
1.12	-	15.75	-	-
1.26	15.73	15.78	1.0	-

(1) 150 hours at 450°C

(2) 150,000 lb/in^2

(3) Minimum anneal temperature required for completion of reaction

Table 6-IV Alpha Phase Formation During Compression of Homogenized Pu-Ga Alloys

3.4.8 The effect of annealing temperature on percent alpha phase retained to room temperature is illustrated in Fig 6-H. Again the proportion of delta phase forming from alpha, beta and gamma phases is related to anneal temperature. Complete transformation to delta phase occurs only after heating to approximately 260°C. In contrast to the cored alloys, it was determined that the delta phase thus formed in homogenized systems containing as little as 0.37 wt.-% Ga is stable. There was no measurable density change with respect to storage at room temperature for times up to three months.

3.4.9 The alpha $\rightleftharpoons$ delta transformation has been considered

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	% Alpha after Compression	T_f (3) °C
	62.0	250
	35.0	260
	17.0	230
	4.5	240
	-	-
	1.0	-

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by Lomer[9]. He proposed a correspondence matrix which relates the lattices, accounting for nearly all of the atomic portions and suggests that the transformation is martensitic.

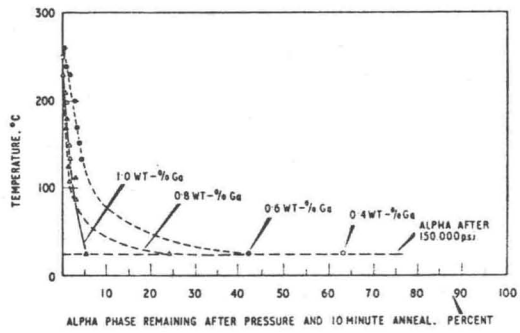

Fig 6-H Effect of 150,000 lb/in^2 Pressure on Alpha Phase Formation and Effect of Anneal Temperature on the Alpha to Delta Transformation.

4 Conclusions

4.1 A lattice parameter determination on solid specimens produces a 0.1% larger lattice parameter for a given composition than when annealed powder is used.

4.2 Lattice parameter and density data decrease with increasing gallium content while hardness increases.

4.3 The density data of all investigators are in good agreement while the hardness data contain discrepancies.

4.4 The compositional dependence of alpha phase formation after application of 150,000 lb/in^2 pressure was found to be in the same direction but displaced from that of a previous investigator. The method of alloy preparation may be the cause.

4.5 In cored alloys, alpha-delta phase mixtures formed by pressure are metastable both with respect to anneal temperature up to 280°C and subsequent room temperature storage. The increase in room temperature stability with increasing anneal temperature was attributed to gallium diffusion resul-

ting in an increase in gallium content in the areas originally low in gallium.

4.6 The alpha phase produced by the application of pressure to homogenized delta phase alloys transforms to delta phase in increasing amounts as the temperature is increased. In contrast to cored alloys, the delta phase thus formed does not revert to alpha phase during room temperature storage.

5 Acknowledgment

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