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Formation and recovery of irradiation and mechanical damage in stabilized δ-plutonium alloys

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Abstract

Based on the results of thermal expansion measurements of aged δ-phase Pu–Ga alloys and comparisons with historical bulk density, thermal expansion, and X-ray diffraction data of δ-phase Pu–Al and Pu–Ga alloys, it is being proposed that Pu-solute atom mixed ⟨1 0 0⟩ dumbbell interstitials, generated by damage processes, induce volumetric swelling and impede $\delta \rightarrow \alpha'$ Martensitic transformation in these alloys. Damage induced volumetric swelling in δ-phase Pu–Al and Pu–Ga alloys is distinct from swelling induced by He bubble formation in that it appears to saturate as a function of damage eventually reaching a constant value which is linearly dependent on solute element atomic concentration. Based on arguments offered in this work, each Pu-solute atom mixed ⟨1 0 0⟩ dumbbell interstitial creates a strain field of approximately 4.8 times the Pu atomic volume over some 20 Å in extent. As the interstitial concentration increases with damage, so does the volumetric swelling up to the point of saturation with a concentration of 1% of the solute atomic concentration. These interstitials appear to anneal out at temperatures of 150 to 200 °C. This relatively high thermal stability is possibly due to an altered local bonding environment for the Pu-solute atom interstitial pair and first-nearest neighbors in response to reduced coordination number and lattice strain field.
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1. Introduction

In 1958, Bochvar et al. authored a paper in which the binary phase diagram for the Pu–Al system exhibited a stabilized δ-phase field which extended to compositions no greater than 14 at.% and to temperatures no lower than 175 °C, at which point the alloy undergoes a eutectoid decomposition into β-Pu and Pu_3Al [1]. This observation sparked a controversy over the correct δ-phase field portion of the Pu binary phase diagram, a controversy which has not yet been resolved but is likely related to crystallographic changes associated with self-irradiation damage. This δ-phase field meta-stability was sited in 1961 by Ellinger, Land and Miner when discussing observed X-ray diffraction data for stabilized δ-phase Pu–Al alloys, which had been aged at ambient temperatures for 10 years [2]. In this work, Ellinger et al. witnessed an aging related lattice expansion (∼0.1%) in Pu–Al alloys varying in composition from 11.5 to 12.8 at.% and attributed this swelling to lattice defects. Similarly, in 1975 Chebotarev and Utkina reported observing damage induced lattice expansion (<0.2%) in δ-phase Pu–Al and Pu–Ga alloys, expansion which saturated as a function of damage to a constant value increasing linearly with alloy concentration, whether the materials had undergone mechanical shear or self-irradiation damage [3]. Based on this experimental data, Chebotarev et al. attributed this lattice expansion to a crystallographic modification, a lattice distortion in the face-centered cubic (fcc) atomic positions. Later work by Inozemtsev et al. showed lattice swelling of similar magnitude in Pu–Al and Pu–Ga alloys exposed to neutron-irradiation [4]. In all reported cases of this behavior, the volumetric expansion was relieved by annealing the alloys at temperatures up to 250 °C [2–4].

Interstitial defects in crystalline structures have often been sited as explaining thermo-physical and mechanical changes which result from the accumulation radiation or mechanical damage. The most stable interstitial atomic configuration of fcc metallic elements is that of the ⟨1 0 0⟩ split interstitial or ⟨1 0 0⟩ dumbbell interstitial [5] where two atoms share an axis aligned along the crystallographic ⟨1 0 0⟩ direction. These defect structures were first uniquely identified in electron-irradiated Al single crystals via diffuse X-ray scattering as inducing

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a lattice expansion and having a tetragonal symmetry [6]. Many studies of radiation damage in fcc alloys have shown that these defect structures can form at substitution sites as a mixed ⟨1 0 0⟩ dumbbell interstitial consisting of an impurity or solute atom and a host atom and these structures are stable to temperatures of 200 K or higher [7]. No matter what atoms combine to form the ⟨1 0 0⟩ dumbbell interstitials in fcc metals, these defect structures not only generate lattice swelling, but also induce unusual changes in the lattice properties such as decreased elastic moduli, large thermal atomic displacements, and decreased Debye-Waller factors [7].

In addition to the volumetric swelling in the damaged lattice of δ-phase Pu–Al and Pu–Ga alloys described above, data from elastic constant [8], X-ray diffraction [9], EXAFS [10], and neutron diffraction [11] experiments in δ-phase Pu–Ga alloys aged at ambient temperature have shown decreased elastic moduli, tetragonal lattice distortions, altered Pu–Ga and Pu–Pu bond lengths, lattice micro-strain, and large thermal atomic displacements correlating with similar radiological and thermo-mechanical histories. Since these changes can be attributed to altered dynamics in a lattice possessing a strain field of preferred direction as occurs in the case of ⟨1 0 0⟩ dumbbell interstitials in fcc metals [7], these data strongly suggest the existence of mixed ⟨1 0 0⟩ dumbbell interstitial in δ-phase Pu–Al and Pu–Ga alloys when compared with other fcc alloys containing mixed ⟨1 0 0⟩ dumbbell interstitial induced by either radiological or mechanical damage [7]. The existence of these interstitials could also explain the absence of void swelling in aged Pu–Al and Pu–Ga alloys under radiation damage conditions, as the formation of these defect structures has been suggested to reduce void swelling [12]. The current proposal is counter to conventional isochronal annealing studies in δ-phase Pu–Ga alloys in which irradiation damage accumulated at cryogenic temperatures is completely resolved below 310 K [13]. If mixed ⟨1 0 0⟩ dumbbell interstitial are the source of damage induced volumetric swelling in δ-phase Pu–Al and Pu–Ga alloys, then these defects must exhibit relatively high thermal stability. This thermal stability may be found in the altered local bonding environment for the Pu-solute atom interstitial pair and first-nearest neighbors in response to reduced coordination number and lattice strain field.

The thermal expansion experiments described here were designed to explore the phenomenon of self-irradiation damage related volumetric changes and other effects of self-irradiation in δ-phase Pu–Ga alloys. Damage caused through mechanical, neutron-irradiation, and self-irradiation damage in δ-phase Pu–Al and Pu–Ga alloys is seen to induce swelling which is annealed at relatively high temperatures. These thermal expansion studies of aged δ-phase Pu–Ga alloys performed as a function of temperature allow for further quantification of the damage related volumetric changes and further exploration into the impact of radiation damage on the δ → α′ Martensitic transformation in these alloys.

2. Experimental description and results

Thermal expansion measurements were performed on samples from cast lots of δ-phase Pu–Ga alloy (2.0 at.% Ga) with low (<0.03%) and high (7.5%) ^{238}Pu isotopic content for the purposes of determining the effects of increasing the rate of self-irradiation damage to the lattice [14,15]. Other than Pu isotopic content variation, the analytic chemistry of these alloys were similar with similar trace impurity compositions. All samples utilized for these experiments were homogenized at a temperature of 450 °C for more than 10 h to assure uniform Ga concentration and characterized by immersion density to assure a single phase alloy. Samples were then sectioned with a low speed diamond wheel saw and hand lapped to final finish to ensure minimal formation of α′-phase Pu on the surface due to mechanical damage. There was no evidence of α′-phase Pu due to mechanical damage in above ambient temperature thermal expansion data; however, sample exposure to below ambient temperature did exhibit evidence of δ → α′ Martensitic transformation in thermal expansion data which is typical for Pu–Al and Pu–Ga alloys in this composition range [16].

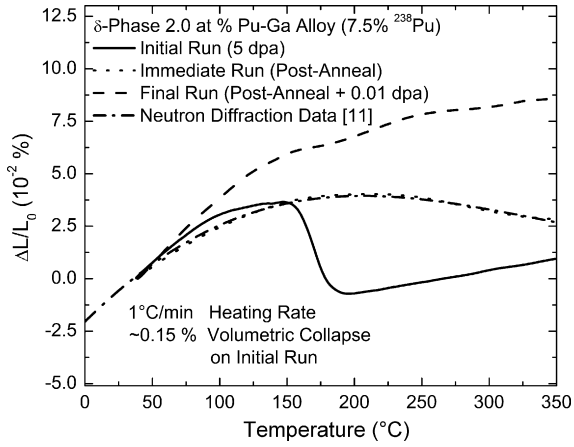

Fig. 1. Thermal expansion data for self-irradiated δ-phase Pu–Ga alloy before and after high temperature annealing (>450 °C). Crystallographic data [11] is included to show that annealing removes bulk defects.

Thermal expansion measurements were conducted in a Netzsch 402 dilatometer under high purity He gas atmosphere to minimize surface oxidation and to ensure good thermal control. These measurements were made on separate samples at low temperatures (−160 °C < T < 150 °C) and high temperatures (30 °C < T < 450 °C). Heating and cooling rates of 1 °C/min were utilized. For the low temperature dilatometer run, a hold time of 1 h. was employed at the lowest temperatures to allow the alloys to carry the δ → α′ Martensitic transformation to completion. All runs were corrected for the thermal expansion of the silica sample holder.

The results from the high temperature and low temperature thermal expansion experiments can be seen in Figs. 1 and 2, respectively. The thermal

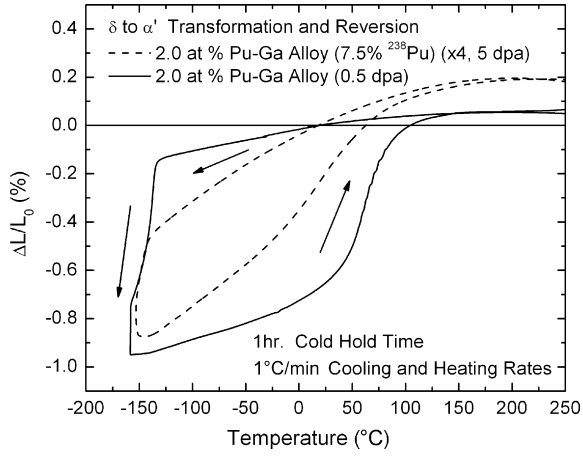

Fig. 2. Thermal expansion data for self-irradiated δ-phase Pu–Ga alloy of differing ^{238}Pu isotopic content exhibiting δ to α′ transformation on cooling and reversion on subsequent heating.

expansion data in Fig. 1 comes from a δ-phase Pu–Ga alloy (2.0 at.% Ga) sample with 7.5% ^{238}Pu isotopic content which was exposed to approximately 5 disintegrations per atom (dpa) of self-irradiation damage [14]. This sample progressed through a series of heating runs, each time being brought back to room temperature prior to being heated for the next run. All sample lengths were normalized to the room temperature value prior to each run. The initial run of the sample exhibited a volume collapse of approximately 0.15% between the temperatures of 150 and 200 °C. After this volume collapse, the sample appears to expand almost linearly with temperature up to the melt of the Pu_6Fe eutectic (not shown in Fig. 1) which tends to sit in the inter-granular spaces. This linear thermal expansion is assumed to be a interplay between the lattice contraction due to the negative thermal expansion coefficient possessed by this alloy Pu–Ga and the lattice expansion which occurs with He bubbles [17]. This sample was then taken to 450 °C and oven cooled to 30 °C.

Within 24 h, this sample with 7.5% ^{238}Pu isotopic content was heated again under identical conditions to 450 °C and oven cooled to 30 °C. During this run the sample exhibited strikingly similar behaviour to that of a sample of δ-phase ^{242}Pu–Ga alloy (2.0 at.% Ga) as determined by neutron diffraction [11]. This would indicate that the high temperature exposure during the initial run had annealed those defects observable by dilatometry and that bulk and crystallographic thermal expansion data agree well in this temperature range. The sample does show the negative thermal expansion coefficient at temperature higher than 200 °C which is typical for 2.0 at.% Ga stabilized δ-phase Pu–Ga alloy. At this point the He bubbles, which do exist, are so large and have such a low gas pressure that they tend to act as holes in the bulk which contribute only slightly to the overall thermal expansion of the sample [17,18].

This same sample with 7.5% ^{238}Pu isotopic content was then allowed to sit under inert atmosphere for approximately 1 month during which time it accumulated some 0.1 dpa self-irradiation damage. The sample was heated again under identical conditions to 450 °C and oven cooled to 30 °C. During this heating cycle, the sample exhibited behaviour somewhat similar to the initial heating run although without the associated volume collapse. However, the sample did exhibit a small yet perceivable variation from linear behaviour in the region from 150 to 200 °C perhaps indicating some defect annealing occurring. Once again, there is an almost linear thermal expansion at higher temperatures due to the combined response of the Pu–Ga alloy lattice and He bubbles.

The low temperature thermal expansion data in Fig. 2 comes from two δ-phase Pu–Ga alloy (2.0 at.% Ga) samples, one containing <0.03% ^{238}Pu isotopic content and the other containing 7.5% ^{238}Pu isotopic content, respectively. Both samples were observed to have a $\delta \rightarrow \alpha'$ transformation onset temperature at approximately −135 °C. However, almost eight times less volume (approximately 0.3%) transformed to α' in the alloy with 7.5% ^{238}Pu than did in the alloy containing <0.03% ^{238}Pu isotopic content, which exhibited approximately 2.6% by volume α' transformation. On heating to 250 °C, both samples exhibited full reversion ($\alpha' \rightarrow \delta$) within experimental error initiating at 50 °C and terminating at 150 °C.

3. Results and discussion

The formation of the mixed ⟨1 0 0⟩ dumbbell interstitial in δ-phase Pu–Al and Pu–Ga alloys in response to mechanical, neutron-irradiation, and self-irradiation damage is very intriguing as it would explain the linear increase in lattice constant with a linear increase in solute concentration as seen by many researchers in damaged alloys [2–4,16,18,19]. In order to gain insight of the proposed defect structure and the implications of its existence, the lattice distortion due to the interstitial should first be determined. Based on the theory of diffuse X-ray scattering on a defect [6], such a defect generates a change in the fcc lattice parameter of

$$\frac{\Delta a}{a} = \frac{C_D P}{3V_A(c_{11} + 2c_{12})} \quad (1)$$

where C_D is the atomic fraction of interstitials or defects, P represents the trace of the dipole tensor, V_A the host lattice atomic volume, and c_{11} and c_{12} are the longitudinal and shear elastic constants for the host lattice. For such defects in δ-phase Pu–Al and Pu–Ga alloys, the values $V_A = V_{Pu} = 1.85 \times 10^{-23}$ cm^3 based on a Pu atomic radius of 1.64 Å [20], and $c_{11} = 36.3$ GPa and $c_{12} = 26.7$ GPa [21] must be employed. The trace of the dipole tensor P for most fcc metals ranges from 40 to 60 eV for a mixed ⟨1 0 0⟩ dumbbell interstitial depending on the interatomic potential utilized in calculating the atomic displacements [7]. Therefore, a value of $P = 50$ eV will be assumed for δ-phase Pu–Al and Pu–Ga alloys. Given these values, the concentration dependence for fcc lattice parameter change for δ-phase Pu–Al and Pu–Ga alloys due to mixed ⟨1 0 0⟩ dumbbell interstitials is

$$\frac{\Delta a}{a} = 1.6C_D. \quad (2)$$

From the relationship between the lattice constant and volumetric changes in an isotropic material,

$$3\frac{\Delta a}{a} = \frac{\Delta V}{V} \quad (3)$$

the volume change in δ-phase Pu–Al and Pu–Ga alloys ΔV per one mixed ⟨1 0 0⟩ dumbbell interstitial is determined to be $4.8\,V_{Pu}$. The associated strain field of the interstitial in the local lattice, modelled as a defect of "strength" $4.8\,V_{Pu}/4\pi$, falls off as the inverse of the radial distance squared [22], and has magnitude of 1% or 0.046 Å at a distance of 10 Å from the defect, so that the interstitial and the associated lattice distortion may be considered a sphere of diameter 20 Å.

When a study is made of a historical collective of lattice and bulk expansion data, a strong correlation is suggested between the fcc lattice parameter change for δ-phase Pu–Al and Pu–Ga alloys and volume fraction of solute atoms in these alloys. This correlation is largely independent of the source of the damage, whether due to self-irradiation [2,3,16,18], mechanical shear [3], rolling [19], or neutrons irradiation [4]. If the proposal for the formation of mixed ⟨1 0 0⟩ dumbbell interstitials in δ-phase Pu–Al and Pu–Ga alloys has validity, then in order to determine the concentration of such defects, there must be a determination of the relation of defect volume fraction to volume fraction of solute atoms in solution in the δ-phase alloys where swelling has be observed. In Fig. 3, the historic damage accumulation data, expressed as $\Delta V/V$, is plotted as a function of alloy solute volume fraction in δ-phase Pu–Al and Pu–Ga alloys. The normalization of alloy solute volume fraction for δ-phase Pu–Al and Pu–Ga alloys originates with δ-phase alloys being substitutional solid solutions with Al and Ga atomic volumes of 16.6 Å^3 and 19.6 Å^3 [23], respectively, in a host lattice of Pu atoms with atomic volume 18.5 Å^3 [20]. These Pu–Al and Pu–Ga solutions exist at concentrations up to 11% by volume [2]. In order to determine the ratio of solute atoms incorporated into mixed ⟨1 0 0⟩ dumbbell interstitials to solute atoms in solution, consider the following. From Eqs. (2) and (3), for mixed ⟨1 0 0⟩ dumbbell

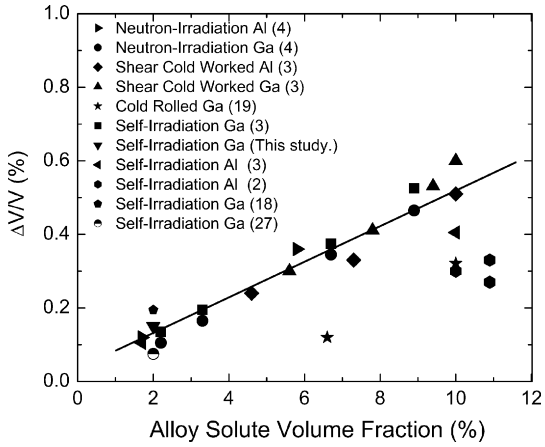

Fig. 3. Historic data and data from this study of $\Delta V/V_{Pu}$ plotted as a function of alloy solute volume fraction in δ-phase Pu–Al and Pu–Ga alloys (reference number identifies data source). The line exhibits a linear fit to the data with slope (see text for discussion).

interstitials in δ-phase Pu–Al and Pu–Ga alloys

$$\frac{\Delta V}{V_{Pu}} = 4.8 C_D. \tag{4}$$

As seen in Fig. 3, there is a strong direct correlation between $\Delta V/V$ and alloy solute volume fraction in δ-phase Pu–Al and Pu–Ga alloys with radiation or mechanical damage, so that to good approximation

$$\frac{\Delta V}{V_{Pu}} = 0.0165 \frac{V_S}{V_{Pu}} C_S. \tag{5}$$

The linear fitting coefficient 0.0165 was determined by a best fit to the data in Fig. 3. There are some outlying data in Fig. 3 which were not included in the fit. It is intriguing that the data for these samples do lie on a line of approximately the same slope of $\Delta V(C_S V_S)^{-1} = 0.0165$ as the other data with a $\Delta V/V$ offset of -2×10^{-3} at zero age. Perhaps the simplest explanation is that the damage in these samples had not generated the necessary concentration of interstitials to reach the point of $\Delta V/V$ saturation as observed in the other studies.

By setting the right hand side of Eq. (4) equal to the right hand side of Eq. (5) one finds

$$\frac{C_D}{C_S} = \frac{0.049}{4.8} \frac{V_S}{V_{Pu}}. \tag{6}$$

For Al, Eq. (6) yields 0.0093. The calculations here suggest that for alloys which have had lattice radiation or mechanical damage, 0.93% of Al atoms in solution are incorporated into mixed ⟨1 0 0⟩ dumbbell interstitials. For Ga Eq. (6) yields 0.011, implying 1.1% of Ga atoms in solution are incorporated into mixed ⟨1 0 0⟩ dumbbell interstitials. Based on these estimates, the approximate uniform concentration of these 20 Å defects would be $4.4 \times 10^{19}\,cm^{-3}$ as compared with the He bubble 20 Å concentration $1.5 \times 10^{17}\,/cm^{-3}$ reported by Schwartz et al. [24] Therefore, these defects should be approximately 300 times more numerous than He bubbles. Such defect structures are likely to impact phase transformations as witnessed in this thermal expansion study due to associated lattice strain and to alter plastic deformation and mechanical yield as seen in δ-phase Pu alloys due to dislocation pinning effects [25,26].

There are a matter of issues which must be addressed to justify the existence of mixed ⟨1 0 0⟩ dumbbell interstitials in damaged δ-phase Pu–Al and Pu–Ga alloys. One is the matter of a how crystalline defect which has an associated volume of lattice distortion can be observed by both bulk density and X-ray diffraction if the defects are not necessarily periodic. Eshelby addressed this issue by examining a uniform density of such defects in a spherical crystal and found that such a configuration would give equal X-ray and bulk geometrical expansions [22]. The second issue to be addressed is that in the study of damage in δ-phase Pu–Ga alloys by isochronal annealing experiments, consisting of accumulating damage at low temperatures and measuring electrical resistivity (e.g., a damage proportional quantity) with ever increasing temperatures, δ-phase Pu–Ga alloys exhibit behaviour to suggest that vacancies, interstitials, and dislocations are annealed in stages well below the temperatures at which these defects appear to exist. Conventional isochronal annealing studies in δ-phase Pu–Ga alloys exhibit irradiation damage, accumulated at 10 to 20 K, to be completely annealed at a Stage V recovery of 310 K [13]. Perhaps the thermal stability of the mixed ⟨1 0 0⟩ dumbbell interstitials defect may be explained by the mechanics and temperature scales of formation. As radiation or mechanical damage is suffered by the lattice, the solute atom becomes wedged in an interstitial position. Due to the relatively high temperatures (i.e., ambient temperatures) at which this event takes place, the lattice then has time to accommodate the resultant lattice distortion without extensive lattice fracture and broken bonds as might be residual from damage at cryogenic temperatures. The Pu-solute atom interstitial pair, first-nearest neighbors, and the extended lattice acquire an energy balance through a reduced coordination number and lattice strain field. This damage accumulated at higher temperature appears to form these stable defect structures perhaps lending to the belief in the meta-stability of δ-phase Pu–Al and Pu–Ga alloys as proposed by Bochvar et al. [1].

Theoretical and experimental research has been conducted in δ-phase Pu–Ga alloys to explore the interaction between He bubble formation and radiation damage [17,24,27], but the mechanics of this phenomenon is not completely understood. However, the He bubble growth and population in stabilized δ-phase Pu alloys is apparently moderated by continuous radiation damage of the lattice through the mechanism of resolution [17]. Perhaps the mechanism that drives the total number of Pu-solute atom mixed ⟨1 0 0⟩ dumbbell interstitials to saturation in δ-phase Pu–Al and Pu–Ga alloys is also radiation damage resolution. In such a picture, the continuous damage and annealing of damage in the lattice eventually generates a fixed concentration of mixed ⟨1 0 0⟩ dumbbell interstitials which is proportional to the solute concentration. Further increase in the concentration of interstitials is limited by the continuous creation and annihilation of interstitials and saturation occurs. This phenomenon requires further investigation. In addition to the thermal expansion data presented here as an argument for mixed ⟨1 0 0⟩ dumbbell interstitials in damaged δ-phase

Pu–Al and Pu–Ga alloys, there are data acquired through other techniques which support this proposal. Migliori et al. have observed elastic constant decreases with time in δ-phase Pu–Ga alloys, an indication of defect structure formation as a function of radiation damage [8]. These elastic constant decreases vary in magnitude with temperature, thus indicating a subtle interplay between radiation damage formation and damage annealing processes which are constantly occurring at ambient temperatures.

4. Conclusions

The existence of ⟨1 0 0⟩ dumbbell interstitials, the most stable interstitial formation for the fcc lattice, accounts for thermo-physical and mechanical changes which result from mechanical and radiation damage accumulation. Many studies of radiation damage in fcc alloys have shown that these defect structures can form at substitutional impurity sites as a mixed ⟨1 0 0⟩ dumbbell interstitial consisting of an impurity atom. The observed thermal expansion behaviour of aged δ-phase Pu–Ga alloys when compared with historical bulk density and X-ray diffraction data of δ-phase Pu–Al and Pu–Ga alloys, strongly suggests the generation of Pu-solute atom mixed ⟨1 0 0⟩ dumbbell interstitials by damage processes, thus inducing volumetric swelling and impedance to $\delta \rightarrow \alpha'$ Martensitic transformation. Based on experimental data, both recently acquired and recorded in scientific literature from mechanical, neutron-irradiation, and self-irradiation damage data, an argument for the formation of these defect structures has been established. Estimates show that these Pu-solute atom mixed ⟨1 0 0⟩ dumbbell interstitials have an associated volume change of $\Delta V = 4.8\, V_{Pu}$, extend approximately 20 Å in diameter, the total population comprising approximately 1% of the solute atoms in these alloys. Volumetric swelling, as observed in these materials in all cases of damage, is removed at temperatures in the range of 150–200 °C. The relatively high thermal stability of these proposed defect structures, when compared to conventional isochronal annealing studies, may be originate in the reduced coordination number and lattice strain field associated with an altered local bonding environment for the Pu-solute atom interstitial pair and first-nearest neighbors. The concentration of these Pu-solute atom mixed ⟨1 0 0⟩ dumbbell interstitials, which appears to saturate as a function of damage to a value proportional to the solute concentration, is likely controlled by radiation damage resolution.

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