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Supporting evidence for double-C curve kinetics in the isothermal $\delta \rightarrow \alpha'$ phase transformation in a Pu–Ga alloy

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Abstract

Time-temperature-transformation (TTT) diagrams for the $\delta \rightarrow \alpha'$ transformation in a number of Pu–Ga alloys were first reported in 1975 by Orme et al. Unlike typical single-C curve kinetics observed in most isothermal martensitic transformations, the Pu–1.9 at.% Ga alloy exhibits two noses, and thus double-C curve kinetics. The authors attributed the occurrence of the double-C to a difference in mechanism: a massive transformation for the upper C and a martensitic transformation for the lower C. Since that time, the nature, and the existence of the double C have received only limited attention. The results of Deloffre et al. suggest a confirmation of this behavior, but the fundamental origin of the double C remains unknown. Here, we apply differential scanning calorimetry (DSC) as an alternative approach to acquiring the TTT data and our experimental evidence suggests a confirmation of the double-C behavior after 18 h of isothermal hold time. In addition, we report three exothermic peaks corresponding to transformations during cooling at 20 °C/min prior to the isothermal holds. These three peaks are reproducible and suggest a number of possibilities for the origin of the unique kinetics: α' forms with different morphologies, or from different embryos in the upper and lower C curves; α' forms directly in one C curve and forms via an intermediate phase in the other C curve; the two C curves result from α' forming by two or more distinct mechanisms (e.g., massive and martensitic transformations).

Keywords: Actinide alloys and compounds; Metals and alloys; Solid state reactions; Phase transitions; Calorimetry

1. Introduction

In many Pu–Ga alloys, the phase observed at 25 °C is the face-centered cubic δ-phase. However, on-going research concerning the equilibrium phase diagrams of the Pu–Ga and Pu–Al systems has revealed that the δ-phase is not in thermodynamic equilibrium below 97 °C as many have thought since the Pu–Ga phase diagram was first reported by Ellinger et al. [1]. Rather, Chebotarev et al. [2] then Timofeeva [3] and then Hecker and Timofeeva [4] showed that the metastable δ-phase decomposes to the monoclinic α-phase and the tetragonal Pu_3Ga intermetallic compound via a eutectoid transformation below 97 °C. The kinetics of this decomposition are exceedingly slow, estimated to be in excess of 10,000 years [3]. Thus, it is not surprising that we typically observe just the metastable δ-phase at ambient conditions; no experimental observations of the eutectoid decomposition have been reported in naturally aged materials.

At sub-ambient temperatures, the metastable δ-phase in a Pu–1.9 at.% Ga alloy undergoes a partial, or incomplete, phase transformation to the metastable α' phase [5]. This monoclinic α'-phase is structurally similar to the α-phase in pure Pu, but α' has Ga supersaturated in the lattice, whereas α does not contain Ga [6]. The presence of Ga in α' gives rise to an expanded cell in which the lattice parameters increase with increasing Ga contents [6]. The kinetics of this isothermal transformation were measured using dilatometry and electrical resistivity by Orme, Faires, and Ward in 1975 [5] and were described in a series of time-temperature-transformation (TTT) diagrams for Pu–1.4 at.% Ga and Pu–1.9 at.% Ga alloys in the temperature range +60 °C to −155 °C. The TTT diagram for the Pu–1.9 at.% alloy is reproduced in Fig. 1. Unlike typical single-C curve kinetics observed in most isothermal martensitic transformations [7] and in Pu–0.6 at.% and Pu–0.7 at.% Ga alloys [5], the Pu–1.9 at.% Ga alloy exhibits two noses (i.e., two temperatures where the transformation rate is highest), and thus

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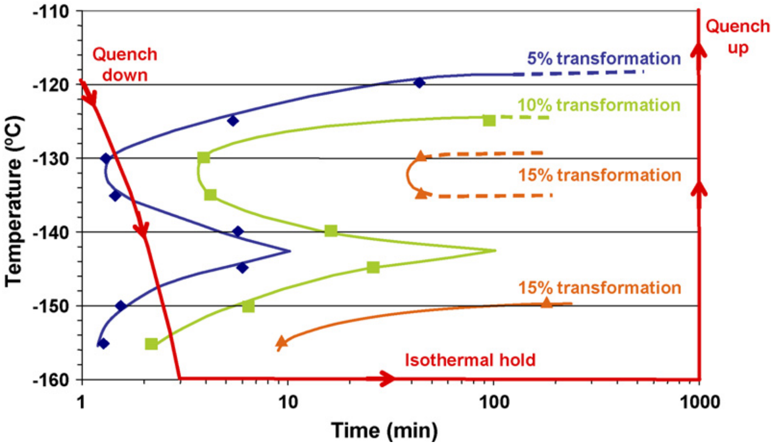

Fig. 1. Time–temperature-transformation diagram for a Pu–1.9 at% Ga alloy [5]. This TTT diagram shows the unusual double-C curve kinetics for the $\delta \rightarrow \alpha'$ transformation. The dark line with arrows shows the thermal profile of an experiment with an 18 h isothermal hold at −160 °C.

Table 1

A summary of the differences in α' formed in the upper- and lower-C curves observed by Deloffre

Pu–1.2 at% Ga alloy		Pu–1.9 at% Ga alloy	
Upper C	Lower C	Upper C	Lower C
Lathe morphology	Lenticular morphology	Wider particles	Thinner particles
Heterogeneous nucleation on grain boundaries	Heterogeneous nucleation	Heterogeneous nucleation preferentially on grain boundaries	No preferential nucleation sites
Inhomogeneous distribution of α' particles within δ grains (preferential formation at grain boundaries)	Homogeneous distribution of α' particles within δ grains		
α' unit cell volume = 323.3(5) Å	α' unit cell volume = 324.7(5) Å		
α' contains some γ'-phase	No γ'-phase	No γ'-phase	No γ'-phase
Reverts to δ either indirectly ($\alpha' \rightarrow \beta' \rightarrow \gamma' \rightarrow \delta$) or directly ($\alpha' \rightarrow \delta$)	Reverts to δ directly ($\alpha' \rightarrow \delta$)	Reverts to δ directly ($\alpha' \rightarrow \delta$)	Reverts to δ directly ($\alpha' \rightarrow \delta$)

double-C curve kinetics. Orme et al. attributed the occurrence of the double-C to a difference in isothermal mechanism: a massive transformation forming α' in the upper C and a martensitic transformation forming α' in the lower C.

Deloffre et al. also investigated the nature of the $\delta \rightarrow \alpha'$ isothermal martensitic transformation with X-ray diffraction, optical microscopy, and dilatometry [8,9]. Although these experiments were not designed to collect data for a TTT diagram, the observations strongly suggest a confirmation of the double-C behavior. Table 1 summarizes the differences that Deloffre observed in the α' formed in the temperature regimes of the upper- and lower-C curves in two Pu–Ga alloys. In each alloy, the α' products formed in these two temperature regimes had distinctly different characteristics. This evidence strongly suggests double-C curve behavior. Deloffre concluded that the lower the isothermal hold temperature, the more the displacive transformation dominates over the diffusional transformation. At an intermediate temperature, the martensite growth occurs by displacive transformation.[1] Thus, Deloffre's conclusions suggest that the origin of the double-C behavior is different mechanisms (massive in the upper-C and martensitic and the lower-C) that form the α' product.

Recently, Sadigh and Wolfer [10] performed density functional theory calculations on the Pu–Ga δ-phase and observed in their calculations a significant lowering of the unit cell volume and total energy of the unit cell when the Ga atom occupied site 8 in the monoclinic α' structure relative to Ga located in the other sites. This led them to propose that the double-C curve kinetics resulted from preferential short-range Ga diffusion to the energetically favorable site 8 lattice site during the $\delta \rightarrow \alpha'$ transformation. Thus, for the upper-C, larger concentrations of this eighth variant can be obtained due to limited Ga diffusion. Below a certain temperature, this diffusion process to site 8 stops and the α'-phase with a random distribution of Ga is produced, resulting in the lower-C. This hypothesis has not been experimentally verified and it is questionable whether or not diffusion

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[1] Although Deloffre's [8,9] description of the martensitic eight $\delta \rightarrow \alpha'$ transformation implies that this transformation has both displacive and diffusional components, we believe that his intention was to describe reconstructive components of the transformation. A martensitic transformation is displacive and does not involve diffusion. However, reconstructive transformations, which include massive transformations, could involve small atomic rearrangements (on the order of one lattice spacing or larger). It follows that a reconstructive transformation would become more difficult at lower temperatures because thermal activation for atomic motion is small.

plays a role in the low temperature range of the transformation (<−100 °C). Thus, many questions regarding the origin of the double-C curve kinetics remain open.

Here, we investigate the $\delta \rightarrow \alpha'$ isothermal martensitic transformation behavior as a function of time and temperature using the technique of differential scanning calorimetry (DSC) for generating alternative TTT data. The goal of this paper is to provide experimental evidence suggesting the existence of the double-C curve kinetics for this transformation. Rather than reproducing the technique employed by Orme et al., we utilize a novel method for collecting and presenting TTT data. This simple technique uses a differential scanning calorimeter (DSC) and is described in detail. We also investigate the isothermal kinetics over a time span that is significantly longer than what Orme et al. considered (<200 min). Our work suggests that even after 18 h, the double-C behavior persists.

2. Experimental details

A single Pu–2.0 at% Ga alloy was used for all the DSC experiments described here. The impurity content, excluding gallium, was 1600 weight ppm, which includes 1000 ppm of δ-stabilizing elements (Al, Am, Ce, etc.) and 400 ppm of α-stabilizing elements (Np, U, etc.). The mass of the DSC sample was 250 mg and it was in thermal contact with the sample pan. For all of these experiments, the heating and cooling rates were 20 °C/min. This is the fastest rate that the DSC can maintain when cooling and heating through the entire temperature range (−160 to 350 °C).

Prior to any of the DSC experiments, the Pu–Ga sample was annealed at 430 °C for 12 h to produce a partially-uniform gallium distribution across the grains and to assure that the sample was entirely in the δ-phase. Before each experimental run, the sample was annealed at 375 °C for 8 h and then conditioned at 25 °C for 12 h. Previous work [11] demonstrates that conditioning treatments of at least 6 h at 25 °C provide the needed time and temperature conditions for reproducible α' formation upon cooling.

Because the Pu–Ga sample was repeatedly scanned and was not removed from the DSC after each cycle, a baseline scan with empty pans could not be performed after each scan, as is conventionally done in DSC experiments. As an alternative, a curve obtained by a third-order polynomial fit of the cooling curve in the temperature range of approximately 150 to −40 °C was subtracted from the raw heating data ($\alpha' \rightarrow \delta$ reversion) as the baseline. Conversely, a polynomial fit of the heating data in the range 0 to −90 °C was subtracted as the baseline from the cooling data ($\delta \rightarrow \alpha'$ transformation). In both cases, a straight line was subtracted to correct the slope. Although this method does not achieve absolute accuracy in the calculation of the heats of transformation or heat capacities, it provides a consistent method for comparing all of the transformation (and reversion) peaks. This comparison is sufficient for the analysis described here.

3. Acquisition of TTT data using a differential scanning calorimeter

To generate TTT diagrams, Orme et al. used resistometry to monitor the isothermal $\delta \rightarrow \alpha'$ transformation in real time [5]. The resistivities of the δ and α'-phases differ by approximately 30% (approximately 40% at room temperature [12,13]), and therefore, after the sample was quenched to the isothermal hold temperature, the resistance of the sample was monitored and the time was recorded when the resistance was consistent with 5%, 10%, and 15% transformation to the α'-phase.

Orme et al. presented their data in a traditional TTT diagram with log time plotted on the x-axis, temperature on the y-axis, and contour lines of constant amount of transformation plotted on the graph. One of these TTT diagrams for the $\delta \rightarrow \alpha'$ transformation in a Pu–1.9 at.% Ga alloy is shown in Fig. 1. To verify the existence of the double-C behavior observed by Orme et al., we devised an alternative method for acquiring time-temperature-transformation data. Thus, the method described below and utilized to collect the data presented here provides supporting evidence of the double-C behavior, rather than a simple reproduction of Orme et al.'s data [5].

The heat release from the exothermic $\delta \rightarrow \alpha'$ transformation during continuous cooling is small (the enthalpy (ΔH_t) of the transformation is approximately 0.85 kJ/mol and the heat is released over the course of about 3 min) [14], and it is not distinguishable from the baseline during isothermal holds. Thus, the progress of the isothermal $\delta \rightarrow \alpha'$ transformation (i.e., the amount of α' formed as a function of time) cannot be monitored in real time in a DSC. Instead, samples were quenched to the isothermal hold temperature at 20 °C/min (for the temperature range of interest, this is the fastest reliable quench rate in the DSC), held for 18 h, and then heated at 20 °C/min to 350 °C to revert the α' that formed during the isothermal hold to the δ-phase. Note, however, that the cooling rate is insufficient to entirely miss the nose of the upper-C (this is illustrated in Fig. 1). The amount of α' formed during the hold was inferred from the area of the $\alpha' \rightarrow \delta$ reversion peak on heating. Because the enthalpy of transformation for the $\alpha' \rightarrow \delta$ reversion (ΔH) is not well known, the specific amount of α' formed is not quantified in these experiments. Instead, the amount of transformation is given as the area of the $\alpha' \rightarrow \delta$ peak, which is assumed to be directly proportional to the amount of α' that reverted to the δ-phase.

There are three assumptions made in this technique for acquiring TTT data. First, we assume that all the products formed during the isothermal hold revert on heating. This allows us to use the $\alpha' \rightarrow \delta$ peak area as a measure of the amount of the transformation product formed during the isothermal hold. This assumption appears to be valid because we can cycle the sample repeatedly, and after a sufficient conditioning at room temperature [11], a reproducible amount of transformation is observed. Second, we assume that the amount of α' formed during the 20 °C/min quench and any additional α' formed during the heating portion of the cycle are small compared to the amount of α' formed during the isothermal hold. Fig. 2 shows DSC scans of the $\alpha' \rightarrow \delta$ reversion with no holding time in the worst case (−160 °C) and with holding times of 6 min and 18 h. The peak area ratio for a holding time at −160 °C for 18 h and a holding time of 0 min is greater than 3, whereas this ratio is greater than 8 for a holding temperature of −120 °C. Thus, this second hypothesis seems mostly valid. Third, we assume that the formation of α' during the quench does not in any way influence the formation of α' during the isothermal hold. Specifically, as the specimen passes through the temperature range of the upper-C, we assume that any small amount of α' that forms in the upper-C does not change the nucleation process or transformation mechanism of α' that forms during the isothermal hold in the lower-C temperature range. At this point, we have no evidence that this third assumption is valid.

The DSC technique for acquiring TTT data does not generate a data set that is conducive to the structure of a traditional-TTT

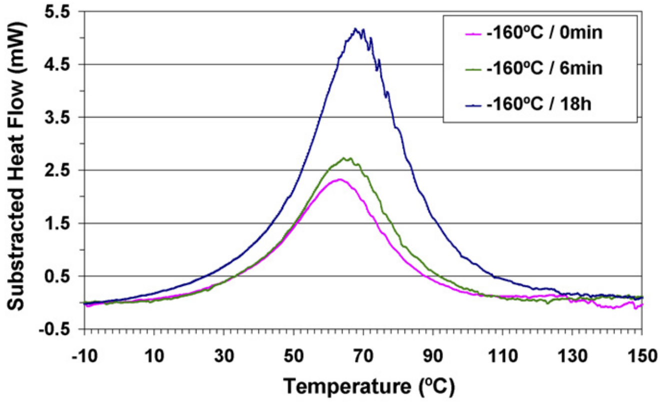

Fig. 2. DSC scans of the $\alpha' \rightarrow \delta$ reversion at −160 °C with no holding time and with holding times of 6 min and 18 h. The amount of α' formed during the 20 °C/min quench and any additional α' formed during heating are lower than the amount of α' formed during the 18 h isothermal hold.

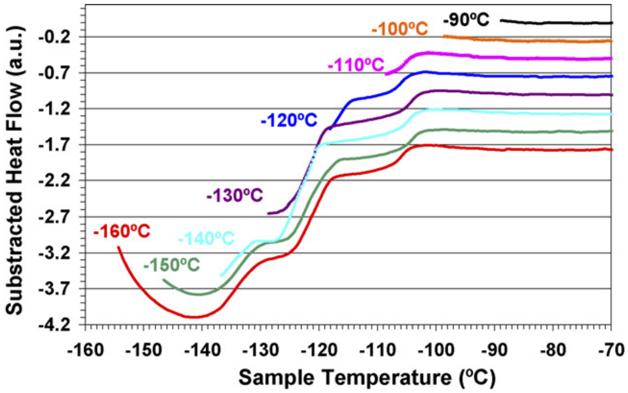

Fig. 3. DSC thermograms corresponding to the $\delta \rightarrow \alpha'$ transformation during continuous cooling at 20 °C/min before each 18 h anneal. This transformation is exothermic and begins at approximately −100 °C. The DSC data indicates that the transformation results in three overlapping exothermic peaks. The data have been offset along the y-axis for clarity.

diagram with time on the x-axis, temperature on the y-axis, and contour lines of constant amount of transformation plotted on the graph (Fig. 1). Rather, this data is more comprehensible when plotted on an alternative-TTT diagram with temperature on the x-axis, amount of transformation on the y-axis, and contour lines of constant time plotted on the graph. It should be noted that the alternative-TTT diagrams are not simply rotated versions of the traditional TTT diagrams. The data plotted on the two types of TTT diagrams are substantially different, but like the traditional-TTT plots, the alternative-TTT plots can show a temperature, or temperatures, where the transformation rate is fastest. Therefore, the unconventional DSC approach can provide supporting evidence of the double-C curve kinetics published by Orme et al. [5].

The paper by Orme et al. [5] does not specify the conditions at time zero. We do not know if these experimenters began timing the experiment when the sample was dropped into the quench bath, when it reached the isothermal hold temperature, or at some other time. We also do not know if the sample was in thermal equilibrium with the quench bath when the experimenters began timing the transformation. Depending on the criteria used for time zero, the C curves could be shifted along the x-axis. Thus, the length of the incubation period prior to transformation as reported on a TTT diagram depends strongly on the conditions at time zero. Also, it should be noted that the 5% contour of transformation is the lowest value reported by Orme et al. [5]. The first transformation to occur will be observed at shorter times and higher temperatures. Thus, if for example, a 1% contour of transformation was plotted, it would be shifted to the left of the 5% contour. In the present work, we define time zero as the time when the sample temperature, as measured by the DSC, reaches −100 °C. For isothermal holds at temperatures higher than −100 °C, time zero is defined as the time when the sample temperature reaches the hold temperature.

4. Results and discussion

A Pu–2.0 at% Ga sample was quenched to a series of temperatures between −90 and −160 °C at 20 °C/min and held isothermally for 18 h. Exothermic peaks in the DSC scans of the quench indicate that some transformation occurred during the quench period, and these scans are shown in Fig. 3. For this reason, a long holding time is important to minimize the effect of partial transformation during cooling. Based on the work by Orme et al. [5], it is not surprising that some transformation occurred during cooling. The thermal profile of a typical DSC experiment is shown in Fig. 1, and it intersects both the upper- and lower-C curves. According to this TTT diagram, only quenches to −130 °C and lower should show transformation during cooling. However, as discussed earlier, time zero is not specified for Orme et al.'s TTT diagram [5], and the C curves could be shifted in time relative to our experiments. Thus, the transformation observed during quenches to −110 and −120 °C is not necessarily inconsistent with Orme et al.'s work [5].

In Fig. 3, it is interesting to note that the cooling data shows three overlapping exothermic peaks: the first begins at approximately −103 °C, the second begins at approximately −117 °C, and the third begins at approximately −131 °C. The upturn in the heat flow at −140 °C corresponds to a slowing of the transformation rate. Based on the double-C TTT diagram published by Orme et al. [5], we might expect to see two overlapping exothermic peaks in the DSC data, with each peak corresponding to one of the C curves. The presence of three overlapping DSC peaks suggests that the TTT diagram may contain a third C curve, or an interesting precursor phenomenon, that was not identified by Orme et al. [5]. More work is required to determine the causes of the three distinct thermal processes, and several possibilities can be considered. First, each exothermic peak could correspond to the formation of a distinct α' product. The products may differ in morphology (i.e., shape, size, or crystallographic orientation), lattice parameter, or composition. Second, each peak could correspond to α' formation from a different type of nucleation site. The α' particles may ultimately be the same, but each of these nucleation and growth process may be activated at a different temperature. There is experimental evidence that α' may nucleate on α embryos, or on intrinsic sites such as grain boundaries and impurities, depending on the prior thermal history [11]. Third, each peak may correspond to the formation of a distinct

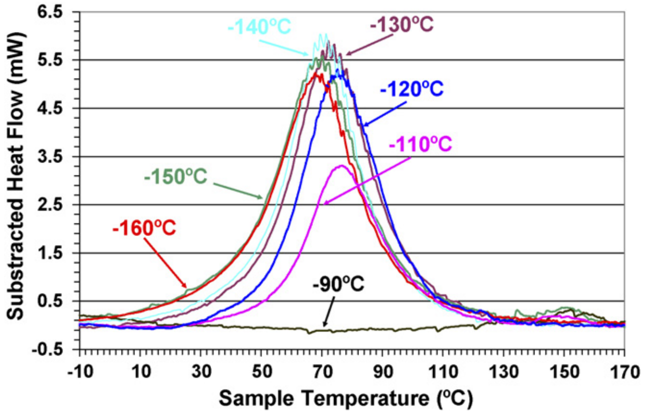

Fig. 4. DSC thermograms corresponding to the $\alpha' \rightarrow \delta$ reversion upon continuous heating at 20 °C/min following each 18 h isothermal hold. The temperatures on the plot correspond to the isothermal holding temperatures. This transformation is endothermic and begins at approximately 25 °C.

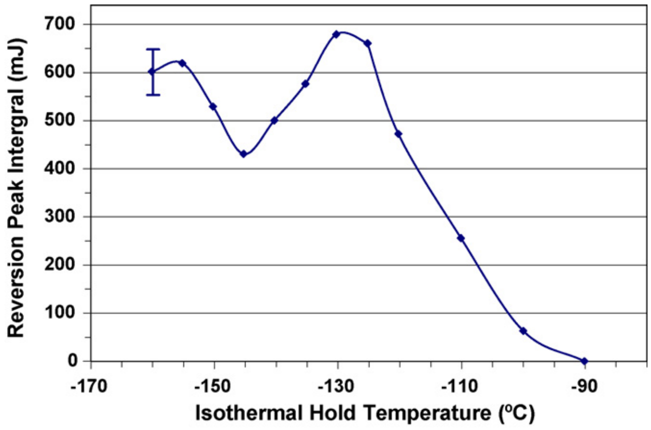

Fig. 5. Integrated peak areas of the $\alpha' \rightarrow \delta$ reversion peaks following 18 h isothermal holds vs. holding temperature. The amount of α' formed is directly related to the integrated area of the $\alpha' \rightarrow \delta$ reversion peak.

phase, including one or more intermediate products. Deloffre et al. [8,9] suggested that γ' may form as an intermediate, and Faure et al. demonstrated this with pressure-induced transformations in a diamond anvil cell [15]. It is possible that one peak corresponds to the $\delta \rightarrow \gamma'$ transformation, another corresponds to the $\gamma' \rightarrow \alpha'$ transformation, and the remaining peak corresponds to a direct transformation from δ to α'. Finally, the three exothermic peaks may correspond to different transformation mechanisms. Orme et al. suggested that a massive transformation may occur at higher temperatures (in the upper-C) and a martensitic transformation may occur at lower temperatures (in the lower-C). Under some circumstances, the mechanism could even contain a diffusional component, as suggested by Sadigh and Wolfer [10]. In this case, the heterogeneity of the sample could play a non-negligible role the transformation.

After the sample is quenched, it is held isothermally for 18 h and then heated to 350 °C to revert the α' formed during the quench and isothermal hold to the δ-phase. DSC scans of the $\alpha' \rightarrow \delta$ reversion are shown in Fig. 4. A single endothermic peak is observed for the isothermal holds between −110 and −160 °C. The isothermal hold at −90 °C showed no reversion peak, indicating that α' did not form within 18 h at this temperature. Although the peak temperatures (the temperature at which the transformation rate is fastest) of the reversion peaks shift slightly depending on the isothermal hold temperature, there appears to be only a single endotherm resulting from the reversion. It is notable that the formation of α' during cooling results from three distinct thermal processes, but the reversion occurs in a single thermal process. If the α' formed in each thermal process was in some way different from the α' formed in each of the other two processes, it is reasonable to expect the reversion to occur in three thermal processes as well. However, it is also possible that there could be there overlapping reversion peaks that appear as a single peak in the DSC data. It is also possible that α' forms via an intermediate phase or via two or more mechanisms. More work is necessary to determine what causes the three exothermic $\delta \rightarrow \alpha'$ peaks and the single $\alpha' \rightarrow \delta$ reversion peak.

We assume the integrated area of the $\alpha' \rightarrow \delta$ peak is directly related to the amount of α' transformation during the quench and isothermal hold, and this area is used to generate the 18 h alternative-TTT diagram shown in Fig. 5. In this figure, the isothermal hold temperature is plotted on the *x*-axis, the amount of α' formed during the isothermal hold is plotted on the *y*-axis, and the data points correspond to 18 h of isothermal holding. These data suggest a confirmation of the double-C behavior reported by Orme et al. [5] because they shows two temperatures, −130 and −155 °C where the amount of transformation to α' that occurs during an 18 h hold is higher than the amount formed during holds for the same length of time at temperatures between these local maxima. This alternative-TTT diagram also indicates that the double-C behavior persists for isothermal holds at least as long as 1080 min (the TTT curves published by Orme et al. [5] only extend to 200 min).

5. Conclusions

The isothermal martensitic $\delta \rightarrow \alpha'$ transformation in a Pu–2.0 at% Ga alloy was investigated. Previously, this transformation was reported to have double-C curve kinetics in a TTT diagram that extended to approximately 200 min. Here, differential scanning calorimetry was used as an alternative approach to investigate this behavior. A Pu–2.0 at% Ga alloy was held isothermally for 1080 min (18 h) at various sub-ambient temperatures between −90 and −160 °C in the DSC, and the amount of α' formed during the quench and isothermal hold was determined by integrating the area of the $\alpha' \rightarrow \delta$ reversion peak on heating. The data indicate two temperatures where local maxima in the amount of transformation occur during isothermal holding, −130 and −155 °C. At −145 °C, less transformation occurs. Thus, a slice through the TTT diagram at a constant time (18 h) provides supporting evidence of the double-C curve kinetics for the Pu–2.0 at% Ga alloy. The thermograms corresponding to the $\delta \rightarrow \alpha'$ transformation during continuous cooling prior to the isothermal holds show three reproducible overlapping exothermic peaks that may result from the formation of α' with three distinct morphologies, structures, or compositions, formation of α' from three different types of embryos, formation of an intermediate product, or formation of α' via two or more transformation mechanisms. More work is required to characterize the Pu–Ga sample at various stages during the transformation and reversion to understand the origin of these three overlapping peaks.

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