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Mechanical properties of delta-stabilized Pu–1.0 wt% Ga alloys

Jack L. Robbins *

Defense Technologies Engineering Division, Lawrence Livermore National Laboratory, P.O. Box 808, L-125, Livermore, CA 94551-0808, USA

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Abstract

This overview paper summarizes how temperature, strain rate, purity, prior fabrication history and prior thermal history affect mechanical properties of delta-stabilized Pu–1.0 wt% Ga alloys (hereafter referred to as delta). The efficacy of comparing torsion and tensile data using the Von Mises criteria (distortion energy theory of yielding) is clearly shown by numerous examples. Delta also follows the Hall–Petch relationship. A reasonably self-consistent set of mechanical properties, i.e., hardness, strength, ductility, creep, and fatigue, are presented; these data (and other information) were used by Los Alamos to benchmark a new constitutive model for delta. The agreement with mechanical property data of other FCC metals is extremely good. Further work in the area of dynamic restoration processes at elevated temperature is suggested. The fairly new high-quality TEM work on delta continues to be another rich area for further research, especially as related to looking at aging effects in delta.

PACS: 61.82.Bg; 62.20.−x; 62.20.Fe; 62.20.Hg

1. Introduction

The delta allotrope of Pu has a face-centered-cubic crystal structure and is stable in the unalloyed state between 319 and 451 °C. The temperature range of stability (or very-long-term metastability) can be extended from well below room temperature to temperatures greater than 451 °C by alloy additions of Al [1], Am [2], Ce [3] and Ga [4]. Of the delta stabilizers, Ga was chosen as the desired element for the majority of metallurgy studies. Consequently, most of the mechanical property information that has been published is for Pu–Ga binary alloys with emphasis on the 1.0 wt% (3.34 at.%) alloy. The largest body of data on this alloy was published in the decade between 1965 and 1975 [5–9,11,12,14,16–18,21–23,25–34,37–46], with limited studies since that time [10,13,24,35].

Fig. 1 is the practical Pu–Ga phase diagram [4]. This diagram shows single delta phase stability for the 1.0 wt% alloy from ambient (actually well below ambient) up to 530 °C. Impurities can lower the delta-to-epsilon phase boundary; for a nominal-purity alloy this boundary can be as low as 508 °C [5].

A large contributor to variability in mechanical properties of delta is the Ga microsegregation (and subsequent alpha phase formation) that occurs during cooling from the melt. This partitioning occurs when the alloy is cooled to room temperature through the liquid-plus-epsilon and epsilon-plus-delta phase fields (peritectic and peritectoid reactions, respectively) after casting. Fortunately, the effects of this double partitioning are simplified somewhat because the high chemical diffusivity in the epsilon phase eliminates the peritectic coring [6,7]. Thus, the resulting as-cast structures are a product of the transition through the epsilon-plus-delta phase field (also called peritectoid encasement). Gallium concentration differences previously observed across grains were dependent on cooling rate; concentrations of 1.6 wt% Ga in the grain centers and of essentially zero at the grain boundaries were reported [8]. These gradients are not removed by rolling, hydroforming, or other

* Tel.: +1-925 422 1795; fax: +1-925 422-1485.
E-mail address: jackr@fone.net (J.L. Robbins).

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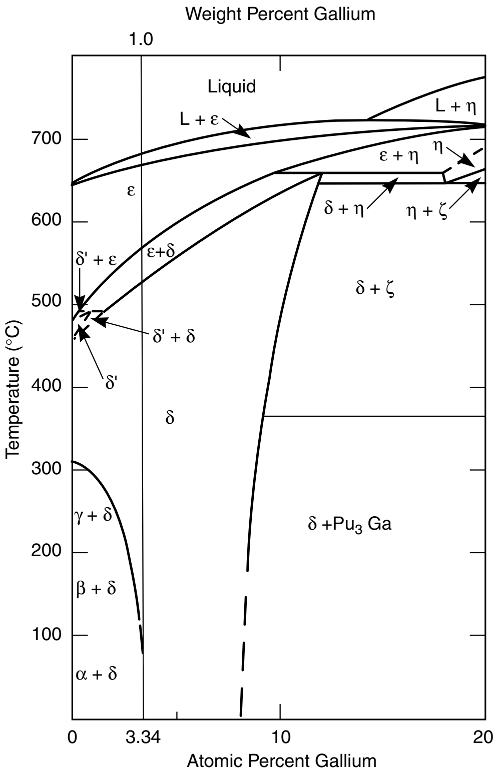

Fig. 1. Pu–Ga phase diagram Pu-rich region.

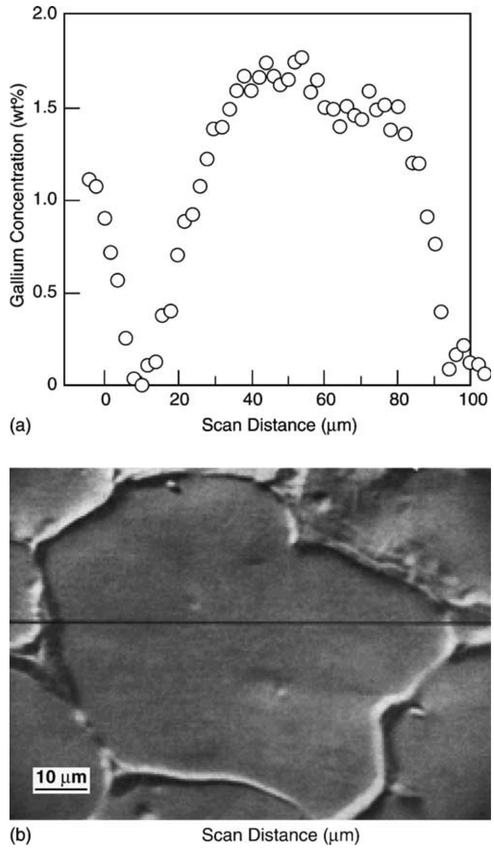

Fig. 2. Back-scattered electron image of delta Pu cooled at 5 °C/min through the $\varepsilon + \delta$ phase field and quenched (as-cast) [9].

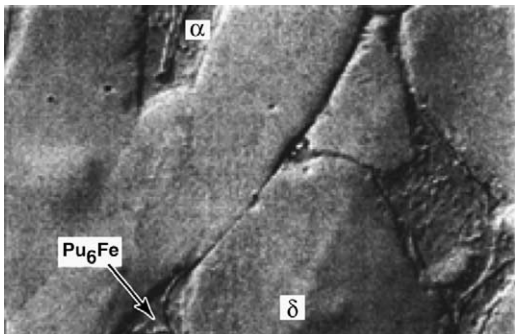

Fig. 3. Photomicrograph of as-cast delta Pu showing both alpha and Pu_6Fe [10].

fabrication procedures; instead, a high-temperature homogenization treatment is required. A detailed paper following the homogenization of a cored delta alloy using heat treatments for various times at 460 °C indicates the length of time required for a truly homogeneous alloy [9]. This paper showed that, for a delta alloy cooled at 5 °C/min through the epsilon-plus-delta phase field and air quenched from 480 °C, the Ga concentration at grain centers was as high as 1.7 wt% and as low as zero Ga at grain boundaries (Fig. 2). At the near-zero Ga regions, the delta transformed to the high-density alpha phase and was clearly evident in the grain boundaries (Fig. 2). Alpha is also seen in another typical photomicrograph [10] of as-cast delta in Fig. 3; also shown are inclusions of Pu_6Fe at triple points (discussed later). Immersion density measurements of the sample in Fig. 2 indicated this alloy contained approximately 6–7 vol.% alpha Pu (each 1 vol.% alpha corresponds to an increase of 0.04 g/cc in density over the nominal 15.75 g/cc for all delta). Thus, we now have not only a solid solution hardened alloy, but an alloy hardened by a second phase as well. Obviously, the mechanical properties of this material are significantly different from a well-homogenized, single-phase delta alloy – containing alpha will be much stronger. In subsequent sections of this paper, such coring and alpha phase formation is a major cause of variability in reported strength data. It is interesting to note that full homogenization (no Ga gradients) required a heat treatment of 720 h at 460 °C [9].

Fig. 4. Photomicrograph of nominal-purity as-cast delta Pu with a grain size of 40 µm [11].

The above discussion was for typical nominal-purity delta with grain sizes in the 40–60 µm region. Fig. 4 is a photomicrograph of such an alloy [11], but one that has been homogenized to eliminate alpha phase formation. A finer grain size will require less homogenization time at a given temperature because the mean free path for Ga diffusion is less.

Common impurities in nominal-purity delta, such as Fe and Ni, have a significant effect on elevated temperature plasticity. There is a eutectic that causes liquid Pu_6Fe to occur at triple points and grain boundaries. Fig. 3 shows the room temperature Pu_6Fe intermetallic. The presence of this intermetallic at triple points and grain boundaries is common for nominal-purity alloys with up to approximately 500 wppm of Fe + Ni [12,13]. The Pu_6Fe liquid leads to the phenomenon of hot shortness, which manifests itself in almost no ductility for delta in the 400–500 °C temperature range. There is also a Pu–Ni eutectic that has a similar effect, although in nominal-purity delta the Fe-to-Ni ratio is typically 2.5:1. Nominal-purity delta (typically 2000 wppm total metallic impurities and numerous PuC inclusions) is stronger than high-purity delta (typically 300 wppm total metallic impurities) at any given temperature. These effects will be discussed in the section on torsion.

Grain size also affects mechanical properties. For nominal-purity delta, the 40–60 µm grain size can be reduced by hot working to grain sizes as low as 1–2 µm through the process of concomitant recrystallization. Fig. 5 [14] is an electron micrograph taken of a Ge-coated replica, which was removed from the polished and etched delta surface of such a hot-worked sample. This delta at room temperature is significantly stronger than delta with the larger grain size.

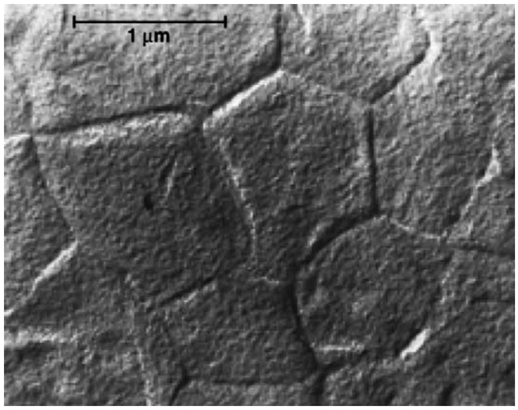

Fig. 5. Electron micrograph of hot-worked delta Pu with a grain size of 1 µm [14].

Finally, it is necessary during casting to get all of the Ga in solution in order to prevent the formation of Pu–Ga compounds with higher melting points. These compounds would lower the effective Ga concentration in the delta phase; this can lead to a metastable alloy and alpha phase formation.

2. Hardness

The word 'hardness' is of significant engineering importance, although a value of hardness for a particular metal or alloy is not considered a fundamental property as is yield strength or ultimate tensile strength (for a given strain rate and temperature). The hardness value typically is a combination of combined properties such as yield strength, ultimate strength, ductility and work-hardening characteristics [15]. The Vickers hardness number is more commonly referred to as the diamond pyramid hardness number (DPHN). For this paper, only DPHN will be considered.

Fig. 6 shows the influence of load on DPHN values at room temperature for delta Pu. Error bars are shown for the data from Sprague and Cramer [16] and it is believed that a similar spread is true for the rest of the investigators [6,17–20]. A number of interesting conclusions can be drawn from Fig. 6. Firstly, DPHN (value of 43.7 ± 2.5) is a constant independent of load from 100 g to 5 kg for fully homogenized delta. This is remarkable agreement for four independent investigators using different material purity and hardness testing equipment. Secondly, the DPHN value from Elliott and Gschneidner [20] is high and out-of-line with the rest of the data. Considering that this data is the oldest and no mention

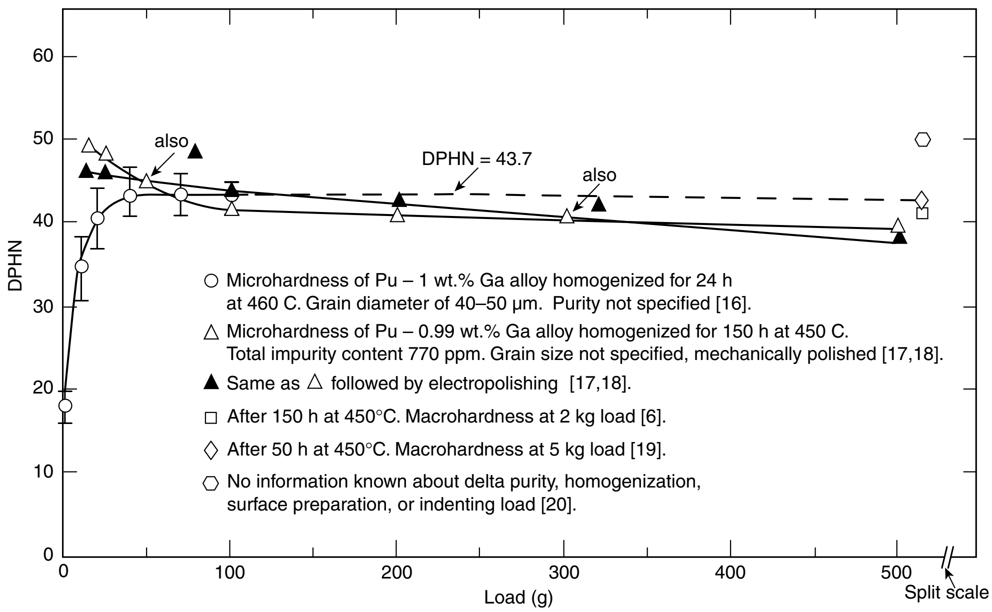

Fig. 6. Variation of hardness number with indenting load for delta Pu.

was made of the processing history for the delta or the test method used, this result is not too surprising. It is speculated that the high value is due either to a cored alloy that contained a substantial amount of alpha phase Pu or to a work-hardened structure such as a rolled plate. Thirdly, notice that the microhardness numbers below 100 g show a variation with load. Two articles by Hays [17,18] (essentially the same data, emphasis is different) indicate a DPHN increase with decreasing load, while Sprague and Cramer [16] show a DPHN decrease with decreasing load. Two reasons lend credibility to the trend in the latter study. First, the same trend was shown for pure copper of approximately the same grain size as delta. Second, at low loads the relative amount of work hardening induced in the specimen is less than at the higher loads and this should lead to a lower value of DPHN. Apparently the above discrepancy was never resolved between the two laboratories.

The influence of temperature on DPHN is shown in Fig. 7. Data from this study [21] is for a cast and homogenized delta alloy. Notice that DPHN is a monotonically decreasing function of temperature. The two high-temperature values (385 and 415 °C) are abnormally low due to creep effects during hardness testing.

For a given set of processing conditions for delta, there is an approximately linear relationship between yield strength and DPHN. This was shown by Hays [21] for yield strength determined in compression and by Wheeler et al. [22] for effective yield strength determined in torsion.

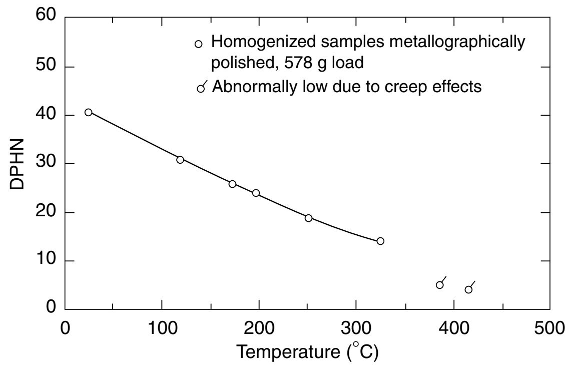

Fig. 7. Variation of hardness number with temperature for delta Pu [21].

3. Tension, compression, and torsion

Strength and ductility as a function of temperature and strain rate will be discussed for tension, compression and torsion together. There is a wealth of published information available; no attempt will be made to cover all aspects in detail. Instead, a perspective is provided on how temperature, strain rate, purity, prior fabrication history (work hardening and texture) and prior thermal history (Ga segregation and grain size) affect strength and ductility.

Fig. 8 shows yield strength (0.2% offset in tension and compression and effective yield strength in torsion) as a function of temperature and strain rate for a number of

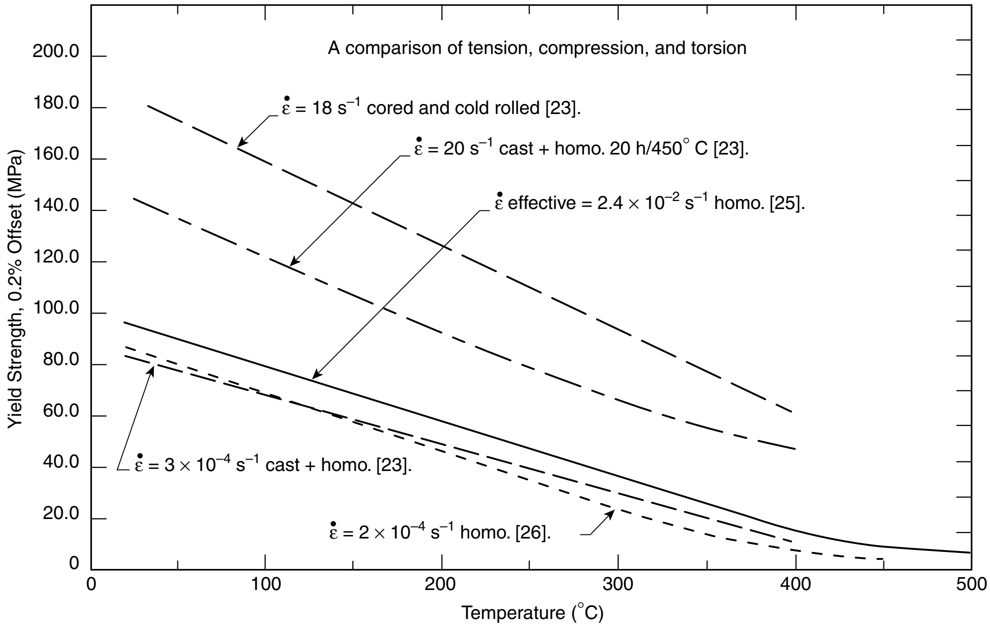

Fig. 8. Influence of temperature and strain rate on the yield strength of Pu–1.0 wt% Ga alloys.

different investigators and materials. Rather than show individual data points (which are provided in the references), just the overall range that can be expected, when all variables discussed above are considered, is shown. The high strain-rate data from Merz [23] is in two groups: the higher values are for a cored and cold rolled alloy (so structure is both second phase hardened with alpha phase and work hardened by rolling), while the lower values are for a cast and homogenized (20 h at 450 °C) alloy. The intermediate strain-rate data is taken from Wheeler and Robbins [25] and the yield strength is calculated from the torsion data by using the Von Mises criteria. The two lowest strain-rate curves are both for cast and homogenized delta taken from research by Merz [23] and Barmore and Uribe [26], respectively. Tension, compression and torsion data are included for the Barmore and Uribe [26] curve. Merz's data [23] does not indicate an increased sensitivity of flow stress (i.e., yield strength) to strain rate at higher temperatures (as is common with other FCC metals and has been observed in delta), whereas Barmore's and Uribe's [26] does. Since it is known that in many FCC metals such as Al, one or more thermally activated deformation mechanisms control plastic deformation at elevated temperature, Barmore and Uribe may be more nearly correct. Their article [26] also discusses strain-hardening exponents.

Notice that there is no discontinuity in strength in the hot short region between 400 and 500 °C even though ductility goes to a minimum (essentially zero) in this region for the higher Fe alloys (this will be discussed later). Yield strength data from Gardner [6] and Gill [27] all fit appropriately within the curves in Fig. 8 when strain rate, purity, solid solution hardening and prior history are taken into account. Data from Beitscher [28] between strain rates of 2×10^1 and 2×10^3/s are consistent for cast, homogenized, rolled and recrystallized delta properties. Flow stress data from Miller et al. [29] for chill cast and homogenized delta is in line with the lower strain rate curves in Fig. 8. Data from Hecker and Morgan [12] between strain rates of 10^{-4} and 10^{-2}/s at room temperature fit the lower strain rate data in Fig. 8 at room temperature; however, their high strain rate data are somewhat lower than the Merz data [23] for cast and homogenized delta.

Other articles [30,31] present further mechanical property data including yield strength in tension and compression, ultimate tensile strength and ductility for both nominal-purity and high-purity delta alloys. There are also discussions about the strain-hardening exponent and the strain-rate exponent.

Recently, Stout et al. [13] have developed a constitutive model to predict the mechanical properties of delta phase Pu–Ga alloys. The model takes into account the effects of test temperature and strain rate on yield and ultimate tensile strengths of delta as well as isolating effects of various microstructural variables. Baselining the model was done largely by using published test results, most all of which are referenced in this paper. Having this baseline will allow future Pu mechanical metallurgists to study the possible effects of aging (such as the long term effects of He generation within the bulk Pu lattice) on subsequent mechanical properties.

Delta, for the most part, is a very ductile alloy much like pure Al. Fig. 9 indicates ductility as a function of temperature for three different investigators [23,25,32] to give representative information. Notice in Fig. 9(a) and (c) the drastic loss in ductility between 400 and 500 °C due to the hot short liquid Pu_6Fe eutectic phenomenon.

A number of the previously cited references dealing with mechanical properties discuss static elastic modulus data for delta Pu. No attempt will be made to summarize that data here; rather, dynamic modulus data as a function of temperature are presented in Fig. 10. Both Young's modulus, E and the shear modulus, G, are a linearly decreasing function of temperature. While data is only shown up to 300 °C for Dunegan [33] and McIlree [34], Calder et al. [35] extended the range to 500 °C with the same linear relationship shown in Fig. 10. One static measurement shows a room temperature value of Young's modulus to be slightly lower than the dynamic values. This is quite common for static measurements taken from a stress-strain curve.

The effect of hot working on the room temperature strength and ductility of delta was studied extensively by Wheeler et al. [22]. Fig. 11 again illustrates the excellent agreement that can be obtained for yield strength, independent of the test method. Torsion, tension and compression all agree within approximately 5% to 8% when compared by means of the effective stress–strain concept.

Fig. 12 [22] shows that the grain-size effect on yield strength can be described by the Hall–Petch relationship,

$$\overline{\sigma_y} = \sigma_o + Kd^{-\frac{1}{2}}, \tag{1}$$

where $\overline{\sigma_y}$ is the effective yield strength at room temperature, d is the grain size in μm and σ_o and K are constants, equal to 63.8 MPa (6.50 kg/mm^2) and 82.8 MPa μm$^{1/2}$ (0.26 kg/mm$^{\frac{3}{2}}$), respectively. The value of K is

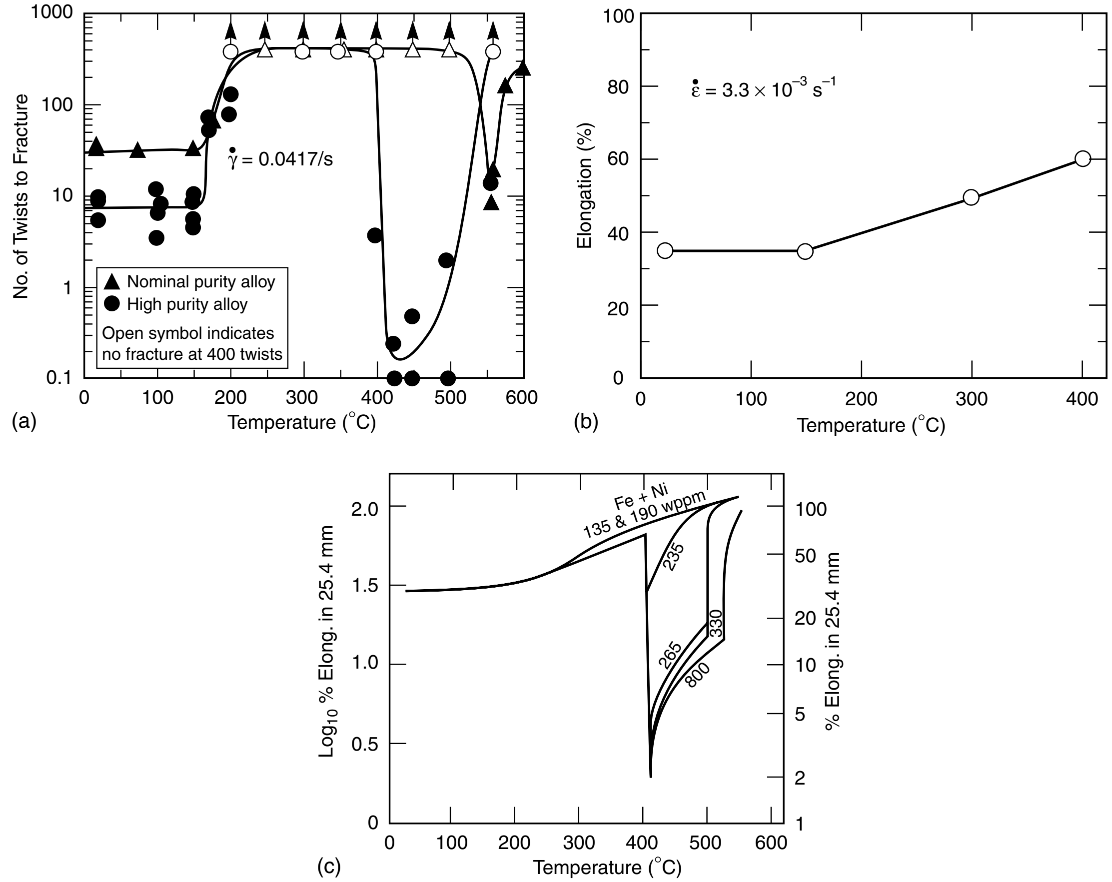

Fig. 9. Ductility of delta Pu. (a) Influence of temperature on the ductility of two Pu/Ga alloys [25].(b) Elongation of cast and homogenized Pu–1 wt% Ga alloy versus test temperature [23]. (c) Ductility of Pu–1 wt% Ga with various Fe + Ni contents [32].

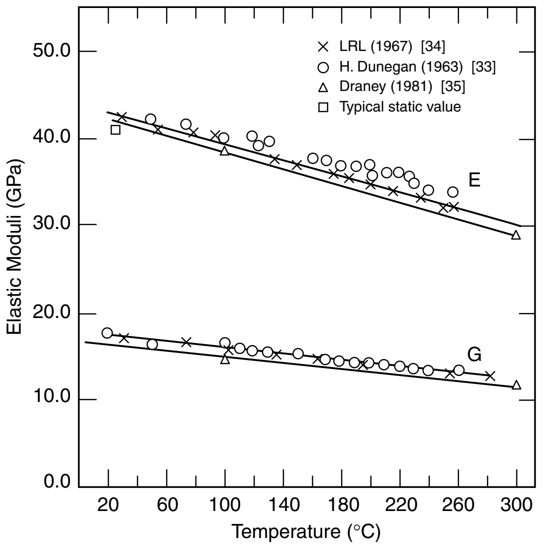

Fig. 10. Dynamic modulus as a function of temperature for delta.

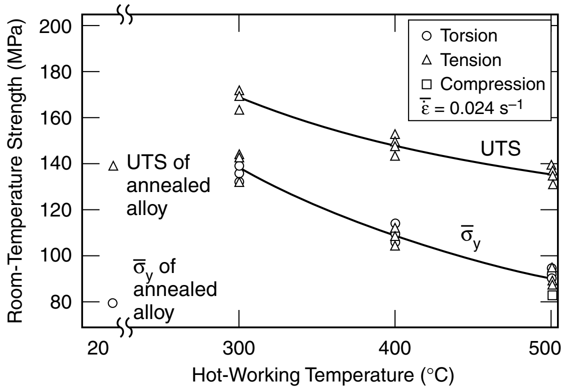

Fig. 11. Influence of hot working temperature on the room temperature yield and ultimate strength of high-purity delta [22].

small and is typical of many face-centered cubic metals [36]; thus, grain-size sensitivity is fairly low.

The very-fine-grained (1 μm) delta formed by hot working (Fig. 5) exhibited superplasticity; i.e., very high elongations in tensile tests without necking and a high value of *m*, the strain rate sensitivity exponent [37]. It appears that this is the only single-phase alloy to exhibit superplasticity. Unalloyed Pu in the beta phase with the proper microstructure also shows tendency for superplasticity.

Recovery and recrystallization in cold-worked and annealed delta have been covered extensively [38–41]. Beitscher [41] speculated from his experiments that the stacking fault energy of delta is high like that of Al.

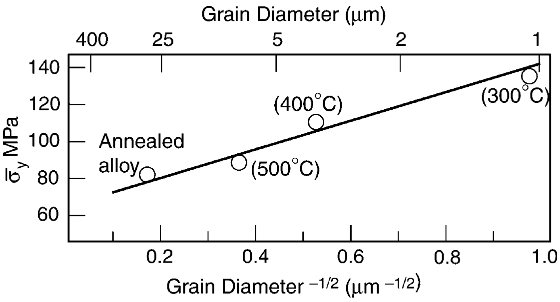

Fig. 12. Influence of grain diameter on the room temperature effective yield strength of delta. Temperatures in parentheses indicate the prior hot working temperature [22].

4. Torsion

A limited amount of torsion testing of delta was done in the study by Wheeler et al. [22] and by Los Alamos National Laboratory [24]. However, the definitive work was done in another study [25] where the torsional ductility and strength properties of nominal-purity and a high-purity delta alloys were studied as a function of temperature between 20 and 600 °C. The alloys were cast, swaged into rods and then homogenized for 24 h at 460 °C. Fig. 13 shows the torque (and consequently the torsional strength) vs. torsional strain curves at various temperatures for the nominal-purity alloy. These curves are reminiscent of standard tensile or compression stress-strain curves for the lower values of strain. It can be seen that appreciable strain hardening was observed at room temperature and decreased with increasing temperature, leading to actual strain softening above 150 °C and to the eventual formation of a torque plateau at temperatures above 175 °C. The strain softening and torque plateau are indicative of some type of dynamic restoration process (either recovery or recrystallization). These phenomena are typical for other materials tested in torsion and not peculiar to the delta alloys studied by

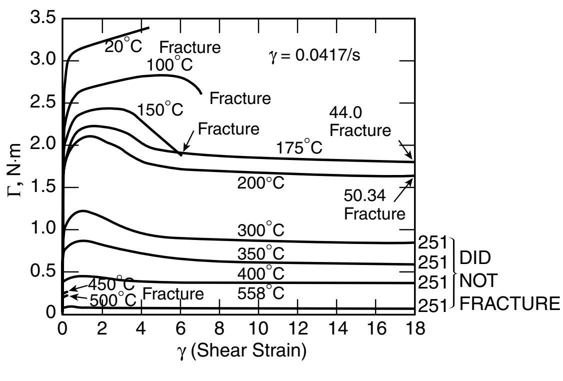

Fig. 13. Influence of temperature on the torque-strain curve for nominal-purity delta-stabilized Pu [25].

Wheeler and Robbins [25]. Most investigators, including Robbins, seem to favor dynamic concomitant recrystallizaton as the mechanism for the restoration process, however more rapid quenching rates and TEM are needed to further elucidate the mechanism of hot deformation. The influence of temperature on ductility from [25] was discussed previously under the topic of hot shortness.

5. Creep

Elevated temperature creep studies of delta are somewhat limited, however two papers [42,43] delineate the salient features for a nominal-purity alloy and a high-purity alloy, respectively. Robbins and Wheeler [42] discussed the compressive creep characteristics of a nominal-purity delta alloy at a constant stress over a temperature range of 234 to 387 °C. This limited temperature range was dictated by two factors: (1) the desire for a temperature range well above one-half the absolute melting point of the FCC delta phase and (2) the necessity of staying below the temperature of the Pu–Pu_6Fe eutectic region.

In all cases, the true strain-versus-time curves manifested a predominantly tertiary type of creep behavior; this behavior was associated, at least in part, with recrystallization during straining. The apparent activation energy for creep was determined to be 33 700 cal/mol independent of strain between strain values of 0 and 0.15.

The study by Lytton et al. [43] provides data on constant stress compression creep tests for a high-purity delta alloy over the temperature range from 252 to 382 °C for stresses from 4.8 to 17.3 MPa. Although the primary creep behavior could not be correlated by established techniques, the creep rates developed after true strains of about 0.15 provided good agreement with the temperature and stress dependence of creep for pure FCC metals and dilute alloys. The activation energy for high-temperature, steady-state creep of this alloy, after correction for variations in modulus of elasticity, was found to be 38 900 cal/mol. This is in good agreement with the value of 36 300 cal/mol for self-diffusion of Pu in a delta alloy [44]. The work of Lytton et al. [43] provides further discussion on the unusual primary creep characteristics of delta as well as stress dependence during steady-state creep.

6. Fatigue

Gardner [45,46] provides a good discussion of the fatigue behavior of a number of delta alloys. Several metallurgical conditions of both as-cast and rolled sheet were fatigue-tested at room temperature in flexure with a superimposed mean stress of 37.3 to 46.2 MPa. The frequency of stress reversal was approximately 215 cycles/s. A number of S-N curves were presented for cored, as-cast and homogenized, 94% cold rolled and annealed at 300 °C and 86% cold rolled sheet. The 0.1% yield strengths for these alloys ranged from 71.1 to 209.8 MPa. Corresponding fatigue strengths only varied from 69 to 103.5 MPa for test durations of 10^7 to 10^8 cycles, suggesting that the fatigue strength was relatively insensitive to metallurgical condition. The superimposed mean stress did not have a great effect on fatigue strength. The presence of alpha phase initially or during testing seemed to enhance resistance to failure.

7. Conclusions

This overview paper presents a fairly complete and self-consistent set of mechanical property data for delta Pu; the physically based constitutive model discussed earlier adds substantial credibility to this statement. While there certainly are rich areas for further research on delta, it appears that the majority of efforts will be devoted to an understanding of aging in Pu and how this aging might affect physical and mechanical properties. Further work in the area of dynamic restoration processes in delta at elevated temperature could help elucidate the contributions of recovery, subgrain formation and recrystallization. With the high-quality TEM work that is now ongoing, it would be of interest to measure the stacking fault energy of delta.

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