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AND DUCTILITY OF A PLUTONIUM-1 WT% GALLIUM DELTA-STABILIZED ALLOY

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THE EFFECT OF HOT WORKING ON ROOM-TEMPERATURE STRENGTH
AND DUCTILITY OF A PLUTONIUM-1 WT% GALLIUM DELTA-STABILIZED ALLOY*

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High-purity Pu-1 wt% Ga specimens (annealed for 24 hours at 460°C) were hot-worked to a torsional strain of 63 (at a torsional strain rate of 0.04 sec^{-1}) at temperatures of 300, 400, and 500°C and quenched to room temperature within 20 seconds. The effects of this hot working were evaluated by measuring the room-temperature strength and ductility of the quenched specimens by torsion, tension, and compression testing; all three methods showed excellent agreement. Hot working at 300°C appreciably increased the room-temperature yield and ultimate strength without loss of ductility. Hot working at 400 and 500°C significantly increased room-temperature ductility with some concurrent increase in strength.

The high capacity for deformation during hot working is associated with a dynamic restoration process which results in a very fine-grained equiaxed room-temperature structure. The mechanism of this restoration process is believed to be concomitant recrystallization. The final grain size increased with increasing hot-working temperature. This grain size increase is due to the fact that grain size is a function of the steady state hot-working stress, and increases with decreasing stress. The effect of grain size on room-temperature yield strength can be described by the Hall-Petch relation.

*This work performed under the auspices of the U. S. Atomic Energy Commission.

Introduction

A recent study⁽¹⁾ on the hot deformation behavior of two Pu-1 wt% Ga alloys indicated that these materials have a high capacity for torsional deformation at temperatures greater than $0.6 T_m$. "Unlimited" ductility regions were found for both materials between 250 and 500°C. In these unlimited ductility regions, both alloys were able to survive a true shear strain of 250 without fracture. It is possible to achieve such large strains under steady state flow conditions (no change in resistance to deformation with increasing deformation) when some dynamic restoration process is operative. The mechanism of the restoration process in the Pu-Ga alloys was thought to be concomitant recrystallization⁽¹⁾. Regardless of the mechanism, it is possible to modify the structure so that it consists of very fine equiaxed grains at room temperature.

Since such a structure modification might have useful application in the fabrication of Pu-Ga alloys for better properties, this investigation was undertaken to assess the room-temperature properties of a high-purity Pu-1 wt% Ga alloy which had previously been subjected to hot working by torsion. It was also hoped that this study might further elucidate the mechanism of hot deformation in the unlimited ductility region.

Materials and Experimental Techniques

The high-purity Pu-1 wt% Ga alloy was prepared by casting, swaging, and vacuum annealing (24 hours at 460°C) in a manner identical to that reported previously⁽¹⁾. The alloy had less than 325-ppm total metallic impurities; the composition is given in Table I. Density measurements were made using an immersion technique; densities of the annealed specimens varied between 15.754 and 15.78 g/cm³ per cubic centimeter.

The microstructures of the annealed alloy and of the hot-worked specimens were studied by both optical microscopy and electron microscopy of carbon replicas. Metallographic samples were all longitudinal sections (along the specimen axis) obtained by cutting the gage section along the midplane parallel to the axis. Metallographically polished samples were either electrolytically etched in a citric acid, nitric acid, ethanol solution or cathodically etched. Two-stage plastic carbon replicas were made from the samples prepared for optical microscopy; these replicas were shadowed at 35 degrees with germanium. A representative microstructure of the annealed alloy is shown in Figure 1. Diamond pyramid hardness measurements (DPHN) were made on the mounted and etched samples with a microhardness tester, (25-g load). Grain sizes were determined by lineal analysis.

Table I. Composition of Pu-Ga Alloy

Spectroscopic Analysis				Chemical Analysis		Isotopic Analysis	
Element	ppm [*]	Element	ppm [*]	Element	ppm	Isotopes	wt%
Ag	< 0.05	Fe	< 20	U	17	²³⁸ Pu	< 0.05
Al	< 5	Mg	< 5	Ta	22.5	²³⁹ Pu	93.17
B	< 1	Mn	2	Fe	24	²⁴⁰ Pu	5.88
Be	< 0.05	Ni	< 50	Am	120 (2/70)	²⁴¹ Pu	0.44
Ca	< 5	Si	< 10	Ga(wt%)	0.98 ± 0.03	²⁴² Pu	< 0.05
Cd	< 10	Sn	< 10	C	33		
Cr	< 5	V	< 25	O	30		
Cu	< 3	Zn	< 10	N	3.9		
				H	3		

*Limit of error is ±40%, which includes analysis error plus individual sample variations.

Hot working was done in vacuum at 300, 400, and 500°C at a constant torsional strain rate of 0.042 sec⁻¹. Specimens were hot-worked to a torsional strain of 63 and quenched to room temperature within 20 seconds. Both the specimen geometry (solid cylindrical gage section, 1 inch long by 0.2 inch in diameter) and the testing apparatus have been described previously⁽¹⁾. Torque was converted to torsional stress by the equation,

$$\tau = \frac{(3.1 + n)\Gamma}{2\pi r^3},$$

where τ is the maximum torsional stress, Γ is the applied torque, r is the radius of the gage section of the specimen, and n is the slope of $\log \Gamma$ versus $\log$ of shear-strain γ at constant strain rate.

The effects of hot working were evaluated by measuring the room-temperature strength and ductility of the quenched specimens by torsion, tension, and compression testing. The distortion-energy theory for yielding⁽²⁾ can be used to compare torsional data with compression or tension data. This theory states that the effective stress $\bar{\sigma}$ is equal to the normal true stress in a compression or tension test and to $\sqrt{3} \tau$ in a torsion test. The effective strain $\bar{\epsilon}$ is equivalent to the true strain ϵ in compression or tension and equal to $\gamma/\sqrt{3}$ in torsion. It has been shown^(1,3) that there is very close agreement between experimental results and the theoretical predictions from the distortion-energy theory. Consequently, the effective stress-strain concept can be used to compare results of the three types of tests.

Room-temperature torsion tests were performed on the same equipment used for the hot-working studies at an effective strain rate $\dot{\epsilon}$ of 0.024 sec^{-1} ($\dot{\gamma} = 0.042 \text{ sec}^{-1}$). The tension and compression tests were done on a standard Instron machine at an initial true strain rate $\dot{\epsilon}$ of 0.024 sec^{-1} . A reduced gage section 0.5 inch long by 0.135 inch in diameter was machined within the original gage section of the hot-worked specimens. These reduced section specimens were then used for both torsion and tensile testing at room temperature. The compression samples were right circular cylinders 0.3 inch long by 0.14 inch in diameter machined out of the center of the hot-worked gage section.

Results and Discussion

Representative room-temperature torque versus torsional strain curves are shown in Figure 2 for the three hot-working temperatures; the as-received (fully annealed) alloy is shown for comparison. Both strength and ductility can be increased as a result of the hot deformation. For example, hot working at 300°C can increase the maximum torsional strength by about 40% with no loss in ductility, while hot working at 400 or 500°C can result in a significant increase in ductility with some concurrent strength increase.

The room-temperature strength enhancement is more clearly seen as a function of hot-working temperature in Figure 3. The effective yield strength $\bar{\sigma}_y$ (at $\bar{\epsilon} = 0.002$ offset) was determined for the hot-working temperatures utilizing torsion, tension, and compression testing. The ultimate tensile strength (UTS) was determined in the usual manner. Hot working at 300°C appreciably increases both $\bar{\sigma}_y$ (by as much as 80%) and UTS (by as much as 30%). The stronger hot-worked alloy showed no significant loss of ductility (Figure 2), which is in contrast to the decrease in ductility that occurs when an annealed alloy is strengthened by cold working. When hot-worked at 400°C , the alloy was somewhat stronger than the annealed alloy, while the 500°C hot-worked alloy showed only slight improvement in strength; in both cases ductility was markedly improved (Figure 2).

Figure 3 illustrates the excellent agreement obtained for yield strength of the Pu-Ga alloy, 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. A linear relationship was also obtained between $\bar{\sigma}_y$ and DPHN, as shown in Figure 4. This linear relation further supports comparison of room-temperature strength properties by means of all of the common laboratory tests.

We previously showed⁽¹⁾ that the very high ductility associated with hot working of Pu-1 wt% Ga alloys was related to some dynamic restoration process which results in a very fine-grained equiaxed room-temperature structure; we tentatively concluded that a

concomitant recrystallization process was operative at temperatures above 250°C and at strains of the order of 20 or greater. There is a diversity of opinion on the mechanisms of such a restoration process, and numerous ideas have been advanced including dynamic recovery⁽⁴⁾, concomitant or dynamic recrystallization⁽⁵⁻⁸⁾, and polygonization⁽⁹⁻¹¹⁾. A recently published⁽¹²⁾ comprehensive review of hot working emphasized that caution needs to be exercised in interpreting hot-worked structures because changes can occur during the quenching phase of the experiment after deformation has ceased. More recently, it has been shown that dynamic recrystallization occurs during hot working of nickel and Ni-Fe alloys⁽¹³⁾, copper⁽¹⁴⁾, and Udimet 700⁽¹⁵⁾.

Optical metallography and electron microscopy replication were done during this investigation to determine the structure which resulted from hot deformation and hopefully to elucidate further the mechanism of the dynamic restoration process. Electron micrographs of these replicated structures are shown in Figure 5; it is evident that hot working resulted in a very fine-grain equiaxed room-temperature structure and that the final grain size increased appreciably with increasing hot-working temperature. This grain size increase is due to the fact that grain size is a function of the steady state hot-working stress and increases with decreasing stress^(1,13).

The electron micrographs of the cathodically etched and replicated hot-worked samples showed numerous parallel striations within the individual grains. Annealed samples similarly prepared showed no such striations. Therefore, we have interpreted these striations as being related to deformation within the grains. This interpretation supports the dynamic recrystallization theory which we previously proposed. If the recrystallization had occurred after testing but during the quench, the grains would be strain free. These conclusions need further substantiation through additional replication on hot-worked samples as a function of strain.

Figure 6 shows that the grain-size effect on yield strength can be described by the Hall-Patch relationship,

$$\bar{\sigma}_y = \sigma_0 + Kd^{-1/2},$$

where $\bar{\sigma}_y$ is the effective yield strength at room temperature, d is the grain size, and σ_0 and K are constants, equal to 9,250 psi (6.50 kg/mm²) and 12,000 psi-in^{1/2} (0.26 kg/mm^{3/2}), respectively. The value of K is small and is typical of many face-centered cubic metals⁽¹⁶⁾; thus, grain-size sensitivity is fairly low.

A number of annealing studies were done on the hot-worked alloy to establish the rate of grain growth as a function of annealing

temperature. Samples from each of the three hot-working temperatures were annealed for 1 hour at 100°C, 30 minutes at 200°C, and 15 minutes at 250°C. None of these treatments resulted in any appreciable change of the initial fine-grain size. This suggests that the hot-worked alloy will show significant strength improvement over the annealed starting material up to at least 250°C.

Summary

The room-temperature strength and ductility properties of a high-purity Pu-1 wt% Ga annealed alloy can be significantly improved by hot working at temperatures between 300 and 500°C. For example, hot working at 300°C increases the effective yield strength by as much as 80% over the initial annealed alloy with no loss in ductility. Hot working at 400 and 500°C can result in significant improvement in ductility with some concurrent strength increase.

Excellent agreement was obtained for room-temperature strength values, independent of the test method. Torsion, tension, and compression all agreed within about 5 to 8% when compared by means of the effective stress-strain concept.

The high capacity for deformation during hot working is associated with a dynamic restoration process which results in a very fine-grain equiaxed room-temperature structure. The mechanism of this restoration process is believed to be concomitant recrystallization. The final grain size increased with increasing hot-working temperature. This increase in grain size is due to the fact that grain size is a function of the steady state hot-working stress, and increases with decreasing stress.

The effect of grain size on room-temperature yield strength can be described by the Hall-Petch relation, and the results are in agreement with those published for other face-centered cubic materials.

The variety of strength and ductility properties obtained by hot working are not readily achievable by normal thermal or mechanical treatments separately. This suggests that structural modification by hot working might be usefully applied to fabrication of Pu-Ga alloys.

The fine grain size resulting from hot working appears to be stable to subsequent low-temperature annealing treatments, suggesting that the hot-worked alloy will show significant strength improvement over the annealed starting material up to at least 250°C.

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Wheeler - Fig. 1

Wheeler - Fig. 2

Wheeler - Fig. 3

Wheeler - Fig. 4

Wheeler - Fig. 5

Wheeler - Fig. 6