The effects of strain rate on the stress-strain behavior of Ni-modified Ti-6Al-4V (Ti-6Al-4V-2Ni) at superplastic forming temperature (815°C), were investigated. The microstructural evolution during superplastic deformation (SPD), produced strain hardening at low strain-rates and strain softening at high strain rates. Maximum attainable superplastic ductility, observed at intermediate strain rates, was associated with a dynamic balance between strain hardening and softening. Metallographic evidence is presented to show that the observed strain hardening is due to deformation-enhanced grain growth in the α and β phases, while the strain softening can be attributed, primarily, to grain size refinement, due to dynamic recrystallization.
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FUNDAMENTAL INVESTIGATIONS OF
FAILURE DURING SUPERPLASTIC FORMING PROCESS

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CORRELATION BETWEEN MECHANICAL PROPERTIES AND MICROSTRUCTURE IN A Ni-MODIFIED SUPERPLASTIC Ti-6Al-4V ALLOY

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SUMMARY

The superplastic deformation (SPD) properties of Ti-6Al-4V modified by the addition of 2 percent Ni (Ti-6Al-4V-2Ni) have been investigated in the temperature range between 750-870°C and strain rates from $10^{-5}$ to $10^{-2}$ s$^{-1}$. It was found that the effect of microstructural changes occurring during SPD produced strain hardening and strain softening. Metallographic evidence is presented to show that the observed strain hardening is due to deformation-enhanced grain growth of both α and β phases. The strain softening was primarily due to grain size refinement. Maximum attainable super-plastic ductility was found to be associated with a dynamic balance between strain hardening and softening. Wedging and pinching off of the α-grains by the more diffusible β-phase through grain boundary diffusion seems to be an important mechanism in the deformation process of the Ni-modified Ti-alloy in the superplastic temperature and strain-rate range studied.

1. INTRODUCTION

Many titanium alloys exhibit superplastic deformation behavior in the temperature range where the α and β phases co-exist. Ti-6Al-4V, the most commonly used Ti alloy, is superplastic at temperatures between 850°C and
950°C, with optimum superplastic properties attained near 925°C [1], the temperature frequently used for superplastic forming (SPF) operations with this alloy. However, lower SPF temperatures would be desirable to reduce oxidation problems, shorten forming cycle times, and reduce die costs. Thus, there is technological interest in lowering the forming temperature of Ti-6Al-4V by addition of a suitable β-stabilizer. Furthermore, alloy additions with high diffusivities can accelerate creep rates, allowing the creep process essential to superplastic deformation to proceed at reasonable rates below the conventional SPF temperatures for the base alloy. Alloy additions of β-stabilizers such as Ni, Fe, Co, that diffuse rapidly in Ti, have been found to lower the optimum SPF temperature of Ti-6Al-4V alloy [2,3]. Among these, Ti-6Al-4V alloy modified by the addition of 2 percent Ni (Ti-6Al-4V-2Ni) appears to possess the best potential in lowering the SPF temperature without sacrificing the ease of its formability. This progress report describes the work carried out to analyze the superplastic deformation properties of the Ni-modified Ti-6Al-4V alloy at SPF temperature and to correlate them to the microstructure. Furthermore, the mechanisms responsible for the observed stress-strain behavior in the Ti-6Al-4V-2Ni alloy and their correlation with the microstructural evolution produced by the deformation, are detailed.

2. EXPERIMENTAL PROCEDURES
The Ti-6Al-4V-2Ni alloy used in this investigation had a nominal composition (wt percent) of 5.78 Al, 0.08 Fe, 0.01 Co, 2.10 Ni and balance Ti. Original 7 kg ingots of the Ni-modified alloy (cast by TIMET) were broken down in the conventional manner, and finish-rolled in the α+β phase field to produce a fine mixture of equiaxed α and β grains. The final material was received as a
sheet with thickness of approx. 1.3 mm, from which test specimens with tensile axis parallel to the rolling direction were machined. To optimize the initial microstructure, tensile samples were annealed in purified argon atmosphere for 1 hour at 815°C.

Uniaxial tensile tests were conducted using an MTS servohydraulic machine interfaced with a PDP/11 computer and fast digital data acquisition system. A Quad Elliptical Radiant heating furnace provided a heating rate of 200°C/min, with a phaser power controller which maintained stable temperatures to within ±1°C. All tests were conducted in a purified argon atmosphere. Upon completion of the tests, specimens were quenched under load in pre-chilled argon. Samples were held at the test temperature for 20 minutes before testing.

To determine the SPD behavior pertinent to this investigation, two types of high temperature tests were conducted: 1) Continuous tensile tests to ascertain the effect of rate of deformation on the microstructural evolution, at constant temperature and for different strain levels up to fracture. 2) To minimize the effect of microstructural changes observed during SPD of the Ni-modified alloy, tensile specimens were pre-strained at temperature of 815°C and strain rate of $2 \times 10^{-4}$ s$^{-1}$ up to a total true strain of 0.20.

Samples from deformed specimens were investigated by scanning (SEM) as well as by transmission (TEM) electron microscopy.

3. RESULTS AND DISCUSSION

It has been determined that a large volume fraction of the α-phase in α-β titanium alloys, is essential to SPD [1]. Furthermore, it has been found that equal volume fraction of the two phases in a duplex alloy, produces optimum superplastic properties [4]. The static annealing of the Ni-modified (α+β) Ti
at 815°C for 1 hour, (SA-1-815) produced volume fractions of the α-phase \( f_\alpha \) and β-phase \( f_\beta \) approximately 1:1 (49 percent α). In addition, this heat treatment also yielded very similar phase sizes \( \lambda_\alpha = 6.3 \, \mu m, \lambda_\beta = 5.7 \, \mu m \). The main purpose of the annealing program was to obtain a suitable and similar initial microstructure for the tensile tests. Furthermore, contrary to earlier belief, it has been determined in previous work [5] as well as in the present investigation, that the microstructures of the base and Ni-modified alloys undergo substantial evolution during SPD. Thus, the concept of constant microstructure during deformation for this alloy becomes irrelevant. Instead, optimization of the initial microstructure for superplasticity is more desirable.

The temperature and strain rates selected for deformation in this investigation were such that the specimens deformed in region II of superplasticity. The results illustrated in Fig. 1, show the effect of the rate of deformation on the SPD behavior on the Ni-modified alloy. These test were conducted at high, intermediate and low strain rates \( (5 \times 10^{-3}, 2 \times 10^{-4}, 5 \times 10^{-5} \, s^{-1}) \) for the optimum temperature of 815°C and up to true strain of 1.0. Fig. 1 also illustrates the processes of strain hardening and strain softening occurring during deformation. The strain softening observed at high strain rate \( (5.0 \times 10^{-3} \, s^{-1}) \), was primarily due to grain refinement of both α and β phases, as illustrated in Fig. 2. This process of grain refinement during SPD is due to dynamic recrystallization, which produces fine equiaxed new grains replacing the large grains. At low strain rates \( (5.0 \times 10^{-5} \, s^{-1}) \), significant strain hardening effect was observed, as shown in Fig. 1. Microstructural correlation is presented in figure 3, where the grain growth produced by static annealing for the same time
(specimen shoulder), is contrasted with the strain-enhanced grain growth at the specimen gauge length. It can be seen that for test conditions leading to strain hardening, there is deformation-enhanced growth of both α (dark) and β (light) phases. In the absence of overt cavitation, the maximum attainable superplastic ductility was associated with a dynamic balance between strain hardening (due to grain growth) and strain softening (due to recrystallization). This behavior was observed at intermediate strain rate (2.0 x 10^{-4} \text{s}^{-1}) as illustrated in Fig. 1. The evolution of the microstructure resulting from dynamic balance conditions can be detailed from Fig. 4. This micrograph illustrates the fact that at superplastic forming temperature and intermediate strain rates, there appears to be a rapid deformation-enhanced distribution of the softer phase β, which injects itself between grains of the harder phase α. Furthermore, there is no significant change in the shape and size of the α-phase, suggesting an effective balance between phase growth-induced strain hardening and phase refinement-produced strain softening.

For further characterization of the stress dependence of superplastic strain-rate, the true activation energy was calculated from pre-strained test samples deformed at constant strain rates of 2.0 x 10^{-4} and 5.0 x 10^{-4} \text{s}^{-1} and variable temperatures. Narrow temperature increments between 780°C (1053K) and 830°C (1153K) were used, to allow some degree of microstructural stability at each temperature and flow stress level. Furthermore, pre-strained specimens were used to obtain an activation energy value consistent with some degree of steady-state condition, with regard to the observed microstructural evolution. The average value of the true activation energy, \( Q_T \), was found to be 145 KJ/mole. The activation energy for lattice
self-diffusion $Q_V$, for the $\beta$-phase in Ti, obtained from radioactive $^{44}$Ti tracer, have been found to be [6] about 248 KJ/mole. Thus, considering the activation energy for grain boundary diffusion, $Q_{GB} = 0.6 Q_V$, we obtain $Q_{GB}(\beta$-Ti) = 149 KJ/mole, which is in good agreement with the $Q_T = 145$ KJ/mole determined. Hence, it appears that the true activation energy for SPD in the Ni-modified Ti alloy, is similar to that of grain boundary diffusion, in agreement with the interphase boundary accommodation process detailed below.

Previous investigations [2] on the mechanism for SPD in Ti-6Al-4V-2Ni, have suggested that the flow stress-strain rate behavior of each phase could be represented by the Ashby-Verrall model [7]. Our microstructural correlations of SPD behavior in the range of temperatures and strain-rates tested suggest otherwise. It appears that the controlling deformation process is that of interphase accommodation in agreement with the Spingarn-Nix model for deformation in two phase materials by diffusion creep [8]. As shown in Fig. 5, there is an apparent wedging and pinching off of the harder phase $\alpha$ by the more diffusible $\beta$-phase during the accommodation process. Furthermore, as predicted by the model, the interphase boundaries -- appear to be curved -- concave in the more diffusive $\beta$-phase, and convex in the harder phase $\alpha$.

CONCLUSIONS

The superplastic deformation of Ti-6Al-4V-2Ni alloy deformed in region II showed strain hardening at low strain rates and strain softening at higher strain rates. The strain softening is primarily due to dynamic grain refinement. The strain hardening is due to deformation enhanced grain growth. Maximum attainable superplastic ductility was associated with a dynamic balance between hardening and softening behavior. The rate controlling deformation mechanism in this alloy appears to be that of
interphase accommodation, in agreement with the Spingarn-Nix model for
deformation in the two phase microstructure by diffusion creep.

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Fig. 1. Effect of strain rate on SPD behavior in Ti-6Al-4V-2Ni at optimum SPF temperature of 815°C and $T_e = 1.0$. 

$T = 815 ^\circ C$

- $\dot{\varepsilon} = 5.0 \times 10^{-3} \, s^{-1}$
- $\dot{\varepsilon} = 2.0 \times 10^{-4} \, s^{-1}$
- $\dot{\varepsilon} = 5.0 \times 10^{-5} \, s^{-1}$
Fig. 2. Dynamic Recrystallization process associated with strain softening during SPD in Ti-6Al-4V-2Ni, at $T = 815^\circ C$, $\dot{\varepsilon} = 5.0 \times 10^{-3} \text{ s}^{-1}$, $\varepsilon = 1.0$. 

$T$
Fig. 3. Deformation enhanced grain growth associated with strain hardening during SPD in Ti-6Al-4V-2Ni at APF temperature (815°C), \( \dot{\varepsilon} = 5.0 \times 10^{-5} \text{ s}^{-1} \), \( \varepsilon = 1.0 \).
Fig. 4. Microstructural evolution as a function of strain showing the effect of deformation-enhanced phase accommodation in Ti-6Al-4V-2Ni at SPF temperature (815°C) and intermediate strain-rate ($\dot{\varepsilon} = 2.0 \times 10^{-4} \text{ s}^{-1}$).
Fig. 5. Accommodation process during SPD in Ti-6Al-4V-2Ni, showing β-phase injection between two α-phase grains. SA-1-815, $T = 815°C$, $\dot{\varepsilon} = 2.0 \times 10^{-4} \text{ s}^{-1}$, $\varepsilon = 0.6$. 
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LIST OF PERSONNEL INVOLVED IN THE RESEARCH

1. Prof. Amiya K. Mukherjee: Principal Investigator
2. Mr. Benjamin Hidalgo-Prada: Research Engineer
3. Mr. Michael Meier: Research Assistant
LIST OF COUPLING ACTIVITIES

1. Dr. Amit Ghosh and Dr. Murray Mahoney: North American Rockwell Science Center. Informal interaction on superplasticity of 7475-T6 aluminum alloy and Ti-6Al-4V alloy.

2. Dr. J. Wadsworth: Lockheed Palo Alto Lab; interaction with respect to Al-Cu-Li alloys with Zr additions for superplasticity studies.

3. Dr. S. Shastri: McDonnell Douglas Corporation Aircraft Company Research Lab, St. Louis, MO; informal interaction on effect of alloy addition in modifying the properties of Ti-6Al-4V base alloy.