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# Effect of hydrogen on the superplasticity and microstructure of Ti-6Al-4V alloy

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### Abstract

The influence of hydrogen on the superplasticity and microstructure in a Ti-6Al-4V alloy has been investigated. The addition of a small amount of hydrogen into the alloy improves the superplasticity, decreases the dislocation density and promotes the flow of  $\beta$  phase along the  $\alpha$ - $\alpha$  grain boundaries. The correlations of the optimum superplasticity with the microstructural factors were discussed.

Keywords: Titanium alloy; Hydrogen; Superplasticity; Microstructure

### 1. Introduction

Hydrogen-induced hot workability in titanium alloys is one of the objects of study currently receiving much attention [1-3]. However, the hydrogen-induced superplasticity of titanium alloys has been less investigated. Ti-6Al-4V alloy is a material widely used in the aeronautical industry. This paper reports the results of an investigation on the influence of hydrogen added into Ti-6Al-4V alloy as a temporary element on the superplasticity and microstructure.

### 2. Material and experimental methods

The material used was a Ti-6Al-4V alloy bar 18 mm in diameter. Its chemical composition (wt.%) is listed as follows: Al, 5.73; V, 4.07; Fe, 0.09; Si < 0.04; C, 0.022; N, 0.016; H < 0.005; Ti, balance. The alloy was annealed at 900 °C for 4 h in order to obtain a stable microstructure. The mean grain sizes of  $\alpha$  and  $\beta$  phases are 6.84  $\mu$ m and 6.77  $\mu$ m respectively. Hydrogenation of the alloy was performed in mixed atmospheres of argon and hydrogen at 650 °C for 1 h. Because of the large diffusion rates of hydrogen in the titanium alloys [4] the through penetration of hydrogen into the specimens took place. The superplasticity tensile tests were conducted on an Instron 1185 machine at 800 °C. The flow stress-strain rate behaviours were examined by the step tensile velocity method. The microstructure and dislocation configurations were examined by optical metallography and transmission electron microscopy.

### 3. Experimental results and discussion

### 3.1. Superplastic deformation behaviours

Fig. 1(a) shows the log  $\sigma$ -log  $\dot{\epsilon}$  curves for specimens with various hydrogen contents tested at T = 800 °C. Hydrogenation causes a decrease of the flow stress of the specimens with 0.09-0.54 wt.% H at the strain rates tested. At  $\dot{\epsilon} > 4 \times 10^{-5}$  s<sup>-1</sup>, the flow stress gradually decreases when the hydrogen content increases from 0.09 to 0.32 wt.%. However, as the hydrogen concentration reaches 0.54 wt.%, the flow stress increases. At  $\dot{\epsilon} = 4 \times 10^{-5} \text{ s}^{-1}$ , the  $\sigma$  value of the specimen containing 0.13 wt.% H is lower than that of the unhydrogenated specimen by about 17 MPa. So it is concluded that hydrogen lowers the flow stress and this decrease is correlated with the strain rate. Generally, at large strain rates (greater than  $10^{-4}$  s<sup>-1</sup>), the specimens with relatively large hydrogen contents (up to 0.32 wt.%) have a great drop in flow stress. The increase of flow stress of the specimen with 0.54 wt.% H is due to the precipitation of titanium hydride ( $\gamma$  phase, as shown later). In contrast, at small strain rates (less than  $10^{-4}$  $s^{-1}$ ), the influence of hydrogen on the flow stress is less and the optimum content is 0.13 wt.% H.

Fig. 1(b) shows the  $m-C_{\rm H}$  curves of specimens tested at 800 °C and at different stress levels. The maximum



Fig. 1. Relationship of hydrogen concentrations with superplastic deformation behaviours: (a)  $\sigma - \dot{\epsilon}$  curves; (b)  $m - C_H$  curves.

value of the strain rate sensitivity m was obtained for the specimen containing 0.13 wt.% H in the case of  $\sigma = 10$  MPa. The m value reduces with increasing hydrogen content.

### 3.2. Microstructures of superplastically deformed specimens

## 3.2.1. Effect of hydrogen on phase constitution and grain sizes of $\alpha$ and $\beta$ phases

Fig. 2 shows the microstructures of specimens deformed at 800 °C to  $\delta = 200\%$  and quenched from this temperature. The unhydrogenized specimen has an equiaxed  $\alpha + \beta$  microstructure (Fig. 2(a)) and the specimen with 0.17 wt.% H has approximately the same microstructure. However, in specimens with hydrogen contents larger than 0.3 wt.% H the  $\gamma$  phase (TiH<sub>2</sub>, f.c.c., a = 0.441 nm) appears. In this case, the  $\alpha$ ,  $\beta$  and  $\gamma$  phases coexist in the specimens (Fig. 2(b)). The



Fig. 2. Influence of hydrogen on microstructures of alloys: (a) not hydrogenized; (b) 0.32 wt.% H; (c) 0.54 wt.% H.

amount of  $\gamma$  phase increases with increasing hydrogen content. The morphology of the  $\gamma$  phase is shown in Fig. 2(c).

The grain sizes of specimens with different hydrogen contents heated at 800 °C and quenched from this temperature were measured and the correlations of the hydrogen content to the grain sizes and the volume fractions of  $\alpha$  and  $\beta$  phases are exhibited in Fig. 3. With increasing hydrogen content, the grain size of  $\alpha$ phase  $d_{\alpha}$  is reduced and that of  $\beta$  phase  $d_{\beta}$  increases. There is a similar but opposite tendency of variation in direction for both phases. In the case of 0.13–0.17 wt.% H, nearly equal fine microstructures ( $d < 8 \mu m$ ) were observed. Accompanying the increase of hydrogen content,  $d_{\beta}$  increases and  $d_{\alpha}$  reduces rapidly. In contrast, in the case of 0.1–0.17 wt.% H, the volume fractions of  $\alpha$  and  $\beta$  phases have much the same value (0.4–0.5). As the hydrogen content increases further, the volume



Fig. 3. Relationship between hydrogen content and microstructural parameters of  $\alpha$  and  $\beta$  phases:  $f_{\alpha}$  and  $f_{\beta}$  denote the volume fractions of  $\alpha$  and  $\beta$  phases respectively;  $d_{\alpha}$  and  $d_{\beta}$  denote the grain sizes of  $\alpha$  and  $\beta$  phases respectively.

fraction of  $\alpha$  phase  $f_{\alpha}$  reduces and the volume fraction of  $\beta$  phase  $f_{\beta}$  enlarges. According to the empirical microstructural conditions of  $\alpha + \beta$  titanium alloys for superplasticity [5,6], fine (less than 10  $\mu$ m) and equiaxed  $\alpha$  and  $\beta$  grains and probably equal volume fractions of both phases at high temperatures are required. Consequently, it is suggested that in view of the microstructure the hydrogenation of the specimens to the range of 0.1–0.17 wt.% H is favourable to the superplastic deformation. Raising the hydrogen content to about 0.3 wt.% H leads to the precipitation of  $\gamma$  phase, so the microstructural conditions for superplasticity are not fulfilled and the superplasticity deteriorates.

# 3.2.2. Effect of hydrogen on dislocation density and $\alpha$ - $\alpha$ grain boundary state

For examination of the dislocation density and the grain boundary state, the specimens deformed to  $\delta = 200\%$  at 800 °C were quenched to room temperature. The dislocation structure was studied by transmission electron microscopy. For classification of dislocation densities, the observed fields were divided into three classes: (1) class A, with none or a few dislocations, (2) class B, with a certain number of dislocations, and (3) class C, with many dislocations. Fig. 4 illustrates the statistical results of 100 observed fields for specimens with various hydrogen contents. It is noticed that, in the range of hydrogen contents of 0.18 wt.% or less, with increasing hydrogen content the number of grains with high dislocation density is reduced and that with low dislocation density is increased. The decrease of dislocation density implies the release of pinned dislocations and the promotion of dislocation climbing probability, which corresponds to the enhanced diffusion ability of alloying elements in the alloy due to hydrogenation in titanium [7].

In order to examine the  $\alpha$ - $\alpha$  grain boundary state, the grain boundaries were divided into three states: (1) no dislocation pile-up (ND), (2) existence of pileup (DP) and (3)  $\beta$  phase flow along the grain boundary ( $\beta$ F). Fig. 5 gives example photographs of the latter two classes. The statistical results of observation shown



Fig. 4. Histogram of the numbers of  $\alpha$  grains with different dislocation densities ( $T_{sp} = 800$  °C;  $\dot{\epsilon} = 1.7 \times 10^{-4}$  s<sup>-1</sup>;  $\delta = 200\%$ ; A, B and C denote class A, class B and class C fields respectively): (a) unhydrogenized; (b) 0.1 wt.% H; (c) 0.18 wt.% H.



Fig. 5.  $\alpha$ - $\alpha$  grain boundary states; (a) pile-ups of dislocations; (b)  $\beta$  phase flow along an  $\alpha$ - $\alpha$  grain boundary.



Fig. 6. Statistics of  $\alpha - \alpha$  grain boundary states ( $T_{sp} = 800$  °C;  $\dot{\epsilon} = 1.7 \times 10^{-4} \text{ s}^{-1}$ ;  $\delta = 200\%$ ): (a) not hydrogenized; (b) 0.18 wt.% H.

in Fig. 6 indicate that hydrogenation reduces the number of grain boundaries with dislocation pile-ups and increases that with  $\beta$  phase flow.

The modern theory of superplasticity emphasises that grain boundary sliding is the basic mode of deformation and that the superplastic strain rate is composed of the flow rate accommodated by diffusion and the creep rate accommodated by dislocations at grain boundaries and in grains [8–10]. It is suggested that the addition of hydrogen into titanium alloy favours the release of pinned dislocations in grains and at grain boundaries and promotes the dislocation gliding and climbing, so that the possibility of  $\beta$  phase flow arises and the dislocation accommodation effect required for gliding along an  $\alpha$ - $\alpha$  boundary is increased.

### 4. Conclusion

(1) The superplasticity of Ti–6Al–4V alloy is improved by additions of small amounts of hydrogen. The optimum superplasticity at 800 °C is obtained by charging the specimens with 0.13–0.32 wt.% H according to the strain rates. The  $\gamma$  phase precipitates in the specimens containing more than 0.32 wt.% H.

(2) Introducing hydrogen into the alloy decreases the dislocation density, promotes the dislocation motion and facilitates the  $\beta$  phase flow.

#### References

- W.R. Kerr et al., *Titanium'80, Science and Technology, New York*, Vol. 4, The Metallurgical Society of AIME, Warrendale, PA, 1980, p. 2477.
- [2] V.A. Kopachev and V.K. Nosov, Fiz. Met. Metallored., 57(1) (1984) 175.
- [3] R.J. Liderich, S.M. Sastry, J.E. O'Neal and W.R. Kerr, in D.F. Hasson and C.H. Hamilton (eds.), *Advancing Processing Methods* for Titanium, 1982, p. 115.
- [4] J.E. Costa, D. Banerjee and J.C. Williams, in R.R. Boyer and H.W. Rosenburg (eds.), *Beta Titanium Alloys in the 1980's*, Metallurgical Society of AIME, Warrendale, PA, 1983, p. 69.

- [5] C.H. Hamilton, A.K. Ghosh and M.M. Mahoney in H.E. Paton and C.H. Hamilton (eds.), *Superplastic Forming of Structural Alloys, Conf. Proc.*, Metallurgical Society of AIME, Warrendale, PA, 1982, p. 129.
- [6] C.H. Hamilton, in S.P. Agrawal (ed.), Proc. Symp. on Superplastic Forming, American Society for Metals, Metals Park, OH, 1984, p. 13.
- [7] L. Zhao, S. Zhang and M. Yan, Mater. Sci. Prog., 4(3) (1990) 237.
- [8] C. Hammond, in N.E. Paton and C.H. Hamilton (eds.), Superplastic Forming of Structural Alloy, Metallurgical Society of AIME, Warrendale, PA, 1982, p. 131.
- [9] R.C. Gifkins, in N.E. Paton and C.H. Hamilton (eds.), Superplastic Forming of Structural Alloys, Metallurgical Society of AIME, Warrendale, PA, 1982, p. 3.
- [10] A. Arieli and A. K. Mukherjee, Mater. Sci. Eng., 45 (1980) 61.