

Effect of hydrogen on superplastic deformation of (TiB $+$ TiC)/Ti–6Al–4V composite

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ABSTRACT

Ti–6Al–4V matrix composite reinforced with TiB plus TiC was prepared and hydrogenated. The phases were identified by X-ray diffraction (XRD). Microstructures were examined by optical microscopy (OM). Dependence of transus temperatures of the composite on hydrogen concentration was determined. The result shows hydrogen decreases transus temperatures of the composite significantly and increases the amount of β phase. Superplastic deformation of the hydrogenated composites was performed. Hydrogen decreases the optimum superplastic temperatures and increases the optimum superplastic strain rate. The flow stresses of hydrogenated composites decrease greatly compared to unhydrogenated composite. The strain rate sensitivity index m of the composite increases with increasing hydrogen concentration and reaches maximum 0.327 at 0.85 wt.% H. Meanwhile, the activation energy Q decreases with increasing hydrogen concentration and ranges in 488–382 kJ/mol. Hydrogen promotes recrystallization of the composite and refines the microstructure during superplastic deformation.

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1. Introduction

Titanium matrix composites (TMCs) reinforced with ceramic particulates have significant potential for structural applications due to their outstanding combination of high specific strength, elevated temperature resistance and stiffness as well as low density [\[1–3\]](#page--1-0). If an isotropic material and its hot forming are needed, the particulate reinforced titanium matrix composites are a better choice than the continuous reinforced metal matrix composites, normally at lower prices [\[4\].](#page--1-0) However, their high hardness limits their ability to be machined. Superplastic forming as a near-net-shape technique has generated strong interest for TMCs to reduce production costs.

Recently, hydrogen as a temporary alloying element has become a powerful tool in improving the titanium alloys hot workability [\[5–8\],](#page--1-0) superplastic forming [\[9–12\]](#page--1-0), powder consolidation [\[13,14\]](#page--1-0), microstructure refinement [\[15–18\]](#page--1-0), diffusion bonding [\[19\]](#page--1-0) and composite fabrication [\[20\].](#page--1-0) The presence of the hydrogen allows the titanium alloy to be: (a) processed at lower stresses and/or lower temperatures, and (b) heat-treated to produce novel microstructures with enhanced mechanical properties [\[21\].](#page--1-0) Machida et a1. [\[22\]](#page--1-0) had studied effect of hydrogenation treatment on grain refinement of TiC/Ti–6Al–4V composite. They produced fine grain structure by severe plastic deformation after hydrogenation treatment and improved mechanical properties of composites.

In this study, Ti–6Al–4V matrix composite reinforced with TiB plus TiC was fabricated using in situ technology. The mechanical properties of the composite were enhanced significantly. However, the high temperature deformation of

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the composite is hard due to the addition of reinforcements. This work is aimed to utilize the hydrogen to improve the superplastic properties of the composite.

2. Experimental procedure

Ti–6Al–4V matrix composite reinforced with TiB plus TiC was prepared by common casting technique based on the following reactions [\[2,23\]:](#page--1-0)

 $5Ti + B_4C \rightarrow 4TiB + TiC$ (1)

$$
Ti + C \rightarrow TiC \tag{2}
$$

The composite with 5 vol.% reinforcements (mole ratio of TiB and TiC is 1:1) was fabricated by consumable vacuum arc remelting. A stoichiometric ratio of sponge titanium, AlV alloy, pure Al, B4C powder and graphite powder were blended thoroughly and melted homogeneously in a consumable vacuum arc-remelting furnace. The ingots were melted twice to ensure chemical homogeneity. After casting, the ingots were hot-forged into a rod of 25 mm diameter. The total deformation degree was over 90%. Before hydrogenation, the composite was annealed to obtain equiaxed microstructure.

Specimens were hydrogenated using high temperature hydrogen charging furnace at 750 $^{\circ}$ C for 2 h. Hydrogen concentrations were determined by weighing the specimens before and after hydrogenation [\[11,13\]](#page--1-0).

Tensile specimens with a gauge section of $4.4 \times 1.6 \times 8$ mm were cut from the above hydrogenated specimen, with axes parallel to the hot-forging direction. The superplastic tensile tests were performed using a SHIMADZU AG-10KNA test machine. Tensile tests were conducted in air at temperatures ranging from 780 $^\circ\textsf{C}$ to 980 $^\circ\textsf{C}$ and initial strain rates ranging from 1 \times 10 $^{-4}$ s $^{-1}$ to 1 \times 10 $^{-2}$ s $^{-1}$. All specimens were coated by enamel in order to resist oxidization.

Optical microscopy (OM) was carried out after superplastic deformation. The samples for OM were prepared using conventional grinding and mechanical polishing techniques. The polished samples were etched in Kroll's reagent (composition: 1-3 ml HF, 2-6 ml HNO $_3$, 100 ml water). Phase identification of the composites was performed by a Siemens D-500 X-ray diffractometer (XRD).

3. Results and discussion

Fig. 1 shows the XRD patterns of hydrogenated composites. The relative intensities of β -Ti peaks increase with increasing hydrogen concentration. Hydrogen improved the stability of β phase and more β phase was retained. Plenty of hydride forms at high hydrogen concentration.

The microstructures of the annealed (0 wt.% H) and hydrogenated composites are shown in [Fig. 2](#page--1-0). The annealed composite has an equiaxed microstructure composed by equiaxed α phase and grain boundary β phase. The mean grain size of α phase is about 10 μ m. The composite with 0.20 wt.% H holds the original morphology. Its microstructure was not easy to etch clearly because hydrogen changed the potential

Fig. 1 – XRD patterns of the composites with (a) 0 wt.% H (b) 0.20 wt.% H (c) 0.45 wt.% H (d) 0.85 wt.% H.

difference of α phase and β phase. The colors of the two phases become undistinguishable. The relatively light-colored phase is β phase, which is dark-colored in annealed composite. It can be seen that the volume fraction of β phase increases significantly. At 0.45 wt.% H, more β phase formed and the colors of the two phases completely reversed. At 0.85 wt.% H, the microstructure of the composite hardly changed and the volume fraction of α and β phase kept constant. The result of OM agreed with the analysis of XRD patterns.

The β transus temperatures of hydrogenated composites were measured by metallographic method. In the metallographic method, the samples with dimension 10 \times 10 \times 10 mm are encapsulated in quartz tubes to prevent hydrogen escape. After holding for 15 min at given temperature, the encapsulated samples are water quenched. The microstructures of the samples quenched from different temperatures are observed and the temperature at which the primary a phase disappears is determined as the β transus temperatures. The interval of quenched temperatures is 10° C. So the precision of this method is 5 \degree C. The results are presented in [Fig. 3.](#page--1-0) The transus temperatures decrease with increasing hydrogen concentration. And at 0.45 wt.% H, the transus temperature reaches 825 °C and hardly changes at higher hydrogen concentrations. This suggests that the line at higher hydrogen concentrations represents eutectoid temperature [\[5\]](#page--1-0). Therefore, a great deal of hydride forms at 0.45 wt.% H or higher hydrogen concentrations. This result agrees with the XRD results.

According to the results in [Fig. 3,](#page--1-0) the temperatures in $\alpha + \beta$ field nearby the transus temperatures were chosen as the testing temperatures for superplastic deformation. [Fig. 4](#page--1-0) shows the elongation of hydrogenated composite as a function of testing temperature at initial strain rate of 1 \times 10 $^{-3}$ s $^{-1}$. On the whole, the elongations of hydrogenated specimens increase at a fixed temperature in $\alpha + \beta$ field. The specimens with 0.45 wt.% H and 0.85 wt.% H possess the elongations of 295% and 305% while the original composite possesses an

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