Sintering Properties of Ti–6Al–4V Alloys Prepared Using Ti/TiH₂ Powders

J.-M. Oh¹, K.-H. Heo², W.-B. Kim¹, G.-S. Choi³ and J.-W. Lim¹,*

¹Mineral Resources Research Division, Korea Institute of Geoscience and Mineral Resources, Daejeon 305-350, Korea
²Division of Management & Planning, Korea Foundation for the Advancement of Science and Creativity, 602, Seollungno, Gangnam-gu, Seoul 135-867, Korea
³Gangwon Industrial Technology Research Center, Research Institute of Industrial Science & Technology, Gangneung 210-340, Korea

Ti–6Al–4V alloy specimens were prepared by sintering Ti/TiH₂ powders with pre-alloyed TiAl and Al–V powders. Changes in the sintered density, Vickers hardness, and phase structure of the Ti–6Al–4V (Ti6Al4V) and hydrogenated Ti–6Al–4V (H-Ti6Al4V) alloys as a function of the vacuum sintering temperature (700–1300°C) were investigated. The sintered density and Vickers hardness of the Ti6Al4V and the H-Ti6Al4V alloys increased as the sintering temperature increased, while the concentration of oxygen considerably decreased with increasing sintering temperature. The oxygen concentration of the H-Ti6Al4V alloy was lower than that of the Ti6Al4V alloy. The sintered density and Vickers hardness values of the Ti6Al4V and H-Ti6Al4V alloys were almost the same. These results suggest that the preparation of Ti6Al4V alloy using TiH₂ powder is more cost-effective and produces sintered products with lower oxygen concentrations.

1. Introduction

The mechanical properties of sintered products made using metal titanium powder are inferior to those of cast products. However, it is very cost-effective for high-performance environments. Therefore, recent research has focused on improving the mechanical properties of titanium sintered products. Accordingly, several researchers have studied the effects of the use of TiH₂ powder on the properties of titanium sintered products.²–⁴ TiH₂ powder is a semi-final product of the hydrogenation–dehydrogenation (HDH) process for the powder metallurgy of titanium. Kim et al.⁵ reported that Ti sintered products made using TiH₂ powder had a higher sintering density and Vickers hardness than those prepared using pure Ti powder. Hattori et al.⁶ revealed that using MH₂ (M = Ti, Zr, Hf) powder results in significantly increased densification of the sintered product with increasing sintering temperature. This densification was described to occur due to the clean metal powders produced via the dehydrogenation reaction (i.e., MH₂ → M + H₂) during sintering. However, few studies have reported the effect of the use of TiH₂ on basic properties, including hardness, density and the microstructure of Ti–6Al–4V alloys.

Therefore, in this study, we investigated the effect of the use of Ti/TiH₂ powders on the phase structure, oxygen concentration, Vickers hardness and sintering density of Ti–6Al–4V (Ti6Al4V) and hydrogenated Ti–6Al–4V (H-Ti6Al4V) alloys.

2. Experimental

Pure Ti and TiH₂ prepared via the HDH process were obtained from SE-JONG Materials (South Korea). The microstructures of the Ti and TiH₂ powders were examined using scanning electron microscopy (SEM). TiAl (1 : 1) and Al–V (2 : 8) pre-alloy powders were used to prepare master alloys; Al is lost via evaporation during sintering at high temperature due to the low melting temperature of Al. The weight ratio of Ti/TiH₂ : Al–V was nearly 90 : 6 : 4. The mixed powders were compacted at 3000 kgf/cm² using a cold isostatic press in a rubber mold (27 mm diameter × 70 mm height). The compacted specimens of the Ti6Al4V and H-Ti6Al4V alloys were sintered at 700, 800, 900, 1000, 1100, 1200 and 1300°C at 1.3 × 10⁻³ Pa for 4 h.

The sintered specimens were sliced every 2 mm and their cross sections were examined for their microstructure and mechanical properties. The sintered density of the specimens was calculated using Archimedes’ method. The oxygen concentrations of the Ti6Al4V and H-Ti6Al4V alloys were measured via inert gas fusion infrared absorption using an O/N analyzer (LECO TC-436). The structures of the specimens were determined via X-ray diffraction (XRD, Rigaku RTP 300 RC) using Cu-Kα radiation. The Vickers hardness was tested using a 250 g load with an Akashi (MVK-E) hardness tester. Excluding the maximum and minimum values, the average value was determined from 10 measurements.

3. Results and Discussion

Images of the morphology of the Ti and TiH₂ powders before vacuum sintering heat treatment are shown in Fig. 1. Both images present a broad distribution of particles ranging from 20 to 100 µm; however, the TiH₂ powders are finer than Ti powders, which is similar to previous results that showed that Ti powders produced by HDH have larger grains than TiH₂ powders.⁵ XRD patterns of Ti6Al4V and H-Ti6Al4V alloys sintered at various temperatures ranging from 700 to 1300°C are shown in Fig. 2. These patterns indicate the presence of Ti and alloying elements at the sintering temperature of 700°C. As shown in Fig. 2, peaks B and C, which correspond to TiAl intermetallic compounds and V
of Al–V alloys, appeared in the temperature range of 700 to 900°C; however, these peaks disappeared above 1000°C. From the XRD patterns, it was determined that the Ti6Al4V and H-Ti6Al4V alloys were successfully prepared from TiAl intermetallic compounds and V of Al–V alloys above the sintering temperature of 1000°C.

The maximum oxygen solubility of Ti is approximately 30 at%, and it has a significant influence on the physical, mechanical, and electrical properties of Ti.7) Accordingly, recent research has focused on the de-oxidation of Ti.8,9) Figure 3 shows the variation of the oxygen concentration in Ti6Al4V and H-Ti6Al4V alloys as a function of sintering temperature. The oxygen concentration in the H-Ti6Al4V alloys was lower than in the Ti6Al4V alloys; the low oxygen concentration of the H-Ti6Al4V alloys is thought to be due to the clean Ti powders produced via the dehydrogenation reaction (i.e., TiH2 → Ti + H2) during vacuum sintering.5) The concentration of oxygen in the Ti6Al4V and H-Ti6Al4V alloys decreases with increasing sintering temperature. The two different slopes shown in Fig. 3 arise due to changes in the microstructural defects such as internal pores of the sintered body that are caused by the low sintering temperature. As the sintering temperature increases up to 1000°C, the concentrations of oxygen in the Ti6Al4V and H-Ti6Al4V alloys reduced to 6720 and 5950 ppm, respectively, which are ~22 and ~28% lower, respectively, than that of the sintered body at 700°C. Presumably, this is due to considerably increased densification of the sintered body with increasing temperature, which reduces the internal pores. Upon increasing the sintering temperature from 1000 to 1300°C, the oxygen concentration in the Ti6Al4V and H-Ti6Al4V alloys remained relatively constant, especially in comparison to the steep slope between 700 and 1000°C. For pure Ti and TiH2 powder sintering, studies have shown that the oxygen concentration increased with sintering temperature which differs from the current results. This difference is presumably due to the high-temperature oxidation resistance of the Al oxide layers in Ti–Al alloys.

The density of the sintered product is very important in the field of powder metallurgy. The variation of the sintered density and Vickers hardness of the Ti6Al4V and H-Ti6Al4V alloys as a function of sintering temperature is shown in Fig. 4. After compaction, the density of the Ti6Al4V and H-Ti6Al4V powders were 3.15 and 3.17 g/cm³, respectively. The packing factor of the H-Ti6Al4V alloy is greater than that of the Ti6Al4V alloy because the theoretical density of TiH2 powders is smaller than that of Ti powders. The sintered density increased from 79 to 96% and from 81 to 97% for the Ti6Al4V and H-Ti6Al4V alloys, respectively, at sintering temperatures from 700 to 1300°C. The respective highest densities of the
Ti6Al4V and H-Ti6Al4V alloys were 96 and 97%; these values are close to those reported. At lower sintering temperatures from 700 to 900°C, inhomogeneous microstructures in the Ti-based alloys generally result in coarse porosity and low density. However, these results revealed a small enhancement of the density of the Ti–6Al–4V alloy with TiH₂ powder at low sintering temperatures; this enhancement from the TiH₂ powder was not found at higher sintering temperatures. For pure Ti and TiH₂ powder sintering, the effect of sintering on the density of TiH₂ powders was greater than that on the density of Ti powder. However, the use of TiH₂ powder for the Ti6Al4V alloy did not affect the densification. This may be due to alloying with Al and V, which affects the density of the Ti/TiH₂6Al4V alloys due to substitution by Al and V during sintering.

Figure 4 shows the effect of the sintering temperature on the Vickers hardness of the Ti6Al4V and H-Ti6Al4V alloys. The Vickers hardness of both alloys almost linearly increased with increasing sintered density as a function of the rise in sintering temperature. At a sintering temperature of 1300°C, the Vickers hardness was ~350 Hv for both the Ti6Al4V and H-Ti6Al4V alloys. For comparison, the hardness of hot-wrought Ti alloys is generally reported to be ~350 Hv. The Vickers hardness of both the Ti6Al4V and H-Ti6Al4V alloys were similar at sintering temperature of 1200 and 1300°C. During high-temperature sintering, the melt of Al particles driven by capillary forces would fill the pore structure or inter-particle spaces within the compacted microstructures of the Ti6Al4V and H-Ti6Al4V alloys. Additionally, Al diffuses interstitially into Ti and V particles according to their respective solubility, which occurs in manner similar to the mechanism for Ti–6Al–7Nb alloy production via powder metallurgy. The variation of the sintered density and Vickers hardness shows that the TiH₂ powder has a no significant effect on the sintering of the Ti6Al4V alloy compared to pure Ti powder.

Thus, there are two major successes in this experiment: First, the preparation of Ti6Al4V alloy using TiH₂ powder can reduce cost and eliminate on step due to the utilization of TiH₂ powder, which is obtained as a semi-final product of the HDH process. Second, the Ti6Al4V alloy made using TiH₂ powder has better mechanical properties and lower oxygen concentration than that prepared using pure Ti powder.

4. Conclusions

We prepared Ti6Al4V alloys via the sintering of Ti/TiH₂ powders with pre-alloyed TiAl and Al–V powders. The sintered densities and Vickers hardness values of the Ti6Al4V and H-Ti6Al4V alloys increased as the sintering temperature increased, while the oxygen decreased considerably with increasing sintering temperature. The concentration of oxygen in the H-Ti6Al4V alloys was lower than that in the Ti6Al4V alloys. It was determined that the Ti6Al4V alloy made using TiH₂ powder has better mechanical properties and lower oxygen concentration than those made using pure Ti powder. Therefore, preparation of Ti6Al4V alloy using TiH₂ powder can reduce costs due to the utilization of TiH₂ powder, which is obtained as a semi-final product of the HDH process.

Acknowledgements

This research was supported by the Basic Research Project of the Korea Institute of Geoscience and Mineral Resources (KIGAM) funded by the Ministry of Knowledge Economy of Korea.

REFERENCES