Evaluation of HDH process of Ti-6Al-4V alloy

João Pedro Valls Tosetti
Flávio Beneduce Neto
Daniel Rodrigues

Metallurgy Division – IPT
Av. Prof. Almeida Prado, 532, São Paulo – SP - 05508-901, Brazil
danielrd@ipt.br

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Abstract: An alternative route to the conventional hydriding and dehydriding process applied to Ti grade 5 (Ti-6Al-4V) were evaluated considering that dehydriding and sintering were carried out in a single step. Microstructure evolution, hydriding degree, chemical composition and shrinkage during sintering were investigated. High hydriding degree was obtained. A shrinkage higher than 15% was observed, and a sintered density 95% of the theoretical was obtained in spite of coarse milled hydride particles were used.

Introduction

Titanium grade 5 (Ti-6%Al-4%V) is the most widely used titanium alloy. Due to the combination of high mechanical and corrosion resistance and low density, this alloy finds application at aerospace and chemical industries and orthopedic application.

There are two basic methods used to produce titanium alloy powders: the blended elemental [1, 2, 3] and the pre alloyed techniques [1, 4]. The usual route is shown in figure 1A, which comprises dehydriding and sintering as two separated and independent steps. An alternative route has been suggested [1] where sintering and dehydriding are carried out in one single step. This route is shown in figure 1B and was adopted in this work.
Experimental Procedure

Ti-6Al-4V forged rods (φ25 mm) were used as raw material. After a careful surface cleaning, in order to remove any organic or inorganic impurities, the rods were submitted to a hydriding cycle of 2 h, under 0.13 MPa of pure H$_2$ at 1000°C followed by a cooling rate of about 1°C/min up to room temperature. The hydrided was then milled in a ring vibrating mill for 5 min. Figure 2 shows the aspects of milled powder. The morphology is irregular and particle size distribution is broad, so particles as fine as 1 µm and as coarse as 20µm can be observed.

The hydrided material was submitted to a thermal analysis (DTA). Other sample was pressed at 200 MPa. Since the hydrde is brittle, a higher pressure would not increase either green density or green strength. The green strength was quite low and it was not possible to measure green density. The
pressed sample was then dehydrided and sintered simultaneously inside a dilatometer at 1300°C, for 30 min, under a dynamic pure argon atmosphere.

**Results and Discussion**

Figure 3 shows the results of (DTA). As a reference, a curve for pure titanium hydride powder is also shown.

![Graph](image)

**Figure 3:** DTA of Ti-6Al-4V and Ti hydride powders. Heating rate 10º/min. Dynamic argon.

One can observe that an endothermic reaction occurs at around 600 C and might be related to the decomposition of the hydride. For the pure titanium hydride this reaction occurs at a slight lower temperature, even considering the accuracy of analysis. Another endothermic reaction at 350 C can be observed and it may be related to a phase transition of the titanium hydride.

Figure 4 shows dilatometric results for a Ti-6Al-4V hydrided pressed sample. It is possible to observe that shrinkage starts at low temperature (around 350ºC) and almost two thirds of all shrinkage (10%) happens at the heating step. The sintered density was 4.22 g/cm³ (95% of the theoretical). As the decomposition of the hydride occurs at 600ºC (Fig. 3) possibly the shrinkage at lower temperature should be related to neck growth between hydride particles.

![Graph](image)

**Figure 4:** Dilatometric curve for the simultaneous dehydriding and sintering Ti-6Al-4V sample.

Figure 5 shows the microstructure of the sintered material. This figure shows α (dark), an intergranular (light) vanadium rich β phase and rounded pores. The presence of pores at grain
boundaries suggests that values of density greater than 4.22g/cm³ can be get considering neck growth by atomic grain boundary diffusion. The phase diagram presented in figure 6 [5] shows that at low temperature the equilibrium phases are $\alpha$-Ti, $\beta$-(Ti,V), and Ti$_3$Al.

**Figure 5:** Microstructure of sintered Ti-6Al-4V. $\alpha$ phase, intergranular $\beta$ phase, and pores. As polished. SEM-BSI (Back Scattered Image).

**Figure 6:** Phase diagram Ti-Al-V (6 wt.%Al vertical section).

Figure 7 shows XRD patterns comparing hydrided powder and sintered samples. No titanium peak could be detected in the hydrided powder, so the hydriding was complete. The sintered sample showed peaks for both $\alpha$ and $\beta$ phases, as already observed in the microstructure and predicted for the equilibrium diagram. The peaks for the hydrided sample are quite broad and it indicates that the hydride crystallographic structure is stressed by the presence of foreign atoms as vanadium and aluminum.
Figure 7: XRD for hydrided Ti-6Al-4V powder and sintered sample. Cu-K_α.

EDS chemical analysis of sintered sample and hydrided powder are presented in table 1. One can see that the β phase has quite higher vanadium content. The hydried powder and sintered samples have basically the same chemical composition, which is around nominal Ti-6Al-4V.

| EDS spectrum results for the matrix and the intergranular phase of the sintered Ti-6Al-4V. |
|---|---|---|
| Matrix (α) | 91.2 | 5.4 | 3.4 |
| Intergranular phase (β) | 82.2 | 4.3 | 13.5 |
| average (global) | 90.0 | 5.4 | 4.6 |
| hydried powder | 89.6 | 6.0 | 4.4 |

Conclusions

- The presence of titanium after hydriding Ti-6Al-4V rods was not detected, i.e., the hydriding was complete.
- Shrinkage during sintering started at low temperature and sintered density (4.22 g/cm³) was quite close to theoretical one.
- The chemical composition, aluminum and vanadium contents, did not show any significant change during hydriding and sintering
- Microstructure of the sintered sample was similar to the obtained by the conventional route. Improvements in the final density can be expected with increasing sintering time at 1300°C.
References


