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Effects of hydrogen content on powder metallurgy characteristic of titanium hydrides

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ABSTRACT

The titanium dihydride (TiH_2) powder metallurgy has been attracted a lot of attention, but TiH_2 powder is difficult to press moulding. In this paper, the titanium hydride powder metallurgy including TiH_2 and unsaturated titanium hydrides ($TiH_{1.5}$) was investigated simultaneously compared with pure titanium metal powder metallurgy. The results indicates that the titanium hydride powder metallurgy is accompanied by the deoxidation self-purification effect during dehydrogenation process for both of TiH_2 and $TiH_{1.5}$, which have higher sintering density than pure titanium. There are the three stages relative to densification rate, namely the slow, rapid and full densification stages for all of three materials. The compressive yield strengths increase rapidly in the rapid densification stage and are unchangeable almost in the full densification stage after holding 2 h at 1300 °C. The titanium hydride powder metallurgy is helpful to obtain much better mechanical properties than the pure titanium metal powder metallurgy. Here the compressive yield strength of the as-sintered TiH_2 compact with the maximum hydrogen content is the best but has very small difference compared with that of the as-sintered $TiH_{1.5}$ compact after full sintering densification.

Introduction

Titanium (Ti) and titanium alloys have many applications, especially in the aerospace industry and human implants, due to the unique combination of excellent mechanical properties, excellent corrosion resistance, low density and good biocompatibility [1–5]. However, the high manufacturing cost with the existing production technologies limits its use in the civil industries, such as the automotive industry [6,7]. Powder metallurgy (PM) has been identified as a viable and promising route to reduce the production costs of titanium metal, owing to the advantages of near-net-shape capabilities and low costs [5,8,9].

Titanium metal has a large affinity with hydrogen, which leads to the formation of titanium hydrides, such as TiH_2, $TiH_{1.5}$, $TiH_{0.71}$ and others [10]. The use of titanium hydride powder instead of titanium metal powder as the starting material provides the advantage of low cost in titanium powder metallurgy because titanium hydride such as TiH_2 is an intermediate product in the hydrogenation - dehydrogenate (HDH) process of titanium sponge [4,7,8]. In addition,

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there are some different performance between titanium hydride and titanium metal in powder metallurgy process, for example, titanium dihydride (TiH_2) powder is brittle extremely and broken easily, thus resulting in different mechanism of compaction and sintering stages [4,8].

Some researchers have expressed great interest in the performance of titanium hydride and other hydrides in powder metallurgy in recent years. And the current researches on titanium hydride are mainly focused on the following points:

(I). The hydrogenation and dehydrogenation mechanism of titanium hydride powder [10,11].
(II). The effects of hydrogen existence on the surface of powder, including the self-cleaning mechanism of dehydrogenation [8,12–14].
(III). The mechanical properties of titanium hydride and titanium alloy hydride in powder metallurgy [15–19].

The researches mentioned above mainly focus on the saturated titanium hydride (TiH_2) as the starting materials, and barely on the unsaturated titanium hydrides ($TiH_{1.5}$) in powder metallurgy. The saturated titanium hydride (TiH_2) is hard and brittle, the compressibility of which is very poor, while the compressibility of the unsaturated hydride titanium ($TiH_{1.5}$) powder is much higher than that of TiH_2 powder. In the paper, we studied the powder metallurgy characteristic of saturated titanium hydride (TiH_2) and unsaturated titanium hydrides ($TiH_{1.5}$) simultaneously compared with pure titanium (HDH-Ti).

Experimental

The bulk sponge titanium was hydrogenated to obtain the TiH_2 and TiH_x at 425 °C in a tube reactor with high-purity hydrogen (99.999%) through controlling the pressure and times of charging hydrogen, which were then crushed and sieved into the powder of 75–100 μm. Some TiH_2 powder was used to dehydrogenate to obtain HDH-Ti powder at higher temperature.

The HDH-Ti, TiH_x and TiH_2 powders were molded under pressure of 200 MPa for 120 s. The green compacts were separately heated to 800 °C, 900 °C, 1000 °C, 1100 °C, 1200 °C and 1300 °C for holding 10 min with the rate of 5 k min^{-1} in the vacuum. And the green compacts were separately holding for 1 h, 2 h, 3 h and 4 h at 1300 °C. The sintering profile of temperature vs time is shown in Fig. 1. The vacuum of the sintering process was 5×10^{-3} Pa, and the samples were cooled to room temperature inside the furnace.

The structure analysis for DHD-Ti, $TiH_{1.5}$ and TiH_2 powder was investigated by X-ray diffraction (XRD, Dan Dong Fang Yuan, DX-2600) with Cu Kα radiation at 35 kV and 25 mA. The evolved gas and weight loss were examined by the TG-MS simultaneously in a thermo-gravimetric analysis (TG, STA449 F3, Netzsch) which is a thermo-balance (MS, QMS403C, Netzsch). The SEM morphology observation of as-sintered ingots was carried out by Hitachi MT3000.

The compression tests for the as-sintered compacts were conducted on an Instron universal testing machine (Model Instron 5569) with a constant speed of 0.5 mm/min at room temperature. The diameter of the samples were measured using vernier caliper with the precision of 0.02 mm to calculate the shrinkage percentage for the sintered samples. Meanwhile, the green density of the samples was calculated by the specific value of weight and volume, where the weight of the samples was obtained using the precision electronic balance (BSA124SCW, precision: 0.1 mg). The sintered density was used by the Archimedes method.

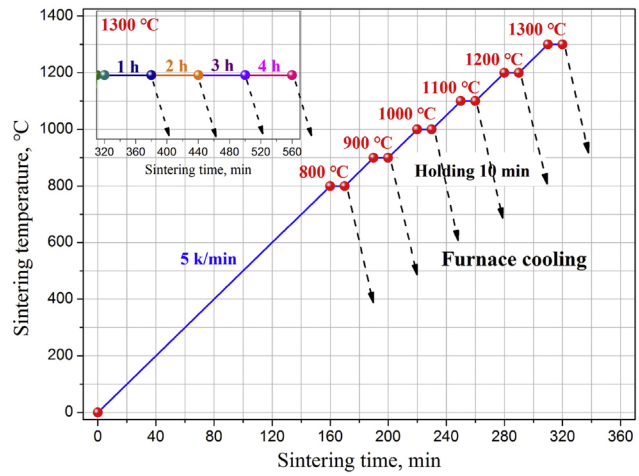

Fig. 1 – Sintering profile of temperature vs sintering time with a heating rate of 5 k min^{-1} in the laboratory vacuum furnace.

Result and discussion

Microstructure change and densification during sintering process

According to the XRD results in Fig. 2, the TiH_2 powder has only $TiH_{1.971}$ phase (PDF: No. 89-4071), and the TiH_x powder consists of $TiH_{1.5}$ (PDF: No. 78-2216) main phase, a small amount of α-Ti (PDF: No. 44-1294) phase and possibly minute

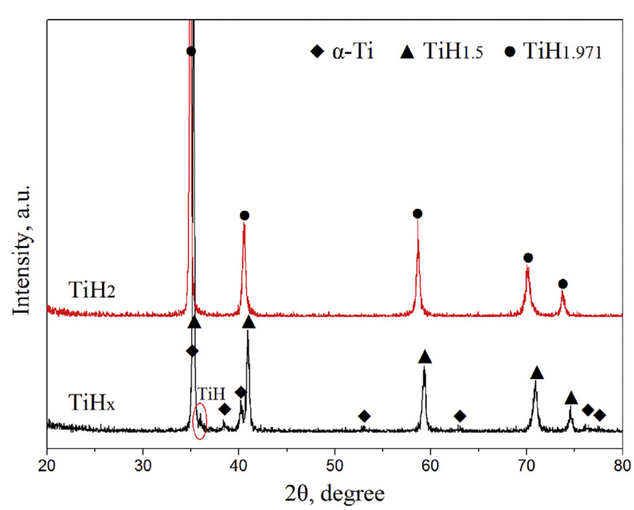

Fig. 2 – The XRD patterns of TiH_2 and TiH_x powders.

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