

# Comparison of hydrogen storage properties of Ti<sub>0.37</sub>V<sub>0.38</sub>Mn<sub>0.25</sub> alloys prepared by mechanical alloying and vacuum arc melting

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#### ABSTRACT

Two process methods, mechanical alloying and vacuum arc melting, were used to prepare  $Ti_{0.37}V_{0.38}Mn_{0.25}$  alloy powders for studying their differences in hydrogen storage capacities.  $Ti_{0.37}V_{0.38}Mn_{0.25}$  samples produced by mechanical alloying showed an amorphous structure and a maximum hydrogen absorption of 1.76 wt%, but those prepared by vacuum arc melting exhibited a single phase BCC structure with no Laves phase, as well as a maximum hydrogen absorption of 3.62 wt%. The hydride in  $Ti_{0.37}V_{0.38}Mn_{0.25}$  alloy after hydrogen absorption was VH<sub>2</sub>, whose low reaction temperature allows for large amount of hydrogen absorption at ambient temperature. The hydride was, however, unstable and decomposed completely at relatively low hydrogen desorption temperature of 200 °C. After absorption–desorption cycling for 100 times, the mechanically alloyed powders, which did not pulverize as much as those of the arc-melting derived powders, showed smaller decline in hydrogen-absorption capability.

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#### 1. Introduction

The search for alternative energy resources has become increasingly important as the massive exploitation of fossil fuels leads to rapid drainage of energy resources [1]. Hydrogen, a clean energy source, can be perpetually supplied with environmental and economical advantages. Currently, the commercial  $AB_5$  type alloy cannot be satisfactorily used as a fuel cell because of its low hydrogen storage capacity. Similarly, the commercial applications of another  $AB_2$  type alloy, though higher in hydrogen storage capacity [2], are also limited by its high working temperature. On the other hand, BCC alloy, because of its excellent hydrogen storage capacity and its absorption–desorption characteristics at ambient temperature, has been regarded as an important hydrogen storage material [3–5]. Of all BCC alloys, vanadium-series alloy is the most promising, exhibiting a maximum storage capacity of 3.8 wt% [6–10]. However the applications of BCC alloys are restricted mainly due to the high price of pure vanadium. In order to bring down the cost of BCC alloys, FeV raw material was used to replace the pure vanadium [11,12], but high hydrogen storage capacities were not obtained. As a result, hydrogen storage ability decreases because of the addition of Fe with impurities of Al and Si. Consequently some researchers focus on the increasing of hydrogen ability by addition of other elements such as Al [13], Ce [14] and Cr [15]. Regarding the practical use, hydrogen absorption–desorption cyclic properties become an important issue. However literatures on the hydrogen absorption and desorption cyclic properties for BCC alloys are scarce. In this study, two

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Fig. 1 – The XRD pattern of  $Ti_{0.37}V_{0.38}Mn_{0.25}$  powders prepared by mechanical alloying.

processing methods, mechanical alloying and vacuum arc melting, were used to prepare TiVMn alloy powders, whose hydrogen storage and absorption–desorption cyclic properties were compared.

#### 2. Experimental

Alloy samples used in this study were prepared by mechanical alloying and vacuum arc melting, followed by mechanical grinding into fine powders. The crystal structures of the powders were examined by an X-ray diffractometer (XRD, Mac Science MXP3). It was operated under 40 kV, 30 mA and 20° to 90° scan angle, using copper target  $K\alpha$  emission with characteristic wavelength of 0.154 nm, and a scanning speed of 3°/min.

Their capability or performance like hydrogen storage curve, cyclic hydrogen absorbing and releasing property was performed with Seivert's type apparatus. Because the formation of a surface-passivation-layer on the samples constitutes a barrier to hydrogen penetration, it is necessary to activate



Fig. 2 – The XRD pattern of  $Ti_{0.37}V_{0.38}Mn_{0.25}$  powders prepared by vacuum arc melting.

the samples before performing hydrogen absorption [16]. To activate the samples, the alloy sample powders (1–1.5 g) were first placed in a hydrogen tank. Next, the tank was vacuumed down to  $8 \times 10^{-3}$  torr with an oil diffusion pump. The samples were then heated to 500 °C and held at this temperature for 1 h. Finally, the heating tank was removed and the samples were allowed to cool to room temperature. The activation process was repeated until a complete reproducibility of the hydrogen sorption capacity was achieved. Alloys activated by this process can absorb hydrogen rapidly. Subsequent hydrogen desorption temperature was kept at 400 °C for 1 h.

#### 3. Results and discussion

## 3.1. Analysis of the absorption–desorption characteristics of $Ti_{0.37}V_{0.38}Mn_{0.25}$ alloy prepared by mechanical alloying

The XRD pattern in Fig. 1 shows the amorphous structure of the powders prepared by mechanical alloying. Fig. 2 shows the XRD pattern of  $Ti_{0.37}V_{0.38}Mn_{0.25}$  prepared by vacuum arc melting. The pattern is identified to be a single phase BCC



Fig. 3 – The hydrogen absorption curve (a) and amount of hydrogen storage (b) of  $Ti_{0.37}V_{0.38}Mn_{0.25}$  prepared by mechanical alloying as a function of pre-treatment temperature.

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