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Introduction

There are many different allotropic forms for TiH₂ such as α, β and δ-phases depending on the temperature, hydrogen concentration and applied pressure [1].

TiH₂ is brittle and can be pulverized to nanocrystalline form by mechanical milling for short periods of time. The hydride can then be dehydrogenated to form Ti powder. The fine powder form of hydride has applications in bonding metals with non-metals and for hydrogen storage [2].

All these applications involve in dehydrogenation of either TiH₂ or lower hydrogen containing compounds under controlled atmosphere. Therefore, a basic understanding of TiH₂ dehydrogenation is essential to develop new applications.

Method

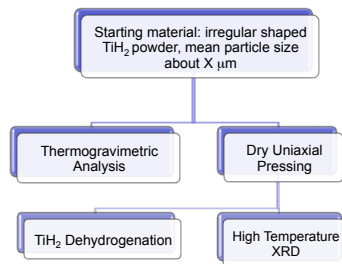


Figure 1: Experimental Procedures

Results and Discussions

1. Thermal Decomposition Characteristics by using TGA

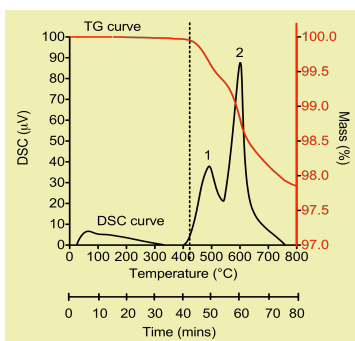


Figure 2: TG and DSC curves for nonisothermal decomposition of Japanese TiH₂ in Argon at 10 °C/min. The dehydrogenation was started from the dashed lines.

The first endothermic peak = the reduction in the stoichiometry of the δ hydride.
The second endothermic peak = the decomposition of the hydride to form β and α phases by progressive heating.

2. High Temperature XRD Observations of TiH₂ Phase Changes at Different Temperatures

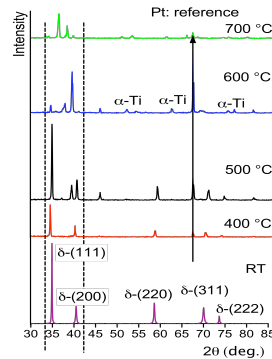
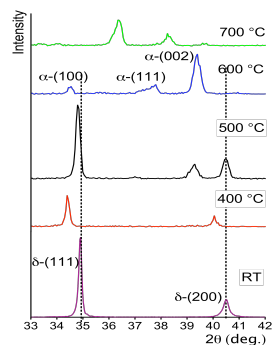


Figure 3: XRD patterns for TiH₂ samples (Japanese) pre-heated in Argon to different temperatures. The window of details measurement on phase changes is marked with dashed lines as below.



Pre-heat treating above 500 °C, some of the peaks is broadened and shifted, indicating hydrogen loss.

Pre-heating at 600 °C, α-Ti become noticeable and little hydride present.

At 700 °C, α-Ti is detected and no hydride present.

Table 1: Lattice Parameter of δ-(111) and δ-(200).

Peaks	d-(111) Lattice Parameter, a (nm)	d-(200) Lattice Parameter, a (nm)
As-received TiH ₂ at RT	0.1484 ± 0.0002	0.1286 ± 0.0003
TiH _{2-x} at 400°C	0.1503 ± 0.0003	0.1299 ± 0.0002
TiH _{2-y} at 500°C	0.1482 ± 0.0007	0.1281 ± 0.0006

The hydrogen concentration increases linearly with lattice parameter

The lattice parameter of the powders increased due to the heat expansion as the temperature increased, then it decreased because of the hydrogen released

3. TiH₂ Dehydrogenation

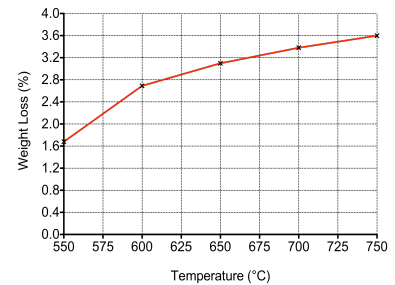


Figure 4: Weight Loss vs. Temperature after TiH₂ dehydrogenation

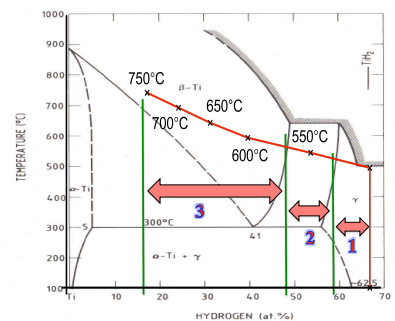


Figure 5: Ti-H Phase Diagram combined with the isothermal Dehydrogenation heated in Argon at different Temperatures by constant Heating Rate of 10 °C/min

Conclusion

- 2 endothermic peaks were identified in TG curve for both hydride powders, accompanied by a stepped weight loss which are due to the reduction in hydrogen and activation energy.
- Lattice Parameter is increased due to the thermal expansion effect. Lattice Parameter decreased when the H₂ concentration decreased.
- In the TiH₂ dehydrogenation results, the δ TiH₂ disappeared with increasing temperature and the β_H phase and the α_H phase gradually appeared.

References

- H.R.Z. Sandim, B.V. Morante and P.A. Suzuki, Materials Research, Vol 8, No 3 (2005) 293.
- V. Bhosle, E.G. Baburaj, M. Miranova and K. Salama, Materials and Engineering A356 (2003) 190.