Determination of the weight loss during dehydrogenation of TiH_2 or ZrH_2 powder

Basic principle

When heated under high vacuum, titanium hydride or zirconium hydride will quantitatively decompose into their respective components. The resulting weight loss of a weighed sample can be expressed as a hydrogen content.

Test procedure

The dehydrogenation is preceded by a drying step. The sample is placed in a drying cabinet and kept at a fixed temperature until its weight is constant.

1 to 1.5 g of the sample is weighed and pelletized with the aid of a hydraulic press (50 kN). This pellet is put into a quartz boat of known weight (precision ± 0.1 mg) which was pretreated by calcination at 1200 °C and cooling down in an exsiccator.

Quartz boat plus sample are weighed again and inserted into a silica tubing which is sealed at one end and equipped with a standard ground joint at the other (length approx. 300 mm, inner diameter 19 mm). The thoroughly greased ground joint is connected to the inlet of a pumping unit. After building up a vacuum of $< 10^{-4}$ mbar, a tubular furnace is positioned around the silica tubing. The furnace is closed and heated to 1200 °C. The heating-up is accompanied by a temporary pressure increase at 500 °C and higher, which is caused by a release of hydrogen.

If the pressure drops below $< 10^{-5}$ mbar, the temperature is kept at 1200 °C for another hour before switching off the oven. The oven is cooled down to < 50 °C, the pumps are subsequently switched off and the pressure within the quartz apparatus is compensated with argon. Once room temperature is reached, the quartz boat is taken out of the tube and its weight is immediately determined.

Determination of the weight loss during dehydrogenation of TiH_2 or ZrH_2 powder

Calculation

The hydrogen content is calculated according to the following equation:

$$\frac{(b-c)\cdot 100}{(b-a)} = \%\,\text{hydrogen}$$

a: empty weight of the calcined quartz boat

b: weight of quartz boat plus sample

c: weight of quartz boat plus residue after heating

The precision of this measuring method according to plausibility studies is ± 2 % regarding the amount of hydrogen released.

Edition: May 2001

Chemetall GmbH

Special Metals Division

Trakehner Straße 3
D-60487 Frankfurt am Main
P.O. Box 90 01 70
D-60441 Frankfurt a.M.
Phone +49 69 7165-3911
Fax +49 69 7165-2523
Mail specialmetals@chemetall.com