

Decomposition behaviour of as-received and oxidized TiH_2 powder

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Metallic foams are excellent engineering materials offering high energy absorbing capacity, reduced thermal and electrical conductivity, as well as enhanced mechanical and acoustic damping. This material combines properties of cellular materials together with those of metals. The high stiffness-to-mass ratio leads to a variety of applications especially in automotive industry. (See also news report on page 15.)

One very promising and proved way to produce metal foams is the powder metallurgy route [1]. A precursor material tablet is produced by compacting a powder mixture including small amounts of blowing agent powder. Heating of this material causes gas release by decomposition of the embedded blowing agent and thus expansion of the material.

The overall aim is to produce a stabilized blowing agent compatible with the foaming process [2]. The alloy composition and the type of the blowing agent have to be chosen such that the blowing agent gives rise to an ideal foaming behavior with formation of a homogeneous pore size.

Up to now titanium hydride (TiH_2) turned out to be the best foaming agent to reach homogene-

ous pore formation and pore size distribution. As there is a mismatch between the melting point of commercial aluminum alloys and the decomposition temperature of TiH_2 (400°C), the latter should be subjected to thermal and/or oxidizing treatments.

By this an oxide layer is formed on the surface of the titanium hydride powder particles. This layer delays gas release, from the particles, so that during heating up of the powder mixture the blowing first takes place when the melting temperature of the alloy is reached. The thickness and composition of the layer depends of the used temperature and time period of oxidation (Fig. 1).

By the present investigation the influence of various pre-treatments in air on the release of hydrogen from TiH_2 is examined. For this purpose the decomposition of TiH_2 powders is examined by differential scanning calorimetry (DSC) [3], thermogravimetric analysis (TGA) [1,4,5] and mass spectrometry (MS, Fig. 2). The morphology of the powder was obtained by scanning electron microscopy (SEM).

Cold pressed TiH_2 powder pre-heated in air at 520°C for 180 minutes was studied in situ during the heating in air. Synchrotron radiation experiments were performed at the KMC2 beamline at BESSY the synchrotron radiation source in Berlin. Powder diffractometry was performed using an area sensitive detector, which allowed the acquisition of the whole range of the diffraction pattern simultaneously. The sample was mounted on in the focus of a radiation heater (Fig. 3). The temperature of the sample was controlled using two thermocouples mounted on the sample stage. In the preliminary experiment the temperature was changed from room temperature up to 700°C with the constant rate of 5 K per minute.

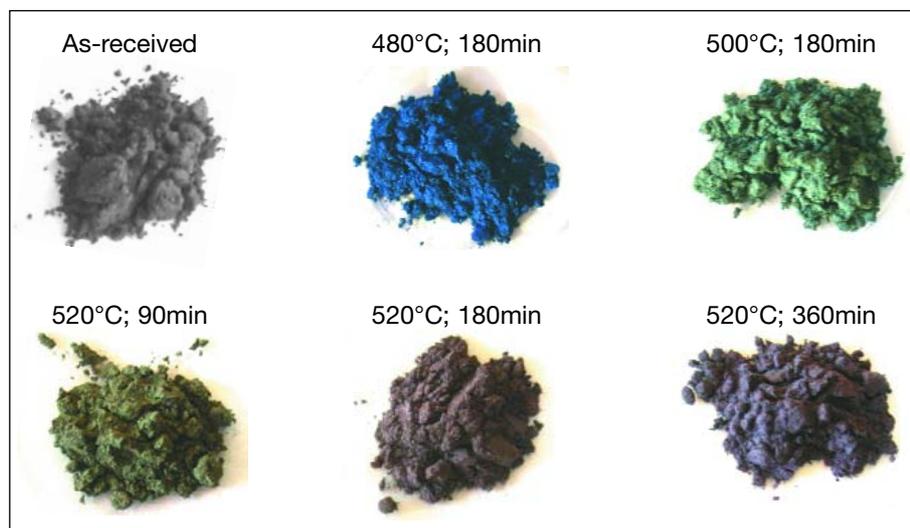


Fig. 1: TiH_2 oxidized at different conditions. The colour of the powder depends on the thickness of the oxygen layer.

The area sensitive detector was covering the angles between 34° and 45° , so it was possible to acquire the most important Bragg reflexes of the different compounds assumed in sample (Fig. 4). Data evaluation showed that we were able to determine the content of hydrogen in the sample and to follow the evolution of the Ti/H ratio using a phase diagram.

We identified the same titanium oxides, which were measured after the pre-treatment on a laboratory X-ray source (see Fig. 4). After the initial change of the TiO_2 intensity, the amount of oxides stayed constant, while the concentration of hydrogen increased considerably. The structure of titanium-hydride changed at higher temperatures to bcc β -phase because of the loss of hydrogen. After the cooling the sample consisted of a mixture of cph α -Ti and fcc δ - $\text{TiH}_{1.5}$. Advantage of the in-situ measurements is the possibility to monitor exactly the phase transition, using only one sample for the whole range of temperatures.

These preliminary in situ X-ray diffraction experiments at BESSY are combined with transmission electron microscopy (TEM) to get some additional information about the structure of the oxide layer covering the hydride particles.

In the future we plan to perform the in-situ diffraction experiment during the pre-treatment of the blowing agent at different temperatures and annealing time, as well as the decomposition of the hydride and in the inert atmosphere.

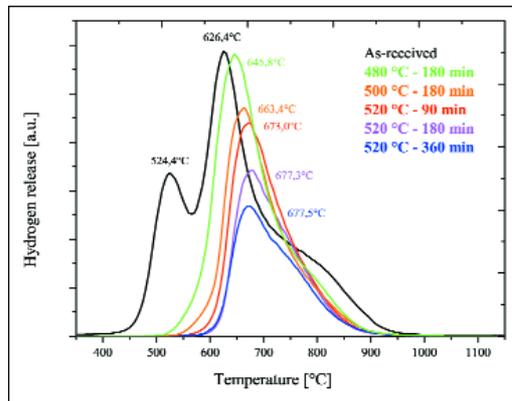


Fig. 2: Release of hydrogen as a function of heat treatment. The decomposition of the TiH_2 is impeded due to the slow diffusion of the hydrogen through the oxide layer.



Fig. 3: The sample was mounted on a ceramic holder and irradiated with a halogen lamp. The incoming X-rays entered the figure from the right-hand side. Scattered photons were reflected through the metal cone on the left side to the detector.

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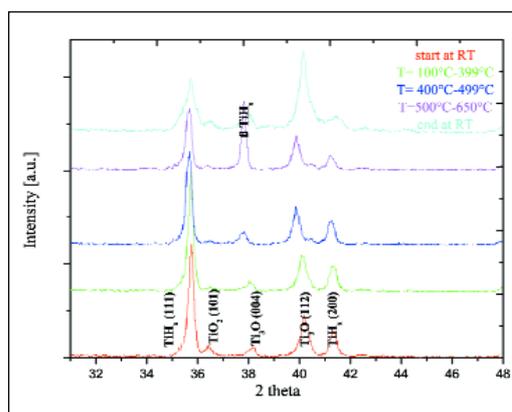


Fig. 4: Alterations of the peak intensities during heating and cooling of a pre-heated powder.