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Effect of the TiH_2 pre-treatment on the energy absorption ability of 6061 aluminium alloy foam

Ľubomír Orovčík [a,c,*], Martin Nosko [a], Peter Švec Sr. [b,c], Štefan Nagy [a,c], Miroslav Čavojský [a], František Simančík [a], Jaroslav Jerz [a]

[a] Institute of Materials & Machine Mechanics, Slovak Academy of Sciences, Račianska 75, 83102 Bratislava 3, Slovakia
[b] Institute of Physics, Slovak Academy of Sciences, Dúbravská cesta 9, 81404 Bratislava 4, Slovakia
[c] Faculty of Materials Science and Technology STU in Trnava, Paulínska 16, Slovakia

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ABSTRACT

Expansion kinetics of aluminium foams is generally driven by foaming agent. Therefore, a proper type and pre-treatment of the foaming is important for improvement of its inner structure, mechanical properties and energy absorption ability. This paper is focused to reveal an effect of long-term oxidation of TiH_2 on foaming kinetics (expansion profile) and structure of heat-treatable 6061 alloy foam. Subsequently, a uniaxial compression test was performed to study the effect of structural changes due to pre-treatment of TiH_2 on the mechanical properties and absorption ability. Structural transformation of TiH_2 was also studied with respect to annealing conditions.

1. Introduction

Mechanical properties of aluminium foams can be modified by heat treatment of heat-treatable aluminium alloys [1–5], improvement of its inner structure by placement of foamable precursor [6], amount of TiH_2 for different densities [7–10] or by pre-treatment of TiH_2, which has received little attention.

Aluminium foams made through powder metallurgy route (PM) [2,4,6] possess more regular pore sizes if H_2 is released from TiH_2 during melting of the alloy [11–15] which leads to formation of rather-homogeneous inner pore structure [6,10–12]. Pre-treatment of TiH_2 is responsible for formation of the oxides surface layer, which delays decomposition of H_2 from TiH_2 at higher temperatures. This fact is responsible for elimination of the complex endothermic double peak on DTA curve between 500 and 550 °C [11,12]. The same effect can be achieved by long-term oxidation at 450 °C/120 min [13]. Pre-treatment of TiH_2 depends on used alloy, for partial improvement of inner structure, in case of AlSi10 it is 450 °C/15 min [10], but its effect on the mechanical properties has not been studied yet. A large amount of early-released H_2 leads to pore coalescence during foaming [11,12,14,16,17]. For improvement of the mechanical properties, the temperature of H_2 release has to be moved to semi-solid state of the alloys due to partial H_2 releasing, which has resulted into formation of the more homogeneous structure [11,13–15].

This work is therefore focused to investigate the long-term oxidation of TiH_2 on mechanical properties and inner structure of the most commonly used heat-treatable alloy AA6061. Pre-treatment of TiH_2 at 500 °C/120 min was chosen to remove more amount of H_2 and to create more surface oxide layer, which delayed H_2 release up to temperature, which could lead to significant improvement of inner foam structure thus absorbed deformation energy during impact. Structural transformation of TiH_2 was also studied with respect to annealing conditions.

2. Experimental

TiH_2 was supplied by Chemetall GmbH Frankfurt (purity 98.8%, particle size 99.9% – 325 mash, nominally ≤ 45 μm, average particle size $d = 5\ \mu m \pm 1\ \mu m$). Pre-oxidized TiH_2 powder was prepared by heating of 5 g of as-received powder in an alumina crucible inside a resistant furnace at temperature 400 °C, 450 °C and 500 °C and holding time 15, 30, 60 and 120 °C for each temperature. TiH_2 was analysed by X-ray diffraction (XRD) using a Bruker AXS D4 Endeavor diffractometer with Bragg–Brentano geometry and Cu Kα radiation at $\lambda = 0.15406$ nm. The amount of O_2 in surface layer was analysed by JEOL JSM 7600F by the WDS, parameters were set to 30 kV after thorough simulation of the reaction between electron beam and TiH_2 via Monte Carlo. Dynamic light scattering (DLS) was used for determined particle size of the as-received TiH_2 before and after pre-treatment. The thermodynamic stability was monitored by high-temperature differential thermal analyser (DTA) in the mode of DSC using Perkin-Elmer DTA7 in Ar atmosphere at a purge rate of

* Corresponding author. Tel./fax: + 421 259309404.
E-mail address: lubomir.orovcik@savba.sk (Ľ. Orovčík).

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30 ml min^{-1}, from 25 °C to 850 °C, with a heating rate of 10 K/min. Foamable precursor was prepared by conventional PM route with chemical composition correspond to 6061 alloy with 0.8 wt% of TiH_2.

The uniaxial compression test was performed for both non-treated and pre-treated TiH_2 at 500 °C/120 min, at least 4 experiments were done for each treatment conditions. To assure reproducibility of the measured data [1,6,18,19], the foamable precursor was placed into mould identically for all experiments. Foamed samples of diameter 30 mm and height 45 mm with density (~0.6 g/cm^3) were prepared for subsequent testing resistance furnace preheated to 750 °C. According to previous measurement [10–12], density of the foam was controlled via thermocouple mounted on the mould cover [6]. Subsequently, heat treatment of the foam itself was performed at annealing temperature 530 °C/30 min and at ageing temperature 170 °C/12 h according to convention 6061 alloys.

To reveal 3-D porous structure, Nanotom 180 GE was used. Compression strength of the foam was estimated via uniaxial compression tests on a Zwick device with maximum testing force 100 kN at the strain rate 0.033 s^{-1} according to DIN 50134 [18–19]. Energy absorption ability was calculated from the region under the stress–strain curve and the longest plateau was determined as last stress drop in densification part area according to DIN 50134 [18–19].

3. Results and discussion

Pre-treatment of TiH_2 leads to its surface oxidation, which is more pronounced with increasing temperature and time of annealing. The long-term oxidation of TiH_2 at 500 °C/120 min. leads to significant pre-oxidation of surface layer (Fig. 1a) and to formation of titanium oxides such as TiO, TiO_2, Ti_3O (Fig. 1b). The particle size d_{50} of as-received TiH_2 increases from 15.05 μm to 18.11 μm for pre-treatment 500 °C/120 min. We therefore assume that pre-treatments of the TiH_2 are responsible for the change of the fcc crystal lattice $TiH_{(1.971,\ 1.924)} \rightarrow TiHx\ (s) + H_2(g)$ reaction, $1.5 < x < 2$ and for the change of unit-cell parameters, which were calculated from different reflections and Rietvelt-refined. The corresponding evolution of unit-cell parameters was thus determined for as-received fcc–$TiH_{1.924}$ in the range between $a = (0.44496 \pm 0.00045)$ nm and $a = (0.44319 \pm 0.0022)$ nm for annealing at 450 °C/60 min, after which no presence of this phase was detected. Transformation of crystal lattice from cubic to tetragonal occurs during annealing at 400 °C/15 min and higher is in progress until 450 °C/120 min (Fig. 1c). Unit-cell parameters changed from $a = (0.44792 \pm 0.0008)$ nm and $a = (0.43817 \pm 0.0008)$ nm to 0.44509 nm and 0.43766 nm at 450 °C/120 min. After long-term oxidation at higher temperatures (450 °C/120 min and at 500 °C), formation of $TiH_{1.5}$ with lattice parameter a ranging from 0.44264 nm to 0.44087 nm is observed. The evolution of the unit-cell volume of the Ti–H phases with the temperature and time of annealing can be seen on Fig. 1c. An intense dehydrogenization started at 570 °C (Fig. 1d) for pre-treated TiH_2, and this fact therefore could result in the formation of the more homogeneous inner structure with thicker cell-walls and decreased amount of defects within structure. At 500 °C, there is observed significant damage of TiH_2 surface, which results in the formation of Ti_3O phase within damaged areas and consequently, oxidation of the TiH_2 surface

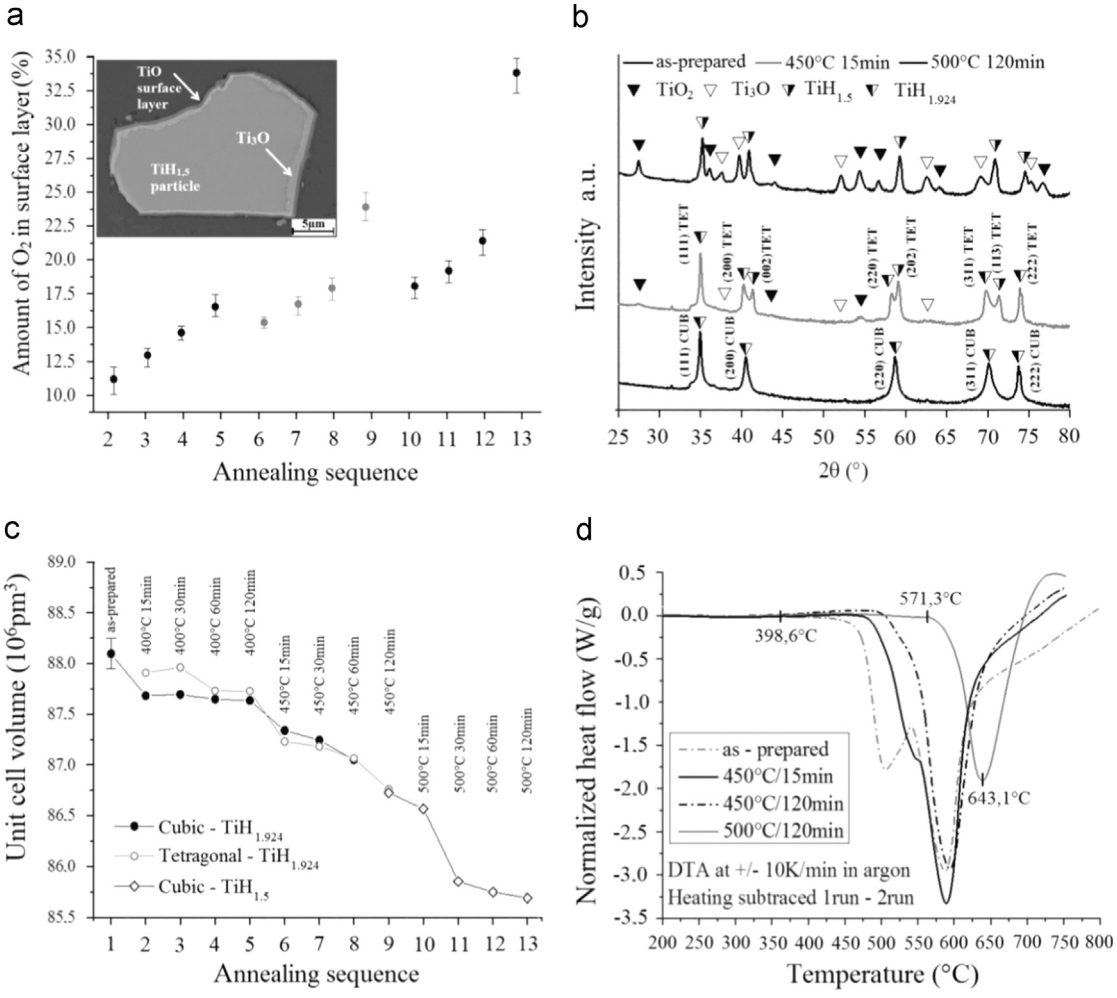

Fig. 1. (a) Increasing of oxides on surface and TiH_2 particles after 500 °C/120 min annealing, (b) RTG analyses of pre-treated TiH_2, (c) changes of unit cell volume, and (d) DSC curves showing decomposition of TiH_2.