

The Influence of Titanium Hydride Pretreatment on the Compressive Properties of Aluminum Foam

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Macrostructure has an important effect on the compressive properties of closed-cell aluminum foams. Meanwhile, the decomposition behavior of a foaming agent has a significant influence on the macrostructure of closed-cell aluminum foams. In order to get optimal compressive properties on aluminum foams, it is important to obtain the optimal decomposition behavior of a foaming agent. In this paper, different heat treatment temperatures and fixed heat treatment were employed to investigate the decomposition behavior of titanium hydride. For a more intuitive understanding of their decomposition characteristics of the pretreated titanium hydrides, closed-cell commercially pure Al foams were prepared by melt foaming method using different types of pretreated titanium hydrides as foaming agent. In addition, the macrostructures and quasi-static compressive properties were used to evaluate the pretreatment effect. The results showed that pretreatments have a significant influence on the macrostructure and compressive properties of aluminum foams. The decomposition characteristics of titanium hydride pretreated at 753 K for 30 min are most suitable for the preparation of closed-cell aluminum foams under present conditions, as the foams possess good combination of pore size distribution, yield strength and energy absorption capacity.

Keywords: foaming agent; pretreatment; aluminum foam; compressive property.

1. INTRODUCTION

Metal foams possess high specific stiffness, high specific strength and good energy absorption properties, which makes metal foam a potential material to absorb the impact energy during a crash of a vehicle. In the past decades, there were a considerable number of investigations on their mechanical characteristics (particularly for aluminum foams) and the results indicated that the cell structures had a significant influence on the yield strength, plateau stress and energy absorption characteristics [1]. Some researchers have investigated the macrostructure and quasi-static compressive behaviors of different types of foams [2–4], and the results showed that foams with relatively smaller average pore size possessed better deformation stability. In addition, macrostructure of aluminum foam is closely related to the foaming agent decomposition behavior [5–9]. Song et al. [5] and Yang et al. [10] investigated the optimal content of TiH₂ through the evolution of aluminum foams macrostructures. Though TiH₂ possessed strong hydrogen release ability and accepted as the best foaming agent for aluminum foams, the further treatment needed to be done to improve the uniformity of aluminum foam structures [11]. Accordingly, more and more researchers have pay attention to the heat treatment of titanium hydride. Malachevsky et al. [12] investigated the thermal evolution of titanium hydride to optimize the fabrication process of aluminum foam and results showed that the temperature of oxidation treatment had an important influence on the hydrogen release behavior. Meanwhile,

Kennedy et al. [13] reported that hydrogen gas normally released at approximately 763 K for untreated TiH₂ and heat treatment in air delayed hydrogen evolution to higher temperatures. Matijasevic et al. [11] studied the improvement of aluminum foam technology by tailoring of foaming agent. Results showed that the expansion potential of the foams and the uniformity of cell size distribution were improved. Meanwhile, the individual cell walls were smoother and less corrugated when the foams were blown with pretreated TiH₂. Though, some people investigated the solidification behavior of aluminum foams, using pretreatment TiH₂ as foaming agent [14–16]. The researches mainly focused on the effect of heat treatment on the shift of TiH₂ decomposition temperature. However, few people pay attention to the influence of pretreated titanium hydride on the compressive properties of closed-cell aluminum foams.

In this paper, different pretreatment temperatures were applied on titanium hydride powders to investigate the effect of oxidation on the decomposition behavior of titanium hydride. The macrostructures and quasi-static compressive properties were used to evaluate the pretreatment effect. The optimal heat treatment temperature was obtained and the reasons were discussed.

2. EXPERIMENTAL

2.1. Preparation procedures

Titanium hydride particles were pretreated in resistance furnace at 673, 713, 753, 793 and 833 K for 30 min, respectively. When being oxidized treatment for 15 minutes, titanium hydride powders were stirred so that they could be

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heated homogeneously. Aluminum foams were fabricated by melt foaming method in this experiment. The matrix material was commercially pure aluminum (with the purity of ~99.9%), calcium granules (commercially pure, granularity between 1 mm~2.5 mm) were used to adjust the viscosity of aluminum melt. Titanium hydride powders (commercially pure, 300 ±20 mesh) were used as foaming agent. The preparation processing mainly includes the following stages [17]: (1) melting certain quality of commercially pure aluminum (~1 kg) in a low carbon steel crucible to a fixed temperature; (2) adding certain quality of Ca granules (2.5 wt%) to the melt accompanied by stirring, with the stirring speed of 450 rpm and stirring time of 10 min. The impellor was driven by an electromotor and the stirring speed was controllable [18]; (3) adding 1.5 wt% of TiH₂ (under original or heat treated at 673, 713, 753, 793, 833 K for 30 min) to the melt accompanied by stirring with the speed of 1000 rpm for 10 min and holding for 120 s; (4) cooling the flux in the air after it was foamed. During the whole procedures the temperature of the melt was maintained at 973 K ±5 K except for the last step.

The morphology of TiH₂ powders was obtained by a Hitachi S4800 scanning electron microscope (SEM) equipped with energy dispersive X-ray spectrometer (EDX). Differential thermal analysis (DTA) was used to analyze the thermal decomposition temperatures of the original or heat treated TiH₂ powders. The instrument type is SDT Q600 (V20.9 Build 20) Simultaneous DSC-TGA. The differential thermal analysis (DTA) was monitored from room temperature to 1173 K in argon atmosphere with the heating rate of 10 K/min.

2.2. Characterization

The specimens were machined by electro-discharging machine into the size of (35 × 35 × 35) mm (length × width × thickness). The samples were finally ground using 2000 grit emery paper. Porosity was calculated by weight and dimension of sample. Weight was measured by electronic balance with the accuracy of 0.0001 g. Caliper was used to measure the accurate dimensions of the foams. Pore size distribution of the samples in the cross-section was determined by software.

Uniaxial compression tests were performed by using SUNS Electron Universal Material Testing Machine, with a maximum load of 300 kN. All tests were performed under displacement control, with a displacement rate of 2.0 mm/min (with the initial strain rate of 0.001/s) at room temperature. Vaseline was used to minimize the friction between the specimen and the plates. Load and displacement were recorded automatically by a computer, stress σ is defined as the load (kN) on specimen divided by specimen cross area; strain ϵ is defined as the displacement (mm) of specimen divided by specimen height. For each test parameters, three specimens were compressed and the average data were used.

3. RESULTS AND DISCUSSION

3.1. Structural features

Fig. 1, a, shows optical cross sections of aluminum foams (with the porosity about 80 %) fabricated by using

the original and pretreated titanium hydrides as foaming agent. It can be seen that the pores are isolated and closed-cell. The pore size and pore number are two important structural parameters which can be used to reflect the pore structure of aluminum foam [19]. From Fig. 1, b, the distribution ranges of pore diameters of aluminum foams are about 0.5–5.5 and 0.5–3.5, 0.5–4.5, 0.5–3.5, 0.5–5, 0.5–4.5 mm, using original and pretreated titanium hydrides (with the pretreated temperatures of 673, 713, 753, 793, 833 K) as foaming agent, respectively. In addition, the percentages (number) of pore size (0.5 mm~2.5 mm) are 55.07 % and 89.33 %, 92.73 %, 97.64 %, 92.00 %, 65.22 %, with the original and pretreated titanium hydrides (with the pretreated temperatures of 673, 713, 753, 793, 833 K) as foaming agent, respectively. That is to say, the uniformity of pore size distribution is increased first and then decreased with pretreated temperature increasing. In addition, almost all of the pore size concentrate on 0.5 mm~2.5 mm, using the titanium hydride (pretreated at 753 K) as foaming agent. However, only about half of total distribute in the range of 0.5 mm~2.5 mm, using the original titanium hydride as foaming agent.

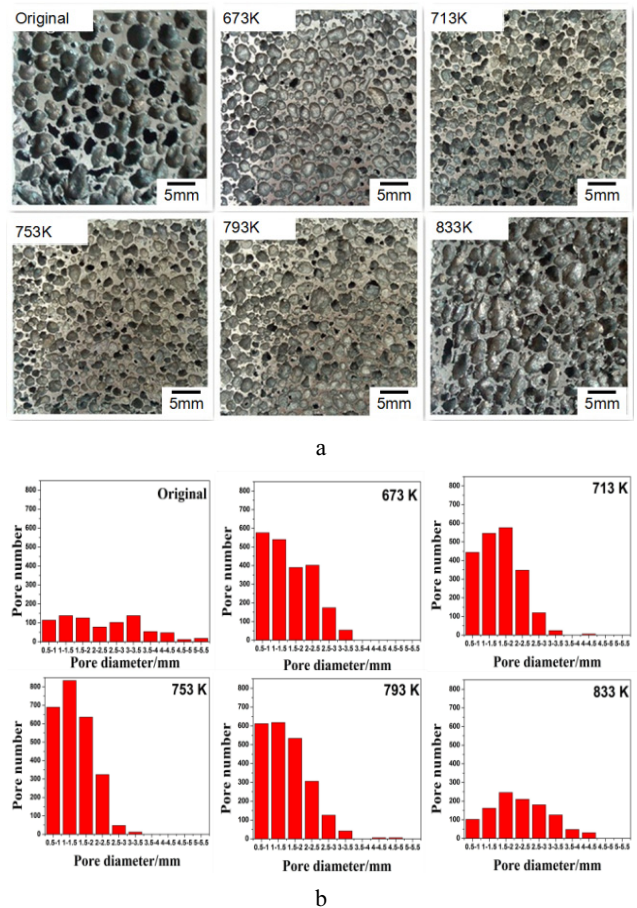


Fig. 1. Optical cross sections (a) of aluminum foams using TiH₂ as foaming agent; the distribution of pore diameters (b)

Bubbles formed in the molten aluminum when TiH₂ decompose to hydrogen gas. Both the hydrogen quantity and decomposition behavior of TiH₂ directly affect the foams macrostructures, especially for pore size distribution. Differential thermal analysis (DTA) was applied to study the decomposition characteristic of titanium hydride

powder. The weights of the original and pretreated TiH₂ (with the pretreated temperatures of 673, 713, 753, 793, 833 K) are 9.325, 7.321, 12.496, 6.436, 11.964 and 5.675 mg, respectively. Meanwhile, heat absorption is related to the weight of the test material. Thus, maximum heat absorption can be observed for TiH₂ pretreated at 713 K in Fig. 2. As shown in Fig. 2, the original and pretreated TiH₂ at 673 K and 713 K have similar decomposition temperatures. It is interesting that the decomposition temperatures of the TiH₂ pretreated at 753, 793 and 833 K are successively delayed. In other words, pretreatment increases the powders decomposition temperatures.

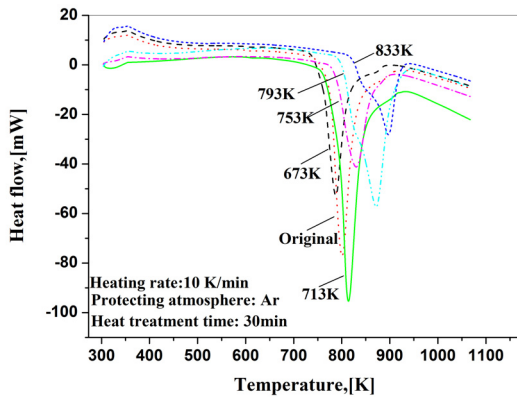


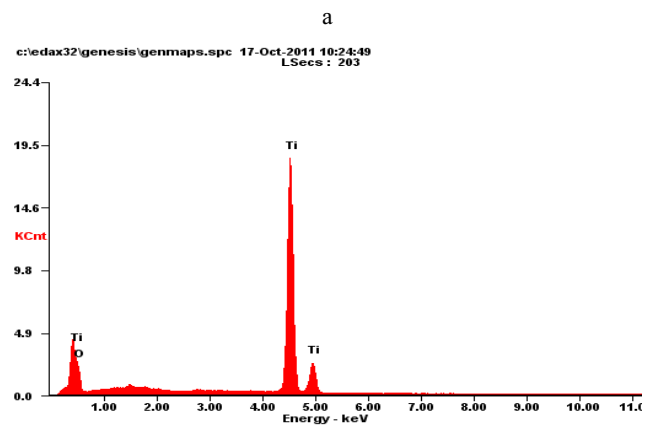
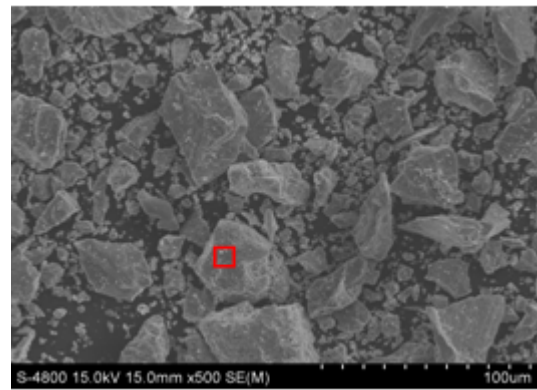
Fig. 2. The DTA curves of original and pretreated titanium hydride

Fig. 3, a, and b show the morphology and the EDX results of TiH₂ powders (pretreated at 753 K for 30 min). As it is known, titanium element has a great affinity to oxygen element and heat treatment can make surface layer TiH₂ decomposed to Ti and H₂, Ti will attach to the un-decomposed TiH₂. Hence, under air atmosphere condition a new compact of titanium oxide film with strong adhesion will form on the surface of un-treated TiH₂ powders as the heat treatment process developing. As a result the hydrogen gas subsequently generated needs more time to pass through the compact oxide layer. While for the un-treated powders hydrogen gas will liberate directly. Meanwhile, as the heating rate is fixed to 10 K/min in the present experiment and the thickness of oxide layer maybe different from different pretreatment temperatures, leading to the differences of the decomposition temperature as shown in Fig. 2. In other words, the delay time maybe changed with different pretreatment temperatures. In addition, the dispersible uniformity of TiH₂ is limited by the available disperse time. As described above, pretreatment increases the available disperse time of the powders. Thus, the uniformity of the powders can be improved, resulting in the homogeneity of the pores in the aluminum foam. The powders pretreated at 753 K may possess the optimal combination of titanium oxide film and the content of un-treated TiH₂, leading to the more concentrated pore size distribution as described above.

3.2. Compressive stress-strain curves

Typical quasi-static engineering stress-strain curves of different types of aluminum foams are shown in

Fig. 4, a. The compressive deformation process exhibits three universal deformation characteristics: an initial linear-elastic region where stress increases linearly with strain increasing due to the elastic bending of cell wall; an extended plateau region where the stress is almost independent of strain as the cells deform plastically; finally a densification region in which the stress-strain curve rises steeply as collapsed cells are compacted together. These deformation characteristics are similar to those of other metal foams [20–24]. It should be noted that pretreatment has an important effect on the compressive properties of the foams. Aluminum foams fabricated using the original titanium hydride has the lowest plateau strength (defined as the average value of the stress between the strain of 0.1~0.5). The plateau strengths of the aluminum foams fabricated using the pretreated titanium hydride at 673 K and 833 K are slightly increased compared with the original. It is also clear that the stresses of aluminum foam using pretreated titanium hydride at 731, 793 and 753 K are further increased successively. Specimens using pretreated TiH₂ at 753 K possess the optimal plateau strength compared with others under the present conditions.



Element	wt%	at%
OK	17.47	38.79
TiK	82.53	61.21
Matrix	Correction	ZAF

Fig. 3. SEM (a) and EDX (b) results of TiH₂ powders after oxidation at 753 K for 30 min

For metallic foams, the first peak stress on the stress-strain curve is defined as yield strength [25, 26]. As can be seen from Fig. 4 the yield strengths of the foams

using original and pretreated titanium hydride at 673, 713, 753, 793, 833 K as foaming agent are about 1.9 and 2.1, 2.7, 3.8, 3.0, 2.1 MPa, respectively. It is obvious that the yield strength increases first and then decreases with the pretreatment temperatures increasing. The yield strength of the foam using pretreated TiH₂ at 753 K for 30 min is about 3.8 MPa, which is about two times of the original one.

It is known that as the cell size decreasing, the cell number would increase to keep the porosity, leading to the increased cell wall surface and new cell walls would form [2]. The cell shape was mainly decided by porosity: as the porosity increased, the spherical cells would contract each other then change from sphere to polyhedron [3]. In our study, the porosity is about 80 % for all samples, so we can infer the cell shape is mainly polygon with different edge numbers. In the Simone and Gibson model, cells were tetrakaidecahedrons and one of the sections was hexagonal [4]. It was indicated that most of the cell walls were bended rather than being pressed in the axial direction [2]. As the cell size decreasing, the cell wall length would decrease. The average compressive force also decreased as more cell walls shared the load. In addition, the moment upon the cell wall decreases as well [2]. Big pores exist in the foams with larger pore size distribution range and these big pores collapse more easily when they are compressed. While the foams with uniform and small pore sizes can withstand greater force.

In summary, Al foams using titanium hydride pretreated at 753 K for 30 min as foaming agent possess homogeneous pore size distribution and relatively small pore size, resulting in the good combination of compressive stresses and yield strength under the present conditions.

3.3. Energy absorption properties

In most cases, metal foams are used in energy absorption fields. Energy absorption capacity is an important aspect to evaluate the properties of metal foams. Namely, the energy absorption capability was area of under the stress-strain and it was calculated by [27]:

$$W = \int_0^{\varepsilon} \sigma d\varepsilon, \quad (1)$$

where W is the energy absorption capability and σ is the stress where the strain is ε . Fig. 4, b, shows the energy absorption capability of aluminum foams calculated according to Eq.(1), which indicates that all energy absorption capabilities increase with strain increasing. However, the amounts of energy absorbed in all stages are obviously different among the six groups of specimens. As shown in Fig. 4, b, the energy absorption capacity for the foams using original and pretreated TiH₂ at 673, 713, 753, 793, 833 K as foaming agent are 1.35 and 1.78, 2.30, 2.92, 2.59, 1.61 MJ/m³ respectively when the strain is 0.60. The aluminum foam fabricated by original TiH₂ possesses lower energy absorption capacity compared with others. The energy absorption capacity of Al foams increase first and then decrease with pretreatment temperatures increasing. Moreover, the energy absorption capacity of aluminum foam using TiH₂ pretreated at 753 K for 30 min is much higher among the six groups of specimens when

the strain is 0.60. That is due to the fact that the uniformity of the cell sizes is beneficial to eliminate the appearance of larger cell edges in the foams under fixed porosity. Meanwhile, the foams with uniform cell sizes possess better deformation stability when being compressed. Uniform cell sizes improve the strength of the foams by sharing the load [2]. In addition, energy absorption capacity displays the same variation trend with the uniformity of pore size distribution. While, pore size distribution of Al foams using titanium hydride pretreated at 753 K for 30 min as foaming agent is more homogeneous and the pore size is relatively smaller. Therefore, specimens prepared using TiH₂ pretreated at 753 K for 30 min as foaming agent possess better energy absorption capacity under the present conditions.

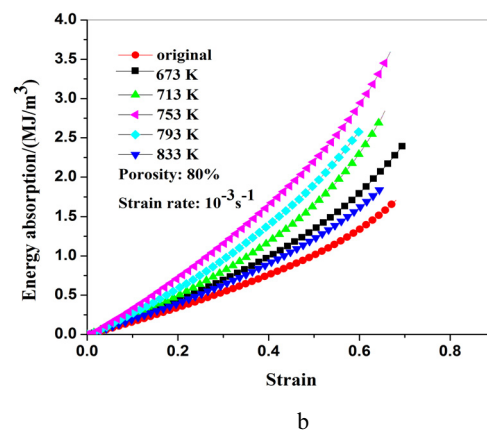
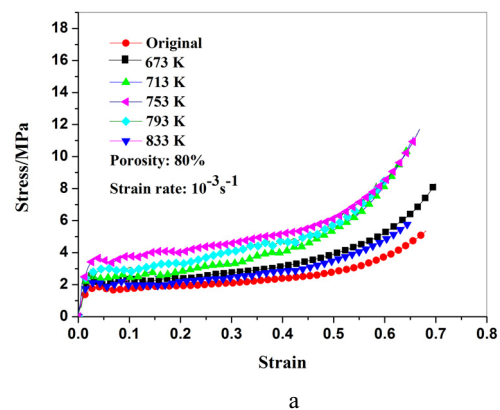


Fig. 4. Stress-strain curves (a) and energy absorption capabilities (b) of Al foams

4. CONCLUSIONS

Compared with the others foams, the aluminum foam prepared using TiH₂ pretreated at 753 K for 30 min as foaming agent possess more homogeneous pore size distribution in the cross section. When pretreated titanium hydrides were added to the molten aluminum, titanium hydride can be homogeneously dispersed as the effect of the titanium oxide film. The decomposition temperature of titanium hydride increases with the pretreatment temperatures increasing. However, excess high pretreatment temperature can result in decreasing of the final amount of hydrogen. In addition, the distribution of the pore sizes has a signification influence on the yield

strength and the energy absorption capability. Al foams prepared using TiH₂ pretreated at 753 K for 30 min as foaming agent possess good combination of pore size distribution, yield strength and energy absorption capacity under present conditions.

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