



Enabling the development of ductile powder metallurgy titanium alloys by a unique scavenger of oxygen and chlorine



Y.F. Yang ^{a,*}, S.F. Li ^a, M. Qian ^b, Q.S. Zhu ^a, C.Q. Hu ^a, Y. Shi ^c

^a State Key Laboratory of Multiphase Complex Systems, Institute of Process Engineering, Chinese Academy of Sciences, No.1 Zhongguancun North St, Beijing, 100190, China

^b RMIT University, School of Aerospace, Mechanical and Manufacturing Engineering, Centre for Additive Manufacturing, GPO Box 2476, Melbourne VIC 3001, Australia

^c State Key Laboratory of Advanced Processing and Recycling of Non-ferrous Metals, Lanzhou University of Technology, No.287 Langongping Road, Lanzhou 730050, China

ARTICLE INFO

Article history:

Received 20 October 2017

Received in revised form

22 May 2018

Accepted 11 June 2018

Keywords:

Powder metallurgy

Sintering

Rare earth alloys and compounds

Metal matrix composites

Metals and alloys

Diffusion

ABSTRACT

NdB₆ can effectively react with the surface titanium oxide films to form an initial NdBO₃ layer at about 670 °C, just prior to their dissolution. Two thirds of all the oxygen scavenged during sintering occurred in the α-Ti phase region through this reaction. NdBO₃ layer continues to grow by diffusion of oxygen until 1130 °C, beyond which NdBO₃ decomposes into Nd₂O₃ dispersoids and Nd₂O₃ further reacts with chlorine to form NdCl_xO_y. The unique oxygen-scavenging process prior to the active dissolution of oxide film makes NdB₆ more effective in scavenging oxygen than other rare earth hydrides, resulting in better tensile elongation. The addition of NdB₆ also leads to the improved sintered density, substantial microstructural refinement and dispersion strengthening by Nd-containing compounds and TiB. The optimal addition level of NdB₆ is 0.3 wt.%, beyond which large Nd₂O₃ aggregates and an increased number of TiB whiskers were formed. This is detrimental to the ductility.

© 2018 Elsevier B.V. All rights reserved.

1. Introduction

The oxygen (O) content has been one of the key issues that affect the powder metallurgy Ti business in terms of the cost of production and the resulting mechanical properties [1–6]. Reducing the detrimental effect of oxygen therefore plays a key role in enabling the development of low-cost high performance PM Ti alloys. The oxygen content of the sintered part strongly depends on the oxygen content of the raw Ti powder used, where the oxygen is mainly present in the form of TiO₂, Ti₂O₃ and TiO from the outermost surface to innermost layer in the surface oxide film on Ti powder [7]. These oxides start to dissolve into the underneath titanium matrix from about 670 °C [7], resulting in the largely reduced ductility.

Currently, in order to reduce the decline of ductility, rare-earth-containing (RE-containing) hydrides have been introduced to scavenge the oxygen from the Ti solid solution during sintering

[8–17]. However, the significant scavenging by RE-containing hydrides occurs after the surface titanium oxide films have disappeared. The oxygen-scavenging process is controlled by the diffusion of oxygen [9,13]. Oxygen is therefore difficult to be fully scavenged, evidenced by the resulting oxygen-deficit RE oxides after even 120 min of scavenging at 1350 °C [13,14]. As a result, the improvement of the ductility is still limited. The practical solution is to develop new scavenger that ideally can consume the surface oxide film prior to its significant dissolution into Ti. The recent study showed that LaB₆ was able to scavenge oxygen before the disappearance of oxide film [7]. NdB₆ is another type of commercially available lower-cost RE-containing compounds (USD \$0.35/g) and has similar crystal structure to LaB₆. However, it remains unknown if the unique oxygen scavenging feature also happens on NdB₆.

This paper presents a detailed study of whether and how NdB₆ can scavenge O from Ti powder during sintering, particularly focusing on whether the surface oxide films can be consumed by NdB₆ before they dissolve into the titanium matrix. In addition, commercially pure Ti (CP-Ti), Ti-6Al-4V and Ti-10V-2Fe-3Al (weight percent throughout) were selected as the base alloy to

* Corresponding author.

E-mail address: yfyang@ipe.ac.cn (Y.F. Yang).

investigate the effect of NdB_6 addition on the sintering densification, microstructure and mechanical properties. In comparison of the effectiveness of NdB_6 in improving the mechanical properties, other RE hydrides were introduced as references. These results are important for capitalizing on the scavenging effect of NdB_6 for the fabrication of low cost high performance PM Ti alloys.

2. Experimental procedure

Hydride-dehydride (HDH) Ti powder (–250 mesh, 99.4% purity, 0.25 wt.% O) supplied by General Research Institute for Nonferrous Metals, China, and two master alloy powder products supplied by Baoji Jia Cheng Rare Metal Materials Co. Ltd., China 66.7V-13.3Fe-20Al (–325 mesh, 99.5% purity) and 40V-60Al (–325 mesh, 99.5% purity) were used. NdB_6 , LaH_2 and YH_2 powders (–500 mesh, purity 99.5%) used were supplied by Yaguang New Materials Co. Ltd., China. Elemental B supplied by the same company was also used in order to distinguish the separate role of B and Nd in the sintering densification.

The powder mixtures were blended in a Turbula mixer with 200 rpm for 60 min and then compacted in a floating die at a pressure of 600 MPa. The mixtures have a good chemical homogeneity. The green density obtained was around 78% of theoretical density. The theoretical density of all alloys was estimated using the mixing rule from the equation,

$$\rho = \frac{(m_{\text{Ti alloys}} + m_{\text{NdB}_6})}{\left(\frac{m_{\text{Ti alloys}}}{\rho_{\text{Ti alloys}}} + \frac{m_{\text{NdB}_6}}{\rho_{\text{NdB}_6}}\right)}$$

where $m_{\text{Ti alloys}}$ and m_{NdB_6} are the weight percentage of Ti alloys and NdB_6 , and ρ are their densities.

Sintering was conducted at 1350 °C for 120 min in a tube furnace under a vacuum of 10^{-3} – 10^{-2} Pa, with heating and cooling both at 4 °C/min. The sintered density was measured using the Archimedes method according to the ASTM standard B328. In order to investigate the scavenging pathway of oxygen, the interrupted sintering experiments of Ti-1.0% NdB_6 (weight percent throughout) and Ti-5.0% NdB_6 compacts were conducted in a high-sensitivity dilatometer system (Model Netzsch 402C, Germany) in flowing argon. Referring to the scavenging process of LaB_6 , the samples were heated to the characterized temperatures of 670 °C, 830 °C, 1130 °C, 1350 °C at 10 °C/min and then were cooled to 200 °C at 40 °C/min. Changing the heating rate from 4 °C/min to 10 °C/min had no noticeable effect on the sintering behaviour [18]. The interrupted Ti-1.0% NdB_6 sample was used for microstructure observation while the Ti-5.0% NdB_6 sample was used for phase analysis.

Samples cut from tensile bars were polished with 50 nm colloidal alumina and etched by Kroll's etchant for microstructure observation. In order to prevent the contamination, the interrupted samples were only polished without etching. The microstructure were examined using scanning electron microscopy (SEM, Model JEOL 6460L, Japan) equipped with energy-dispersive spectroscopy (EDS, Model JEOL, Japan). XRD (Rigaku D/max III, Japan, Cu K α target) was used to identify the phase composition. Pro plus 5.0 software (Media Cybernetics, Bethesda, MD, USA) was used to quantify the microstructure. For each as-sintered material, the average prior- β grain size was measured from 500 grains and the average length of the α -Ti laths in as-sintered Ti-6Al-4V and Ti-10V-2Fe-3Al was measured from 1000 α -Ti laths.

Tensile specimens (3 mm \times 4.5 mm cross-section and 15 mm gauge length) were machined from as-sintered bars and tested on an Instron mechanical tester (Model 5054, USA) with a crosshead speed 0.5 mm/min, where a video extensometer was used to

monitor the displacement. The fractographs of tensile samples were characterized by SEM.

3. Results

3.1. Phase identification

Fig. 1 shows the XRD results of CP-Ti-1.0% NdB_6 , Ti-6Al-4V-1.0% NdB_6 and Ti-10V-2Fe-3Al-1.0% NdB_6 after sintering at 1350 °C for 120 min. NdB_6 disappeared in each case. Apart from the α and β phases, Nd_2O_3 is detected in each as-sintered sample, indicative of scavenging of oxygen by NdB_6 .

Fig. 2 shows the as-sintered microstructures of CP-Ti and CP-Ti-0.3% NdB_6 after sintering at 1350 °C for 120 min. The bright particles observed in the as-sintered CP-Ti-0.3% NdB_6 were confirmed to be all Nd-containing particles by EDS. Two types of Nd-containing particles were confirmed by spot EDS analyses: binary neodymium oxides and Nd-Cl-O ternary compounds, see Fig. 2d, suggestive of scavenging of both O and Cl by NdB_6 . In addition, short TiB fibres (dark contrast) were observed in the as-sintered microstructure although it was absent in the XRD patterns. Both Nd-containing particles and TiB short fibres were also easily visible in the as-sintered microstructures of Ti-6Al-4V-0.3% NdB_6 and Ti-10V-2Fe-3Al-0.3% NdB_6 , as shown in Fig. 3.

3.2. Scavenging of O and Cl by NdB_6 : microstructural evolution

The interrupted sintering experiments at 670 °C, 830 °C, 1130 °C and 1350 °C were designed to clearly understand the scavenging process of O and Cl by NdB_6 . Fig. 4 shows the XRD patterns of these corresponding interrupted sintered Ti-5.0% NdB_6 samples. NdBO_3 was detected at 670 °C and its content increased with increasing the temperature to 1130 °C. No NdB_6 particles were detected at 1130 °C. Further increasing the temperature to 1350 °C led to the formation of Nd_2O_3 .

Fig. 5 shows the corresponding microstructure of the interrupted samples. Fig. 5a shows a NdB_6 particle in a green compact of

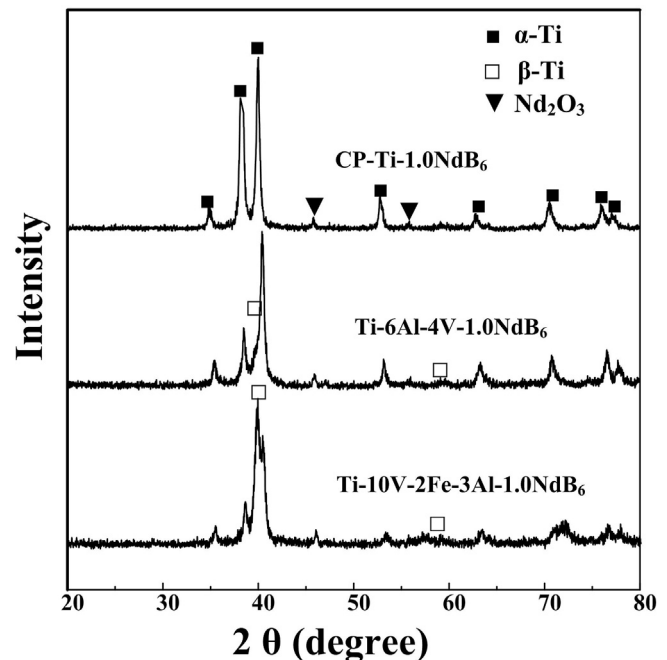


Fig. 1. XRD results of CP-Ti-1.0% NdB_6 , Ti-6Al-4V-1.0% NdB_6 and Ti-10V-2Fe-3Al-1.0% NdB_6 after sintering at 1350 °C for 120 min.

Download English Version:

<https://daneshyari.com/en/article/7990684>

Download Persian Version:

<https://daneshyari.com/article/7990684>

[Daneshyari.com](https://daneshyari.com)