Journal of Alloys and Compounds 769 (2018) 37–44

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

In situ formation of low interstitials Ti-TiC composites by gas-solid reaction

Ce Zhang, Zhimeng Guo, Fang Yang*, Haiying Wang, Yanru Shao, Boxin Lu

Institute for Advanced Materials& Technology, University of Science and Technology Beijing, Beijing 100083, China

ARTICLE INFO

Article history:
Received 5 June 2018
Received in revised form
26 July 2018
Accepted 30 July 2018
Available online 1 August 2018

Keywords:
Titanium matrix composites.
TiC
CH_4
Gas-solid reaction
Strengthening mechanism

ABSTRACT

In this work, in situ formation of low interstitials Ti-TiC composites with a high relative density has been fabricated by the combination of TiH_2-CH_4 reaction and vacuum pressure-less sintering. During the gas-solid reaction, TiH_2 reacted with CH_4 to form TiC particles on the powder surface. As a consequence, low interstitials contents (O < 0.21 wt%, N < 0.062 wt%, H < 0.011 wt%) were obtained in as-sintered materials. TiC particles uniformly distributed in the Ti matrix with a particle size of 3–12 μm, resulting in the grain refinement from 87.89 μm to 34.76 μm. The as-sintered Ti-35 vol%TiC exhibited high tensile strength of 644 MPa in YS and 736 MPa in UTS. Considering about ductility, the Ti-15 vol%TiC obtained comprehensive mechanical properties, about 628 MPa in YS, 715 MPa in UTS and 12.1% in elongation. The acceptable ductility (elongation > 10%) is important for following thermomechanical processing. In summary, the achievement of high performance Ti-TiC composites was attributed to the grain refinement, in-situ TiC strengthening, as well as a solid solution strengthening of the O, N and C elements in the Ti matrix.

1. Introduction

Titanium and its alloys have excellent properties, such as low density, high strength and fracture toughness, good corrosion resistance [1]. However, titanium use is far less widespread relative to other structural metals due to its high costs. One important lower-cost way is taking advantage of cheap elements (C, B, N, Si) in titanium matrix composites (TMCs) instead of expensive elements (V, Nb) in titanium alloys. In addition, TMCs reinforced by ceramic phases possess considerable potential for extensive application area like aerospace, war industry and civil industry because of their high specific strength, good specific modulus, preferable wear resistance, better mechanical strength and durability at high temperatures [2,3].

For TMCs, the reinforcement materials and the processing method are two important factors should be considered. In terms of TiC particles (TiC_p) reinforced TMCs, there are a few reinforcement materials can be used, such as C powder (graphite [4,5], carbon black [6]), innovative C nanomaterials (carbon nanotubes [7,8], graphene [9]) and C-containing compound (B_4C [10,11], VC [12], CrC [13]). Normally, the reinforcement material powders are usually as fine as possible to form in-situ fine TiC_p. As a drawback, the finer powder is easy to agglomerate due to its stronger van der Waals forces, especially the C nanomaterials. So, in order to achieve the uniform distribution of TiC_p, the blended powder must undergo long term ball-milling, which may increase the content of O and N, resulting in a detrimental effect on the ductility of TMCs.

As for processing method, TMCs can be better manufactured by the in situ technique due to their better wettability and stronger interface bonding between ceramic particles and the matrix compared to ex situ technique. And different in situ preparative techniques have been developed recently. Ingot metallurgy (IM) is most common in situ method. The Ti-TiC composites was prepared by vacuum melting utilized the reaction between Ti and C, which have low interstitials concentration (<0.2 wt%) [14–16] in general. But the as-cast Ti-TiC composites may have casting defects such as coarse microstructures, pores and the agglomeration of the reinforcing phase, leading to the elongation less than 10% once TiC volume fraction is over 10% [17–19]. The mechanical properties can be improved by subsequent hot working [16].

Powder metallurgy (PM) has long been regard as a promising method to produce in situ Ti-TiC TMCs, in which blend or pre-alloyed powder compacts are densified by hot pressing (HP) [20,21], spark plasma sintering (SPS) [22], vacuum sintering (VS)

* Corresponding author.
E-mail address: yangfang@ustb.edu.cn (F. Yang).

https://doi.org/10.1016/j.jallcom.2018.07.344
0925-8388/

[12,13,23,24] and laser melting deposition (LMD) [25,26]. PM TMCs can obtain a uniform distribution of reinforcement and homogeneous matrix microstructure compared with liquid phase solidification techniques. But the main drawback associated with PM is the difficulty in controlling the interstitials due to the impurity in powder and interstitials pick-up during powder handing process. The O content in PM TMCs is over 0.3 wt% in general, which leads a brittle Ti matrix [27] so that the elongation is less than 5% when TiC volume fraction is over 10% [20,22–24]. Different from IM TMCs, the following thermomechanical processing is difficult to improve due to the brittle matrix by high interstitials concentration.

Gas-solid reaction is a new low-cost way to produce TMCs. Kim has previously demonstrated the formation of TiC_p in sintered materials by pre-reaction between Ti compacts and CH_4 [28]. However, the previous study did not obtain a better comprehensive mechanical property especially ductility. They reported a high yield strength of 1100 MPa, but an extremely low elongation of <2% for Ti-6Al-4V-10% vol.%TiC [29]. Our previous research [30] has obtained a full density Ti-6Al-4V with low interstitials content and good ductility by sintering the hydrogenation dehydrogenization (HDH) Ti powder. Therefore, this investigation provided a new and simple method combined with gas-solid reaction and HDH process to fabricate low interstitials content Ti-TiC composites. In addition, the strengthening mechanism of the gas-solid reaction in TMCs had been systematically investigated.

2. Experimental procedure

2.1. Process outline

The starting materials were TiH_2 powder with a 180–830 μm particle size (−80/+20 mesh) and CH_4 with 99.99% purity. A schematic illustration of the experimental process is shown in Fig. 1, and the detailed description is given below:

I. Fine powder preparation: TiH_2 powder (180–830 μm) was crushed by vibratory milling under vacuum for 2 h. Then, fine powder with particle size below 20 μm was sieved under Ar atmosphere.
II. Gas-solid reaction and dehydrogenation: Put the TiH_2 powder (<20 μm) in a rotary furnace, which was evacuated to vacuum and back-filled with Ar to atmospheric pressure in advance, ensuring the powder away from being oxidized. Subsequently, the furnace was evacuated to vacuum again and then back-filled with CH_4. When the CH_4 pressure in the furnace was slightly higher than atmospheric pressure, open the gas outlet to obtain a stable CH_4 flow. Meanwhile, the furnace was heated to 600, 700, 800 and 900 °C for 30 min with a heating rate at 3 °C/min, respectively. After gas-solid reaction, the furnace was evacuated to vacuum once again and kept at 600 °C for 10 h to remove the residual hydrogen in the powder.
III. Powder compacting and densification: Green compacts with relative density 82.8 ± 0.6% were prepared by isostatic cool pressing (CIP). Lastly, the compacts were sintered at 900–1300 °C for 2 h with a heating rate 2 °C/min under vacuum of 10^{-3} Pa. It should be pointed out all the powder transfer and handing processes were in the sealed container or glovebox under Ar atmosphere.

2.2. Materials characterization

The interstitial O/N/H content in these as-sintered samples were measured with Eltra ONH-2000 apparatus. The test samples were about φ3*3 metal block, machining from five places in the depth range of 100–150 μm under the as-sintered materials surface. These metal blocks were loaded into nickel basket (The nickel capsule was used as for powder measurement) and put into the graphite crucible, then melted by pulse-heating under He atmosphere. The O content of the samples was measured by non-dispersive infrared analysis. The N/H contents were determined by the thermal conductivity method. The C content was measured with HORIBA EMIA-820 V, by high frequency combustion and infrared absorption method. The volume fraction of TiC in the

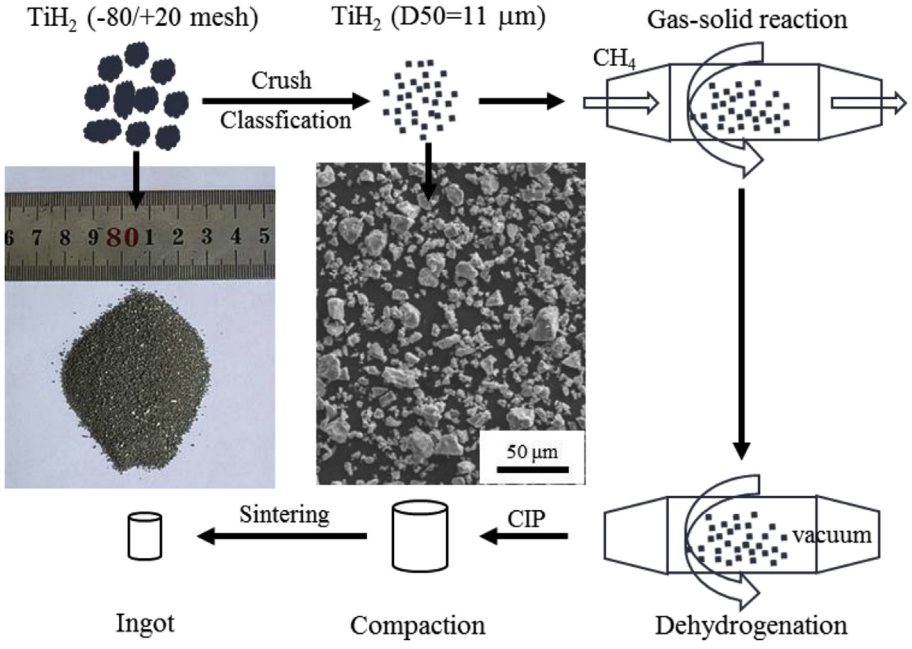

Fig. 1. A schematic illustration for the experimental process of low interstitials Ti-TiC composites by gas-solid reaction.