



# Low volume fraction in situ (Ti<sub>5</sub>Si<sub>3</sub> + Ti<sub>2</sub>C)/Ti hybrid composites with network microstructure fabricated by reaction hot pressing of Ti–SiC system



L.J. Huang<sup>a</sup>, S. Wang<sup>a</sup>, L. Geng<sup>a,\*</sup>, B. Kaveendran<sup>a</sup>, H.X. Peng<sup>a,b</sup>

<sup>a</sup>School of Materials Science and Engineering, Harbin Institute of Technology, P.O. Box 433, Harbin 150001, China

<sup>b</sup>Advanced Composites Centre for Innovation and Science (ACCIS), Bristol University, Bristol BS8 1TR, United Kingdom

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## ABSTRACT

In situ hybrid Ti<sub>5</sub>Si<sub>3</sub> rods and Ti<sub>2</sub>C particles reinforced pure titanium matrix composites ((Ti<sub>5</sub>Si<sub>3</sub> + Ti<sub>2</sub>C)/Ti) with a low volume fraction of reinforcements and a novel network microstructure were successfully fabricated by reaction hot pressing (RHP) of the Ti–SiC system. In order to tailor the network microstructure, large spherical pure Ti particles and fine SiC particles were selected followed by low energy ball milling and reaction hot pressing. Ti<sub>5</sub>Si<sub>3</sub> rods and Ti<sub>2</sub>C particles were in situ synthesized by the reaction between the Ti and SiC phases and dispersed around the large pure Ti particles forming a unique network microstructure. The yield strength or ductility of the fabricated composites were remarkably improved by tailoring a novel network microstructure and introducing in situ hybrid reinforcements. In particular, both the strength and the ductility of the composite, having 1 vol.% reinforcement, are significantly enhanced compared with that of monolithic pure Ti. This can be attributed to formation of the network microstructure, matrix grain refinement and synthesis of in situ hybrid reinforcements. Furthermore, with increasing volume fraction of reinforcements above 1 vol.%, an increased strength and a decreased ductility were observed.

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## 1. Introduction

Titanium and its alloys have extensive applications in the aerospace, military and automotive industries due to their good mechanical properties [1]. In order to further enhance the mechanical properties of titanium based materials for some critical applications, much attention has been paid on titanium matrix composites (TMCs) due to its superior properties such as high specific strength, high specific stiffness at room temperature and high temperature durability [2,3]. In particular, discontinuously reinforced titanium matrix composites (DRTMCs), fabricated by in situ methods are sought-after due to their superior and isotropic properties along with low cost [4–7]. Moreover, powder metallurgy (PM) coupled with in situ reaction is regarded as one of the most promising in situ methods to fabricate DRTMCs due to microstructural control, near net shape processing, minimal material waste and low cost [2,3,8]. However, the low fracture ductility, in some case, the extreme brittleness of titanium alloy matrix composites with a homogenous microstructure fabricated by conventional PM seriously restricts their practical applications [2,9,10].

By an unusual way, a novel network microstructure of TiB whisker reinforced Ti matrix (TiBw/Ti) composites was successfully designed and fabricated [4,11]. The novel network microstructure can be treated as the Hashin–Shtrikman (H–S) upper bound structure. It can also be viewed as a grain boundary strengthening structure of one hard phase encapsulating one soft phase [12,13]. It is also coincidental with the multi-scale hierarchical structures proposed by Lu [14]: compared with conventional or homogenous composite structure, the strengthening ratio can be further enhanced by assembling components in a controlled way to form a novel reinforcement architecture or hierarchical structure [14]. Therefore, the titanium matrix composites with a network microstructure exhibited both superior tensile strength and superior tensile ductility. This work resolved the bottleneck problem of extreme brittleness of DRTMCs fabricated by PM, and further improved the strengthening effect of the composite.

Apart from TiB whiskers and TiC particles, SiC particles were usually selected as reinforcement of TMCs [2,15,16]. However, in the past 30 years, researchers have always been cautious and therefore done their best to restrict the reaction between the SiC reinforcement and Ti matrix [2,15,16]. In fact, as stated by Poletti et al. [16], the reactions between metal matrices and ceramic particles do not always imply a degradation of the mechanical properties. A thorough reaction may well be exploited for further

\* Corresponding author. Tel.: +86 451 86418836; fax: +86 451 86413922.

E-mail address: [ljuanghit@yahoo.com.cn](mailto:ljuanghit@yahoo.com.cn) (L. Geng).

improving the mechanical properties of TMCs. Additionally, the hybrid strengthening effect may also play a key role due to the different reaction products, such as  $Ti_5Si_3$  and  $Ti_2C$ . Moreover, the novel network microstructure might contribute to a superior combination of mechanical properties. Therefore, the aim of the present work is to fabricate in situ hybrid ( $Ti_5Si_3 + Ti_2C$ )/Ti composites with a novel network microstructure and evaluate their mechanical properties. Of particular interest is a low reinforcement volume fraction ranging from 1% to 5%. The present work can be useful and important to guide and improve performances of TMCs by material design.

## 2. Experimental procedures

### 2.1. Composite fabrication

A system of pure Ti and SiC was selected to fabricate the in situ composites due to their reaction capability [15,16]. In order to tailor a novel network microstructure, large spherical pure Ti particles with an average particle size of 85  $\mu m$  and fine polygonal SiC particles of 2  $\mu m$  were selected as shown in Fig. 1. Before mixing, SiC particles were pretreated by acid cleaning and drying in order to remove impure elements such as S, O and Ni on the particle surface [17]. Pure Ti particles and cleaned SiC particles were blended using low energy milling at a low speed of 150 rpm for 5 h. The process was used to make the fine SiC particles adhere onto the large Ti particles without causing much deformation to the inherent shape of the Ti particles (Fig. 1c). Furthermore, this process can be used to better exploit the toughening effect of the matrix and decrease processing time along with cost [11]. The blending processes were carried out in a pure argon atmosphere. The blended mixtures were hot pressed in vacuum ( $10^{-2}$  Pa) at 1200  $^{\circ}C$  under a pressure of 20 MPa for 60 min. Duda's work indi-

cated that  $Ti_2C$  phase is easily formed when Ti is sufficient, while TiC phase is formed when C is rich [18]. Aksyonov et al. [19] also pointed out that the process of  $Ti_2C$  formation is energetically favorable if TiC is in contact with titanium. Considering the designed network microstructure, only low volume fractions of reinforcements are permitted [20]. Therefore, the reaction between Ti and SiC is hypothetically described as follow:



On the basis of the above equation, 1 vol.%, 3 vol.% and 5 vol.%( $Ti_5Si_3 + Ti_2C$ )/Ti composites with a network microstructure were fabricated by controlling the added weight of the SiC raw material. For comparison, monolithic pure Ti sample was fabricated using the same processing parameters.

### 2.2. Microstructure examinations

Microstructural characterization was carried out using optical microscopy (OM, OLYMPUS PEM-3) and scanning electron microscopy (SEM, Hitachi S-4700). The samples for microstructural observation were etched using the Kroll's solution (5 vol.%HF + 10 vol.%HNO<sub>3</sub> + 85 vol.%H<sub>2</sub>O) for 8 s after mechanical polishing. Fracture characterization was also done using SEM.

### 2.3. Mechanical property tests

Room temperature tensile tests were carried out using an Instron-5569 universal testing machine at a constant crosshead speed of 0.5 mm/min (approximate strain rate is  $5.5 \times 10^{-4}$ /s). High temperature tensile tests were carried out using an Instron-1186 universal testing machine at the same constant crosshead

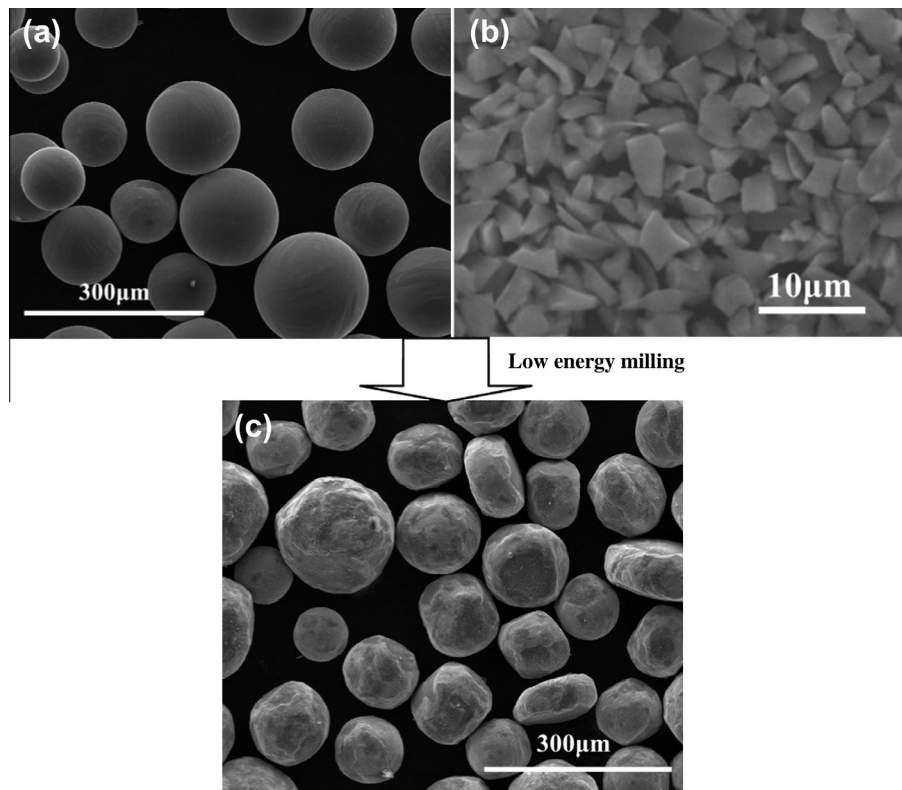


Fig. 1. SEM micrographs of (a) pure Ti particles (85  $\mu m$ ), (b) SiC particles (2  $\mu m$ ), and (c) mixtures of Ti and SiC particles.

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