Materials Research Bulletin 44 (2009) 966–969

Contents lists available at ScienceDirect

Materials Research Bulletin

journal homepage: www.elsevier.com/locate/matresbu

Controlling the oriented growth of Ti_2SnC grains with carbon fiber as a reactive template in the Ti–Sn–C system

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ARTICLE INFO

Article history:
Received 13 March 2007
Received in revised form 11 June 2007
Accepted 18 November 2008
Available online 27 November 2008

Keywords:
A. Ceramics
A. Carbides
B. Crystal growth
D. Microstructure

ABSTRACT

Carbon short fibers with a length of ~3 mm used as a reactive template were mixed with Ti and Sn powders. The mixture was pressurelessly sintered at 1200 °C for 2 h in a vacuum atmosphere. The microstructure shows that Ti_2SnC grains with a plate-like shape grow along a preferred direction, forming a 'string' structure in which Ti_2SnC platelets pack themselves closely with a top–bottom–top–bottom sequence. With increasing the length of the C fibers, the length of the 'string' structures increases. A large amount of long and aligned 'string' structures have formed after sintering the samples prepared by infiltration of the carbon short fibers with a length of ~50 mm in a Ti–Sn slurry and then stacking and cold pressing. X-ray diffraction and scanning electron microscopy, respectively were used to analyze and observe the phase composition and microstructure.

1. Introduction

The layered ternary compounds $M_{n+1}AX_n$ (n = 1, 2, 3), where M is a transition metal, A is a group IIIA or IVA element, and X is C or N, such as Ti_3SiC_2, Ti_3AlC_2, Ti_2SnC, etc., have attracted much world-wide attention in recent years owing to their outstanding properties [1]. Among these compounds, Ti_2SnC is one of the most interesting materials because of its high electrical conductivity, self-lubricity, machinability, etc. [2–7].

The crystal structure of Ti_2SnC is hexagonal with strongly anisotropic chemical bonding, which causes anisotropic behavior along the *a*- and *c*-axes. For example, its electrical conductivity along the *a*-axis is much higher than along the *c*-axis [6]. In addition, its grains also exhibit an anisotropic grain growth behavior, with plate-like and rod-like shapes [5].

To obtain the best properties along *a*- or *c*-axes, researchers have tried to fabricate the $M_{n+1}AX_n$ materials with an optimum microstructure such as a single crystal or a highly textured microstructure. However, it is difficult to obtain the $M_{n+1}AX_n$ single crystals, but a textured Ti_3SiC_2 ceramic with good mechanical properties was first obtained using a hot forging technique [8]. In addition, Ti_3SiC_2 and Ti_3AlC_2 thin films with oriented grains and a textured microstructure have been fabricated by elemental target magnetron sputtering [9–12]. However, no information on the fabrication of a textured Ti_2SnC ceramic is available. Therefore, it needs an effective method to fabricate the textured Ti_2SnC ceramic used to investigate its anisotropic properties.

Recently, the reactive-templated grain growth (RTGG) has been proved to be one of the most effective methods for the fabrication of the textured piezoelectric ceramic [13,14]. In the RTGG process, reactive template particles with a specific crystal structure are mixed with reactants. After sintering, the target material will be textured, with an oriented grain growth. Hence the reactive template material plays an important role in the formation of the textured structure. For Ti_2SnC, if a suitable reactive template was chosen, a textured microstructure with an oriented grain growth would be obtained using the RTGG process.

Recently, it was reported that a double layer including a thin inner layer and an outer layer was formed around the unreacted C particles or fibers during the synthesis of Ti_2SnC [5,15]. The inner layer is composed of TiC grains, and the outer layer consists of Ti_2SnC grains, which grow in a preferred orientation normal to the surface of the inner layer. This feature suggests that the C fiber can be chosen as a reactive template in the Ti–Sn–C reaction system to fabricate the textured Ti_2SnC ceramic.

The main purpose of this work is to investigate the effect of the C fiber as a reactive template in the Ti–Sn–C reaction system on the oriented grain growth of Ti_2SnC.

2. Experimental procedures

Commercial powders of Ti (−325 mesh, >99.2% purity), Sn (−200 mesh, >99.5% purity) and C fiber bundles (denoted as C_f bundles, each fiber less than 8 μm in diameter) were used in the present study.

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doi:10.1016/j.materresbull.2008.11.017

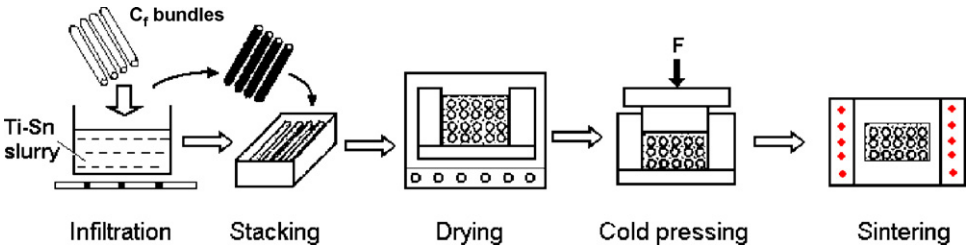

Fig. 1. Schematic diagram of stacking process.

The C_f bundles were cut to short fibers (C_{sf}) with size ~3 mm in length and mixed with Ti and Sn powders with a mole ratio of Ti:Sn:C_{sf} = 2:1.1:0.9 (denoted as 2Ti–1.1Sn–0.9C_{sf}) in a polypropylene bottle for 10 h. 10 mol.% of more Sn was introduced in the system is on the assumption of the loss of Sn at high temperature. During mixing, some agate balls with sizes of 3–20 mm in diameter were put into the container to ensure better mixing effect. The mixed powders were cold-pressed to form compacts with a diameter of ~20 mm and a height of ~5 mm. Then the compacts were put into a graphite crucible and pressurelessly sintered at 1200 °C for 2 h in a vacuum atmosphere. The as-fabricated samples were pulverized to identify the phase composition using a D/Max 2200PC X-ray diffractometer at 40 kV and 40 mA with Cu Kα radiation.

In another set of experiment, Ti and Sn powders with a mole ratio of 2:1.1 were mixed with 40 wt.% ethanol as a solvent in a polypropylene bottle for 10 h to form a Ti–Sn slurry. The C_f bundles with 50 mm in length were cut, and then infiltrated in the Ti–Sn slurry. The infiltration process was performed in an ultrasonic vibrator (KQ-100E, China) with a frequency of 40 kHz for 20 min to guarantee the slurry uniformly spreading through the bundles. The infiltrated C bundles were aligned and stacked layer by layer in a stainless steel mold with a 50 × 20 mm^2 die channel and then dried in an oven at 60 °C for 30 min. After drying, the sample in the stainless steel mold was cold-pressed under 120 MPa to form a compact with a volume of 50 × 20 × 3–5 mm^3. The green compact was then pressurelessly sintered under the same condition as that mentioned above. The whole process is schematically described in Fig. 1.

The microstructure of the sintered samples was observed by a STEREOSCAN 360 scanning electron microscopy (SEM) equipped with energy-dispersive spectroscopy (EDS).

3. Results and discussion

Fig. 2 shows a series of SEM images of the as-synthesized samples made of 2Ti–1.1Sn–0.9C_{sf} after sintering at 1200 °C for 2 h. An SEM micrograph in Fig. 2(a) displays that a large amount of

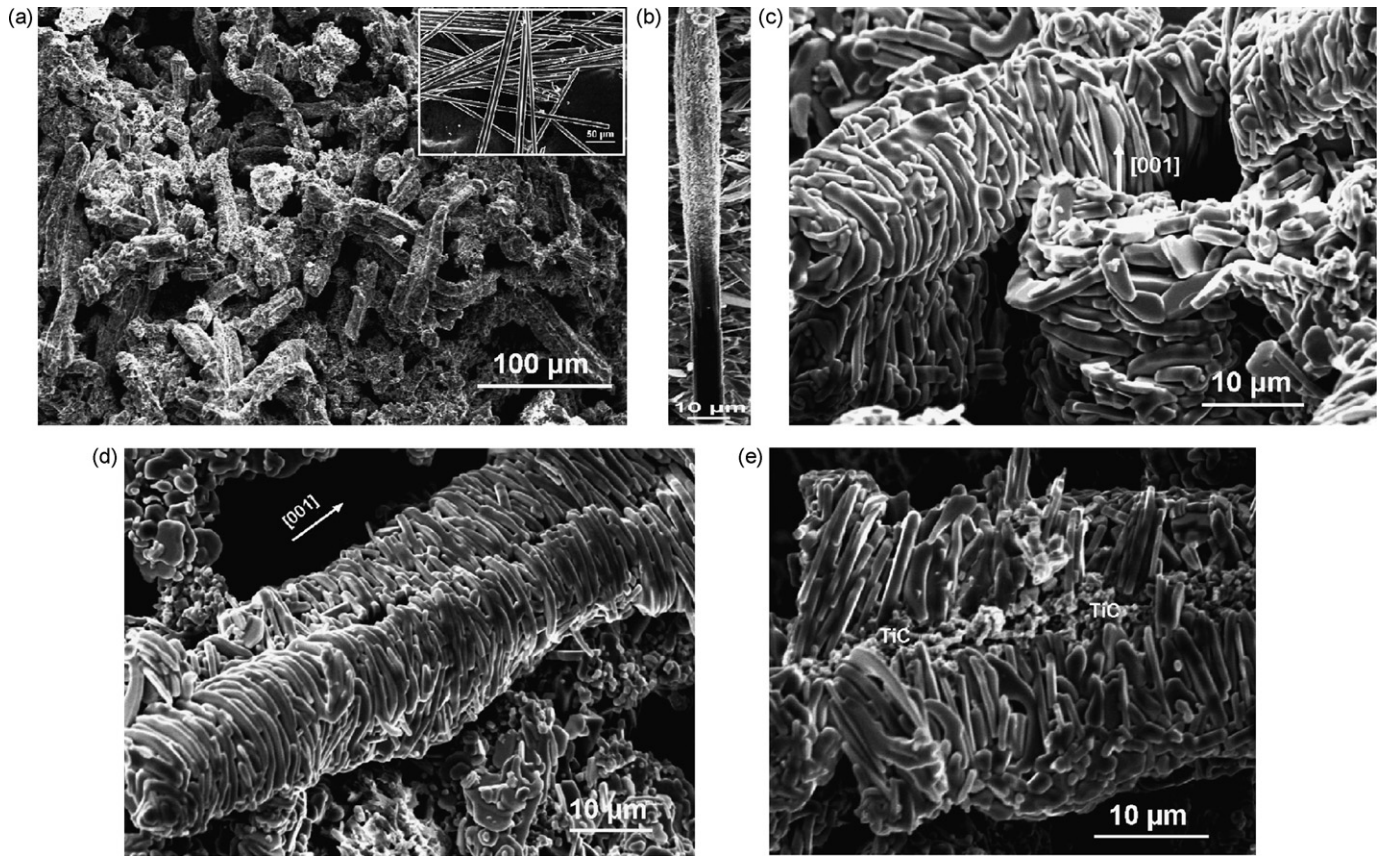

Fig. 2. Scanning electron microscopy images of the fracture surface of the sample made of 2Ti–1.1Sn–0.9C_{sf} after sintering at 1200 °C for 2 h. (a) A low magnification. Inset in (a) shows the morphology of the carbon short fibers; (b) an image of a residual carbon fiber, showing a reacted section with swollen feature and an unreacted section; (c)–(e) are high magnifications, showing the oriented growth of Ti_2SnC grains. Smaller TiC grains distributed in the central site along the string's axis are shown in (e).