Biomorphic Synthesis of TiC Hollow Fibers from Cotton Fibers

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Abstract

Biomorphic titanium carbide fibers have successfully been prepared using natural fiber of cotton as bio-templates. Cotton fibers charcoal are infiltrated with titanium oxide sol into hollow fiber and subsequently sintered in inert gas at high temperature to produce the final titanium carbide fibers. The morphology of resulting titanium carbide fibers, as well as conversion of cotton fibers to ceramic fiber, has been investigated by SEM and XRD techniques. Experimental results showed that the biomorphic fiber morphology of cotton fiber charcoal is remained in the TiC fibers.

Key words: Biomorphic titanium carbides, Hollow fibers, Carbothermal reduction, Infiltration, Sol-gel process

Introduction

Recently, it was reported that synthesis of ceramic materials from natural grow plant structures has received increasing interests. In comparison to synthetic materials, natural materials such as wood, jute, bamboo, etc., exhibit a multi-scale built anatomy, developed and optimized in a long-term evolution process. Materials scientists have attempted to utilize these natural as well as the pre-processed biological plant materials for the fabrication of various oxide or carbide eco-ceramic materials, such as SiC, SnO₂, and Al₂O₃.(1-3) However, the morphology of matured cotton fiber is more uniform compared with other kinds of plant fiber. So in this paper, it was selected to serve as a bio-template to prepare biomorphic titanium carbide (TiC ) fibers.

In this study, the effect of infiltrated cycles and holding time of TiO₂-sol in synthesizing biomorphic TiC fibers were presented.

Thermodynamic Analysis

In this study carbon perform coated with TiO₂-sol were used as precursors for the carbothermal reaction. The overall chemical reaction can be express as:

\[ \text{TiO}_2(s) + 3\text{C}(s) = \text{TiC}(s) + 2\text{CO}(g) \]  \hspace{1cm} (1)

Thermodynamic equilibrium calculations were performed using a computer program based on Gibbs energy minimization method to determine the most stable composition of the materials as function of temperature.

The equilibrium composition of TiO₂-C system at different temperatures was calculated using Gibbs energy minimization method and the results are shown in Figure 1.

Figure 1. Equilibrium composition of TiO₂-C system in Ar-gas atmosphere.\(^{(4)}\)

Figure 1. Shows that it is thermodynamically feasible to fabricate TiC ceramic by using carbothermal reduction process. When increases temperature to about 1270°C, TiO₂ is reduced by carbon and produces TiC. The reaction will continue as the temperature increases until...
complete reaction at about 1320°C. The resulted phases can be maintained as TiC ceramic after cooling down process under Ar gas atmosphere.

**Materials and Experimental Procedures**

Dried natural cotton fibers (Figure 2 (a)) were first carbonized in inert gas at 700°C for 1 hr and cooling down to room temperature with flowing argon gas of 3 LPM to obtain cotton fibers charcoal (Figure 2(b)). After calcinations, the morphology of the cotton fiber charcoal has been shrunked and rather round. The TiO$_2$ solution prepared from titanium tetra-isopropoxide (TTIP: C$_{12}$H$_{28}$O$_4$Ti, 97%, Fluka) added to 2-propanol ((CH$_3$)$_2$CHOH, 99.7%, LabScan) under stirring within 15 minute. Subsequently, 2M HCl was added to the solution. The resulting TiO$_2$ colloid solution was kept under stirring for 30 minute before left it in refrigerator for 24 hours to get stable TiO$_2$-sol. The carbon fiber preform were vacuum-assisted infiltrated with titanium dioxide sol for one cycle of 1,2,3 and 4 minute and two cycles of 4 minute. All the experimental conditions were shown in Table 1. After drying at 100°C for 24 hr, the resulted carbon fibers coated with TiO$_2$ were obtained. The TiO$_2$-coated carbon fibers were loaded into high temperature furnace (Carbolite, CTF 18/75/600) and the samples were synthesized by carbothermal reduction process at 1500°C for 60 minute with the heating rate of 5 °C/min under flowing of argon gas (3LPM). The resulted biomorphic TiC hollow fibers were obtained as shown in the Figure 2 (C).

<table>
<thead>
<tr>
<th>Holding time (min)</th>
<th>Cycle infiltrate</th>
<th>Soaking time (min)</th>
<th>TiO$_2$ / C wt. Ratio</th>
<th>Code</th>
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<td>60</td>
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<td>2</td>
<td>60</td>
<td>2.229</td>
<td>4/2 R60</td>
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</table>

**Results and Discussion**

**XRD Analysis**

Figure 3(a) shows the XRD patterns of resulted TiC fibers obtained from cotton fibers charcoal with one cycle- infiltrated of TiO$_2$-sol at different holding time. The results show that completed reaction to get only TiC phase occur when using condition of 4 minute of one cycle infiltration. When decrease the infiltrated holding time, the resulted products shown uncompleted reaction and left TiO$_2$ in the resulted products. This may be due to their natural nature of quite wide range in shape and size of cotton fiber that eventhough lower TiO$_2$ to carbon weight ratio found in less holding time (Figure 4 (a)) but still left TiO$_2$ in the system after the synthesis. When increase infiltrated cycle at the same holding time of 4 minute (Figure3 (b)), the resulted phases of TiC with small amount of Ti$_2$O$_3$ phase was obtained. The reason of titanium oxide left in the system may be from the additional excess amount of TiO$_2$ coated on carbon preform as shown in Figure 4(b).
**SEM Analysis**

Figure 5. Shows the SEM images of the resulted biomorphic TiC fibers derived from cotton fiber of 1 and 2 infiltrated cycles at 4 minute holding time. As shown in this figure these biomorphic TiC fibers are hollow with diameter ranging from 8 μm to 10 μm and relatively smooth inner surface.

Figure 3. XRD analysis of TiC formation after carbotermal reduction process at 1500°C, 60 min. (a) Variable of holding time. (b) Variable of cycle infiltrates.

Figure 4. TiO2 to Carbon weight ratio as function of (a) infiltrated holding time (b) infiltrated cycles at 4 minute holding time.

**Conclusions**

Titanium carbide fibers were produced via carbotermal reduction synthesis of cotton fibers. The product fibers from one cycle of vacuum infiltrated at 4 minute holding time was shown only TiC phase. When increased infiltrated cycle or decrease holding time, the resulted products shown uncompleted reaction and lift TiO2 in the resulted products.
Acknowledgements

The authors are pleased to acknowledge the financial support for this research by the National Research Council of Thailand (NRCT) and Prince of Songkla University.

References


