Determination of Non-stoichiometry of Tubular Titanium Carbide Formed by Self-Propagating High Temperature Synthesis

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Abstract

Titinium carbide (TiC_x) was produced by self-propagating high temperature synthesis (SHS) method. The morphology and non-stoichiometric number of the SHS product were observed by scanning electron microscopy and neutron diffractometry, respectively. Tubular titanium carbide with hole inside was formed with different non-stoichiometric number (x), which value increased with combustion temperature.

Keywords: titanium carbide, self-propagating high temperature synthesis, neutron diffraction, non-stoichiometry

1. Introduction

Titanium carbide like fibrous or tubular shape can give more wider engineering applications because of the possibility of precise electrical controlling such materials as bio-medial filter, separator of geans, nano-sensor and hydrogen storage materials.[1] In this study titanium carbide with tubular shape was prepared by self-propagating high temperature synthesis, their morphology and non-stoichiometric number were determined by neutron diffractometry.

2. Experimental method

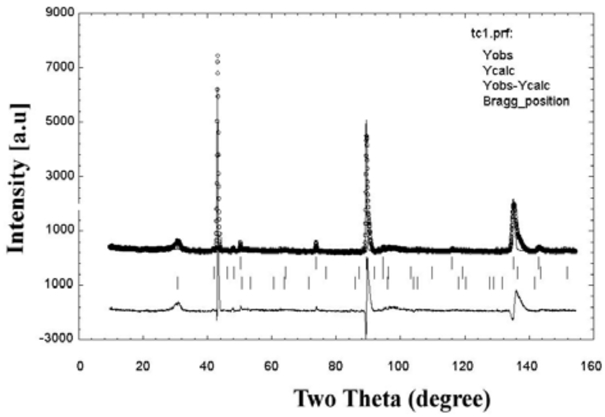
Titanium powders (99.999% purity, < 325 mesh), carbon black (99.999% purity, 7 μm) and carbon fibers(99.999% purity, ϕ 18 nm) were examined. Stoichiometric amounts of titanium and carbon (molar ratio of 1:1) were mechanically blended in an inert atmosphere and ignited for SHS. Combustion was determined by optical pyrometer and two-thermocouple method.[2] After the SHS reaction, the products were leached at nitric acid and heat treated at 500-700^oC for 6 hours. The phase identification and microstructure of the final product were performed by a neutron diffractometry (HANAO, KAERI, Korea) and scanning electron microscopy (JEOL 35C Japan).

3. Results and Discussion

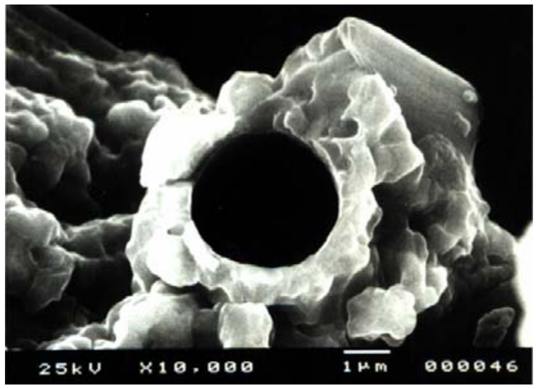
Combustion temperatures of the SHS reaction were above 1750^oC. Fig. 1 is the typical neutron-diffraction spectra and final morphology. The Rietvelt analysis showed that the non-stoichiometric number is 0.87 at the combustion temperature of about 1750^oC, whereas, it is 0.93 at the temperature of 1820^oC. Fig. 1-b is the tubular titanium carbide observed by scanning electron microscopy. It is interesting how the tubular carbide was formed during the combustion reaction. Considering equilibrium shape formed through nucleation and growth, a fibrous shape with a hole inside can be formed through heterogeneous nucleation and anisotropic growth during the combustion reaction.[3] Since the combustion temperature is higher than the melting point of titanium and lower than the melting point of carbon, initial liquid nucleus was formed on the solid carbon fiber surface. Lateral direction has high curvature, the carbon atom on the lateral surface faster diffuses out into liquid titanium than the carbon atom on cross sectional surface by inter-diffusion process.
Since the diffusion rate of titanium atom into solid carbon is relatively slower than that of carbon atom into liquid titanium, the final product like titanium carbide was formed between liquid titanium and solid carbon. Un-reacted carbon, if it is remained, is separated during cooling due to the difference of thermal expansion coefficient. Therefore, the formation mechanism of the carbide observed in Fig. 1-b is related to a liquid-solid reaction including the preferential diffusion process of carbon atom into liquid titanium.

4. Summary

Tubular shape of titanium carbide (TiC_x) was synthesized by SHS. The Rietvelt analysis showed that the non-stoichiometric number is 0.87 at the combustion temperature of about 1750^oC, whereas, it is 0.93 at the temperature of 1820^oC, respectively. The formation.



(a)



(b)

Fig. 1. Typical neutron diffraction spectra (a) and morphology (b) of TiC_x formed by SHS.

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6. References

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