Synthesis of Nanostructured TiC/Co Composite Powder by the Spray Thermal Conversion Process

Gil-Geun Lee[1,a], Gook-Hyun Ha[2,b], and Byoung-Kee Kim[2,c]

[1]Division of Materials Science and Engineering, Pukyong National University, San 100, Yongdang-Dong, Nam-Gu, Busan, 608-739, Korea
[2]Materials Technology Department, Korea Institute of Machinery and Materials, 66, Sangnam-Dong, Changwon, Kyungnam, 641-831, Korea
[a]gglee@pknu.ac.kr, [b]ghha@kmail.kimm.re.kr, [c]kbk1649@kmail.kimm.re.kr

Abstract

In the present, the focus is on the synthesis of nanostructured TiC/Co composite powder by the spray thermal conversion process using titanium dioxide powder has an average particle size of 50 nm and cobalt nitrate as raw materials. The titanium-cobalt-oxygen based oxide powder prepared by the combination of the spray drying and desalting methods. The titanium-cobalt-oxygen based oxide powder carbothermally reduced by the solid carbon. The synthesized TiC-15wt.%Co composite powder at 1473K for 2 hours had an average particle size of 150 nm.

Keywords : titanium carbide, carbothermal reduction, hard metals

1. Introduction

The mechanical properties of the tool materials depend not only on the chemical composition but also on their microstructure.[1)] The hardness, fracture toughness and wear properties of the tool materials were strongly influenced by the size of the hard particles and the distance of the mean free path among the hard particles. These mechanical properties increased simultaneously with decreasing particle size and the distance of the mean free path. To manufacture high performance tool material with an ultra fine microstructure, raw powder materials with an ultra fine particle size should be used. Recently, there are several methods proposed for the synthesis of ultra fine particles.[2,3)]

The titanium carbide (TiC) has been used as a dispersion particle in the WC-TiC-Co system tool materials for increasing high temperature hardness and decreasing reaction with steel alloy during machining, and also extensively used in the cermet tool materials of TiC and TiCN systems.[4)] A number of processes exist for synthesizing titanium carbide, such as carbothermal reduction of titanium dioxide, direct carbarization of titanium, chemical reaction of titanium chloride ($TiCl_4$), self-propagating high temperature synthesis, amongst others. Generally, these processes need over temperature range of 1700-2100K for the commercial product of titanium carbide powder. So that, these processes have the difficulty of decreasing particle size due to the agglomeration of each particles at high synthesis temperature.

Recently, a new spray thermal conversion process is proposed for the synthesis of ultra fine powder.[2,5)] This process is composed of three steps; synthesis of precursor powder by spray drying of aqueous solution of the metallic salts, desalting treatment of precursor powder and thermochemical conversion of the desalted powder. This new spray thermal conversion process can be obtains metal/metal and carbide /metal composite powders as final products. The synthesized carbide/metal composite powder expected has a more homogeneous mixing state of the carbide and metal than conventionally mixed powders, therefore this can be used as a raw powder for the tool materials.

In the present study, the focus is on the synthesis of titanium carbide/cobalt composite powder by the spray thermal conversion process. And also the analysis of carbothermal reduction of titanium-cobalt-oxygen based oxide powder by solid carbon for the optimizing synthesis process of ultra fine titanium carbide/cobalt composite powder.

2. Experimental and Results

Figure 1 shows the X-ray diffraction patterns of synthesized titanium-cobalt-oxygen based oxide powder for the final composition of the TiC-15wt.%Co by spray drying and desalting processes. The starting slurry was prepared by suspending titanium dioxide powder has an average particle size of 50 nm, TiO_2, in an aqueous solution of cobalt nitrate, $Co(NO_3)_36H_2O$, to obtain a final composition of TiC-15wt.%Co. Spray drying was performed using a rotary atomizer with a slurry feed rate of 20 mL/min and an atomizer rotating speed of 11000 rev/min in a hot air (523 K) stream. A precursor powders obtained by spray drying were burnt out at 1073 K for 2 hours in air atmosphere to remove organic salt components and form a titanium-cobalt-oxygen based oxide powder. The synthesized oxide powder shows diffraction peaks of anatase-TiO_2 and $CoTiO_3$ phases.

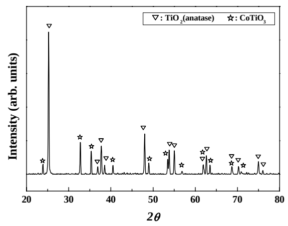

Fig. 1. X-ray diffraction pattern of synthesized titanium-cobalt-oxygen based oxide powder.

The titanium-cobalt-oxygen based oxide powder was mixed with carbon black (mean particle size: 0.5μm) for 24 hours using a tumbler-ball mill. The mixture was placed in a graphite crucible and then heat treated at a temperature from 1073 to 1673 K for 30 minutes in a tube furnace under a flowing stream of argon atmosphere. Figure 2 shows the X-ray diffraction patterns of the mixture of titanium-cobalt-oxygen based oxide powder and carbon black heat treated in the tube furnace under a flowing stream of argon at a specific temperature for 30 minutes. At 1073 K, there were

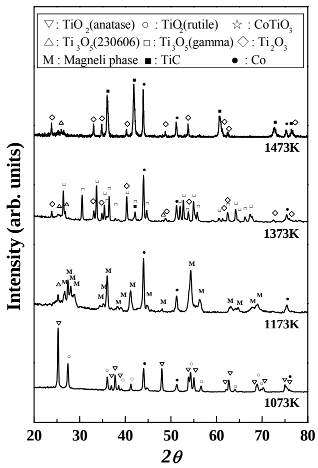

Fig. 2. X-ray diffraction patterns of the mixture of titanium-cobalt-oxygen based oxide powder and carbon black heat treated at a specified temperature for 30 minutes.

three kinds of phases in the diffraction pattern: anatase-TiO_2, rutile-TiO_2 and cobalt. The TiC peak can be observed in the X-ray diffraction pattern of mixture heat-treated at 1373 K. The cobalt oxide, $CoTiO_3$, in the titanium-cobalt-oxygen based oxide powder reduced at about 1073 K. The titanium oxide in the one reduced at about 1173 K, and titanium carbide formed at about 1373 K.

Figure 3 shows the TEM micrograph of synthesized TiC/Co composite powder at 1473 K for 2 hours. The synthesized titanium carbide and cobalt particles have an average particle size of 150 nm.

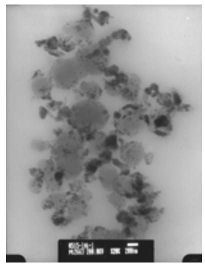

Fig. 3. TEM micrograph of synthesized TiC/Co composite powder.

3. Summary

The TiC/Co composite powder has an average particle size of 150 nm can be synthesized by the spray thermal conversion process using titanium dioxide powder and cobalt nitrate as raw materials. The titanium-cobalt-oxygen based oxide powder carbothermally reduced to the titanium carbide/cobalt composite powder through three steps of carbothermal reduction steps with increasing temperature; reduction of $CoTiO_3$, reduction of TiO_2 and formation of titanium carbide.

Acknowledgements

This research was supported by a grant from the Center for Advanced Materials Processing (CAMP) of the 21st Century Frontier R&D Program funded by the Ministry of Commerce, Industry and Energy, Republic of Korea.

4. References

1. K.J.A.Brookes, Word Directory and Handbook of Hardmetals and Hard Materials, 6th ed., International Carbide Data, Marsh Barton, pp.95-102, 1996.
2. G.G.Lee, G.H.Ha and B.K.Kim, Powder Metallurgy, **43**, 79(2000).
3. K.E.Gonsalves, S.P.Rangarajan and J.Wang, Handbook of Nanostructured Materials and Nanotechnology edited by H.S.Nalwa, Academic Press, London, pp. 1~56, 2000.
4. M.Sherif El-Eskandarany, J. of Alloys Compound, **305,** 225(2000).
5. B.K.Kim, G.H.Ha, D.W.Lee and G.G.Lee, Advanced Performance Materials, **5**, 341(1998).