

Refining of $\text{TiO}(\text{OH})_2$ slurry and carbon control of ultrafine TiC-Co powder

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1. Introduction

TiC based-alloy has been widely used as cutting tools and dies due to a high hardness and wear resistance. In order to increase the wear resistance of TiC based-alloy, ultrafine TiC powder must be developed. In industry, TiC has primarily been produced by the carbothermal reaction of TiO_2 with carbon at about 1700°C and the produced TiC size is about $1\sim 2\ \mu\text{m}$.

In this study, spray conversion process was applied to synthesize ultrafine TiC powders. The effect of washing treatment of $\text{TiO}(\text{OH})_2$ slurry on sulfur elimination were studied. The control of carbon content of TiC powders was also investigated in manufacturing processes including H_2 and vacuum atmospheres.

2. Experimental

The material $\text{TiO}(\text{OH})_2$ slurry produced by sulfate process was used as a raw material. The $\text{TiO}(\text{OH})_2$ slurry was washed 3 times by water, and then added water and NH_4OH as PH controlling agent in water. The NH_4OH was added until PH value in water reached in the range of $7\sim 9$. After adding NH_4OH , the slurry was washed by distilled water and dried.

The precursor solution of $\text{TiO}(\text{OH})_2$ and Co nitrate was spray-dried and calcined at 700°C for 3 hours. The calcined Ti-Co oxide and carbon ($100\%\sim 140\%$) were ball-milled for 24 hours. Reduction/carburization treatments of the milled composite oxide powder with different carbon were conducted at $1200\sim 1250^\circ\text{C}$ for $3\sim 6$ hours in H_2 and vacuum atmospheres.

The morphology and microstructure of the powders were examined by FE-SEM. The chemical analyses of TiC-Co powders were also performed with LECO TC-436 for oxygen and with LECO TC-600 for carbon.

3. Results and discussion

a) Refining of $\text{TiO}(\text{OH})_2$ slurry

Raw material $\text{TiO}(\text{OH})_2$ slurry contains impurity sulfur as much as 4.40 wt%. In order to decrease S content, slurry was washed by water, and then new water and $\text{NH}_4(\text{OH})$ were added in $\text{TiO}(\text{OH})_2$ slurry. After separation and washing of $\text{TiO}(\text{OH})_2$ powder, sulfur content in $\text{TiO}(\text{OH})_2$ was drastically decreased. The effect of $\text{NH}_4(\text{OH})$ addition was explained as follows;



b) Carbon control of TiC-5%Co powder during synthesis in H_2 atmosphere

Fig. 1 shows the carbon contents in TiC-5%Co powders heat-treated at 1250°C for 6 hours in H_2 atmosphere. By increasing added carbon amount in calcined Ti-Co based composite oxides, the carbon content in TiC-5%Co powder increased. After adding 120% of carbon, the carbon and oxygen contents are 18.8% and 1.41%, respectively. This carbon content in powder is very similar to the theoretical carbon content(19.04%) of TiC-5%Co composition. The formation temperature of TiC particle is very low when compared to that of conventional process due to a high reactivity of fine particle in Ti-Co oxides. Fig. 2 show the FE-SEM micrograph of TiC-5%Co powder formed by addition of 134% carbon. The particle sizes in TiC-5%Co powder were found to be below 200 nm. The small particles existed on the surface of large powder formed at 1250°C for 6 hours.

c) Carbon control of TiC-5%Co powder during synthesis in vacuum.

The reduction/carburization process in vacuum has some advantage such as batch-type . After heat-treatment at 1200°C for 3 hours in vacuum, TiC phase was formed and oxygen content in TiC-5%Co powders is below 0.52%. Fig. 3 shows the variations of carbon content in TiC-5%Co powders with added carbon content and heat-treatment time. 100% of carbon

addition is sufficient to control carbon in TiC-5%Co powder due to small carbon loss during vacuum heat-treatment.

Fig. 4 shows the FE-SEM micrographs of TiC-5%Co powders formed at 1200°C for 3 hours in vacuum. The TiC particle size decreased as the added carbon content increased because the TiC particle growth inhibited by excess carbon particles.

3. Conclusion

The sulfur content in TiO(OH)₂ slurry decreased by the treatment of NH₄OH addition and washing. Ultrafine TiC-5%Co powders were successfully synthesized at 1200~1250°C for 3 hours in H₂ and vacuum atmosphere. The optimum added carbon contents necessary for formation of TiC-5%Co powder in H₂ and vacuum processes were 120% and 100%, respectively.

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 Science & Technology, Republic of Korea.

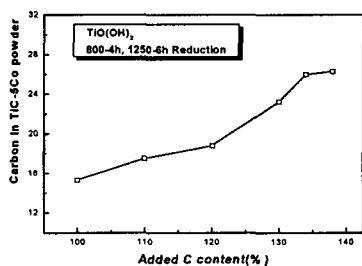


Fig. 1 The carbon contents in TiC-5%Co powders heat-treated at 1250°C for 6 hours in H₂ atmosphere.

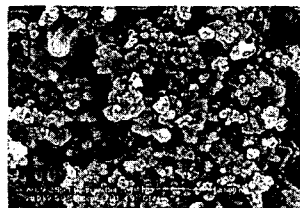
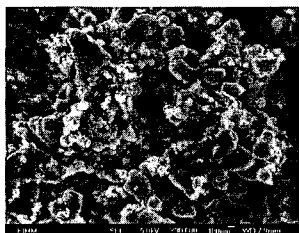


Fig. 2 The FE-SEM micrograph of TiC-5%Co powder formed by addition of 134% carbon.

Fig. 3 Variations of carbon content in TiC-5%Co powders with heat-treatment time and added carbon.



1200°C, 3h(100%C)



1200°C, 3h(120%C)

Fig. 4. The FE-SEM micrographs of TiC-5%Co powders.