Development of Nano-sized WC Powder for Hardmetals

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Abstract

In order to develop the nano-sized WC powder that improved the hardness of hardmetals, carbothermal reduction of WO3 by C was examined by using the thermogravimetric analysis. At the direct carburization reaction path of $WO_3 \rightarrow WO_{2.72} \rightarrow WO_2 \rightarrow W \rightarrow W_2C \rightarrow WC$*, the nano-sized grain was generated at the reaction stage* $WO_{2.72}$ *to* WO_2 *and W. For trial production, the intermediate products which consists of metal and carbide phases obtained by the first heating has been carburized to the final WC powder. We succeeded in the development of the WC powder of about 70nm. In addition, the nano-sized WC powder in which the vanadium of the most effective grain growth inhibitor was uniformly dispersed was developed.*

Keywords : tungsten carbide, nano-sized, direct carburization

1. Introduction

In hardmetal tools used for producing high-precision electronics device, the WC-Co hardmetal with finer WC crystal is desired. Due to ultrafine WC-Co hardmetal is high hardness and strength [1], WC powder with 300 to 500 nm are used for hardmetal production of high hardness and strength. Nano-sized of raw WC powder can be expected to improve hardness, strength and a high surface accuracy in processing of WC-Co hardmetal.

In order to achieve the manufacturing of a nano-sized WC powder, in this report, the morphological change occurred during direct carburization (reduction and carburization reaction of WO_3 powder by carbon) were researched by thermogravimetric analysis, and an experimental production for WC powder with an average particle size less than 70 nm was aimed by the direct carburization technique. In addition, milling and classification effects of the intermediate and final products, and the behavior of vanadium addition for grain growth inhibitor during direct carburization process, were also researched.

2. Experimental and Results

The nano-sized WO_3 and carbon powder were used as raw materials. The raw material powders were mixed uniformly. For an experimental production, the mixed raw powder was pelletized to 2～3 mm. Reaction of the mixed powders were reacted using a thermobalance in N_2. The reaction process and morphological change of the direct carburization were examined by XRD and SEM. Fig. 1 shows mass loss, DTA, and phase of the products obtained by thermobalance tests. The direct carburization reaction proceeded to final WC via various kinds of intermediate products such as $WO_3 \rightarrow WO_{2.90} \rightarrow WO_{2.72} \rightarrow WO_2 \rightarrow W \rightarrow W_2C$. The active reduction reaction from $WO_{2.72}$ to WO_2 took place approximately 1280 K. The intermediate $WO_{2.72}$ grain grew into thick-whisker shape due to the reduction reaction that takes place at the high temperature. In the following reduction stage to WO_2 or W, nano-sized grains formed by nucleation. (A) in Fig.2 shows the nuclear generation process from the thick-whisker $WO_{2.72}$ to the nano-sized WO_2 or W grains.

The reaction of the direct carburization consists of first stage of intermediate generation with nano-sized particle and second stage from intermediate product to WC powder.

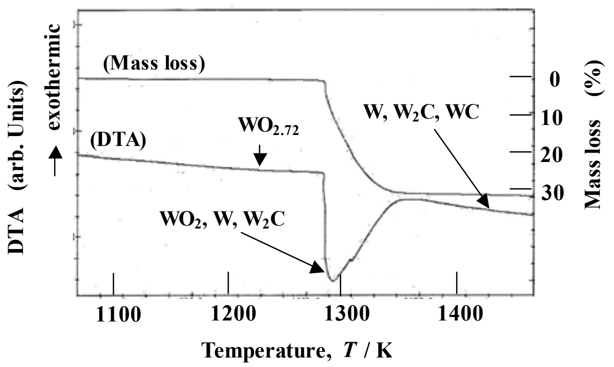

Fig. 1. DTA and mass loss curves for the mixture of WO_3 and C in N^2, and phase of products indentified by XRD.

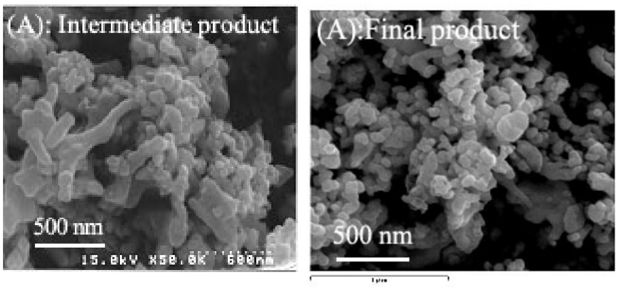

Fig. 2. SEM images of intermediate products obtained by DTA and final product of experimental

The appropriate condition examined by thermobalance was developed to experimental production. The intermediate product was carburized to WC powder of single phase by second stage reaction at 1373 K. (B) in Figure 2 shows the SEM image of the WC powder of 78nm (by BET method) obtained by experimental production.

In order to obtain finer particles and a narrow particle size distribution, milling and classification of the nano-sized WC powder by jet mill were studied. The nano-sized WC powder was crushed at the maximum air jet flow of the jet mill, and the milled powder was classified by controlling the rotational speed of classification rotor, and it was collected with a cyclone collector and a bag filter. Table 1 shows the properties of the nano-sized WC powders milled and classified by jet mill. Figure 3 shows the SEM image of each WC powder in Table 1. By the classification function of the jet mill, coarse particles collected in the cyclone, and finer particles collected in the bag filter. The coarse agglomerated particle remaining without being crushed by jet milling was collected in cyclone. The collected particles in the bag filter is less agglomeration and the average particle size of 61 nm.

Table 1. Properties of the nano-sized WC powder milled by jet mill.

	Before jet mill	Jet milling	
		Cyclone	Bag-filter
BET m^2/g	5.28	5.03	6.28
nm by BET	72	76	61
O %	0.48	0.48	0.5

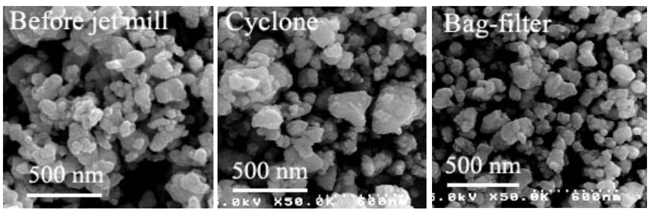

Fig. 3. SEM images of the nano-sized WC powder mill ed by jet mill.

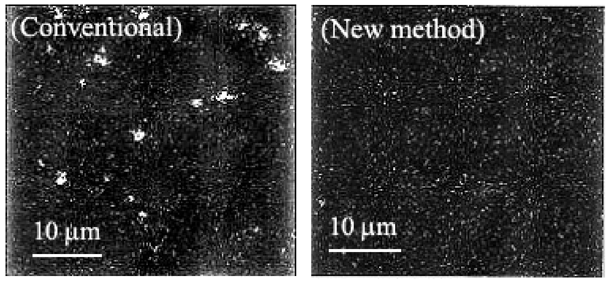

Fig. 4. X-ray dot map of vanadium of WC powder containing VC.

As a grain growth inhibitor of sintering process of ultrafine WC-Co hardmetal, VC is the most effective [2]. In order to more effectively inhibit the WC grain growth during sintering of nano-sized WC-Co hardmetal, some kind of addition technique of VC to the WC powder was examined. In general, several microns VC powder has been mixed when WC powder of the raw material is mixed with the Co powder. (Conventional) in Fig.4 shows dispersibility of VC mixed with the WC powder by the above-mentioned processing. It was observed that VC existed in the particle of the added raw VC powder. On the other hand, (New method) in Fig.4 shows dispersibility of VC added with solution uniformly at direct carburization process. Uniform distribution of VC is clear.

3. Summary

By direct carburization, nano-sized WO_2 and W particle are generated, and carburized to nano-sized WC powder. The agglomerated particles of nano-sized WC powder is the crushed by jet mill, Then using the classification function of the jet mill, the nanosized WC powder is classified into finer and coarse particles. WC powder of 61 nm was obtained by direct carburization and combined jet milling. Uniform dispersibility of VC obtained by new method of addition with solution of vanadium.

4. Acknowledgement

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