Compaction of Ultra-fine WC Powder by High-Speed Centrifugal Compaction Process

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Abstract

High-Speed Centrifugal Compaction Process is one of slip-using compacting method originally developed for processing of oxide ceramics. In this study, we apply the HCP to ultra-fine (0.1 micron) WC powder. Organic liquid of heptane was chosen as dispersing media to avoid possible oxidation of WC. The mixing apparatus was a key to obtain dense compacts. Only the slips mixed by high energy planetary ball mill were packed up to 55% by the HCP, and sintered to almost full density at 1673 K without any sintering aids. This sintered compact marked Vickers hardness of Hv 2750 at maximum.

Keywords : Binder-less WC, Slip, Dispersion, Sintereing performance

1. Introduction

High-Speed Centrifugal Compaction Process (HCP) is one of slip-using compacting method suitable for fine powders. In the HCP, powders in slips are compacted under a huge centrifugal force of about 10,000 g. In the last ten years, authors applied the HCP to alumina and got affirmative results of dense green microstructures, improved sinterability as well as mechanical properties[1-3].

Compacting mechanism of the HCP differs from other slip-using methods (usually based on filtration) [1,2]. The unique compacting mechanism of the HCP leads a number of characteristics such as a higher compacting speed, wide applicability for net shape formation, flawless microstructure of the green compacts [4], etc.

In the present study, we are going to apply the HCP to ultra-fine (0.1μm) WC powder.

2. Experimental

Starting powder and slip preparation

For starting powder, we chose an ultra-fine (0.1μm)pure WC (WC02NR, A.L.M.T. Corp., Japan). An organic liquid of heptane was used as dispersing medium. WC powder was dispersed in 33 mass% of heptane, and 0.1-0.9 mass% of dispersing agent (sorbitan monostearate) is added. Three milling apparatus of (i) conventional ball mill, (ii) turbula mill (WAB Corp., Swiss), and (iii) planetary ball mill (P-6, Fritsch, Germany) were used.

Compaction (HCP)

The slip was poured into aluminum cylindrical tube (φ8×100) die, then rotated at 5,000 rpm for 1 h in high-speed centrifuge (#7820, Kubota, Japan) with a rotor of 120 mm. A green compact of 20 mm high was obtained.

Sintering and evaluation of sintered compacts

The green compacts were heated in vacuum furnace to 773 K and held for 1 h for de-binding, then heated to 1073 K and held for 1 h for pre-sintering. Sintering was also performed by vacuum furnace, at 1623 to 1823 K with a holding time of 1.5 h. No sintering aids, such as Co or Ni, were added.

Vickers hardness was measured from Vickers indentation with a load of 98 N. Fracture toughness (K_{IC}) was also measured from cracks formed around the Vickers indentation with a load of 196 N (IF method). X-ray fluorescence analysis was performed to measure carbon content in sintered compacts. XRD diffraction was also used to determine sintered phases.

3. Results

Effect of milling apparatus on packing density

Packing densities of green compact made with slips milled by distinct apparatus are summarized in Fig. 1. The slips mixed by conventional ball mill or turbula mill are scarcely densified by the HCP. In contrast, the slips mixed by high energy planetary ball realize higher packing density up to 55% in relatively short period of milling (24 h).

Sintered density and microstructure

The green compacts of defferent densites are sintered at 1773 K for 1.5 h. The sintered density of these compacts are shown in Fig. 2 and corresponding microstructures are shon in Fig. 3. The green comapct of the lowest density (a) is scarcely densified, but sintering performances are dramatically improved with denser compacts. However, microstructure of the compact of 45% (b) is much coarser than more denser compacts of (c) and (d). Only dense green compacts realize dense and fine sintered microstructures.

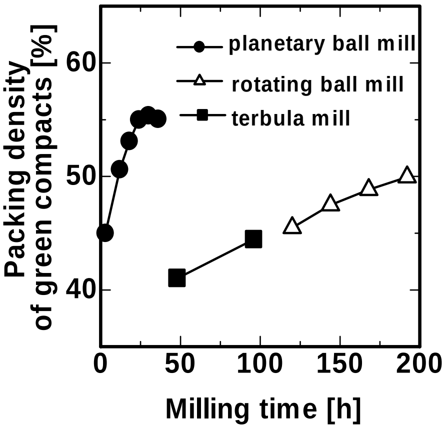


Fig. 1. Packing densities of green compacts with slips prepared by distinct apparatus.

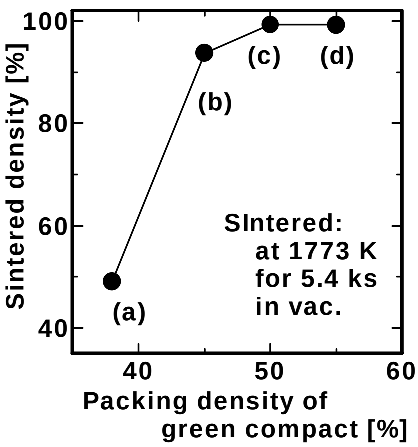


Fig. 2. Sintered densities as a function of packing density of green compacts.

Mechanical properties

Maximum Vickers hardness and fracture toughness are Hv 2750 at 1723 K sintering and K_{IC} 5.8 [MPa $m^{1/2}$] at 1798 K, respectively.

Carbon content in sintered compacts

Carbon content of starting powder is 6.21 % but it decreases to 5.8 % after sintering. This concentration is less than the stoichiometric amount of WC and is hazardous for formation of deteriorative phase. Small peak of W_2C are observed by XRD diffraction. Huge surface area of ultra-fine WC powder may cause decarburization. The absence of bonded phase (usually Co-WC euteutic) also reduces the WC phase band [5]. Severere control of carbon potential during sintering may be required to realize WC single phased material.

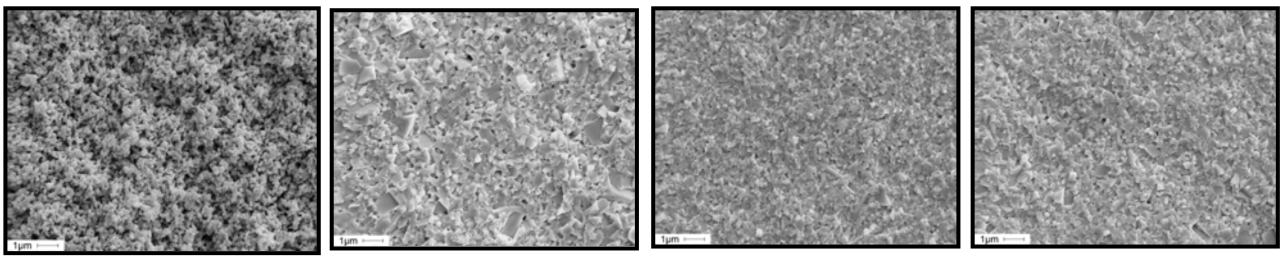
4. Summary

Ultra-fine WC powder was prepared as slip, compacted by High-Speed Centrifugal Compaction Process (HCP) and sintered without any sintereing aids.

For slip preparration, heptane and sorbitan-monostearate were selected as dispersing medium and agent, respectively. Only the slips mixed by high energy planetary ball mill were packed up to 55% by the HCP. Higher green density improves not only densification performance but also reduces concurrent grain growth during sintering. The green body of 55% was sintered to almost full density at 1673 K without any sintering aids. This compact marked Vickers hardness of Hv 2750 at maximum.

5. References

1. H.Y. Suzuki and H. Kuroki: Metal and Mater. Int., Vol. 10 (2004), p. 185-191
2. H. Y. Suzuki, K. Shinozaki, S. Tashima, and H. Kuroki: J. Jpn. Soc. Powder Powder Metallurgy, Vol. 51 (2004), p. 423-434 [in Japanese].
3. H. Y. Suzuki and H. Kuroki: Science of Sintering Current Problems and New Trends, Edited by M. Ristic, Serbian Academy of Science and Arts, Beograd, (2003) p. 29-36.
4. H. Y. Suzuki, K. Shinozaki, and H. Kuroki: Sintering Science and Technology, edited by R. M. German, G. L. Messing, and R. G. Cornwall, Penn-State Univ., (2000) p. 207-212.
5. P. Rautala and J. T. Norton: J. Metals, Vol. 4 (1952) p. 1054.



(a) 39 % **(b) 45 %** **(c) 49 %** **(d) 55 %**

Fig. 3. Fracture surfaces of sintered compacts made from green bodies of different packing densities.