Binder-free Tungsten Carbide Fabricated by Pulsed Electric Current Sintering

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Abstract

In this paper, we show some experimental results of binder-free WC sintered by Pulsed Electric Current Sintering (PECS) also known as Field Assisted Sintering Technology (FAST). These binder-free WC have extremely hardness and stiffness. However, these mechanical properties are dependent on the sintering condition, e.g., maximum temperature, applied pressure, etc. We show some relationship between mechanical properties and sintering condition to improve to sinter the binder-free WC.

Keywords : Tungsten Carbide, micro-die, Binder-free, Pulsed Current Sintering, Field Assisted Sintering

1. Introduction

High precise micro devices are widely used in various fields such as electric appliances, information and communication systems, bio and medical fields and so on. Development of high-performance micro devices is desired to lead to further progress of these applied fields. Therefore, it is very important to establish some micro fabrication techniques for the high precise micro devices.
In order to fabricate high functional micro devices with various raw materials at low cost, one of the suitable ways is to use precise metal die[1]. From the viewpoint of metal die, a tungsten carbide (WC) based cemented carbide is one of the suitable materials for precise micro-dies because of its high strength and high stiffness. However, it is very difficult to make the micro-dies made of WC based cemented carbide in precise. For example, it is very difficult to fabricate the micro-dies with sub-micrometer precision by mechanical manufacturing such as mechanical machining or electric discharge machine (EDM). A new fabrication method using a focused ion beam (FIB)[2] has been reported so far. FIB machining is based on ion sputtering phenomena. From its principles, any materials can be machined finely and precisely [3]. It is well known that for ion sputtering, the surface topography is affected by spatial variation in projectile fluencies resulting from surface scattering, crystallographic effects, atomic diffusion, evaporation, re-deposition of sputtered atoms and so on[4]. The surface roughening of a polycrystalline WC-Co after FIB machining is mainly attributed to the presence of the Co phase because the binding energy of Co is much lower than that of WC[5]. Hence, in order to improve the surface roughness after FIB machining, WC free from metal binders is suitable. However, it is difficult to sinter binder-free or binder-less WC by conventional sintering processes, and hence the pulsed electric current sintering (PECS) process also known as Field Assisted Sintering Technology is needed to sinter binder-free WC[6-8].
In this study, new developed Nano-WC powder were consolidated without metallic binders by PECS, and the density, hardness and toughness of the binder-free WC were investigated.

2. Experimental Procedure

In this paper, a new ultra fine nano particle tungsten carbide powder developed by A.L.M.T. Corp., Japan, was used. The particle size of WC powder was 82 nm.
Sintering was carried out by a PECS system, SPS-2030, produced by Sumitomo Coal Mining Co., Ltd.. In the sintering process, an applied pulsed electric current heats the WC powders directly in a graphite die. This PECS system controls the temperature of the specimen by measuring the surface temperature of a graphite die by a pyrometer. Therefore, this measured temperature was assumed as the sintering temperature. The vacuum was maintained to be less than 10 Pa during the sintering process.
The graphite die was covered with a carbon felt to maintain the heat balance of the die in the consolidation process. Theinner diameter of carbon die is 25 mm, and the weight of WC powder is 100g. Experiments were carried out under various conditions as follows; the maximum sintering

temperature were 1973K, 2013K or 2053K, the holding time of the maximum sintering temperature were 1800 sec., 3600 sec. or 7200 sec.. Applied pressure of the punches was controlled as 40 MPa or 60 MPa in stable during the sintering and cooldown processes. After sintering, the hardness and fracture toughness of sintered WC were measured according to JIS Z2244 and JIS R1607, respectively, by HV-115 Vickers hardness tester produced by Mitsutoyo corp.

3. Results and Discussion

From measurement results of mass density, Vickers hardness at 9.8 N, and plane strain fracture toughness at 9.8 N based on indentation-fracture (IF) of sintered tungsten carbide, the higher sintering temperature, longer holding time and higher applied pressure caused of increment of mass density. Experimental results also shows that the applied pressure is very effective for hardness. The calculated mass density of pure WC is 15.7 g/cm^3 and some sintered WC were over this calculated mass density. Therefore, XRD measurements were carried out to specimens, and these XRD spectra had large peaks of WC and W_2C.
The maximum temperature and the holding time also affected the hardness and the fracture toughness. The hardest binder-free tungsten carbide was obtained at the maximum temperature 2053 K. In case of this high temperature, the shorter holding time would be higher hardness of the sintered WC. On the contrary, in case of the maximum temperature as 2013 K, increase of the holding time was caused of increase of hardness of the WC.
Sintered specimens were classified into two groups based on the fracture toughness. One is over 5.0 with density is under 15.7 g/cm^3, other is around 4.8 with density is over 15.7 g/cm^3. This means that the amount of W_2C affected to the fracture toughness of the sintered WC.
Figure 1 shows the micrographs of fracture surface of the sintered WC at 2053K, holding time was 3600 sec., applied pressure was 60MPa. From this figure, the grain sizes of WC were almost 100 nm - 300 nm andno significant grain growth was observed.

4. Conclusions

Binder-free WC made from the Nano-WC powder werefabricated at the various sintering temperature of 1973–2053 K by PECS process with various sintering conditions. After sintering, density, grain size, hardness and toughness of them were also investigated. The experimental results are concluded as follows,

1. The applied pressure is very important for the densification. Higher applied pressure is caused for the better densification.
2. Vicker’s hardness of the hardest WC sintered at 2053 K with 3600 sec. holding time and applied pressure at 60 MPa, is Hv 2764 in average and some area of the specimen is over Hv 2800.
3. The significant grain growth could not be observed in any sintering conditions.

The binder-free WC with the density over 15.7 g/cm^3 would be obtained under certain sintering conditions, and these WC were more brittle than that of under 15.7 g/cm^3. It is considered that the reason would be the amount of W_2C.

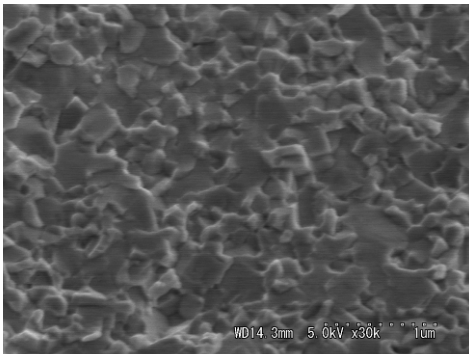

Fig. 1. SEM image of the binder-free WC sintered at 2053K, holding time was 3600 sec., applied pressure was 60MPa.

5. Acknowledgement

The authors gratefully acknowledge the financial support from the project "Integrated Development of Materials and Processing Technology for High Precision Components" by Ministry of Economy, Trade and Industry of Japan.

6. References

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