Paper

Mechanical Properties of Binder-free WC Fabricated by PCS Process

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SYNOPSIS

Binder-free WC was fabricated at the sintering temperature of 1873 K–2023 K by pulsed current sintering (PCS) process, and the density, grain size, hardness and toughness of them were investigated. As a result, the WC sintered at 1973 K showed a very high hardness more than HV10 2700 because it was almost fully dense and no significant grain growth occurred during sintering process.

KEY WORDS

WC, Binder-free, PCS, Ultra fine WC powder

1 Introduction

In recent years, high precision 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 promote further progress of these applied fields. Therefore, it is important to establish micro fabrication techniques for development of the high precision micro devices.

Some micro devices are fabricated by lithography methods developed for semiconductor circuits, such as LIGA¹⁻⁷⁾ processes or electroforming methods. However, these methods need large and special facilities and/or a long time to fabricate precision micro parts, resulting in high cost. Another problem of fabrication of micro devices by these methods is limitation of a raw material of micro devices, that is, some kinds of semiconductors like silicon, some kinds of soft metals like pure nickel and some kinds of functional polymers like PMMA can be only applied to these methods. Thus, it is very difficult to fabricate micro devices which can be used everywhere in long life.

In order to fabricate high functional micro devices made of various raw materials at low cost, one of the suitable ways is to use metal die. For macro devices, fabrication with metal die is an ordinary way to make mechanical parts with various raw materials at low cost. Recently, the micro-forging process has been proposed as a new microfabrication method⁸⁻¹⁰⁾. The microforging process can lead to mass

production of micro devices. To perform microforging of various materials such as polymers, glasses and metals, precise micro-dies made of hard and tough materials are required. From the viewpoint of forming, a 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 difficult to make the micro-dies made of a WC based cemented carbide by LIGA process. Also, it is 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) for metals^{11,12)} has been reported so far. FIB machining is one of the promising micro and nanometer-order manufacturing methods. From its principles, any materials can be machined finely and precisely by FIB process¹³⁻¹⁵⁾. 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^{16,17)}. Recently, it was reported that a polycrystalline WC-Co micro-die was machined by FIB¹⁸⁾. A polycrystalline WC-Co alloy is one of the suitable materials for micro-dies because of high stiffness and high toughness. However, the surface of micro-die fabricated by FIB was badly rough because of the difference of material properties between WC and Co. The smooth surface with

no or little irregularities is required for high precision MEMS (Micro Electro Mechanical Systems) components because a large decrease in mechanical properties of the micro components is often caused by the surface irregularities of the components¹⁹⁾.

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 WC¹⁸⁾. 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 current sintering (PCS) process is needed to sinter binder-free WC²⁰⁻²²⁾.

In this study, fine WC powder were consolidated without binders by PCS at 1873 K–2023 K, and the density, grain size, hardness and toughness of the binder-free WC were investigated.

2 Experimental Procedure

A commercial pure WC ultra fine powder, WC-02N, produced by A.L.M.T. Corp., Japan, was used. The particle size of most WC powder was 0.1 to 0.13 μm , as shown in Fig. 1. The total carbon ratio was $6.20 \pm 0.05\%$, and the free carbon ratio was less than 0.20%. The powder included 1 mass% Cr_3C_2 as grain growth control ingredients.

Sintering was carried out at 1873 K, 1923 K, 1973 K and 2023 K by a PCS system, SPS-2030, produced by Sumitomo Coal Mining Co., Ltd. In the sintering process, an applied pulsed electric current heated the WC powders directly in a graphite die. This system measured the temperature of surface of graphite die by a pyrometer. Therefore, this measured temperature was assumed as the sintering

Fig.1 SEM micrographs of Ultra fine WC powders.

temperature. The graphite die was covered with a carbon felt to control the heat balance of the die in the consolidation process. The vacuum was maintained to be less than 10 Pa during the sintering process. The heating rate was set to be 80 K/min. A pressure of the punches was controlled to be 2 MPa in an initial state and 40 MPa in a holding stage. The holding time was 15 minutes. Diameter of sintered WC is 20 mm and thickness is 5 mm.

The hardness and fracture toughness of sintered WC were measured according to JIS Z2244 and JIS R1607, respectively, by HV-115 Vickers hardness tester Akashi corp.

3 Results and Discussion

Fig. 2 shows the variation in displacement, pressure and temperature with time for the sintering process at 1973 K. It can be seen that shrinkage occurred over 1450 K, in spite of a low pressure under 5 MPa.

Fig. 3 shows the density of sintered WC at the various sintering temperatures. This figure shows that almost the full density of sintered WC can be obtained at the sintering temperature over 1973 K.

Fig.2 Example of sintering condition, where the sintering temperature is 1973 K.

Fig.3 Density of the binder-free WC sintered at various temperatures.

Fig. 4 shows the results of Vickers hardness tests at 98 N and plane strain fracture toughness tests at 98 N based on indentation-fracture method. From this figure, the hardness of the WC sintered at the temperature lower than 1973 K was lower than that of the WC sintered at the temperature over 1973 K. This is probably attributed to the difference in density, namely, there were more pores in the WC sintered at the temperature lower than 1973 K. On the other hand, the fracture toughness tended to decrease with increasing sintering temperature. Cha and Hon reported that the grain growth of WC decreases the fracture toughness²²⁾, hence the difference of the grain size of sintered WC may be a reason to decrease the fracture toughness.

It is noted that the WC sintered at 1973 K exhibited a very high hardness more than HV2700. This is because almost the full density was attained and no significant grain growth occurred for the WC sintered at 1973 K.

Fig. 5 shows the micrographs of fracture surface of the

Fig. 4 Vickers hardness and plane strain fracture toughness of the binder-free WC sintered at various temperatures.

(a)

(b)

(c)

(d)

Fig. 5 SEM micrographs of fracture surface of the binder-free WC sintered at (a) 1873 K, (b) 1923 K, (c) 1973 K and (d) 2023 K.

WC sintered at the various temperatures. From these figures, the grain size of WC sintered at 1873 K and 1923 K was 200 nm – 400 nm and the one at 1973 K was 200 nm – 500 nm. However, the grain size of the WC sintered at 2023 K was 2 or more times larger than that of others, in particular, some large grains over 1 μm were observed. Therefore, significant grain growth in the WC sintered at 2023 K is likely to be responsible for a decrease in hardness shown in Fig. 4.

4 Summary

Binder-free WC was fabricated at the sintering temperature of 1873 K – 2023 K by PCS process, and the density, grain size, hardness and toughness of them were investigated. The experimental results are concluded as follows,

- (1) Sintered at 1873 K and 1923 K, the WC was not fully dense, though the grain size was small.
- (2) Sintered at 1973 K, it was almost fully dense and no significant grain growth occurred during sintering. As a result, a very high hardness more than Hv 2700 was attained.
- (3) Sintered at 2023 K, it was almost fully dense, however, significant grain growth occurred during sintering. The significant grain growth is responsible for a decrease in hardness.

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