Spark Plasma Sintering and Hot Pressing Sintering of Nanocrystalline WC-10Co-0.8VC

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Abstract

WC-10Co-0.8VC nanocrystalline powders were sintered by spark plasma sintering (SPS) and hot press sintering (HPS), and the microstructure and properties were compared. Results show that dense WC-10Co-0.8VC can be obtained by SPS in several minutes when the sintering temperature is >1200 °C. Sintered at a temperature of 1300 °C, the sample prepared by SPS for 3 minutes has higher density, finer grains and better properties than that prepared by HPS for 60 minutes. SPS can be used to prepare nanocrystalline WC-10Co-0.8VC with improved properties when suitable sintering parameters are chosen.

Keywords: nanocrystalline material, mechanical alloying, spark plasma sintering

1. Introduction

Nanostructured WC–Co cemented carbides have received increasing attention because of superior properties \([1,2]\). However, it is difficult to prepare nanostructured WC–Co since nanocrystalline WC tends to coarsen during sintering due to high interface area between WC particles and cobalt phase. From the viewpoint of the growth retardance, a decrease in sintering time is more effective than the addition of grain growth inhibitors, since an additional 30s results in an increase in WC grain size from 0.2 um to 2 um when WC-10Co is heated at 1400 °C[3]. Spark plasma sintering (SPS) is a novel powder consolidation method by which materials can be densified in only several minutes at relatively low temperatures. In this paper, WC-10Co-0.8VC nanocrystalline powders were sintered by SPS to investigate whether it was possible to prepare nanocrystalline cemented carbides, and the microstructure and properties were compared with hot press sintering (HPS).

2. Experimental and Results

WC-10wt%Co-0.8wt%VC nanocrystalline powders (average grain size of WC: 11 nm), were sintered by SPS at 1200 °C for 5 minutes and 1300 °C for 3 minutes. For comparison, WC-10Co-0.8VC powders were also hot press sintered at 1300 °C for 60 minutes. The densities of samples were measured by the Archimedes method. The microstructures of polished and etched sample surfaces were examined using an S-570 scanning electron microscope (SEM) and H-11 II scanning probe microscopy (SPM).

The Vickers hardness was measured on polished surfaces using AKASHI AVK-A under 30kgf load. Fracture toughness was calculated from the length of cracks produced by the Vickers indentation, according to Eq.1 \([4]\)

$$K_{IC} = 0.0889(HP/L)^{1/2} \quad (1)$$

where H is the hardness, P is the applied load and L is the total length of cracks produced by the Vickers indentation. After subtracting the expansion of the graphite die, the relative shrinkage of nanocrystalline WC-10Co-0.8VC powder with temperature during SPS is shown in Fig.1. Shrinkage is sensitive to temperature and most of the shrinkage takes place before 1200 °C. 97.7% and 95.1% of theoretical density can be obtained by SPS at 1300 °C for 3 minutes and at 1200 °C for 5 minutes, respectively. Sintered at the same temperature of 1300 °C, the sample prepared by SPS for only 3 minutes has higher density than that prepared by HPS for 60 minutes. Since quick densification can be realized by SPS in several minutes, this should make it possible to restrain the grain growth of WC during sintering.

SEM observation shows that sintered at 1300 °C, the sample prepared by HPS has bigger WC grains than that by SPS owing to the longer sintering time. The decrease of spark plasma sintering temperature is more beneficial to retard the grain growth of WC during sintering, thus the sample by SPS at 1200 °C has a more uniform and finer microstructure than at 1300 °C. It is worth noticing that some exaggerated WC grains >1um are still observed on the samples by SPS at 1300 °C and 1200 °C. Considering that WC-10Co-0.8VC nanocrystalline powders prepared by
mechanical alloying are very active, WC grains grow quickly and easily during sintering.

Fig. 1. The relative shrinkage of nanocrystalline WC-10Co-0.8VC with temperature during SPS

Fig. 2 shows SPM micrographs of WC-10Co-0.8VC prepared by SPS. The size of most WC grains in the by SPS at 1200°C is smaller than 100nm and, likewise, about 50% of the sample at 1300°C. Hence, nanocrystalline WC-10Co-0.8VC cemented carbides can be obtained by SPS if suitable sintering temperature and time are chosen.

Fig. 2. SPM micrographs of WC-10Co-0.8VC prepared by SPS (a) 1300°C-3min, (b) 1200°C-5min

The properties of samples prepared by SPS and HPS are compared in Fig. 3. In spite of the same sintering temperature of 1300°C, the sample prepared by SPS possesses higher hardness and fracture toughness than that prepared by HPS owing to the finer grains. The properties further improve when most of the WC grains are smaller than 100 nm. The sample prepared by SPS at 1200°C has a HV30 of 1887, and $K_{IC}$ of 11.5 MPam$^{1/2}$, although it has a lower density value than the sample prepared by SPS at 1300°C. This work indicates that SPS is a promising consolidation technique for the preparation of nanocrystalline WC-10Co-0.8VC with improved properties.

3. Summary

Dense WC-10Co-0.8VC can be obtained by SPS in several minutes when the sintering temperature is >1200°C. SPS can be used to prepare nanocrystalline WC-10Co-0.8VC with improved properties when suitable sintering parameters are chosen.

4. References