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Mechanical properties and erosion resistance of ceria nano-particle-doped ultrafine WC–12Co composite prepared by spark plasma sintering

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ABSTRACT

Ultra-fine grained WC–Co composites possess higher hardness or strength and wear resistance, compared to their coarse counterparts. However, abnormal grain growth of ultra-fine WC particles often occurs during the traditional pressureless liquid sintering, which substantially impairs the mechanical properties and erosion resistance of the material. Thus, controlling the abnormal grain growth becomes one of the key issues in fabricating ultra-fine grained cemented carbides. In this study, ultra-fine grained WC–Co composites containing different amounts of ceria nano-particles were prepared by spark plasma sintering and effects of the nano-ceria on mechanical properties and erosion behavior of the WC–Co composites were studied. The results demonstrated that trace nano-ceria addition effectively suppressed the abnormal grain growth of WC, leading to uniform and fine microstructures. Such ultra-fine grained WC–Co composites have both improved hardness and fracture toughness, resulting in enhanced resistance to high-speed solid-particle erosion. However, when the added nano-ceria was more than 0.1 wt%, Co pools started to form, which lowered the material density, hardness and toughness and consequently the erosion resistance.

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1. Introduction

WC–Co composites have been widely used for machining, cutting, mining and drilling tools, due to their high hardness and excellent wear resistance [1]. When the WC grain size is reduced to submicron or nanometer scales, the WC–Co composites possess improved mechanical properties and wear resistance, compared to their coarse-grained counterparts [2]. Thus, ultrafine-grained and nanostructured WC–Co composites have attracted great interest in the field of high-performance hard materials [3]. However, abnormal grain growth of ultrafine WC particles in WC–Co composite occurs easily during the traditional pressureless liquid sintering, which substantially deteriorates their performance. Thus, controlling abnormal grain growth has become one of the key issues in fabricating ultrafine WC–Co composites [4].

The grain growth can be inhibited to a certain extent by using special sintering technologies with accelerated heating rate, increased densification rate, decreased sintering temperature and short holding time [5], such as microwave sintering [6], rapid hot pressing sintering [7], and spark plasma sintering (SPS) or pulse electric current sintering (PECS) [8]. In particular, SPS is a novel consolidation process that combines fast joule heating, established by flowing a pulsed electric current through the die/punch/powder compact set-up under an applied pressure. The fast densification process is reported to be benefited from grain boundary diffusion, surface diffusion, volume diffusion, and plastic deformation [9]. During past few years, SPS process was successfully used to synthesize ultra-fine/nano-WC–Co composites [10–12].

Although many studies are focused on the development of sintering techniques, one of the basic and successful ways for controlling the WC grain growth is the addition of small amounts of WC grain growth inhibitors, such as VC, Cr_3C_2, NbC or Mo_2C, to the starting powder mixture, typically less than 1.0 wt% of a metallic carbide [13]. Vanadium carbide (VC) and chromium carbide (Cr_3C_2) are the most effective grain growth inhibitors due to their high solubility and mobility in the cobalt phase at lower temperatures [14,15]. For all transition metal carbides, however, the mechanism for grain growth inhibition has been related to the slowing down of the solution/re-precipitation reactions at the WC–Co interfaces [16]. These carbides are effective in preventing anomalous grain growth but the drawback is that they may cause the product to be brittle, especially for vanadium carbide [15]. Rare earths and their oxides are effective inhibitors in WC–Co composites [17–19], which may increase the wetting power of Co to WC grains, control the formation of η

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phase in cemented carbides, raise the bending strength and wear-resistance, and also increase the corrosion resistance of cemented carbides [20]. The previous studies [19] on traditional rare earth as additives, have shown that the rare earths improve mechanical properties of WC–Co and optimal performance is observed when the content of added rare earths or their oxides such as CeO_2 is more than 1.0 wt% [19–21]. The high melting point of ceria (2673 K) makes it in a solid state during sintering of cemented carbides even using the conventional liquid sintering method in the temperature range of 1723 K. CeO_2 particles cannot diffuse effectively during the sintering process, so that the positive effect of rare earths would be minimized when their primary particle size is at or above micron scale. Further, the segregation of rare-earth oxide particles at boundary would weaken their positive effect and could even be detrimental. The unstable performance due to the non-uniform distribution of rare-earth oxide particles in cemented carbide and the high cost limited their industrial applications.

Our previous study showed that nano-CeO_2 is much more effective in cemented carbides and the mechanical properties of WC–Co composites are optimized with only 0.1 wt% nano-CeO_2 [3]. To our knowledge, no studies on the WC–Co composites with nano-CeO_2 by other researchers have been reported in the literature. In this study, ultra-fine grained WC–Co composites containing different amounts of CeO_2 nano-particles were prepared by spark plasma sintering and effects of the nano-CeO_2 on mechanical properties and erosion behavior of the WC–Co composites were studied.

2. Experimental

2.1. Materials preparation

Ultrafine WC powder ($\geq$99.5%, ~200 nm, free carbon 0.11 wt%, shown in Fig. 1a) and Co powder ($\geq$99.6%, ~60 nm) were used as raw materials. A small quantity (0.05–0.6 wt%) of nano-CeO_2 powder (~10 nm, shown in Fig. 1b) was added to WC–12Co composite powder. The compositions of samples with different amounts of nano-CeO_2 are shown in Table 1. The ultrafine WC–12Co composite powders were wet ball-milled for 0.5 h using ethanol as a medium in a high vibration ball milling machine. The ball material was tungsten and the volume ratio of the ball to the material was 5:1. The powders were dried at 333 K in an oven for 10 h.

The mixed powders were then sintered using an SPS apparatus (SPS1050, Sumitomo Coal Mining Co., Ltd., Tokyo, Japan). The schematic of SPS apparatus was shown in Fig. 2 [22].The powders were put into cylindrical graphite dies with an inner diameter of 30 mm. The temperature was monitored using an optical pyrometer aimed at a non-through hole of 0.5 mm in diameter and 2 mm in depth on the graphite die. The heating rate was 100 K/min, and a pressure of 50 MPa was applied during sintering. The sintering temperature was set at 1473 K with a holding time of 5 min. During the consolidation process, the dimensional changes of powders were monitored by measuring the position of the lower ram, which acted as an electrode to produce high pulsed current within the powder compact.

2.2. Characterization

The phase constitution of the composites was determined using a Rikagu X-ray diffractometer with Cu K_α radiation. A field

Table 1
Compositions of WC–12Co composites for this study.

No.	WC	Co (wt%)	CeO_2 (wt%)
1	Balance	12	0
2	Balance	12	0.05
3	Balance	12	0.1
4	Balance	12	0.3
5	Balance	12	0.6

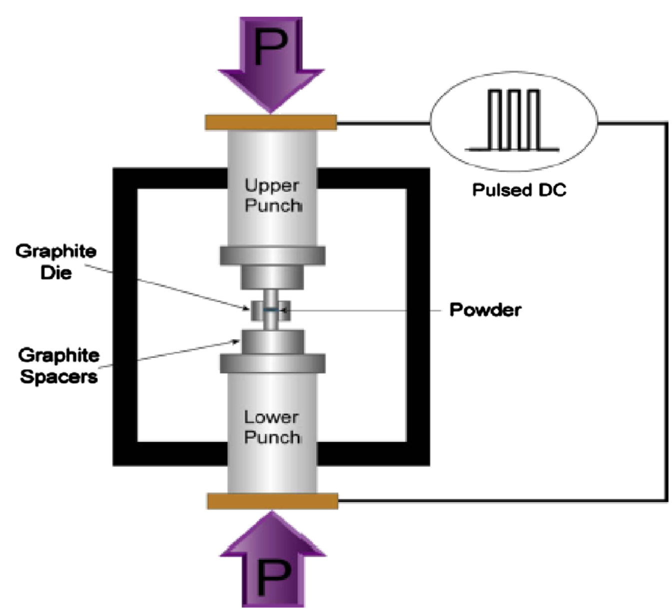

Fig. 2. Schematic illustration of SPS apparatus [19].

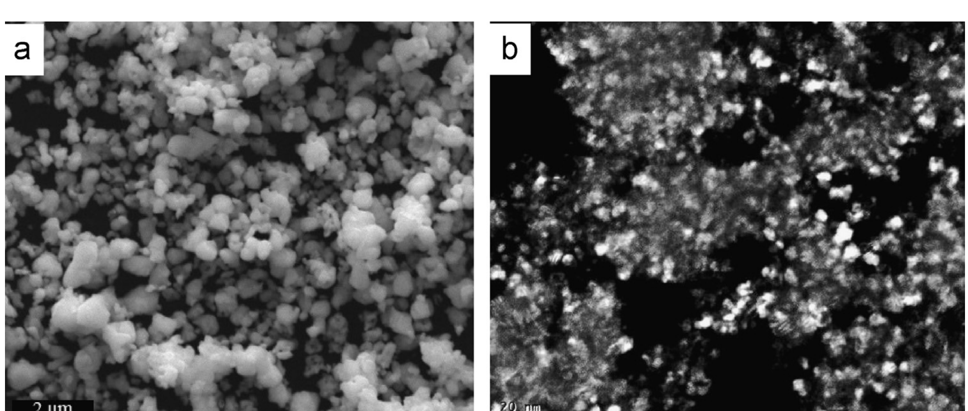

Fig. 1. SEM image of WC powder (a) and TEM image of nano-ceria powder (b).

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