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ScienceDirect
Ceramics International 34 (2008) 2043–2049
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

# Oxidation of ultrafine-cemented carbide prepared from nanocrystalline WC–10Co composite powder

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Received 26 April 2007; received in revised form 5 July 2007; accepted 26 July 2007
Available online 19 August 2007

**Abstract**

The oxidation behavior and associated properties, phases and microstructure of ultrafine WC–10Co-cemented carbide using WC–10Co nanocomposite powder prepared by spray pyrolysis-continuous reduction and carbonization technology, were investigated in the 450–700 °C temperature range at 50 °C intervals. The results showed that the working temperature of the cutting edge should be lower than 550 °C in air without coolant in order to assure the lifespan and working efficiency of ultrafine WC–10Co-cemented carbide materials as cutting tools.


*Keywords:* WC–10Co; Nanocomposite powder; Ultrafine-cemented carbide; Oxidation; Working temperature

## 1. Introduction

WC–Co cemented carbides are universally used for miniature drills for printed circuit boards, pins for dot-printers, wood machining, dental work, cutting tools, rock drill tips and other wear resistant parts due to their unique combination of hardness, toughness and strength [1–3]. Recently, ultrafine WC–Co cemented carbide with both high hardness and high strength are demonstrating their excellent potentialities [4–6]. In most circumstances, hardness directly indicates the wear resistance of tool. Thus, the harder the grade, the higher the wear resistance of the cutting edge. In cemented carbide for metal cutting purposes, the quality of a cemented carbide grade is dictated substantially by its high-temperature properties, and the lifespan and working efficiency of ultrafine WC–Co cemented carbide materials as cutting tools in air are strongly depending on its oxidation resistance.

In recent years, researchers have made concentrated efforts to study the high-temperature hardness and wear characteristics of WC–Co cemented carbide. The hardness of a simple WC–10Co-cemented carbide at 800 °C will feature only about one third of that at room temperature, whereas the specimen containing additions of TiC and (Ta, Nb)C will feature about half of its hardness at room temperature [7]. Luyckx S [8] investigated the high-temperature hardness of WC–Co–Ru from room temperature to 900 °C in high vacuum. The data available on the high-temperature hardness of WC–Co–Ru was also found in a patent [9]. A wear test between cemented WC–Co alloy and carbon steel was conducted to observe the wear behavior of cemented WC–Co alloy at 400 °C by Marui E et al. [10]. Hegeman JBJW et al. [11] observed the micrographs of polished and heat-treated WC–10Co specimens in high vacuum with subsequent slow cooling. Acchar W et al. [12] researched the transverse rupture strength (TRS) measured in three point-bending in air at temperatures between room temperature and 1000 °C. Basu SN et al. [13] investigated the oxidation behavior of coarse WC–Co samples in flowing Ar–$O_2$ gas mixtures at 600 °C, 700 °C and 800 °C, respectively. Casas B et al. [14] studied the oxidation-induced strength degradation of WC–Co hardmetals containing different binder phases with the same carbide mean grain size (2.5 μm) at 700 °C in air.

However, the oxidation resistance of ultrafine WC–10Co-cemented carbide without additives in air is seldom touched. In this paper, an investigation was carried out to study the oxidation resistance of ultrafine WC–10Co-cemented carbide based on WC–10Co nanocomposite powder prepared by spray pyrolysis-continuous reduction and carbonization technology.

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0272-8842/$34.00 
doi:10.1016/j.ceramint.2007.07.029

The microstructure, phases and properties of the oxidized cemented carbides were investigated.

## 2. Experimental

WC–10 wt.%Co nanocomposite powder without additives, produced by spray pyrolysis-continuous reduction and carbonization [1], was used for this study. The powder was ball-milled in acetone for 48 h, and dried at 90 °C in vacuum oven. The particle size was characterized by a Brunauer–Emmet–Teller (BET) analyzer. The green compacts were consolidated in a vacuum sintering process at 1380 °C for 60 min. Sintered specimens, 25 mm × 10 mm and a thickness of 10 mm, were characterized for microstructure and grain size by a JSM-5610LV scanning electron microscopy (SEM). To investigate the oxidation behavior, differential scanning calorimetry/thermogravimetric (DSC/TG) (Netzsch STA 449C Simultaneous Thermal Analyzer) analysis was applied in air with a heating rate of 10 °C/min. Before oxidation, the sintered samples were polished with diamond pastes of 6 μm, 3 μm and 1 μm, respectively, to obtain a smooth surface. After each sequential polishing step, the surface was cleaned with acetone. Then, the samples were oxidized in the temperature range of 450–700 °C at 50 °C intervals for 4 h in muffle furnace in air with subsequent slow cooling.

The micrographs of the oxidized surface layer and specimen microstructure were investigated by SEM. The phases in the oxidized surface layer were characterized by X-ray diffraction (XRD). In order to remove the oxidized surface layer before the Vickers indentations were made, the samples were polished mechanically with emery papers down to grade 1200, and with 0.05 μm wet polishing diamond pastes, the surface was also cleaned with acetone. The Vickers hardness (3 kg) was measured at room temperature with a micro-Vickers hardness tester, and the Rockwell A hardness (HRA) (50 kg) was also measured. The TRS was measured with an MTS-810 Teststar Iis Electro-Hydraulic Servocontrolled Testing System. The Vickers hardness indentations were observed by an optical microscopy. Saturated magnetization and coercivity force were measured by a saturation induction measuring system and a förster-koerzimat 1.095, respectively.

## 3. Results and discussion

The properties of the WC–10Co nanocomposite powder are summarized in Table 1. The specific surface area of the nanocomposite powder was 5.12 $m^2\ g^{-1}$ and the equivalent mean particle size was about 80 nm.

Table 1
Properties of WC–10Co nanocomposite powder

| | |
|---|---|
| Total carbon content [wt.%] | 5.54 |
| Free carbon content [wt.%] | 0.18 |
| Oxygen content [wt.%] | 0.25 |
| Cobalt content [wt.%] | 10.08 |
| Specific surface area [$m^2\ g^{-1}$] | 5.12 |

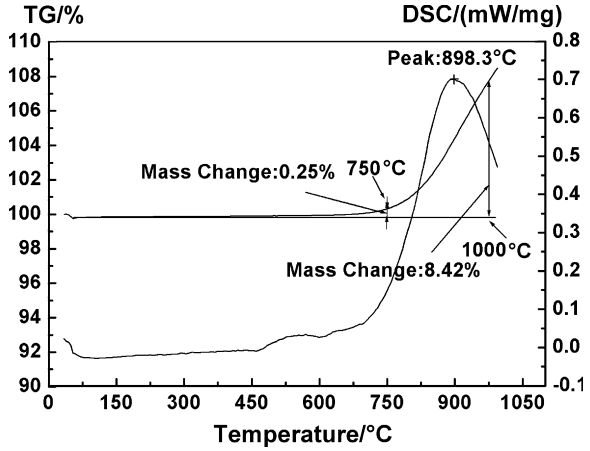


Fig. 1. DSC/TG pattern of ultrafine WC–10Co-cemented carbide from room temperature to 1000 °C in air.

The DSC/TG pattern of ultrafine WC–10Co-cemented carbide from room temperature to 1000 °C in air is shown in Fig. 1. Only one peak was observed in the DSC curve. The ultrafine WC–10Co-cemented carbide began to oxidize at about 425 °C in air. An obvious exothermic peak appeared at 898.3 °C, which can be explained by furious oxidation [15]. As shown by the TG curve, the mass increment was 0.25 wt.% from room temperature to 750 °C, which corresponds to a limited oxidation of cobalt and WC. The mass increment of 8.42 wt.% from 750 °C to 1000 °C corresponds to a prominent oxidation of Co and WC.

As shown in Fig. 2, the surface layers of WC–10Co-cemented carbide after oxidation processes at 450 °C, 500 °C, 550 °C, 600 °C, 650 °C and 700 °C, all contained $WO_3$, $Co_3O_4$ and $CoWO_4$ phases.

As shown in Fig. 3, the oxidation layer thickness was 39.1 μm, 83.1 μm, 117 μm, 619 μm, 1.25 mm and 2.12 mm, respectively. The oxide layer thickness increased with increasing oxidation temperature. When the oxidation temperature reached 700 °C, the thickness of the oxidized surface layer was 2.12 mm.

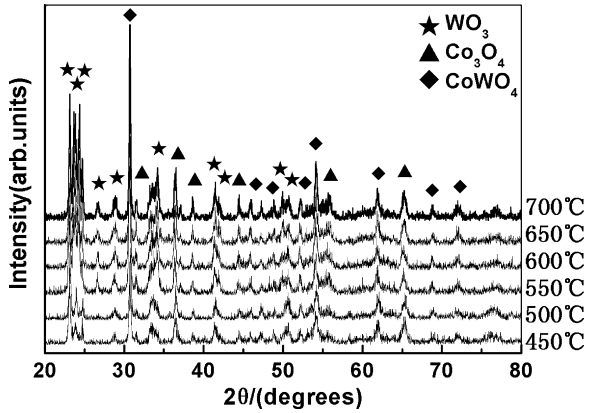


Fig. 2. XRD patterns of the surface of the ultrafine WC–10Co-cemented carbide, oxidized at different temperatures.