

Two-step hot-pressing sintering of nanocomposite WC–MgO compacts

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Abstract

Two-step hot-pressing sintering (TSS) was applied to consolidate nanocomposite tungsten carbide–magnesia (WC–MgO) powders. The first step sintering was employed at a higher temperature to obtain an initial high density, and the second step was held at a lower temperature by isothermal sintering for several hours to increase bulk density without significant grain growth. The experimental results showed the sintering temperature plays an important role in densification and grain growth of WC–MgO compacts. The optimum TSS regime consisted of heating at 1750 °C (1st step) and 1550 °C (2nd step), resulting in the formation of near full dense microstructure (0.99 TD) with suppressed grain growth (2.59 μm). Accordingly, the improvement on the mechanical properties, including increase in the hardness (from 16.7 to 18.4 GPa), fracture toughness (from 10.2 to 12.95 MPa m^{1/2}) and flexural strength (from 976.6 to 1283.7 MPa), was also observed due to the grain refining and full dense bulk. © 2011 Elsevier Ltd. All rights reserved.

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1. Introduction

Pure tungsten carbide (WC) powders are usually sintered in the presence of binding materials (typically Co, Fe or Ni) in the liquid phase by various techniques. Metallic binder is introduced to improve WC interparticle binding and to increase the compact toughness. However, metallic binders result in reduced hardness and corrosion/oxidation resistance,¹ and enhance grain growth, particularly in conventional liquid phase sintering due to rapid diffusion in the liquid phase.² Therefore, efforts to obtain harder materials have attempted the use of hot-pressing to consolidate WC with low amounts of Co³ and WC with no metal binder.^{4–6}

Among them, a new composite material, WC–MgO is considered as an ideal material for use in industrial applications. Compared with the commercial micron- and submicron-grained structure WC–Co composites, the WC–MgO can achieve superior high value of hardness and toughness combination.⁷ Researches^{8–11} have been focused on the synthesis of the pow-

ders, but fewer on its consolidation details. We have recently attempted to prepare this MgO particulate toughened WC matrix composite using hot-pressing sintering method. It has been found that the sintering activity and densification response of the hot-pressing processed WC–MgO were not as good as those consolidated *via* spark plasma activated sintering (PAS). The PAS method has yielded such benefits as a rapid sintering rate, high densification, and fine grain size production. Nevertheless, PAS method is hardly accessible and scarcely used for practical applications.

Fortunately, the experimental results revealed that the aforementioned problems can be well-resolved by adding the rare earth (RE) oxide in the elemental powder system. In our previous works, La₂O₃ was selected as the addition to WC–MgO during its bulk synthesis process. The results indicated that the La₂O₃–WC/MgO compact can achieve the high relative density and homogenous microstructure consisting of small grains.¹² However, the effectiveness of the additives greatly depends on the homogeneity of their distribution. While excess La₂O₃ was added to WC–MgO, these particles located at grain boundaries may result in the increasing of the grain boundary width. It may cause technique problems for the fabrication of uniform, dense and ultrafine structure.

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Another way to control the grain growth during consolidation is to use novel processing technique to tailor the microstructure. In recent years, a two-step sintering (TSS) method has been proposed to achieve the densification of ceramic bodies without significant grain growth in the final stage of sintering.¹³ The TSS procedure consists of the following steps: (1) reaching a high temperature (T_1) to conduct first-step sintering until the relative sample density $>75\%$ theoretical density (TD) is achieved; (2) lowering the temperature to T_2 to conduct second-step sintering during which there is only densification and no grain growth.^{13,14} So far, the TSS method has been successfully applied to the sintering of Y_2O_3 ,^{13,15} Ni–Cu–Zn ferrite,¹⁶ $BaTiO_3$,^{17–19} Al_2O_3 ,^{20,21} and liquid-phase sintering of SiC ²² as well as doped ZnO varistors.²³

According to the reported works, achieving a high density and a small grain size is very important for the structural ceramic materials because it can bring about an improvement of mechanical properties such as hardness, wear resistance, strength or fracture toughness.²⁴ In this work, two-step sintering is applied on the preparation of high-density WC–4.3 wt% MgO compact with refined grains. Preliminary investigation on the bulk density and microstructural evolution during constant-heating-rate and isothermal sintering are carried out in order to determine the appropriate heating temperatures T_1 and T_2 . Thereafter, the suggesting temperatures T_1 and T_2 are practiced in the present work, and the effects of the applied TSS process on the structural properties of WC–MgO are discussed. A comparison is also made of the mechanical properties of the compacts made by TSS technique with those of the normally hot-pressing sintered ones.

2. Experimental

Experiments consisted of synthesis, characterization, sintering and mechanical properties determination of WC–MgO samples according to the following methods.

2.1. Powder synthesis and characterization

Nanocrystalline WC–4.3 wt% MgO was synthesized via mechanical alloying process in a QM-1SP4 high energy planetary ball mill machine. The starting materials were WC (75 μm , 99.5% purity) and MgO (48 μm , 98.5% purity). The ball mill process was carried out under argon gas atmosphere at a rotational speed of 350 revolutions per minute (rpm) for 50 h. The ball-to-powder weight ratio was 10:1. The milling balls (10 mm in diameter) and vial were made of cemented carbide materials.

The morphologies of the as-milled powders were investigated by transmission electron microscope (TEM) on a JEOL JEM-2100F electron microscope. The phases identification were observed by X-ray diffraction (XRD) on a D/max-2550 PC (Rigaku Co., Japan) X-ray diffractometer with a Cu K_α radiation ($\lambda = 0.154$ nm) at 40 kV and 300 mA. The specific surface area of the powders was determined using Brunauer–Emmett–Teller (BET) surface area analyzer (Autosorb-1, Quantachrome, United States). The particle size of the powders was measured

by dynamic laser light scattering method (DLS, BI-200SM, Brookhaven, United States).

2.2. Conventional hot-pressing sintering and two-step hot-pressing sintering

Sintering of the samples was implemented by conventional hot-pressing sintering (CS) and two-step hot-pressing sintering (TSS). The as-milled composite powders were hot-pressed in a graphite die at 1450–1900 °C in a vacuum of 1.3×10^{-1} Pa with 50 °C temperature intervals and a heating ramp of 10 °C/min. The specimens were held at the highest temperature for 3 min with the pressure of 39.6 MPa in order to obtain a uniform temperature throughout the sample. The consolidated bulks with a dimension of $\varnothing 15 \times 15$ mm³ were obtained for the properties tests. For the first step of TSS, samples were heated under the same conditions as conventional sintering. The cooling rate of TSS between T_1 and T_2 was 60 °C/min. The samples were held at T_2 up to 8 h with the pressure of 39.6 MPa so as to reach a higher density.

The densities of the sintered bulks were determined by Archimedes' principle, using deionized water immersion, with an analytical balance. Comparing this value with that calculated from the rule of mixture principle, the relative density of the compacted bulks was achieved. The samples were ground and polished by standard ceramographic methods and then etched in a Murakami's reagent consisting of $Fe_3[K(CN)_6]$ (10 g), KOH (10 g) and distilled water (100 ml) for 5 min to expose the grain boundaries. The microstructure of the bulks was observed by scanning electron microscope (SEM, S-4800, Hitachi Co., Japan) of the polished and etched surface. The grain size was estimated by the linear intercept method. For each sample, at least three images were taken of the microstructure; in each image a minimum of five line segments were assessed. The particulate/matrix interfacial microstructures were characterized by scanning probe microscope (SPM, NanoScope IV, United States).

2.3. Mechanical properties

The hardness was determined using a HVS-50Z Vickers indenter with a load of 30 kg and an indentation time of 10 s. The resulting indentation cracks were used as an indication of the fracture toughness (K_{IC}) of the sample.²⁵ We should note that the Vickers hardness measurements that were made are a rough approximation for the valid K_{IC} test. Hence, they were an approximation of the expected fracture-toughness measurements, not an absolute determination. At least ten indentations were made and the average value was obtained. Flexural strength of sintered samples was measured using three-point flexure test according to the ASTM B312 standard. Sample bars were cut from the sintered pellets and then ground and polished into 15 mm $\times$ 5 mm $\times$ 3 mm specimens. Three-point flexure tests were carried out at room temperature with a span of 12 mm and cross-head speed of 0.01 mm/s. The fracture strength of consolidated samples (the average of five tests) was calculated

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