



Fabrication of tungsten carbide foam through gel-casting process using nontoxic sodium alginate



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ABSTRACT

In this study, nontoxic sodium alginate was utilized in gel-casting process to fabricate tungsten carbide (WC) micro-porous foam. Suspensions containing 20 and 25 vol.% WC and 1 wt.% sodium alginate were used. Calcium phosphate and sodium hexa-metaphosphate were employed as solidifier agent and chelator, respectively. The green bodies were dried at room temperature for 36 h and pre-sintered at 1450 °C for 4 h. The influence of chelator and calcium salt on strength of dried green body was evaluated. Tungsten carbide foams with 50–60% porosity were successfully produced. SEM micrographs of tungsten carbide foams show a uniform porous micro-structure, with average size of 0.75 μm.

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1. Introduction

Macroporous ceramics have found many applications in various industrial processes, including thermal insulation, catalytic reaction supporter, and filtration of molten metals, exhaust gases, and hot corrosive gases [1]. These porous media were also employed to fabricate various types of metal matrix composites via liquid metal infiltration method [2]. High melting point, high corrosion and wear resistances are their outstanding properties, in combination with the features gained via replacement of solid material by voids in the components [1]. Such macroporous ceramics exhibit pore width larger than 50 nm, and are produced by various methods such as replica, sacrificial template and direct foaming. The sacrificial template technique usually consists of a continuous matrix of ceramic particles or ceramic precursors and a dispersed sacrificial phase. The biphasic composite is prepared by forming a two phase suspension that is subsequently processed by wet colloidal routes [1].

Gel-casting is also widely applied to the fabrication of porous and complex-shaped ceramics (e.g., microbeads, rutile capacitor, thin-wall rutile tube, refractory nozzle). The gel-casting process was first developed in the Metals and Ceramics Division–Ceramic Processing Group at Oak Ridge National Laboratory (ORNL), Oak Ridge, USA by Omatete and Janney during the 1990s [3]. The in situ free radical polymerization of acrylamide monomers, which originally developed for dense ceramics, was successfully used for setting ceramic foams of various compositions by Binner and Sepulveda [4,5], however, the neural toxicity of the acrylamide monomers, which are commonly employed in the process, limits their

applications [6]. Many researchers studied nontoxic or low toxicity polymers in the process. The temperature or pH-induced gelation of gelatin, ovalbumin, and bovine serum albumin, for instance, and temperature-induced gelation of polysaccharides such as sucrose, agar, and carrageenan gum have been recently applied as nontoxic processing route to fabricate porous ceramics [1]. Alginate is a type of gelling polysaccharide which is soluble in water at room temperature and gels after reaction with divalent metal ions [7]. The result is construction of a three dimensional (3D) network, suspending ceramic particles over the network [8].

Several reports have been presented on gel-casting of alumina and silicon carbide using sodium alginate [6–11], while fabrication of high density tungsten carbide via gel-casting route remains a serious challenge, as its density (15.8 g/cm³) [12] is 4–5 times of alumina (3.96 g/cm³) [7] and silicon carbide (3.22 g/cm³) [11] densities, and its suspension over a 3D network appears to be troublesome. In the present work, it is attempted to fabricate tungsten carbide foam via a nontoxic gel-casting route, and a natural innocuous polymer–sodium alginate was then employed to coagulate the tungsten carbide suspension. The resulted micro-porous green body was characterized by SEM, TG/DTA and XRD tests and mechanical tests were carried out to evaluate its green strength.

2. Experimental procedure

2.1. Materials

Tungsten carbide with purity of 99.6 wt.%, particle size range of 0.2–10 μm and mean particle size of 0.9 μm, was employed in the present study. The morphology and particle size distribution of tungsten carbide

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powder were shown in Figs. 1 and 2, respectively. The particle size distribution of the powder was determined by particle size analyzer (FRITSCH Particle Sizer Analysette 22).

2.2. Procedure

Controllable gel-casting and solidification of the suspensions become possible through the use of appropriate amounts of sodium alginate, the solidifier agent, the chelator and the hexanedioic acid, as listed in Table 1.

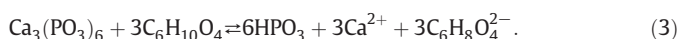
Sodium alginate undergoes chemical gelation to form a 3D network in the presence of multivalent cations (e.g., calcium). The irreversible gels can form very quickly through reaction between calcium ions and sodium alginate, as shown in Eq. (1) [13].



Gelation occurs during casting with a fast gelation rate and thus results in various cross-linking densities and heterogeneity within the gel [13]. The reaction rate of the gelation is controlled by chelator ($(\text{NaPO}_3)_6$) and initiator ($\text{C}_6\text{H}_{10}\text{O}_4$). The gelation reaction is delayed with increasing chelator because the free calcium ions in the solution were limited due to the chelation [6]. A stable complex forms from the reaction between chelator and calcium salt. It allows chelated calcium ions to be uniformly distributed in the alginate solution and prevents immediate gelation, as shown in Eq. (2) [10]:



Upon adding hexanedioic acid ($\text{C}_6\text{H}_{10}\text{O}_4$) to the ceramic suspensions, the complex decomposes and gradually releases calcium ions, in which subsequently the ions react with sodium alginate, and gelation occurs, according to the following equation [10]:



Flow chart of the forming process is illustrated in Fig. 3. First; sodium alginate was dissolved in the deionized water; then tungsten carbide powder, dispersant (ammonium citrate tribasic – $(\text{NH}_4)_3\text{C}_6\text{H}_5\text{O}_7$), calcium salt ($\text{Ca}_3(\text{PO}_4)_2$) and chelator (sodium hexa-metaphosphate – $(\text{NaPO}_3)_6$) were added to the solution, and the resulting suspension

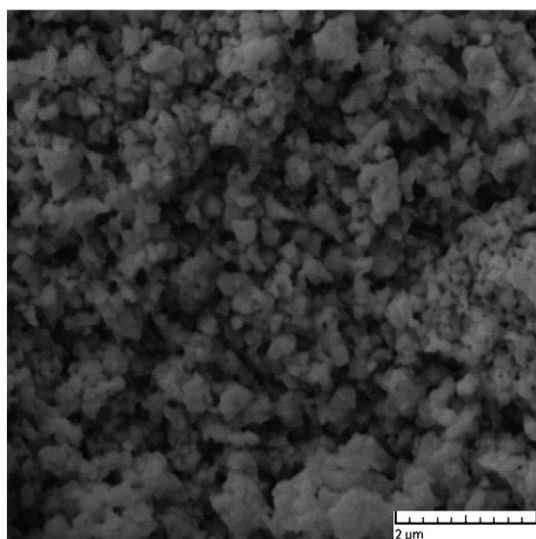


Fig. 1. SEM micrograph of WC powder.

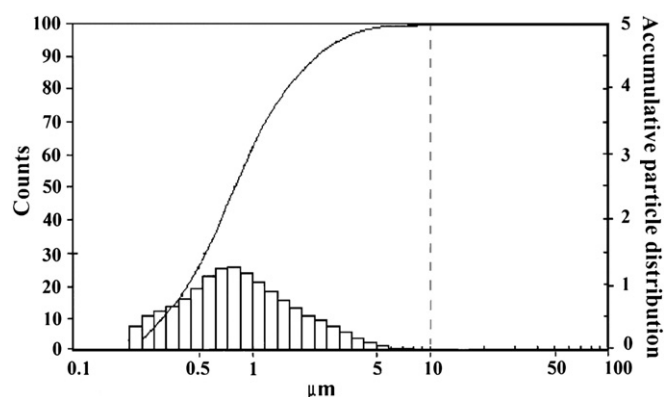


Fig. 2. Particle size distribution of tungsten carbide powder.

was then ball milled for 24 h to break down the agglomerates and to make a uniform slurry. After degassing the slurry in a rotary evaporator under vacuum, hexanedioic acid was introduced to the slurry, before casting it into a nonporous mold. After 24 h, the gelled wet green body were removed from the mold and dried at room temperature for 36 h.

Slurries with different volume fractions of tungsten carbide (20 and 25 vol.%) and 1 wt.% sodium alginate (water-based) were prepared and cast, as described in Fig. 3. The influence of chelator and calcium salt amounts on the dried green body strength was examined (Table 2).

Drying and sintering shrinkages of the final complex shapes decrease by increasing loaded solid (i.e. tungsten carbide) in the slurry. However, an increase in loaded solid enhances viscosity of the slurry and reduces its fluidity to fill thin and complex molds [11]. Therefore, it is essential to precisely verify the slurry composition. The sufficient amount of dispersant (based on ceramic powder) decreases viscosity of the slurry due to an increase in the density of electron cloud on the surface of ceramic particles and static repulsive forces [10]. Thus, the dispersant provides the opportunity to increase the solid loading volume fraction (i.e. WC) in the slurries and it decreases the shrinkage during drying and sintering. The utilized dispersant in this work was 0.3 wt.% [14].

2.3. Measurements

The room temperature bending strength of dried green body samples, which had been cut to a size of $20 \times 10 \times 3 \text{ mm}^3$, was determined by a three-point flexure test, taking into account ASTM C1341-95 standard, with span sizes of 15 mm and 0.5 mm/min loading rate (ZWICK, Z050). Binder burnout behavior of the green bodies was determined by thermogravimetric (TG) and differential thermal analyses (DTA) (NETZSCH, STA 409 PC Luxx) at the heating rate of $5^\circ\text{C}/\text{min}$ up to 1069°C under argon atmosphere. Phase analyses of the specimens were achieved by X-ray diffractometer (GNR, MPD 3000). Fracture surfaces of the green bodies (after flexural strength test) were pre-sintered at 1450°C for 4 h [15]. According to ASTM C373-88 standard, the Archimedes's method was applied to measure the bulk density of the

Table 1
Additives of gel-casting suspensions.

Binder (gelation reagent)	Sodium alginate (NaAlg)
Dispersant	Ammonium citrate tribasic, $(\text{NH}_4)_3\text{C}_6\text{H}_5\text{O}_7$
Solidifier agent (calcium salt)	Calcium phosphate tribasic, $\text{Ca}_3(\text{PO}_4)_2$
Chelator	Sodium hexameta phosphate, $(\text{NaPO}_3)_6$
Acid	Adipic acid, $\text{C}_6\text{H}_{10}\text{O}_4$

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