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Effect of the addition of polyvinylpyrrolidone as a pore-former on microstructure and mechanical strength of porous alumina ceramics

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Abstract

The effect of the solvent on the properties of porous alumina ceramics was studied when polyvinylpyrrolidone (PVP) was used as an organic pore-former. In particular, porous alumina ceramics were produced by dry-pressing of mixed PVP–alumina powder; the mixing of PVP and alumina powder was achieved via ball milling using water or acetone as solvent, or dry ball milling. Due to the different solubility of PVP in water and acetone, porous alumina ceramics with different pore structures and mechanical properties were obtained. Because of its cylindrical pores being aligned to some extent, the sample prepared using acetone as solvent exhibited the highest bending strength (140.2 MPa) and Young's modulus (57.4 GPa), which were 1.6 times and 3.4 times higher compared to that prepared without PVP. Moreover, the addition of PVP via wet ball milling led to more uniform dispersion of PVP in alumina, hence limiting the grain growth during sintering process and increasing the grain bonding.

Keywords: Mechanical properties; Porous ceramics; PVP; Microstructure

1. Introduction

Materials containing tailored porosity exhibit special properties and features that usually cannot be achieved by their conventional dense counterparts. Therefore, porous materials find numerous applications as end products and in several technological processes. Contrary to porous polymers, porous ceramics are more inert to various chemicals, bacteria and harsh environment such as high temperature and high pressure, and they are widely used as filters, catalyst supports, electrodes for solid oxide fuel cells and chemical or electronic sensors [1–3]. However, the brittleness of porous ceramics is the main concern for those applications requiring very good mechanical properties, such as ceramic hollow membranes. Especially, as separation membranes, a high porosity is needed to minimize the permeation resistance, and this leads to a decrease in the toughness of ceramic membranes. It still remains a challenge to retain the mechanical strength while a large volume of pores is introduced into the ceramic structures[4]. Therefore, there is a need for developing an effective way to prepare porous ceramics with excellent mechanical properties for industrial use.

There are numerous methods for increasing porosity of ceramics, such as by partial sintering or selection of a granular composition for initial molding mixture [5,6]; introduction of additives (PMMA, PVA, starch, $Al(OH)_3$, etc.) and their subsequent removal by evaporation, dissolution, burning-out or decomposition [7–10]; involvement of air into ceramic suspension or gas bubbles arising from chemical reaction or decomposition of pore-former [11]; extrusion of plasticized ceramic mixtures [12]. Porous ceramic materials are usually made by several shape-forming processes such as dry-pressing, isopressing, slip-casting, freeze-casting and so on. The simple dry-pressing method was used in the present work to study how the additive affects the properties of porous alumina ceramics.

Polyvinylpyrrolidone (PVP) is a water-soluble polymer and is usually used as an additive in the fabrication of ceramic

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membranes [13,14]. However, there are few studies on the relationship between different pore structures and mechanical properties of porous ceramics prepared using different solvents in dissolving and mixing the pore-former PVP and ceramic powder. As a water-soluble polymer, PVP can be readily dissolved in polar solvents such as NMP and ethanol while it has a limited solubility in non-polar solvents such as acetone. The solubility difference in different solvents can lead to different PVP particle sizes, which is expected to affect the pore morphology in the final porous ceramic materials and thus their mechanical properties. In this study, water and acetone were used as solvents to prepare mixed PVP–alumina powders for formation of porous alumina ceramics by the dry pressing method, in comparison with direct use of the raw PVP powder. The pore morphology and mechanical properties of the final porous ceramics were characterized and compared, with the goal of improving our understanding of how the procedure for addition of PVP affects the microstructure and mechanical structure of porous alumina ceramics.

2. Experimental

2.1. Preparation of porous ceramics

15 g of acetone (purity≥99.0%, Merck Schuchardt OHG) or deionized water were used to dissolve 1.5 g of PVP (average molecular weight 40,000, Sigma-Aldrich), respectively, followed by adding 15 g of alumina powder (d_{50}=1.2 μm, PP5010, Shell-lap Supplies Pty Ltd) and ball milling. The weight ratio of solvent and alumina was 1:1. After ball-milling for 48 h, the powder was dried and ground with a mortar and pestle. For comparison, dry ball-milling of PVP–alumina powder was carried out. The methods for preparing PVP–alumina powders were referred to as Method A, Method W, and Method D (dry milling) for the use of acetone, water and dry milling respectively. The PVP–alumina powder was uniaxially pressed into rectangular bars with the size of 50.9 mm×5.9 mm×3.0 mm at a pressure of 20 MPa. The porous alumina bars were then obtained by sintering the green compacts using a heating profile as follows: 600 °C for 1 h, to 1000 °C for 1 h and then to 1500 °C for 10 h at a heating rate of 3 °C/min. Samples are indicated as *X*–*Y*, while *X* refers to the method used, *Y* refers to the quantity (in percentage) of PVP added, e.g. A-10 means acetone was used to prepare 10 wt% PVP/alumina powder.

2.2. Characterization

The mean particle size of PVP in water and acetone was determined using a Mastersizer 2000 (Malvern instruments Ltd, UK). The porosities of the samples were measured using the Archimedes' method with deionized water as a liquid medium. The pore size distribution of the samples was measured by mercury porosimetry (Auto pore III mercury porosimeter, Particle and Surface Sciences Pty. Ltd. USA).The morphology and pore structures of the porous alumina were observed by scanning electron microscopy (Nova Nano SEM, FEI company), and the average grain size of sintered samples was measured by the linear intercept technique based on SEM images [15]. The linear sintering shrinkage of the samples was determined by measuring the length of the samples before and after sintering.

The mechanical strength was determined by the three-point bending test (Mini-instron) with a span of 20 mm and a crosshead speed of 0.5 mm/min. For each batch, at least five specimens were tested and the average strength was calculated and the standard deviation *s* was also calculated to show the variability of the tested samples.

3. Results and discussion

3.1. Dissolution of PVP in different solvents

PVP is slightly dissolved in acetone, and well dissolved in water. The mean PVP particle size in acetone was determined to be 5.26 μm while its mean particle size was only 39 nm in water. From the SEM images of raw samples shown in Fig. 1, it can be seen that individual particles are clearly observed in A-10 compact while the particles are floccule-like in W-10 compact, indicating PVP is better dispersed in W-10 than A-10. This can be attributed to different particle sizes of PVP in the preparation of PVP–alumina mixtures, i.e., PVP nanoparticles were uniformly dispersed and well bound with alumina particles.

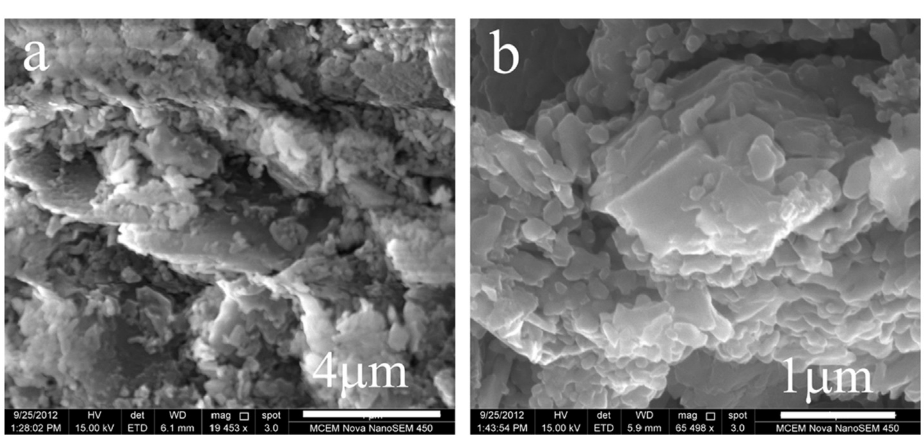

Fig. 1. SEM images of compact samples: (a) W-10 and (b) A-10.