



ELSEVIER

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

The influence of gel-casting parameters on the preparation of Si porous bodies



P. Tabrizian^{a,*}, F. Golestanifard^a, A. Alem^b, E. Ghassemi^c

^a Metallurgy and Materials Engineering Department, Iran University of Science and Technology, Iran

^b Mechanical and Industrial Engineering Department, Concordia University, Canada

^c Institute for Colour Science and Technology, Iran

ARTICLE INFO

Article history:

Received 20 January 2016

Received in revised form

30 April 2016

Accepted 16 July 2016

Available online 18 July 2016

Keywords:

Gel-casting

Reaction bonding

Rheological behaviour

Porous

ABSTRACT

This study aimed to investigate and optimise the rheological behaviour and gel-casting parameters of Si suspensions prepared to fabricate RBSN bodies. Si green bodies with 30 vol% solid content possessing roughly 40 MPa flexural strength and 50 vol% porosity. The gelation time decreases by heating, and the Si gel-cast body dried in PEG1000 solution shows less than 2% shrinkage. Based on FESEM analysis and adsorption-desorption of nitrogen gas, the final Si bodies show open pores and uniform distribution.

© 2016 Elsevier B.V. All rights reserved.

1. Introduction

Porous Si₃N₄ bodies due to its special features were extensively used in various industrial applications, such as biomedicine, gas, oil and the automobile [1]. Of different methods have been used to fabricate porous Si₃N₄ ceramics, utilisation of gel-casting (GC) and reaction bonding in combination is much faster and more economical [2,3]. To this end, the investigation into GC Si bodies in aqueous systems is important.

GC is a wet processing method based on the in situ polymerisation and consolidation of suspensions invented by ORNL in 1990 to improve the uniformity and near-net shaping of bodies [4]. In the process, ceramic powders are dispersed in the pre-mixed monomer solution to prepare a stable and castable suspension. After polymerisation, the particles and water in slurry are held together with cross-link network of polymers [5–7]. The notable merit of GC is machinability of the sample under green condition due to the high strength green bodies [8]. This promising process has been applied to various fine ceramics, such as SiC, Al₂O₃, SiO₂ and Si₃N₄ [6,7,9].

In this work, the authors' investigate the rheological behaviour of Si suspensions and gelation parameters to fabricate Si porous gel-cast bodies with minimal rate of shrinkage and high flexural

strength. Si by its very nature is extremely reactive and silica forms readily in aqueous systems, so preparation of GC Si body is inherently difficult and need accurate investigation [10]. The effects of different gelation techniques and drying methods to prepare defect-free Si bodies were studied.

2. Experimental procedure

2.1. Materials

Si bodies were fabricated based on the GC of Si powder (H.C. Starck, 99.995%, D₅₀:7.38 μm). P-hydroxybenzoic acid (PHBA, Merck 99-96-7) was used as a dispersant. Acrylamide (AM, C₂H₃CONH₂, Merck) was used as a monomer and N,N'-methylenebisacrylamide (MBAM, (C₂H₃CONH₂)₂CH₂, Merck) was used as a cross linker. Ammonium persulfate (APS, (NH₄)₂S₂O₈, Sigma-Aldrich) and N,N,N',N'-tetramethylethylenediamine (TEMED, C₆H₁₆N₂, Sigma-Aldrich) were used as the initiator and catalyst, respectively.

2.2. GC process

A premix solution containing water, monomer and crosslinker was prepared. The dispersant and Si powder were then added to the premix solution. The suspension was then mixed for 4 h in a

* Corresponding author.

E-mail address: parinaz_tabrizian@yahoo.com (P. Tabrizian).

jar mill. The suspension was degassed in a rotary evaporator under a vacuum for 5 min to remove air bubbles produced during mixing and milling.

Two gelation methods were employed: (a) adding catalyst and, (b) heating. In adding catalyst, an initiator and catalyst were added to the suspension after degassing. Catalyst and initiator volumes must be carefully controlled to ensure that the suspension remains completely fluid during casting. After casting and gelation, the sample was removed from the mould to undergo drying. Heat-set sample moulds were placed in an oven at 60 °C for 45 min to produce a gel and then left to dry.

2.3. Testing method

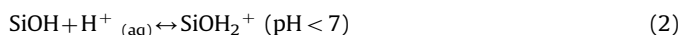
Suspensions with different mixing times were prepared and their viscosity measured with a controlled-rate rotary rheometer (Pro2+, Brookfield). Sedimentation tests were also performed for 24 h to determine the sediment height. During this test, the suspensions were poured in the test tube to a height of 10 cm, after 24 h the height of sediment was measured.

Samples were kept in a room with 5% humidity at a temperature of 28 °C for 48 h, and then dried at a temperature of 120 °C for 5 h, or samples were kept in a room with 85% humidity at a temperature of 28 °C for 48 h then drying at a temperature of 120 °C for 5 h. In other method, samples were placed in a liquid desiccant composed of water and PEG1000 for 2 h then the samples were washed with water and kept at a temperature of 28 °C for 46 h, then dried at a temperature of 120 °C for 5 h. The resulting bodies were heated at a rate of 1 °C/min and kept at a temperature of 450 °C for 3 h for polymer pyrolysis. Porosity was measured by way of nitrogen adsorption–desorption (Quantachrome Corporation NOVA 2200). The microstructural properties of the GC bodies were inspected using a field-emission scanning electron microscope (FESEM, TESCAN-MIRA3).

3. Result and discussion

3.1. Effect of mixing time on the colloidal behaviour of Si suspension

In order to prepare stable Si suspensions, Si reactions in aqueous systems must be investigated. As shown in Eq. (1), Si oxidises in aqueous systems resulting in hydrogen gas release. Moreover, SiO₂ is hydrolysed and leads to the creation of silanol groups. Depending on the suspension's pH, Eqs. (2) or (3) may occur.



The suspension's pH affects the intensity of the reaction in Eq. (1). Depending on the surface chemistry of the Si powder, the optimum pH value may be different [11,12]. In this study, with respect to the surface chemistry of the Si powder, the suspension pH was set at 8–9, which led to the production of stable fluid suspensions with less hydrogen release [3,4,10].

To optimise the mixing time, 6 suspensions at a pH of 8–9 with 30 vol% solid content were mixed for 1, 2, 4, 8, 16 and 24 h. Fig. 1 shows the viscosity of the suspensions at a range of speeds, from 90 to 190 RPM.

The results of sedimentation test of the suspensions mixed for different times are illustrated in Fig. 1, as well. Increasing the mixing time makes the suspension more fluid, in contrast the stability of the suspension decreases. So, the suspension mixed for

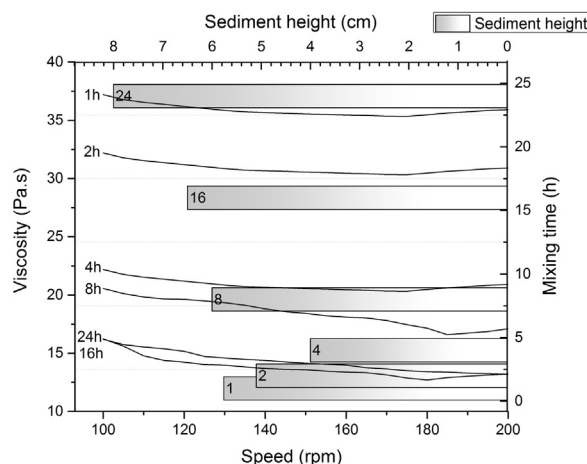


Fig. 1. Viscosity and sediment height of the suspensions with 30 vol% solid content at a pH of 8–9 for different mixing times.

4 h had 4 cm sedimentation height out of 10 cm, but the suspension mixed for 24 h had 8 cm sedimentation height.

In addition, by increasing the mixing time to 24 h, the viscosity of the suspension increases due to agglomeration and segregation. The resulting suspension was also unstable this non-uniformity is a sign of demixing [13].

So, the mixing time has strong impact on the stability and fluidity of the suspension. When the suspension mixed less than 4 h had a viscosity in the range of 30–40 Pa.s and it was more unstable and inhomogeneous compared to the suspension mixed for 4 h. Suspensions possessing these characteristics will encounter problems during casting and gelation, especially when the solid content is higher than 30 vol%.

On the other hand, by increasing the time of mixing the severity of the reaction in Eq. (1) and the consequently released hydrogen are increased. This gaseous release causes the uncontrolled porosity within the Si GC bodies. Following this reaction, the surface charge characteristics of electrical double layer of Si particle which plays a major role in suspension stability, fundamentally changes [4,10,13]. So, the suspensions mixed more than 4 h due to high ionic strength, become weakened in terms of the stability compared to the suspension mixed for 4 h. In short, although increasing the mixing time reduces the viscosity of the suspension, it also makes the suspension unstable.

3.2. Properties of Si GC bodies based on gelation method

The time it takes and the temperature at which a suspension becomes a gel affects the properties of the GC bodies [14,15]. This study focused on the differences between the time and temperature of gelation for two commonly used methods of gel preparation. Fig. 2 displays the comparison of gelation under heating and adding catalyst conditions.

By changing the gelation method from adding catalyst to heating, the time and temperature of gelation increased by 360 s and 33 °C, respectively. It is concluded that the dissolution of APS and react with AM is much faster by adding catalyst compared to heating utilisation. The differences between the time and temperature cause different percentage of shrinkage in bodies, so the heat-prepared GC bodies showed a 15% more linear shrinkage than bodies prepared in the presence of the TEMED. Fig. 2 shows the samples prepared by adding catalyst and heating. Heat-prepared GC bodies had crack and become brittle invariably affecting the machinability of the green bodies. Kokabi et al. used TEMED to prepare Si GC bodies, their results showed that by controlling the amount of APS and TEMED at the constant solid load of 50 vol%,

Download English Version:

<https://daneshyari.com/en/article/1641037>

Download Persian Version:

<https://daneshyari.com/article/1641037>

[Daneshyari.com](https://daneshyari.com)