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Short communication

Extrusion of alumina fibers using zirconia sol as binder

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Abstract

Zirconia sol was used as an extrusion aid for the preparation of alumina fibers. The amount of zirconia sol required for getting an extrudable slurry has been optimised. The only phase present in the dried and sintered fibers is α-Al_2O_3. Sintering studies showed that a dense microstructure is formed at 1600 °C. SEM micrographs revealed intergranular fracture to be the predominant fracture mode. Tensile strength is the highest for fibers sintered at 1600 °C.

Keywords: A. Sol–gel process; A. Sintering; D. Al_2O_3; D. ZrO_2

1. Introduction

Extrusion is one of the ceramic fabrication techniques considered to be the most suitable method for producing a variety of shapes with constant cross-section [1–3]. It has been widely used to produce ceramic tube [4], ceramic fiber [5] and ceramic matrix composite [3], etc. Because of the non-plastic nature of ceramic materials, binders are generally used during shaping. Conventional binders are generally organic in origin but reports highlight inorganic binders in view of their advantages of fewer binder problems and lower carbon residues [6]. Sol–gel assisted colloidal processing is one of the promising technique for the preparation of ceramic paste with nano particles. The advantages of this method are increased wetting of powder surface, accurate control over desired moisture level, elimination of agglomeration and contamination, good compatibility with the matrix, elimination of binder burn out, non-contamination of ceramic mass with metal parts and fast sintering [7]. Ananthakumar et al. has studied the rheology and packing characteristics of alumina ceramics [2] using boehmite gel as binder. He has also studied the effect of boehmite and organic binder on extrusion of alumina [7]. Sunil Kumar et al. used boehmite gel as an extrusion aid for alumina ceramics [6] and alumina–zirconia composites [1]. Del Olmo et al. extruded ceramic fibers of modified lead titanate using sol–gel precursor [5]. Several reports are available using zirconia as second phase but not as grain boundary segregation in alumina matrix. In this present work, alumina fibers were prepared using zirconia sol as binder and characterised. A simple method has been used for the preparation of fibers and so there is no control over the diameter of fiber. This process can be upgraded to industrial level, to get fine fibers.

2. Experimental procedure

Alumina powder (A 16 SG, ACC-ALCOA Chemicals, India) having 99.8% purity, BET surface area of 8.6 m^2/g and average particle size of 0.3 μm was used as starting material. Zirconium oxychloride (Otto Kemi, Mumbai) was dissolved in distilled water to a molar concentration of 1 M. Oxalic acid was separately dissolved in distilled water to a molar concentration of 1 M. Both the solutions were mixed and stirred continuously until the solution becomes transparent. Different amount of zirconia sol having a viscosity of 200 cP was tried to make an extrudable paste and the nature of mixture is given in Table 1. It was found that 15 ml of zirconia sol with 10 ml of water is suitable for making an extrudable paste, out of 100 g of alumina. The volume fraction of zirconia in alumina is 0.0062.

A laboratory model vertically mounted screw type extruder was used to prepare fibers. The mixed paste like material was

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Table 1
Observations on the alumina paste with zirconia sol, containing 100 g of alumina powder

S. no.	Zirconia sol (ml)	Water (ml)	Comments
1	5	0	Powdery
2	10	0	Powdery
3	15	0	Agglomerated powders
4	20	0	Agglomerated powders
5	25	0	Paste with high viscosity
6	5	5	Powdery
7	10	5	Powdery
8	15	5	Paste with high viscosity
9	20	5	Paste with low viscosity
10	25	5	Paste with low viscosity
11	5	10	Powdery
12	10	10	Agglomerated powders
13	15	10	Paste suitable for extrusion
14	20	10	Paste with low viscosity
15	25	10	Paste with low viscosity

fed into the extruder chamber and pressure was applied through the screw from the top. Alumina fibers were drawn and dried at room temperature. The dried fibers were then sintered at three different temperatures (1200, 1400 and 1600 °C) for 2 h at a heating rate of 5 °C/min.

X-ray diffraction analysis (XRD) was performed at room temperature (Model XD-DI, SHIMADZU operating with Cu Kα). Micrographs were recorded by scanning electron microscope (Model JSM-840A, JEOL) operating at 20 kV. Specimens were prepared by embedding fibers on the surface of an adhesive carbon film. Density measurement was carried out by the water displacement method. Tensile strength was determined by Instron tensile testing machine (4301). Fibers were mounted with adhesive on chart paper tabs for aligning and gripping. A 5 mm gauge length and crosshead speed of 0.5 mm/min were used in all these tests. The fracture load was converted to tensile strength by measuring the cross-sectional area of the fiber with an optical microscope. Ten samples were tested for each batch, the average was calculated and reported.

3. Results and discussion

XRD patterns of dried and sintered alumina fiber are shown in Fig. 1. The dried fiber contains only α-Al_2O_3. This is due to the large amount of α-Al_2O_3 mixed with the sol. The phase present in the sintered fiber is also α-Al_2O_3. Zirconia is not observed because of its low content.

The average diameter of the fibers sintered at 1600 °C was found to be ∼480 μm. The surface morphology of alumina fiber sintered at 1200, 1400 and 1600 °C is shown in Fig. 2. Sintering is incomplete at 1200 and 1400 °C as is evident from SEM microstructure. The powdery nature of the particles is retained after these sintering treatments and there are large amount of open and interconnected porosity present through out the surface. A dense microstructure is formed at 1600 °C. The grain size of alumina varies from 1 to 6 μm, while zirconia

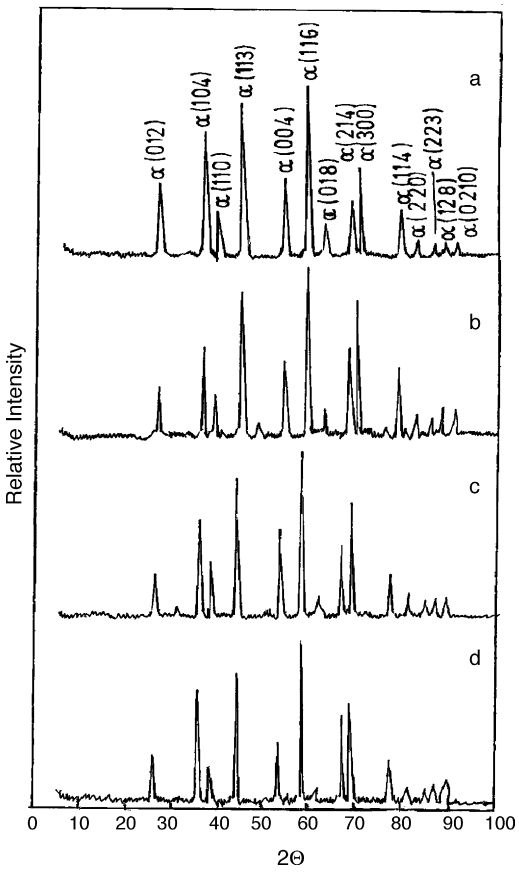

Fig. 1. X-ray diffraction patterns of alumina fiber (a) dried and sintered at (b) 1200 °C; (c) 1400 °C; (d) 1600 °C.

grains of size varying from 1.5 to 2 μm are occasionally observed. It was reported by Low and McPherson [8] that the presence of zirconia up to 8 wt.% in a corundum matrix does not inhibit grain growth at high temperature. In another study [9], boehmite sol was used as an extrusion aid for the preparation of alumina fiber. It was found that, even after sintering at 1600 °C, a dense microstructure is not formed (Fig. 3).

Fracture surfaces of alumina fibers heat-treated at 1200, 1400 and 1600 °C are shown in Fig. 4. An intergranular fracture path is observed in all sintered samples. Incomplete sintering at 1200 and 1400 °C with the presence of pores is also apparent.

The density of alumina fibers sintered at 1200, 1400 and 1600 °C is reported in Table 2. The density is highest for the fibers sintered at 1600 °C. Incomplete sintering with the

Table 2
Density of alumina fibers sintered at various temperatures

Sintering temperature (°C)	Density (% theoretical density)
1200	76
1400	85
1600	95

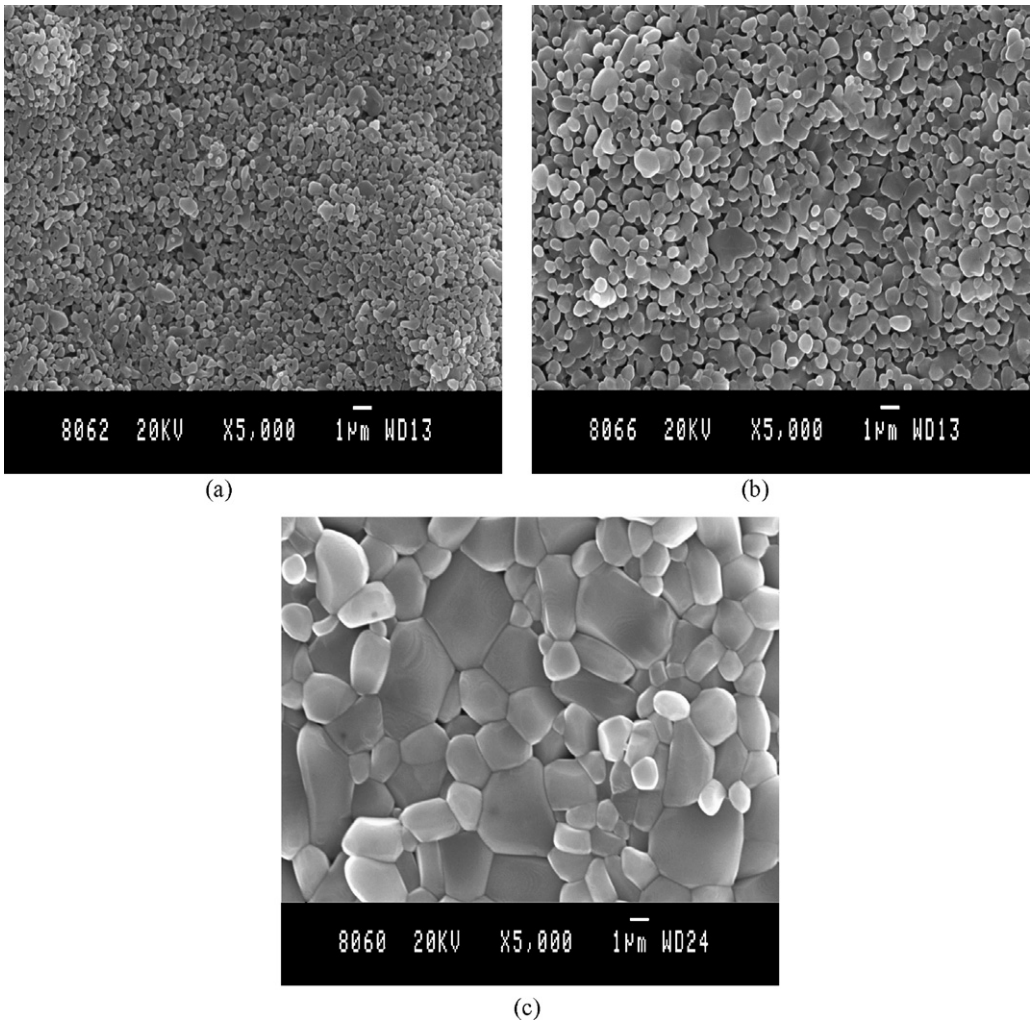

Fig. 2. Microstructure of zirconia sol assisted alumina fiber sintered at (a) 1200 °C; (b) 1400 °C; (c) 1600 °C.

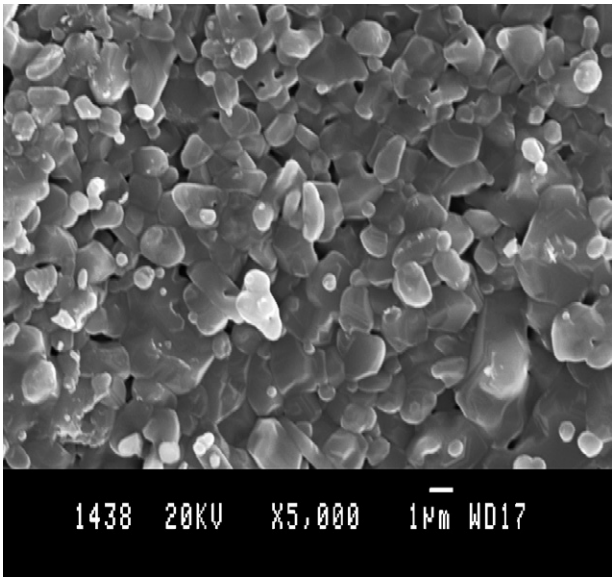

Fig. 3. Microstructure of alumina sol assisted alumina fiber sintered at 1600 °C.

presence of pores is the reason for the lower density at 1200 and 1400 °C.

The tensile strength values of alumina fiber sintered at 1200, 1400 and 1600 °C are reported in Table 3. It was found that the tensile strength increases with the increase in the sintering temperature. The tensile strength is the highest for the fiber sintered at 1600 °C. This is due to the dense microstructure, as it is evident from the surface and the fracture morphology. Incomplete sintering at 1200 and 1400 °C with the presence of pores is the reason for the lower tensile strength values.

Table 3
Tensile strength of alumina fibers sintered at various temperatures

Sintering temperature (°C)	Tensile strength (MPa)
1200	98 ± 28
1400	125 ± 18
1600	198 ± 25

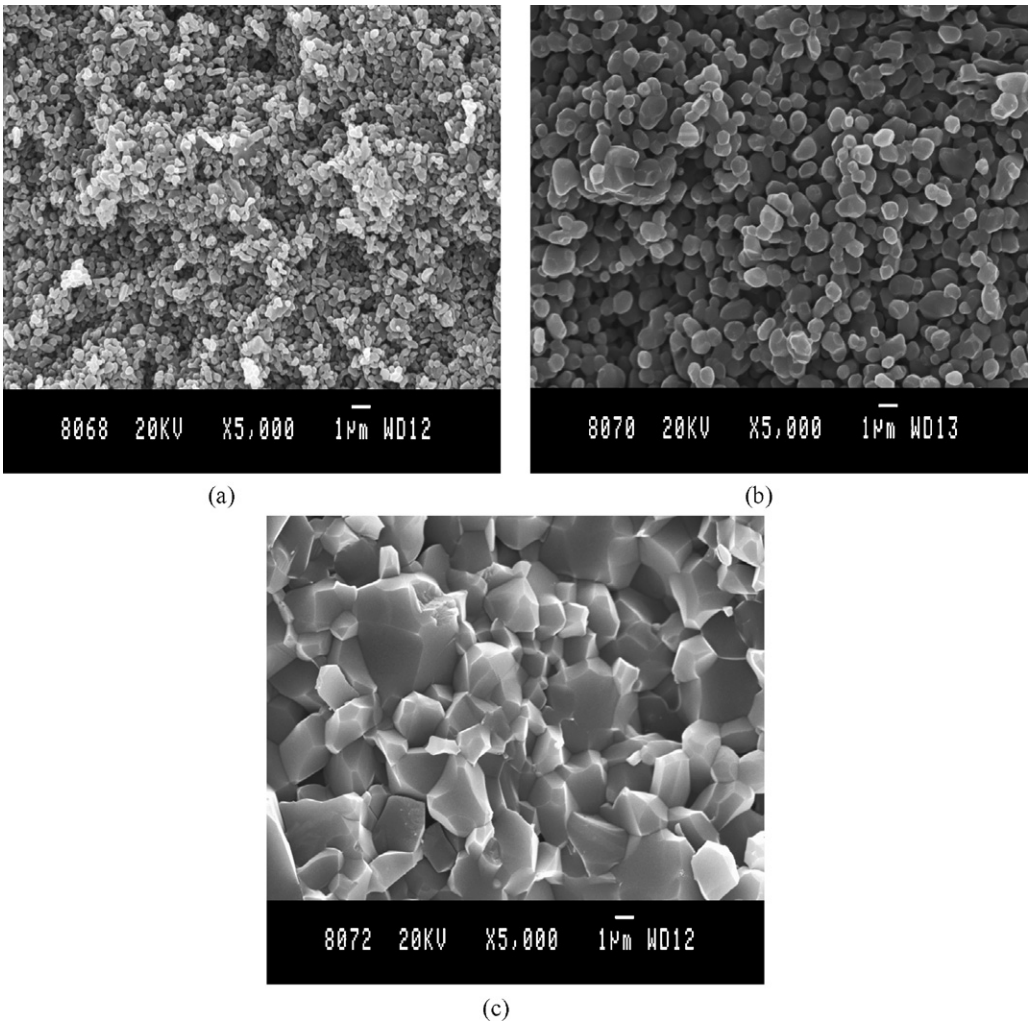

Fig. 4. Fracture morphology of zirconia sol assisted alumina fiber sintered at (a) 1200 °C; (b) 1400 °C; (c) 1600 °C.

4. Conclusions

Alumina fibers were extruded using zirconia sol as binder. The density and tensile strength were found to be the highest for fibers sintered at 1600 °C. Zirconia sol can be used as an extrusion aid for making fibers, tubes, rods, etc. using alumina powder.

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References

[1] C. Sunil Kumar, U.S. Hareesh, B.C. Pai, A.D. Damodaran, K.G.K. Warrier, Aqueous extrusion of alumina–zirconia (12 mol% ceria) composite using boehmite as extrusion aid, Ceram. Int. 24 (1988) 583–587.

[2] S. Ananthakumar, A.R.R. Menon, K. Prabhakaran, K.G.K. Warrier, Rheology and packing characteristics of alumina extrusion using boehmite gel as binder, Ceram. Int. 27 (2001) 231–237.

[3] S. Ananthakumar, K.G.K. Warrier, Extrusion characteristics of alumina–aluminium titanate composite using boehmite as a reactive binder, J. Eur. Ceram. Soc. 21 (2001) 71–78.

[4] C. Kaya, S. Blackburn, Extrusion of ceramic tubes with complex structures of non-uniform curvature made from nano powders, J. Eur. Ceram. Soc. 24 (2004) 3663–3670.

[5] L. Del Olmo, L. Pardo, J.I. Pina, J. Mendiola, Extruded ceramic fibers of modified lead titanate including sol–gel precursors, J. Mater. Sci. 22 (1987) 4133–4138.

[6] C. Sunil Kumar, U.S. Hareesh, A.D. Damodaran, K.G.K. Warrier, Monohydroxy aluminium oxide (boehmite, AlOOH) as a reactive binder for extrusion of alumina ceramics, J. Eur. Ceram. Soc. 17 (1997) 1167–1172.

[7] S. Ananthakumar, P. Manohar, K.G.K. Warrier, Effect of boehmite and organic binder on extrusion of alumina, Ceram. Int. 30 (2004) 837–842.

[8] M. Low, R. McPherson, Crystallisation of gel derived alumina and alumina–zirconia ceramics, J. Mater. Sci. 24 (1989) 892–898.

[9] J. Chandradass, M. Balasubramanian, Extrusion of alumina fibres by sol–gel precursor, J. Mater. Sci., in press.