

Extrusion of alumina fibre using sol-gel precursor

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Abstract Alumina fibres were prepared by extrusion using boehmite sol as binder. The microstructure and mechanical properties of the fibres were studied. The fibres sintered at 1400 °C have relatively dense microstructure and the highest strength.

Introduction

Ceramic fibres have number of advantage over bulk ceramics. Brittle materials can develop higher strength and moduli in fibrous form. In particular, fine fibres are reasonably flexible and can be manipulated without risk of breakage [1]. Recent trends in material design suggest that properties such as toughness and creep resistance of parts for thermo-mechanical applications, traditionally made from high-strength alloys, could be implemented by using ceramic fibre (SiC, Si₃N₄, Al₂O₃) reinforced ceramic or metal matrix composite [2]. Ceramic fibres, in particular alumina fibres, are of growing interest and importance for a variety of applications such as high temperature insulating material in the form of mats, blankets and boards [3], catalyst support in high temperature reactions [4], and as reinforcements in ceramic as well as metal matrix composites [5]. 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 [6–8]. It has been

widely used to produce ceramic tube [9], ceramic fibre [10], rubber toughened polymer matrix composites [11], ceramic matrix composite [8], etc. Binders are employed in the shaping of ceramic materials because of their non-plastic nature [12].

There exist a few practical difficulties in using conventional organic binders, such as the uniform dispersion of particulate powder into viscous binder, distribution of moisture uniformly throughout the matrix and the elimination of hard agglomerates. The homogenous mixing of these binders with the matrix is achieved by high intensity mixture, in which problem of contamination arises. Further, the conventional binder form micro/macro pores and cracks if they are not burnout properly [7]. These problems are eliminated by using inorganic binder in view of their advantages of fewer binder problem and lower carbon residues [12]. Ananthakumar et al. have 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 powder [13]. Sunilkumar et al. [12] used boehmite gel as an extrusion aid for alumina ceramics and alumina-zirconia composites. Del Olmo et al. [10] extruded ceramic fibres of modified lead titanate including sol-gel precursor of approximately 1 mm diameter. In the present work, alumina fibres were prepared using boehmite gel as an extrusion aid and their microstructures were characterized by scanning electron microscope.

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Experimental procedure

Alumina powders (A 16 SG, ACC-ALCOA Chemicals, India) having 99.8% purity, BET surface area of 8.6 m²/g

and average particle size of 0.3 μm were used as the starting material. Chemical composition of the alumina powder is given in Table 1. Boehmite sol was prepared according to the procedure described by Yoldas [14]. Aluminium-tri-isopropoxide ($\text{Al}[\text{OCH}(\text{CH}_3)_2]_3$) (20.4 g) was dissolved in 100 ml of distilled water, refluxed under air atmosphere at 80 $^\circ\text{C}$ for 3 h in the presence of acid catalyst (0.07 mol nitric acid) and then cooled.

Fifteen wt.% of boehmite sol containing 0.2 wt.% of organic binder (hydroxy ethyl cellulose) having viscosity of 100 cps was mixed with 85 wt.% of alumina powder. Small amount of distilled water was added to convert the solid paste appropriate for extrusion.

Laboratory model vertically mounted screw type manually operated extruder was used to prepare fibres. The mixed paste like material was fed into the extruder chamber and pressure was applied through screw from the top. Alumina fibres were drawn and dried at room temperature. The dried fibres were then sintered at three different temperatures (1,200, 1,400 and 1,600 $^\circ\text{C}$) for 2 h at a heating rate of 5 $^\circ\text{C}/\text{min}$.

The dried and sintered fibres were characterized using X-ray powder diffractometer with Cu-K_α radiation (SHIMADZU, XD-DI), Fourier Transform Infrared spectroscope (FTIR) by the KBr method (Perkin–Elmer, RXI), Thermogravimetric analyser (NETZSCH STA 409PC) and Scanning electron microscope (QUANTA 200). Tensile strength was evaluated by universal testing machine (Instron 4301). Fibres were mounted with adhesive on chart paper tabs for aligning and gripping. A 5 mm gauge length and cross-head 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 fibre with an optical microscope. Ten samples were tested for each composition.

Results and discussions

The thermogravimetric analysis of alumina powder mixed with boehmite sol is shown in Fig. 1. The total weight loss is 2.8%. Most of the weight loss occurs up to 400 $^\circ\text{C}$. This loss corresponds to the evolution of adsorbed water and small amount of organic additives present in the boehmite sol.

Table 1 Chemical composition of alumina powder

Chemical composition	Al_2O_3	Na_2O	Fe_2O_3	MgO	SiO_2	CaO
wt. %	99.8	0.06	0.02	0.03	0.03	0.02

The photograph of the extruded and sintered alumina fibre is shown in Fig. 2. The diameter of the sintered fibre is 330–370 μm . Scanning electron micrograph of single fibre in horizontal and vertical direction is shown in Fig. 3. The diameter of the fibre is found to be 350 μm .

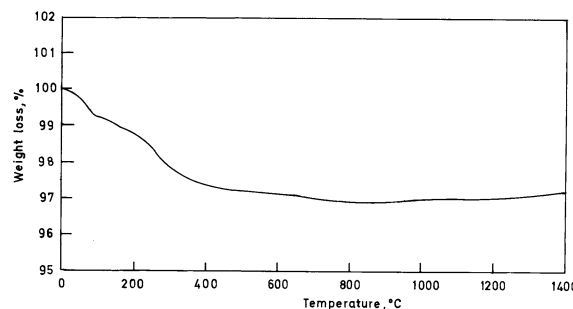


Fig. 1 Thermogravimetric analysis of dried alumina fibres

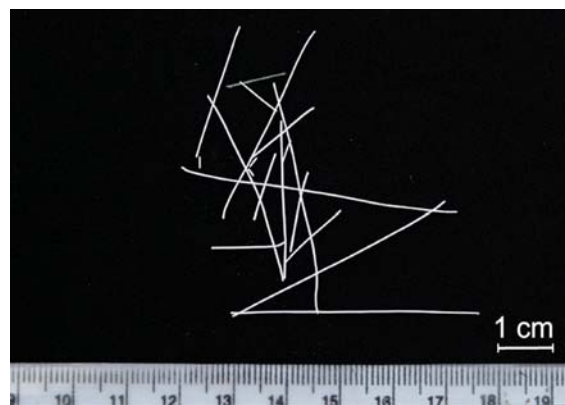


Fig. 2 Photograph of alumina fibres sintered at 1,200 $^\circ\text{C}$

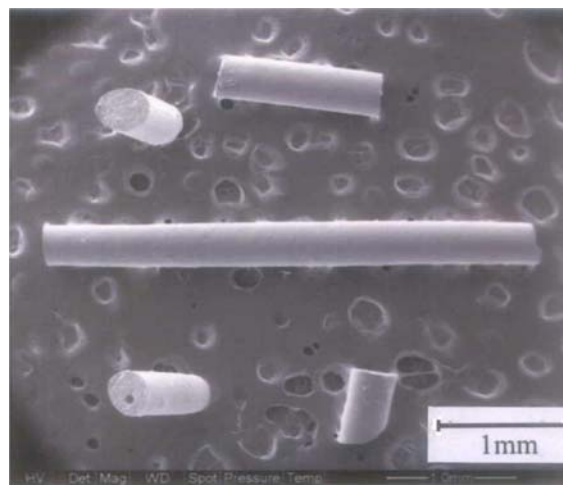


Fig. 3 SEM micrograph of alumina fibres sintered at 1,600 $^\circ\text{C}$

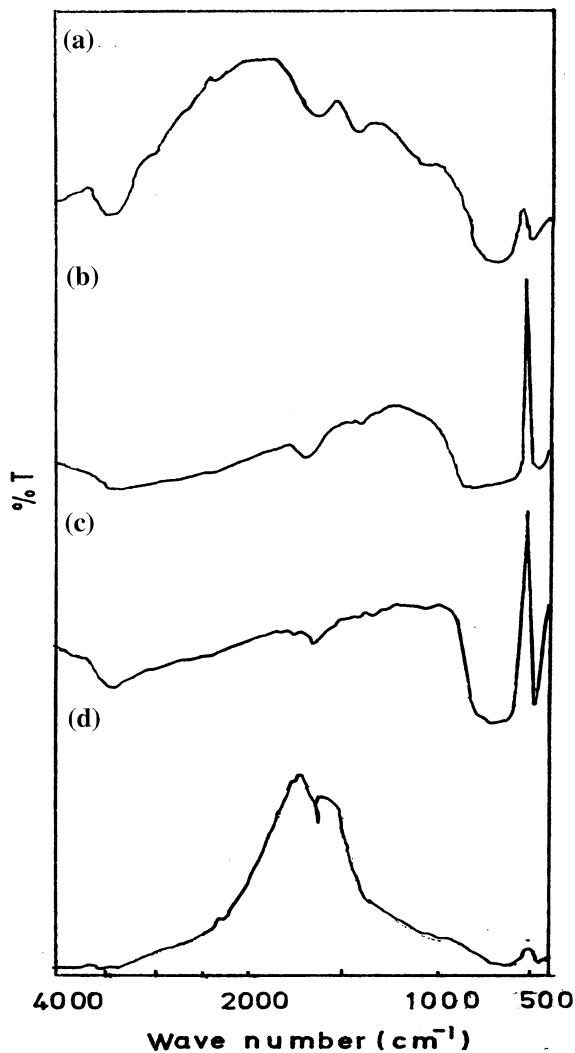


Fig. 4 FTIR spectra of alumina fibres (a) dried; (b) sintered at 1,200 °C; (c) sintered at 1,400 °C; (d) sintered at 1,600 °C

FTIR spectra of dried and sintered fibres are shown in Fig. 4. FTIR involves measuring the intensity of a beam of infrared radiation before and after it interacts with the sample as a function of wavenumber. From the plot of intensity versus wavenumber, the nature of chemical bonds can be determined. The dried fibre shows absorption band at $\sim 3400\text{ cm}^{-1}$ [15] due to the presence of molecular water. This band is also present in fibre heated at 1,200 °C with reduced intensity. As the sintering temperature increased to 1,400 °C the absorption band vanishes. The absorption band at $\sim 1,650\text{ cm}^{-1}$ [16] is due to O–H bending mode confirming the presence of adhesive water. This band is present with reduced intensity after 1,200 °C and absent after 1,400 °C. The absorption band observed at $1,380\text{ cm}^{-1}$ [17] corresponds to Al–OH bending mode. This band is also present in the 1,200 °C sintered fibre

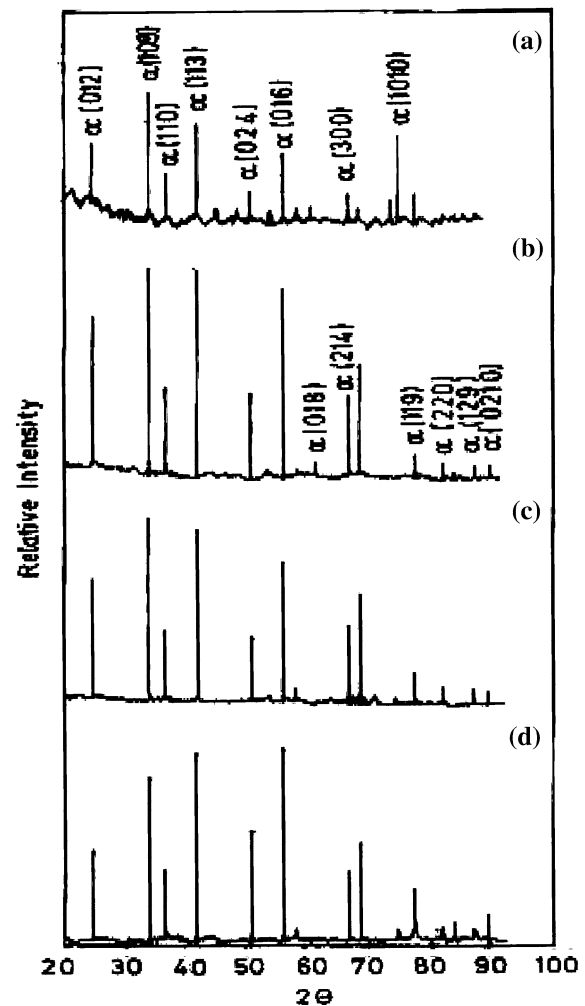


Fig. 5 X-ray diffraction patterns of alumina fibres (a) dried; (b) sintered at 1,200 °C; (c) sintered at 1,400 °C; (d) sintered at 1,600 °C

and absent in the 1,400 °C sintered fibre. The broad band in the region $400\text{--}1,000\text{ cm}^{-1}$ corresponds to Al–O vibration. The doublet in the region 645 and 607 cm^{-1} [18] corresponds to AlO_6 vibration. This band is also present in the fibres after 1,200 and 1,400 °C treatment. A well-defined absorption band at 460 cm^{-1} [17] corresponds to Al–O vibration of $\alpha\text{-Al}_2\text{O}_3$. This band is also present in the sintered fibre.

Figure 5 shows the X-ray diffraction patterns of dried and sintered alumina fibres. The dried fibre has only $\alpha\text{-Al}_2\text{O}_3$ phase. This is due to the large amount of $\alpha\text{-Al}_2\text{O}_3$ mixed in the sol. The phase present in the sintered fibres is also $\alpha\text{-Al}_2\text{O}_3$ phase.

Figure 6 shows the microstructure of alumina fibre after sintered at 1,200, 1,400 and 1,600 °C. Since there existed some pores inside the compacts as is evident from the SEM microstructure, sintering is incomplete

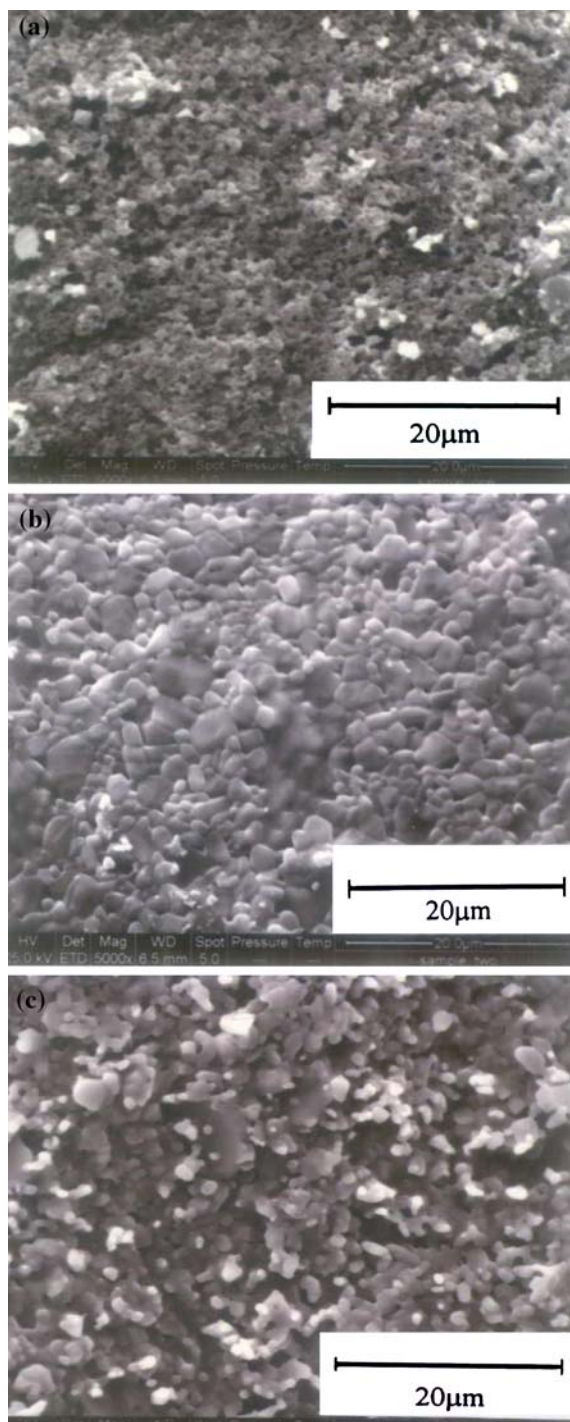


Fig. 6 Microstructures of alumina fibre sintered at (a) 1,200 °C, (b) 1,400 °C and (c) 1,600 °C

at 1,200 °C. The powder nature of the particle is retained after this treatment and there is large amount of porosity. Dense microstructure is obtained after 1,400 and 1,600 °C treatment.

Table 2 Tensile strength of alumina fibre after sintered at different temperatures

Sintering temperature	1,200 °C	1,400 °C	1,600 °C
Average tensile strength (MPa)	151	165	145
Standard deviation (MPa)	5.07	6.88	4.12

The tensile strength values of alumina fibre sintered at 1,200, 1,400 and 1,600 °C are given in Table 2. Tensile strength value corresponds to average tensile strength and scatter is given by standard deviation. Tensile strength value is higher for the fibres sintered at 1,400 °C and this is due to the dense microstructure. Sintering is almost complete at 1,400 °C and the higher sintering temperature leads to grain growth. This may be the reason for the slight decrease in tensile strength value for the fibres sintered at 1,600 °C. Incomplete sintering at 1,200 °C with the presence of many pores accounts for the lower tensile strength value.

Conclusion

Alumina fibre was prepared using boehmite sol as inorganic binder. α - Al_2O_3 is the only phase present in the alumina fibre after sintered at high temperatures. The tensile strength is higher for the fibres sintered at 1,400 °C.

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