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Influence of spinning parameters on synthesis of alumina fibres by centrifugal spinning

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ABSTRACT

The present study investigates the synthesis of alumina fibres of 5–15 μm dia. by sol-gel process through centrifugal spinning. Among various spinning parameters such as rpm, viscosity, humidity, chamber temperature etc., effect of rpm and viscosity on quality of spun fibres was examined in order to spin less defective (less shots) fibres. From the rpm vs viscosity experiments, it was concluded that viscosity between 13 and 42 Pa.s with 3000 rpm was favourable for obtaining fibres containing less shots. It was shown that besides viscosity, refractive index and percent weight loss of the aged sol can be used as indicator for determining spinnability of alumina precursor sols. Influence of only preheating and continuous heating of the chamber during spinning were also investigated and it was found that only preheating did not help in producing fibres. Besides this, influence of hot air blower (HAB) aligned in different directions with respect to the spinnerette, on fibre quality was tested. It was observed that the quality of fibres did not change due to change in HAB direction.

1. Introduction

High temperature insulation (> 1000 °C) requires use of ceramics in various forms such as refractory bricks, fibres and foams [1–3]. Though refractory bricks are widely used, fibre and foam based products have low thermal mass, better thermal insulation properties (increases efficiency up to 73%) than refractory bricks [4]. Foam based monolithic products are prone to fracture while fibre based insulations are damage tolerant [5]. Besides making fibreboards for use as insulation in furnaces, fibres can also be used for fire resistant textiles and structures, and for use as reinforcements in composites.

Ceramic fibres can be synthesized by melt spinning, template method, extrusion, electrospinning and centrifugal spinning etc [6]. Melt spinning involves melting of the ceramic material to a very high temperature (usually > 2100 °C) and then spinning. This process consumes a lot of energy [6–10]. Template method involves coating of natural or synthetic template material with a sol followed by burning out the template to get the fibre. This increases the production cost. Extrusion has the problem of clogging of the sol in the spinnerette holes [6]. Nano ceramic fibres can be produced by electrospinning. Less productivity and high operating costs are the disadvantages of this method [6]. Considering the above pros and cons it was decided to use centrifugal spinning of sol followed by gelation.

Various defects such as sticking of fibres, shots, cracked fibres, curly fibres, core and sheath fibres, rough fibres are generally produced during spinning, drying and calcination of the fibres [10]. Among all the defects, shots remains as the major one and they reduce the thermal performance of fibre or fibre based products [11]. Though shots can be eliminated at a later stage also either by sieving or by washing [11], it is desirable to reduce them during synthesis stage itself so that the material loss can be minimized. This can be achieved by optimizing the spinning parameters. "Fibre Index" is used to define the ratio of weight percent of fibre to shots [11]. Low fibre index indicates more amount of shots which reduce thermal conductivity [11].

In the present work, synthesis of alumina fibres using sol-gel technique with centrifugal spinning has been reported. The principal objective of the present work is to investigate the influence of spinning parameters like rpm and viscosity etc. on fibre quality. Besides this, effect of preheating of the spinning chamber during fibre spinning and direction of hot air blown with respect to the spinnerette has also been explored.

2. Materials and methods

Aluminium nitrate nonahydrate (Al $(NO_3)_3 \cdot 9H_2O$, ANN, $\geq$ 95%, Merck) was dissolved in distilled water (pH = 7.02, conductivity = 18.2 μS/cm at 28 °C). Aluminium powder (Al, 99%, MMPIL, Nagpur, India, average particle size 3 μm (measured by DLS)) was added to the above solution. This made alumina precursor sol (APS). APS was refluxed at 110 °C in an oil bath. Removal of nitrate was confirmed by brown fumes

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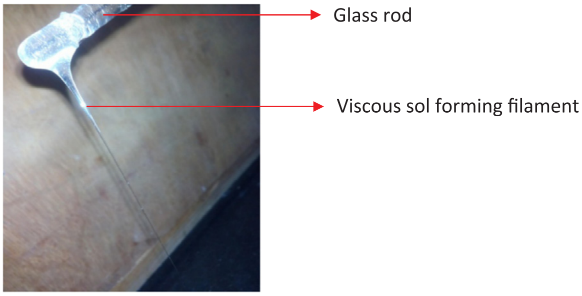

Fig. 1. Spinnability check of the sol - hand drawing of the viscous sol by a glass rod.

emanating during the refluxing step. The outgoing water vapor was condensed in order to maintain the concentration of the solution constant [8]. pH of all the sols before and after refluxing was 0 and 2 respectively. The refluxed sols were aged for different time periods to attain viscosities of 0.4 Pa.s, 8 Pa.s, 13 Pa.s, 21 Pa.s and 42 Pa.s. These viscosity values were measured at 28 °C using a parallel plate geometry (40 mm) in rotational mode by a rheometer (Model no.: Discovery HR-1, TA instruments). Besides viscosity, another two parameters, refractive index and weight loss during aging are proposed as markers to indicate spinnability of sols. Refractive index of sol aged to different time periods was measured by Reichert digital refractometer (AR200). Weight loss due to water removal during aging was also calculated. Spinnability of sol was checked by hand drawing the viscous sol with a glass rod (Fig. 1).

In order to understand the influence of spinning parameters (viscosity and rpm), sols with different viscosities obtained by aging, as mentioned above, were spun at three different rpms (500, 2000 and 3000 as 3000 is the maximum possible rpm for the present set up) in a self fabricated centrifugal spinning machine which had a spinnerette of 600 mm dia. Apart from this, few experiments were conducted to understand whether only preheating of the chamber to 150 °C and spinning helps in producing fibres at least for a batch process (~ 50 ml spinnable sol) and also to see whether hot air blower (HAB) direction influences the quality of the fibres synthesized. HAB was either kept tangential with respect to the spinnerette (in its direction of rotation) (Fig. 2(a)) or it was kept vertically down (along with the height of the spinnerette) (Fig. 2(b)).

The spun fibres were calcined at 1200 °C for 4 h. at a heating rate of 5 °C/min., sieved first by a 120 µm sieve and second by 90 µm sieve.

Aluminium nitrate nona hydrate+Aluminium powder + DI water

↓

Refluxing at 110°C for 4 hrs.

↓

Aging at 80°C for various time periods to achieve viscosities of 0.4 Pa.s, 8 Pa.s, 13 Pa.s, 21 Pa.s, and 42 Pa.s

↓

Centrifugal spinning of the sol

↓

Drying of the gel fibres at 100°C

↓

Calcination at 1200°C for 4 hrs.

↓

Phase formation (α - Alumina fibres)

↓

Sieving of the fibres using 120 µm sieve followed by 90 µm sieve

↓

Sintering of the fibres at 1600°C for 3 hrs.

Fig. 3. Flow chart of alumina fibre synthesis via sol-gel with centrifugal spinning [8,12].

Sieved fibres were sintered at 1600 °C for 3 h. The flow chart of the whole process is shown in Fig. 3.

Phase studies of the fibres at various stages were done by XRD (X'Pert Pro XRD Panalytical). Morphology of the fibres was imaged by SEM (Hitachi-S-3400N) and FESEM (JSM-7600F). Grain size of the fibres was measured by Image J software on the respective SEM / FESEM images.

3. Results and discussions

The overall reactions happen as per the Eqs. (1) and (2). Aluminium nitrate nonahydrate reacts with water and produces a complex ion. The

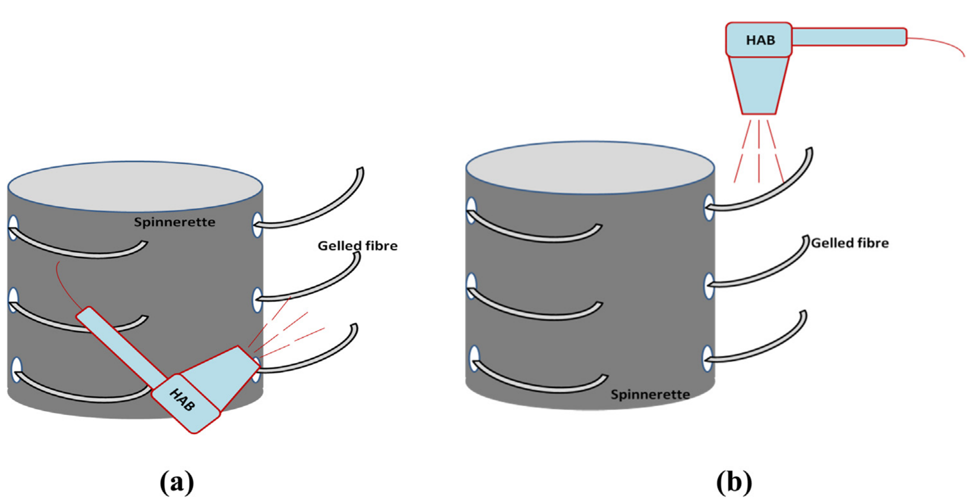

Fig. 2. Schematic representation of gelled fibres coming out of spinnerette when (a) HAB was in tangential direction (b) HAB was vertically down.