



EFFECT OF MAGNESIUM OXIDE ON SOL-GEL SPUN ALUMINA FIBRES

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ABSTRACT

Alumina fibres were prepared by sol-gel process. The sol was prepared by mixing aluminium-tri-isopropoxide in distilled water and refluxed in the presence of acid catalyst. Magnesium oxide was added in different wt.% (0,1,2) to the sol and stirred. 10 wt.% hydroxyethyl cellulose was added to the sol to obtain spinnability. At the appropriate viscosity, the sol was taken in a syringe and fibres were drawn in the ammonia solution. The fibers were dried at room temperature and then sintered at 1600°C for 2 hours. α -alumina is the only phase present in the sintered fibres is confirmed by X-ray diffraction analysis in corroboration with Fourier transform infrared spectroscopy. Differential thermal analysis indicated that the phase transition to α -alumina phase with sintering aid is higher. Thermogravimetric analysis indicated the removal of most of the volatile up to 600°C. Scanning electron microscopic analysis reveals as the MgO content increases to 2 wt.% the grain size decreases. Tensile strength is found to be good for sample containing 2 wt.% magnesium oxide.

Keywords: fibres; sol-gel process; sintering; Al_2O_3

1. INTRODUCTION

Conventionally ceramic fibers have been prepared by drawing or blowing the high temperature melt through the orifice. This melting method requires conversion of raw materials to a homogeneous, high temperature melt, making it difficult to prepare fibres of compositions, which are high melting or immiscible in the liquid state. In the case of glassy and amorphous fibres uncontrollable crystallization may inhibit fiber drawing from the melt¹. The sol-gel technique² applied to fibre preparation is free from these difficulties. In the sol-gel technique the sol is hydrolyzed and then condensed and/or polymerized until an appropriate viscosity for spinning is achieved for the production of alumina fibre³. Pure or high alumina polycrystalline fibres find wide applications in two major areas as reinforcement of metals or ceramics in the form of continuous fibres and as high temperature insulating material in the form of mats, blankets, boards etc⁴. The incorporation of alumina fibres in metal alloys and ceramics improves the mechanical properties (e.g. tensile strength, elastic modulus at low and elevated temperatures, wear resistance, etc.) and modifies some physical properties (e.g. density and thermal conductivity)⁵.

It has been long recognized that very small amounts of MgO additions can lead to densification of alumina to theoretical level⁶. One theory that is agreed upon frequently states that grain growth is inhibited by the reduction of grain boundary mobility upon forming a solid solution, i.e., by the mechanism of solid drag⁷. Other explanations for accelerated densification of alumina by MgO additions are increased lattice diffusion coefficients⁸. One of the most recent hypotheses is based on the assumption that grain boundary movement in alumina occurs by the addition and subtraction of atoms to and from kinks in steps on adjoining grains. It is suggested

that Mg^{2+} ion can poison these kinks in steps on attachment or detachment and thus reduce grain boundary mobility. Experimental evidence suggests that the role of MgO can be attributed mainly to its ability to lower the grain boundary mobility^{9,10}. In this paper the effect of magnesium oxide on sol-gel spun alumina fibres is reported.

2. EXPERIMENTAL PROCEDURES

Alumina sol was prepared according to the procedure described by Yoldas¹¹. Aluminium-tri-isopropoxide (CDH, New Delhi) was dissolved in distilled water in a molar Concentration of 1M refluxed at 80°C for 3 hours in the presence of acid catalyst (0.07 mole nitric acid) and then cooled. MgNO_3 was added such that the final composition contains 1 and 2 wt.% MgO in alumina sol. Hydroxyl ethylcellulose (HEC) was added as binder to the sol to impart green strength and plasticity. The sol was then concentrated by the removal of water to achieve required solid loading. When the sol was appropriate for forming fibres, it was taken in a 10-ml syringe and then fibres were drawn in a rectangular glass tray containing ammonia solution. The gelled fibres were taken out from the ammonia solution and dried at room temperature. The dried fibres were then sintered at 1600°C for 2 hours at a heating rate of 5°C/min.

The fibres were characterized using X-ray powder diffractometer with Cu-K_α radiation (SHIMADZU XD-DI), Thermal analyzer (NETZSH STA409PC) and Fourier Transform Infrared spectrometer by the KBr method (PERKIN ELMER RXI). The tensile strength was determined by Universal tensile testing machine (Instron 4301). Fibre was mounted with adhesive on chart paper tabs for aligning and gripping. A 5mm gauge length and a crosshead speed of 0.5mm/min was 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 set and the average values are reported.

3. RESULT AND DISCUSSIONS

The photograph of the sintered alumina fibres of Al_2O_3 -1wt.%MgO is shown in Fig.1. The diameter of the sintered fibre varies from 115-200 μm . Differential thermal analysis of alumina fibre containing 0, 1 and 2 wt.% MgO was performed at 10°C/min. and is shown in Fig.2. The curve has an endothermic peak at ~128°C corresponds to the loss of adsorbed water. The exothermic peak at ~215°C corresponds to decomposition of organic binder. The small exothermic peak around 1100°C in 10 wt.% HEC without MgO corresponds to crystallisation of α - Al_2O_3 . As the MgO content increases to 2 wt.% the transformation temperature of α - Al_2O_3 is not apparent, so a first order derivative was taken and it was found that, the α - Al_2O_3 phase crystallization temperature was found at ~1250°C which is higher compared to the temperature reported by Saraswathi et al¹². This is due to resistance to the phase transformation increase with decreasing grain size as reported by Lange et al¹³.

The TGA curves of alumina fibre containing 0, 1 and 2 wt.% MgO is shown in Fig.3. It shows a total weight loss of 48%, 51% and 53% respectively. The curve shows three stages of weight loss at different temperatures, one up to 200°C and other between 200 and 300°C, the first two stages of weight loss, which is attributable to loss of adsorbed water and organic binder burn out, as is evident from the DTA curve, shown in Fig.3. The weight loss between 300 and 500°C is due to the loss of structural water (decomposition of structural -OH group). FTIR analysis was performed for both the gel and oxide fibres in the wavenumber region of 4000-400 cm^{-1} . The spectra of dried and sintered alumina fibre at different temperature are shown in Figs.4 and 5. The absorption recorded at about 3450 cm^{-1} ¹⁴ is due to the presence of molecular water. This is also present in the fibres sintered at 1600°C for 2 hours. This may be due to the absorption of moisture during testing. The broad band at 2060 cm^{-1} ¹² indicates the presence of hydrogen

bonded OH. This is absent in sintered fibre. The absorption at 1650 cm^{-1} due to O-H bending mode confirming the presence of adhesive water. This is also present in sintered fibre due to moisture absorption. The bands observed at 1380 cm^{-1} and 1070 cm^{-1} ¹⁶ correspond to Al-OH bending mode. These absorption bands are also present in sintered fibre. The broad band in the region 540 cm^{-1} ¹⁷ corresponds to Al-O vibration mode. This is absent in sintered fibre. In addition to that, a band is observed at 610 cm^{-1} ¹⁸ corresponds to AlO_6 vibration. A well-defined absorption at 460 cm^{-1} corresponds to Al-O vibration of $\alpha\text{-Al}_2\text{O}_3$ ¹⁶. Fig.6. shows the X-ray diffraction patterns of dried fibres containing 0, 1, 2 wt.% MgO. The dried fibre exhibit very broad peak ascribed to the presence of boehmite phase. The phase present at 1200° , 1400° and 1600°C , when heated for 2 hours is $\alpha\text{-Al}_2\text{O}_3$ (Fig.7). Fig.8 shows the microstructure of alumina fibre with 0, 1 and 2 wt.% MgO. As the MgO content increases to 2 wt.% the grain size decreases. The grain size of alumina fibre without MgO varies from 1-4 μm to as the MgO content increases to 2 wt.%. The decrease in grain size varies from 0.35-2.7 μm . This might be due to grain growth inhibition by the reduction of grain boundary mobility upon forming a solid solution, i.e., by the mechanism of solid drag⁷ or due to increased lattice diffusion coefficients⁸. The tensile strength values are shown in Table 1. The tensile strength of alumina fibre was found to increase with increasing MgO. This is due to the decrease in grain size, as is evident from SEM analysis of Alumina fibres containing α -alumina phase was prepared by sol-gel process. The tensile strength was found to increase with increasing MgO content, due to decrease in grain size.

4. ACKNOWLEDGEMENT

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5. REFERENCES

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TABLE AND FIGURES

Table.1 Tensile strength values of alumina fibres

Sample	Tensile Strength (MPa)
Al_2O_3	128 ± 10
Al_2O_3 -1wt.%MgO	162 ± 10
Al_2O_3 -2wt.%MgO	183 ± 10

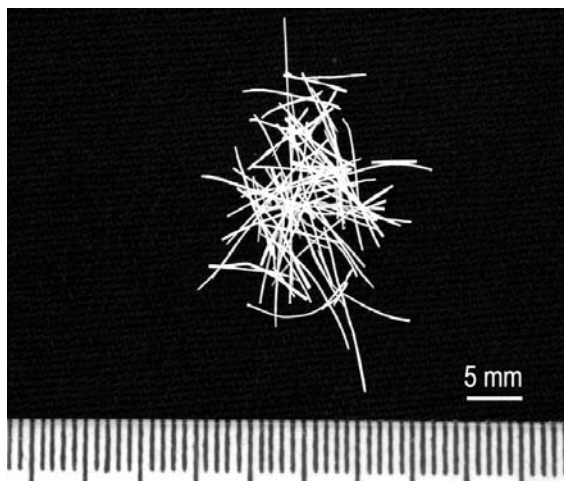


Fig.1. Photograph of sintered Al_2O_3 -1 wt.% MgO

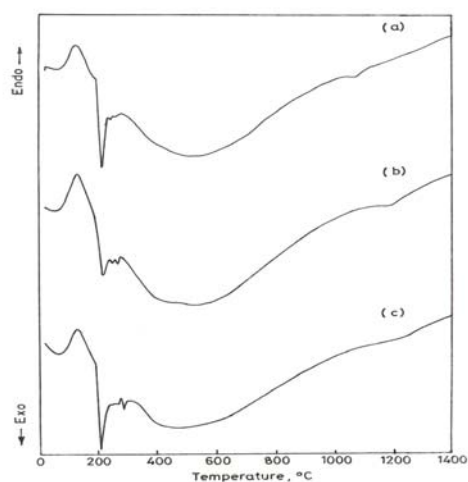


Fig.2. Differential thermal analysis of alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO (c) 2 wt.% MgO

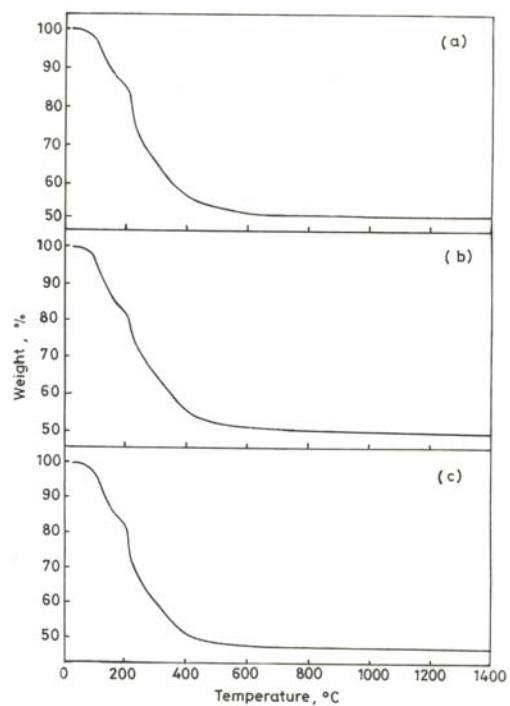


Fig.3. Thermogravimetric analysis of alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO (c) 2 wt.% MgO

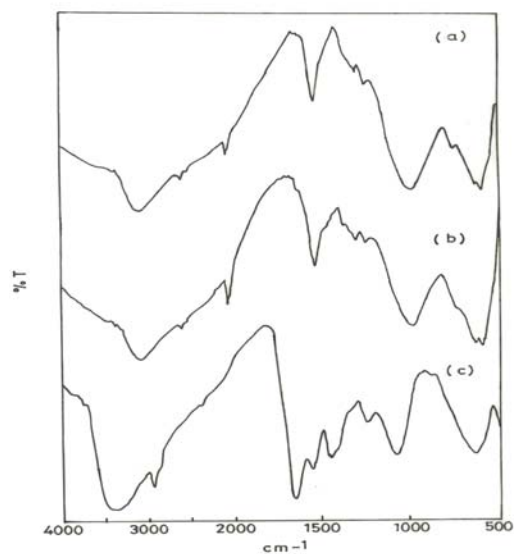


Fig.4. FTIR analysis of dried alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO (c) 2 wt.% MgO

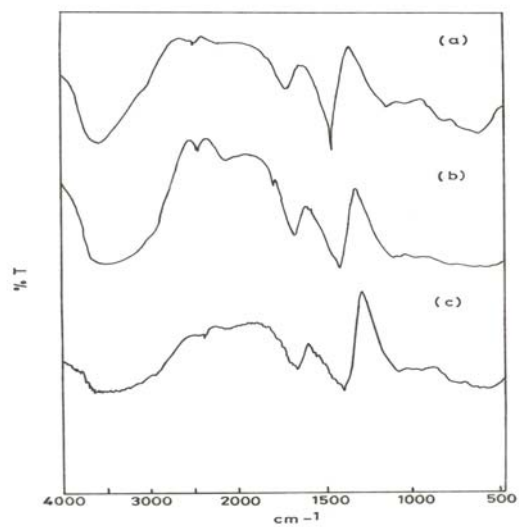


Fig.5. FTIR analysis of sintered alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO
(c) 2 wt.% MgO

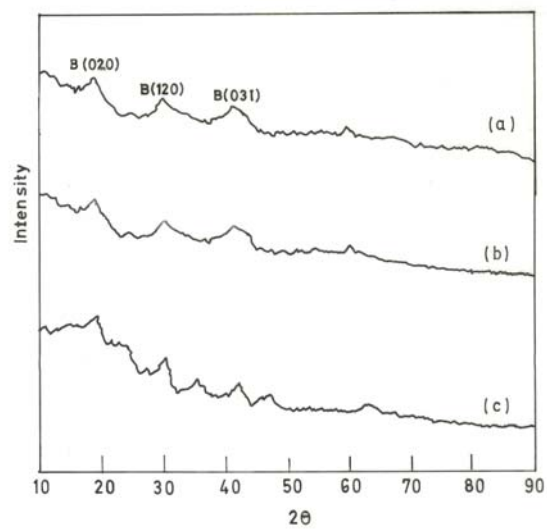


Fig.6. X-ray diffraction pattern of dried alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO
(c) 2 wt.% MgO

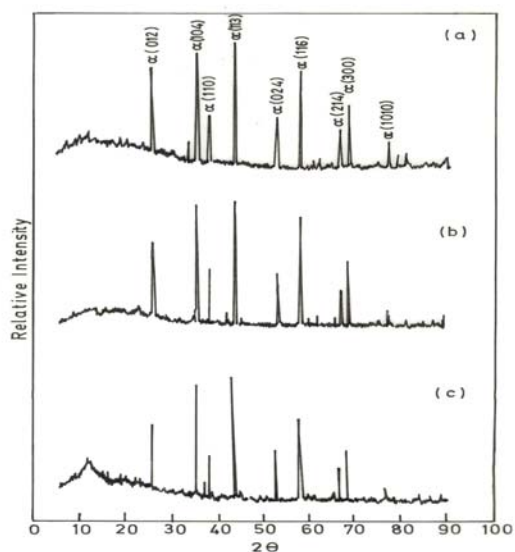


Fig.7. X-ray diffraction pattern of sintered alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO (c) 2 wt.% MgO

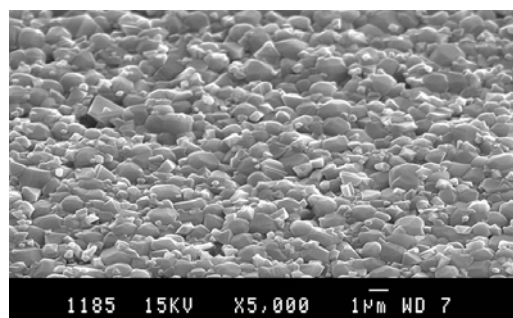
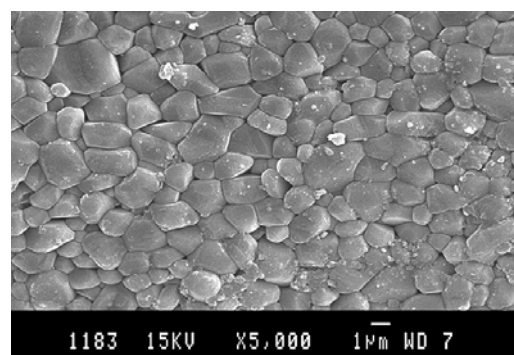
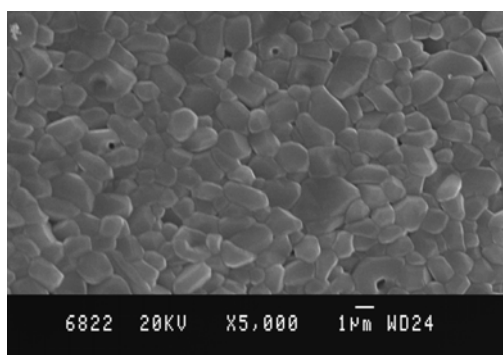


Fig.8. SEM micrograph of alumina fibre (a) 0 wt.% MgO (b) 1 wt.% MgO (c) 2 wt.% MgO