PREPARATION AND CHARACTERIZATION OF SPIN COATED ALUMINA FILM

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ABSTRACT

Aluminium oxide or alumina, the most widely used fine ceramics material was synthesized using sol-gel method. The yield harvested was spin coated on alumina and quartz substrates. The films were subjected to sintering at 600ºC and 1000ºC. The effect of temperature on the surface morphology of the film was investigated using SEM and AFM analysis.

KEYWORDS: Alumina film, sol gel – spin coating process, FTIR, SEM, AFM.

1. INTRODUCTION

Ceramics are classified as oxides and non-oxides depending on the methods of processing; however oxide ceramics such as alumina are widely used because of their favourable chemical and thermal compatibilities.[1] The need to enhance the electrical and mechanical properties of materials, led to the development of oxide ceramics.[2] Alumina or aluminium oxide with the chemical formula Al_2O_3, is a polymorphic white granular material having melting point about 2,000°C and specific gravity about 4.0. It is insoluble in water and organic liquids and very slightly soluble in strong acids and alkalis. Based on the differences in crystal structure, aluminium oxide exists in many forms of which α-Al_2O_3, which is frequently called the corundum, is found to exhibit the highest stability.[3]

Alumina is conventionally synthesized by sol-gel process; the advantages of sol-gel method are its versatility and the possibility of obtaining high purity materials, the provision of easy introduction of trace elements, synthesis of special materials with low energy consumption at low processing temperature.[4] The sol-gel method is preferred among the other techniques, as it produces pure samples with large specific surface area and provides better synthesis environment.[5] Spin coating method, a combination of outward fluid flow and evaporation is predominantly used for producing uniform thin films which are used in the manufacture of storage magnetic disks, monitor screens and integrated circuits, apart from being used as coatings, abrasives, heat resistant materials and cutting tools in industries.[6,1] In addition to chemical inertness, these materials are characterized by favourable thermomechanical and wear resistant properties.[7,2] Aluminum oxide as a thin film is being used in a wide variety of applications such as wear protective coatings, high temperature and semiconductor devices owing to the excellent combination of mechanical and electrical properties.[8] Fabrication of magnetic head recording due to the highest hardness, low impurities and appreciable combination of chemical stability and fracture resistance properties are the predominant characteristics of Alumina film.[9]

This paper reports the synthesis of Alumina by sol-gel technique, preparation of Alumina films and the characterization studies.

2. EXPERIMENTAL PROCEDURE

Alumina is synthesized using sol-gel technique. Sol-gel technique is a most feasible low temperature technique. Inorganic aluminum chloride ($AlCl_3$) was used as the precursor in the synthesis of Al_2O_3. 0.1M of $AlCl_3$ and ethanolic solution (sol) was prepared which was stirred vigorously for several hours at room temperatures resulting in the formation of clear sol. The addition of a suitable additive disperses the inorganic salts in solvent forming a stable sol and eliminating the cracks often encountered in the sol-gel process.[10] The gel was formed with the addition of 28% NH_3 as additive.

The gel was coated on quartz and alumina substrates which were well cleaned with dilute HF solutions. At the speed of 5000 rpm and at 60 s, the resulting product was dried at room temperature for 15 min and then sintered at 600ºC and 1000ºC for 2 hours to form a dry gel on the substrate. The gel was left to maturate for 30 hours at room temperature and then dried at 100ºC for 30 minutes. The characterization study was performed for as prepared powder, sintered powder, alumina and quartz films which were subjected to Fourier Transform Infrared Spectroscopic (FTIR), Scanning Electron Microscopic (SEM) and Atomic Force Microscopic (AFM) analysis.

3. RESULTS AND DISCUSSION

3.1 Fourier Transform Infrared Spectroscopy

The FTIR spectra of the as prepared and sintered samples (1000ºC) were measured in the range 400 to 4000 cm^{-1} as shown in Fig. 1 & Fig. 2. The stronger broadening band 3600-3100 cm^{-1} occurs due to the hydrogen band between the various hydroxyl groups in the as prepared and sintered sample. The stronger broadening bands between 600-450 cm^{-1} corresponds to Al-O vibration. The peaks were in the range 1200-500 cm^{-1} and between 500-450 cm^{-1}. The IR spectrum of 1000ºC sintered powder highlights transmission peaks at around 3500 cm^{-1} indicating O-H stretching and bending mode that imply some hydroxyl still present and their bonds strengths were high. The absorption band in the range 450-600 cm^{-1} represents Al-O stretching mode.

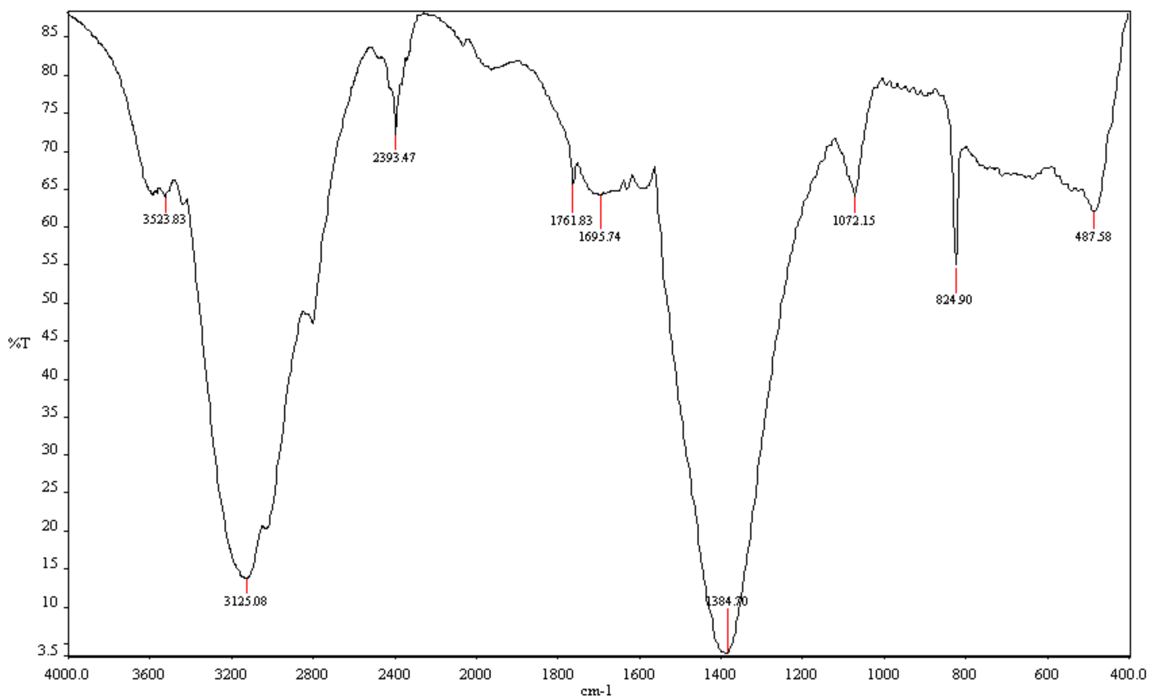


Fig. 1. FTIR Spectrum of As Prepared Alumina Powder

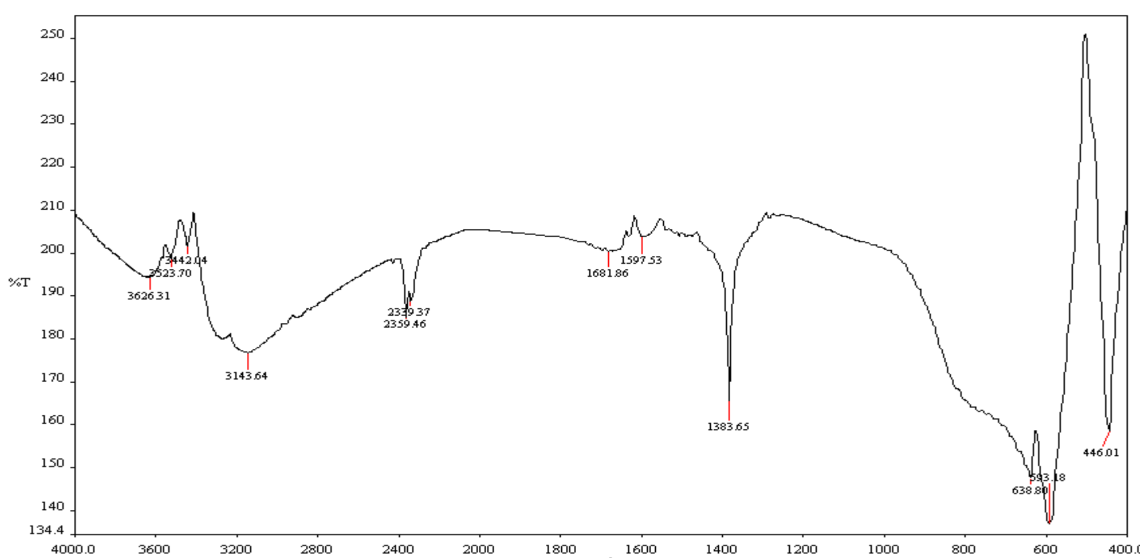
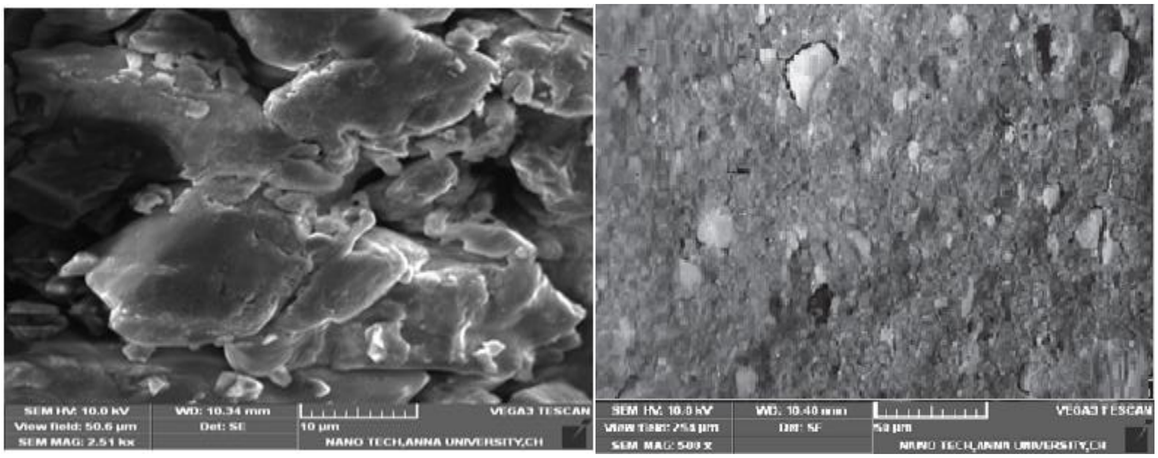


Fig. 2. FTIR Spectrum of Sintered Alumina Powder

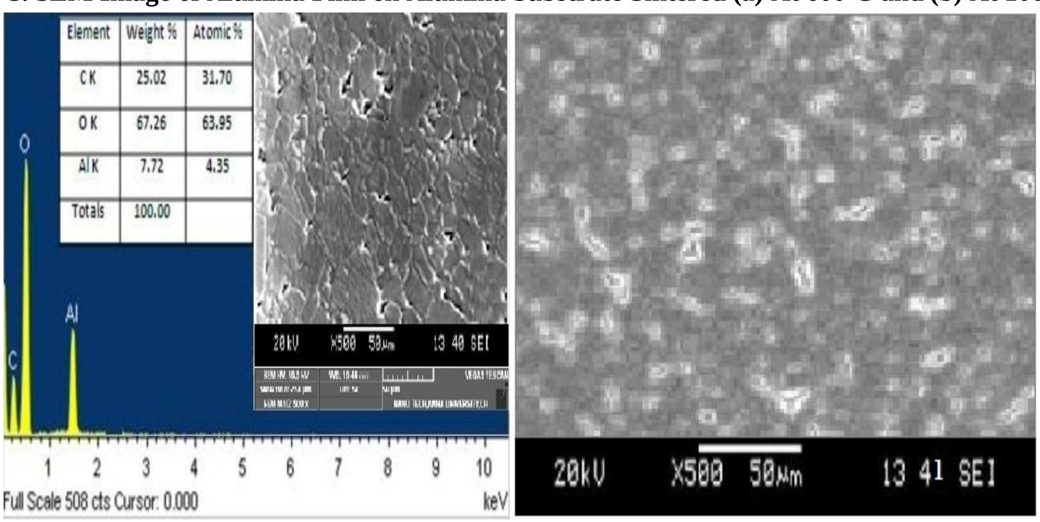
3.2 Scanning Electron Microscopy

The SEM images of Alumina film on alumina substrate sintered at 600ºC and 1000ºC is shown in Fig. 3(a & b). The Alumina film sintered at 600ºC shows coarse particle with lot of agglomeration in Fig. 3(a). This infers the presence of residues (small substances) present in sol. The SEM images of Alumina film on quartz substrate sintered at 600ºC and 1000ºC is highlighted in Fig. 4(a & b). The presence of Al and O in all the spectra is evident and small amount of unburnt carbon in the films. The films sintered at different temperatures indicate some dense structure and cracks on the surface. The Alumina film on alumina and quartz sintered at 600ºC shows coarse particles with greater agglomeration and more cracks on the surface than the sintered alumina films at 1000ºC.



(a) 600ºC (b) 1000ºC

Fig. 3. SEM Image of Alumina Film on Alumina Substrate Sintered (a) At 600ºC and (b) At 1000ºC



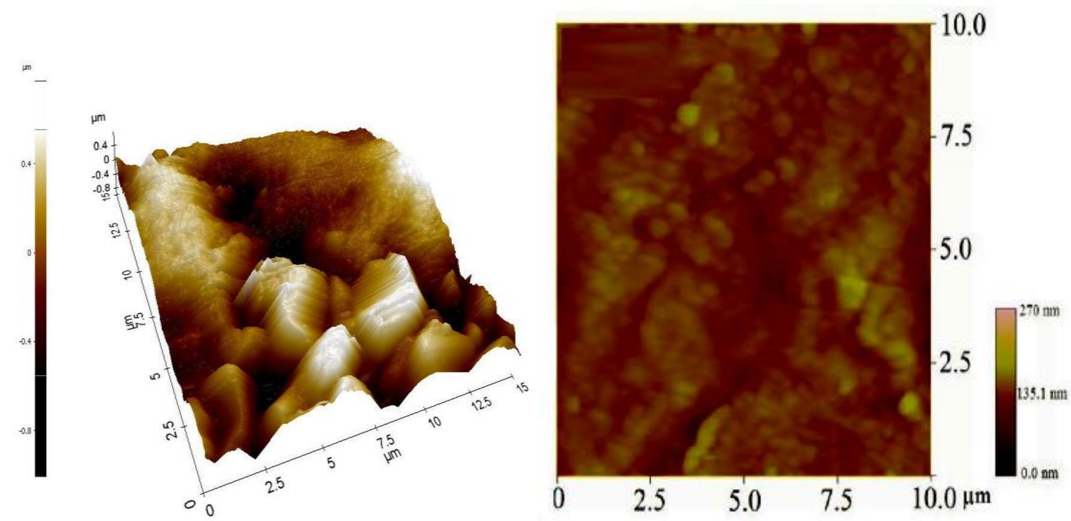
(a) 600ºC (b) 1000ºC

Fig. 4. SEM Image of Alumina Film on Quartz Substrate Sintered (a) At 600ºC And (b) At 1000ºC

3.3 Atomic Force Microscopy

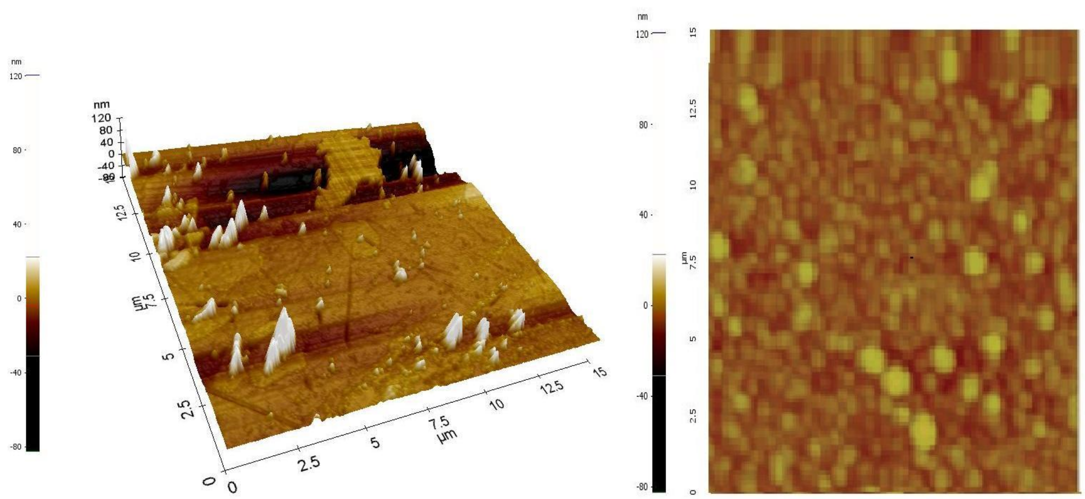
The results presented in AFM images distinctly reveal that Alumina films prepared at 600ºC and 1000ºC on alumina and quartz substrates have quite different surface topographies. The presented images show that the AFM technique is a very useful tool for studying film morphology, providing insight, in agreement to that obtained by SEM. The AFM images of Alumina film on alumina substrate sintered at 600°C, indicate the presence of randomly distributed small particles having size from 40nm up to 40–60nm as shown in Fig. 5(a). These particles may be interpreted as the residue after decomposition of inorganic part of the alumina precursor in the first steps of the heat treatment. Samples sintered at higher temperature (1000ºC), are homogeneous and exhibit less agglomeration as shown in Fig. 5(b).

The AFM images of Alumina film on quartz substrate sintered at 600°C, indicate the presence of randomly distributed small particles and cracks on the surface as shown in Fig. 6(a). Film sintered at higher temperatures show discontinuities as small pores visible in the alumina coating sintered at 1000°C, due to the shrinkage of the material at this temperature as shown in Fig. 6(b).



(a) 600ºC **(b) 1000ºC**

Fig. 5. AFM Image of Alumina Film on Alumina Substrate Sintered (a) At 600ºC and (b) At 1000ºC



(a) 600ºC **(b) 1000ºC**

Fig. 6. AFM Image of Alumina Film on Quartz Substrate Sintered (a) At 600ºC and (b) At 1000ºC

4. CONCLUSION

Alumina films formed via sol-gel technique and spin coating process is commonly prone to the formation of cracks. This is due to the application of polydentates, which results in the agglomeration of particles causing bond weakening and crack propagation. In order to eliminate the crack often encountered in film formation, monodentate ammonia is used as an additive. The additive combines with the inorganic solvent to form a stable sol. These alumina films formed can be used as armor plating for protection of tanks in aeronautics, hydraulic parts, enameling the surface of insulators and other ceramic materials like tiles. The detailed study on the different ways of coating methods and surface morphology will have appreciable impact in future for optimizing parameters like film homogeny, particles shape and size and effect on properties. Sintering causes densification at higher temperatures leading to standardization of the surface of the film.

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