

# We are IntechOpen, the world's leading publisher of Open Access books Built by scientists, for scientists

6,900

Open access books available

185,000

International authors and editors

200M

Downloads

Our authors are among the

154

Countries delivered to

TOP 1%

most cited scientists

12.2%

Contributors from top 500 universities



WEB OF SCIENCE™

Selection of our books indexed in the Book Citation Index  
in Web of Science™ Core Collection (BKCI)

Interested in publishing with us?  
Contact [book.department@intechopen.com](mailto:book.department@intechopen.com)

Numbers displayed above are based on latest data collected.  
For more information visit [www.intechopen.com](http://www.intechopen.com)



## Synthesis of Alumina Nanofibers and Composites

Mohamad Ridzuan Noordin<sup>1</sup> and Kong Yong Liew<sup>1,2</sup>

<sup>1</sup>*Faculty of Industrial Science and Technology, Universiti Malaysia Pahang, Kuantan,*

<sup>2</sup>*College of Chemistry and Materials Science,  
South Central University for Nationalities, Wuhan,*

<sup>1</sup>*Malaysia*

<sup>2</sup>*China*

### 1. Introduction

Alumina,  $\text{Al}_2\text{O}_3$ , is a ceramic metal oxide of great importance as building material, refractory material, electrical and heat insulator, attributed to its high strength, corrosion resistance, chemical stability, low thermal conductivity, and good electrical insulation. One phase of alumina, the  $\gamma$ -phase is widely used as catalyst, catalyst support and adsorbent because its high porosity and surface area. It is conventionally prepared by heating the hydrated trihydroxides, gibbsite and bayerite, to temperature of  $> 1000^\circ\text{C}$  to obtain the  $\alpha$ -alumina, corundum, a material with great hardness and low surface area. The trihydroxides, gibbsite, bayerite, both are  $\text{Al}(\text{OH})_3 \cdot n\text{H}_2\text{O}$  and the monohydroxyl oxide, boehmite,  $\text{AlO}(\text{OH}) \cdot a\text{H}_2\text{O}$ , have the monoclinic crystal structure with similar lattice parameters ( $a=0.866$  nm,  $b=0.506$  nm,  $c=0.983$  nm,  $\beta=94^\circ34'$  in boehmite,  $a=0.868$  nm,  $b=0.507$  nm,  $c=0.972$  nm,  $\beta=94^\circ34'$  in gibbsite, and  $a=0.867$  nm,  $b=0.506$  nm,  $c=0.942$  nm,  $\beta=90^\circ26'$  in bayerite [1, 2]. In the process of heat treatment, the trihydroxide undergoes a series of transformations. It loses the water of hydration, then dehydroxalate at  $< 300^\circ\text{C}$  to form the monohydroxyl oxide boehmite,  $\text{AlO}(\text{OH})$ , which on further heating to increasingly higher temperatures, changes to the transition aluminas, including the  $\eta$ -,  $\gamma$ -,  $\delta$ -, and  $\theta$ -phases, which then transform finally to the  $\alpha$ -form. These transition aluminas are crystalline solids with high porosity and surface areas as well as acidic and basic properties, which make them suitable as adsorbents, catalyst, catalyst support and fabricated into filtration membranes as well as used as fillers or components in polymer/ inorganic composite materials with enhanced mechanical properties. The conventional alumina as obtained are usually powder of particulates. These could be fabricated into alumina fibers by various method, through melt growth techniques including the internal crystallization method and extrusion, electrospraying [3] and electrospinning [4-8] have been developed for producing  $\text{Al}_2\text{O}_3$  fibers.

The high surface areas of transition aluminas are due to the presence of pores. These pores are irregular in sizes and size distributions, and are mainly micropores of diameter less than 2 nm. Since the synthesis of mesoporous materials was first reported in the late 1980s, a number of methods for the synthesis of mesoporous alumina have been reported [8,9]. More recently, interest in nano-materials and nanotechnology has spurred a flurry of investigations

Source: Nanofibers, Book edited by: Ashok Kumar,  
ISBN 978-953-7619-86-2, pp. 438, February 2010, INTECH, Croatia, downloaded from SCIYO.COM

on the preparation and the applications of nano-materials including that of inorganic oxides such as aluminum oxide. Nanoparticles can be of 1, 2 or 3 dimensions. One dimensional nano-structured alumina may be described as fiber, wire, belt, rod, laths and tubes [9-17]. Nanofibers are one dimensional material with high aspect ratio of length to diameter of 1000:1 and diameter of 1 to 100 nm, which may be solid fibers or hollow nanotubes. The final properties of these nanofibers depend on the size and homogeneity of the fibrous structure, which shall enable tailoring  $\text{Al}_2\text{O}_3$  nanofibers to advanced technological usage with the enhanced sintering ability, strength, catalytic activity, and absorption ability. However, for the present report, lower aspect ratio materials are also included.

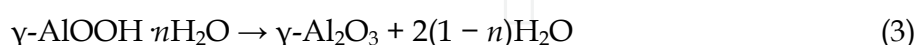
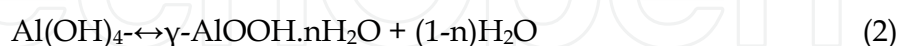
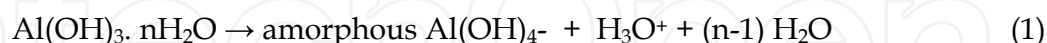
## 2. Synthesis of alumina nanofibers

Various routes including hydrothermal or solvothermal, sol-gel techniques, electrospinning and extrusion, template techniques [19,20], chemical vapor deposition in flame[30], and mercury mediated technique [29] have been developed for producing  $\text{Al}_2\text{O}_3$  nanofibers. Many of the nanofibers have been produced using hydrothermal treatment, with or without surfactant, [18-25] and sol-gel [17,27] techniques of various alumina precursors, subsequent calcinations dehydrate the solid from the sol-gel solution produced by the hydrothermal process. The solid, which consists of nanofibrous boehmite, may be calcined to obtain the  $\alpha$ -alumina nanofibers. The gel has high viscosity and may be electrospun to obtain the nanofibers. The solid either from the hydrothermal or sol-gel processes or the electrospun fibers is dehydrated by heat treatment leading to transition aluminas and finally to the nanofibrous  $\alpha$ -alumina. Other methods such as vapor-liquid-solid deposition to burn aluminum together with silica nanoparticles at high temperature between 1300 and 1700 °C, and by using other 1-D materials as hard template have also been reported [13-15, 19, 30]. Most of the earlier reports are on the generation of  $\text{Al}_2\text{O}_3$  fibers of diameters in the submicrons to several tens of microns range. Only more recently did the synthesis of nanofibrous alumina appeared. However, information regarding the growth and particulate properties of  $\text{Al}_2\text{O}_3$  nanofiber still remains very limited. For the hydrothermal and sol-gel processes, the aluminum precursors could be Al metal, the alkoxide or aluminum salts. The size and morphology of aluminum hydrate nanoparticles synthesized by these processes can be controlled by manipulating the pH value of the reaction mixture, the hydrothermal temperature and aging time, the solvent and the surfactant used. [2,3,20-27]

### 2.1 Hydrothermal and sol gel processes

The first step in the synthesis of nanofibrous alumina both these processes is the hydrolysis of the starting aluminum compound or elemental Al powder to the hydrated hydroxide of aluminum, boehmite, which are then aged subsequently. In the hydrothermal process, the aging could be done in an enclosed reactor under its own autogenic pressure, the colloidal solution obtained is either filtered and the solid further processed. In the sol gel process, the sol is transformed to a gel after the aging. The two processes are closely related and the reactions that take place are similar, these processes are discussed together. The boehmite formed in the first step has the formula,  $\text{AlOOH} \cdot n\text{H}_2\text{O}$ . It may be amorphous particles initially which then transform into network structures linked by hydrogen bonds and forms nanosheets or sheets of crumpled nanostructures. The nanosheets could be rolled up and be transformed into hollow tubular structures or nanofibers[17, 31], which may or may not be crystalline. The initial spherical particles of boehmite are sol solution with low viscosity. As

it aged, the network structures and fibers are formed and the sol changed into gel with high viscosity. The solid in the gel is initially spherical nanoparticles [17], which on aging gradually transform into the crystalline boehmite. The solid on heating to higher temperature dehydrates and dehydroxylates forming the different phases of transition aluminas in stages at progressively higher temperature than 400°C, which finally transforms to the final  $\alpha$ -alumina at temperature of 1000°C or higher. The reactions may be represented as follows [13-16, 18, 19, 21-27, 31]:



The  $\gamma$  crystalline boehmite is formed under suitable synthesis condition and the morphology is retained in the various transition aluminas as well as in the final  $\alpha$ -alumina as demonstrated by the TEM and Xrd characterizations, although the crystal structures are different. The boehmite formed nanosheets in the sol and gelling process and the curled up fibers or tubes were visible in the TEM images of the boehmite.

Alumina nanofibers can be synthesized with metallic aluminum particles [10,22, 43, 59], Al alkoxides [36; 46, 17] or its salts [27, 45, 47, 48, 49, 51] as the starting materials. Other starting materials that have been used include Al pentanedionate, [50], aluminum hydroxide, [52], alumina composite, [53] aluminum film, [42] and sodium aluminate. [54] However, whatever the starting materials used in the synthesis, the nanofibrous boehmite intermediate will have to be formed [22] for the formation of the nanofibers as mentioned above. The starting Al precursor is first hydrolyzed with water to form the hydroxide [17, 22, 32, 37, 38], aged to the viscous gel fibers of hydrated AlOOH, which can be calcined directly or electrospun or drawn from the viscous gel and converted to ceramic fibers after firing [16, 27, 31-37]. The metallic aluminum powder used for the process is prepared by exploding the metal wire by a high density current pulse normally produced by discharging a capacitor bank. The produced particles are spherical of about 80-120 nm [22, 43].

For the case in which metallic Al nanoparticles is used the first step of the reactions are depicted as follows:[17, 22]



or



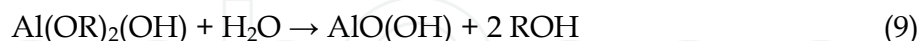
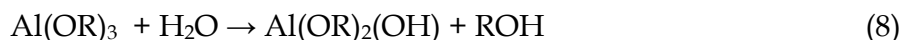
reaction (5) leads to boehmite, while reaction (6) leads to formation of the trihydroxide, bayerite, which on dehydration via a series of transformation forms the boehmite:



The boehmite will further dehydrate to convert to the transition aluminas which will further dehydrate to  $\gamma\text{-Al}_2\text{O}_3$  up to 700°C without significant change in morphology. With the aluminum salts, hydrolysis occurs forming aluminum hydroxide, which then proceeds as in

eq.7. Na aluminate reacts with acid to form the aluminum hydroxide and proceeds to be dehydrated similarly.

With alkoxides,  $\text{Al(OR)}_3$ , as the starting materials, the initial hydrolysis with water yield alcohol and the hydroxylated aluminum centers as follows: [17]



In the initial stage of the sol gel reaction, small three dimensional oligomers are formed with the  $-\text{Al-OH}$  on the outer surface. Linkage of  $-\text{Al-OH}$  groups occurs via polycondensation and results in oxypolymers. When sufficient interconnected  $\text{Al-O-Al}$  bonds are formed, they respond cooperatively to form the colloidal particles or a sol. These sol particles aggregate and increase in size with time forming three dimensional network and randomly distributed spherical nanoparticles as shown by TEM in the earlier stage of the process. With further aging, the nanosheets are shown to curl up forming the nanofibers. The morphology was seen to be preserved even after calcinations at  $1200^\circ\text{C}$  [17,21, 45, 55].

The size and morphology of the end product of the hydrolysis and subsequent dehydration of the intermediate boehmite from the hydrolysis depend on the conditions in which these processes are performed. The primary factors involved the nature of the starting material, whether a surfactant or templating agent is used, the amount of water used, the aging time in the hydrolysis, the pH value of the reaction mixture, and the hydrothermal temperature [17-19, 21-23, 45-47, 55-59]. Even a simple procedure of stirring the hydrolyzing reaction mixture of Al particles in water, affect the outcome of the resulting hydroxylate product. Without stirring, boehmite was produced, while with stirring, the trihydroxide, bayerite, was resulted.[22]. In the sol-gel synthesis using isopropoxide as the starting material [17], the reaction had to be carried out at the range of  $R_w=60-65$ , outside of this range no fibrous material was formed. Under acidic conditions, hydrolysis was enhanced and that the rate of polycondensation and aggregation to larger particles was increased too high for nanofibers formation. Additionally, the presence of organic solvent influences the reaction rate significantly. Of the solvent tested only isopropanol in the hydrolysis mixture led to nanofibers formation. Although the morphology is retained during the calcinations of the nanofibrous alumina, the physical property such as the pore size increased with calcinations temperature from the as prepared samples to  $1000^\circ\text{C}$ , however, the surface area decreased at temperature  $> 550^\circ\text{C}$ . Electron microscopic observations of microstructure, together with studies of the evolution of X-ray and electron diffraction patterns, have been used to provide mechanistic information on the dehydration of boehmite to  $\gamma\text{-Al}_2\text{O}_3$ . Calcinations of boehmite at  $400^\circ\text{C}$  produces slow development of a fine, lamellar, porous microstructure, oriented parallel to  $(001)_\gamma$ , corresponding to the loss of oxygen atoms of the boehmite lattice. The mechanism proposed for the dehydration is controlled by diffusion in a direction determined by the hydrogen bond chains in the boehmite structure and involves counter migration of the Al cations and protons with formation of voids in a coherent cubic close-packed matrix. Final reorganization to give the spinel structure of  $\gamma\text{-Al}_2\text{O}_3$  is suggested to involve gradual filling of the tetrahedral cation sites [3].



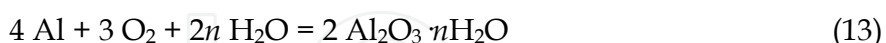
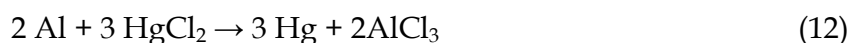
To control the hydrolysis and the polycondensation rates, much works have been reported for the synthesis of the nanofibers with the addition of various surfactants and other additives as templating agents to direct the growth of the fibers. Alumina nanofibers have been successfully grown with the use of polyethylene oxide polyethylene glycol, hexamethyltetraamine, and organic solvents [14, 15, 17, 25, 41, 45, 48]. The surfactant directs the fibrils growth by forming rodlike micelles. Alumina nanofibers were successfully prepared by electrospinning technique using aluminum acetate and aluminum nitrate as alumina precursor or other different precursors with a combination of PVA and PEO as polymer precursor [48]. Recently, a one pot synthesis of high area alumina nanofibers were synthesized from aluminum isopropoxide and acetic acid in supercritical CO<sub>2</sub> (SCF) as the solvent has been described in which the water in the process was limited by the reaction between the alkoxide and the acetic acid [60]. Another report on the use of supercritical CO<sub>2</sub> as a drying agent has also been described for the synthesis of unidirectional nanostructures such as nanotubes, nanofibers, and nanorods. The morphology of the final product depends on the nature of the employed surfactants [25].

Another method to ensure the formation of the nanofibrous alumina is by using the hard template route. Aluminum surfaces created by anodic oxidation electrically with nanosize pores could be used as the nanoreactors for the synthesis of the oxide fibers [14, 15, 19].

## 2.2 Other methods

### 2.2.1 Mercury mediated method

Alumina nanofibers were successfully synthesized in mercury media at room temperature. The procedure consisted of immersing high purity aluminum strips in HgCl<sub>2</sub> solution for a few minutes, removed and exposed to air. After some time, the white alumina powder was formed on the surface of the aluminum strips. The as-grown aluminas were heated and calcined in air at 850°C for 2 h to. The product was initially amorphous with diameters of 5-15 nm and length of several μm but converted to γ-alumina after calcinations. During the process, the following reactions took place:



The Al reacted with mercuric chloride forming Al amalgam by metathesis and got oxidized by air when removed from the solution. The dissolution of the Al into the Hg was a continuous process. The final product is the alumina nanofibers [28].

### 2.2.2 Flame aerosol method

Flame aerosol technology is used for large-scale manufacture of ceramic commodities such as pigmentary titania, fumed silica and alumina. The dynamics of formation and growth of these particles are reasonably well understood. The formation of alumina nanofibers in carbon-containing coflow diffusion flames with pure oxygen support, using trimethylaluminum vapor as the precursor has been reported recently. The alumina nanofibers formed were non-crystalline, typically 2–10 nm in diameter and 20–210 nm in length. These fibers were mixed with spherical nanoparticles along with carbon nanotubes formed at the same time. No alumina nanofiber formation was found in flames supported

by air or in pure  $H_2$  flames. The alumina nanofibers were formed from gas-phase aluminum-containing species in the flame. Gas-phase carbon-containing species such as CO or hydrocarbon were probably crucial to the formation of the alumina nanofibers. The nanofibers were formed in the region past the maximum temperature zone of the flame. Sufficiently high temperature may be due to the higher concentration of aluminum in the gas phase, while the presence carbon nanotubes or nanofibers may serve as templates or mediating agent for the alumina nanofibers formation [29, 64].

### 2.2.3 Vapor-Liquid-Solid process

Crystalline alumina nanowires were synthesized at elevated temperatures in a catalyst assisted process using iron as catalyst. Nanotrees formed by alumina nanowires were also found. The typical nanowires are crystalline of size around 50 nm in diameter and around 2  $\mu\text{m}$  in length. The tree trunks of the nanotrees are around 100 nm in diameter and around 10  $\mu\text{m}$  in length. The results are explained in terms of growth mechanism based on a vapor-liquid-solid (VLS) process. The process involved mixing commercially available aluminum powder, iron powder and silicon carbide powder in an appropriate ratio and then sprinkled on tungsten (W) boat. The boat was then placed at the center of the vacuum bell jar and evacuated down to about  $5.0 \times 10^{-2}$  Torr. The boat was gradually heated up by passing current through it, and kept at  $1700^\circ\text{C}$  for 1 h under flowing argon. The treated powders contained in the W boat were taken out and cooled down to room temperature in flowing argon [64]. Single-crystal  $\alpha\text{-Al}_2\text{O}_3$  fibers by vapor-liquid-solid deposition from aluminum and silica powder and  $\text{Al}_2\text{O}_3$  nanowires were synthesized by heating a mixture of Al,  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$  catalyst.  $\text{Al}_2\text{O}_3$  nanowires and nanotrees have also being grown on silicon carbide particles' surface in thermal evaporation process and using iron as catalyst [20].

## 2.3 Electrospinning

Electrospinning is a old technology for the production of polymer fibers. This process has experienced renewed interest for the synthesis of nanofibers of polymers, ceramics, and their composites. The process uses electrical force to produce fibers with nanometer-scale diameters from its solution. Nanofibers have a large specific surface area and a small pore are being used or finding uses in filtration, protective clothing, biomedical applications including wound dressings and drug delivery systems, structural elements in artificial organs, and in reinforced composites. Recently, this potentially commercially viable process has been much investigated for the production of ceramic including alumina nanofibers [4-7, 51, 53].

The process setup consists of a capillary tube, connected to a reservoir of the colloidal solution or melts, with a small orifice through which the fluid could be ejected and a collector. A high voltage is used to create an electrically charged jet of solution or melt out of the capillary solution fluid. This induces a charge on the surface of the droplet formed at the tip of the capillary and held by its surface tension. As the intensity of the electric field is increased, the hemispherical surface of the droplet at the tip of the capillary tube elongates to form a conical shape known as the Taylor cone. On further increasing the electric field, a critical value is attained with which the repulsive electrostatic force overcomes the surface tension and the charged jet of the fluid is ejected. The ejected colloidal solution jet undergoes

an instabilization and elongation process, which causes the jet to become very long and thin. Meanwhile, the solvent evaporates, leaving behind a charged fiber. In the case of the melt the discharged jet solidifies when it travels in the air.

Many reports have appeared in the literature in the past few years on the fabrication of alumina nanofibers as the viscosity of the boehmite gel is well suited for the electrospinning technique [5,34, 48,50,51,53]. The gel solution for the spinning consisted of nanofibrous colloid prepared by the sol-gel or hydrothermal processes, however, the precursor mixture may also be used. A typical solution for electrospinning is prepared by mixing suitable concentration of the solution of an aluminum salt with a polymer solution such as polyvinyl alcohol or polyethylene oxide. The distance between the capillary tip and the collector electrode, the flow rate, the voltage were adjusted suitably. Polymer-alumina composite nanofibers were obtained which can then be processed by further drying or calcinations [48].

### 3. Alumina nanofiber composites

Alumina nanoparticles and nanofiber show interesting properties such as ability to be formed into structures that enhance the functions of osteoblast for bone replacement [65, 66], to form ultrathin alumina hollow fiber microfilm membrane [67] for separation processes, to form novel nanofilter for removal and retention of viral aerosols [68] and to serve as high performance turbidity filter [69]. The preparation of the final form of alumina fiber may involve the use of binders such as acid phosphate and silica colloid binder or polymers [70]. In the case where polymer was used it would be burnt off during high temperature treatment of the prepared.

Many composite materials comprising of alumina nanoparticles or nanofibers as minor or major component in the presence of polymer or inorganic substrate also show similar interesting properties. S. Sundarajan [71] for example explored the fabrication of nanocomposite membrane comprising of polymer and alumina nanoparticles for protection against chemical warfare stimulants and found alumina based materials to be prospective candidates. While traditionally alumina film has been used as protective film on metal substrate [72, 73], alumina presence thermoplastic and thermosetting polymeric materials are also gaining wide application as surface coatings. Landry [74] for example studied the preparation of alumina and zirconia acrylate nanocomposites for coating wood flooring. It was found that for both the alumina and zirconia nano-composites, the conversion of acrylate resin is faster and more important when silane is used as the coupling agent.

The addition of the nanoparticles and nanofibers is meant to enhance the mechanical and thermal properties compared to in absence of such constituents. It is generally agreed that the large surface to volume ratio of the nanoscale constituents plays a key role to the improvement.

#### 3.1 Alumina in polymer substrate

The effect of alumina in polyaniline, diglycidyl ether of bisphenol A type epoxy resin, carbon fiber epoxy resin composite and PA1010 has been studied recently. Generally it has been found that the presence of low loading of alumina nanoparticles and nanofibers tend to enhance the thermal and mechanical properties of the polymer matrix. In many instances



the strength of the composites are below the strength of neat resin due to non-uniform particle size distribution and particle aggregation. Ash et.al (as noted in [75]) studied the mechanical behavior of alumina particulate/poly(methyl methacrylate) composites. They concluded that when a weak particle matrix interphase exists, the mode of yielding for glassy, amorphous polymers changes to cavitation to shear, which leads to a brittle-to-ductile transition.

Two challenges have been observed to be overcome to facilitate the enhancement of the properties of the polymer substrate. First is the need to disperse the nanoparticles and nanofibers uniformly throughout the polymer substrate and secondly to facilitate the interaction between the nanofibers and the molecules of the polymer substrate. At low loading the nanoparticles could be distributed uniformly across the substrate. However at high loading, there is the tendency for the fibers to cluster together and hence limit the enhancement of the mechanical properties and in fact lowered it. It has also been observed that while the tensile strength increases with reduction in particles size for micron-scale particles, the tensile strength decreased with reduced particle size for nano-scale particles [76]. The changes were attributed not to the strength of bonding between the particulate with the matrix but more to the poor dispersion of particles.

Various preparative methods have been adopted to facilitate good dispersion. This include mechanical milling [77] and Mechanical milling followed by hot extrusion [78]. In both studies the alumina nanofibers was found in the as sintered product. M.I Flores-Zamora concluded that the presence of alumina based nanoparticles and nanofibers seemed to be responsible for the reinforcement effect.

Attempts to meet the second challenge involve functionalizing alumina particles such as in the on fiber and epoxy resin composites [79]. The functionalizing of the alumina surfaces is meant to enhance the miscibility of the alumina particles in the polymer substrate and also the facilitate bridging between alumina surfaces with the substrate. It was however observed that where the functionalised alumina, L-alumoxane is miscible with the resin, high loading results in a marked decrease in performance due to an increase in brittleness. This was proposed to be due to weak inter-phase bonding between resin and the alumina fiber.

### **3.2 Alumina in inorganic and carbon based substrate**

The composites comprising of carbon and alumina is also of significant interest. Study on this category of composite material includes fabrication of macroscopic carbon nanofiber (CNF)/alumina composite by extrusion method for catalytic screening [80]. It was found that the synthesized composite possessed a mesoporous structure with a relatively high surface area (340 m<sup>2</sup>/g) and a narrow particle size distribution, displaying a good thermal stability. A comparison of the surface acidity between the composite and commercial alumina demonstrated that the total number of acid sites on composite was significantly increased along the distinct decrease in the number of strong acid sites, which may enhance the activity and anti coking property as a promising industrial catalyst support in petroleum industry.

Hirato [81] fabricate carbon nanofiber-dispersed alumina composites by pulsed electric-current pressure sintering and their mechanical and electrical properties. High bending and

fracture strength were observed on the composites compared to that of monolithic alumina. The electrical resistivity of the composite material was also observed to reduce by  $10^{17}$  order of magnitude. Xia [82] studied the fracture toughness of highly ordered carbon nanotube/alumina nanocomposites. The result of the study demonstrate that nanotube bridging/sliding and nanotube bridging necessary to induce nanoscale toughening, and suggest the feasibility of engineering residual stresses, nanotube structure and composite geometry to obtain high toughness nanocomposites.

### 3.3 Future development of alumina nanocomposite

There are wide applications of alumina nanofiber composites. These potential would continue to drive research interest in this field. In the case of applying alumina composites for application that is dependent on its strength, future work is like to further elucidate the factors that would first facilitate good dispersion of the fiber in the host matrix and second that would enhance the interphase interaction between alumina nanofiber and the matrix. The effect of additive that could facilitate dispersion or/and bonding between alumina fiber and the matrix may be of great interest. The application for catalysis may be of special interest especially the matrix between alumina and carbon nanofibers and similar substances. It may be argued that catalysts are in nearly all cases composite materials, however the interaction between purposely prepared nano size precursors may yield effect not seen when prepared through the traditional mode.

## 4. References

- [1] H. Knoezinger, R. Ratnasamy, Catal. Rev. (1978), 17(1):31
- [2] V. Saraswati, G.V.N. Rao, G.V. Rama Rao, Structural evolution in alumina gel, J. Mater. Sci. (1987), 22(7):2529;
- [3] S.J. Wilson, The dehydration of Boehmite,  $\gamma$ -AlOOH, to  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, J. Solid State Chem. 30(2), (1979):247
- [4] A. Jawaorek, A.T. Sobczyk, Electrospinning route to nanotechnology: an overview, J. Electrostatic (2008), 60:197
- [5] A.M. Azad, Fabrication of transparent alumina nanofibers by electrospinning, Mater. Sci. Engineer. A. (2006), 435-436:468;
- [6] T. Subbiah, G.S. Bhat, R.W. Tock, S. Parameswaran, S.S. Ramkumar, Electrospinning of nanofibers, J. Appl. Polym. Sci., (2005), 96(2):557;
- [7] D. Li, Y. Xia, Electrospinning of nanofibers: reinventing the wheel?, Advanced Mater. (2003), 16(14):1151
- [8] K. Niesz, P. Yang, G.A. Somorjai, Chem. Comm., (2005), 1986,
- [9] S.A. Bagshaw, T.J. Pinnavaia, Mesoporous Alumina Molecular Sieves, Angew. Chem. (Intern.Ed.English) (1996), 35 (10): 1102-1105.
- [10] X. S. Peng, L. D. Zhang, G. W. Meng, X. F. Wang, Y. W. Wang, C. Z. Wang, G. S. Wu, Photoluminescence and Infrared Properties of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> Nanowires and Nanobelts, J. Phys. Chem. B, 2002, 106 (43)
- [11] J.C. Hulthen, A general template-based method for the preparation of nanomaterials, J. Mater. Chem. (1997), 7(7):1075;

- [12] L. Pu, X. Bao, J. Zou, D. Feng, Individual alumina nanotubes, *Angew Chem.* (2001), 40(8):1490;
- [13] H. Hou, Y.Xie, Q. Yang, Q. Guo, C. Tan, Perapration and characterization of  $\gamma$ -AlOOH nanotubes and nanorods, *Nanotechnol.*, (2005), 16(6):714;
- [14] Y. Xia, Y. Yang, P. Sun, Y.Wu, B Mayers,, Gates, Y.Yin, , F.Kim, , H.Yan, One-dimensional nanostructures: Synthesis,characterization, and applications, *Advanced Mater.*, 15(5),(2003):353
- [15] J. G. Lu, P. Chang, Z. Fan, Quasi-one-dimensional metal oxide materials—Synthesis, properties and applications *Mater. Sci. Engineer. R: Reports*, 52(1-3), (2006): 49-91
- [16] W. Cai, J. Yu, S. Mann, Template-free hydrothermal fabrication of hierarchically organized  $\gamma$ -AlOOH hollow microspheres, *Microp. Mesop. Mater.* 122(1-3), (2009):42
- [17] G. L. Teoh, K. Y. Liew, W. A. K. Mahmood, Synthesis and characterization of sol-gel alumina nanofibers, *J. Sol-Gel Sci. Technol.*, (2007) 44(3): 177
- [18] M.G. Ma , J.F. Zhu, A facile solvothermal route to synthesis of  $\gamma$ -alumina with bundle-like and flower-like morphologies, *Mater. Lett.* 63 (2009): 881;
- [19] Y. Zuo, Y. Zhao, X. Li, N. Li, X. Bai, S. Qiu, W. Yu Synthesis of alumina nanowires and nanorods by anodic oxidation method, *Mater. Lett.*, 60( 24),(2006):2937;
- [20] Y.Z. Jin, Y.Q. Zhu, K. Brigatti, H.W. Kroto, D.R.M. Walton, Catalysed growth of novel aluminium oxide nanorods *Appl. Phys. A: Mater. Sci. Proc.*, 77(1), (2003): 113.;
- [21] H. C. Lee, H. J. Kim, S. H. Chung, K. H. Lee, H. C. Lee, J. S. Lee, Synthesis of Unidirectional Alumina Nanostructures without Added Organic *Solvents* *J. Am. Chem. Soc.*, (2003), 125 (10):2882
- [22] J. H. Park, M. K. Lee, C. K. Rhee, W. W. Kim, Control of hydrolytic reaction of aluminum particles for aluminum oxide nanofibers. *Mater Sci.Eng. A*, 375-377, (2004):1263
- [23] W. Cai, H. Li, Y. Zhang, Influences of processing techniques of the  $H_2O_2$ -precipitated pseudoboehmite on the structural and textural properties of  $\gamma$ - $Al_2O_3$ . *Colloids Surf. A: Physicochem. Engin Asp*, 295( 1-3), (2007), 185
- [24] V. Valcárcel, A. Pérez, M. Cyrklaff, F. Guitián, Novel Ribbon-Shaped  $\alpha$ - $Al_2O_3$  Fibers, *Advanced Mater.* 10(16):1370
- [25] H. Y. Zhu, X. P. Gao, D. Y. Song, Y. Q. Bai, S. P. Ringer, Z. Gao, Y. X. Xi, W. Martens, J. D. Riches, R. L. Frost, Growth of Boehmite Nanofibers by Assembling Nanoparticles with Surfactant Micelles. *J. Phys. Chem. B*, (2004), 108 (14): 4245
- [26] W. Cai, H. Li, Y. Zhang, Azeotropic distillation-assisted preparation of macro-mesostructured  $\gamma$ - $Al_2O_3$  nanofibres of crumpled sheet-like morphology, *Mater Chem. Phys.*, 96(1),(2006):136;
- [27] J. Wang, Y. Wang, M. Qiao, S. Xie, K. Fan, A novel sol-gel synthetic route to alumina nanofibers via aluminum nitrate and hexamethylenetetramine, *Mater. Lett.*, 61(28),(2007):5074;
- [28] Q. Yang, Y. Deng, W. Hu, Synthesis of alumina nanofibers by a mercury-mediated method. *Ceram. Intern* 35(1), (2009):531

- [29] B. Guo, H. Yim, Z.P. Luo Formation of alumina nanofibers in carbon-containing coflow laminar diffusion flames, *J. Aerosol Sci.*, 40(4), (2009):379
- [30] A. Jagminas, J. Kuzmarskytė, L. Malferrari, M. Cuffiani A new route of alumina template modification into dense-packed fibrilous material, *Mater. Lett.*, 61(14-15), (2007):2896
- [31] R. Ma, Y. Bando, T. Sasaki, Directly rolling nanosheets into nanotubes, *J. Phys. Chem.B*, 108(7), (2004):211
- [32] J. Wang, Y. Wang, M. Qiao, S. Xie, K. Fan, A novel sol-gel synthetic route to alumina nanofibers via aluminum nitrate and hexamethylenetetramine, *Mater. Lett.*, 61(28), (2007):5074
- [33] X. Liu, Z. Wu, T. Peng, P. Cai, H. Lv, W. Lian Fabrication of alumina nanofibers by precipitation reaction combined with heterogeneous azeotropic distillation process, *Mater. Res. Bul.*, 44(1), (2009):160-167
- [34] V. Maneeratana, W. M. Sigmund Continuous hollow alumina gel fibers by direct electrospinning of an alkoxide-based precursor, *Chem. Engineer. J.*, 137(1), (2008):137
- [35] J. Chandradass, M. Balasubramanian, Extrusion of alumina fibre using sol-gel precursor *J. Mater. Sci.*, 41(18), (2006):6026
- [36] E. Elaloui, A. C. Pierre and G. M. Pajonk, Influence of the Sol-Gel Processing Method on the Structure and the Porous Texture of Nondoped Aluminas, *J. Catal.*, 166(2), (1997):340
- [37] J. Chandradass, D.S. Bae, D.S., M. Balasubramanian, Synthesis and characterization of sol-gel alumina fiber by seeding  $\alpha$ -alumina through extended ball milling, *Mater. Manuf. Proc.* 23(8), (2008):786
- [38] R. Venkatesh, P.K. Chakrabarty, B. Siladitya, M. Chatterjee, M. D. Ganguli, Preparation of alumina fibre mats by a sol-gel spinning technique, *Ceramics Intern.*, 25(6), (1999):539-543
- [39] G. Zhihong, N. Guilin, L. Yuan, C. Yu, Heat-induced transformation between nanospheres and nanofibers of boehmite, *Proc. 1st IEEE-NEMS 2006*, Article number 4135091, Pages 886-889
- [40] R. Ma, Y. Bando, T. Sasaki, Directly rolling nanosheets into nanotubes, *J. Phys. Chem.B*, 108(7), (2004):2115
- [41] Z. Zhu, H. Liu, H. Sun, D. Yang, PEG-directed hydrothermal synthesis of multilayered alumina microfibers with mesoporous structures, *Microp Mesop. Mater.*, 123( 1-3), (2009): 39-44
- [42] Y. Kobayashi, T. Ishizaka, Y. Kurokawa, Preparation of alumina films by the sol gel method., *J. Mater Sci.*, (2005), 40:263
- [43] F. Tepper, L. Kaledin, Nanosize electropositive fibrous adsorbent, (2005) US patent:6838005;
- [44] KMS. Khalil, Synthesis of Short Fibrous Boehmite Suitable for Thermally Stabilized Transition Aluminas Formation, *J Catal.* (1998), 178(1):198
- [45] J. Wang, Y. Wang, M. Qiao, S. Xie, K. Fan, A novel sol-gel route to alumina nanofibers via aluminum nitrate and hexamethylenetetramine, *Mater. Lett.* (2007), 61(28):5074.

- [46] T. Yogo, H. Iwahara, Synthesis of  $\alpha$ -alumina from modified aluminum alkoxide precursor, *J. Mater Sci.*, (1992), 27:1499;
- [47] J.F. Hochepped, P. Nortier, Influence of precipitation conditions :pH and temperature on the morphology and porosity of boehmite particles, (2002), *Powder Technol*, 128(2-3), 268
- [48] P.K. Panda, S. Ramakrishna, Eletrospinning of alumina nanofibers using different precursors, *J. Mater Sci*, (2007),42: 2189
- [49] K. Song, K. Woo, Y. Kang, Preparation of alumina fibers from aluminum salts by the sol gel methods, *Korean J. Chem. Eng.* 16(1), (1999):75
- [50] A.M. Azad, Fabrication of transparent alumina nanofibers by electrospinning, *Mater. Sci. Eng. A*, (2006), (435-473):468
- [51] R.W. Tuttle, A. Chowdury, E.T. Bender, R.D. Ramsier, J.L. Rapp, M.P. Espe, Electrospun ceramic fibers: Composition, structure and the fate of precursors, *Appl. Surf. Sci.*,(2008), 254:4928;
- [52] SC. Shen, W.K. Ng,Q.Chen, X.T. Zeng, R.B.H. Tan, Novel synthesis of lace-like nanoribbons of boehmite and  $\gamma$ -alumina by dry gel conversion method, *Mater lett*, (2007),61(21):4280 steam crystallization
- [53] K.M. Sawicka, P.Gouma, Electrospun composite nanofibers for functional applications, *J. Nanopart. Res.*(2006), 8:769
- [54] H.Y. Zhu, X.P. Gao, D.Y. Song, S.P. Ringer, Y.X. Xi, R.L. Frost, Manipulating the size and morphology of aluminum hydrous oxide nanoparticles by soft chemistry approaches, *Microp. Mesop. Mater.* (2005), 85(3):226
- [55] H.Y. Zhu, J.D. Riches and J.C. Barry,  $\gamma$ -alumina nanofibers prepared from aluminum hydrate with poly(ethylene oxide) surfactant, *Chem. Mater.*, 14, (2002): 2086
- [56] X. Liu, Z. Wu, T. Peng, P. Cai, H. Lv, W. Lian Fabrication of alumina nanofibers by precipitation reaction combined with heterogeneous azeotropic distillation process, *Mater. Res. Bul.*, 44(1), (2009):160
- [57] H.Y. Zhu, X.P. Gao, D.Y. Song, Simon P. Ringer, Y.X. Xi, Ray L. Frost, Manipulating the size and morphology of aluminum hydrous oxide nanoparticles by soft-chemistry approaches, *Microp. Mesop. Mater.* 85(3), (2005):226
- [58] W. Cai, H. Li, Y. Zhang, Influences of three post-precipitation techniques, *Colloids Surf. A: Physicochem. Engin Asp*, 295( 1-3), (2007), 185,
- [59] M. Thiruchitrambalam, V. R. Palkar, V. Gopinathan, Hydrolysis of aluminium metal and sol-gel processing of nano alumina, *Mater Lett.*, 58(24), (2004):3063
- [60] M. B. I. Chowdhury, R. Sui, R. A. Lucky, P. A. Charpentier, A One-Pot Procedure to Synthesize High Surface Area Alumina Nanofibers using Supercritical Carbon Dioxide, *Langmuir* (in press)
- [61] H. C. Lee, H. J. Kim, C. H. Rhee, K. H. Lee, J. S. Lee, S. H. Chung, Synthesis of nanostructured  $\gamma$ -alumina with a cationic surfactant and controlled amounts of water, *Microp. Mesop. Mater.*, 79(1-3), (2005): 61-68
- [62] J. Zhang, F. Shi, J. Lin, S. Y. Wei, D. Chen, J. M. Gao, Z. H., X. X. Ding, C. Tang, Nanoparticles assembly of boehmite nanofibers without a surfactant, *Mater. Res. Bul.*, 43(7), (2008): 1709



- [63] S. E. Pratsinis, Flame aerosol synthesis of ceramic powders, *Prog. Energy Comb. Sci.*, 24(3), (1998):197
- [64] J. Zhou, S. Z. Deng, J. Chen, J. C. She, N. S. Xu, Synthesis of crystalline alumina nanowires and nanotrees, *Chem. Phys. Lett.*, 365(5-6). (2002):505
- [65] R.L. Price, K.M. Heberstroh, T.J. Webster, 2003, Enhanced functions of osteoblasts on nanostructured surfaces of carbon and alumina, *Med. Biol. Eng. Comp.*, (2003):41, 372.
- [66] T. J. Webster, E. L. Hellenmeyer, R. L. Price, Increased osteoblast function on theta + delta nanofiber alumina, *Biomaterials* 26, (2005): 953
- [67] I. H. Choi, I. C. Kim, B. R. Min, K. H. Lee, Preparation and characterization of ultrathin alumina hollow fiber microfiltration membrane, *Desalination*, 193 (2006):256
- [68] H. Li, C. Wu, F. Tepper, J. Lee, C. N. Lee, Removal and retention of viral aerosols by a novel alumina fiber filter, *Aerosol Science* 40, (2009): 65
- [69] F. Tepper, L. Kaledin, C. Hartmann, High Performance Turbidity Filter, February 2005, 55-57
- [70] J.A. Fernando, D.D.L. Chung, Thermochemical properties of alumina fiber membrane, *Ceram. Intern.*, 31, (2005): 453
- [71] S. Sundarajam, S. Ramakrishna, Fabrication of nanocomposite membranes from nanofibers and nanoparticles for protection against chemical warfare stimulants, *J. mater. Sci.*, (2007), 42: 8400
- [72] C. Jing, X. Xu, J. Hou, Preparation of compact Al<sub>2</sub>O<sub>3</sub> film on metal on metal for oxidation resistance by polyvinylpyrrolidone, *J Sol-Gel Sci Technol* (2007) 43: 321
- [73] C. Jing, X. Xhao, Y. Zhang, Elimination of a zero-growth in thickness of Al<sub>2</sub>O<sub>3</sub> protective film deposited by cycles of dip coating method, *J Sol-Gel Sci Technol* (2007) 42: 151
- [74] V. Landry, B. Riedl, P. Blanchet, Alumina and zirconia acrylate nanocomposites coatings for wood flooring: Photocalorimetric characterization, *Progr. Org. Coatings*, 61, (2008):76
- [75] E. T. Thostenson, C. Li, T. Chou, Nanocomposites in context, *Composites Sci. Techn.*, 65 (2005): 491
- [76] L. Sun, R. F. Gibson, F. Gordaninejad, J. Suhr, Energy absorption capability of nanocomposites: A Review, *Composites Sci. techn.* 69 (2009):2392
- [77] A. Santos-Beltran, V. Gallegos-Orozco, I. Estrada-Guel, L. B. Gomez, F. E. Magana, M. M. Yoshida, R. M. Sanchez, *J. Alloys Comp.* 434-435, (2007):514
- [78] M.I. Flores-Zamora, I. Estrada-Guel, J. Gonzalez-Hernandez, M. Miki-Yoshida, R. Martinez-Sanchez, Aluminium-graphite composites produced by mechanical milling and hot extrusion, *J. Alloys Comp.* 434-435, (2007):518
- [79] N. Shahhid, R. G. Villate, A. R. Barron, Chemically functionalized alumina nanoparticles effect on carbon/epoxy composites, *Composites Sci. Techn.* 65 (2005):2250
- [80] J.H. Zhou, C. Chen, R. Guo, X. C. Fang, X. G. Zhou, Synthesis and characterization of carbon nanofiber/alumina composites by extrusion casting, *Carbon* 47 (2009): 2077

- [81] K. Hirota, Y. Takaura, M. Kato, Y. Miyamoto, J. Mater. Sci (2007), 42: 47920.
- [82] Z. Xia, W.A. Curtin, B.W. Sheldon, Fracture toughness of highly ordered carbon nanotube/alumina nanocomposites, Transaction of ASME, 126,(2004):239

IntechOpen

IntechOpen



## **Nanofibers**

Edited by Ashok Kumar

ISBN 978-953-7619-86-2

Hard cover, 438 pages

**Publisher** InTech

**Published online** 01, February, 2010

**Published in print edition** February, 2010

“There's Plenty of Room at the Bottom” this was the title of the lecture Prof. Richard Feynman delivered at California Institute of Technology on December 29, 1959 at the American Physical Society meeting. He considered the possibility to manipulate matter on an atomic scale. Indeed, the design and controllable synthesis of nanomaterials have attracted much attention because of their distinctive geometries and novel physical and chemical properties. For the last two decades nano-scaled materials in the form of nanofibers, nanoparticles, nanotubes, nanoclays, nanorods, nanodisks, nanoribbons, nanowhiskers etc. have been investigated with increased interest due to their enormous advantages, such as large surface area and active surface sites. Among all nanostructures, nanofibers have attracted tremendous interest in nanotechnology and biomedical engineering owing to the ease of controllable production processes, low pore size and superior mechanical properties for a range of applications in diverse areas such as catalysis, sensors, medicine, pharmacy, drug delivery, tissue engineering, filtration, textile, adhesive, aerospace, capacitors, transistors, battery separators, energy storage, fuel cells, information technology, photonic structures and flat panel displays, just to mention a few. Nanofibers are continuous filaments of generally less than about 1000 nm diameters. Nanofibers of a variety of cellulose and non-cellulose based materials can be produced by a variety of techniques such as phase separation, self assembly, drawing, melt fibrillation, template synthesis, electro-spinning, and solution spinning. They reduce the handling problems mostly associated with the nanoparticles. Nanoparticles can agglomerate and form clusters, whereas nanofibers form a mesh that stays intact even after regeneration. The present book is a result of contributions of experts from international scientific community working in different areas and types of nanofibers. The book thoroughly covers latest topics on different varieties of nanofibers. It provides an up-to-date insightful coverage to the synthesis, characterization, functional properties and potential device applications of nanofibers in specialized areas. We hope that this book will prove to be timely and thought provoking and will serve as a valuable reference for researchers working in different areas of nanofibers. Special thanks goes to the authors for their valuable contributions.

### **How to reference**

In order to correctly reference this scholarly work, feel free to copy and paste the following:

Mohamad Ridzuan Noordin and Kong Yong Liew (2010). Synthesis of Alumina Nanofibers and Composites, Nanofibers, Ashok Kumar (Ed.), ISBN: 978-953-7619-86-2, InTech, Available from:  
<http://www.intechopen.com/books/nanofibers/synthesis-of-alumina-nanofibers-and-composites>

**INTECH**  
open science | open minds

[www.intechopen.com](http://www.intechopen.com)

**InTech Europe**

University Campus STeP Ri  
Slavka Krautzeka 83/A  
51000 Rijeka, Croatia  
Phone: +385 (51) 770 447  
Fax: +385 (51) 686 166  
[www.intechopen.com](http://www.intechopen.com)

**InTech China**

Unit 405, Office Block, Hotel Equatorial Shanghai  
No.65, Yan An Road (West), Shanghai, 200040, China  
中国上海市延安西路65号上海国际贵都大饭店办公楼405单元  
Phone: +86-21-62489820  
Fax: +86-21-62489821

IntechOpen

IntechOpen

© 2010 The Author(s). Licensee IntechOpen. This chapter is distributed under the terms of the [Creative Commons Attribution-NonCommercial-ShareAlike-3.0 License](https://creativecommons.org/licenses/by-nc-sa/3.0/), which permits use, distribution and reproduction for non-commercial purposes, provided the original is properly cited and derivative works building on this content are distributed under the same license.

IntechOpen

IntechOpen