Synthesis and Characterization of α-Al_2O_3 Nanorods Prepared by a Simple Aluminum-Water Reaction

M. A. Shah and J. Kumar*
Department of Physics, Faculty of Science, King Khalid University, Post Box 9100 Abha, Kingdom of Saudi Arabia
**Crystal Growth Center, Anna University, Chennai-India*

α-Al_2O_3 nanorods have been successfully synthesized by a simple reaction of aluminum metal powder with water in the temperature range of 50-200^{o}C. The Field Emission Scanning Electron Microscopy (FESEM) shows that the diameters of nanorods are relatively uniform, ranging from 40-60 nm, with the length of several micrometers. Nanoribbons-like nanostructures are believed to result from a combination of increased reaction time and temperature. A plausible mechanism is proposed for the formation of these nanorods/nanoribbons and is expected that this synthetic technique can be extended to obtain other metal oxides.

1. Introduction

One-dimensional nanostructures have received considerable attention as a result of their peculiar and fascinating properties and applications superior to their bulk counterparts. The ability to generate such minuscule structures is essential for modern science and technology. While the materials with one dimensional nanostructures have potential applications in magnetic, optoelectronic and micromechanical devices, and so on, they also contribute to the basic fundamental understanding of physical and chemical properties of materials as the transition from bulk to the confined nano scale occurs [1-4].

Aluminum oxide is known to exist in a number of metastable polymorphs in addition to thermodynamically stable α-Al_2O_3 or corundum form. This material is one of the most versatile ceramic oxides and has been used in a wide range of applications in engineering and biomedical areas depending on its purity and crystallinity. It is widely used as a catalyst, an adsorbent and as a support for industrial catalysis in hydrocarbon conversion. Owing to its special properties, such as high elastic modulus, thermal and chemical stability, high strength, toughness and excellent dielectric properties, alumina has been regarded as a material of outstanding performance, especially under tension or bending conditions [5]. In contrast to metals and polymers, however, the thermal stability of ceramics above 700^{o}C makes them suitable materials for high temperature applications. The technological importance of alumina has stimulated intensive investigations on the synthesis of alumina and the characterization of its physical and chemical properties. Over the past decade, much effort has been focused on creating nanoscale alumina structures, including nanoparticles and nanocomposites. One dimensional Al_2O_3 nanostructures with various morphologies including nanowires, nanoribbions, nanorods, nanofibres, nanobelts and nanotubes have been prepared by various methods [6-9]. However, the synthesis temperature in the above techniques is usually near or even higher then 1200^{o}C and have made use of frequent catalysts that can produce unintentional defects. Therefore, it provides motivation and is desirable for device application point of view to synthesize Al_2O_3 nanostructures at low temperatures using simple techniques. In this communication, we describe a novel and facile method for preparation of α-Al_2O_3 nanorods by a simple reaction of aluminum metal powder with water in the temperature range of 50-200^{o}C. The diameters of nanorods are relatively uniform ranging from 40 to 60 nm. The aim of the study is to provide the feasibility of the simple route for the preparation of aluminum oxide nanostructures. This method is convenient, economical and fast.

2. Experimental producer

The procedure employed by us for the synthesis of aluminum oxide nanostructures is as follows: In a

Corresponding author: shah11nit@gmail.com

typical synthesis, 5 mg of aluminum metal powder was added to 10 ml of distilled water. The reaction mixture was well sonicated for 20 minutes, transferred into a stainless steel autoclave and then sealed. Samples prepared have been kept at different temperatures ranging from 50C^o to 200C^o. At 50 C^o, 100 C^o and 150 C^o, the reaction mixture was kept for 12h, whereas at 200 C^o the reaction time was 24h. The resulting suspension was centrifuged to retrieve the product and was vacuum dried. High resolution FESEM (FEI NOVA NANOSEM) coupled with EDX were employed for characterization. The molecular ratio of Al/O of the nanorods, calculated from EDX and quantitative analysis data is close to that of bulk Al_2O_3 crystal. X-ray powder diffraction was carried out on a Siemens X ray Diffractometer with Cu-Kα ($\lambda = 0.1514$nm) radiation.

3. Results and discussions

Figure 1 shows Field Emission Scanning Electron Microscopy (FESEM) images of the as prepared samples obtained by reacting micrometer sized large aluminum particles with water under different conditions. Nanorods/nanobelts were not observed for a sample reacted for 12h at room temperature, whereas needle like products radiating from and normal to the original large aluminum particle surfaces were produced for sample heated at 50^{o}C for 12 h (Figure 1a). At higher temperature of 100^{o}C using the same reaction time of 12h, needles become prominent (Figure 1b). The image reveals short nanorods with an average diameter of 40 nm. Further, at 150^{o}C for 12h, the product consists of a large quantity of rods like nanostructures. The nanorods having diameters in the range of 40 to 60 nm with an average diameter of 50 nm have been observed (Figure 1c). The average length of nanorods is in micrometers. At 200^{o}C for 24 h, ribbon like structures resulted (Figure 1d). Most of the nanoribbons have uniform widths along entire lengths. The thickness of about 50 nm is found to be varying along the length of the ribbon. All results suggest that the time and temperature in the reaction process plays a dominant role on the formation of Al_2O_3 nanostructures. Nanoribbons like nanostructures are believed to result from a combination of increased reaction time and temperature. The EDX measurement on the nanorods indicates that rods are composed of Al and O as shown in Fig. 2. The elements corresponding to various peaks are indicated.

The formation of nanorods by the reaction of aluminum metal with water can be explained as follows.

$$2Al\ (s) + 3H_2O\ (l) \rightarrow\ Al_2O_3\ (s) + 3H_2\ (g)$$

Here (s), (l) and (g) represent solid, liquid and gas respectively. Aluminum metal gives out hydrogen when react with water. The growth of nanorods probably occurs by making use of oxide nuclei that may be present on the metal surface. The similar study has been reported earlier in case of iron oxide nanorods, where evolution of hydrogen has been documented [10]. The mechanism for 1D Fe_2O_3 nanorod growth has been discussed when nanorods were formed by oxidizing iron in an air system. Moreover, α-Al_2O_3 nanobelts and nanosheets with different morphologies and sizes have been successfully synthesized by a simple chemical route from water and Al powder in an argon atmosphere, where the temperature distribution inside alumina tube is the critical factor for determining the sizes of nanostructures [11]. In our work, we attribute the nanorod formation to hydrothermal mechanism, although the precise mechanism is unclear at present. Water at elevated temperatures plays an essential role in the precursor material transformation because the vapour pressure is much higher and the state of water at elevated temperatures is different from that at room temperature. The solubility and the reactivity of the reactants also change at high pressures and high temperatures, and high pressure is favorable for crystallizations. Further, Al_2O_3 layer formation on the rod surface, acting as a passivation layer, will inevitably reduce the dissolution rate of Al substantially and thus prevent further growth of nanorods. This explains why the length and width of nanorods do not grow in an unlimited fashion, when an increased reaction time and temperature are used.

4. Conclusion

In this communication, we describe a novel method for the syntheses of α-Al_2O_3 nanorods/nanoribbions from a simple Al-H_2O reaction in the temperature range of 50-200^{o}C. This method is a contribution to the field of synthesis with its advantages as:

1. It is a simple route involving only water and does not require any additives as reported in literature.

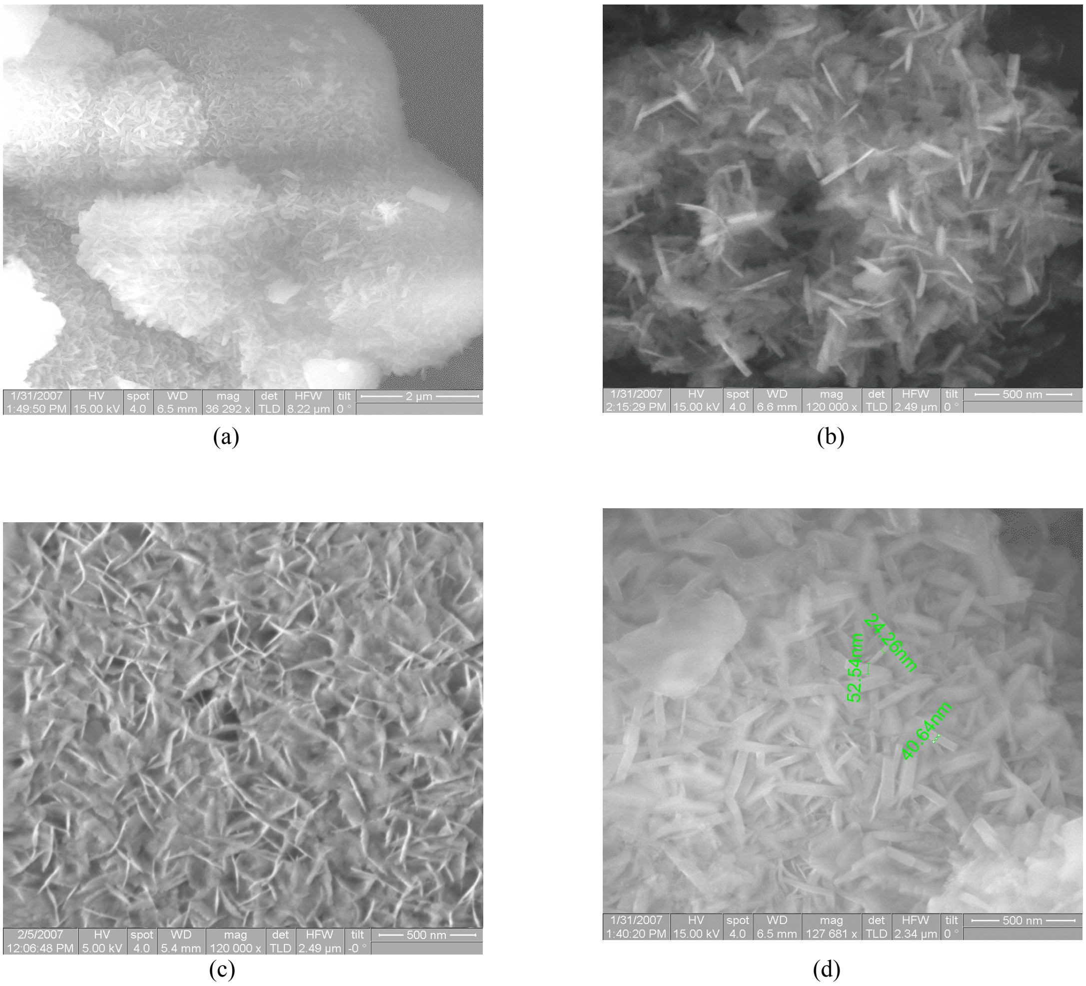

FIG. 1: FESEM images of prepared samples at: (a) 50 °C for 12 h, (b) 100°C for 12 h, (c) 150°C for 12 h and (d) 200°C for 24 h.

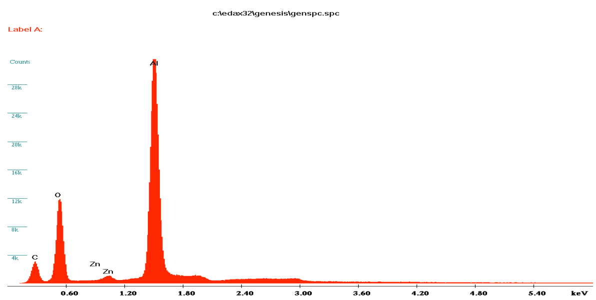

FIG 2: EDX of pattern of prepared samples at 200°C.

2. It is an economical, reproducible and low temperature process.
3. It is template free method and does not require any substrate.
4. This direct and efficient route, extendable to other metal or alloy oxide nanorods has the potential to be further scaled up towards production of large quantities.

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