J. Am. Ceram. Soc., 90 [10] 3311–3313 (2007)
DOI: 10.1111/j.1551-2916.2007.01866.x

Journal

Electron Beam-Induced "Nanocalcination" of Boehmite Nanostrips to Mesoporous α-Alumina Phase

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Phase transformation of boehmite (AlOOH) nanostrips to α-Al_2O_3 nanolaces was investigated under electron beam irradiation of a 200 keV transmission electron microscope (TEM). X-ray diffraction pattern showed that the powder consisted of boehmite phase before the electron exposure, while selected area electron diffraction patterns demonstrated the formation of stable α-alumina phase after bombardment with high-energy electrons. The *in situ* TEM observations revealed that the initial morphology remained unchanged while a lace-like mesostructure of α-alumina was developed.

I. Introduction

Alumina nanostructures have extensive technological and industrial significances,[1,2] due to their wide range of novel characteristics in catalytic[3] and electronic[4] applications. Al_2O_3 exists in different crystallographic polymorphs such as metastable γ, δ, θ, κ, ρ, η, and χ aluminas as well as the thermally stable α-phase.[5,6]

The phase transformations that occur during the calcination of the hydrated alumina phase (γ- AlOOH) to α-alumina (α-Al_2O_3) are of fundamental importance in designing ceramic processing procedures.[7] Upon heating, γ-AlOOH decomposes to γ-Al_2O_3 with a disordered cubic close-packed lattice. Then through a series of polymorphic phase transformations, γ-Al_2O_3 changes to the more ordered cubic close-packed θ-Al_2O_3, which undergoes a reconstructive transformation at elevated temperatures, e.g., 1050°–1200°C, to form the thermodynamically stable hexagonal close-packed α-phase. Generally, the mentioned transformation sequence was reported as follows[8,9]:

$$\gamma\text{-AlOOH} \rightarrow \gamma\text{-Al}_2\text{O}_3 \rightarrow \delta \rightarrow \theta \rightarrow \alpha\text{-Al}_2\text{O}_3$$

The transformation of θ to α-Al_2O_3 is believed to be achieved by a nucleation and growth mechanism and belongs to the first-order phase transformation classification.[10,11]

Electron irradiation has been applied to induce phase transformations in various metallic systems such as Ni–Mo alloy,[12] $Zr_{66.7}Ni_{33.3}$,[13] $Zr_{66.7}Cu_{33.3}$, and $Zr_{66.7}Pd_{33.3}$ metallic glasses,[14] oxides and nitrides, e.g., PbO_2,[15] $SrFeO_{3-\delta}$,[16] and Fe_4N,[17] or in the reduction of oxides to the elements.[18] But to the best of our knowledge, there is no report on phase transformations induced by electron exposure in alumina systems. In this paper, we have prepared nanostrips of boehmite via an innovative method and performed "Nanocalcination" under electron beam exposure to obtain a mesoporous α-Al_2O_3 structure locally.

J. Blendell—contributing editor

Manuscript No. 22736. Received January 27, 2007; approved May 22, 2007.
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II. Experimental Procedure

The boehmite nanostrips were prepared according to the following experimental route: 0.36 g $AlCl_3 \cdot 6H_2O$ was dissolved in 10 mL ethanol under vigorous stirring at room temperature. Then 40 mL water was added slowly to the solution and mixed for 2 h. The resultant solution was put into a teflon-lined stainless-steel autoclave at 200°C for 24 h. At this temperature, the autogenous pressure inside the autoclave was estimated to be about 1.5 MPa. A gelatinous precipitate was obtained, which was filtered, washed with deionized water, and dried at 70°C for 24 h. The crystal structure of the obtained powders was characterized with X-ray diffraction analysis (XRD, Philips X'Pert diffractometer, Eindhoven, the Netherlands) with Bragg's

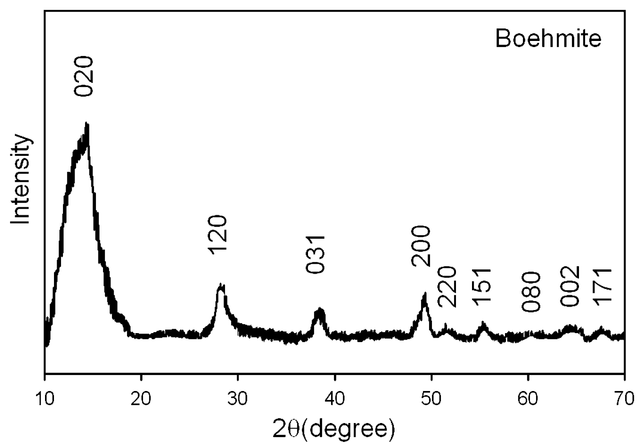

Fig. 1. X-ray diffraction pattern of the obtained powder prior to the electron beam irradiation.

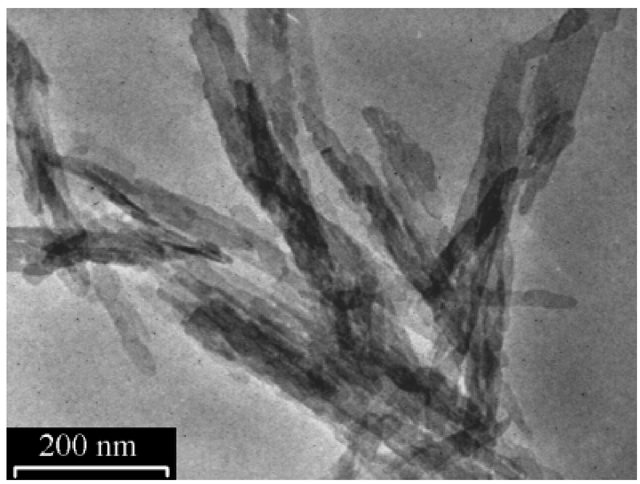

Fig. 2. Transmission electron spectroscopy image of the obtained boehmite nanostrips.