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Crystalline Structure and ELNES of Branched Al_2O_3 Nanowires

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Nanoscale α-Al_2O_3 has attracted attention as a candidate for vaccine delivery enhancement due to its ease of functionalization toward organic molecules and its low cytotoxicity [1]. Herein a study of the structural properties of branched α-Al_2O_3 nanowires is presented to further understand their growth mechanisms so that they may be optimized for the previously mentioned applications.

Synthesis of α-Al_2O_3 nanowires was carried out in a chemical vapor deposition (CVD) reactor by mixing anatase TiO_2 powder (as an oxygen feedstock) with Al foil in an Ar environment. This process was carried out at 1150°C for 60 min and 1050°C for 30 min.

High temperature growth processes yielded branched Al_2O_3 nanowires with six-fold symmetry about a main trunk (Fig. 1a). For ease of imaging, primary (Fig. 1f) and secondary (Fig. 1b) branches were separated from their trunks by sonication in ethanol. From selected area electron diffraction (SAED) and high resolution TEM imaging (HRTEM) of the primary branches (Figs. 1e, 1g) we see that the alumina is α-phase and they grow in the [110] direction, radially outward from the main trunk which runs in the [001] direction. Secondary branches (Fig. 1b) grow from the primary branches along the [200] direction (though these directions are equivalent due to the hexagonal close-packed structure of α-Al_2O_3). A model of α-Al_2O_3 is shown (Fig. 1d) to illustrate concordance with HRTEM image (Fig. 1g). SAED patterns reveal that the primary and secondary branches have planar spacings along their growth directions of 2.379 Å and 2.375 Å, respectively. The concurrence of the two within 0.168% suggests that the difference in morphology is not due to a strained lattice or bulk defects.

Low temperature growth processes yielded similar nanowires with diminished crystalline quality and superlattice defects as visible in HRTEM imaging (Fig. 2b) and SAED (Fig. 2c). Quantitative energy dispersive x-ray spectroscopy (EDS) yielded neither characteristic Ti x-rays nor a significant change in Al:O atomic ratios, indicating that these defects are not due to incomplete liberation of oxygen from the feedstock particles. Electron energy loss near edge spectra (ELNES) of the Al $L_{2,3}$ edge shows that the low temperature process yields an increase in the edge at 85eV. This can be attributed to an increase in the amount of amorphous α-Al_2O_3 and a decrease in average Al-O and Al-Al bond lengths [2].

References

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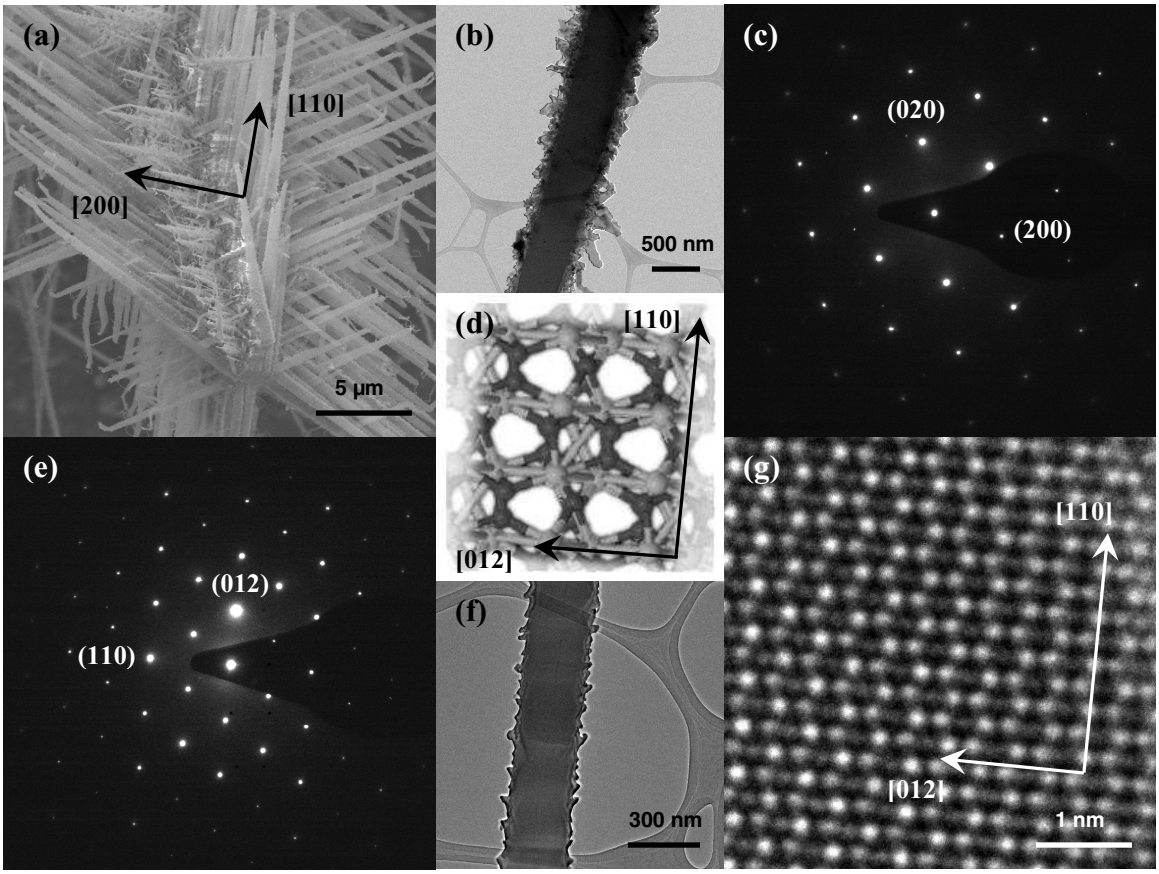

Figure 1. (a) SEM image of [001] trunk with [110] and [200] branches grown at 1150°C for 60 min, (b) BF TEM image of [200] branch and (c) corresponding SAED pattern. (d) Model of α-Al_2O_3 oriented to match [110] branch depicted in Fig 1f. (e) SAED pattern of (f) [110] branch and (g) corresponding HRTEM image.

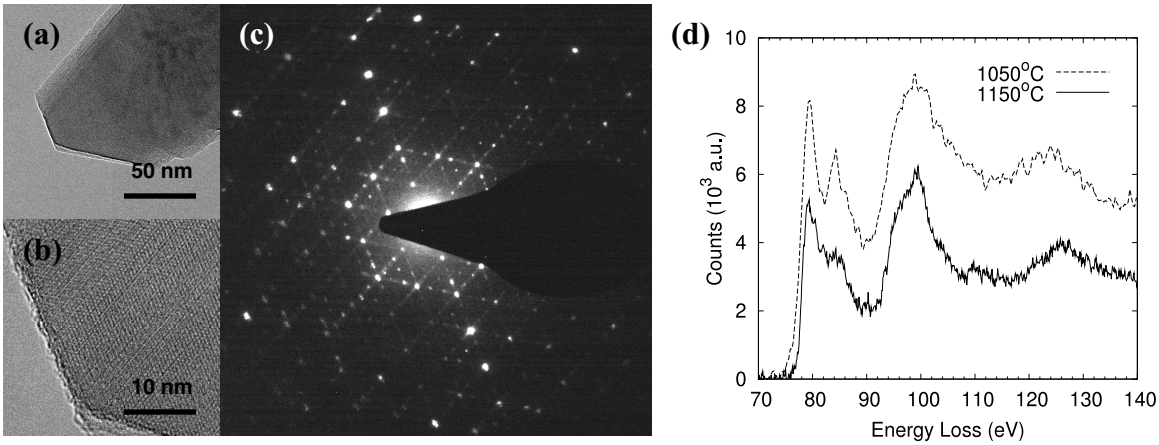

Figure 2. (a) BF TEM and (b) HRTEM image of nanowires grown at 1050°C for 30 min and (c) corresponding SAED pattern showing superlattice structure. (d) EEL spectra featuring Al $L_{2,3}$ edges of nanowires grown at 1050°C and 1150°C.

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