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Fabrication of hollow coaxial $ZnAl_2O_4$ high aspect ratio freestanding nanopillars based on the Kirkendall effect

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Recent progress in optical metamaterial research has highlighted the importance of developing the technology that would allow to pattern high quality optical materials on nanoscale. Optical cloaking, far field imaging with super resolution, and materials with hyperbolic dispersion are only a few examples among several topics that require realization of highly periodic metal-dielectric three dimensional structures with subwavelength feature sizes [1]. The topology of metamaterials typically consists of an array of nanopillars, trenches and flat multilayers. Realization of multi-coaxial cylindrical structures enables development of new promising materials with unusual and exiting responses to light. Latest reports brings a hope in achieving discrete multilayered structures by implementing Kirkendall effect in Al_2O_3/ZnO nanolaminates [2],[3]. During the annealing the diffusion of ZnO into Al_2O_3 leads to creation of crystalline $ZnAl_2O_4$ compound. This solid state reaction is highly sensitive to temperature, annealing time, layer thickness and other parameters. This work presents a fabrication approach of free standing high aspect ratio $ZnAl_2O_4$ hollow coaxial nanopillars.

The nanopillars were fabricated by combining atomic layer deposition (ALD) [4] with advanced deep reactive ion etching (DRIE) techniques. A similar approach was recently developed for fabrication of high aspect ratio Al_2O_3 and TiO_2 trenches [5]. The method is based on ALD deposition on high aspect ratio silicon structures (mold) with subsequent selective removal of the silicon. The use of ALD in combination with a sacrificial silicon mold is a novel way to create high aspect ratio metal oxide structures. Figure 1 shows the fabrication steps in pillars patterning. Homemade silicon-on-insulator (SOI) wafers (Fig. 1a) were selected as substrates. Then, using deep-UV lithography and DRIE, a square lattice of holes (Fig. 1b) with a diameter of 300 nm and a lattice constant of 400 nm was patterned. Next, the holes were filled with Al_2O_3/ZnO/Al_2O_3 layers (thicknesses of 50 nm/25 nm/50 nm, respectively) by ALD (Fig. 1c) using diethyl zinc, trimethylaluminum, and water as precursors at 200°C. The top part of the ALD layers was removed using Ar^+ ion milling, thereby exposing the silicon matrix between coated pillars (Fig 1d). Prepared samples were annealed at 800°C for 12h in N_2 environment at atmospheric pressure in order to induce Kirkendall effect (Fig. 1e). Scanning electron-microscopy (SEM) images (Fig. 2a and 2b) reveals surface morphology before and after the annealing. ZnO migrated into Al_2O_3 areas resulting the formation of large voids. Last step in fabrication was silicon removal between pillars. It was done by reactive ion etching with SF_6 process gas (Fig 1f). The success of this procedure is heavily dependent on the ability to etch silicon selectively, without affecting the ALD coatings. Conventionally, in semiconductor industry silicon dry etch is based on SF_6 process gas. This work demonstrates that silicon can be etched very selectively to ZnO based materials with no observable influence on the pillars.

Each fabrication step was carefully evaluated using SEM analysis. Figure 2c shows SEM image of fabricated free-standing high aspect ratio $ZnAl_2O_4$ cylindrical nanopillars. Additional work requires for improvement the separation and minimizing porosity between coatings. This should be handled in combination with adjusting multilayers thicknesses during ALD deposition and improved annealing conditions.

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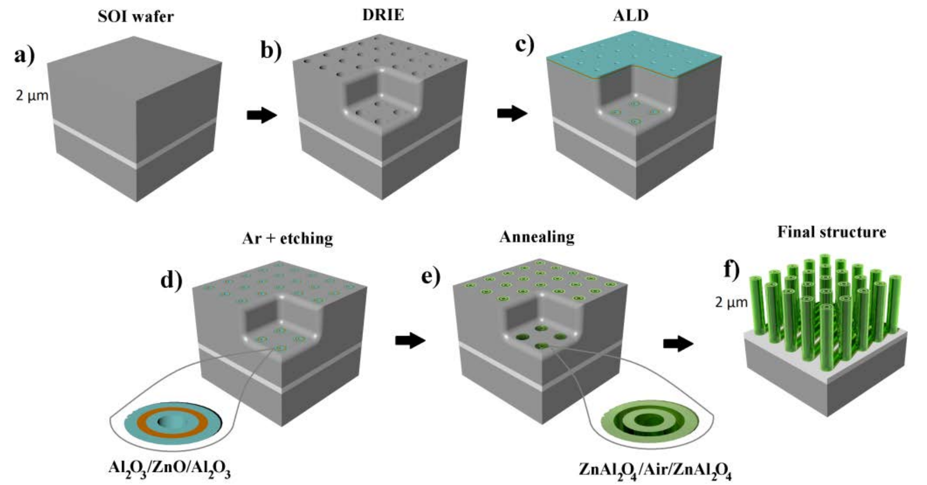

Figure 1. Schematic drawing of the multistep fabrication

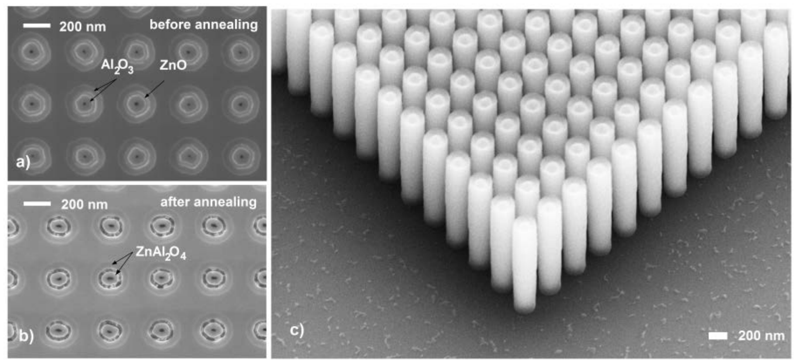

Figure 2. Electron micrograph images. (a) ALD deposition of initial coatings. (b)Temperature induced Kirkendall effect and the formation of $ZnAl_2O_4$ hollow coaxial pillars. (c) Final structure after selective silicon removal.