

Creation of zirconium silicotungstic oxide nanorod crystals using electron beams

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ABSTRACT

The heteropoly acid $H_4[SiW_{12}O_{40}] \cdot nH_2O$ (silicotungstic acid: STA) is a strong Brønsted acid with a Keggin structure and is used as a catalyst and proton conductor. On the other hand, zirconia is a Lewis acid, which is an electron pair acceptor. Therefore, in a hybrid structure, the silicotungstic acid donates (releases) protons, and zirconia becomes a proton receiving site, allowing for the formation of a proton hopping pathway. Furthermore, in zirconium silicotungstic oxide nanorod crystals, nanoconducting paths are formed, and high-speed proton transfer can be expected.

This study reports for the first time on zirconium silicotungstic oxide nanorod crystals obtained by electron beam irradiation. 5 g of STA and 50 ml of H_2O aqueous solution were mixed with 0.9 g, 0.5 g, and 0.1 g of $ZrOCl_2$ (30%) in 50 ml of H_2O aqueous solution. Hydrothermal synthesis ($80^\circ C$, 24 h) was performed, and the mixture was filtered to obtain silicotungstic zirconia (STA- ZrO_2) hybrid particles (Fig. 1a). The chemical composition determined by ICP and water analysis was $SiO_2 \cdot 8ZrO_2 \cdot 12WO_3 \cdot 32-34H_2O$. XRD analysis showed a d spacing of 1.1 nm, and according to the Sherrer equation, the particle size was 3.1-3.5 nm (Fig. 1b). FTIR showed peaks at Zr-OH (638 cm^{-1}) and Zr-O-Zr (458 cm^{-1}) (Fig. 1c). Using STA- ZrO_2 particles, zirconium silicotungstic oxide nanorod crystals were created using a TEM (JEM2100F) instrument (Fig. 2). When an acceleration voltage of 200 kV was applied to the particles (Fig. 2a), nanorod crystals were created (Fig. 2b). The crystal planes grew parallel (Fig. 2c) or perpendicular (Fig. 2d) to the rod. The growth mechanism is unknown, but it is speculated to be related to the crystal axis of the grid (Cu). Furthermore, compositional analysis using an Energy Dispersive X-ray Spectrometer (EDS) detected peaks of Zr, Si, W, and O, indicating that it is zirconium silicotungstic oxide nanorod crystals.

REFERENCES

[1] J. Kim; Japan patent no. 7330460.

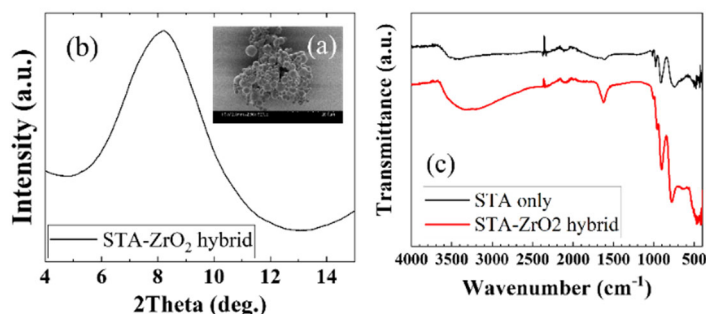


Fig. 1 (a) SEM image, (b) XRD, and (c) FTIR properties of STA- ZrO_2 hybrid particles.

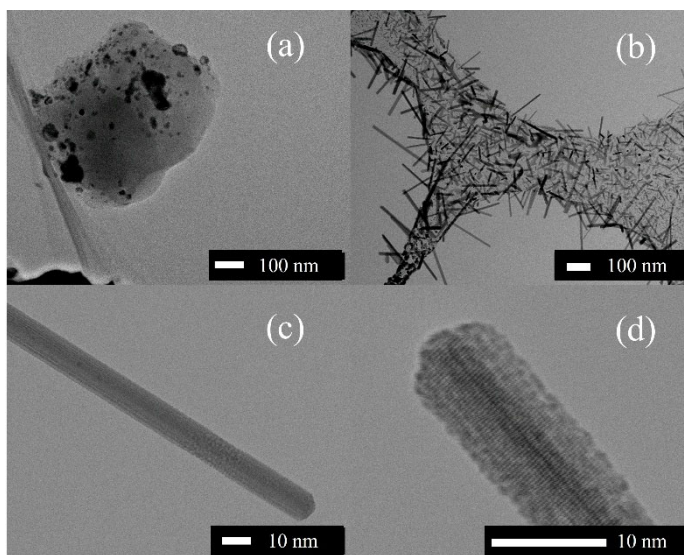


Fig. 2 TEM images; (a) STA- ZrO_2 particle, (b) zirconium silicotungstic nanorod, (c, d) crystal nanorods.