

Effect of Atmosphere on Heat-Treated Electro-Spun TiO₂ Fibers

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The effect of size and morphology on the properties of materials is fundamental when seeking new applications or developing novel technologies [1]. Several methods have been implemented to prepare ceramic materials with specific structures in order to enhance such properties as their catalytic activity or selectivity. The fabrication of fibers with nanometric dimensions has been identified as a promising approach in this field. Electrospinning has been used in recent years for synthesizing nanofibers of both organic and composite materials; such fibers have applications in catalysis, as membranes, and for sensor applications due to their high surface area and small pore size. The possibility of producing nanotubes or nanowires composed entirely of oxides has also been studied. Among other ceramics, TiO₂ appears as a good candidate because of its many applications in such diverse fields as cosmetics, dye-sensitized fuel cells and catalysts.

The process involves the preparation of a solution (polymer and inorganic precursor), the spinning of the solution to obtain composite fibers, and heat treatment of the resulting mat to obtain the final oxide fibers. In this work, air and N₂ were used to study the effect of the atmosphere on the morphology and composition of TiO₂ nanofibers during heat treatment. TiO₂/PVP composite and TiO₂ nanofibers have been prepared using the following approach. Anhydrous ethanol (3 mL), glacial acetic acid (3 mL) and titanium isopropoxide (Sigma Aldrich, 1.5 g) were taken in a vial; anhydrous ethanol (7.5 mL) and polyvinylpyrrolidone (PVP, Sigma Aldrich, MW~1,300,000, 0.45 g) were prepared separately. The two vials were placed in a bath maintained at 60°C and stirred. The titanium isopropoxide solution was then added to the PVP solution to produce a clear, yellow precursor solution, which was then stirred for 1.5 h in the bath at 60°C, cooled to room temperature for 30 min and transferred to a plastic syringe for electrospinning [4]. A vertical set-up was used for the electrospinning process in which a syringe pump was used to direct a controlled flow of the solution through a capillary tube to a stainless-steel needle that was connected to a high-voltage supply. For this study, the nanofibers were collected on an aluminum foil wrapped around a rotating drum. The drum was electrically grounded so as to generate an electric field of 25 kV between the tip of the needle and the foil, which were separated by a distance of ~17 cm. Mats of TiO₂/PVP composite nanofibers were collected at two different locations on the aluminum foil for solution flow rates of 0.5 mL/h and 1.2 mL/h, respectively. The mats were allowed to stand overnight under ambient conditions. Part of the mat spun at 1.2 mL/h was removed from the foil and heat-treated in air on a titanium hot plate for 3 h at 500°C in order to selectively remove PVP. A second section of the same mat was heat-treated in N₂ atmosphere on a titanium hot plate for 3 h at 500°C.

The characterization of the as-spun material was previously studied [2]. SEM and TEM were used to characterize the morphology of heat-treated fibers. The mats produced under different atmospheres

show variations in color and strength. The SEM image in Figure 1a shows the characteristic morphology of the fibers after heat treatment under N₂ atmosphere. The fibers within the mat look like cylindrical fibers. Samples were embedded and ultra-microtomed for TEM observation. This procedure allowed the observation of cross-section and several slices of the fiber. Figure 1b shows a low-mag image where several sections of fibers are exposed. The TEM images in Figure 1c and 1d show the geometrical shape of the TiO₂ nanostructures. After heat treatment, fibers show a circular cross section, in the range of 50 to 150 nm. Differences in contrast suggest the fibers are polycrystalline with variations in particle size. [3]

References:

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- [2] A. Suresh, M. J. Arellano-Jiménez, J. McCutcheon and C. B. Carter. 'Polycrystalline TiO₂ Produced by Electrospinning' *Microscopy and Microanalysis* 2012 19 (suppl. 2) 1392-3
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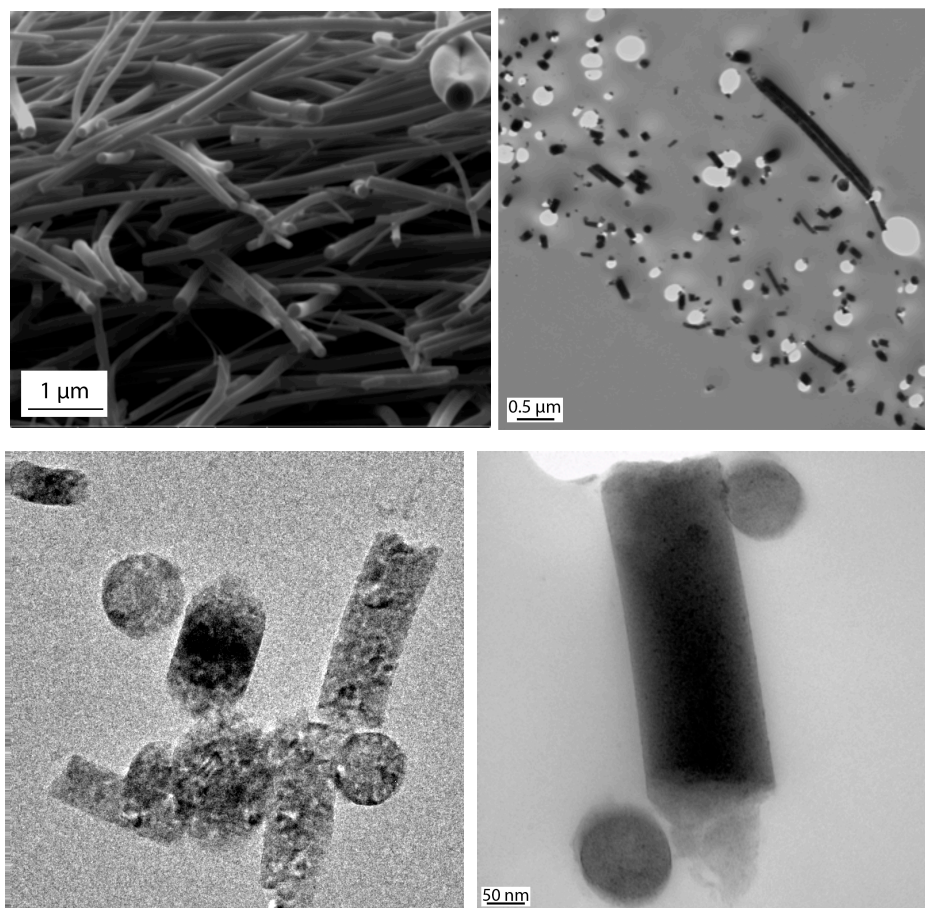


Fig. 1. A and b) TiO₂ fibers after heat treatment under N₂ atmosphere, a) SEM image of the fibers b) TEM image of ultra-microtomed material, c and d) Cross and longitudinal sections of TiO₂ fibers after heat treatment in air (c) and N₂ (d) atmosphere (same scale).