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Microtomy on Heat-Treated Electro-Spun TiO_2 Fibers

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The use of microtomy for sample preparation has been widely applied to the study of soft tissue in the biological area, but has been also successfully applied in materials science. The implementation of microtomy has been a valuable tool to prepare samples for the study of many different materials such as polymers, carbon fibers, ceramics and metallic alloys [1]. It is a widely used method to prepare polymers or composite materials. Samples prepared by this method can be analyzed using several techniques including VLM, SEM and TEM, which allows a more extensive characterization of the material. The fabrication of ceramic with nanometric dimensions has been identified as a promising approach to enhance their catalytic activity or selectivity. Several methods have been implemented to prepare ceramic materials with specific structures because the effect of size and morphology on the properties of materials is fundamental when seeking for new applications [1]. The possibility of producing nanotubes or nanowires composed entirely of TiO_2 has been studied because of its many applications in such diverse fields as dye-sensitized fuel cells and catalysts. Electrospinning has been used in recent years for synthesizing nanofibers of both organic and composite materials; membranes fabricated by this method exhibit high surface area and small pore size. The shape and distribution of the ceramic component within the fiber is a critical factor in the behavior of the final material. It then becomes important to have a suitable method to produce samples where the distribution and orientation of materials can be analyzed using different techniques. The use of microtome sections allows the analysis of the bulk structure in large samples. As shown in the present study, the microtome can provide excellent results in producing samples from electrospun ceramic fibers.

The process involves the spinning of a polymer solution, or polymer-precursor, to obtain fibers. Final oxide fibers result after heat treatment of the resulting mat. In this work, polymer and polymer-TiO_2 precursor were used to study the morphology and composition of TiO_2 nanofibers. TiO_2/polymer composite and TiO_2 nanofibers have been prepared using an approach reported before [2]. 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 between the tip of the needle and the foil, which were separated by a distance of ~17 cm. Mats of TiO_2/polymer composite nanofibers were collected on aluminum foil. The mats were allowed to stand overnight under ambient conditions. Part of the mat was removed from the foil and heat treated on a hot plate.

Samples were embedded and ultramicrotomed 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 1 show the geometrical shape of the TiO_2 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. Selected 80-nm-thick sections were used for the TEM analysis. This combination of microtomy and microscopy techniques allowed the examination of the same area in the micro- and nano-scale.
The STEM image in Figure 1 shows the characteristic morphology of the fibers after heat treatment. The material appears as a mat formed by cylindrical fibers. The TEM image in Figure 1 clearly shows the geometrical shape of the TiO2 nanostructures. The synthesized fibers have a uniform width in the range of 50 to 150 nm. The contrast observed in the images confirms that the material is crystalline; the DP corresponds to polycrystalline anatase [3].

References:

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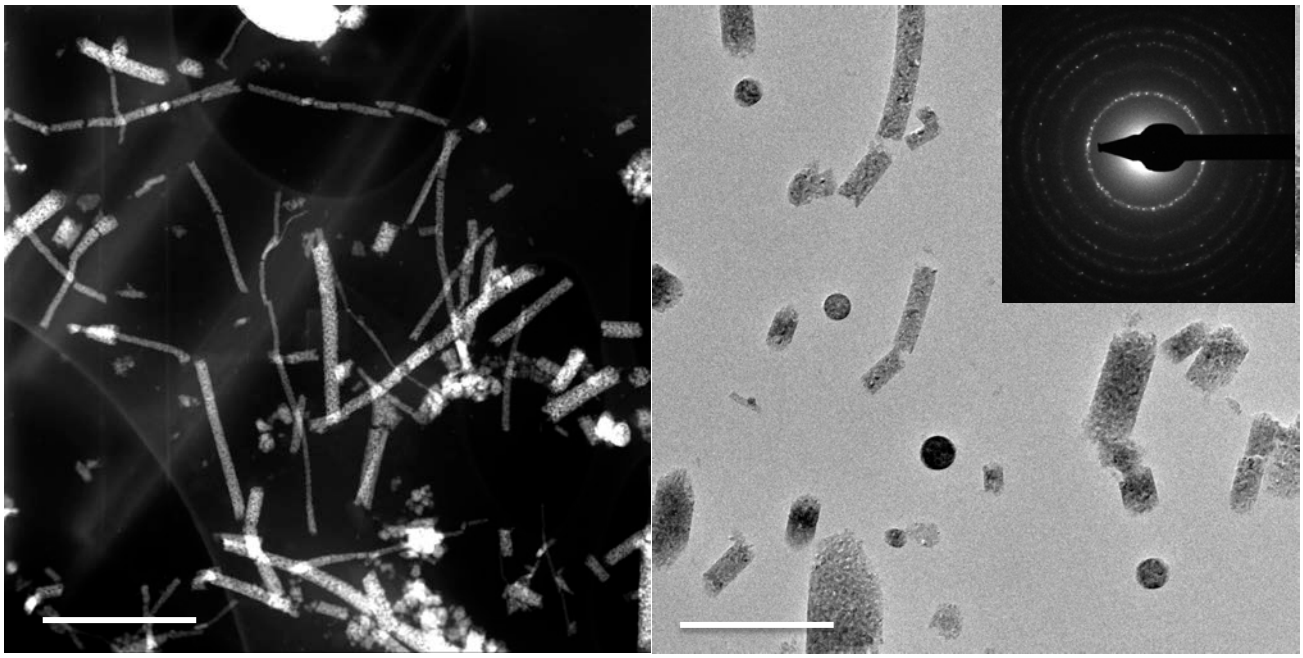

Fig. 1. TEM images of materials produced by electrospinning. Microtomed sections allow the observation of fibers in all different directions such as cross and longitudinal sections. TiO_2 nanofibers are approximately 50-150 nm wide. c) SADP confirms the material is polycrystalline anatase. Scale bar 500 nm and 200 nm.

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