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Material Characterization for Industrial Processes of Thin Titanium Nanotube Films

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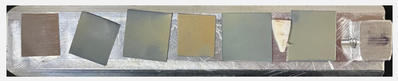
Abstract

Material characterization is an important step in the experimental processes. This step ensures that the sample's processing yields the intended results. This work outlines the process taken to analyze and characterize a set of industrial thin films. Our characterization starts with an elemental baseline from scanning electron microscopy (SEM). The composition is verified by SimNRA (Simulation Nuclear Reaction Analysis) calculations fitting Rutherford Backscattering (RBS) data collected from a silicon surface barrier (particle) detector.

Purpose

Access to pure compounds for material creation can be a barrier to the use of certain material processes. By characterizing thin film samples, shown in Figure 1, the process can be validated. In the case of TNT films, the shape creates an increase in surface area which is advantageous in cases such as energy storage.

Figure 1: TNT samples arranged on a stick for PIXIE analysis.



Methods

To properly identify the samples 2 measurements were required, the material composition and the film thickness. To find the material composition x-ray diffraction, done with the Scanning Electron Microscope, gave a preliminary composition, which aided in the RBS fitting. To measure the thickness and supplement the material composition as measured by the SEM, Rutherford Backscattering data was collected by exposing the samples to a proton beam at energies of 2 MeV and 3.4 MeV. The RBS data is then fit using SimNRA, giving measurements of both thickness and composition.

Scanning Electron Microscopy

The first step to the characterization of the thin film samples was done with a Scanning Electron Microscope (SEM) and x-ray diffraction. The SEM works by using a beam of electrons instead of light, which allows for improved resolution, an image of a sample can be seen in Figure 2. X-ray diffraction works by exposing the sample to x-rays, which put particles into an excited state. When the particles transition back into a relaxed state, they give off different energies of light corresponding to the given element. By fitting the spectra the surface composition can be found as shown in Figure 3. The results can be overlaid as seen in Figure 4.

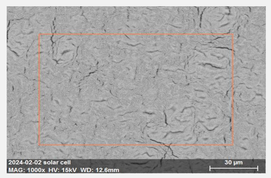


Figure 2: Image of TNT K4 sample at 1000X Magnification

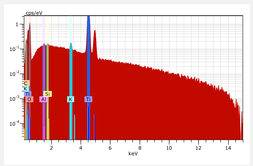


Figure 3: Fit of TNT K4 Sample With SEM

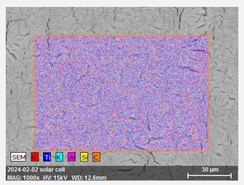


Figure 4: Composition Data Overlay for TNT K4 Sample

Rutherford Backscattering (RBS)

The second portion of the characterization was done with Rutherford Backscattering (RBS) data from a proton beam, created by the Hope College 1.7MV particle accelerator. A key difference between the proton radiation and the x-ray radiation is the depth that the radiation can penetrate, largely due to protons possessing mass. By increasing the depth it allows for the sample thickness to be measured. To record a measurement the samples are placed in front of the beam in the test chamber, shown in Figure 5.

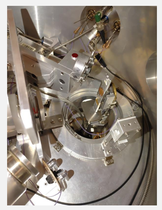


Figure 8: South RBS line test chamber

SimNRA Fitting

SimNRA fits the RBS data collected with the particle accelerator. The initial characterization that is done by the x-ray diffraction gives a set of initial parameters for the fit. By giving the software the detector geometry and beam energy, a spectra for each particle can be created and scaled to match the experimental data. When running the accelerator at higher energies non-Rutherford reactions can occur, requiring differing models to be applied. The initial beam energy of 3.4 MeV created too many reactions which impacted the fit shown in Figure 6. To reduce the number of reactions and increase the density of counts, a second run of data was collected at 2.0 MeV, which gave an improved fit, shown in Figure 7.

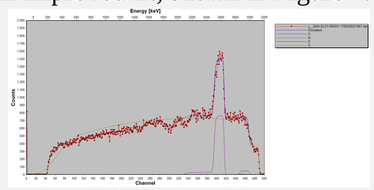


Figure 6: Fit of TNT sample at 3.4 MeV

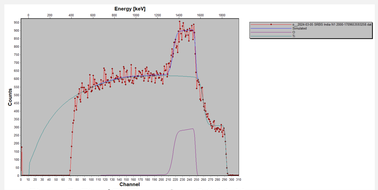


Figure 7: Fit of TNT sample at 2.0 MeV

Results

The thickness and material composition of all 6 TNT samples was measured by fitting in SimNRA, the results are shown in Figure 8. By examining the ratio of each of the samples' composition and thickness, the creation of TiO2 nano-tubes, Figure 9, was verified by comparing to the expected values. In fitting the data there were 3 layers, the expected nanotube sample (Average of 2.43µm), a thin oxidized layer of titanium, and a final thick layer of titanium as the substrate. The sample thickness shown here is for the first layer corresponding to the nano-tubes.

Sample	Thickness	Composition (By At. %)
TNT	2.14 µm	Ti - 33.854% O - 66.146%
TNT K3	2.34 µm	Ti - 25.850% O - 64.192% K - 9.958%
TNT K4	2.49 µm	Ti - 29.723% O - 65.265% K - 5.012%
TNT K5	2.38 µm	Ti - 31.636% O - 64.778% K - 3.586%
TNT K7	2.55 µm	Ti - 30.588% O - 66.080% K - 3.362%
TNT K9	2.69 µm	Ti - 30.140% O - 64.562% K - 5.298%

Figure 8: Table of sample thicknesses and composition

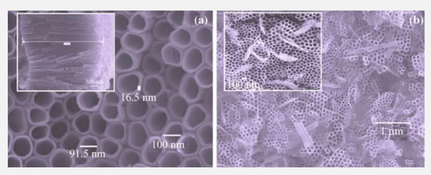


Figure 9: TNT Sample

John K, A., Naduvath, J., Mallick, S. *et al.* Electrochemical Synthesis of Novel Zn-Doped TiO_2 Nanotube/ZnO Nanoflake Heterostructure with Enhanced DSSC Efficiency. *Nano-Micro Lett.* **8**, 381–387 (2016). https://doi.org/10.1007/s40820-016-0099-z

Future Work

After analyzing the fit for the different TNT samples, it was noticed that the particle steradians was significantly lower than expected. The initial thinking is the roughness of the TNT sample reduced the back scattering, which in turn reduced the counts. More time should be spend to better understand the discrepancy in this measurement. Additional thin film samples were also sent from Union Christian that will also be measured to confirm their composition.

Acknowledgements

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