Growth and Re-Crystallization of Titanium Dioxide For Use in Dye-Sensitized Solar Cells

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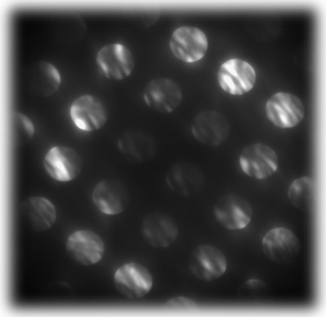

Figure 1- Convergent Beam Electron Diffraction Pattern

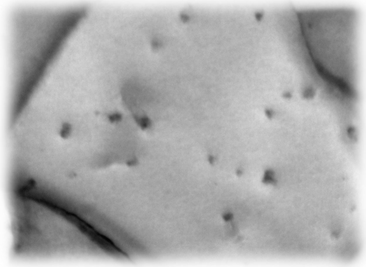

Figure 2- TEM Image of TiO2 Sample at 50,000x

Objectives

- Attempt grid deposition using various methods to produce our own samples
- Obtain data from the in-situ heating of the sample in the TEM
- Verify the phase of the sample using convergent beam electron diffraction
- Analyze the data to determine average grain size

Procedures

- Sample Preparation
  - RF sputtering done at South Dakota State
  - Put onto molybdenum grids with a carbon/silicon support layer
- TEM Operation and Grid Heating
  - Used *in Situ* heating of samples to observe crystallization
  - 20,000X magnification as standard
  - Heated between 200°C and 400°C
- Beam Temperature Measurement
  - Attempted to analyze effect of beam on sample
  - Measured change in temperature before and after beam
- Crystallization Verification
  - Used convergent beam electron diffraction (CBED) to check the phase of the crystals
  - Also used Kikuchi Lines to find different zones of crystal

Abstract

This poster describes the process used in our investigation of titanium dioxide, also known as titania. The Titania sample is first deposited onto molybdenum grids in its amorphous stage using RF sputtering. The samples are then heated to temperatures ranging from 200-400°C and the nucleation and crystallization is observed in the TEM. Using classical phase change kinetics and simple image analysis to find the crystalline volume fraction we were able to produce an analysis of the crystalline density and percent of the sample that was crystallized. From this, the average grain size of each sample is determined. Further investigation is needed to determine what temperature produces the best grains and how this can be applied for use in low cost solar cells.

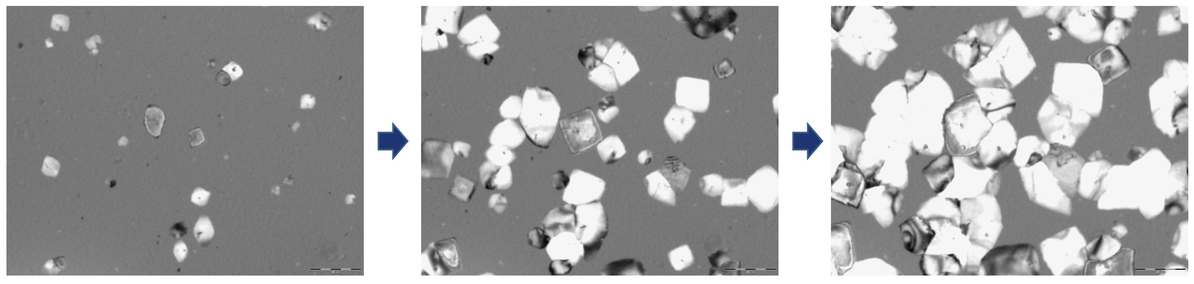

Figure 3- Sample A7 During Various Stages of Crystallization

Results

- Table 1 Below shows the samples data was collected on and the parameters used
- The effect of beam heating was analyzed and an average heating of ~8°C was found
- This heating is most likely lower than the actual heating due to the effect of having to spread the beam out to heat the whole sample instead of the small area usually observed during data collection
- Convergent Beam Electron Diffraction was used to find multiple patterns that can be used in future research to determine the phase and structure of the crystals
- Personal goals of research experience and lab experience were gained, as well as operation of various equipment

Table 1- TiO2 Sample Collection Specifications

Sample Designation	Magnification	Heating Time (seconds)	Temperature (°C)	Notes/Film
A7*	20,000x	~1400	240	Beam area only/C
A8*	20,000x	~600	275	Beam area only/C
B6**	12,000x	~1800	300	Beam area only/C
C7	20,000x	~300	325	Si
C8	20,000x	~300	300	Si
C10	20,000x	~7200	260	Beam area only/Si

* Sample was reheated to 300° C to finish crystallization
** Sample was reheated at 350° C to finish crystallization

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Conclusions

With the onset of dye-sensitized solar cells, the application of the crystallization of titania has greatly increased and warrants further research. The research done on the re-crystallization of titania should continue and the analysis should be completed in order to maximize the grain size and discover the best size for future use. It is worth noting that the effect of the beam should be taken into account and more research conducted in that area to determine the precise effect of the beam heating on the crystallization to further verify that there are no unexpected effects.

Figure 4- Image of TEM

Future Work

- Test more samples to get a more complete data set
- Find a way to produce our own samples
- Determine which temperature produces the best grains
- Implement this method into practical uses such as solar cells

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