

Confocal Raman Characterization of Sputter coated TiO₂ Nanotubes on Functional Substrates

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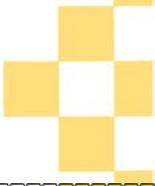
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Outline



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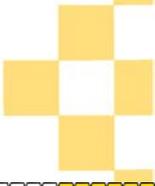
- **Introduction**
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- **Aim & Objectives**
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 - Anodization Technique
 - Characterization
- **Results and Discussion**
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 - X-Ray Diffraction (XRD)
 - Confocal Raman Spectroscopy (CRS)
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- **Acknowledgements**



Introduction



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- Energy demand has intensified research on renewable energy sources.
- Efforts have been devoted to develop high efficient solar cell devices.
- Dye sensitized solar cells (DSSCs) are deemed as the most promising candidates, which commonly use TiO_2 -NPs as photo-anode.

Background



- Random connections in TiO_2 -NPs, bring about charge recombination.
- TNTs-FS may stimulate DSSCs due to vectorial charge transport and charge collection efficiencies.
- Complexity in fabricating highly conductive thin film, limit the application of TNTs in DSSCs.

Background



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- This study focuses on synthesis and CRS characterization of TNTs on functional substrate.
- Hence, it will allow a more qualitative study and understanding of TNTs structural properties.



Aim & Objectives

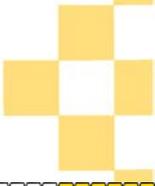


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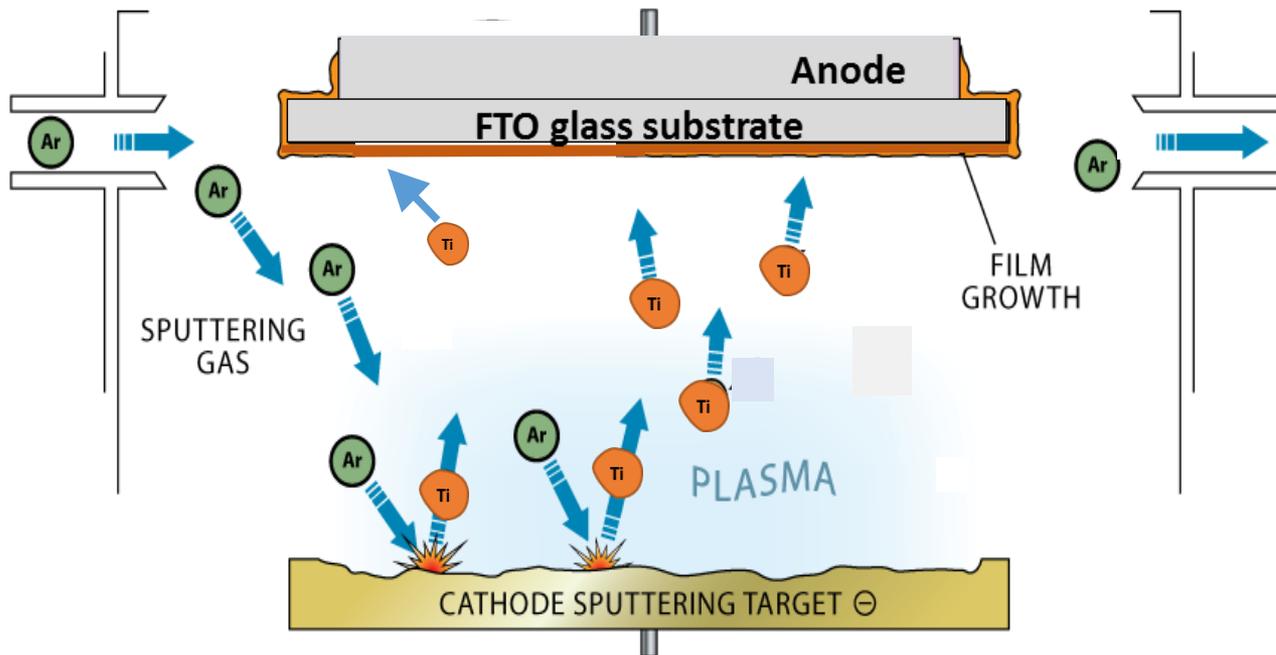


- **Aim**
 - Synthesis of TNTs on FTO glass substrate using electro anodization technique and CRS characterization of TNTs.

- **Objectives**
 - To evaluate the structural and morphological properties of TNT annealed at different temperatures using SEM and XRD
 - To evaluate the structural phase distribution of TNT using Confocal Raman Spectroscopy through Large Area Scans and Depth profiling.



RF SPUTTERING PROCESS

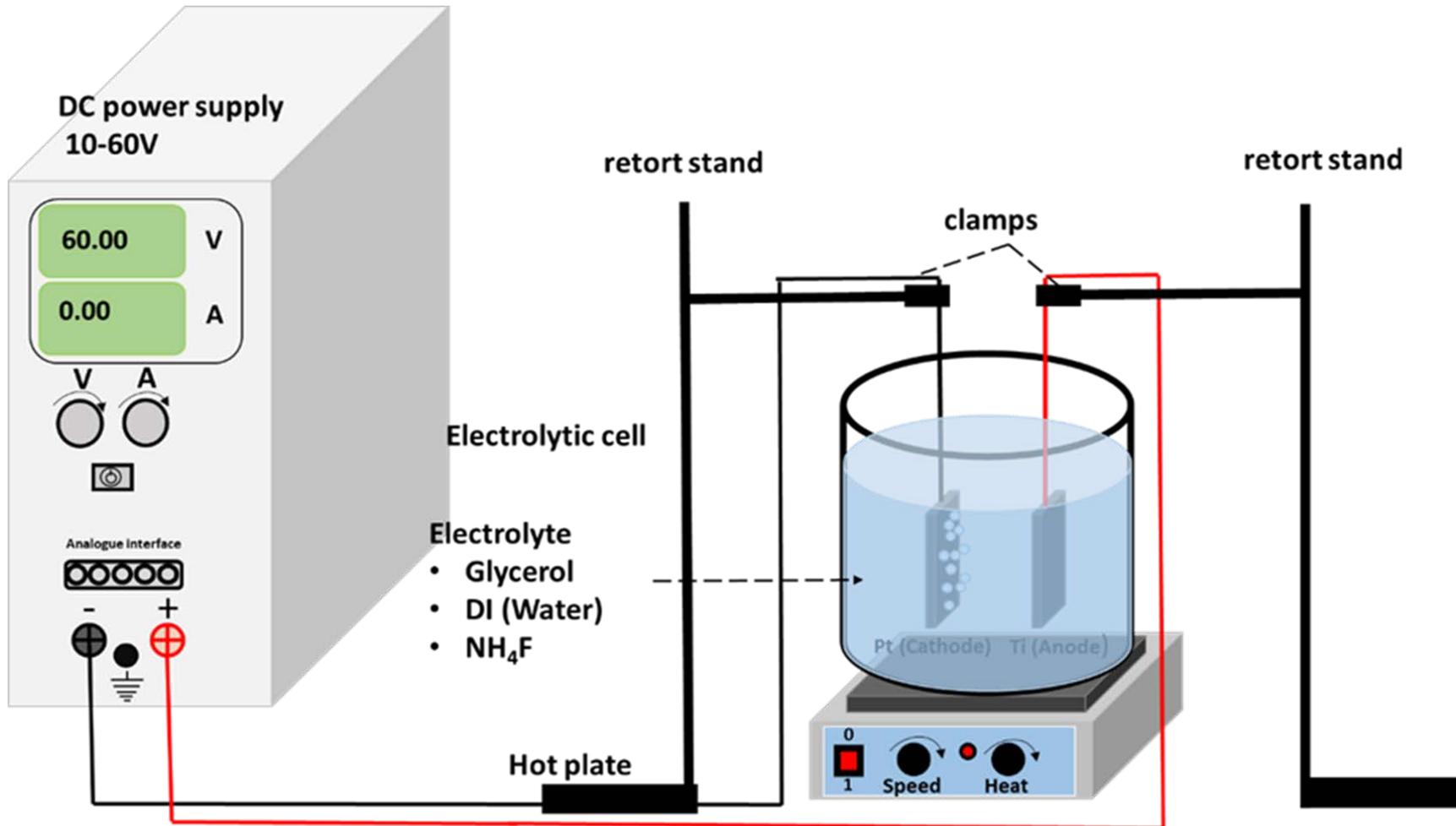


SPUTTERING CONDITIONS

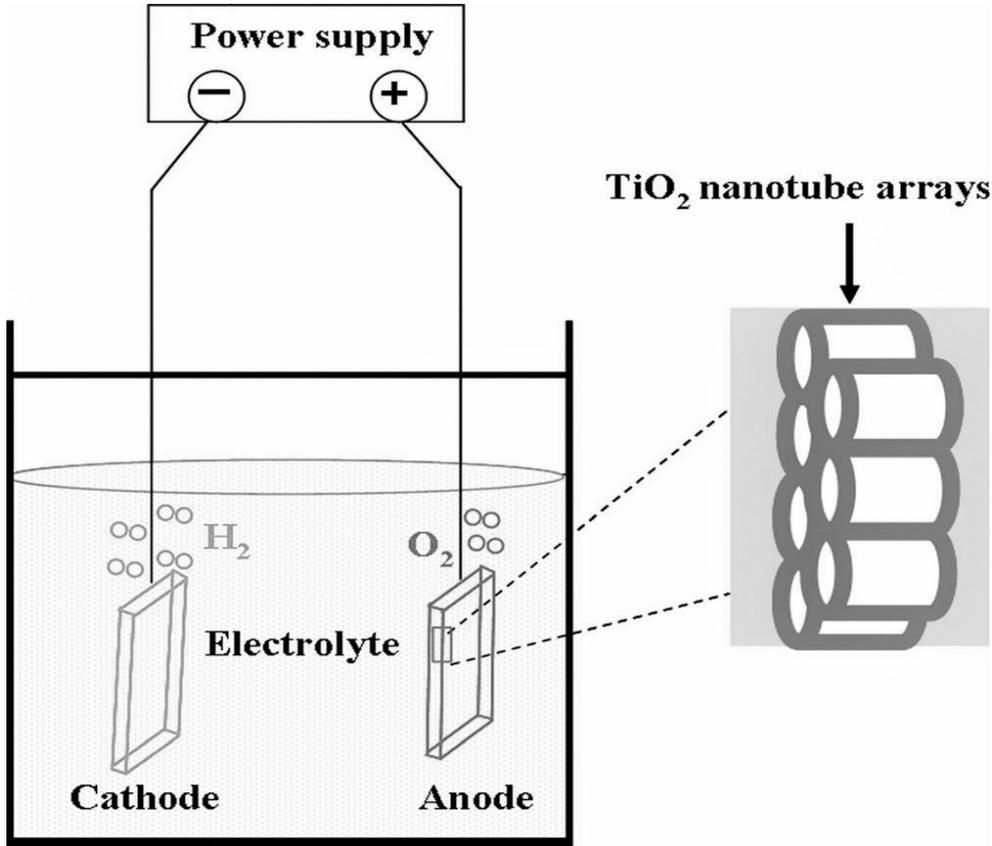
- RF power - 150W
- Pressure - 39 mbar
- Temp - 200 °C
- Period - 4 hours
- Thickness - 10 μm



Experimental setup



Electro-anodization



Reactions at anode:

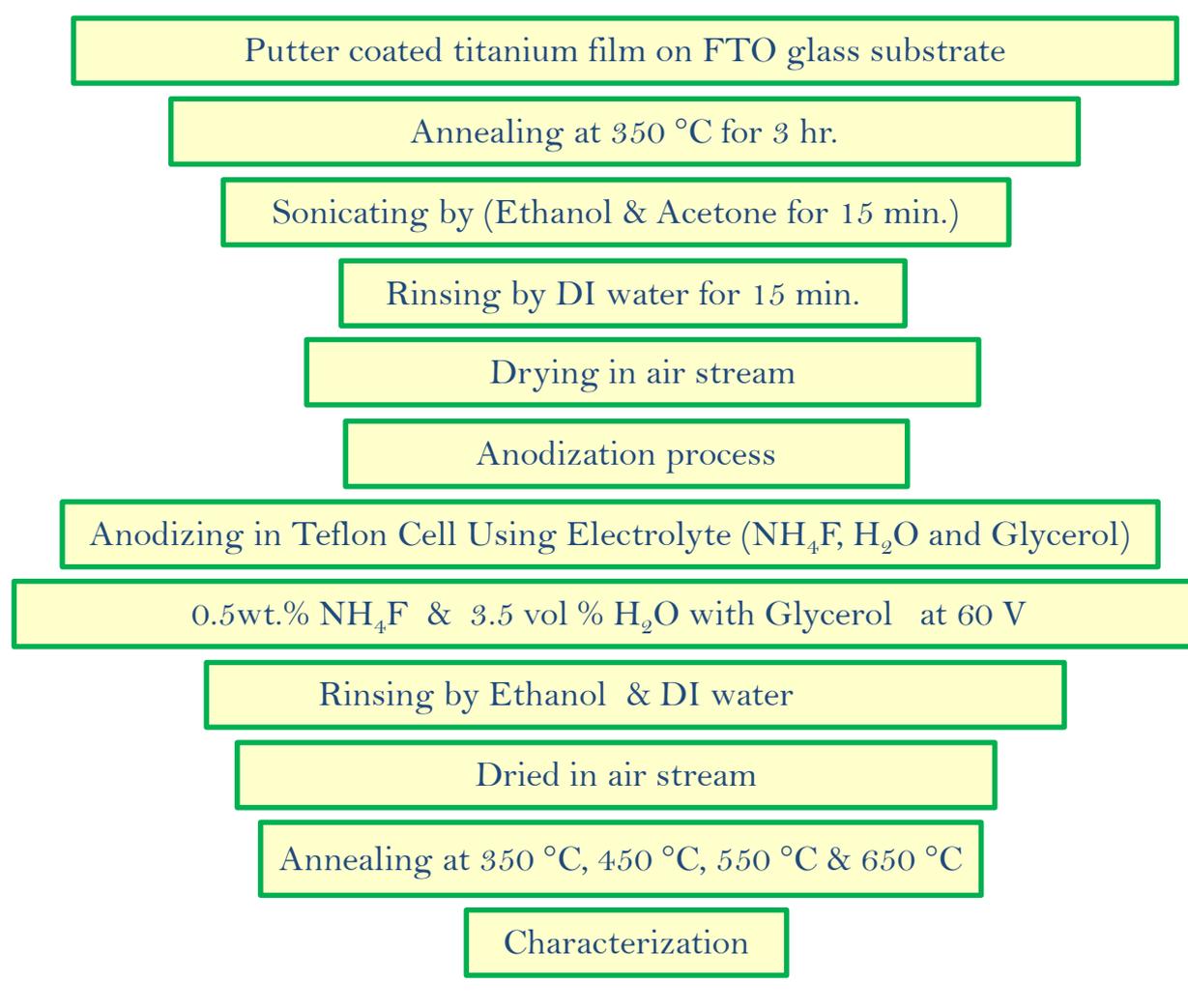
1. $\text{Ti} \rightarrow \text{Ti}^{+4} + 4\text{e}^-$
2. $\text{Ti}^{+4} + 2\text{H}_2\text{O} \rightarrow \text{TiO}_2 + 4\text{H}^+$
3. $\text{TiO}_2 + 4\text{H}^+ + 6\text{F}^- \rightarrow [\text{TiF}_6]^{-2} + 2\text{H}_2\text{O}$

- Anodization of Ti occurs as a result of the competition between oxide formation and chemical dissolution of the oxide by F⁻.

Methodology



TiO₂ nanotubes synthesis flow chart



Characterization techniques



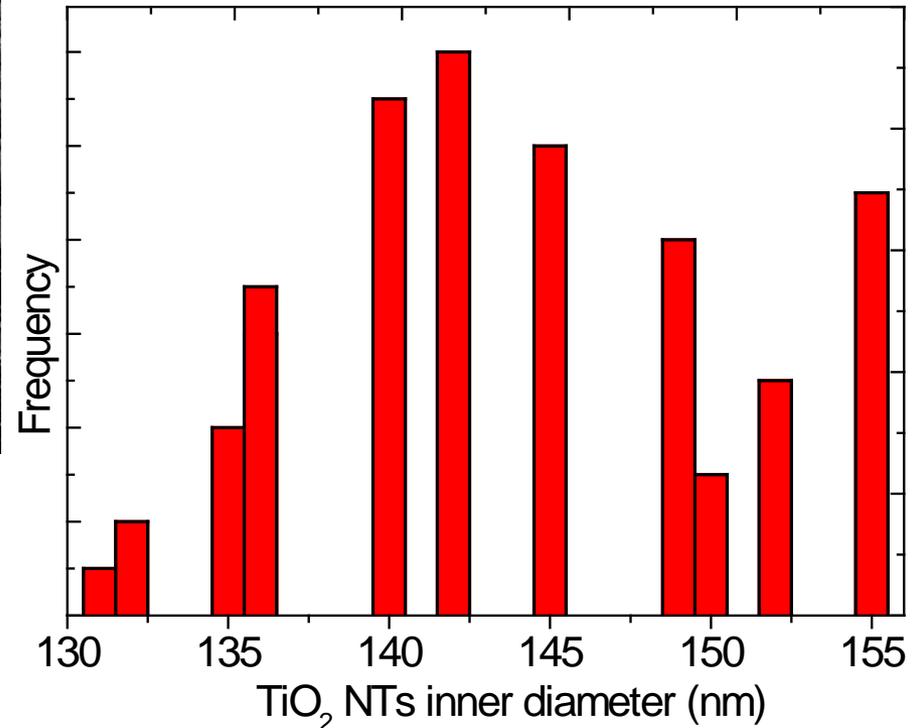
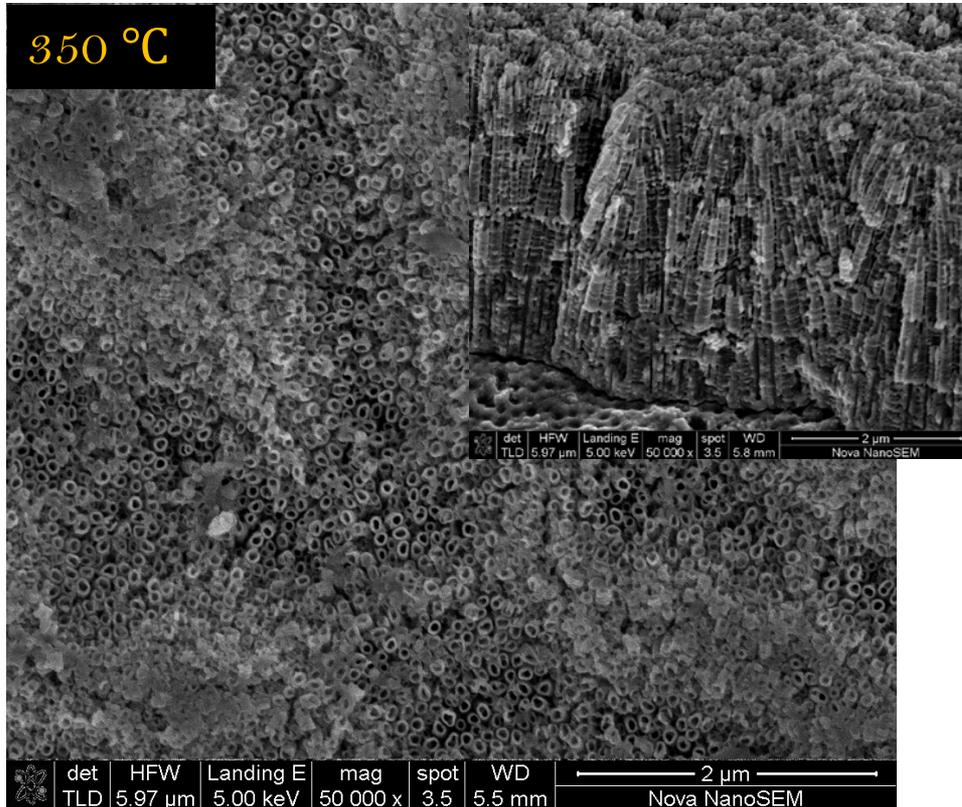
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- The following techniques were used to determine the structural and morphological properties TNTs grown on FTO glass substrate.
 - Scanning Electron Microscopy
 - X-ray Diffraction
 - Confocal Raman Spectroscopy
 - Large area scan
 - Depth profiling

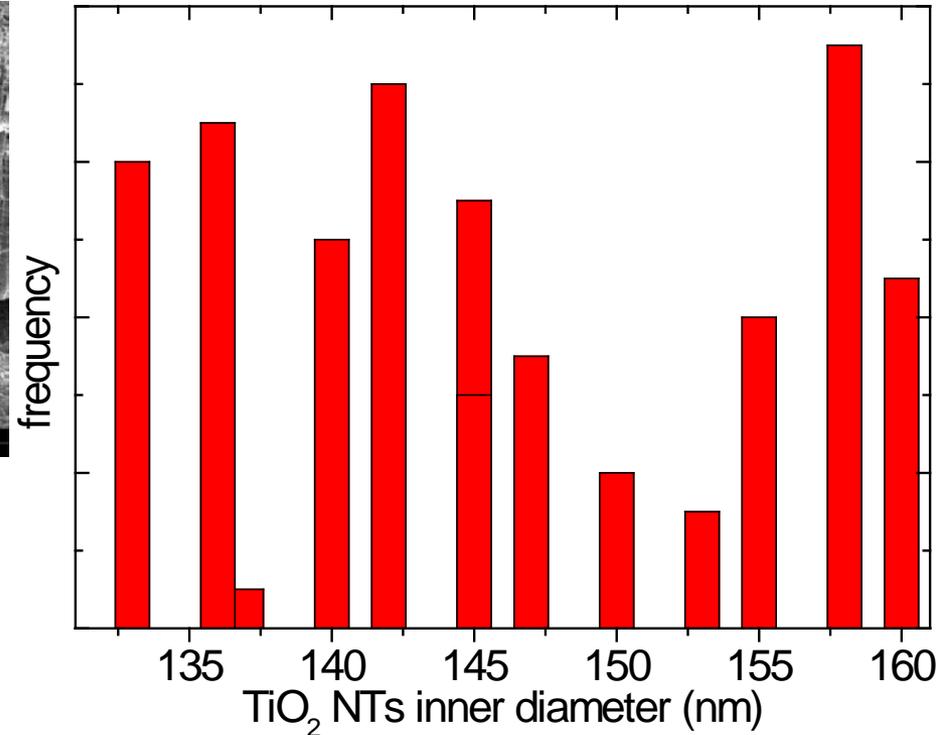
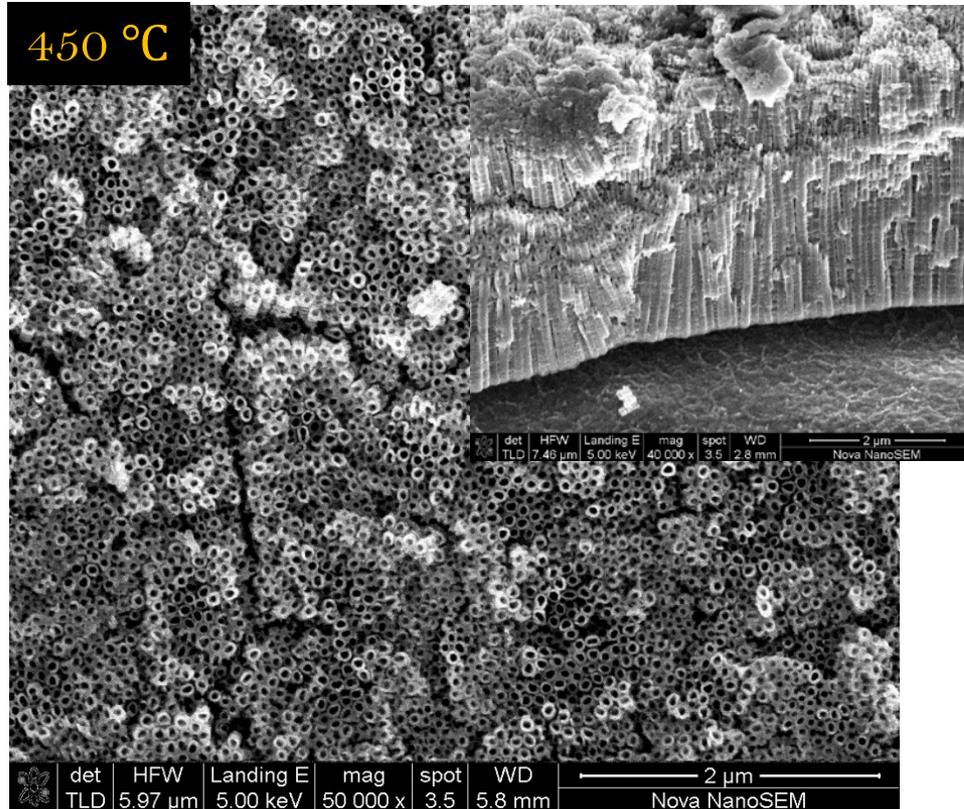
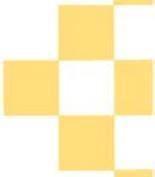


Results : SEM



- SEM micrographs have revealed regular TNTs covered by barrier oxide layer.
- Pore diameter of TNTs are in the range of 130- 155 nm.

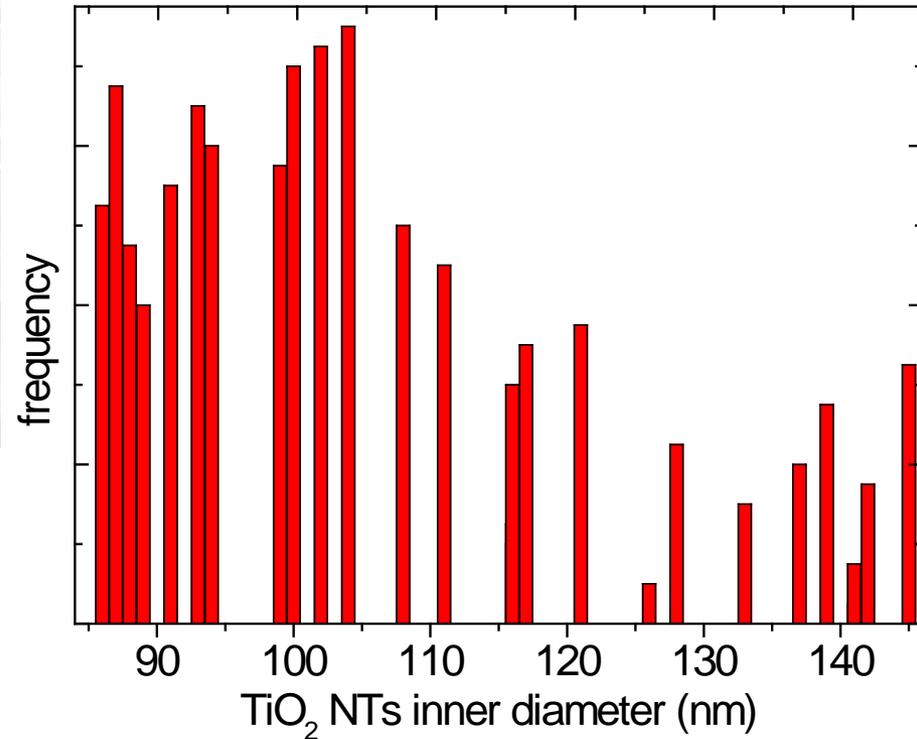
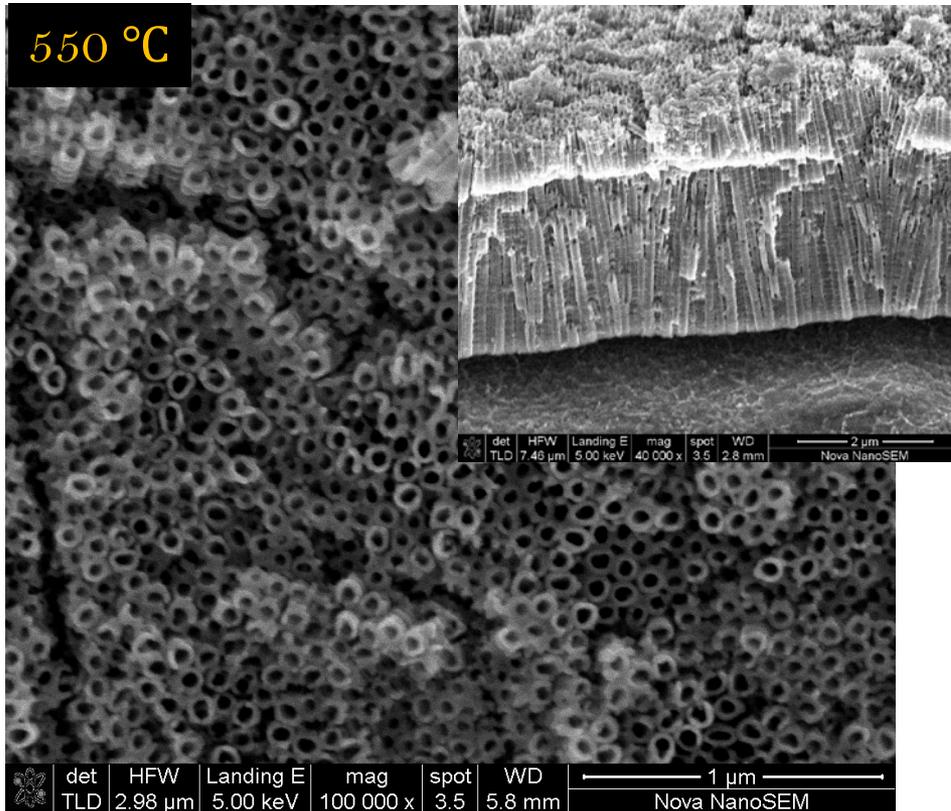
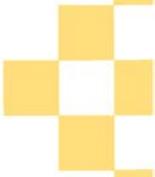
Results : SEM



- Well orientated surface morphology of TNTs on FTO glass substrate.
- Increased TNT inner diameter size (150- 170 nm).



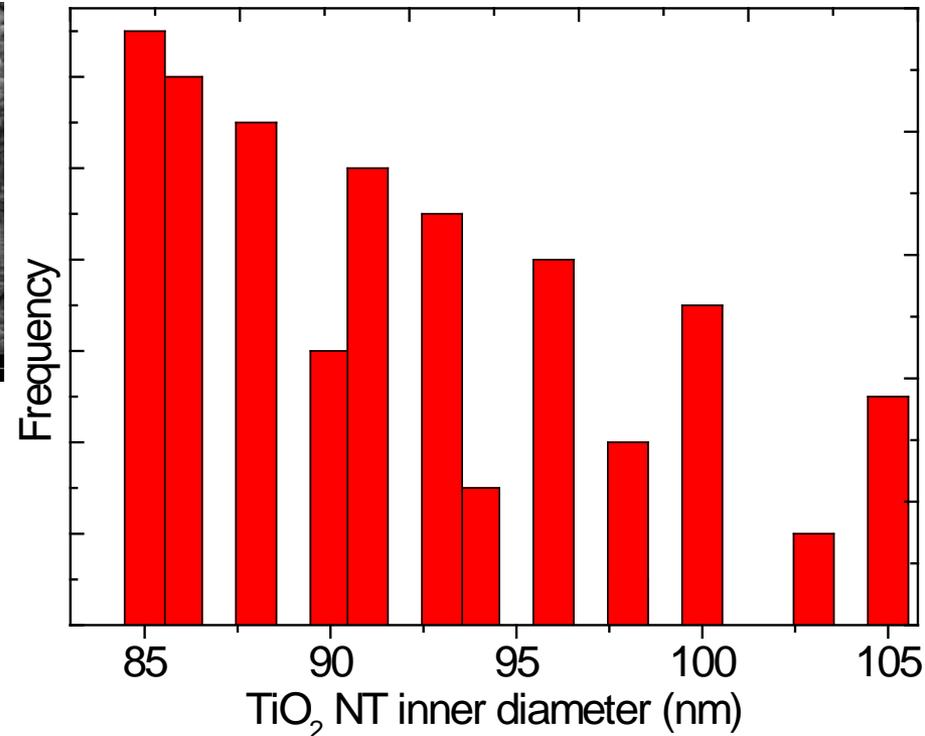
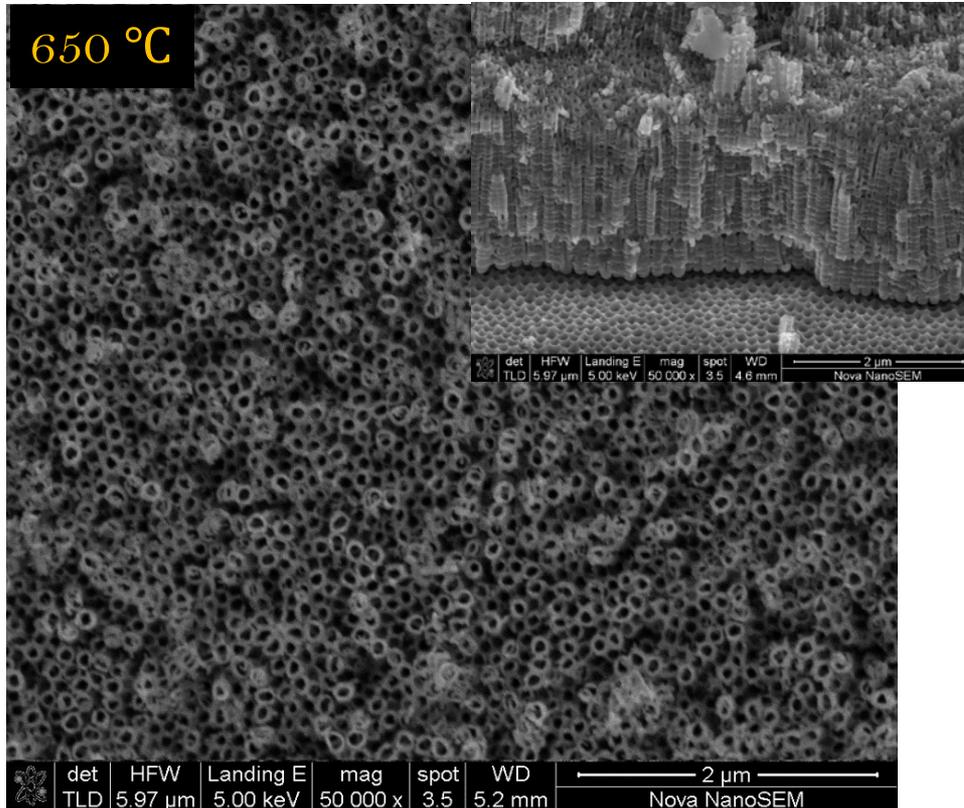
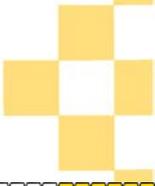
Results : SEM



- Cross sectional view shows smooth & well aligned TNTs.
- Increase in temperature results in greatly enhanced morphology of TNTs.



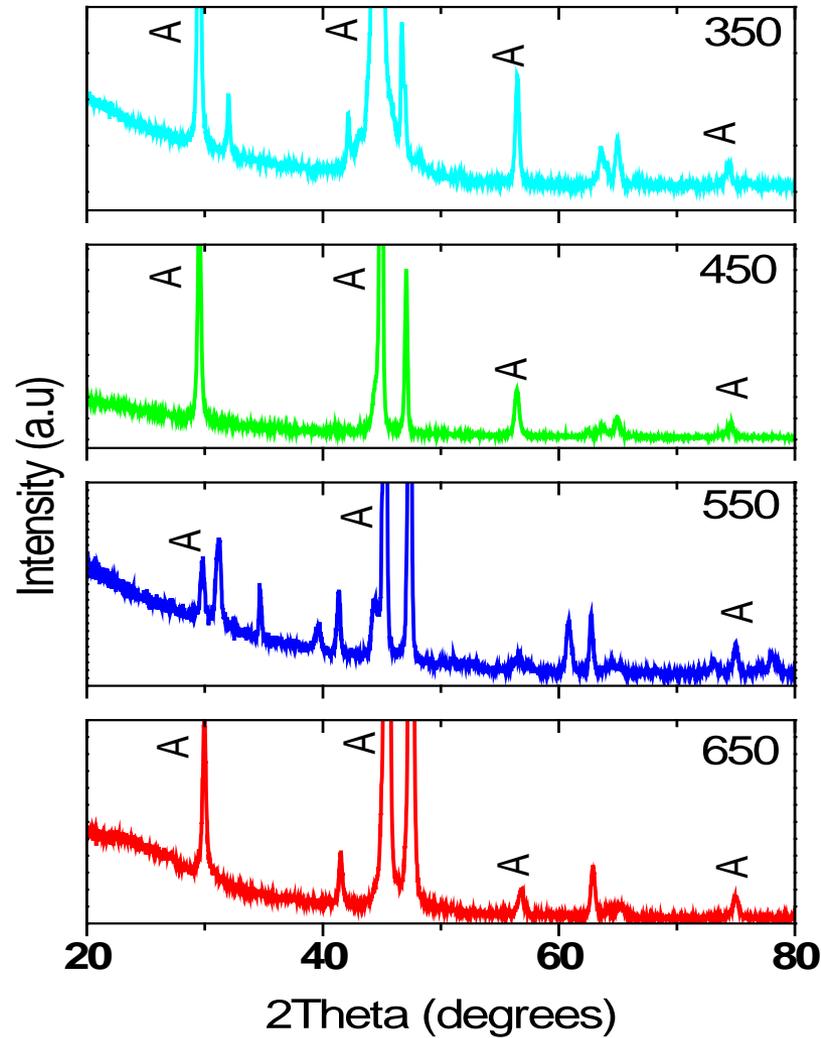
Results : SEM



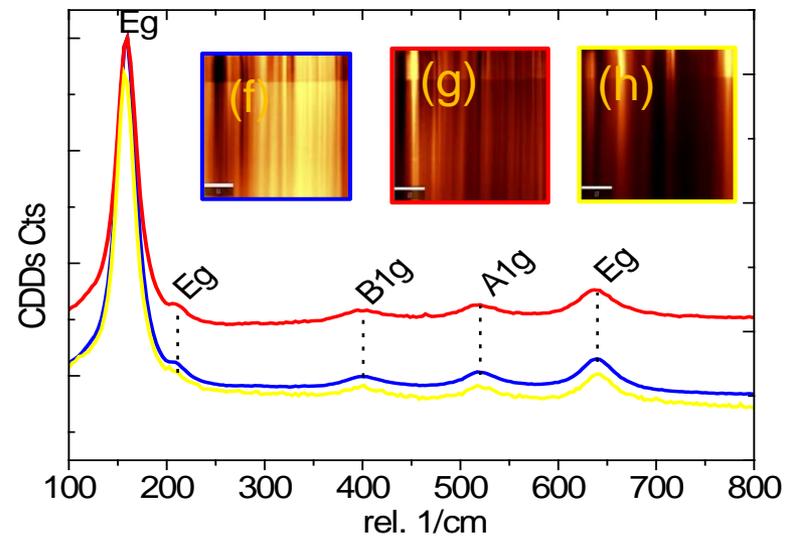
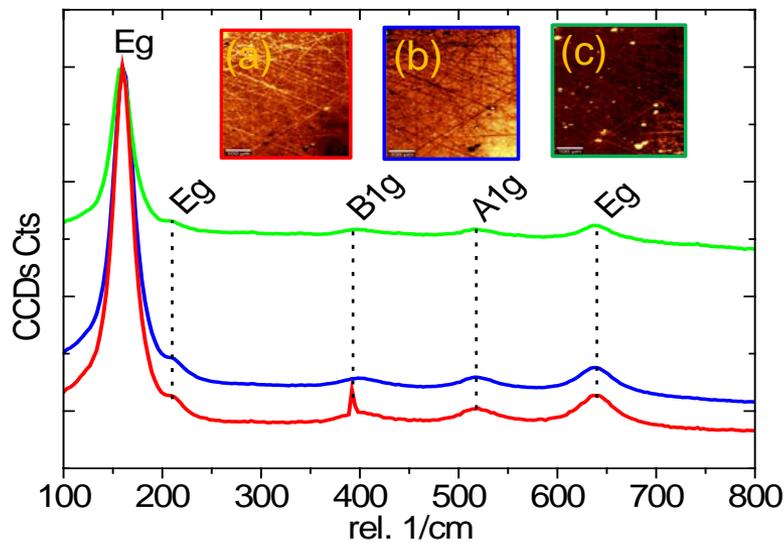
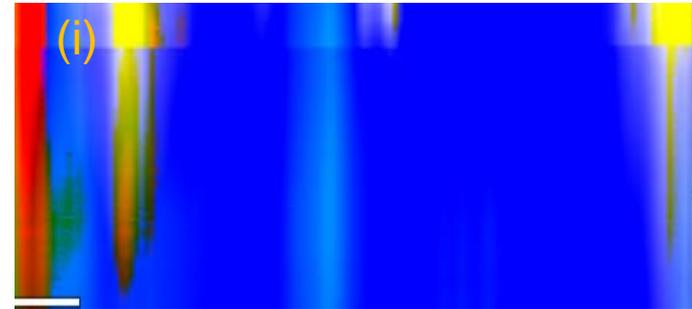
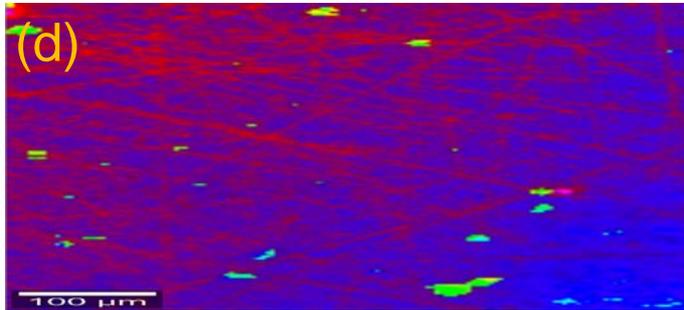
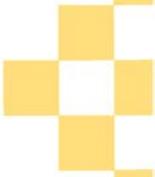
- SEM revealed orientated surface morphology of TNTs grown on FTO glass substrate.
- No morphology disruption or deformation observed of TNTs at 650 °C.



Results : XRD



Results : CRS

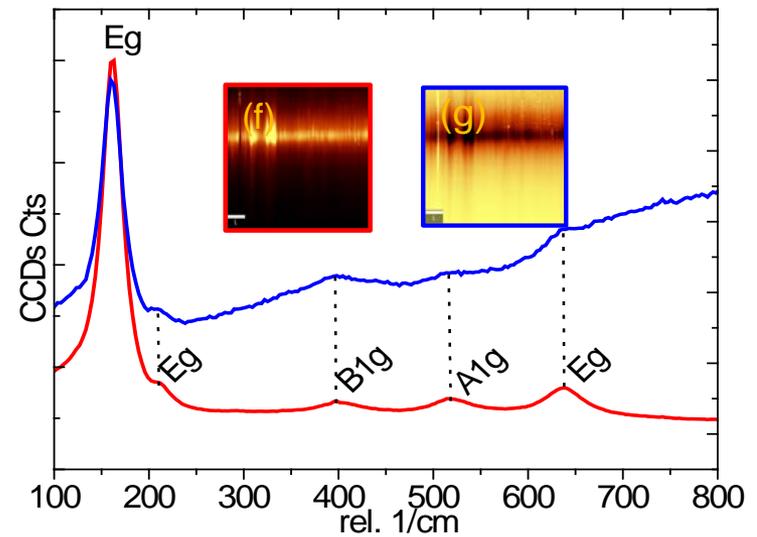
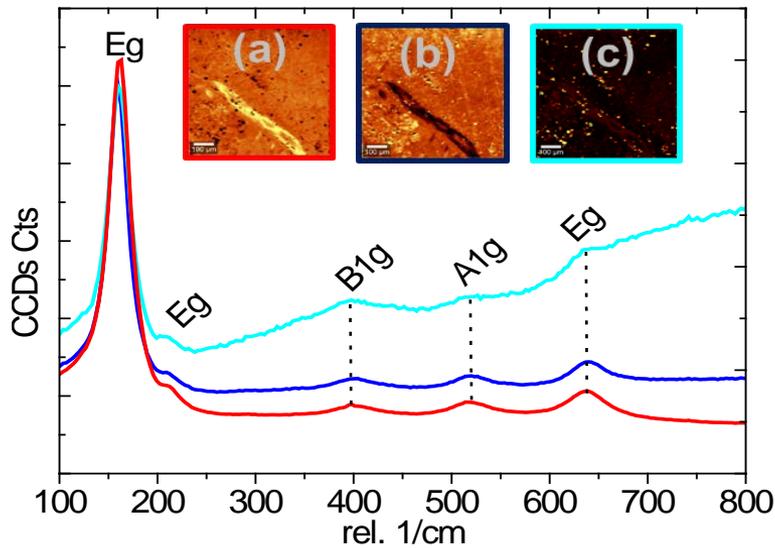
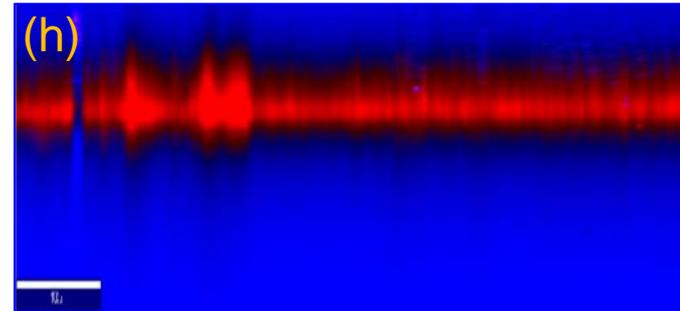
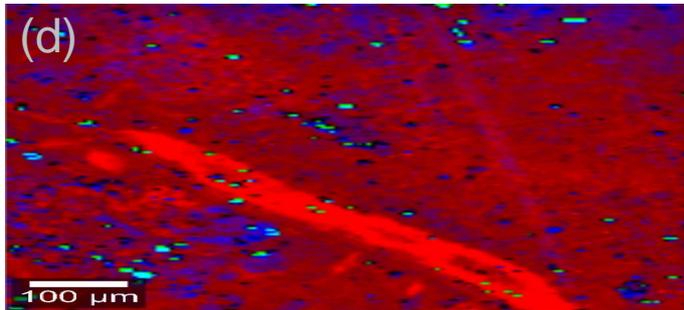
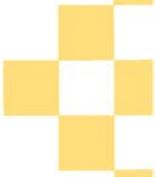


LAS (XY) TNT 350 °C

Depth (XZ) TNT 350 °C



Results : CRS

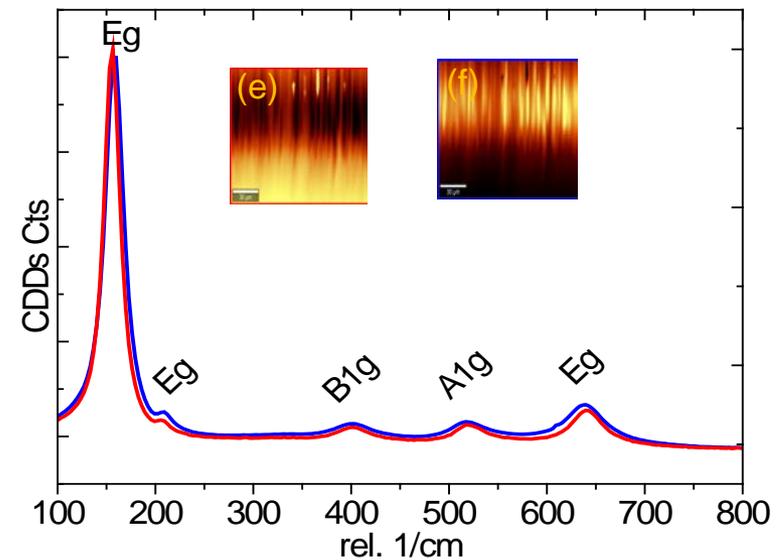
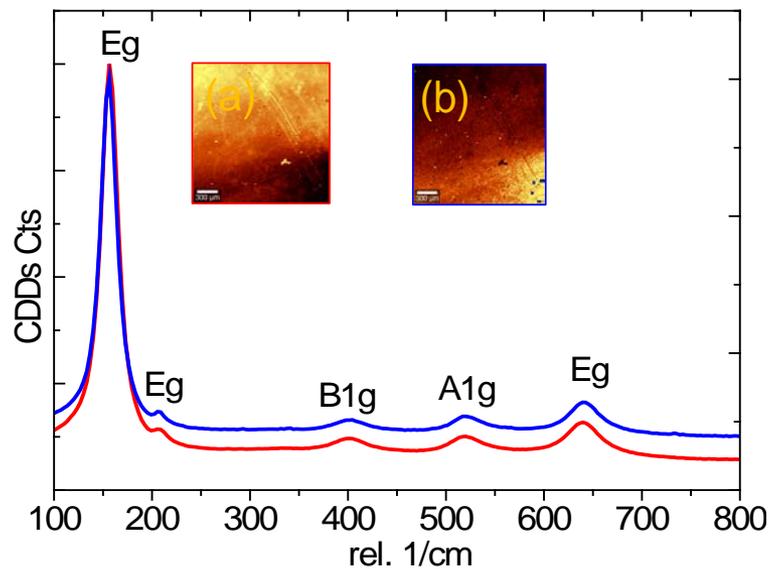
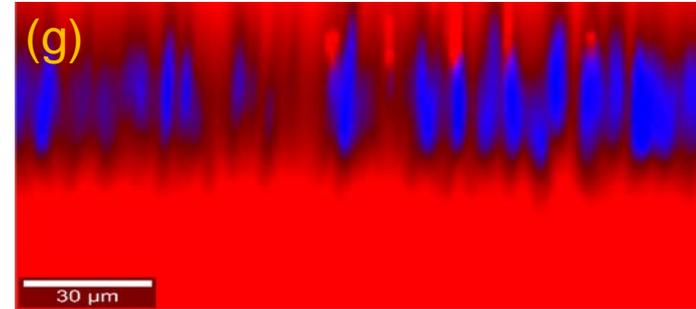
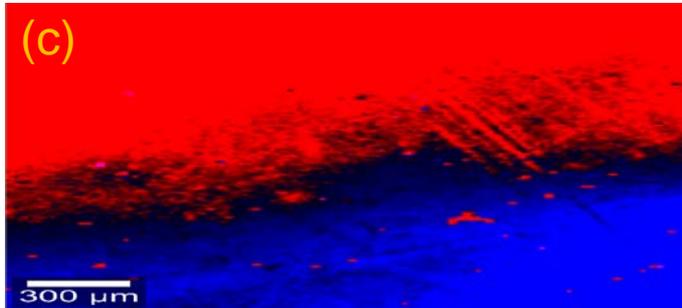


LAS (XY) TNT 450 °C

Depth (XZ) TNT 450 °C



Results : CRS

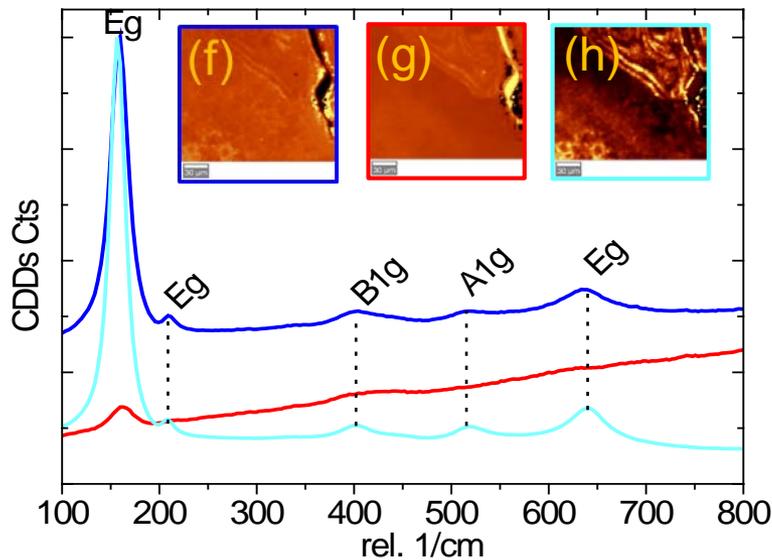
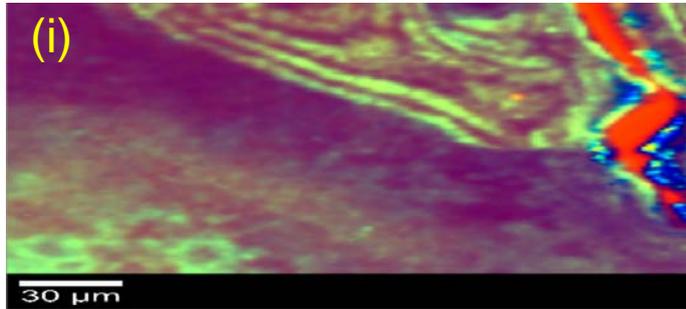


LAS (XY) TNT 550 °C

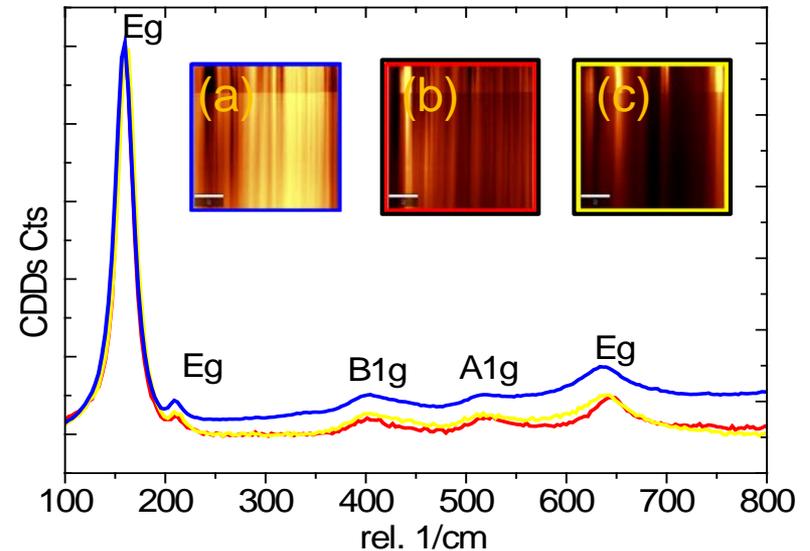
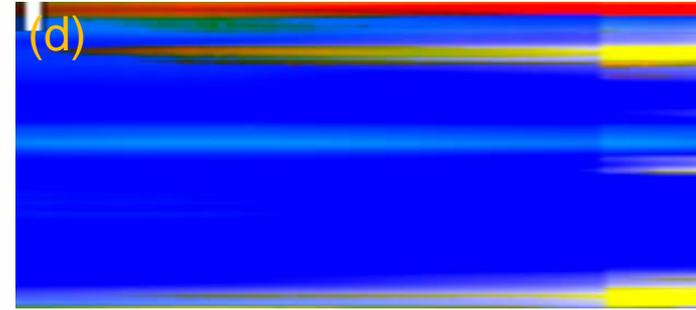
Depth (XZ) TNT 550 °C



Results : CRS



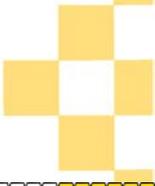
LAS (XY) TNT 650 °C



Depth (XZ) TNT 650 °C



Conclusion

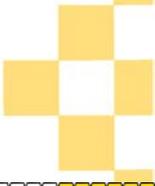


- TNT arrays with a thickness of $\pm 10 \mu\text{m}$ and a pore diameter range 85 – 170 nm were successfully grown on transparent conductive FTO substrates by anodizing the sputtered Ti films.
- SEM micrographs show the regular morphology with no disruption or deformation of TNTs on FTO substrate even at elevated temperature (550 °C & 650 °C).
- CRS analysis (LAS & Depth) & XRD confirmed the presence of Anatase TNTs on FTO glass substrate with increase in temperature resulting in high peaks intensities, thus high structural phase maturity.
- The enhanced morphology & presence of only Anatase phase TNTs make these substrates suitable for DSSCs.

Acknowledgements



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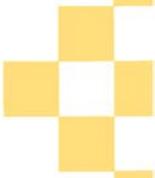
- I would like to thank God almighty for the gift of life and wisdom.
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THANK YOU ALL

