

Report on the experiment “GISAXS study of ordering in graded mesoporous titanium dioxide films” (MA-7) on the beam-line ID01 of the ERSF on July 12. – 15. 2006

We have characterized a series of 13 samples with successively deposited  $\text{TiO}_2$  nanocrystalline layers on conducting glass support using GISAXS measurement methods at  $\lambda=1.24 \text{ \AA}$  ( $E=10 \text{ keV}$ ). The series was designed to obtain cross-references about the structure of organized  $\text{TiO}_2$  mesoporous layers in different layers. The samples were differently doped and processed at several different temperatures. The GISAXS experiments were supposed to help in determination of the ordering, roughness, and overall density of the individual layers, and densification of layers at high temperature during the thermal treatment. The Fig. 1 is an SEM image of the typical organized  $\text{TiO}_2$  structure prepared by a templated growth. The Fig. 2 shows reflective scans that point to the overall density of the layers and their collapsing at high thermal treatments. Scanning under different angles provided us useful information about the types of porosity, pore size and particle size. In addition, we could see difference in the structure between the top and under-layers (Fig. 3 is showing the X-ray penetration depth profile). Extending the scan range to wide angles (Fig. 4) allowed us obtaining valuable information about the crystallinity of the primary particles and helped in the determination of the crystallite size. The study was extremely helpful and it has moved our research significantly forward. Interpretation of results is in progress. We need to say thank to the ID01 beam-line staff, especially to our instructor Gerardina Carbone. Her help and guidance made our measurements efficient and smoothly going.

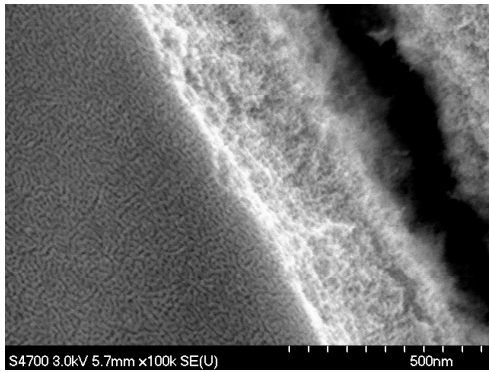


Fig 1

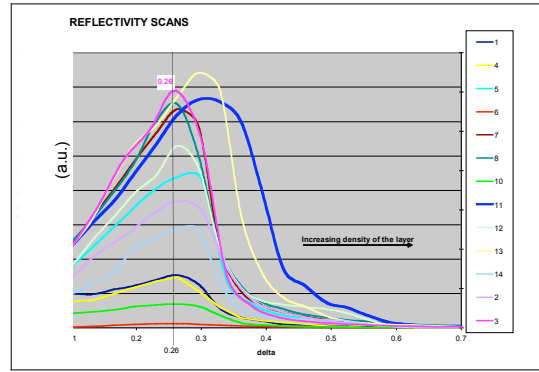


Fig 2

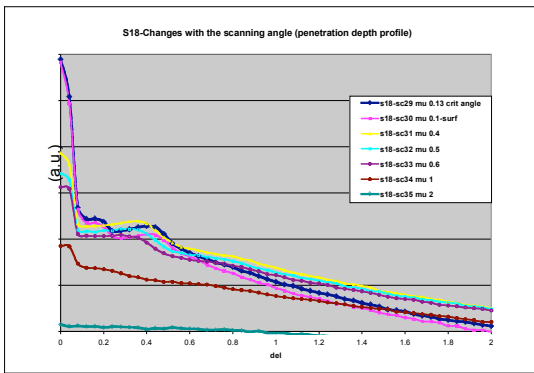


Fig 3

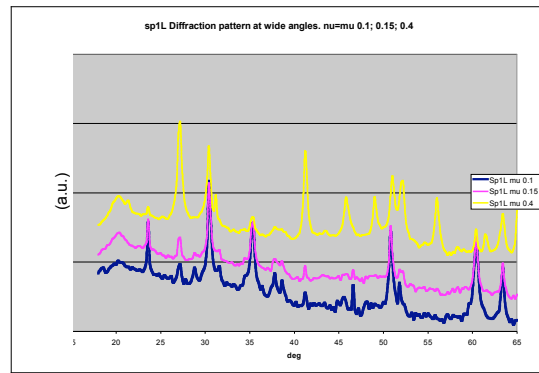


Fig 4