

# Phase Characterization of TiO<sub>2</sub> Powder by XRD and TEM

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## ABSTRACT

In this study, the commercial TiO<sub>2</sub> nanopowder and micropowder of anatase phase and rutile phase have been characterized by x-ray diffraction (XRD) and transmission electron microscopy (TEM). XRD patterns of nano-TiO<sub>2</sub> in rutile and anatase phases exhibit broad peaks whereas both phases of micro-TiO<sub>2</sub> demonstrate very sharp peaks. TEM images show that the grain size of TiO<sub>2</sub> micropowders and TiO<sub>2</sub> nanopowders are 0.3-0.7 μm and 10 nm, respectively. The selected-area electron diffraction patterns of TiO<sub>2</sub> nanopowders in rutile and anatase phases are consistent with XRD results.

**Key words:** TiO<sub>2</sub>, anatase, rutile, micro and nanopowders, TEM, XRD

## INTRODUCTION

Titanium dioxide or titania (TiO<sub>2</sub>) was first produced commercially in 1923. It is obtained from a variety of ores. The bulk material of TiO<sub>2</sub> is widely nominated for three main phases of rutile, anatase and brookite (Kim *et al.*, 2005). Among them, the TiO<sub>2</sub> exists mostly as rutile and anatase phases which both of them have the tetragonal structures. However, rutile is a high-temperature stable phase and has an optical energy band gap of 3.0 eV (415 nm), anatase is formed at a lower temperature with an optical energy band gap of 3.2 eV (380 nm) and refractive index of 2.3 (Brady, 1971).

TiO<sub>2</sub> is mainly applied as pigments, adsorbents, catalyst supports, filters, coatings, photoconductors, and dielectric materials. In recent years, TiO<sub>2</sub> has been well known as a semiconductor with photocatalytic activities and has a great potential for applications such as environmental purification, decomposition of

carbonic acid gas, and generation of hydrogen gas (Zhang *et al.*, 2000). In most of these cases, the size of the TiO<sub>2</sub> particles is an important factor affecting the performance of the materials. It is not surprising; therefore, that much research has been focused upon the reduction of the particle size. Much effort has been devoted to the preparation of TiO<sub>2</sub> nanopowders, including sol-gel route, homogeneous precipitation, hydrothermal methods, flame synthesis and relatively new molten salts method (Bilik and Plesch, 2007). They were usually found that different routes often produced different results. Even for the same route, using different amount of the starting materials, the obtained powder size is different (Li *et al.*, 2002).

Consequently, phase and particle size are the important parameters that influence physical properties of material. Several techniques could be used for the investigation of them. However, the particle size determination can be based on direct observation of particles by transmission

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electron microscopy (TEM) or scanning electron microscope techniques, in this case, we can also receive the important information on the shape of particles. Data on particle size can be obtained by X-ray diffraction (XRD) technique as the particle size is related to the diffraction peak broadening. It is important to note that TEM and XRD methods allow not only to measure the particle size, but also to identify crystalline phases.

## MATERIALS AND METHODS

Four commercial TiO<sub>2</sub> samples used in this experiment were provided by Aldrich: (i) 99.7 % anatase, nanopowder; (ii) 99.8 % anatase, micropowder; (iii) 99.5 % rutile, nanopowder; (iv) 99.9 % rutile, micropowder.

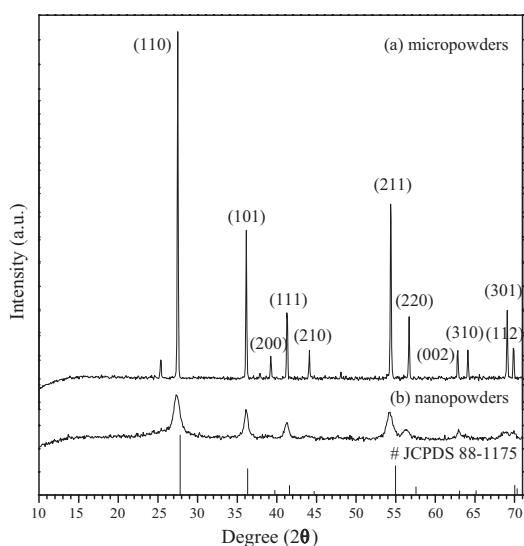
XRD measurements were performed on the Bruker D8 Advance diffractometer operating in the reflection mode with Cu-K $\alpha$  radiation (35 kV, 30 mA) and diffracted beam monochromator, using a step scan mode with the step of 0.075° (2 $\theta$ ) and 4 s per step. Diffraction patterns of both anatase and rutile TiO<sub>2</sub> powders were compared with reference to JCPDS database. Additionally, the morphology of the powder was observed by

TEM with a JEOL JEM-2100 ultra high resolution TEM operating at 160 kV.

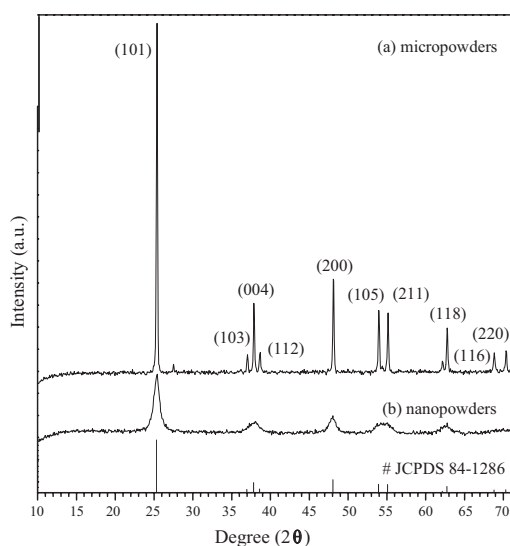
## RESULTS AND DISCUSSION

### XRD

XRD patterns of nano-TiO<sub>2</sub> and micro-TiO<sub>2</sub> in rutile and anatase phases are shown in Figure 1 and Figure 2, respectively. In Figure 1, XRD patterns exhibited strong diffraction peaks at 27°, 36° and 55° indicating TiO<sub>2</sub> in the rutile phase. On the other hand, in Figure 2, XRD patterns exhibited strong diffraction peaks at 25° and 48° indicating TiO<sub>2</sub> in the anatase phase. All peaks are in good agreement with the standard spectrum (JCPDS no.: 88-1175 and 84-1286). From Figure 1 and Figure 2, they were shown that the diffraction pattern peak intensity of the TiO<sub>2</sub> increases with increasing particles size. These results suggested that the nano-TiO<sub>2</sub> powder is composed of irregular polycrystalline. Amorphous revealed a broad pattern with low intensity; however, the effect of the amorphous materials on the broadening of the XRD patterns of nanosized TiO<sub>2</sub> is negligible.



**Figure 1** X-ray diffraction of rutile TiO<sub>2</sub> (a) micropowders and (b) nanopowders.

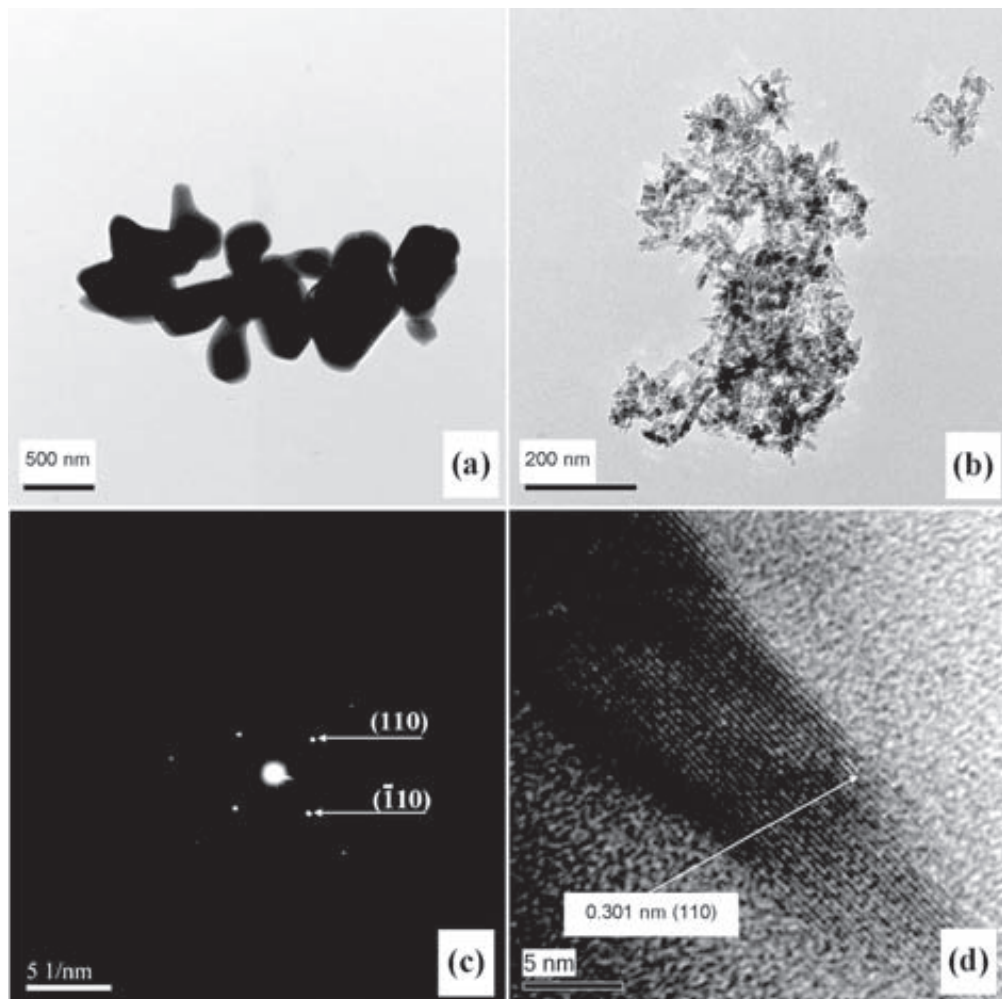


**Figure 2** X-ray diffraction of anatase TiO<sub>2</sub> (a) micropowders and (b) nanopowders.

## TEM

TEM was used to further examine the particle size, crystallinity and morphology of samples. TEM bright field images of  $\text{TiO}_2$  micropowders in rutile and anatase phases are shown in Figure (3a) and (4a), respectively. It is clearly seen that the  $\text{TiO}_2$  powders in rutile phase consist of both spherical and rod shapes but the particle of  $\text{TiO}_2$  powders in anatase phase are mostly spherical morphology. Furthermore, it can be estimated that the particle size of samples in Figure (3a) and (4a) are microscale with the grain size about 0.3-0.7  $\mu\text{m}$ .

TEM bright field images of  $\text{TiO}_2$  nanopowders in rutile and anatase phases are shown in Figure (3b) and (4b), respectively. It can be estimated that the particle size of powders in Figure (3b) and (4b) are nanoscale with the grain size less than 10 nm. The corresponding selected-area electron diffraction (SAED) patterns of nano- $\text{TiO}_2$  powders in rutile and anatase phases are shown in Figure (3c) and (4c), respectively. These are in agreement with XRD results in Figure (1b) and (2b), respectively. In Figure (3c), the SAED patterns of nano- $\text{TiO}_2$  powders in rutile phase shows spotty ring patterns without any additional



**Figure 3** Images of rutile phase. (a) TEM image of micro- $\text{TiO}_2$  powder; (b) TEM image of nano- $\text{TiO}_2$  powder; (c) SAED pattern of nano- $\text{TiO}_2$  powder and (d) HRTEM image of nano- $\text{TiO}_2$  powder.

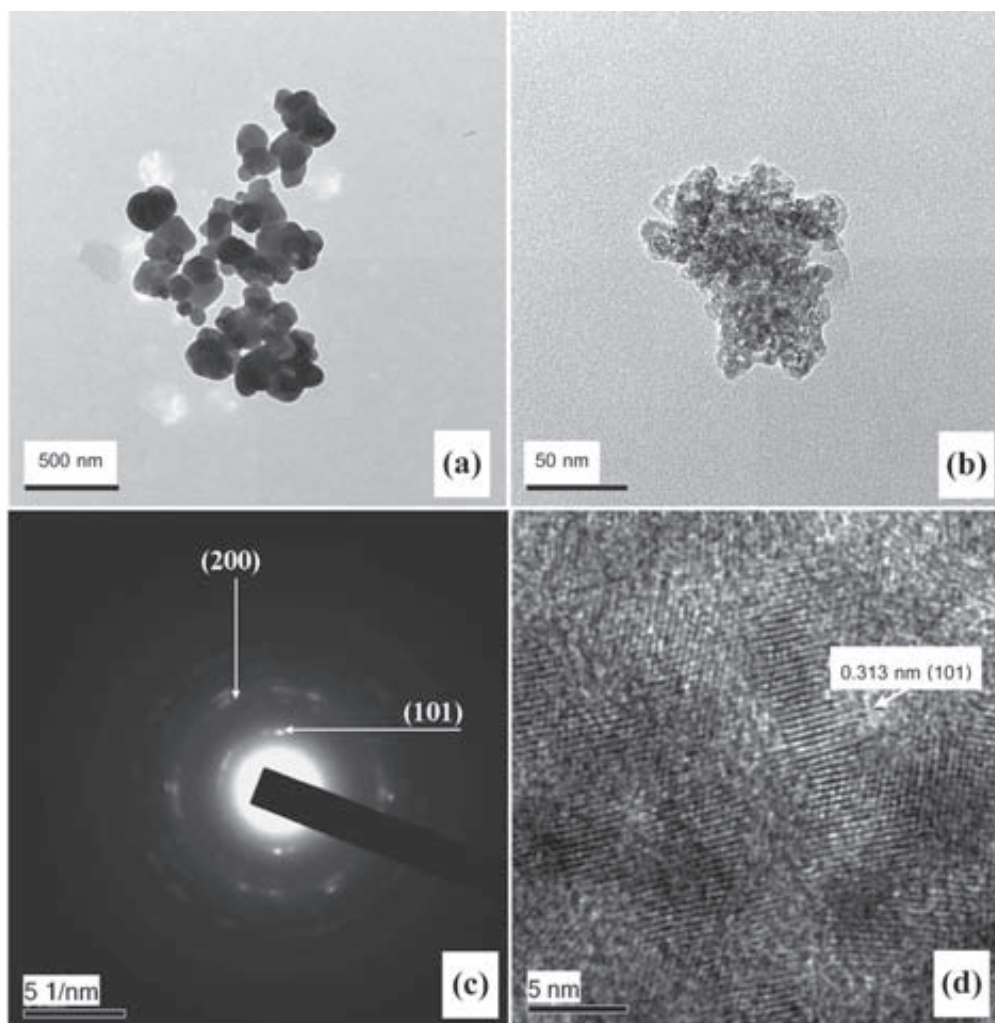
diffraction spots and rings of second phases, revealing their well crystalline. On the other hand, the SAED patterns of nano-TiO<sub>2</sub> powders in anatase phase (Figure (4c) shows that the brightness and intensity of polymorphic ring is weak, so they are poorly crystallized and partly amorphous.

The crystallinity of nano-TiO<sub>2</sub> powders can also be observed by phase-contrast images or Moire patterns. Figure (3d) and (4d) show crystal lattice planes of nano-TiO<sub>2</sub> in rutile and anatase phases, respectively. It is seen that, for rutile phase,

only one crystal lattice plane(110) with d-spacing of 0.301 nm is obtained, whereas those of anatase give many crystal lattice planes with d-spacing of 0.313 nm for the plane (101).

## CONCLUSION

In this work, a study has been carried out on the identification of phase and particle size of TiO<sub>2</sub> powders using XRD and TEM techniques. All high purities samples were commercial TiO<sub>2</sub>. From the results, X-ray diffraction patterns can



**Figure 4** Images of anatase phase. (a) TEM image of micro-TiO<sub>2</sub> powder; (b) TEM image of nano-TiO<sub>2</sub> powder; (c) SAED pattern of nano-TiO<sub>2</sub> powder and (d) HRTEM image of nano-TiO<sub>2</sub> powder.

confirm the TiO<sub>2</sub> phases. Furthermore, the particle size can be clearly indicated by characteristic of XRD pattern; the diffraction pattern peak intensity of the TiO<sub>2</sub> increases with increasing particles size. Additionally, TEM was used to further examine the crystallite/particle size, the crystallinity and morphology of samples. TiO<sub>2</sub> powders in rutile phase consist of both spherical and rod shape; on the contrary, the particle of TiO<sub>2</sub> in anatase phase has mostly spherical morphology.

#### ACKNOWLEDGEMENTS

This work is financially supported by the Department of Physics, the Faculty of Science, King Mongkut's University of Technology Thonburi and the Nano Technology Center (NANOTEC).

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