

PREPARATION AND UV CHARACTERIZATION OF TiO₂ NANOPARTICLES SYNTHESIZED BY SANSS

Ching-Song Jwo¹, Der-Chi Tien², Tun-Ping Teng³, Ho Chang⁴,
Tsing-Tshih Tsung⁴, Chih-Yu Liao⁵ and Chi-Hsiang Lin¹

¹Department of Air-Conditioning and Refrigeration Engineering, National Taipei University of Technology, Taipei, 10608, Taiwan, R. O. China

²Institute of Mechatronic Engineering, National Taipei University of Technology, Taipei, 10608, Taiwan, R. O. China

³Institute of Mechanical and Electrical Engineering, National Taipei University of Technology, Taipei, 10608, Taiwan, R. O. China

⁴Department of Mechanical Engineering, National Taipei University of Technology, Taipei, 10608, Taiwan, R. O. China

⁵Department of Electrical Engineering, National Taipei University of Technology, Taipei, 10608, Taiwan, R. O. China

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Abstract. This paper proposes preparation and investigation of UV characterization of TiO₂ nanocomposite synthesized by SANSS. A Submerge Arc Nano particles Synthesis System (SANSS) to produce TiO₂ nano particles in deionized water without any of dispersant [1,2] has been successfully accomplished in this study. Uniform dispersion, deep UV optical absorption, and crystalline structure of TiO₂ nano particles are investigated. Particularly, deep UV absorbance is the unique characteristic of TiO₂ nano particles synthesized by SANSS. The evidence of deep UV absorbance reveals the unique quantum effect of TiO₂ nano particles suspend in deionized water. As a result, the observed absorbance pattern indicates absorbent energy of TiO₂ nano fluids synthesized by proposed method is higher than that of TiO₂ nano fluids prepared by other conventional fabrication process. The observed UV absorbance of the nano fluids demonstrates the TiO₂ molecular tends to react with higher activation energy. After being exposed to the UV light irradiation, the TiO₂ nano particles will spontaneously correspond for photo catalytic reaction mechanism through hydroxyl radicals with higher kinetic energy which can cause decomposition of high energetic covalent bond such as C=C, C-H, etc. (Legan, 1982) [3]. The result of the experiment indicates that VOC (volatile organic compounds) with C-C, C=C, C-H structures such as Toluene now is more effectively to be decomposed by UV irradiation with existence of TiO₂ nano particles synthesized by proposed SANSS process.

1. INTRODUCTION

Nanomaterials reveal new functions which are derived both from the geometric size of nanostructures and from their material-specific properties. In the investigation of material properties, compounds that are employed as photocatalysts include oxides such as TiO₂, ZnO, Nb₂O₅, WO₃, SnO₂, and ZrO₂, as well

as sulfides such as CdS and ZnS. Among these, titanium dioxide (TiO₂) emerges with its advantages of lower cost, biologically and chemically inert. In addition, it has received increasing attention for water and air purification, due to energy of UV photon irradiation it has used to generate holes and electrons, which oxidizes toxic organic pollutants to

Corresponding author: Ching-Song Jwo, e-mail: s2458034@ntut.edu.tw

environmentally harmless components. Although there are many literatures dealing with the subject of producing TiO₂ nano particles in various ways, but few literatures investigate the TiO₂ nano particles fabricated by arc-discharge process.

According to the basic principles of the gas condensation method and using deionized water as dielectric liquid, a Submerge Arc Nanoparticles Synthesis System (SANSS) for preparing TiO₂ nano particles suspension [1,2] under low temperature and pressure has been proposed. Under certain high voltage and regulated current, a pair of titanium bars, as the electrodes, will be close enough to ionized, melted and vaporized in deionized water. Through arc-discharge process, an oxidized and vaporized TiO₂ particles are rapidly quenched by the specially designed cooling system with low atmosphere condition. Then nanocrystalline TiO₂ particles will nucleate and form. The experimental results indicate that uniform distribution and well-controlled size of nanocrystalline particles can be generated by maintaining a low operating temperature in the vacuum chamber and by applying an adequate electric current to the titanium electrodes.

2. EXPERIMENTAL

2.1. Experimental description, background, method

In the process, a bulk metal as the electrode is submerged in dielectric liquid in a vacuum chamber.

Using electric energy source to generate an adequate arc with a high temperature ranging from 6000 to 12000 °C [4]. During the process, arcing area of titanium electrodes on both side are melted and vaporized in deionized water, H₂O molecules with high pressure are generated due to the high temperatures condition of surrounding dielectric liquid. The high-pressure water vapor promoted effectively push away the vaporized titanium aerosol from the electrodes. Then, the vaporized aerosol present in the dielectric liquid changes from its current gas phase to the nucleating, growing and solidifying stages, and eventually becomes metal nanoparticles dispersed in the dielectric liquid. Because the dielectric liquid is controlled at a low temperature and low pressure, the vaporized metal can be condensed more efficiently in the dielectric liquid than that of nanoparticles prepare by normal atmosphere condition. Meanwhile, since the submerged arc is generated steadily and the pressure of the vacuum chamber and the temperature of the

dielectric liquid are controlled at a desired level, the vaporized metal gas can be effectively transformed into homogenous particles in nano-scale range through particle nucleation and grain growth.

In this work, the suspension with well-dispersed TiO₂ nanoparticles was successfully prepared.

2.2. Experimental system

The schematic diagram of the experimental setup is shown in Fig. 1a. It is composed primarily of an electrical arc-discharge system, a servo control system, a pressure control system, and a temperature control system.

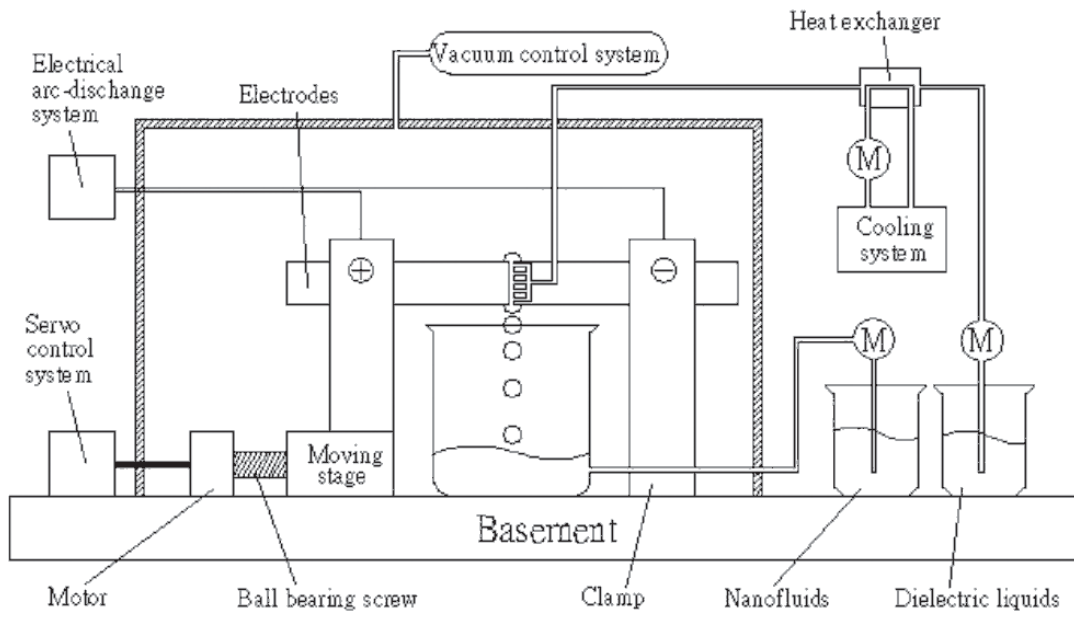
2.3. Experimental procedures

Fig. 1b illustrates the procedures for preparing nanoparticle suspension. Titanium rods were used as both positive and negative electrodes. First, the submerged arc in the reacting chamber heated the pure metallic rod. The parameter control system was deployed to regulate various process parameters shown in Table 1. The instruments operated by the combined SANSS, which include the electrical arc-discharge system, servo control system, constant pressure system, and isothermal system.

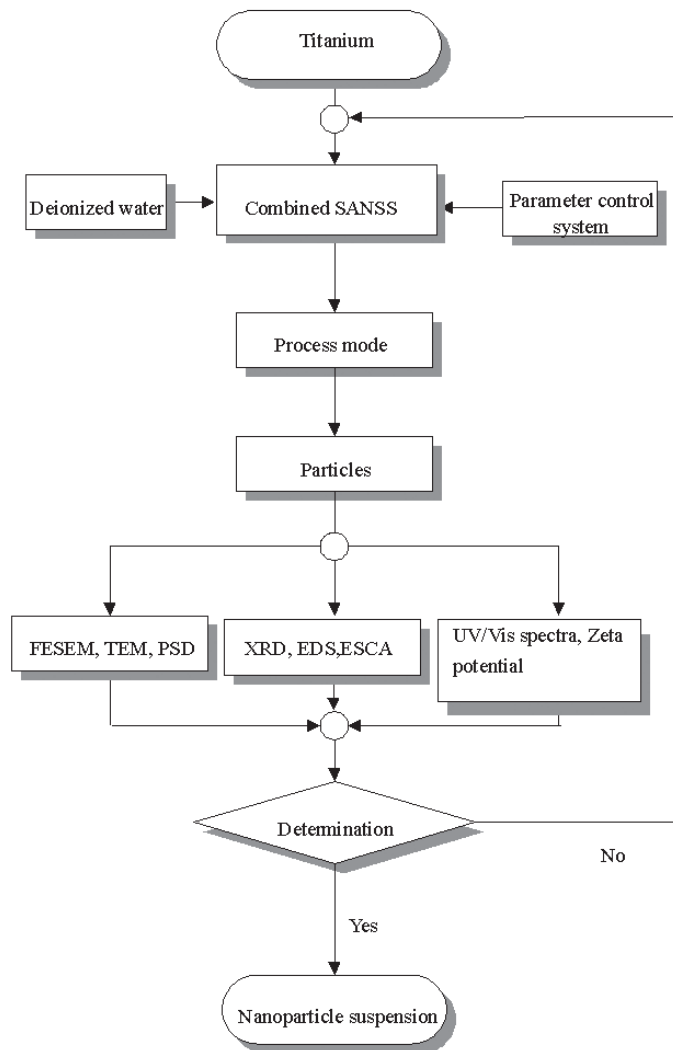
The governing parameters of these instruments, such as the applied electric current, pulse duration, off-time duration, breakdown voltage, electrode gap, pressure of the vacuum chamber and the temperature of deionized water, are crucial determinants of the nanoparticles suspension. After the titanium particles have been vaporized and condensed for a period of time, the nanoparticle suspension was sampled to a collector for inspection and analysis to determine the characteristics of nanoparticles, such as particle size, structure, zeta potential, optical property and stability of the nanoparticle suspensions.

2.4. Sample characterization

The prepared nanoparticles were characterized for microstructural properties by the Transmission Electron Microscope (TEM, JEOL JSM-1200EX2 and Hitachi-H7100) and the Field Emission Scanning Electron Microscope (FESEM, EM0093). The chemical composition was verified using the electron spectroscopy for chemical analysis (ESCA, Omicron Multiprobe Compact). The dry nanoparticle powders could be obtained by heating the particle suspension with an appropriate temperature. The crystalline phase was determined by X-ray Diffrac-



(a)



(b)

Fig. 1. (a) Schematic diagram of the Submerged Arc-Discharge Nanoparticle Synthesis System. (b) Flow chart of nanoparticle suspension prepared by SANSS.

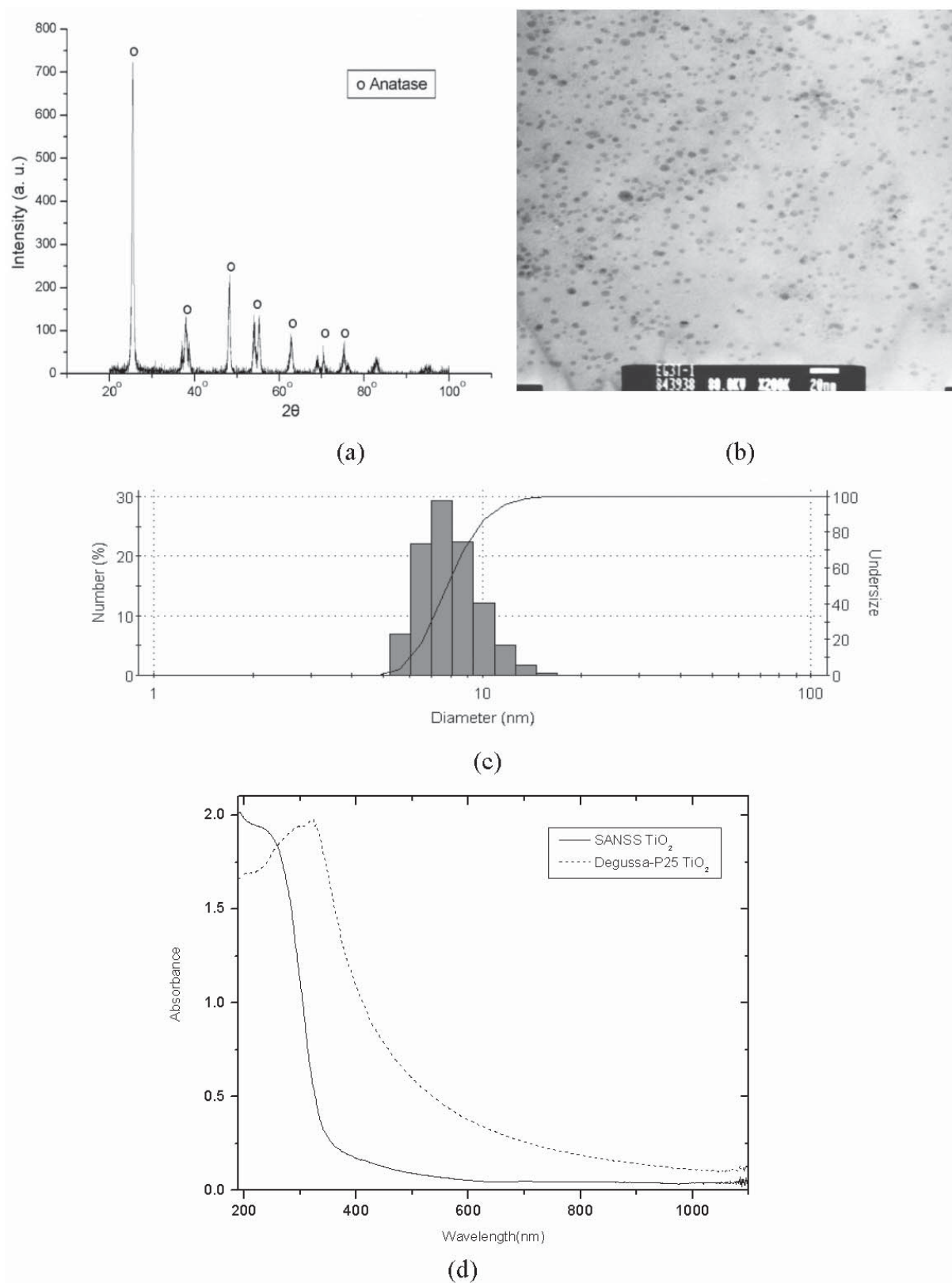


Fig. 2. Instrument measurements: (a) XRD pattern of SANSS TiO₂; (b) TEM image of SANSS TiO₂; (c) Particle size of SANSS TiO₂; (d) Deep UV absorption spectra of SANSS TiO₂.

Table 1. Process variables for TiO₂ nanofluid production.

Open voltage	100 V
Peak Current	2.5 A
Pulse duration	10 μs
Off time	30 μs
Temperature of dielectric	0.5 °C
Pressure	50 Torr
Capacity of dieionzed water	130 ml
Working time	10 min
Tool polarity	Positive

tion (XRD, MAC-MXP18). All peaks measured by XRD analysis were assigned by comparing with those of Joint Committee on Powder Diffraction Standards (JCPDS) data. The Particle Size Distribution (PSD) of the nanoparticles was acquired by dynamic light-scattering measurement (Horiba-LB500). The Energy Dispersive X-ray Spectroscopy (EDS, EM0093) is also used to analyze the component of the Nanoparticle suspension. Meanwhile, a Ultra-violet-Visible (UV-Vis) spectrophotometer (UV-500, U-2001) is used to analyze the optical property of the Nanoparticles.

3. RESULTS AND DISCUSSION

This study uses the SANSS to prepare TiO₂ nanofluids and X-ray Diffraction (XRD) to confirm the crystal phase of the particles. As indicated by the comparison in Fig. 2a, the nanoparticles produced from the process are Anatase TiO₂; Table 1 summarizes the working conditions conducive to the preparation of ideal nanoparticles by the SANSS. The TEM image of the nanoparticle suspension prepared using the process variables in Table 1 is shown in Fig. 2b. Fig. 2c is the particle size distribution, under volume distribution derived from the HORIBA particle size distribution analyzer, which indicates a mean particles size of 30 nm. With the aid of low temperature and low pressure which can reduce the particle size below 10 nm as shown in Fig. 2b. Fig. 2d shows a series of UV-Vis absorption spectra of TiO₂ nanoparticles suspension produce by SANSS. The observed UV absorbance pattern indicates that TiO₂ particles react with deep UV, and corresponds for photo catalytic reaction mechanism, which tends to de-bond the atoms with higher volant bond. The Photo catalytic experiment shows the degradation

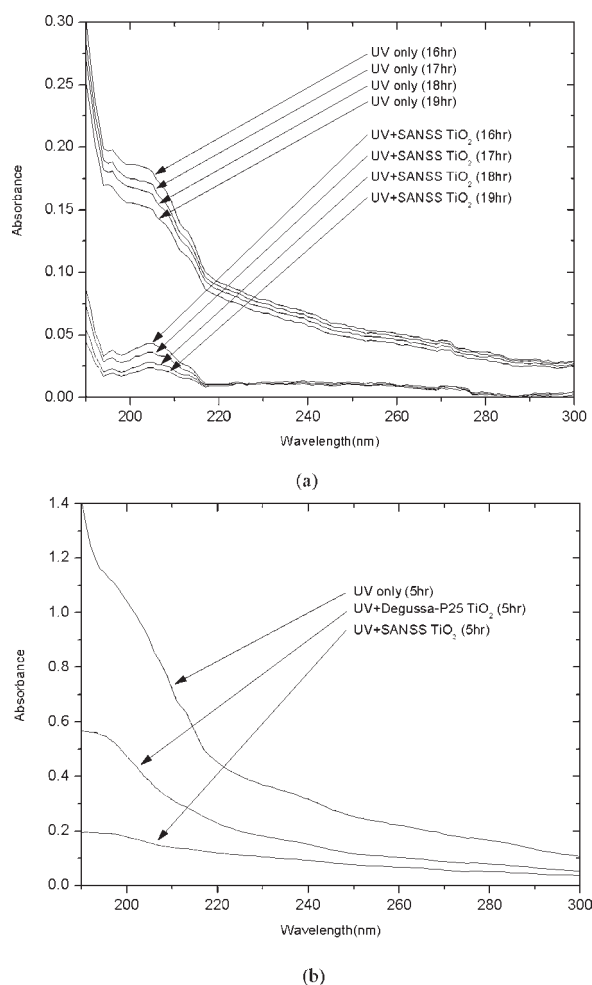


Fig. 3. (a) Degradation of Toluene with/without SANSS TiO₂ under UV exposure. (b) Comparison of the Toluene degradation of by SANSS TiO₂ and Degussa-P25 TiO₂ under UV irradiation.

of toluene and its residue will be incompletely decomposed, over a long period by intensively exposing to UV irradiation only. However, with the aid of incorporated SANSS TiO₂, which inherits the deep UV characteristics, toluene residue will be resolved entirely in the chamber by UV exposure for an adequate time, as shown in Fig. 3a. As a result, Fig. 3a shows that the organic chemical compound treated by UV irradiation with or without TiO₂ will obtain significant difference in decomposition of toluene molecules.

4. CONCLUSION

According to the basic principles of the gas condensation method, a Submerge Arc Nanoparticle Synthesis System (SANSS) for preparing TiO₂

nanofluids have been successfully developed. From the experimental results and the discussion above, the following conclusions can be stated.

- (1) The stable state of suspension is the unique characteristic of TiO₂ nano particles synthesized by SANSS.
- (2) The deep UV characteristic resulted from the particle structure size around 10 nm.
- (3) The total decomposition of Toluene by UV light irradiation only is not successfully achieved [5]. However, under the photo catalytic effects of incorporated TiO₂ nanoparticles, the residue of toluene can be easily removed.
- (4) The comparison of the decomposition performance obtained using SANSS TiO₂ and Degussa-P25 TiO₂ (see Fig. 3b) indicates that SANSS TiO₂ particles with deep UV absorbance characteristic can dissociate Toluene more efficiently than Degussa-P25 TiO₂ particles.

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