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**Output จากโครงการวิจัยที่ได้รับทุนจาก สกว.**

1) International Symposium on Functional Nanomaterials 2007 (ID : Conf -248)

"Phase Transformation of Nano-Grain Size Titanium Dioxide Films"

2) 6th Asian Meeting on Electroceramics 2008 (ID : P-A-59)

"The influence of calcination method on optical properties and nanostructure of sol-gel Ag-$TiO_2$ films"

Paper ID : Conf-248

# PHASE TRANSFORMATION OF NANO-GRAIN SIZE TITANIUM DIOXIDE FILMS

Samart Kongtaweelert, Usa Sukkha

*Department of Chemistry, Faculty of Science, King Mongkut's Institute of Technology Ladkrabang, Bangkok 10520 THAILAND

**Abstract:**

In this project, nano-grain titanium dioxide thin films were prepared by sol-gel spin coating technique. The precursor solution was prepared by dissolved titanium(IV)isopropoxide in mixed solution, 2-propanol, glacial acetic acid and acetylacetone, then the precursor solution were dropped on glass substrate. The substrate was spin with 7000 rpm for 2 s in each layer. The calcining behaviors of xero-gel were analyzed by thermal gravimetric analyzer (TGA) and differential thermal analyzer (DTA). From the result of TGA and DTA were shown thermal degradation at 240 °C and 317 °C that corresponding to previously work. Therefore, the precursor films were calcined at 400, 500 and 600 °C. The crystallization of calcined films was characterized by X-ray diffraction technique. The surface roughness of calcined films was observed by atomic force microscope (AFM). The transmittances of calcined films were study by ultraviolet-visible (UV) spectrophotometer. From the XRD result, the $TiO_2$ anatase structure was found at 500 °C and phase transformation, anatase to rutile, at 600 °C. The AFM results were found the highly homogeneous films with the average grain size about 50 and 150 nm for 500 to 600 °C films respectively. From UV spectrophotometer result was found the band gap energy in the range of 3.96-4.20 eV.

**KEY WORDS**: Titanium oxide ($TiO_2$), sol-gel, thin film, nano-grain.

## 1. Introduction

Titanium dioxides has gained extensive interest due to their unique physical and chemical properties such as large energy gap, high refractive index and transparency in the visible regions. The $TiO_2$ thin films have potential applications, such as gas sensor[1-4], bactericide[5, 6], self-cleaning glasses[7], photocatalysts[8-11], etc. Its application is extremely promoted by the preparation technology. Therefore, the preparation of nano-$TiO_2$ film is ceaselessly researched to obtain the product with high quality at low coast. Usually, $TiO_2$ thin films can be prepared by different techniques such as micro-arc oxidation[12], thermal plasma chemical vapor deposition (TPCVD)[13], pulse laser deposition (PLD)[14], ion beam-induced chemical vapour (IBICVD)[15] etc. These method have advantage in having control over film growth, neverthless, these techniques are complicate, energy-intensive and high temperature. Alternatively, the sol-gel spin-coating process is one of the most appropriate technologies for preparation of $TiO_2$ film due to simplicity, excellent compositional control, high homogeneity, lower crystallization temperature and low cost for raw material. However, the sol-gel spin-coating process, organo metallic precursors in the organic solvents are used, it is necessary to control the viscosity of precuesor solution and spin speed.

In this work, we report simple, less expensive and efficient method for the preparation of $TiO_2$ thin films. The characteristics and optical properties of $TiO_2$ thin films are presented and discussed.

## 2. Experiment procedure

### *2.1 Preparation of $TiO_2$ precusor solution*

Titanium(IV)isopropoxide, 2-propanol, glacial acetic acid and acetylacetone were used as starting materials. The 0.15 M of precursor solution were prepared by dissolved Titanium(IV)isopropoxide in mixed solution of 2-propanol, glacial acetic acid and acetylacetone, then stirred continuously for 30 min at 50 °C . The precursor solution was used as starting solution for thin films preparation.

### *2.2 Preparetion of $TiO_2$ films*

The cover glass (22×22×0.13 mm) were used as substrates. Before the deposition, the substrates were ultrasonically cleaned in cleaning solution for 15 min, water, a mixture of 0.5M HCl solution and 0.5M $HNO_3$ in a molar ratio was 1:1 for 20 min and then were thoroughly rined with deionized water and acetone, respectively. Finally they were dried at 95 °C for 30 min. The $TiO_2$ thin film were deposited on substrates by spin-coating technique at room temperature. The precursor solution were droped on glass substrates, which were rotated with 7000 rpm for 25 s then the as-coated films were dried at 120 °C for 3 min. The film thickness can be controlled by repetition of the cycle from spinning to drying for 5, 10 and 15 time respectively. Finally, the green-film were calcined at 400, 500 and 600 °C for 2 h.

The xero-gel obtained by drying precursor at 100 °C for 24 h, then the xero-gel were calcined in the same temperature program as films.

### *2.3 Characterization*

The thermal behavior of xero-gel were monitored by thermal gravimetric analyzer and differential thermal analyzer (TG-DTA) (Pyris 1 TGA, Perkin-Elmer). The crystallinity of calcined $TiO_2$ films and xero-gel were identified by X-ray diffraction (XRD) with a diffractometer (type 08 Advance, Bruker AG). The surface roughness of calcined films was observed by atomic force microscopy (AFM). Spectroscopic analysis of calcined films were performed by using a uv-vis spectrophotometer (JASCO 7800, Shimadzu JASCO 7800).

## 3. Results and discussion

### *3.1 Transformation of xero-gel into crystalline form*

The TG-DTA curves of xero-gel is shown in Fig.1. The thermo-gram of TGA-DTA in each sample are shows two stages of weight loss with endothermic peak at 240 and 317 °C respectively. The first one at 240 °C is the minor loss by 5% w/w which is due to the loss of absorbed water in the $TiO_2$ gel and 22% w/w at 317 °C of the second one is due to the decomposition of organic compounds contained in the xero-gel. There is no further weight loss can be seen in range 400-700 °C

From the XRD result, it is shown that the phase transformation of calcined powders were dependent on there's calcining temperature as show in the Fig.2. The XRD pattern of 400 °C powders was not show any crystalline phase. The phase

transformation form amorphous to polycrystalline was found while calcining at higher temperature from 400 °C to 500 °C and show highly percent crystallinity from 500 °C to 600 °C . The diffraction pattern of $TiO_2$ in anatase phase was found in 500 °C powders and more strongly peak in 600 °C powders. It was corresponding to previous study that because of the crystalline size were increasing while increasing calcining temperature.

*3.2 Surface morphology*

Corresponding to XRD patterns, the AFM micrographs of 400 °C films with 5, 10 and 15 layers was show the amorphous surface film and it's became to be more crystalline surface films after calcined at 500 °C to 600 °C of the 15 layers films as show in Fig.3-5. The average grain size of films are increasing from 50 to 150 nm with calcined of 500 to 600 °C respectively that because the result of thermodynamic rearrangement at the grain boundary. The calcining temperature was affected directly to the surface energy at the grains. This energy will be increasing the grain growth rate that related to Arrhenious equation

$$k = Ae^{-E}a/RT$$

when k is the rate constant of grain growth , A is the Arrhenious constant, $E_a$ is the activation energy, R is gas constant and T is temperature.

*3.3 Optical properties of $TiO_2$ films*

The UV-Vis spectra of $TiO_2$ thin films, in the wavelength range 200-400 nm was show in Fig.6-8. From the spectral, it's observed that the $TiO_2$ thin films are transparent in visible region, transmittance values decreases with increasing cycle times (Table 1), and the absorption edge in UV region at a wavelength range 290-315 nm

The band gap energy ($E_g$) is calculated by the formula:

$$E_g = \frac{hc}{\lambda} = \frac{1243.1}{\lambda}. \quad (1)$$

Where $E_g$ is band gap energy (eV) of the sample, h is the Planck's constant, c is the speed of light and $\lambda$ is the cut-off wavelength of the spectrum (nm) [12]. The band gap energy values determined for all studied samples after calcining at 400-600 °C have been summarized in Table 1. From Table 1, it can be clearly observed that for all samples the band gap energy are a blue shift which indicates that the particles which make up the film are very fine in size of about a few nanometers.

## 4. Conclusions

The $TiO_2$ amorphous structure was found at 400 °C and phase transformation, amorphous to anatase, at 500 °C. The $TiO_2$ thin films are transparency in the visible region while its have the absorbance edge in the 290-315 nm wavelength range and bandgap energy is 3.96-4.20 eV. We obtained nano-$TiO_2$ thin films with a highest homogeneous films and average grain size about 50 nm by the sol-gel spin-coating technique of 15 layers at 500 °C for 2 hours.

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**Caption of Table**

Table 1 Summary of bandgap energy values calculated and transmittance values for all studied $TiO_2$ thin films.

| Calcining temperature (°C) | coating cycles | % transmittance | Bandgap energy (eV) |
|---|---|---|---|
| 400 | 5 | 78 | 4.13 |
| | 10 | 71 | 4.14 |
| | 15 | 61 | 4.14 |
| 500 | 5 | 80 | 4.13 |
| | 10 | 77 | 3.96 |
| | 15 | 65 | 4.18 |
| 600 | 5 | 75 | 4.13 |
| | 10 | 67 | 4.05 |
| | 15 | 65 | 4.20 |

## Caption of Figures

Fig. 1 TG/DTA curves of xerogel

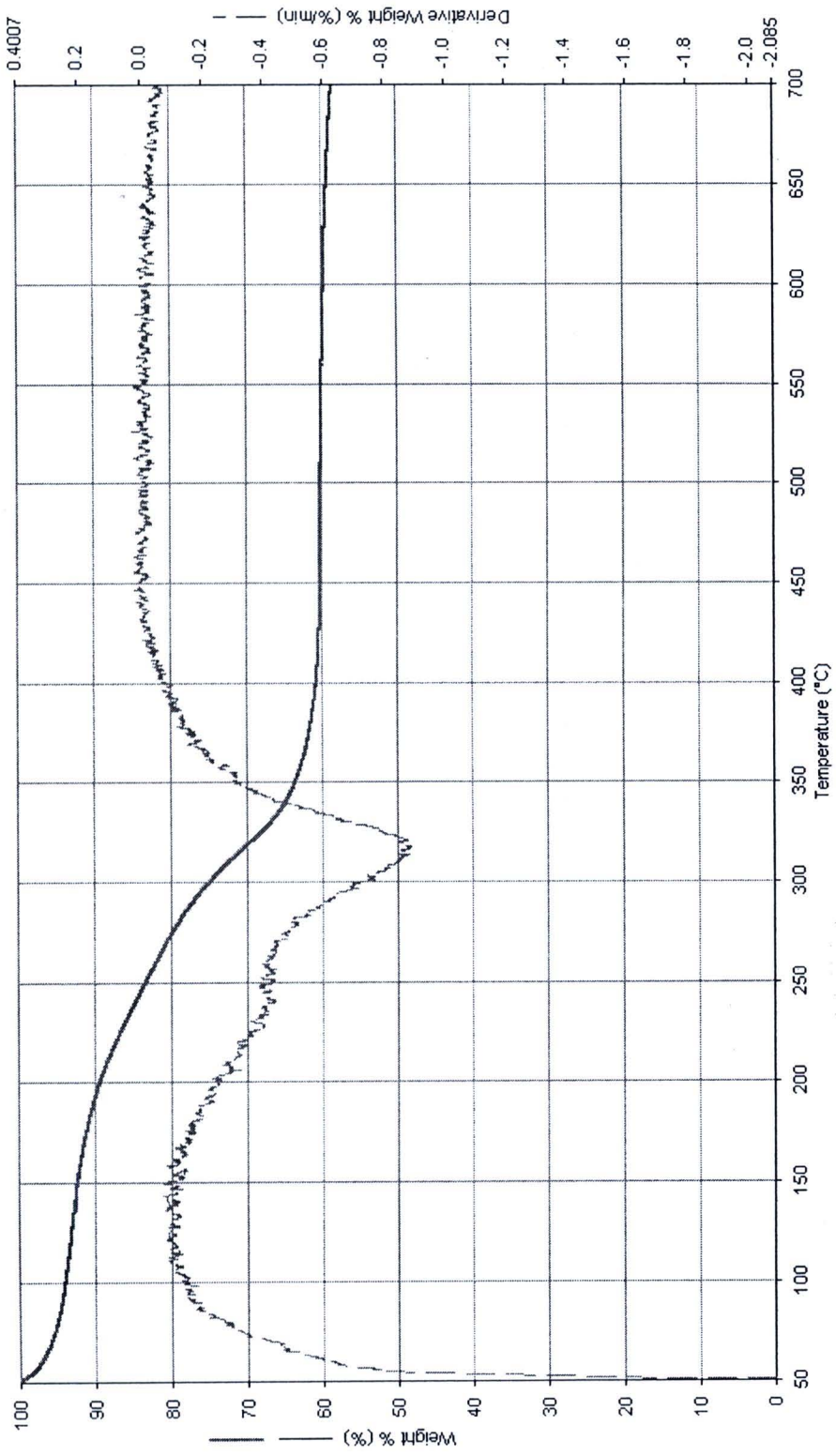


Fig. 2 XRD patterns of xerogel calcined at different temperatures for 2 h.

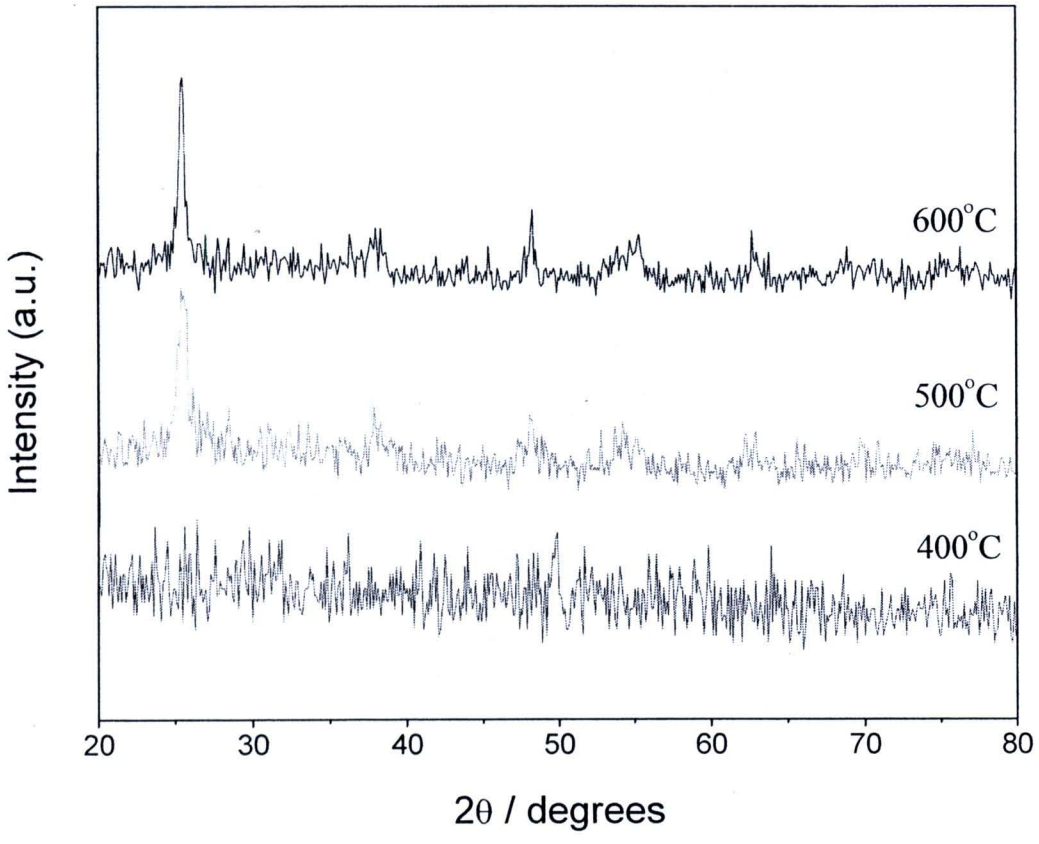


Fig. 3 AFM micrographs of $TiO_2$ thin film prepared by repeating 15 coating cycles and calcined at 400 °C for 2 h.

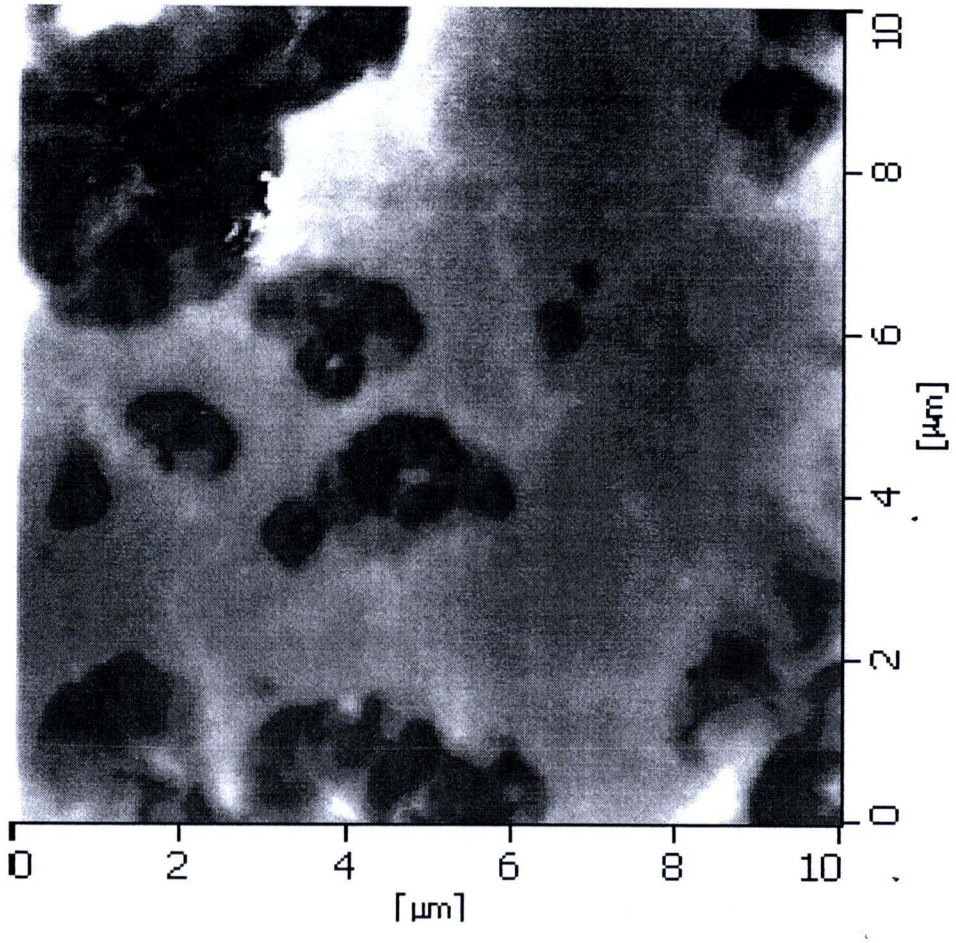


Fig. 4 AFM micrographs of $TiO_2$ thin film prepared by repeating 15 coating cycles and calcined at 500 °C for 2 h.

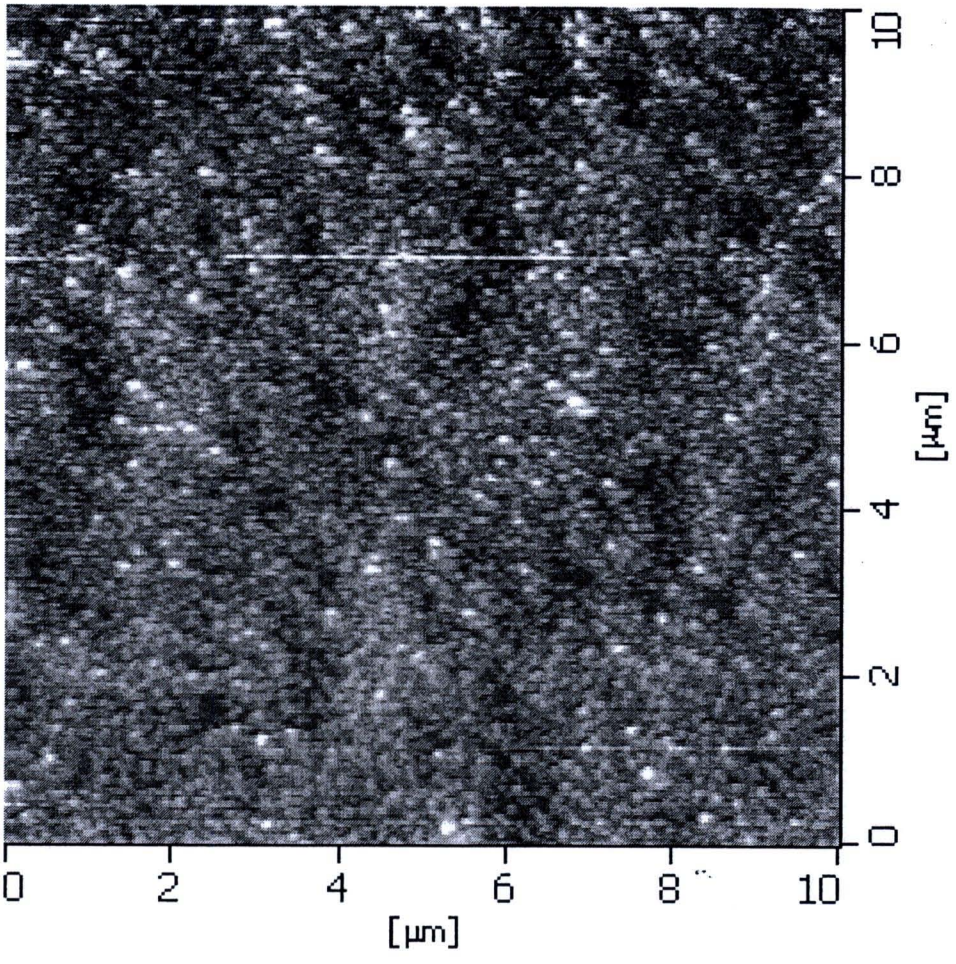


Fig. 5 AFM micrographs of $TiO_2$ thin film prepared by repeating 15 coating cycles and calcined at 600 °C for 2 h.

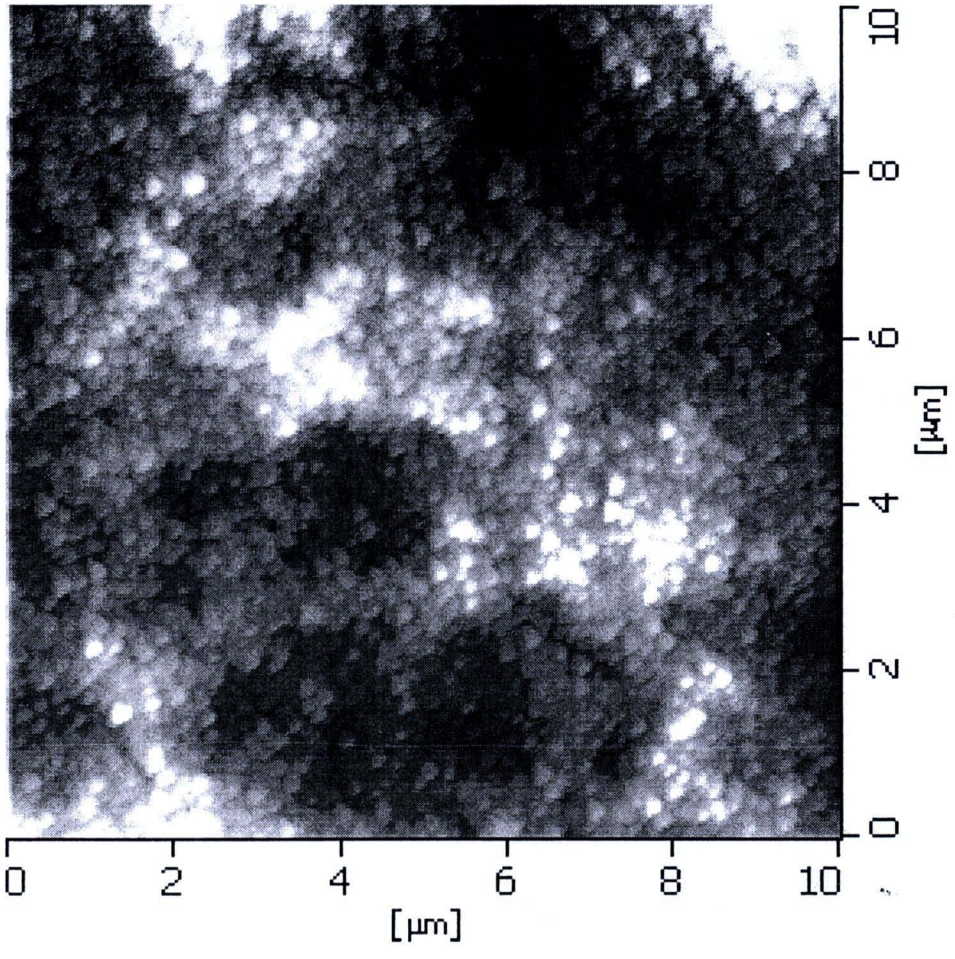


Fig. 6 Optical transmittance spectra of $TiO_2$ thin films prepared by repeating (a) 5, (b) 10 and (c) 15 coating cycles and calcined at 400 °C for 2 h.

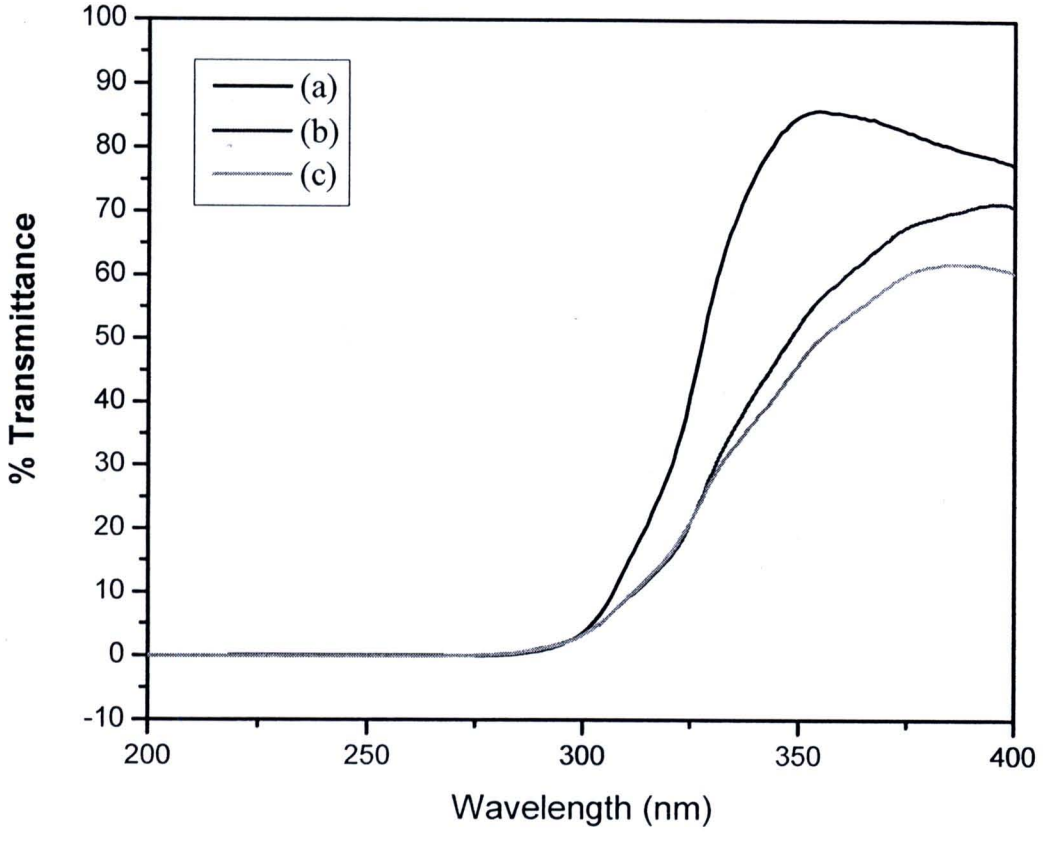


Fig. 7 Optical transmittance spectra of $TiO_2$ thin films prepared by repeating (a) 5, (b) 10 and (c) 15 coating cycles and calcined at 500 °C for 2 h.

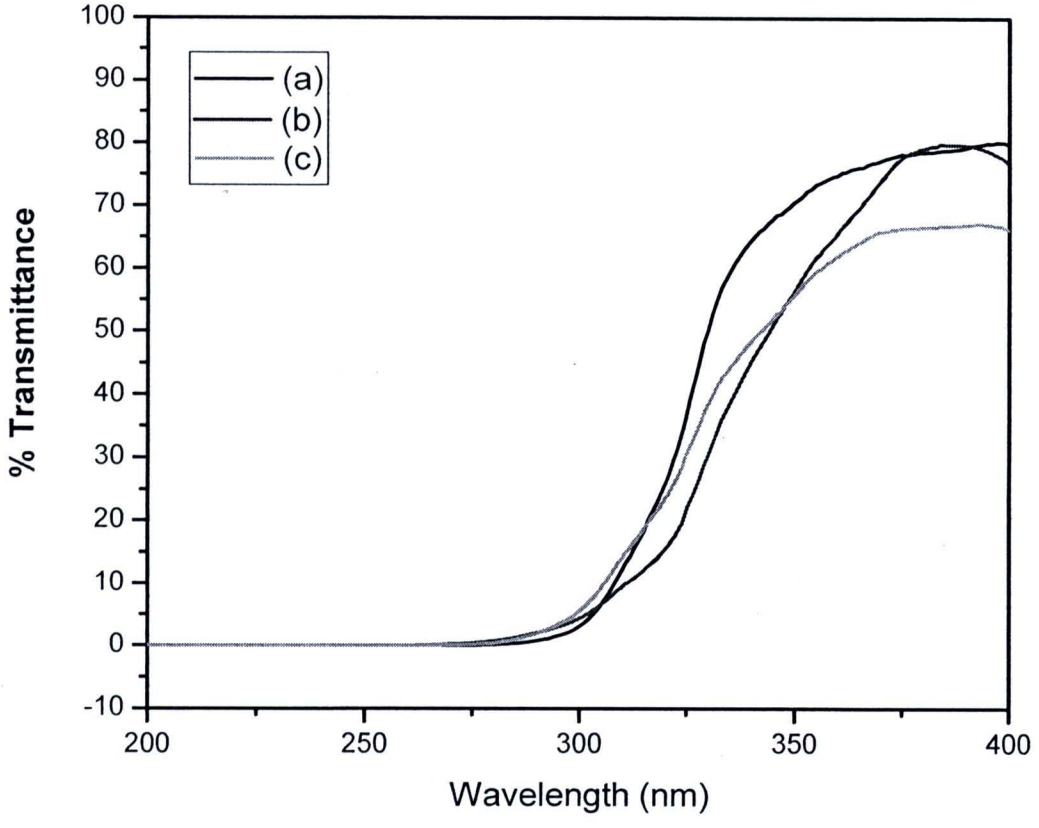
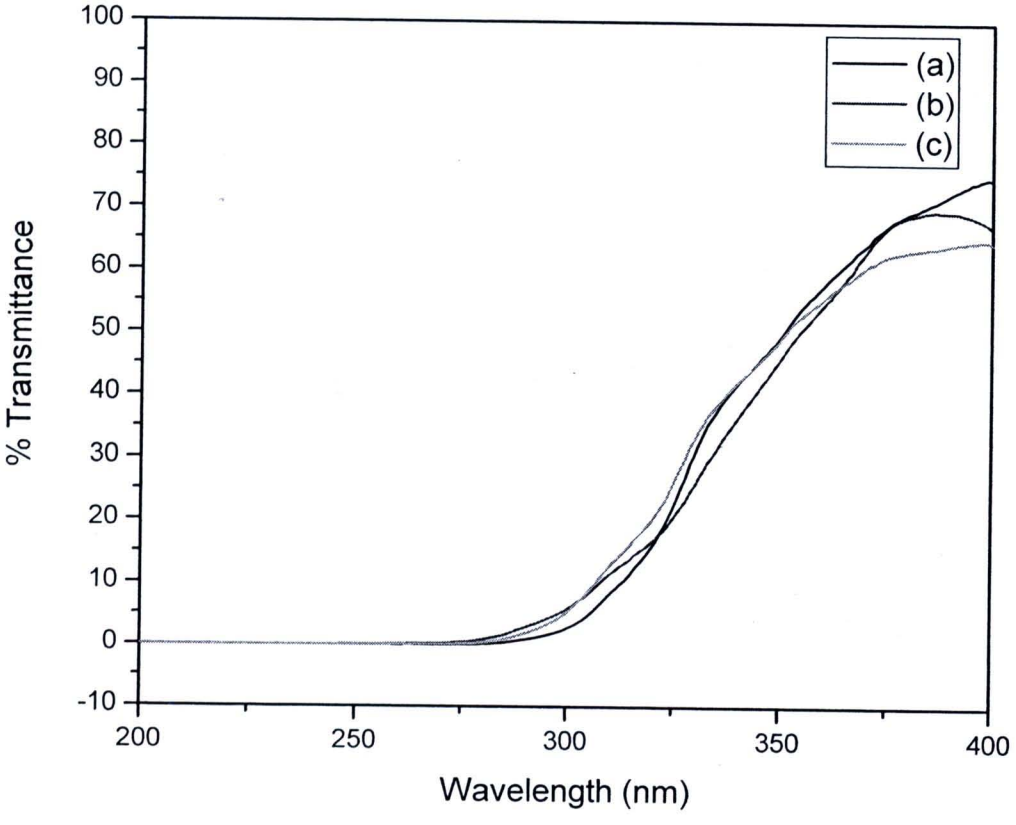


Fig. 8 Optical transmittance spectra of $TiO_2$ thin films prepared by repeating (a) 5, (b) 10 and (c) 15 coating cycles and calcined at 600 °C for 2 h.



# The Influence of Calcination Method on Optical Properties and Nanostructure of Sol-gel Ag-$TiO_2$ Films

(Department of Chemistry, Faculty of Science, King Mongkut's Institute of Technology Ladkrabang, Bangkok 10520 THAILAND) *Usa Sukkha, Sakda Trisak, Samart Kongtaweelert
E-mail:sa_kab@yahoo.com

**Abstract**

The nano-grain of silver-doped $TiO_2$ films were prepared by sol-gel spin coating technique. The titanium(IV)isopropoxide, silver nitrate, 2-propanol, glacial acetic acid and acetylacetone were used as starting material. The thermal behaviors of silver-doped $TiO_2$ xero-gel were analyzed by thermal gravimetric analyzer (TGA) and differential thermal analyzer (DTA). From the result of TGA and DTA were shown thermal degradation at around 120°C and 250°C. The green films of silver-doped $TiO_2$ were calcined by conventional and 2-step method. The crystallization of calcined films was characterized by X-ray diffraction (XRD) technique. The surface morphology of calcined films was investigrated by scanning electron microscope (SEM). The transmittances of calcined films were observed by ultraviolet-visible (UV-vis) spectrophotometer. From the XRD result, the $TiO_2$ anatase structure was found at 300 °C and phase transformation, anatase to rutile, at 550 °C. From the SEM results, grain of 2-step method films are found to be smaller and denser than conventional method films. Whereas Ag particles at grain boundaries of 2-step films are smaller than in conventional method films. From UV spectrophotometer result, the band gap energy of Ag-$TiO_2$ are in the range of 3.6-3.85 eV.

**Keywords:** Ag-$TiO_2$ film, 2-step calcinations, sol-gel, nano-grain

**References**

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## *Wednesday Afternoon, October 22*

***Room: P  Poster session A  13:50-14:40***

P-A-01 Dielectric Properties of 0-3 Lead Magnesium Niobate Titanate (PMNT)-Ordinary Portland Cement (PC) Composite

*A.Chaipanich, N.Jaitanong (*Chiang Mai University, Thailand)

P-A-02 Ferroelectric and Electrical Properties of Bismuth titanate Ceramics with Nd/V co-doping

*Jin Soo Kim, Jung Hyun Jeong (*Pukyong Nat'l Universty, Korea)

P-A-03 Dielectric and Impedance Spectroscopy of lead-free $(Na_{0.5}K_{0.5})NbO_3$-$LiNbO_3$ Ferroelectric Ceramics

*Jin Soo Kim, Jung Hyun Jeong (*Pukyong Nat'l Universty, Korea)

P-A-04 Poling Effect on the Piezoelectric Properties of BNT-based Solid Solutions

*Yuji Hiruma, Hajime Nagata, Tadashi Takenaka (*Tokyo University of Science, Japan)

P-A-05 Piezoelectric Properties of Bismuth Layered-Structure Ferroelectric $Bi_2WO_6$ Mono-Domain Crystals

*Hiroaki Takeda, Masaya Nishida, Yoshinori Sakaguchi, Takashi Nishida, Kiyoshi Uchiyama, Tadashi Shiosaki (*Nara Institute of Science and Technology, Japan)

P-A-06 Phase Diagram of Barium Titanate - Pottasium Niobate System Ceramics and Their Piezoelectric Property

*Satoshi Wada, Momoyo Nitta, Nobuhiro Kumada, Daisuke Tanaka, Chikako Moriyoshi, Yoshihiro Kuroiwa (*University of Yamanashi, Japan)

P-A-07 Effect of Particle size on the Dielectric Properties of Lead Magnesium Niobate Titanate-Portland Cement Composites.

*N. Jaitanong, A. Chaipanich, R.Rienyoy, L.jariensuk, R.Potong (*Chiang Mai University, Thailand)

P-A-08 Dielectric relaxations in multiferroic $BiMn_2O_5$ ceramics

*Y. J. Wu, Y. Q. Lin, X. M. Chen (*Zhejiang University, China)

P-A-09 Sintering Behavior of Silver Particles in Electrode for Multilayer Ceramic Substrate

*Atsuyuki Okada, Takashi Ogihara (*Daiken Chemical Co.,Ltd, Japan)

P-A-10 .A study of capacitance aging of Ni-MLCC under DC electrical field.

*Shigeki Sato (*TDK Corporation, Japan)

P-A-11 Sintering of $Ba_{1-X}Sr_XTiO_3$.powders prepared using KOH-$KNO_3$ molten salt method

*Tomohiko Kishiki, Masashi Higuchi, Takashi Asaka, Keiichi Katayama (*Tokai University, Japan)

P-A-12 The Influence of $BaTiO_3$ Tetragonality by Solid State Synthesis on MLCC's Dielectric Characteristics

*Masayuki Hogiri, Hiroshi Kagata (*Panasonic Electronic Devices Co., Ltd., Japan)

P-A-13 Enhanced Giant Dielectric Response in $Ba[(Fe,Al)_{0.5}Ta_{0.5}]O_3$ Ceramics

*Z. Wang, X. M. Chen, X. Q. Liu (*Zhejiang University, China)

P-A-14 Giant Dielectric Response in (Li, V)-doped NiO Ceramics Prepared by A Polymer-pyrolysis Method

*S. Pongha, P. Thongbai, S. Maensiri, T. Yamwong (*Khon Kaen University, Thailand)

P-A-15 Effects of Rare Earth Substitution (R=Sm,Eu,Dy,Er) on Dielectric Properties of $Bi_2O_3$-ZnO-$Nb_2O_5$ Pyrochlore Ceramics

*Tongqing Yang, Qinghua Xu, Xi Yao (*Tongji University, China)

P-A-16 Crystallographic aspects of microwave dielectric properties.

*Hitoshi Ohsato Isao Kagomiya, Ken-ichi Kakimoto, Eiichi Koga

(*Nagoya Institute of Technology, Japan)

P-A-17 Influence of Structural Characteristics on Dielectric Properties of Mg-based Ceramics at Microwave Frequencies

*Eung Soo Kim, Seock No Seo, Hyeong Joon Kim (*Kyonggi University, Korea)

P-A-18 Evolution of Tin Films in Oxidative Atmosphere

*Shunichi Hishita, Hajime Haneda (*National Institute for Materials Science, Japan)

P-A-19 Characteristics of $La_2O_3$ and $Al_2O_3$ Films as Charge Trapping Layer in SONOS-type Flash Memory Device

*Doo Jin Choi, Hyo June Kim, Seoung Yong Cha (*Yonsei University, Korea)

P-A-20 Fabrication of hydrogen sensors using Pd-polycrystalline 3C-SiC Schottky diode for harsh environments

*Gwiy-sang Chung, Jeong-hak Ahn (*University of Ulsan, Korea)

P-A-21 Bottom-Up Fabrication of High-k Dielectric Nanofilms using Oxide Nanosheets as a Building Block

*Minoru Osada, Takayoshi Sasaki (*National Institute for Materials Science, Japan)

P-A-22 Preparation of N-doped ZnO films by MOCVD

*K. Kobayashi, H. Kobayashi, Y. Tomita, Y. Maeda, Y. Khono (*Shizuoka University, Japan)

P-A-23 Composition Dependence of Electric Properties of $(Ba_{1-x},Sr_x)TiO_3$ Thin Films on Glazed Alumina Substrate

*Takashi Nozaka, Yoji Mizutani, Bhakdisongkhram Gun, Yuta Kawakami, Masahiro Echizen, Takashi Nishida, Hiroaki Takeda, Kiyoshi Uchiyama, Tadashi Shiosaki

(*Yokohama Denshi Seiko Co., Ltd., Japan)

P-A-24 Polarity of ZnO films grown by pulsed laser deposition

*Yutaka Adachi, Naoki Ohashi, Takeshi Ohgaki, Isao Sakaguchi, Hajime Haneda, Hideki Yoshikawa, Shigenori Ueda, Keisuke Kobayashi, Tsuyoshi Ohnishi, Mikk Lippmaa

(*National Institute for Materials Science, Japan)

P-A-25 Dielectric Properties of Lead Titanate-Dispersed Zinc Oxide Ceramic Nanocomposites

*Rewadee Wongmaneerunga, Supon Anantab, Rattikorn Yimnirunb (*Maejo University, Thailand)

P-A-26 Synthesis and Characterization of Samarium doped Ceria ($Ce_{0.8}Sm_{0.2}O_{1.9}$) Nano-Particle by Aerosol Plasma Pyrolysis

*Kenichi MYOUJIN, Araki TORIYAMA, Takashi OGIHARA (*University of Fukui, Japan)

P-A-27 Ion Exchange of Layer Structured Crystal $CsTiNbO_5$ and Its Application as Cathode Material in a Rechargeable Lithium Battery

*Masao Ohashi (*Tokuyama College of Technology, Japan)

P-A-28 Electrocatalytic Activities of Trirutile-type Oxides $MTa_2O_6$ (M=Co,Ni,Mg) toward Oxygen Reduction Reaction

*Takayuki Konishi, Hideki Kawai, Morihiro Saito, Jun Kuwano, Hidenobu Shiroishi

(*Tokyo University of Science, Japan)

P-A-29 Improvement of UV Luminescence Properties of Gallium Nitride Powder by Hydrogen Radical Irradiation
*Takamasa Ishigaki, Norio Kobayashi, Rubin. Ye, Takayuki Watanabe
(*National Institute for Materials Science, Japan)

P-A-30 New titanium Oxide Compound for Photocatalytic Water Splitting
*S. Nakajima, S. Watanabe, K. Uematsu, T. Ishigaki, K. Toda, M. Sato
(*Niigata University, Japan)

P-A-31 Electrical Properties of Pt/Nb-doped $SrTiO_3$ Schottky Junctions
*Jianyong Li, Takeshi Ogaki, Ryota Matsuoka, Hideyo Okushi, Naoki Ohashi
(*National Institute for Materials Science, Japan)

P-A-32 Electrical properties of Conductive Paste with Silver Nanoparticles and Its Application to Flexible Substrate
*Yukio Yamamoto, Takashi Ogihara (*Daiken Chemical Co. Ltd, Japan)

P-A-33 Application of Nanoporous Silicon for Metal-Semiconductor-Metal Visible Light Photodetector
*Narin Atiwongsangthong, Surasak Niemcharoen, Wisut Titiroongruang
(*King Mongkut's Institute of Technology, Thailand)

P-A-34 Magneto-Optical Properties of Mn-Substituted Titania Nanosheets
*X.P. Dong, M. Osada, Y. Ebina, Y. Kotani, K. Ono, S. Ueda, K. Kobayashi, K. Takada, T. Sasaki
(*National Institute for Materials Science, Japan)

P-A-35 Synthesis of Nanosphere Phosphor Particles Using an Ion-exchange-polymer/Water/Oil Emulsion Method
*Hiroyuki Sano, Seiichirou Harada, Mikio Shimizu, Makoto Kuwabara (*Kyushu University, Japan)

P-A-36 Fabrication of polycrystalline 3C-SiC micro resonator and its characteristics
*Tae-won Lee, Gwiy-sang Chung (*University of Ulsan, Korea)

P-A-37 Fabrication of the ultra low power consumption microhotplates using a poly 3C-SiC thin membrane
*Jae-min Jeong, Gwiy-sang Chung (*University of Ulsan, Korea)

P-A-38 Fabrication of Barium Titanate/Silver Composites from a Particle Preparation Stage.
*T.Kojima, M.Sugihara, N.Uekawa, K.Kakegawa (*Chiba University, Japan)

P-A-39 "Growth of (100) and (111) platinum thin films on single-crystal $SrTiO_3$ by pulsed laser deposition
*Isao Sakaguchi, Yutaka Adachi, Tsubasa Nakagawa, Naoki Ohashi, Kazunori Takada
(*National Institute for Materials Science, Japan)

P-A-40 Diffusion of oxygen in Mg-doped ZnO ceramics
*I. Sakaguchi, T. Nakagawa, K. Matsumoto, Yutaka Adachi, Naoki Ohashi, Hajime Haneda
(*National Institute for Materials Science, Japan)

P-A-41 Redistribution of unintentional defects induced by heavy ion implantation in ZnO ceramics
*I. Sakaguchi, T. Nakagawa, K. Matsumoto, S. Hishita, Y. Adachi, N. Ohashi, H. Haneda
(*National Institute for Materials Science, Japan)

P-A-42 Blocking of oxygen diffusion by grain boundary in $BaTiO_3$ ceramics

*Tsubasa Nakagawa, Kenji Matsumoto, Isao Sakaguchi, Yutaka Adachi, Naoki Ohashi, Hajime Haneda (*National Institute for Materials Science, Japan)

P-A-43 Low Temperature Synthesis of $TiO_2$ from Acid Solution

*S. Watanabe, S. Nakajima, K. Uematsu, T. Ishigaki, K. Toda, M. Sato (*Niigata University, Japan)

P-A-44 Effect of. Vibro-Milling Time on Phase Formation and Particle Size of $ZnNb_2O_6$ Nano- powders

*A.Ngamjarurojana, R. Yimnirun, S. Ananta (*Chiang Mai University, Thailand)

P-A-45 Ferroelectric Properties of $Pb(Zr_{1/2}Ti_{1/2})O_3$-$Pb(Zn_{1/3}Nb_{2/3})O_3$ Ceramics under compressive stress applied perpendicular to electric field.

*M. Unruan, A. Prasatkhetragarn, A. Ngamjarurojana, Y. Laosiritaworn, S. Ananta, R. Yimnirun (*Chiang Mai University, Thailand)

P-A-46 XPS Characterization of Relaxor Perovskite PNN Ceramics Prepared By Corundum Precursor Method

*L. Srisombat, O. Khamman, R. Yimnirun, S. Ananta, T.R. Lee (*Chiang Mai University, Thailand)

P-A-47 Characteristic of Diamond Film Synthesized by Using CACVD Technique at High Temperature

*Y. Wongprasert, S. B. Pongsai (*King Mongkut's Institute of Technology, Thailand)

P-A-48 Investigation of Oxygen Vacancies in Micro-patterned PZT Thin Films using Raman spectroscop

*Ken Nishida, Takashi Yamamoto, Hironari Takeuchi, Takashi Katoda, Minoru Osada, Shintaro Yokoyama, Takafumi Kamo, Takashi Fujisawa, Hiroshi Funakubo
(*National Defense Academy, Japan)

P-A-49 Microstructural and Polarization Study of the $(1-x)Bi(Mg_{1/2}Zr_{1/2})O_3$-$xPbTiO_3$ Piezoelectric Ceramics

*G. Shabbir, S. Khalid (*Pakistan Institute of Nuclear Science & Technology , Pakistan)

P-A-50 Fabrication and characterization of organic materials based gas sensor

*M. Atique, G. Shabbir (**Pakistan Institute of Nuclear Science & Technology , Pakistan)

P-A-51 $Sr_xFe_yO_{z-\delta}$ Synthesis, Structure and Thin Film Gas Sensing Properties

*Xiaomei Du, Michael Post, James Tunney (*National Research Council of Canada, Canada)

P-A-52 Heat Transfer Model during Laser-Assisted Cutting Of Mullite.

*C. Y. Ho, C. Ma, M. Y. Wen (*Hwa Hsia Institute of Technology, Taiwan)

P-A-53 Investigation into Laser Drilling of Alumina CeramicsUsing Three-Dimensional Model

*C. Y. Ho, C. Ma, M. Y. Wen (*Hwa Hsia Institute of Technology, Taiwan)

P-A-54 Atomic diffusion mechanism in.a-$Al_2O_3$ by fist principles calculation and SIMS

*Nobuaki Takahashi, Teruyasu Mizoguchi, Tsubasa Nakagawa, Isao Sakaguchi, Takahisa Yamamoto, Yuichi Ikuhara (*The University of Tokyo, Japan)

P-A-55 Photo-assisted VOC Detection by Transparent pn Junction

*Yoshinobu Nakamura, Yusuke Morita, Yasuhiro Yoshida, Satoru Fujitsu
(*The University of Tokyo, Japan)

P-A-56 "Dielectric properties and structure of Ni-doped (0.8-x)PMT-0.2PZ-xPT ternary system near morphotropic phase boundary"

*Hua Hao, Hanxing Liu, Cao Minghe, Yu Zhiyong, Ouyang Shixi
(*Wuhan University of Technology, China)

P-A-57 Properties of Barium Stannate Titanate with Boron Oxide Addition.

*Gobwute Rujijanagul, Nattaya Tawichai, Sukum Eitssayeam, Kamonpan Pengpat, Uraiwan Intatha (*Chiang Mai University, Thailand)

P-A-58 Microstructure and Microwave Dielectric Properties of $Ca[(Mg_{1/3}Ta_{2/3})_{1-x}Tix]O_3$ Complex Perovskite Ceramics.

*M. S. Fu, X. M. Chen, X. Q. Liu (*Zhejiang University, China)

P-A-59 The Influence of Calcination Method on Optical Properties and Nanostructure of Sol-gel Ag-$TiO_2$ Films.

*Usa Sukkha, Sakda Trisak, Samart Kongtaweelert

(*King Mongkut's Institute of Technology, Thailand)

P-A-60 Preparation and characterization of Indium Tin Oxide by sol-gel coprecipitate method

*Patchraporn It-thivisit, Samart Kongtaweelert (*King Mongkut's Institute of Technology, Thailand)

P-A-61 Structural, Dielectric, Ferroelectric and Piezoelectric Characterization of Nickel Oxide (NiO) Modified PZT Ceramics

*Subhash B. Deshpande (*National Chemical Laboratory, India)

P-A-62 Synthesis and luminescence properties of non-doped long persistent phosphors : Mg2SnO4

Akiko Torisaka, Hiroaki Kaneko, Kentaro Nomizu, Sho Abe, *Tadashi Ishigaki, Kenji Toda, Kazuyoshi Uematsu, Mineo Sato (*Niigata University, Japan)