

# ICAMMP2011

## 第二届材料与制造工艺研究进展 国际学术会议

2011 International Conference on Advanced in Materials and  
Manufacturing Processes

(ICAMMP 2011)

December 16-18, 2011 Guilin, China

Co-organized by:

University of Wollongong, Australia

Northeastern University, China

University of Science and Technology Beijing, China

Hubei United University, China

Hong Kong Industrial Technology Research Centre

# CONFERENCE MAP GUIDE

## Guilin, China



## Guilin Da Zheng Hot Spring Holiday Hotel



ICAMMP 2011 SECRETARIAT

Any question, please contact the secretariat: +86 13826049112

**The Second International Conference on  
Advanced in Materials and Manufacturing Processes  
(ICAMMP 2011)**

**Co-organized By:**

University of Wollongong, AU  
Northeastern University, China  
University of Science and Technology Beijing  
Hebei United University, China  
Hong Kong Industrial Technology Research Centre

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Prof. Zhengyi Jiang, University of Wollongong, AU  
Prof. Xianghua Liu, Northeastern University, China  
Prof. Jingtao Han, University of Science and Technology Beijing  
Prof. Sihai Jiao, Research Institute, Baosteel, China  
Prof. Jinglong Bu, Hebei United University, China

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 Prof. Xiaoming San, Hebei United University, China  
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 Prof. Walid Mahmoud Shewakh, Beni Suef university, Egypt  
 Prof. Yun-Hae Kim, Korea Maritime University, Korea  
 Prof. Yu-Shiang Wu, China Univerisity of Sci. and Tech., Taiwan  
 Prof. Heinz-Gunter, Brokmeier, Technische Universitat Clausthal, Germany  
 Prof. Zhengyi Jiang, University of Wollongong, Australia  
 Prof. Yun-Hae Kim, Korea Maritime University, Korea  
 Prof. Christian Kloc, Nanyang Technological University, Singapore  
 Prof. Jacques Noudem, CRISMAT laboratory, France  
 Mr. Mohammad Hadi Hafezi, National university of Malaysia, Malaysia

### **Conference Website**

<http://www.icammp.org>

## SCHEDULE OF THE CONFERENCE

### December 16 (Friday)

10:00—21:00

Registration, Sequence Hall, Guilin Da Zheng Hot Spring Holiday Hotel

12:00—13:00

Lunch, Aurantiacus Chinese Restaurant, 1/F (丹桂轩)

18:00—19:30

Dinner, Aurantiacus Chinese Restaurant, 1/F (丹桂轩) ①

### December 17 (Saturday)

8:30—8:40

Open Ceremony, Ballroom, 3/F (大正厅)

8:40—9:55

Keynote Speeches

9:55—10:15

Tea Break

10:15—11:30

Keynote Speeches

12:00—

Lunch, Aurantiacus Chinese Restaurant, 1/F ②

14:00—15:30

Parallel Sessions (A, B, C)

15:30—15:50

Tea Break

15:50—17:20

Parallel Sessions (A, B, C)

18:00—18:40

Dinner, Aurantiacus Chinese Restaurant, 1/F ③

### December 18 (Sunday)

8:30—10:00

Parallel Sessions (A, B, C)

10:00—10:20

Tea Break

10:20—11:50

Parallel Sessions (A, B, C)

12:00—

Lunch, Aurantiacus Chinese Restaurant, 1/F ④

## Morning, December 17, 2011

**Plenary Session (8:30 AM—11:30 AM)**

**Ballroom, 3/F (大正厅)**

### **Chairmen**

**Prof. Zhengyi Jiang, University of Wollongong, AU**

**Prof. Xianghua Liu, Northeastern University, China**

**Prof. Jingtao Han, University of Science and Technology Beijing**

**Prof. Sihai Jiao, Research Institute, Baosteel, China**

**Prof. Jinglong Bu, Hebei United University, China**

8:30—8:40 Opening Speech

8:40—9:05 Plenary

***Prof. Jingtao Han, University of Science and Technology Beijing, China***

9:05—9:30 Plenary

***Prof. Xianghua Liu, Northeastern University, China***

9:30—9:55 Plenary

***Prof. Sihai Jiao, Research Institute, Baosteel, China***

9:55—10:15 Tea Break

10:15—10:40 Plenary

***Prof. Xungai Wang, Deakin University, Australia***

10:40—11:05 Plenary

***Prof. Zhengyi Jiang, University of Wollongong, Australia***

11:05—11:30 Plenary

***Prof. Tsuyoshi FURUSHIMA, Tokyo Metropolitan University, Japan***

12:00— Lunch

## Afternoon, December 17, 2011

14:00 PM—17:20 PM

### Session A: Advanced Materials

1/F VIP Building, Tuoyuan Room (贵宾楼驼园厅)

Chairmen: Prof. Woei-Yun Ho, Mingdao University, Taiwan

Prof. Guang Xu, Wuhan University of Science and Technology, China

(12 minutes for each presentation, including 2-3 minutes of answering questions)

1. Dielectric and Ferroelectric Properties of  $(K_{0.5}Na_{0.5})(Nb_{1-x}V_x)O_3$  Ceramics (N2477)  
*Yanmin Huang, Lijun Liu, Daping Shi, Shaoying Zheng, Shuangshuang Wu*
2. Effect of Envelope Films on Thermal Properties of Vacuum Insulation Panels with Glass Fiber (N2742)  
*Wangping Wu, Zhaofeng Chen, Jieming Zhou and Xueyu Cheng*
3. Decolorization of Coking Wastewater by Mineral Adsorbents (N1844)  
*Kaining Yu, Cheng Wang, Yang Yu, Yan Li*
4. Failure Analysis of the Stuffing Box in a Super High Pressure Compressor (N2185)  
*Xiaohui Li, Yiliang Zhang, Xuedong Xu*
5. Secondary Hardening during Tempering of Cr-W-Mo-V High Alloy Medium-Upper Carbon Steel and its Hardness Forecast (N1327)  
*Yongqing Ma, Hongtao Gao, Yufen Liang, Xiaojing Zhang*
6. Study the Non-Linear Heat-Elasto-Plastic Constitutive Relation (N1524)  
*Xiaojiu Feng, Lifu Liang, Siyuan Wang*
7. Fully Automatic Wafer-Scale Micro/Nano Manipulation Based on Optically Induced Dielectrophoresis (N2176)  
*Yanli Qu, Maijuan Zheng, Wenfeng Liang, Zaili Dong*
8. Fabrication and Thermoelectric Properties of Magneli Phases by Adding Ti into  $TiO_2$  (N2316)  
*Yun Lu, Yusuke Matsuda, Katsuhiro Sgura, Hao Liang, Takahito Otomitsu, Hiroyuki Yoshida*
9. Fabrication of  $TiO_2/Cu$  Composite Photocatalyst Thin Film by 2-Step Mechanical Coating Technique and its Photocatalytic Activity (N2317)  
*Yun Lu, Liang Hao, Keisuke Toh, Hiroyuki Yoshida*
10. Extraction Feature of Gas Fluctuation of Guideway System Based on the Wavelet Transform (N2291)



*Dongju Chen, Jinwei Fan, Feihu Zhang*

11. Low Temperature Fabrication of Platinum/Carbon Black Powder Coating (N3089)  
*Phuri Kalnaowakun*
12. Nonlinear Constitutive Equation and Elastic Constant of Rubber Material (N2043)  
*Li Chen, Z hao Li*
13. Phase-Field Model for Spinodal Decomposition of Dilute Solute Field in Al-Ag Alloy (N1390)  
*Yingjun Gao, Zhirong Luo*
14. The Time Response of Exponential Doping NEA InGaAs Photocathode Applied to near Infrared Streak Cameras (N1961)  
*Weidong Tang, Chuandong Sun, Wenzheng Yang, Zhipeng Cai*
15. Theoretical Energy Distributions of Electrons from a Large Exponential-Doping GaAs Photocathode (N2178)  
*Zhipeng Cai, Weidong Tang, Wenzheng Yang, Xun Hou*
16. The Effect of Bainitic Transformation on Martensite Start Temperature of a Fe-C-Mn-Si Alloy (N1732)  
*Guang Xu, Hang Zou, Conghua Bu*
17. Corrosion Behavior of CrN/AlSiN Multilayer Coatings on AISI 304 Stainless Steel in Aluminum Alloy Melt (N1731)  
*Wei-Yu Ho, Chia-Hang Tsai, Cheng-Hsun Hsu, Woei-Yun Ho*
18. Preparation and Mechanical Properties of a Bulk Icosahedral Quasicrystalline Ti-Zr-Sc-Ni Alloy (N2419)  
*Xinlu Wang, Wanqiang Liu, Shanshan Zhang, Limin Wang*
19. Characterization of LiClO<sub>4</sub>-SiO<sub>2</sub> Composite Electrolyte Prepared by Modified Sol-Gel Method (N1634)  
*Hafizul Mat, Nor Sabirin Mohamed, Ri Hanum Yahaya Subban*
20. Preparation and Characterization of Ag Nanowires by Hydrothermal Method (N2108)  
*Ligang Liu, Zhiqiang Wei, Xiaojuan Wu, Ge Zhang*

## Afternoon, December 17, 2011

14:00 PM—17:20 PM

**Session B: Materials Processing Technology**

**3/F The First Meeting Room (大正 1 号会议室)**

**Chairmen: Prof. Mont Kumpugdee Vollrath, University of Applied Sciences Berlin,  
Germany**

**Prof. Cheng Zhao, Tongji University, China**

(12 minutes for each presentation, including 2-3 minutes of answering questions)

1. A Study on Brazing Process and Mechanical Properties of A Z31 Magnesium Alloy (N1310)  
*Xian Xie, Gaofeng Quan, Xiue Gu, Xingming Liu, Jiale Sun*
2. Ergonomic Analysis of the Master of Minimally Invasive Surgical Robot (N3539)  
*Qingbo Zhang, Chen Zhao, Xiaoming Luo*
3. An Approach to Analyze Pose Accuracy of Master Slave Surgical Robot with Matlab (N3541)  
*Jianye Zhang, Wei Wang, Chen Zhao, Heng Zhang*
4. Effect of Current on the Interfacial Structures of the Dissimilar Metals Diffusion Bonding (N1706)  
*Lifang Hu, Fuming Wang, Shaoping Chen, Qingsen Meng*
5. Research Analysis and Experiment Study on Flow Field of Hydrodynamic Coupling (N3677)  
*Rui Zhang, Chengjian Sun, Yue Wang*
6. Semi-Continuous Extraction of Agarwood Oil with Subcritical Water: Optimization of Plug Flow by Response Surface Methodology (N3595)  
*Aunyarut Apibalsri, Supawan Tantayanont, Somkiat Ngamprasertsith*
7. A New Approach for Colloidal Crystal Fabrication with the Template of Reversible Stress Wrinkle (N3626)  
*Qing Xu, Zhaoting Yang, Chaorong Li, Benyong Chen, Wenjun Dong*
8. Picosecond Laser Microstructuring for Black Silicon Solar Cells (N3303)  
*Xiaoning Zhu, Hongliang Zhu, Dewei Liu, Yongguang Huang, Xiyuan Wang, Haijuan Yu, Shuai Wang, Xuechun Lin, Peide Han*
9. Evaluation of Non-Uniform Deformation of Multi-Layered Ceramic Sheets with Printed Electrodes during Compression (N2703)  
*Fumio Naruse, Naoya Tada*

10. The Learning Styles of Taiwanese Mechanical Engineering Students (N3350)  
*Chin Min Hsiung, Chichang Lin*
11. Preparation of Three-Dimensional Ordered Macroporous Perovskite-Type LaFeO<sub>3</sub> Oxides (N2977)  
*Kun zhao, Fang He, Xinai Li, Zhen Huang, Haibin Li*
12. The Assembly Success Rate and the Estimation of Assembly Time for a Flexible Assembly System (N3566)  
*Jun Wang, Zhijun Yao, Zhi Guo*
13. Classification of Fresh N36 Pineapple Crop using Image Processing Technique (N2887)  
*Shuhairie Mohammad, Kamarul Hawari Ghazali, Nazriyah Che Zan, Siti Sofiah Mohd Radzi, Rohana Abdul Karim*
14. Increasing Solubility of Poorly Water Soluble Drug Resveratrol by Surfactants and Cyclodextrins (N3460)  
*Mont Kumpugdee-Vollrath, Yvonne Ibold*
15. Effect of Ce Addition on the Microstructures and Mechanical Properties of a Ni-Co-Based Superalloy (N1605)  
*Chuanyong Cui, Guoming Han, Xiaofeng Sun*
16. Evaporation Characteristics of Ammonium Formate-Urea-Water Solution Droplet for SCR Systems (N2340)  
*Seung Yeol Lee, Seung Wook Baek*
17. Experimental Observation on the Uniaxial Cyclic Deformation Behaviour of TA16 Titanium Alloy (N1868)  
*Qianhua Kan, Wenyi Yan, Guozheng Kang*
18. Synthesis and Characterization of Siloxane-Modified Polyurethane Dispersion/Clay Nanocomposites (N3175)  
*Heun Hyo Noh, Jong Keun Lee, Xing Liu, Yong Man Choi*
19. A New Interwell Tracer Technology ---Particle Tracer Technology (N1564)  
*Yifei Liu*

## Afternoon, December 17, 2011

**14:00 PM—17:20 PM**

### **Session C: Advanced Manufacturing Technology**

**2/F The Third Meeting Room (大正 3 号会议室)**

**Chairmen: Prof. Abel Cherouat, University of Technology of Troyes, France**

(12 minutes for each presentation, including 2-3 minutes of answering questions)

1. Effects of a High Magnetic Field on the Solidification Behavior of Binary Al-Si and Ag-Cu Systems (N2563)  
*Tie Liu, Qiang Wang, Hongwei Zhang, Guojian Li, Keiji Nakajima, Jicheng He*
2. Key Issues in Microscale Temperature Sensing with Thermocouple Array (N2139)  
*Weiqiang Sun, Haixiao Liu, Shengyong Xu*
3. GaN-based LEDs grown on cone-shaped patterned sapphire substrates with peripheral air voids by lateral etching (N2374)  
*Nan Ming Lin, Shih Chang Shei, Shouu Jinn Chang, Wei Chih Lai, Ya Yu Yang, Wun Cin Lin, Hsin Ming Lo*
4. Finite Element Simulations of the Sinus Lifting Process (N1432)  
*Ching-Chieh Huang, Li-Wen Chen, Nan-Ming Yeh, Wu Dong-Feng, Yung-Chuan Chen*
5. Mechanical Bending Simulation of Thin Stainless Steel by Using Isotropic and Orthotropic Properties (N2461)  
*Somsak Siwadamrongpong, U sanee Kitkamthorn, Chaiyapak Sajjawattana*
6. Realization of a Prototype System for Point Cloud Data Processing in Reverse Engineering (N1141)  
*Yong Zhuo, Juan Peng, Yanjun Wu*
7. Observer Design and Stabilization for a Class of Switched System (N2008)  
*Ming-Yuan Shieh, Juing-Shian Chiou*
8. Phase-Field Numerical Simulation of Pure Free Dendritic Growth Using Wheeler and Karma Model (N2021)  
*Yun Chen, Namin Xiao, Xiuhong Kang, Dianzhong Li*
9. A Comparison of Cover Coat Methods for Electronic Flexible Printed Circuit (E-FPC) Based on Peeling Strength (N2498)  
*Sirisak Rangklang, Paphakorn Pitayachaval, Suradet Tantriratn, Jiraphon Srisertpol*
10. Strain Hardening Analysis Coupled to Remeshing Procedure Application to Sheet Hydroforming Processes (N3426)

*Faouzi Slimani, Abel Cherouat, Mahfoudh Ayadi, Mohamed Ali Rezgui*

11. Modification Method by Increasing and Decreasing Elements in the Emergent Design System (N2403)  
*Akira Kito, Yuki Mizumachi, Mitsuhiro Sekiguchi, Koichiro Sato, Yoshiyuki Matsuoka*
12. Mechanical Properties of Aluminium-Alumina Welding by Friction Welding (N3675)  
*Tummasook Mingmuang, Sukangkana Lee, Chawalit Thinvongpituk*
13. Inhibited and Enhanced Spontaneous Emission Using Silicon-Based on Finite Thickness Photonic Crystal Waveguides (N3601)  
*W. Amorntep, P. Wanchai*
14. Simulation and Experimental Measurement of Ultrasonic Waves Propagation Velocity in Trabecular Bone (N3699)  
*Abderrazek Bennamane, Tarek Boutkedjirt*
15. Optimization of Diamond-Coated Drawing Dies for Stainless Steel Tubes Based on the FEM Simulation (N3543)  
*Zichao Lin, Bin Shen, Fanhong Sun, Zhiming Zhang, Songshou Guo*
16. Investigation and Evaluation of the Safety Properties of Hardwood Athletic Floor System in China (N3581)  
*Yuping Zhang, Limin Peng, Bin Lv, Feng Fu*
17. Wear Resistance Improvement of Titanium Bearings by Laser Gas Nitriding (N3725)  
*Edson Costa Santos, Katsuyuki Kida, Justyna Rozwadowska, Takahashi Honda, Hitonobu Koike, Yuji Kashima, Kenji Kanemasu, Ryo Matsumoto*
18. Epitaxial Growth of Ni-Based Superalloys Using Laser and Spark Deposition (N3728)  
*Edson Costa Santos, Katsuyuki Kida, Justyna Rozwadowska, Masaaki Kidera, Changjun Chen*

## Morning, December 18, 2011

08:30 AM—11:50 AM

Session A: Advanced Materials

1/F VIP Building, Tuoyuan Room (贵宾楼驼园厅)

Chairmen: Prof. Ning Fang, Utah State University, USA

Prof. Sutham Niyomwas, Prince of Songkla University, Thailand

(12 minutes for each presentation, including 2-3 minutes of answering questions)

1. Microstructure Characterization of Al Nanoparticles Prepared by Anodic Arc Plasma (N2814)  
*Zhiqiang Wei, Xiaojuan Wu, Ligang Liu, Ge Zhang*
2. Application of Benzyl Ester of Modified Vegetable Oils as Rubber Processing Oils (N2505)  
*Hasleena Boontawee, Charoen Nakason, Azizon Kaesaman, Anoma Thitithammawong, Sopa Chewchanwuttiwong*
3. The Biocompatibility and Bioactivity of Gradient Composite Bioceramic Coating with Different Content of Nd<sub>2</sub>O<sub>3</sub> Fabricated by Wide Band Laser Cladding (N2118)  
*Ming Xiao, Qibin Liu, Yizhi Zhu*
4. The Influence of the Residual Mg Content in the Ductile Cast Iron on the Formation Law of Spheroidal Graphite (N2117)  
*Zhiqing Shen, Hongliang Zheng, Tongtong Li, Yan Xu, Rongfu Xu, Xuelei Tian*
5. Effect of Casing Design to Microstructure and Mechanical Properties of 3 Mm TWDI Plate (N1695)  
*Johnny Wahyuadi Soedarsono, Bambang Suharno, Rianti Dewi Sulamet-Ariobimo*
6. The Synthesis and Characterization of N-alkyl Dialkyl-Ester Derivatives with Trialkyl Borate (N3181)  
*Yiwen Jin, JunSeong Park, Yong Sung Park, Je-Wan Woo*
7. Influence of Laser Power on the Microstructure and Anti-Corrosion Performance of LHPs NiCrAlY Coatings (N1884)  
*Shuqing Li, Shuili Gong, Yuping Duan, Shunhua Liu*
8. Effect of Casting Velocity on Microstructures of AlMg<sub>0.9</sub>Si<sub>0.7</sub> Alloy by Low-Superheat DC Casting (N2199)  
*Dazhi Zhao, Fuxiao Yu, Fang Liu, Kezhun He*
9. Development of Processing Maps for Coiled Tubing Steel (N2132)  
*Zhendong Zhang, Haitao Zhou, Xianghua Liu, Sijun Li, Jie Dong, Xiao Zhou*
10. Organic Light Emitting Diodes with Metallocene Compounds as Cathode Interfacial Layers (N2308)  
*Keunhee Park, Donghyun Yeo, Seungsik Oh, Hoonbae Kim, Myung Hoon Ha, Hyojin Oh, Minwoo*

*Park, Jisoo Park, Daewon Park, Donggeun Jung, Heeyeop Chae, Hyoungsub Kim, Jin-Hyo Boo*

11. Performance Enhancement of Organic Solar Cells with the LiF/Al Cathode Structure by the Pyromellitic Dianhydride Layer (N2306)  
*Seungsik Oh, Donghyun Yeo, Keunhee Park, Hoonbae Kim, Myung Hoon Ha, Hyojin Oh, Minwoo Park, Jisoo Park, Daewon Park, Donggeun Jung, Heeyeop Chae, Hyoungsub Kim, Jin-Hyo Boo, Eunkyoung Nam*
12. Tensile Properties of Hot Extruded Mg-Zn-Nd-Y-Zr Alloy at Elevated Temperatures (N2133)  
*Xiao Zhou, Haitao Zhou, Zhendong Zhang, Ruirui Liu, Libin Liu*
13. Characteristics of Two-Stage  $\Gamma$ -Gate on AlGaAs/InGaAs/AlGaAs DH-HEMTs by Using AlGaAs/InGaP Etching-Stop Layers (N2522)  
*Jia-Chuan Lin, Meng-Kai Hsu, Fu-Xiang Zeng, Jia-Na Wei, Chi-Ting Liu, Hsi-Ting Hou*
14. Effect of Neodymium Salt in the Anodization of Aluminum in Sulphuric Acid (N1337)  
*Xiaozhen Liu, Jinhua Yang, Aibing Yu, Gang Wang, Lingling Song, Xiaoli Zhang*
15. Preparation and Performance of Controlled-Release Tablets of Salsalate (N1532)  
*Xiaozhen Liu, Jinhua Yang, Zhongfang Lai, Lingling Guo, Lingling Song, Yingzhen Shi*
16. The Effect of Thai Compost on Biodegradability of Polylactic Acid Based on ISO 14855-2 Method (N3035)  
*Prakit Sukyai*
17. Nano-Sized Silica Reinforcement of Epoxidized Natural Rubber Prepared in Latex State (N2321)  
*Methakarn Jarntong, Charoen Nakason, Zheng Peng, Natinee Lopattananon*
18. Synthesis of Alumina-Tungsten Carbide Composites by Self-Propagating High Temperature Synthesis Process (N2792)  
*Sutham Niyomwas*
19. Wavelet-Based Denoising of Vibration Signals for Tool-Edge Wear Detection in High Speed Machining of Inconel 718 (N1490)  
*Ning Fang, P. Srinivasa Pai, Nathan Edwards*
20. Al Ion Implantation of SiC Coated Carbon Fiber-Reinforced SiC Matrix Composites by Metal Vapor Vacuum Arc Ion Source (N2560)  
*Guojia Ma, Guoqiang Lin, Hongchen Wu*
21. The Nanocrystallinity Enhancement of Sol-Gel Derived TiO<sub>2</sub> Nanoparticles by Pre-Hydrothermal Treatment (N2363)  
*Akhmad Herman Yuwono, Yu Zhang, John Wang*

Morning, December 18, 2011

08:30 AM—11:50 AM

Session B: Materials Processing Technology

3/F The First Meeting Room (大正 1 号会议室)

Chairmen: Prof. Chun-Huy Wang, Nan-Jeon Institute of Technology, Taiwan

Prof. Yunze Long, Qingdao University, China

(12 minutes for each presentation, including 2-3 minutes of answering questions)

1. Fabrication and Characterization of Regenerated Cellulose /TiO<sub>2</sub> Nanocomposite Hybrid Fibers (N3236)  
*Chaorong Li, S hunxin Shu*
2. Synthesis, Electrical and Humidity Sensing Properties of BaTiO<sub>3</sub> Nanofibers via Electrospinning (N3545)  
*Hongdi Zhang, Yunze Long, Zhaojian Li, Bin Sun, Chenhao Sheng*
3. Influence of Thermal Behavior of Spindle on Machining Accuracy in Micro-Endmilling (N3712)  
*Osamu Horiuchi, Mitsuyoshi Nomura, Boxiao Ma, Takayuki Shibata, Yoshihiko Murakami, Masami Masuda*
4. Growth of Cu<sub>2</sub>ZnSnS<sub>4</sub> Thin Films by Sulfurization of Co-Sputtered Metallic Precursors (N3276)  
*Xinyi Li, Dacheng Wang, Qinyang Du, Weifeng Liu, Guoshun Jiang, Changfei Zhu*
5. Development of Tooling Cost Model for High Speed Hard Turning (N1296)  
*Erry Yulian Tribilas Adesta, Muataz Al Hazza, Delvis Agusman, Agus Geter E. Sutjipto*
6. Self-Assembly of Large Scale and High-Quality Colloidal Particle Films by Spin-Coating (N3630)  
*Zhaoting Yang, Qing Xu, Chaorong Li*
7. Dislocation Generated by Electron Irradiation (N3266)  
*Suryanto*
8. The Research of CAD System in Three-Plate Parallel Indexing Cam Mechanism (N3769)  
*Feng Xu, Yanhui He, Wei He, Hongtai Li*
9. Synthesis of Phenothiazine Derivatives Containing Hetero Atom and Analysis of Conductive Polymer (N3234)  
*Dae-Hee Yun, Han-Sol Yoo, Yong-Sung Park, Je-Wan Woo*
10. Effect of Voltage Pattern on Intensity of Electromagnetic Emission during Electric Flame-Off Process of Ball Bonding Machine (N3468)  
*Sarunya Puapairoj, Chiranut Sa-ngiamsak*



11. Primary Study on Electron Beam Surface Sculpt of Ti-6Al-4V (N3113)  
*Xichang Wang, Shuili Gong, Enming Guo, Haiyan Zhao, Hengdong Xu*
12. Design, Development & Experimental Investigation of Electro-Discharge Diamond Surface Grinding of Ti-6Al-4V (N2787)  
*Manoj Modi, Gopal Agarwal*
13. Process Optimization and Properties of Laser Cladding High Speed Steel Coatings on Nodular Cast Iron (N1580)  
*Jianbin Lv, Zhengyi Wang, Changsheng Liu*
14. Effect of Bi<sub>2</sub>O<sub>3</sub> Addition on the Physical and Electrical Properties of Lead-Free (Na<sub>0.5</sub>Bi<sub>0.5</sub>)TiO<sub>3</sub>-Ba(Sn<sub>0.08</sub>Ti<sub>0.92</sub>)O<sub>3</sub> Ceramics (N3094)  
*Chun-Huy Wang*
15. Improved Properties of CENR/PP Blends by Using Compatibilizers for Promoting the Interfacial Adhesion between Phases (N2195)  
*Anoma Thitithammawong, Adam Kaewsare, Charoen Nakason*
16. A Comparative Study of the Effect of Crosslink Agents and Chemical Modification on Properties of Natural Rubber Vulcanizates (N2345)  
*Anoma Thitithammawong, Rasmawatee Kasoe, Charoen Nakason*
17. Thermal Characterization of Polyurethane from Castor Oil Reinforced Sugarcane Straw Composites (N2564)  
*Patrícia Câmara Miléo, George Jackson de Moraes Rocha, Adilson Roberto Gonçalves*
18. Effect of Microstructure on the Corrosion Behavior of TiN Coatings (N2519)  
*Marwan Azzi, Jolanta-Eva Klemberg-Sapieha*
19. A Research on Static and Dynamic Fracture-Resistance of Welding Parts of Coolant Piping Material (N2334)  
*Haochuang Li, Kwang-Hyeon Lee, Jea-Mean Koo, Chang-Sung Seok*
20. Numerical Simulation of Fiber orientation of Short Fiber Reinforced Polypropylene in Injection Molding  
*Hesheng Liu, Aihua Xiong, Xingyuan Huang, Jiamei Lai*

Morning, December 18, 2011

**08:30 AM—11:50 AM**

**Session C: Advanced Manufacturing Technology**

**2/F The Third Meeting Room (大正 3 号会议室)**

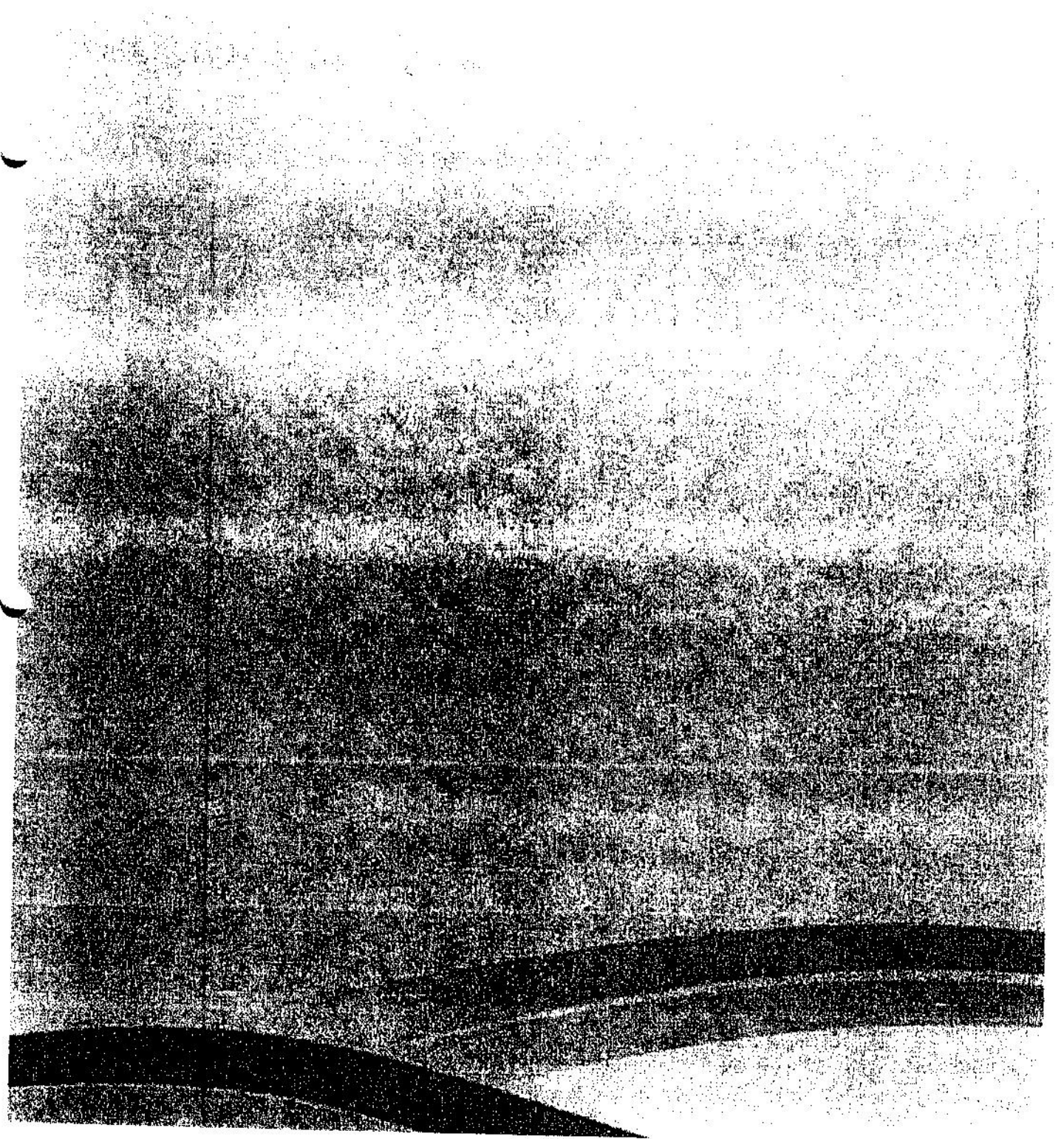
**Chairmen: Prof. Chien-Jong Shih, Tamkang University, Taiwan**

**Prof. Zhengzhou Wang, Tongji University, China**

(12 minutes for each presentation, including 2-3 minutes of answering questions)

1. Tool Wear in Sheet Metal Stamping(N2000)  
*Wenyi Yan, Michael P. Pereira, Bernard F. Rolfe*
2. Finite Capacity Scheduling - Large System Design (N3230)  
*Rodney L. Martin*
3. Nozzle Flow Model of High Pressure Variable-Rate Spraying Based on PWM Technology (N2651)  
*Changyuan Zhai, Xiu Wang, Dayin Liu, Wei Ma, Yijin Mao, Yongbing Ma*
4. Numerical Simulation Analysis on the Ten Cross Rolls Straightening Process of the Heavy Calibre Seamless Steel Tube (N2301)  
*Huagui Huang, Huanping Zheng, Fengshan Du, Wenzhang Wang*
5. ZM Solid Catalysts for Synthesis of 1-Butoxy-2-Propanol with High Selectivity (N3004)  
*Patuan Alfon, Johny W Soedarsono, Dedi Priadi, Sulistijono*
6. The Effects of Compression Pressure Applied on the Manufacture of Carbon Composite Bipolar Plate for PEMFC by Utilizing Graphite Waste Products (N2155)  
*Yunita Sadeli, Johny W.S, Bambang Prihandoko, Sri Harjanto*
7. Transformation of System Action for Reconfigurable Automation Work System (N2446)  
*Muhamad Arfauz A Rahman, John P. T.M o*
8. Flame Retardation of PP/EPDM Composites Containing Ammonium Polyphosphate and Its Microcapsule (N3146)  
*Zhengzhou Wang, Zhongqian Gu, Pingkai Jiang*

9. Coating of Ti64 Bearings in Air by Using a Q-Sw Laser (N3729)  
*Justyna Rozwadowska, Katsuyuki Kida, Edson Costa Santos, Takashi Honda, Hitonobu Koike, Kenji Kanemasu*
10. Investigation of Crack Initiation and Propagation during Rolling Contact Fatigue of SUJ2 Steel Bearings Using a Newly Developed one-Point Testing Machine (N3731)  
*Justyna Rozwadowska, Katsuyuki Kida, Edson Costa Santos, Takashi Honda, Hitonobu Koike, Yuji Kashima, Ryo Matsumoto*
11. Inhomogeneous 3D Finite Element Model for Prediction of Free Surface Roughening (N2436)  
*Tsuyoshi Furushima, Tetsuro Masuda, Ken-ichi Manabe, Sergei Alexandrov*
12. Deformation and Heat Transfer Analysis for High Speed Dieless Drawing of AZ31 Magnesium Alloy Tubes (N2434)  
*Tsuyoshi Furushima, Takuma Ikeda, Ken-ichi Manabe*
13. An Intake Three-Pipeline Model Using Experimental Optimization for a Small Engine (N2629)  
*Chien-Jong Shih, Ting-Hao Chang, Tso-Liang Teng*
14. Low Cost Diffuser Based Micropump Using Pinch Actuation (N2496)  
*Pei Song Chee, Ruzairi Abdul Rahim, U.Hashim, Rashidah Arsat, Pei Ling Leow*
15. Evaluation of Welding Residual Stress Based on Temper Bead Welding Technique (N1630)  
*XianShi, Yiliang Zhang, Jianping Zhao, Ruibin Gou*
16. Conductivity Studies on  $\text{Li}_1\text{-XAl}_x\text{Ti}_2\text{-X(PO}_4)_3$  ( $\text{X}=0.0\text{-}0.5$ ) Due to the Addition of  $\text{Al}^{3+}$  Trivalent Cation (N2907)  
*Maziidah Hamidi, Syafawati Nadiyah Mohamed, M uhd Zu Azhan Yahya*
17. Synthesis of Norbornene Derivative Using Diels-Alder Reaction (N3229)  
*JunSeong Park, H yun-Chul Oh, Yong Sung Park, Je-Wan Woo*
18. Magnetic Abrasive Finishing - A Review (N1985)  
*M.G.Patil, K.Chandra, P.S.Misra*
- ✓ 19. The Production of poly(L-Lactic acid) from 2-steps direct polycondensation in 100 kg scale (N3643)  
*Sommai Pivsa-Art, Weraporn Pivsa-Art, Sumonman Niamlang, K iyoaki Ishimoto, Hitomi Ohara*



2011 International Conference on  
Advances in Materials and Manufacturing Processes  
December 16-18, Guilin, China

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The Scientific Committee has completed its review of your paper submitted for the 2nd/2011 International Conference on Advances in Materials and Manufacturing Processes (ICAMMP 2011). The final decision is made base on the peer-review reports, the scientific merits and the relevance.

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Manuscript Number	N3643
Authors	Sommai Pivsa-Art, Weraporn Pivsa-Art, Sumonman Niamlang*, Kiyooki Ishimoto and Hitomi Ohara
Title	The Production of poly(L-Lactic acid) from 2-steps direct polycondensation in 100 kg scale

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2011-10-13

# Production of Poly(L-lactic acid) from 2-Steps Direct Polycondensation in a 100 kg Scale

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**Keywords:** Poly(L-lactic acid), mass scale production, 2-steps direct polycondensation, solid state polymerization

**Abstract.** Synthesis of poly(L-lactic acid), PLLA, in a mass scale production using 2-steps direct polycondensation methods was investigated. One hundred kilogram of L-lactic acid was esterified in a designed reactor under reduced pressure at elevated temperature in the first step, followed solid state polymerization, SSP. The synthesized PLLA from both laboratory (5 g) and mass (100 L) scales show comparable melting temperature,  $T_m$  (151 – 172 °C) and molecular weight ( $M_n$ ) (10,000 – 32,000) at the similar polymerization conditions. The appearances of synthesized PLLA are yellow-white solid powder. The results show high potential to produce environmental friendly polymer, PLLA, using non-complex facilities process.

## Introduction

Recently, many countries and states are banning petroleum based plastic grocery bags due to its non-degradable in environment, so biodegradable polymer has attracted much attention as the biodegradable plastic bag for the replacement of petroleum based plastic bag. Polyesters such as polyhydroxyalkanoates (PHA), polyhydroxybutyrates (PHB) and polylactic acid (PLA) play a predominant role as biodegradable plastics due to their potentially hydrolysable ester bond (Nampoothiri *et al*, 2010). Poly(L-lactic acid) PLLA is widely used to produce the biodegradable plastic bag, since the raw materials can be produced on a mass scale by the microbial fermentation of agricultural by products mainly the carbohydrate rich substances. PLA have the outstanding mechanical properties over other biodegradable polyester and in absence of enzymes or catalyst, PLLA degrade to lactic acid by simple hydrolysis [1]. In commercial production of PLLA is performed by using the ring opening polymerization (ROP) of the dimer of L-lactic acid, L-lactide. The synthesized PLLA from ring opening polymerization of lactide, a process that uses catalysts such as tin, zinc, aluminum and lead, initiators such as *n*-, *sec*- and *tert*-butyl lithium, and solvent such as diphenyl ether, toluene and chloroform. Many of these components are toxic or flammable. As a result, this process requires purification to remove the unwanted materials from the product, which requires complex facilities [2]. The alternative route to produce PLLA is the direct polycondensation of L-lactic acid or oligomer of PLLA. However the synthesis of high molecular weight PLLA by this method is considered to be difficult, because of instability of the lactic acid oligomer and the difficulty in removing the water produced in the polymerization, which can encourage the depolymerization [3-4]. Thus, PLLA synthesized with the organic solvent and via melt-solid polymerization have been investigated. The direct polycondensation polymerization has 3



steps of polymerization: dehydration, polycondensation and solid state polymerization [5]. However, the use of solvent increases the difficulty of process control and polymer purification. These reasons increase the process cost and limits the process to small scale production [6]. To solve these limitation, the utilization of binary catalyst system of stannous (II) chloride dehydrate/para-toluene sulphonic acid monohydrate ( $\text{SnCl}_2/\text{TSA}$ ) was studied. The higher molecular weight of PLLA was produced [7]. To obtain reduce the production cost and time in large scale, the low number of steps in production line is required. Thus a 2-steps direct polycondensation is developed; Step1 dehydration and polycondensation, Step2 solid state polymerization [8]. This 2-steps direct polycondensation is the process that has the possibility to scale up to large scales of PLLA production (100 kg Batch) for responsive to the higher demand in PLLA.

In this present work, PLLA was synthesized via 2-steps direct polycondensation in lab and mass scale to determine the potential of produce PLLA via environmental friendly and non-complex facilities process. The thermal properties and  $^1\text{H-NMR}$  of synthesized PLLA from both scales were investigated.

## Experiment

### Materials

L-lactic acid (Galactic) with a monomer concentration of 90% w/w was used as received. *p*-Toluenesulfonic acid, PTSA was used as catalyst and used as received. Chloroform was used as the eluent in gel permeation chromatography (GPC). The polystyrene standard for GPC calibration was supplied by Sigma Aldrich Chemical Co. (USA).

### Synthesis of PLLA

PLLA were synthesized by 2 steps direct polycondensation polymerization to obtain the PLLA oligomer and further polymerization by solid state polymerization to obtain high molecular weight PLLA.

#### Lab scale (5 g batch)

Direct polycondensation polymerizations were conducted in 25 ml custom made test tube with a mechanical stirrer and condenser connected to an inline cold trap and a vacuum pump. Thermometer was used to measure the exterior oil temperatures of the test tube. The reactor pressure was measured and controlled by vacuum controller (VCN500, OKANO). The pressure in the reactor was lower incrementally using a vacuum pump (G-50DA, ULVAC-KIKO).

Five grams of L-LA and 0.5%w/w of PTSA were added to reactor (test tube), PLLA was synthesized by direct polycondensation (DP) through three operations: distillation, oligomerization and polymerization. The temperature and pressure during the polymerization were precisely controlled at 150 °C 30 torr for 4 hr. At the end of the reaction, the products were poured in the plastic tube and allowed to cool to room temperature. After solidification, the product was grinded and annealed at 100 °C for 2 hr. Annealed product was further solid state polymerized in the reactor to obtain high molecular weight PLLA as 3 following steps: step1 110 °C 10 torr 5 hr, step 2 130 °C 10 torr 5 hr and step 3 150 °C 10 torr 5 hr.

#### Mass scale (100 kg batch)

For study the potential to produce PLLA in mass scale from direct polycondensation polymerizations, the similar reaction condition of lab scale was selected to compare the thermal properties and molecular weight of synthesized PLLA on the effect of reactor size. The reaction was conducted in 100 kg reactor with a paddle and condenser connected to an inline cold trap and a vacuum pump. Thermocouple was used to measure the internal of reactor and exterior oil temperatures. The reactor pressure was measured and controlled by vacuum controller (VCN500,

OKANO). The pressure in the reactor was lower incrementally using a rotary vacuum pump (K7503, OSAKA VACUUM Ltd.)

One hundred kilograms of L-LA and 0.5%w/w of PTSA were added to reactor, PLLA was synthesized by direct polycondensation (DP) through three operations: distillation, oligomerization and polymerization. The temperature and pressure during the polymerization were precisely controlled. Polymerization was done for 4 hr at 30 torr after reaching 150°C. At the end of the reaction, the products were discharged at 150 °C in stainless bats and allowed to cool to room temperature for 7 hr and were kept in dry ice. After solidification, the product was grinded and annealed at 90 °C for 2 hr. Annealed product was further solid state polymerized in the reactor to obtain high molecular weight PLLA. The reaction temperature has to lower than melting temperature,  $T_m$  of sample to prevent the molten of PLLA [8-10]. Thus, the temperature was gradually increase step by step as following steps: step1 100 °C 10 torr 23 hr; step 2 115 °C 10 torr 8 hr; step 3 120 °C 10 torr 14 hr. and step 4 135 °C 10 torr 6 hr.

#### Analytical method

The PLA's weight average molecular weight (MW), number average molecular weight (Mn) and Mw/Mn ratio were determined using a gel permeation chromatography (GPC) system equipped with two chromatography columns and a RI detector. Chloroform was used as the eluent at a flow rate of 1 mL/min, and the molecular weight were calibrated to a polystyrene standard at 40 °C. The thermal properties (melting temperature,  $T_m$  and glass transition temperature,  $T_g$ ) were measured by using DSC at heating rate 20 °C/min under  $N_2$  atmosphere.

To confirm the successful of synthesized PLLA,  $^1H$ -NMR was use to investigate the chemical structure of L-LA and PLLA.

#### Results and discussion

##### Thermal and physical properties of synthesized PLLA

The PLLA oligomer, that obtained from direct polycondensation were annealed and further polymerized by solid state polymerization, SSP, respectively, as previously described. In SSP, the polymerization temperatures have to lower than melting temperature,  $T_m$  to maintain the reaction in solid phase. Thus the reactions were stopped to sampling the synthesized PLLA for determining  $T_m$  by DSC before further polymerization. The physical and thermal properties of synthesized PLLA from both lab scale and mass scale are tabulated in Table 1 and 2, respectively. For both scale, thermal and physical properties show the similar results. After direct polycondensation, the low molecular weight PLLA or PLLA oligomer are obtained. Then synthesized PLLA oligomers were further polymerized via SSP at temperature below  $T_m$ . As longer solid state polymerization time, higher MW of PLLA is obtained due to the longer time and higher probability of oligomer to interact each other [8-9]. Melting temperatures,  $T_m$  also show the similar results,  $T_m$  of synthesized PLLA gradually increase with increasing SSP time as shown in Figure 1 and 2 for lab scale and mass scale, respectively. For lab scale,  $T_m$  of polymerized PLLA are 137, 140, 150, 162 and 172 °C for direct polycondensation, annealing, SSP step1, SSP step 2 and SSP step 3, respectively. For mass scale,  $T_m$  of polymerized PLLA are 130, 131, 130, 143, 150, 150 and 157 °C for direct polycondensation, annealing, SSP step1, SSP step 2, SSP step 3 and SSP step 4, respectively. As longer SSP time, higher and shaper DSC thermograms are obtained due to the increasing of reaction time [11-13]. The  $T_m$  and MW values of the synthesized PLLA from lab scale and mass scale are comparable at the same conditions. Thus the size of reactor does not show the effect on the  $T_m$  and MW of synthesized PLLA. For the physical characteristic also show the similar characteristic which are yellow-white solid power.

For mass scale, there are comments involved bumping of lactic acid during direct polycondensation and the agglomerations of molten PLLA during SSP due to low molecular weight of PLLA oligomer. To avoid bumping of lactic acid during direct polycondensation, gradually adjust



temperature and pressure are required. For preventing the agglomeration of PLLA in SSP, pre-anealling reaction at 40-50 °C is needed.

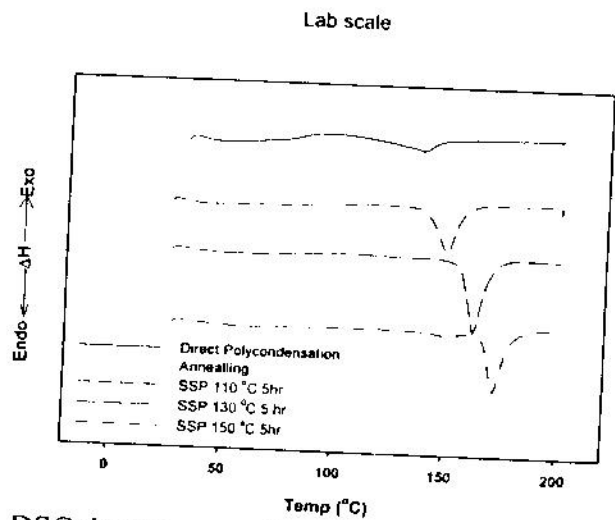


Figure 1. DSC thermogram of synthesized PLLA from mass scale.

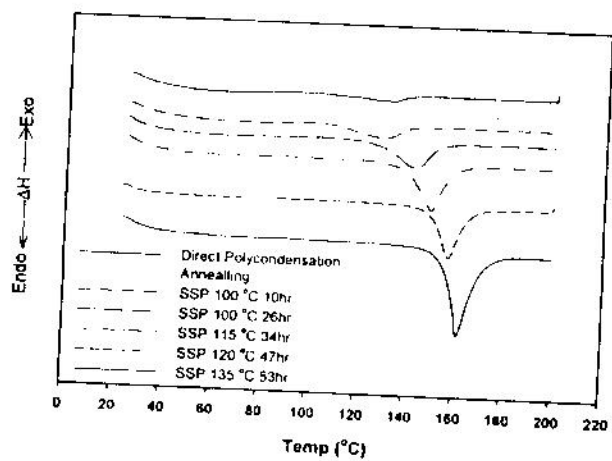


Figure 2. DSC thermogram of synthesized PLLA from mass scale.

**Table1** Thermal and physical properties of synthesized PLLA from lab scale (5 g)

Step	Condition	Yield (%)	Tc (°C)	Tm (°C)	MW	PDI	Characteristics	
							Appearance	Color
Melt Polymerization	150 °C 30 torr	62.75	96	137	3730	2.37	Solid-powder	White
Annealing	100 °C 1 atm		56	140	4220	2.08	Solid-powder	White
Solid state Polymerization/ 5h	110°C 10 torr 5 hr			150	7160	2.44	Solid-powder	White
Solid state Polymerization/ 10h	130°C 10 torr 5 hr			162	21300	2.17	Solid-powder	White
Solid state Polymerization/ 15h	150°C 10 torr 5 hr			151, 172	32400	2.19	Solid-powder	Yellow - white

**Table2** Thermal and physical properties of synthesized PLLA from mass scale

Step	Condition	Yield (%)	Tc (°C)	Tm (°C)	MW	PDI	Characteristics	
							Appearance	color
Melt Polymerization	150 °C 30 torr	75.25	92	130	3160	2.41	Solid-powder	White
Annealing	90 °C Atm 3 hr			131	3570	1.93	Solid-powder	White
Solid state Polymerization/ 10 hr	110°C 10 torr 7 hr			130	3790	1.89	Solid-powder *	Yellow -brown
Solid state Polymerization/ 26 hr	100°C 10 torr 16 hr			143	7050	2.14	Solid-powder, rock of melt part	Yellow -white
Solid state Polymerization/ 34 hr	115°C 10 torr 8 hr			150	10800	2.14	Solid-powder, Small rock	Yellow - white
Solid state Polymerization/ 47 hr	120°C 10 torr 13 hr			150	20200	2.36	Solid-powder, Small rock	Yellow - white
Solid state Polymerization/ 53 hr	135°C 10 torr 6 hr			157	24000	2.16	Solid-powder, Small rock	Yellow - white

\* Partially melt

### <sup>1</sup>H-NMR characterizations

To confirm the successful of synthesise of PLLA, <sup>1</sup>H-NMR of lactic acid, PLLA oligomer and synthesized PLLA were investigated. For lab scale, the synthesized PLLA from lab scale was successfully synthesized as shown in Figure 3. Peaks at 5.1-5.3 ppm are the methane protons in LA repeat units, while the signals of methine protons in the end groups of the polymers appear at 4.4 ppm. The peaks at 1.6 ppm represent to methyl protons, which often interfere with those of residual lactic acid monomer [14-15].

For mass scale, PLLA were also successfully synthesized and confirmed by using <sup>1</sup>H-NMR as shown in Figure 4. After direct polycondensation reaction, synthesized PLLA oligomer shows two equilibria of L-lactic acid - PLLA (K1) and PLLA - lactide (K2) at the same time [16] as shown in Figure 5.

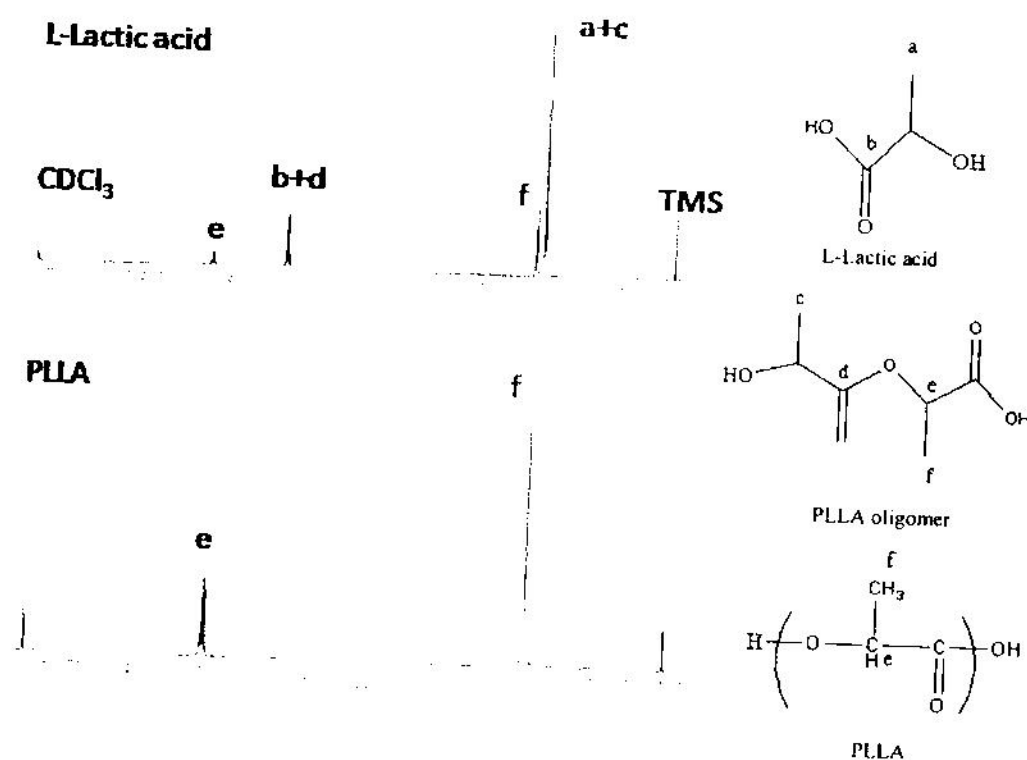


Figure 3. <sup>1</sup>H-NMR of synthesized PLLA from lab scale.

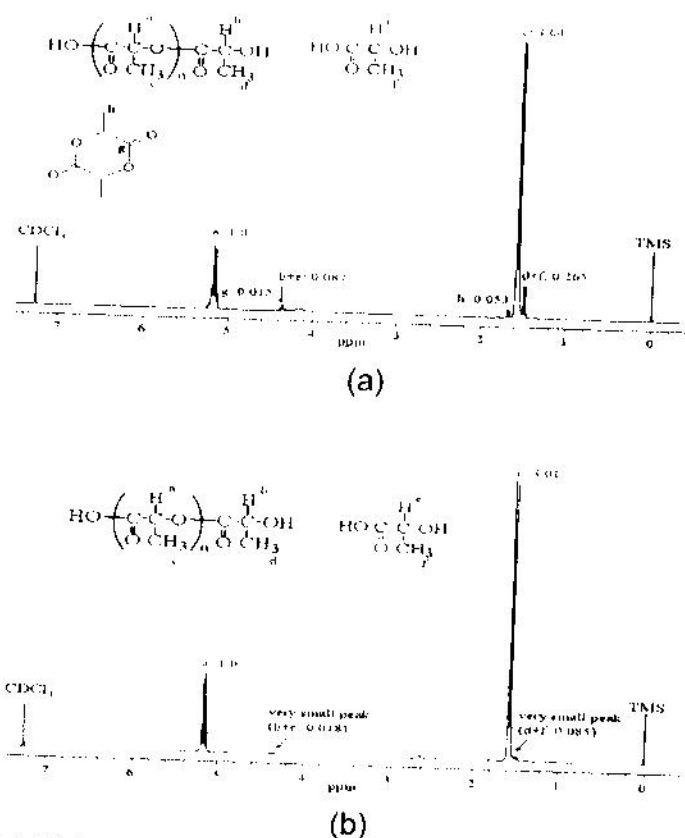


Figure 4.  $^1\text{H}$  NMR of a) PLLA oligomer and b) synthesized PPLA from mass scale.

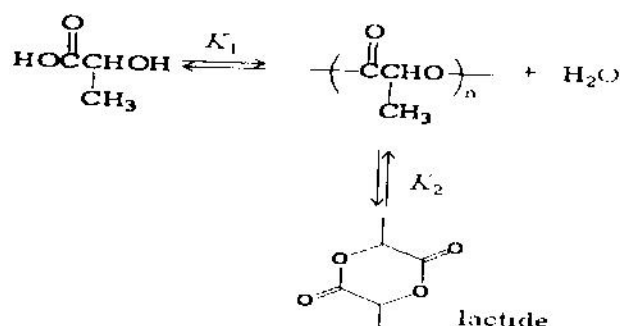


Figure 5. Schematic diagram of 2 reaction equilibria involved in direct polycondensation reaction.

## Conclusions

To develop the environmental friendly and non-complex facilities process of PLLA production. In this work, PLLA was successfully synthesized via direct polycondensation and solid state polymerization in lab and mass scale and confirmed by using  $^1\text{H}$ -NMR. The synthesized PLLA from both lab and mass scale shows the comparable melting temperature,  $T_m$  and MW. Thus there are high potential to produce PLLA from new process. The investigations of effect of time and reaction condition for high efficiency and economized reaction are required in future works.

### Acknowledgements

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