PLASMA PHYSICS LABORATORY

National Institute of Physics
College of Science, University of the Philippines
Diliman, Quezon City

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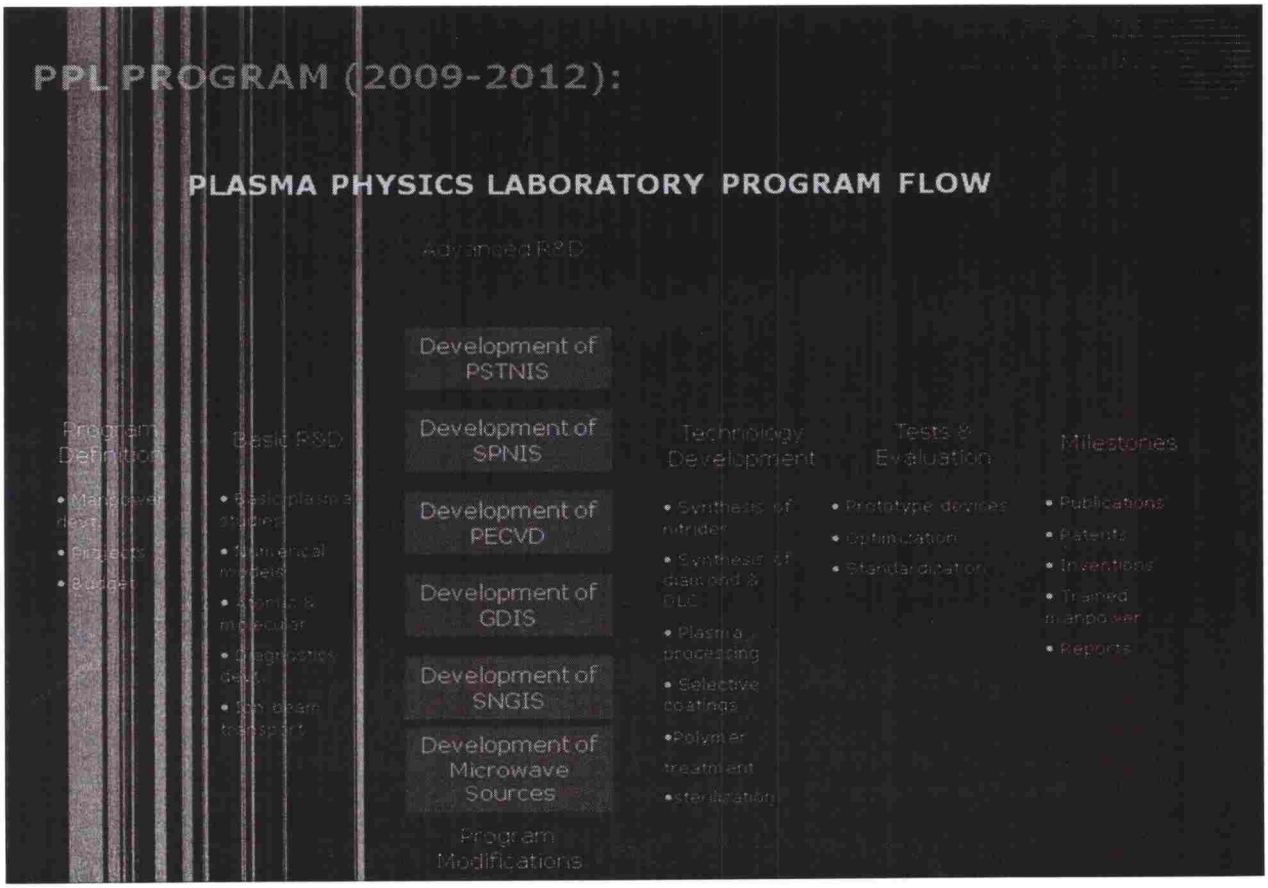
A. Introduction

The laboratory continued implementation of its program supported in part by the Department of Science and Technology (DOST) and other agencies in 2012. It has pursued the second year of implementation of projects on microwave systems, funded by DOST and donated equipment from IBF Electronic, GmbH, Germany. It has continued Research Service Agreements on coating technology with Philippine companies. It has also started the implementation of a DOST- Technicom grant towards commercialization of TiN coating developed in the laboratory. These programs and other projects are summarized in this report. They constitute the research and development program in plasma science and technology for the period 2009-2012. The program flow is shown in the diagram.



The acronyms stand for Plasma Sputter-type Negative Ion Source (PSTNIS), Sheet Plasma Negative Ion Source (SPNIS), Plasma Enhanced Chemical Vapor Deposition (PECVD), Gas Discharge Ion Source (GDIS), and Streaming Neutral Gas Injection System (SNGIS). A description of the R&D activities on each device follows.

IA. Project Title: Research Service Agreements on Coating Technology

Funding Source: Various Philippine Companies
Amount of Funding : 10% of cost of uncoated tools
Duration: January 2012- December 2012

IB. Project Title: Market testing and process optimization of industrial prototype plasma enhanced chemical vapor titanium nitride coating technology
Total Funds: P 9,797,092.00
Source: DOST-TECHNICOM
Duration: November 16, 2011 – November 15, 2012

The Sheet Plasma Negative Ion Source (SPNIS) was developed from previous DOST-funded projects purposely for the production, extraction and enhancement of negative hydrogen ions (H^-) using a mixture of hydrogen, argon and magnesium plasma. The H^- ions extracted using a modified Wien filter was used in the synthesis of silicon hydride. Selective solar (IR) coatings of tin-bismuth on various metal substrates were also deposited using the facility. The facility was subsequently configured for TiN coating of metal tools used by various industries (tool and dye, etc.) operating in the Philippines.

A similar machine is being developed starting November 16, 2011 under a DOST-TECHNICOM project titled "Market testing and process optimization of industrial prototype plasma enhanced chemical vapor titanium nitride coating technology". Costing PhP 9,797,092.00, the one year project builds an industrial prototype of the magnetized sheet plasma source for coating of TiN to service the manufacturing needs of industry. The prototype machine will be based at the DOST-Metals Industry Research and Development Center (MIRDC). Once commissioned, it will be operated under a research and patent licensing memorandum of agreement with the Asian Semiconductor Electronics Technologies (ASET) Corporation and the university. The project has been extended until February 15, 2013.

II. Project Title. Plasma Etching Using Low Energy Ions from a Gas Discharge Source

Funding Source: NIP MOOE Allotment
Amount of Funding : PhP 100,000.00
Duration: January 2012- December 2012

A Gas Discharge Ion Source (GDIS) was developed as an example of a low energy ion beam source. Ion beams of carbon tetrafluoride (CF_4) plasma are used for surface modification applications such as ion etching on silicon carbide (SiC) samples. The ion treatment that the sample surfaces undergo changes their physicochemical properties. The modification is of great significance in the moisture absorption of the material improving its characteristic features like contact angle and other physical characteristics.

III. Project Title. Beam Transport in a Sputter-type Ion Source

Funding Source: NIP MOOE Allotment
Amount of Funding : PhP 100,000.00
Duration: January 2011- December 2011

In this study, Zirconium (Zr), Copper (Cu), Silver (Ag), Niobium (Nb), Aluminum (Al), Titanium (Ti) and Gold (Au) are used as metal targets to produce negative metal ions. The metal ions are fully characterized in terms of ion beam energies and ion currents using a retarding-type electrostatic energy analyzer. The behavior of ion energies and ion currents towards increasing target voltages and differences in beam characteristics between each metal used are studied. The effect of the chamber pressure to the ion beam characteristics is also studied.

IV. Project Title: Physical Vapor Deposition of Advance MAX Phase
Funding Source: Philippine Council for Industry, Energy and Emerging Technology Research and Development, Department of Science and Technology
Amount of Funding : PhP 4,051,296.00
Duration: April 1, 20012-March 31, 2014

Physical vapor deposition (PVD) is fundamentally a vaporization coating technique, involving transfer of material on an atomic level. It is an alternative process to electroplating. The process is similar to chemical vapor deposition (CVD) except that the raw materials/precursors, i.e. the material that is going to be deposited starts out in solid form, whereas in CVD, the precursors are introduced to the reaction chamber in the gaseous state. The synthesis of new materials that possess a unique combination of metallic and ceramic properties has been of much interest lately. A large solid group of compounds with similar attributes are called "MAX phases", where M is a transition metal, A is an A-group element, and X is C and/or N

Most PVD syntheses of MAX phases are constrained with high-temperature requirement. Substrate temperatures in the range 800–1000 °C are usually reported, limiting the use of temperature-sensitive substrates. Hence, a promising novel route in the experimentation of these ternary compounds is the use of the magnetized sheet plasma ion source (MSPIS) where no substrate heating is required.

The objective of the project is to establish a reliable and reproducible PVD procedure using a magnetized sheet plasma facility for obtaining advanced MAX thin films with desirable properties for functional and decorative applications. Combinations of remarkable properties of MAX phases open up new applications for heating elements, burner nozzles, reactor heat exchangers, high temperature bearings and components in the chemical and related petrochemical industries.

V. Project Title: Studies on Microwave Plasma Systems

Project A: Adhesion enhancements of metal and plastic surfaces with adhesives using microwave-induced atmospheric plasma jets
Total Funds: P6,850,713.50 (cost of equipment donation)
Source: IBF Electronic GmbH & Co. KG, Ober-Ramstadt, Germany
Duration: August 2010 – July 2012

The R&D aims to develop an atmospheric plasma jet from a 2.45 GHz microwave source and use it for adhesion enhancements of epoxy on industrial materials. The R&D preliminary targets are the studies on the stability of the plasma jet in terms of ignition,

minimum reflection, and heating using high-ripple 2 kW and low ripple 6 kW magnetrons. An initial experiment on a low pressure microwave plasma source shows that the treatment of stainless steel surfaces have enhanced its adhesion with an epoxy adhesive. Based on tensile test results, the adhesion strength of epoxy-bonded O_2/Ar plasma treated surfaces was increased to 3816.0 N from 3038.3 N for the epoxy-bonded untreated surfaces. These results would be replicated in the atmospheric microwave plasma jet.

Project B: Development of microwave-induced plasma jets
Total Funds: P1,966,423.00
Source: DOST-PCIEERD
Duration: September 2010 – August 2011 and December 2011 – November 2012

Continuing R&D is pursued on an atmospheric plasma jet developed from an existing microwave waveguide system (MWS) and equipment donated by IBF Electronic, GmbH & Co. KG, Germany. The plasma chamber and vacuum systems of the MWS are replaced with customized tapered waveguide and gas nozzle system, specifically designed for enhanced stability of the plasma jet. The atmospheric microwave plasma jet is developed to enhance the paintability of various industrial materials such as stainless steel, glass, plastics, etc. Plasma treatment can quickly enhance the surface paintability of industrial materials to ordinary paints without the need for primers. From initial results, it has been shown that the plasma jet treatment of stainless steel can achieve superhydrophilic surfaces using argon and argon-nitrogen plasmas.

B. R&D Highlights

Sheet Plasma Negative Ion Source (SPNIS) Projects

The Asian Semiconductor Electronics Technologies (ASET) Corporation has agreed to provide financial and other support for conducting research and development under the DOST-Technicom project and to license the patented technology.

Continuing studies on the sheet plasma specifically on high dynamic range imaging resulted in the papers presented in C.b1.1 and C.c1.10 The device was also used in the synthesis of tin oxide, details of which are summarized in the papers C.b2.1 C.c1.4 and the thesis D.1.5. Two papers on the MAX phase synthesis of TiCdC and NbAlC are reported in C.b2.2 and C.b2.3, respectively. Thermally induced magnetization of ilmenite (thesis D.2.2) is also demonstrated using the device.

Plasma Enhanced Chemical Vapor Deposition (PECVD) Projects

Flame endurance of wood samples was increased using chemical and plasma treatment. The chemical treatment involved the conventional method of spraying the solution over the wood surface at atmospheric condition and chemical vapor deposition in a vacuum chamber. In the plasma treatment, wood samples were immersed in reactive plasmas comprised of various ratios of flame retardant solutions and/or reactive gases. Based on the thermo-gravimetric analysis (TGA) results, chemical treatment by exposure to vapor was found to be more effective compared to the direct application. However, comparing the chemical treatment and plasma treatment, the 25 mA phosphoric acid plasma treatment was found to yield superior thermal stability and flame retardant properties; in terms of the

highest onset temperature and temperature of maximum pyrolysis, highest residual char% and comparably low total weight%. These results are detailed in the papers C.a1.3.

Plasma surface modification to improve adhesion properties of *S. contorta*, *G. arborea* and *A. mangium* wood specimens were done with the DBD machine. Results are published in the papers C.a1.1 and C.a1.2.

Microwave/DBD Projects

Characterizations of the. operation of atmospheric plasma jet and plasma pen (APJ/P) are described in the papers/thesis C.b.3.1, C.b.3.2, C.c1.1, C.c1.3, C.c1.7 and D.2.6. Treatment of materials using the APJ/P is demonstrated via the following papers: C.b.3.2, C.b.3.3, C.c1.5.

Next generation medical sterilization equipment based on a dielectric barrier discharge (DBD) and atmospheric plasma jets/pens (APJ/P) are described in papers C.b.3.4, C.b.3.5, C.c1.1, C.c1.2, C.c1.9 and in the theses D.1.3 and D.2.5. The equipments are safe, simple, low cost and can perform powerful sterilization in a short time. The efficacy of bacterial inactivation of DBD and APJ/P are compared. Test samples of a) *Bacillus subtilis, b) P. aeruginosa, c) Candida tropicalis,* and *d) Candida utilis* laced on packaging material, metal or polypropylene are exposed to H_2O_2, oxygen, argon and nitrogen mixed species plasma and the decimal reduction value (D-value), that is, the time required to deactivate 90% of the bacteria, are determined. The D-value for argon-oxygen atmospheric plasma at relatively low absorbed microwave power is about a minute for the inactivation of a) and 20s for b). For c) and d) a mixture of oxygen and nitrogen gave rise to a D-value of a few tens of seconds. In the case of the DBD, a mixture of oxygen and hydrogen peroxide plasmas lead to D-values of about 10 minutes for the inactivation of a) but at lower temperatures compared to the treatment using the APJ/P. The D-values reported in these papers highlight the merits of plasma sterilization over conventional techniques.

Other Highlights

Publications of the laboratory in SCI-indexed international journals (6), proceedings of international (11) and local conferences (12) and papers presented in various other conferences are summarized in Section C of this report. The laboratory trained five (5) undergraduates and six (6) graduate theses students who were awarded their respective degrees in 2012.

One Ph.D. student (Ms. Michelle Villamayor) was sent on research visit under the auspices of the DOST-JSPS bilateral agreement to the Department of Applied Physics, Doshisha Universit, Kyotanabe, Japan for two weeks in November and December, 2012.

C. 2012 Publications and Conference Papers Presented (*italicized authors are collaborators, otherwise affiliated with the laboratory*)

a1. SCI –indexed international journal

1. *M. N. Acda, E. E. Devera, R. J. Cabangon*, K. G. Pabelina, and H. J. Ramos, "Effects of dielectric barrier discharge plasma modification on surface properties of tropical hardwoods at low pressure", Journal of Tropical Forest Science **24** (3) (2012) 416–425 (2012)

2. *Menandro N. Acda, Edgar E. Devera, Rico J. Cabangon*, and Henry J. Ramos, "Effects of plasma modification on adhesion properties of wood", International Journal of Adhesion & Adhesives **32** (2012) 70–75

3. Karel G. Pabeliña, Carmencita O. Lumban, and Henry J. Ramos, "Plasma impregnation of wood with fire retardants", Nuclear Instruments and Methods in Physics Research B **272** (2012) 365–369.

4. *L. Jirkovsky*, L.Ma. Bo-ot, *A. Muriel*, and H. Ramos, "Time development of two and three-dimensional self-gravitating system using initial Dirac delta function distribution", Physica A **391** (2012) 1128–1132

5. Giovanni M. Malapit, Christian Lorenz S. Mahinay, Matthew D. Poral, and Henry J. Ramos, "Electrostatic energy analyzer measurements of low energy zirconium beam parameters in a plasma sputter-type negative ion source", Rev. Sci. Instrum. **83** (2012), 02B704

6. Luis Ma. Bo-ot, *Yao-Hong Wang, Che-Ming Chiang and Chi-Ming Lai*, "Effects of a Green Space Layout on the Outdoor Thermal Environment at the Neighborhood Level", Energies **5**(10), (2012) 3723-3735.

b1. Paper presented at the 22nd International Toki Conference (ITC22) , Ceratopia, Toki, Gifu, Japan, November19 - 22, 2012.

1. Michelle Marie S. Villamayor, Leo Mendel D. Rosario, Rommel Paulo Viloan, Ma. Camille Lacdan, Julie Ting, Beverly Anne Suarez, *Shuichi Kato*, Roy B. Tumlos, *Maricor N. Soriano*, *Motoi Wada*, Henry J. Ramos, "High Dynamic Range Imaging of Atmospheric Jet Plasma and Low Pressure Magnetized Sheet Plasma", 22nd International Toki Conference, November 19-22, 2012, Toki City, Gifu, Japan.

b2. Papers presented at the International Conference of Young Researchers on Advanced Materials 2012, July 1-6, 2012, Singapore

1. Aubrey Faith Mella, Janella Salamania, Michelle Villamayor, Henry J. Ramos, "Synthesis of Tin Oxide (11) Thin Films on Silicon and Glass Substrates using Magnetized Sheet Plasma Ion Source"

2. Matthew Villanueva, Henry J. Ramos, "Deposition of TiCdC Thin Film Via Sheet Plasma Negative Ion Source (SPNIS)"

3. Janella Salamania, Aubrey Faith Mella, Henry J. Ramos, "Thin Film Deposition of NbAlC on Stainless Steel Substrates using Sheet Plasma Negative Ion Source "

b3. Papers presented at the 11th Asia-Pacific Conference on Plasma Science and Technology (APCPST), Kyoto, Japan, 2-5 October 2012.

1. L.M.D. Rosario, H.V. Lee, Jr., J.A.S. Ting, H.J. Ramos, and R.B. Tumlos, "Generation of single filament and flame in microwave-induced atmospheric plasmas"

2. R.B. Tumlos, L.M.D. Rosario, J.A.S. Ting, H.V. Lee, Jr., M.C.C. Lacdan, C.P. Romero, E. Tinacba, T. Tuballa, J.R. Uy, and H.J. Ramos, "Microwave-plasma devices for material surface treatment"

3. J.A.S. Ting, L.M.D. Rosario, H.J. Ramos, R.B. Tumlos, and R.V. Fischer, "Enhanced wettability of aluminum and PMMA surfaces using a coaxial plasma bulb device"

4. J.K.T. Soriano, L.M.D. Rosario, M.C.M. Lacdan, H.V. Lee, Jr., J.A.S. Ting, H.J. Ramos, *M.A.T. Siringan*, and R.B. Tumlos, "Microwave atmospheric jet plasma (AJP) disinfection of Candida tropicalis suspension"

5. C.F.P. Romero, K.G. Pabelina, L.M.D. Rosario, M.C.C. Lacdan, H.V. Lee, Jr., J.A.S. Ting, H.J. Ramos, *M.A.T. Siringan*, and R.B. Tumlos, "Bacterial inactivation using atmospheric-pressure plasma jet".

b4. Paper presented at the International. Journal Arts and Sciences Conference, Vienna, April 2012.

1. *Simon Formoso* and L. Ma. T. Bo-ot, 'Development and Initial Application of a Prototypical Tapping Machine'

b5. Paper presented at the Conference for Sustainable Business in Asia 2012, Bangkok, November 2012.

1. *Raymond Clarin* and L. Ma. T. Bo-ot, "Alternative Methods of Construction in Projects with Short Turnover Time"

c1. Proceedings of the 30th SPP Physics Congress, De La Salle Health Sciences Institute, Dasmarina City, Cavite, Philippines October 24-26, 2011, ISSN 1656-2666, Vol. 8.

1. Ma. Camille Lacdan, Julie Anne Ting, Leo Mendel Rosario, Henry Lee, Roy Tumlos, Henry Ramos, "Diagnostics of Microwave induced atmospheric plasma"
2. Jenica Rozette Uy, Joey Kim Soriano, Leo Mendel Rosario, Camille Faith Romero, Roy Tumlos, *Maria Auxilia Siringan*, "Bacterial inactivation of Bacillus subtilis using microwave atmospheric pen plasma"
3. Leo Mendel Rosario, Julie Anne Ting, Ma. Camille Lacdan, Henry Lee, Henry Ramos, Roy Tumlos, "Constriction of Ar/N_2 plasma flame using tapered nozzles"
4. Aubrey Faith Mella, Michelle Villamayor, Rommel Viloan, Henry Ramos, "Effect of oxygen content on tin oxide thin film deposition on silicon and glass using a sheet plasma negative ion source"
5. Erin Joy Tinacba, Julie Anne Ting, Leo Mendel Rosario, Henry Lee, Henry Ramos, Roy Tumlos, "Atmospheric microwave plasma cleaning of oil-contaminated aluminum surface"

6. Meryll Viernes, Hannah Cosinero, Aiko del Rosario, Christian Mahinay, "Simulation of the experimental potential of einzel lens and kinetic energy of gold ion to focus the ion beam in plasma sputter-type negative ion source"

7. Aren Renz Centeno, Henry Lee, Leo Mendel Rosario, "Comparison of the output power of a 2.45 GHz continuous wave magnetron using power meter and calorimetric measurements"

8. Joey Kim Soriano, Leo Mendel Rosario, Ma. Camille Lacdan, Henry Lee, Julie Ting, Henry Ramos, Maria Auxilia, Roy Tumlos, "Fungal sterilization via microwave atmospheric pen plasma (APP)"

9. Joanna Abigael Daseco, Karel Pabelina, Henry Ramos, Armando Somintac, "Hydrophilic and hydrophobic glass by direct current glow discharge low pressure plasma treatment"

10. Michelle Marie S. Villamayor, Henry Ramos, Maricor Soriano, "Comparison of High Dynamic Range images rendered via Response function recovery, Adobe Photoshop CS 5 and Matlab"

11. Angelo Aquino and Luis Ma. T. Bo-ot,"Multivalued Behavior from the Two-Level System Using Homotopy Analysis"

c2. Proceedings of 11th Annual Conference. on Architectural Research and Education, De La Salle-Dasmarinas, Dasmarinas, Cavite, January 2012.

1. *Simon Formoso* and L. Ma. T. Bo-ot, 'Development and Initial Application of a PrototypicalTapping Machine',

D. Manpower trained

D.1 Bachelor of Science (BS) Physics/Applied Physics

1. Catherine Joy dela Cruz, "Deposition of Hydrogenated Amorphous Carbon Thin Films on Glass by Hollow Cathode Discharge Facility", BS Physics Thesis, University of the Philippines Diliman, March 2012.

2. Aja Corinne Corcuera,"Deposition of SiC Thin Films with 3C and 6H Polytypes on Si(111) & Si(100) Substrates Using a Sheet Plasma Negative Ion Source", BS Applied Physics Thesis, University of the Philippines Diliman, October 2012.

3. Joey Kim Soriano, "Atmospheric Plasma Disinfection of *Candida Tropicalis* – Infected Peptone Water Medium", BS Physics Thesis, University of the Philippines Diliman, October 2012.

4. Joanna Abigael Daseco, "Comparative Study on the Use of Different Metal Electrodes in Low Pressure Glow Discharge Plasma Sterilization", BS Physics Thesis, University of the Philippines Diliman, October 2012.

5. Aubrey Faith Mella, "Tin Oxide Thin Film Deposition on Silicon and Glass Substrates Using the Sheet Plasma Negative Ion Source", BS Physics Thesis, University of the Philippines Diliman, October 2012.

D.2 Master of Science (MS) Physics/Materials Science and Engineering

1. Jhoelle Roche M. Guhit, "Efficacy of Glow Discharge Argon and Oxygen Plasma Treatment on the Physical and Chemical Properties of Philippine Coconut Fibers", MS Physics Thesis, University of the Philippines Diliman, March 2012.

2. Christian M. Pesigan, "Surface Modification and Nanostructure Formation by CF_4 Gas Plasma Treatment of Polypropylene", MS Materials Science and Engineering Thesis, University of the Philippines Diliman, April 2012.

3. Beverly Anne T. Suarez, "Thermally Induced Magnetization of Ilmenite ($FETIO_3$) Synthesized from Hot Isostatic Pressure of Titanium Nitride Thin Films on Stainless Steel with Oxygen Ambient Gas", MS Physics Thesis, University of the Philippines Diliman, May 2012.

4. April M. Ulano, "Production of Au-, Nb-, and Si- Ions Using a Sputter-Type Negative Ion Source", MS Physics Thesis, University of the Philippines Diliman, May 2012.

5. Camille Faith P. Romero, "Bacterial Inactivation Using Atmospheric-Pressure Plasma Jet", MS Physics Thesis, University of the Philippines Diliman, May 2012.

6. Ma. Camille Lacdan, "Diagnostics of Microwave-Induced Atmospheric Plasmas ", MS Physics Thesis, University of the Philippines Diliman, October 2012.

PREPARED BY:

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Coordinator, January 25, 2013