## 國外短期進修心得報告

單位:國防大學理工學院應用化學及材料科學系

報告人級職:中校副教授

姓名: 黃其清

進修國家:美國

進修期間: 95.07.25.至 95.12.31

報告日期:96.01.30

職深獲國家栽培,奉准赴美短期進修半年(命令請見附件一), 進入德州農工大學化學工程學系郭育教授(Dow Professor/Dr. Yue Kuo)主持之 Thin Film Nano & Microelectronics Research Laboratory 研究、學習。

郭育教授,為薄膜電晶體(Thin Film Transistors,TFTs)專家,著作等身,擁有多項尊榮的學術頭銜(請參見其個人網頁 http://yuekuo.tamu.edu)。職有幸全程修習其開授之"Microelectronics Process Engineering"課程(該課程之大綱、第一次上課時學生需簽訂之「榮譽信條(Honor Code)」、以及最後一節課發予學生填寫之「教師評鑑表」等請參見附件二),且每週排定固定時間與郭育教授進行一對一討論,並參加其研究群之學術研討;在郭教授嚴格要求下,自覺受益甚多。茲擇要點簡述如下:

- (一)藉修習其開授之課程,職除了瞭解「元件物理」及「VLSI 製造技術」外,對化學工程學理─「質量傳送」、「反應動力學」在「薄 膜製程」之應用有更深刻的認識,對職提昇學能與精進教學亟有幫助。
- (二) 職與郭教授、國內核能研究所學術合作,進行「提昇薄膜電晶體 (Thin Film Transistors, TFTs) 對電磁脈衝 (ElectroMagnetic Pulse, EMP) 與高能輻射防護能力」之研究。並已有初步具體成果 (會議論文之 Short Abstract 投稿證明、該研究之構想、以及職在美

期間對此研究所做簡報之投影片請參見附件三)。目前亦持續進行三邊研究合作,相信可提供國軍 C<sup>4</sup>ISR 電子儀器裝備抗輻射能力之參考。

(三)在美短期進修期間,職之研究生成功合成出「四腳氧化鋅晶鬚(tetrapod ZnO whisker,t-ZnOw)」。據文獻報導,此物質製作之變阻器(Varistor)性能較優,可提供更佳之電磁脈衝防護能力<sup>[1]</sup>;且此物有微波吸收特性<sup>[2]</sup>,甚至可吸收紅外線<sup>[3]</sup>,在軍事上可用做反偵測材料,相當值得研究發展,亦契合職短期進修之目的。職曾與郭教授多次討論,借助其學識與經驗,設計「低壓三區加熱裝置反應器(Low Pressure 3-Zone Heating Element Reactor)」,以期獲得型態尺寸均勻、且結晶度佳、且高產量之t-ZnOw產物。該研究構想業已申請96年度國科會專題研究計畫(計畫書封面、該計畫英文摘要、反應器設計圖、及儀器設備費需求表等請見附件四)。

另外,職有一不成熟之想法,建請 鈞長卓參。德州農工大學內有軍校生部隊(Cadet Troop),事實上,其在 1876 年創立之初即是軍校起家;如今,該校在工程領域排行全美第 14 名(2005 年 US News & World Report),在政治、經濟方面則有布希學院(George Bush School)的加持而聲譽日隆,其發展歷程值得吾人深思。但另一重點是一前校長 Robert M. Gates 於 2006 年底新任美國國防部長;因此,

是否可能在「華美軍事合作」相關會議(談)上能協商以軍售訓練經費及作法送訓國軍人員赴德州農工大學攻讀學位或短期進修,以解決目前及以後可能面臨的准予出國進修員額縮減的問題。

## 參考文獻

- [1] Wu Jun, Xie Changsheng, Bai Zikui, Zhu Bailin, Huang Kaijin, Wu Run, "Preparation of ZnO-glass varistor from tetrapod ZnO nanopowders,"

  \*\*Materials Science and Engineering B59 (2002) pp. 157-161.
- [2] Zuowan Zhou, Longsheng Chu, Shuchun Hu, "Microwave absorption behaviors of tetra-needle-like ZnO whiskers," *Materials Science and Engineering B*126 (2006) pp. 93-96.
- [3] Run Wu, Changsheng Xie, "Formation of tetrapod ZnO nanowhiskers and its optical properties," *Materials Research Bulletin* 39 (2004) pp. 637-645.

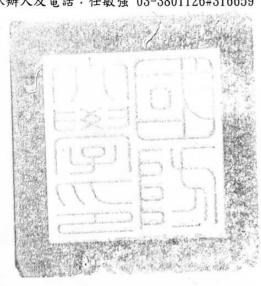
## 國防大學 今

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傳真:(03)4890543

承辦人及電話:任敏強 03-3801126#316659





受文者: 黄丰清中校

發文日期:中華民國95年5月4日 發文字號:集暉字第 0950002224 號

速别:

密等及解密條件或保存期限:

附件:簡歷冊,紙本,1,頁。

主旨:核定貴學院所屬黃其清中校乙員赴美國德州農工大學化學 工程學系短期進修半年,請照辦!

## 說明:

- 一、依據貴學院95年4月20日集錠字第0950000884號文辦理。
- 二、黃員以「94年度國外短期進修員額」赴美國德州農工大學 化學工程學系短期進修,期限自95年6月30日起至95年12 月31日止。

正本:國防大學中正理工學院、國防大學中正理工學院黃其清中校、國防大學總務處

副本:內政部警政署入出境管理局(台北市廣州街15號)、國防部政治作戰局、國防 部主計局、外交部駐美代表處(駐休仕頓台北經濟文化辦事處 11 Greenway Plaza, Suite 2006 Huston, TX. 77046, U.S.A.)、國防部人力司人事管理處、國 防部人力司人力培育處、國軍綜合財務處(均含附件,請查照並登記資料)

校長陸軍二級上將 曾

本件保存

缩影:

檔號:

第1頁,共1頁

國防大學	出國進修人員簡歷冊 表 1
單位及單位編制號	中正理工學院(單位代碼 06G00)
身分證字號	E121256098
軍 種 官 科	陸軍化學
級職	中校副教授
姓名	黃其清
英 文 姓 名	Chyi-Ching Hwang
出生年月日	56 年 9 月 17 日
出 生 地	高雄市
學歷	中正理工學院 79 年班應用化學系學士 成功大學化學工程博士
經歷	連長、助理教授、副教授
任 官 日 期	79年7月2日
進修院校及科系	德州農工大學化工系 ( Department of Chemical Engineering, Texas A&M University )
進修期限	半年
開 學 日 期	95年7月1日
畢 業 日 期	95 年 12 月 27 日
預定出(返)國日期	95年06月30日出國; 95年12月29日返國
上次進修回國日期	無出國紀錄
本階最大年限退伍日	103 年 7 月 1 日
返國後延役預定退伍日	96年12月29日
返國後預派單位及職務	國防大學中正理工學院應用化學系中校副教授
出國期間列管報到單位	國防大學中正理工學院
在台通信地址(中文)	33509 桃園縣大溪鎮瑞源里 24 鄰慈安六村 58 之 1 號
國外通信地址(英文)	Thin Film Nano & Microelectronics Research Lab, 235 J. E. Brown Engineering Building, MS 3122, Texas A&M University, College Station, TX77845-3122, U.S.A.

## **CHEN 475 Microelectronics Process Engineering**

**Description:** This course is for <u>senior level undergraduate</u> and <u>graduate students</u> who

would like to learn microelectronics, especially VLSI, fabrication

processes. It includes fundamental unit processes, thin film materials, and device operation principles for semiconductor transistors and capacitors.

Instructor: Professor Yue Kuo, 235 J. E. Brown Engineering Building,

Tel. 845-9807, Email: yuekuo@tamu.edu http://yuekuo.tamu.edu

Office Hours: Monday and Wednesday 4:00-5:30 pm, other time by appointment.

Prerequisites: Physical Chemistry, Thermodynamics, Transport Phenomena, or

Instructor's permission

Schedule: Monday, Wednesday, Friday 11:30 am - 12:20 pm, Zachry Room 119B

Textbook: VLSI Fabrication Principles, S. G. Ghandhi, 2<sup>nd</sup> Ed., John Wiley & Sons, 1994.

References: ULSI Technology, C. Chang and S. Sze, McGraw-Hill, 1996.

Amorphous and Poly Silicon Thin Film Transistors, Y. Kuo, Kluwer, 2004.

Midterm Exam30%Final Exam30%Report30%Homework, attendance, etc.10%

## Course Outline

- 1. Microelectronics technology and Industry over view
- 2. Semiconductor Material Properties
- 3. Solid State Transistor Operation Principles and IC Structures
- 4. Single Crystal Growth
- 5. Thin Film Depositions
  - Epitaxy
  - Oxidation
  - Chemical Vapor Deposition
  - Physical Vapor Deposition
  - Others
- 6. Doping
  - Diffusion
  - Ion Implantation
- 7. Etching and Contamination Cleaning
  - Wet and Dry
  - Contamination Cleaning
- 8. Lithography
  - Photoresist Materials and Processes
  - Pattern Transfer
- Packaging
- 10. Process Integration (to make a complete IC)

## Term Paper of CHEN 475 10/31/2006

Microelectronics Process Engineering
Instructor: Dr. Yue Kuo <u>yuekuo@tamu.edu</u>

Report Form:

10 pages in MS Word format

single space

total pages of figures < 2 pages, ≥ 3 figures per page

references (max. 1 page)

Criteria for grading: grading standard

1. Content (complete literature search, reviews) 30%

2. Organization (disciplines, rules, differentiation of items ...) 30%

3. Analysis (data, theories, problems, advantages, disadvantages...) 30%

4. Summary 10%

Due date:

Dec. 1, 2006, 5:00pm

submitted by internet attachment to yuekuo@tamu.edu

Topics of choice: Choose only one specific topic or a portion of the topic

1. Materials: High dielectric constant (high-k) materials (with equivalent oxide

thickness less than 5 nm)

2. Epitaxy: epitaxy growth of Germanium on silicon wafer

3. Oxidation: oxidation of polycrystalline silicon (mechanism, processes, ...)

4. Diffusion: diffusion of P, B, etc. in the high-k materials (mechanism, rate, comparison with diffusion in other materials, ...)

5. Ion Implantation: shallow junction ion implantation (for example, < 10 nm)

6. CVD: deposition of high-k films by atomic layer deposition (ALD)

7. Etching: plasma etching of metal nitride or metal oxide films (such as, tantalum nitride, titanium nitride, aluminum oxide, tantalum oxide, etc.) ...

8. Sputtering: reactive sputtering of metal oxides (e.g., hafnium oxide, tantalum oxide, aluminum oxide, etc.)

9. Lithography: immersion lithography (principles, new photoresist, etc.)

10. Other topics upon the approval of the instructor (OLED, transistors on flexible substrates, etc.)

## Americans with Disabilities Act (ADA) Policy Statement

Signature of student

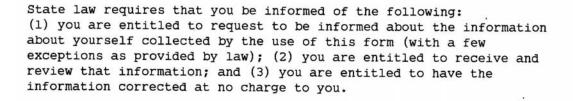
The Americans with Disabilities Act (ADA) is a federal anti-discrimination statute that provides comprehensive civil rights protection for persons with disabilities. Among other things, this legislation requires that all students with disabilities be guaranteed a learning environment that provides for reasonable accommodation of their disabilities. If you believe you have a disability requiring an accommodation, please contact the Department of Student Life, Services for Students with Disabilities, in Room 126 of the Koldus Building or call 845-1637.

Academic Integrity Statement
"An Aggie does not lie, cheat, or steal or tolerate those who do."
Aggie Honor Code and the Honor Council Rules and Procedures on the web <a href="http://www.tamu.edu/aggiehonor">http://www.tamu.edu/aggiehonor</a>
It is further recommended that instructors print the following on assignments and examinations:
"On my honor, as an Aggie, I have neither given nor received unauthorized aid on this academic work."

Date

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## Comments for LEC

What are the most positive aspects of this course?

Grading has been fair and consistent. Circle Yes or No. If No, tell why.

What qualities did you like most about your instructor?

What qualities did you like least about your instructor?

Additional comments about the course and the instructor.

Effects of gamma ray irradiation on the electric characters of thin film transistor (TFT) devices

## **Abstract**

In this project, the electric characters' stabilities of gamma ray irradiated TFT devices and their composing materials will be evaluated under various dosages and dose rates of gamma ray irradiation. Both staggered and inverted, staggered types of hydrogenated amorphous silicon (i.e., a-Si:H) TFTs, as well as the composing materials acting as substrates (i.e., glass or plastic), electrodes (such as aluminum, chromium, nickel, and molybdenum), gate dielectrics (for example, silica, silicon nitride, and tantalum oxide), and a-Si:H will be used for testing, respectively. Gamma ray irradiations will be performed at room temperature with the total dose and dosage rate up to 25 Mrad and 50 Krad/min by using Cobalt-60 as the radiation source. Comparing the electric characters (i.e., mobility, on-current, off-current, leakage current, threshold voltage, and subthreshold slope) of the pre-irradiation TFT devices with those of post -irradiation ones, it is considered that these results may have implications in hardness assurance testing. On the other hand, the composing materials will be examined with electron spin resonance (ESR) after irradiation. In addition, the dependences of annealing conditions and aged time on the recovery property of radiation-damaged samples will also be explored. Judging from these experimental results, coupling with the microscopic analysis, a possible failure mechanism of TFT devices suffered from the gamma ray irradiation may be proposed.

## 1. Introduction

Thin film transistor (TFT), a promising electric device with rapidly increasing usages in numerous fields, has provided itself for applications including displays, imagers, pixel or circuit drivers, and a wide rage of biological, chemical, gases, magnetic, photonic, ionic, and electronic sensors. Therefore, TFTs are rather important for obtaining information and for the purposes of commanding, controlling, communicating, and detecting.

Many scenarios exist that can require TFT devices to operate during or after irradiation because they may be exposed to irradiation for a period of time in aerospace plane and satellite systems, nuclear powder plant systems or weapon systems.

It is known that, generally, the solid-state devices irradiated with high-energy radiations may lead to the following three events:

- Formation of electron-hole pairs
   They result from the photoelectric effect and can generate the leakage current and cause
   the leakage passageway;
- Inverting spin orientation of electrons
   They situate an exciting state and thus against the Hund rule;
- 3) Thermal effect.

The device's temperature is raised by the released energy due to the recombination of electron-hole pairs. The device may be damaged by thermal shock, or a phenomenon of thermal diffusion. These may occur on the interfaces of thin films so as to change the compositions and the properties.

These events may cause the permanent damage or recoverable failure to the electric devices. Although considerable efforts have been performed to investigate the influences of irradiation on the response of other solid-state devices such as MOSs and SOIs, there is no literature reporting the irradiation effects on TFT response. Also, the relevant testing standards and the requirements of military specifications with respect to the TFTs' irradiated-hardness have not been established yet. It is thus worth researching this subject, which may provide useful data to estimate the radiation hardness of TFTs. Besides, it is also important to obtain an understanding of the effects of irradiation intensity on the TFTs' composing materials acting as substrates (i.e., glass or plastic), electrodes (such as aluminum, chromium, nickel, and molybdenum), gate dielectrics (for example, silica, silicon nitride, and tantalum oxide), and a-Si:H so as to assist in improving the radiation-proofing performances of TFTs.

## 2. Research scheme

Although TFTs have been widely used in various areas and the applications are growing continuously, their structures can be divided into three categories: 1) staggered, 2) inverted, staggered, and 3) coplanar. Among them, the former two structures are commonly used in a-Si:H TFTs which possess unique fabrication characteristics: low-temperature, high-throughput, and large-area. Therefore, a-Si:H TFTs with the two structures are selected as the objects in this study.

In the real situation of irradiation, the radiation frequency certainly is continuous and distributes in a wide range. The gamma ray emitted from cobalt-60 is discrete with two characteristic peaks appearing on the electromagnetic frequency spectrum. Even though, cobalt-60 will be used as the radiation source because it is readily available (that is, cobalt-60 is convenient to obtain and easy to control).

The a-Si:H TFTs are manufactured by the Nano Thin Film & Microelectronics Research Laboratory. The basic unit processes include thin film deposition (using sputtering and PECVD techniques), photolithography, wet etching and plasma etching, annealing, and cleaning.

When batches of TFTs have been prepared, their electric properties should be measured prior to irradiation test.

The common requirements of a TFT are high field-effect mobility, high ratio of on current to off current, low threshold voltage, and low subthreshold slope. It is thought that the above items should be measured for the pre- and post-irradiated TFT samples; and thus their radiation hardness can be evaluated by comparing these characters values of pre- and post-irradiation.

When the design or the structure of the a-Si:H TFT is fixed, its performance is dependent on factors such as the properites of composing materials, the dimensions of thin films, and influences of other process steps (for example, the etch method, the substrate temperature, the power and frequency of PECVD, the temperature and time of heat treatment, and so on). Therefore, the composing materials will also be examined with irradiation in this study. Moreover, the properties of the a-Si:H channel and annealing conditions will be selected as the experimental parameters.

By understanding the correlation among the change of electric characteristics, effects of experimental parameters, and the testing results of the irradiated composing materials, it is expected that the radiation hardness of the a-Si:H TFT may be improved and a possible radiation-damaged mechanism may be proposed as well.

## 3. Experimental procedures

The TFT samples will be fabricated by means of the Thin Film Nano & Microelectronics Research Laboratories' PECVD process. (The whole unit steps and the specifications for TFTs should be described briefly.) In addition, the composing materials will also be prepared in a plate, bulk form for testing.

Gamma-ray irradiations will be performed under the delidded condition using Co-60 as the radiation source. The irradiation effects for the TFT samples and individual composing materials can be examined through the variations of the electric characteristics and the analysis of electron spin resonance (ESR), respectively. The irradiation conditions, both the total dosage and the dose rate, will be set in the ranges of 1-25 Mrad and 1-50 Krad/min, respectively. It should be noted that the dose rate and the total dosage can be adjusted by the exposed time and the distance between the irradiated objects and cobalt-60, respectively.

An HP 4140B DC parameter analyzer, inside a black box, is employed to measure the relevant electric characteristics (i.e., mobility, on-current, off-current, threshold voltage, subthreshold slope, etc.) of the pre- and post-irradiation TFT samples at room temperature in air.

The irradiated TFT samples with abnormal electric characters; will be aged (at 25 degrees centigrade, 65% relative humidity) or annealed (less than 400 degrees centigrade) for a period of time. Then, the electric characteristics of the aged TFT samples or annealed ones will

be inspected again to investigate their recoverable properties.

To examine the potential influence of irradiation-induced heat on the damage /failure of TFTs, the thin films and thier interfaces of the irradiated TFT samples will be observed by TEM and analyzed with WDS.

## 4. Possible problems & Solving ways

It is not necessary to design a new TFT structure and to change the fabrication process; this proposal seems to be practical and inexpensive.

However,

- there is a gap between my specialty and this research. I am lack of the knowledge about semiconductor physics and microelectronics.
- 2) These key items, such as preparation of the TFT samples, measurement the experimental data and expertise of the obtained results, have to rely on the Thin Film Nano & Microelectronics Research Laboratory.

## 5. Expected goals of this research

- 1) Offering the attained outcomes to the applications of national defense
- 2) Cultivating mutual cooperation
- 3) Expanding my academic field and enrich my research ability
- 4) At least one paper published on a famous journal

## 6. Further development

If everything goes well and the goals set can be accomplished, I'd like to...

- 1) test other types of TFTs and various potential composing materials
- 2) use synchronous radiation, Alpha particles, or protons as radiation sources
- 3) take the Taguchi method to analyze the relationship among experimental parameters, material properties, and electric characters so as to obtain the optimum radiation hardness of TFTs.



## Effects of radiation on TFT devices



- the purposes commanding, controlling, communicating, TFTs are important for obtaining information and for and detecting.
- It is required that TFTs can operate during or after irradiation (such as aerosapce plane and satellite systems, nuclear power plant systems, or weapon systems, etc.).
- There is no literature reporting the irradiation effect of irradiation on TFTs. The testing standard and required specifications have not been established. S.



## A scheme for my further research

## Effect of gamma ray irradiation on the electric characters of thin film transistor (TFT) devices

Chyi-Ching Hwang Sept. 22, 2006 TAMU





## Purposes of this research



To get an understanding of the radiation effects on TFT's composing materials so as to assist in improving TFT's radiation-proofing performances.

So, it is worth doing this research, right?



# Events result from high-energy radiation

☐ Formation of electron-hole pairs	leakage current & leakage passageway	☐ Inverting spin orientation of electrons

againsting the Hund rule

Heat effect

thermal shock & thermal diffusion



# Selections of research objects (1/3)

- The a-Si:H TFTs possess unique fabrication characteristics:
- 1. low-temperature
- 2. high-throughput
- 3. large-area.

Therefore, a-Si:H TFTs with the 1) staggered and 2) inverted, staggered structures are selected as objects.



# Selections of research objects (2/3)

- The r-ray emitted from <sup>60</sup>Co is discrete with two characteristic In the real situation of irradiation, the radiation frequency certainly is continuous and distributes in a wide range.
- Use of the r-ray emitted from 60Co is readily available because peaks appearing on the electromagnetic-frequency spectrum.
- 1. convenient to be obtained
- 2. easy to be controlled

Therefore, 60Co will be selected as the radiation source.





## Measurements

of electric characteristics for TFTs

- ☐ Common requirements of TFTs:
- 1. high field-effect mobility
- 2. high Ion/Ioff
- 3. low threshold voltage
- 4. low subthreshold slope
- These items are measured by using the HP 4140B DC parameter analyzer.

comparing these characteristic values of preand post-irradiated TFT samples. The radiation hardness can be evaluated by



# Selections of research objects (3/3)

- ☐ Factors affecting TFTs' performances (fixed design & process steps)
- 1. properties of composing materials
- 2. dimensions of thin films
- 3. Process parameters

to examine their irradiation stabilities analyzed Composing materials will also be irradiated by ESR.



## Self-evalution (1/2)

## ☐ Advantages:

- 1. It is not necessary to design a new TFT structure and to change fabrication process.
- 2. This project seems to be practical and inexpensive.

Let's give it a try!





## Self-evalution (2/2)

- ☐ Possible problems & Solving ways
- 1. A gap between my specialty and this research.
- 2. Without key techniques and instruments.

Just study hard and keep touch with you closely!



# Expected goals of this research





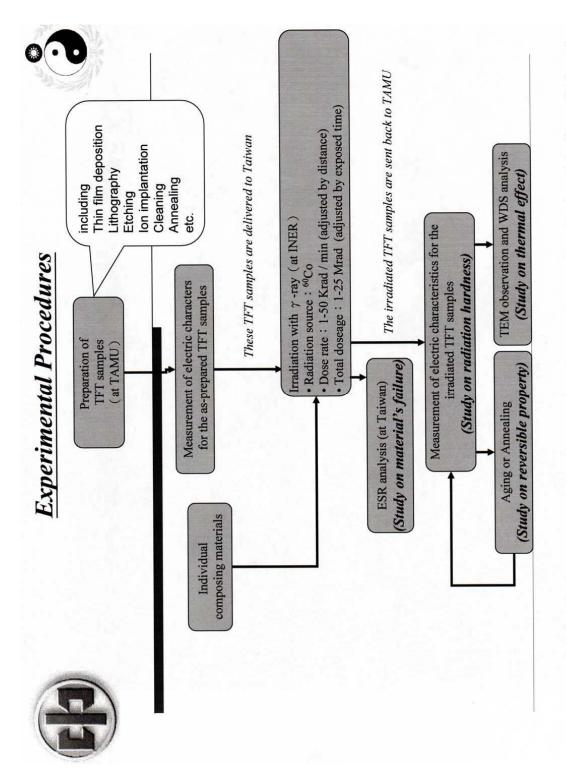
## Further Developments

If everything goes well and the goals set can be accomplished, I'd like to...

	as
f potential	or protons
a variety o	narticles
test other types of TFTs and a variety of potential composing materials	$\square$ use synchronous radiation. $\alpha$ particles, or protons as
test	) ASII

	ar
radiation sources	□ take the Taguchi method to analyze the relationship among experimental parameters, material properties, at
1	ш

pu electric characteristics so as to obtain the optimum radiation hardness of TFTs.



Department of Applied Chemistry Chung Cheng Institute of Technology National Defense University

## Thank you for listening





## 行政院國家科學委員會專題研究計畫申請書

申請條碼:95WFE0900007 一、基本資料: 本申請案所需經費(單選)A類(研究主持費及執行計畫所須經費) 計 畫 類 別 ( 單 選 ) 一般型研究計畫 別個別型計畫 研 究 型 屬工程處 計 畫 鯑 申請機構/系所(單位) 國防大學應用化學及材料科學學系 身分證號碼 \*\*\*\*\*\*098 職 副教授 本計畫主持人姓名 黄其清 稱 文 燃烧法合成多腳氧化鋅晶鬚---反應器研製,產物性質檢測,及前導性應用研究 本計畫名稱 Studies on combustion synthesis of multipod ZnO whiskers---reactor design, product characterizations, and preliminary studies on potential applications 整合型總計畫名稱 身分證號碼 整合型總計畫主持人 全 程 執 行 期 限 自民國 96 年 08 月 01 日起至民國 98 年 07 月 31 日 名 稱(如為其他類,請自行填寫學門) 門代碼 研究學門(請參考本申請書所 附之學門專長分類表填寫) 材料在化工之應用 E0209 究 性 質導向性基礎研究 本年度申請主持國科會各類研究計畫(含預核案)共\_\_\_\_\_件。(共同主持之計畫不予計入) 本件在本年度所申請之計畫中優先順序(不得重複)為第\_\_1。 本計畫是否為國際合作計畫:否 本計畫是否申請海洋研究船:否 本計畫是否有進行下列實驗:(勾選下列任一項,須附相關實驗之同意文件) 人姓名: <u>黃其清</u> 電話:(公) (03)3891716 ext.312 (宅/手機) 0912539206 計 連 畫 址 桃園縣大溪鎮三元一街 190 號國防大學中正理工學院應用化學及材料科學學系 訊 地

申請人簽章:

真

號

碼 (03)3892494

單位系所主管簽章:

執行機關簽章:

表 C001 計畫主持人: 黃其清 申請條碼編號: 95WFE0900007 共 1 頁 第 1 頁

E-MAIL : cchwang1@ccit.edu.tw

十一、研究計畫中英文摘要:請就本計畫要點作一概述,並依本計畫性質自訂關鍵詞。

- (一)計畫中文摘要。(五百字以內)
- (二)計畫英文摘要。(五百字以內)

## **ABSTRACT**

Among all kinds of whiskers, ZnO whiskers are the only one with the multipod shape (i.e., m-ZnOw). They possess many good properties, such as semiconductivity, photoelectric characters, wear resistant, vibration insulation, and microwave absorption. They can be widely applied as both functional and structural materials because of their peculiar shape and single crystalline character. They are also less expensive to produce than other fibers or whiskers used mainly as reinforcement. In our recent study on the synthesis of ZnO powders with different types of morphologies, a combustion method has been developed by using zinc nitrate, metallic zinc and glycine as reactants. It is found that gas phase reaction seems to play an important role in the formation process of rod-like and tetrapod ZnO. The metallic Zn, after evaporation, reacts with O<sub>2</sub> or NO<sub>x</sub>, generated by the decomposition of NO<sub>3</sub> ions, to form ZnO. And its nucleation and subsequent growth lead to the formation of the rod-like and tetrapod structures. In this 2-year project, the multipod ZnO will be directly prepared quickly via a practical and economical oxidation/combustion approach, different form traditional thermal evaporation methods at high temperature and aqueous solution routes with slow production rate. A horizontal furnace with 3-zone heating element together with an appropriate design is utilized as the synthesizing instrument, whereas metallic Zn is used as the starting material. It is expected that the proposed mechanism during synthesis reaction, including evaporation of Zn, following oxidation/combustion, formation of ZnO nuclei, and growth of ZnO, can be controlled via the adjustments of Zn particle size, pressure of gas mixture (N<sub>2</sub>+O<sub>2</sub>) and oxygen fraction, temperature distribution, and the resident time in each zone. As a result, the yield, morphology (i.e., shape, size, and uniformity) and properties (such as specific surface area, PL character, etc.) of the product can be controlled. The goal of the first-year work is to

establish a technique to prepare m-ZnOw, that is, to set up the synthesizing instrument and to define the processing operation procedure. The objective of the second-year study is to examine the effects of relevant experimental parameters as mentioned above on the products? yield, morphology, and properties, which will be analyzed with chemical separation, XRD, SEM, TEM, AES, PL spectra, etc. Moreover, the related researches such as nhancement of m-ZnOw PL character by doping with In<sub>2</sub>O<sub>3</sub> or GeO<sub>2</sub> using this technique? and ? Microwave-absorption behavior of m-ZnOw? will also be preliminarily performed.

Keywords: multipod ZnO whiskers; Combustion Synthesis; Reactor; Morphology; Characterizations

此時直接從氣相傳來的粒子,經與晶粒碰撞後相結合,形成晶粒的一部份而繼續成長。而推動晶粒繼續成長的驅動力(driving force),則來自晶粒尺寸增加所調降之整體自由能, $\triangle G$ 。且須在其附著於任何界面或隨氣流逸散前即完成晶粒成長形成粉體顆粒而沉降。因此反應器的設計與操作,應儘可能讓有利於氣相成核/長晶(gas phase nucleation/growth)的條件發生。因此,反應氣體(鋅蒸氣與氧氣)及氣相產物在反應器內的濃度,應控制在飽和點之上,而此濃度之控制,則與鋅蒸氣濃度、氧氣分壓、操作溫度、管內流路設計有關。然而,必須注意避免氣相產物濃度及操作溫度太高,以致碰撞頻率過於激烈因而干擾正常的成核/成長過程,使產物的形態與尺寸不均勻;亦應避免原本個別且獨立的產物晶粒因成長所增加之尺寸,使其大至足以與附近其他晶粒接觸而聚結(coalescence),復因高溫晶粒間進行內擴散(inter-diffusion)而發生類似燒結(sintering)的現象。因此,管內產氣相產物濃度與溫度分佈對於成核/成長之控制,至為重要。

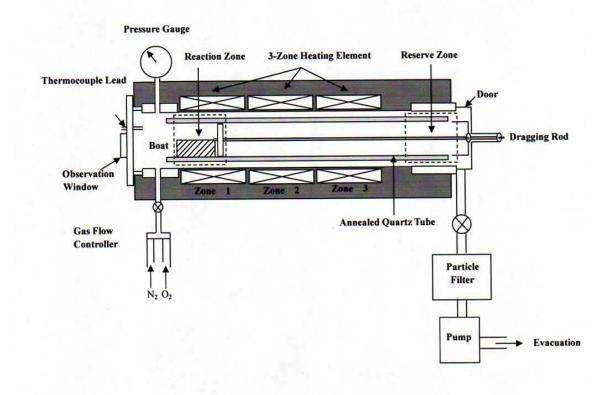


圖 9. 「低壓三區加熱裝置反應器(Low Pressure 3-Zone Heating Element Reactor)」示意圖

儀器及資		本計畫使用「低壓三	1	450,000	450,000	450,000	無
訊設備	裝置反應器	區加熱裝置反應器」					
	/Low Pressure	是一種水平爐管熱壁			1		
	3-Zone Heating	式的反應器。反應器		İ	1		
	Element	本身是以經回火的石			}		
	Reactor	英管、一組「三區加			)	3	
		熱裝置」、用以量測爐			1		i i
		管温度的熱電偶、以		1 1	1		
	1	及抽氣系統的幫浦與		1	1		
		管閥組件所構成。					
		所謂三區加熱裝置,					
		即是一組用來加熱爐					r <sup>ie</sup>
		管之熱阻式加熱器,		1	1		
		環繞於石英管外圍。					
		此加熱器分成三個部					
		分,對爐管的前、中、					
		後等三個區域個別加			1	1	
		溫,以調節爐管的溫			1		
		度分佈。			1		£1:
		氣體混合物			1		
		[N2+O2, 其中 N2 做				)	Ĭ
		為吹淨與稀釋用途,			1		
		O2 與鋅蒸氣反應生成			1		
		ZnO]於爐管前方送入					
		爐管內,反應物置於			J	1	
		同樣已回火之石英載			1		
		皿中,由爐門放進爐					1
	1	管內部,藉由位於爐		}	1		V
		管外圍的三區加熱裝			1		
		置所提供之熱能,達			1		
		到「鋅蒸發、氧化、					
	1	成核、成長」所需之			6		
	1	温度,以進行多腳氧			}		
	)	化鋅晶鬚					
		(multipod-ZnO					
		whisker, m-ZnOw)合					
		成反應。在設計上,		1		1	
		此三區溫度可單獨控					
		制以期獲得所欲之溫					
		度梯度,最高温度可					
		達 1200 ℃,溫控誤差					
		在±5°C間。石英爐管					
		長 120 cm,每區加熱					
		長度約為 25 cm,剩餘 長度做為預備區,在					
	1						
	1	三區加熱裝置到達設					
		定溫度之預熱期間存					
C006	計畫主持人:	放鋅粉及載皿之用; 此長度為 45 cm,應足	条碼編號	: 95WFB09	00007	共 3 頁	第 2 頁
		以遠離第三區,使鋅	11				
	1	粉不受其溫度影響			1		

## 附件五(第六頁)

	合		196-		計			4:	50,000	450, 000	
5 2 年 類別	設備名稱 (中文/英文)	說	明	數	量	單	價	金	額	本會補助	費 來源 提供配合款之機 構名稱及金額
										<b>建</b> 英而 70	将石骨从亚坝
1	<b></b>	72 XX			計						

表 C006 計畫主持人: 黃其清 申請條碼編號: 95WFE0900007 共 3 頁 第 3 頁