行政院國家科學委員會專題研究計畫 成果報告

功率與節能應用之塊狀與磊晶碳化矽的創新研究
研究成果報告(精簡版)

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計 畫 主 持 人：馮哲川

計畫參與人員：碩班研究生：黃品方、練修成、李振昇、黃初旺、洪上宇、
劉宇倫、鄭立琦、郭庭瑋

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行政院國家科學委員會補助專題研究計畫成果報告

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計畫主持人：馮哲川教授

本成果報告包括以下應繳交之附件：
出席國際學術會議心得報告及發表之論文各 份

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執行單位：台灣大學光電工程研究所

中 華 民 國 96 年 12 月 30 日

行政院國家科學委員會專題研究計畫成果報告
計畫編號：NSC 95-2221-E-002-118-
執行期間：95 年 08 月 01 日至 96 年 10 月 31 日
主持人：台灣大學光電工程研究所 馮哲川教授

一、中文摘要

本報告總結國科會計畫 NSC 95-2221-E-002-118-「功率與節能應用之塊狀與磊晶碳化矽的創新研究」(95年08月01日至96年10月31日)的執行成果。在這段時間之內，計畫主持人(暨合作者和學生)已經發表了10篇在SCI上的期刊論文。主要的結果及成就包含了以下幾項：(A) 矽基板上化學氣相磊晶成長的立方體碳化矽的光學性質，(B) 氮化銦鎵/氮化鎵多量子井發光二極體載子侷限的研究，(C) 矽基板上分子束磊晶成長的奈米薄膜氮化鎵的研究，(D) 砷化鎵基板上化學氣相磊晶成長的磷化鋁銦鎵的表面和光學性質，(E)以氫氟酸–甲基氰形成多孔矽的光學和表面及核心技術研究，(F) 不同電介質薄膜沈積和退火中的As化銦鎵/磷化銦多量子井的帶溝藍移，(G)拉曼散射研究的氮化鎵的甲基氰性質，(H)改良溶化技術成長的氧化鋅材料的光學檢測，(I) 矽基板上化學氣相磊晶成長的立方體碳化矽的光學性質紅外線反射研究，(J) 氮化銦鎵/氮化鎵多量子井中的V缺陷的檢測，(K) 提供給20多名研究生的良好訓練。

關鍵詞：碳化矽,化學氣相磊晶,金屬氧化物化學氣相磊晶,微電子,節能功率器件,奈米結構,檢測

Abstract

This report reviews the performance on the project NSC 95-2221-E-002-118-, for 8/1/2005 – 10/31/2006. In this period, P.I. (and collaborators as well as students) have published 10 journal papers to be listed in SCI [1-10], and more conference/proceeding papers. Main results and achievements include: A) Optical Properties of Cubic SiC Grown on Si substrate by Chemical Vapor Deposition [1], B) Study of carrier localization in InGaN/GaN quantum well blue light emitting diode structures [2], C) Pulse Source Injection Molecular Beam Epitaxy and Characterization of Nano-scale Thin GaN Layers on Si substrates [3], D) Surface and optical properties of AlGaInP Films Grown on GaAs by Metalorganic Chemical Vapor Deposition[4], E) Combined optical, surface and nuclear microscopic investigation of porous silicon formed in HF-acetonitrile [5], F) Band-gap Blue Shift of InGaAs/InP Multiple Quantum Wells by Different Dielectric Film Coating and Annealing [6], G) Anisotropic Properties of GaN Studied by Raman Scattering [7], H) Optical Characterization of ZnO Materials Grown by Modified Melt Growth Technique [8], I) Infrared Reflectance Study of 3C-SiC Grown on Si by Chemical Vapor Deposition [9], J) Observation of V Defects in Multiple InGaN/GaN Quantum Well Layers [10], and K) good trainings for more than 20 graduate students.

Keywords: SiC, CVD, MOCVD, epitaxy, microelectronics, energy-saving, power electronics

二、緣由與目的

該計畫為馮哲川教授於92學年加入台灣大學光電所暨電機系後申請的第5個國科會計畫。當今世界文明的高度發展導致了地球資源的快速消耗，其將摧毀人類的將來。因此國際間紛紛提出各種節約能源的計畫，希望能促進人類的永續發展。眾所皆知，台灣本身就是一個缺乏能源的國家，所以節約能源就是一個十分重要的課題。在人類的生活世界裡，電能消耗是很厲害的，例如， 大量電能在各種變電站，電力轉換匣，功能電子元件中被無功地消耗掉。如果我們能發展出新型節能電子元

件，其能工作在高功率和高溫度環境下，那將是一即好的解決方法。碳化矽(SiC)是當今被認可做為"21 世紀高功率元件半導體"的材料。由於生長大塊狀碳化矽結晶的困難和先進的矽科技工業所碰到的瓶頸，它的研究發展經歷過早年的低迷, 但在最近 10 年內，特別是在近些年，碳化矽的研究和發展在全世界引起人們的強烈的興趣。然而碳化矽在台灣的研究發展和工業製造是非常弱的，不像矽基積體電路以及氮化鎵材料和 III-V 材料的發光二極體的案例那樣,在台灣研究發展和工業製造中占有重要市場佔有率。

這個計畫繼續發展碳化矽材料研究以服務於新型節能電子系統。在一年時間裡, 我們將目標集中在研究 3C-、6H-、和 4H-SiC 材料的光學、結構以及表面性質，並探索新的設計，或是改良現有的化學氣相蒸鍍系統。而其目標是為台灣工業建立良好的基礎去拓展碳化矽高溫化學氣相蒸鍍的研究發展。使台灣在碳化矽的研究發展上，趕上全球的先進水平，並做為學術和工業之間的橋樑，以促進台灣在碳化矽節能電子元件工業的發展。

計畫主持人自 1983 年開始一直研究碳化矽並且作出許多極好的貢獻。尤其是由二家著名的科學出版商在最近出版的關於碳化矽的兩本研究專著, 使得他在這一領域佔據着重要的位置。自加入國立台灣大學大約才 4 年的光景，他的研究發展仍繼續遍及許多不同的領域並且成果豐。以他在碳化矽方面的強勢經驗和創新能力, 以及在台灣發展碳化矽事業的宏願，現在和未來向國科會提出的計畫將會一一完成。

拾多名研究生已積極參與先前和本計畫工作並從事於不同的課題得到極好的訓練, 獲取寬帶隙半導體的知識, 各種量測技術, 金屬氧化物化學氣相磊晶理論和實作經驗。13 名碩班學生已畢業並加入了台灣的光電與電子公司過去三年時間裡。12 名碩班學生非常努力工作。5 個國科會計畫的執行已給一年級研究生們打下堅實的基礎並有益於第二年及博士班的進一步學習與深造。

三、研究結果與討論

In performing this project, we have produced 10 journal papers listed in SCI [1-10], and 7 international (abroad) [11-17] and 18 (in Taiwan) conference and proceeding papers, within 2006 only 1-year. The 10-items of them are reviewed and discussed one-by-one item (some contents modified according to the research results) as follows.

1) Optical Properties of Cubic SiC Grown on Si substrate by Chemical Vapor Deposition

We have performed photoluminescence (PL), Raman scattering (RS), Fourier transform infrared (FTIR) transmission and reflectance measurements of a series of CVD-grown 3C-SiC/Si samples with varied film thickness up to 17μm. Typical key or main features on these optical spectra are obtained. It has shown that the thinner (less than 3 lm) 3C-SiC grown on Si have their PL, RS and FTIR optical spectral features, mainly associated with defects. After a certain thickness (>10 lm) of CVD film growth, good and high quality of single crystalline cubic SiC films on Si were obtained, evidenced by PL, RS and FTIR spectra. From comparative measurements for 3C-SiC grown on Si and with Si substrate removed, the stress and strain effects on 3C-SiC/Si are exposed. Tensile stress in the 3CSiC films grown on Si is identified and measured. A relation formulas on two deformation potentials, a and b, for 3C-SiC is deduced from the PL and Raman data. Further interesting variations on the intensities from bound exciton lines of 3C-SiC due to layer tensile stress are described, to be studied further.

2) Study of carrier locali-zation in InGaN/GaN quantum well blue light emitting diode structures

We have investigated the carrier localization of InGaN/GaN QW LEDs with

different structures. Crosssectional HAADF-STEM images observations revealed that the deep localization of excitons (or carriers) originates from the In-rich regions acting as QDs. From TD-PL and EPD-PL experiments, we can expect efficient luminescence in InGaN originates due to carriers radiative recombination through localizing states. The fact that the localized energy depth depends on the structural parameters can be observed from our PL analysis. Generally speaking, stronger localization effect increases the higher internal quantum efficiency at room temperature. Furthermore, the deeper localized potential fluctuations are highly susceptible to rapid state filling as the excitation level is increased.

3) Pulse Source Injection Molecular Beam Epitaxy and Characterization of Nano-scale Thin GaN Layers on Si substrates

A pulse source injection MBE technique was successfully developed to grow extremely smooth thin GaN films. This pulsed-source growth process is different from the atomic layer epitaxy (ALE) process which provides one atomic layer of material growth for each complete source injection period. In the PSIMBE process, multilayers of GaN is grown for each source injection period. Calculations based on the growth rate, indicated that the pulsed source injection MBE process grew eight GaN lattice constants of material for a 3 min source injection period. The PSIMBE process has a faster growth rate than the ALE process. It is believed that this pulsed source injection process provides a sufficiently long relaxation time to allow high mobility Ga atoms to move to the nuclear sites at island boundaries and thereby form layer by layer 2-dimensional growth. Therefore, it can be treated as a migration enhanced growth process. However, detailed growth studies need to be continued to completely understand the growth mechanisms for further process optimization.

4) Surface and optical properties of AlGaInP Films Grown on GaAs by Metalorganic Chemical Vapor Deposition

We have grown two sets of $(Al_xGa_{1-x})_{0.5}In_{0.5}P$ thin films on GaAs substrates with x near 24% and 18%, respectively, by low pressure MOCVD and performed a combined characterization by Nomarski microscopy, atomic force microscopy, photoluminescence, and Raman spectroscopy. Nomarski microscopy observe the cracking line and twist defect line on the surface of the first set of AlGaInP films, while the second set have almost clear surface. AFM revealed the cross-hatch dislocation patterns from the first set of films but no cross-hatch observed from the second set of films. Room temperature PL and Raman spectra showed corresponding variations. The degree of variations in compositions and film quality with the growth conditions were found from the spectral analyses. Raman spectral features are more sensitive to the sample growth parameter variations. The line shape analysis of line width, integrated intensity ratio and spatial correlation model fitting leads to information about the order of the sample crystalline quality. The optimum growth conditions have been obtained from our comprehensive analyses. These nondestructive techniques offers us a better understanding of MOCVD-grown $In_{0.5}(Ga_{1-x}Al_x)_{0.5}P$/GaAs and a useful way to optimize the growth parameters of high quality quaternary semiconductor materials.

5) Combined optical, surface and nuclear microscopic investigation of porous silicon formed in HF-acetonitrile

We have prepared porous silicon (P-Si) on various Si substrates by photoelectron-chemical etching of silicon in HF acetonitrile (MeCN) solutions, a new type of HF solutions for forming P-Si. Different types of Si wafers, Si(100), Si(111) and polycrystalline Si, with different resistivities, were employed to produce 10–30 Am thick P-Si layers. A combined optical, surface and nuclear microscopic assessment of these P-Si layers was performed using photoluminescence (PL), Raman scattering, X-ray photoelectron spectroscopy (XPS) and Rutherford backscattering spectroscopy (RBS). Red

wavelength PL was emitted from these P-Si samples, prepared from (100) Si and polycrystalline Si, which showed clear porous structures by SEM. The PL emission intensities are dependent on the properties of Si wafers prepared for forming P-Si. But there is no visible light emitted from samples prepared from (111) Si wafers in which no porous structures were formed. All samples exhibit dominant Raman bands with a slight downshift and a broader/asymmetric shape compared to the c-Si Raman band. The extent of Raman line shape variation is found to be a function of the resistivity of the substrate. XPS studies reveal different surface and oxidation properties from these P-Si layers. Strong C 1s, O 1s, O KLL and C KLL signals together with Si 2s and Si 2p were observed. The amount of contaminant C is varied with q. The surfaces of these samples are shown to be largely SiO2 with different amount of elemental Si, which increases with decreasing q of single crystalline Si. P-Si obtained by etching of poly-Si showed the largest amount of elemental Si on the top. RBS data reveal the existence of F on the top in addition to Si, O and C. Simulations of RBS data indicate that there exists a large difference in the thickness and composition of the P-Si layers grown from different wafers. Ion channeling measurements distinguish the crystalline variations between the porous and non-porous regions and from sample to sample. It is worthy to make further investigation on P-Si prepared in HF-MeCN solutions, their luminescence mechanisms and various properties.

6) Band-gap Blue Shift of InGaAs/InP Multiple Quantum Wells by Different Dielectric Film Coating and Annealing [6]

In this research, summarizing results of photoluminescence and SIMS characterization for three kinds of InGaAs/InP samples are obtained: 1] The band gap of quantum well structure of InGaAs/InP can be tailed by IFVD at certain conditions. 2] The blue shift of the band gap of InGaAs/InP structure depends on the dielectric layers, such as SiO_2, Si_3N_4, SOG, and annealing condition. 3] The blue shift of the band gap of InGaAs/InP structure also depends on the combination between cladding layer and dielectric layer. 4] The biggest blue shift of 30 meV can be reached with the combination of InP cladding layer and Si_3N_4 dielectric covered layer annealed at 800 ^oC for 10 s.

7) Anisotropic Properties of GaN Studied by Raman Scattering

The interface properties of the phonon anisotropy property of GaN wurtzite crystal have been studied using the angular dependent Raman spectroscopy both theoretically and experimentally. The polarized Raman scattering spectra were recorded from the cross-section of c-axis oriented GaN films as a function of the angle between the polarization direction of the incident laser and the film normal direction in three different polarization configurations, the perpendicular, parallel, and un-polarized configuration. The Raman intensity of A1(TO) showed a sinusoidal dependence on the rotating angle, as did the E1(TO) mode. The theoretical fit is weighed in the susceptibility contribution and the phase differential of the different vibrating elements. The intensity of E2 is quite different to the A1 and E1, because E2 mode has the same vibrating elements but the A1 and E1 have two types of the elements and will have the phase differential contributing to the susceptibility of the polarized angle.

8) Optical Characterization of ZnO Materials Grown by Modified Melt Growth Technique

We had performed the optical characterization on a series of ZnO crystal wafers, undoped and doped with various of dopants, including Ga, Er, Co, Ho, Fe, Mn, and co-dopant of Mg-Li, have been grown by a modified melt growth method, and characterized by optical techniques of Raman scattering, photoluminescence and optical UV-visible transmission. Raman spectroscopic measurements have identified the wurtzite structure with (0001) orientation along the normal surface direction for wafers after cutting and polishing. The observation of weak Raman signals from selection-rule-broken-modes indicated a

small degree of imperfection in the crystalline structure of experimental ZnO samples. RT PL have showed most of ZnO bulk wafer samples with the major band edge emissions from wurtzite ZnO crystal, which are affected only slightly by dopings, and a lower energy emission band depending on the dopants. UV-visible optical transmission measurements revealed the optical absorption gap varied greatly, depending on dopants in ZnO, although they show band edge emissions closely. A large shift in the optical absorption edge has been observed from these Mn/Co/Fe-doped bulk ZnO crystals in comparison with un-doped ZnO, with information of the impurity levels of these dopants obtained. This interesting phenomenon is significant and can be useful in the fabrication of optical waveguide devices based upon ZnO materials. In this study, the computer fitting processes have provided an easy and essential way to deepen our understanding on the optical properties of the ZnO bulk materials.

9) Infrared Reflectance Study of 3C-SiC Grown on Si by Chemical Vapor Deposition

Infrared (IR) reflectance of 3C-SiC on Si (100) has been studied both theoretically and experimentally. Influences of structural and electric properties on reflectance are investigated in detail. Information can be inferred about roughness at film surface and possible presence of interfacial layer at film-substrate interface. A damping behavior of the interference fringes away from the reststrahlen band and a dip or notch within the "flat top" are observed from some samples while they does not occur in high quality 3C-SiC/Si samples. We performed an extensive theoretical simulation study of the IR reflectance from the cubic SiC/Si system with various structural features. We assumed an interfacial transition layer existed between SiC-Si and a rough surface to be responsible to the fringe damping. The dip-notch feature can be demonstrated by a three-component effective medium model. This work shows that IR spectroscopy can be a useful tool to reveal the structural features on 3C-SiC/Si and other material systems.

10) Observation of V Defects in Multiple InGaN/GaN Quantum Well Layers

We have observed an InGaN/GaN multiple quantum well (MQW) layer by high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) and conventional transmission electron microscopy (CTEM), and established the structure of V defects, confirming the formation mechanism of the V defects which was previously proposed by Shiojiri et al. 1) Specimen used in this experiment comprised five 4-nm $In_{0:18}Ga_{0:82}N$ QWs spaced with 40-nm GaN barriers, which were deposited at a reactor temerature of 800^{o}C on the 2-mm GaN layer. The MQW was covered with the 50-nm p-AlGaN capping layer. In HAADF-STEM images the $In_{0:18}Ga_{0:82}N$ QWs appeared as thin bright bands and the GaN barriers appeared as thick dark bands between the QWs. 2) In the MQW, V defects were observed nucleating on a threading dislocation (TD) crossed with the InGaN QW just above the underlying GaN layer. HAADF-STEM gave undoubted evidence that the V defects have a thin six-walled structure with InGaN/GaN {10_111} QWs. 3) The segregation of In atoms was observed at the starting points of the V defects, by CTEM and HAADF-STEM. This might work as the In-rich mask which induced the {10_111} facets on the GaN crystal grown at the low temperature, as proposed in the formation mechanism of the V defects. 4) The TD incorporated with the V defect propagated to the free surface through the V defect buried with the cap layer. This provides evidence for the fact that the InGaN QWs and GaN barriers were connected in good lattice coherence with each other on the {10_111} interfaces, to make the cellurally same structure as a whole. 5) The corners connecting the {10_111} interfaces on the walls of a V defect with the (0001) interfaces in the main MQWs were curved. This is explained as a result of the layer-by-layer growth on the (0001) and {10_111} surfaces where each monolayer did not cover over its under-monolayer for lack of atom. The successive growth of these monolayers formed an interface with step-wise lattices

near the corner, which is observed as the curved corner of the QW in the low-magnified HAADF-STEM images.

In additions, we have produced 7 international (abroad) and 18 (in Taiwan) conference and proceeding papers, within 2006 only 1-year, with some examples listed [11-17].

(四) 成果自評

P.I. has joint NTU since 2003 Fall and in performing the NSC 計畫, NSC 95-2221-E-002-118 "**功率與節能應用之塊狀與磊晶碳化矽的創新研究**"(95 年 08 月 01 日至 96 年 10 月 31 日. P.I. (and collaborators as well as students) have published 10 journal papers (SCI), plus 7+18 conference papers/reports. All the works would stand among the world advanced class levels. Continual penetrating research will be further processed. The outstanding results and achievements include:

A) Optical Properties of Cubic SiC Grown on Si substrate by Chemical Vapor Deposition, B) Study of carrier locali-zation in InGaN/GaN quantum well blue light emitting diode structures, C) Pulse Source Injection Molecular Beam Epitaxy and Characterization of Nano-scale Thin GaN Layers on Si substrates, D) Surface and optical properties of AlGaInP Films Grown on GaAs by Metalorganic Chemical Vapor Deposition, E) Combined optical, surface and nuclear microscopic investigation of porous silicon formed in HF-acetonitrile, F) Band-gap Blue Shift of InGaAs/InP Multiple Quantum Wells by Different Dielectric Film Coating and Annealing, G) Anisotropic Properties of GaN Studied by Raman Scattering, H) Optical Characterization of ZnO Materials Grown by Modified Melt Growth Technique, I) Infrared Reflectance Study of 3C-SiC Grown on Si by Chemical Vapor Deposition, J) Observation of V Defects in Multiple InGaN/GaN Quantum Well Layers, and K) good trainings for more than 20 graduate students.

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行政院國家科學委員會補助專題研究計畫成果報告

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※ 功率與節能應用之塊狀與磊晶碳化矽的創新研究 ※
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計畫類別：個別型計畫
計畫編號：NSC 95-2221-E-002-118-
執行期間：95 年 08 月 01 日至 96 年 10 月 31 日

計畫主持人：馮哲川教授

本成果報告包括以下應繳交之附件：
出席國際學術會議心得報告及發表之論文各　份

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執行單位：台灣大學光電工程研究所

中　華　民　國　96 年 12 月 30 日

出席國際學術會議心得報告

計畫編號：NSC 95-2221-E-002-118-

執行期限：95 年 08 月 01 日至 96 年 10 月 31 日

主持人：台灣大學光電工程研究所 馮哲川教授

出席國際學術會議心得報告及發表之論文

P.I. has attended four conferences in China, 1-19 July 2007: i] International Conference on Smart Materials and Nanotechnology in Engineering, 1-4 July, Harbin. Two oral presentations were presented: “Luminescent Properties of Cadmium Telluride Films Prepared by Molecular Beam Epitaxy”, and “Luminescent Properties of YAG Fluoride Single Crystals”; ii] 5th International Conference on Surface Engineering, 7-10 July, Dalian. One oral presentation was given: “Surface Chemical Properties of Mg doped GaN heteroepitaxial films on Sapphire”; iii] IMR Symposium on Materials Science & Engineering-Frontier & Future of Advanced Functional Materials, 11-13 July, Shenyang. Two invited presentations were given: “III-Nitride quantum materials and nano-structures for new generation light emitting devices”, and “ Advanced SiC power semiconductor materials in the 21st century”; iv] 15th International conference on composites/nano engineering, 15-19 July 2007, Haikou. One invited presentation was given: “Nano-structural Characteristics of InGaN/GaN multiple quantum well light-emitting diodes grown by metalorganic chemical vapor deposition”. All were warmly welcome by audience, and all presentations have been published in conference proceedings.

補助機構及經費:台灣大學光電所展翅計畫+國科會 NSC 95-2221-E-002-118-, 145,126 元.

P.I. has attended the 2006 IEEE INTERNATIONAL CONFERENCE ON SEMICONDUCTOR ELECTRONICS (ICSE), 29 November - 1 December, 2006, Kuala Lumpur, Malaysia.

補助機構及經費: **2006 年台積電贊助半導體合作研究專案** 30,063 元,

Oral presentation was given: “Synchrotron Radiation X-ray Diffraction and X-ray Photoelectron Spectroscopy Investigation on Si-based Structures for sub-micron Si-IC Applications”, which has been published in ICSE 2006 Conference Proceedings, p. 981-984.

P.I. has also attended the 3rd International Workshop on Nanophysics and Nanotechnology (3rd IWONN), 609 December 2007, HaLong, Vietnan. 補助機構及經費: **2006 年台積電贊助半導體合作研究專案+台灣大學光電所展翅計畫** 45,160 元. Oral presentation was given: “Surface science and optical investigation on nano-scale thin films for Si-based 65-nm intergrated circuits application”, which has been published in the 3rd IWONN conference proceeding, p. 716-718.

Through friendly exchanging and discussion with many scientists in these conferences, many knowledge and significant ideals have been obtained and raised. These are very helpful to us to open the eyes and deepen the scientific and technological sense. Indeed, some knowledge obtained from these conferences improves or deepen several important ideals in the research items listed in this research plan and for several papers or manuscripts.