



異質金屬雷射鐸接介金屬特性分析

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研究背景

- 交通工具逐漸走向**電動化**的趨勢，電池儼然成為極其重要的部分，鋁(Al)-銅(Cu)異質材料接合技術廣泛運用在電池產業。
- Al-Cu兩者在物理性質上具極大差異，造成鐸件的品質下降；為了降低上述的影響，高能量密度且高效率的**光纖(Fiber)雷射(LASER)鐸接**，能降低上述的問題。
- 但 LASER鐸接需要面臨Cu的高反射率，且LASER接合後會在介面形成不同型態之**介金屬化合物(Intermetallic compounds, IMCs)**，對接合點的機械性質及導電性皆有負面影響。
- 本研究將使用Fiber LASER鐸接並透過不同鐸接參數及填料添加進行鐸接，並分別針對鐸接參數設計、機械性質、微組織及相鑑定等進行實驗及數據分析，進而了解**IMCs成因及對鐸接件性質的影響**。
 - Key Words:**鋁-銅鐸接、光纖雷射、介金屬化合物、機械性質



實驗步驟

鐸接參數設計

填料備製

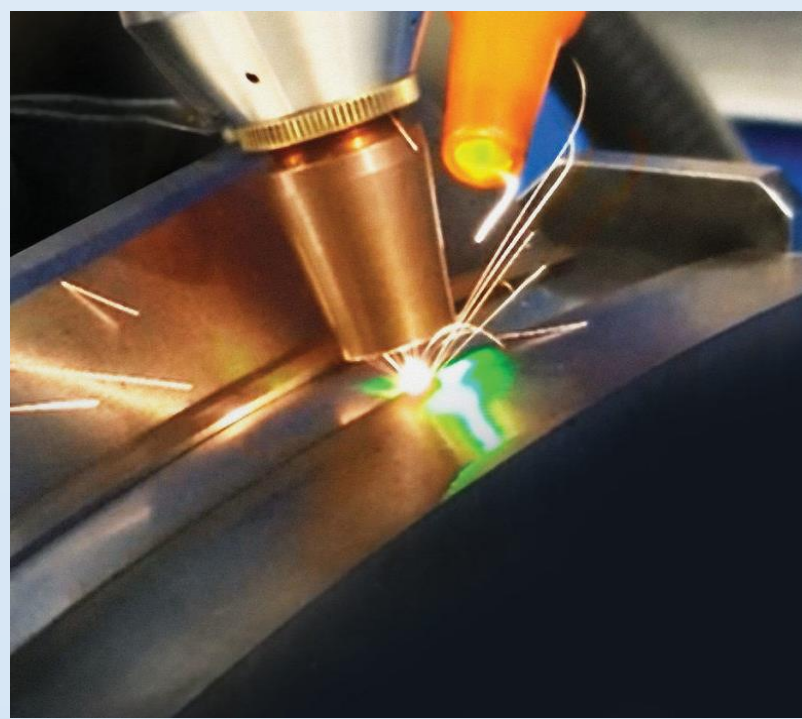
Fiber LASER鐸接

1. OM
2. SEM/EDS
3. FIB&TEM

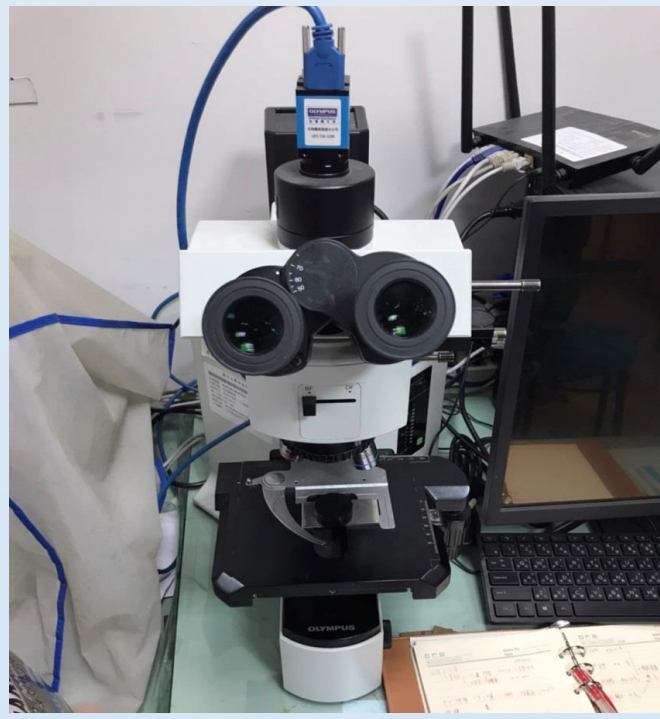
拉伸測試
MTS

1.破斷面觀察
2.機械性質測試

實驗儀器



Fiber Laser



光學顯微鏡



掃描式電子顯微鏡



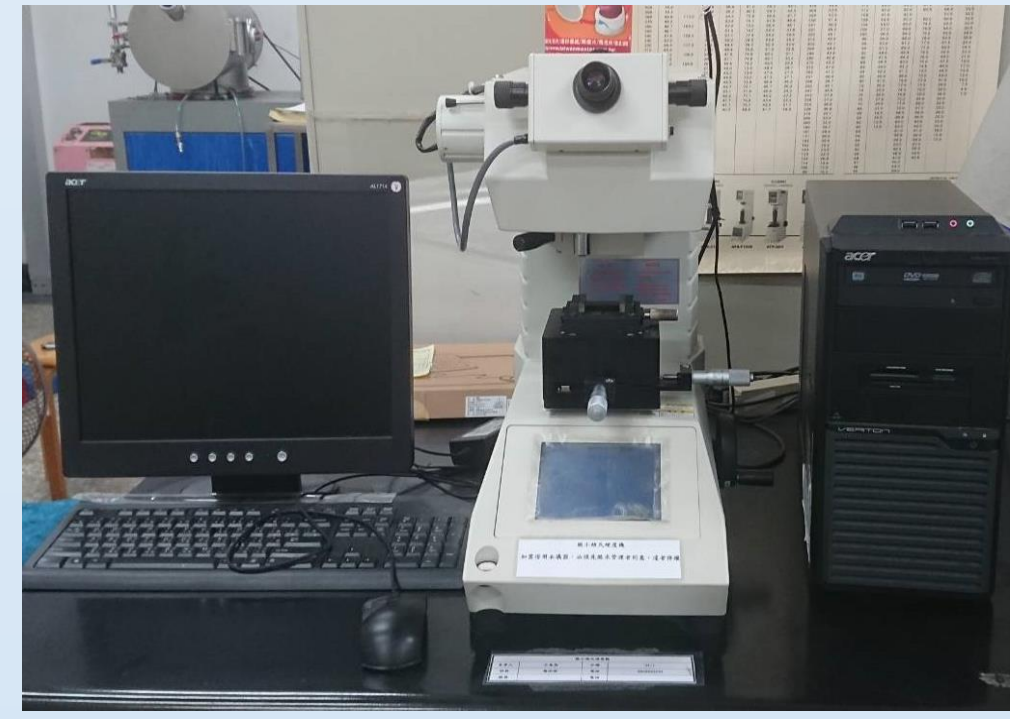
FIB



穿透式電子顯微鏡



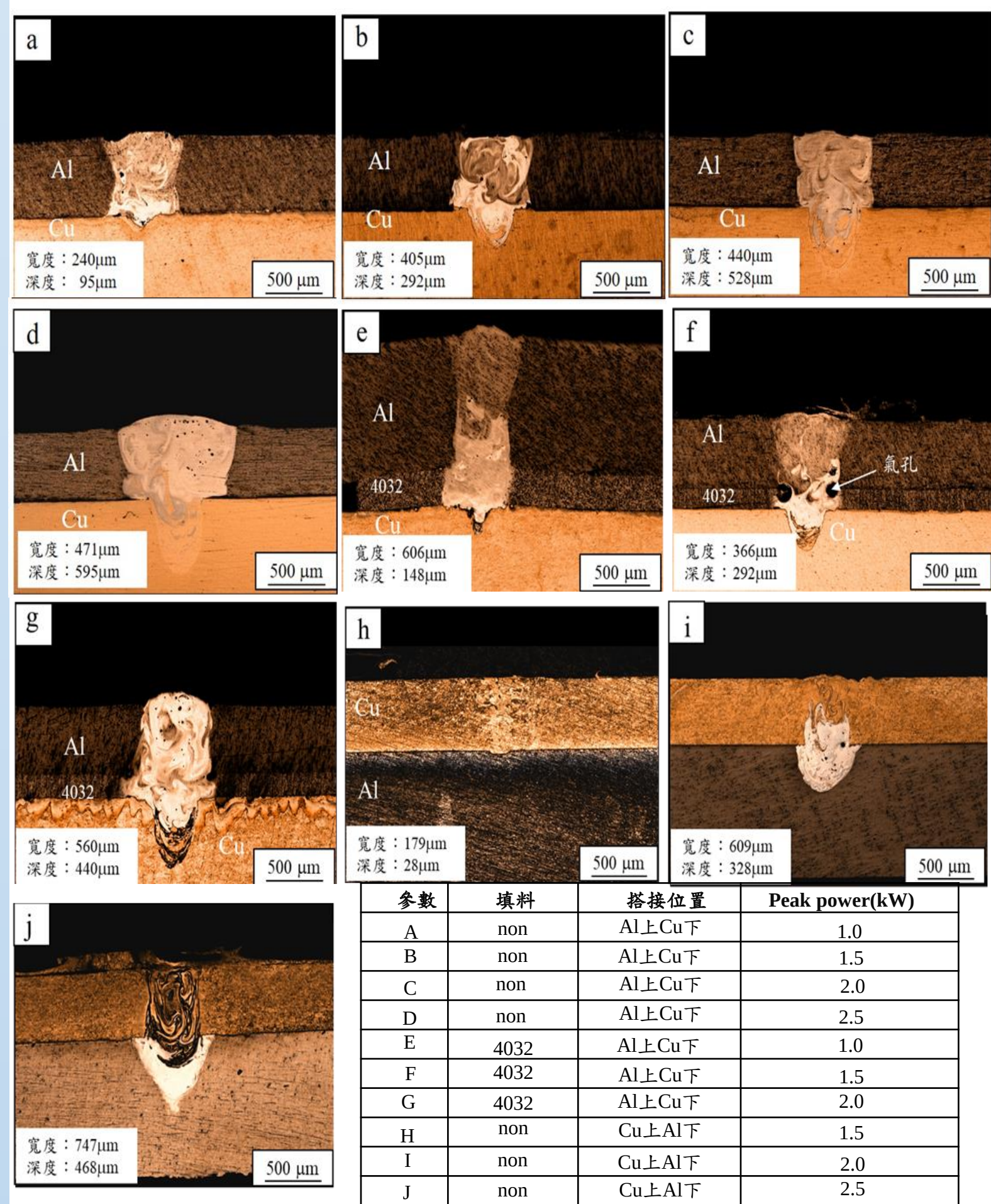
MTS拉伸試驗機



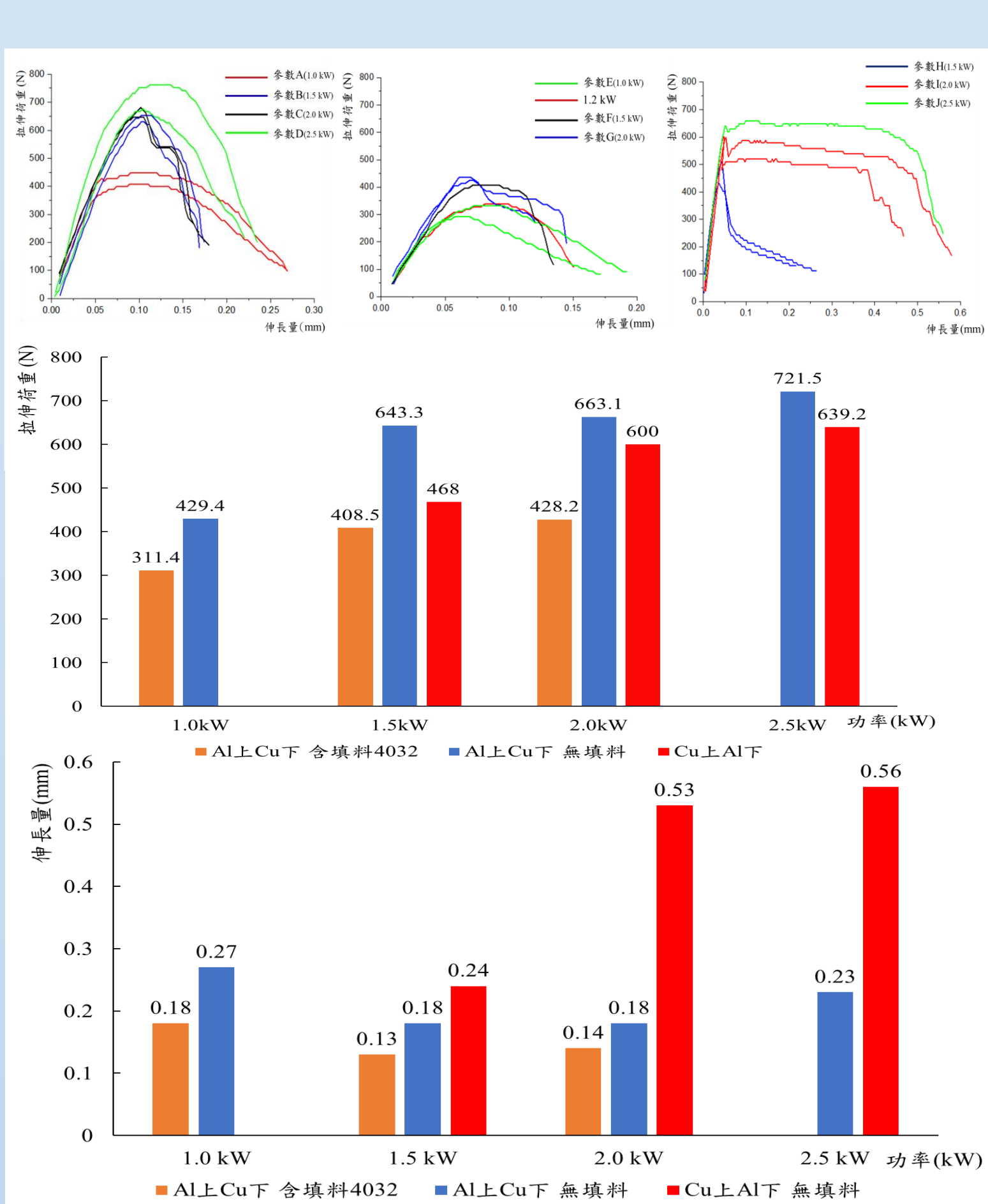
Vickers hardness

結果與討論

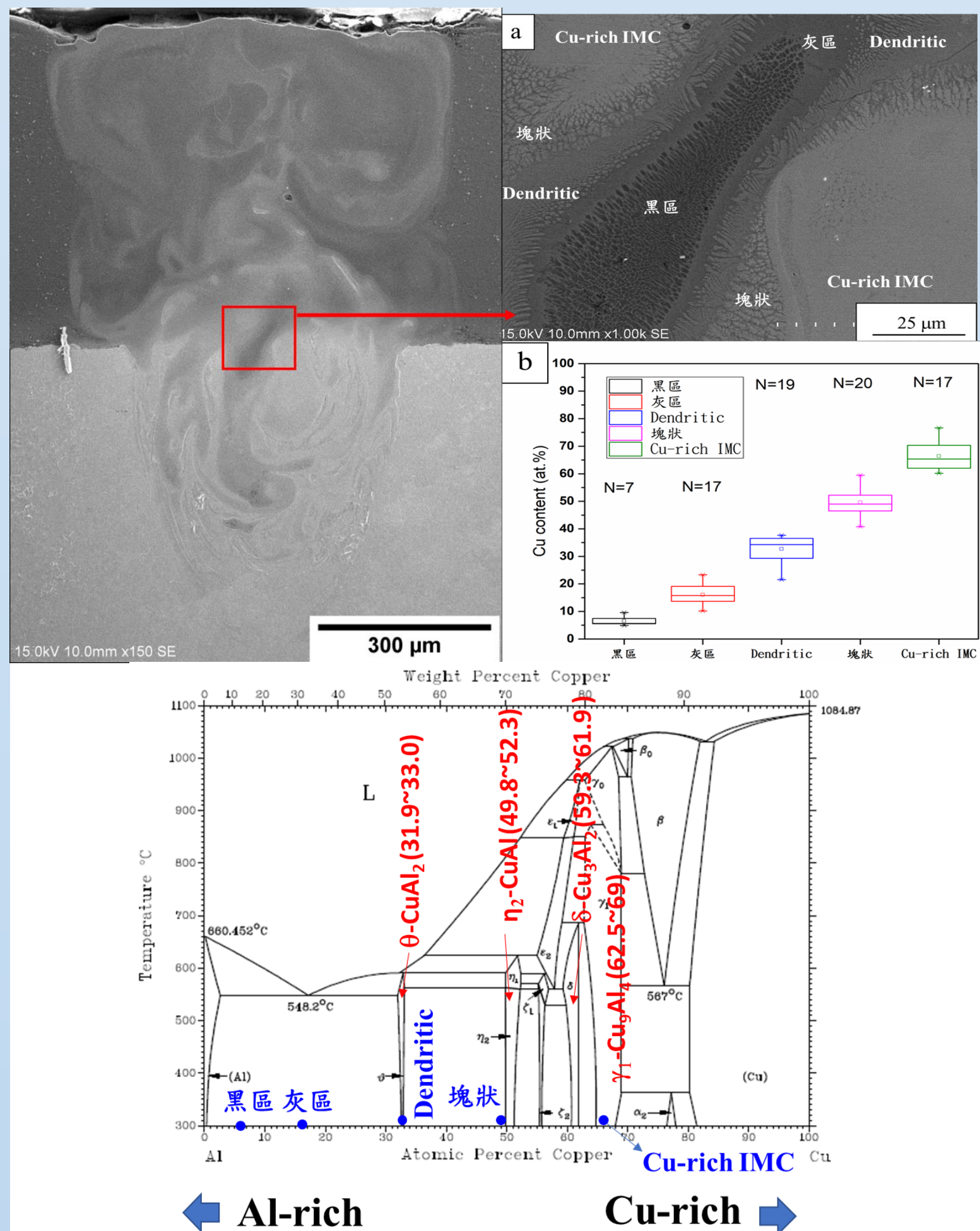
鐸接參數設計及鐸接後橫截面觀察



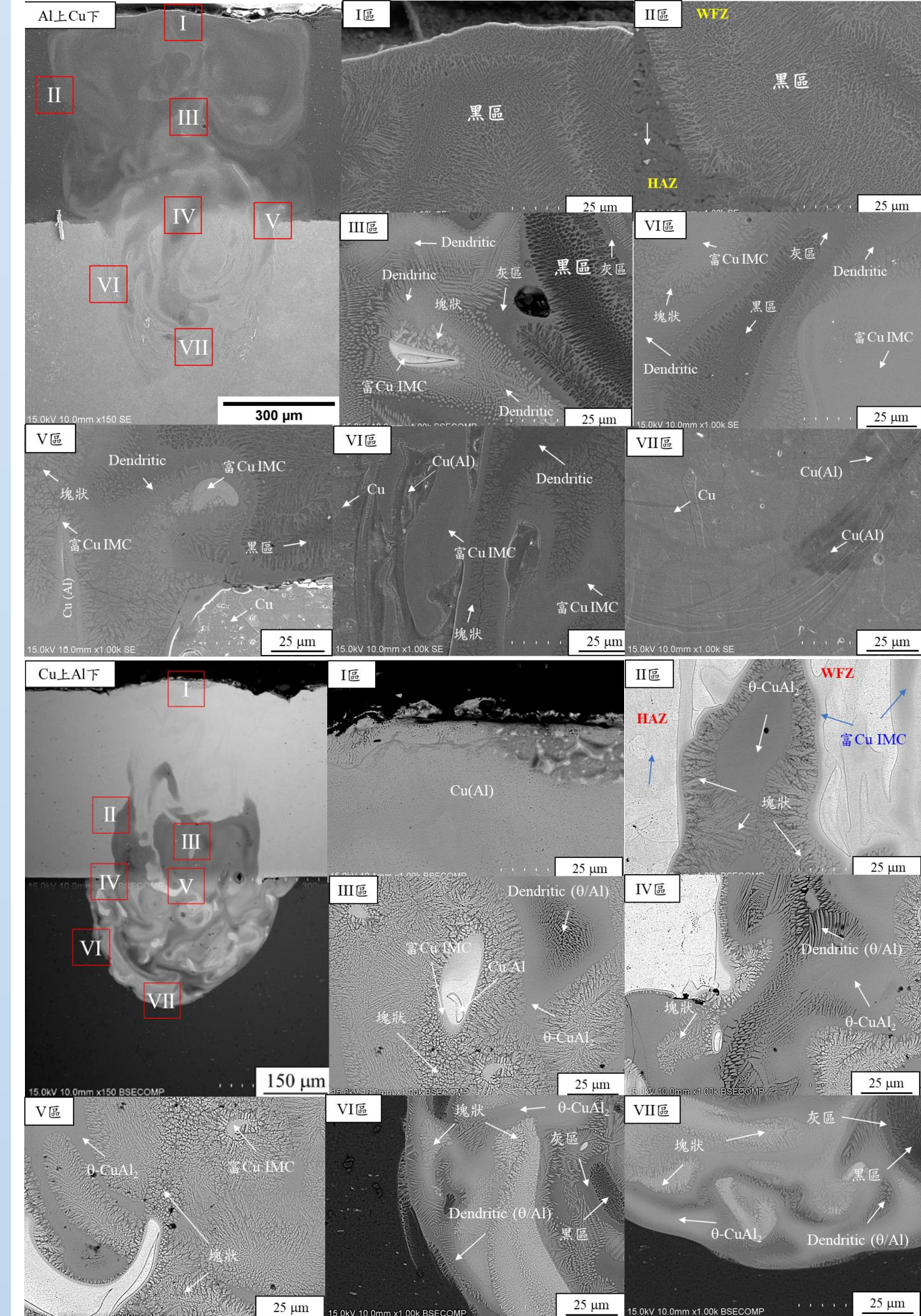
鐸接參數與拉伸荷重及伸長量關係圖



鐸後微組織觀察(關聯性相圖)



鐸後微組織觀察(鐸道各區域觀察)



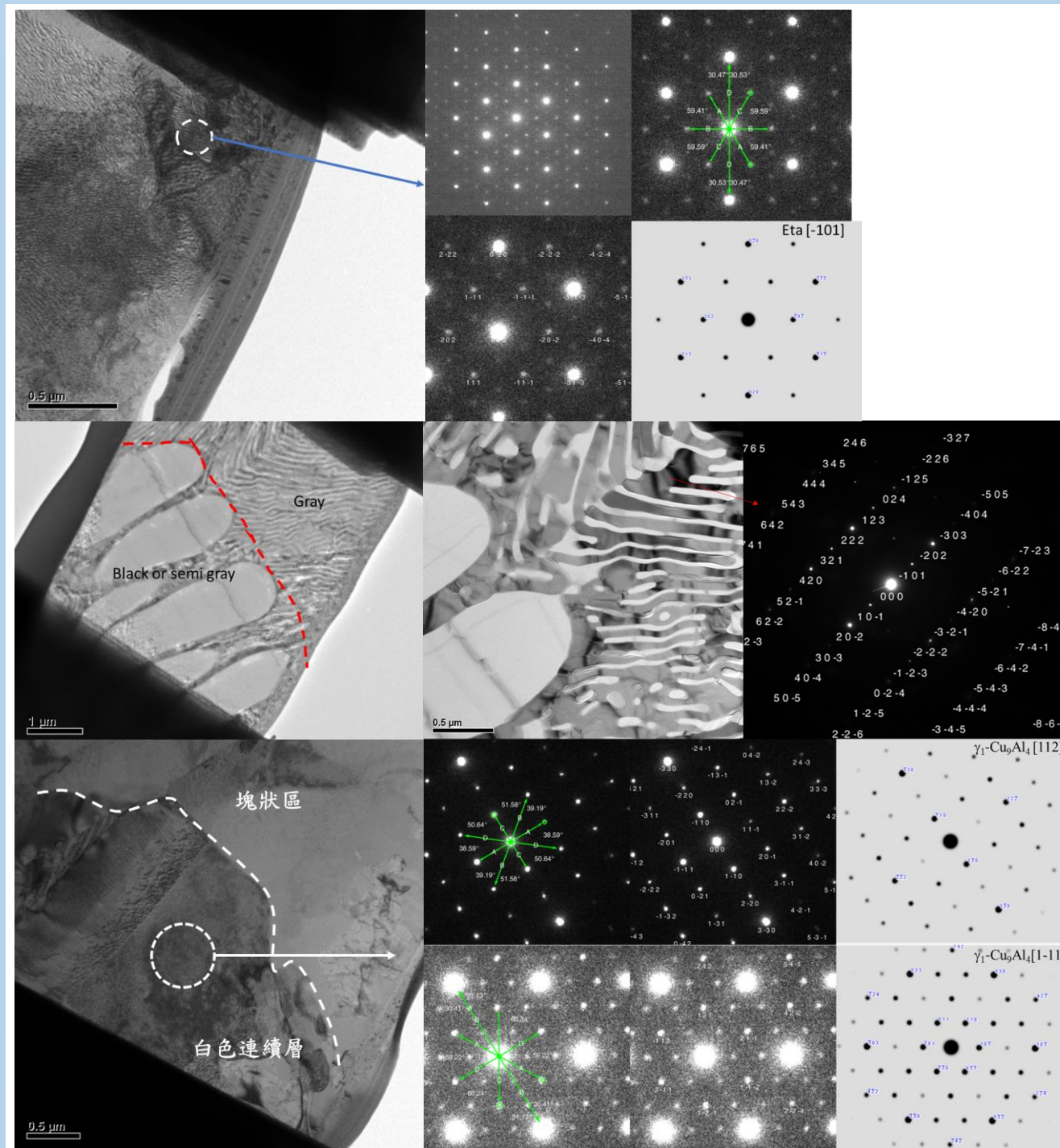
- 鐸接參數設計如表，其他參數:波長:1064nm、spot size:0.125mm、鐸接速度:275mm/s、無保護氣體。
- 當功率達到2.0kW以上，可以使鐸件之鐸道深度具292μm以上，其中以Al上Cu下無填料鐸道深度最佳，Al、Cu鐸接其功率需大於2.0 k具較佳深度、深寬乘積。

不同功率中拉伸荷重Al上Cu下無填料具較佳表現，Cu上Al下搭接方式雖具較佳延展性，但其拉伸荷重相對不高，4032 Al-Si合金填料的添加，對延展性及拉伸荷重的幫助有限。

根據不同形貌特徵定義五種特徵組織，從中間富鋁且對比較黑之區域定義為**黑區**，隨著銅含量增加依次分別為**灰區**、**樹枝狀區 (Dendritic)**、**塊狀結構區**及**富銅介金屬化合物區 (IMC)**等五區。

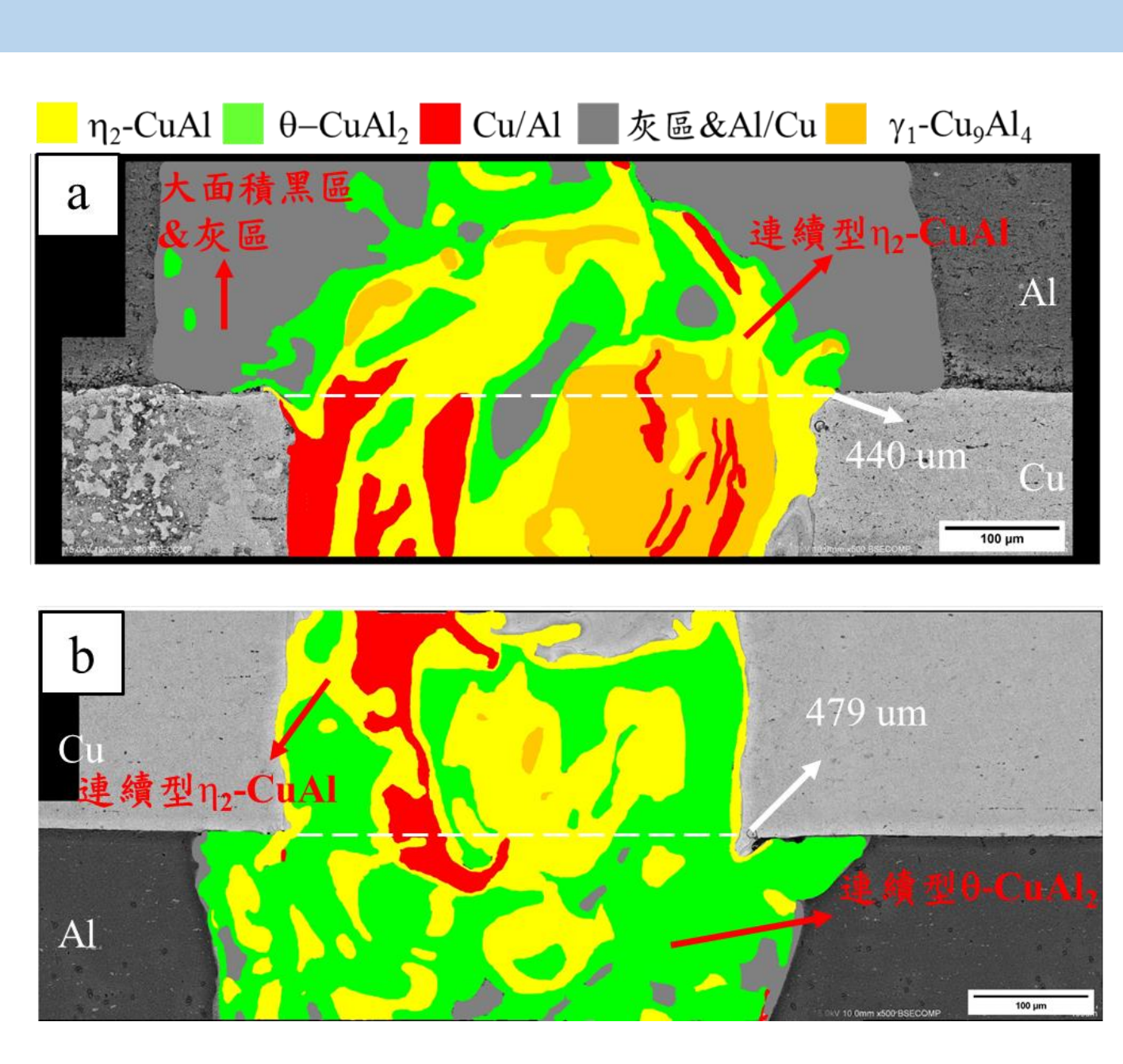
- Al上Cu下無填料鐸道組織，**鋁側鐸道**主要以**富Al黑區及灰區組織**為主；**鐸道Al/Cu界面**則可發現**各種特徵組織**；**銅側鐸道**則以**塊狀組織及富Cu IMC**等富銅組織為主。
- Cu上Al下，在**接近銅側內部Cu(Al)混溶區**界面有**連續富Cu IMC塊狀組織**。Cu/Al界面部位有大面積**θ-CuAl₂**相包覆著硬脆**γ₁-Cu₉Al₄**及**η₂-CuAl**。

鐸道TEM相鑑定



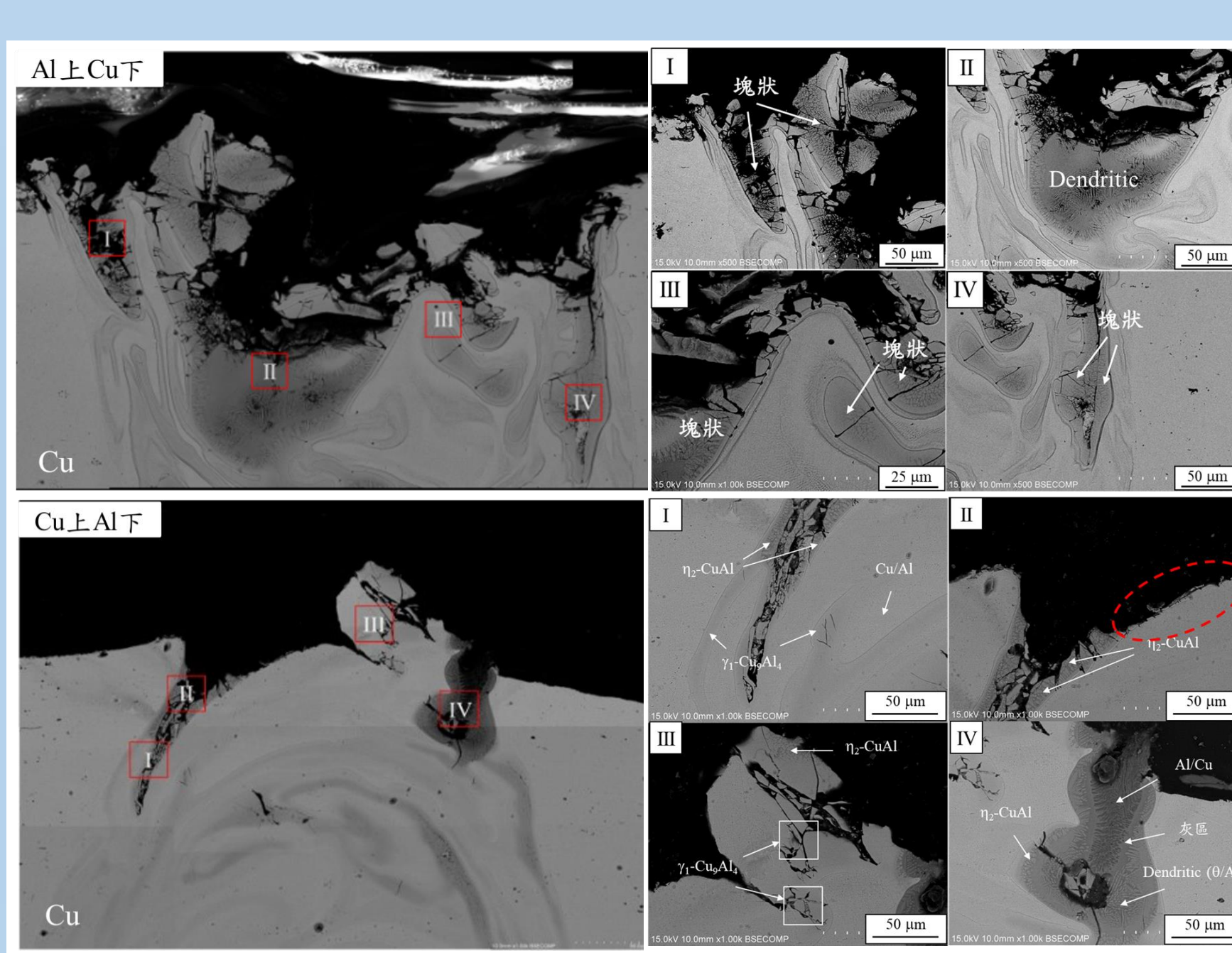
根據TEM相鑑定可以得知，**白色連續層**為**γ₁-Cu₉Al₄**相，**塊狀區**為**η₂-CuAl**，**灰區**為**Al(Cu)固溶體及θ-CuAl₂**兩相層狀共晶組織

鐸道IMCs分布



- Al上Cu下因連續η₂-CuAl、γ₁-Cu₉Al₄分佈在界面處導致其拉伸荷重較高、延展性較低，
- Cu上Al下則是大量θ-CuAl₂相包覆著硬脆相η₂-CuAl導致其延展性較高、拉伸荷重較低。

破斷面與IMCs關聯性



- 破斷面位置主要發生在原本銅側鐸道部位。
- Al上Cu下多處部位呈現破碎狀等脆性破裂特徵形貌，裂痕主要發生在**塊狀組織**。
- Cu上Al下裂痕則出現在η₂-CuAl及γ₁-Cu₉Al₄。

IMCs硬度測試

No.	組織	硬度值(HV)
a	Al(Cu)	135
b	η ₂ -CuAl	609.5
c	γ ₁ -Cu ₉ Al ₄	421
d	θ-CuAl ₂	295

裂痕主要出現在η₂-CuAl及γ₁-Cu₉Al₄上，顯示η₂-CuAl及γ₁-Cu₉Al₄呈現相對較低破裂韌性機械性值。

結論

- 不論試片搭接方式及是否添加填料，其拉伸荷重與鐸接功率、鐸道深度及深寬乘積存在一關聯性，當鐸接功率越大，底層鐸道深度及鐸道面積越大，使最大拉伸荷重的數值有上升的趨勢。
- 根據TEM繞射圖解可以得知**Cu(Al)與塊狀組織間連續白色層**為**γ₁-Cu₉Al₄**相，**塊狀組織**主要為**η₂-CuAl**相。**灰區**為**Al/(Cu)固溶體及θ-CuAl₂**兩相層狀共晶組織，而**黑區**凝固樹枝狀組織為**Al(Cu)固溶體**，**中間呈現灰白區**為**θ-CuAl₂**相。
- 硬度測試中，裂痕會出現在塊狀結構(**η₂-CuAl**)相、**富Cu IMC(γ₁-Cu₉Al₄)**相，顯示組織破裂韌性較差。
- 拉伸荷重以Al上Cu下無填料具較佳表現，延展性則是以Cu上Al下具較佳表現。
- 從破斷面探討，裂痕應該先發生位於界面處**塊狀組織**，再沿著**塊狀組織及富Cu IMC**界面或直接穿過**塊狀組織**延伸成長。

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摘要



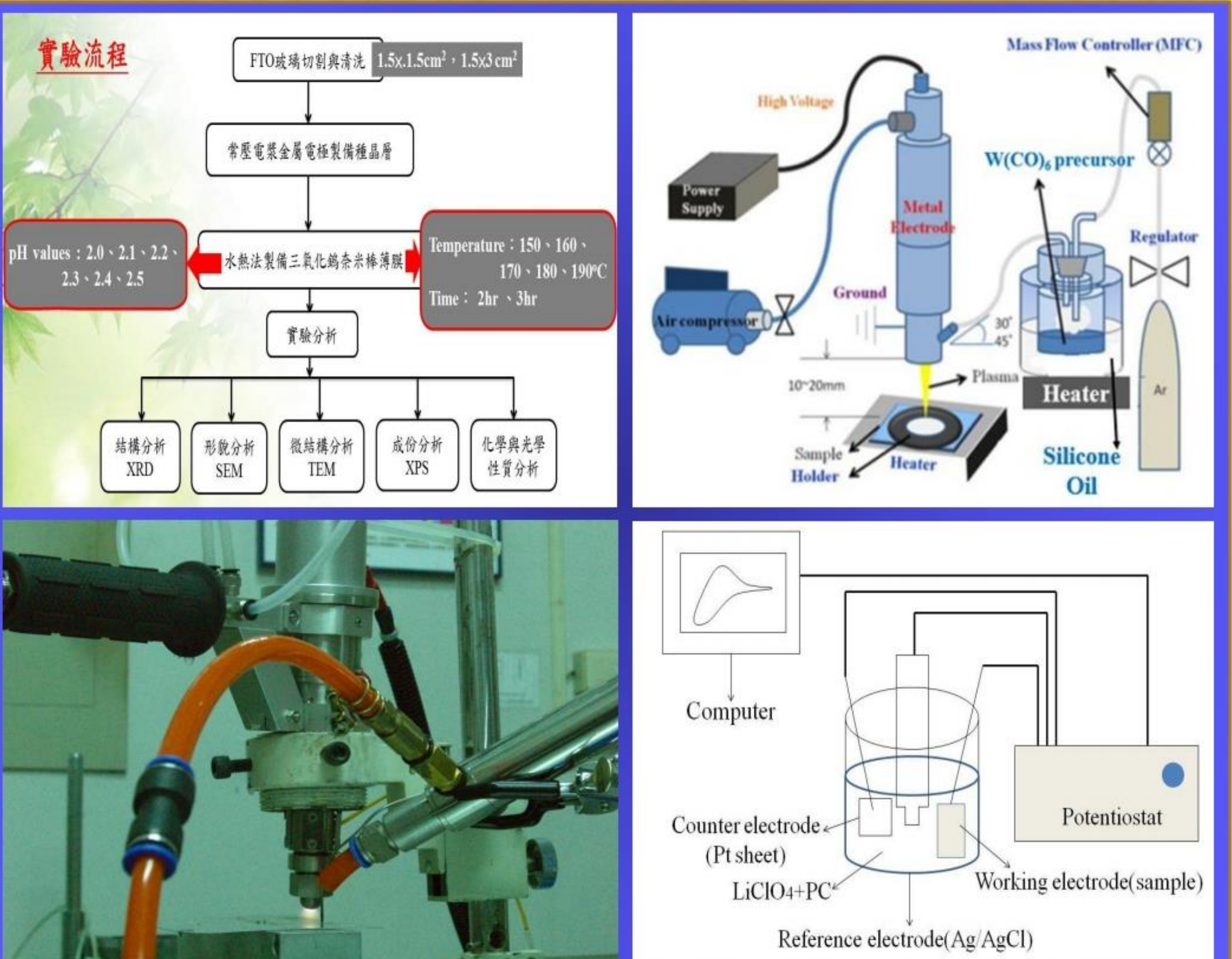
本研究目的為利用種晶層輔助水熱法製備一維方向成長之六方晶結構(Hexagonal structure)三氧化鎢(WO_3)奈米棒薄膜型態應用於電致變色元件之變色層，並探討其微觀結構對電致變色元件之電化學性質影響。本實驗首先以羰化鎢粉末($\text{W}(\text{CO})_6$)當作前驅物，透過改變電漿參數進行最佳化 WO_3 種晶層之沉積實驗。接著將已鍍有最佳化 WO_3 種晶層之FTO基板置入由鎢酸鈉(Na_2WO_4)、鹽酸(HCl)草酸($\text{H}_2\text{C}_2\text{O}_4$)及醋酸鈉(CH_3COONa)所調配之水熱前驅溶液中，透過濃度參數、水熱反應溫度及時間的改變進行 WO_3 奈米棒膜層成長。最佳實驗參數試片呈現良好的電致變色特性，主要為具備高比表面積及多孔結構，利於電解液之鋰離子進行遷入遷出反應；同時，經由循環伏安2000次測試結果顯示穩定性相當優異，未來將利用於產氧光觸媒研究。

關鍵字：常壓電漿、水熱法、三氧化鎢、奈米棒、電致變色

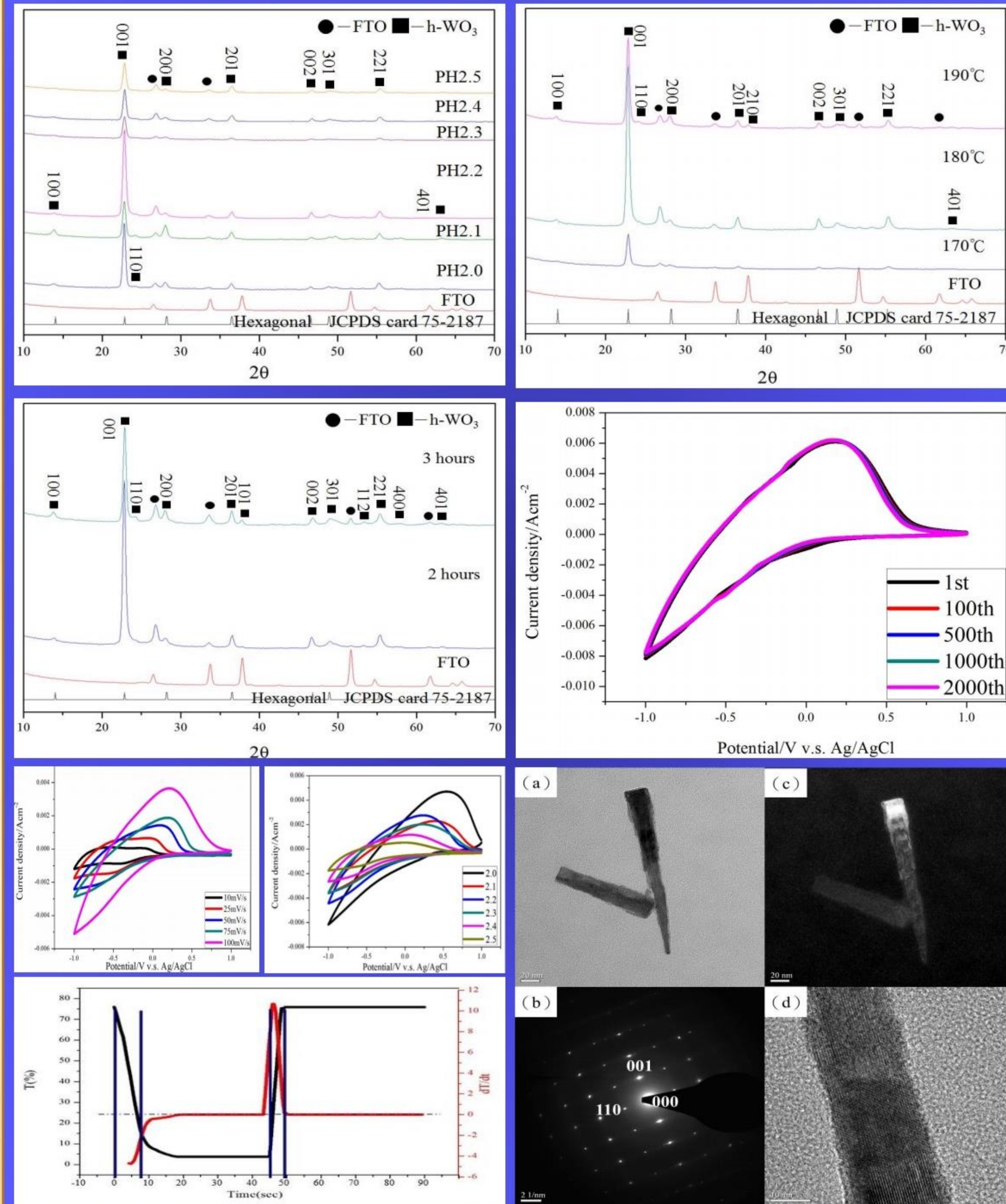
前言

三氧化鎢(WO_3)六方晶結構(HCP)具有優良的開放式隧道構造，適合作為陽離子進出之位置，因而被認為是最適合做為電致變色材料之一。一維奈米材料之薄膜較具開放性之型態，因而可提供離子快速的擴散路徑，以提升電致變色材料的光調節速度，且達到較高的著色與去色效率，此過程中只需少量的電荷遷入與遷出奈米結構材料薄膜層中。本實驗室團隊先前著手於旋鍍法製備種晶層對水熱法影響之研究，但其結果顯示元件穩定性不佳，故嘗試利用本實驗室成功研製的常壓電漿鍍膜系統進行種晶層沉積，以期能改善元件穩定性之效果。同時，參考眾多國外期刊，並未有學者前輩使用醋酸鈉(CH_3COONa)作為包覆劑，故本實驗室團隊決定採用醋酸鈉做為此實驗之水熱法包覆劑。

實驗方法

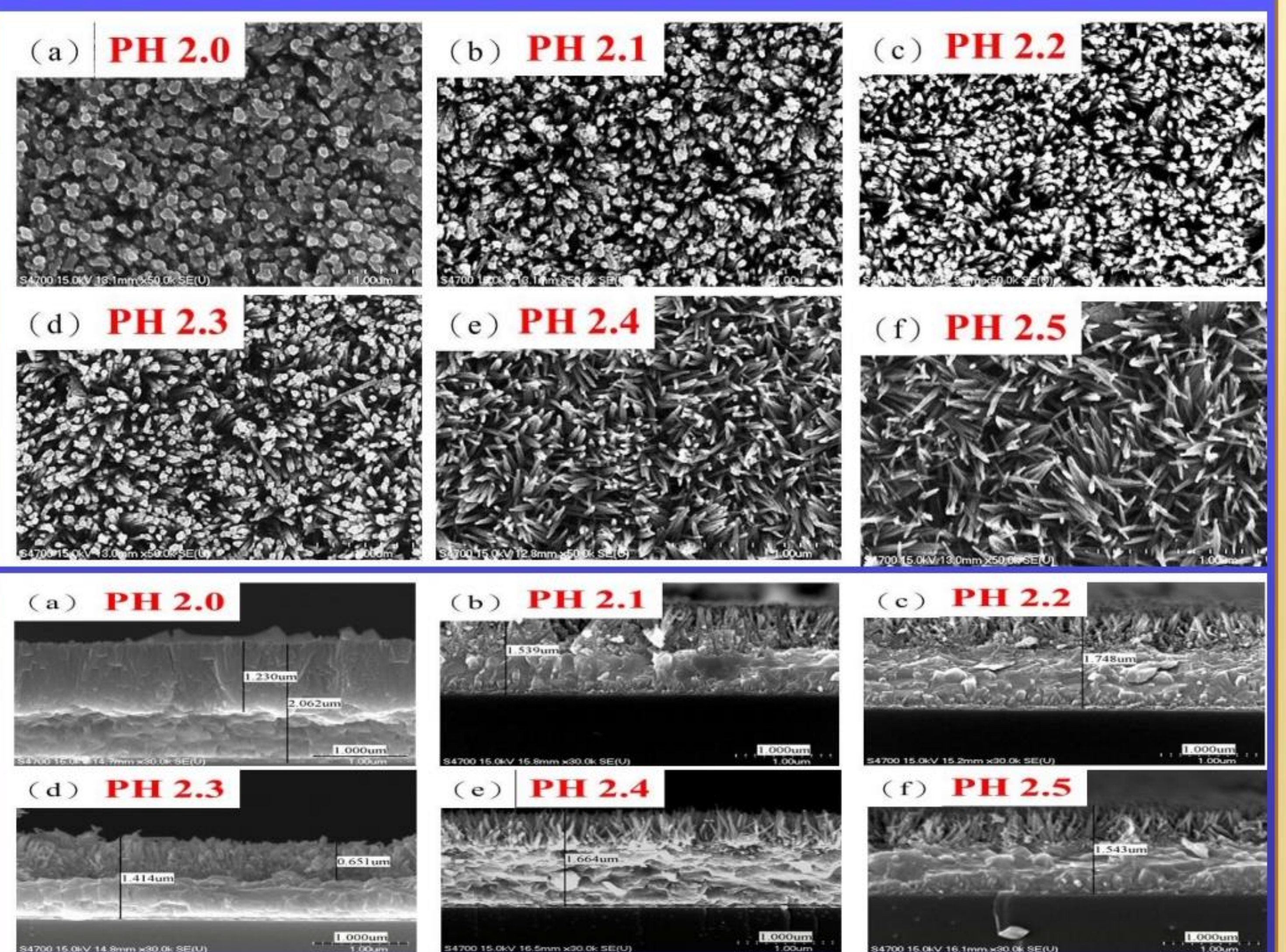


結果與討論

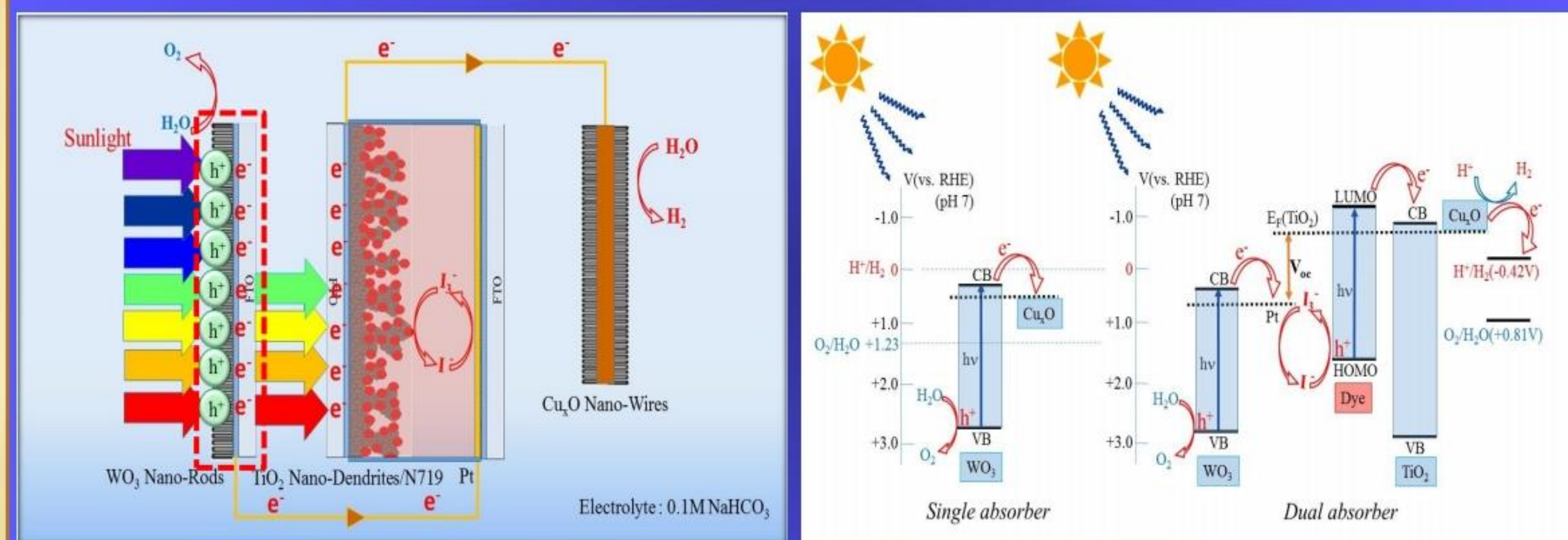


結果與討論

未來努力方向



不同PH值之SEM上視圖及橫截面圖。(a) PH 2.0；(b) PH 2.1；(c) PH 2.2；(d) PH 2.3；(e) PH 2.4；(f) PH 2.5。



結論

本實驗成功利用常壓電漿噴流沉積系統於FTO玻璃基板上製備單斜體 WO_3 種晶層及水熱法製備出垂直性、結晶性佳之六方晶體 WO_3 奈米棒。同時， WO_3 電致變色元件呈現不錯的穩定性及著去色效果。而高比表面積、多孔結構及能隙介於2.7~2.85 eV，將有助於用於未來產氧光觸媒之研究。

致謝

本研究在此感謝中央大學有機太陽電池研究量測實驗室之新世代光驅動電池模組研究中心、國家奈米元件實驗室、義守大學精密分析實驗室於實驗分析儀器上的協助。

常壓電漿技術沉積二氧化鈦膜層於染料敏化太陽能電池之應用研究

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摘要

本實驗目的為使用自行開發之常壓電漿噴流(Atmospheric Pressure Plasma Jet)金屬電極沉積系統，以化學輔助氣相沉積法製備奈米樹狀型態之銳鈦礦結構(Anatase structure)二氧化鈦(TiO_2)膜層於FTO透明導電玻璃上，應用於染料敏化太陽能電池(Dye Sensitized Solar Cell, DSSC)之工作電極，搭配N719染料及含碘離子的電解液組裝成DSSC元件，並探討其微觀結構對DSSC元件之光電性質影響。在實驗過程中，透過調整電漿功率、沉積溫度、沉積距離、主電漿流量、前驅物流量及沉積時間等製程參數，尋求最佳之樹狀結構高度、密集性及孔隙度，達到增加膜層對於染料的吸附量及呈現較佳的電子傳輸性質之目標，本研究最佳光電轉換效率已達12.08%，未來將整合DSSC元件於光電化學產氫之應用研究。

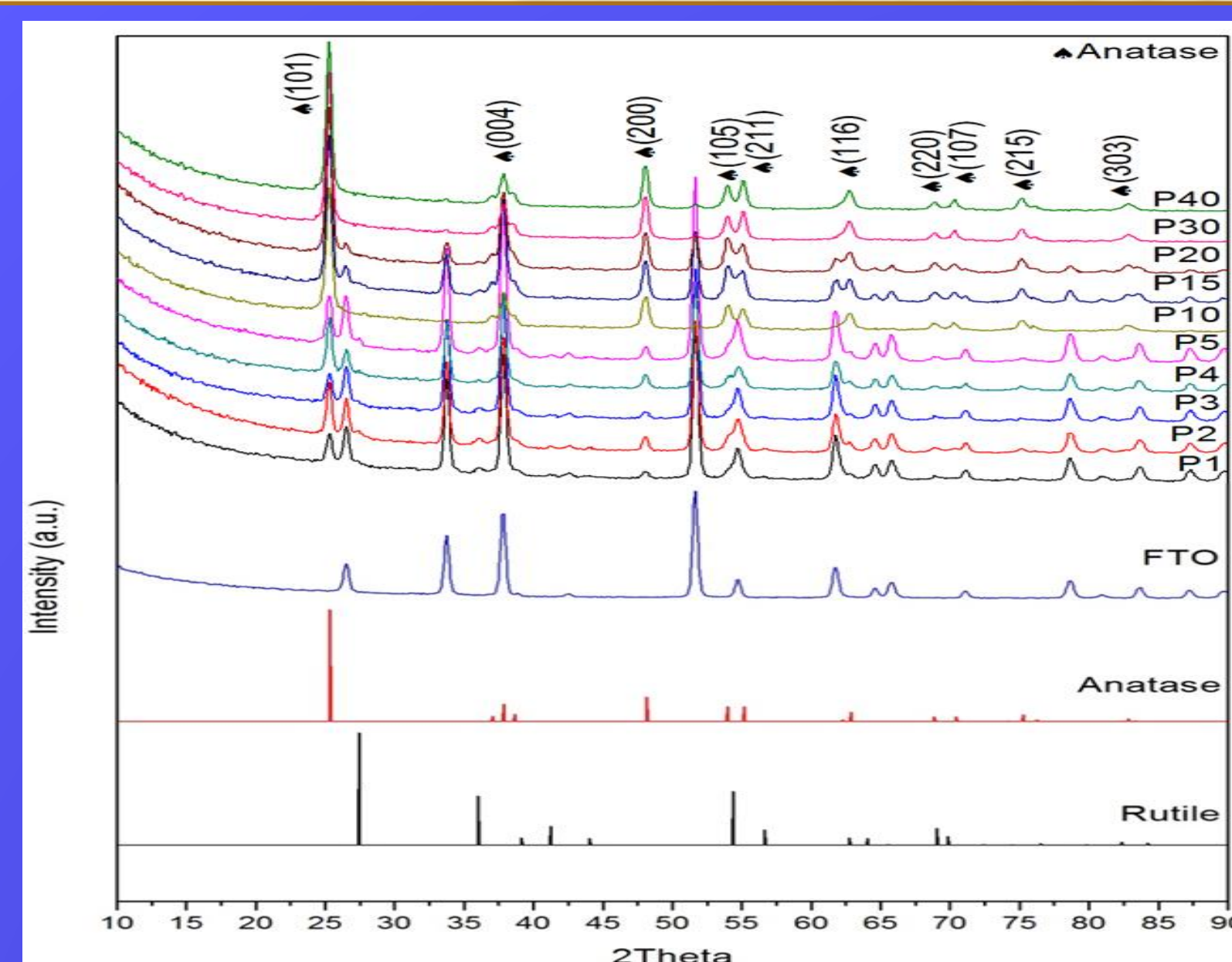
關鍵字：常壓電漿、二氧化鈦、化學氣相沉積、奈米結構、染料敏化太陽能電池

前言

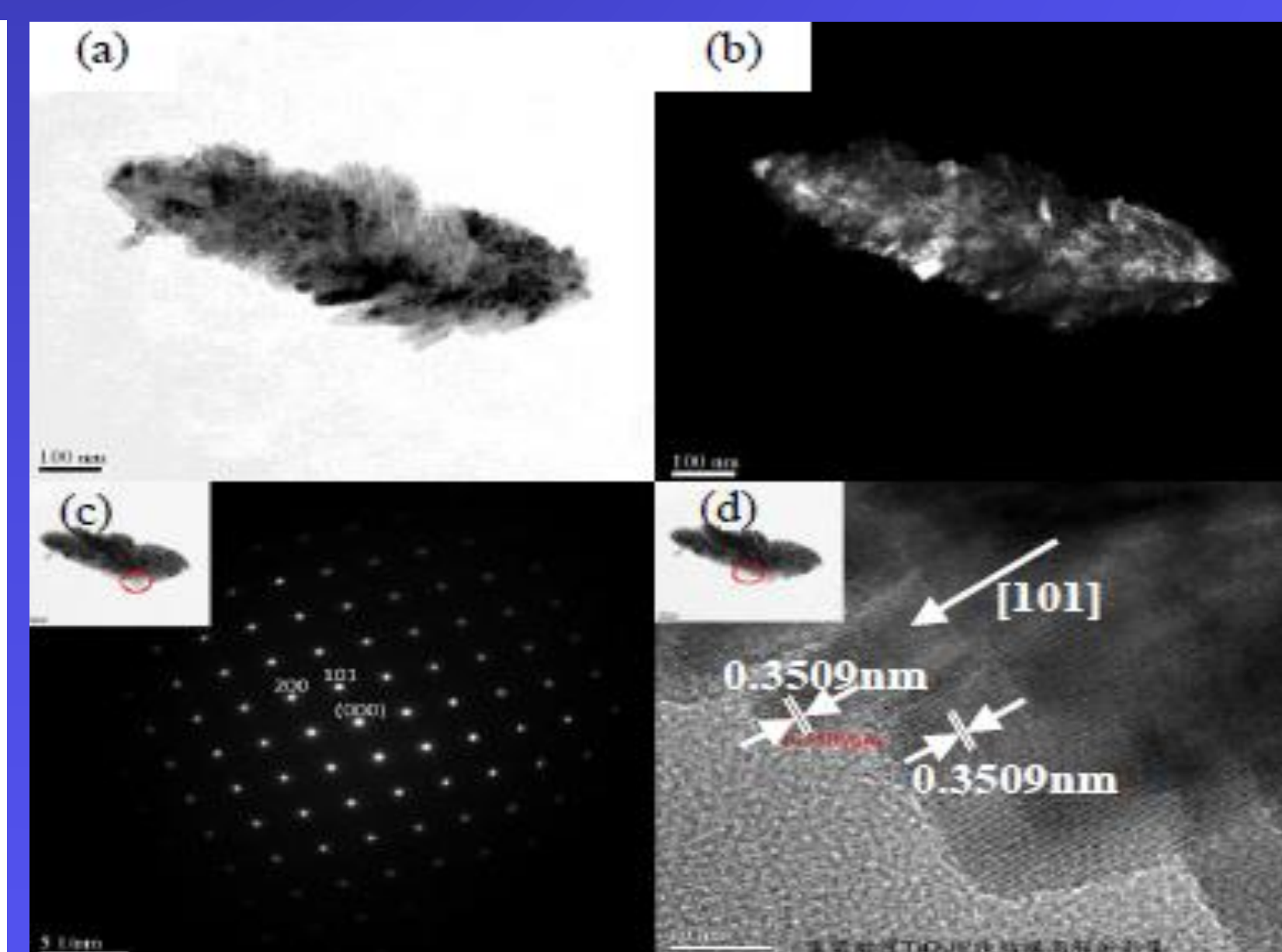
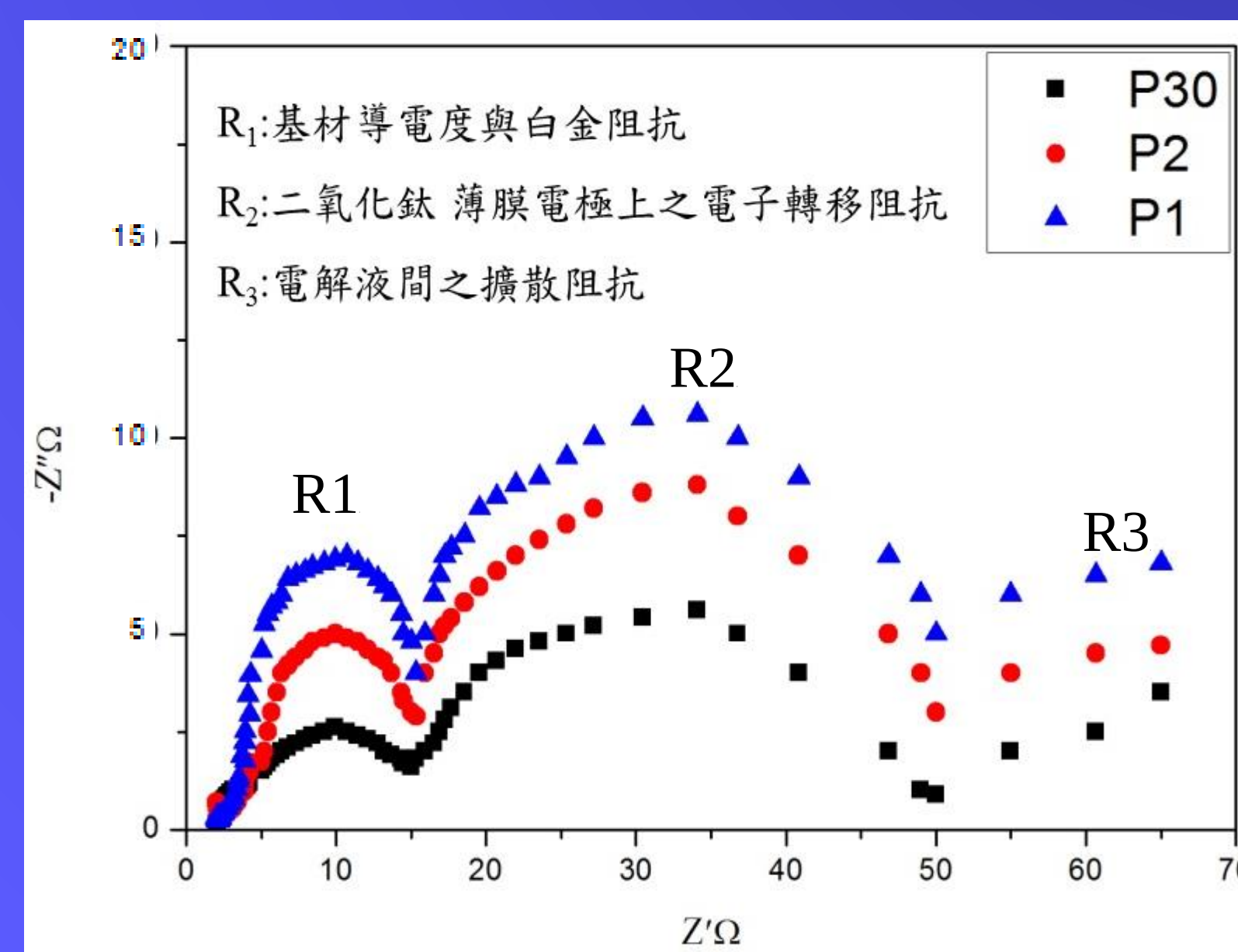
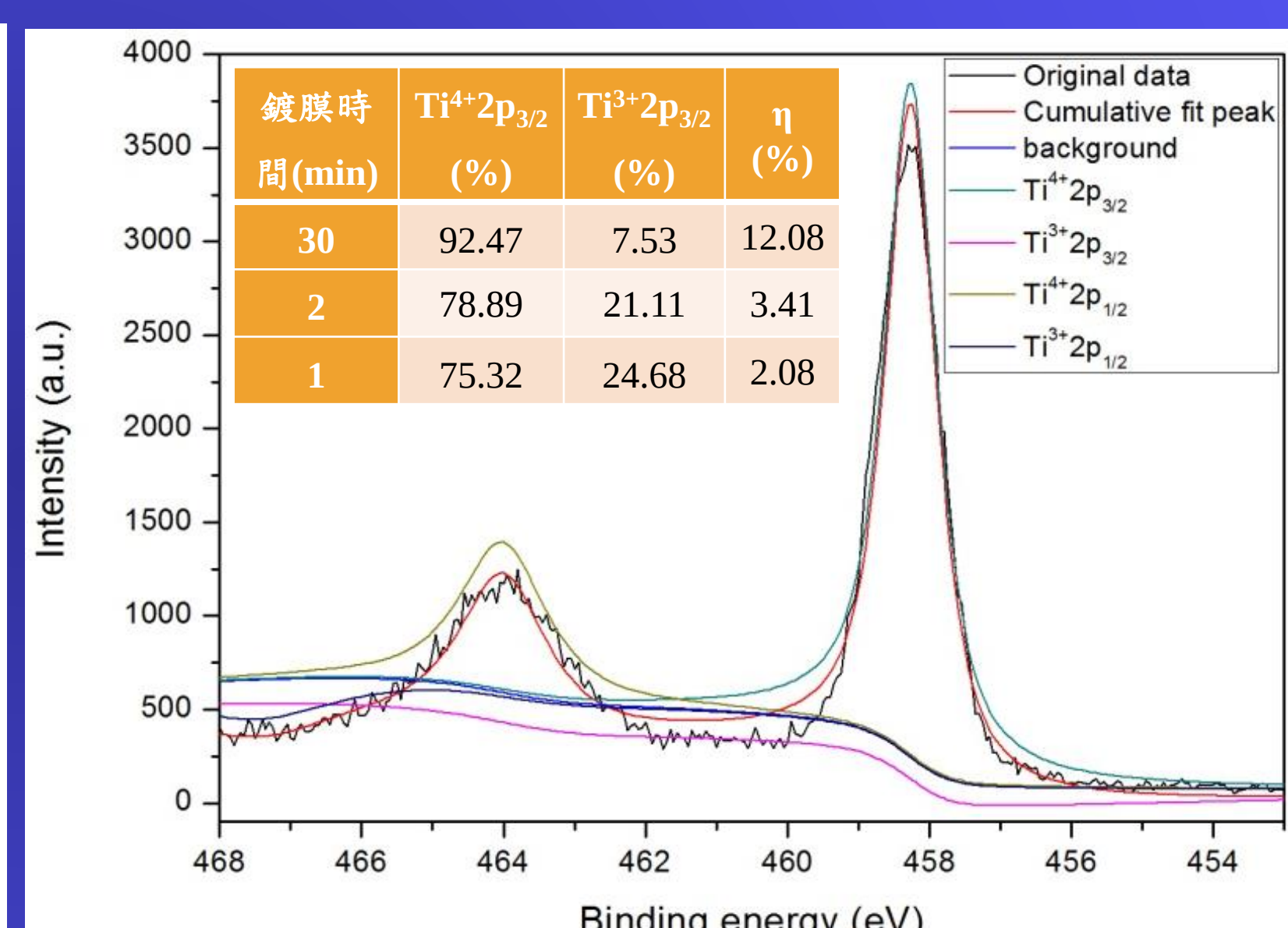
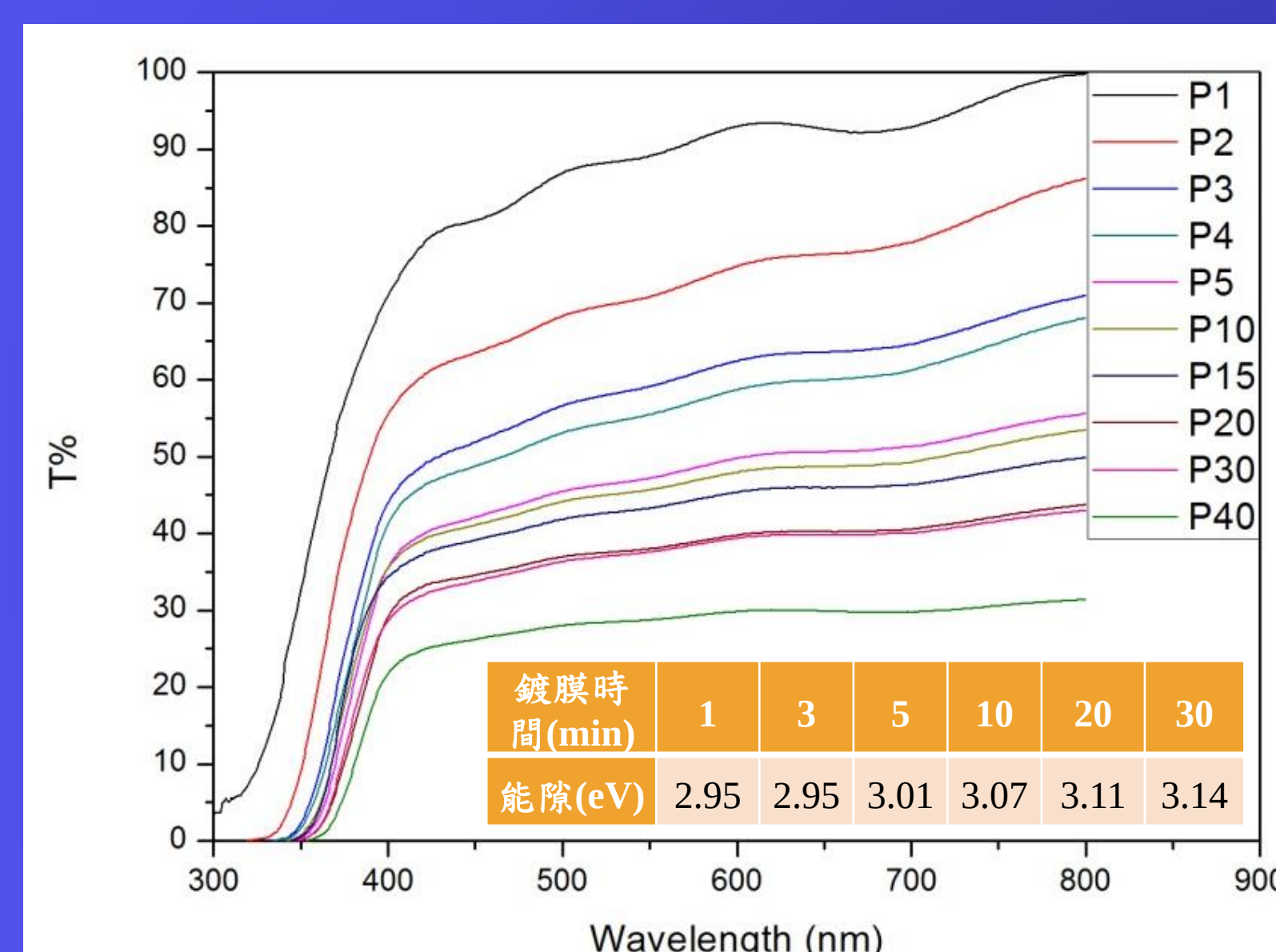
目前鈦鈦礦太陽能電池(Perovskite Sensitized Solar Cell, PSSC)在研發上相當卓越，但其仍面臨需要長時間進行可靠度及耐久性的考驗。因此，在轉換效率高於12%且發展成熟之 TiO_2 奈米結構染料敏化太陽能電池(Dye Sensitized Solar Cell, DSSC)，在具備低成本、容易組裝、大面積高產出、穩定性佳等優勢下，仍有發展競爭的機會。截至目前為止，高轉換效率DSSC的主要技術在於光陽極具備高捕光、高比表面積及高電子傳輸一維結構。常壓電漿除了具有沉積速率高、操作簡易、容易製作大面積膜層等優勢外，捨棄了昂貴的真空系統，大幅降低製程成本因此在近幾年成為眾多研究技術上的熱門話題，同時也成為本實驗室主力研究設備。

貝 爾 斯 公 司

結果與討論

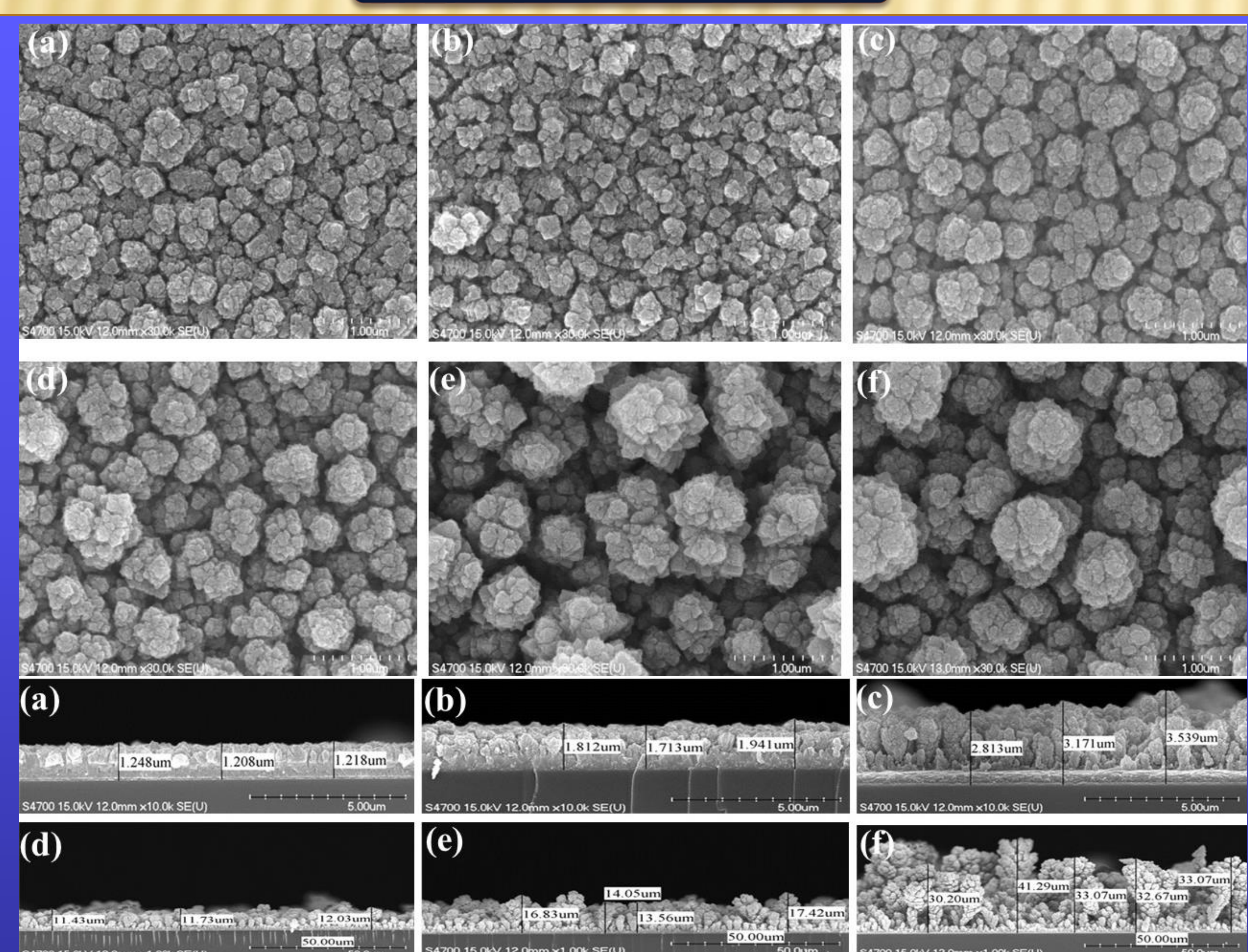


沉積時間 (min)	開路電壓 (V)	短路電流密度 (mA/cm ²)	填充因子 (%)	光電轉換效率 (%)
1	0.70	5.48	54.27	2.08
3	0.70	10.10	65.23	4.61
5	0.70	15.33	49.99	5.37
10	0.70	26.31	53.59	9.87
20	0.70	30.01	57.46	12.08
30	0.70	24.24	52.50	8.91

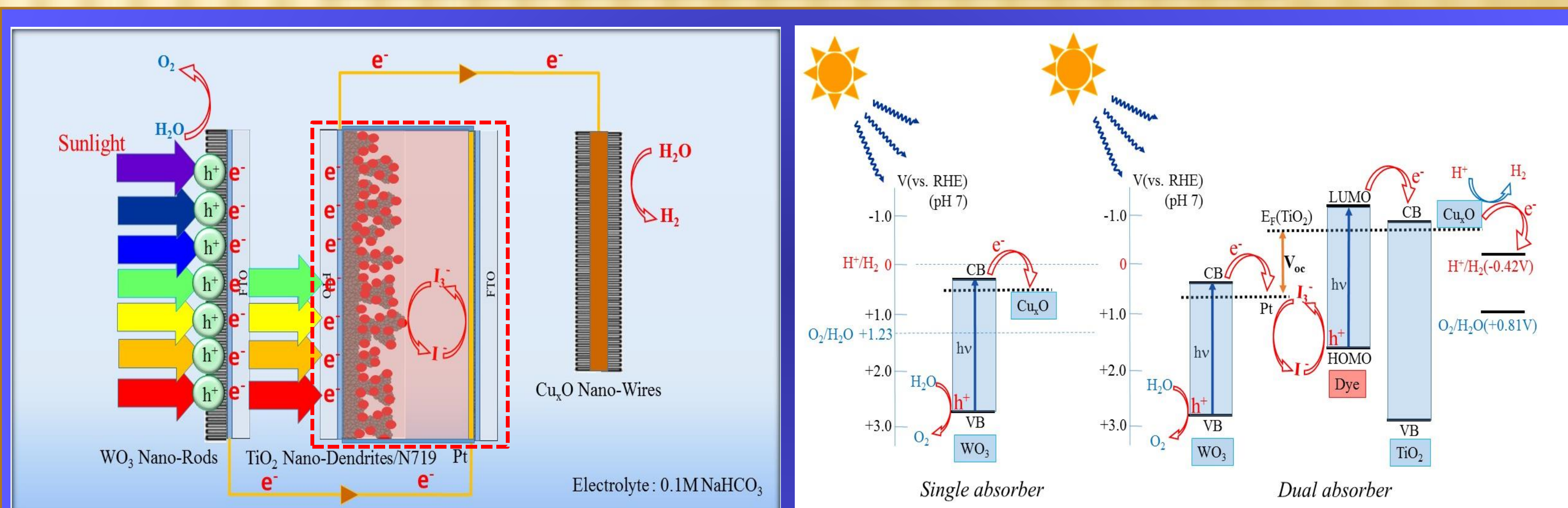


未來努力方向

結果與討論



不同電漿沉積時間之SEM上視圖及橫截面圖。(a) 1分鐘；(b) 3分鐘；(c) 5分鐘；(d) 10分鐘；(e) 20分鐘；(f) 30分鐘。



結論

本實驗成功利用常壓電漿噴流沉積系統於FTO玻璃基板上製備結晶性佳之奈米樹狀型態銳鈦礦結構 TiO_2 之膜層，透過系列實驗參數調整，呈現高比表面積、多孔結構及能隙介於2.95~3.14eV紅移現象，樣品尺寸1.5cm²之最佳光電轉換效率12.08%；樣品尺寸20cm²之最佳光電轉換效率9.50%；樣品尺寸35cm²之最佳光電轉換效率9.01%。最佳製程參數之TEM分析發現樹狀型態皆由許多奈米棒狀型態堆疊而成，晶格繞射圖顯示單晶優選方向結構，有效降低電子電洞的合併作用，高解析影像分析其晶格排列整齊並有一致性，故有利電子的傳輸，因此光電轉換效率高。未來將整合DSSC元件於光電化學產氫之應用研究。

誌謝

本研究在此感謝中央大學有機太陽電池研究量測實驗室之新世代光驅動電池模組研究中心、國家奈米元件實驗室、義守大學精密分析實驗室於實驗分析儀器上的協助。



Deposition of Cobalt Oxide films on *a*-Plane Sapphire Substrate by Ultrasonic Assisted Mist Chemical Vapor Deposition

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Department of Material Science and Engineering I-SHOU University , Kaohsiung ,Taiwan R.O.C
(Ministry of Science and Technology Program Code : MOST 109-2813-C-214-002-E)

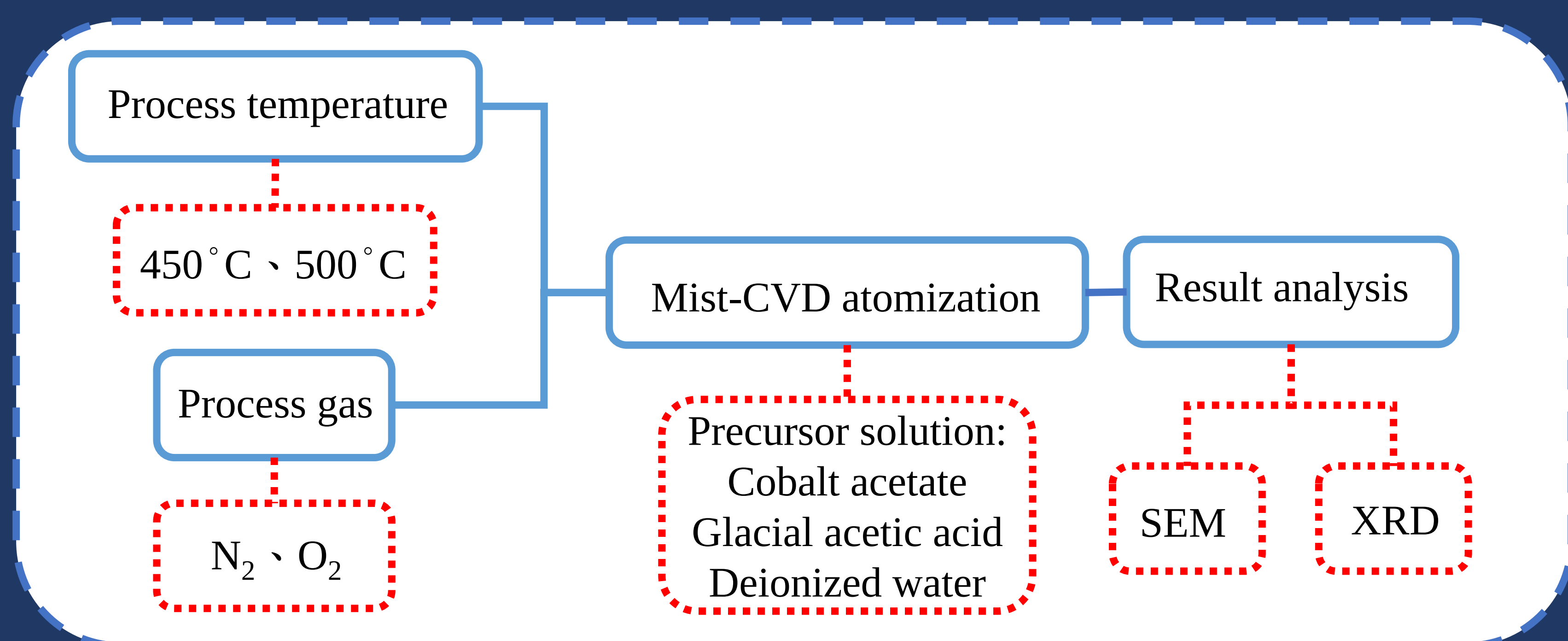
Background and Introduction

- Mist Chemical Vapor Deposition(Mist CVD) is a low cost, high safety, environment friendly and “ Green ” process technology to growing epitaxial films on specific temperature and pressure . This experiment attempt to grow epitaxy of cobalt oxide thin films on *a*-plane sapphire substrate by this technic processes.
- In our experiment , we divide chamber temperature into 450 °C and 500 °C, and divided carrier gas into nitrogen and oxygen . In the last, we analyze surface morphology and crystal structure of four different experiment condition of cobalt oxide films by Scanning Electron Microscope and X-Ray Diffraction.
- The consequence show that, there is generation of excellent epitaxy quality corundum structure cobaltous oxide in nitrogen atmosphere at 450 °C. When temperatures up to 500 °C with oxygen atmosphere , it can generate great epitaxy quality spinel structure tricobalt tetroxide.

Characteristic

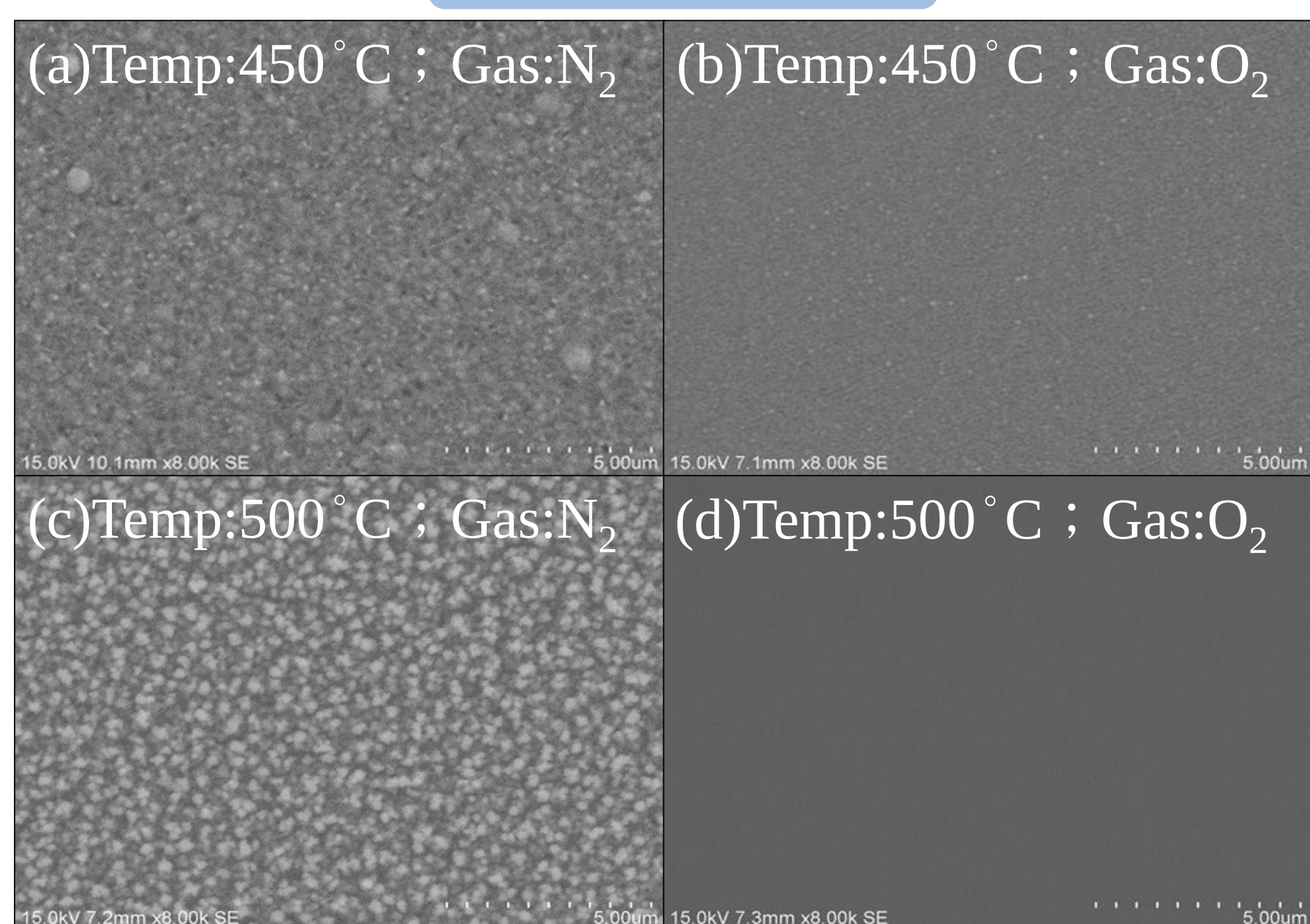
According to the research, there would generate two type of stable composition, CoO and Co₃O₄. Those are the p-type and wide band gap semiconductor. In this study , specific of cobalt acetate aqueous solution is used as the precursor. We attempt to obtain high quality epitaxial films cobalt oxide by simple - atmospheric Mist-CVD process. The result can connect to n-type semiconductor and become p-n junction .

Experiment process



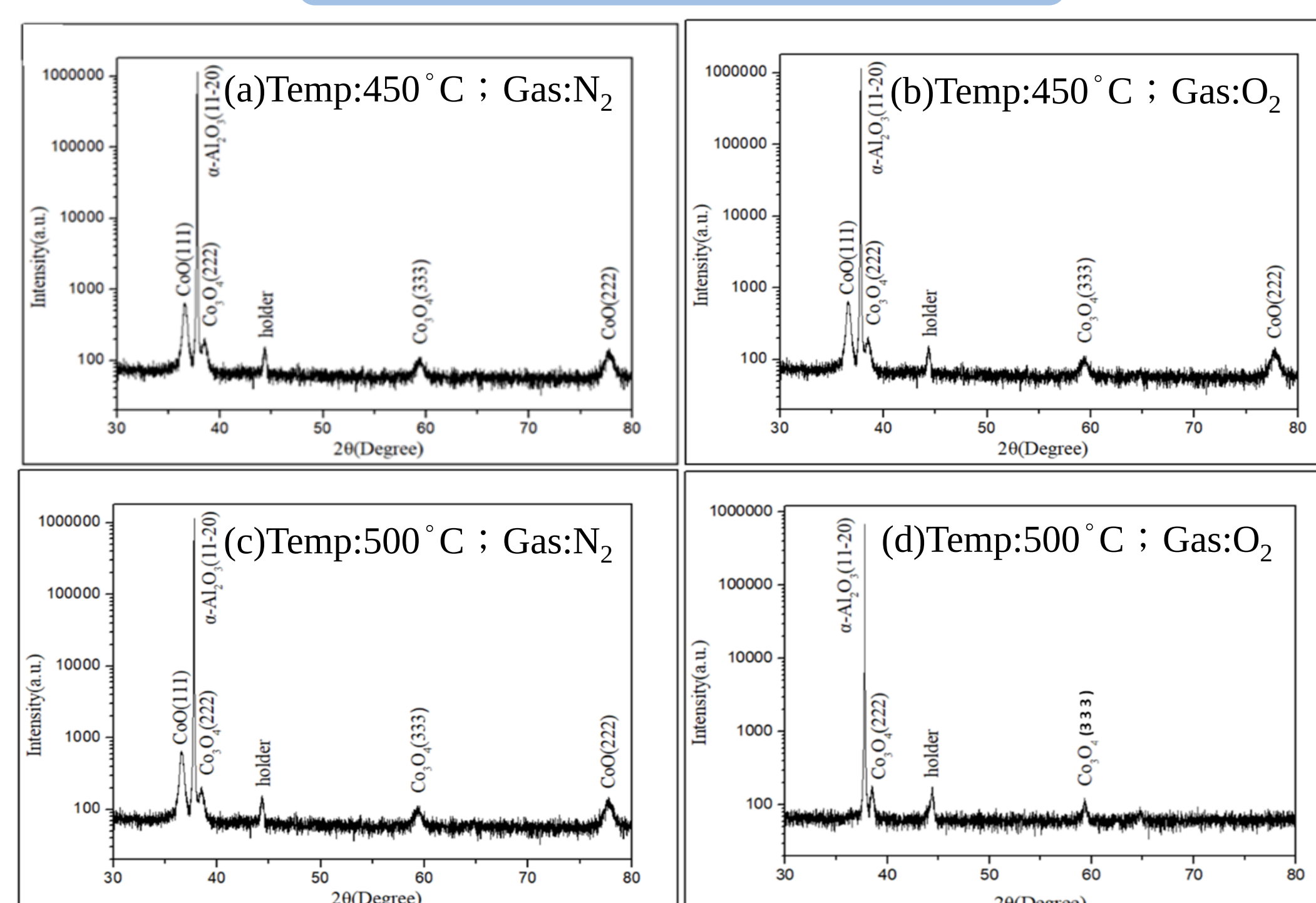
Experimental results

Surface analysis

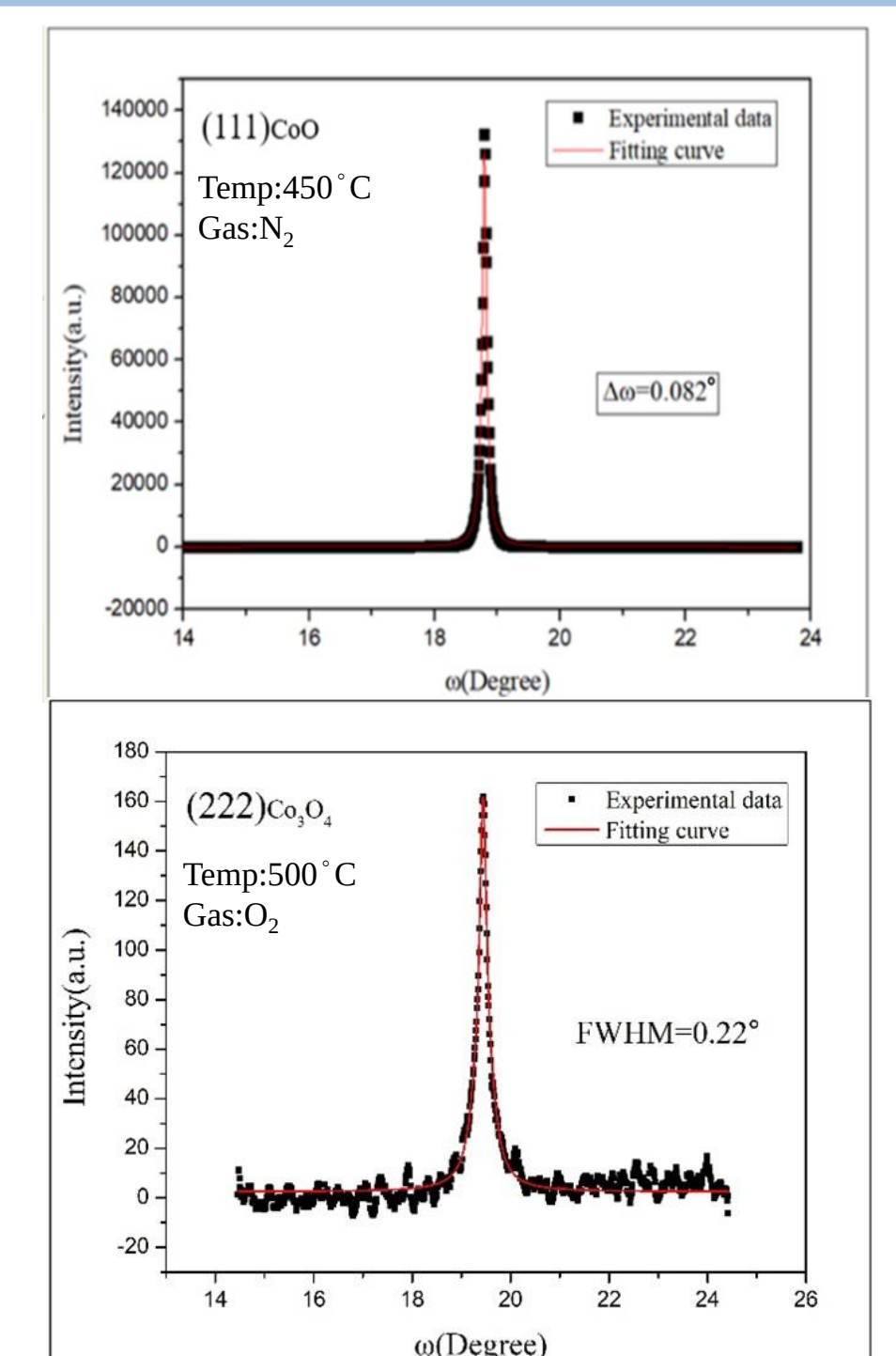


- (a) The specimen obtains fine particles and homogeneous epitaxial surface films layer with cobalt monoxide structure.
- (b) The specimen obtains fine particle and smoothly surface. But it is a multi-phased structure with cobalt monoxide and tricobalt tetroxide structure.
- (c) The specimen obtains fine particle and flatness surface. But it is a multi-phased structure with cobalt monoxide and tricobalt tetroxide structure. However, the grain size is larger than specimen(b).
- (d) The specimen obtains the excellent flatness and homogeneous surface films layer. It is single-phase epitaxy with tricobalt tetroxide structure.

X-ray diffraction analysis



Rocking curve analysis



- The half-height width peak of the CoO₍₁₁₁₎ is 0.082 °(degree).
- The half-height width peak of Co₃O₄₍₂₂₂₎ is 0.22 °(degree).

Conclusion

- Mist-CVD process can grow excellent quality of cobalt monoxide epitaxial films on *a*-plane sapphire substrate in nitrogen atmosphere at high temperature(450°C), it also can grow great quality of tricobalt tetroxide epitaxial films on *a*-plane sapphire substrate in oxygen atmosphere at higher temperature(500°C).
- The epitaxial orientation of cobalt monoxide epitaxial films or tricobalt tetroxide epitaxial films with *a*-plane sapphire substrate : (111)[11 $\bar{0}$]_{CoO,Co₃O₄}|| (112 $\bar{0}$)[0001]_{Al₂O₃}.
- Mist-CVD process can grow more excellent epitaxial films than PLD or MOCVD process.

Acknowledgements

We are extremely thanks to the Ministry of Science and Technology of the R.O.C. and ISU University ManaLab's assistance. Without their support and allowance, we can't complete the project smoothly.

Study on the properties of epitaxial ZnO thin films on c-plane sapphire by hydrothermal method

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In this experiment, ZnO films were deposited on c-plane sapphire substrates by a radio-frequency reaction magnetron sputtering as buffer layers at various oxygen atmosphere ratios(20~80%), by using low-cost hydrothermal method to prepare p-type K-doped ZnO epitaxial film, and discuss the effects of its film properties. The experimental results show that the surface morphology changes from roughness to flatness with the increase of K concentration. Especially while the films growth on buffer layers of sputtering at 50% of O₂ / Ar+O₂ flow ratio, by using the hydrothermal method at 0.006 M K-doped ZnO. In terms of electrical and optical properties, it has the lowest resistance (48.46 Ω-cm), the highest average visible light transmittance (95.24%), and the highest figure of merit is 4.83×10⁻⁷.

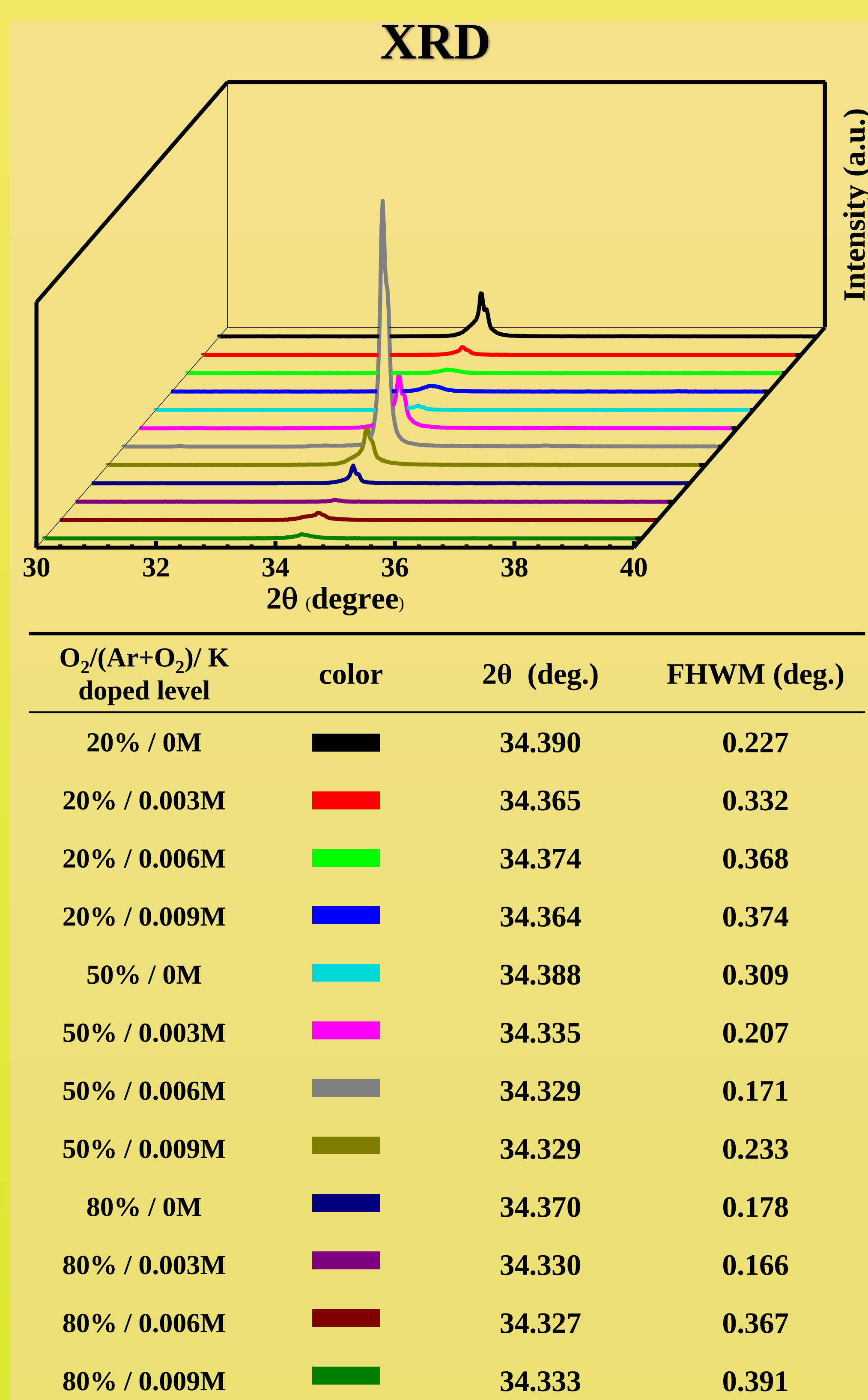


Figure 1.The crystal structure analysis of epitaxial K-doped ZnO films grown on ZnO-buffer layers c-plane sapphire substrates at 90°C temperature with hydrothermal time of 90 minutes, where the ZnO buffer layers are deposited for different oxygen atmosphere ratio.

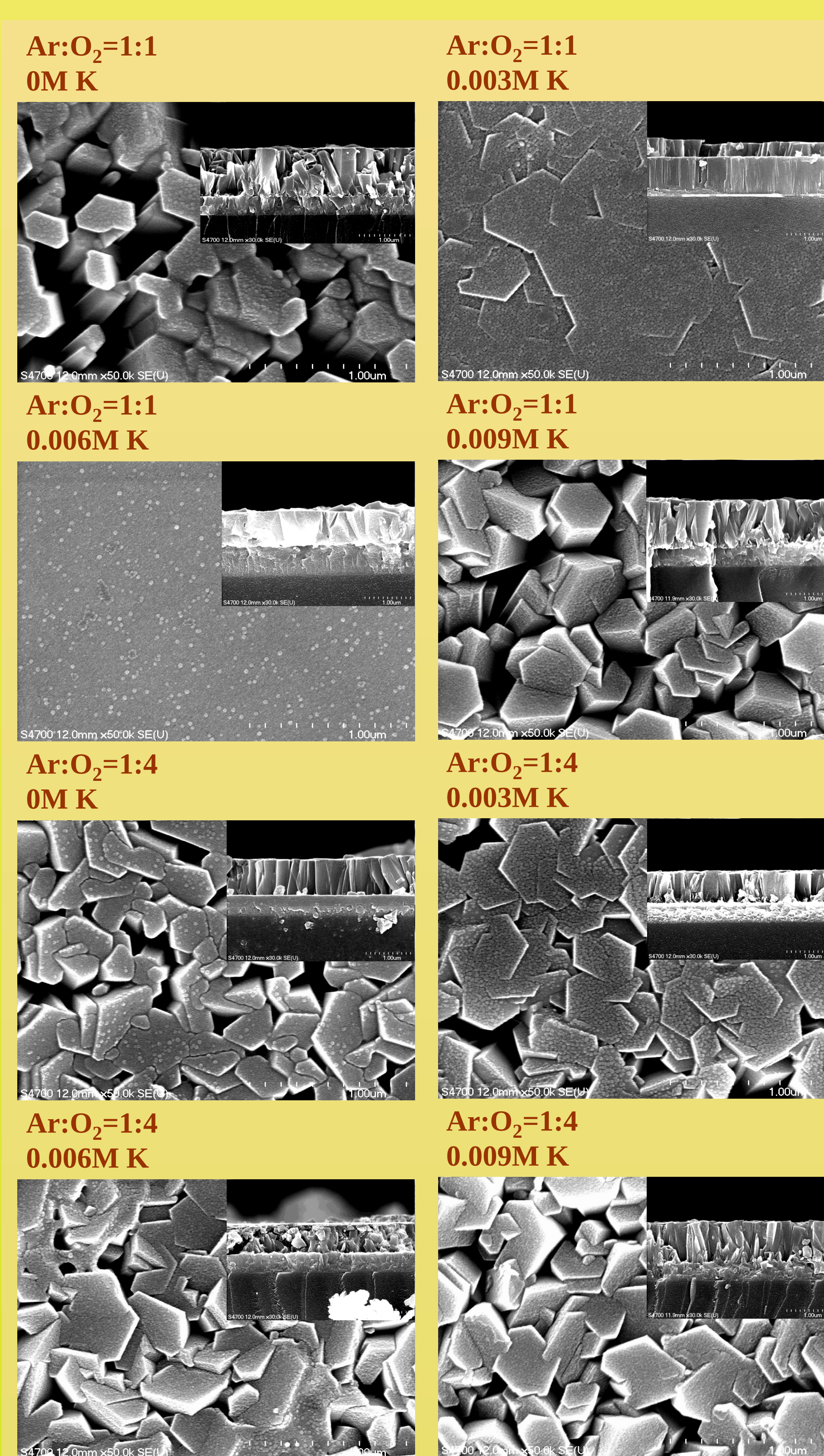
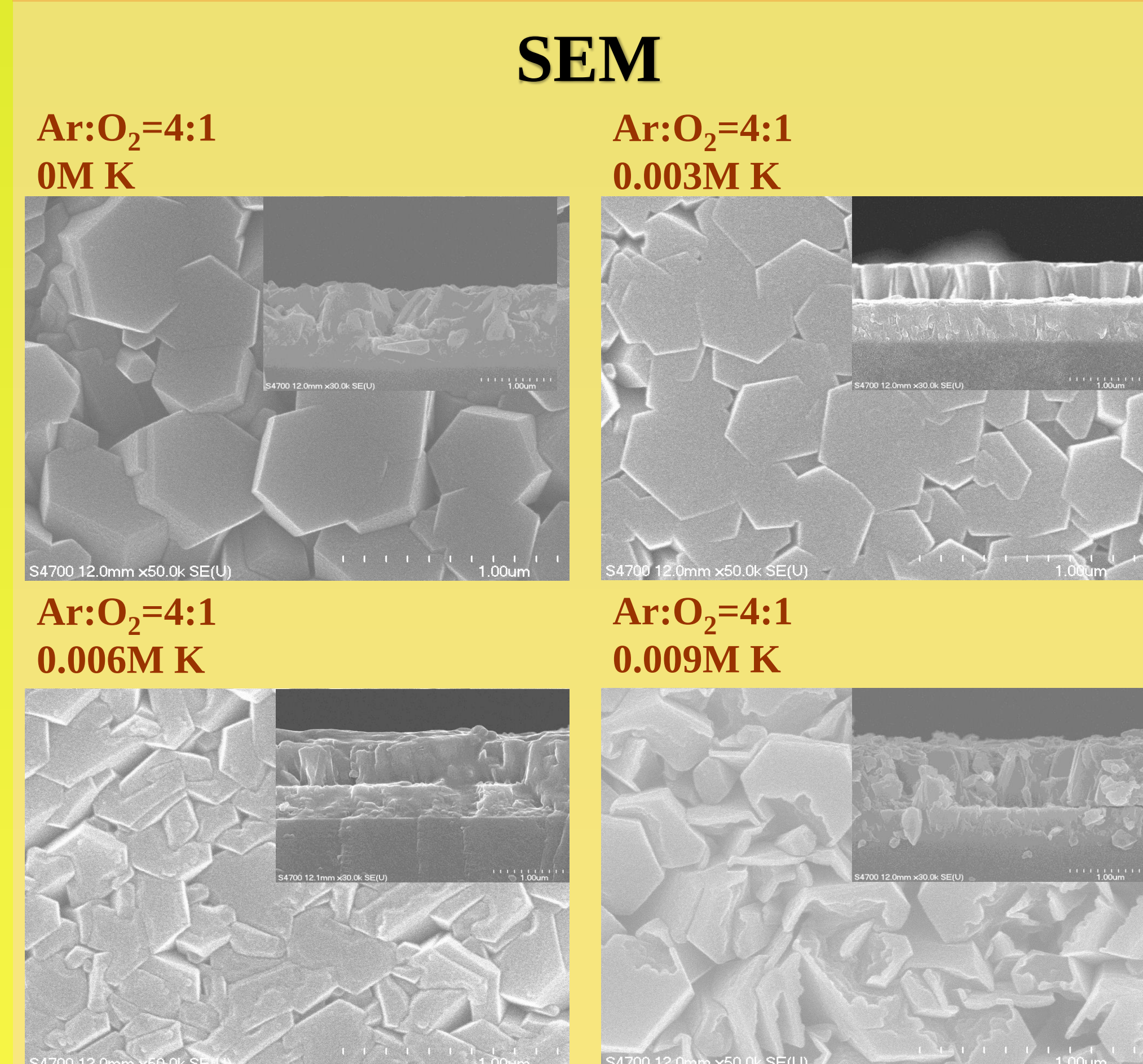


Figure 2.The SEM surface structure and cross section of K-doped ZnO films grown on ZnO-buffer layers c-plane sapphire substrates at 90°C temperature with hydrothermal time of 90 minutes, where the ZnO buffer layers are deposited for different oxygen atmosphere ratio.

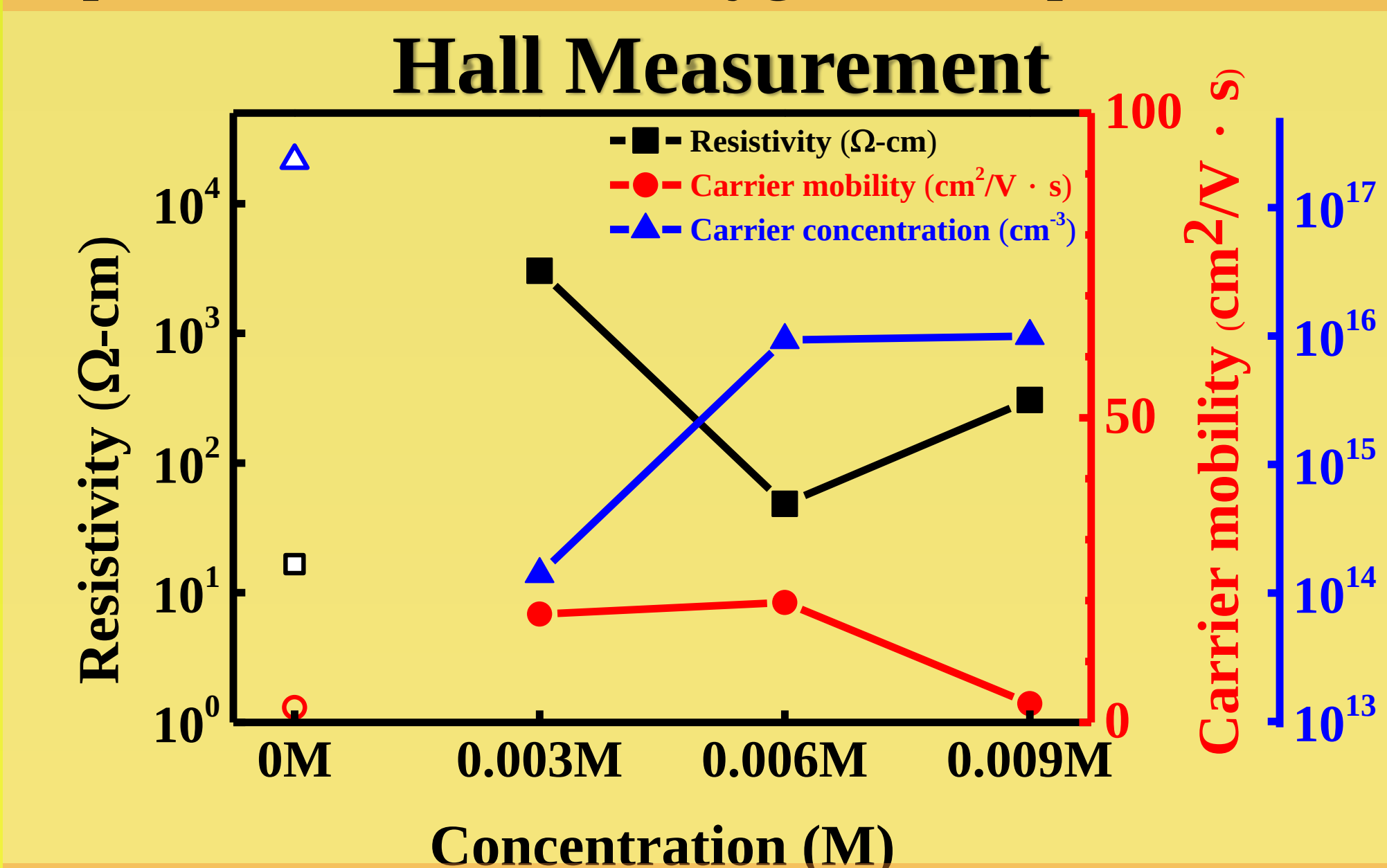


Figure 3.The electrical properties of K-doped (0M~0.009M) ZnO films grown at 90°C temperature with hydrothermal time of 90 minutes on ZnO-buffer layers are deposited for 50% oxygen atmosphere ratio.

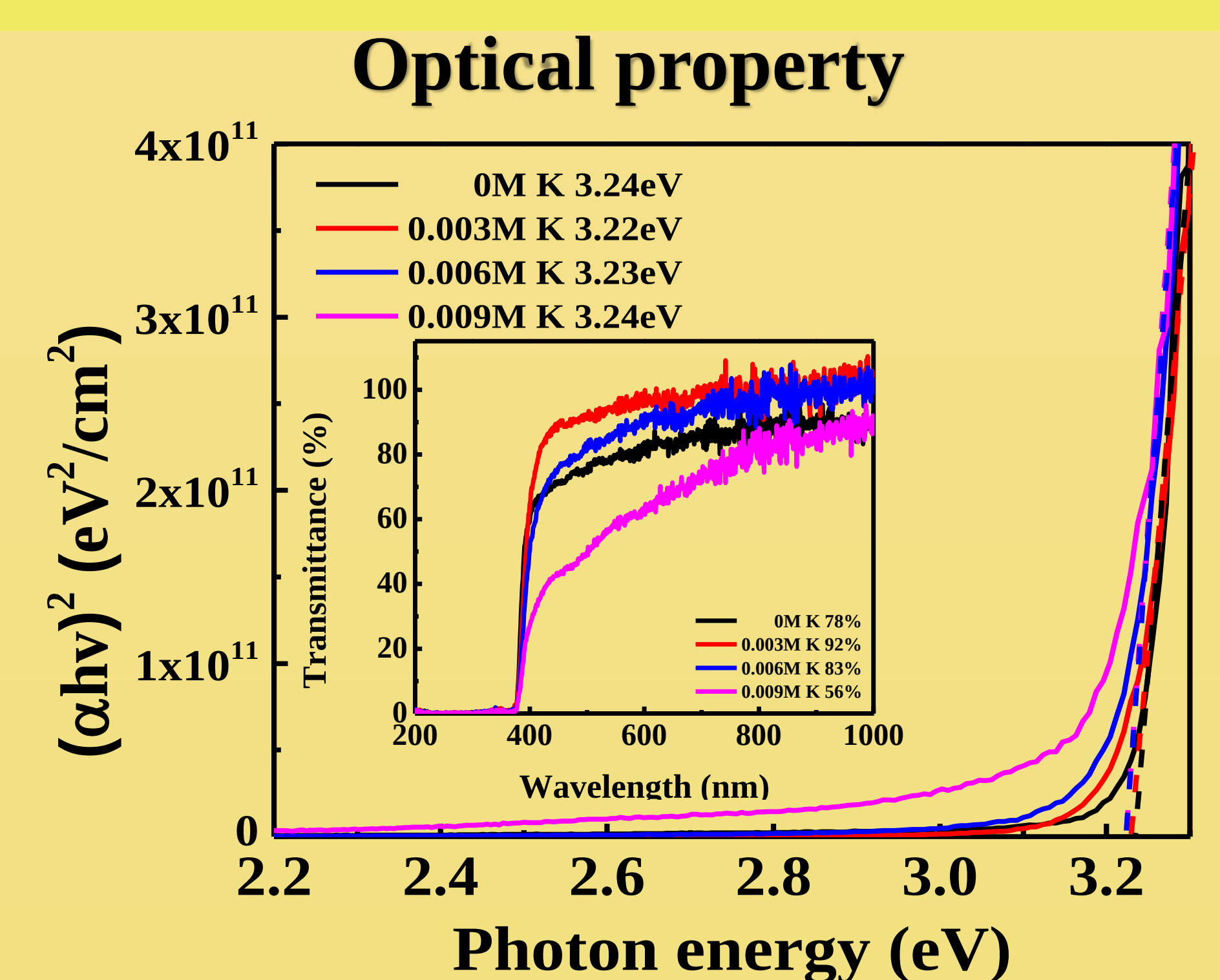


Figure 4.The optical properties of K-doped (0M~0.009M) ZnO films grown at 90°C temperature with hydrothermal time of 90 minutes on ZnO-buffer layers are deposited for 50% oxygen atmosphere ratio.

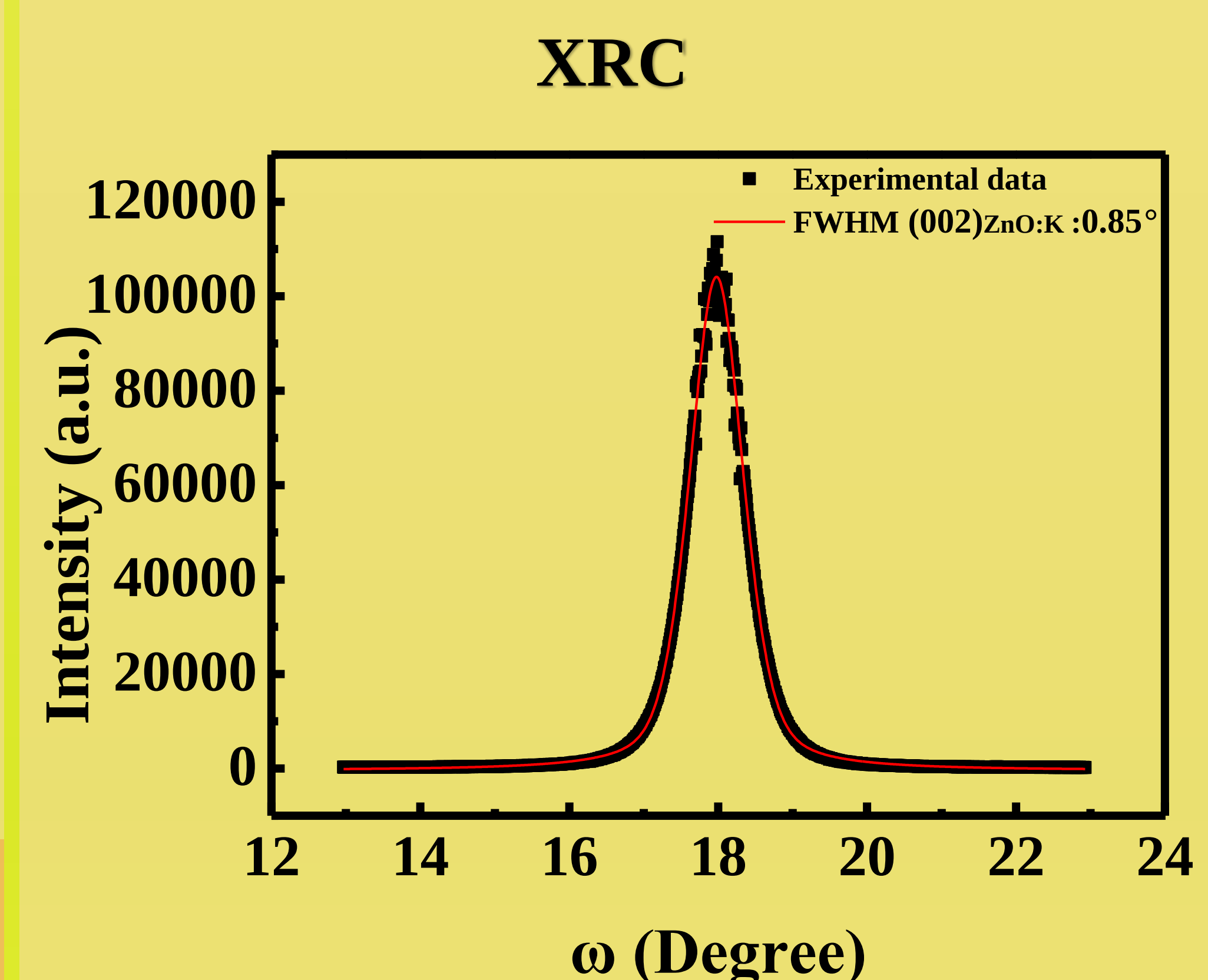


Figure 5.X-ray rocking curve of K-doped (0M~0.009M) ZnO films grown at 90°C temperature with hydrothermal time of 90 minutes on ZnO-buffer layers are deposited for 50% oxygen atmosphere ratio.

Conclusions

The XRD results at various K-doped concentration of ZnO film show a highly preferred growth direction along the c-axis. SEM images of the p-type K-doped ZnO epitaxial films reveal that the surface morphologies are much smooth and reduced porosity, the optical properties increase at least to 80% Especially while the films growth on buffer layers of sputtering at 50% of O₂ / Ar+O₂ flow ratio, by using the hydrothermal method at 0.006 M K-doped ZnO has fine crystallization, epitaxy, and photoelectric properties.

以抽氣過濾及旋鍍改質法製作奈米碳材生物感測紙研究

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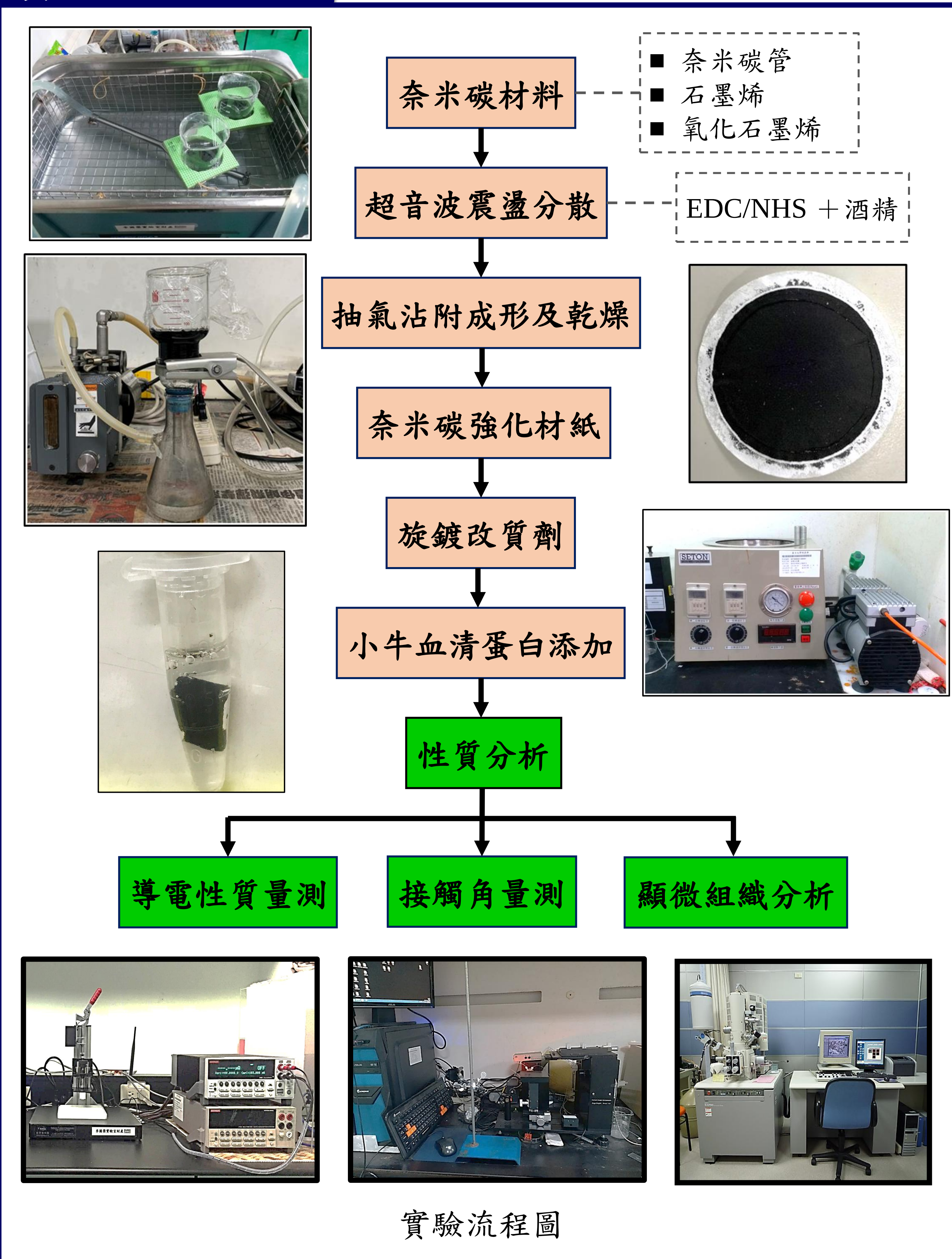
¹義守大學材料科學與工程學系、²義大醫院心臟內科

摘要

癌症是全世界死亡的主要原因之一，但在檢測癌症上，傳統酶聯法程序複雜且成本相對較高，因此改善其缺點，成為了重要的一項課題。

本研究將分別採用**奈米碳管**、**奈米碳管-石墨烯**與**奈米碳管-氧化石墨烯**，以抽氣沾附法製作成奈米碳強化材紙，再針對各種試紙利用旋鍍的方式，分別鍍上不同改質劑，藉此提升試紙的表面親水性，最後再添加不同濃度之小牛血清蛋白，藉此觀察碳強化材紙在蛋白質添加前後的電性變化趨勢，以評估其作為早期癌症篩檢應用的可行性。實驗結果顯示，利用單寧酸(Tannic acid, TA)改質的碳強化材紙不僅於接觸角上變化最為明顯，其電阻變化率也較添加聚(4-苯乙烯磺酸鈉) (Poly(Sodium 4-Styrenesulfonate), PSS)來的優異，在碳強化材紙後續添加蛋白質後，利用TA改質的試片，更有利於蛋白質吸附。

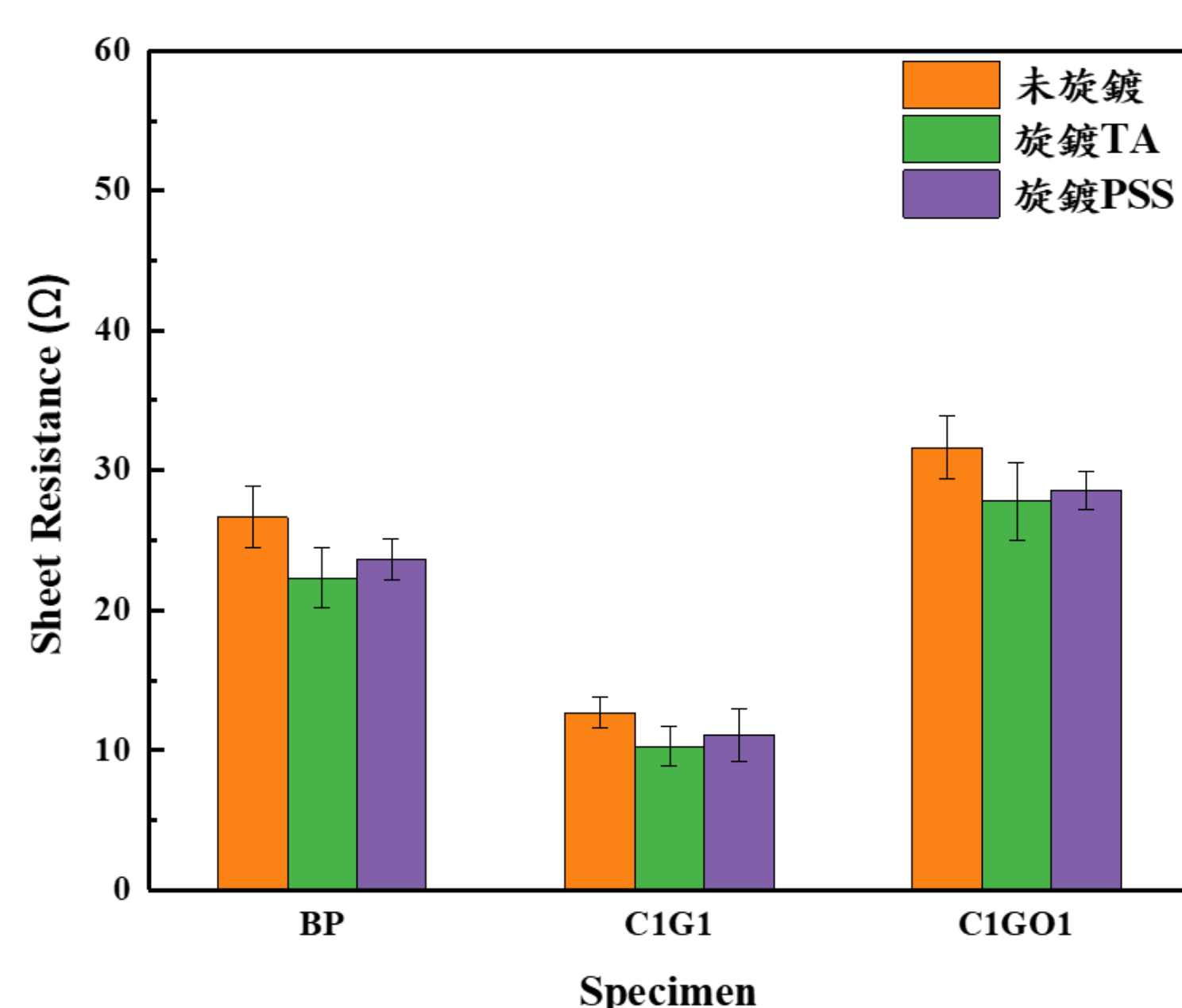
實驗方法



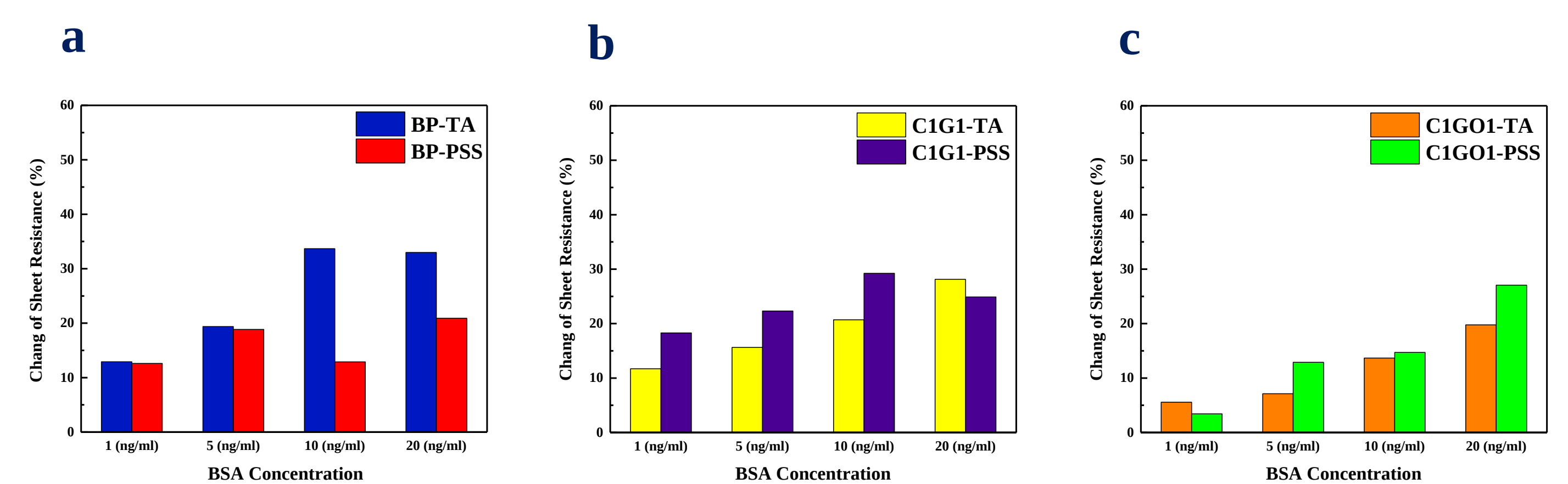
實驗結果



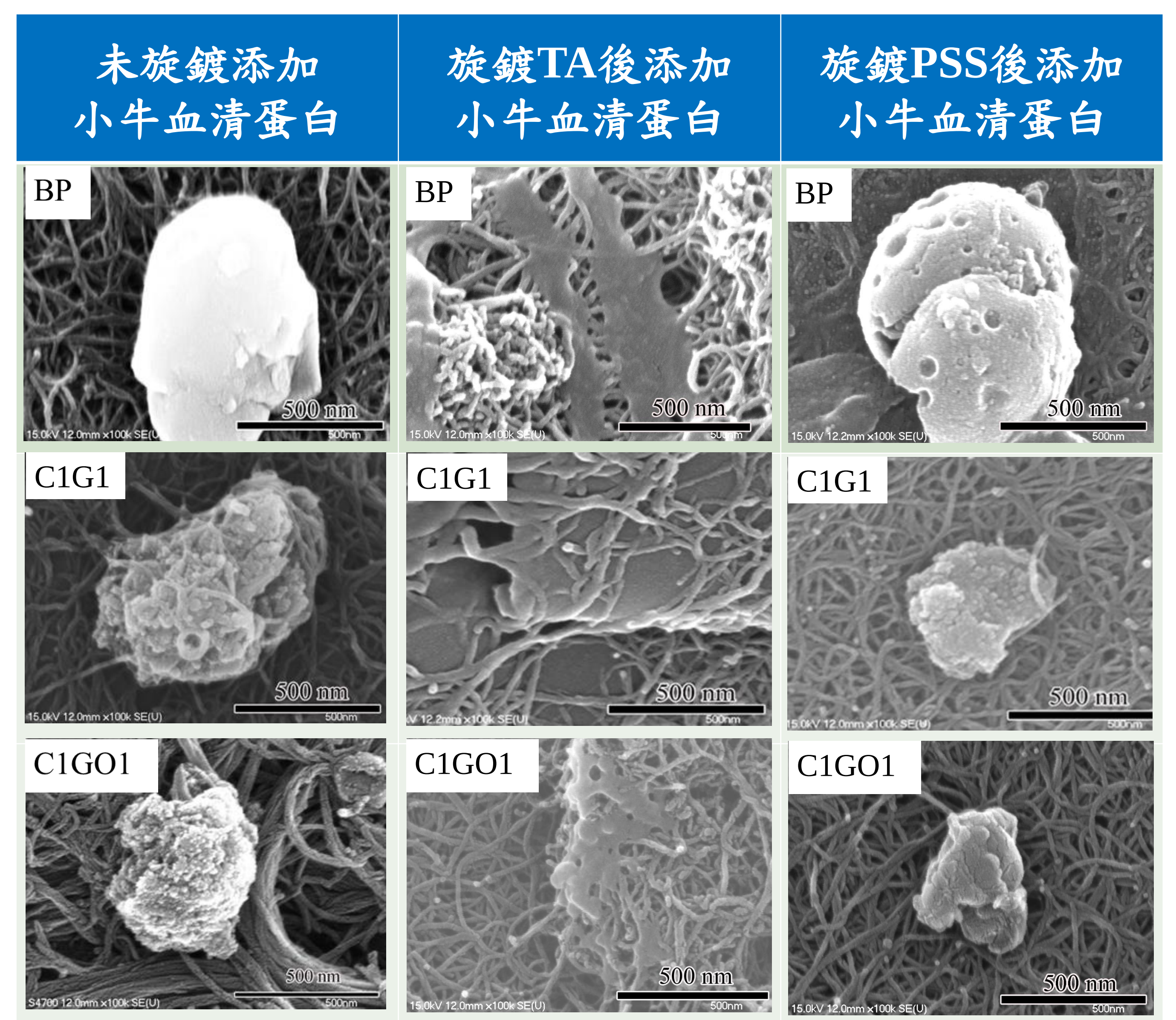
BP試片(a)未旋鍍(b)旋鍍TA(c)旋鍍PSS改質劑後之接觸角



導電性質量測，BP、C1G1與C1GO1旋鍍前後試片電阻變化



導電性質量測，(a) BP、(b) C1G1、(c) C1GO1旋鍍後試片添加小牛血清蛋白之片電阻變化率



各類碳強化材紙旋鍍改質劑後添加蛋白質前後之顯微結構

結論

1. 改質後之各類奈米類碳強化材紙，由疏水表面轉變為親水表面，其中以利用TA改質劑之變化最為顯著。
2. 改質後之各類奈米碳強化材紙，其電阻都明顯下降，導電性較佳，其中利用TA改質之試片最為明顯。
3. BP與C1G1試片經改質並且添加蛋白質後，其電阻變化率明顯增加，其中以C1G1-TA試片變化較為明顯且穩定。

致謝

本研究承蒙義守大學暨義大醫院經費補助(計畫編號：ISU-106-IUC-04)，及共同主持人一義大醫院心臟內科王朝平博士之協助和科技部經費補助(MOST 109-2221-E-214-020)和支持，使本研究得以順利進行，謹此致謝。

以奈米碳強化材紙製作奈米碳材/碳基複合材料研究

郭育維 劉士葆 林則諺 洪沂鈴 楊凌竹 劉冠汝 安卓均

指導教授：李國榮 教授

義守大學材料科學與工程學系

研究動機與目的

碳/碳複合材料是高溫高強度、高彈性係數結構材料(高於 2000°C)，同時具有低熱膨脹係數、低密度、耐磨擦、抗疲勞與抗腐蝕等優異性質，主要是以碳纖維作為碳/碳複合材料中的**強化材**，但其各項性質已臻極致。

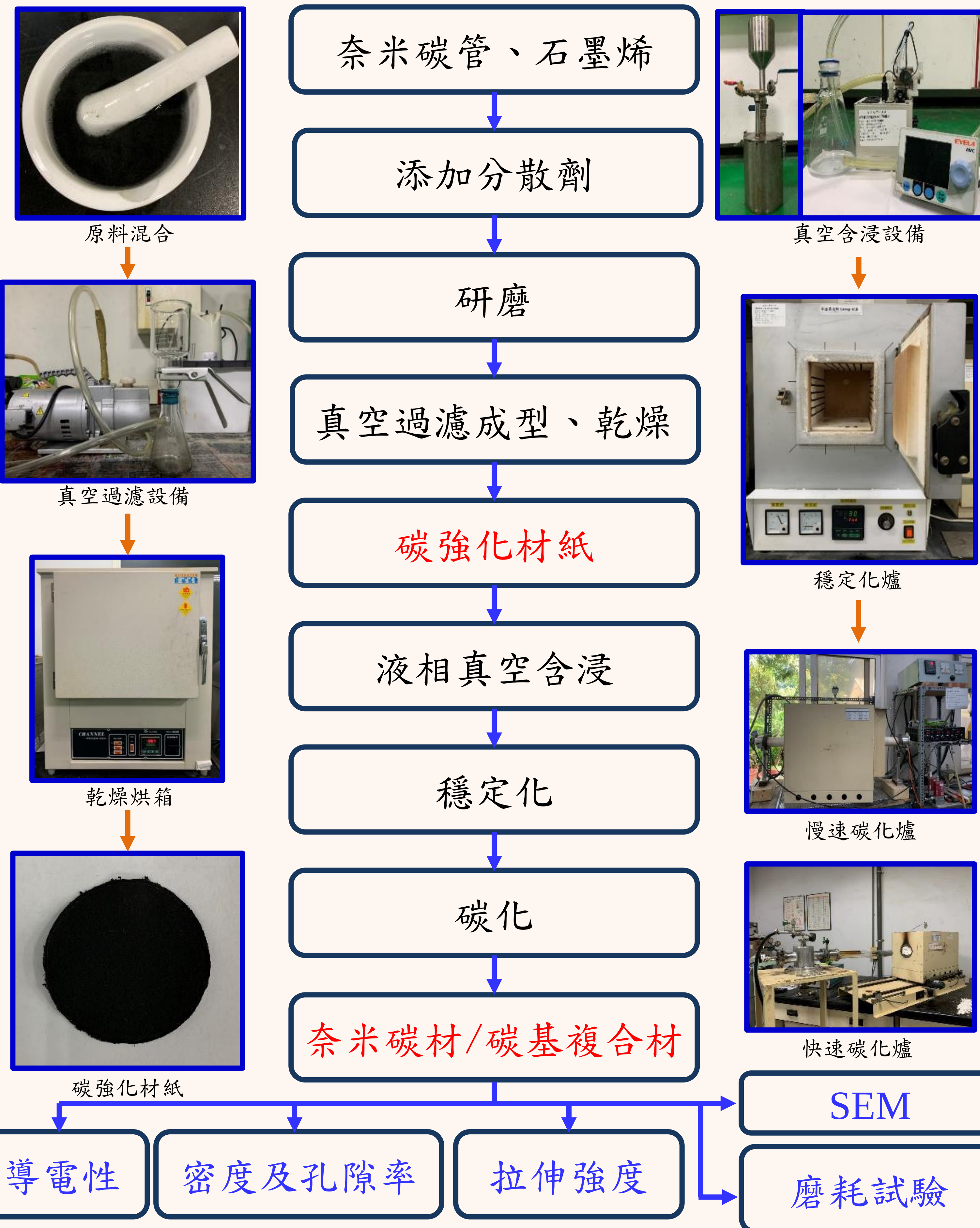
奈米碳管與**石墨烯**作為合適的強化材在各性質上都優於碳纖維，具有**取代碳纖維**作為**奈米碳材/碳基複合材料**的潛力，但也存在**操縱不易**與**團聚**的缺點，所以必須克服缺點，才能進行後續研究。

本研究利用**奈米碳管**與**石墨烯**，取代傳統碳纖維作為強化材，並克服奈米碳材不易操控與團聚的缺點，製成**奈米碳強化材紙**，並利用**不同的碳化速率**，進一步製成**奈米碳材/碳基複合材料**，進行導電性量測、密度及孔隙率量測、拉伸試驗、磨耗試驗與顯微結構分析。

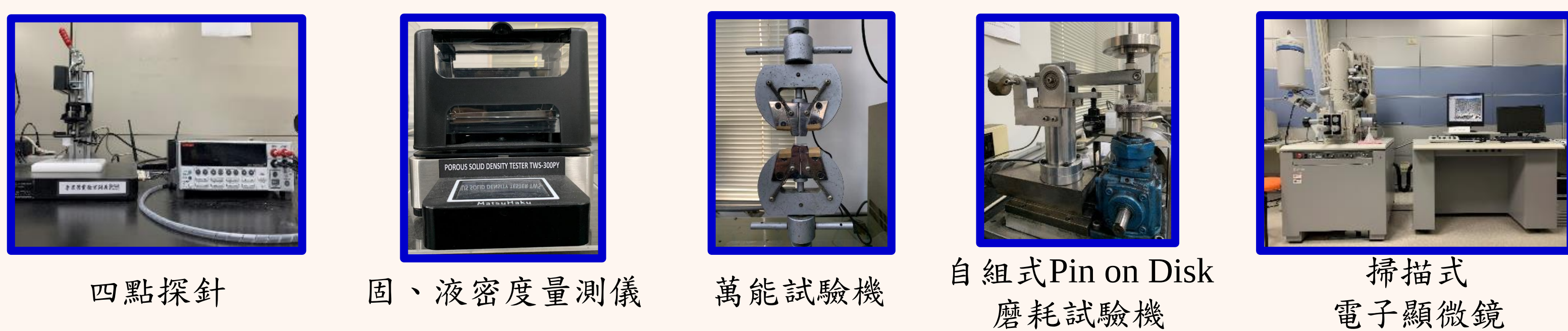
實驗方法

各成分比例與碳化時間表

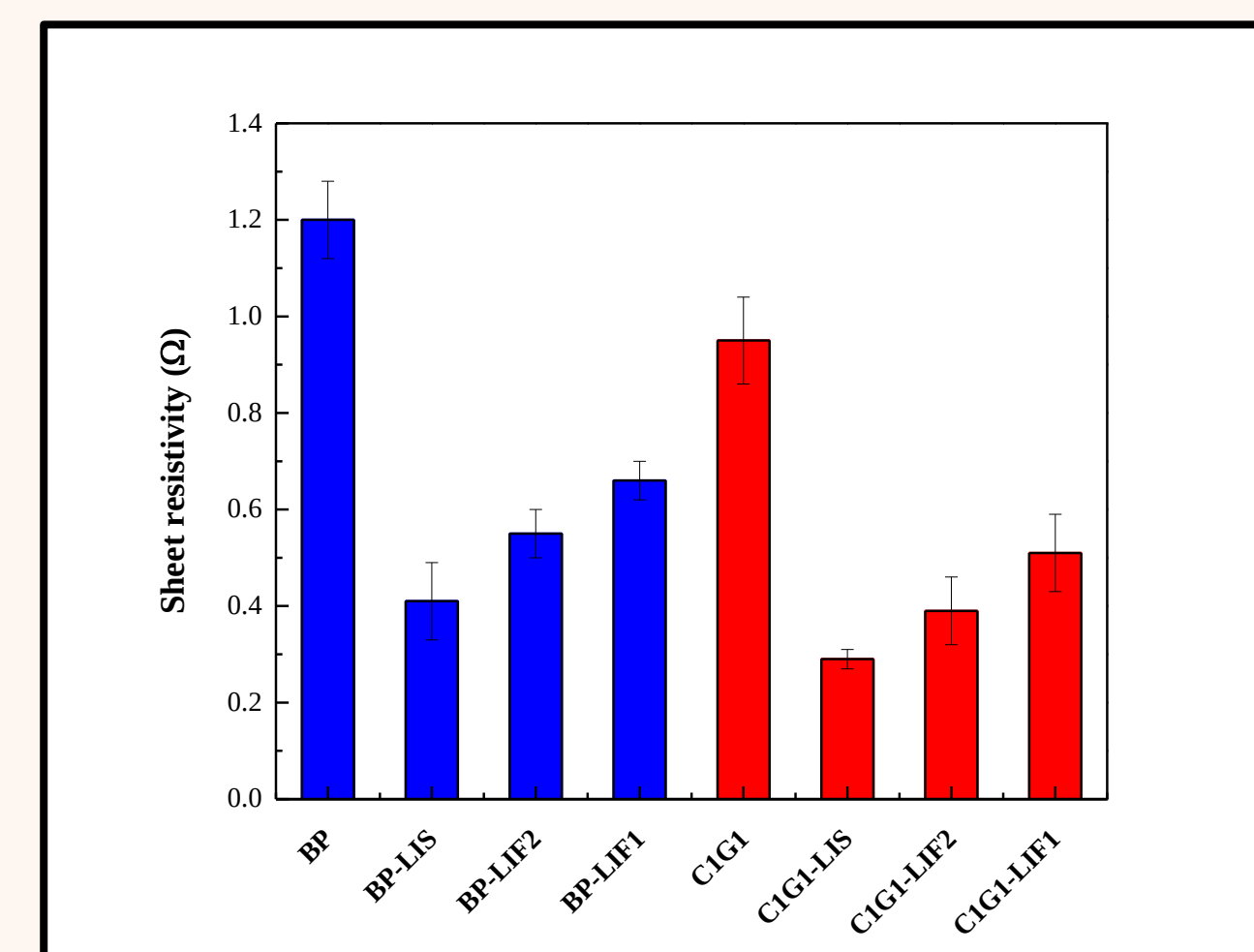
	比例	碳化時間 (min)
BP	CNT 0.08g	0
C1G1	CNT 0.04g + Graphene 0.04g	0
(BP、C1G1) - LIS	1. CNT 0.08g 2. CNT 0.04g + Graphene 0.04g	990
(BP、C1G1) - LIF2	1. CNT 0.08g 2. CNT 0.04g + Graphene 0.04g	166
(BP、C1G1) - LIF1	1. CNT 0.08g 2. CNT 0.04g + Graphene 0.04g	106



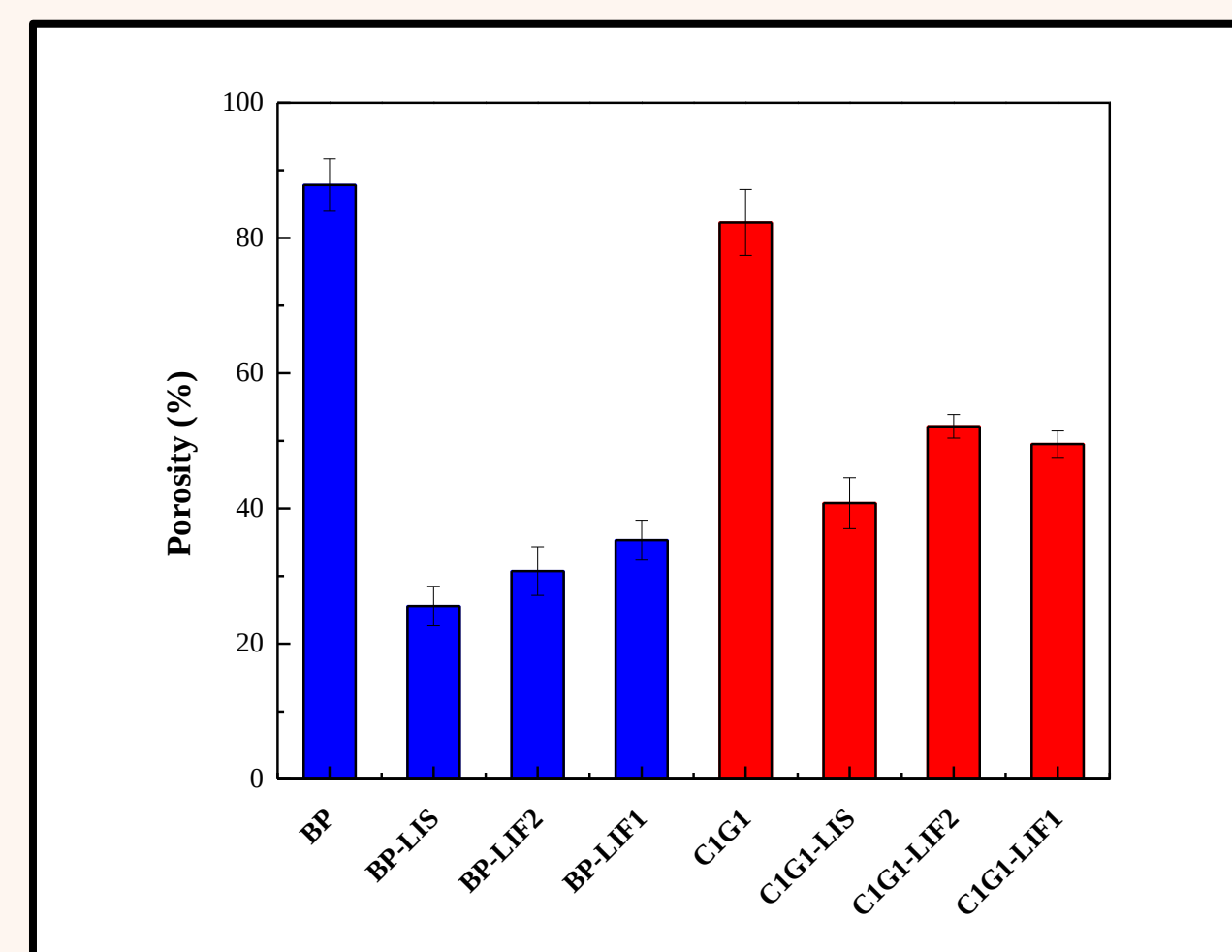
實驗流程圖



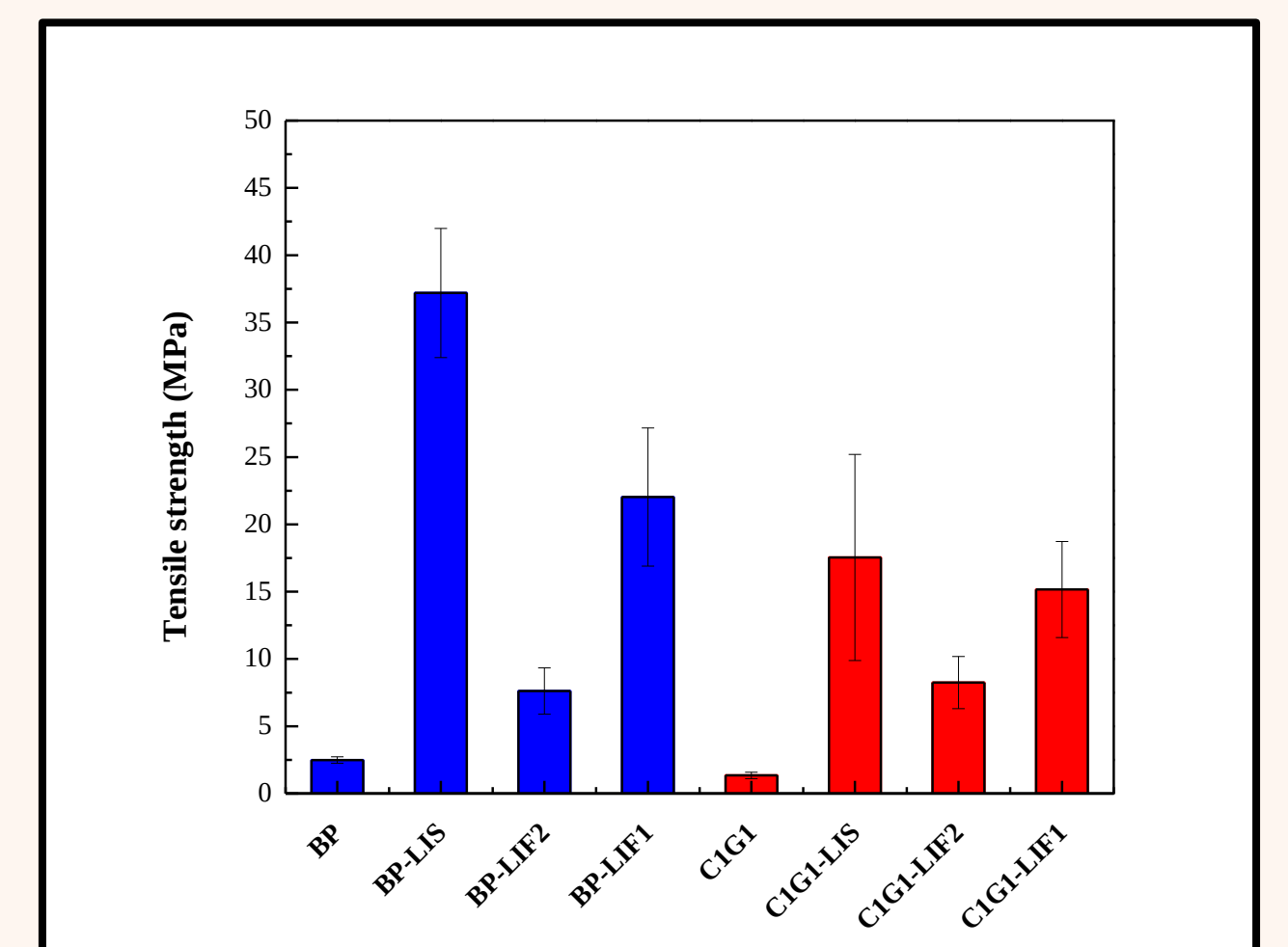
結果與討論



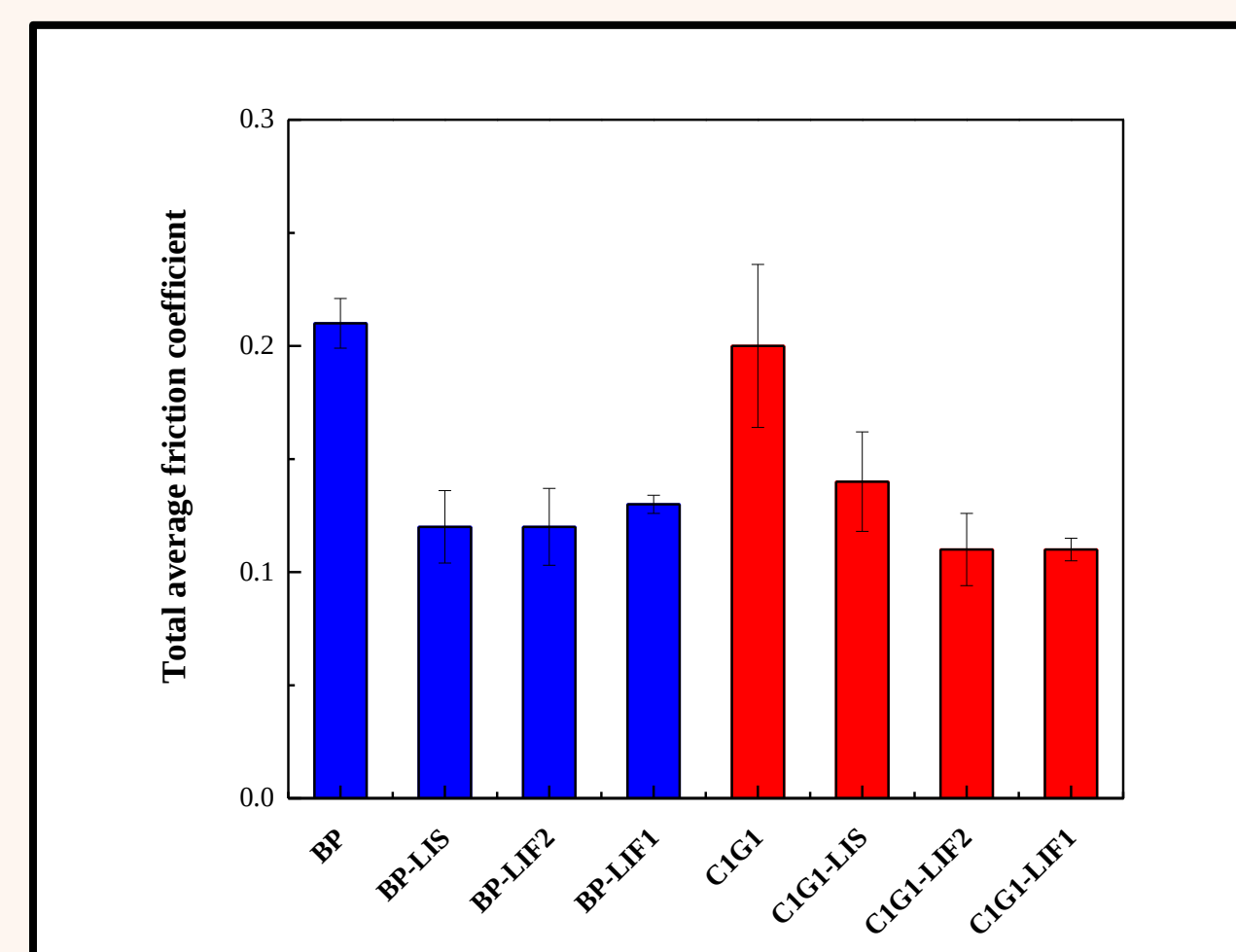
各類試片片電阻



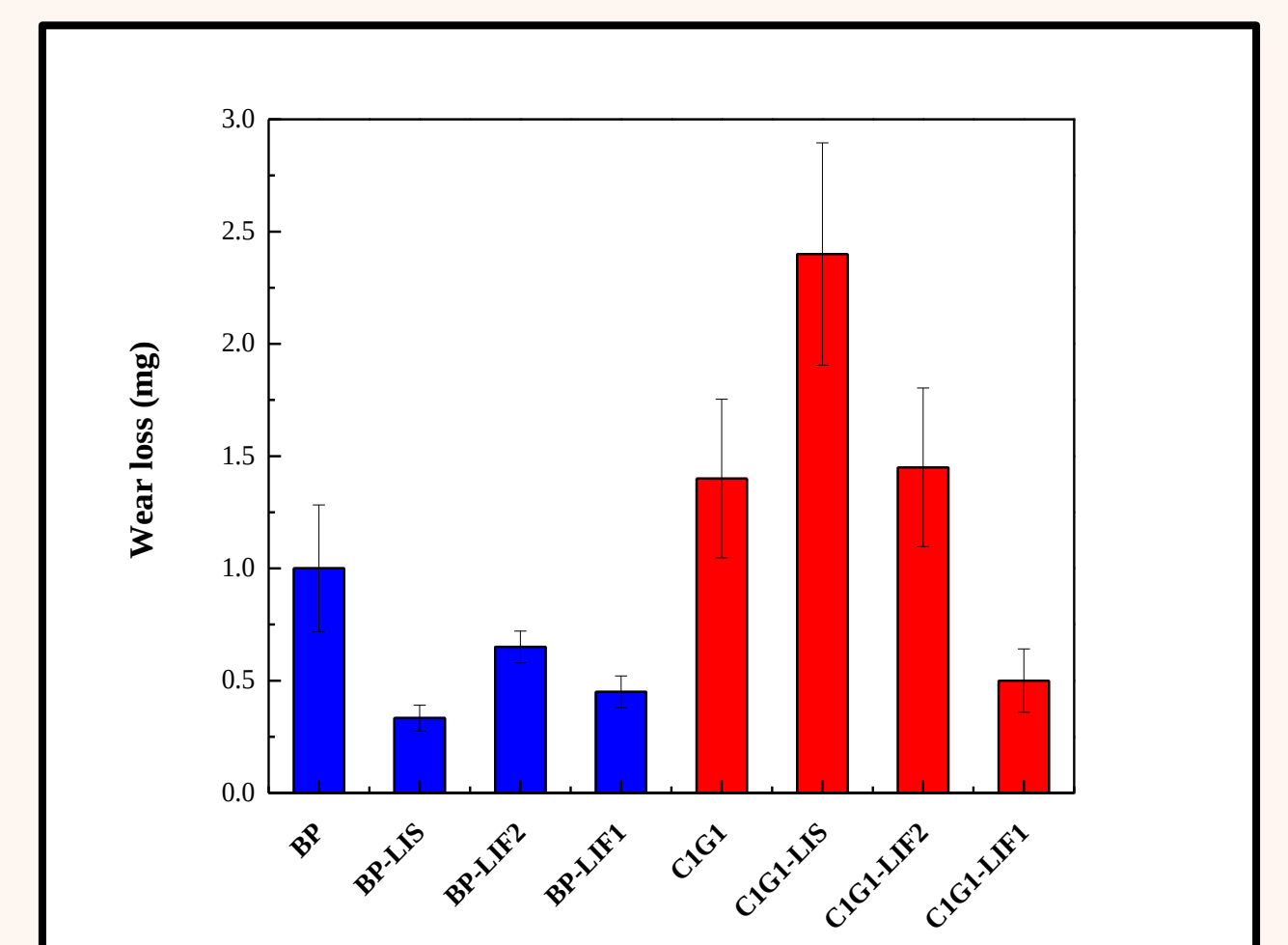
各類試片孔隙率



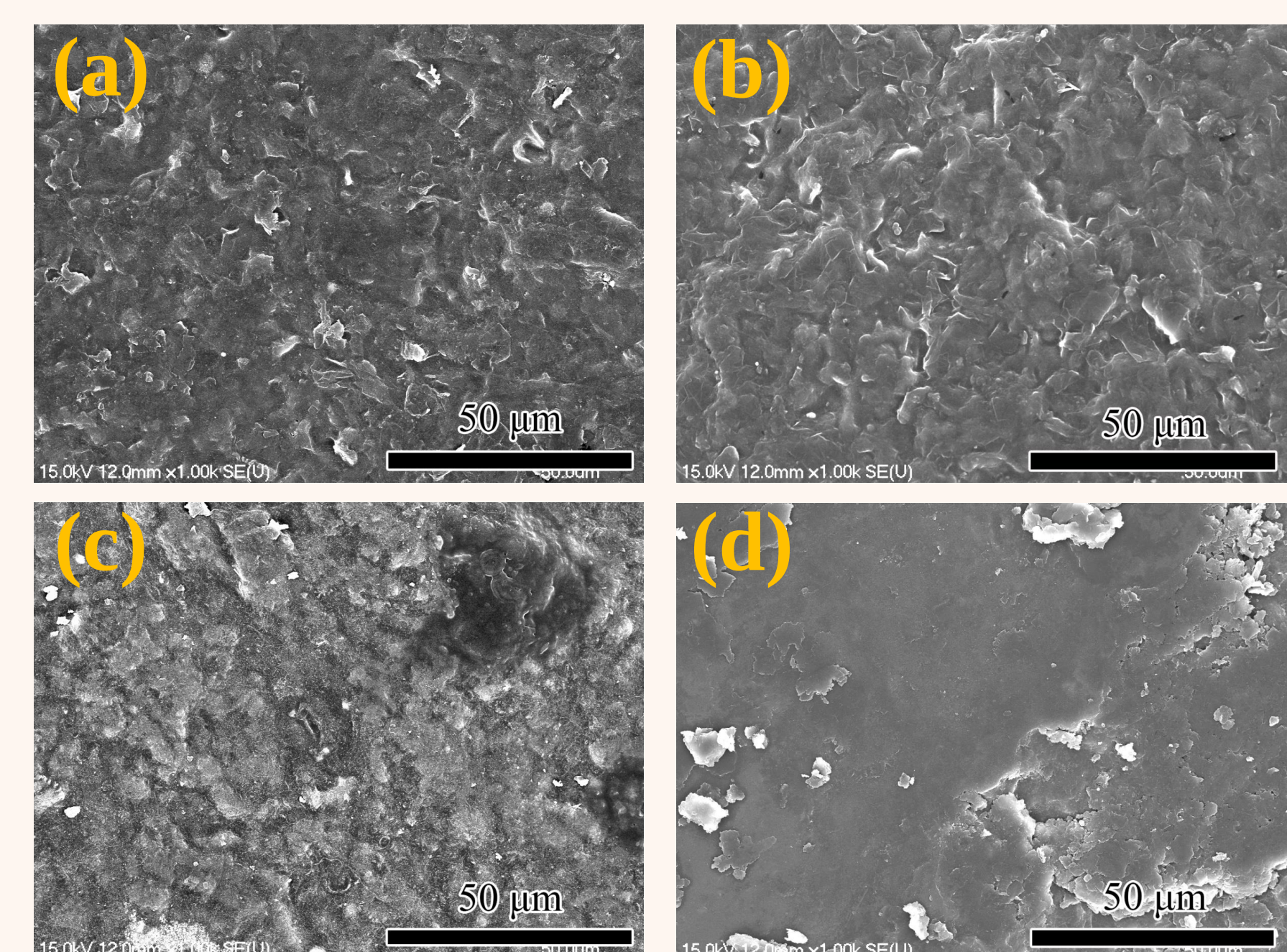
各類試片拉伸強度



各類試片磨擦係數



各類試片磨耗量



快速碳化1小時磨耗前後顯微結構

(a)磨耗前BP-LIF1 (b)磨耗後BP-LIF1

(c)磨耗前C1G1-LIF1 (d)磨耗後C1G1-LIF1

結論

- 1.在各類試片中，篩選出(BP-LIS、BP-LIF1、C1G1-LIF1)三種試片，在各項性質均為優異，其中以BP-LIS為最佳。
- 2.若考慮總製程時間的話，經快速碳化1hr試片在各方面性質與慢速碳化接近，後續也可藉由多次緻密化，改善快速碳化孔隙率高與抗拉強度低的缺點，所以BP-LIF1與C1G1-LIF1具有進步的空間。
- 3.在快速碳化製程中，添加石墨烯試片具有優異的低電阻值與穩定且低的磨擦係數，所以C1G1-LIF1為快速碳化中最佳材料。

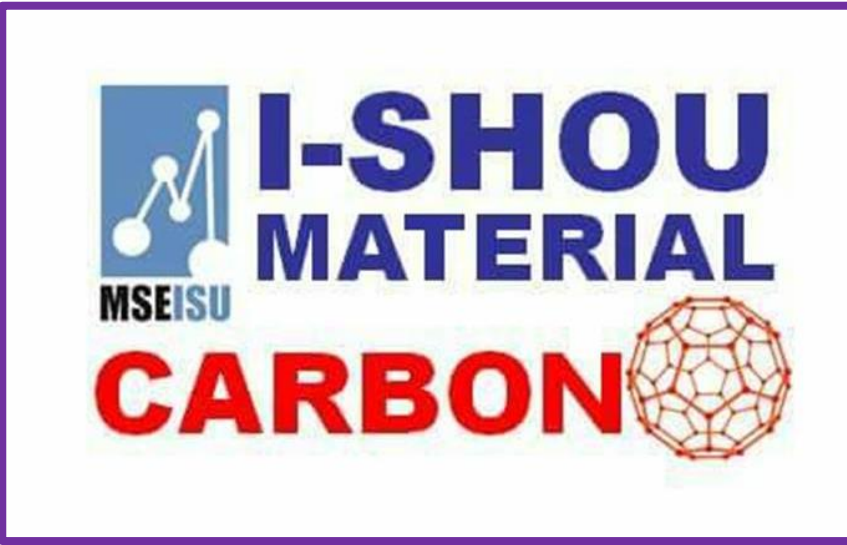
致謝

MOST108-2221-E-214-015)，也感謝義守大學貴重儀器中心的支援，使研究得以順利完成。

以可可殼及稻殼製備多孔生質碳電極之研究

陳奕廷 李奕昌 *李國榮 鄭鉅齊 王浚宇 顏世卿 許心怡 王瑄圻 張淑琪

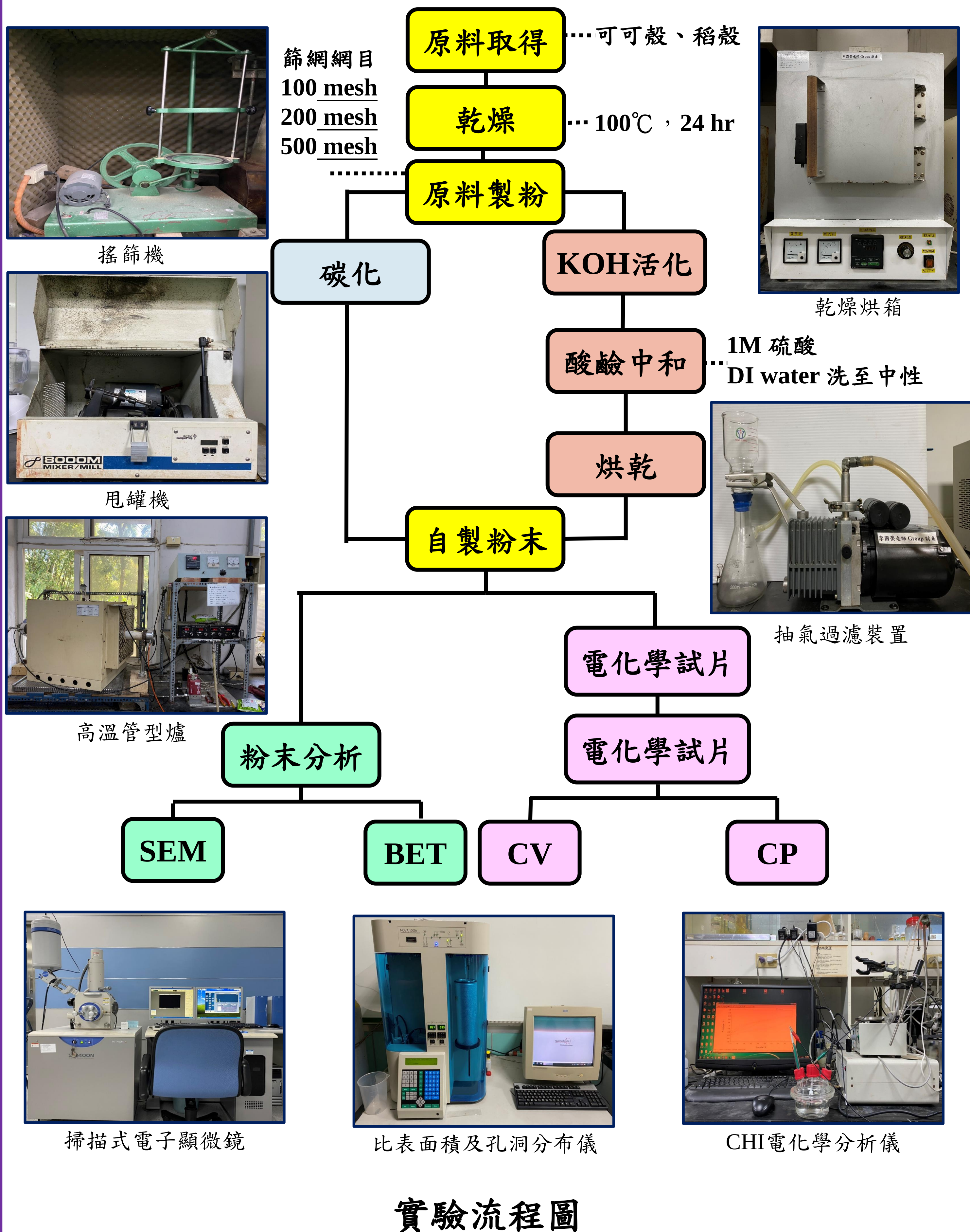
義守大學材料科學與工程學系



摘要

本研究中嘗試使用兩種不同的生質材料(可可殼、稻殼)進行碳化及活化表面改質方式製備為生質碳，以作為碳電極之應用。在本研究中探討活化的製程、粉體粒徑及材料的種類等參數，其比表面積、微中孔結構對比電容量之影響。研究結果顯示在三種粒徑試片當中(大、中、小)以小粒徑試片的比表面積與微中孔比例較好，當比表面積越大與微中孔比例越好時比電容也會跟著提升，研究發現在經過相同KOH比例的活化方式試片當中，以活化可可殼小粒徑試片的電化學性質最佳，各類材質當中以可可殼在電化學性質的表現最好。

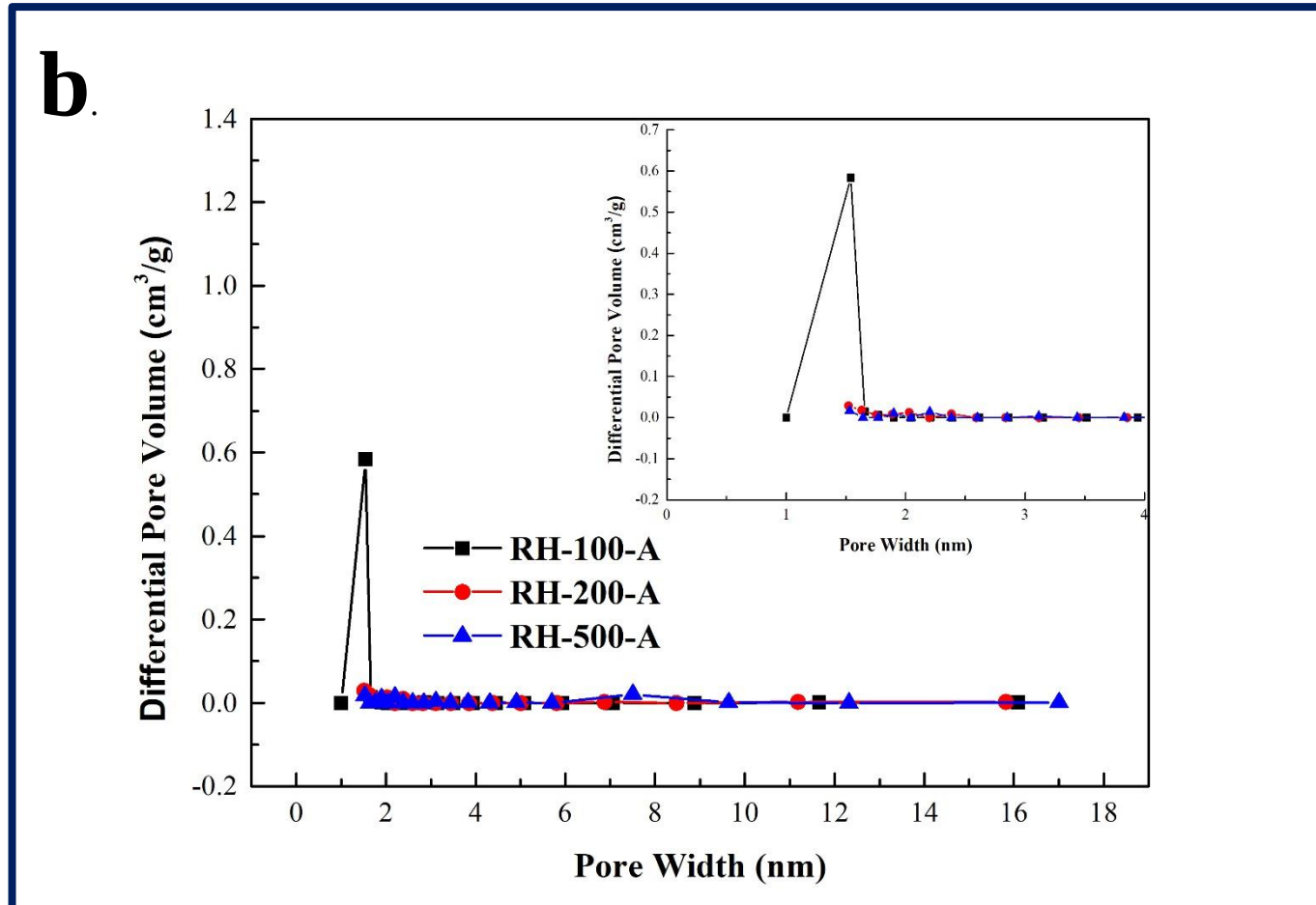
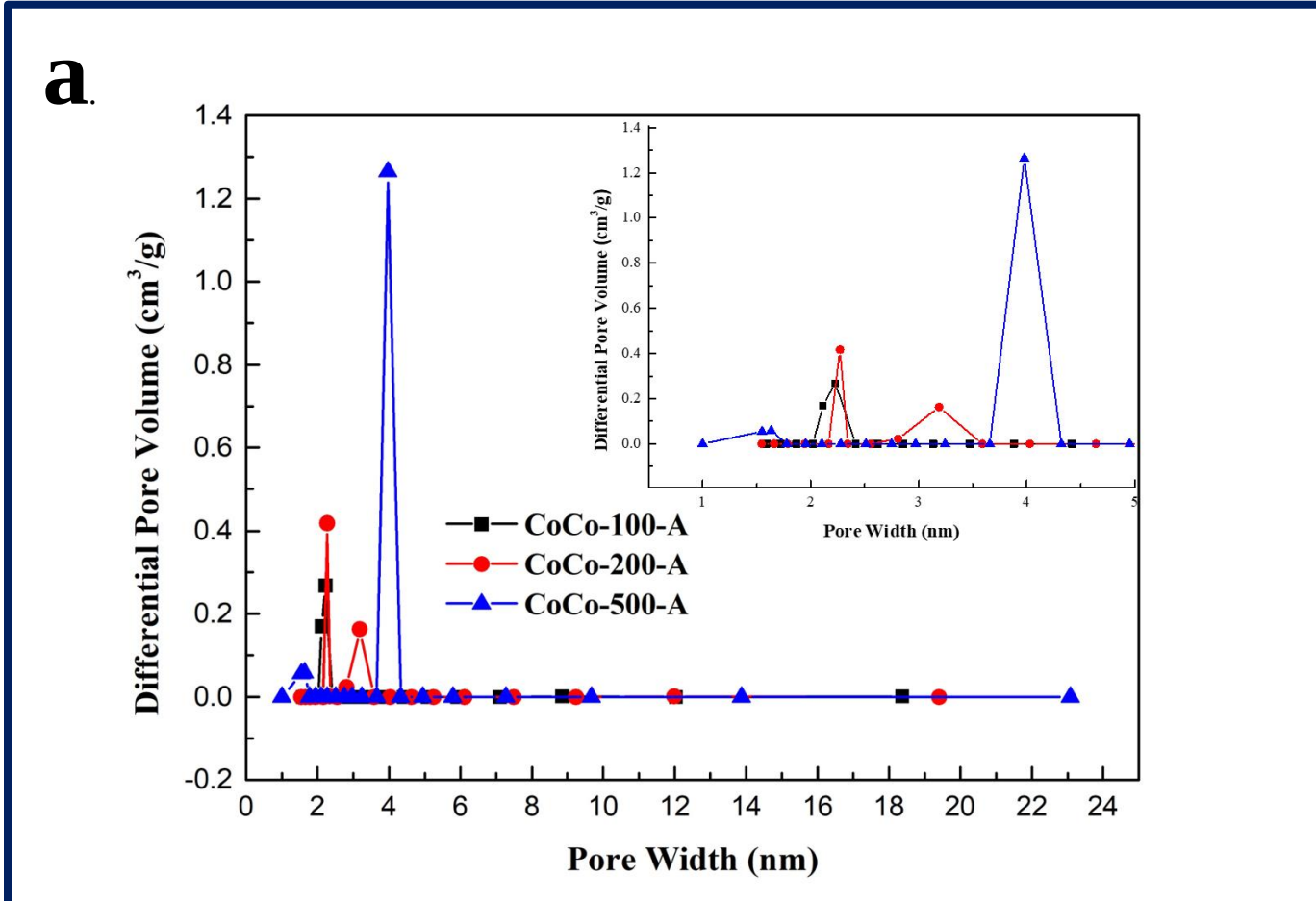
實驗方法



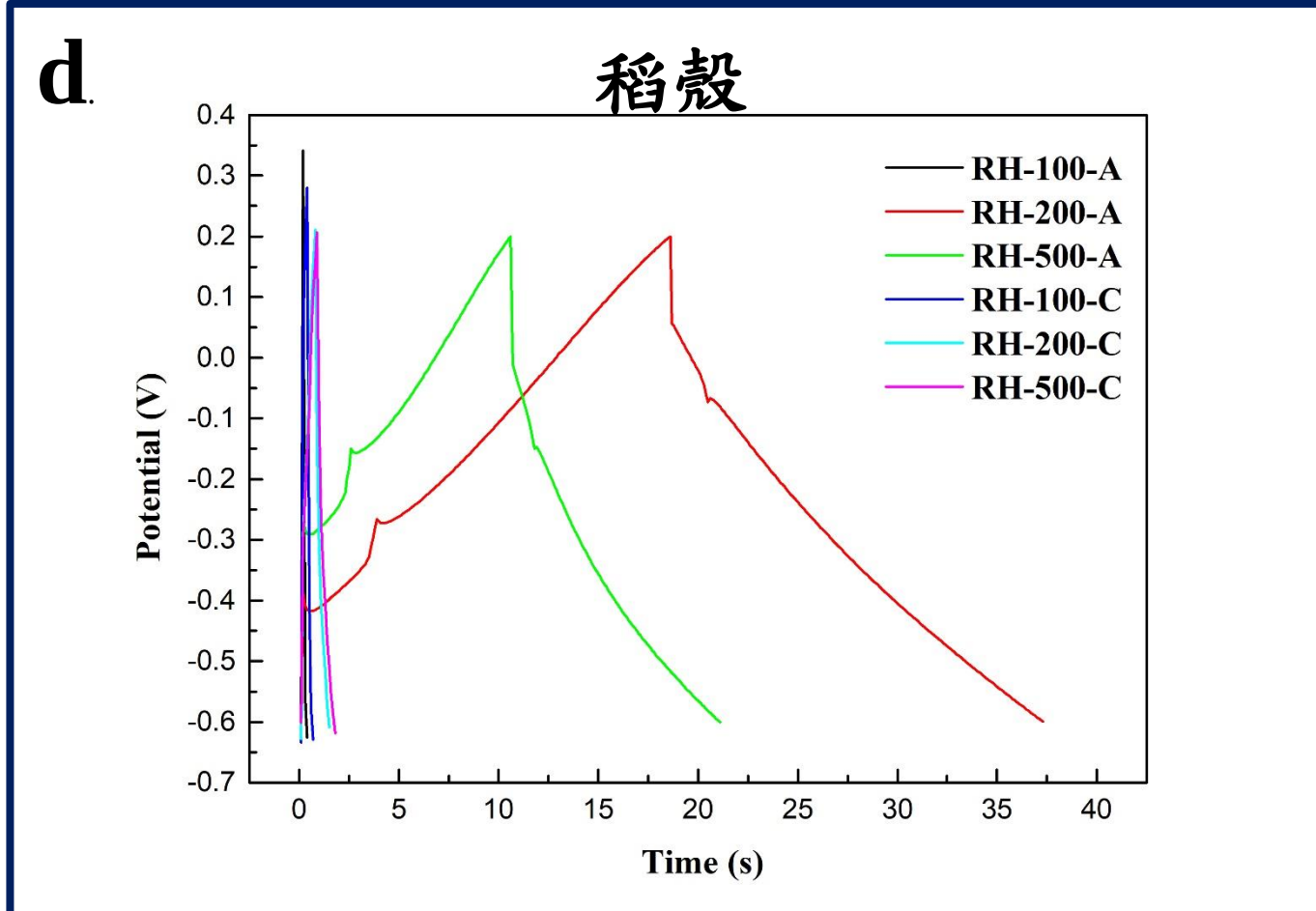
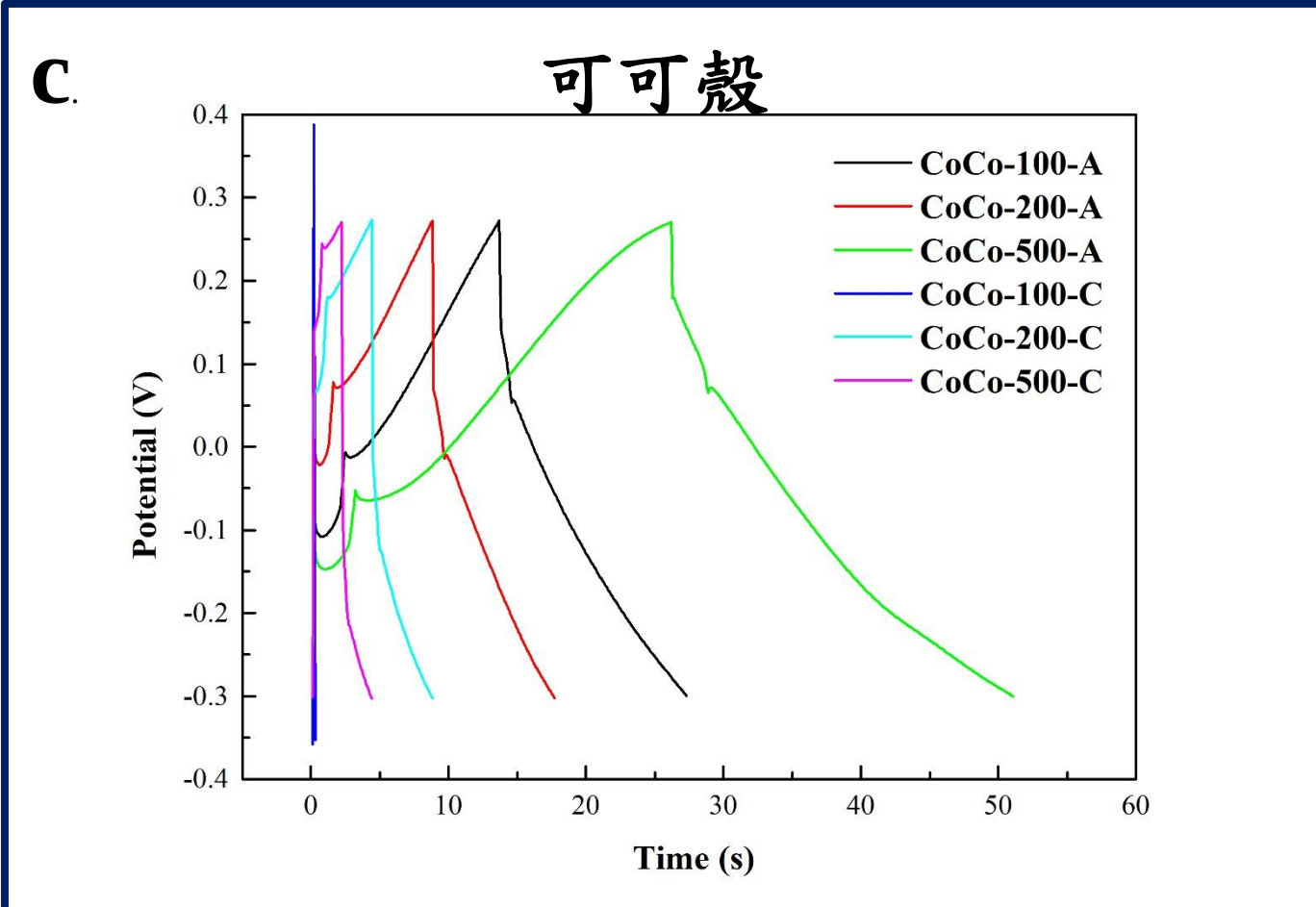
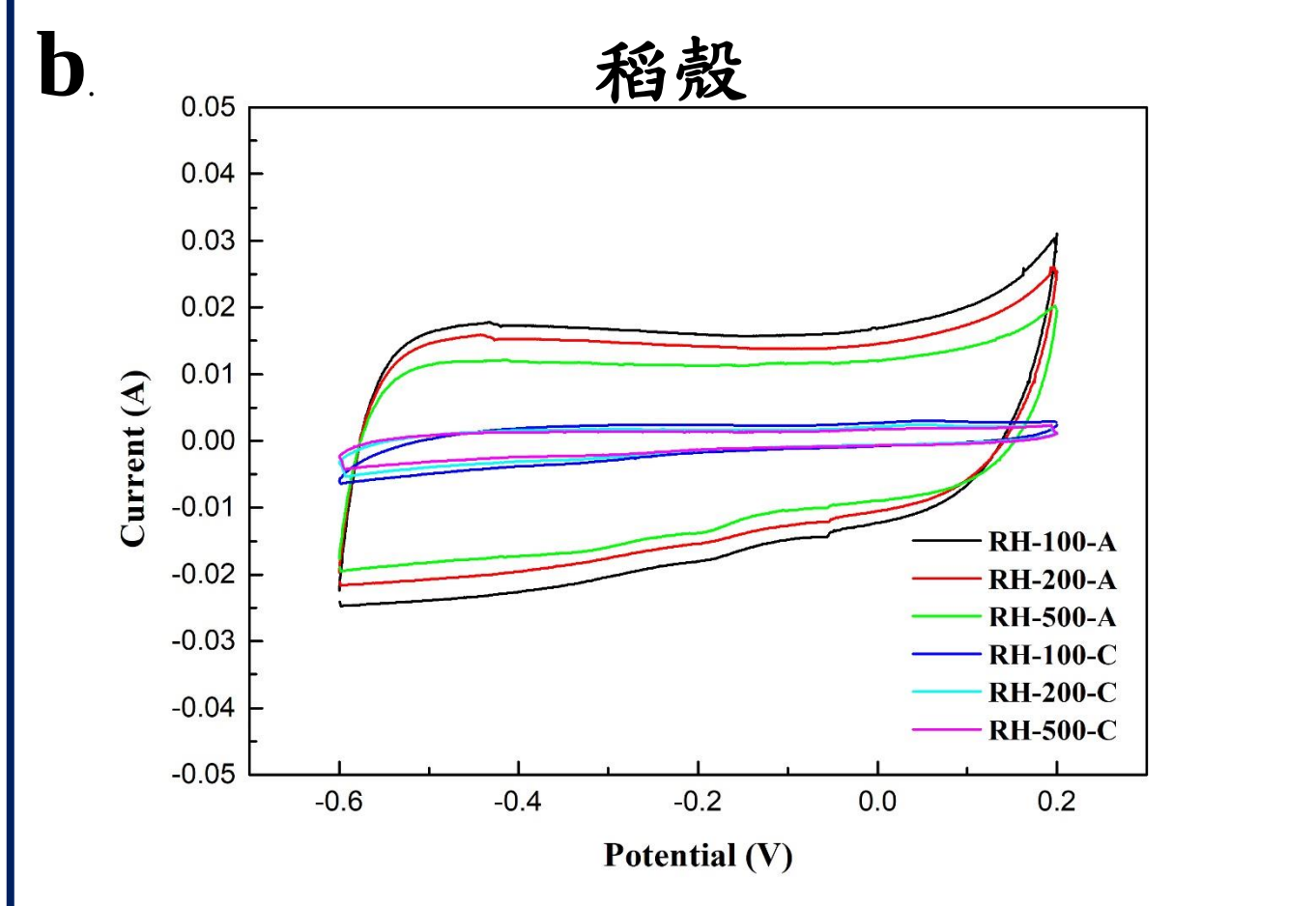
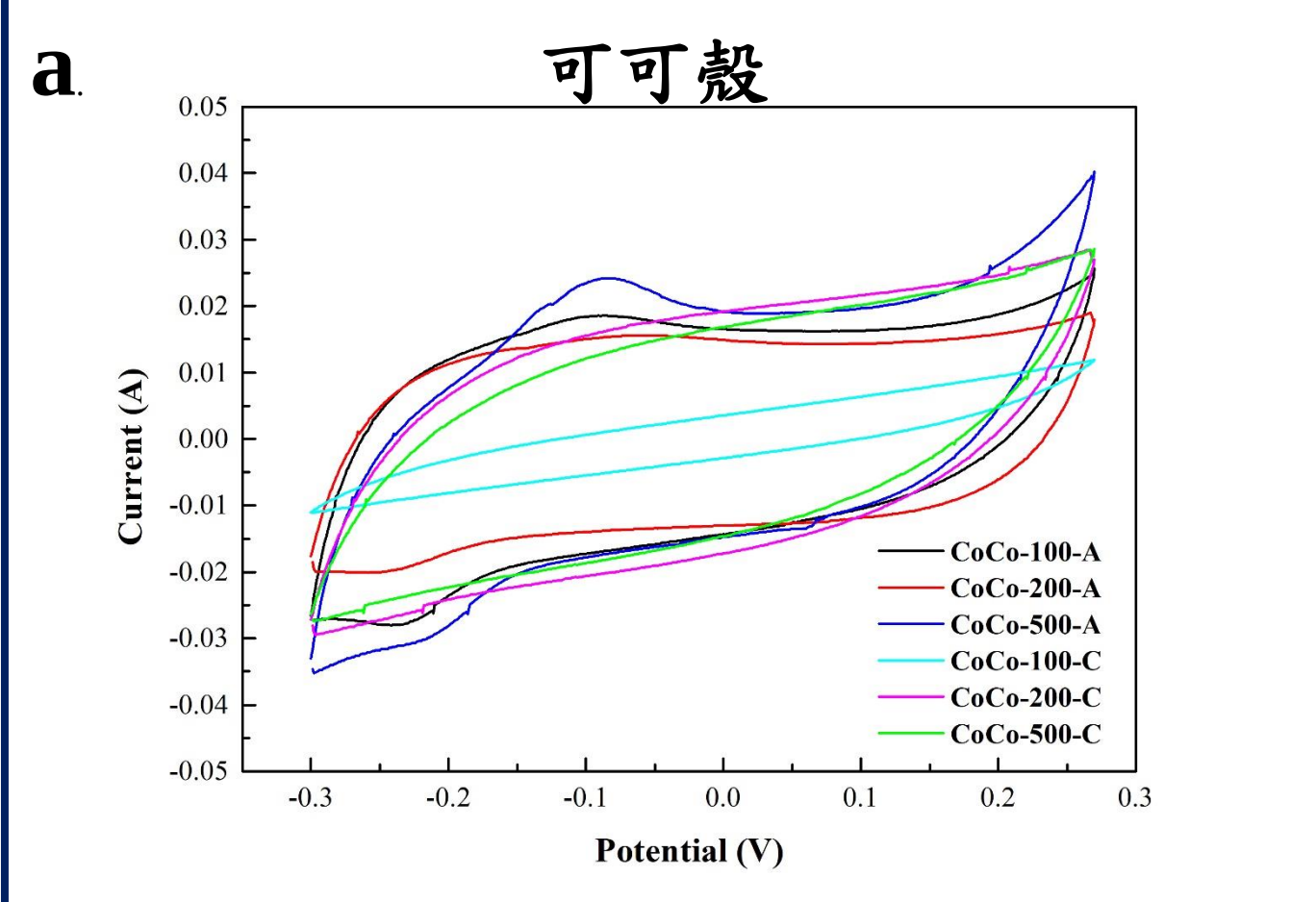
實驗流程圖

可可殼及稻殼活化之BET分析表

製程	可可殼活化後			稻殼活化後		
量測種類	100	200	500	100	200	500
比表面積	690.497 (m ² /g)	619.418 (m ² /g)	1561.097(m ² /g)	677.905 (m ² /g)	687.471 (m ² /g)	513.839 (m ² /g)
孔體積	0.059 (cm ³ /g)	0.101 (cm ³ /g)	0.181 (cm ³ /g)	0.085 (cm ³ /g)	0.027 (cm ³ /g)	0.070 (cm ³ /g)
微孔 (< 2 nm)	0 %	0 %	8.3 %	99.6 %	69.7 %	39.3 %
中孔 (2~50 nm)	100 %	100 %	91.7 %	0.4 %	30.3 %	60.7 %



比表面積分析，(a) 可可殼活化之孔徑分布圖、(b) 稻殼活化之孔徑分布圖



電化學量測，(a) 可可殼CV曲線圖、(b) 稻殼CV曲線圖、(c) 可可殼CP曲線圖、(d) 稻殼CP曲線圖

結果與討論

製程	碳化後		活化後	
生質材 目數	可可殼	稻殼	可可殼	稻殼
100				
200				
500				

各生質材料碳化及活化之SEM顯微結構圖

結論

- 由比表面積分析的結果顯示，可可殼活化500 mesh有最高的比表面積。
- 在可可殼活化改質方式的試片當中，以可可殼 500 mesh的電化學性質最佳。說明當比表面積越高，且具有中孔及微孔結構，其吸附電解液量越多，CV與CP之比電容值越好。

致謝

本研究承蒙科技部計畫(MOST 108-2221-E-214-015)及(MOST 109-2221-E-214-020)、義守大學貴儀中心的支持，研究得以順利進行，僅此致謝。

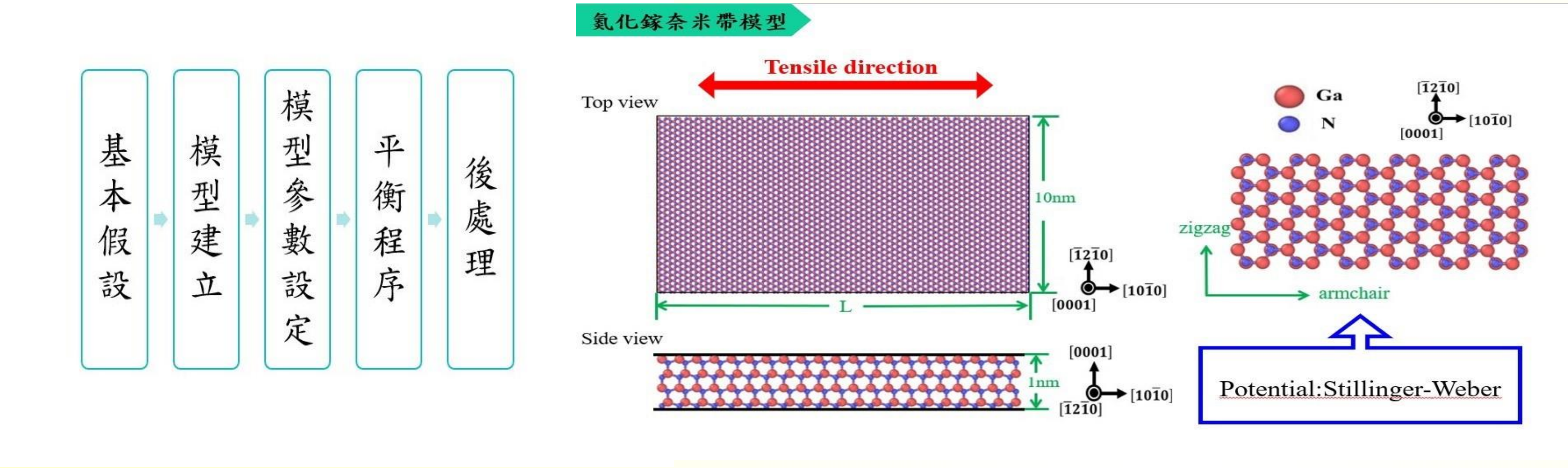
氮化鎵奈米帶之相變化行為研究

指導教授: 簡騰瑞 教授 專題生: 吳承翰、陳咨廷、蘇緣庭、李庭宇

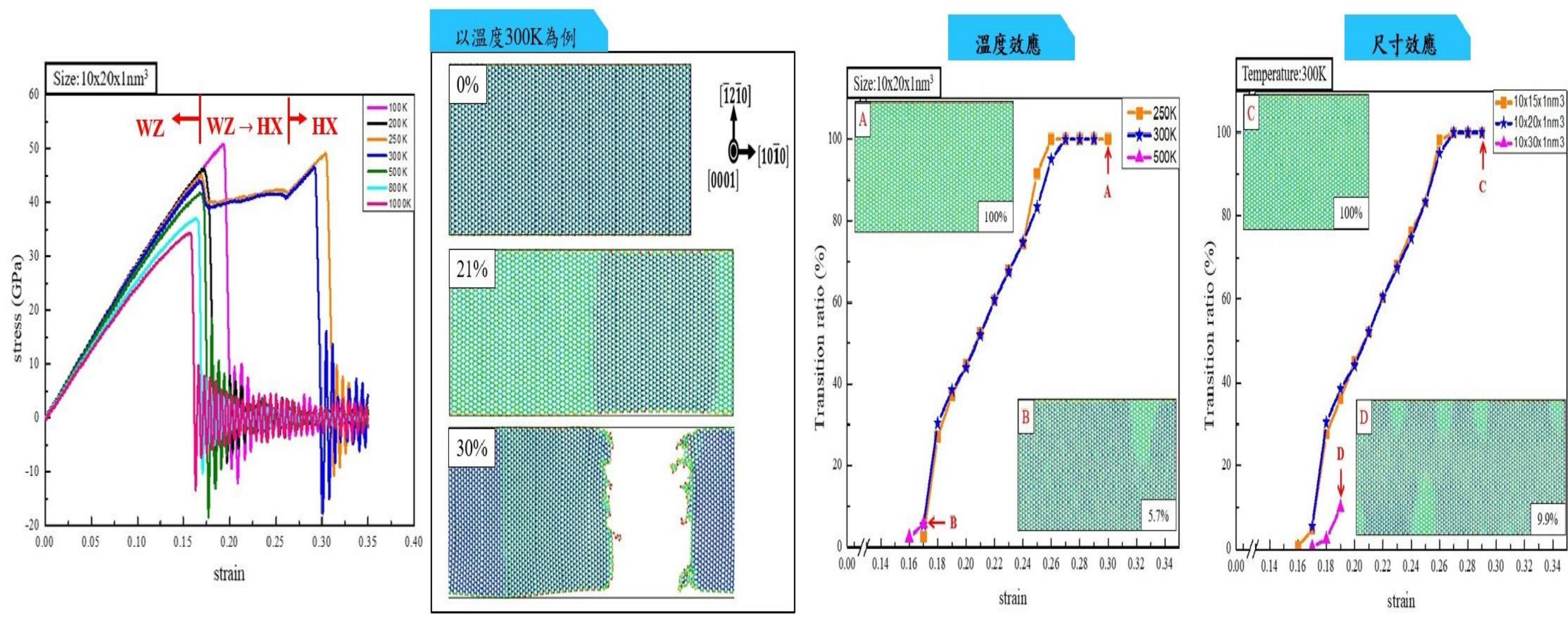
一.摘要

本研究以分子動力學模擬氮化鎵奈米薄膜在拉伸過程中之相變化行為與機制，並以Stillinger-Weber (SW) 势能函數來描述原子間的相互用力，探討不同軸向拉伸及溫度效應之影響。模擬結果顯示，在拉伸過程中，發現沿 armchair 軸向進行拉伸，其Wurtzite (WZ)結構轉換至Hexagonal (HX)結構(WZ→HX)之相變行為，則沿zigzag 軸向 拉伸，則未發生此種行為。其中，氮化鎵奈米帶會在300K(室溫)下，發生相變化行為，而高溫與低溫環境較不易發生，會因溫度影響原子排列結構，無法引發相變轉化的行為。

二.模擬步驟



三.結果與討論



四.結論

- 當溫度低於250K或高於500K均不會發生相變化。
- 在溫度250K、300K下，其WZ極限應力分別為45.1GPa、43.8GPa，HX極限應力分別為49.2GPa、46.6GPa。
- 在 $10 \times 15 \times 1\text{nm}^3$ 尺寸下，會以每1應變下遞增9的相變轉化率。然而， $10 \times 20 \times 1\text{nm}^3$ 尺寸下，則會以每1應變 % 下遞增7的相變轉化率。但是， $10 \times 30 \times 1\text{nm}^3$ 尺寸下，相變轉化率只會達到9.9%。

Study on the properties of LaSrCuOS films prepared by rf-sputtering

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In this experiment, the Lanthanum Copper Oxide Sulfur (LaSrCuOS) films were deposited on quartz substrates by using the RF magnetron sputtering. The preparation of LaSrCuOS films was carried out by the two-step sulfurization process. The first step is to fix the substrate temperature and the gas volume percentage in the argon-oxygen atmosphere to deposit the LaSrCuO film. The second step is to change the sulfurization temperature (700°C, 750°C, 800°C, 850°C) and the Sr content in the LaSrCuOS film (25%, 27%, 30%, 33%, 35%). Then the thin films are scrutinized by employing different analytic instruments of XRD, SEM, UV-Visible and Hall Effect to explore the effect of sulfurization treatment on the properties of LaSrCuOS films.

Experimental

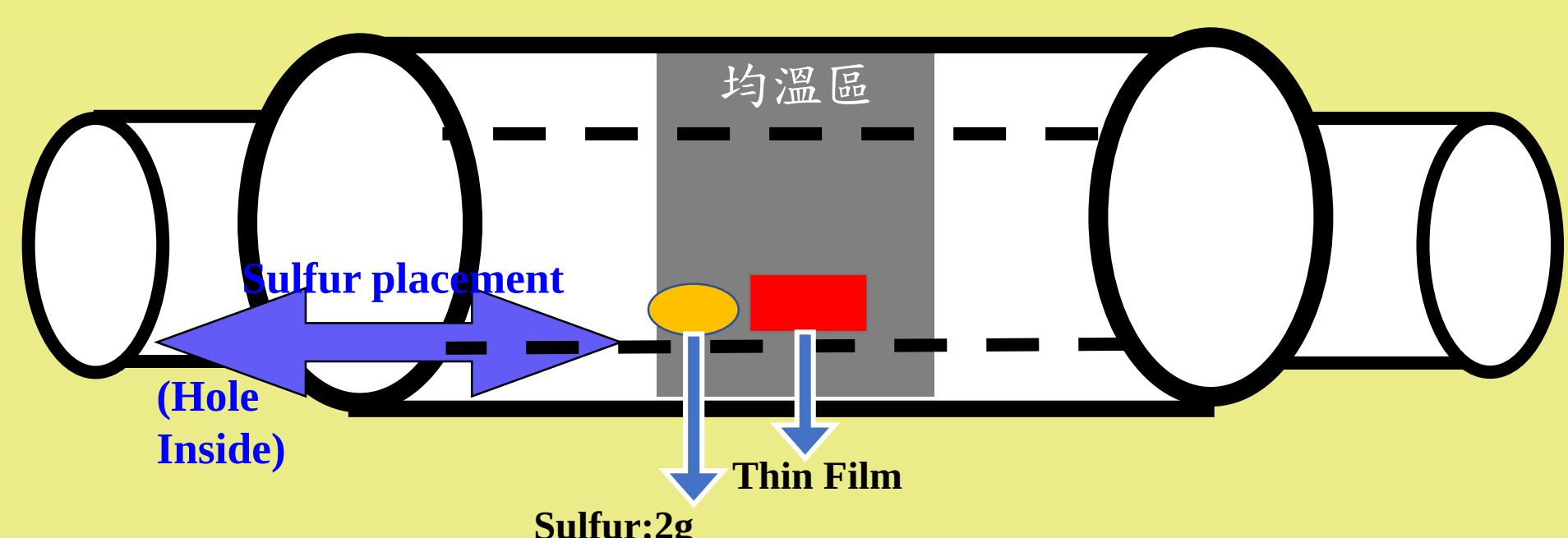


Fig1. The quartz tube structure for the sulfurization treatment

SEM

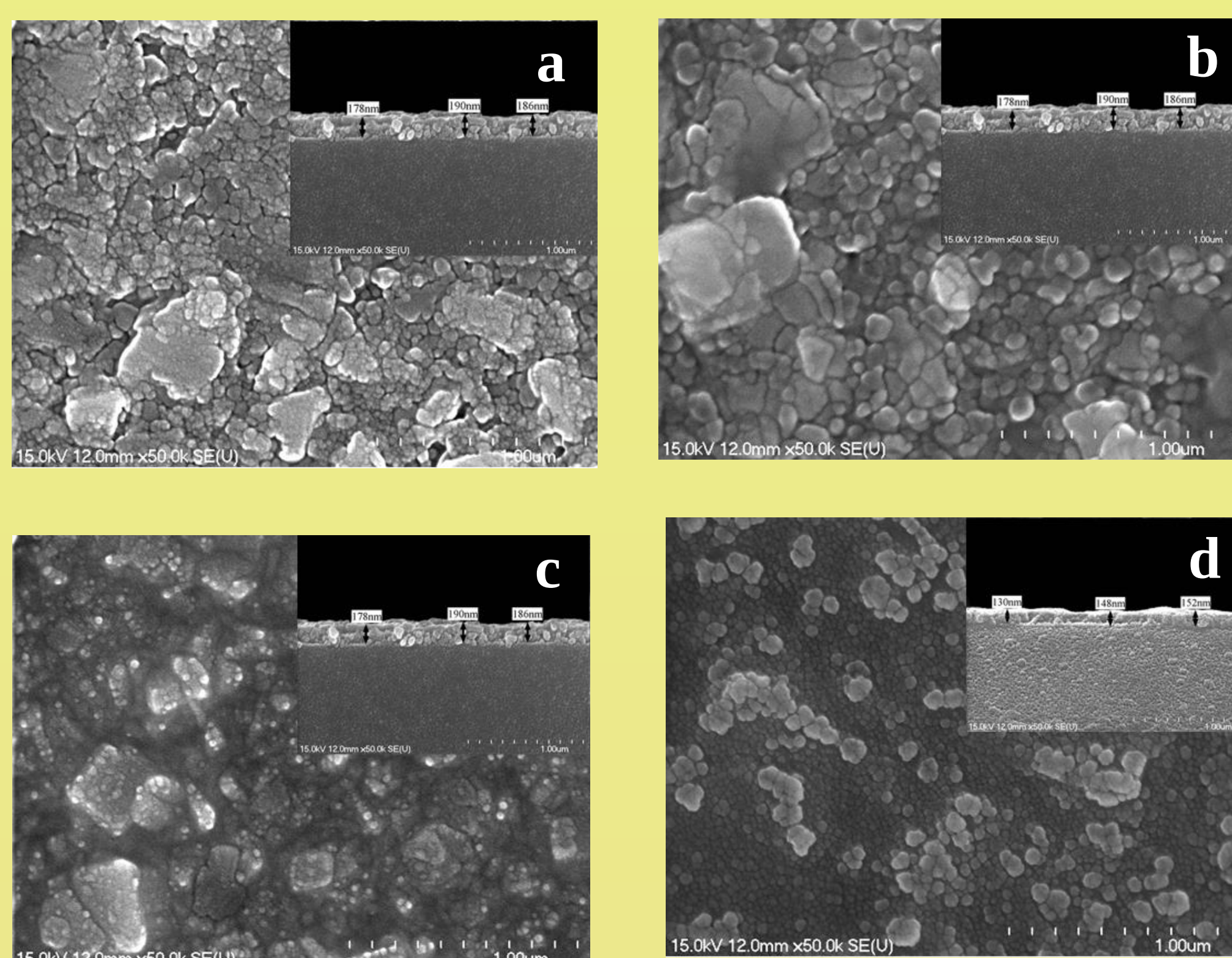


Fig.5 The plane and cross-section SEM analysis of La-Sr-Cu-O-S films, by changing the sulfurization : (a)700°C , (b)750°C , (c)800°C , (d)850°C

XRD

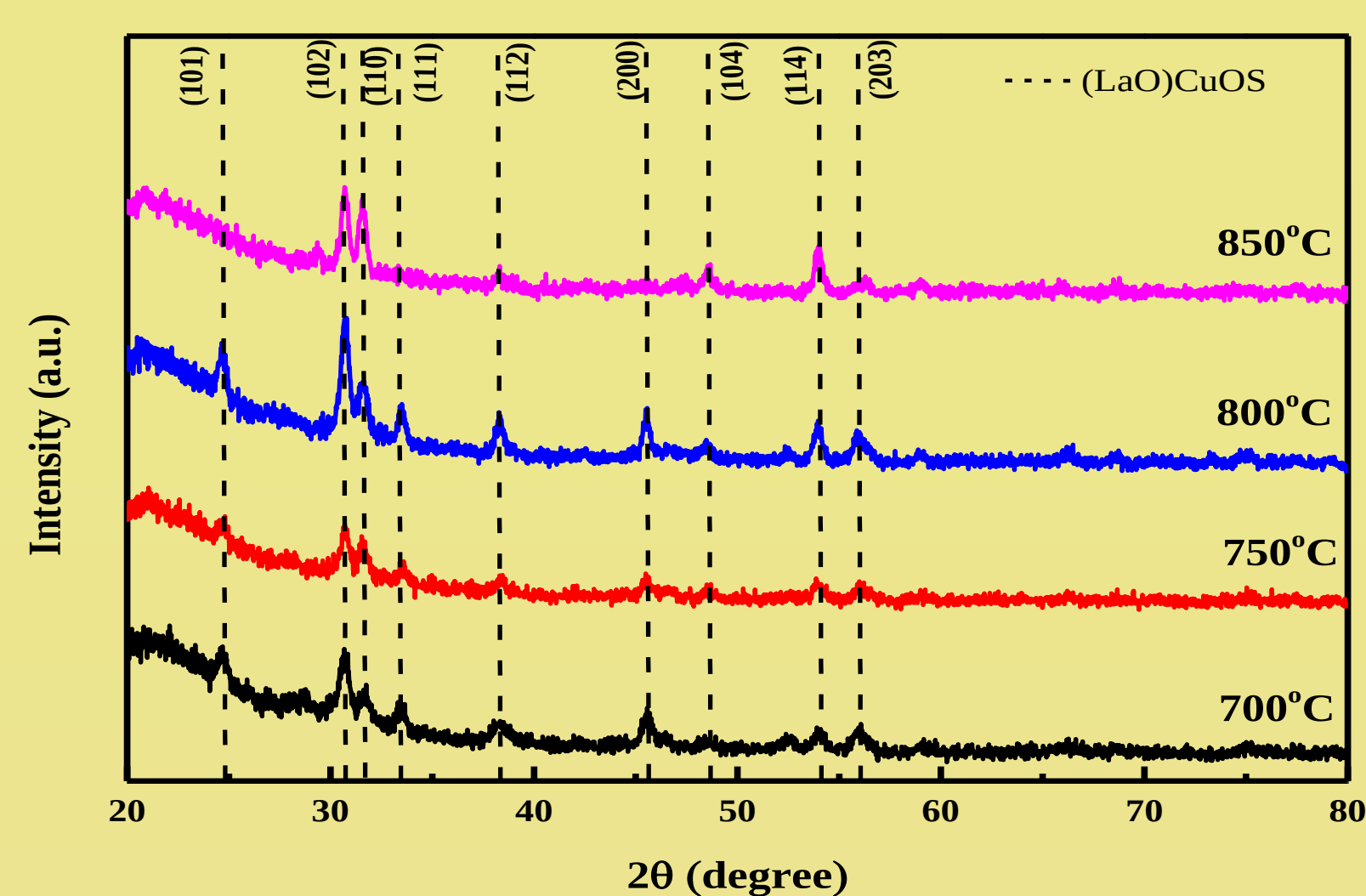


Fig.2 The XRD patterns of La-Sr-Cu-O-S films, by changing the sulfurization temperature

Hall Measurement

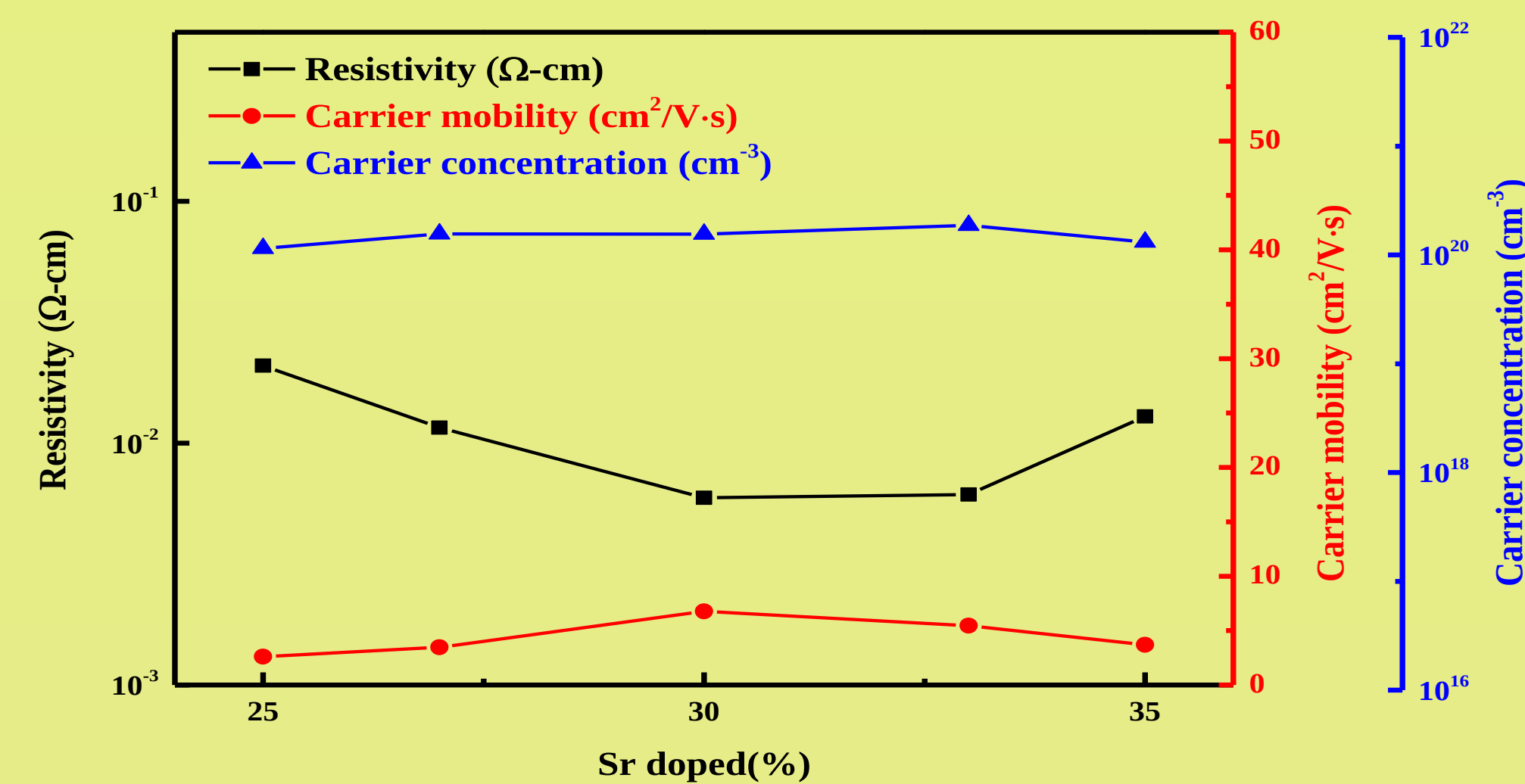


Fig.9 The Hall Measurement of La-Sr-Cu-O-S films, by changing the Different doping concentration

Hall Measurement

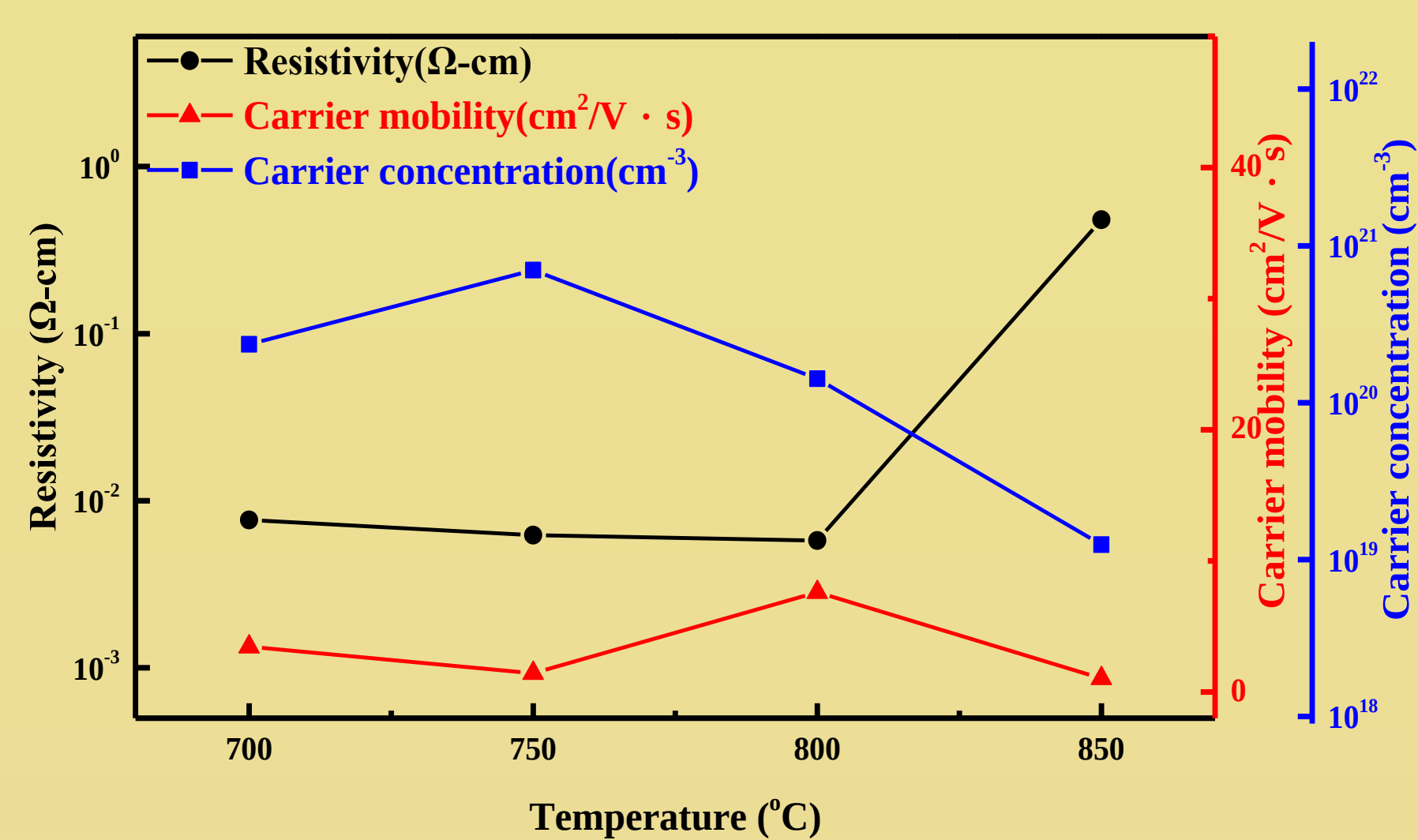


Fig.3 The Hall Measurement of La-Sr-Cu-O-S films, by changing the sulfurization temperature

UV-Visible&Energy Gap

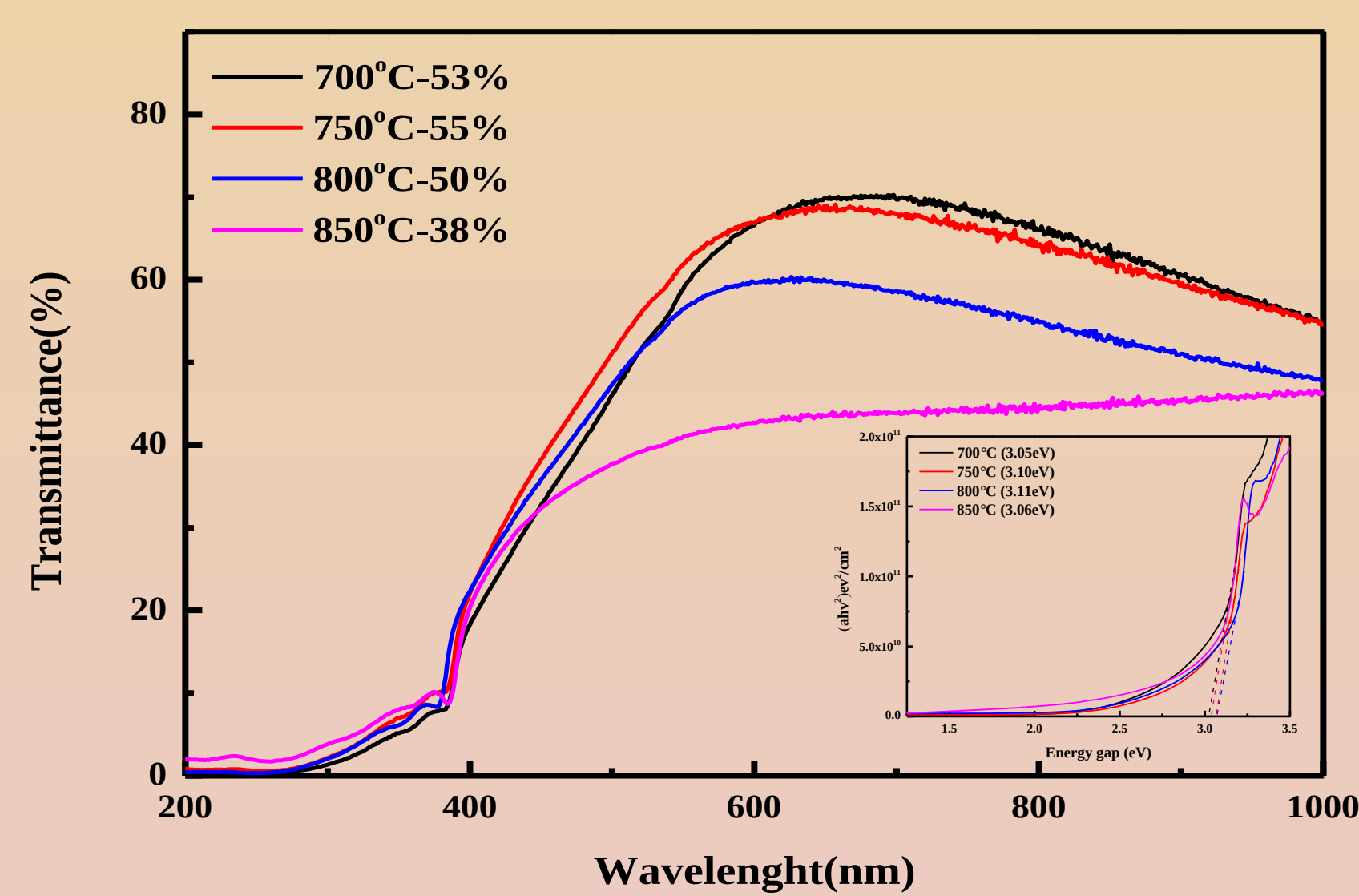


Fig.4 The UV-visible spectra and the band gap energy of La-Sr-Cu-O-S films, by changing the sulfurization temperature

FOM

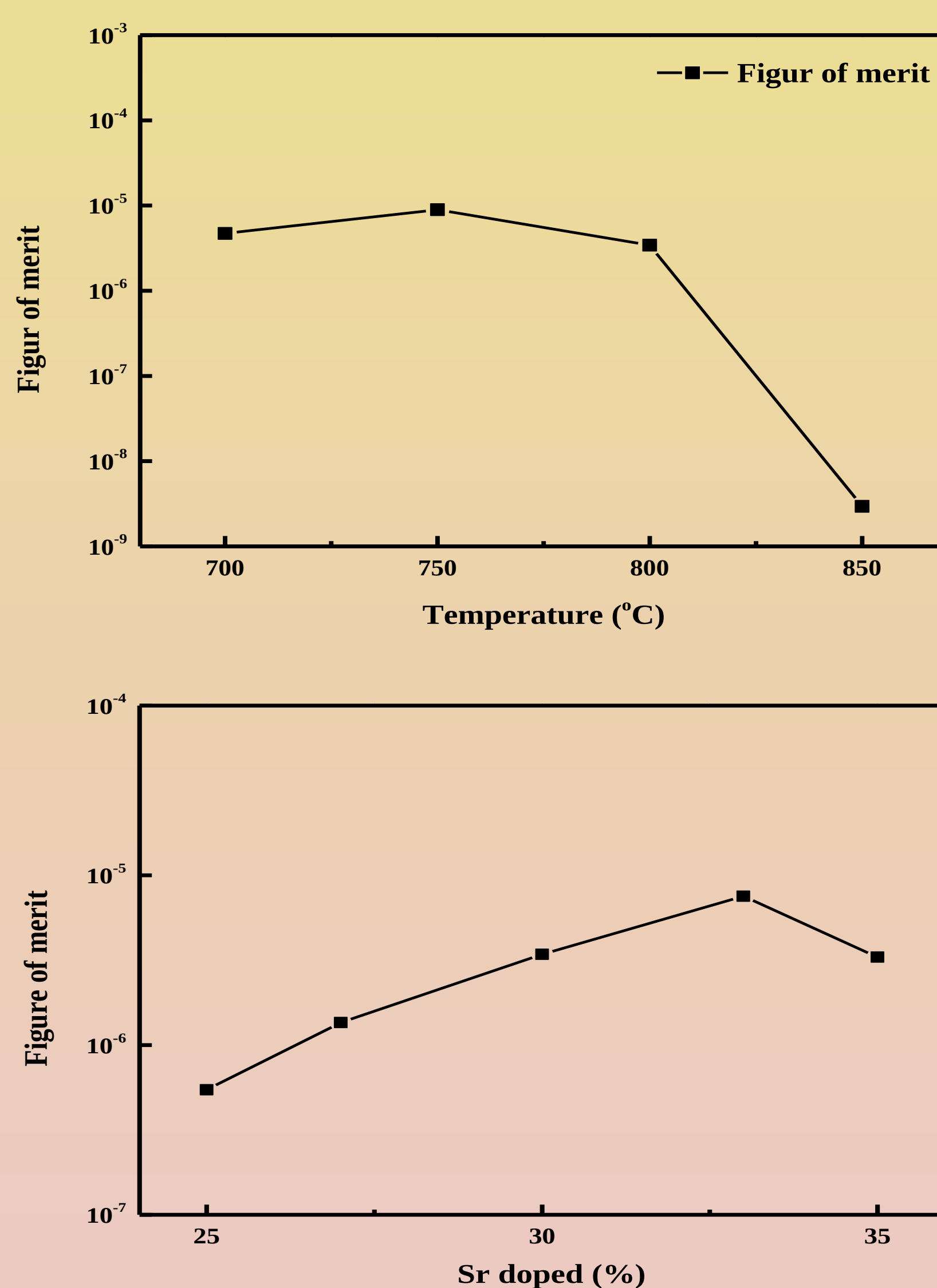


Fig 7. The FOM patterns of La-Sr-Cu-O-S films, by changing the sulfurization temperature and different doping concentration

Conclusions

The experimental results show that when 30% at.% Sr is doped, the main phase of LaCuOS can be obtained by sputtering deposition process at room temperature under 50% argon content. In the subsequent sulfurization process, by changing the sulfurization temperature (700°C ~ 850°C), the thin films are thoroughly investigated. As the sulfurization temperature increases, the grain size first increases respectively (264nm, 268nm, 270nm, 272nm). The optical properties first increase and then decrease (from 53% to 55% to 38%). The sulfurization temperature is 750°C, the band gap energy is 3.1 (eV), the best resistivity is 6.23×10^{-3} (Ω-cm), the carrier mobility is 1.43 (cm² / Vs) and the carrier concentration is 6.99×10^{20} (cm⁻³), can achieve the best quality factor (FOM).

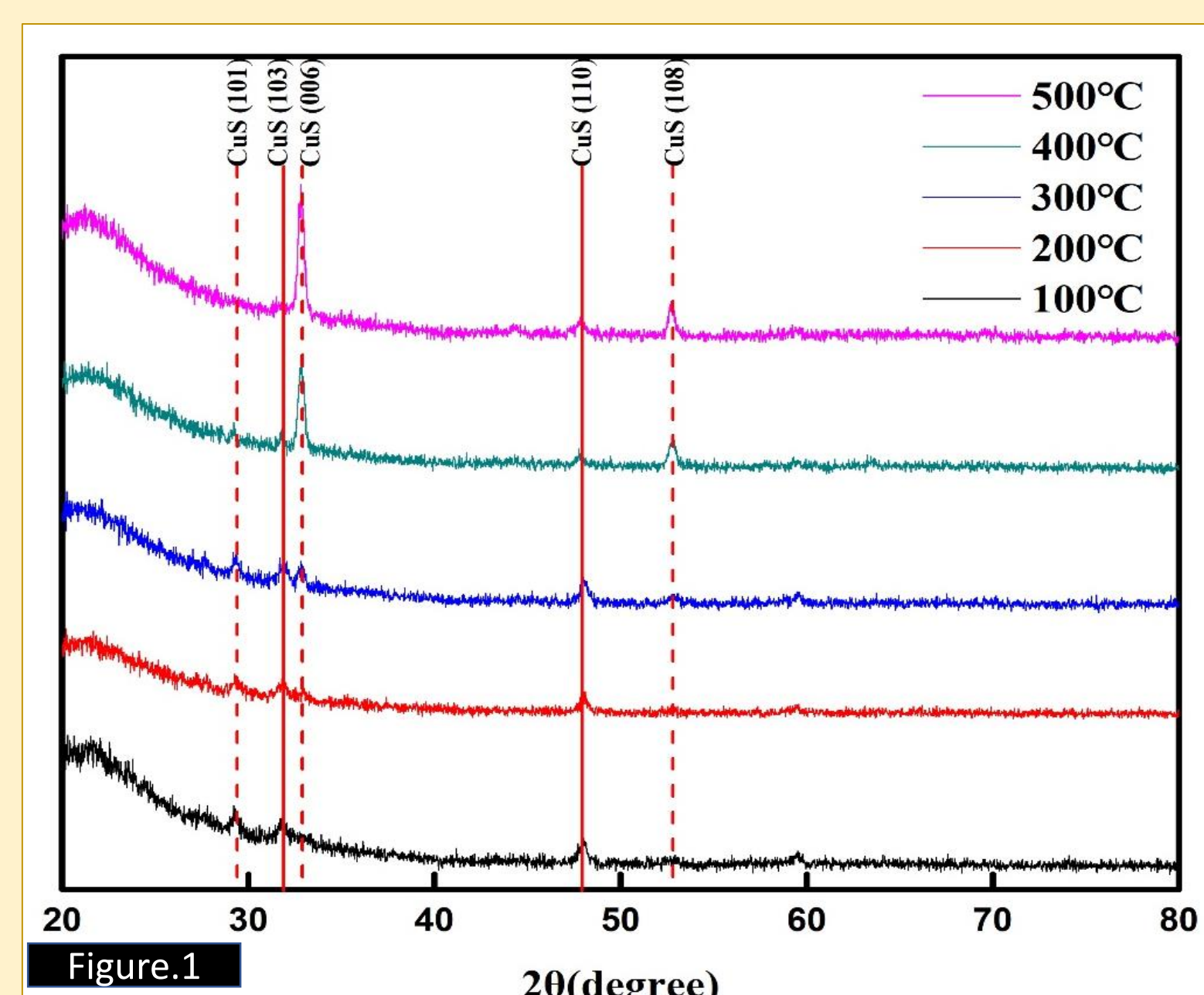
After depositing the LaSrCuO film at room temperature and 50% atmosphere ratio of argon by change of Sr doping amount (25%~35%) the thin films are thoroughly investigated. The experimental results show that the main LaCuOS phase. When Sr doping is increased to 30% at.% the optimal resistivity is 5.95×10^{-3} (Ω-cm), carrier mobility is 6.76 (cm²/Vs) and the carrier concentration is 1.55×10^{20} (cm⁻³). When Sr doping is increased to 33% at.%, the average transmittance in the visible light region increases from the lowest point 47.38% to 54.50%. Although the Sr doping increased to 33% at.% the resistivity is not good enough, after the calculation of FOM factor, the Sr doping increased to 33% at.% is the best because of its overriding effect of good optical property. The role of its quality factor is 7.54×10^{-6} .

Study on prepavation and properties of sulfide copper films by thermal evaporation

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Shih Yun Hui Min-Syuan Hou Wan-Jia Chen
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In this experiment, the sulfide copper films were deposited on quartz glass substrates by thermal evaporation. we control sulfide temperature (100°C, 200°C, 300 °C, 400 °C, 500 °C) to discuss the temperature influence of CuS film. experiment result to express that the best condition is 400°C. CuS film band gap energy is 2.35 eV and 2.8 eV. Optical transparency arrived 65%. It is all conditions best. Resitivity of $4.7 \times 10^{-3} (\Omega\text{-cm})$, Carrier mobility of $3.81 (\text{cm}^2/\text{Vs})$, Carrier concentration of $7.42 \times 10^{20} (\text{cm}^{-3})$ also is best conditions .

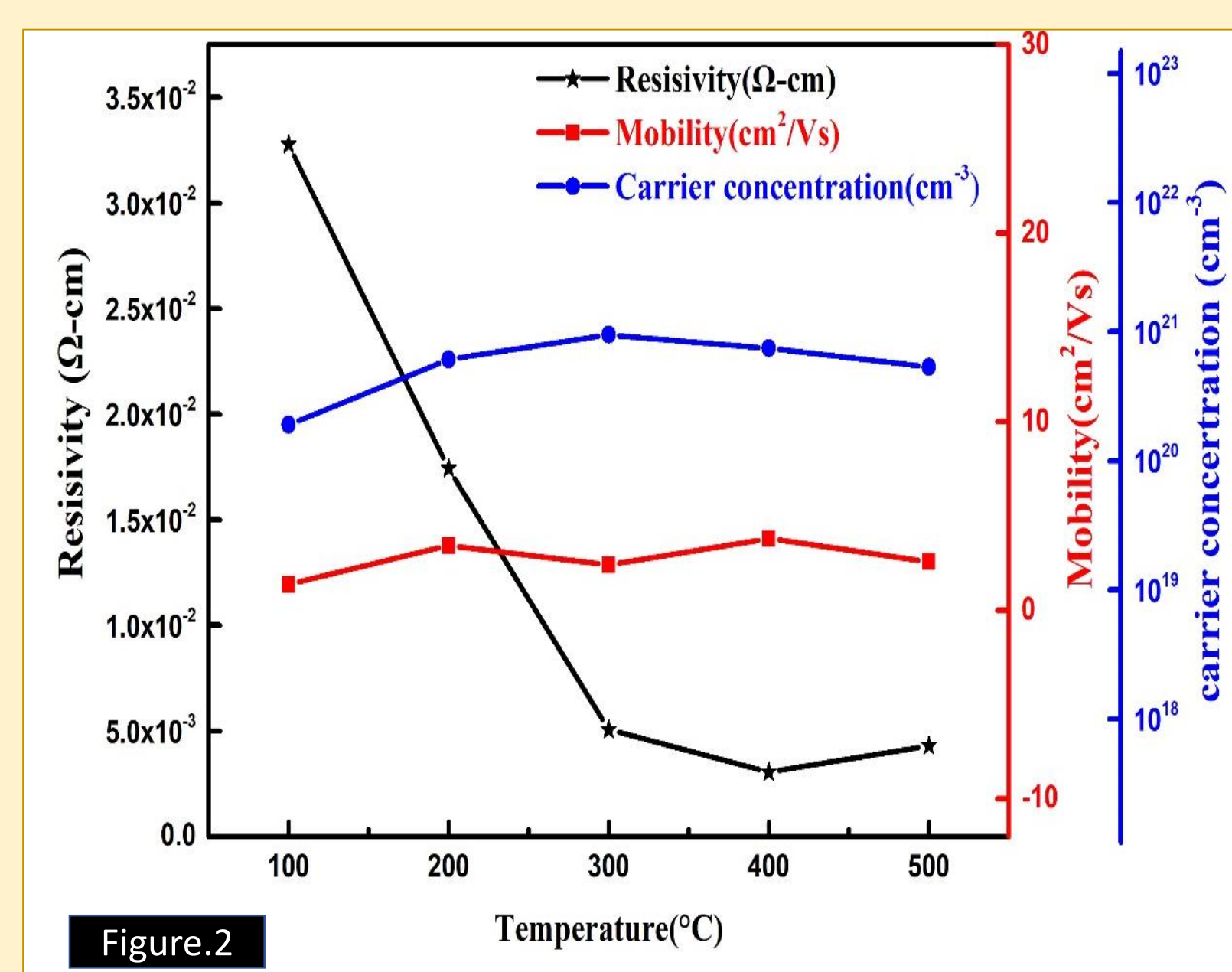
XRD



硫化 溫度 (°C)	2θ (°)	半高 寬 (2θ)	平面間 距 (Å)	晶粒尺 寸 (nm)	晶格常數 a0(Å) C(Å)	相對強度 (a.u.)
100	31.81	0.506	1.89	23.21	A=B=3.79 C=16.31	100%
200	31.79	0.562	1.89	20.05	A=B=3.78 C=16.36	58%
300	31.89	0.521	2.8	43.73	A=B=3.78 C=16.29	74%
400	31.76	0.217	2.73	32.75	A=B=3.80 C=16.27	90%
500	31.80	0.319	2.72	36.66	A=B=3.80 C=16.32	73%

The XRD results show that the main direction is CUS ,corresponding to star cards 060464 found the preferred direction of 006

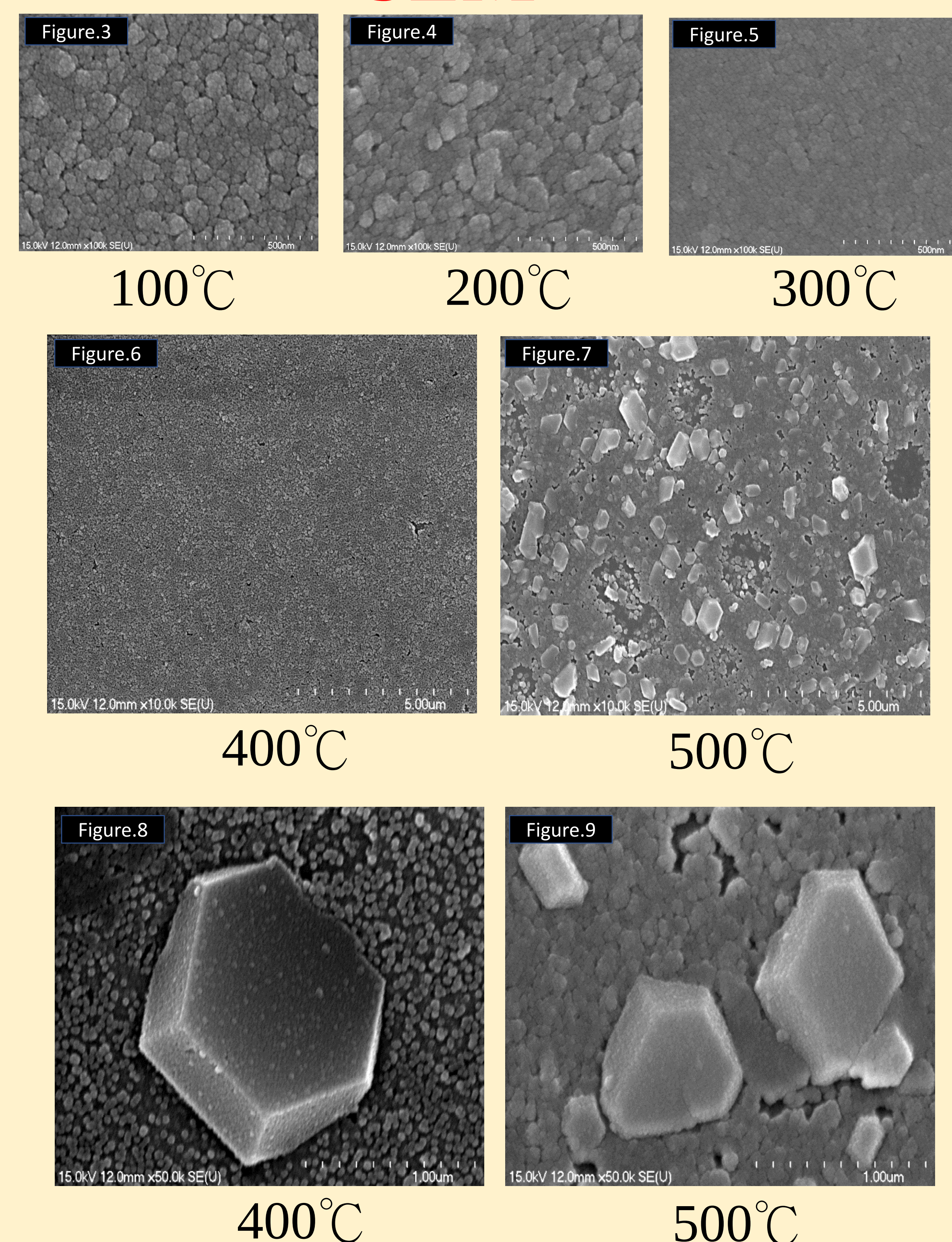
Hall Measurement



退火溫度 (°C)	Resistivity (Ωcm)	Mobility (cm²/Vs)	Carrier concentration (cm⁻³)
100 °C	3.28E-2	1.38	1.89E+20
200 °C	1.74E-2	3.44	4.33E+20
300 °C	5.06E-3	2.42	9.46E+20
400 °C	3.06E-3	3.81	7.42E+20
500 °C	4.3E-3	2.58	5.34E+20

The range of mobility falls between 1.38-3.81 (cm² / Vs), and the range of carrier concentration falls between 1.89 to 9.46 (cm⁻³), there is not much difference. 400°C is the best condition. Resistivity reaches 3.06E-3 Ωcm

SEM



Through SEM to observed can find temperature at 400°C -500°C appear Hexagonal crystal. 100°C -200°C not appear crystal property only has CuS reunion phenomenon. At 300°C film of surface incline smooth .According to S-Cu phase diagrams, 507°C will appear rhombohedral crystal but our experiment appear rhombohedral crystal at 500°C. Infer the reason is thermal energy accumulation effect let 500°C happen rhombohedral crystal. 400°C of grains size and dispersion are average. 500°C of grain size obvious more big but not continuous .

Optical property

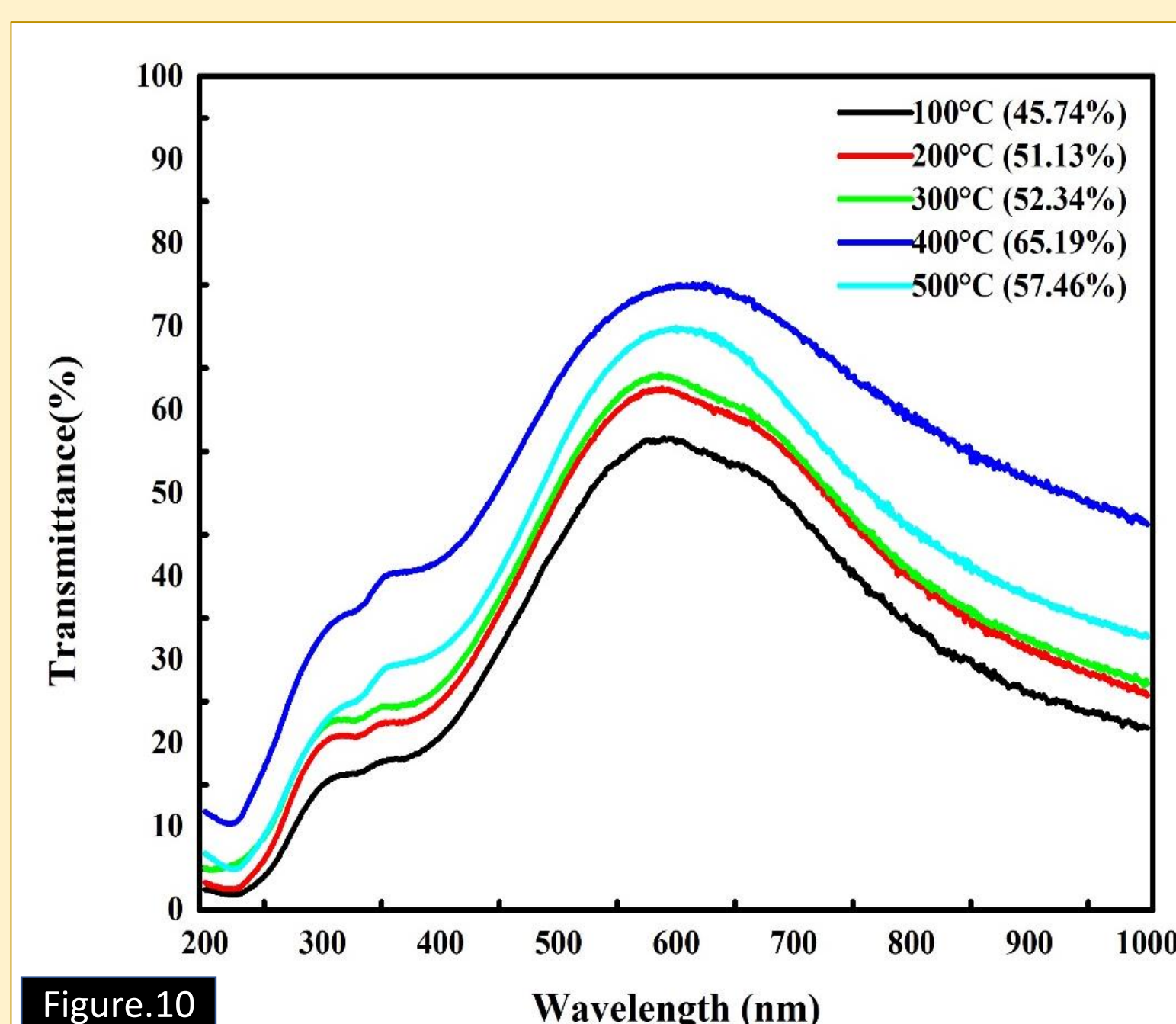


Figure.10 according different sulfied temperature of UV-visible spectar chart ,can discover when CuS film at high temperature the Optical transparency will be upturned. There will turning at 500°C. We are inference to cause this behavior about the crystal structure. At sulfied 400°C the grain size consistency and the grain homogeneity are better than sulfied 500 °C. There cause 500°C of CuS film is discontinuous, let sulfied 500 °C of CuS film transparency to decrease.

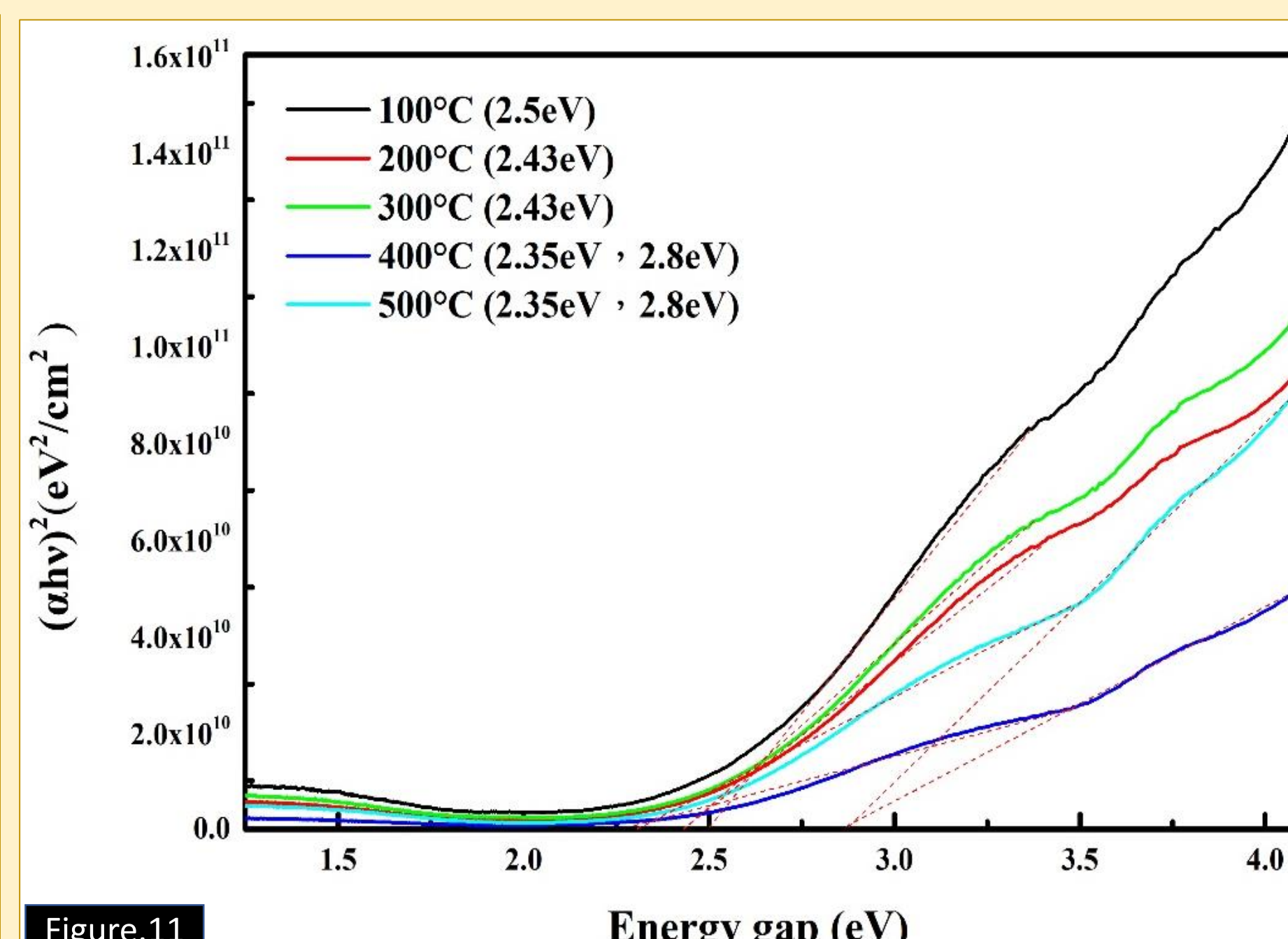


Figure.11 is energy gap of 100 °C 、200 °C 、300 °C 、400 °C 、500 °C, copper sulfide in the literature is about 1.2eV-3eV, the widest energy gap conditions in this experiment are 400°C, 500 2.8 eV.

Conclusions

Experiment result shows that the grain distributed average and grain size raise. reduce the resistivity rate of film., The structure is Covellite between 100°C to 400°C, the Diegenite structure at 500°C and it is Cu_{1.8}S. Grain has obvious growth at 400°C and 500°C, grain growth bring better electrical properties, electrical properties increased with copper content are direct ratio. On the whole, the temperature of 400°C is the best condition, the highest optical transmittance (65%) and best electrical properties, the resistance is $4.7 \times 10^{-3} (\Omega\text{-cm})$, the carrier mobility is $3.81 (\text{cm}^2/\text{Vs})$, the carrier concentration is $7.42 \times 10^{20} (\text{cm}^{-3})$.

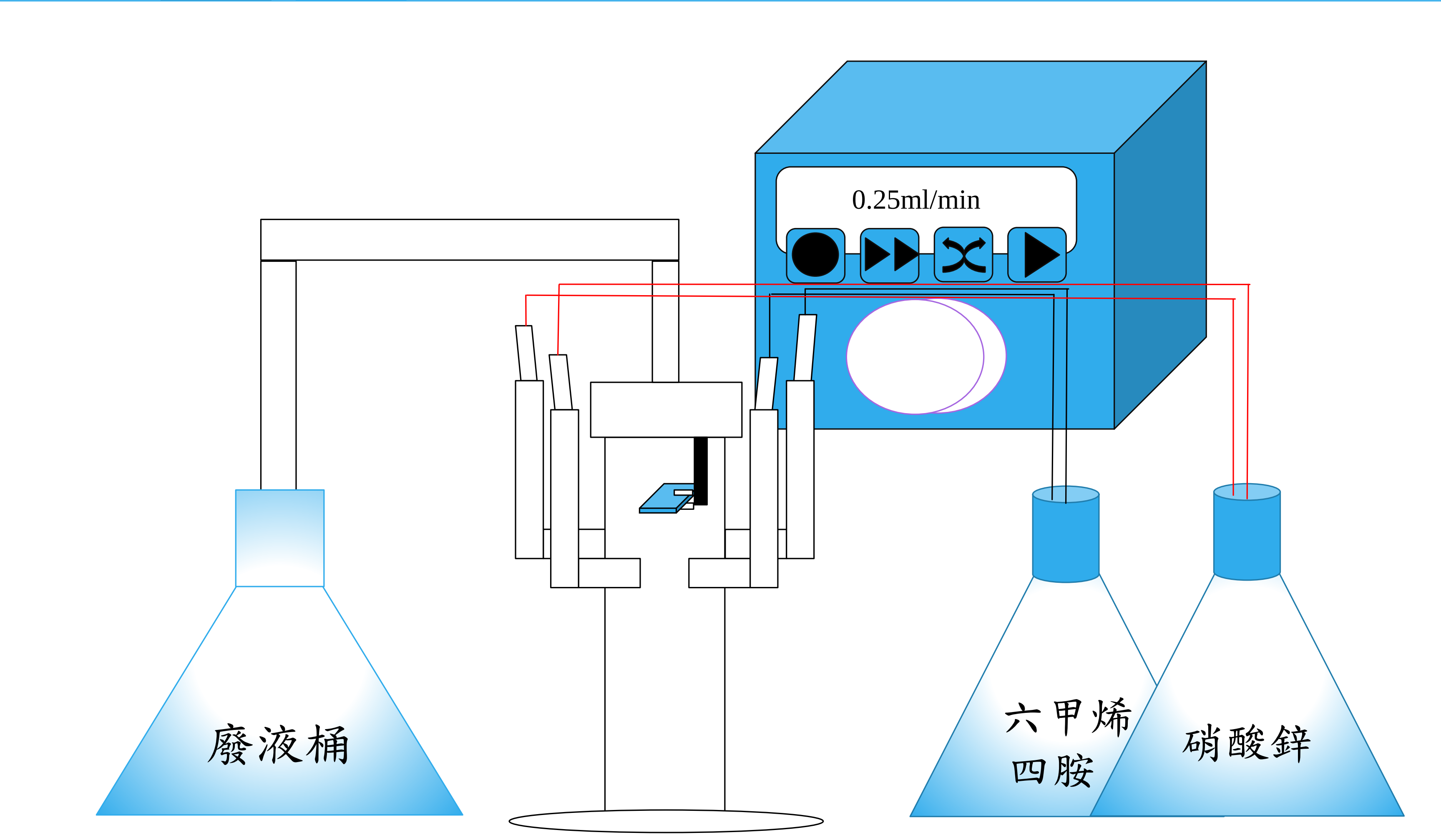
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摘要

在本次實驗中先利用RF射頻濺鍍鍍上氧化鋅薄膜，再透過連續流動式反應槽體進行氧化鋅磊晶的成長，藉由這種其低溫、常壓、且低價，是一種低溫化學溶液法，且槽體因為是開放式，可以使得液體流動，進而去進行實驗，而不使用在槽體內是受到限制且封閉的壓力鍋進行成長，接著再用SEM觀察磊晶及霍爾量測

連續流動式反應槽體



實驗方法及流程

利用RF射頻濺鍍在藍寶石基板上鍍氧化鋅薄膜

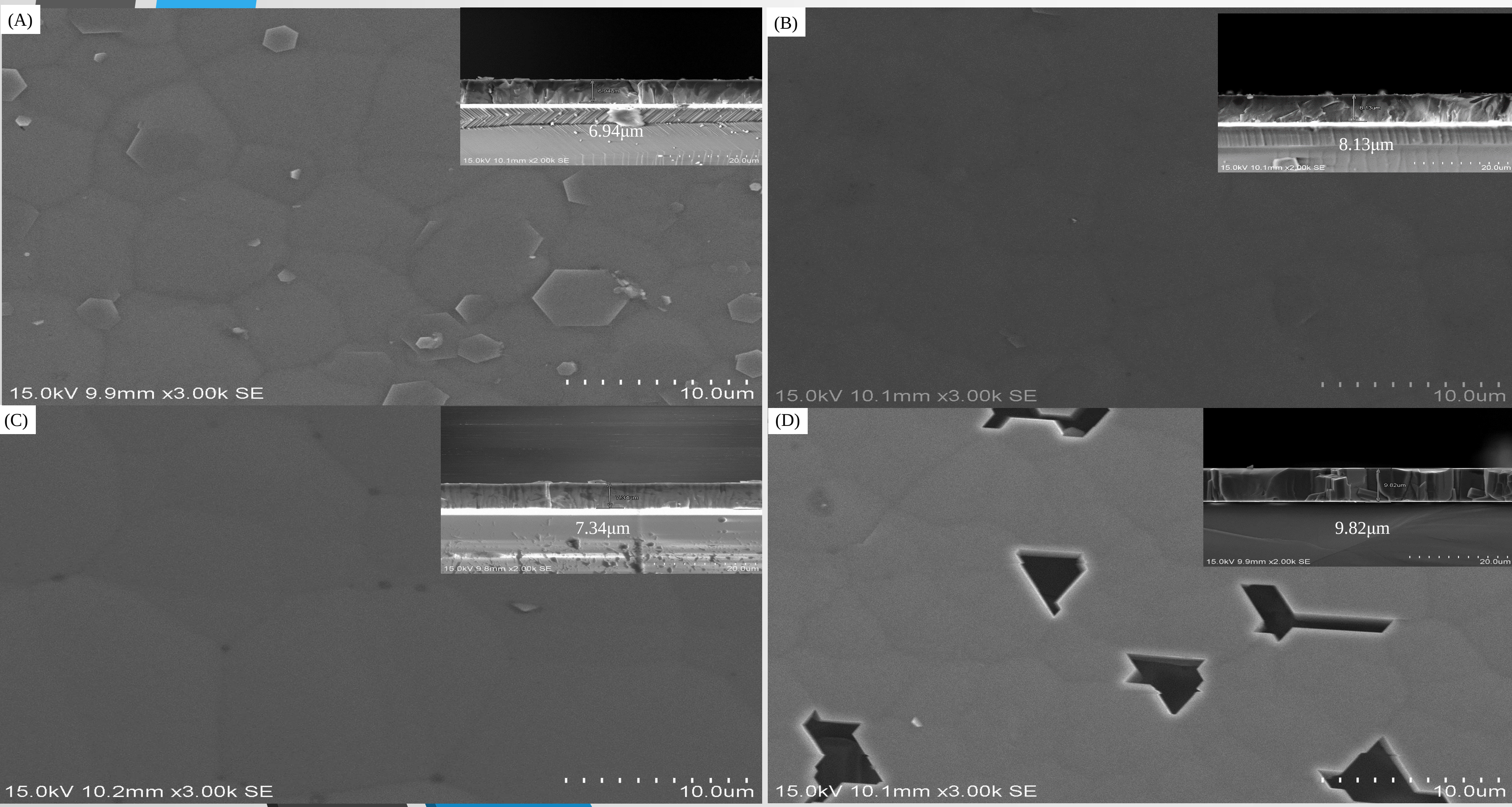
↓

將試片放入連續流動式反應槽體中進行氧化鋅磊晶成長

↓

SEM顯微觀察及霍爾量測

SEM分析



參數	硝酸鋅	六甲烯四胺	厚度
A	0.1M	0.1M	6.94μm
B	0.12M	0.12M	8.13μm
C	0.12M	0.1M	7.34μm
D	0.1M	0.12M	8.92μm

結論

- 從SEM分析可以看到濃度相當時，試片生長平整且癒合狀況較佳，且濃度提升後，厚度也會隨之成長。
- 當硝酸鋅濃度增加時，試片生長平整且癒合狀況較佳，雖然有些許雜晶，但厚度成長相較同濃度(0.12M)的比例下成長幅度較少。
- 當六甲烯四胺濃度增加時，試片生長平整但癒合狀況較差，可以發現提升六甲烯四胺濃度時，試片的厚度與同濃度(0.12M)的比例下成長幅度大。
- 透過霍爾量測後，可得知其試片大致都為N-type半導體。且載子濃度與遷移率對於濃度改變影響不大。

霍爾電性量測

比例濃度 硝酸鋅:六甲烯四胺	載子濃度(cm^{-3})	電阻率($\Omega\text{-cm}$)	載子遷移率(cm^2/Vs)	半導體類型
0.1M:0.1M	3.61E+18	0.0102	10.3	N-type
0.12M:0.12M	6.4E+18	0.048	23.0	N-type/ P-type
0.12M:0.1M	7.54E+17	0.0139	23.8	N-type/ P-type
0.1M:0.12M	2.14E+18	0.0235	22.3	N-type