



電化學

Journal of Electrochemistry

《电化学》期刊公益系列讲座

现代电化学研究方法 之 原位红外光谱

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复旦大学化学系

2020-06-13



主要内容

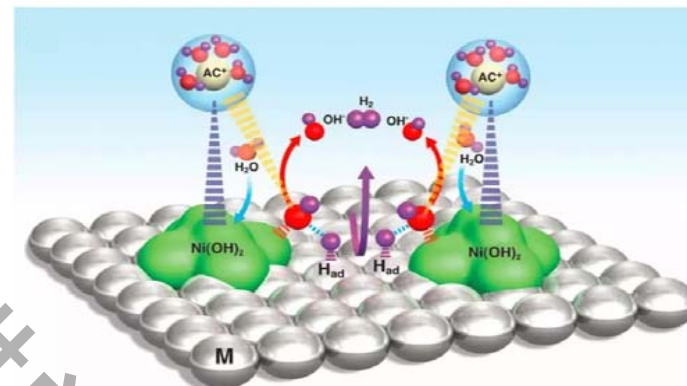
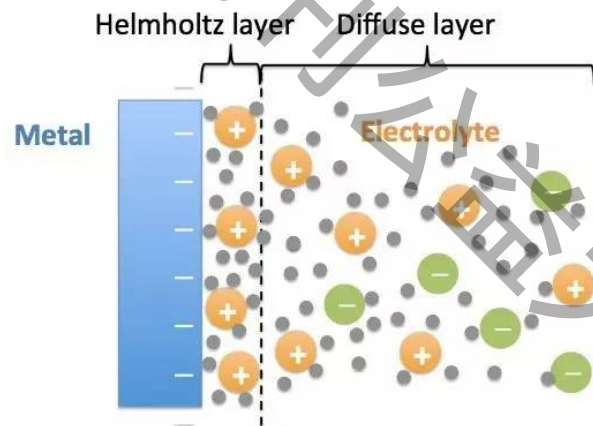
- 电化学研究方法---传统和现代
- 电化学反射红外光谱法基础
- 电化学反射红外光谱应用
- 前沿挑战与展望



电化学

- 电化学是研究两类导体形成的带电界面现象及其上所发生变化的科学，它在能源、材料、金属腐蚀与防护、生命科学、微电子等领域获得了广泛的应用；
- 从最初物理化学的分支发展成为多学科的交叉：纳米电化学、生物电化学、光谱电化学、材料电化学、量子电化学等

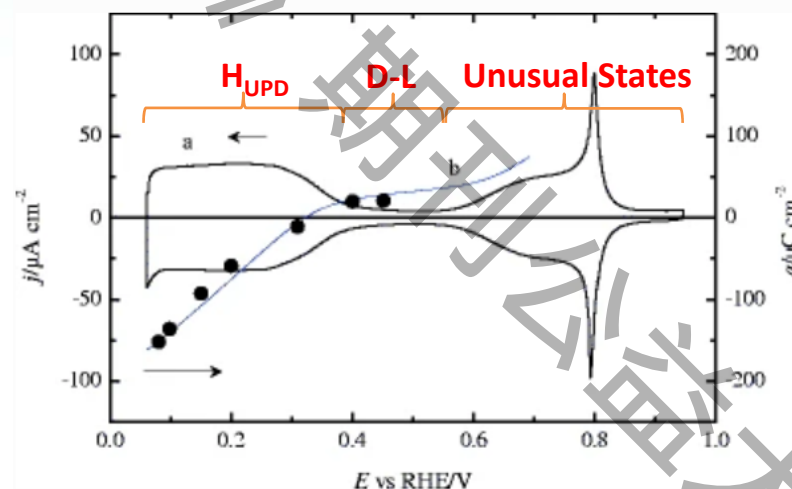
电极-溶液界面是电化学核心



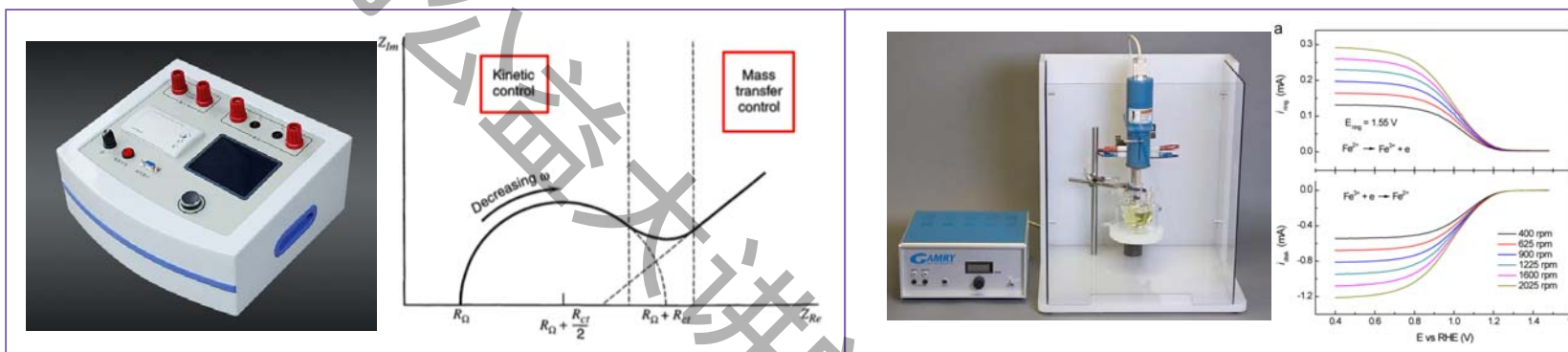
经典电化学研究方法

基于电学参数的宏观测量和分析方法，难以获得分子水平和微观结构的信息

- 循环伏安
- 计时电流
- 交流阻抗
- 旋转(环)盘
- 超微电极.....

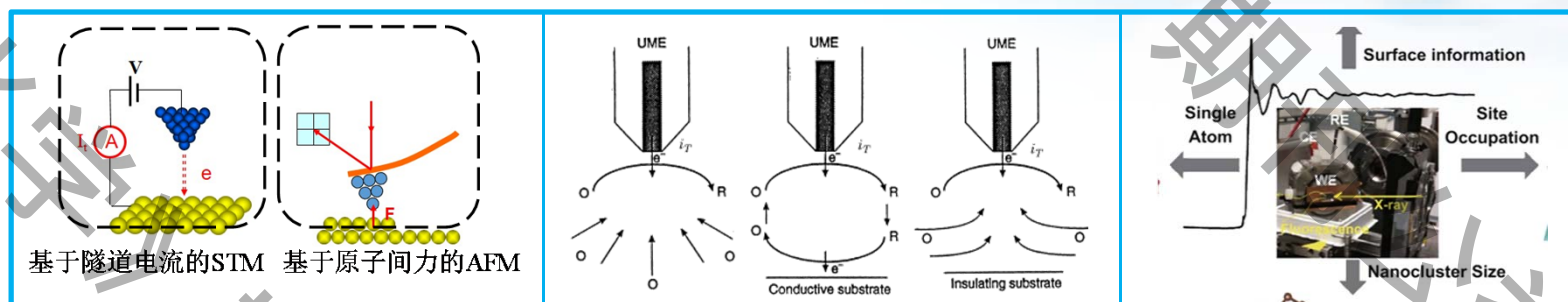


Pt(111) in 0.1 M HClO₄, 50 mV/s

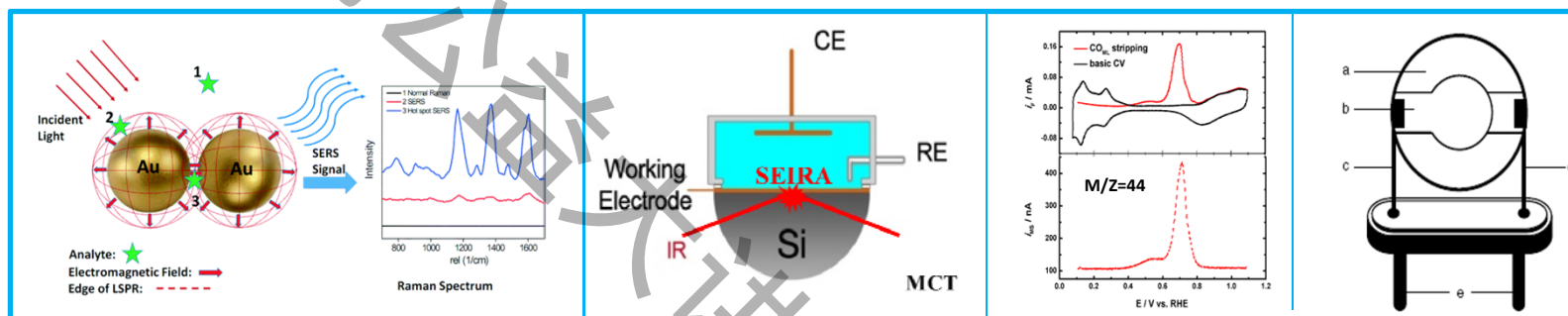


现代电化学研究方法

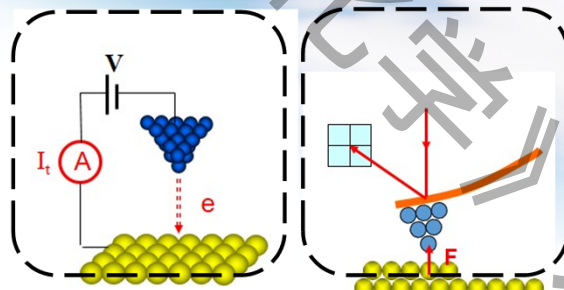
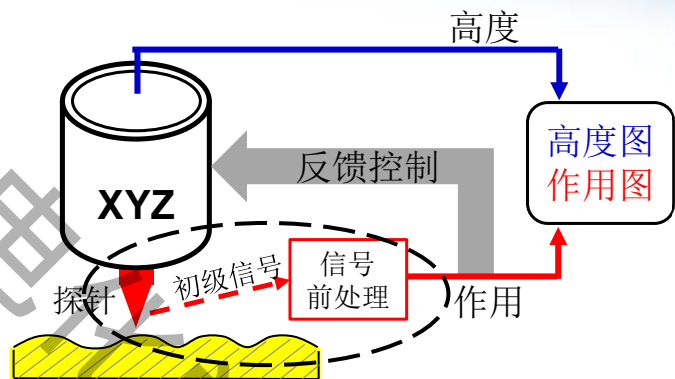
- 从宏观唯象到微观（纳米、分子、原子）层级演进
- 电化学SPM, SECM, XAFS 等



- 电化学 **Raman**, **IR**, **SFG**, DEMS, EQCM等



电化学扫描探针显微术

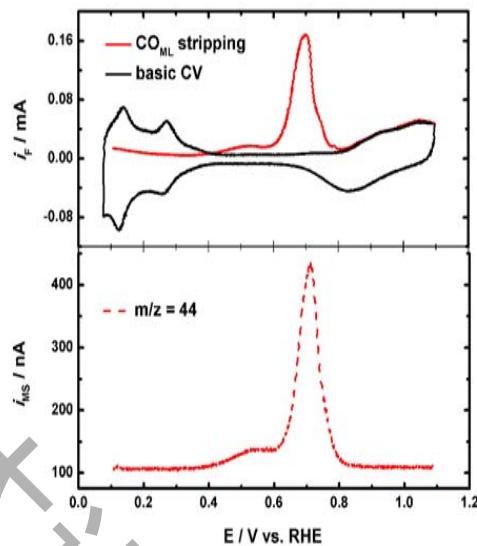
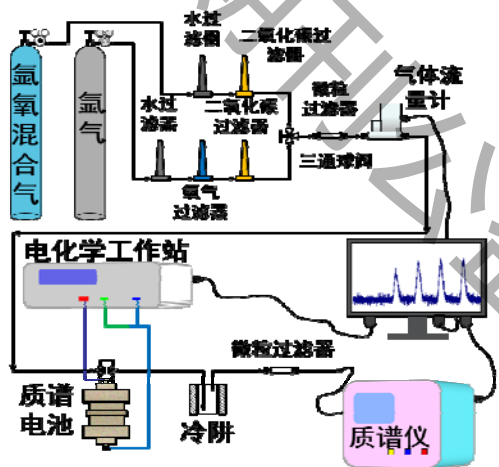


基于隧道电流的STM 基于原子间力的AFM



毛秉伟 教授

电化学微分质谱



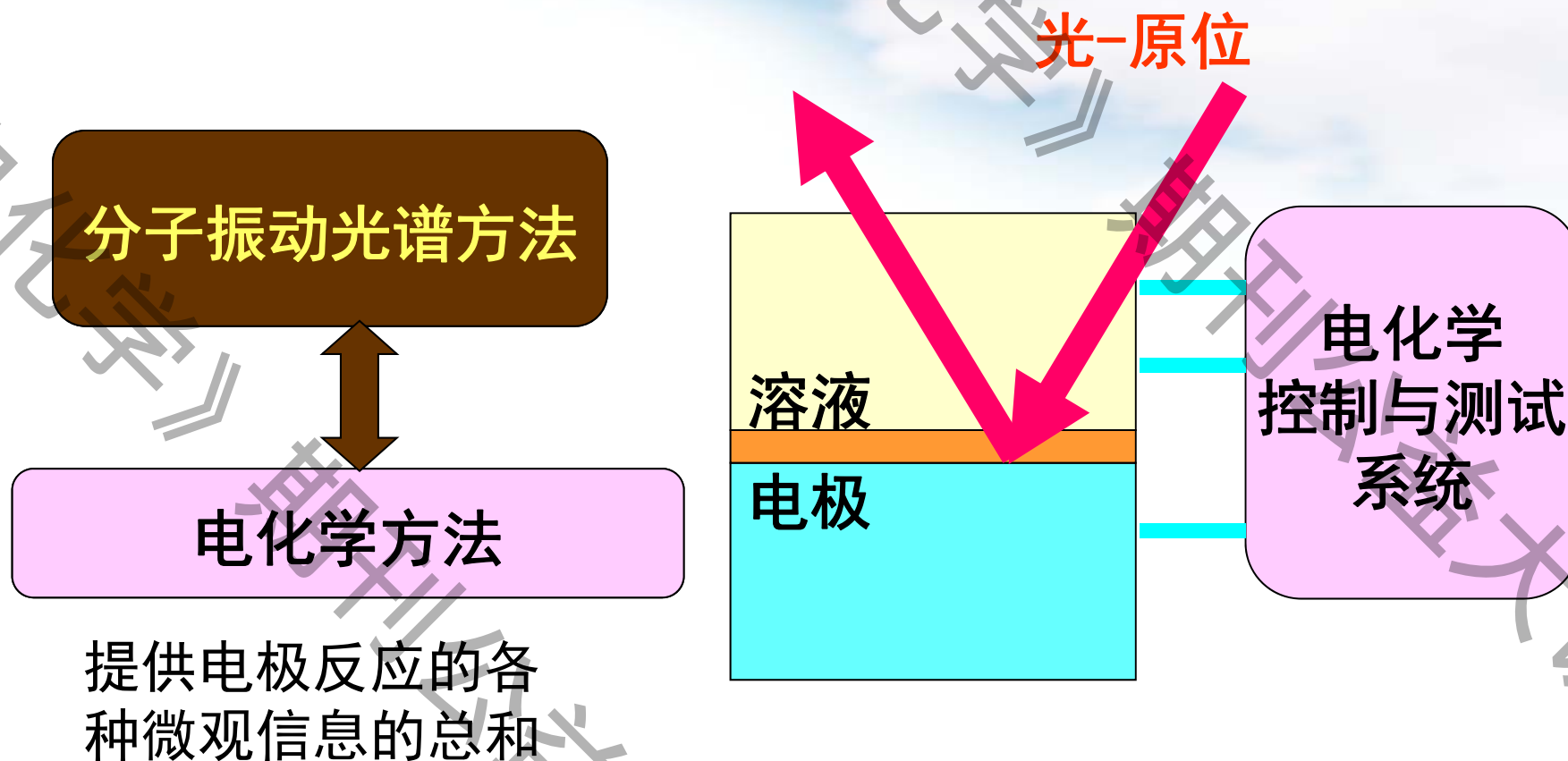
CO电氧化: 10 mV/s扫速, 0.5 M H₂SO₄电解质



彭章泉 教授



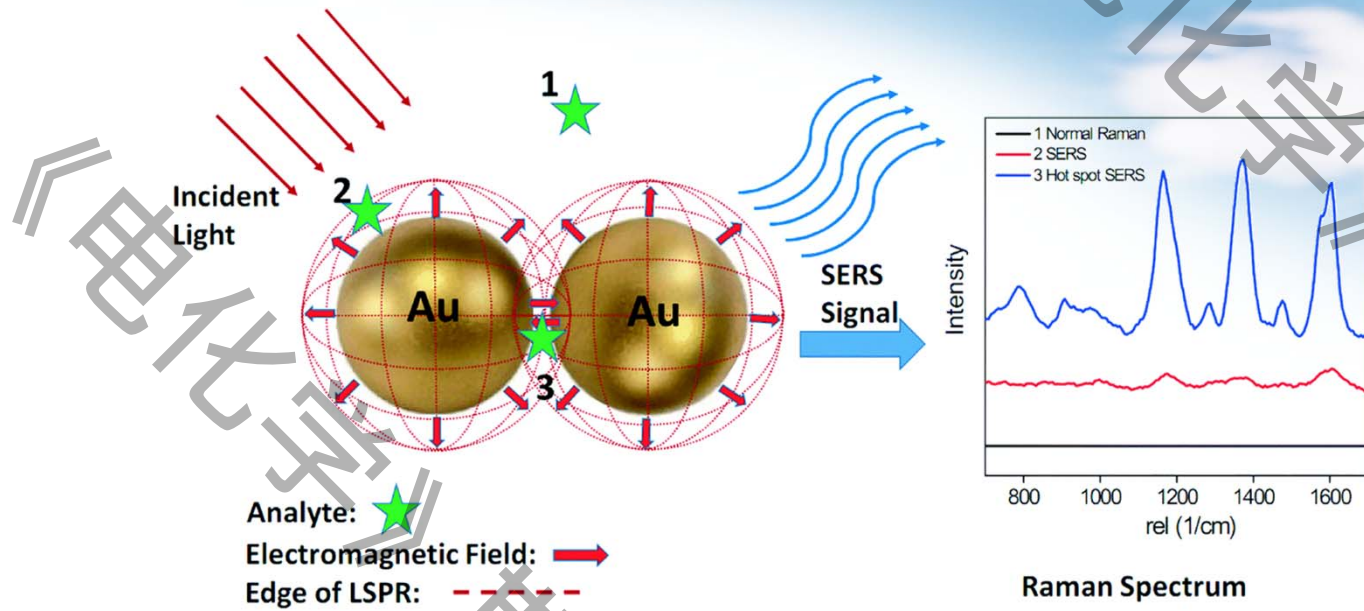
电化学表面振动光谱



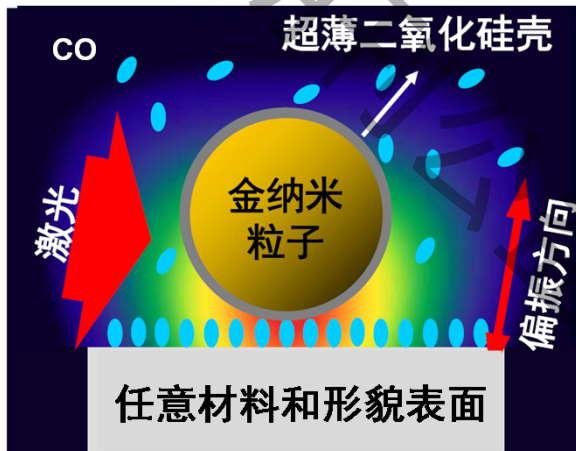
电化学表面IR、Raman、SFG光谱



电化学表面增强拉曼光谱



$$EF_{SERS} = \frac{I_{SERS} / N_{surf}}{I_{ref} / N_{bulk}}$$

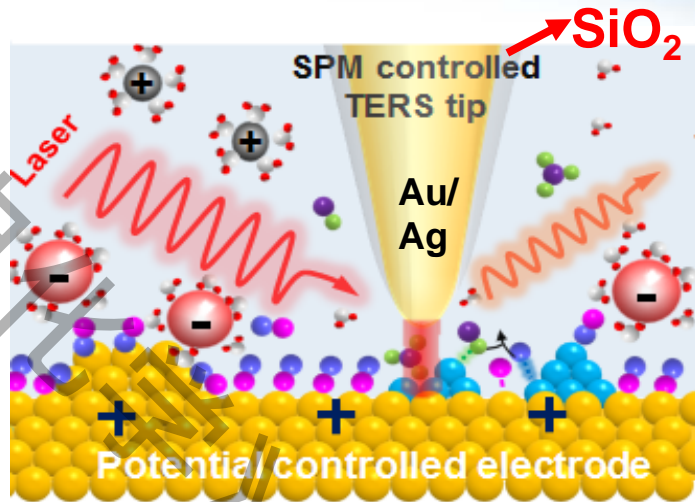


壳层隔绝纳米粒子增强拉曼光谱 SHINERS

- 内核金粒子：“拉曼信号放大器”
- 二氧化硅壳：惰性、极薄且无针孔—化学隔绝

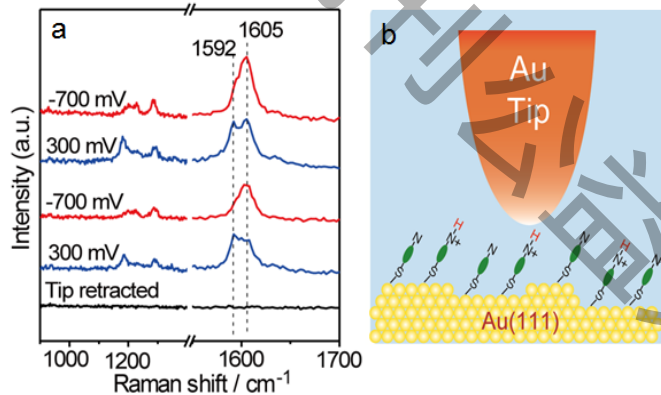


针尖增强拉曼光谱

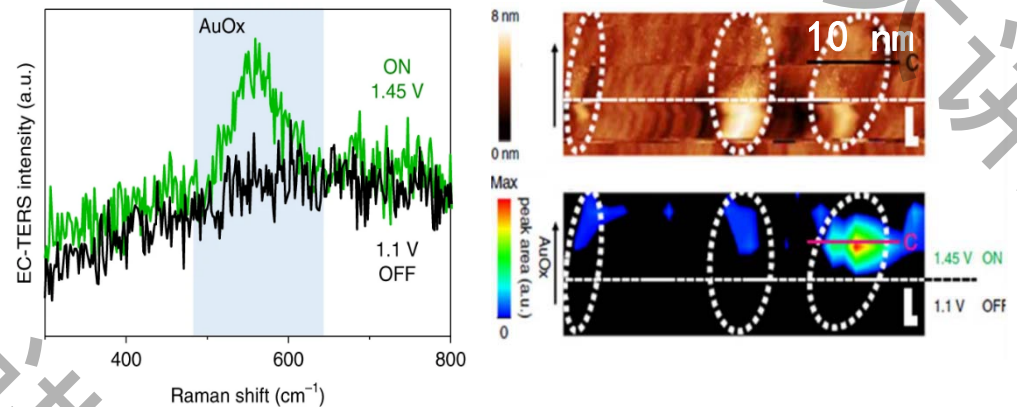


- **EC-TERS:** 电化学、SPM和针尖等离激元增强拉曼技术的联用
- **优势:** 同时获得表面形貌和光谱成像，以高敏度、纳米分辨、分子水平、原位表征电化学界面过程

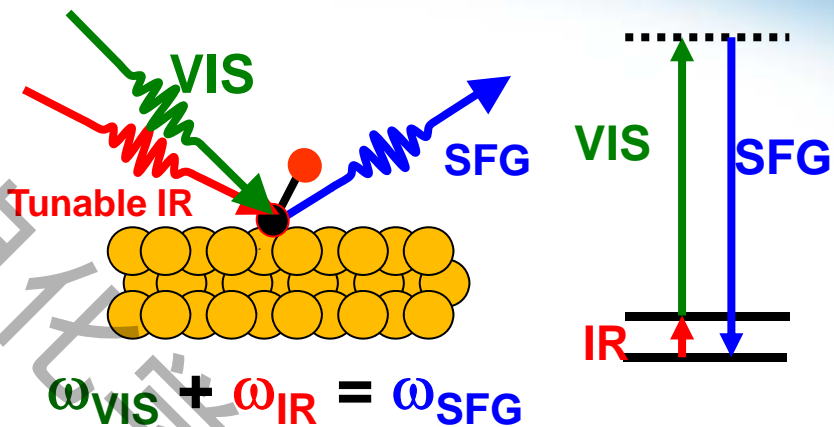
结构明确单晶表面吸附分子
灵敏反映界面变化



10 nm 光谱成像
揭示电化学构效关系



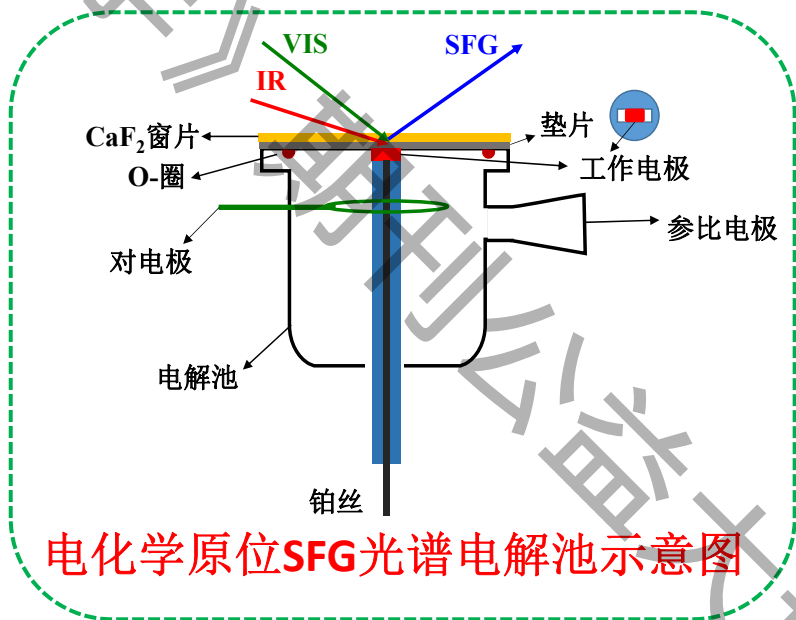
电化学和频光谱 (SFG)



$$I_{SFG} \propto |\vec{P}_{SFG}|^2 = \left| \chi^{(2)}(\omega_{IR}, \omega_{VIS}) \vec{E}_{IR} \vec{E}_{VIS} \right|^2$$

时间分辨
高灵敏
非线性
多维光谱

表面选择性
分子取向
振动去相位
排列方式



电偶极近似的条件下, 均匀体相中
 $\chi^{(2)}(\vec{r}) = -\chi^{(2)}(-\vec{r})$ $\chi^{(2)}(\vec{r}) = \chi^{(2)}(-\vec{r})$
 $\chi^{(2)}(\vec{r}) = 0$
 界面上 $\chi^{(2)}$ 不为0 (界面选择性)



主要内容

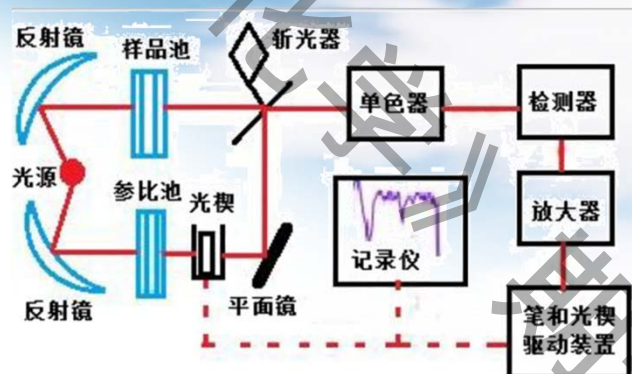
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电化学研究用红外光谱仪

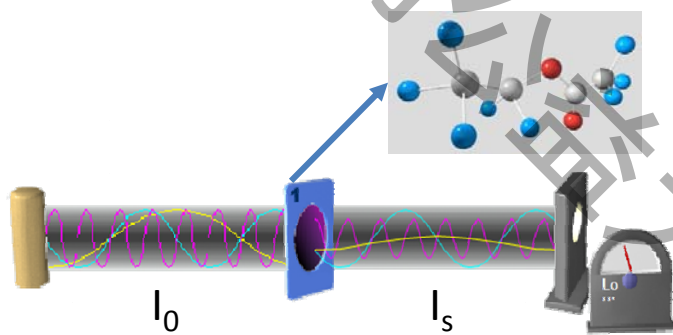
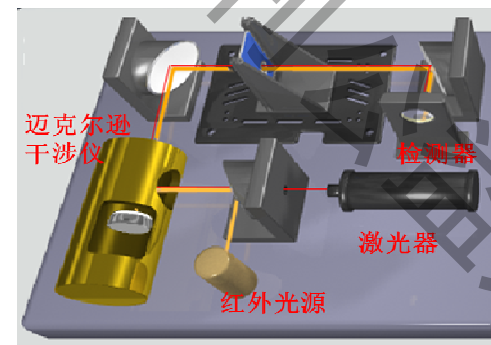
色散型红外光谱仪

早期



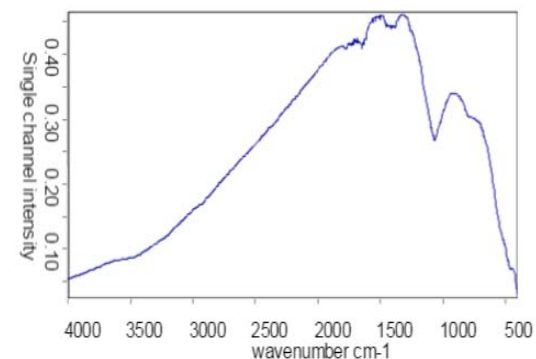
傅里叶变换红外光谱仪

最常用



◆ 数模转换

◆ 傅里叶变换



电化学反射红外光谱

• 主要测量难点

- 溶液（主要是水）对红外光的强吸收
- 电极表面吸附物种数量少 ($10^{14} \sim 10^{15} \text{ cm}^{-2}$)，信号弱

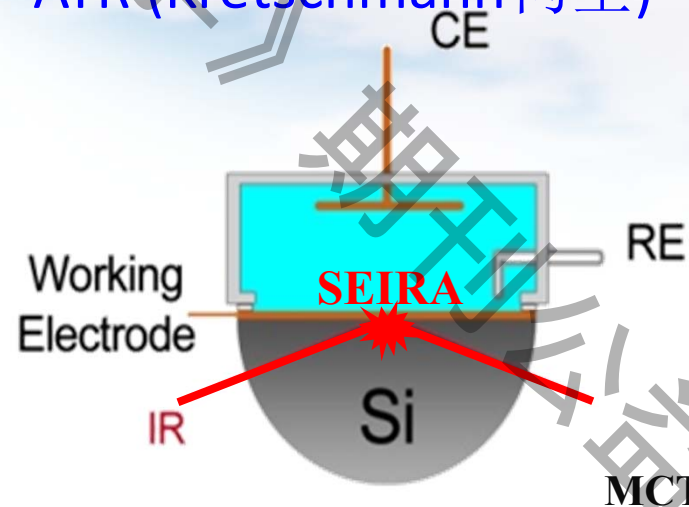
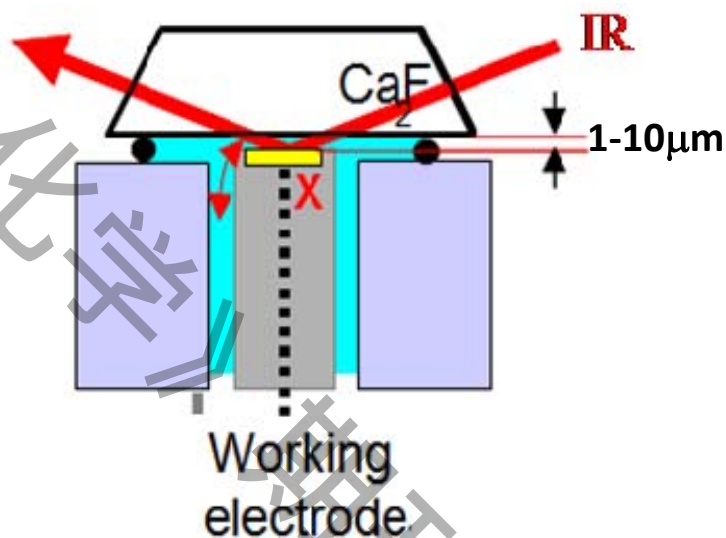
• 解决方法

- 设计原位红外薄层池 ($< 10 \mu\text{m}$) 减少液层吸收或利用ATR模式，结合电位差谱
- 采用针对弱信号的检测方法，如电化学或红外调制、锁相检测等，利用SEIRA效应



电化学反射红外光谱基础

ATR (Kretschmann构型)



外反射模式红外光谱IRAS

- 电极种类不受限；溶解产物
- 欧姆降；传质限制问题

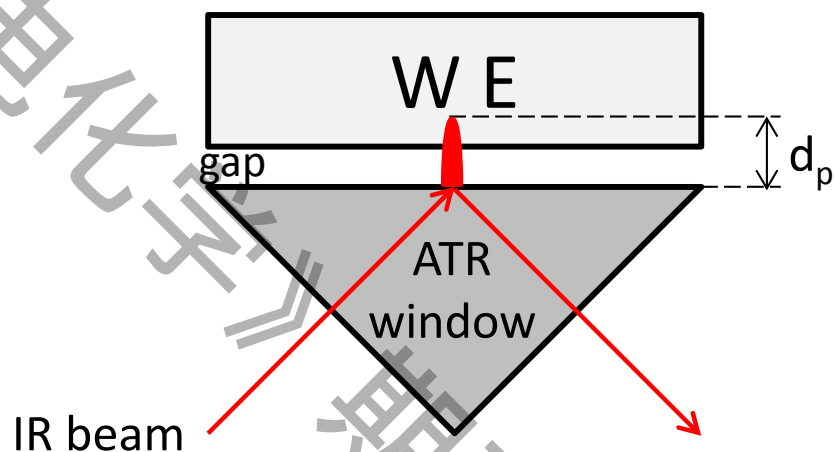
内反射ATR-IR (SEIRAS, FTIR)

- 原位动态跟踪中间表面物种
- 适宜的窗口；表面膜制备



电化学反射红外光谱基础

ATR-IR (OTTO 构型)



- 利用消逝波透过溶液到达工作电极表面，可检测溶液相和吸附态物种。
- 检测到吸附态物种的前提是：

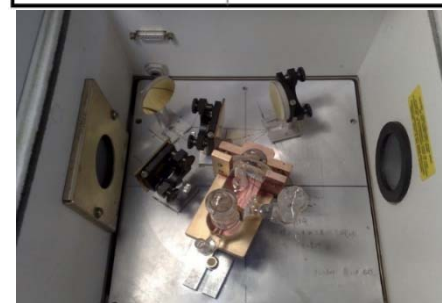
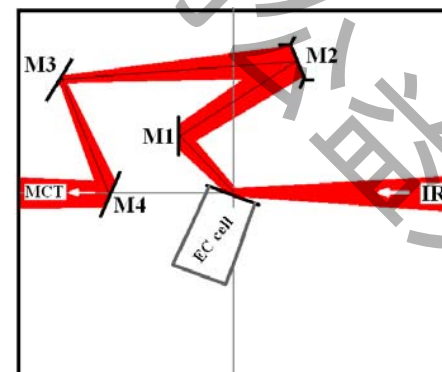
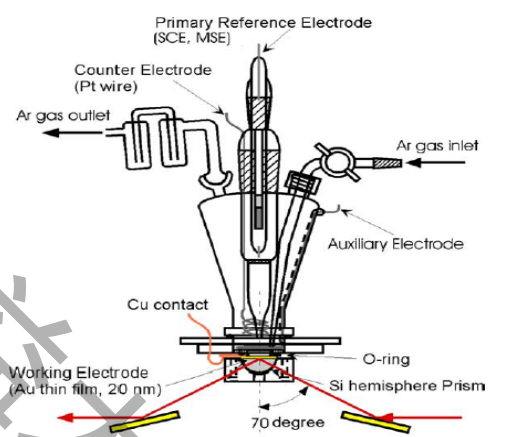
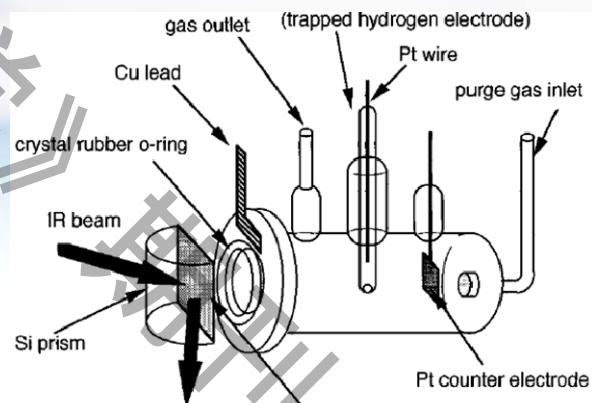
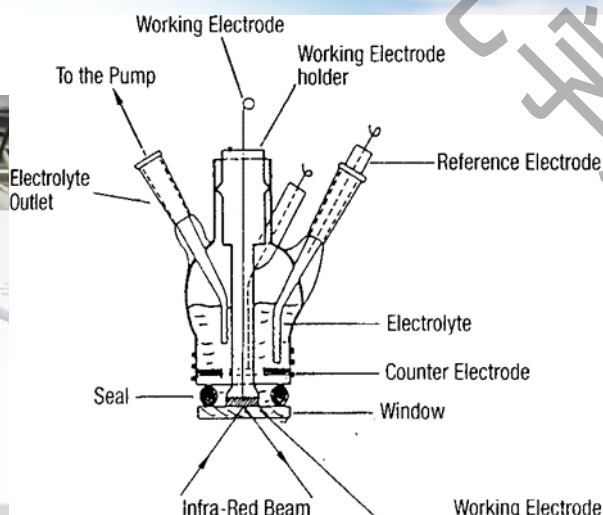
$$gap < d_p = \frac{\lambda}{2\pi n_1} \left[\sin^2 \theta - \left(\frac{n_2}{n_1} \right)^2 \right]^{-\frac{1}{2}}$$

n_1 : 红外窗口的折射率, n_2 : 电解液的折射率

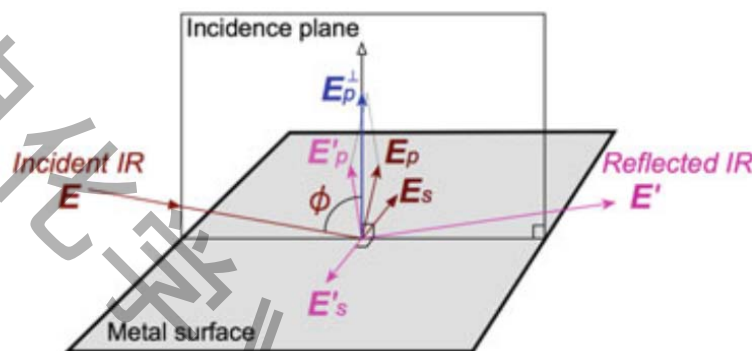
- 优点：适用的电极材料范围广。与Kretschmann-ATR模式相比，可用于单晶电极；与外反射模式相比，可用于粉末电极等非镜面反射电极。
- 缺点：同外反射模式，薄层结构溶液电阻大，电位响应慢，传质受阻，电流分布不均等。



电化学反射红外光谱光路设计

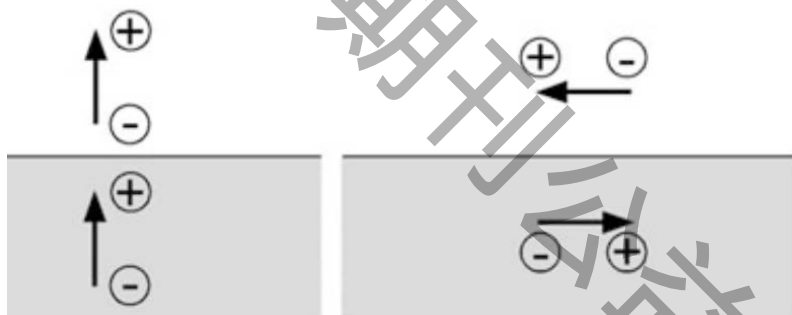


电化学反射红外光谱基础



p-偏振：入射光和反射光在**金属**表面相叠加而增大；**s-偏振**：入射光和反射光相位相差 π 在金属表面抵消殆尽。对于非偏振光而言，只有**p**-组分对**金属**表面红外信号的检测有贡献。

$$I \propto C_{\text{surf}} \left| \vec{E} \cdot \frac{d\vec{u}}{dQ} \right|^2 = C_{\text{surf}} \cos^2 \theta |\vec{E}|^2 \left| \frac{d\vec{u}}{dQ} \right|^2$$

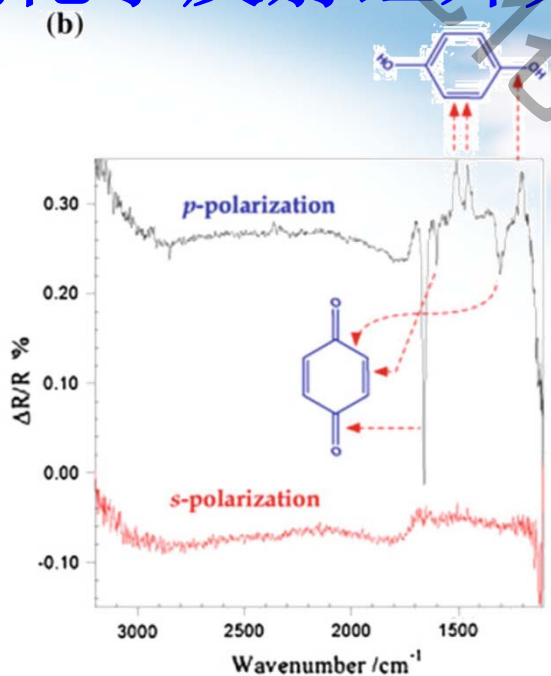
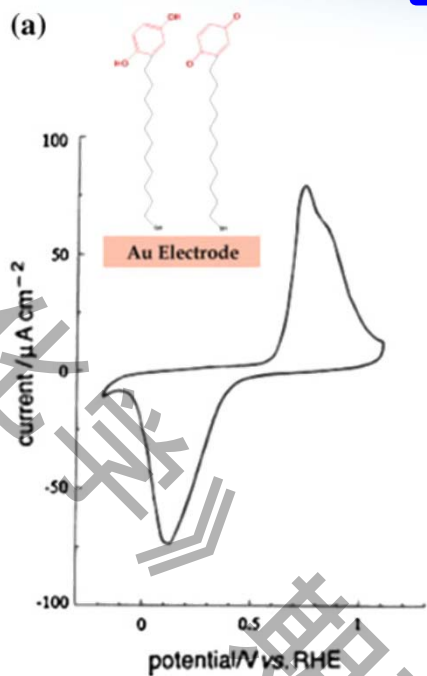


只有垂直于金属表面偶极矩非零的振动模式才具有红外活性
---表面红外选律--

镜像电荷理论



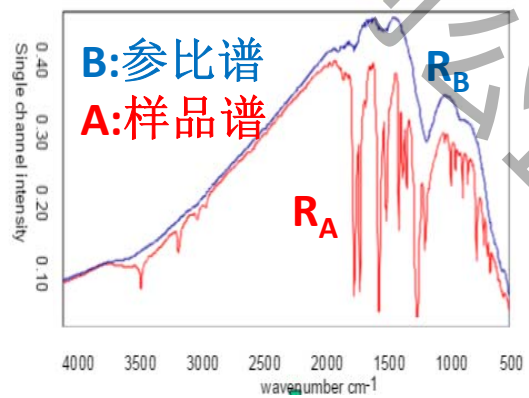
电化学反射红外光谱基础



p-polarization
vs
s-polarization

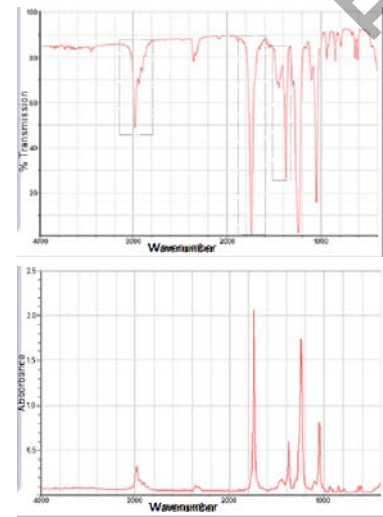
红外光谱数据的数学运算

Au表面自组装单层氢醌的电氧化



$$\Delta R/R = (R_A - R_B) / R_B$$

$$Abs = -\log(R_A / R_B)$$

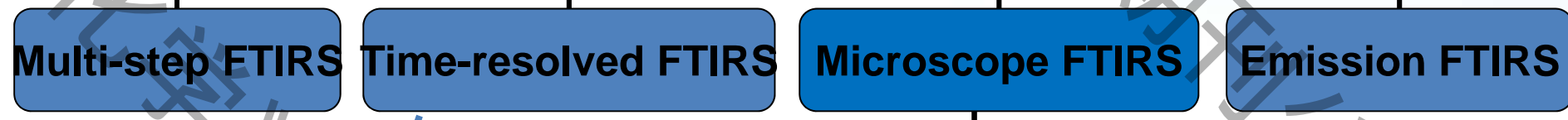


孙世刚院士团队在电化学红外光谱发展中的贡献

外反射模式IRAS
In situ IR techniques

J.M. Xu, S.G. Sun, et al.
Spect. & Spectral Anal.,
2007,27:247-249

S.G. Sun et al.
JEAC, 1988, 240, 147-159;
JEAC, 1990, 289,177-187

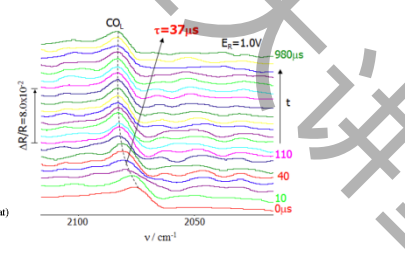
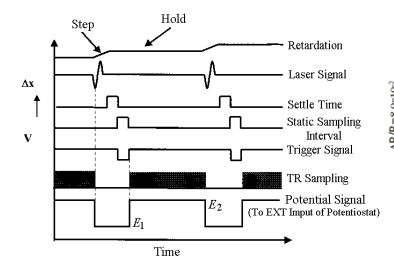
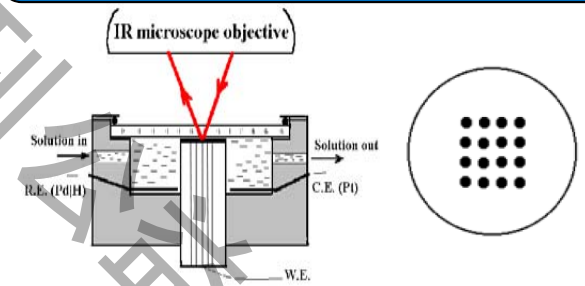


W.F. Lin, S.G. Sun,
Electrochim.Acta,
1996,41:803-809

Combinatorial approach

Fast Time-resolved

S.G. Sun, Y. Lin,
Electrochim.Acta,
1998,44:1153-1162



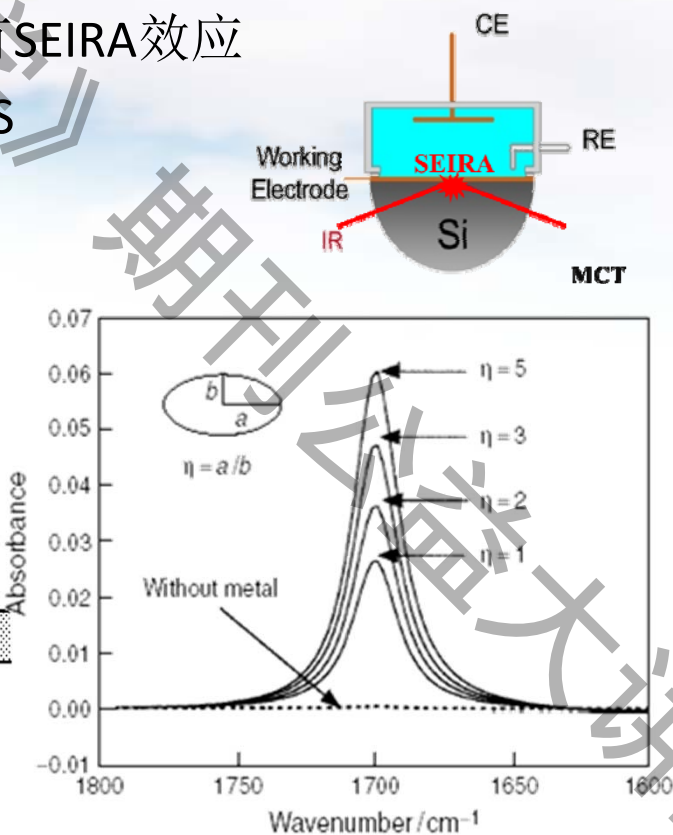
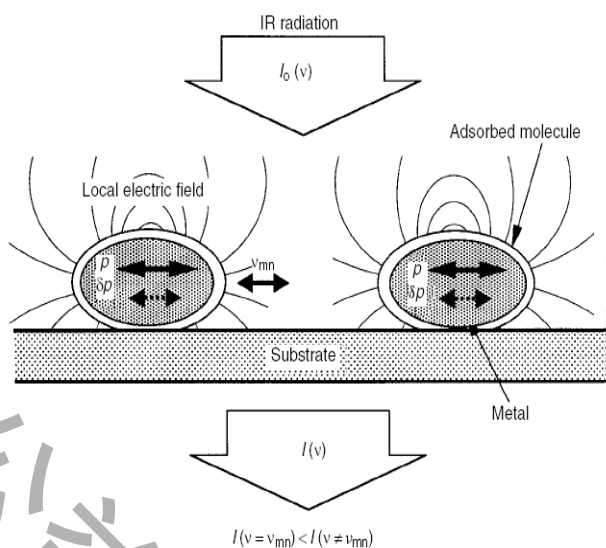
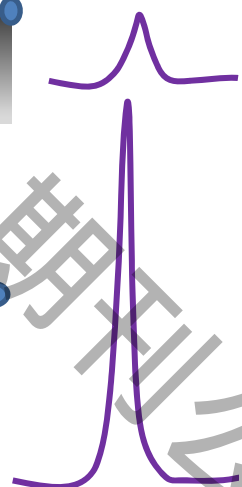
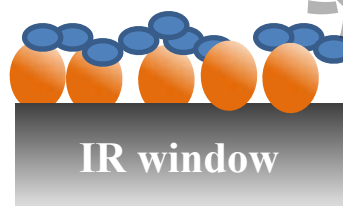
H. Gong, S.G. Sun, et al.,
J.Phys.Chem.B,
2004, 108:11575-11584

Z.Y. Zhou, S.G. Sun,
Electrochem. Commun.,
2005,7:490-495



电化学表面增强红外光谱ATR-SEIRAS

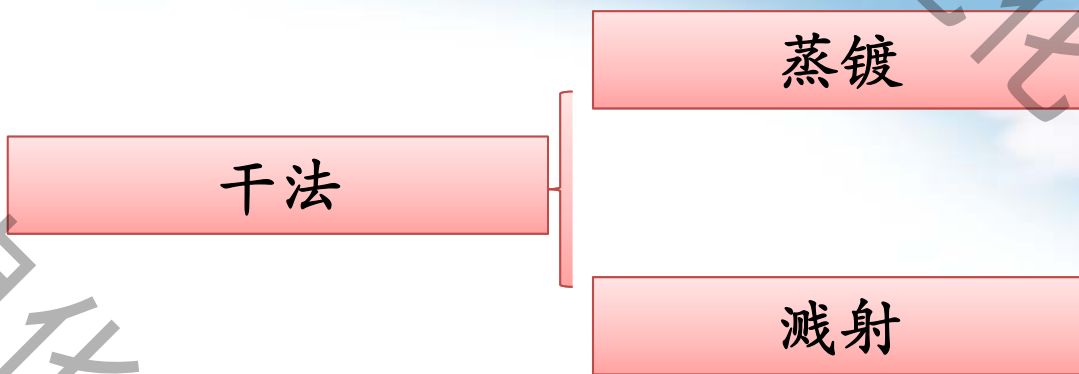
- 1980年Hartstein最早发现Ag岛上吸附 PNBA具有SEIRA效应
- 1992年Osawa等应用于电化学测量, ATR-SEIRAS



10 to 10^3 enhancement depending on size, shape and proximity of nanoparticles



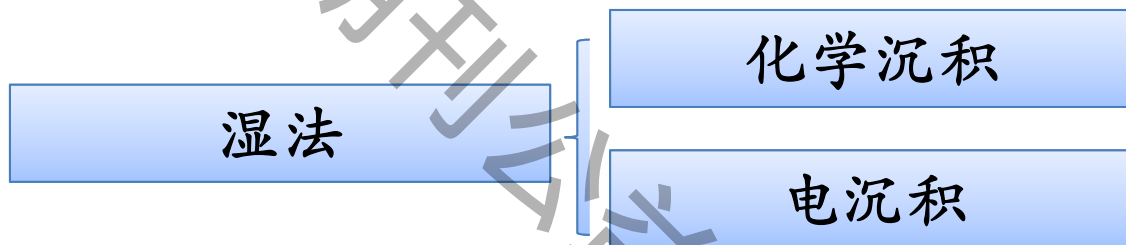
ATR-SEIRAS薄膜电极的构建



◆ 传统方法

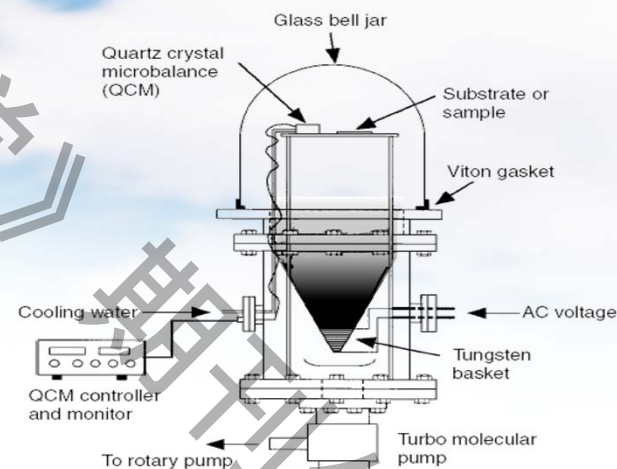
◆ 价格高，耗时久，重现性差

◆ 粘附力差，峰形异常



◆ 克服以上缺点

◆ 仅限部分金属

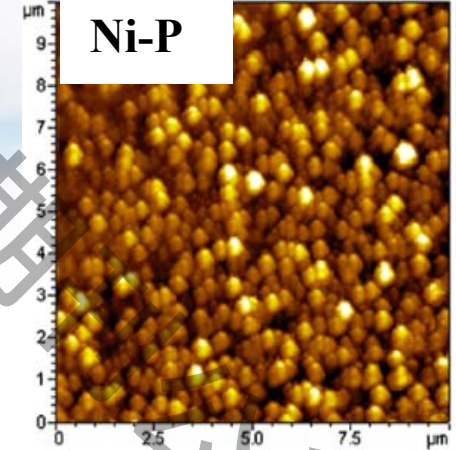
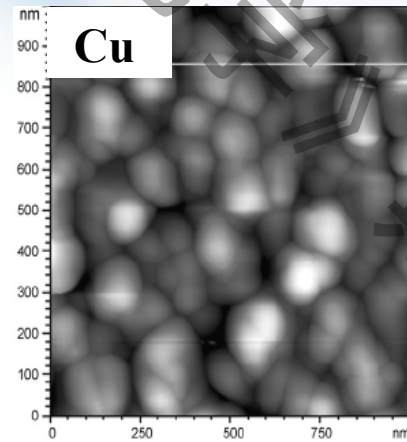
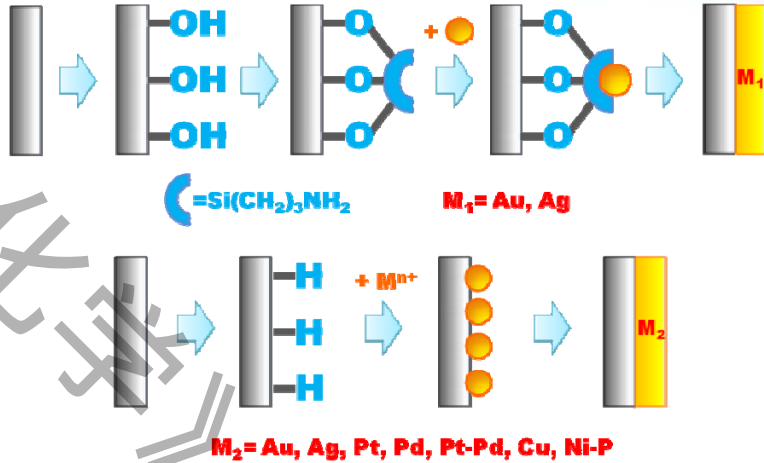


VS.

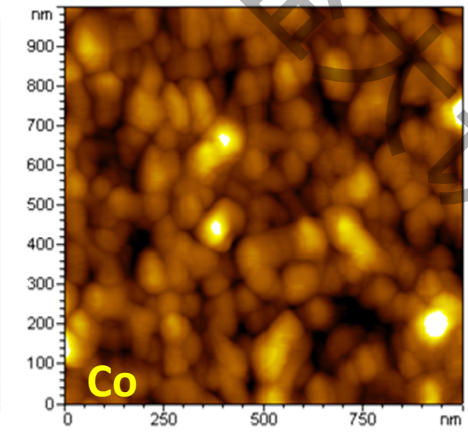
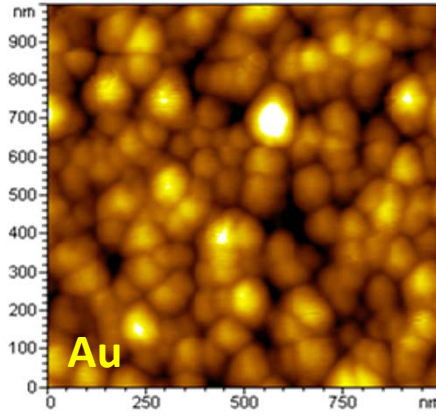
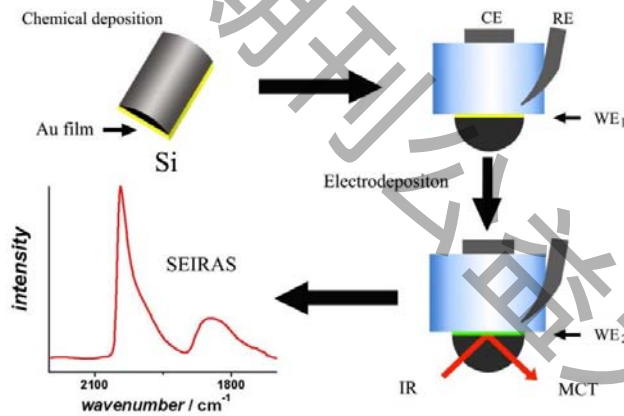


纳米薄膜电极的湿法构建技术

晶种生长法



化学镀+电沉积的通用二步法



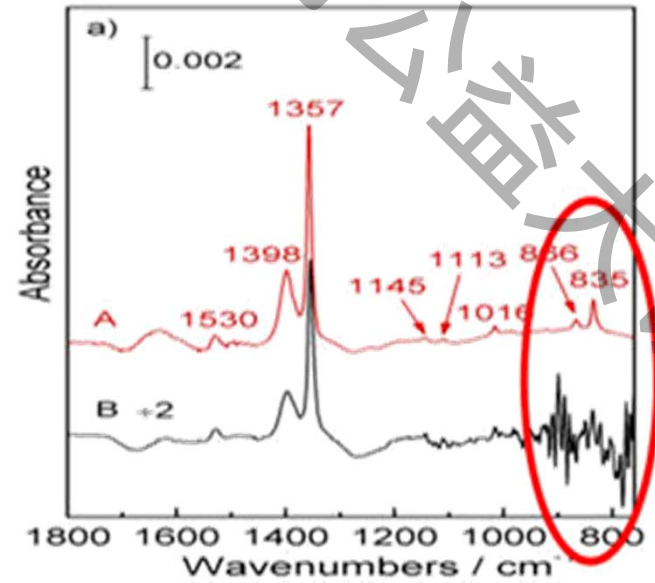
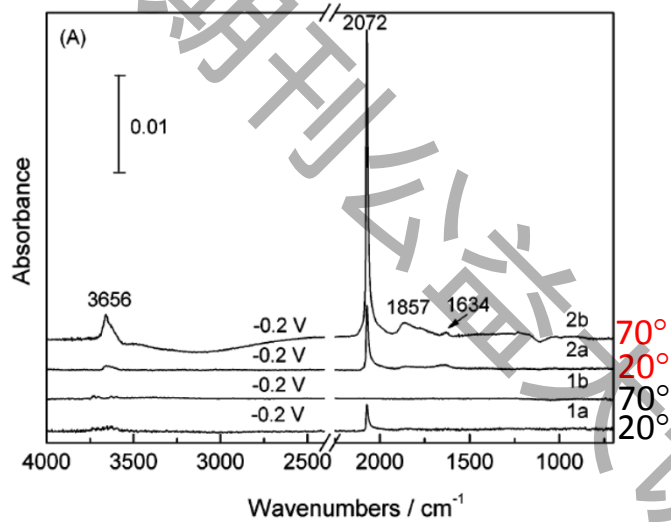
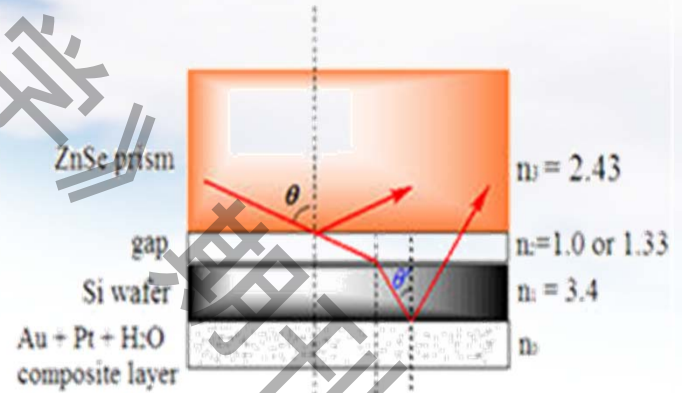
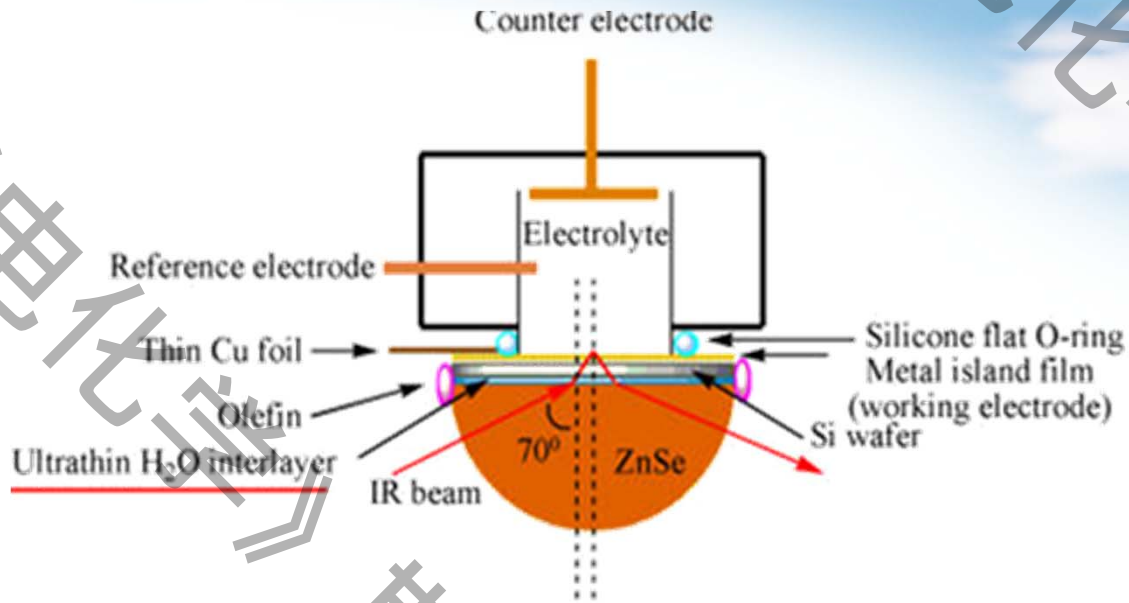
J. Phys. Chem. B 109 (2005) 7900; J. Phys. Chem. B 110 (2006) 4162;
 Appl. Spectrosc. 63 (2009) 1162; Anal. Chem., 82 (2010) 5117
 J. Phys. Chem. B 109 (2005) 15985; Electrochim. Acta 52 (2007) 5950
 Electrochim. Acta 54 (2009) 1834; J. Phys. Chem. B 110 (2006) 25721

表面增强红外光谱拓展到高亮的金属电极

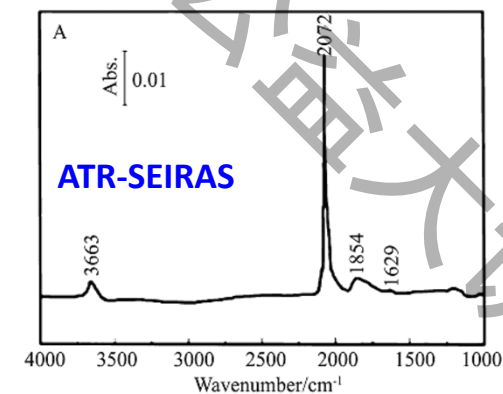
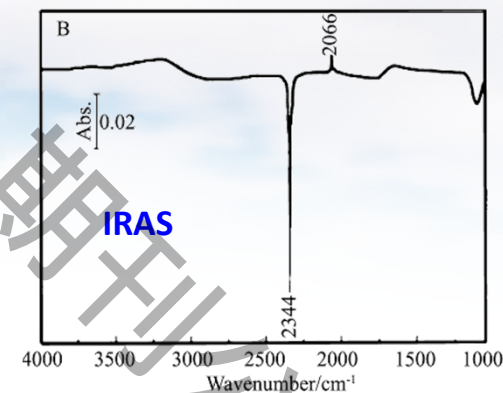
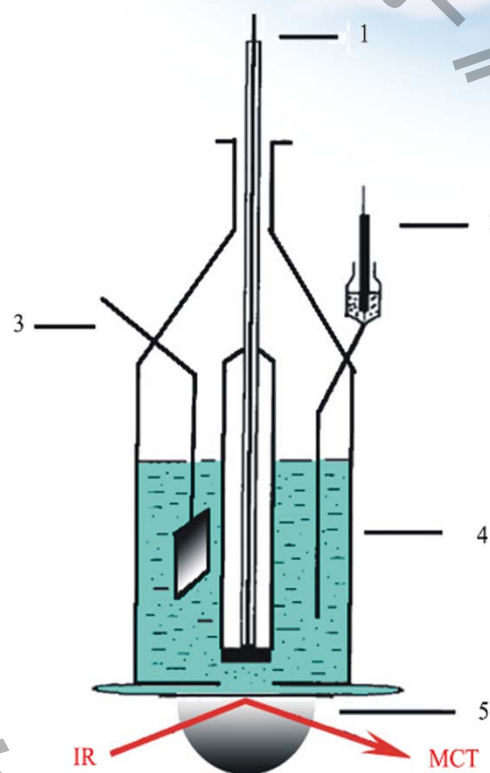
3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8 VIII	9 VIII	10 VIII	11 IB	12 IIB
21 ³ D _{3/2} Sc Scandium 44.955910 [Ar]3d ¹ 4s ² 6.5615	22 ³ F ₂ Ti Titanium 47.867 [Ar]3d ² 4s ² 6.8281	23 ⁴ F _{3/2} V Vanadium 50.9415 [Ar]3d ³ 4s ² 6.7462	24 ¹ S ₀ Cr Chromium 51.9961 [Ar]3d ⁵ 4s ¹ 6.7665	25 ⁶ S _{5/2} Mn Manganese 54.938049 [Ar]3d ⁵ 4s ² 7.4340	26 ⁵ D ₄ Fe Iron 55.845 [Ar]3d ⁶ 4s ² 7.9024	27 ⁴ F _{3/2} Co Cobalt 58.933200 [Ar]3d ⁷ 4s ² 7.8810	28 ³ F ₂ Ni Nickel 58.6934 [Ar]3d ⁸ 4s ² 7.6398	29 ² S _{1/2} Cu Copper 63.546 [Ar]3d ¹⁰ 4s ¹ 7.7264	30 ¹ S ₀ Zn Zinc 65.409 [Ar]3d ¹⁰ 4s ² 9.3942
39 ² D _{3/2} Y Yttrium 88.90585 [Kr]4d ¹ 5s ² 6.2173	40 ³ F ₂ Zr Zirconium 91.224 [Kr]4d ² 5s ² 6.6339	41 ⁶ D _{1/2} Nb Niobium 92.90638 [Kr]4d ⁴ 5s ¹ 6.7559	42 ¹ S ₀ Mo Molybdenum 95.94 [Kr]4d ⁵ 5s ¹ 7.0924	43 ⁶ S _{5/2} Tc Technetium (98) [Kr]4d ⁵ 5s ² 7.28	44 ⁵ F ₅ Ru Ruthenium 101.07 [Kr]4d ⁷ 5s ¹ 7.3605	45 ⁴ F _{3/2} Rh Rhodium 102.90550 [Kr]4d ⁸ 5s ¹ 7.4589	46 ¹ S ₀ Pd Palladium 106.42 [Kr]4d ¹⁰ 8.3369	47 ² S _{1/2} Ag Silver 107.8682 [Kr]4d ¹⁰ 5s ¹ 7.5762	48 ¹ S ₀ Cd Cadmium 112.411 [Kr]4d ¹⁰ 5s ² 8.9938
	72 ³ F ₂ Hf Hafnium 178.49 [Xe]4f ¹⁴ 5d ² 6s ² 6.8251	73 ⁴ F _{3/2} Ta Tantalum 180.9479 [Xe]4f ¹⁴ 5d ³ 6s ² 7.5496	74 ⁵ D ₀ W Tungsten 183.84 [Xe]4f ¹⁴ 5d ⁴ 6s ² 7.8640	75 ⁶ S _{5/2} Re Rhenium 186.207 [Xe]4f ¹⁴ 5d ⁵ 6s ² 7.8335	76 ⁵ D ₄ Os Osmium 190.23 [Xe]4f ¹⁴ 5d ⁶ 6s ² 8.4382	77 ⁴ F _{3/2} Ir Iridium 192.217 [Xe]4f ¹⁴ 5d ⁷ 6s ² 8.9670	78 ³ D ₃ Pt Platinum 195.078 [Xe]4f ¹⁴ 5d ⁹ 6s ¹ 8.9598	79 ² S _{1/2} Au Gold 196.96655 [Xe]4f ¹⁴ 5d ¹⁰ 6s ¹ 9.2255	80 ¹ S ₀ Hg Mercury 200.59 [Xe]4f ¹⁴ 5d ¹⁰ 6s ² 10.4375



改进型宽频检测窗口



IRAS 和 ATR-SEIRAS 测量模式的切换

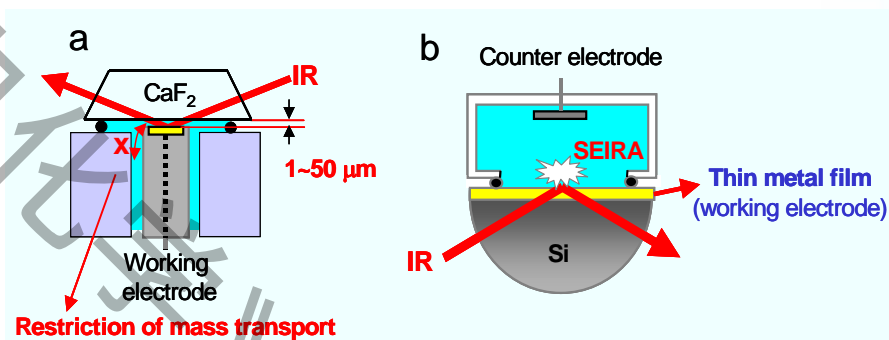


主要内容

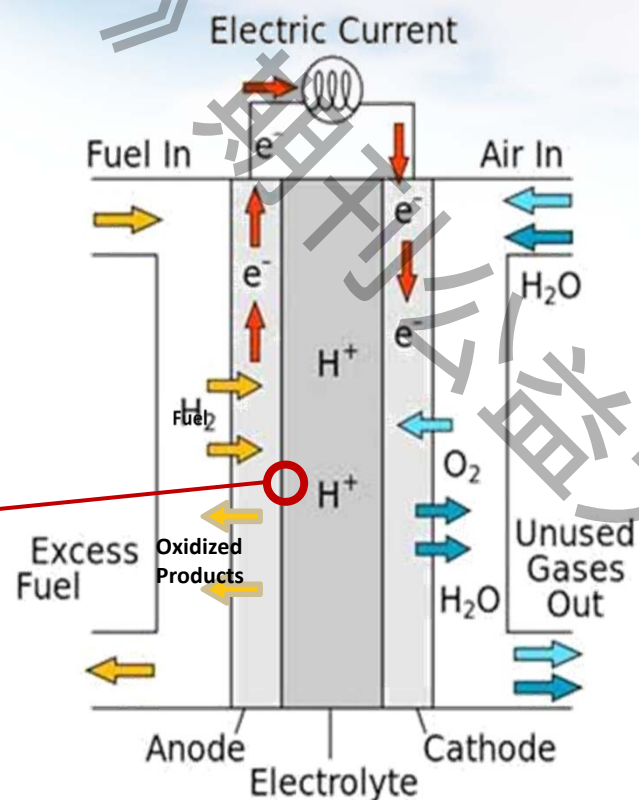
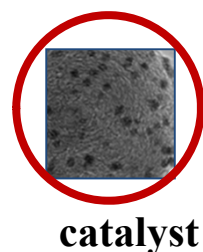
- 电化学研究方法---传统和现代
- 电化学反射红外光谱法基础
- 电化学反射红外光谱应用
- 前沿挑战与展望



原位红外光谱在能源电催化中的应用



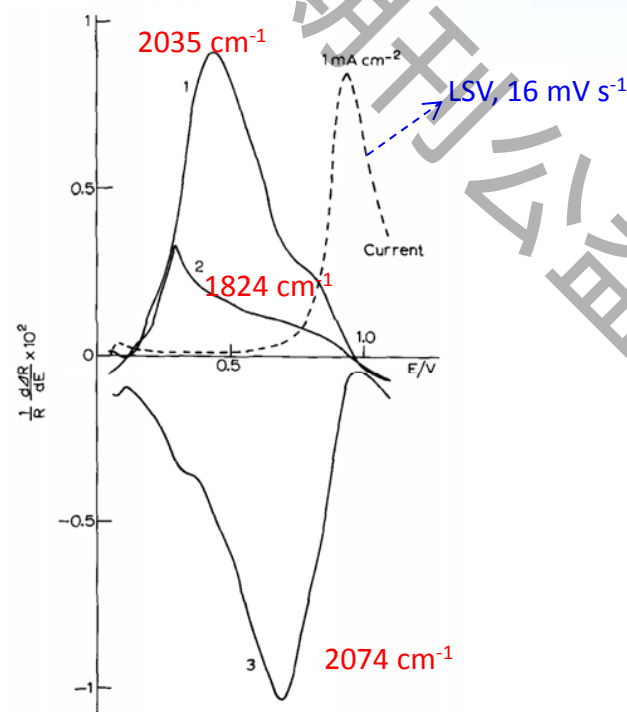
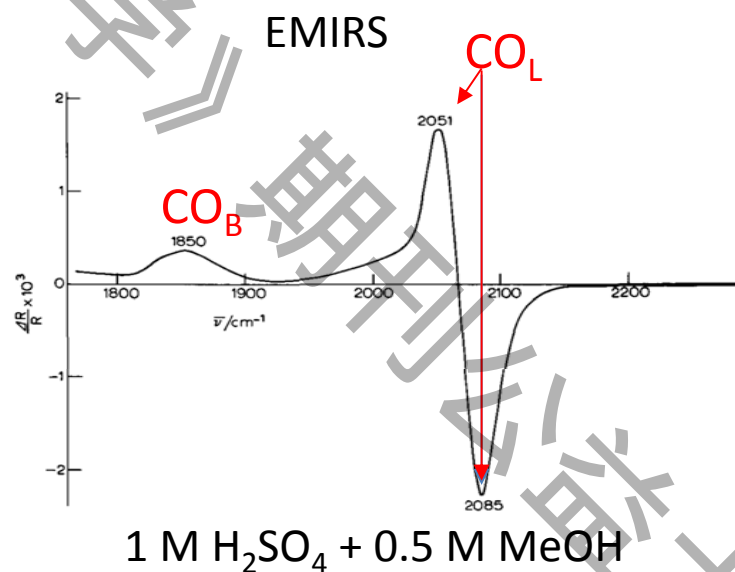
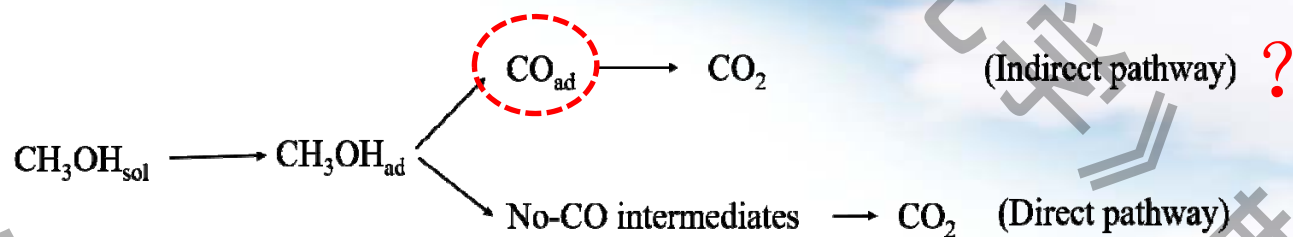
- 揭示电催化反应机理
- 明晰催化剂构效关系
- 指导高效电催化剂研制



直接液体燃料电池示意图



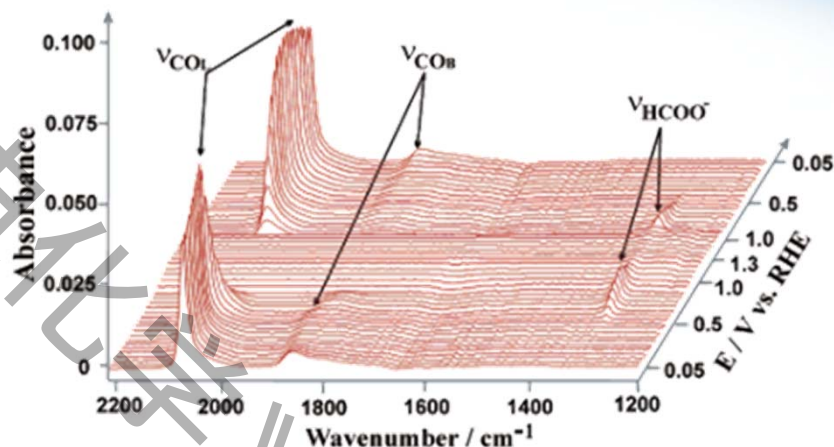
甲醇电氧化中间体CO的确认



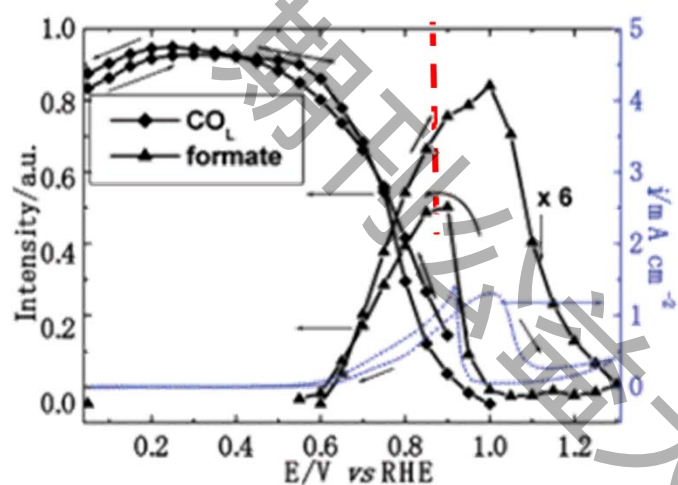
Bewick 利用IRAS首次确认了Pt上甲醇电氧化存在吸附态CO



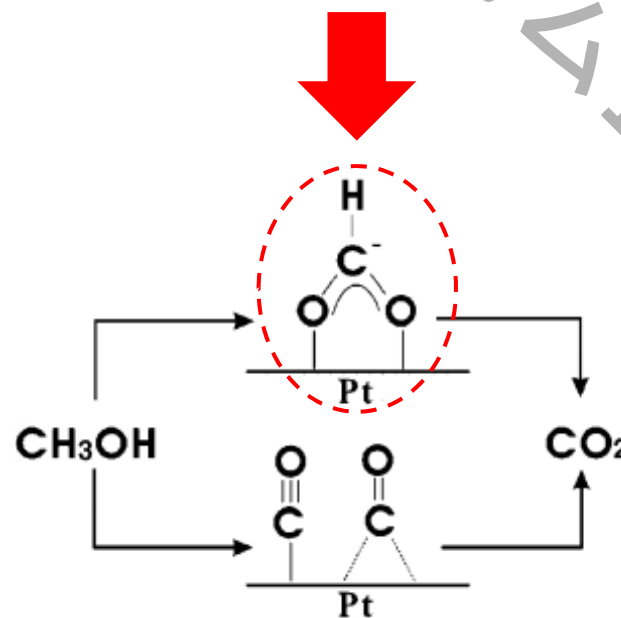
酸性体系中铂上甲醇电氧化机制



- 甲酸根的吸附强度变化趋势与甲醇氧化电流趋势相同
- 利用薄液层红外光谱池控制电极表面电解液流速，排除HCOO_B是溶液相HCOOH吸附

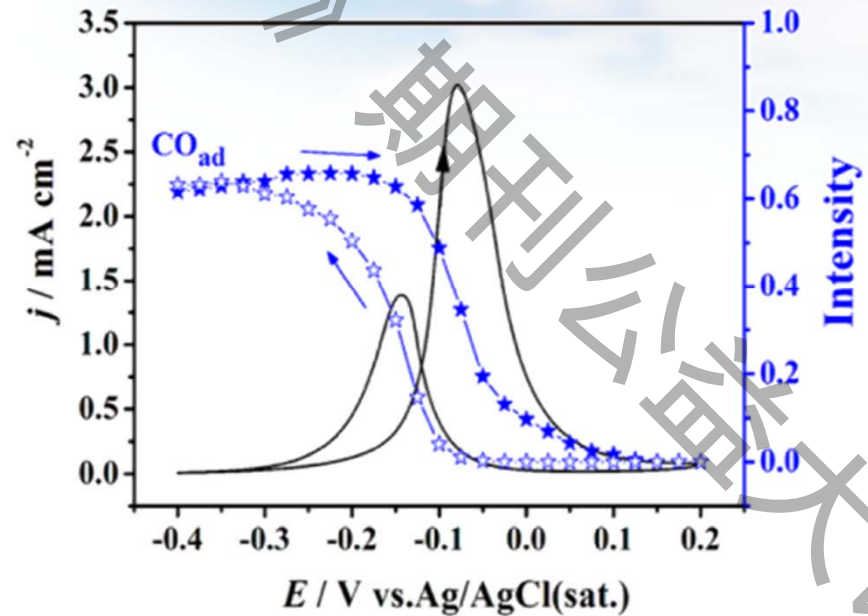
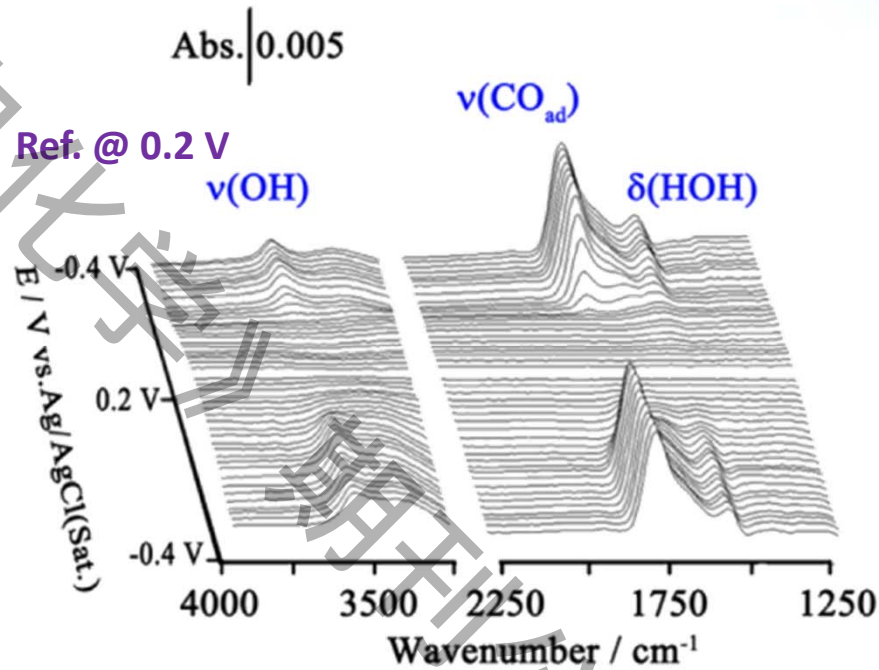


0.5 M CH₃OH + 0.1 M HClO₄



碱性体系中钯上甲醇电氧化机制

ATR-SEIRAS测量表面吸附态物种

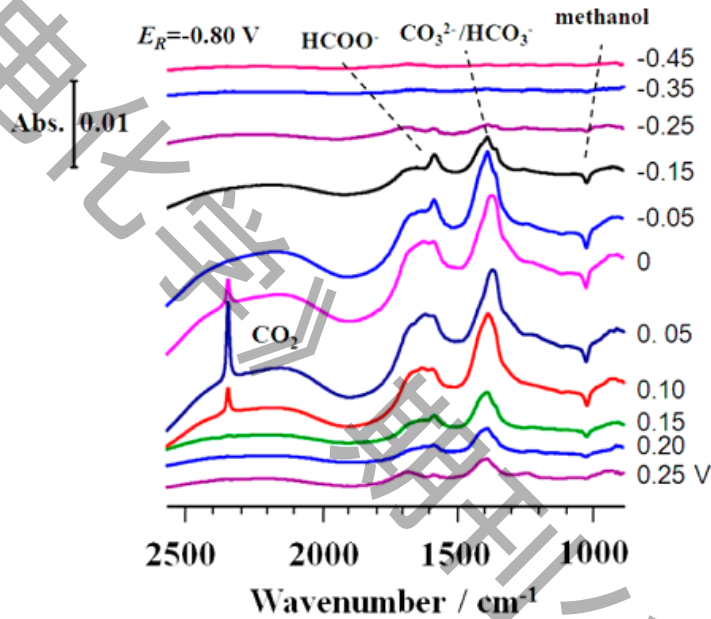


- CO_{ad} 物种，界面共吸附水
- CO_{ad} 氧化起始电位比甲醇的正 (-0.15 V vs. -0.2 V)
- 未测得桥式 HCOO_{B} (1320-1330 cm^{-1}) (弱吸附的甲酸根)



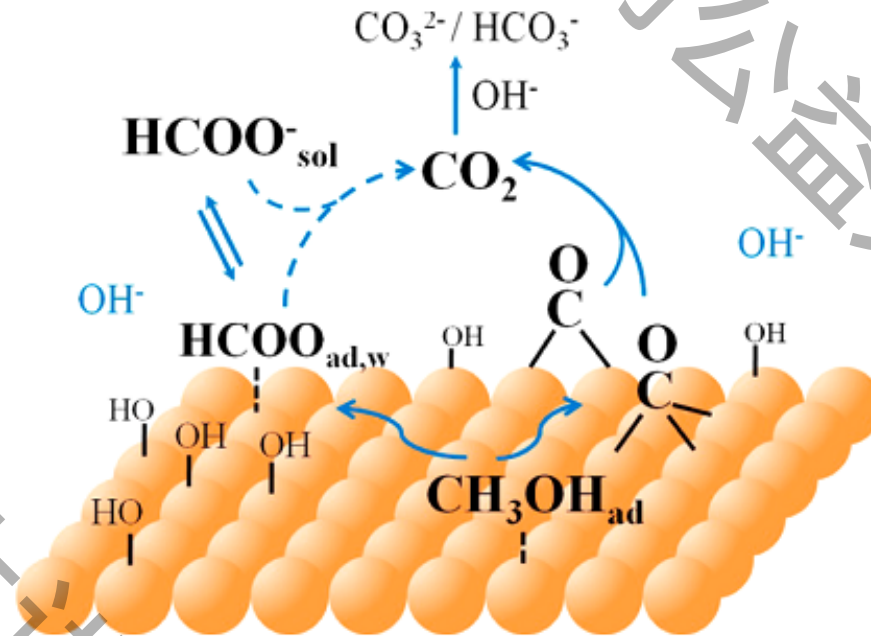
碱性体系中钯上甲醇电氧化机制

IRAS 获得液相产物分布信息



- 吸附的甲酸根一部分快速氧化

$$\text{HCOO}^- + 3\text{OH}^- \rightarrow \text{CO}_3^{2-} + 2\text{H}_2\text{O} + 2\text{e}^-$$
- 另一部分扩散至本体溶液作为副产物

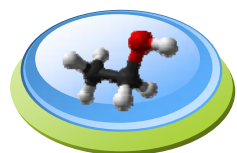


ATR-SEIRAS和IRAS相结合

- 连续脱氢解离成 CO_{ad}
- 弱吸附的甲酸根中间体
- 明确产物电位分布



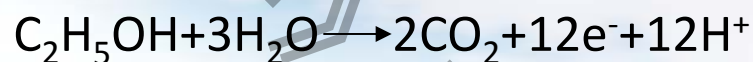
乙醇电氧化反应机理研究



能量密度高
8.1 kWh / kg

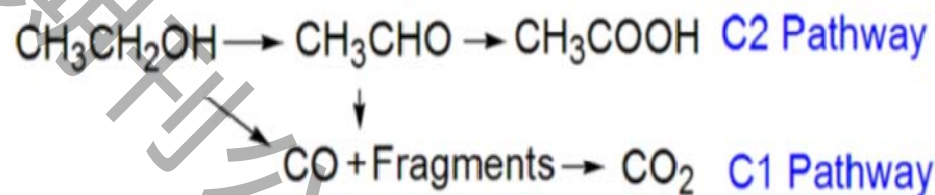
安全、无毒
可再生

设想的阳极反应:



存在的瓶颈问题:

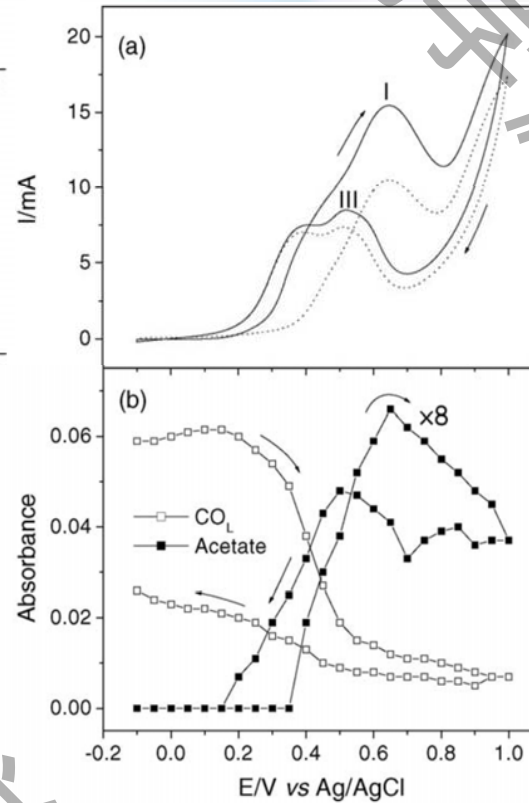
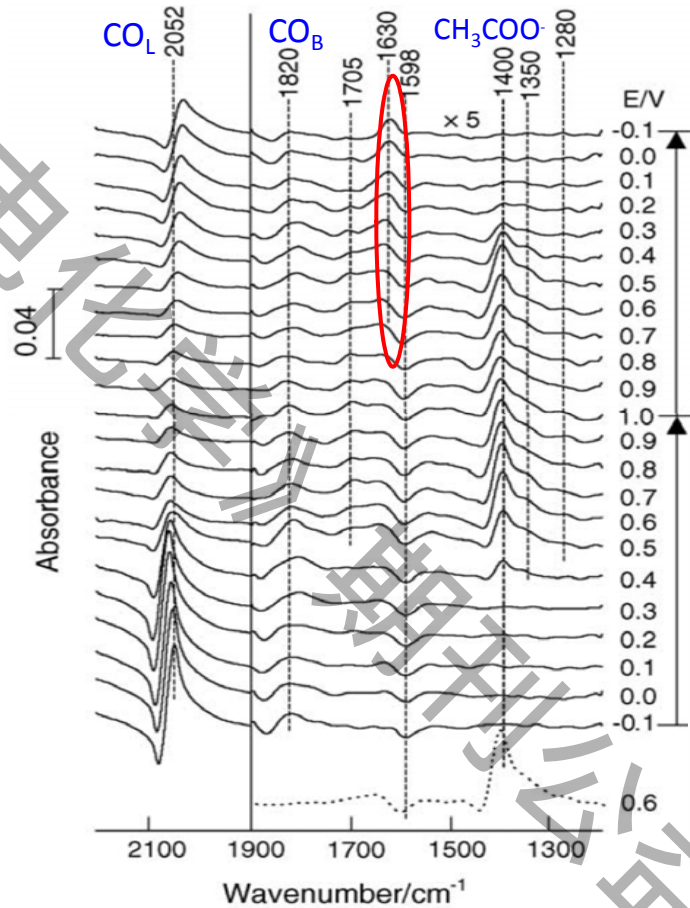
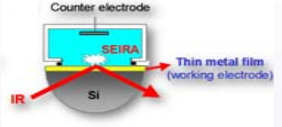
- 乙醇电氧化的动力学迟缓
- 乙醇断裂C-C键困难，能量利用率低



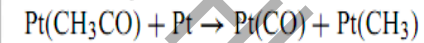
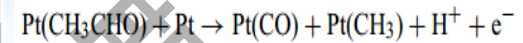
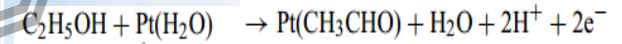
- ◆ 反应关键中间体：乙醛？乙酰？
- ◆ C-C键断裂的机制，C1路径的比例
- ◆ 解离强吸附物种对EOR反应的影响



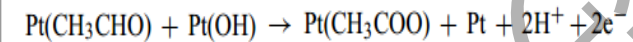
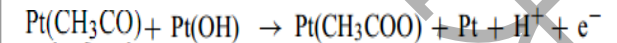
酸性体系铂上乙醇电氧化机制



1630 cm⁻¹



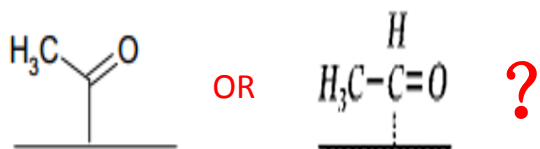
1400 cm⁻¹



- 证实了乙醇电氧化中存在吸附态CO和乙酸根
- 乙醛或乙酰吸附物种中间体?
- 高电位下仍存在吸附态乙酸根, 阻碍了反应进行



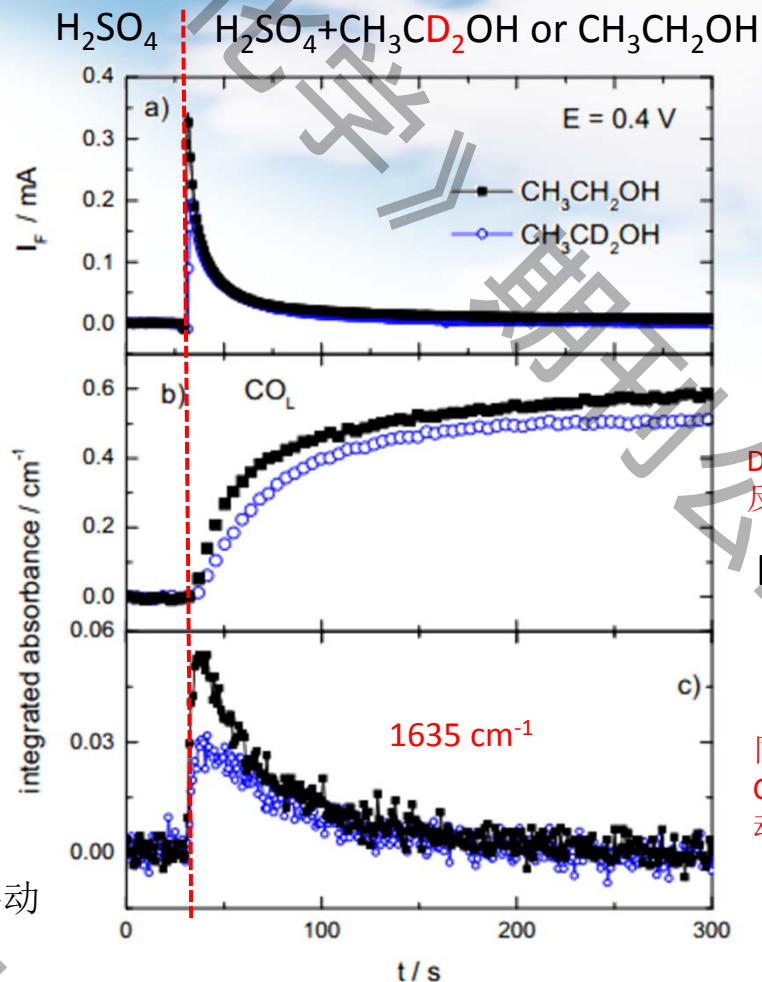
结合同位素效应辨析反应中间体



$$\nu = \frac{1}{2\pi c} \sqrt{\frac{k}{\mu}}$$

折合质量

同位素取代后，吸附态乙醛振动频率会向低波数移动



D取代H后，
反应动力学变缓

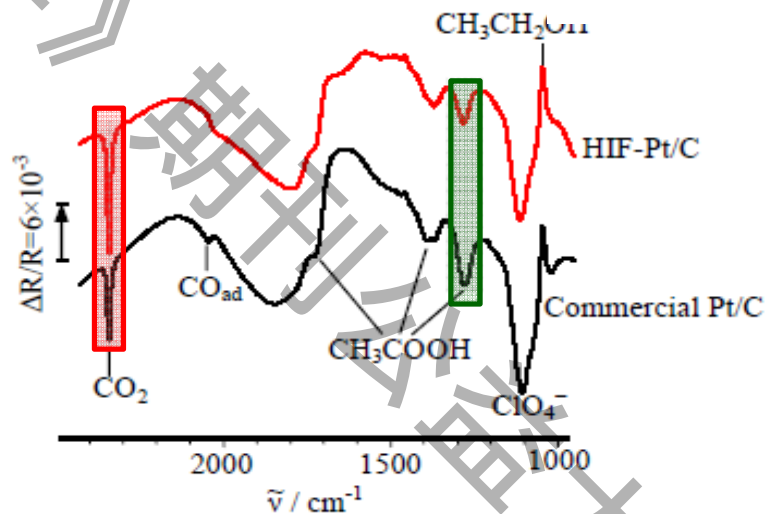
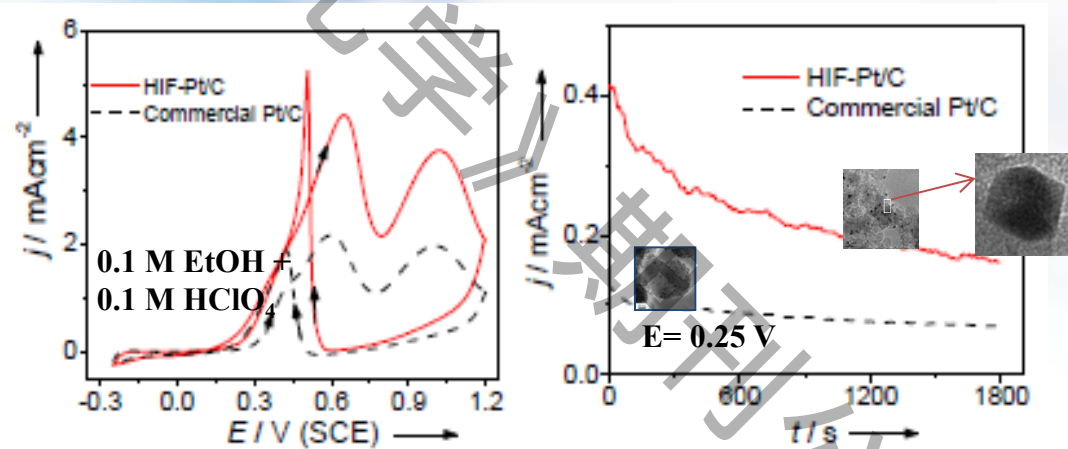
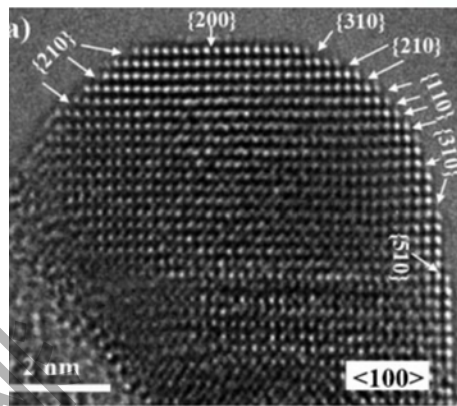
KIE效应

同位素取代后，
C=O谱峰未发生移动：
1635 cm⁻¹

酸性条件，乙酰是乙醇在Pt上电氧化的关键中间体



催化剂表面结构对乙醇电氧化路径的影响



$$S = \frac{I(\text{CO}_2, 2342\text{cm}^{-1})}{I(\text{CH}_3\text{COOH}, 1280\text{cm}^{-1})}$$

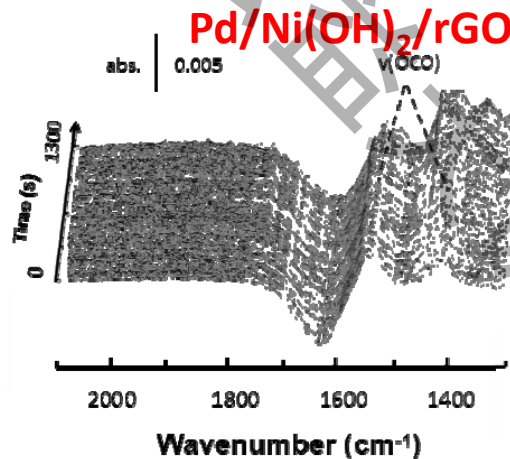
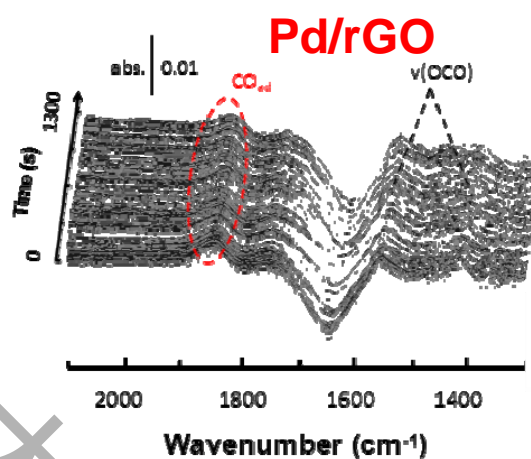
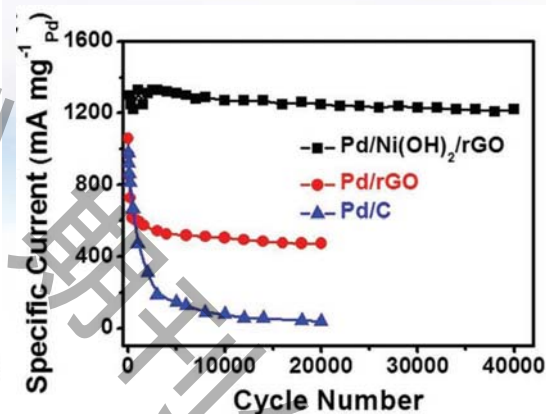
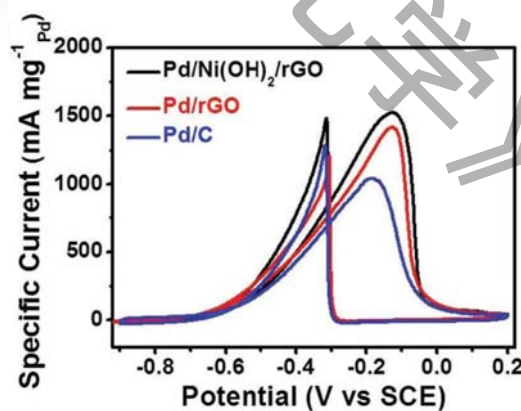
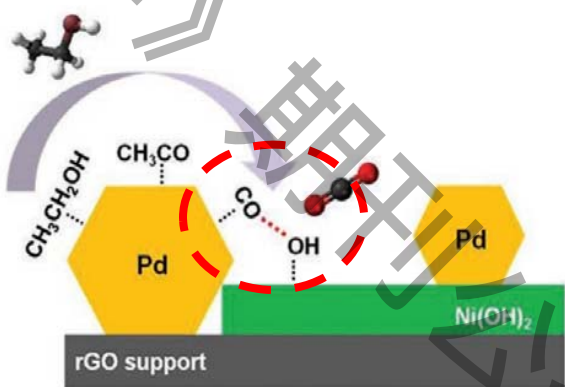
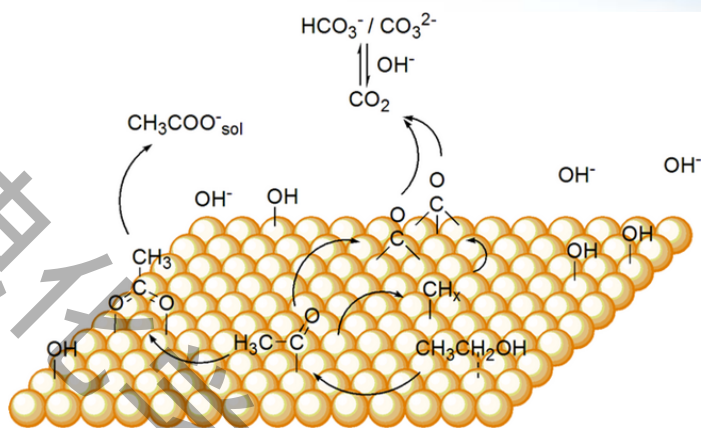
$$S = 1.06$$

$$S = 0.47$$

台阶位原子具有更高的断裂乙醇C-C键能力



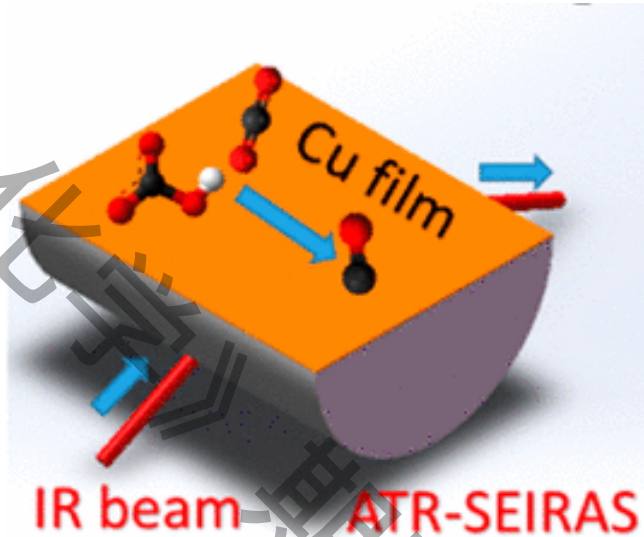
机理认识指导下的高效催化剂合成



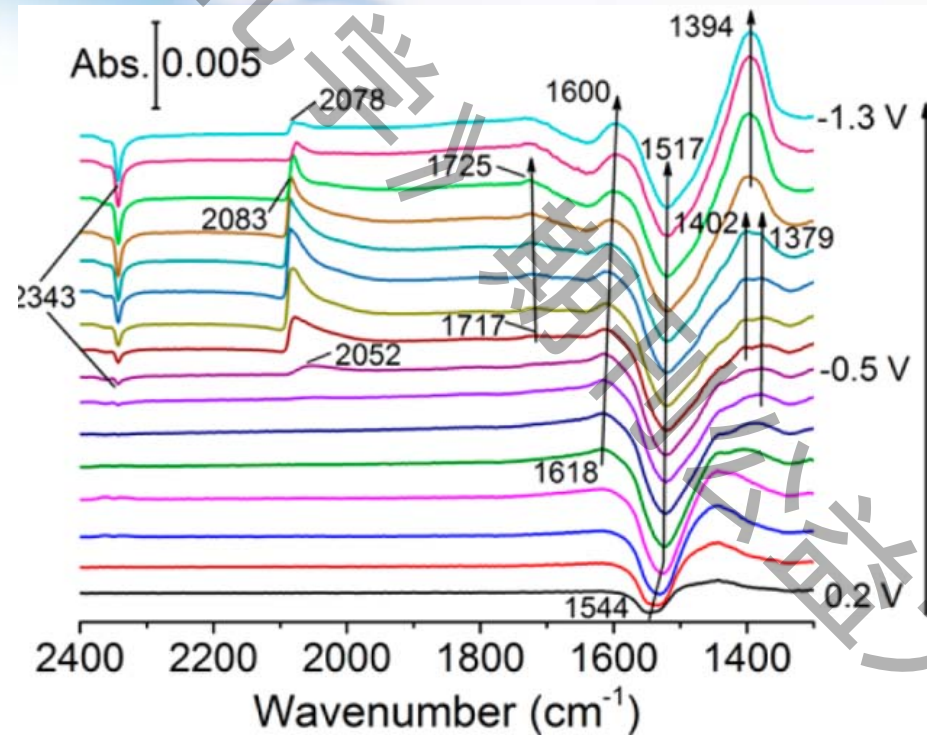
ATR-SEIRAS证实： $\text{Ni}(\text{OH})_2$ 载体的引入可以有效促进中间体 CO_{ad} 的氧化，从而大幅提升其催化乙醇的稳定性和C1路径的比例



铜电极上 CO_2 电还原机理探究



CO_2 饱和0.1 M KHCO_3 溶液



At -0.5 V, 2083 and 2052 cm^{-1} , *CO; 产乙烯和甲烷等的中间体

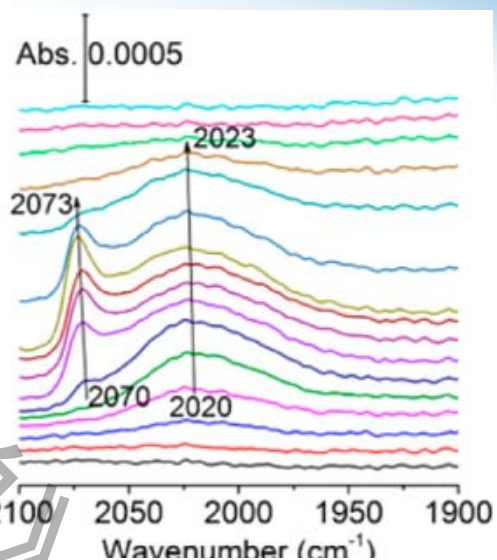
-0.6 和 -1.2 V, 1720 cm^{-1} , *CHO; 产CO中间体

-0.7 和 -0.8 V, 1402 cm^{-1} , bidentate *COO⁻; 1379 cm^{-1} , *COOH; 产甲酸的中间体

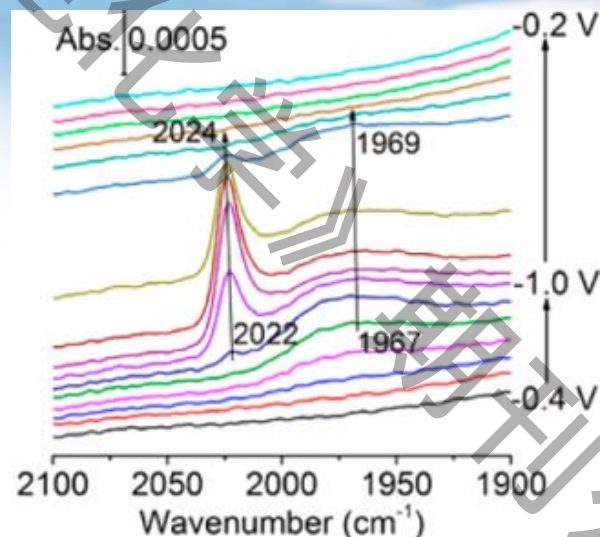
< -0.9 V, 1394 cm^{-1} , 溶液中的 CO_3^{2-}



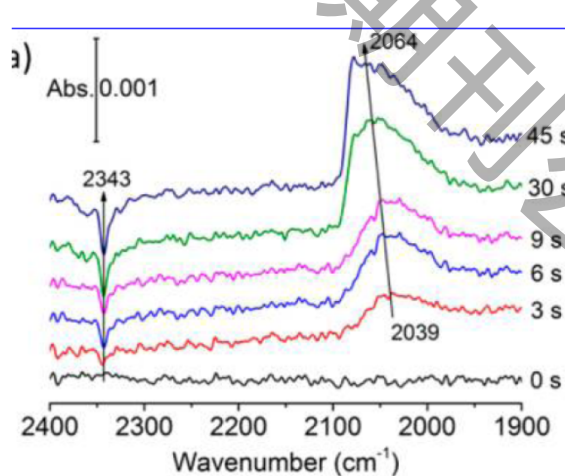
CO₂电还原反应中碳酸氢根的作用



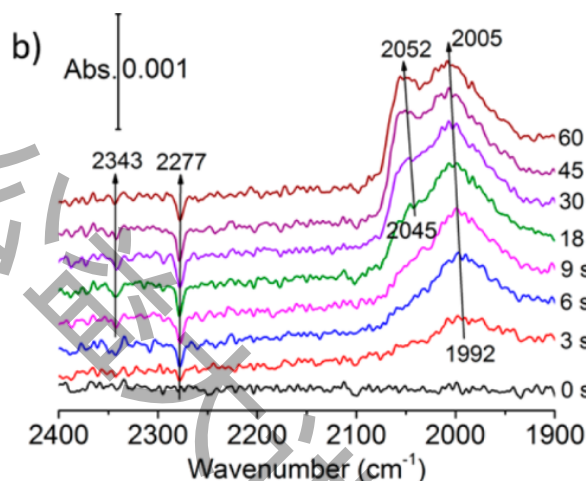
Ar-saturated 0.1 M (a) KH¹²CO₃



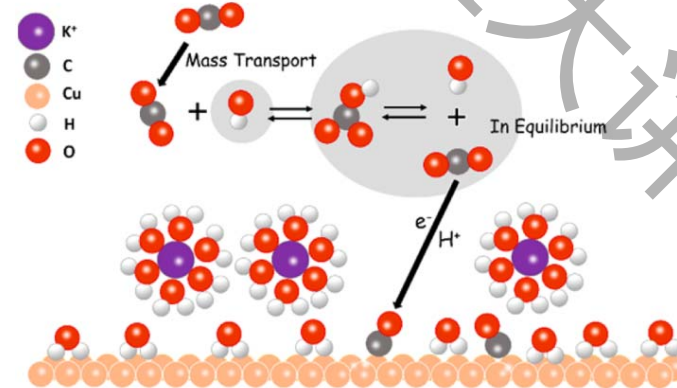
Ar-saturated 0.1 M (b) KH¹³CO₃



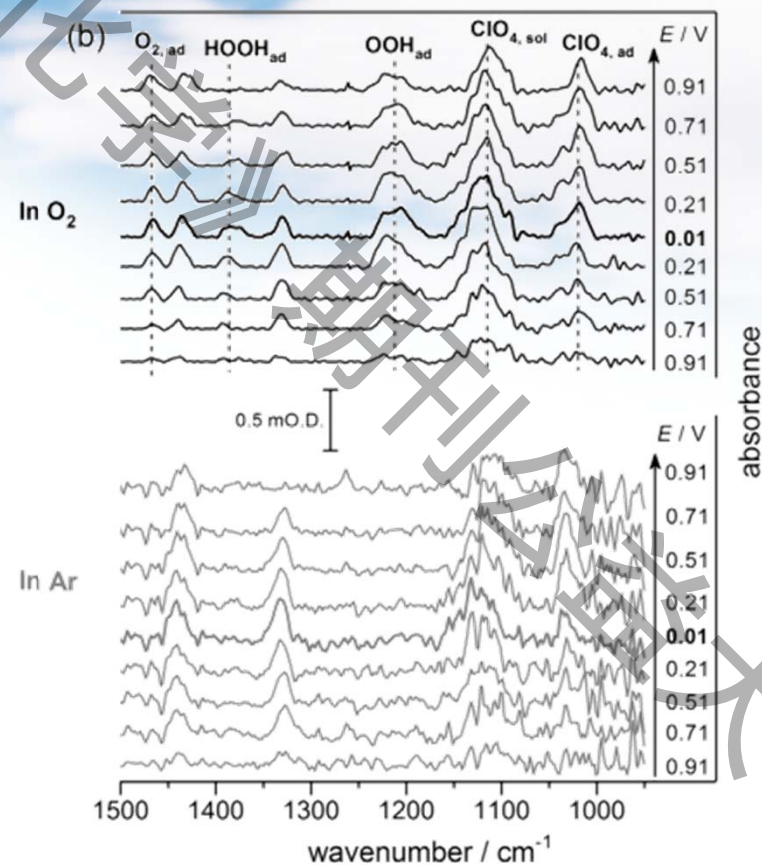
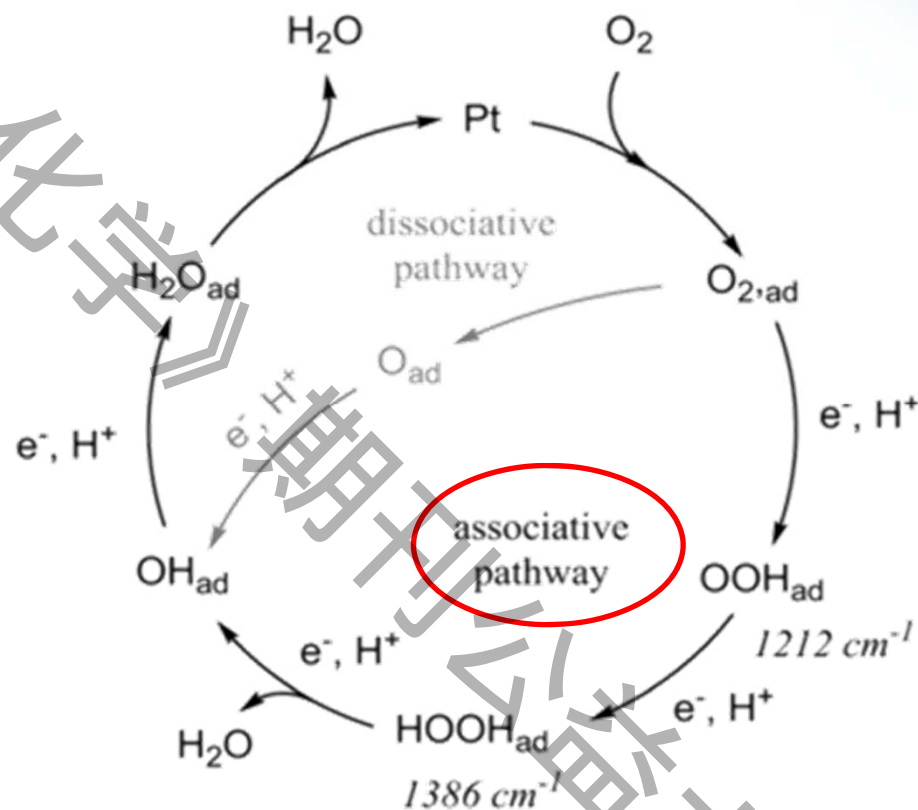
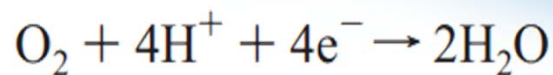
¹²CO₂-0.1M KH¹²CO₃



¹²CO₂-0.1 M KH¹³CO₃



ATR-IR方法研究Pt/C上氧气还原反应路径



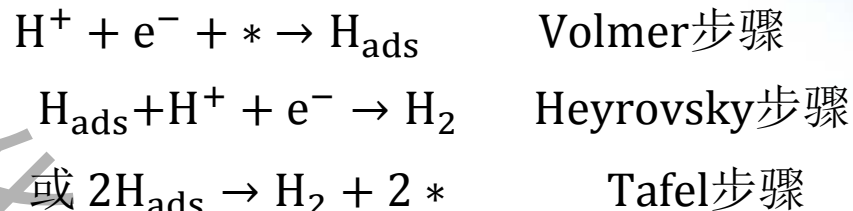
OOH _{ad}	1212 cm ⁻¹
HOOH _{ad}	1386 cm ⁻¹
O _{2,ad}	1468 cm ⁻¹

ATR-IR确认了 ORR反应包含associative pathway

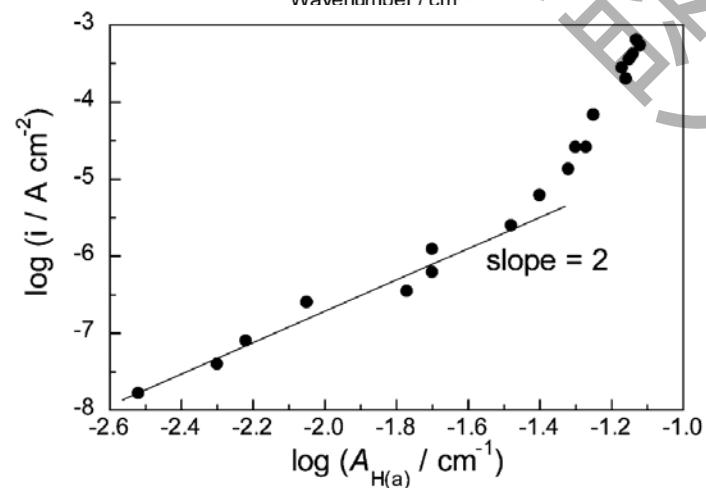
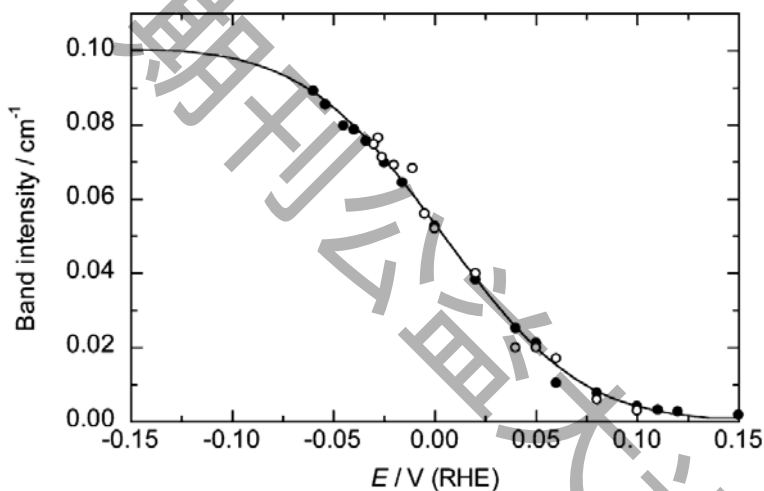
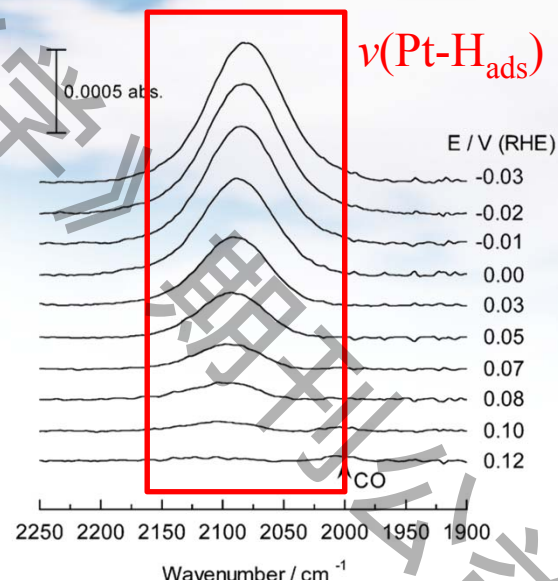


ATR-SEIRAS方法研究Pt上HER反应机理

HER反应的基元步骤:



经典电化学方法研究表明, Pt上的HER反应为Volmer-Tafel机理, 其中Tafel步骤为决速步

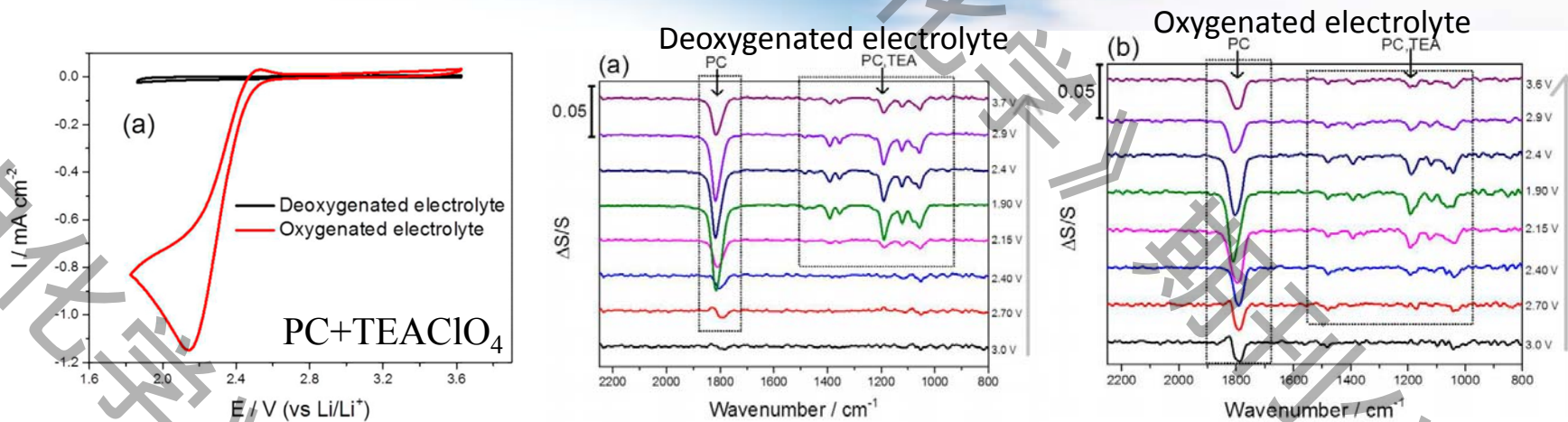


利用ATR-SEIRAS可以从分子层面探究HER反应机理。

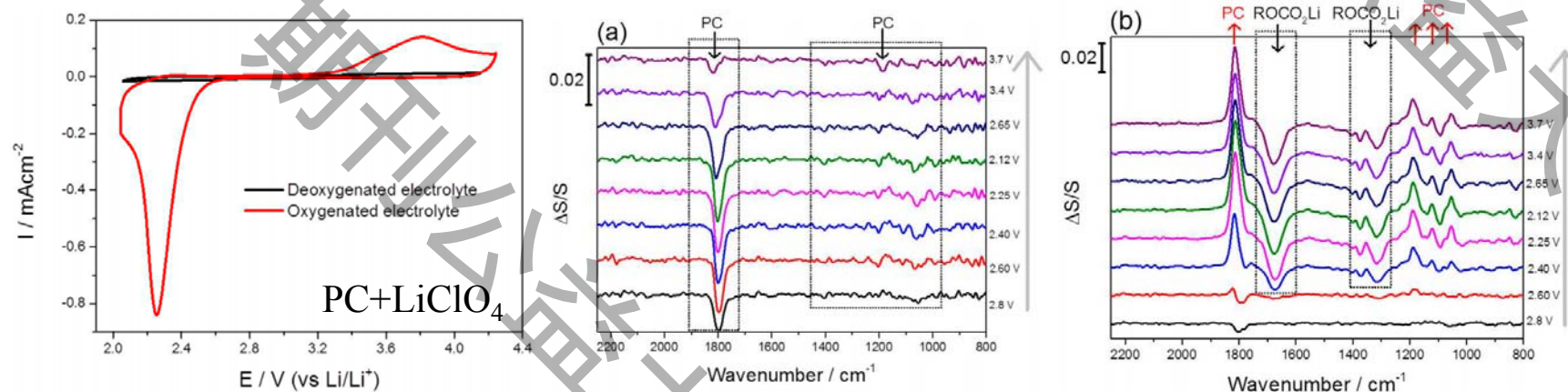


原位红外光谱在锂空电池研究中的应用

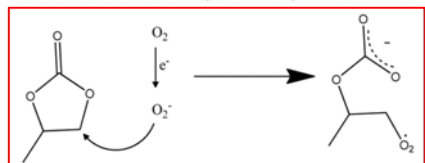
- SEIRAS研究超氧根诱导的碳酸丙烯酯(PC)电解液开环



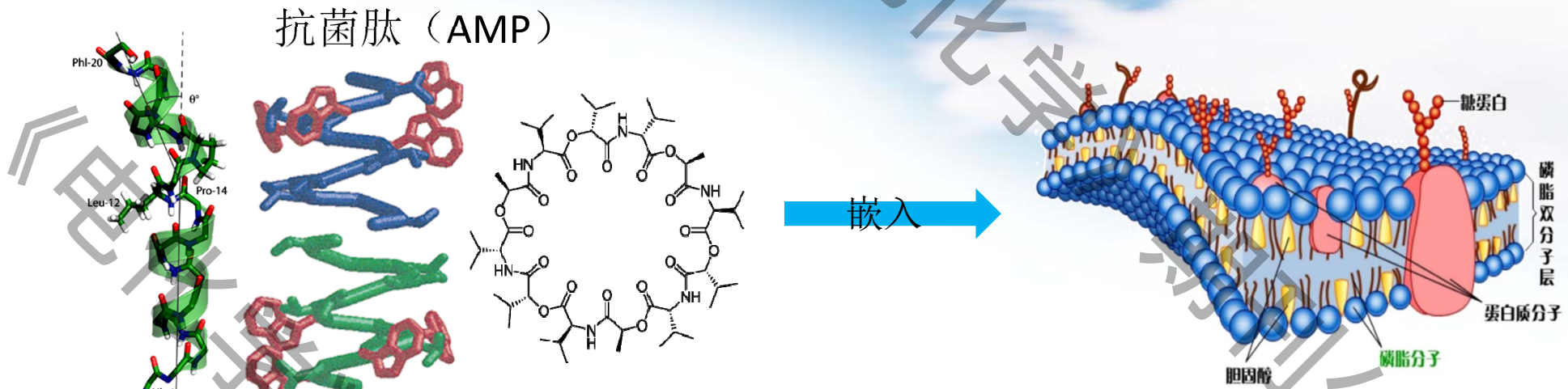
在非盐体系下，界面处未检测到副产物的信号



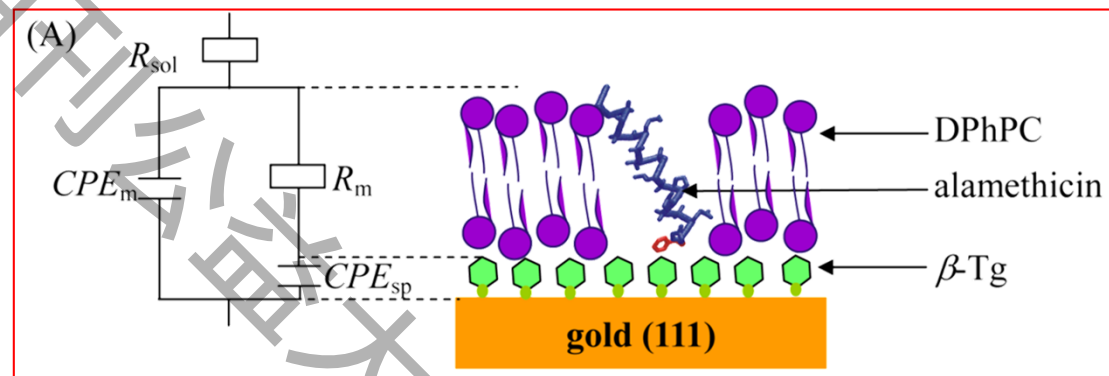
锂盐体系中，电解液(PC)分解，发生开环反应



原位红外光谱在生物化学中的应用



抗菌肽能够选择性地嵌入细菌的生物膜，并形成一个离子通道，改变细菌细胞内外的渗透压，从而杀死细菌。

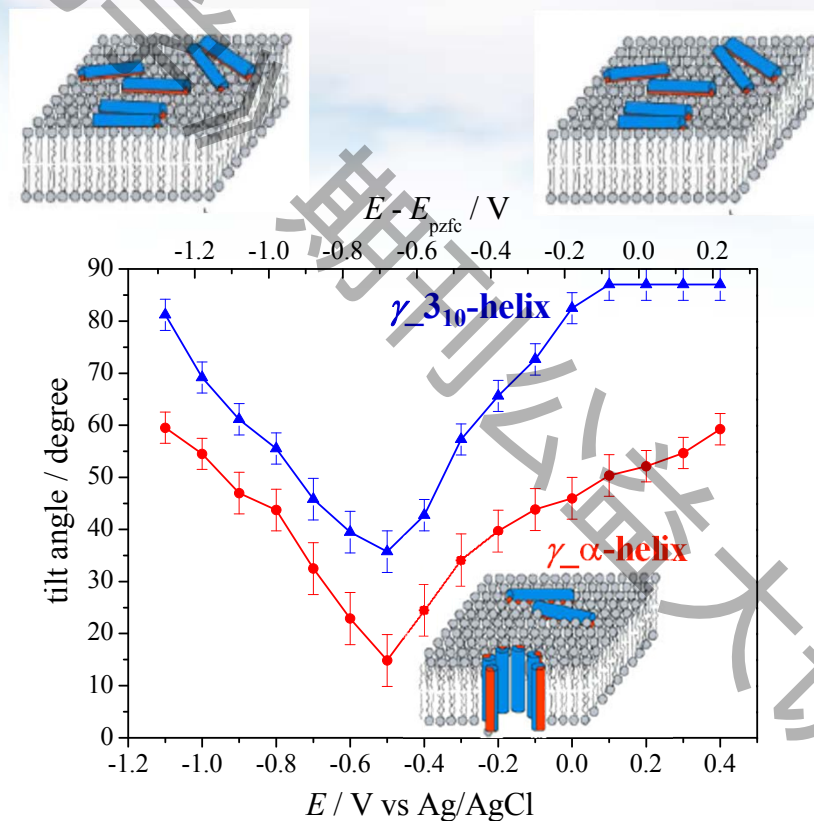
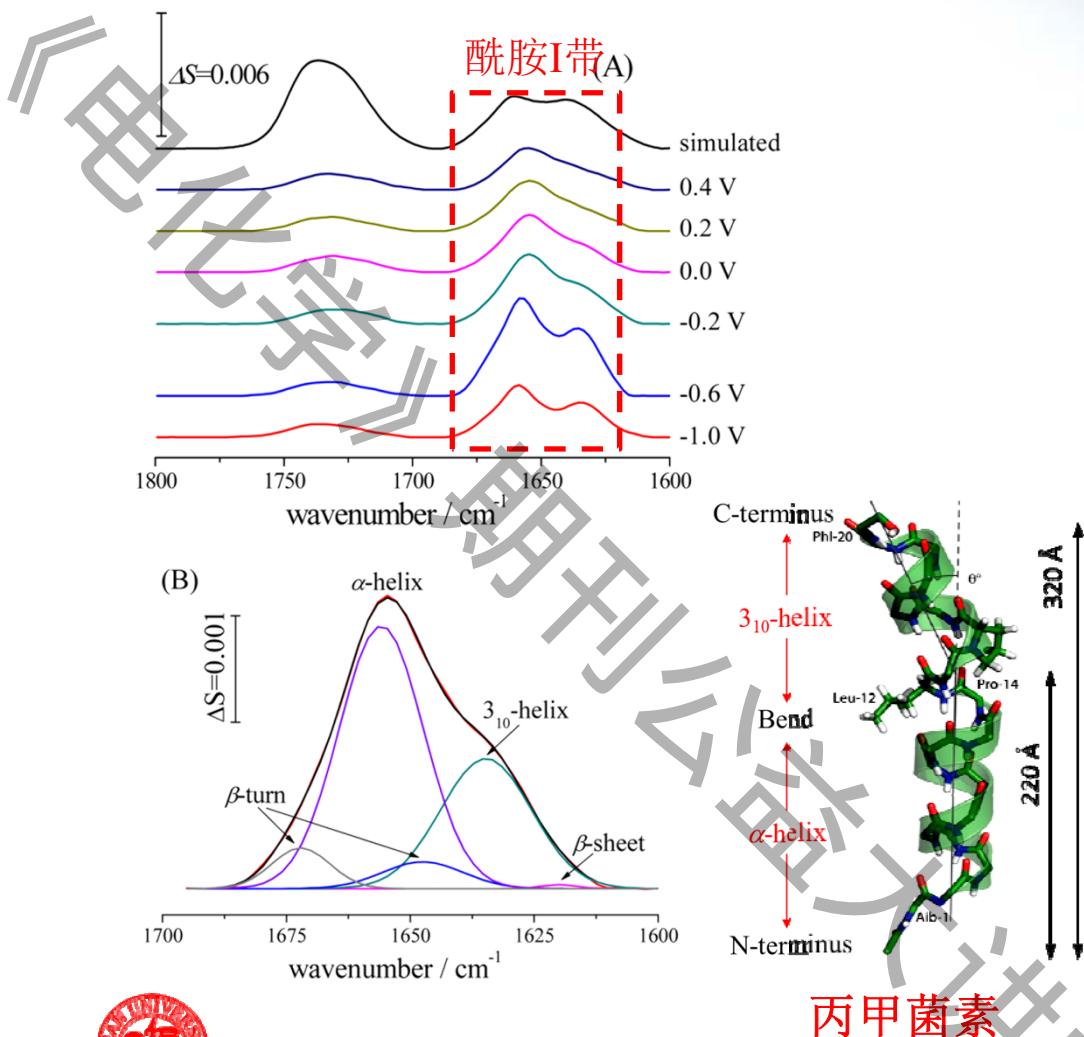


利用电化学方法模拟跨膜电位
利用红外光谱获得生物膜中的化学信息



跨膜电压对丙甲菌素透化脂双层的影响

- 模拟跨膜电压（外加电场）对丙甲菌素在磷脂双分子层中嵌入的影响

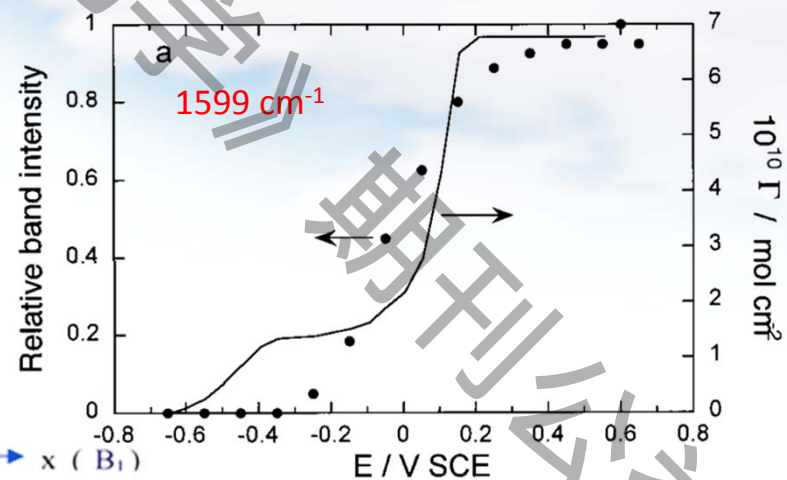
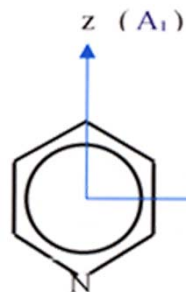
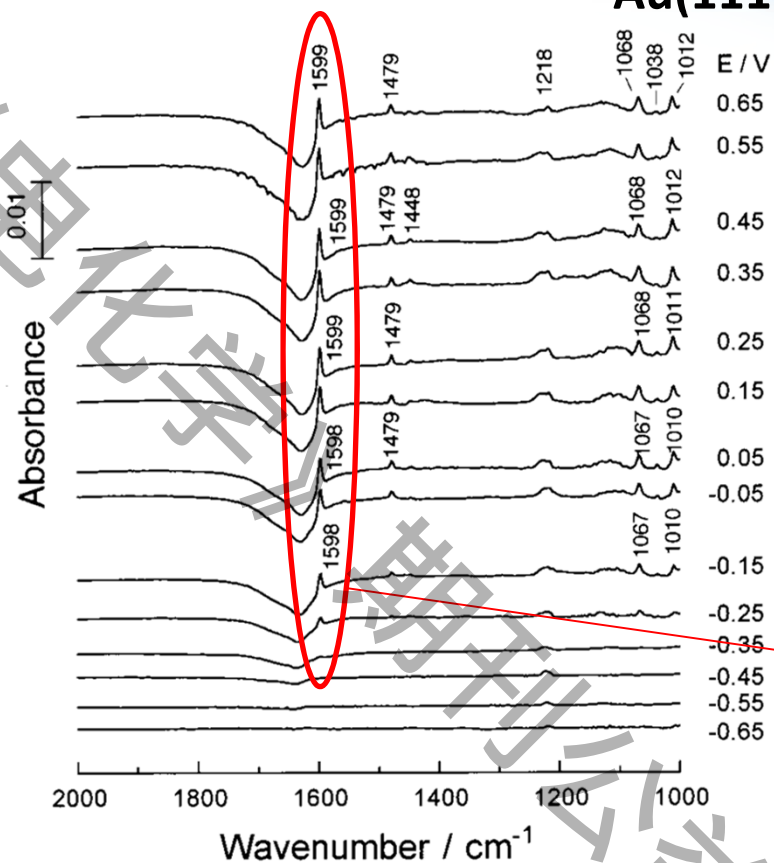


- 电位较正时，丙甲菌素分子平躺在磷脂双分子层表面；
- 当电位负移时，丙甲菌素已经嵌入到磷脂双分子层中，并形成了一个离子通道。



原位红外光谱与STM结合研究

Au(111)上吡啶吸附构型转变



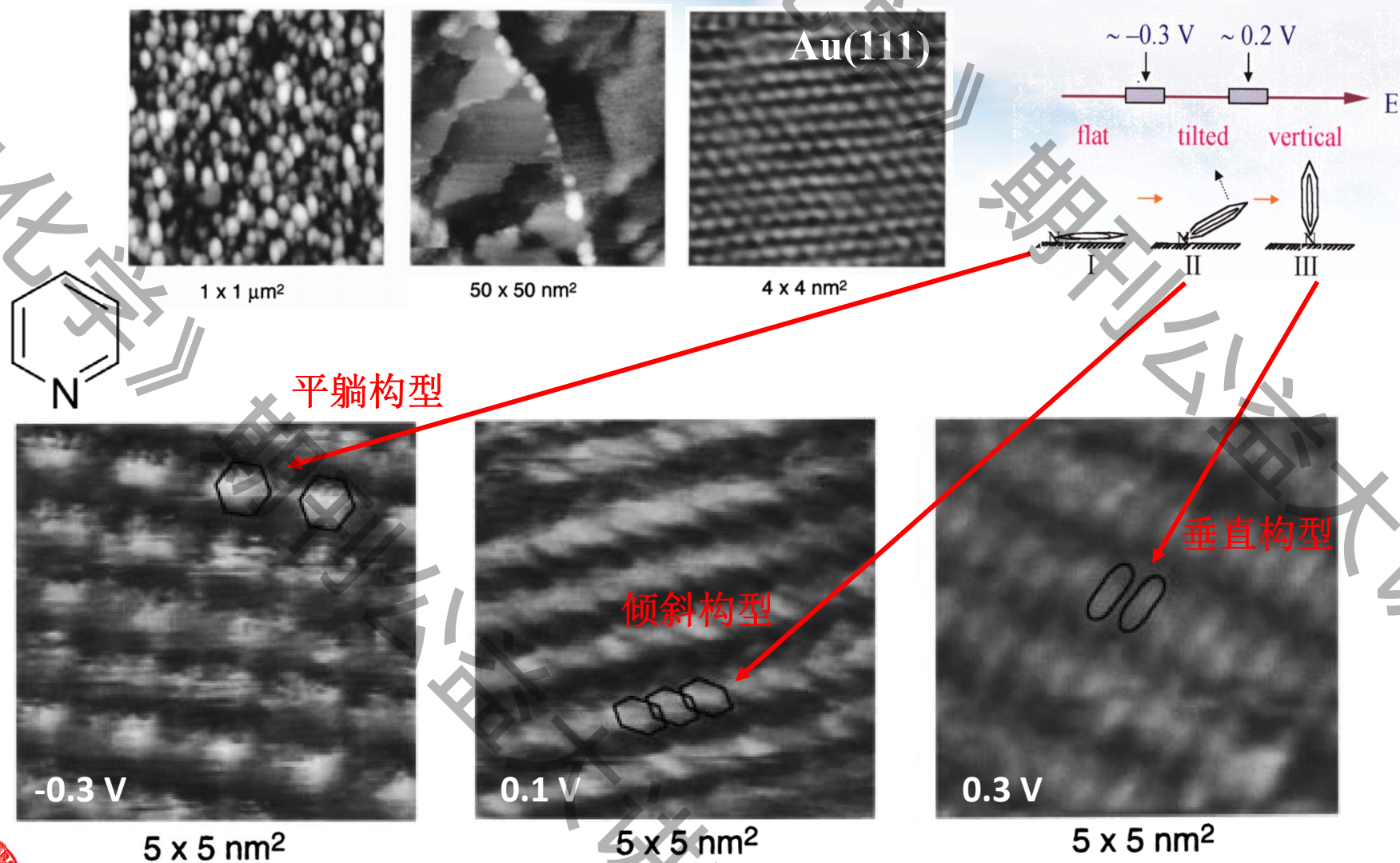
mode no. ^a	symmetry ^a	pyridine/Au(111)	liquid pyridine
ν_{8a}	A_1	1599 vs ^b	1580 m
ν_{8b}	B_1		1572 s
ν_{19a}	A_1	1479 m	1482 m
ν_{19b}	B_1	1448 w	1439 vs
ν_{14}	B_1		1375 vw
ν_{9a}, ν_3	A_1, B_1	1218 w	1218 w
ν_{15}	B_1		1148 w
ν_{18b}	B_1		1085 m
ν_{18a}	A_1	1068 m	1068 m
ν_{12}	A_1	1038 w	1029 m
ν_1	A_1	1012 m	992 m

$$I \propto C_{\text{surf}} \left| \vec{E} \cdot \frac{d\vec{u}}{dQ} \right|^2 = C_{\text{surf}} \cos^2 \theta |\vec{E}|^2 \left| \frac{d\vec{u}}{dQ} \right|^2$$



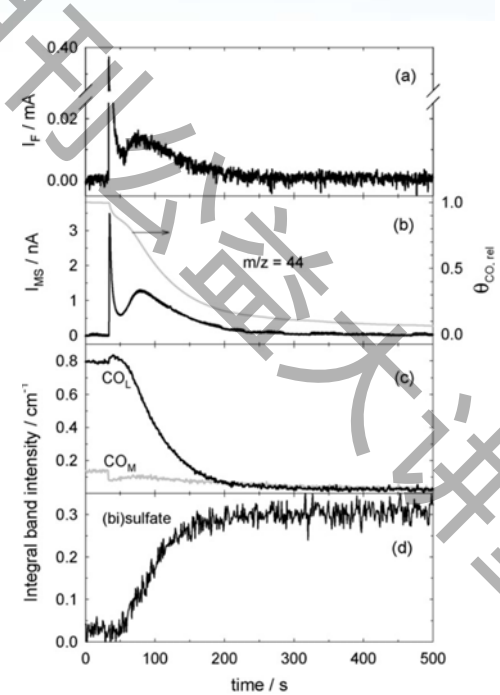
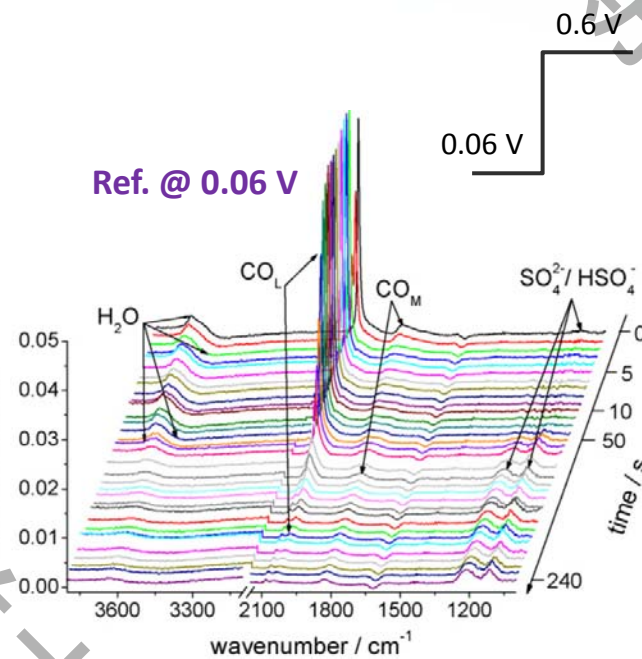
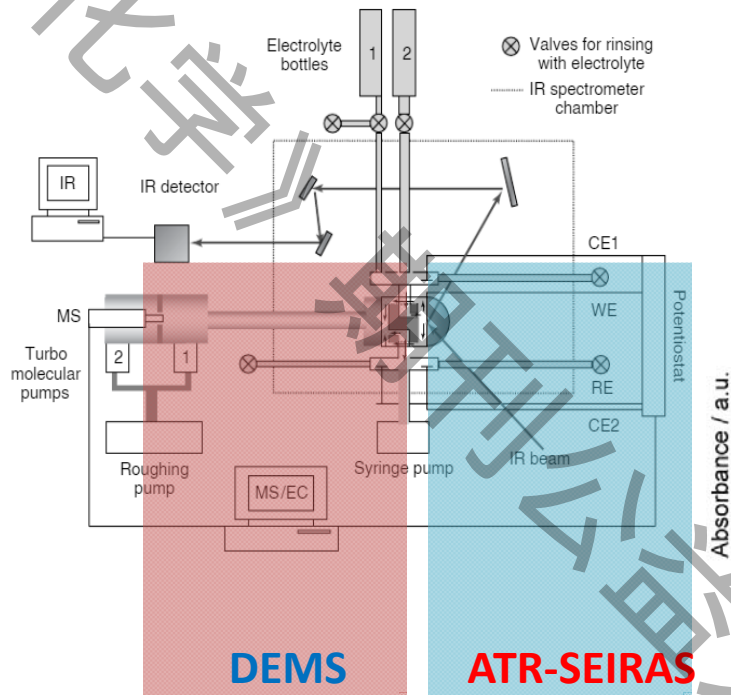
原位红外光谱与STM结合研究

Au(111)上吡啶吸附构型转变



原位红外光谱与在线质谱的联用

- 对于电化学反应过程中，**表面吸附物种 (IR)** 和**挥发性中间体/产物 (DEMS)** 的同步检测，定量分析可挥发性产物和副产物

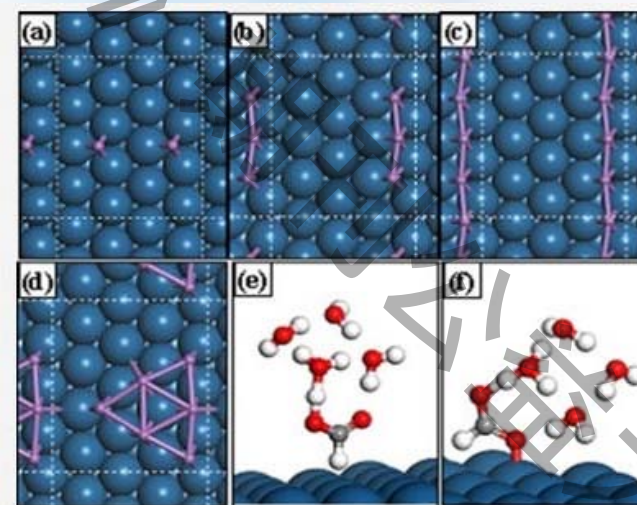
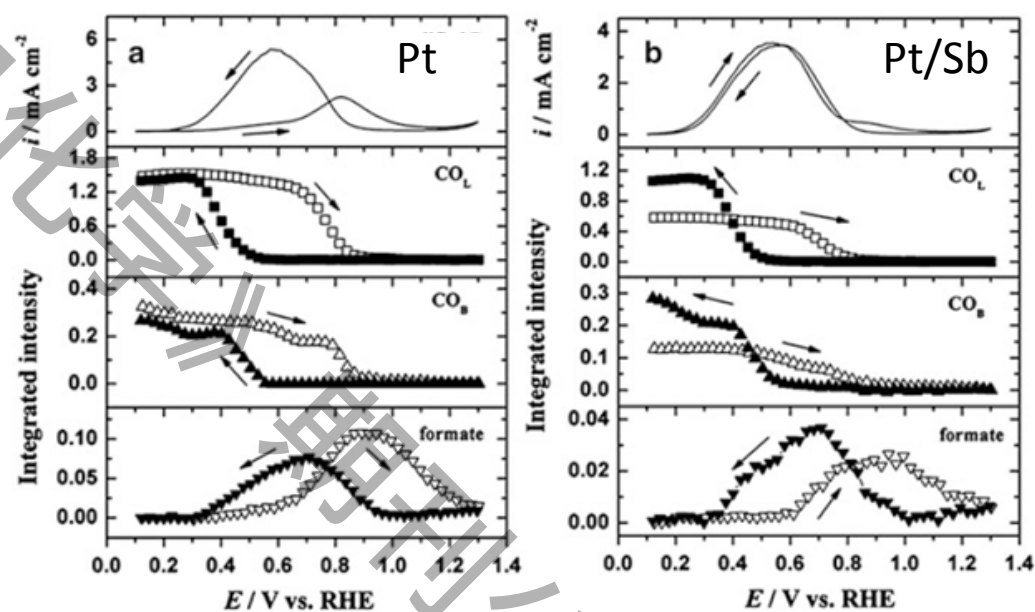


Pt上单层吸附CO的定电位氧化



电化学红外光谱测量和理论计算的结合

- Pt表面Sb修饰提升甲酸电催化活性



- 谱学证据: HCOO_B 和CO的覆盖度均下降
- DFT计算: Sb修饰降低CO在Pt上的吸附能; 甲酸以C-H端接近电极, 利于脱氢反应



主要内容

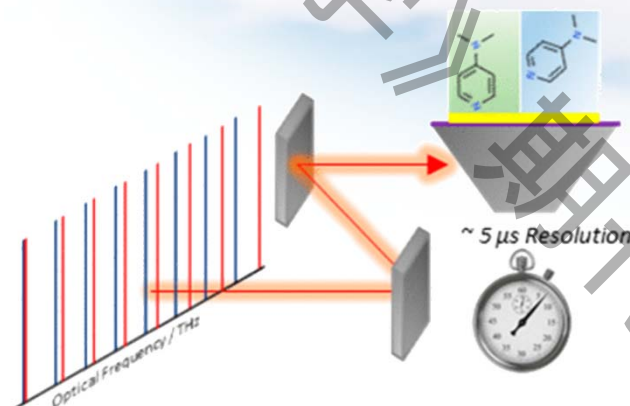
- 电化学研究方法---传统和现代
- 电化学反射红外光谱法基础
- 电化学反射红外光谱应用
- 前沿挑战与展望



电化学红外光谱发展的前沿挑战与展望

- 原位红外光谱测量技术发展:

- ✓ 时间分辨光谱

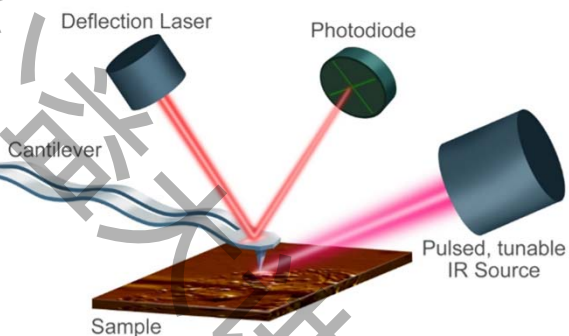


双光梳红外光谱

- ✓ 空间分辨光谱: **EC-AFM-IRFEL**;

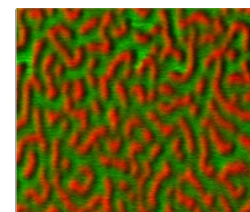
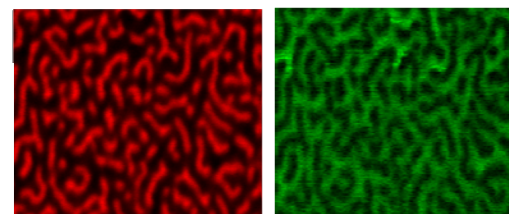
AFM: 不具备分辨化学组成的能力

IR: 空间分辨率受光学衍射极限所限



PMMA 1730 cm^{-1}

PS 1492 cm^{-1}

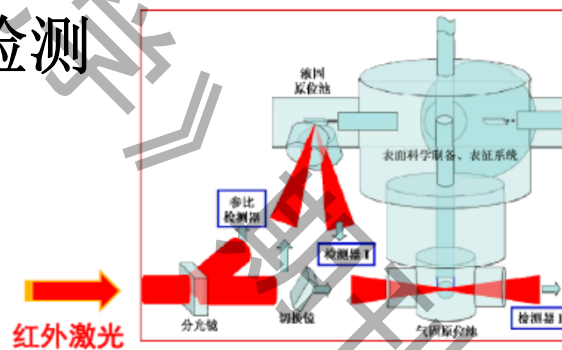


Combined



电化学红外光谱发展的前沿挑战与展望

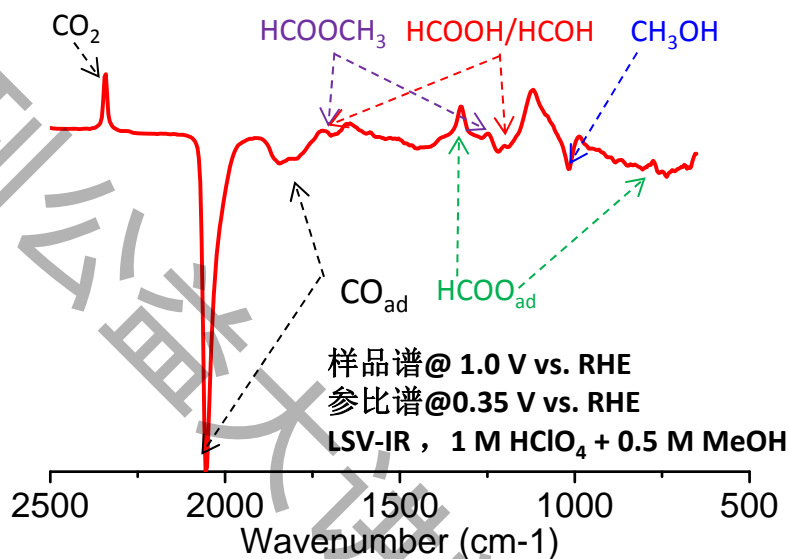
✓ 低覆盖度、低浓度反应中间物种检测



IRFEL-IRRAS

光检测实验站系统图

✓ ATR-IR低频、吸附态、溶液态物种全检测



电化学红外光谱发展的前沿挑战与展望

- 与其他实验和理论方法结合或联用，获得对复杂的结构效应和表界面反应过程全面认识
- 研究体系的拓展，外场（如光、电、磁）调制下的电化学反应
- 工况条件下电化学反应体系的监测
-



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