中子敏感的原子层沉积型微通道板探测器的物理与关键技术研究

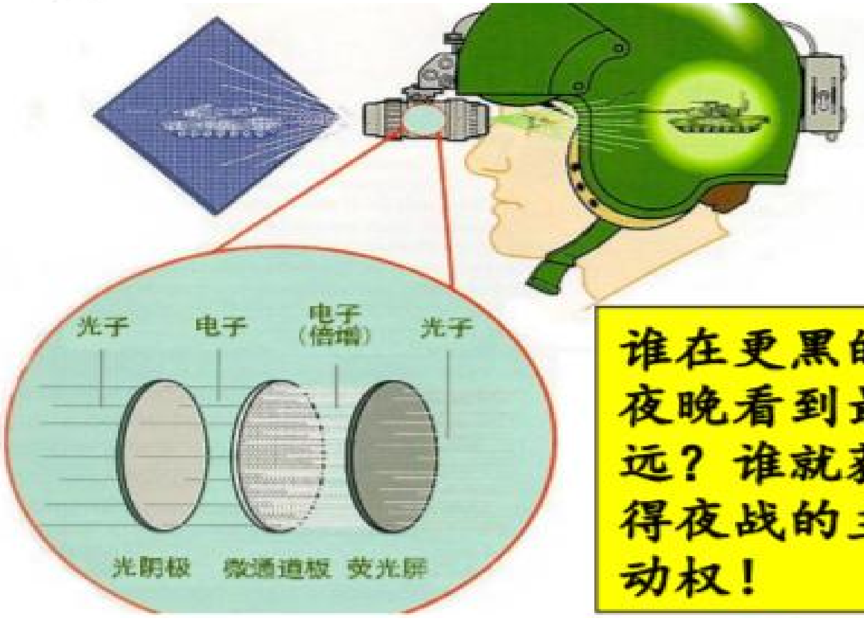
报告人：王玉漫
合作导师：孙志嘉、刘术林
报告时间:2022/06/30

中子敏感的原子层沉积型微通道板探测器的物理与关键技术研究

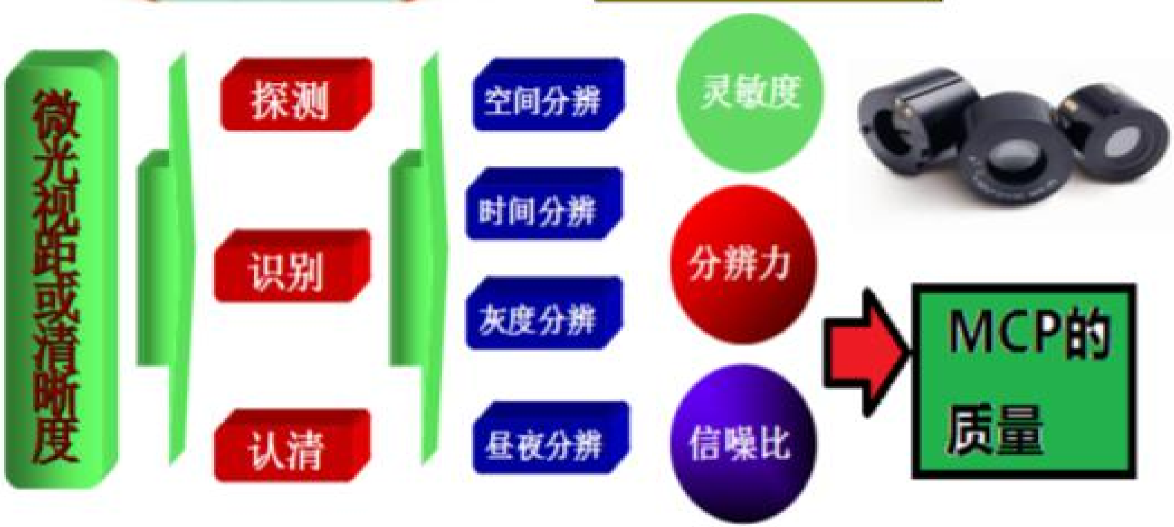
报告人：王玉漫
合作导师：孙志嘉、刘术林
报告时间:2022/06/30

目录

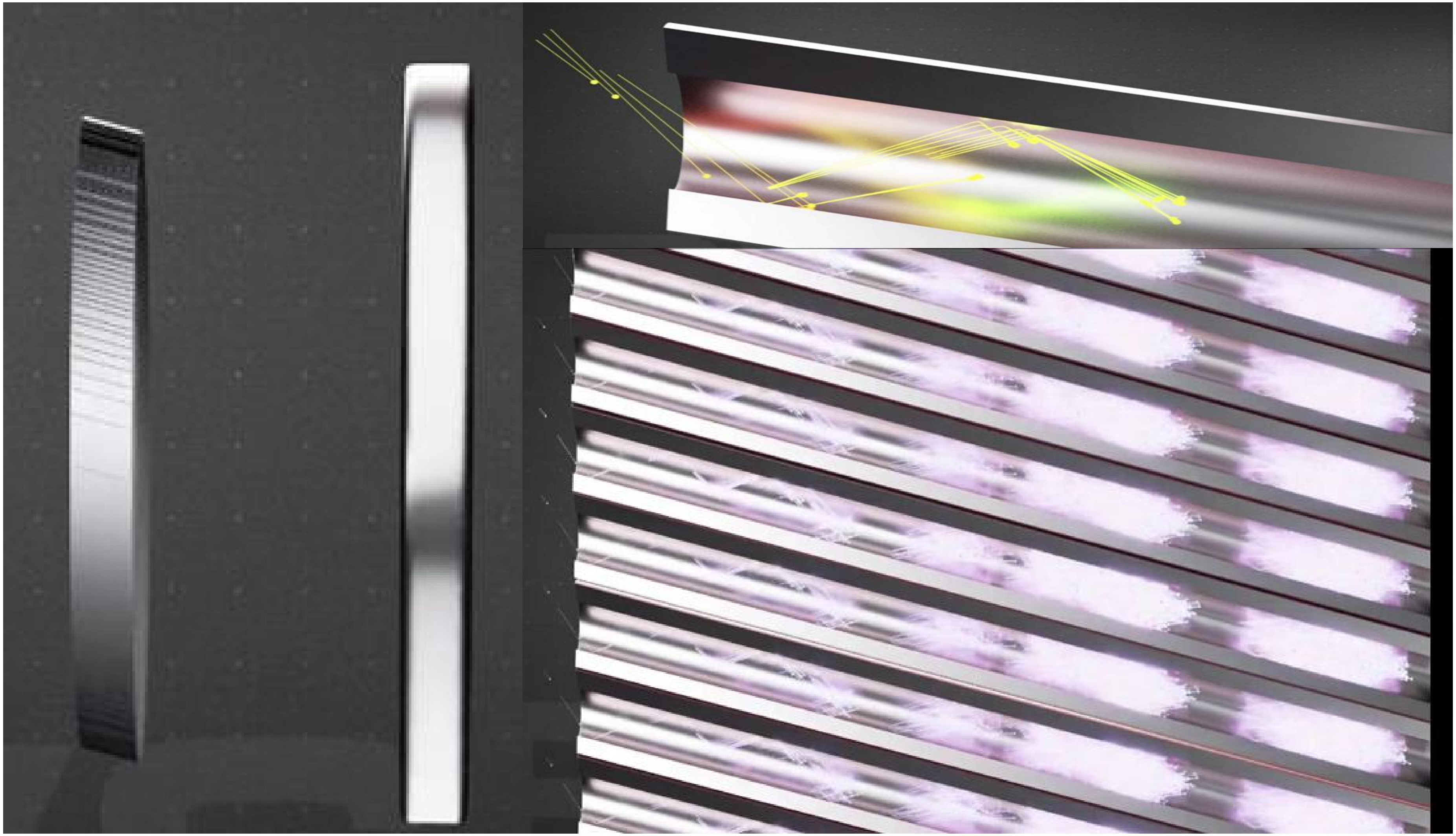
1.1微通道板



谁在更黑的夜晚看到最远？谁就获得夜战的主动权！

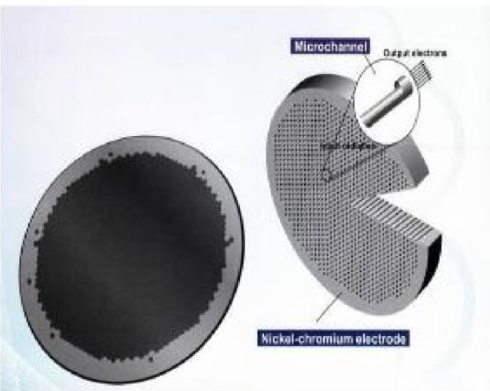


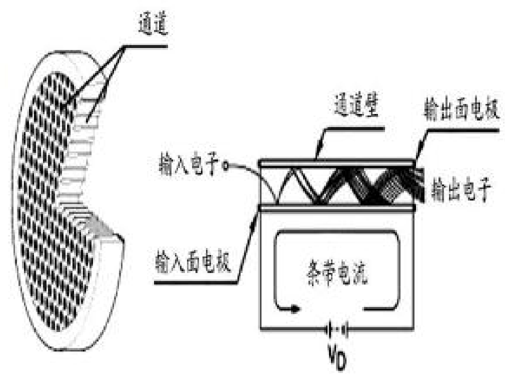
1.1微通道板

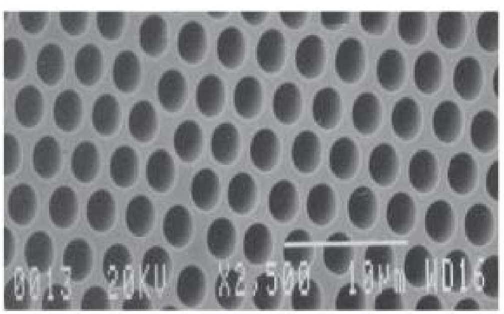


1.1微通道板

微通道板（MCP）是由数百万个通道构成的二维连续电子倍增器阵列，当在其输入和输出两端面上加上工作电压时，就能够实现二维电子的有效倍增，或者把其它事例转化成电子后的二维倍增。**MCP具有时间响应快、分辨力好、增益高、噪声低和体积小等优点，是高精密探测器的核心元件！**





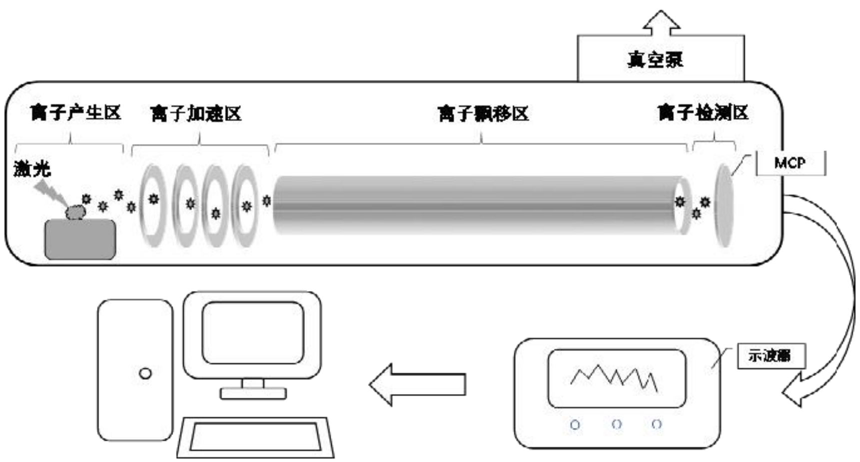


其本身对特定范围的含能光子（DUV~VUV~X射线）、荷电粒子（电子、离子）具有一定的探测效率

Types of Radiation	Energy or Wavelength	Detection Efficiency (%)
Electron	0.2 keV to 2 keV	50 to 85
	2 keV to 50 keV	10 to 60
Ion (H^+, He^+, Ar^+)	0.5 keV to 2 keV	5 to 58
	2 keV to 50 keV	60 to 85
	50 keV to 200 keV	4 to 60
UV	300 Å to 1100 Å	5 to 15
	1100 Å to 1500 Å	1 to 5
Soft X-ray	2 Å to 50 Å	5 to 15
Hard X-ray	0.12 Å to 0.2 Å	to 1
High energy particle (ρ, π)	1 GeV to 10 GeV	to 95
Neutron	2.5 MeV to 14 MeV	0.14 to 0.64

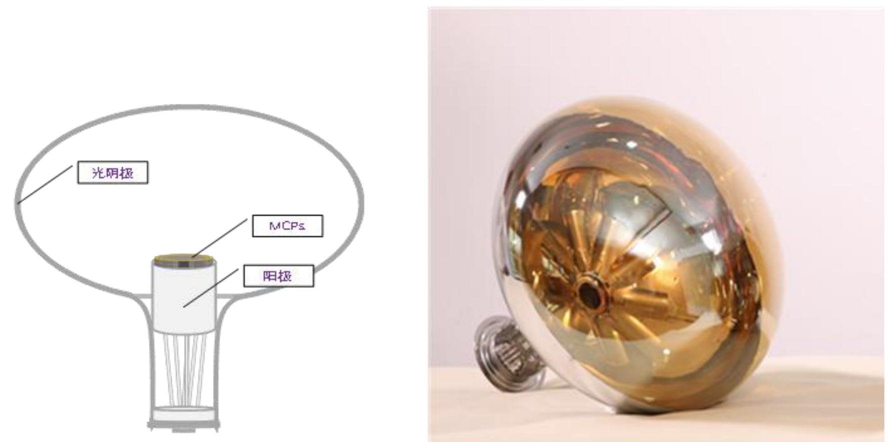
1.1微通道板

飞行时间质谱



（a）

光电倍增管



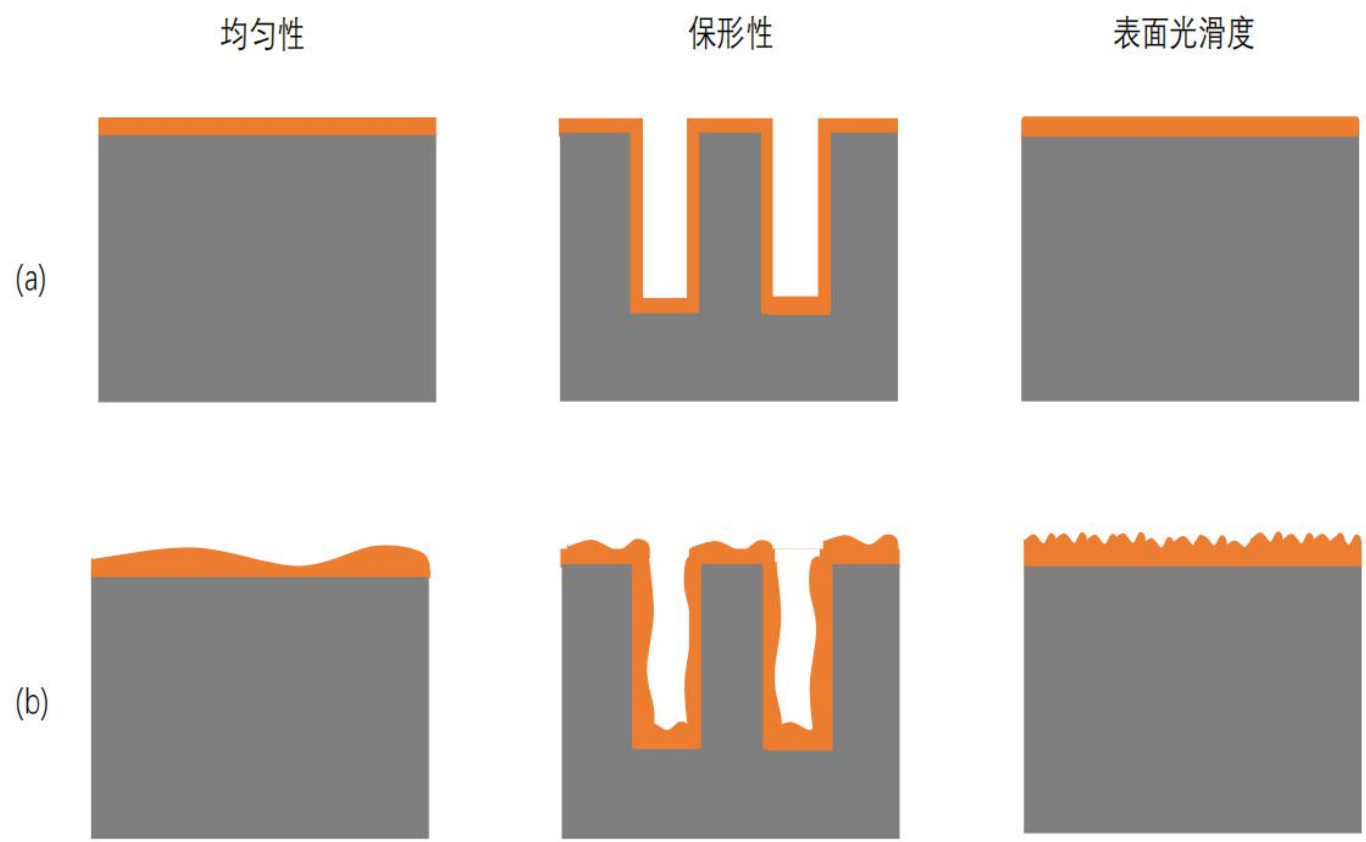
(a) (b)

（b）

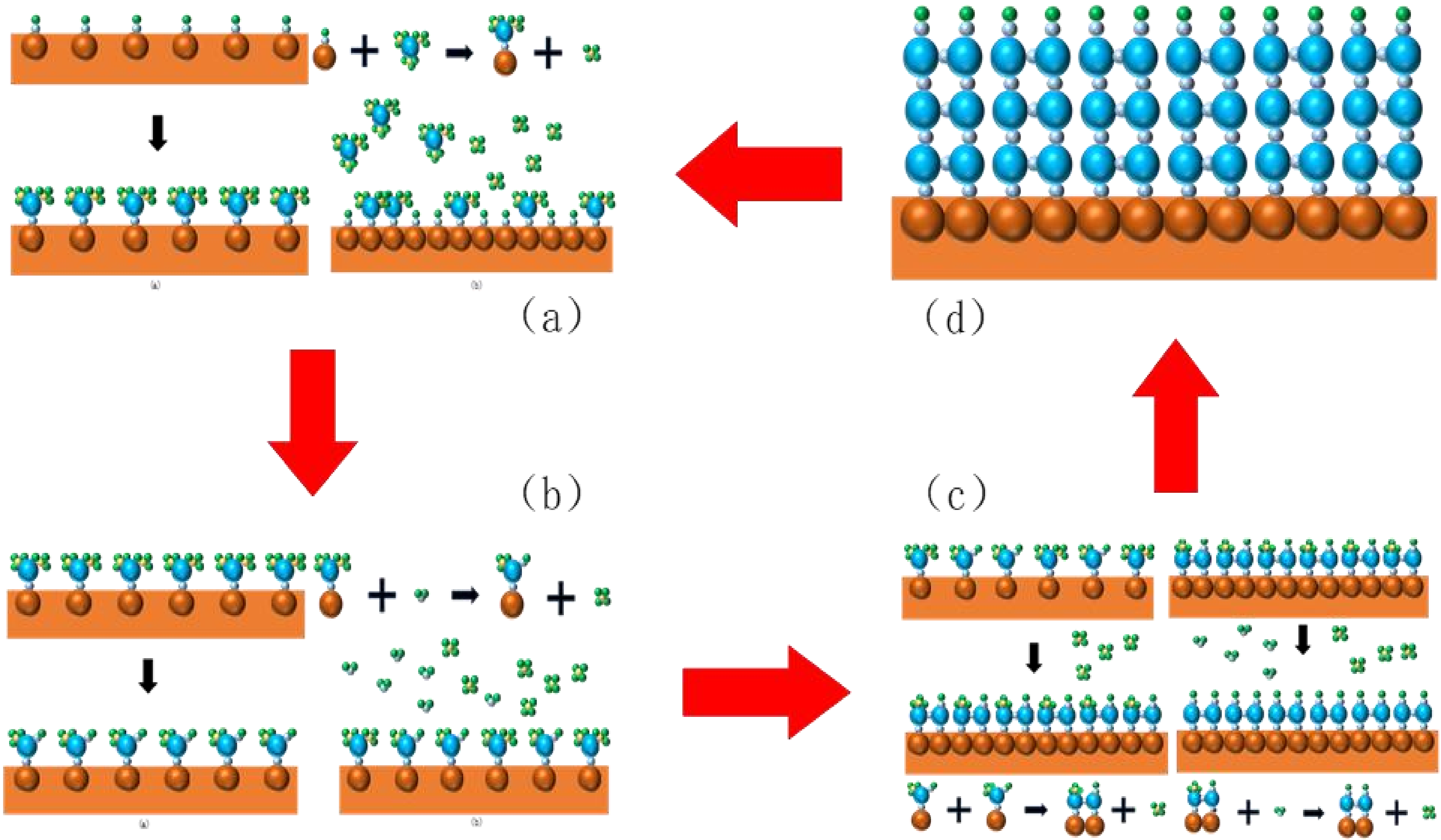
1.2原子层沉积技术

原子层沉积技术（ALD）是一种前驱体气体和反应气体速率可控的交替进入基底，在其表面发生物理和化学吸附或表面饱和反应，将物质以单原子膜的形式一层一层沉积在基底表面的技术。基于自限制反应，原子层沉积技术可以生产出连续的无针孔薄膜，具有出色的台阶覆盖率，并且可以控制原子级膜的厚度和组成。

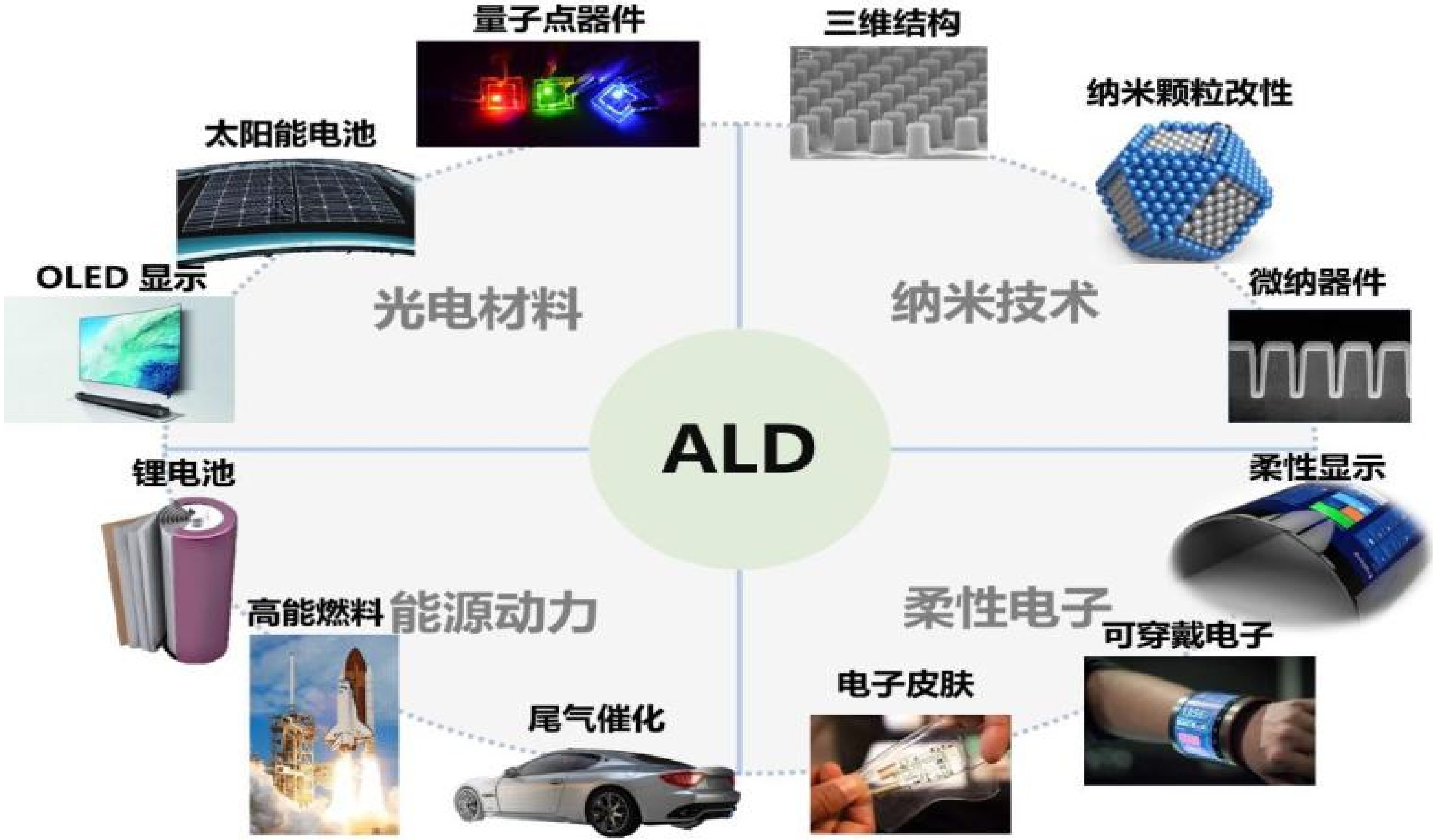
ALD技术发明人：
Tuomo Suntola
托莫·桑托拉
（2018年技术界诺贝尔奖—千禧年技术奖获得者）



1.2原子层沉积技术



1.2原子层沉积技术



量子点器件
三维结构
纳米颗粒改性
太阳能电池
OLED 显示
光电材料
纳米技术
微纳器件
ALD
锂电池
柔性显示
高能燃料
能源动力
柔性电子
可穿戴电子
电子皮肤
尾气催化

1.3原子层沉积型微通道板

MCP-PMT阵列

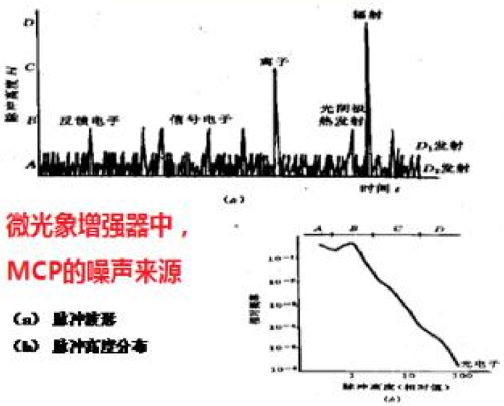
JUNO
江门中微子实验

LHASSO
高海拔宇宙线观测站

1.3原子层沉积型微通道板

国内外传统MCP存在的问题及原因 → 玻璃材料本身和腐蚀及其氢还原工艺

噪声大

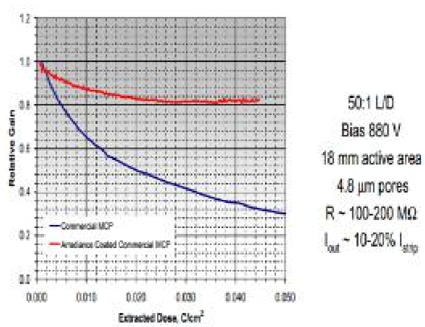


微光象增强器中，MCP的噪声来源

(a) 脉冲波形
(b) 脉冲高度分布

化学腐蚀除掉芯料
↓
通道内表面微观上出现大量起伏的沟壑
↓
二次电子发射系数存在较大的涨落
↓
噪声增加，信噪比降低

电子增益低



Applied over commercial glass MCPs

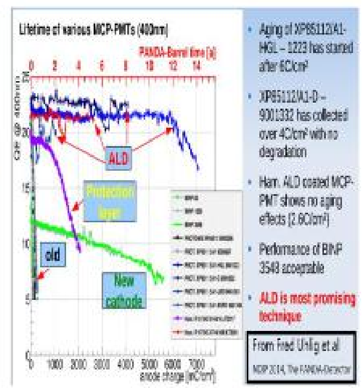
皮料玻璃本身 δ≤3

化学腐蚀 ⇄ 电子清刷除气

通道表面成分和结构的微小变化

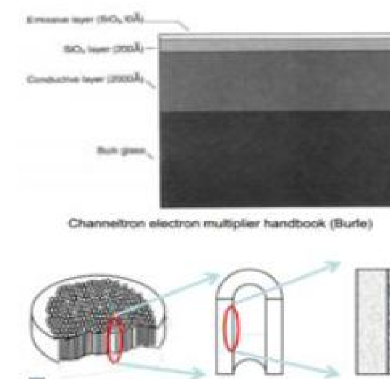
⊕
↓
MCP经彻底除气后，其增益不高

寿命短



累积拾取电荷量不高
↓
电子清刷除气又耗费了一部分电荷
↓
有效工作寿命缩短

成品率低、一致性差

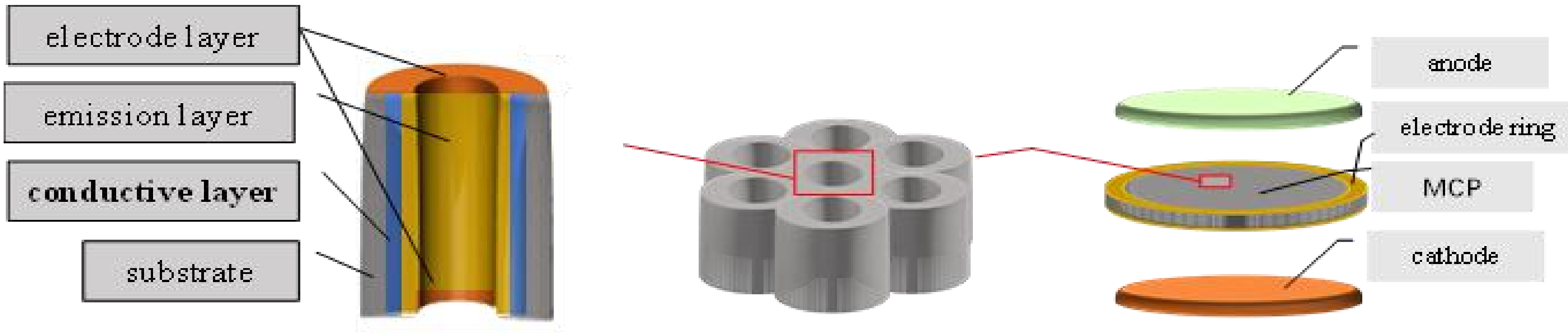


- R层、δ层难以独立控制
- 工艺本身流程长
- 部分工艺难以精确控制

成品率低，一致性不好

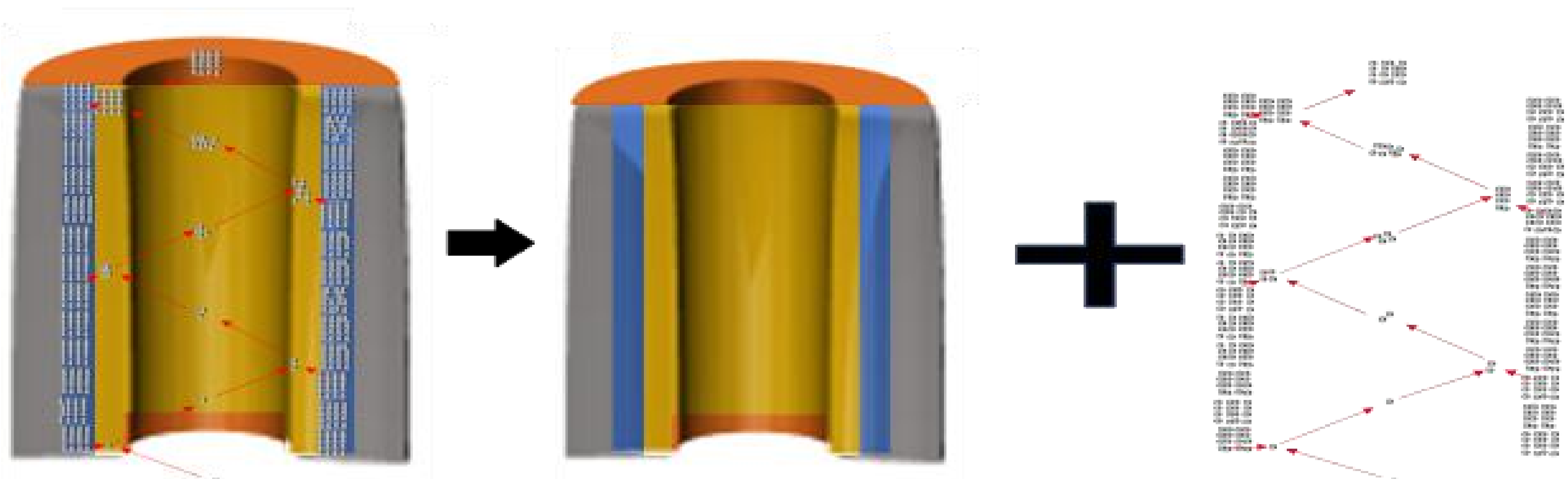
1.3原子层沉积型微通道板

(A)



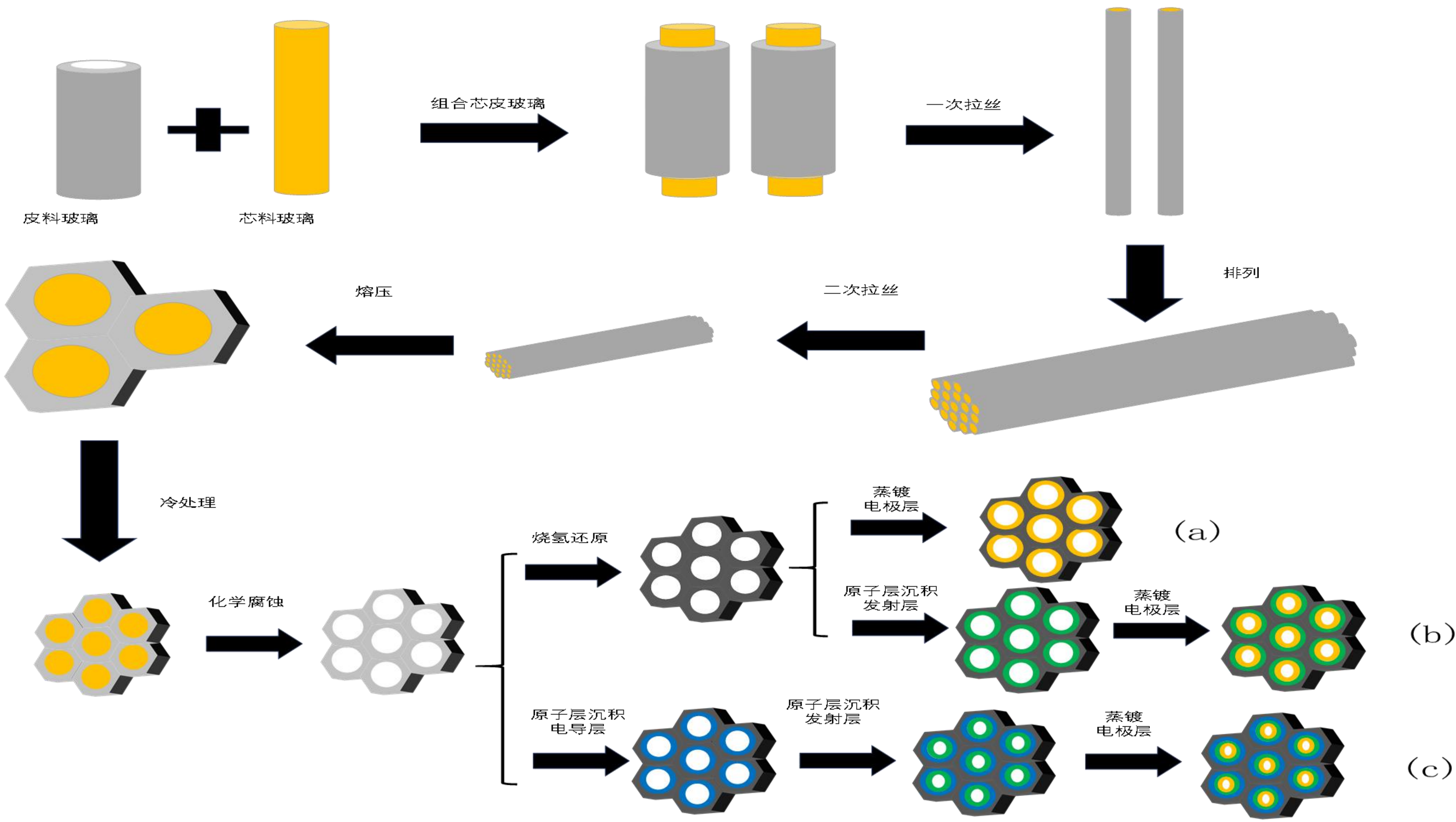
(a) (b) (c)

(B)



(a) (b) (c)

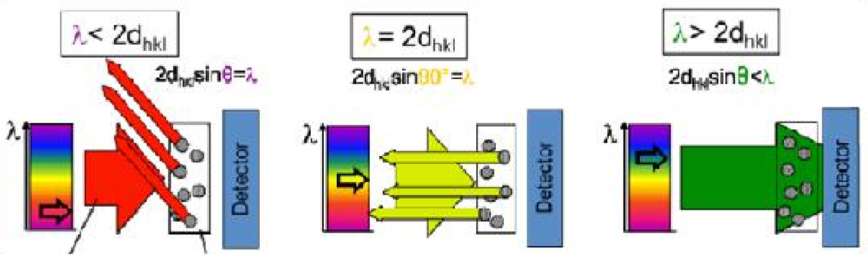
1.3原子层沉积型微通道板



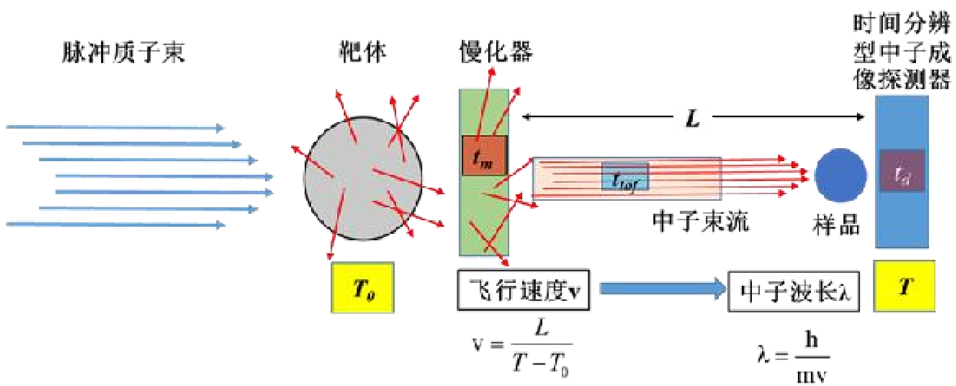
1.4中子敏感型微通道板

能量分辨中子成像

布拉格衍射： $2d\sin\theta=\lambda$



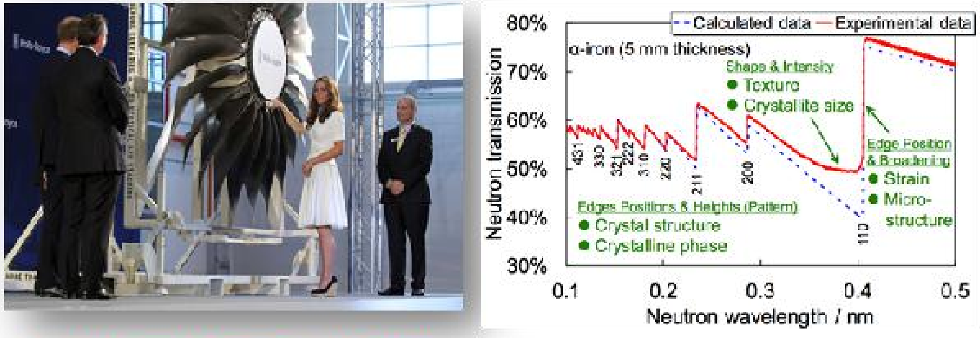
利用飞行时间的方法实现中子能量分辨



$$v=\frac{L}{T-T_0}$$

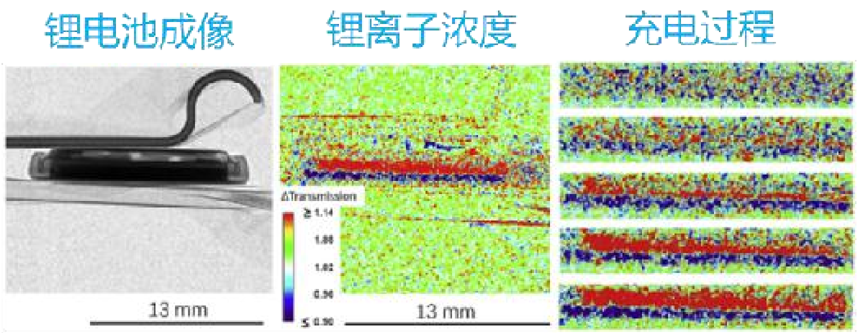
$$\lambda=\frac{h}{mv}$$

航空发动机叶片应力的无损检测



被誉为工业皇冠上的明珠

锂电池在充放电时的迁移过程

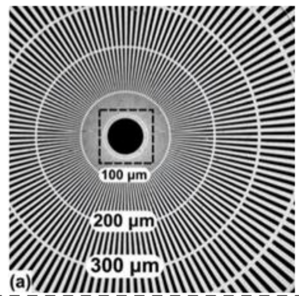


能量分辨中子成像在锂电池和工程材料研究中发挥不可替代作用

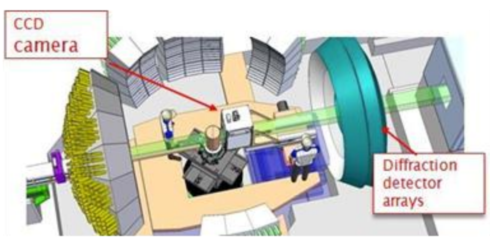
1.4中子敏感型微通道板

散裂中子源+高速/高分辨中子探测器

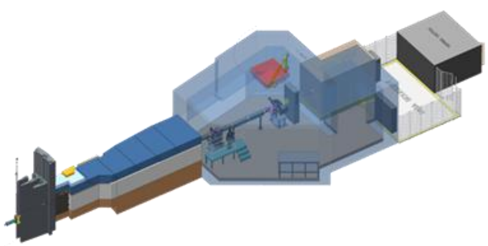
日本RADEN@J-PARC



英国IMAT@ISIS

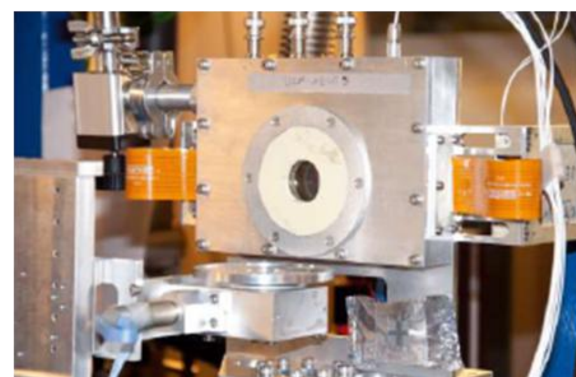


美国VENUS@SNS



高速相机(日本，对中国禁运)

nMCP探测器
(美国、英国，对中国禁运)

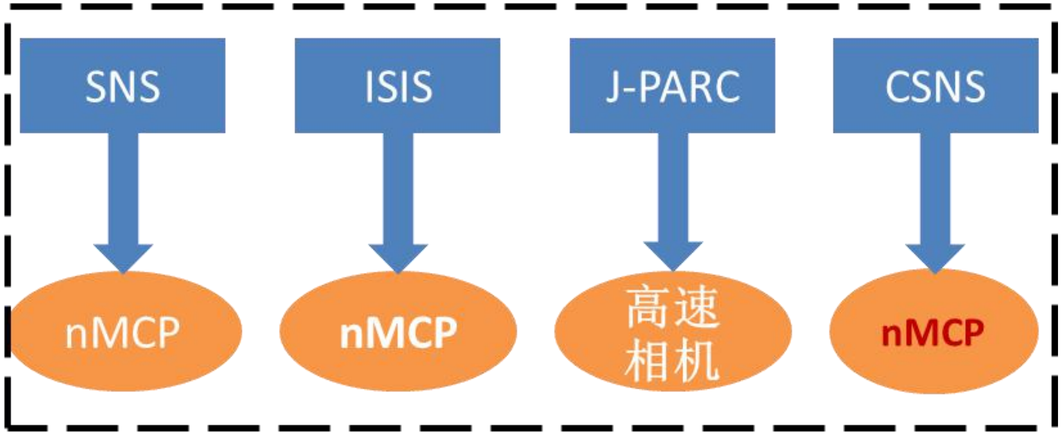


1　日本J-PARC：　闪烁屏+高速相机
2　英国 ISIS：　中子MCP(nMCP)
3　美国SNS：　中子MCP(nMCP)

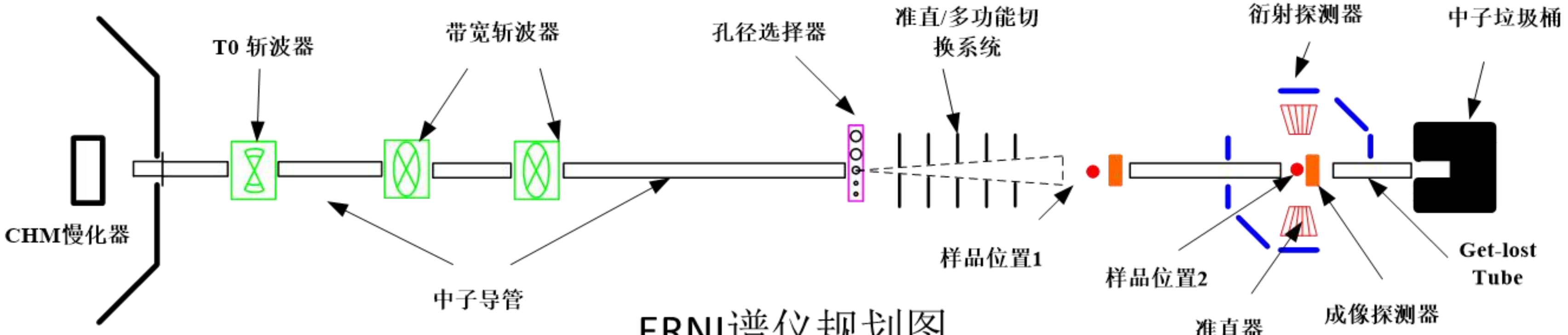
1.4中子敏感型微通道板

国际贸易形式复杂

国际前沿科学研究竞争



CSNS大装置建设的迫切需求

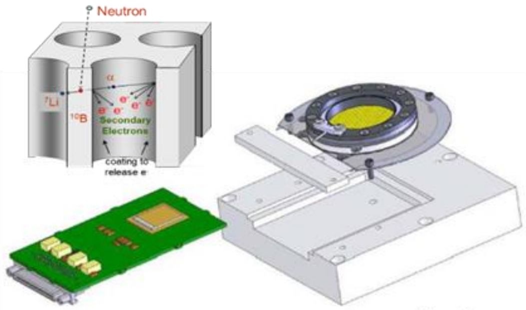


ERNI谱仪规划图

*解决高速成像探测器的"卡脖子"问题，满足大装置以及科技前沿领域的需求

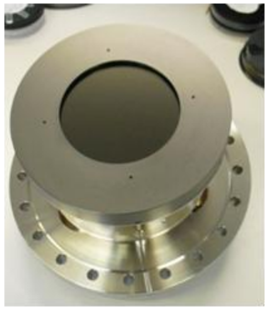
1.4中子敏感型微通道板

nMCP @U.C Berkeley

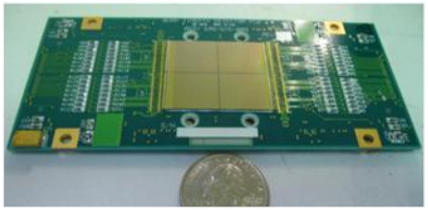


Chervon MCPs + Timepix芯片

美国NOVA 公司



高性能像素芯片
Timepix@CERN

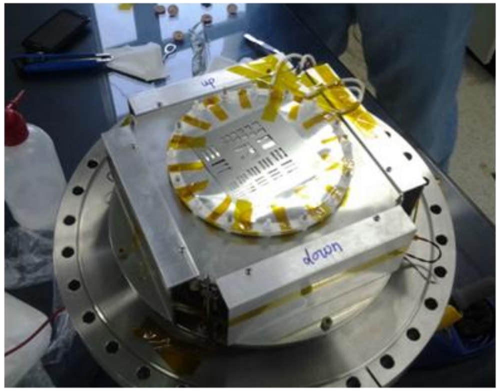


国际上唯一供货商对中国禁运

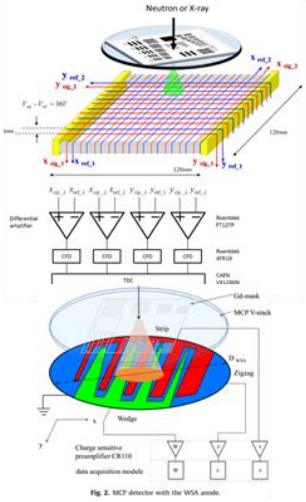
主要技术指标:

- 探测效率:~20%
- 像素尺寸: 55 μm
- 伽马灵敏度：≤1%
- 时间分辨率: 10ns

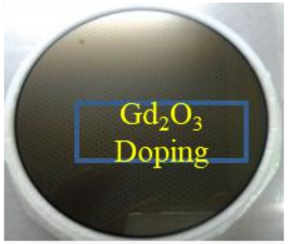
nMCP@清华大学

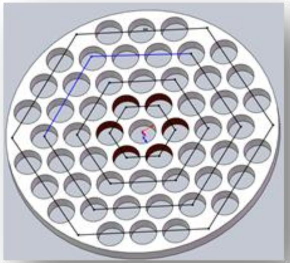


延迟线读出



北方夜视生产的掺Gd nMCP





国内研究现状:

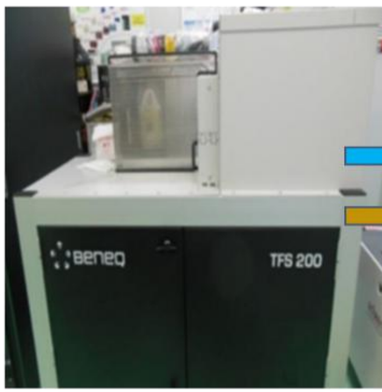
- 技术方案： 掺Gd nMCP经延迟线读出，Muli-Hit严重，Gd-nMCP 伽马甄别能力差，不适合脉冲强流中子源。
- 楔形阳极: ~130μm
- 计数率：~100kHz

需要研制掺硼-nMCP经像素型ASIC芯片实现高速读出，确保技术先进性

2研究基础

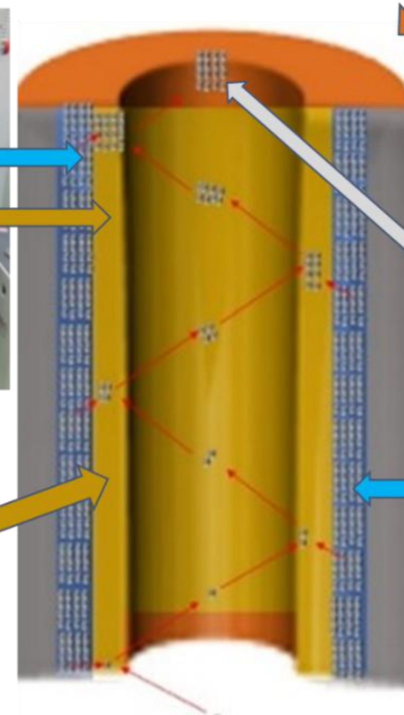
实验设备

原子层沉积设备（ALD）
电导层/发射层的制备



电极膜的制备

高真空电阻蒸发镀膜设备



发射层的SEE测试

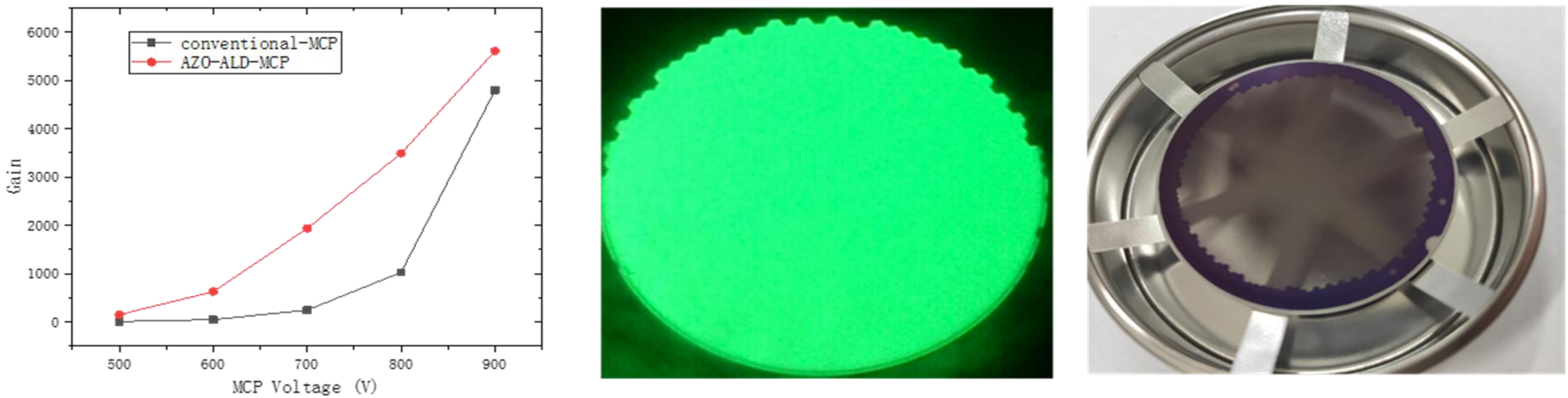
二次电子发射系数测试设备

微通道板组件处理及测试设备

电导层的电阻，ALD-MCP增益的测试

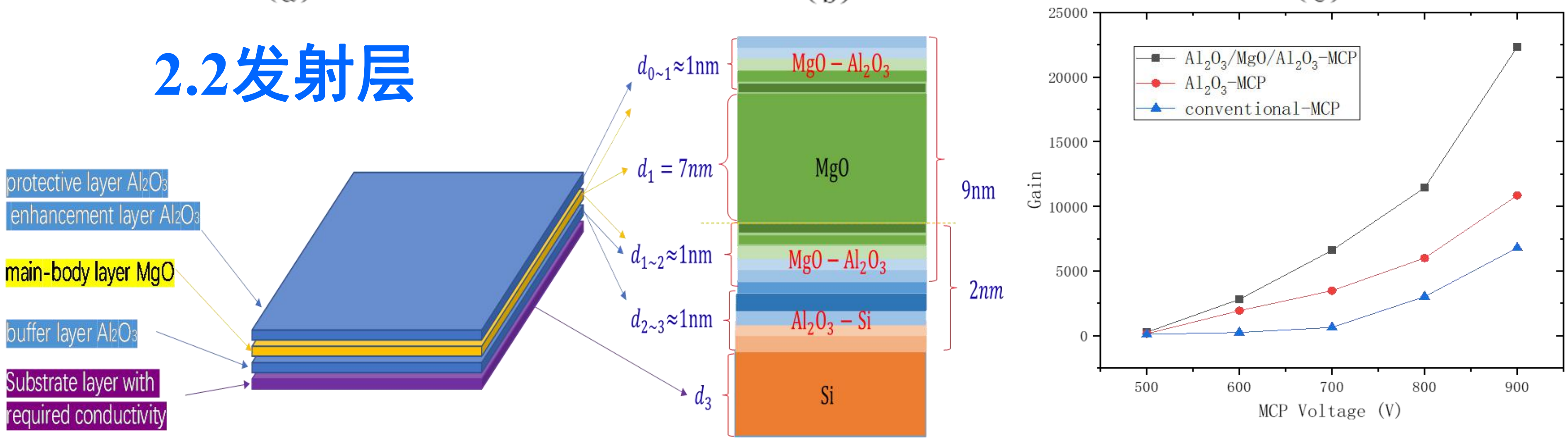
2研究基础

2.1电导层



(a) (b) (c)

2.2发射层



(a) (b) (c)

2研究基础

Wang et al. Nanoscale Res Lett (2021) 16:151
https://doi.org/10.1186/s11671-021-03606-y

Nanoscale Research Letters

NANO EXPRESS Open Access

The Design of the Emission Layer for Electron Multipliers

Yuman Wang[1,2], Baojun Yan[2,3*], Kaile Wen[2], Shulin Liu[2,3*], Ming Qi[1*], Binting Zhang[2,3], Jianyu Gu[2] and Wenjing Yao[2]

Abstract

The electron multipliers gain is closely related to the secondary electron emission coefficient (SEE) of the emission layer materials. The SEE is closely related to the thickness of the emission layer. If the emission layer is thin, the low SEE causes the low gain of electron multipliers. If the emission layer is thick, the conductive layer can't timely supplement charge to the emission layer, the electronic amplifier gain is low too. The electron multipliers usually choose Al_2O_3 and MgO film as the emission layer because of the high SEE level. MgO easy deliquescence into $Mg(OH)_2$ $Mg_2(OH)_2CO_3$ and $MgCO_3$ resulting in the lower SEE level. The SEE level of Al_2O_3 is lower than MgO, but Al_2O_3 is stable. We designed a spherical system for testing the SEE level of materials, and proposed to use low-energy secondary electrons instead of low-energy electron beam for neutralization to measuring the SEE level of Al_2O_3, MgO, MgO/Al_2O_3, Al_2O_3/MgO, and precisely control the film thickness by using atomic layer deposition. We propose to compare the SEE under the adjacent incident electrons energy to partition the SEE value of the material, and obtain four empirical formulas for the relationship between SEE and thickness. Since the main materials that cause the decrease in SEE are $Mg_2(OH)_2CO_3$ and $MgCO_3$, we use the C element atomic concentration measured by XPS to study the deliquescent depth of the material. We propose to use the concept of transition layer for SEE interpretation of multilayer materials. Through experiments and calculations, we put forward a new emission layer for electron multipliers, including 2–3 nm Al_2O_3 buffer layer, 5–9 nm MgO main-body layer, 1 nm Al_2O_3 protective layer or 0.3 nm Al_2O_3 enhancement layer. We prepared this emission layer to microchannel plate (MCP), which significantly improved the gain of MCP. We can also apply this new emission layer to channel electron multiplier and separate electron multiplier.

Keywords: Electron multipliers, Secondary electron emission, Al_2O_3, MgO, ALD

Introduction

The secondary electron emission coefficient (SEE) of a material is defined as the ratio of the emitted secondary electrons number to the incident electrons number on the material. The application field of secondary electrons is very wide, mainly divided into the field of electron multiplication, the field of material surface composition and structure analysis, and the field of suppressing micro-discharge. The field of electron multiplication includes channel electron multiplier (CEM), microchannel plate (MCP), separate electron multiplier, micro-pulse gun (MPG), dielectric window, atomic clocks, etc. [1–9]. The field of material surface composition and structure analysis includes transmission electron microscope (TEM), scanning electron microscope (SEM), auger electron spectrometer (AES), electron diffractometer, etc. [10–13]. The field of suppressing micro-discharge includes the electron cloud problem on the inner surface of the ring-accelerator, the reliability and life of high-power microwave vacuum devices in space, the dielectric

*Correspondence: yanbj@ihep.ac.cn; liusl@ihep.ac.cn; qming@nju.edu.cn
[1] School of Physics, Nanjing University, Nanjing 210093, China
[2] State Key Laboratory of Particle Detection and Electronics, Institute of High Energy Physics, Chinese Academy of Sciences, Beijing 100049, China
Full list of author information is available at the end of the article

Wang et al. Nanoscale Res Lett (2021) 16:55
https://doi.org/10.1186/s11671-021-03515-0

Nanoscale Research Letters

NANO EXPRESS Open Access

The Design of the AZO Conductive Layer on Microchannel Plate

Yuman Wang[1,2], Shulin Liu[2,1*], Baojun Yan[2], Ming Qi[1*], Kaile Wen[2], Binting Zhang[2,3], Jianyu Gu[2] and Wenjing Yao[2]

Abstract

When the resistivity of the AZO conductive layer is within the MCP resistance requirement, the interval of the Zn content is very narrow (70–73%) and difficult to control. Aiming at the characteristics of the AZO conductive layer on the microchannel plate, an algorithm is designed to adjust the ratio of the conductive material ZnO and the high resistance material Al2O3. We put forward the concept of the working resistance of the MCP (i.e., the resistance during the electron avalanche in the microchannel). The working resistance of AZO-ALD-MCP (Al2O3/ZnO atomic layer deposition microchannel plate) was measured for the first time by the MCP resistance test system. In comparison with the conventional MCP, we found that the resistance of AZO-ALD-MCP in working state and non-working state is very different, and as the voltage increases, the working resistance significantly decreases. Therefore, we proposed a set of analytical methods for the conductive layer. We also proposed to adjust the ratio of the conductive material of the ALD-MCP conductive layer to the high-resistance material under the working resistance condition, and successfully prepared high-gain AZO-ALD-MCP. This design opens the way for finding better materials for the conductive layer of ALD MCP to improve the performance of MCP.

Keywords: ALD-MCP, The AZO conductive layer, The working resistance, ZnO, Al_2O_3

Introduction

Microchannel plate (MCP) is an electron multiplier composed of two-dimensional pore arrays by thin glass plate form integration, length of 0.5–5 mm, a 4–40 μm diameter and with a bias angle usually 5°–13° to the normal of the plate surface; the open area ratio of the plate is up to 60%, and the high length-to-diameter ratio in each pore is about 20:1 to 100:1 [1].

As shown in Fig. 1, incident electrons entering the microchannel collide with the walls causing secondary electrons to be generated on the surface of the microchannel walls. Multi-collisions with the microchannel walls will lead to an increasing number of secondary electrons, resulting in an electron avalanche inside the microchannel and the emission of a cloud of electrons from the output of the microchannel. The secondary electron electrons will be further accelerated along the microchannel by a bias voltage. The MCP gain is 10^3–10^4 at a working voltage 700–900 V [2–9].

Each microchannel is as a detector and an electron multiplier. By having millions of microchannels working independently, MCP has the characteristics of high spatial resolution, high timing resolution and wide range of gain used to identify the photons, electrons, neutrons and ions. MCP can integrate into various kinds of instruments, including photoelectric detector, photomultiplier tubes (PMTs), ultraviolet spectrometer, cathode ray tube, scanning electron microscope, field emission displays, residual gas analyzer, medical imaging, time-of-flight mass spectrometry, night-vision goggles, etc. [1, 4, 7–9]. The hydrogen firing of the traditional process makes the

*Correspondence: liusl@ihep.ac.cn; qming@nju.edu.cn
[1] School of Physics, Nanjing University, Nanjing 210093, China
[2] State Key Laboratory of Particle Detection and Electronics, Institute of High Energy Physics, Chinese Academy of Sciences, Beijing 100049, China
Full list of author information is available at the end of the article

2研究基础

2研究基础

ALD-MCP功能薄膜

电导层

1. WYM算法
2. 电导层厚度公式

1. 宽范围的MCP工作电阻测试系统。
2. MCP增益测试平台

通过工作电阻进行判断MCP电导层是否合适

定义的κ_R, L_R, %ZnO, r等进行电阻数据的分析。

制备方案

测试仪器与方法

评价方法

分析方法

发射层

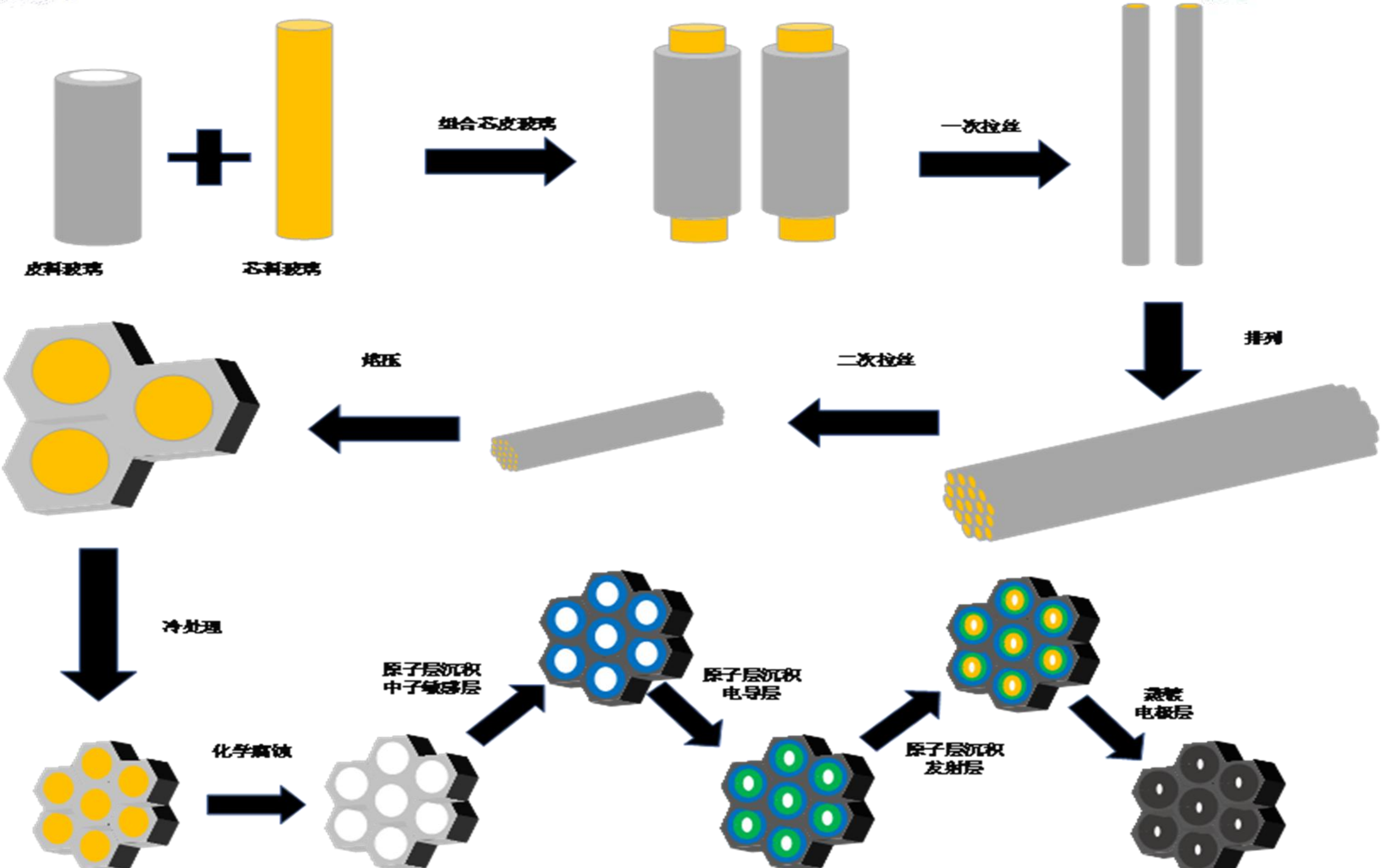
1. 过渡层思想
2. 三明治结构

1. 球形SEE测试系统
2. 低能SEE中和方法

椭偏仪测试材料厚度及对材料进行一次电子能量分区，进而在中能区获得最优SEE厚度关系式

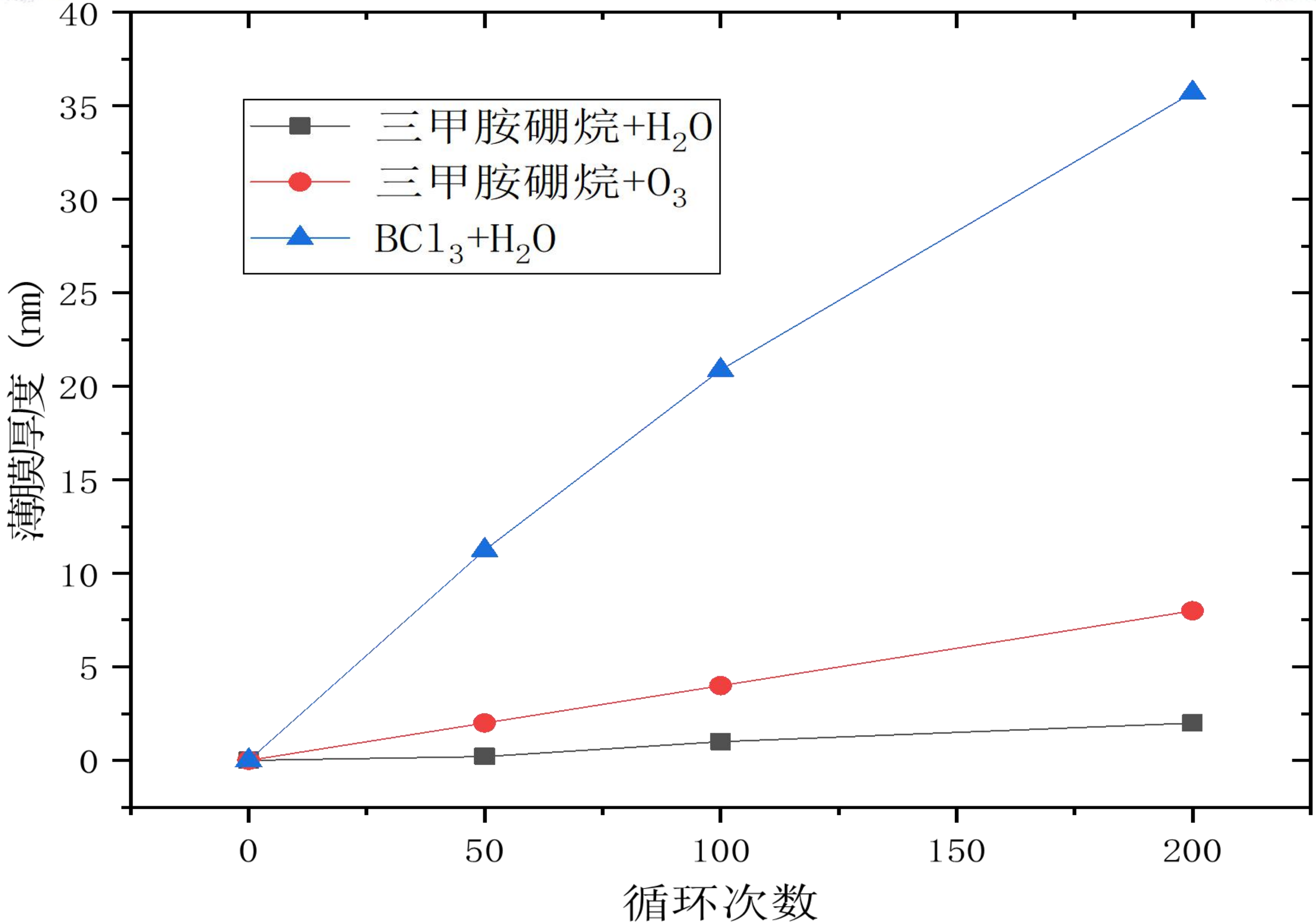
XPS与刻蚀交替实验获得元素内部分布信息

3研究成果



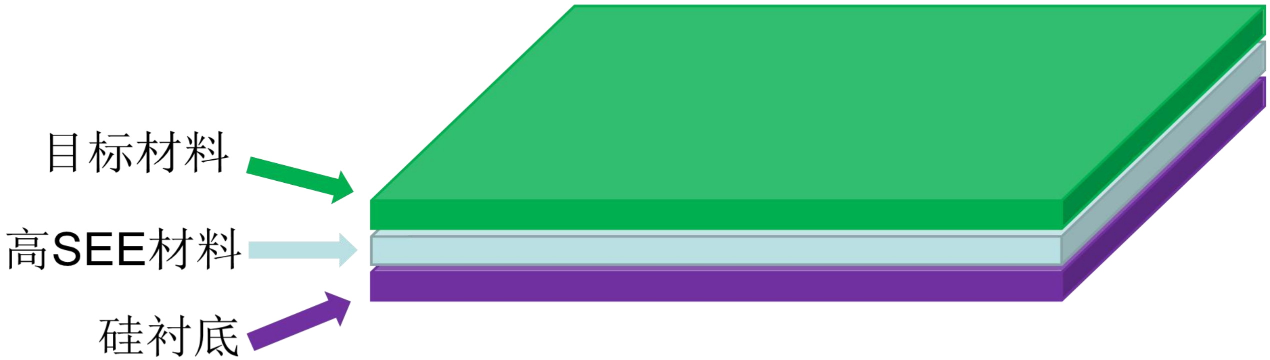
组合芯皮玻璃
一次拉丝
皮料玻璃
芯料玻璃
排列
二次拉丝
熔压
冷处理
化学腐蚀
原子层沉积
中子敏感层
原子层沉积
电导层
原子层沉积
发射层
蒸镀
电极层

3研究成果

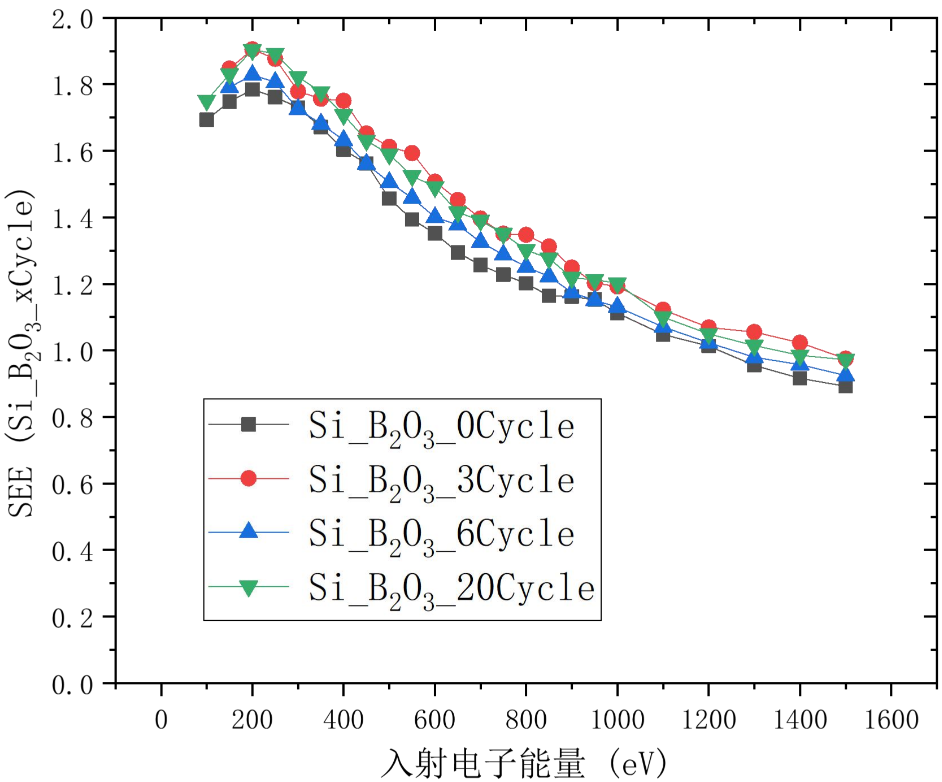


40
35
30
25
20
15
10
5
0
薄膜厚度（nm）
三甲胺硼烷+H2O
三甲胺硼烷+O3
BCl3+H2O
0
50
100
150
200
循环次数

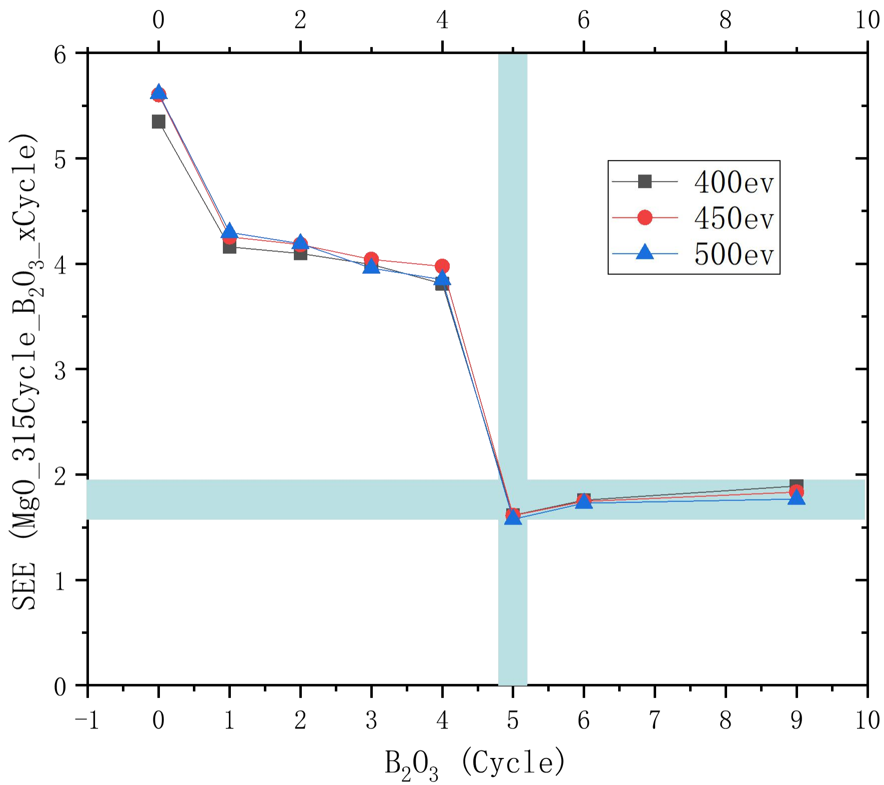
3研究成果



目标材料
高SEE材料
硅衬底



SEE (Si_B2O3_xCycle)
入射电子能量 (eV)
Si_B2O3_0Cycle
Si_B2O3_3Cycle
Si_B2O3_6Cycle
Si_B2O3_20Cycle



SEE (MgO_315Cycle_B2O3_xCycle)
B2O3 (Cycle)
400ev
450ev
500ev

3研究成果

PCNP220142

国 家 知 识 产 权 局

523000
东莞市东城区鸿福东路二号东莞农商银行大厦副楼十一楼
广东众达律师事务所 张雪华(13556708787)

发文日：

2022年05月14日

申请号或专利号：202210521586.0　　发文序号：2022051400179460

专 利 申 请 受 理 通 知 书

根据专利法第28条及其实施细则第38条、第39条的规定，申请人提出的专利申请已由国家知识产权局受理。现将确定的申请号、申请日、申请人和发明创造名称通知如下：

申请号：202210521586.0
申请日：2022年05月13日
申请人：散裂中子源科学中心,中国科学院高能物理研究所
发明创造名称：用于中子探测及成像的原子层沉积型微通道板及制作方法

经核实，国家知识产权局确认收到文件如下：
说明书附图 每份页数:6页 文件份数:1份
说明书摘要 每份页数:1页 文件份数:1份
专利代理委托书 每份页数:2页 文件份数:1份
发明专利请求书 每份页数:6页 文件份数:1份
说明书 每份页数:8页 文件份数:1份
实质审查请求书 每份页数:1页 文件份数:1份
权利要求书 每份页数:2页 文件份数:1份 权利要求项数： 9项

提示：
1. 申请人收到专利申请受理通知书之后，认为其记载的内容与申请人所提交的相应内容不一致时，可以向国家知识产权局请求更正。
2. 申请人收到专利申请受理通知书之后，再向国家知识产权局办理各种手续时，均应当准确、清晰地写明申请号。
3. 国家知识产权局收到向外国申请专利保密审查请求书后，依据专利法实施细则第9条予以审查。

审 查 员：自动受理　　审查部门：专利局初审及流程管理部

200101 2019.11 纸件申请，回函请寄：100088 北京市海淀区蓟门桥西土城路6号 国家知识产权局受理处收
电子申请，应当通过电子专利申请系统以电子文件形式提交相关文件。除另有规定外，以纸件等其他形式提交的文件视为未提交。

PCNP220134

国 家 知 识 产 权 局

523000
东莞市东城区鸿福东路二号东莞农商银行大厦副楼十一楼
广东众达律师事务所 张雪华(13556708787)

发文日：

2022年05月01日

申请号或专利号：202210473415.5　　发文序号：2022050100984570

专 利 申 请 受 理 通 知 书

根据专利法第28条及其实施细则第38条、第39条的规定，申请人提出的专利申请已由国家知识产权局受理。现将确定的申请号、申请日、申请人和发明创造名称通知如下：

申请号：202210473415.5
申请日：2022年04月29日
申请人：散裂中子源科学中心,中国科学院高能物理研究所
发明创造名称：精确测量低二次电子发射系数材料最薄贡献厚度的方法

经核实，国家知识产权局确认收到文件如下：
专利代理委托书 每份页数:2页 文件份数:1份
说明书附图 每份页数:6页 文件份数:1份
实质审查请求书 每份页数:1页 文件份数:1份
说明书 每份页数:6页 文件份数:1份
权利要求书 每份页数:2页 文件份数:1份 权利要求项数： 7项
说明书摘要 每份页数:1页 文件份数:1份
发明专利请求书 每份页数:6页 文件份数:1份

提示：
1. 申请人收到专利申请受理通知书之后，认为其记载的内容与申请人所提交的相应内容不一致时，可以向国家知识产权局请求更正。
2. 申请人收到专利申请受理通知书之后，再向国家知识产权局办理各种手续时，均应当准确、清晰地写明申请号。
3. 国家知识产权局收到向外国申请专利保密审查请求书后，依据专利法实施细则第9条予以审查。

审 查 员：自动受理　　审查部门：专利局初审及流程管理部

200101 2019.11 纸件申请，回函请寄：100088 北京市海淀区蓟门桥西土城路6号 国家知识产权局受理处收
电子申请，应当通过电子专利申请系统以电子文件形式提交相关文件。除另有规定外，以纸件等其他形式提交的文件视为未提交。

感谢各位老师、同学，
报告结束！