

# 中子深度剖面分析应用研究进展

报告人：肖才锦

中国原子能科学研究院

2025年7月



## 目 录

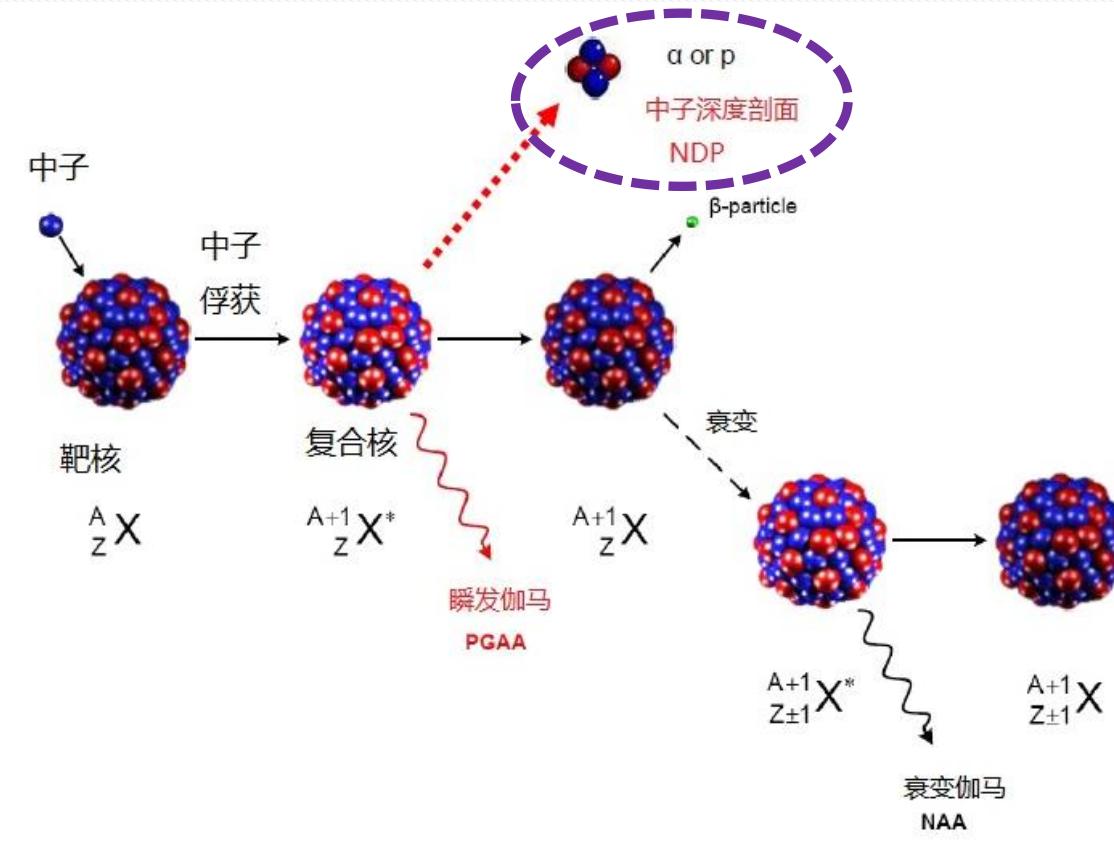
### 01 中子活化分析简介

### 02 中子深度剖面研究进展

# 一. 中子活化分析简介

## 中子活化分析

中子活化分析通常是利用反应堆中子轰击待分析的样品，通过核反应使其中多种元素（每种元素的至少一种同位素）生成放射性核素，根据这些核素衰变中发射特征射线的性质和强度，对相应元素进行定性、定量分析的方法。



### 中子活化分析原理图

包含INAA、PGAA、NDP等技术。

# 一. 中子活化分析简介

## 中子活化分析一直被当做元素分析的基准方法 (primary method)

Tian, W. Z. Metrological role of neutron activation analysis. IA. Inherent characteristics of relative INAA as *a primary ratio method of measurement*. Accred. Qual. Assur. 2001, 6, 488–492.

Robert R. Greenberg , Neutron activation analysis: *A primary method of measurement*, Spectrochimica Acta Part B 66 (2011) 193–241.

### 中子活化分析的独特优势

非破坏

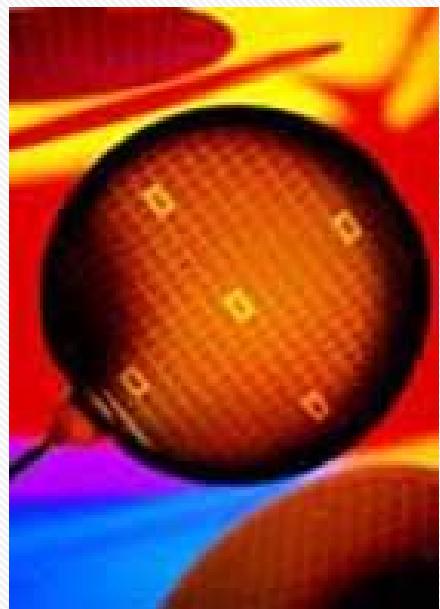
高精度

多元素

# 一. 中子活化分析简介

## 中子活化分析应用

深空探测、地球科学、环境科学、材料科学、考古学、标准物质、半导体等。



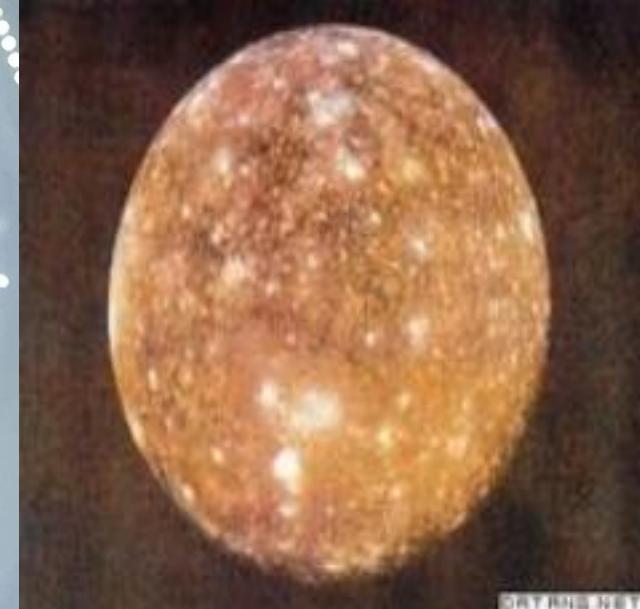
高纯材料



考古



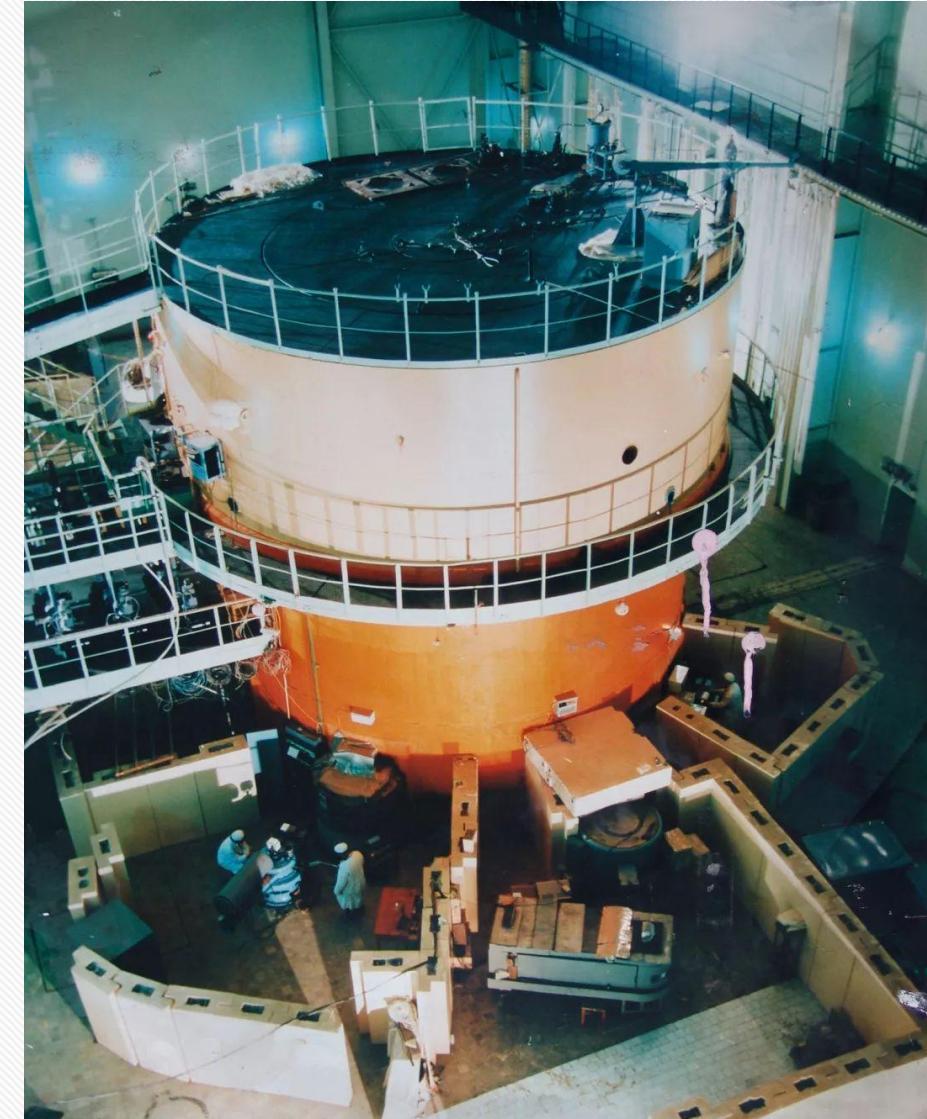
月壤



宇宙尘

# 一. 中子活化分析简介

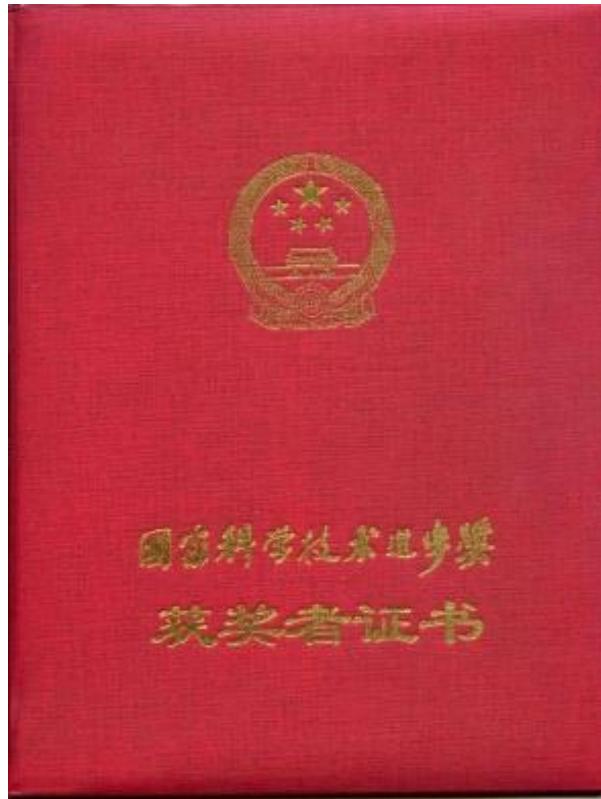
- 首次活化分析实验：1960年NAA分析了高纯 $\text{UO}_2$ 中的稀土元素。
- 1971年，原子能院活化分析实验室成立。



重水反应堆

# 一. 中子活化分析简介

□ 获得过**4项国家科技进步奖**, 十余项省部级奖。



- 1) 反应堆中子活化分析的应用;
- 2) 用缓发中子计数法测铀;
- 3) 堆中子活化分析中裂变干扰的参量法校正
- 4) 反应堆中子活化分析全面参量化研究

## NAA在标准物质定值中的应用

- 为国内众多岩石、土壤、生物、农业等不同类型的国家一级标准物质定值；
- 美国NIST、日本、欧盟，IAEA、FNCA等国际组织机构的标准物质定值；

肖才锦 同志：

参加定值的“化肥成分分析系列标准物质（9种）”已获批国家一级标准物质，编号为 GBW06132~GBW06140。

特此证明！

天津农学院

2021年11月18日

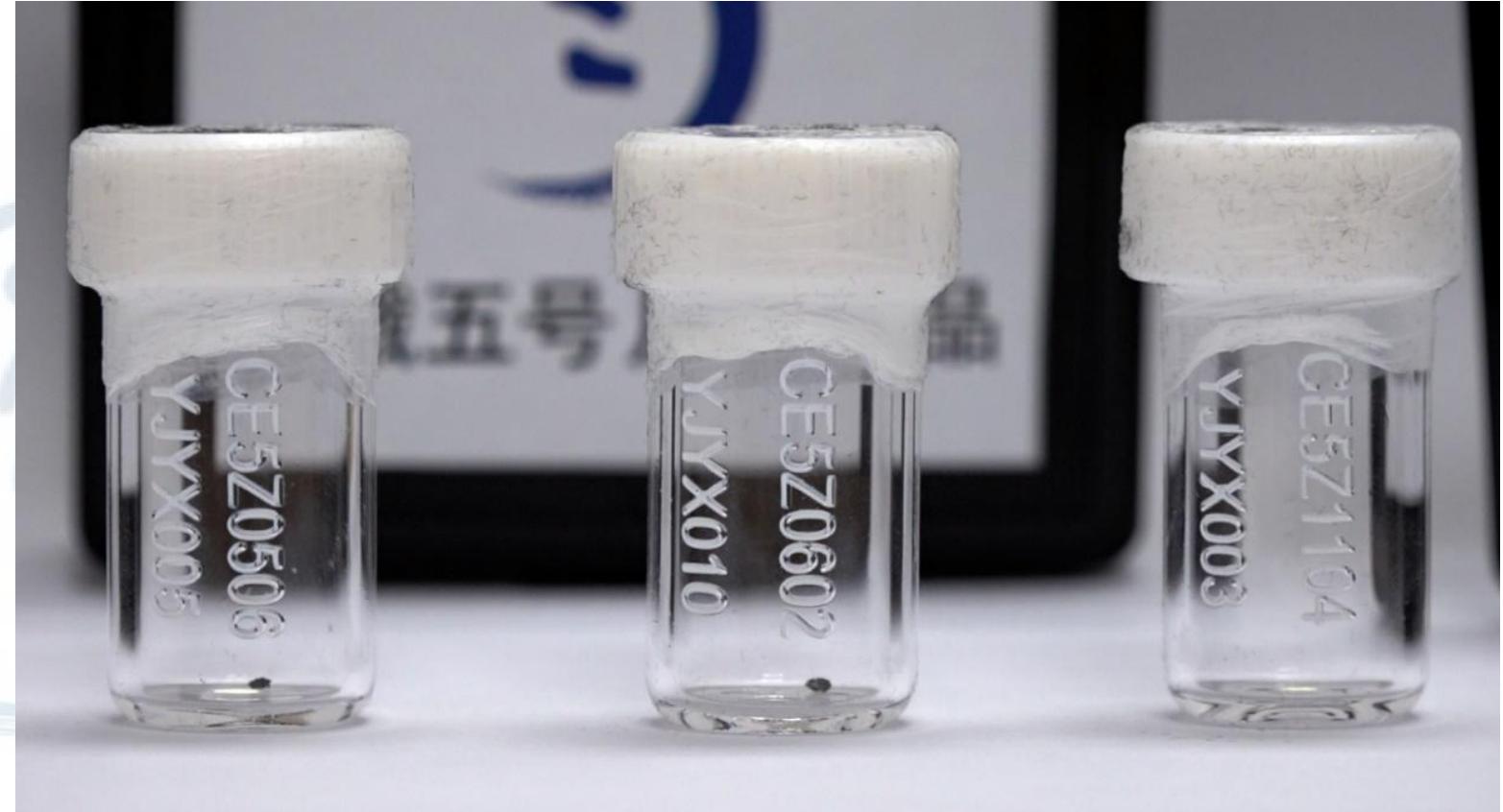
Collection and preparation of SRM 1577c were performed by M.P. Cronise and C.N. Fales of the NIST Measurement Services Division, and E.A. Mackey, R.O. Spatz, and R. Zeisler of the NIST Analytical Chemistry Division. The bovine liver material was collected at Texas A&M University (College Station, TX) with the assistance of W.D. James of the Center for Chemical Characterization and Analysis, and R.R. Riley of the E.M. (Manny) Rosenthal Meat Science and Technology Center.

The technical measurements were performed by S.J. Christopher, R.R. Greenberg, S.E. Long, E.A. Mackey, K.E. Murphy, B.J. Porter, S.A. Rabb, R.O. Spatz, B.E. Tomlin, L.J. Wood, L.L. Yu, and R. Zeisler of the NIST Analytical Chemistry Division, and the following collaborating laboratories and analysts: China Institute of Atomic Energy, Beijing, China: C. Xiao, B. Ni, W. Tian; Massachusetts Institute of Technology, Nuclear Reactor Laboratory, Cambridge, MA: J. Che, L.-W. Hu; Nuclear Physics Institute ASCR, Řež, Czech Republic: J. Kučera; Texas A&M University, College Station, TX, Department of Chemistry: W.D. James and College of Veterinary Medicine: R.J. Taylor; University of São Paulo, Institute of Chemistry, São Paulo, Brazil: C.S. Nomura, P.V. Oliveira; and USDA Beltsville Agricultural Research Center, Human Nutrition Research Center, Beltsville, MD: J. Harnly, E. Greene.

美国NIST SRM1577C证书

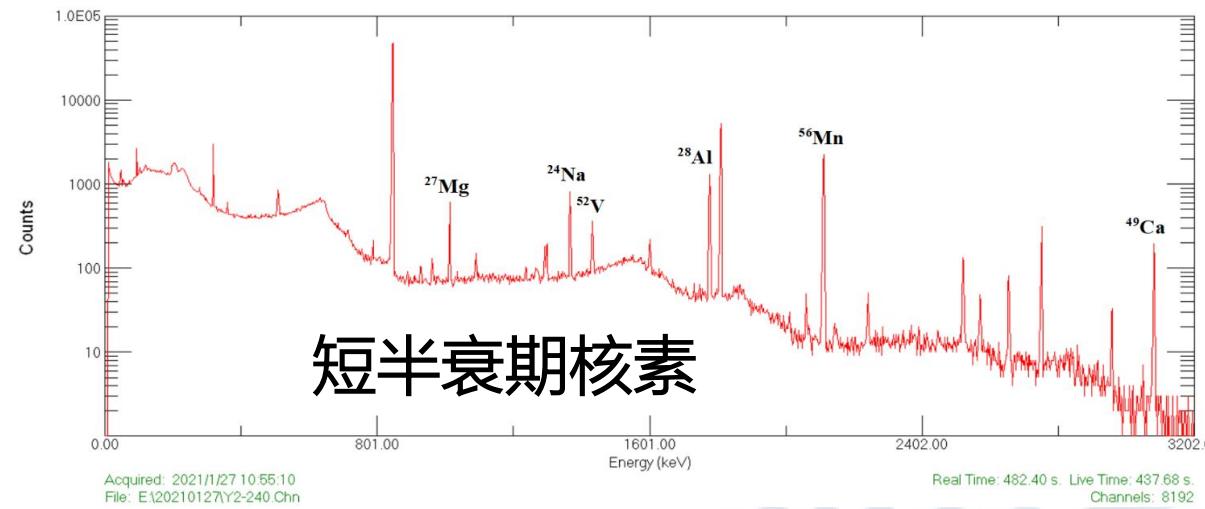
# 一. 中子活化分析简介

## NAA在月壤分析中的应用

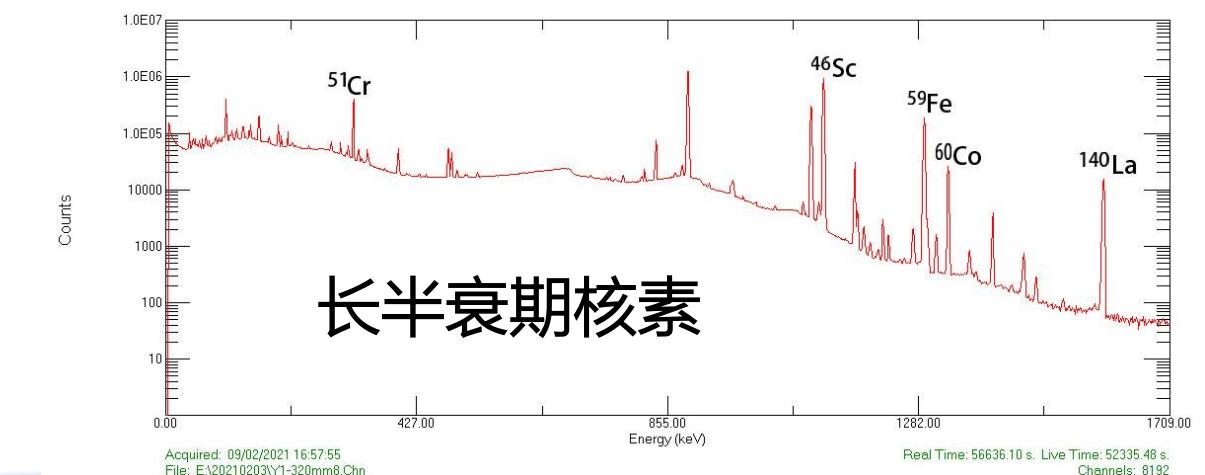


## 月壤中子活化分析研究

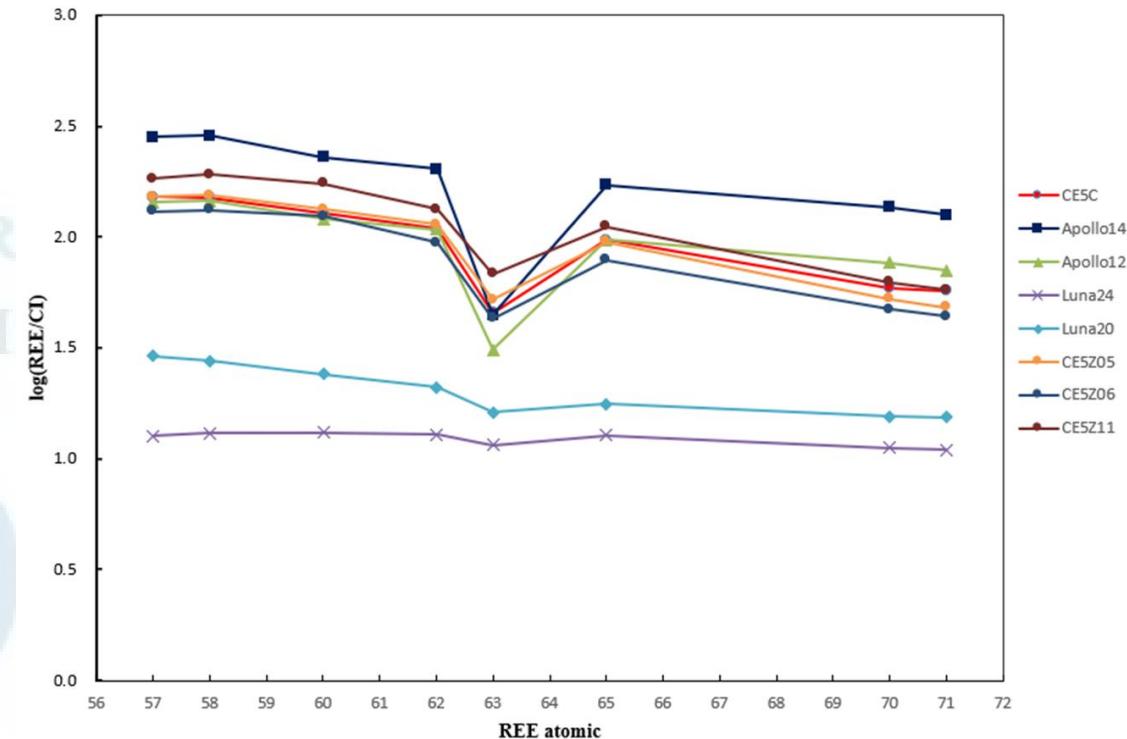
## 嫦娥五号中子活化分析实验



短半衰期核素



长半衰期核素



嫦娥五号月壤稀土模式

## 一. 中子活化分析简介

## 分析结果

- 测定40多种元素含量，有很多新的发现；
  - Au、Ir等铂族元素只有NAA测试数据；
  - 整个分析过程样品无损失，原样返还；

1 <b>H</b> hydrogen																		2 <b>He</b> helium
3 <b>Li</b> lithium	4 <b>Be</b> beryllium																	
11 <b>Na</b> sodium	12 <b>Mg</b> magnesium																	
19 <b>K</b> potassium	20 <b>Ca</b> calcium	21 <b>Sc</b> scandium	22 <b>Ti</b> titanium	23 <b>V</b> vanadium	24 <b>Cr</b> chromium	25 <b>Mn</b> manganese	26 <b>Fe</b> iron	27 <b>Co</b> cobalt	28 <b>Ni</b> nickel	29 <b>Cu</b> copper	30 <b>Zn</b> zinc	31 <b>Ga</b> gallium	32 <b>Ge</b> germanium	33 <b>As</b> arsenic	34 <b>Se</b> selenium	35 <b>Br</b> bromine	36 <b>Kr</b> krypton	
37 <b>Rb</b> rubidium	38 <b>Sr</b> strontium	39 <b>Y</b> yttrium	40 <b>Zr</b> zirconium	41 <b>Nb</b> niobium	42 <b>Mo</b> molybdenum	43 <b>Tc</b> technetium	44 <b>Ru</b> ruthenium	45 <b>Rh</b> rhodium	46 <b>Pd</b> palladium	47 <b>Ag</b> silver	48 <b>Cd</b> cadmium	49 <b>In</b> indium	50 <b>Sn</b> tin	51 <b>Sb</b> antimony	52 <b>Te</b> tellurium	53 <b>I</b> iodine	54 <b>Xe</b> xenon	
55 <b>Cs</b> caesium	56 <b>Ba</b> barium		72 <b>Hf</b> hafnium	73 <b>Ta</b> tantalum	74 <b>W</b> tungsten	74 <b>Re</b> rhenium	76 <b>Os</b> osmium	77 <b>Ir</b> iridium	78 <b>Pt</b> platinum	79 <b>Au</b> gold	80 <b>Hg</b> mercury	81 <b>Tl</b> thallium	82 <b>Pb</b> lead	83 <b>Bi</b> bismuth	84 <b>Po</b> polonium	85 <b>At</b> astatine	86 <b>Rn</b> radon	
87 <b>Fr</b> francium	88 <b>Ra</b> radium		57 <b>La</b> lanthanum	58 <b>Ce</b> cerium	59 <b>Pr</b> praseodymium	60 <b>Nd</b> neodymium	61 <b>Pm</b> promethium	62 <b>Sm</b> samarium	63 <b>Eu</b> europium	64 <b>Gd</b> gadolinium	65 <b>Tb</b> terbium	66 <b>Dy</b> dysprosium	67 <b>Ho</b> holmium	68 <b>Er</b> erbium	69 <b>Tm</b> thulium	70 <b>Yb</b> ytterbium	71 <b>Lu</b> lutetium	
89 <b>Ac</b> actinium	90 <b>Th</b> thorium	91 <b>Pa</b> protactinium	92 <b>U</b> uranium	93 <b>Np</b> neptunium	94 <b>Pu</b> plutonium	95 <b>Am</b> amercurium	96 <b>Cm</b> curium	97 <b>Bk</b> berkelium	98 <b>Cf</b> californium	99 <b>Es</b> einsteinium	100 <b>Fm</b> fermium	101 <b>Md</b> mendelevium	102 <b>No</b> nobelium	103 <b>Lr</b> lawrencium				

# 一. 中子活化分析简介

## 研究成果

在国际化学领域顶级期刊之一  
《美国化学会志》发表研究文章



[pubs.acs.org/JACS](https://pubs.acs.org/JACS)

### Instrumental Neutron Activation Analysis of Chang'E-5 Lunar Regolith Samples

Yonggang Yao, Caijin Xiao,\* Pingsheng Wang, Chunlai Li, and Qin Zhou

Cite This: *J. Am. Chem. Soc.* 2022, 144, 5478–5484

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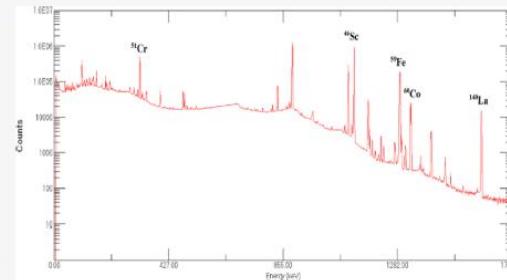
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**ABSTRACT:** The Chang'E-5 (CE-5) lunar samples were analyzed nondestructively for more than 40 elements by instrumental neutron activation analysis (INAA) with a strict quality assurance and quality control. Based on the INAA results, some new observations and discoveries were made. Major, minor, and trace elements in CE-5 are very different from those of the Earth and Apollo lunar samples. The rare earth element (REE) pattern indicated that the CE-5 lunar samples are mare basalts with a clear negative anomaly in Eu. Element correlations are a very interesting discovery; for example, the ratios of Ba/La and FeO/MnO in CE-5 are almost identical to those of the Apollo lunar samples. These observations and discoveries will enrich the understanding of the formation and evolution of the Moon.



## 在《国家科学评论》研究文章

### RESEARCH ARTICLE

National Science Review

9: nwab188, 2022

<https://doi.org/10.1093/nsr/nwab188>

Advance access publication 14 October 2021

EARTH SCIENCES

### Characteristics of the lunar samples returned by the Chang'E-5 mission

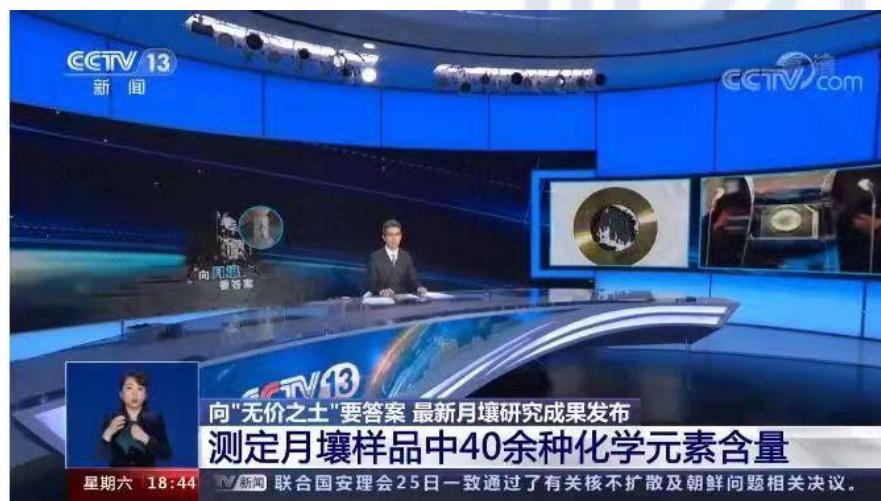
Chunlai Li <sup>1,\*</sup>, Hao Hu <sup>2,\*</sup>, Meng-Fei Yang <sup>3,\*</sup>, Zhao-Yu Pei <sup>2</sup>, Qin Zhou <sup>1</sup>, Xin Ren <sup>1</sup>, Bin Liu <sup>1</sup>, Dawei Liu <sup>1</sup>, Xingguo Zeng <sup>1</sup>, Guangliang Zhang <sup>1</sup>, Hongbo Zhang <sup>1</sup>, Jianjun Liu <sup>1</sup>, Qiong Wang <sup>2</sup>, Xiangjin Deng <sup>3</sup>, Caijin Xiao <sup>4</sup>, Yonggang Yao <sup>4</sup>, Dingshuai Xue <sup>5</sup>, Wei Zuo <sup>1</sup>, Yan Su <sup>1</sup>, Weibin Wen <sup>1</sup> and Ziyuan Ouyang <sup>1,6</sup>

### ABSTRACT

Forty-five years after the Apollo and Luna missions returned lunar samples, China's Chang'E-5 (CE-5) mission collected new samples from the mid-latitude region in the northeastern Oceanus Procellarum of the Moon. Our study shows that 95% of CE-5 lunar soil sizes are found to be within the range of 1.40–9.35  $\mu\text{m}$ , while 95% of the soils by mass are within the size range of 4.84–432.27  $\mu\text{m}$ . The bulk density, true density and specific surface area of CE-5 soils are 1.2387 g/cm<sup>3</sup>, 3.1952 g/cm<sup>3</sup> and 0.56 m<sup>2</sup>/g, respectively. Fragments from the CE-5 regolith are classified into igneous clasts (mostly basalt), agglutinate and glass. A few breccias were also found. The minerals and compositions of CE-5 soils are consistent with mare basalts and can be classified as low-Ti/low-Al/low-K type with lower rare-earth-element contents than materials rich in potassium, rare earth element and phosphorus. CE-5 soils have high FeO and low Mg index, which could represent a new class of basalts.

**Keywords:** Chang'E-5, lunar soils, physical properties, petrography, mineralogy, chemistry

# 一. 中子活化分析简介



相关成果被央视《综合频道》、《朝闻天下》、《新闻直播间》、《晚间新闻》等电视媒体报道

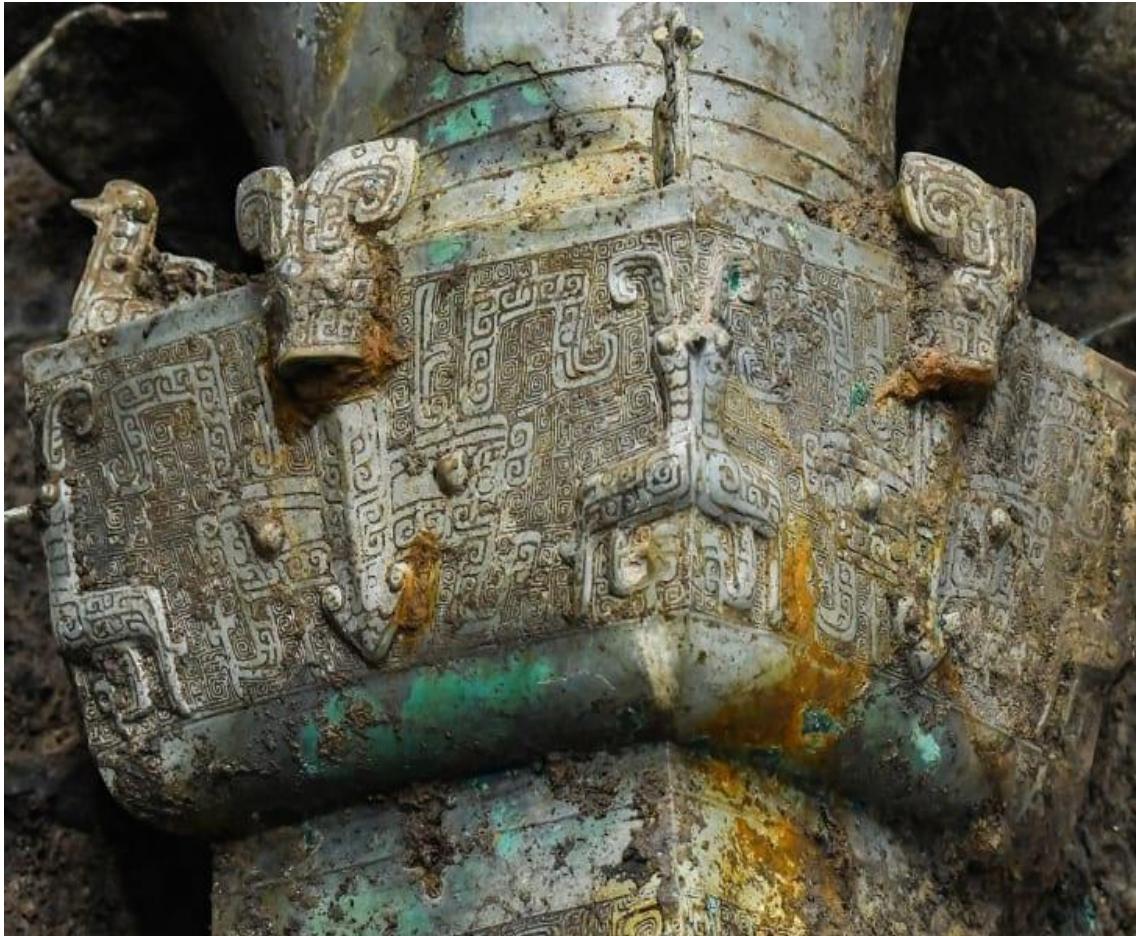
# 一. 中子活化分析简介

月壤研究下一步计划：与行星科学研究人员合作，对月壤成分的系统研究。

- 月壤样品中**岩屑、玻璃、包裹体**高精度中子活化分析；
- 不同深度的钻取月壤样品中子活化分析，研究月壤成分随**深度**的变化规律；
- 月壤中**3He**无损分析；

# 一. 中子活化分析简介

三星堆考古样品分析及产地溯源研究



# 一. 中子活化分析简介

- 口 三星堆青铜器采用陶范法铸造，铸造过程要经过制模、翻范、制芯、合范浇铸等工序，器物铸造完成后，泥芯常被封闭或半封闭在器物里，一般不再取出，从而残留于出土青铜器的耳、銎、足等部位。
- 口 残留泥芯可能包含青铜器“出生地”信息；
- 口 用中子活化分析对三星堆青铜器取样的13个青铜器泥芯样品进行了分析。

# 一. 中子活化分析简介

- **主量氧化物的元素:** Al、Mg、Fe、K、Na、Ca、Ti、Mn。
- **稀土元素:** La、Ce、Nd、Sm、Eu、Tb、Dy、Yb、Lu计9项。
- **基性造岩元素:** V、Cr、Co、Sc计4项。
- **酸性高场强元素:** Th、U、Hf、Ta计4项。
- **碱金属与碱土金属:** Sr、Ba、Rb计3项。
- **热液成矿元素:** Sn、Mo、Cu、Zn、Au、Ag、As、Sb、Hg计9项。

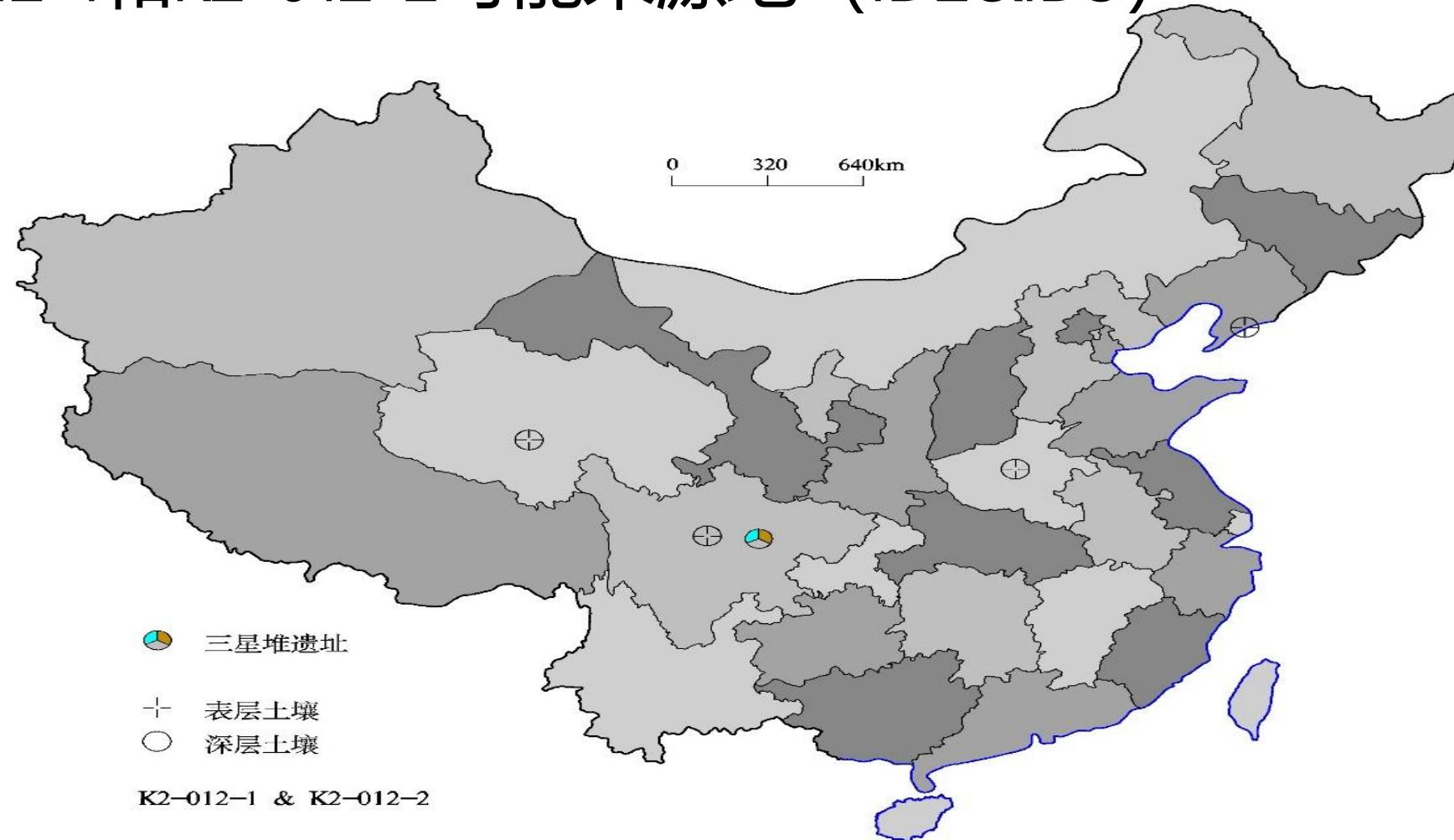
# 一. 中子活化分析简介

与中国地质大学合作，利用**土壤基因**的方法进行产地溯源。

- 针对NAA数据构建：**LG04A01和LG04A02, LG05; LG04B01和LG04B02; LG04C01和LG04C02岩性基因。**
- 样品与全国**表层土、深层土**进行比对；
- 对每一种样品的**土壤基因**进行编码，然后进行分类和**溯源**。

# 一. 中子活化分析简介

K2-012-1和K2-012-2可能来源地 (ID2&ID3)



# 一. 中子活化分析简介

结果

- 用NAA方法对样品主量元素、微量元素等40余种元素进行测定；
- 通过中子活化分析数据对三星堆文明进行溯源。
- 为三星堆文明的起源提供了重要的科学数据；

# 一. 中子活化分析简介

大气颗粒物PM2.5、PM10研究

**1994年-现在，在北京采集了30多年的PM2.5/PM10，采用NAA等核分析方法元素分析和源解析，建立了30年的大气颗粒物样本库和数据库。**

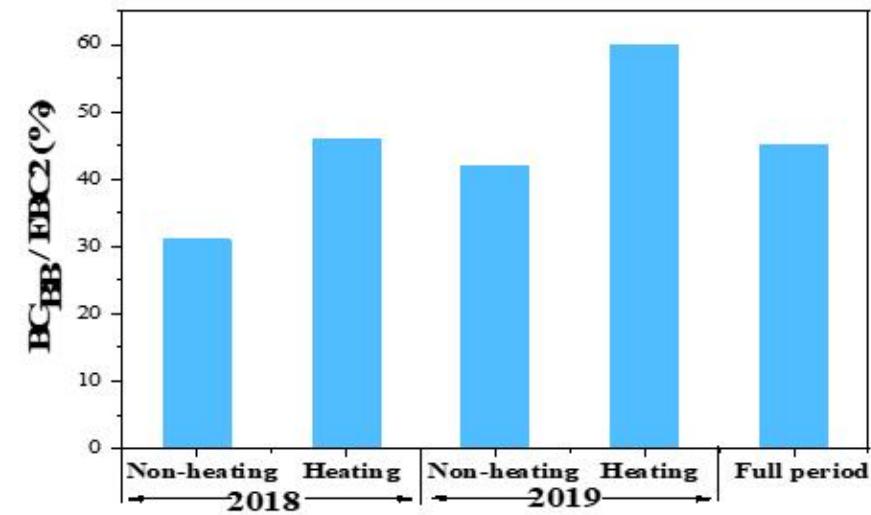
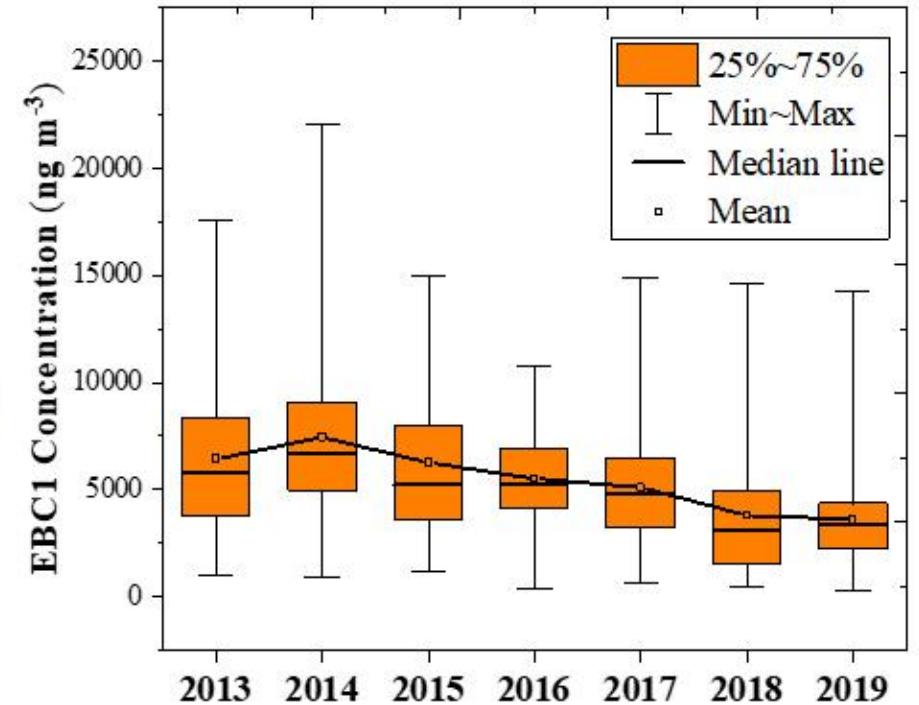
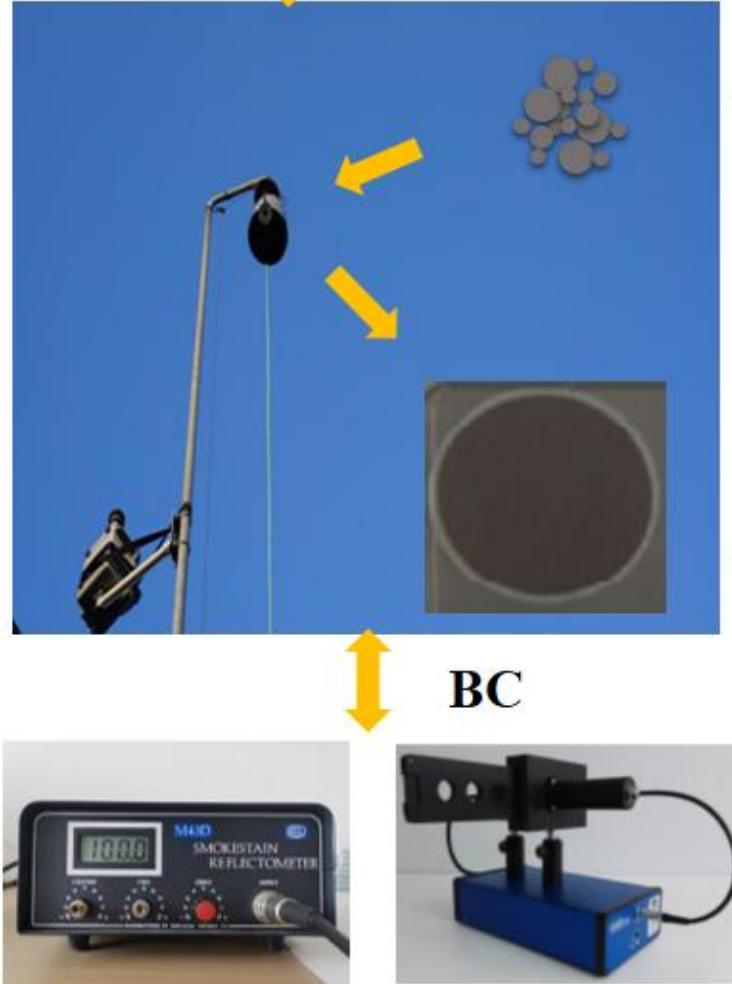


一次典型的重污染过程（2015）



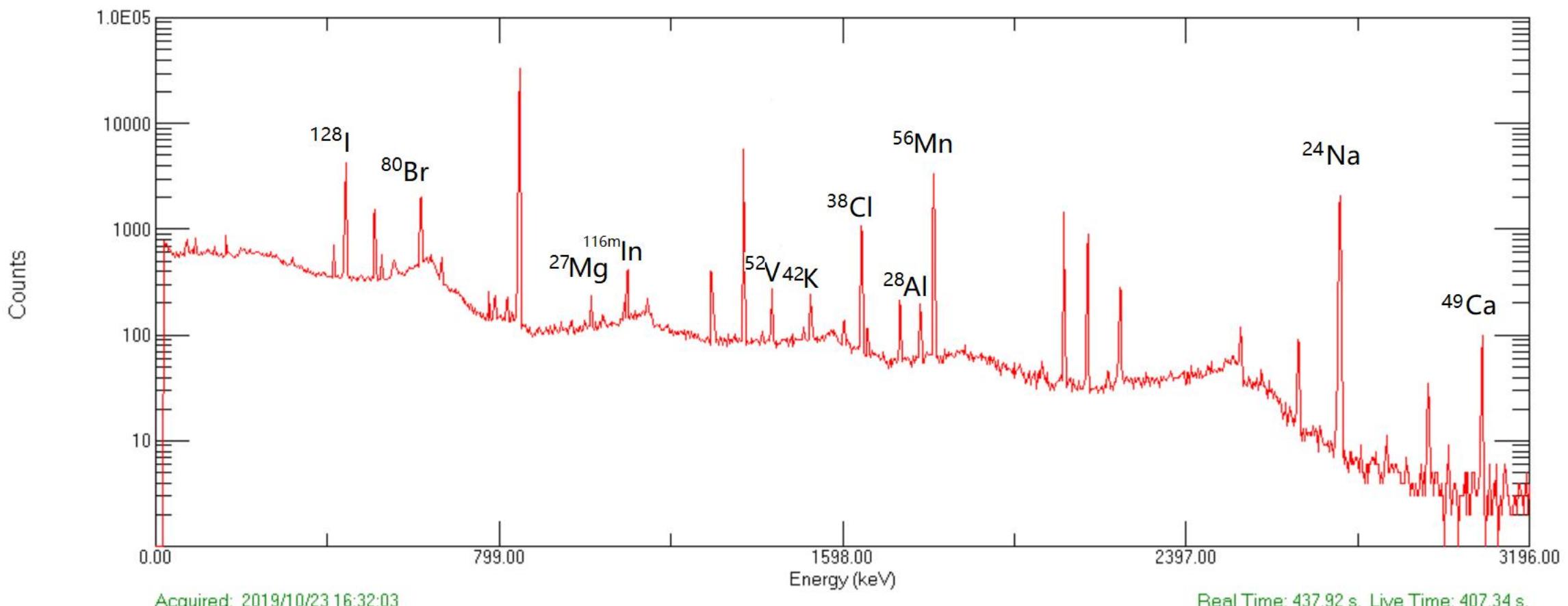
**Anthropogenic emissions  
Local meteorology  
Tracer gases**

**Influencing Factors**



**Source contribution**

## 一. 中子活化分析简介

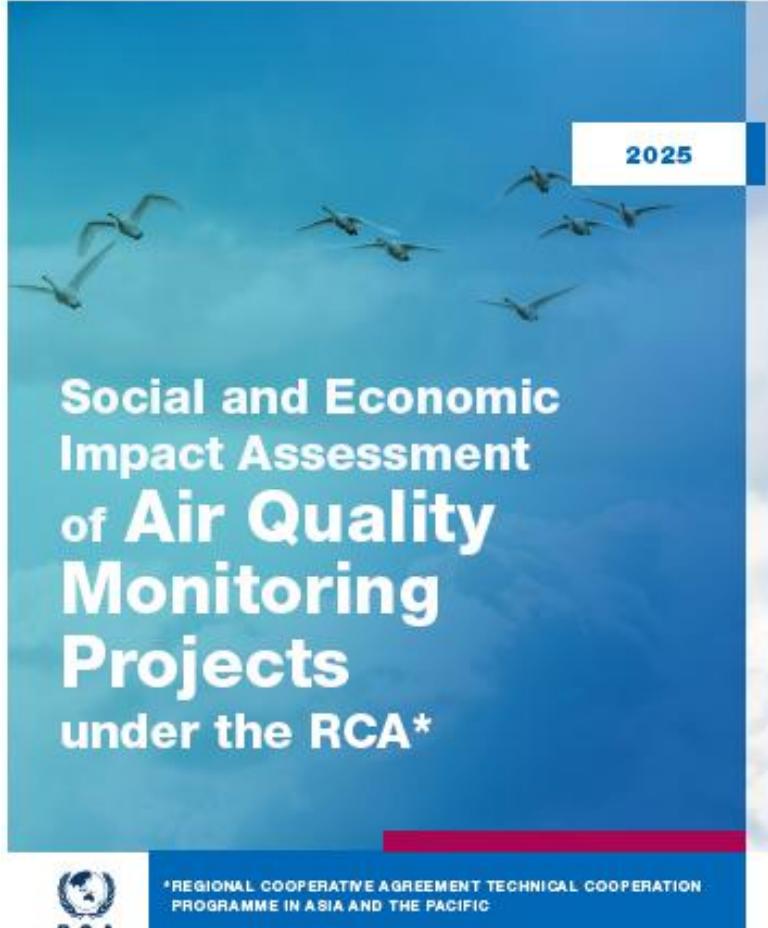


PM2.5短半衰期核素伽马能谱





IAEA



NU  
L

## Annex II: Air quality monitoring under RCA in China – case example

# 一. 中子活化分析简介

## 发表文章：

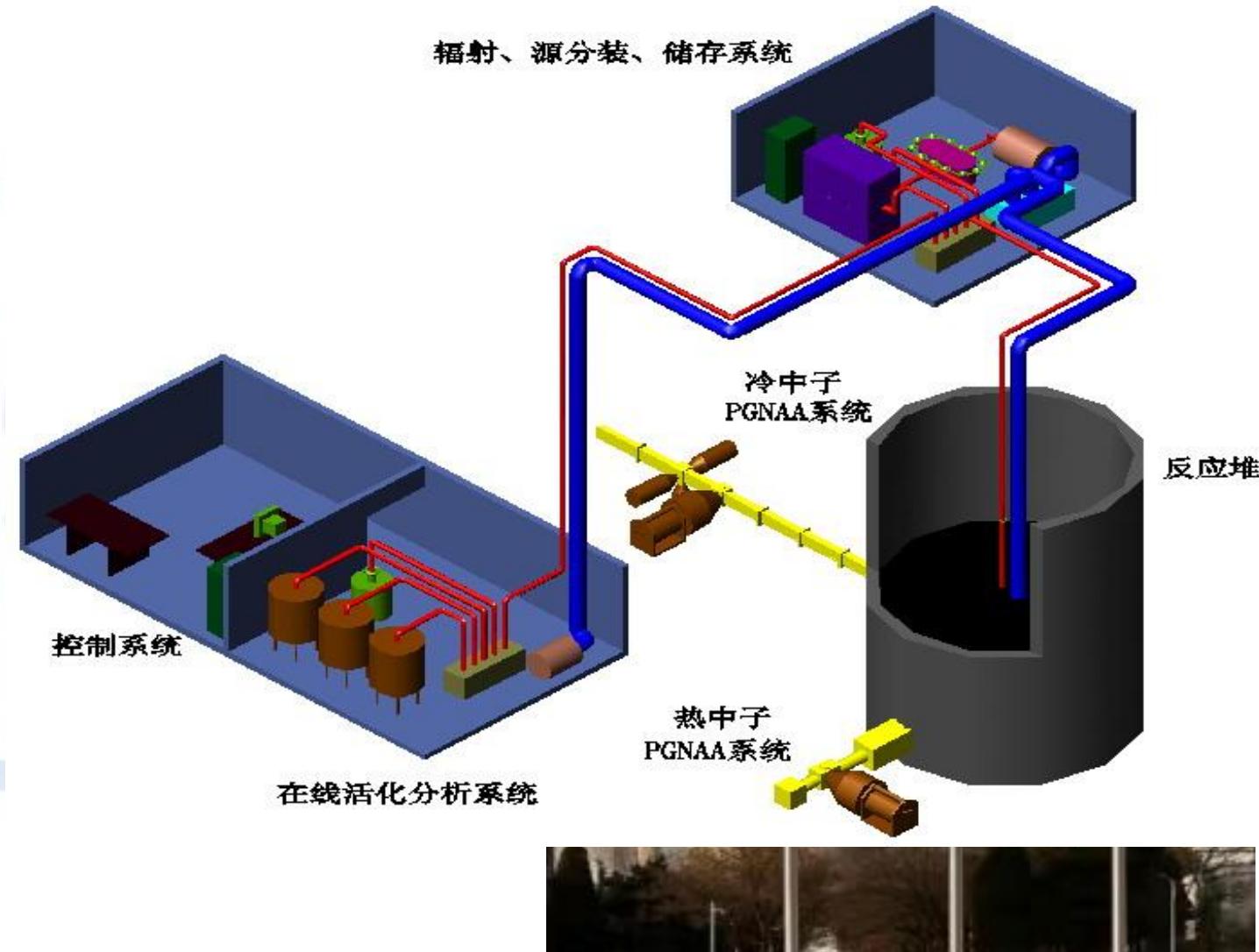
- Junxin Zhang , Yonggang Yao , Caijin Xiao a,\*et al., Atmospheric Pollution Research 14 (2023) 101669
- JunXin Zhang, Caijin Xiao\*, et al., Journal of Radioanalytical and Nuclear Chemistry, vol. 331, pages 609–617 (2022);
- Junkai Yang, Guoping Liu, et al., Journal of Radioanalytical and Nuclear Chemistry, vol. 323, pages 457–463 (2020);
- Xiangchun Jin, Caijin Xiao, et al., Journal of Radioanalytical and Nuclear Chemistry, vol. 307, pages 2147–2154 (2016);
- Caijin Xiao, Guiying Zhang, et al., Journal of Radioanalytical and Nuclear Chemistry, vol. 291, pages 95–100 (2012);
- Philip K. Hopke, David D. Cohen, et al., Science of The Total Environment, Vol. 409, Issue 19, 1 September 2011, Pages 4140;
- Lei Cao, Weizhi Tian, et al. Atmospheric Environment, Vol. 36, Issue 12, April 2002, Pages 1951-1956

# 一. 中子活化分析简介

## CARR中子活化分析研究平台

有五种分析技术：

- 常规中子活化分析INAA
- 缓发中子测量技术DNC
- 瞬发伽马活化分析PGAA
- 中子深度剖面技术NDP
- 微束中子活化分析



中子活化分析可实现全元素分析。

# 一. 中子活化分析简介

## CARR 堆参数



Item name	Parameter
Maximum nuclear power	60 MW
Maximum flux at center of active area	$1 \times 10^{15}$ $n/cm^2/s$
Max Flux at HW reflecting area	$8 \times 10^{14}$ $n/cm^2/s$
Pressure of Coolant at reactor core	0.793/0.127 MPa
T of Coolant at gate of in & out of reactor core	35/56.2 °C
Main loop flow	2386m <sup>3</sup> /hr
Enrichment factor of $^{235}U$	19.75 wt%
Height of active area/equivalent diameter	850/399.2 mm
Depth of pool water/inner diameter	15.0/5.5 m
Diameter of heavy water Reflector	479/2200 mm



## 目 录

01 中子活化分析简介

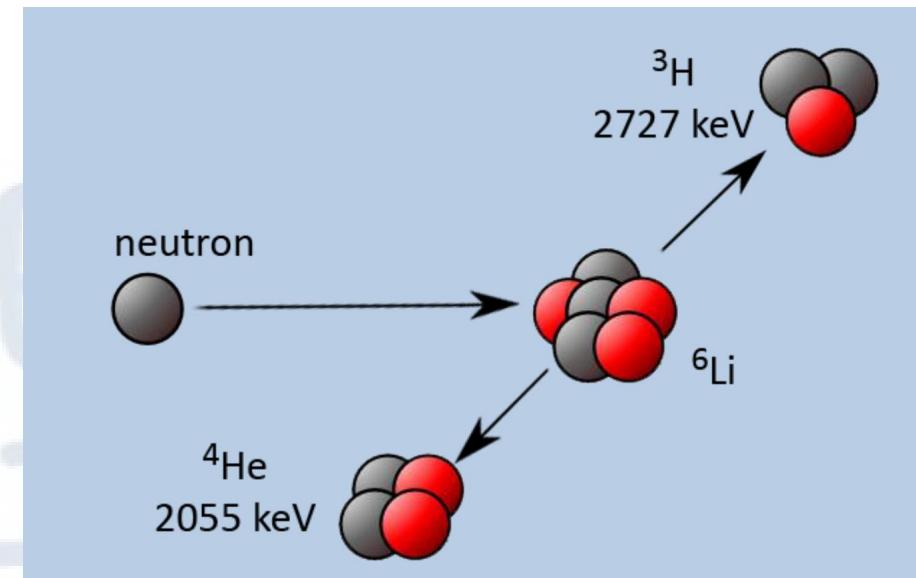
02 中子深度剖面研究进展

## 二. 中子深度剖面分析技术研究进展

## 中子深度剖面分析技术

Li、B等**轻元素**核素俘获热中子后发生(n,p)或(n,α)反应，出射粒子具有特定的能量，通过测定出射粒子的能量进行元素的定性和定量，从反应发生的位置到样品表面的能损则是该元素位置(深度)的量度；

$$x = \int_{E(X)}^{E_0} dE / S(E)$$



## 二. 中子深度剖面分析技术研究进展

### NDP技术特点 (SIMS、XPS等方法) :

1. 无损分析方法;
2. 分析深度几十um, ~10nm分辨率
3. He、Li、B等轻元素分析。
4. 高精度定量 ( $U<1\%$ ) ;
5. 原位/工况分析;



## NDP分析元素以及相关参数

元素	反应	%丰度	能量keV		截面b	探测极限 (原子/cm <sup>2</sup> ) <sup>a)</sup>
He	$^3\text{He}(\text{n},\text{p})$	0.00014	572	191	5333	$1.5 \times 10^{12}$
Li	$^6\text{Li}(\text{n},\alpha)$	7.5	2055	2727	940	$9.0 \times 10^{12}$
B	$^{10}\text{B}(\text{n}, \alpha)$	19.9	1472	840	3837	$2.1 \times 10^{12}$

a) 中子束强度 $6 \times 10^9 \text{ cm}^{-2}\text{s}^{-1}$ 估算

## 二. 中子深度剖面分析技术研究进展

### 中子深度剖面分析技术

在CARR堆建立了中子深度剖面分析装置，中子注量率可达 $10^9 n \cdot cm^{-2} \cdot s^{-1}$ ，先进的探测系统和数据处理方法、软件，CARR NDP整体性能处于国际先进水平。

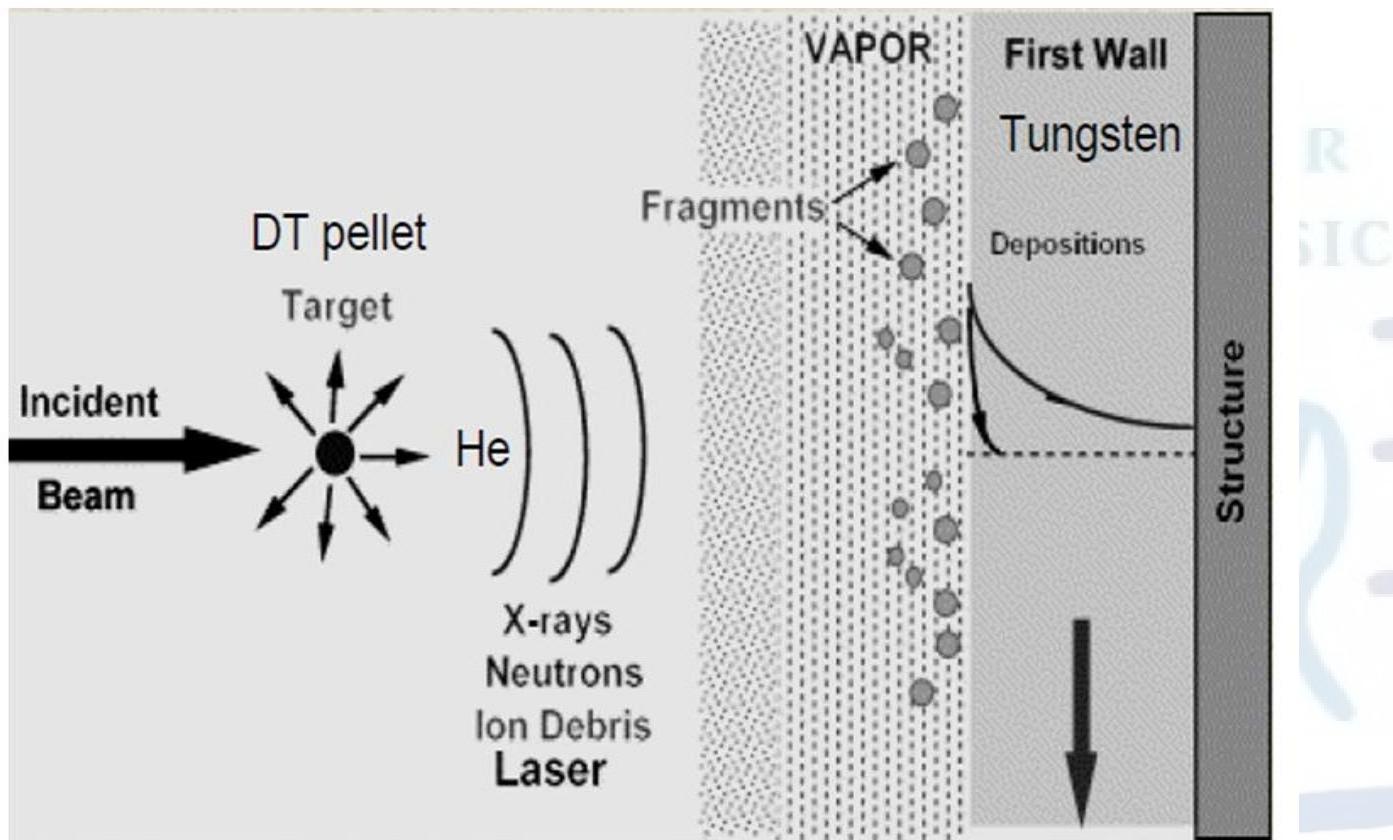


**CARR NDP装置图**

## 二. 中子深度剖面分析技术研究进展

中子深度剖面分析技术应用：

1. 锂电池测试；
2. 半导体B元素掺杂。
3. 核工业中应用 (He、B、Li)：聚变堆第一管壁材料He脆研究，氚增殖材料LiAlO<sub>2</sub>锂的扩散研究；玻璃固化体中B、Li等元素扩散研究；中子屏蔽材料、核材料中B、Li含量测定等；



聚变堆第一管壁He的含量随深度的变化

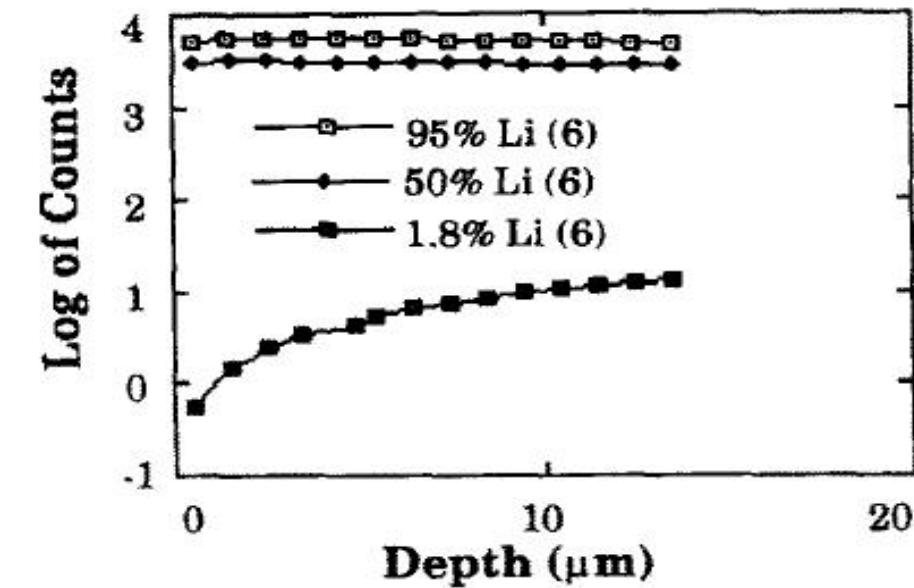


Fig. 9. Concentration of profiles of  ${}^6\text{Li}$  in  $\text{LiAlO}_2$  ceramics.

$\text{LiAlO}_2$ 锂陶瓷表面锂的深度分布

## 二. 中子深度剖面分析技术研究进展

**IAEA NP-T-5.3****不可或缺的**

However, the method gained renewed importance with the propagation of the lithium-ion batteries [10, 11], and NDP has emerged as an **indispensable** tool in the investigation of lithium-ion batteries. New NDP facilities were put into operation [4] or several older ones were upgraded to satisfy the increasing needs of energy-storage researchers [12, 13]. It is now possible to monitor the charge/discharge processes in operando mode, e.g. the accumulation of the lithium ions in silicon-graphite anodes [14] or in silicon-electrolyte interface (SEI) [15]. A few examples related to IAEA's SDGs are listed below, in all cases connected to the role of NDP in the development and improvement of lithium-ion batteries:

**NDP是一种锂电池不可或缺的测试手段。**

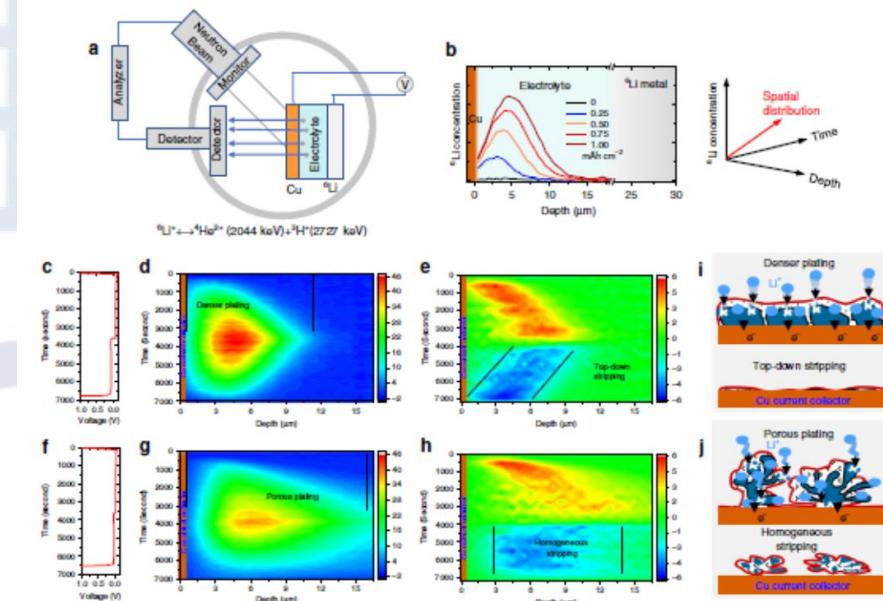
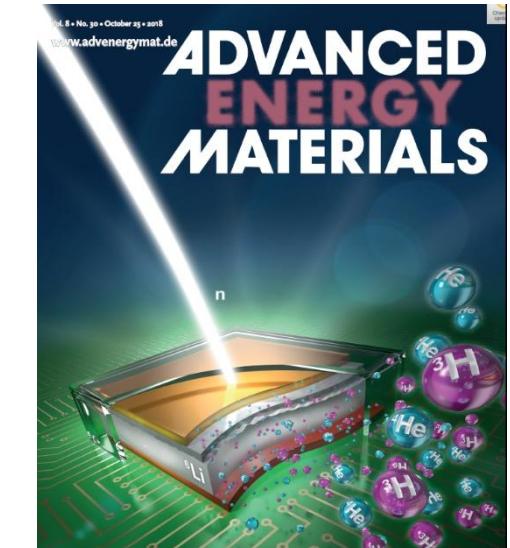
## 二. 中子深度剖面分析技术研究进展

- 近几年，NDP技术在锂电池测试需求和应用的带动下，发展迅速。
- 很多NDP的测试应用成果发表在nature、JACS、AEM等顶刊；

Lv, S. et al. Operando monitoring the lithium spatial distribution of lithium metal anodes. Nat. Commun. 9, 2152 (2018).

Han, F. et al. High electronic conductivity as the origin of lithium dendrite formation within solid electrolytes. Nat. Energy 4, 187–196 (2019).

Wang, Q. et al. Interface chemistry of an amide electrolyte for highly reversible lithium metal batteries. Nat. Commun. 11:4188 (2020).



## 二. 中子深度剖面分析技术研究进展

功能：表征锂离子在电池中的传输过程！！！

- 1) 锂电池材料中（正负电极、电解质）中锂的分布；
- 2) SEI、CEI等界面形成过程、锂离子分布；
- 3) 锂浓度梯度变化(锂的脱嵌过程)；
- 4) 锂电池的失效、老化过程；
- 5) 锂电池材料电导率测定；
- 6) 锂枝晶的形成过程，电池短路，锂电安全评估；
- 7) 锂电池材料性能评估；

## 二. 中子深度剖面分析技术研究进展

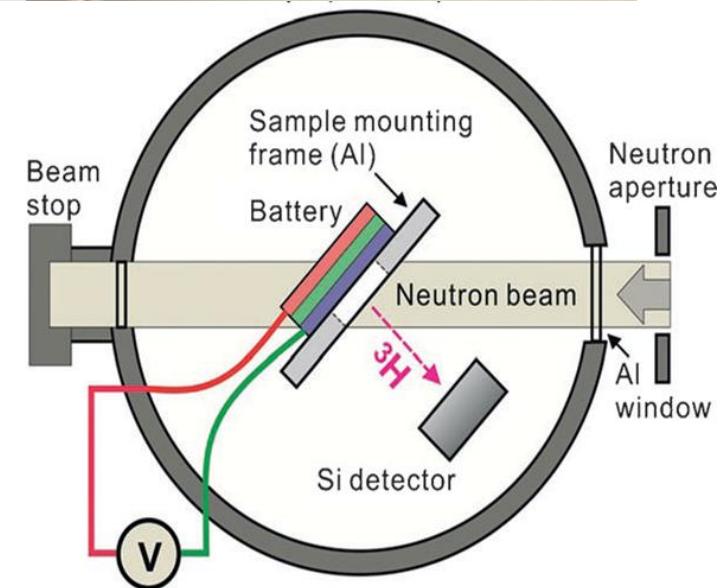
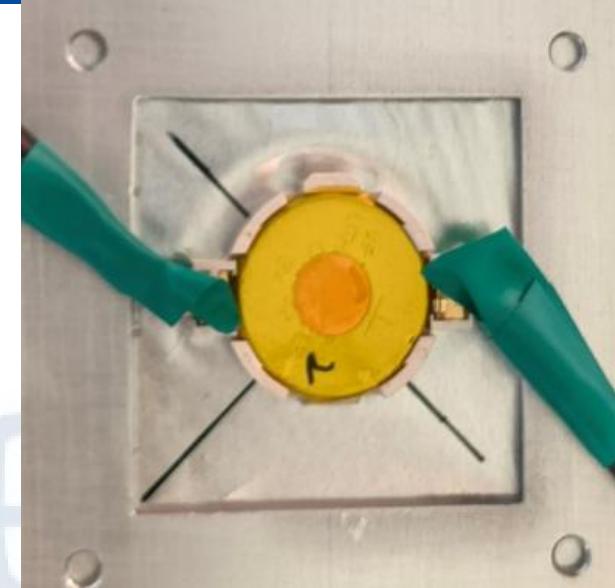
### NDP锂电池原位实验

1) 不同类型电池原位NDP

实验 (扣式、软包；液  
态、固态)；

2) 原位温控装置 ( $100^{\circ}\text{C}$ )

3) 实时测试充放电条件下  
锂在电极及界面的分布；

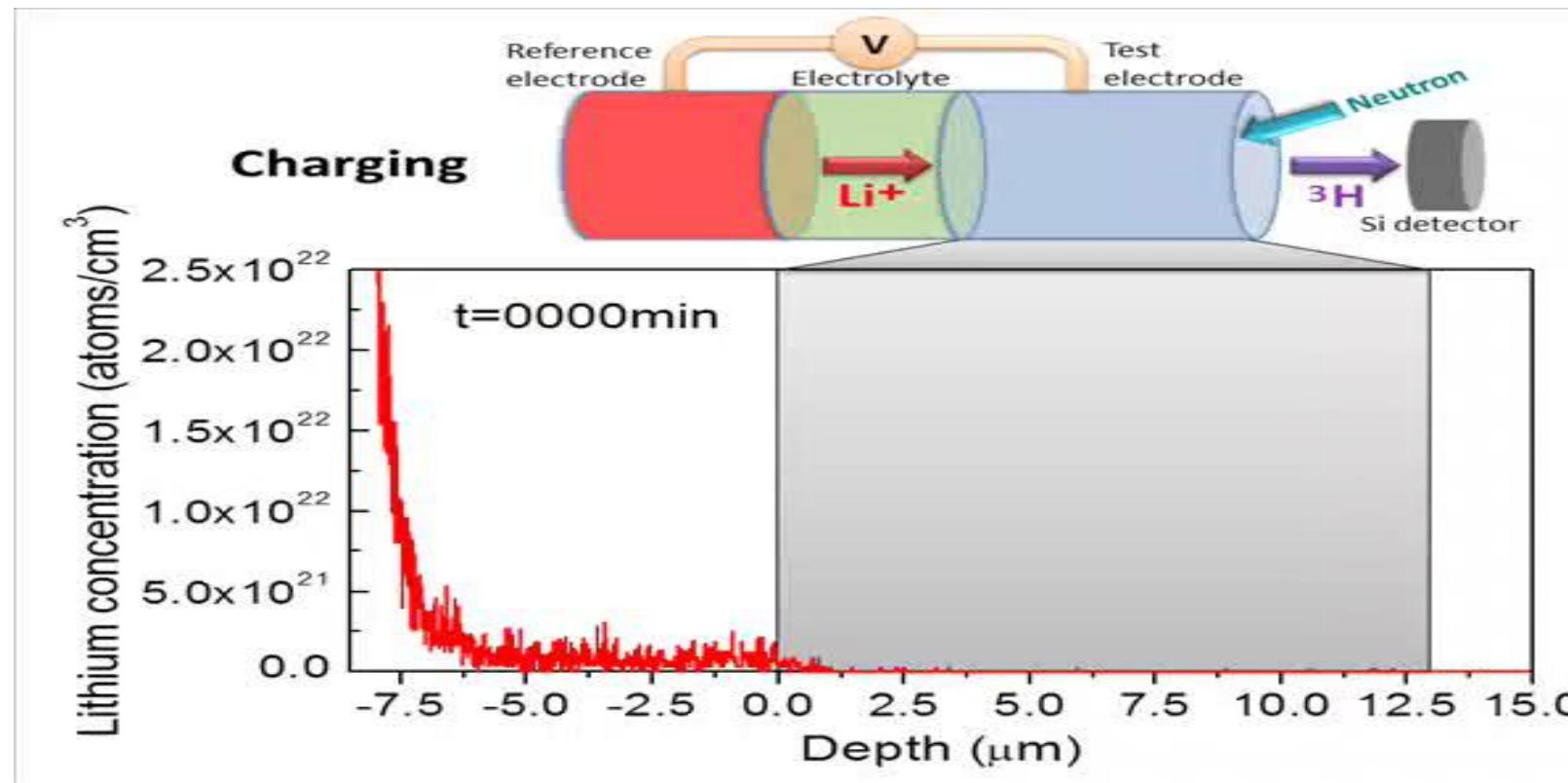


## 二. 中子深度剖面分析技术研究进展

## NDP用于锂电池原位测试

锂含量

深度



## 二. 中子深度剖面分析技术研究进展



### 口电池非原位实验:

- 1) 全自动换样测量;
- 2) 精确定位, 标准物质质控, 锂高精度定量;
- 3) 锂元素灵敏度高;
- 4) 1-4月, 已分析超过1000个电极样品;



## 二. 中子深度剖面分析技术研究进展

### ✓ 样品形式：

将不同条件（如SOC，倍率xC，颗粒度、包覆、压力、温度等）极片或电解质样品放置在铝板上，表面贴薄膜(10umPET)。

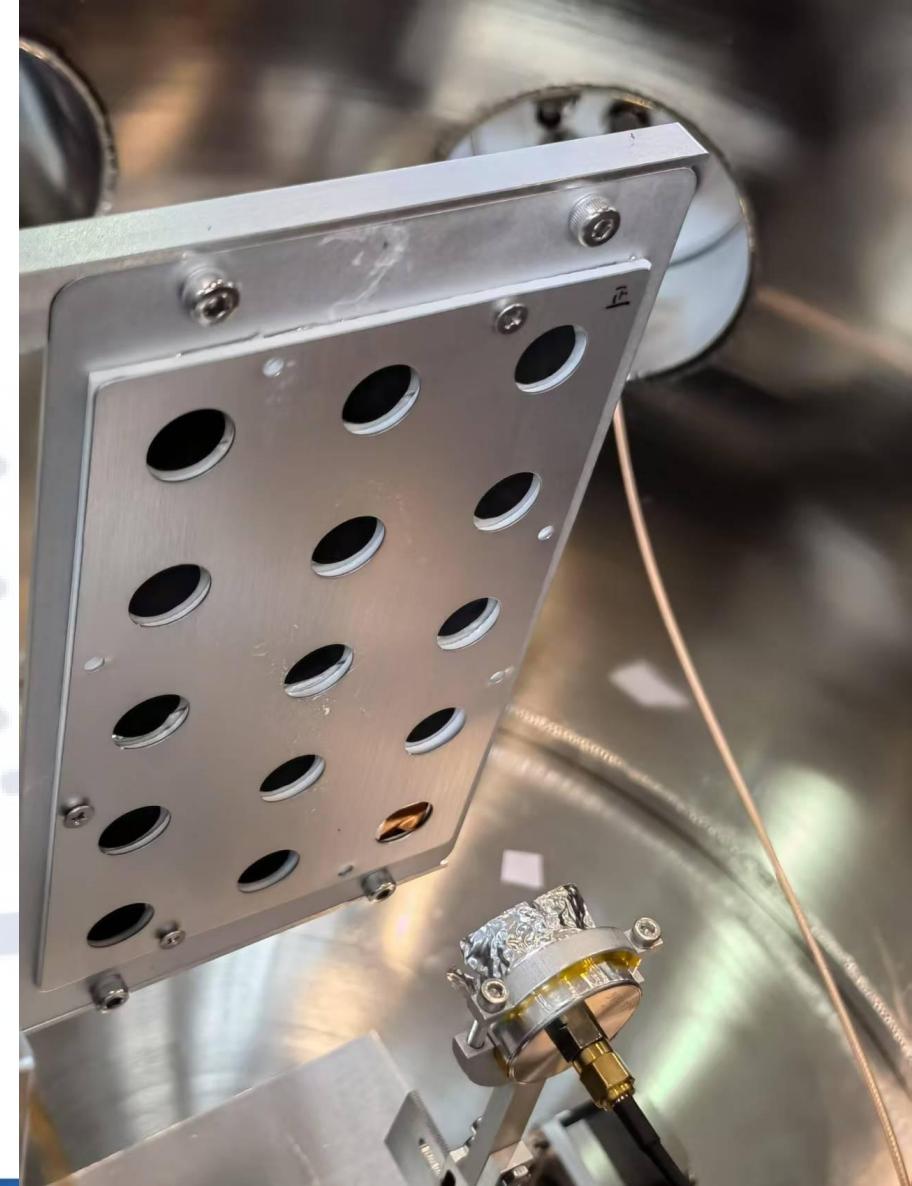


铝板尺寸：20mm\*20mm\*0.5mm

## 二. 中子深度剖面分析技术研究进展

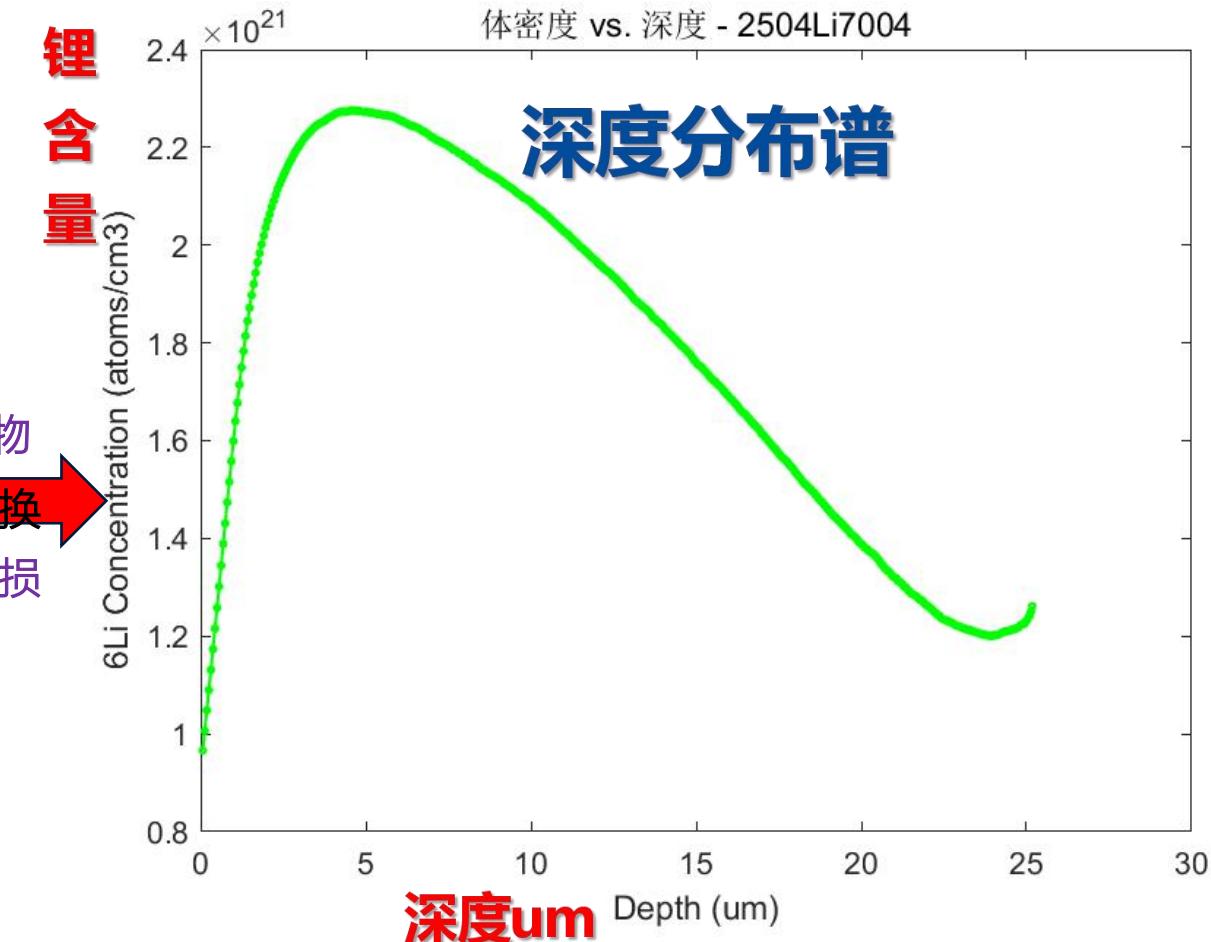
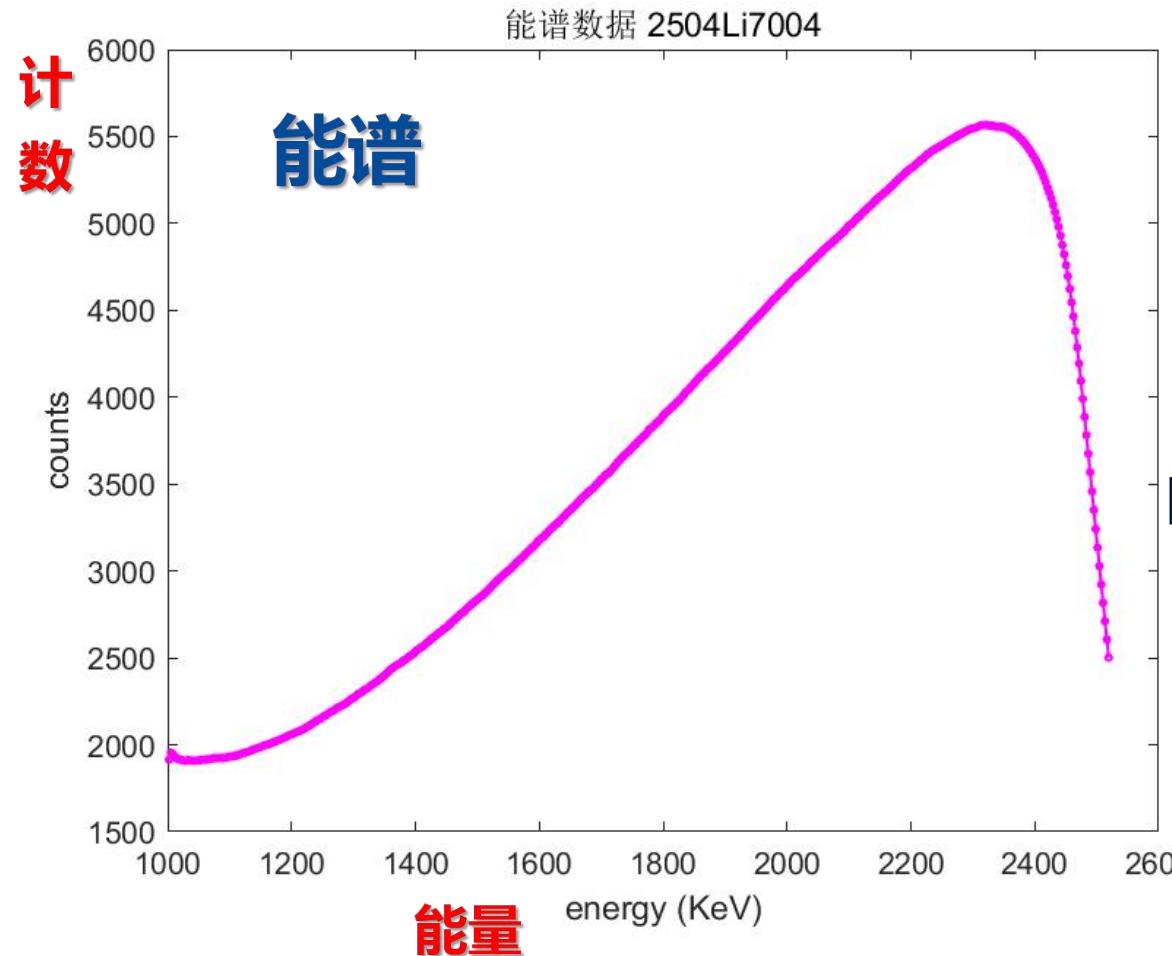
✓ 样品盘一次可以放置15个电极样品，  
实现样品自动测量！

✓ 由于NDP无损分析的特点，NDP测  
试完的样品还可以继续用于SEM、  
XPS、XRD等其他实验研究！

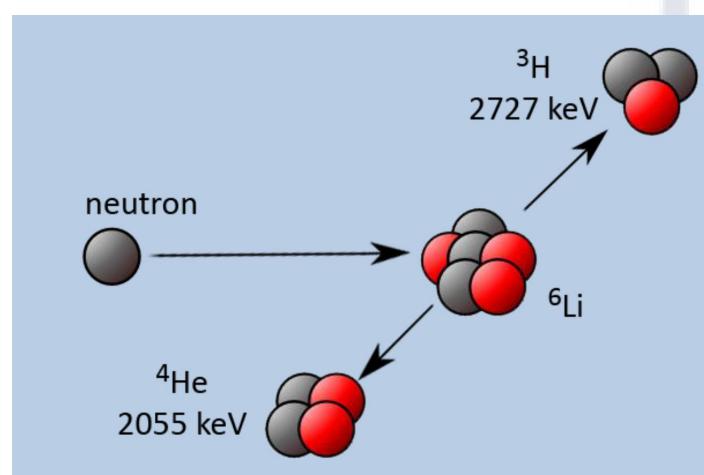
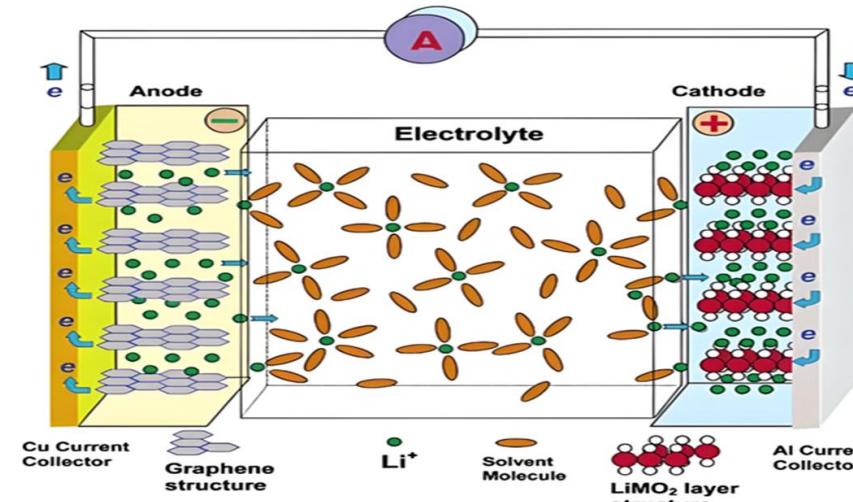


## 二. 中子深度剖面分析技术研究进展

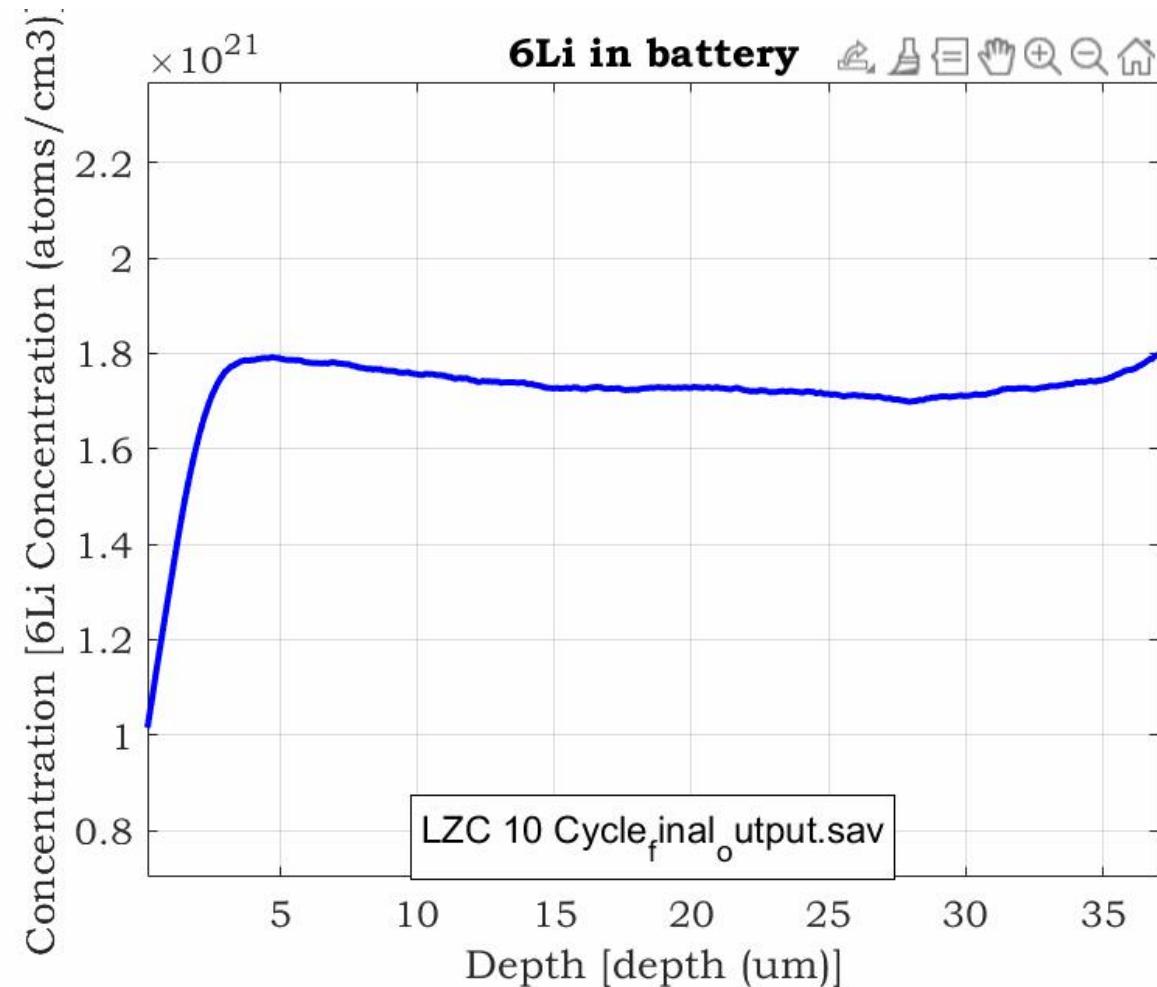
## NDP数据处理方法:



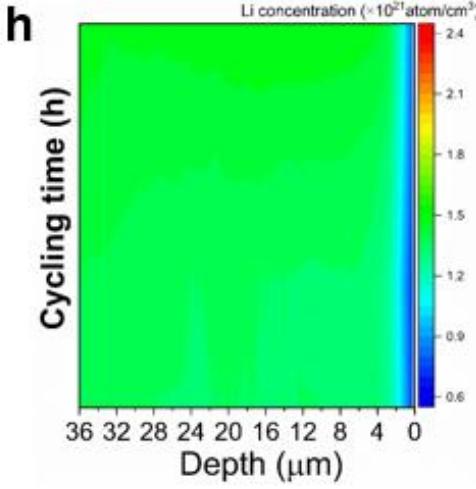
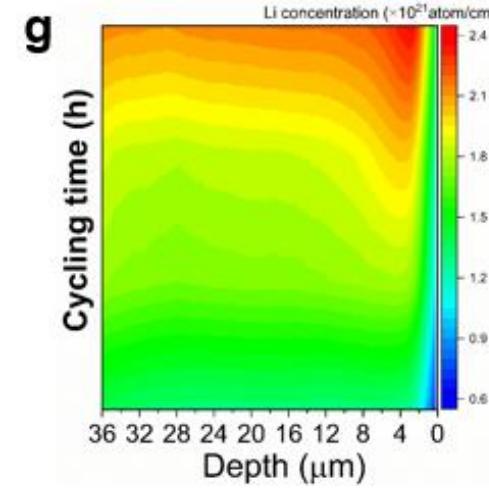
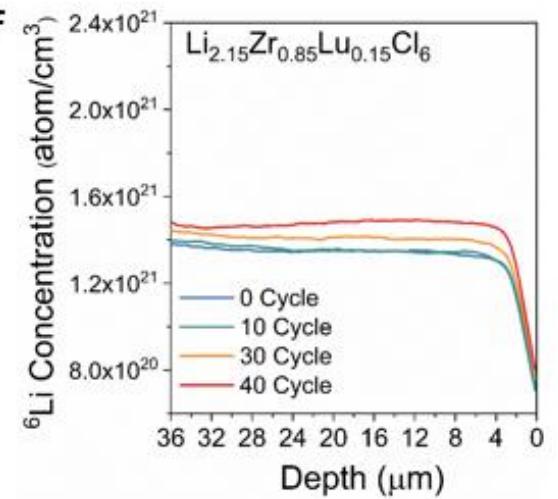
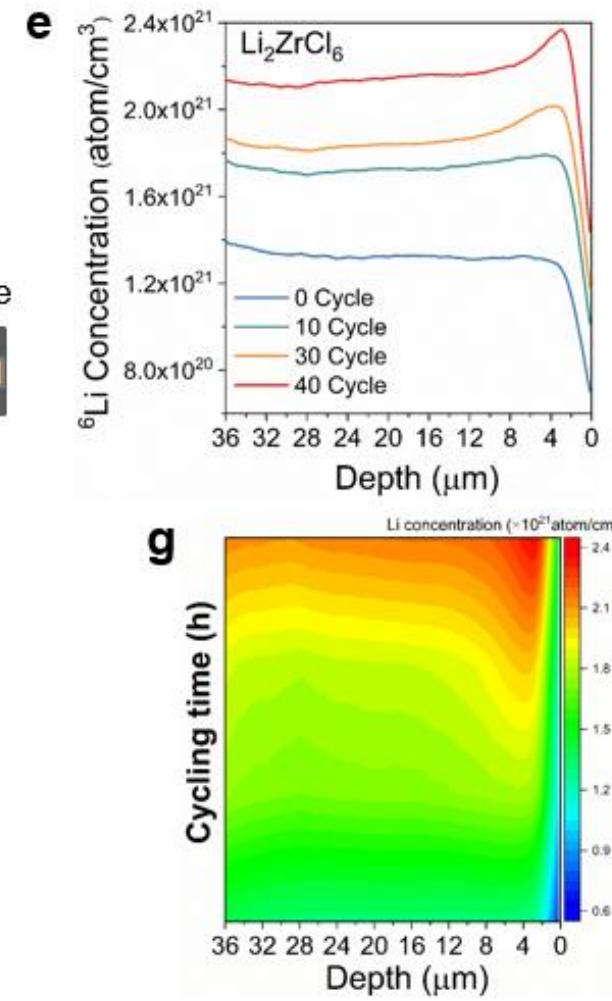
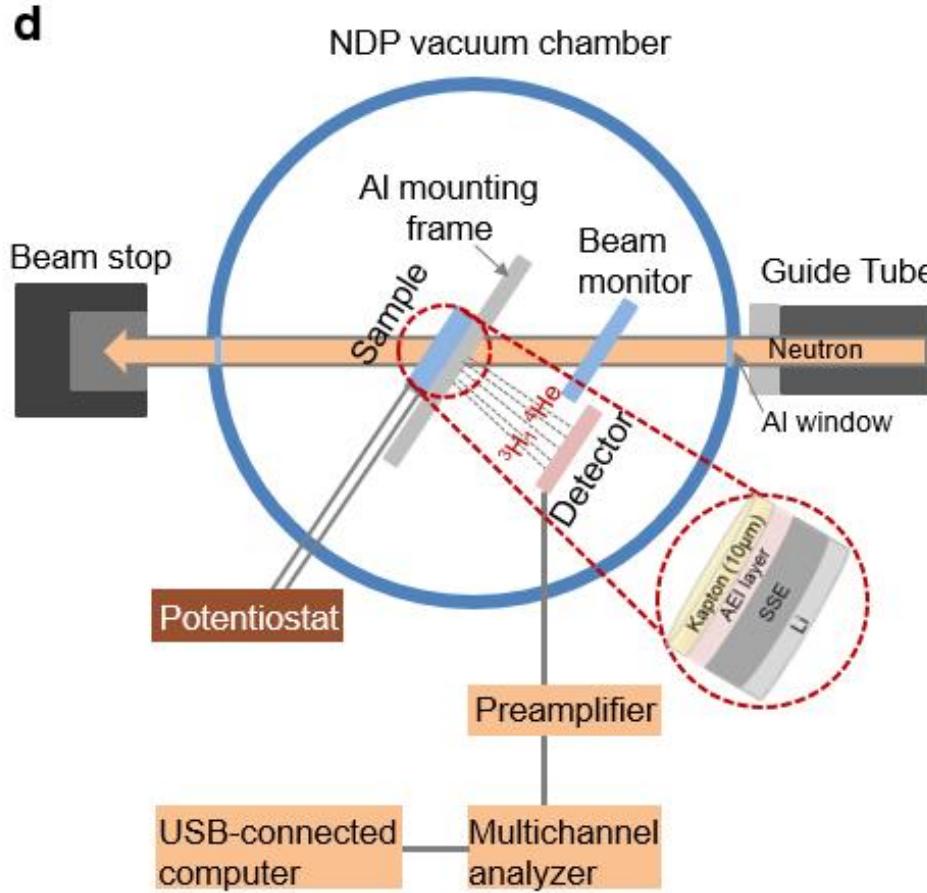
## 二. 中子深度剖面分析技术研究进展



✓ 多个样品测试结果合在一起可得到变化趋势；



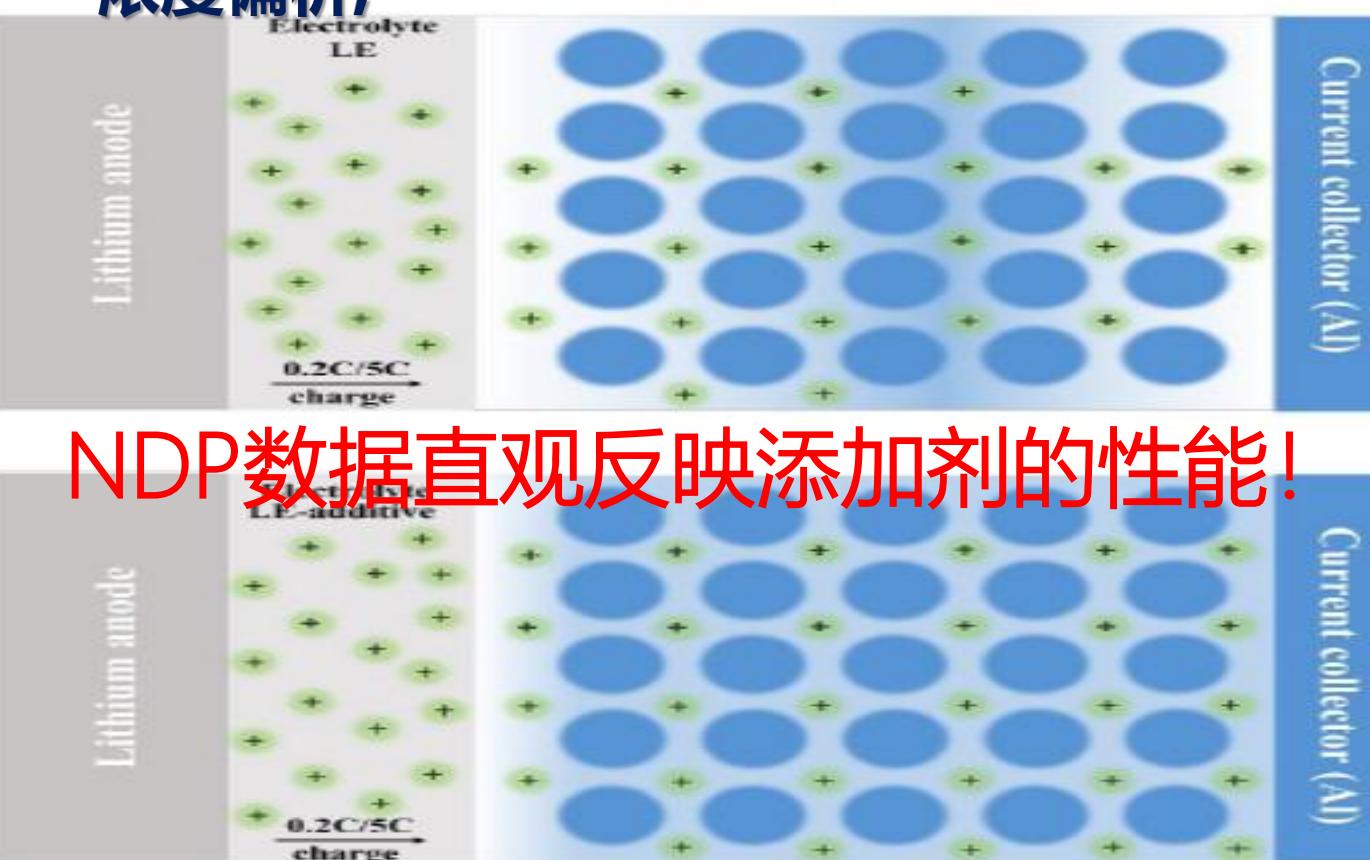
# 口用NDP技术表征卤化物固态电解质：



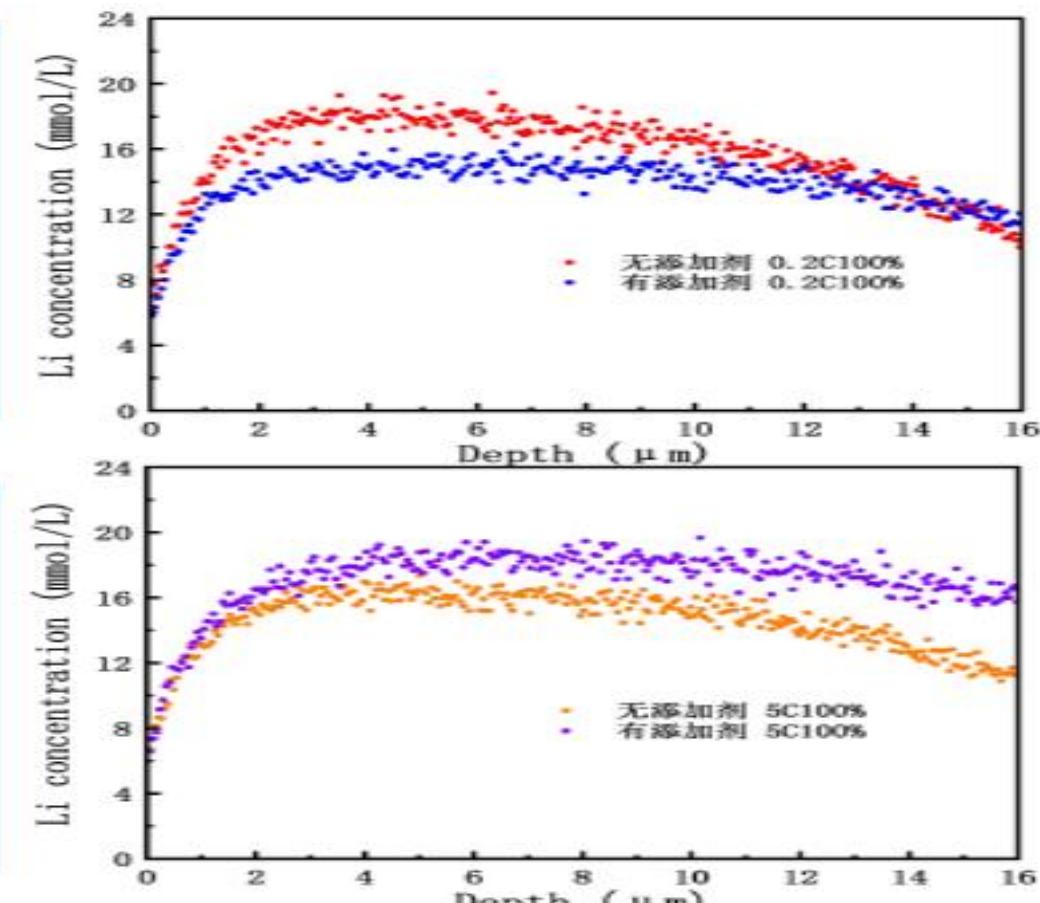
## 二. 中子深度剖面分析技术研究进展

口用NDP技术表征锂电池正极电解质界面锂的空间分布，获得正极的相变动力学信息。

➤ 放电过程中，无论是低倍率或高倍率，添加剂的存在有利于降低正极材料嵌锂时的锂浓度偏析；

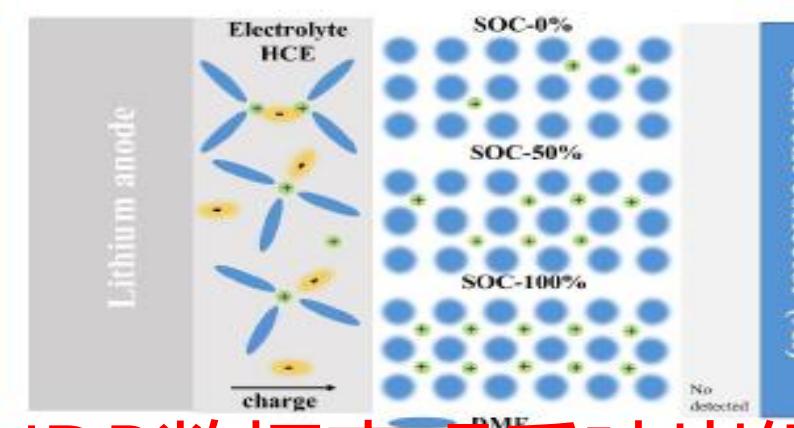
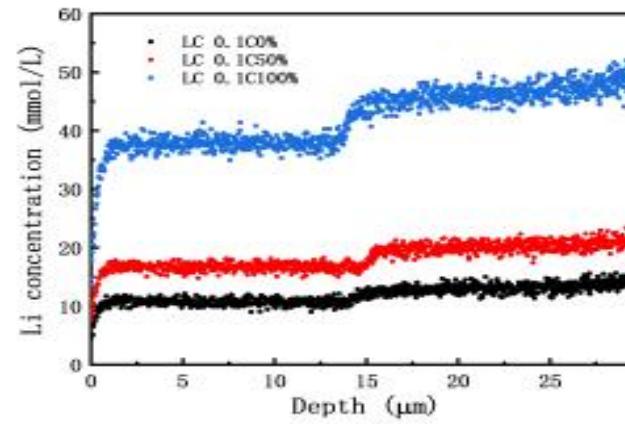
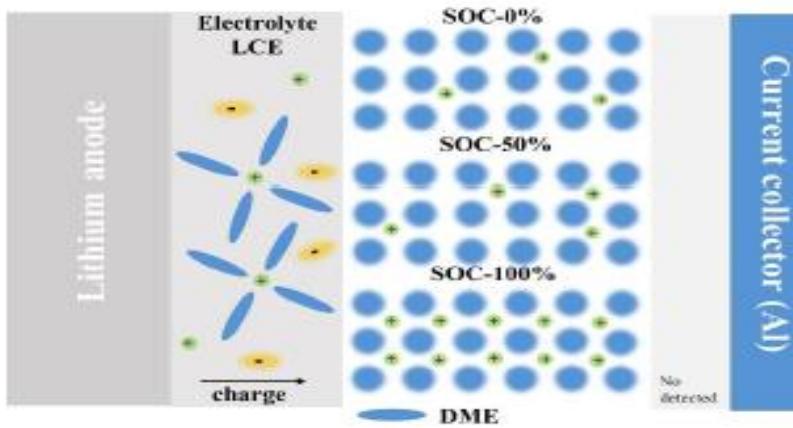


NDP数据直观反映添加剂的性能！

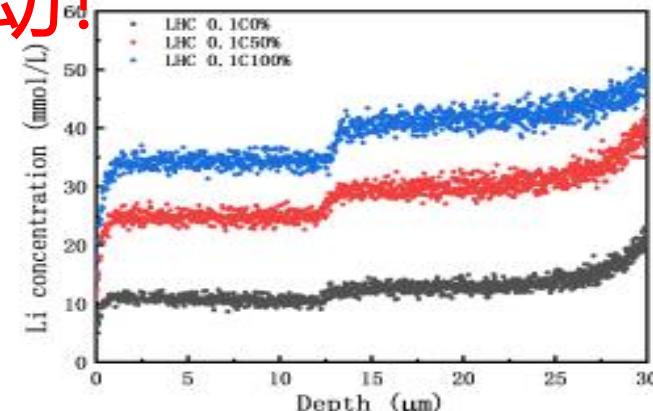
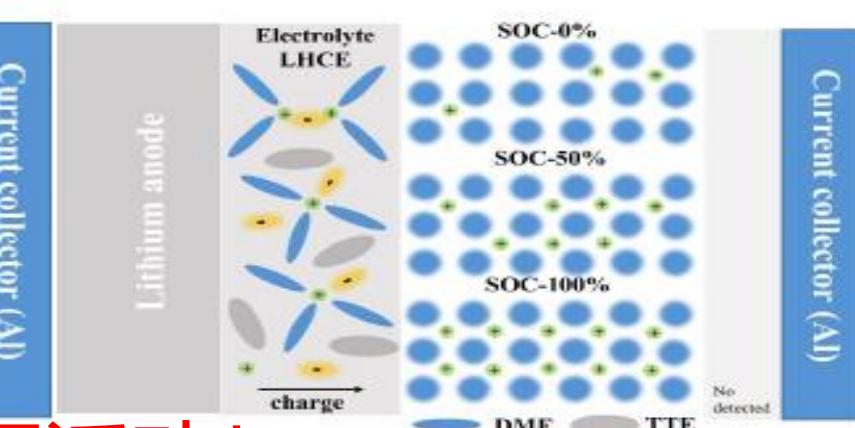
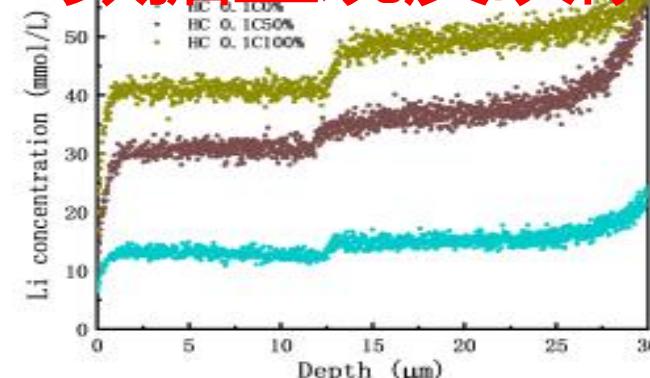


## 二. 中子深度剖面分析技术研究进展

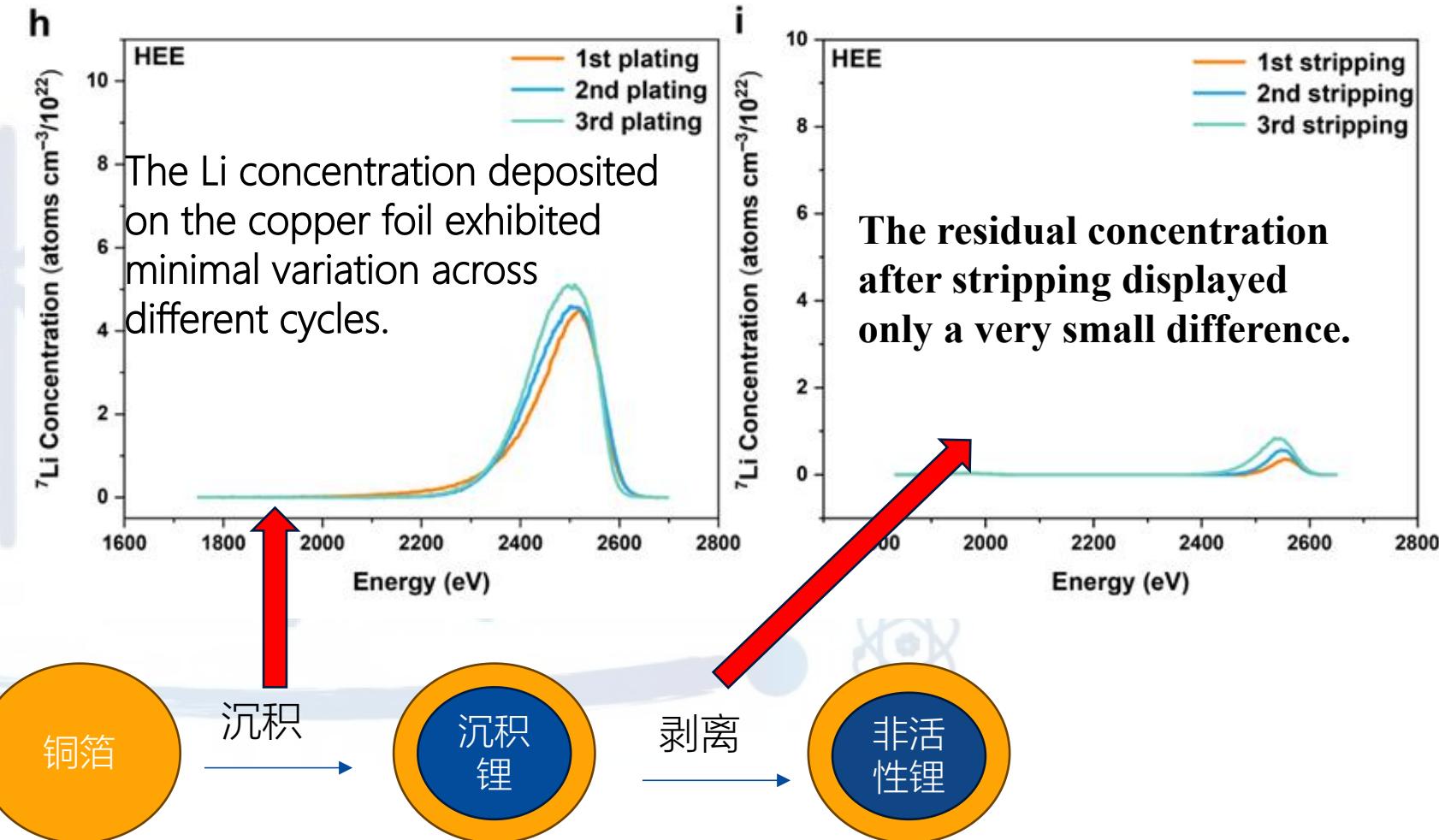
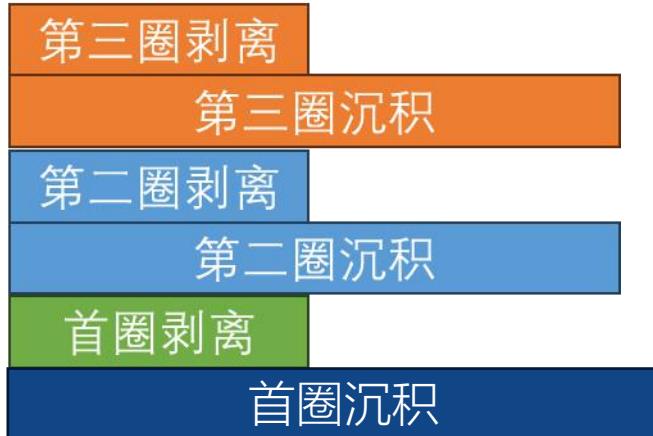
- **低倍率下,** 低浓电解液放电过程中正极材料嵌锂活动主要发生在50%~100%SOC;
- **低倍率下,** 高浓和局域高浓电解液放电过程中正极材料嵌锂活动主要发生在0%~50%SOC;



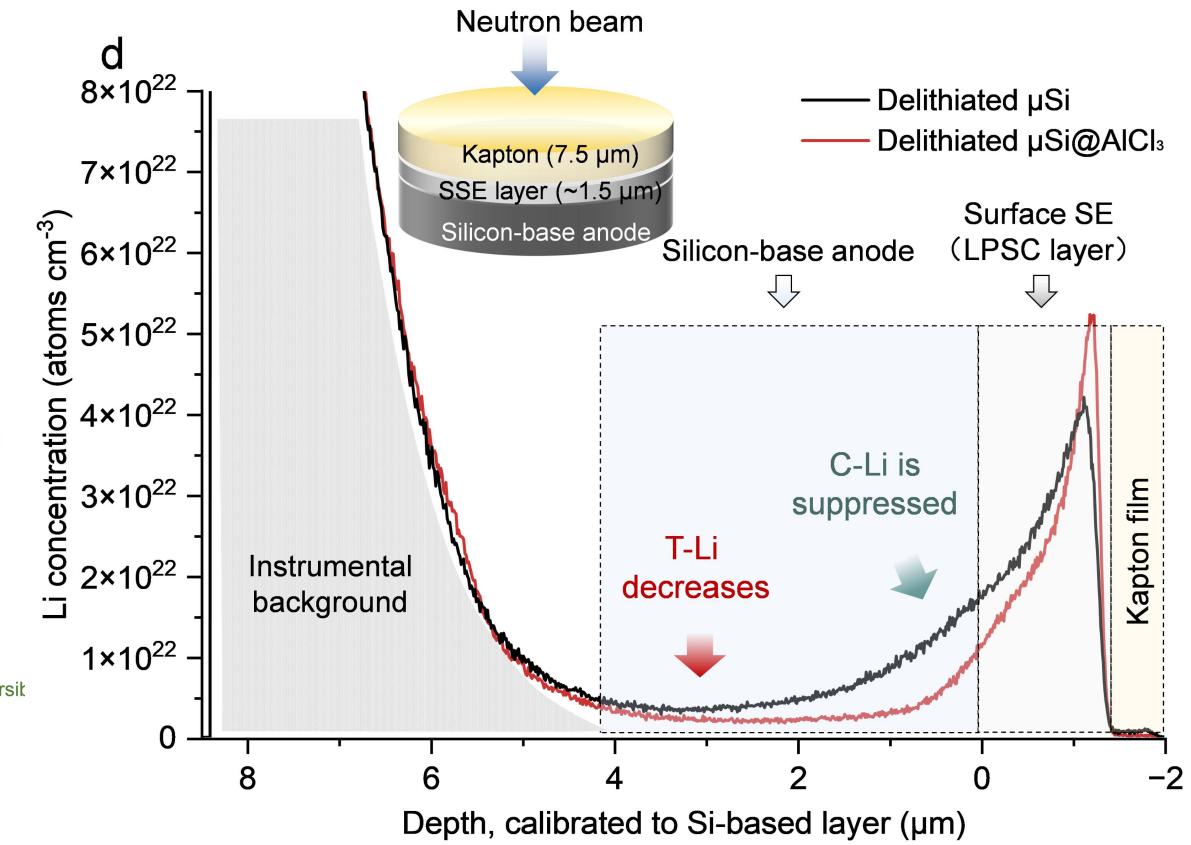
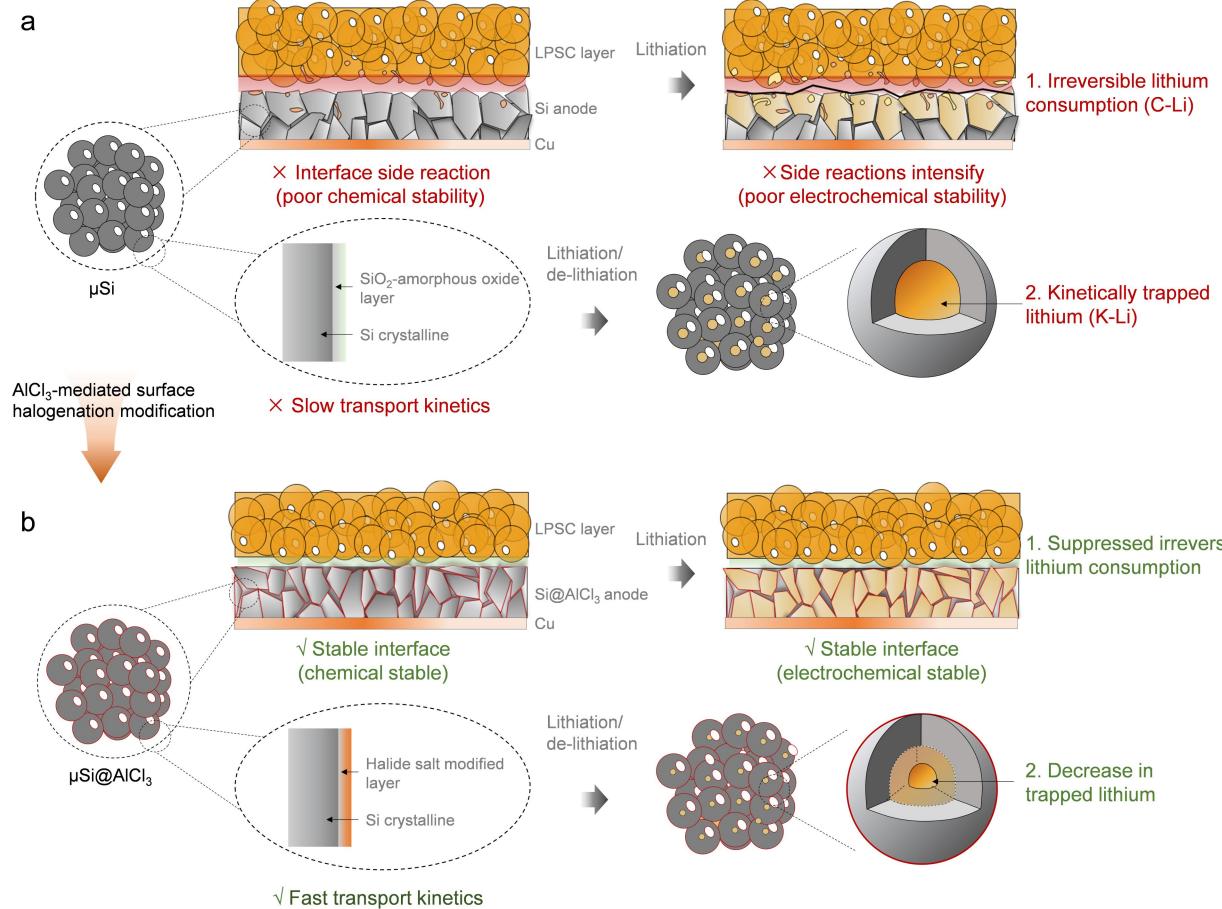
NDP数据直观反映嵌锂活动!



# 用NDP技术表征SEI层形成过程。



# 口用NDP技术表征SEI层，副反应抑制效果。



The results reveal a significant reduction in irreversible Li loss in Si@AlCl<sub>3</sub> compared to pristine Si anodes.

## 二. 中子深度剖面分析技术研究进展



*Journal of The Electrochemical Society*, 156 (11) A886-A890 (2009)  
0013-4651/2009/156(11)/A886/5/\$25.00 © The Electrochemical Society



### Neutron Depth Profiling Applications to Lithium-Ion Cell Research

S. Whitney,<sup>a,\*</sup> S. R. Biegalski,<sup>a,z</sup> Y. H. Huang,<sup>a,b</sup> and J. B. Goodenough<sup>a</sup>



<sup>a</sup>Mechanical Engineering, The University of Texas at Austin, Austin, Texas 78712, USA

<sup>b</sup>School of Materials Science and Engineering, Huazhong University of Science and Technology, Wuhan, Hubei, China

This research proved that neutron depth profiling (NDP) is a useful technique that provides insight into lithium transport within lithium-ion cells. Studies were conducted utilizing NDP to examine lithium-ion cell behavior under three environmental and operational parameters: (i) storage of cells at temperature, (ii) cell charge/discharge cycling, and (iii) charge/discharge rate and state of charge. During the first study, where cells were stored at 50°C for up to 140 days, the solid electrolyte interface growth rate was determined to range up to 4.8 nm per day depending on the cell capacity. The second study involving cell charge/discharge cycling did not reveal a statistically significant buildup of lithium near the cathode-electrolyte surface; however, the depth profiles showed an increased fluctuation in lithium concentrations as the number of cycles increased. Depth profiles from the third area of investigation quantified the linear relationship between lithium buildup near the cathode-electrolyte surface and the rate of charge (ranging from 0.1 to 4 C in this study).

© 2009 The Electrochemical Society. [DOI: 10.1149/1.3216033] All rights reserved.

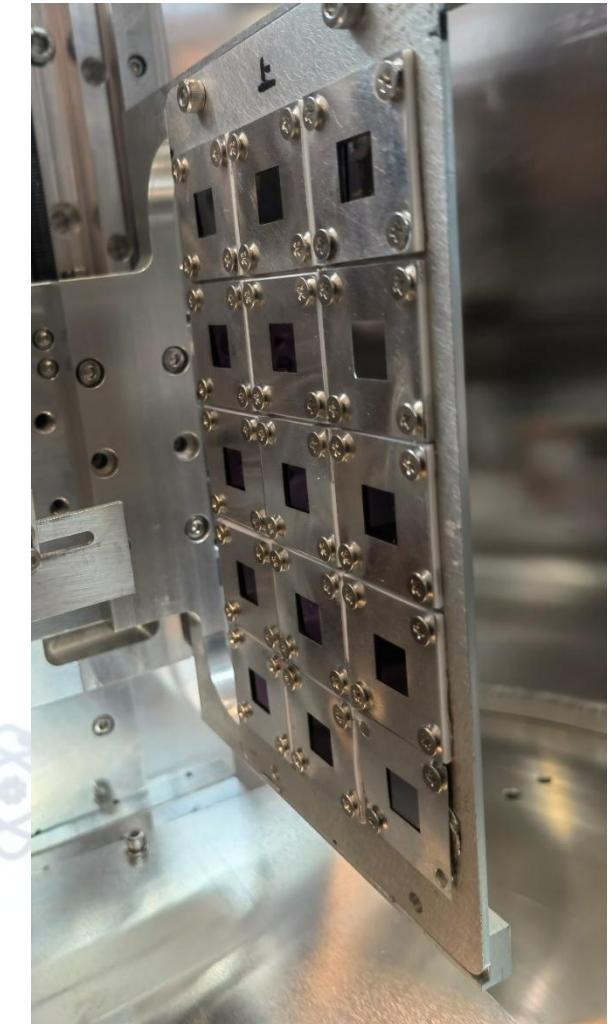
Manuscript submitted April 22, 2009; revised manuscript received July 21, 2009. Published September 14, 2009.

2009年，诺贝尔化学奖得主John B. Goodenough用NDP测量了磷酸铁锂性能和SEI层的形成过程，并预言这种技术在锂电池材料研发和性能评估上有重要作用。NDP已从离线测试发展成为原位/工况测试，成为锂电池一种重要表征方法。

## 中子深度剖面技术研究

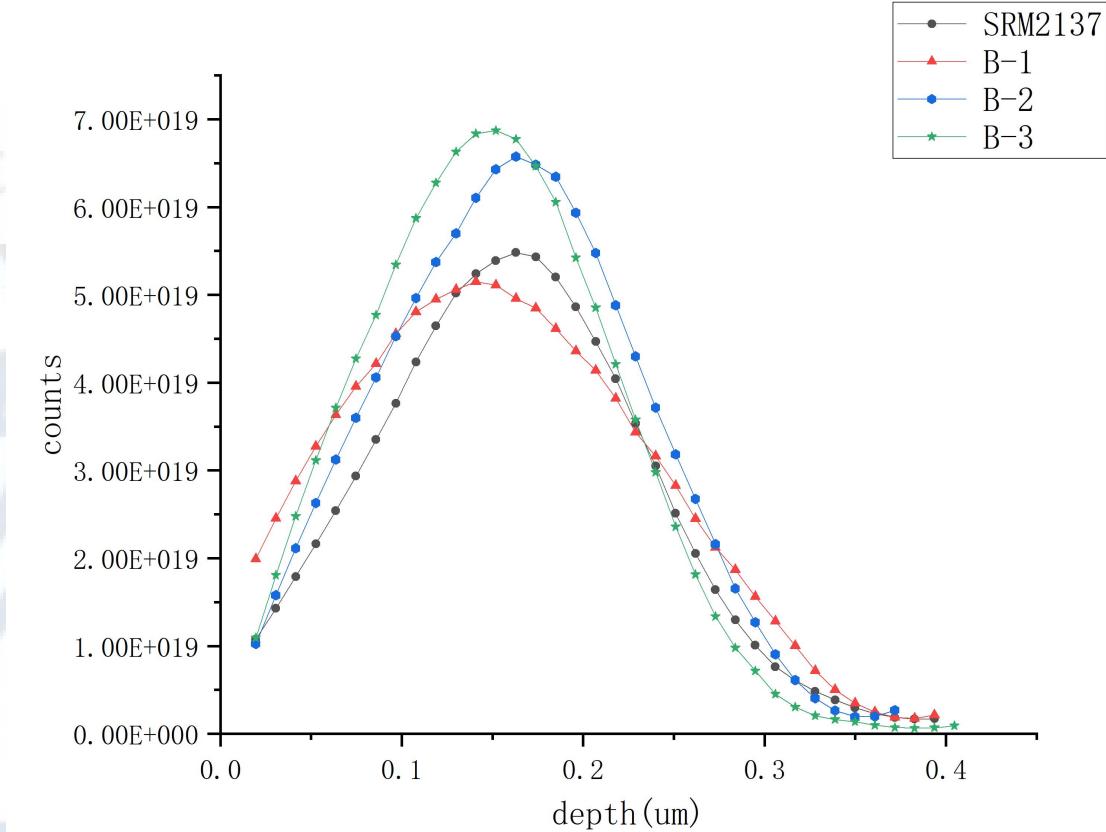
## 半导体样品批量测试分析：

- 口 实现分析不确定度小于1%，深度分辨小于10纳米，整体性能达到国际先进水平；
- 口 测试了外延片、离子注入、BPSG等半导体芯片制造不同过程的样品；
- 口 BPSG测试速度快，可达：1个/分钟；分析精度高；
- 口 这些半导体的NDP精确测试都属国内首次；
- 口 NDP技术对我国半导体生产制造有非常重要的意义。



## 中子深度剖面技术研究

已完成多批次样品分析，为**半导体掺杂**工艺改进和标准物质研制提供了精确测量数据和技术支撑。



B离子注入分布曲线

## 中子活化分析半导体相关标准

## NAA在半导体中能发挥重要作用：

- ① Si/SiC等基体**杂质元素分析**（体分析）；
- ② 光刻胶等半导体制程中的**材料分析**；
- ③ 高纯Cu、Al等**金属靶材杂质元素分析**；
- ④ B、As、P、N等**掺杂元素分析**；
- ⑤ 半导体材料中H的分析；
- ⑥ U、Th、K、Pb等**放射性元素/核素分析**；

ICS 77.040  
H 17中华人民共和国国家标准  
GB/T 32277—2015

## 硅的仪器中子活化分析测试方法

Test method for instrumental neutron activation analysis (INAA) of silicon

SEMI PV10-1110  
TEST METHOD FOR INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS (INAA) OF SILICON

This standard was technically approved by the global Photovoltaic Committee. This edition was approved for publication by the global Audits & Reviews Subcommittee on August 27, 2010. Initially available at [www.semi.org](http://www.semi.org) in October 2010.

## 1 Purpose

1.1 Neutron Activation Analysis (NAA) is a highly sensitive method for multi-element quantitative and qualitative analysis of various materials.<sup>1</sup> In particular, it is an established method in the semiconductor industry for analyzing and qualifying poly-silicon (poly-Si), the feedstock for growing semiconductor grade single crystalline silicon (c-Si) as well as silicon wafers, regarding trace element contamination. The increasing demand for feedstock Si in the photovoltaic industry resulted in a higher volume of production of poly-Si and in developing alternative methods for purifying raw Si, resulting in materials that are generically called solar grade silicon (sog-Si) in the present document. This material may occur in a variety of shapes, like chunks, powder, and granules.

1.2 The growing volume of production of poly-Si and sog-Si requires also an increase of quality control for these materials, for which Instrumental Neutron Activation Analysis (INAA) is one method of choice. INAA is an NAA where the analysis is conducted directly on the irradiated samples, in contrast to Radiochemical Neutron Activation Analysis (RNAA) where the irradiated samples are subjected to chemical separation for removing interfering species or for concentrating the radioisotope of interest.<sup>1</sup> Currently INAA is performed by laboratories using different irradiation sources and conditions, electronic equipment and preparation methods, and it is applied to a wide morphological variety of Si. Standardizing INAA for application to silicon will remove differences in the analytical approach and will establish a common reference for the analyses.

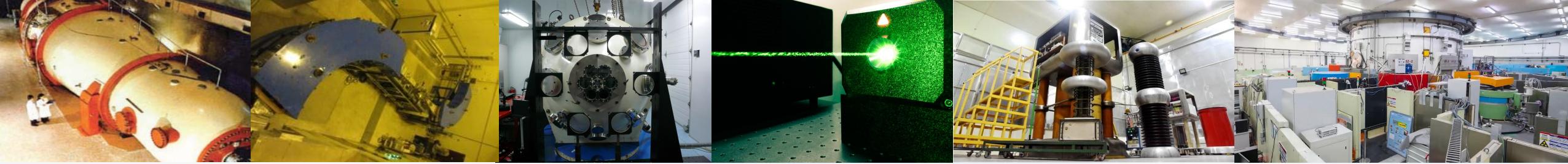
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## 二. 中子深度剖面分析技术研究进展

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6. 石丛, 肖才锦\*等, Inverse iteration algorithm for neutron depth profiling, J Radioanal Nucl Chem (2018) 297 (3) ;
7. 石丛, 肖才锦\*等, 中子深度剖面技术的反演迭代计算, 同位素, 2017 30 (3) ;

- NAA具有高准确度、非破坏、多元素等特点；
- NAA在**月壤**等地外样品分析中发挥重要作用；
- 在地质、考古、标准物质、材料等学科有着广泛的应用；
- NDP技术已成为**锂电池**、**半导体**一种重要测试表征手段；



请各位批评指正！

谢谢！

以身许国  
敢为人先 严谨求实