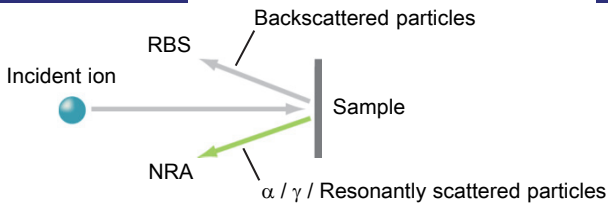


RBS/NRA depth profile of LIB negative electrode after charge – discharge cycles

Lithium Ion Battery (LIB) negative electrode can be analyzed by RBS / NRA method. Keeping charge - discharge state, depth profile can be obtained from surface to bulk. Generally, the quantitative value is considered to be as accurate as ICP-AES.

Principle



- RBS : Rutherford Backscattering Spectrometry
 - Accurate composition depth profile
- NRA : Nuclear Reaction Analysis
 - Li quantification (information depth : ~ 30 μm)

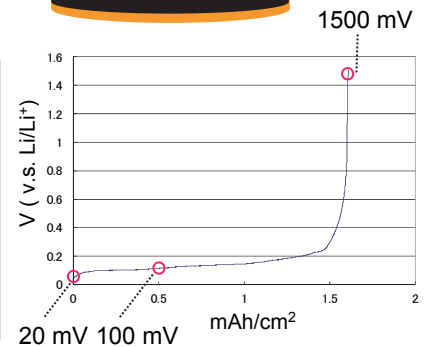
RBS, NRA : do not need ion etching for depth profile
 → **Accurate depth information without sample degradation**

Charge - discharge characteristic

OSample

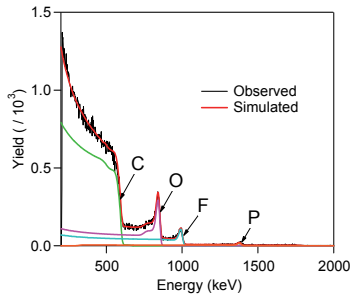
LIB negative electrode : Graphite

Charging potential (v.s. Li / Li ⁺)	State
20 mV	Fully Charged
100 mV	Intermediate
1500 mV	Fully Discharged

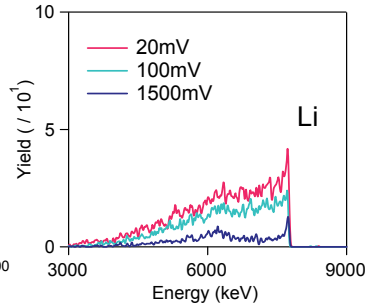


Adjusted charging potential, each sample is introduced into RBS / NRA instrument, **without air exposure**.

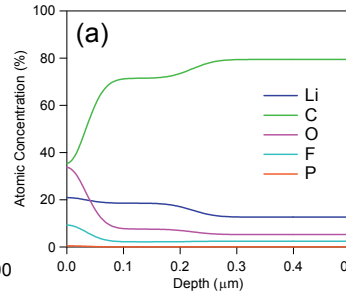
Composition of electrode at each charging potential



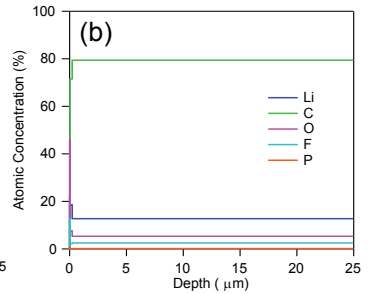
RBS spectrum (20 mV)



NRA spectra



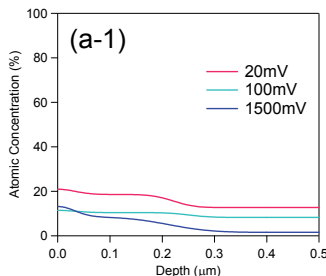
Depth profile (20 mV) (a) : surface, (b) : bulk



(a) Li, O, F, P concentration is high at surface region

(b) Li concentration in deeper region is constant

(a) Surface



Comparison of (a-1) : Li, (a-2) : O depth profile

- **Li concentration** : gradually decrease till ~ 250 nm
- **O concentration at surface** : 20 mV, 100 mV > 1500 mV

(b) Bulk composition

Charging potential [mV]	atomic%					C / Li	Estimated structure
	Li	C	O	F	P		
20	12.7	79.4	5.3	2.5	0.1	6.3	LiC ₆
100	8.3	84.2	5.4	2.0	0.1	10.1	LiC ₁₂
1500	1.6	93.5	2.1	2.7	0.1	58.8	C

bulk composition (atomic%), and C/Li ratio

• **High accurate composition**※ → comparable with structure directly

※ General accuracy of RBS/NRA analysis : ~±3%

RBS / NRA - Accurate depth profile can be obtained, from surface to bulk region