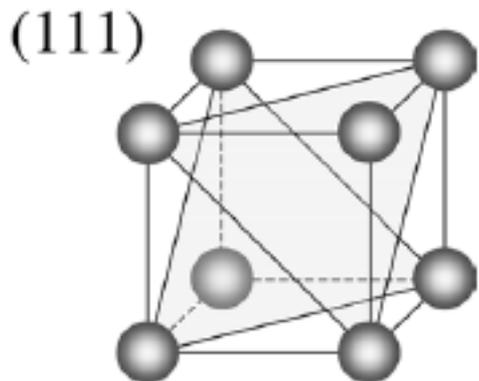
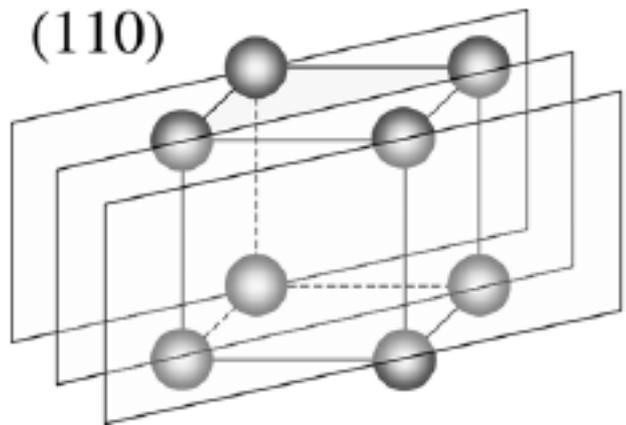
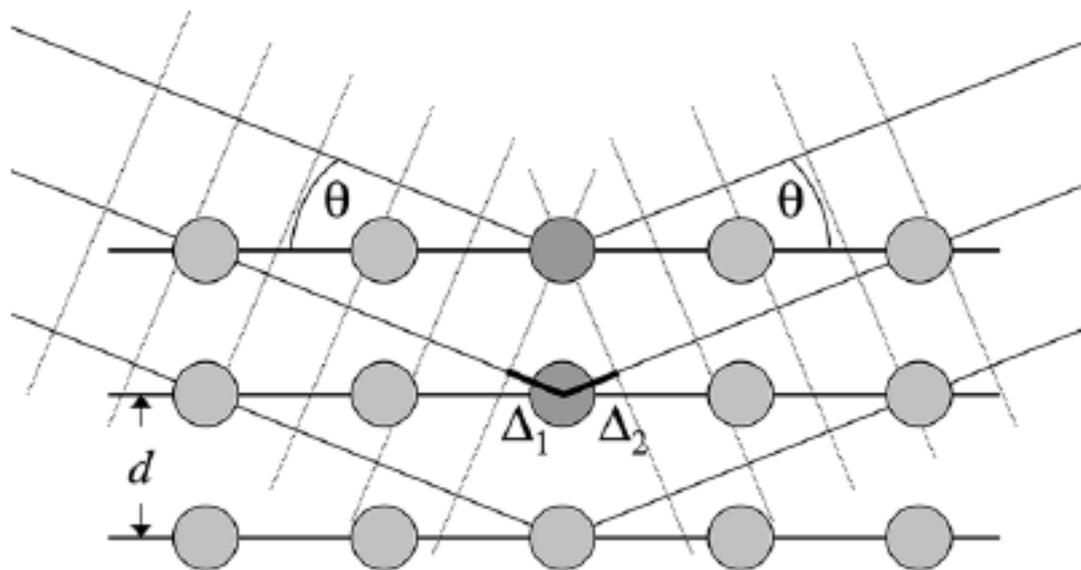


Методы, основанные на использовании рентгеновского излучения, нейтронов, электронного пучка

- фазовый анализ твердых материалов
- особенности для частично разупорядоченных кристаллов и малых частиц
- возможности для исследования полимеров, растворов и гетерогенных жидкостей
- организация *in situ* эксперимента



$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$



$$2d \sin \theta = \lambda$$

Межплоскостные расстояния d

Тип решетки, фазовый состав

Bismuth Selenide (Paraguanajuatite), Bi_2Se_3

CAS registry no.
12068-69-8

Sample
The sample was obtained from Alfa Products,
Thiokol/Ventron Division, Danvers, MA.

Color
Unground, gray metallic
Ground, dark gray

Structure
Hexagonal, $R\bar{3}m$ (166), $Z = 3$, isostructural
with tetradymite, $\text{Bi}_2\text{Te}_2\text{S}$. The structure
of Bi_2Se_3 was refined by Nakajima [1963],
following earlier work by Semiletov and
Pinsker [1955].

Lattice constants of this sample

$a = 4.1396(4)\text{Å}$
 $c = 28.636(4)$

$c/a = 6.9176$

Volume
 424.97Å^3

Density
(calculated) 7.676 g/cm^3

Figure of merit
 $F_{30} = 54.4(0.012, 44)$

$$2d \sin \theta = \lambda$$

CuK α_1 $\lambda = 1.540598\text{Å}$; temp. $25 \pm 1\text{°C}$ Internal standard Ag, $a = 4.08651\text{Å}$			
$d(\text{Å})$	I^{rel} $\sigma = \pm 1$	hkl	$2\theta(\text{°})$
9.56	7	0 0 3	9.24
4.777	19	0 0 6	18.56
3.559	16	1 0 1	25.00
3.478	3	0 1 2	25.59
3.205	7	1 0 4	27.81
3.183	4	0 0 9	28.01
3.040	100	0 1 5	29.36
2.698	1L	1 0 7	33.18
2.534	2	0 1 8	35.39
2.386	1L	0 0 12	37.67
2.238	26	1 0 10	40.27
2.106	9	0 1 11	42.92
2.070	26	1 1 0	43.70
1.9085	8	0 0 15	47.61
1.8998	8	1 1 6	47.84
1.8780	2	1 0 13	48.43
1.7893	2	0 2 1	51.00
1.7782	1L	2 0 2	51.34
1.7392	2	0 2 4	52.58
1.7349	2	1 1 9	52.72

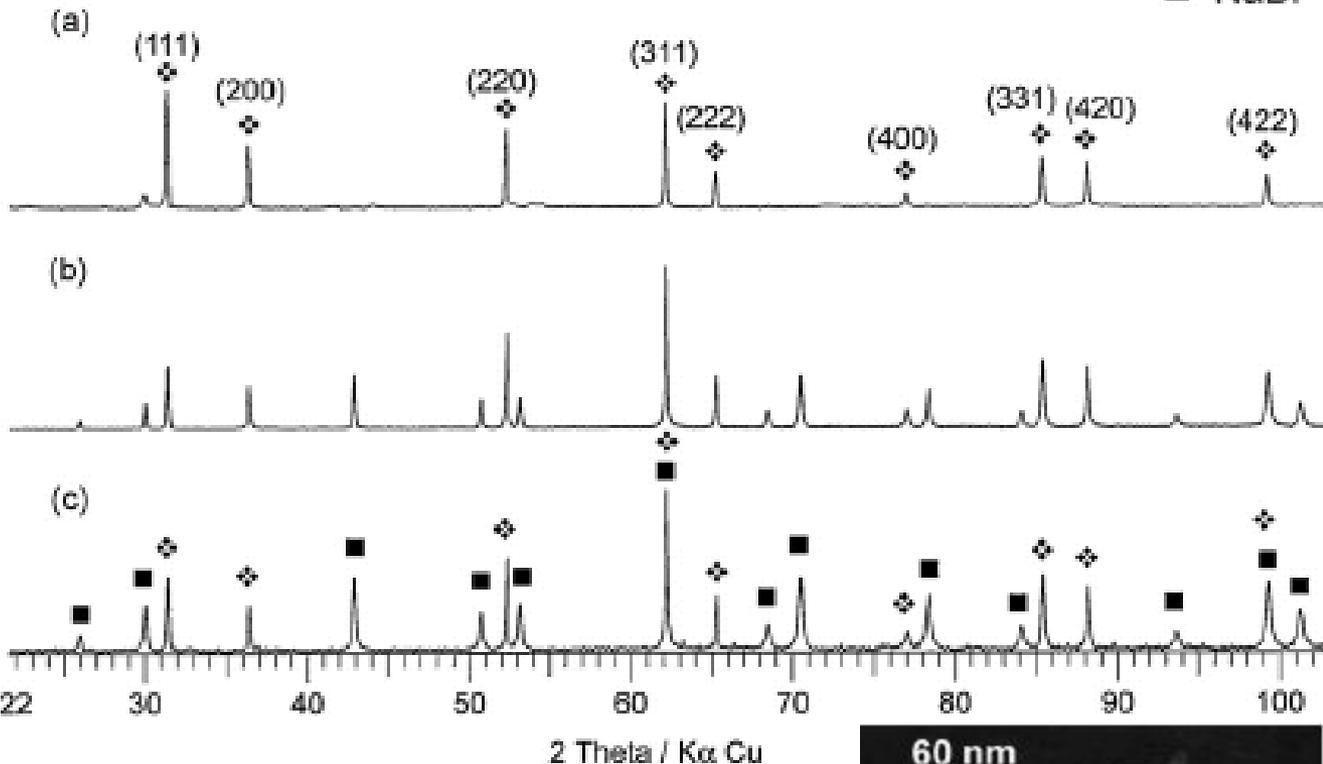
Коллоидный металл

Свинец

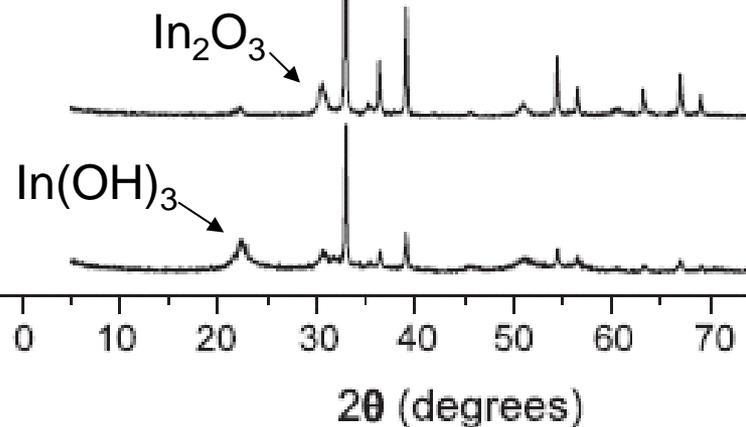
◆ Pb

■ NaBr

Окисление
(примеси воды)



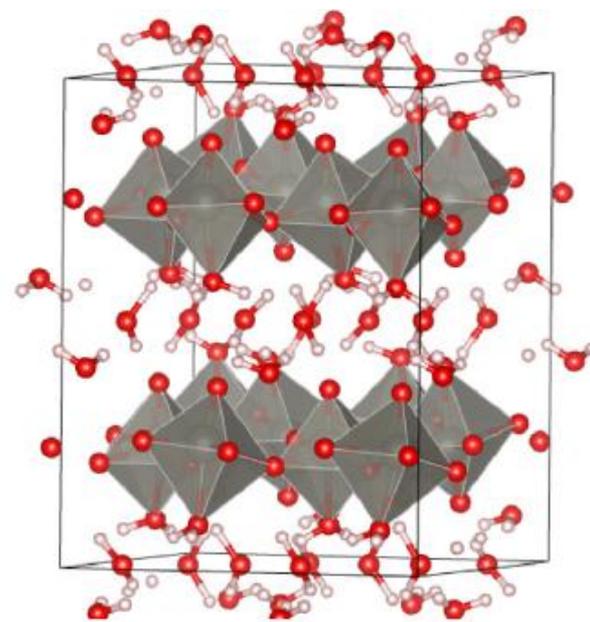
Индий



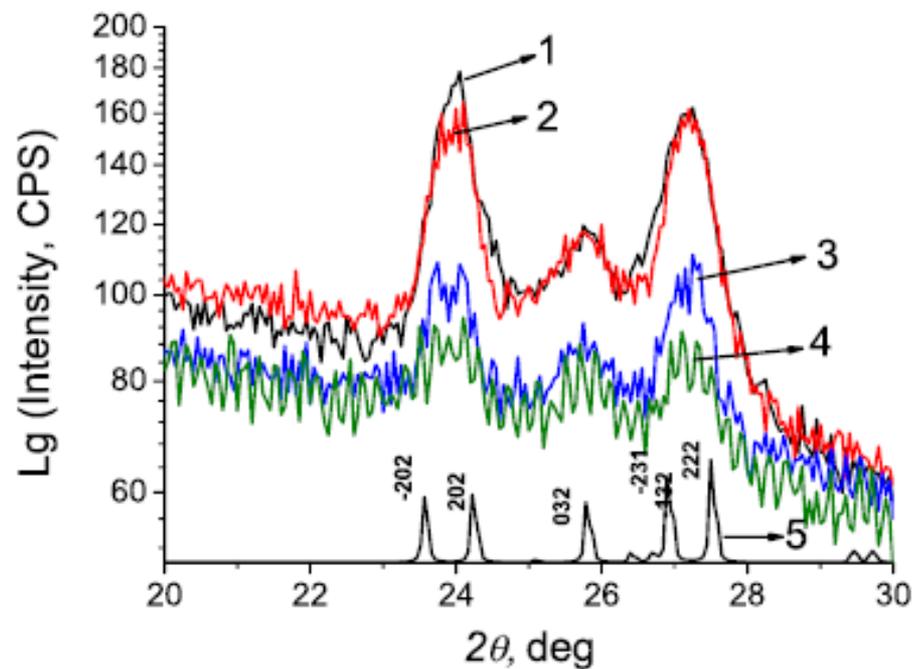
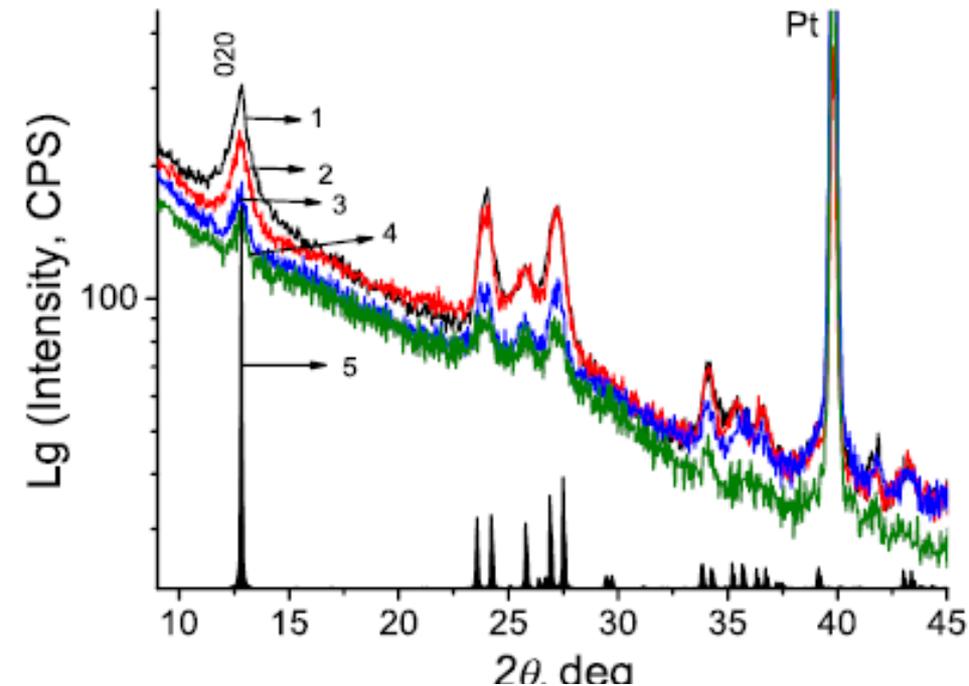
Загрязнение
компонентами
раствора синтеза



**Тонкая пленка на подложке
(электроосажденный оксид
вольфрама)**



Кристаллизация при старении



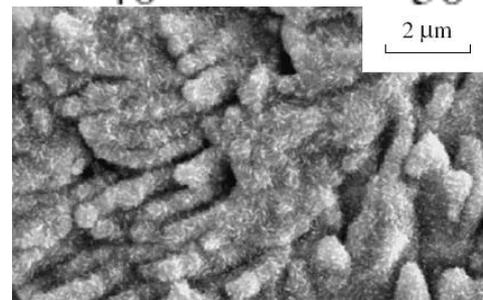
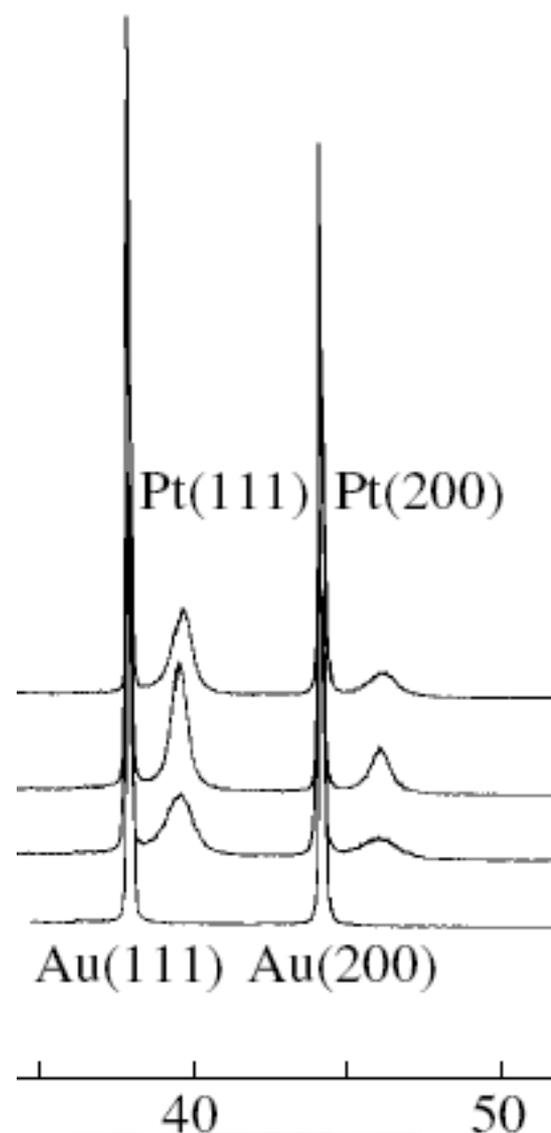
$$\beta \cdot \cos\theta = \frac{\lambda}{D} + 4\varepsilon \cdot \sin\theta$$

↑
интегральная
ширина

↑
размер областей
когерентного рассеяния

↑
Микронапряжения в решетке

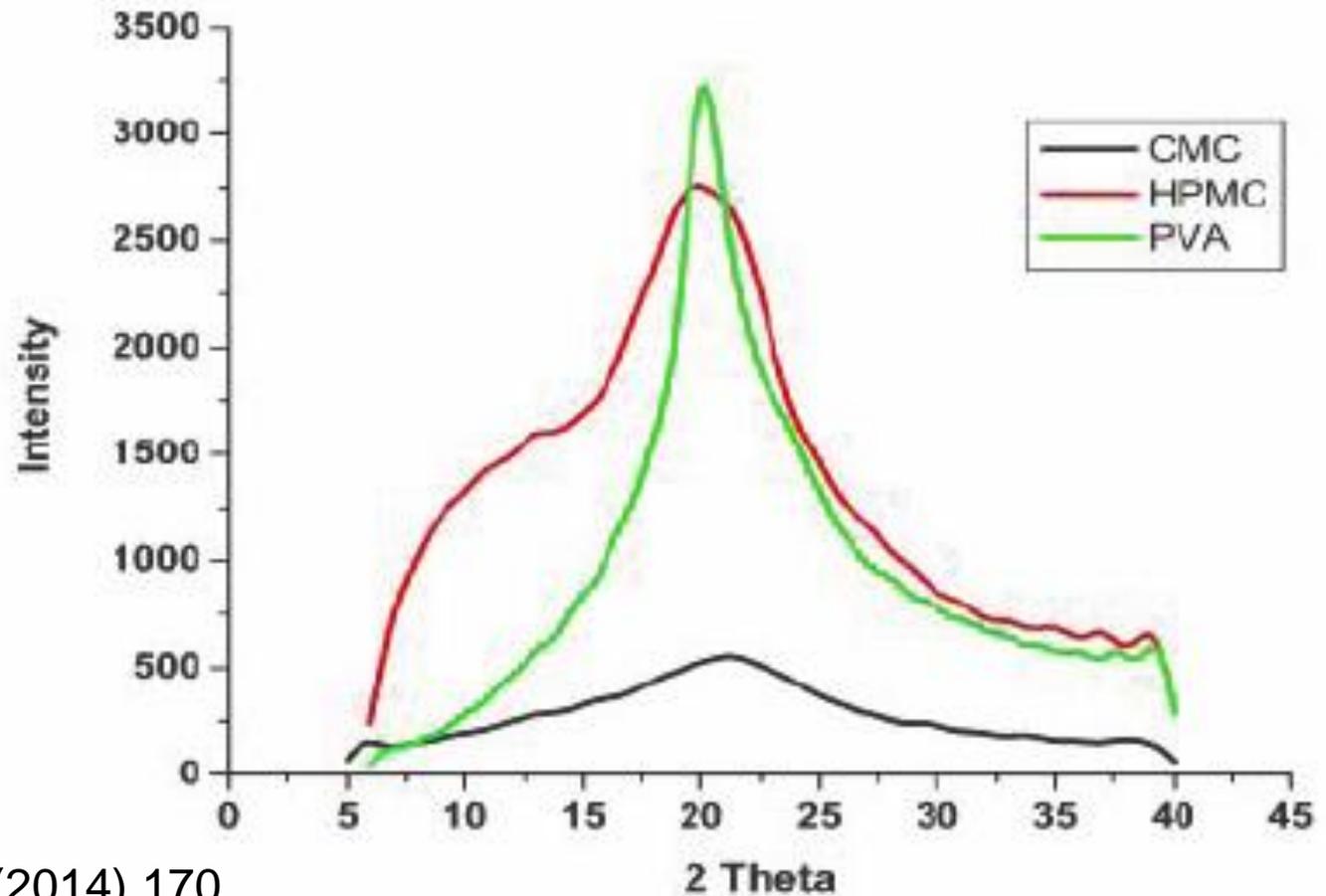
Function	$B_{\text{int}(111)}$	$B_{\text{int}(222)}$, deg 2θ	D , nm	$\Delta d/d$
Gauss	0.790	1.491	15.4	0.0059
Lorentz	1.112	2.317	12.7	0.0099
Pseudo-Voigt	0.975	1.776	11.9	0.0068



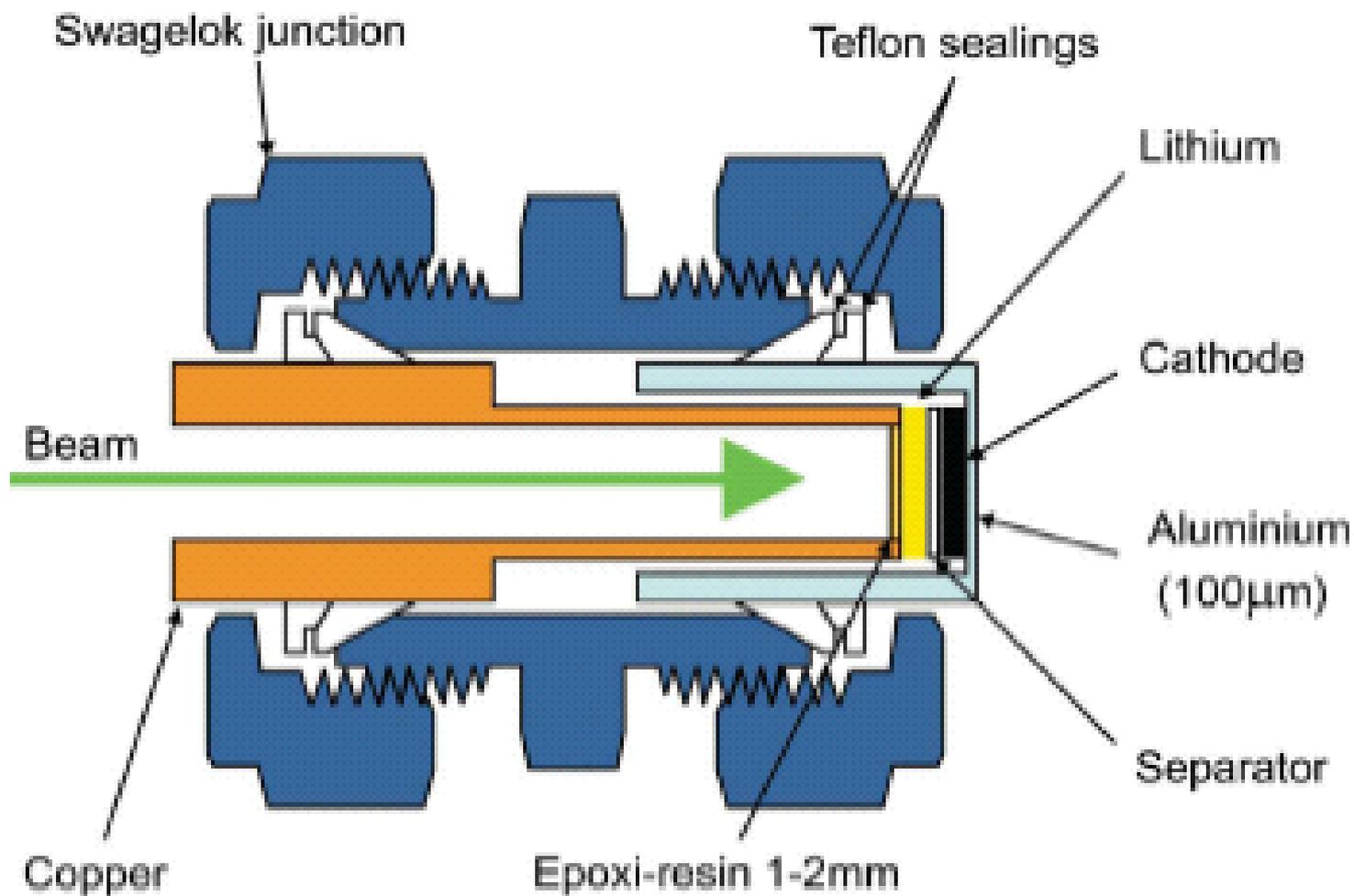
См. С.В.Цыбуля, С.В.Черепанова, Введение в структурный анализ нанокристаллов, НГУ, 2008

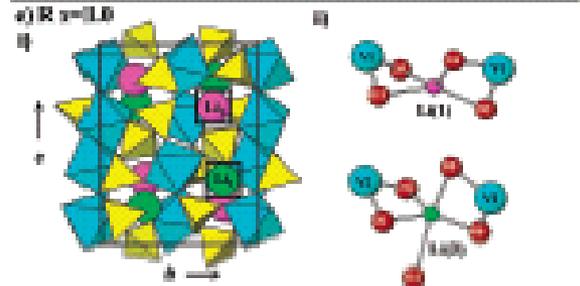
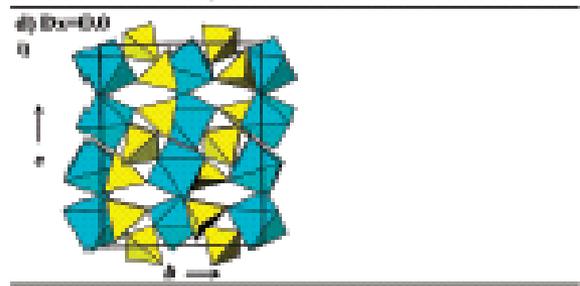
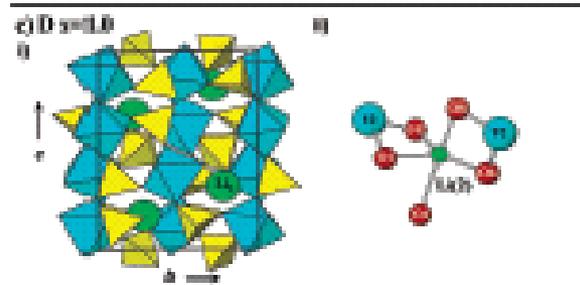
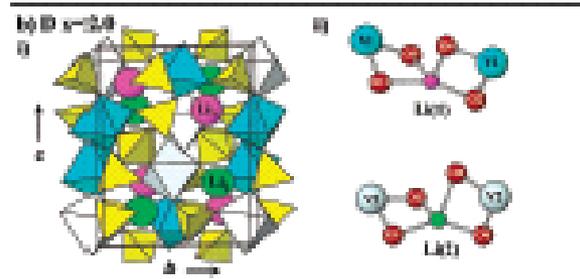
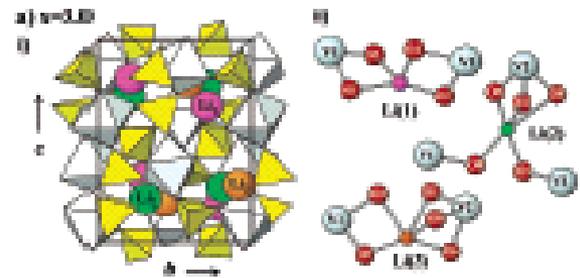
Полимеры

Sample	Average Crystallite size in Å	Average crystallite strain in %
CMC	18.8 ± 0.3	$(6.2 \pm 0.13)\%$
HPMC	19.1 ± 0.5	$(7.1 \pm 0.32)\%$
PVA	70.8 ± 0.8	$(6.5 \pm 0.27)\%$

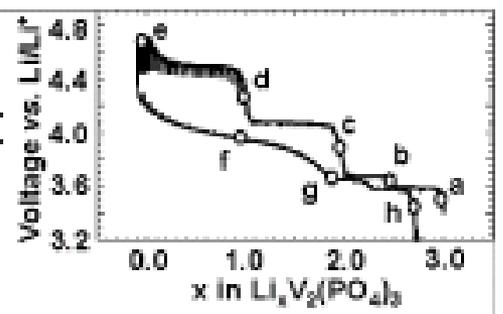


In situ дифракция

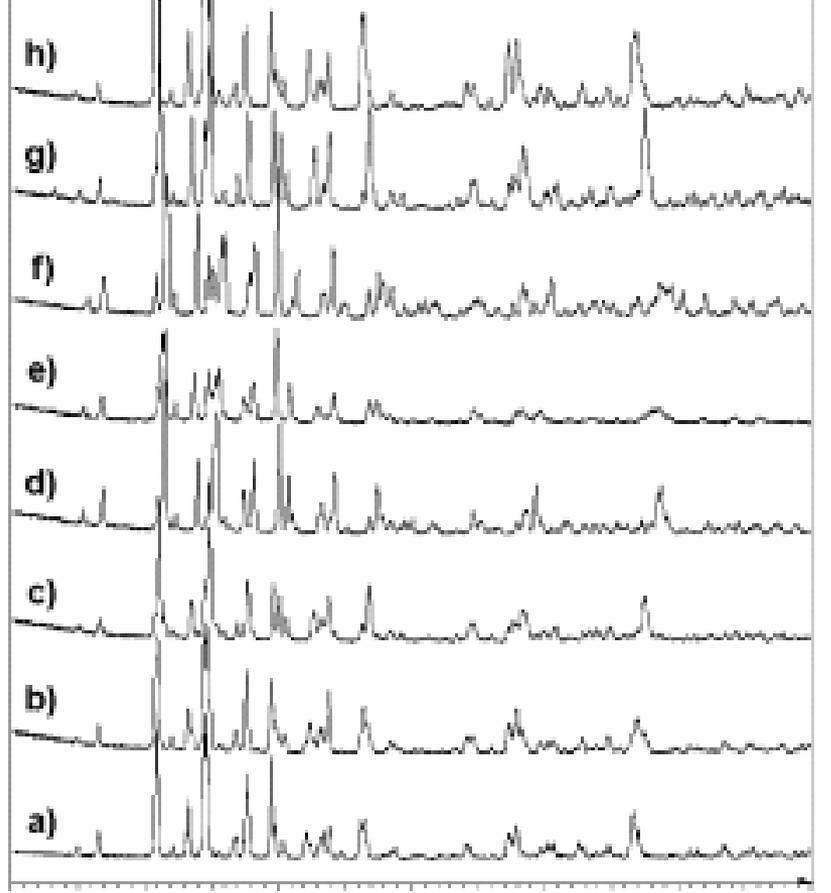




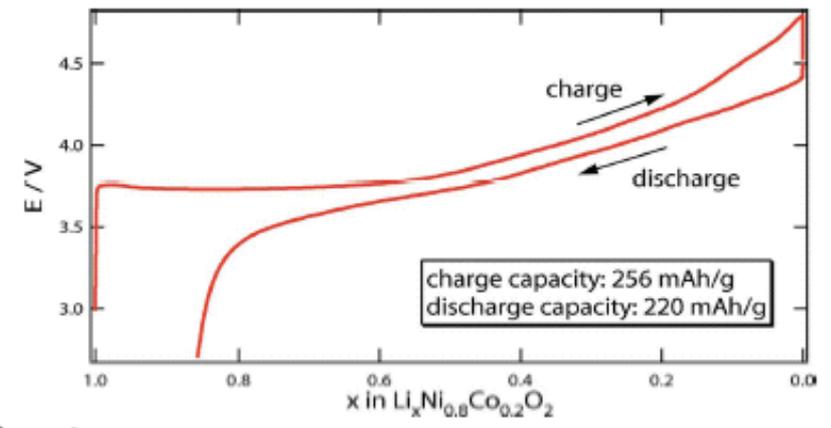
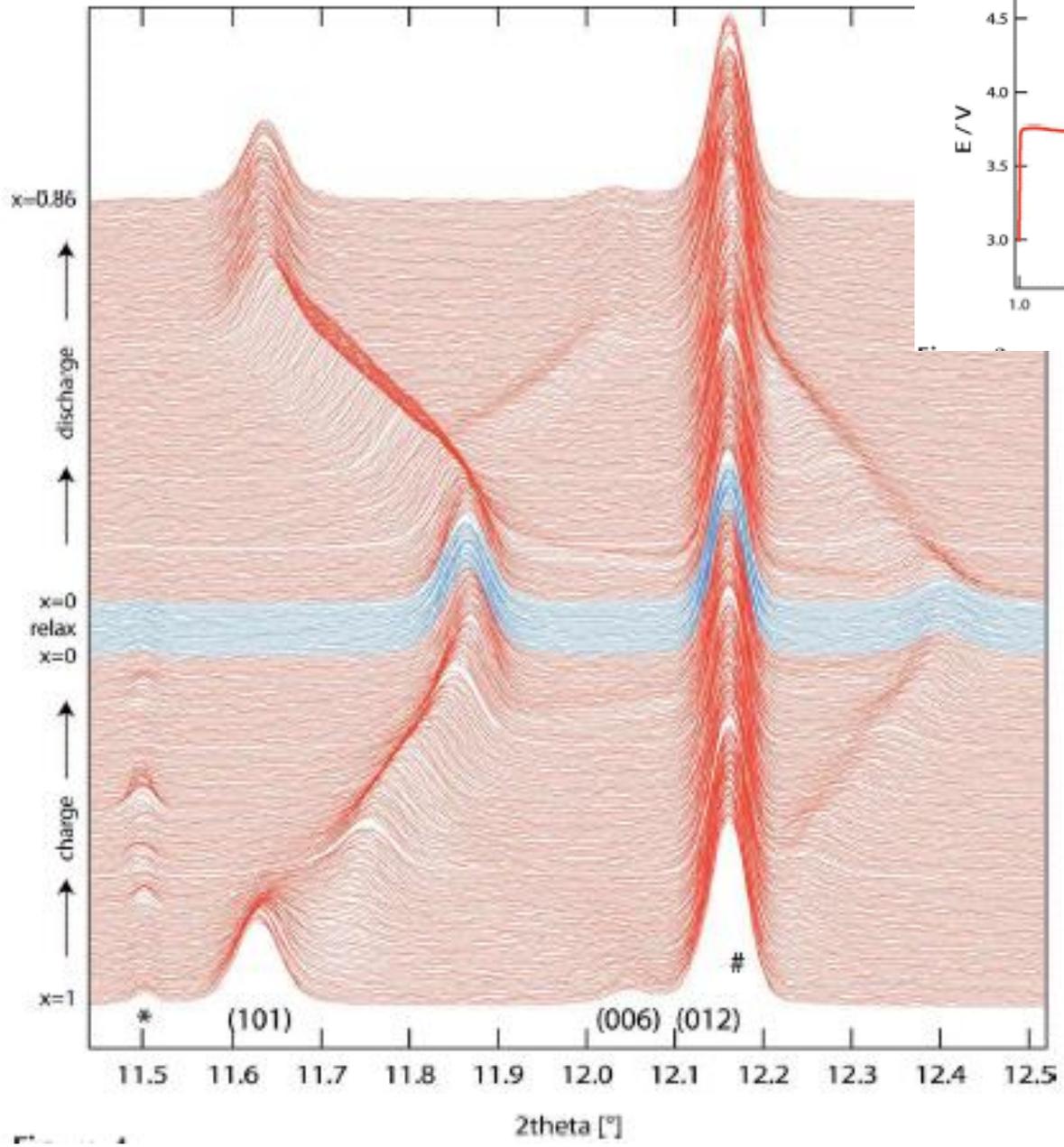
$\text{Li}_3\text{V}_2(\text{PO}_4)_3$:



Intensity (a. u.)



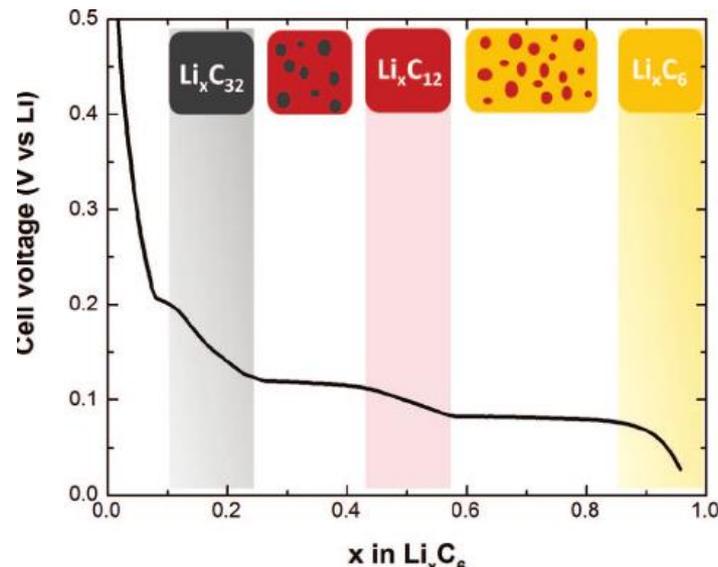
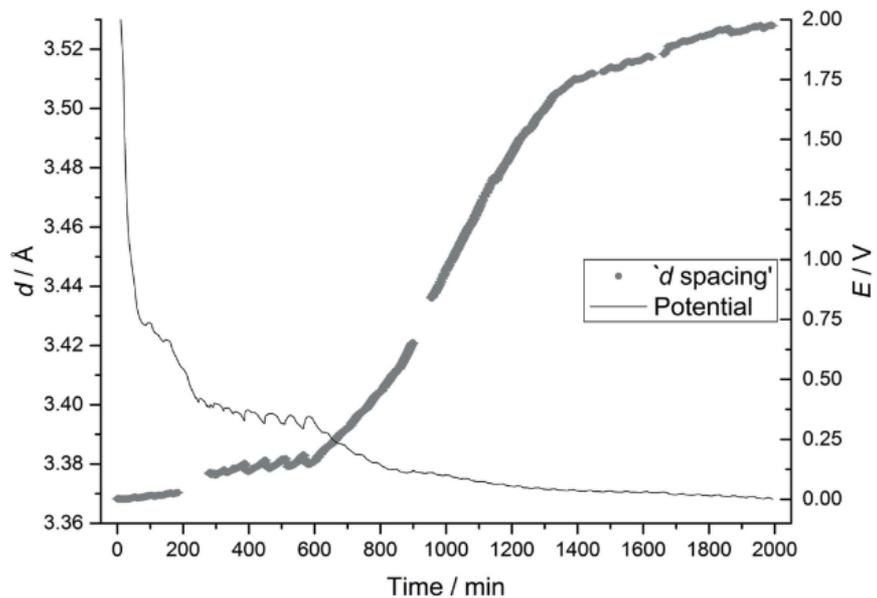
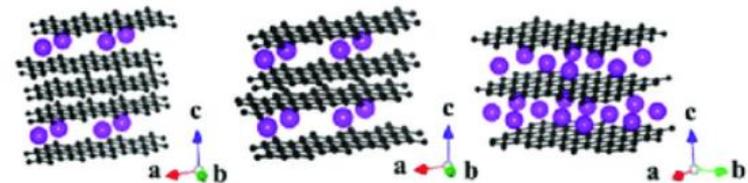
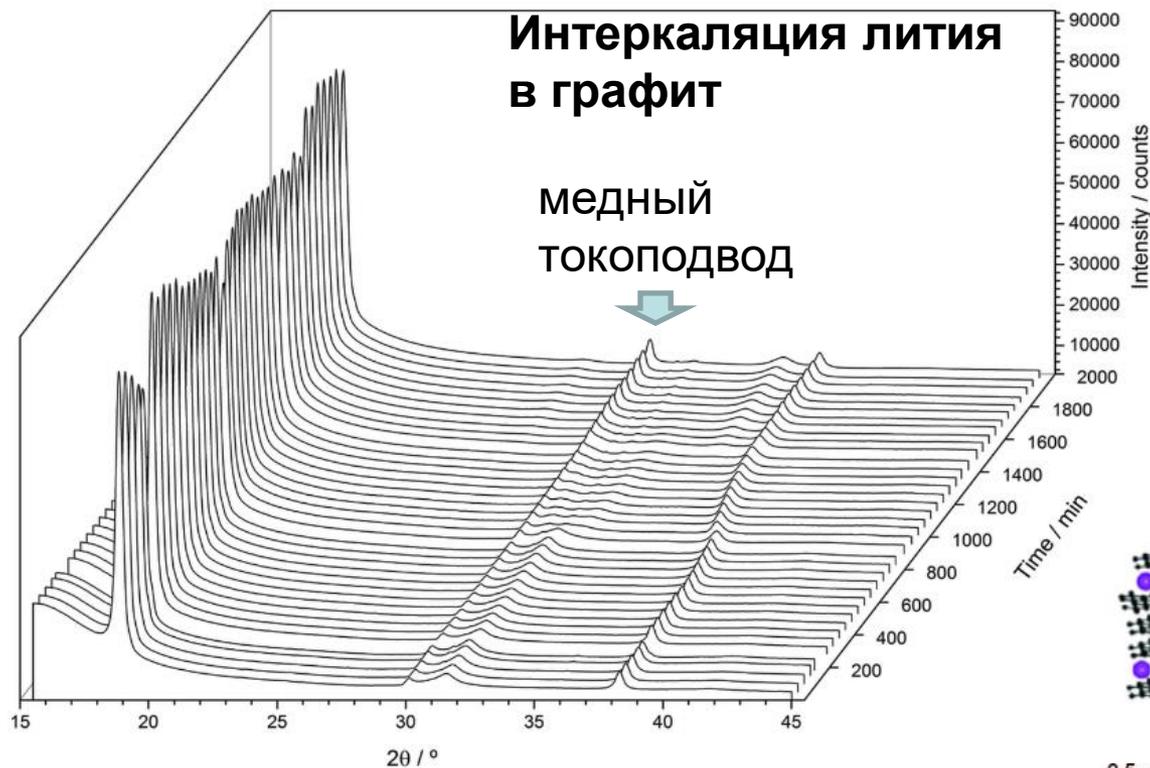
Angle 2θ (degrees)

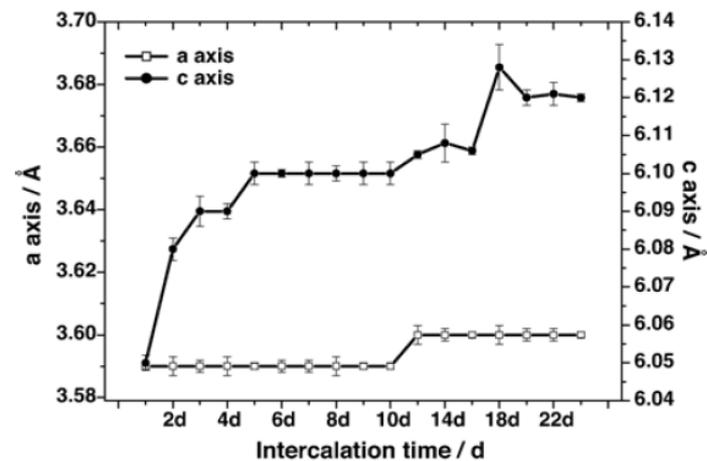
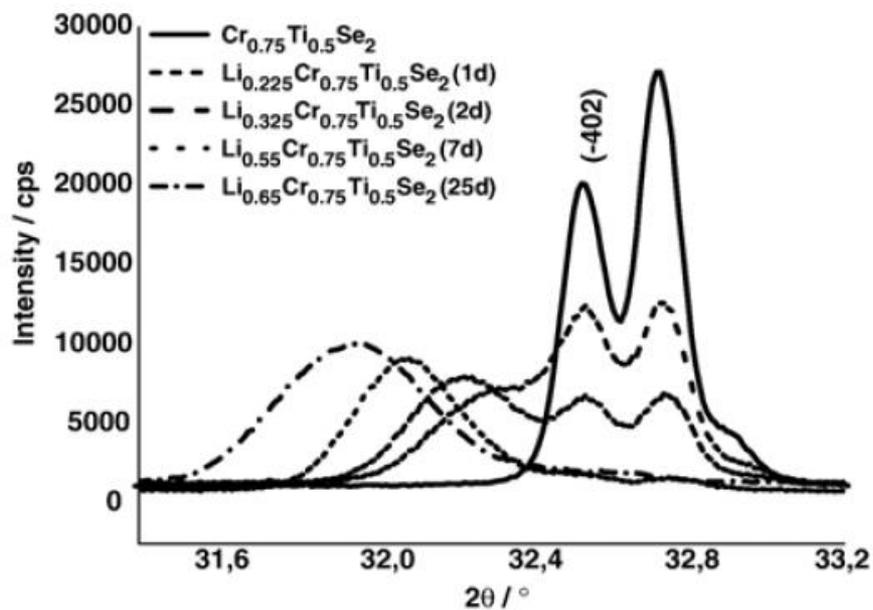
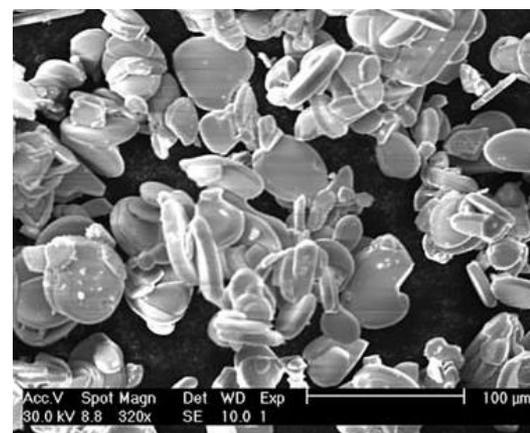
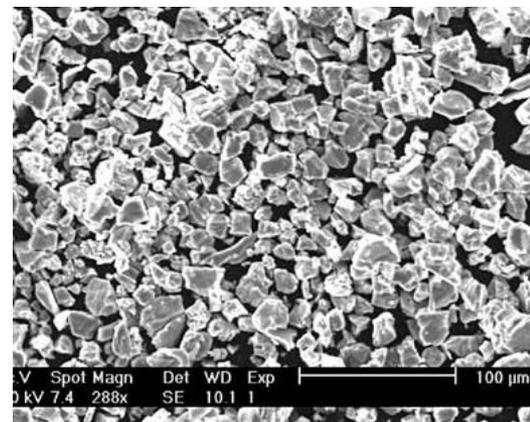
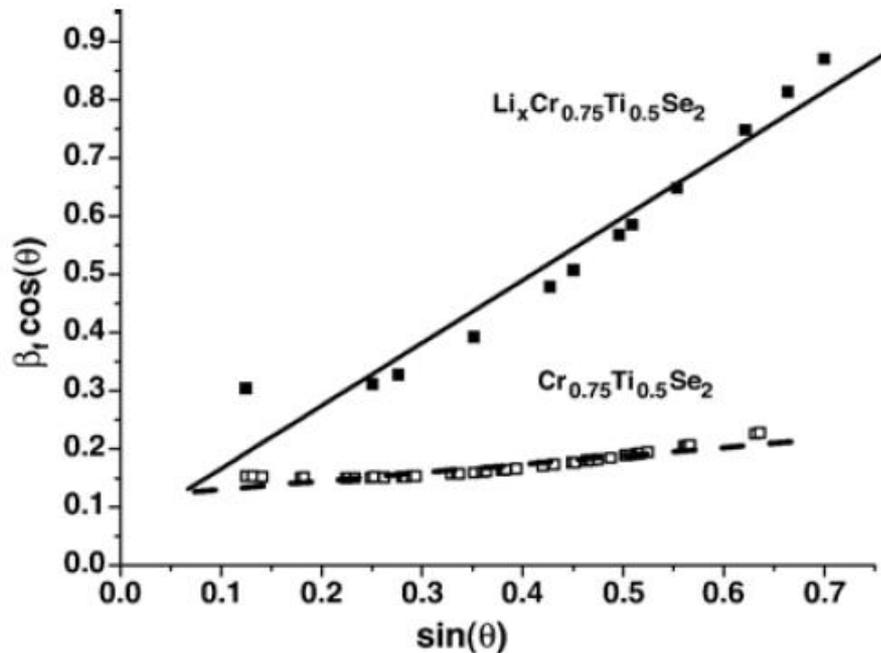


In situ дифракция

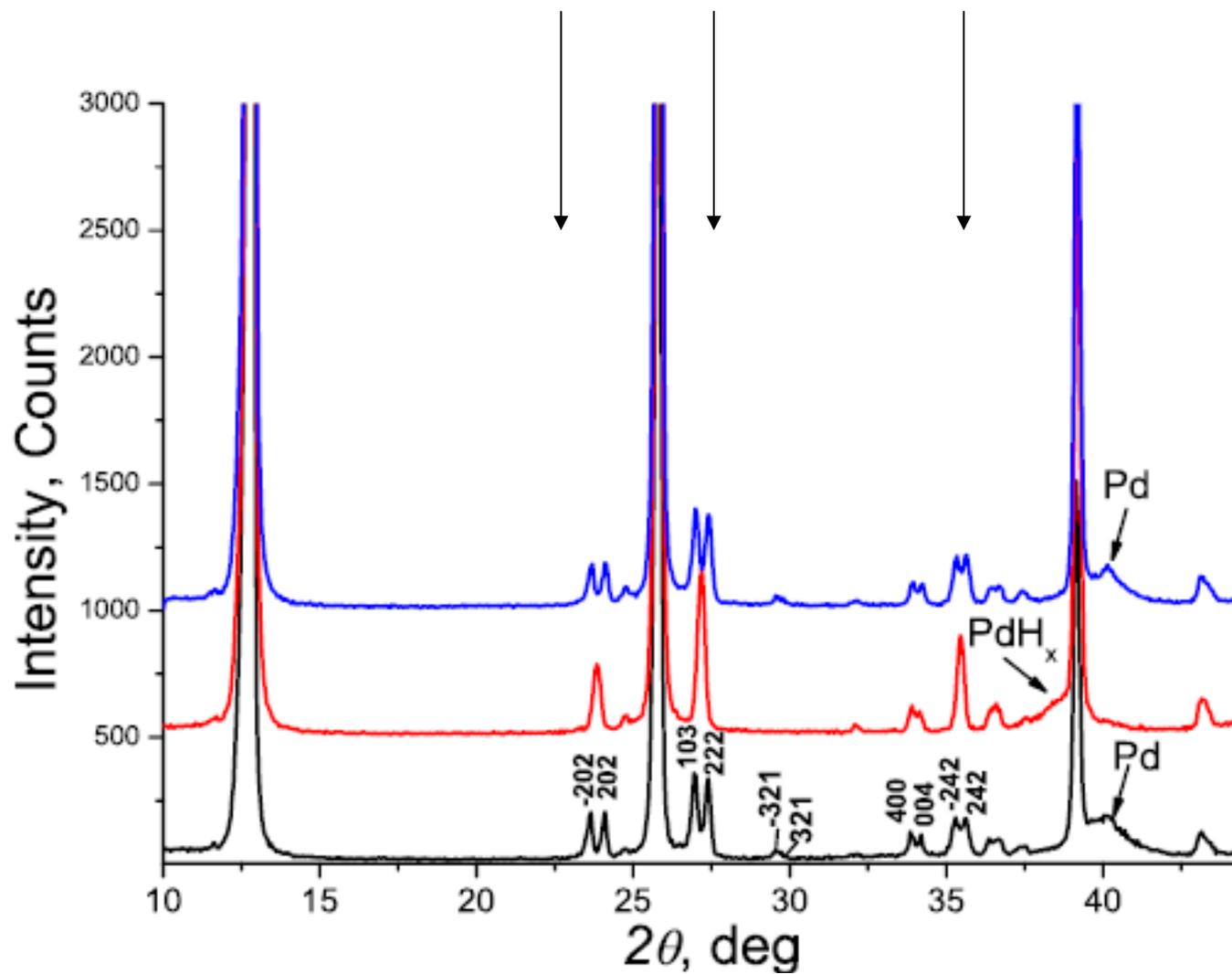
Интеркаляция лития в графит

медный
ТОКОПОДВОД

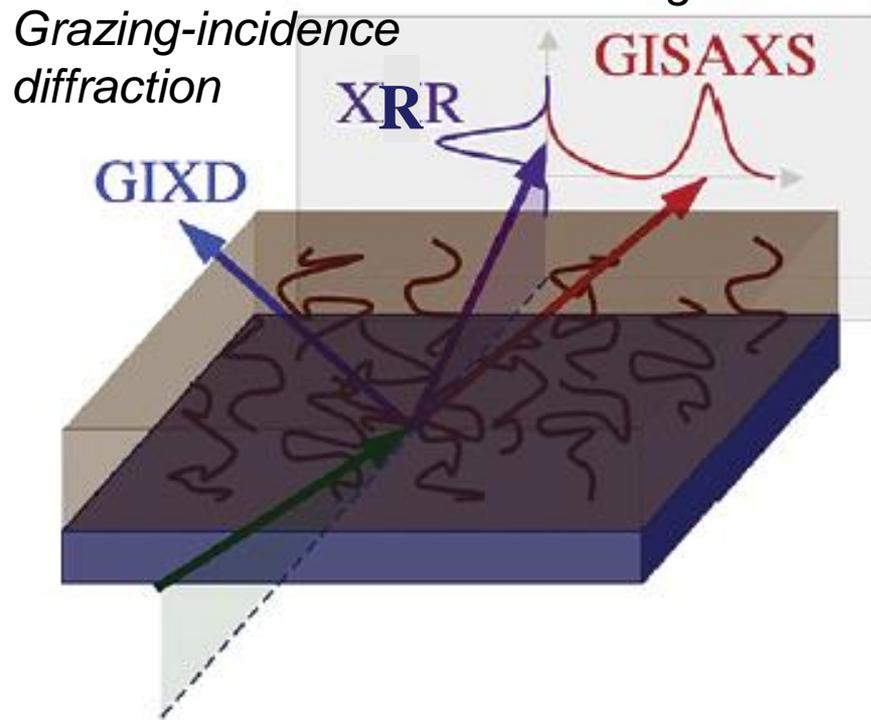
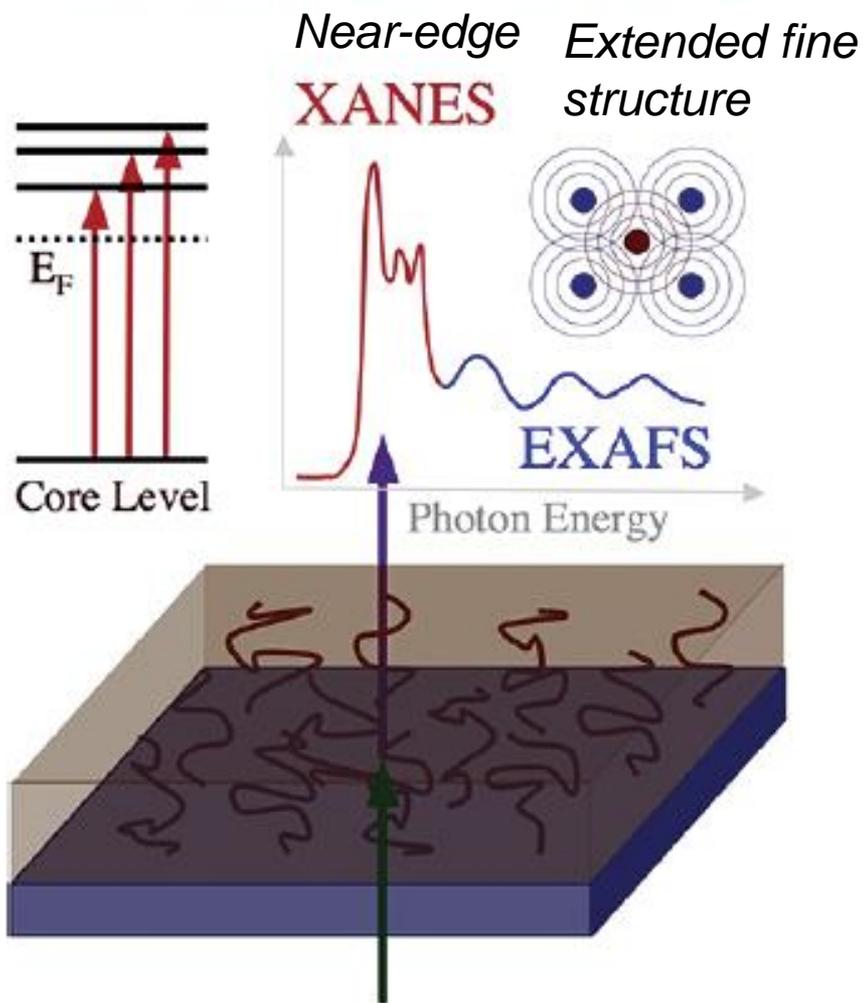




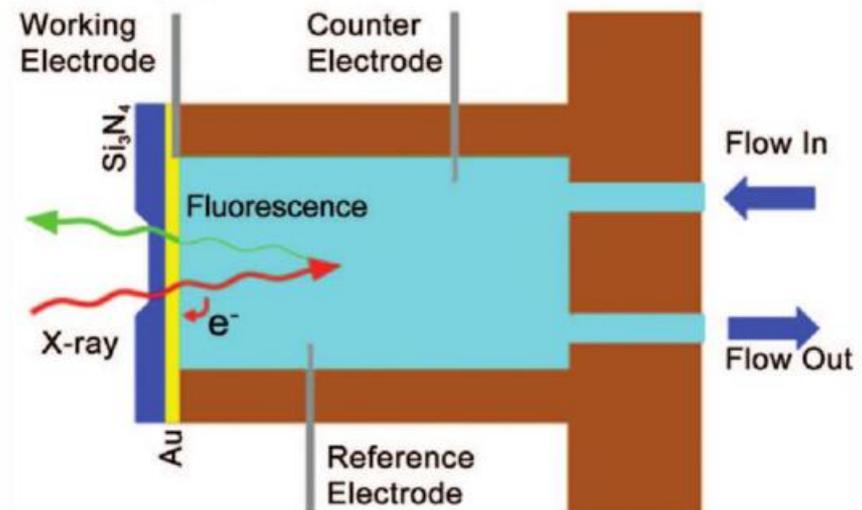
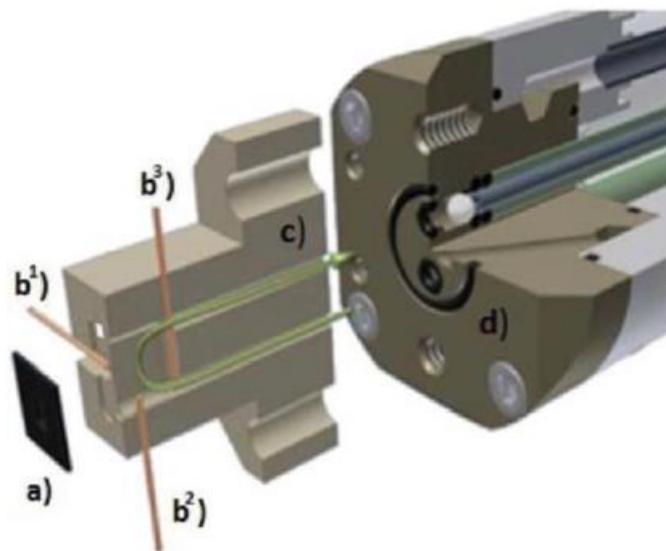
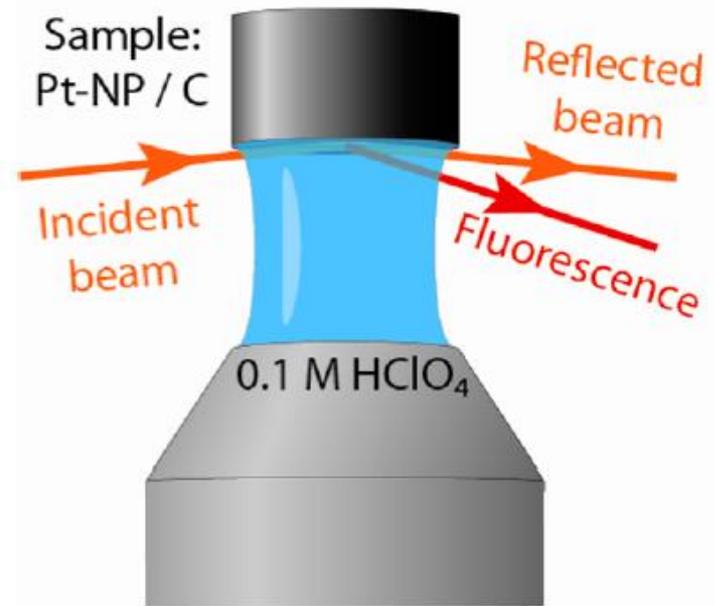
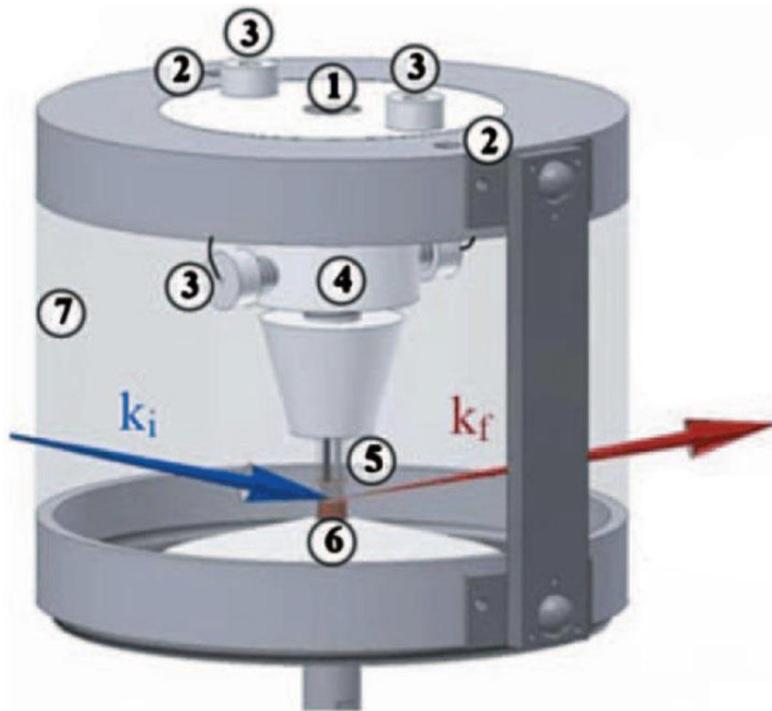
In situ XRD в газовой фазе: гелий – водород - гелий



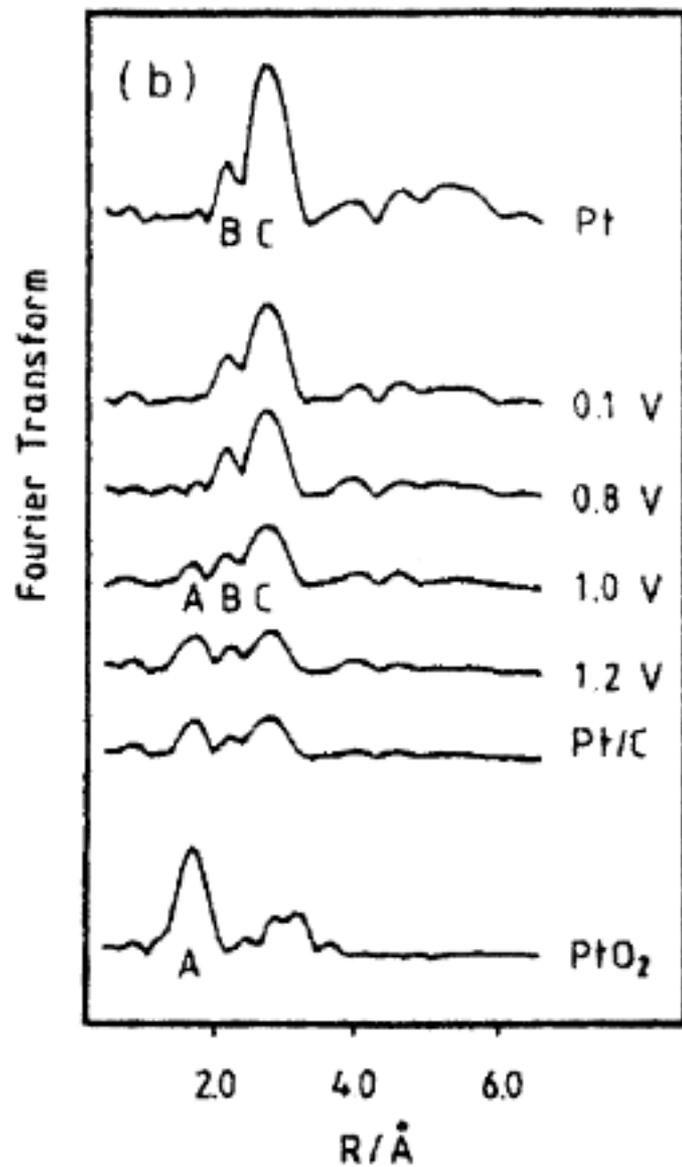
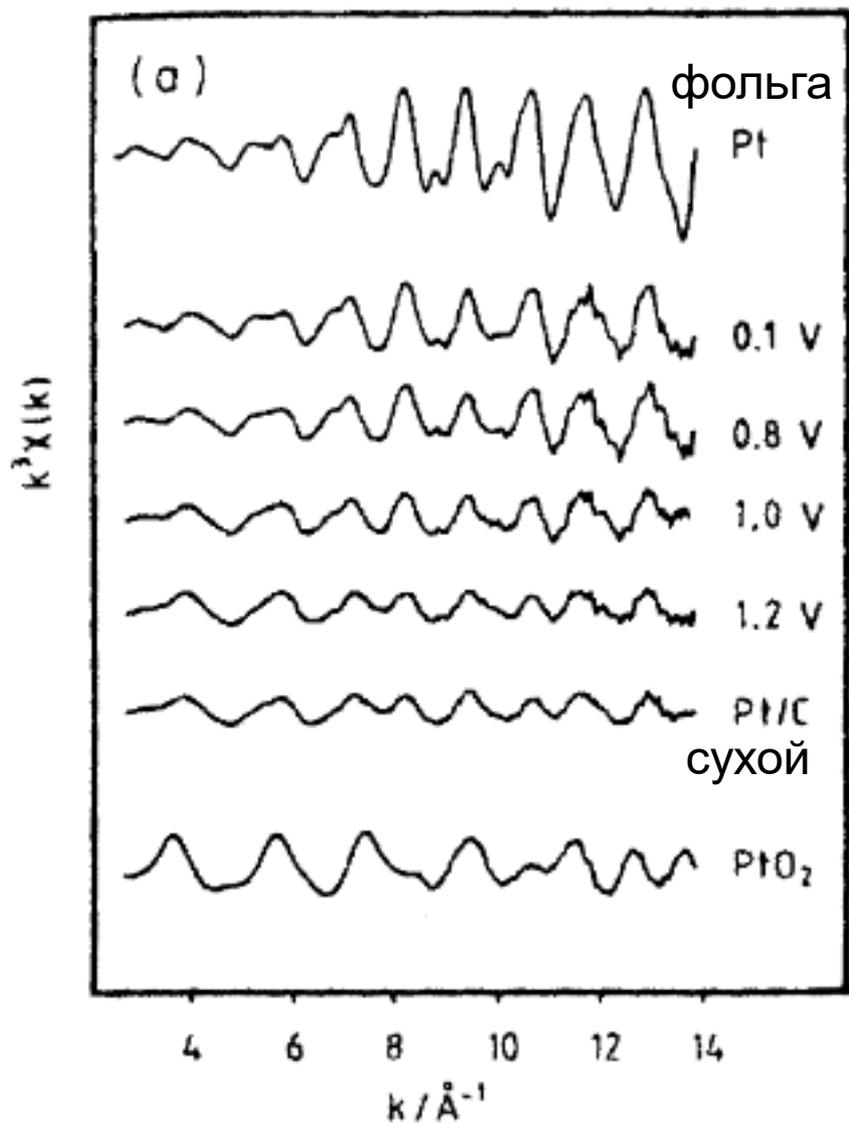
X-Ray Absorption



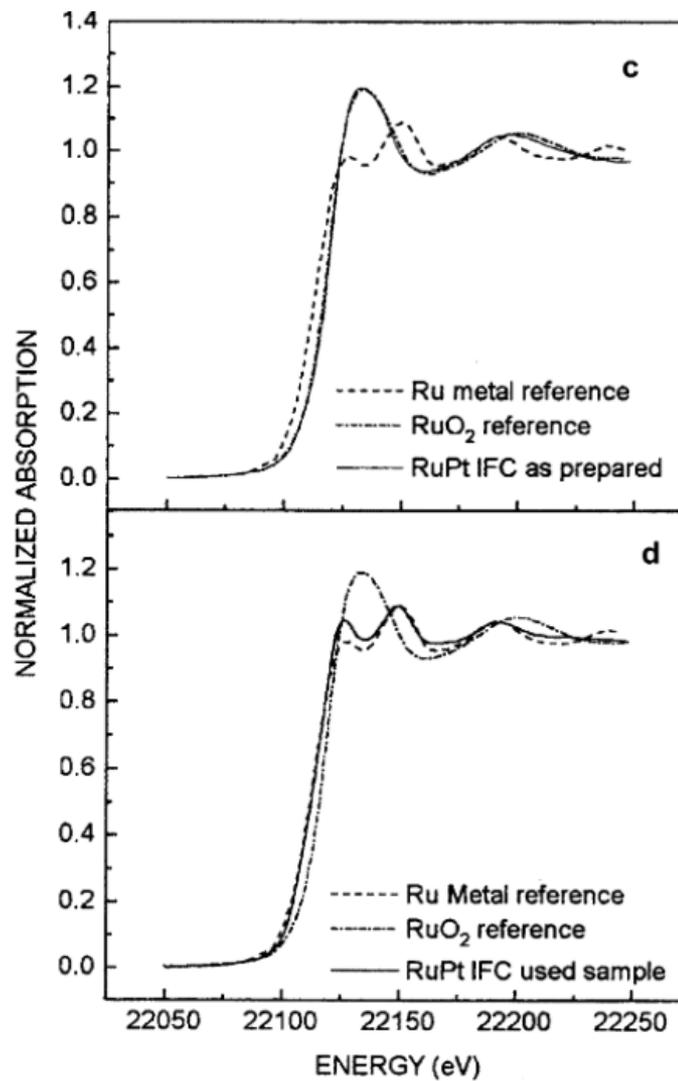
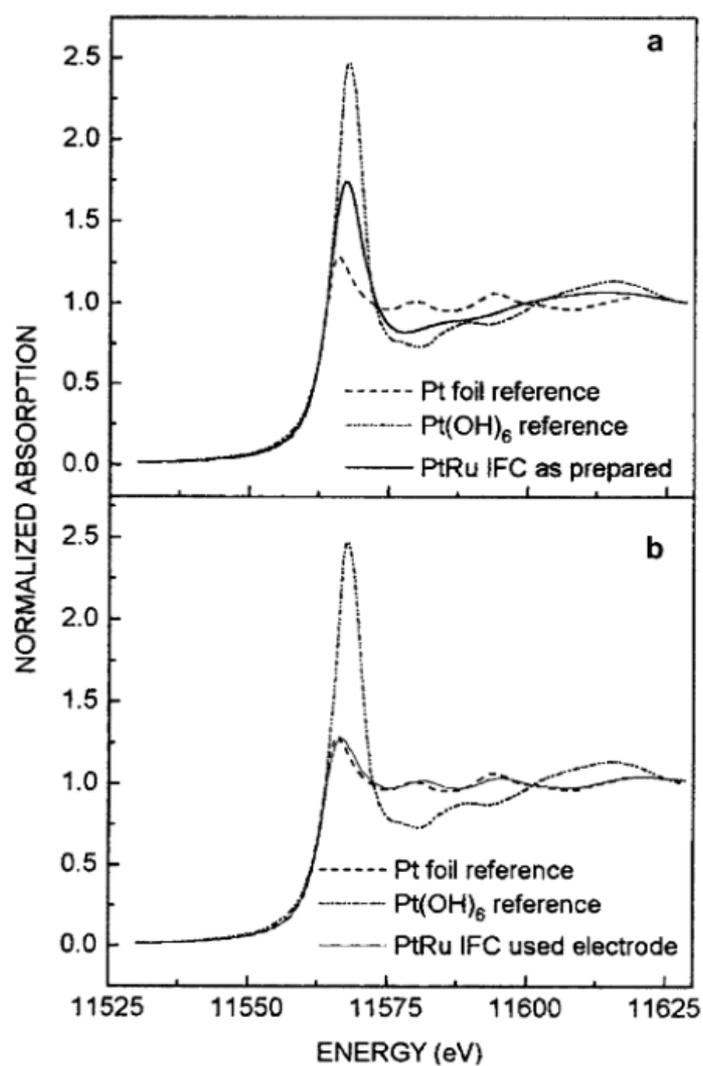
X-Ray Scattering and Diffraction



EXAFS, Pt/C (потенциалы по нас. к.э.)



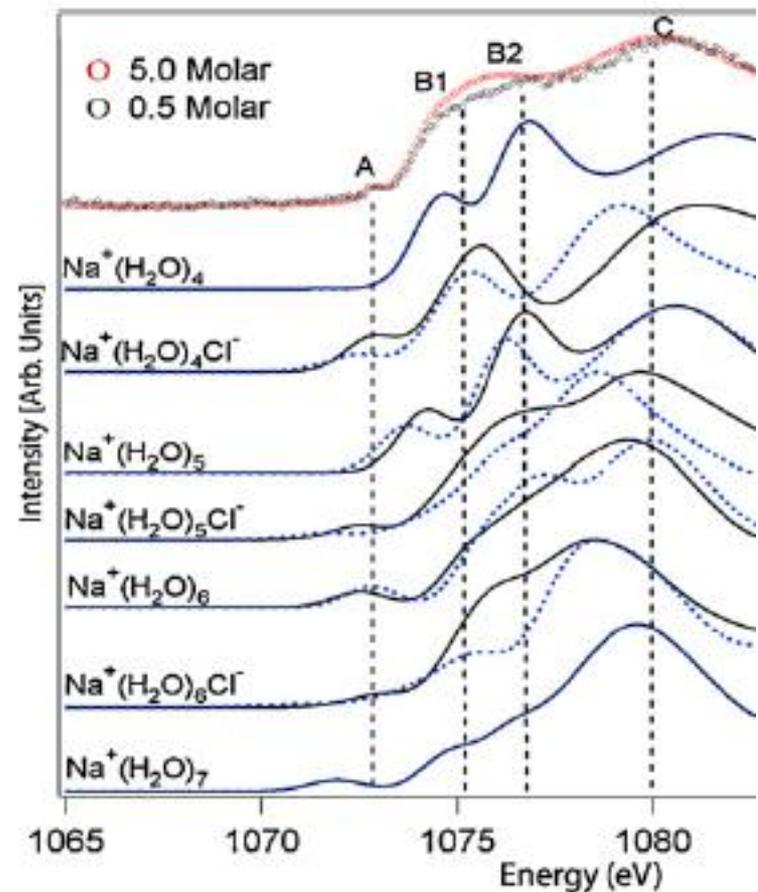
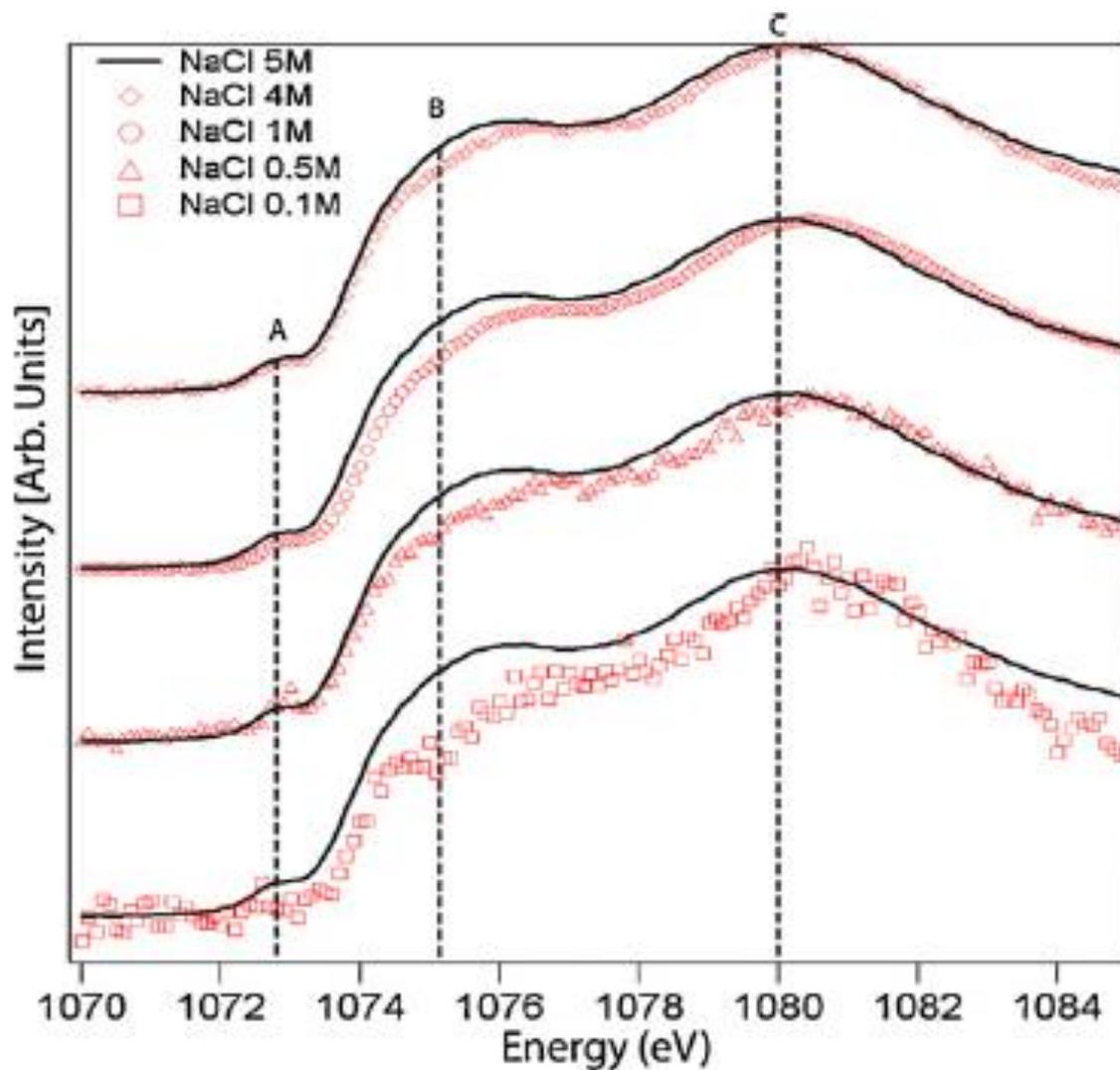
XANES, Pt/Ru

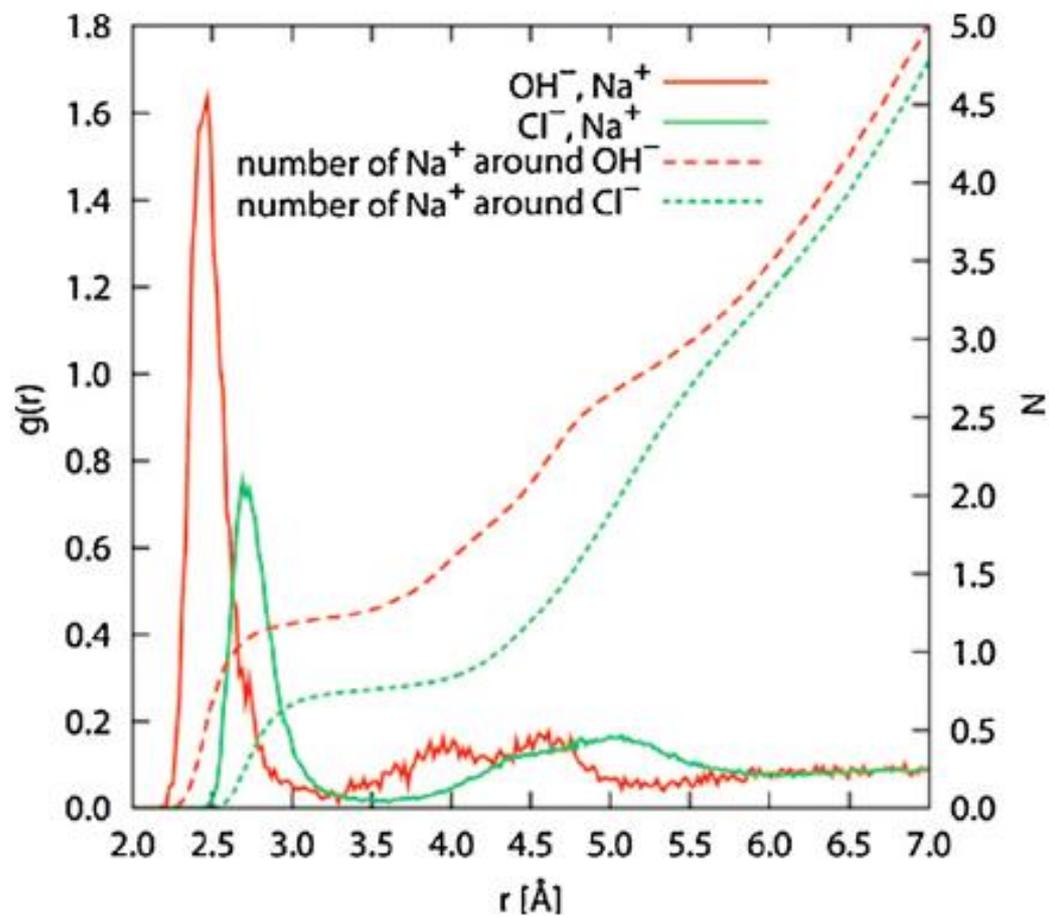
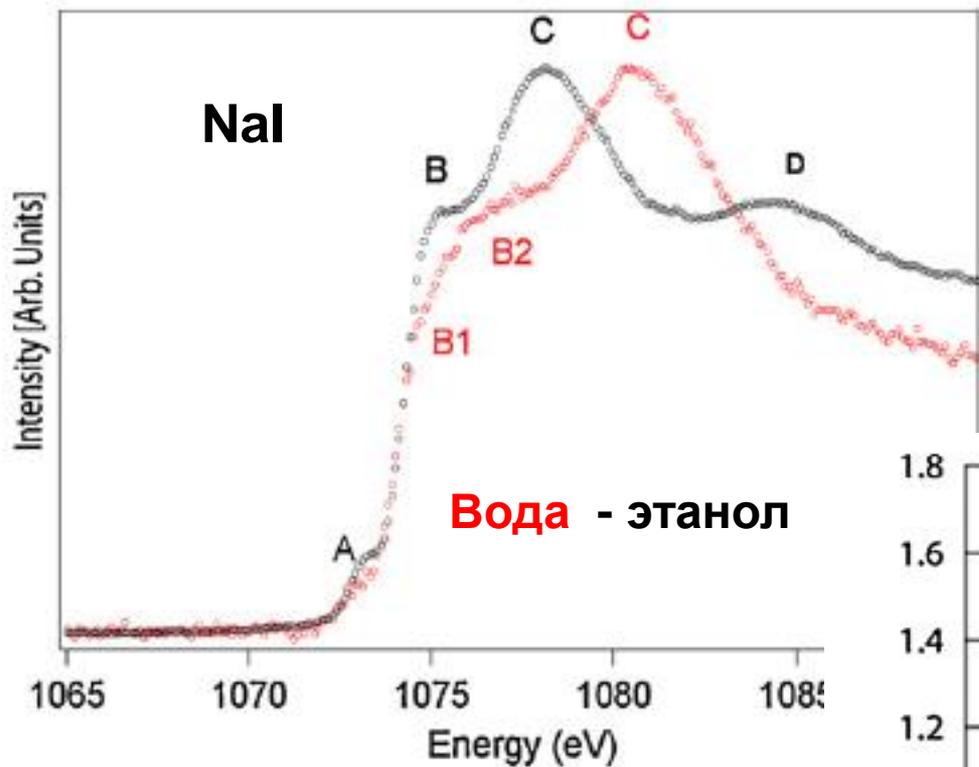


ДО

ПОСЛЕ
ОКИСЛЕНИЯ
МЕТАНОЛА

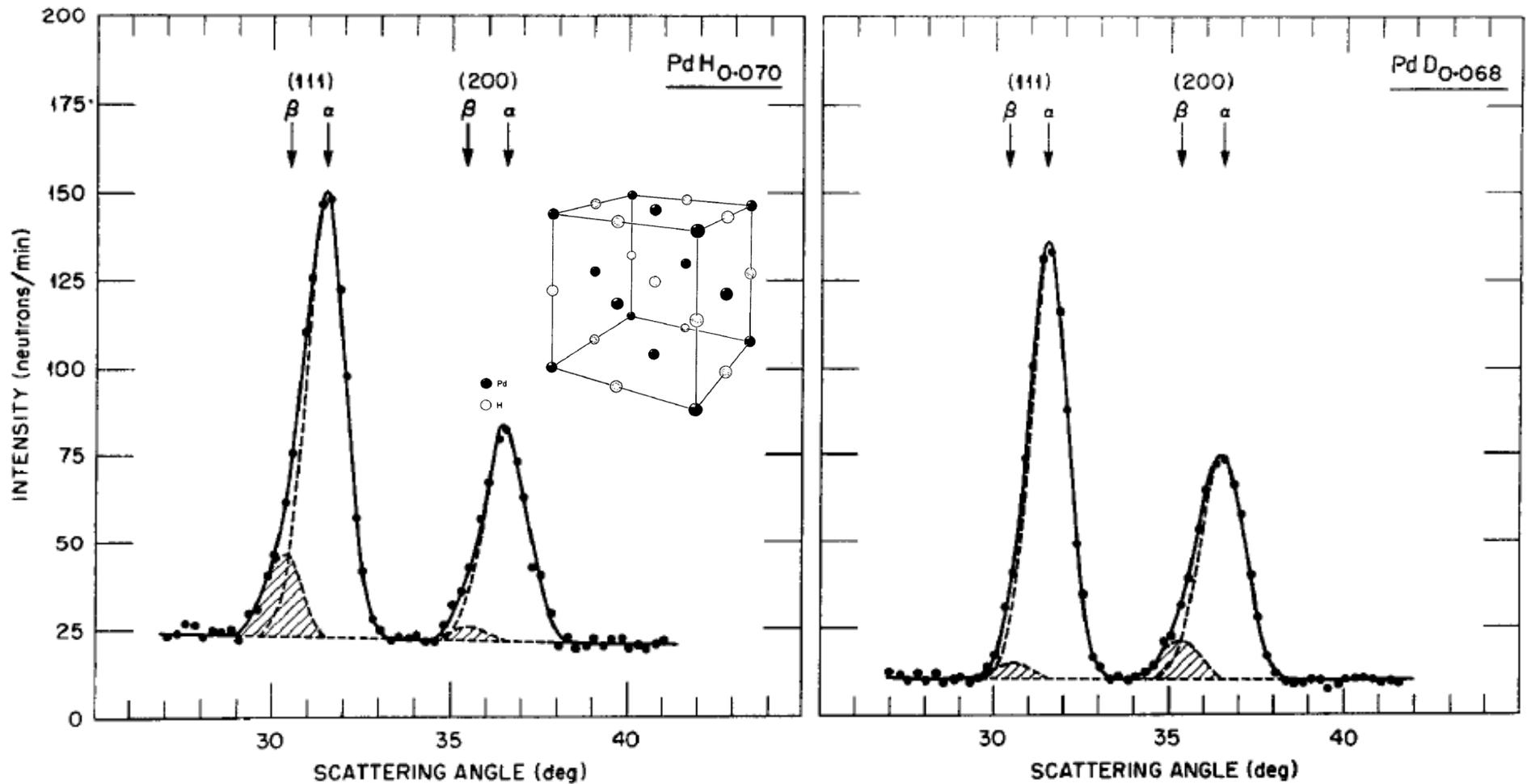
Рентгеновская спектроскопия растворов



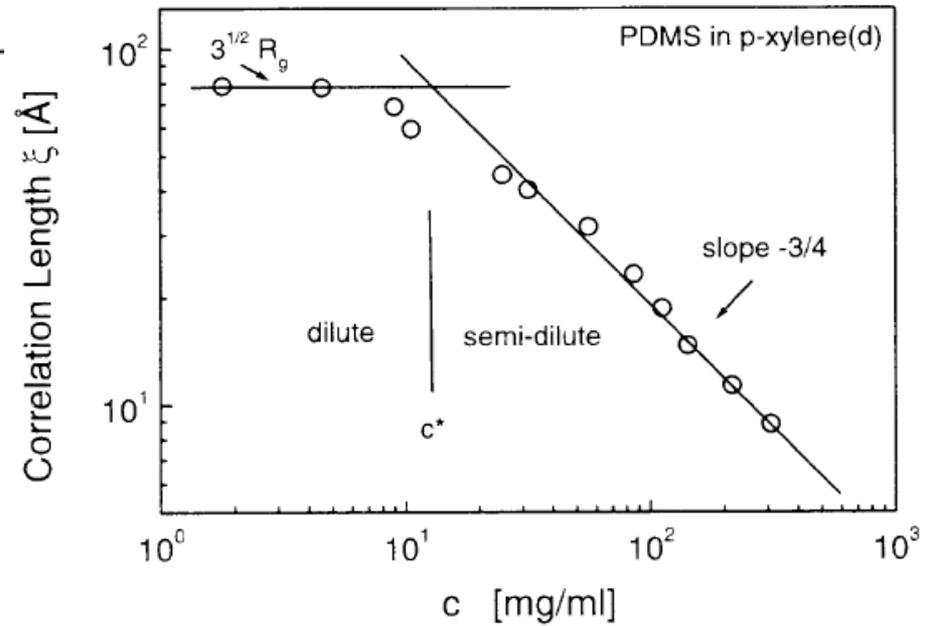
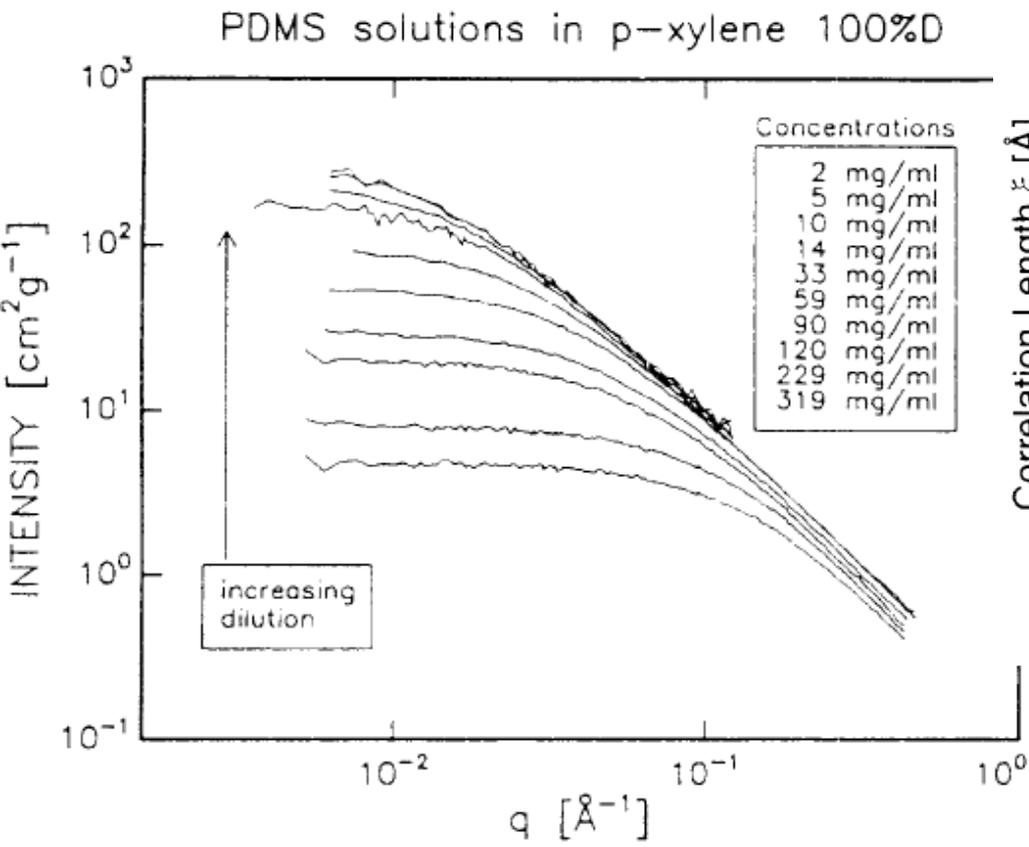
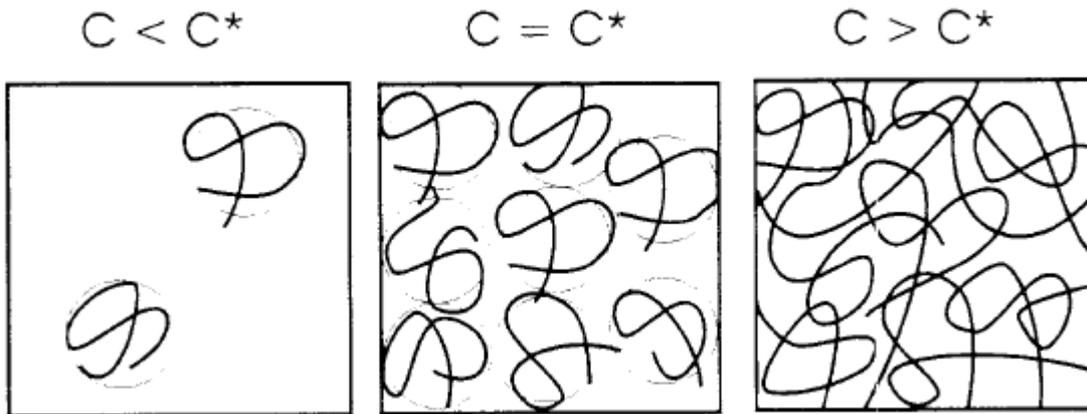


Нейтронная дифракция (легкие атомы)

Образование гидридов палладия (водород в октаэдрических позициях)

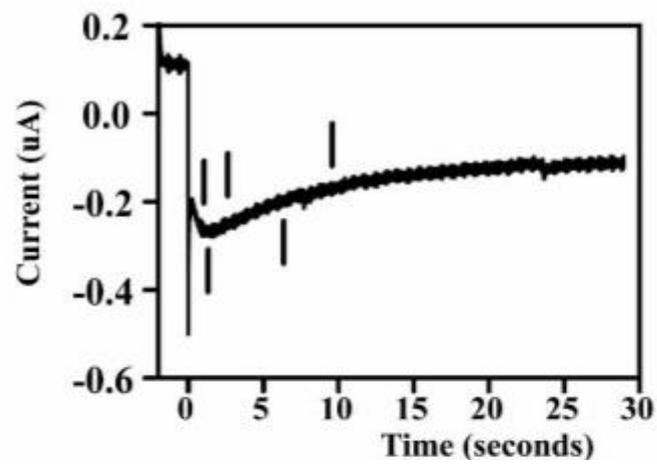


Нейтронное рассеяние - растворы полимеров

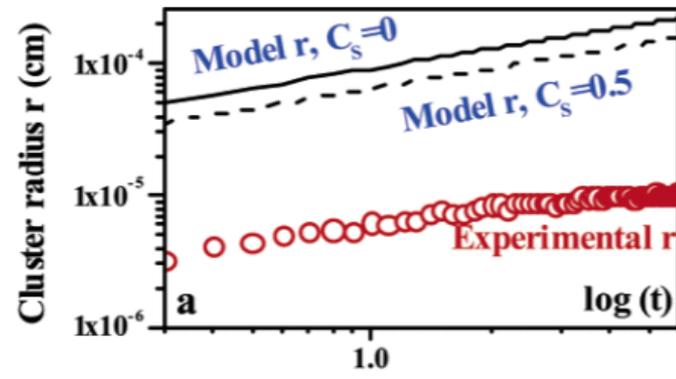


In situ просвечивающая электронная микроскопия (TEM)

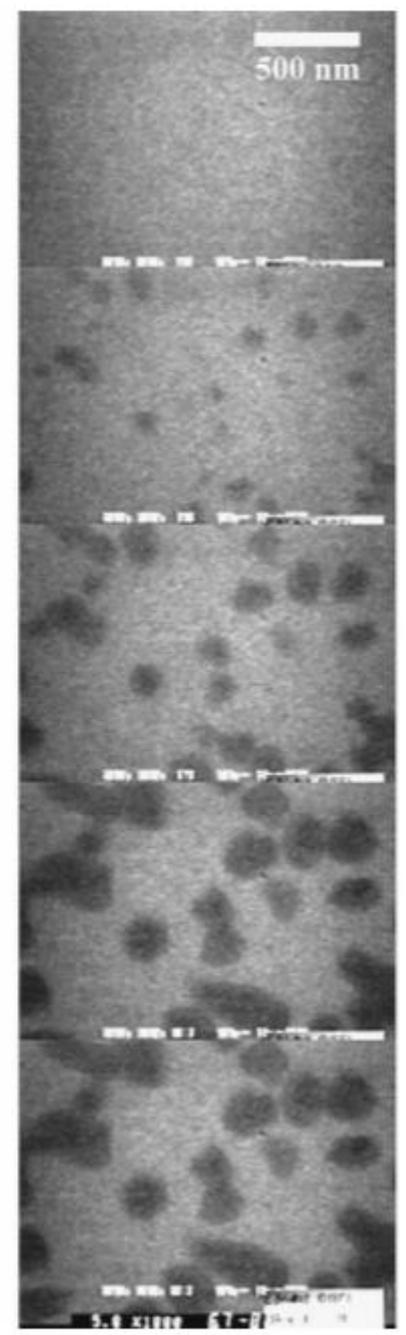
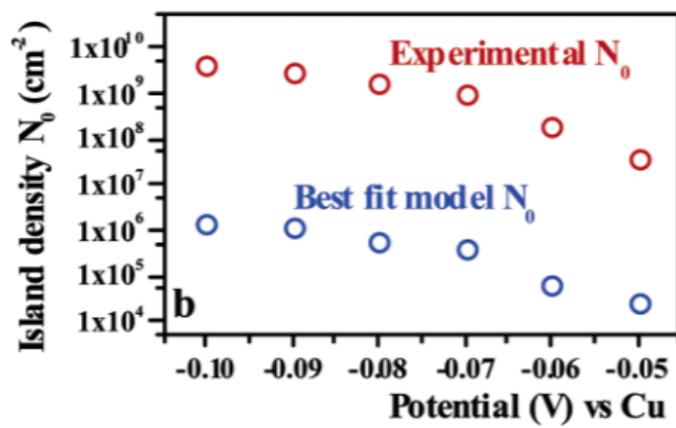
Существующие модели позволяют извлекать число центров N_0 из транзientа тока.



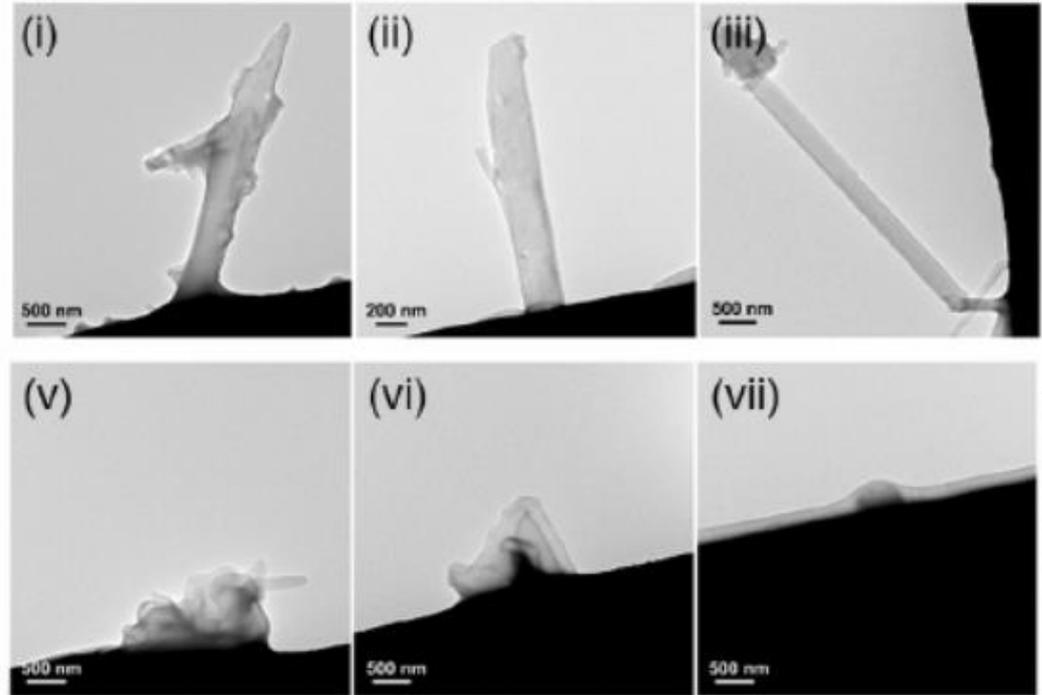
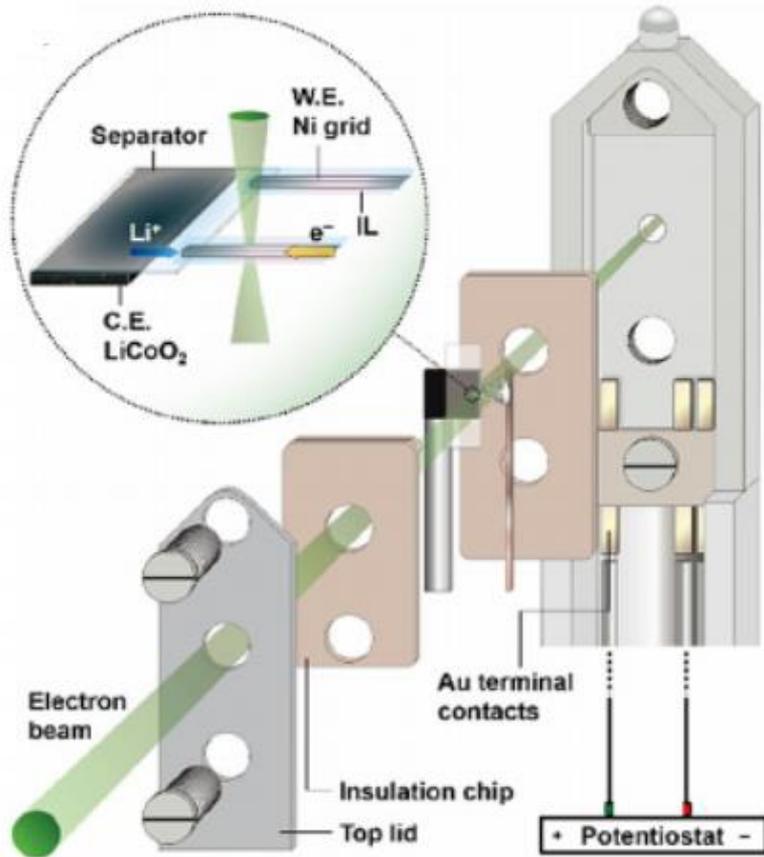
При фиксированном пропущенном заряде от N_0 зависит размер растущего зародыша.



Ранее отклонение рассчитанных N_0 от реальных были известны по данным ex situ микроскопии для сильно разросшихся зародышей.

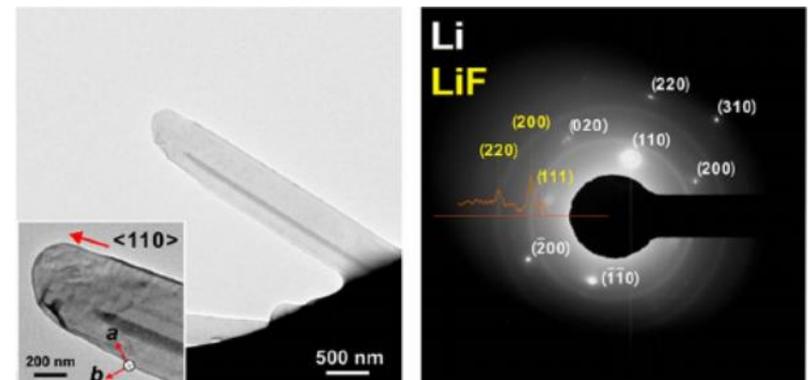


In situ просвечивающая электронная микроскопия (TEM)



Дендриты при переосаждении лития

Small Struct. 2 (2021) 2100018



Обзоры по *in situ* и *operando* для электрохимических систем

ACS Catal. 11 (2021) 1136–1178 (*Operando* Methods in Electrocatalysis)

Chem. Soc. Rev. 50 (2021) 5832-5850 (Electrocatalysis - scanning tunnelling microscopy)

Ann. Rev. Analyt. Chem. 14 (2021) 87-107 (*In Situ* X-Ray Techniques for Electrochemical Interfaces)

Adv. Mater. Technol. 5 (2020) 2000555 (*In Situ/Operando* Optical Microscopy for Probing Electrochemical Energy Systems)

Current Opinion Electrochem. 15 (2019) 18-26 (*In situ* studies: electrochemistry and scattering)

J. Synchrotron. Rad. 25 (2018) 151-165 (*In situ* electrochemical synchrotron radiation for Li-ion batteries)

Nano Energy 11 (2015) 196–210 (Liquid cells for *in situ* TEM electrochemical investigation of lithium-ion battery)

ChemElectroChem 2 (2015) 1427-1445 (Probing *Operating* Electrochemical Interfaces by Photons and Neutrons)