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IMAGING INTERNAL STRUCTURES IN LITHIUM-ION AND SODIUM-ION BATTERIES BY NEUTRON AND X-RAY TOMOGRAPHY

This paper presents the application of combined non-destructive diagnostic methods—neutron and X-ray tomography, conducted using the TITAN neutron radiography and tomography facility at the WWR-K reactor, as well as the IMAX X-ray tomography facility, to visualize the internal structure of 2032 and 18650 battery cells provided by the Research, Design, and Technology Center for Electrochemical Engineering at Dubna State University. The obtained X-ray and neutron tomography data revealed inhomogeneities in the distribution of the base materials, as well as areas of low and high radiation attenuation, gaps, and voids at the boundaries between the electrodes and the battery case. The analysis results demonstrate the high efficiency of X-ray and neutron tomography for quality assessment during the manufacture of such batteries. The conducted test demonstration experiments allow for the development of methods for the combined application of these methods and provide a basis for their further use in the research and development of new battery systems.

Keywords: x-ray tomography, neutron tomography, li-ion and sodium-ion battery.

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Нейтрондық және рентгендік томография көмегімен литий-ионды және натрий-иондық батареялардың ішкі құрылымдарын бейнелеу

Бұл жұмыста аккумуляторлық 2032 және 18650 типті өлшемді элементтердің ішкі құрылымын бейнелеу үшін бұзбайтын диагностиканың біріктірілген әдістері, яғни нейтрондық және рентген томографиясы қолданылды. Нейтрондық томографиялық зерттеулер ССР-Қ (сумен салқындатылатын реактор) реакторында орналасқан "TITAN" нейтрондық радиография және томография қондырғысында, ал рентгендік томография IMAX қондырғысында жүргізілді. Зерттеу объектілері Дубна мемлекеттік университетінің электрохимиялық машина жасау саласындағы ғылыми - конструкторлық және технологиялық орталығы тарапынан ұсынылды. Алынған рентгендік және нейтронды томографиялық мәліметтер базалық материалдардың таралуының біркелкі еместігін, сондай-ақ электродтар мен аккумулятор корпусының арасындағы шекаралардағы төмен және жоғары радиациялық әлсіреу аймақтарын, саңылаулар мен бос орындарды анықтады. Талдау нәтижелері осындай батареяларды өндіру кезінде сапаны бағалау үшін рентгендік және нейтрондық томографияның жоғары тиімділігін көрсетеді. Өткізілген сынақ-демонстрациялық тәжірибелер осы әдістерді біріктіріп қолдану әдістерін жасауға мүмкіндік береді және оларды жаңа аккумуляторлық жүйелерді зерттеу мен әзірлеуде одан әрі пайдалану үшін негіз болады.

Түйін сөздер: рентгендік томография, нейтрондық томография, литий-иондық және натрий-иондық батарея.

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Визуализация внутренних структур в литий-ионных и натрий-ионных батареях с помощью нейтронной и рентгеновской томографии

В данной работе представлено применение комбинированных методов неразрушающей диагностики – нейтронной и рентгеновской томографии, проведенных с использованием установки нейтронной радиографии и томографии «ТИТАН» на реакторе ВВР-К, а также рентгеновской томографической установки IMAX, для визуализации внутренней структуры аккумуляторных ячеек типоразмеров 2032 и 18650, предоставленных Научно-конструкторским и технологическим центром электрохимического машиностроения Государственного университета «Дубна». Полученные данные рентгеновской и нейтронной томографии выявили неоднородности распределения материалов основы, а также области пониженного и повышенного ослабления излучения, зазоры и пустоты на границах электродов с корпусом аккумулятора. Результаты анализа демонстрируют высокую эффективность рентгеновской и нейтронной томографии для оценки качества при изготовлении таких аккумуляторов. Проведенные тестовые демонстрационные эксперименты позволяют разработать методики комбинированного применения этих методов и создают основу для их дальнейшего использования при исследовании и разработке новых аккумуляторных систем.

Ключевые слова: рентгеновская томография, нейтронная томография, литий-ионные и натрий-ионные батареи.

Introduction

In 2015, the Paris Agreement was signed, committing 195 countries to reducing greenhouse gas emissions and slowing global climate change. As a result, more and more countries are focusing on renewable energy and green technologies. However, solar panels and wind turbines face a significant challenge: they are climate-sensitive. To ensure constant electricity availability, reliable energy storage systems are needed.

The limited supply of fossil energy sources, as well as the widespread deterioration of the environmental situation, leads to the need to increase the share of renewable energy consumption and the inevitable use of electric drives for vehicles. The widespread use of renewable energy sources and electric vehicles is limited by the lack of high-capacity and energy-efficient energy storage devices, among which rechargeable chemical current sources (CCS) occupy an important place. In the field of "green" energy, CCS is necessary to equalize the load in electrical networks, regulate the frequency and provide consumers with renewable energy in times of power generation failures that occur when using wind, solar or wave power plants. In this regard, an urgent task is to improve existing, search for and develop new energy-intensive and efficient rechargeable batteries.

One of the most common options is rechargeable electrochemical storage devices such as ion batteries, which are considered as a promising solution. Today, lithium-ion batteries (LIBs) are the dominant technology for electronics, electric vehicles, and stationary energy storage systems due to their high energy, long service life, and technology maturity.

Nevertheless, the shortage of lithium and the uneven distribution of its resources in the world, as well as the increasing demand for large-scale storage devices, are stimulating active research into alternative electrochemical cells such as sodium ion batteries (SIBs). Sodium-ion batteries are positioned as a widespread and cheap electrochemical cell, therefore sodium is competitive and especially promising for use in the energy sector [1].

Microstructural defects, porosity, grain boundaries, and interfacial boundary stability directly affect ion transport, energy storage efficiency, and battery durability. Diagnostic and analytical methods are used to study the battery at the micron level and identify defects. Such methods mainly describe the spatial arrangement of the cells and the internal structure of the battery, so it is necessary to use non-destructive testing methods. Neutron and X-ray (X-ray) radiography and tomography have proven themselves well among the methods of non-destructive testing [2]. X-ray tomography provides high spatial resolution and is sensitive to heavy elements, which makes it particularly useful for analyzing battery structure, while neutron tomography demonstrates high sensitivity to light elements such as hydrogen and lithium, allowing direct observation of lithium distribution, electrolyte dynamics, and interfacial processes inaccessible to X-ray methods. These techniques contributes to a deep understanding of the structure, morphology, chemistry, and kinetics of LIBs, allowing for selective analysis to optimize electrodes and improve overall battery efficiency [3].

The combined use of these approaches opens up unique opportunities for the complex analysis of electrochemical systems. The use of these methods allows not only to observe the general morphology, but also to identify internal defects and the uniformity of the distribution of materials in the battery[4].

Methodology

Material preparation. In this study, we chose a lithium- and sodium-ion battery (LIB and SIB) of coin-cell type 2032 and commercial battery 18650 as a test sample. The main direction of research aimed at visualize the lithium ion batteries structure inside the cell in the initial and final states of charging processes.

Neutron tomography facility. Figure 1 shown Neutron tomography studies were performed at the TITAN (Transmission imaging with ThermAl Neutrons) facility located on the 1st horizontal channel of the WWR-K research reactor. At the TITAN facility, a thermal neutron beam was formed by a system of collimators with an aperture diameter of 2 cm, L/D parameter 350. The size of the neutron beam at the sample position is 8.6 × 8.6 cm^2. The neutron projections were recorded by a position-sensitive detector consisting of a scintillation screen based on ZnS(Ag) and ^{6}LiF with a thickness of 100 microns, and a CCD matrix with dimensions of 2048×2048 pixels. The exposure time for one radiography frame was 20 seconds. The spatial resolution of the images obtained was about 200 microns with a pixel size of 42 × 42 microns. [5].

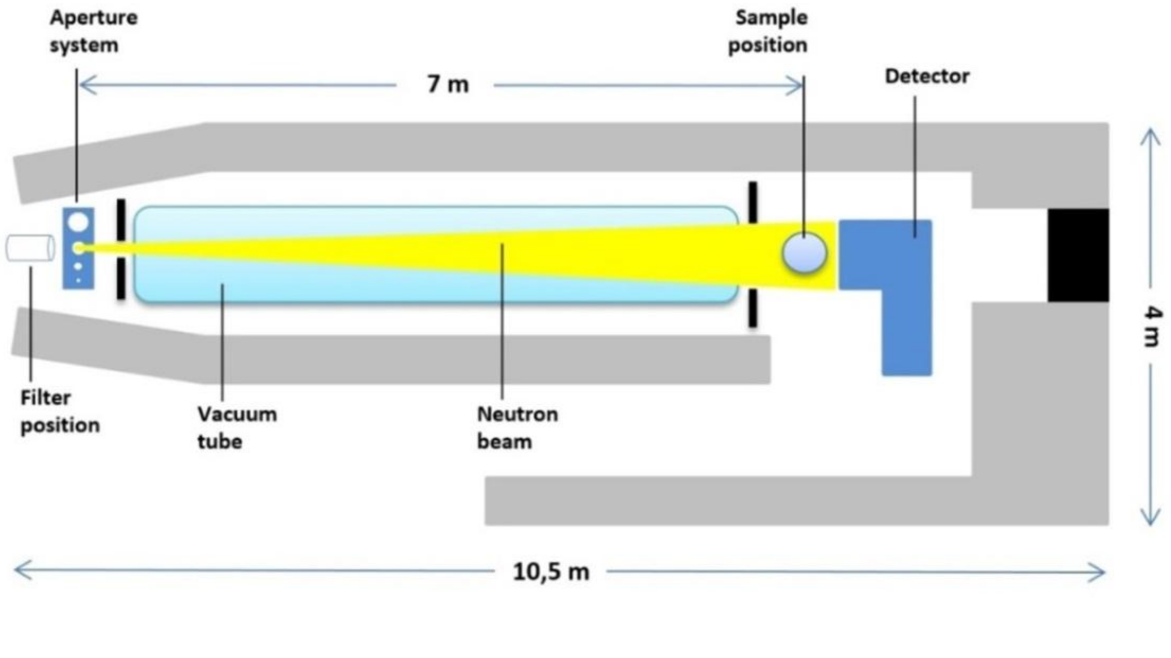


Figure 1 – Scheme of the experimental neutron tomography facility TITAN

The ImageJ software package was used for primary image processing [6], projection and analysis assemblies, whereas the STP Syrmep Tomo project was used for tomography reconstruction of a three-dimensional model of the studied samples [7], The visualization and segmentation processes were performed in the Imagej program [Schneider C. A., Rasband W. S., and Eliceiri K. W. NIH Image to ImageJ: 25 years of image analysis // Nature Methods, – 2012, – Vol.9 , – P. 671–675.].

X-ray tomography facility. Experiments on X-ray tomography were carried out on a compact IMAX facility at the Institute of Nuclear Physics, Almaty, Kazakhstan in figure 2. An X-ray source (Spelman XRB011) with a size of about 30 microns is installed on the IMAX (IMAging with X-ray) installation. A microfocus source with a voltage range from 35 to 80 kV and a current range of 0-700 Ma. The distance from the focus (the point where the X-ray radiation occurs) to the outer window of the tube is 32mm. The X-ray beam exits at an angle of 40° and additionally there is a copper filter with different thicknesses for filtering low-energy parts. The field of view of the detector is 114x145 mm, the pixel size is 64 microns [8].

In this work, the time of one tomography experimental work was about 2 hours.

a)

b)

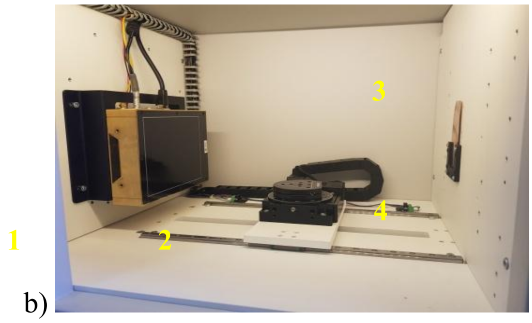


Figure 2 – a) Real photo of outside view b) inside view:
1) detector 2) rotational and linear stage 3) copper filter 4) source.

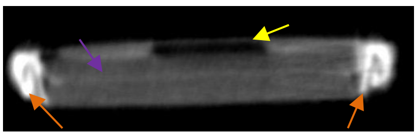
Results and discussion

Coin cell. Neutron tomography examination of a coin-type 2032 lithium-ion cell has provided detailed information about its internal structure. The main structural elements are clearly visible on the horizontal section (Figs. 3b and 4b). An area with reduced attenuation is visible along the central horizontal axis, which differs from the main structure (indicated by the purple arrow), which corresponds to the polypropylene separator layer. A bright area with high neutron attenuation is also visible on the side (orange arrow), due to the presence of an insulating plastic ring between the housing of the positive and negative electrodes, which also effectively attenuates the neutron flux. In figure 3b and 4b, on both tomography sections, elongated dark areas with low attenuation are observed in the upper part of the battery housing (yellow arrow), which corresponds to a pressure metal punch. In Figures 3c and 4, on color tomography images, the regions with low neutron attenuation are displayed in blue shades, while the zones with high attenuation are visualized in red-green colors.

To obtain additional information using X-ray tomography, an experiment of 3D reconstruction of coin cell was conducted, but due to the fact that the housing and electrodes strongly scatter and absorb X-rays, the image turned out to be blurred and distorted. As a result, it was not possible to restore the internal structure of the battery correctly.

a)

b)



c)

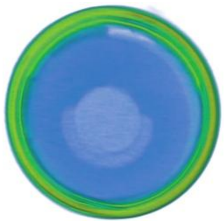


Figure 3 – a) Real photo of A/K coin cell, b) Horizontal slice, c) 3D Neutron tomography drawing

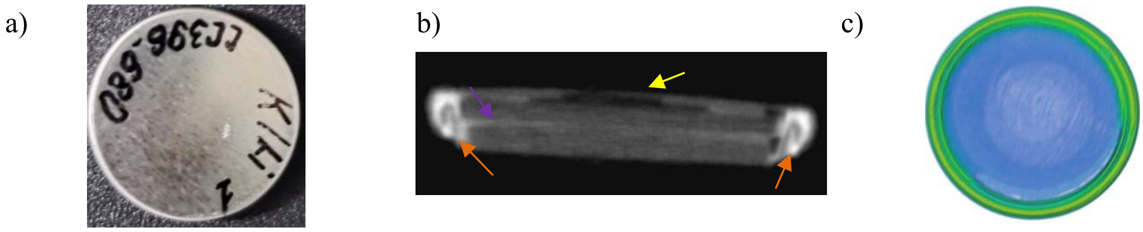


Figure 4 – a) Real photo of K/Li coin cell, b) Horizontal slice, c) 3D Neutron tomography drawing

Sodium 18650 sample. The neutron tomography results made it possible to visualize the internal structure of a cylindrical 18650 sodium-ion battery and identify a number of design features. Figure 5b shows a neutron-radiography image of the battery. Figure 5c shows a tomography section on which the structure of the electrode block is clearly distinguishable. An elongated dark area is observed in the central part, and Figure 5c shows that it has a cylindrical shape and corresponds to an empty space arising from the winding rod of the cell. To the left of this central area, there is an additional elongated dark stripe (yellow arrow), which may indicate the presence of a gap and an uneven fit of the electrodes or separator to each other. Also, another vertical elongated dark stripe (red arrow) is visualized on the slice on the left, which can be interpreted as a void in the center of the twist due to the presence of a positive current outlet. The area marked with purple arrows is characterized by a uniform attenuation of neutrons, which may indicate the presence of an electrolyte. In the right part of the image, there are areas with signs of voids or bulges on the boundary between the electrode material and the housing (black arrow), which may be due to a violation of contact. The left side, on the contrary, demonstrates a tighter fit of the electrodes to the housing. In the upper part of the housing, both on the left and on the right, there are areas with increased attenuation (orange arrow), which corresponds to a plastic insulator between the lid (positive contact) and the housing (negative contact) of the cell. All these features can also be traced in the three-dimensional reconstruction shown in Figure 5d.

Figure 5d shows a three-dimensional reconstruction of a Sodium 1500 battery obtained by neutron tomography. The use of a color scale made it possible to clearly visualize the internal structure and distribution of materials by volume. The blue and cyan hues correspond to regions with low neutron attenuation, which indicates the presence of less dense materials, components with low interaction cross-section, or voids. The yellow-red areas show areas with high attenuation, corresponding, for example, to a metal body.

Figure 6 a),b) shows an X-ray vertical section with a pixel resolution of 16 microns of the upper part of an 18650 cylindrical cell. The internal structure of the electrode block is more clearly discernible in this image than in the X-ray data. The central region is represented with a low attenuation of the X-ray beam, which corresponds to a void as described above. In the left part of the slice, a thin vertical line with low attenuation is visible among the layers of the electrode winding (red arrow), but larger than the central cylindrical part. The connection line to the upper contact area is also visible (blue), which corresponds to the aluminum current lead connecting the positive electrode to the cell cover. It is worth noting that this connecting line was not visible in the neutron tomography section. The X-ray tomography section clearly shows the unevenness of the winding of the electrode block (horizontal yellow dotted line). Similar to the results of the neutron tomography section, the X-ray tomography section also shows a distortion of the electrode block in the central part (yellow arrow). However, according to X-ray tomography, it can be unequivocally said that this bend is associated precisely with the inner layer of the separator, since in the upper part it is he who protrudes beyond the electrodes.

Conclusions

The use of neutron, X-ray, and tomography provides comprehensive information on the internal structure and processes in lithium-ion and sodium-ion batteries. The resulting data on degradation mechanisms and the uneven distribution of active components can be used to optimize battery design, material selection, and operating modes, directly contributing to the creation of more reliable and durable energy devices.

Future research plans include using in situ and operando modes to monitor dynamic changes in batteries during operation. This includes tracking lithium and sodium dendrite formation, gas formation, and changes in electrode density and structure during charge-discharge cycles.

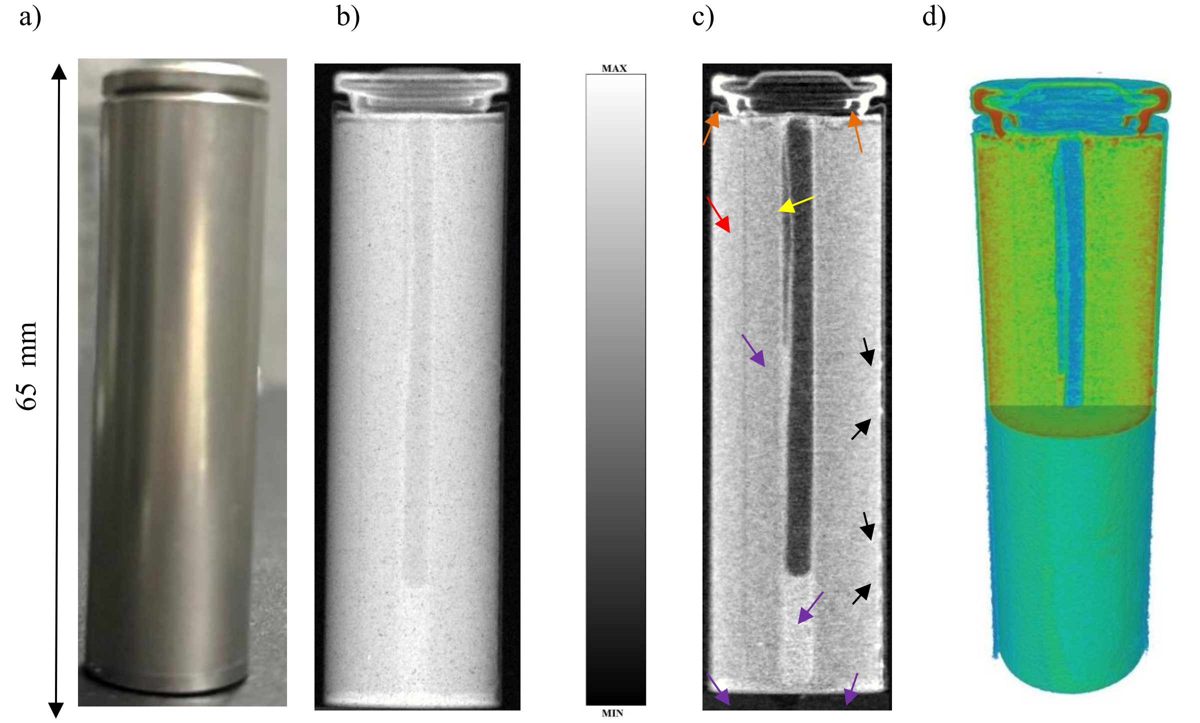


Figure 5 – a) Real photo of Sodium 1500 b) radiography image c) vertical section of a neutron tomography image d) 3D neutron tomography blue

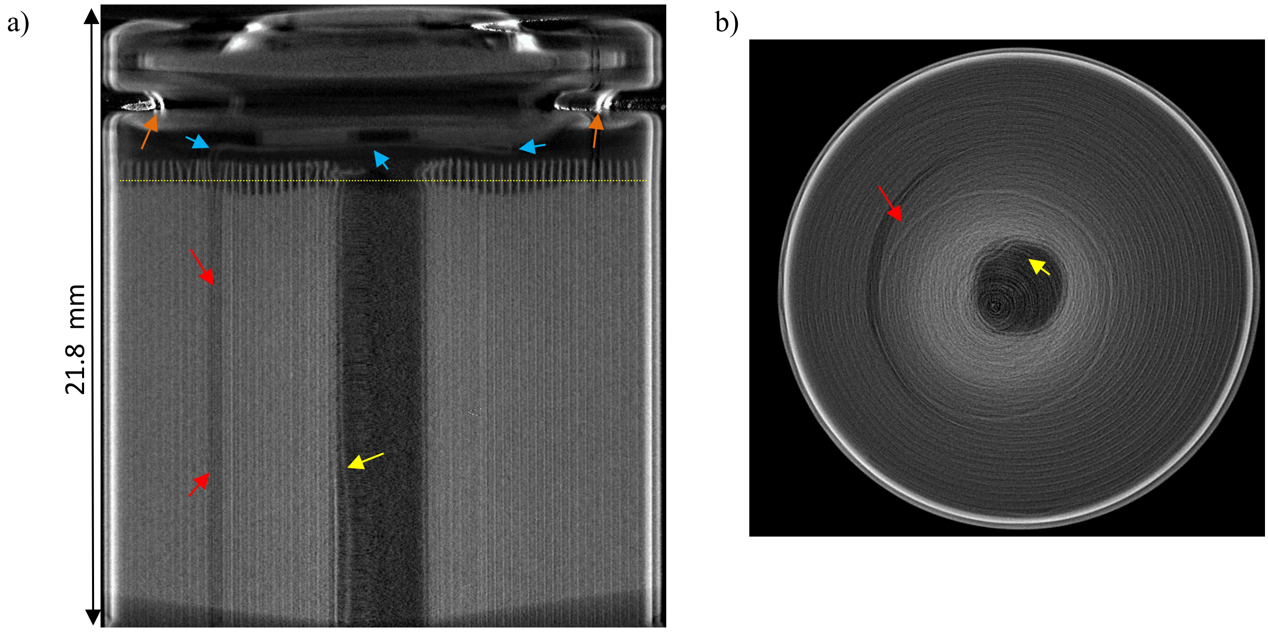


Figure 6 – a) X-ray (Top of battery) b) X-ray tomography slice

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