Progress report on the project "A system for neutron operando monitoring and diagnostics of materials and interfaces for electrochemical energy storage devices at the IBR-2 reactor"

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The project is aimed at developing neutron scattering techniques that would allow studying the structure of electrochemical interfaces and electrode materials of different types in the course of their operation (*operando* and *in situ* modes). It suggests the development of the environment for the effective use of neutron diffraction, reflectometry and small-angle scattering for various types of electrochemical interfaces and electrodes and creation of specialized experimental cells and surrounding equipment for such type of research.

Neutron diffraction (ND). The new ND electrochemical cell for real time diffraction experiments on changes in crystalline structure of electrodes in lithium intercalation/deinteracaltion processes has been manufactured and successfully tested at the HRFD and RTD diffractometers of IBR-2. The cell uses special constructional materials and makes it possible to perform enhanced operando studies of the electrodes in conditions closest to those in real electrochemical energy storage devices. The studies are made together with complementary electrochemical experiments and X-ray diffraction diagnostics carried out in a specialized laboratory at FLNP.

Neutron reflectometry (NR). The three-electrode NR electrochemical cell for studying nanoscale changes at the interfaces of thin-film electrodes (deposited on massive single crystal silicon substrates) and liquid lithium-containing electrolytes under applied potential, has been modified to achieve better performance characteristics including the neutron beam collimation, air-tight conditions and electrical circuit arrangements (choice of material and location of reference electrode relatively to working and counter electrodes). The cell has been tested at the GRAINS reflectometer of IBR-2. The proper combinations of the components in the working electrode heterostructure on silicon substrate has been experimentally studied with the aim at increasing the sensitivity of the NR method regarding the detection of the thin (~ 5 nm) layers forming on the electrode surface in the course of the cell operation.

Small-angle neutron scattering (SANS). A specialized cell for studying nanoscale modifications in bulk carbon electrodes in lithium-oxygen energy storage devices has been designed and manufactured. It is based on the standard vacuum equipment for X-ray spectrometers with proper choice of window, electrode and support materials to provide maximum transmission of neutron scattered in electrodes by the products of the electrochemical reactions forming nanostructures. The cell provides a regulated continuous reaction under electric potential in wetted carbon electrode and keeps the required humidity. The tests of the cell at SANS diffractometer YuMO of IBR-2 are in progress.

The project is performed in the framework of the theme 04-4-1121-2015/2020 ‘Investigations of Condensed Matter by Modern Neutron Scattering Methods’.