NEW ULTRA-HIGH RESOLUTION ELECTRON MICROSCOPE IN THE SSC RIAR HOT LABORATORY

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European Working Group “Hot Laboratories and Remote Handling”
RIAR’s hot laboratory has quite a number of devices and certified techniques.

However, there is a necessity to perform new specific examinations of nano-structured materials to be used in new types of nuclear reactors, radiation engineering, medicine and ecology.

These examinations require super-advanced microscopes and spectrometers equipped with remote handled devices that do not exist yet. Such equipment should be used to modeling and predicting material properties under long-term operation also.
Since RIAR’s hot lab faces such task, a decision was made to purchase a new ultra-high resolution scanning electron microscope intended for examination of irradiated materials.

In March 2008, a unique research unit comprising a field-emission super-high resolution scanning electron microscope Zeiss SUPRA55VP, energy dispersive X-ray spectrometer Inca Energy 350, wave dispersive X-ray spectrometer Inca Wave 500 and HKL EBSD Premium system for registration and analysis of backscattering electron diffraction was purchased and commissioned.
Introduction
BASIC PARAMETERS OF NEW RESEARCH UNIT

Spatial resolution:
- resolution in secondary electrons = 1nm (at 15 kV)
- resolution in secondary electrons = 1.7nm (at 1 kV)
- resolution in secondary electrons = 4.0 nm (at 100 V)
- resolution in backscattering electrons = 1.3 nm
- resolution under STEM = 0.8 nm

Accelerating voltage range 100V – 30kV
The operation at low accelerating voltages allows spatial resolution of the microanalysis system up to 0.1\(\mu\)m to detect ultra-little concentrations on ultra-small areas.
Besides, the operation at low accelerating voltages (from 100V) provides examination of super-fine specimens and samples sensitive to irradiation.
BASIC PARAMETERS OF NEW RESEARCH UNIT

Stability of electron beam current of field-emission electron source no worse than 0.2% per hour, that guarantees the highest result of the quantitative analysis in calculations by wave-type spectrometer.

Magnification range:
- 12x - 900 000x in the secondary electron mode.
Technical features of the Gemini column allowing examination of magnetized specimens within the whole magnification range.

Work current range:
- 4 nA - 20 nA (if required, up to 40 nA)
BASIC PARAMETERS OF NEW RESEARCH UNIT

Low vacuum range:
   1-133 Pa
The in-built low vacuum system allows non-conductive specimens to be examined.

The chamber is 300mm in diameter and 270mm high.

Shift of the stage:
   X/Y = 130 mm, Z = 50 m, slope - 3° - +70°, rotation - 360°;
Our unit intended for examination of irradiated materials is equipped with an HKL CHANNEL 5 system used for back-scattered electron diffraction pictures analysis. This system allows indexing of phases both with high and low symmetry and with pseudo-symmetry as well.

There is software to perform texture analysis of any crystalline material by the method of pole figures and inverse pole figures, distribute orientation/disorientation, etc.

In our case research unit is equipped with an INCA Synergy system based on the energy-dispersive analyzer Inca Energy and EBSD HKL Channel 5 for simultaneous analysis of structure, texture and chemical composition.
Installation of the research unit and first experimental results.

In addition to a special basement, exhausting ventilation and grounding, a room to locate the research unit has:

- closed water cooling system;
- system to maintain temperature and moisture required for microscope operation;
- uninterruptable power supply system
- special biological protection.
Tungsten carbide nano-particles
Fracture surface of irradiated beryllium specimen
Conclusion

The research unit comprising a field-emission super-high resolution scanning electron microscope Zeiss SUPRA55VP, energy-dispersive spectrometer Inca Energy 350, wave spectrometer Inca Wave 500 and HKL EBSD Premium system for registration and analysis of reflected electron diffraction was commissioned in the RIAR’s hot laboratory to examine irradiated structural materials.

Application of this microscope will allow to carry out precision researches of a microstructure, texture and elementary composition of new NPP structural materials as well as their changes after operation.
Thank you very much for your attention!