

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

YU.D. GONCHARENKO, V.D. RISOVANY, A.E. NOVOSELOV, L.A. EVSEEV
*State Scientific Center - Research Institute of Atomic Reactors
433510, Dimitrovgrad, Russia*

ABSTRACT

Recently, requirements to the safety and efficiency of the operated and newly developed nuclear reactors of different types have become more and more strict. Due to this, it is proposed to use core units and components made either from completely new structural materials (ODS steels) or already known materials that have to be operated under elevated parameters (temperature, fluence, coolant parameters, etc.). So, the specialists from hot laboratories face now a task to examine samples of such materials, as well as items made from them, irradiated in research reactors. The following data are to be got: distribution and state of fine-dispersed strengthening phase in the ODS-materials, including fracture surfaces after mechanical tests, change of the texture characteristics in quite local areas of zirconium-based irradiated materials.

As RIAR's materials testing laboratory faces the above task, a decision was made to purchase a new ultra-high resolution electronic microscope intended for examination of irradiated materials. A unique research unit comprising a field-emission scanning electron microscope of super-high resolution Zeiss SUPRA55VP, Carl Zeiss AG (Germany), 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 purchased and commissioned. First examination results of the state of strengthening phase in irradiated ODS materials have been obtained as well as on of texture in local areas of irradiated zirconium-based items.

1. Introduction

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 electron microscope intended for examination of irradiated materials. In March 2008, a unique research unit comprising a field-emission scanning electron microscope of super-high resolution Zeiss SUPRA55VP, Carl Zeiss AG (Germany), 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 purchased and commissioned.

If the Bragg diffraction conditions for some planes of crystal lattice being satisfied, in this case two cone-like beams of diffracted electrons appear for each family of crystalline planes. These cones of electrons can be visible by means of a phosphorescent screen and high-sensitive chamber (e.g. CCD camera) installed behind it to perform observations. As a rule, the chamber is installed horizontally to let the phosphorescent screen be closer to the specimen, the diffraction picture entrance angle being very wide. Thin stripes appearing as a result of crossing of cone-like electron beams with the phosphorescent screen are called Kikuchi stripes. Each of these stripes corresponds to a certain group of crystalline planes. The resulting DOE pictures consist of numerous Kikuchi stripes. Special software determines automatically the location of each Kikuchi stripe and compares it with the theoretical data on the corresponding crystalline phase. Thus, a 3D crystallographic orientation is quickly determined.

To have crystallites orientations maps, the electron beam is gradually moved over the regular point mesh. The DOE picture is made for each point. The data on the orientation and phase composition is stored in a file. Then these data are used to have either orientation or phase maps of the microstructure. The obtained results can complement well the results of ordinary X-ray analysis. However, the peculiar feature of the reflected electron diffraction is that only surface layers of the examined specimen form the diffraction picture. In this case, the requirements to the quality of the specimen surface polishing are very high. The orientation and size of grains of the polished surface must not change after the polishing. At present, the surface polishing process of the specimens to be examined by the back-scattered electron diffraction is mastered.

4. 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 operations.