APPLICATION OF SYSTEM FOR REGISTRATION AND ANALYSIS OF BACKSCATTER ELECTRONS TO STUDY TEXTURE OF PRODUCTS MADE FROM ZIRCONIUM ALLOYS AT SSC RIAR HOT MATERIAL TESTING LABORATORY

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ABSTRACT

Recently, requirements to the safety and efficiency of the operated and newly developed nuclear power reactors have become stricter. Due to this, it is proposed to use core units and components made from zirconium-based alloys, since their texture allows either increasing the lifetime of a product or improving its operating parameters, other conditions being equal. Therefore, the texture of various core components made from zirconium-based alloys should be systematically measured both before and after irradiation. Besides, changes in the texture should be evaluated in small areas of products, e.g. over the fuel rod cladding thickness. One of the possible ways to measure pole figures on small surface areas is to apply the system for registration and analysis of backscatter electrons that can be installed on the up-to-date electron scanning microscopes. The HKL EBSD Premium System for registration and analysis of backscatter electrons was installed and put into operation on the super-high resolution electron microscope Zeiss SUPRA55VP, Carl Zeiss AG, Germany. The first examination results have been obtained both for irradiated and unirradiated zirconium alloy samples. The techniques for preparation of sample surfaces for examination have been tried out.

1. Introduction

Recently, as a result of tightened requirements to the safety and efficiency of operated and newly developed nuclear power reactors, new items made from zirconium-based materials are proposed to be used in the reactor cores. These items have specially made textures that allow either increasing the lifetime or improving other operating parameters, all material parameters being equal. Therefore, the texture of various core components made from zirconium-based alloys should be systematically measured both before and after irradiation. Besides, changes in the texture should be evaluated in small areas of products, e.g. over the fuel rod cladding thickness. One of the ways to measure pole figures on small surface areas is to apply the system for registration and analysis of backscatter electrons that can be installed on the up-to-date electron scanning microscopes.

2. General

The HKL EBSD Premium System for registration and analysis of backscatter electrons was installed and put into operation on the super-high resolution field-emission electron microscope Zeiss SUPRA55VP, Carl Zeiss AG, Germany (Fig.1).

Fig.1. Super-high resolution field-emission scanning electron microscope Zeiss SUPRA55VP, Carl Zeiss AG, Germany, energy-dispersive spectrometer Inca Energy 350 (a), wave spectrometer Inca Wave 500 (b) and HKL EBSD Premium system for registration and analysis of backscatter electron diffraction. (c).

2.1 Basic technical parameters of the research unit on the basis of scanning electron microscope Zeiss SUPRA55VP

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 reflected electrons	1.3 nm
- resolution under STEM	0.8 nm
- HKL CHANNEL 5 system (phase)	25nm
- accelerating voltage range	100V – 30kV
Magnification range:	12x - 900 000X in the secondary electron mode
Low vacuum range:	1-133Pa

The in-built low vacuum system allows non-conductive specimens to be examined.
The range of INCA ENERGY 350 element detection is Be-Pu
The range of INCA Wave 500 element detection is B-Pu
The operation at low accelerating voltages allows spatial resolution of the microanalysis system up to 0.1μm 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.

2.2 Adaptation of the research unit

Both microscope and its components (spectrometers, add-on devices) were not designed for examination of radioactive materials.
Our research unit is a part of hot lab facilities and intended, first of all, for examination of irradiated materials. For this purpose, we had to adapt it.

First of all, when selecting the design of the research unit, we gave our preference to the components that had more capabilities in protecting them from ionizing emission. A special lead-sheet shielding was developed to protect an operator from ionizing emission when examining irradiated samples. This shielding does not disturb either the operator’s work or operation of microscope components.

A three-level shielding was developed to protect the energy-dispersive spectrometer from ionizing emission when performing examinations that do not require this spectrometer.

Energy-dispersive spectrometer Inca Energy 350 with a variable diaphragm was selected.

When the diaphragm is closed completely, the inlet channel of the spectrometer is also closed and thus, a part of γ-emission is avoided. This is the first level of protection.

If the emission from a sample is intensive, and there is no need to use the energy-dispersive spectrometer, then the Inca Energy 350 can move the spectrometer channel inside the microscope body increasing the distance from the spectrometer detector to the sample by more than 10cm. This is the second level of protection.

When examining samples with activity up to 1000μR/s and there is no need to use the energy-dispersive spectrometer, additional stationary shielding in the form of a plate of complicated shape 15mm thick is used. Through the process holes, this plate is fixed on the inner surface of the cell and protects the spectrometer detector.

Radiation protection was also developed for the system for registration of backscatter electron diffraction pictures. It is used when there is no need to apply this system.

Components of this system - phosphorescent screen and digital CCD camera - are the most sensitive to ionizing emission. The phosphorescent screen is also very sensitive to any contamination on its surface, so, if this system is not used, the manufacturer (the Oxford Instruments) recommends to use a thin foil to protect it from dust.

Instead of thin foil, a plate 15mm thick was made from the same material that is used to protect the energy-dispersive spectrometer. This shielding is installed in the niche inside the microscope vacuum chamber.

The HKL EBSD Premium system for analysis of phase structure and texture of crystalline materials by the method of backscatter electron diffraction was also modified. The modification consisted in the fabrication of a special holder for samples to measure the texture of irradiated materials and evaluate the possibility of measurements if the quality of sample surface is not good. This was needed to examine ring samples cut off from a spent fuel rod cladding and to analyze the texture change both along the fuel rod and over the cladding thickness. In this case, a ring sample cut off from the cladding is placed into the metallographic drum to prepare the cross-section surface for examination. To decrease the time of microscope operator’s contact with irradiated samples, there was a need to develop a special holder that would allow examinations of samples textures to be held just in the metallographic drum. A standard holder can be used for miniature samples only, e.g. plates 0.5*05 in size and 1mm thick. We developed a special holder that allows us to conduct examinations even if a sample is fixed on the metallographic drum (Fig.2).

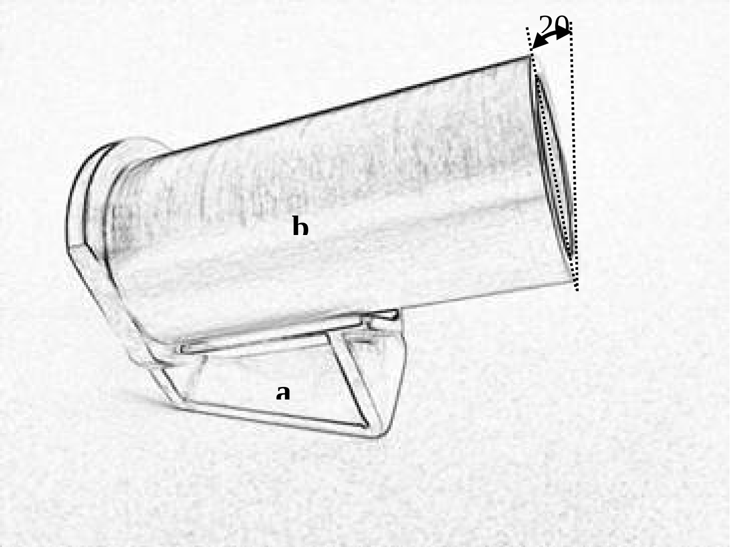

Fig.2 A special sample holder developed for the HKL EBSD system (a) and a standard metallographic drum installed on it (b).

The procedure to prepare the surface for examination recommended by the manufacture is well described in the open literature and corresponds to the procedure to prepare surfaces of very good cross-sections. In case of zirconium alloys, it is good mechanical polishing ending with electro-refining in 20% of $HClO_4$, 80% C_2H_5OH. When preparing the surfaces of irradiated samples for examination, their quality is worse as compared to that of ordinary samples even if all the requirements are met. It is related, first of all, to the remote mode of preparation of irradiated samples surfaces.

Figure 3 presents the polished surface of the cross-section of spent fuel rod cladding (dark quarters are areas of registration of backscatter electron diffraction pictures).

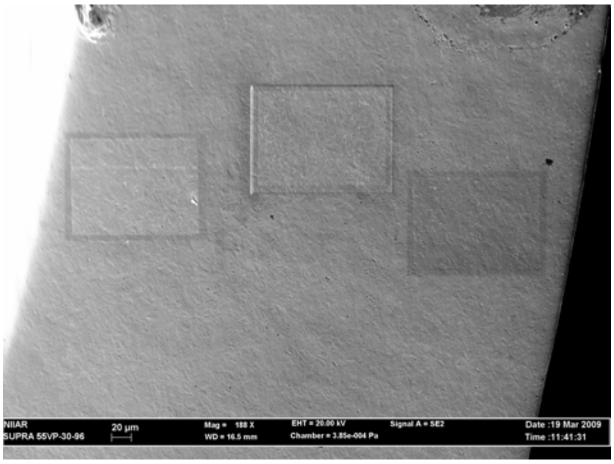

Fig. 3 Areas of registration of backscatter electron diffraction pictures

Figure 4 presents the surface of an area, where the backscatter electron diffraction pictures were registered and texture was analyzed.

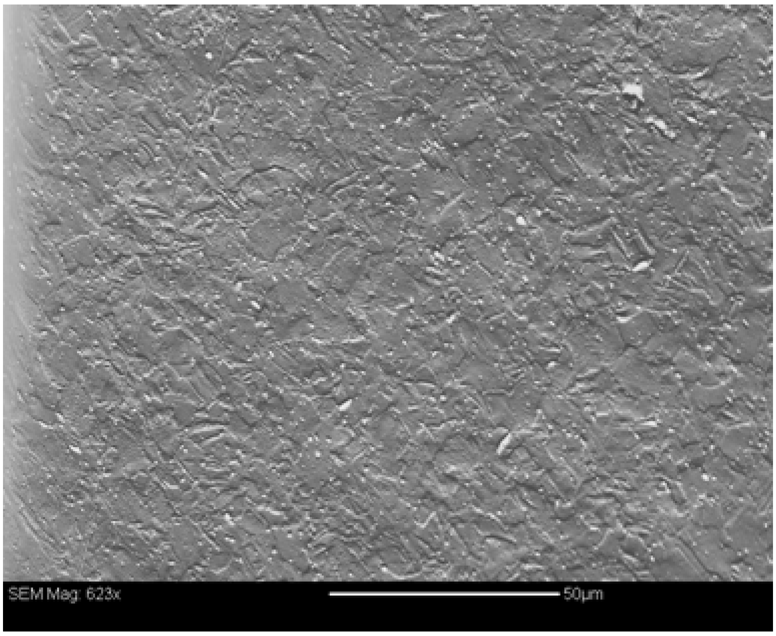

Fig. 4 Surface in backscatter electrons where backscatter electron diffraction pictures were registered.

Figure 5 presents the same area; the color of separate areas depends on their orientation (on Euler’s angle of separate points of this area).

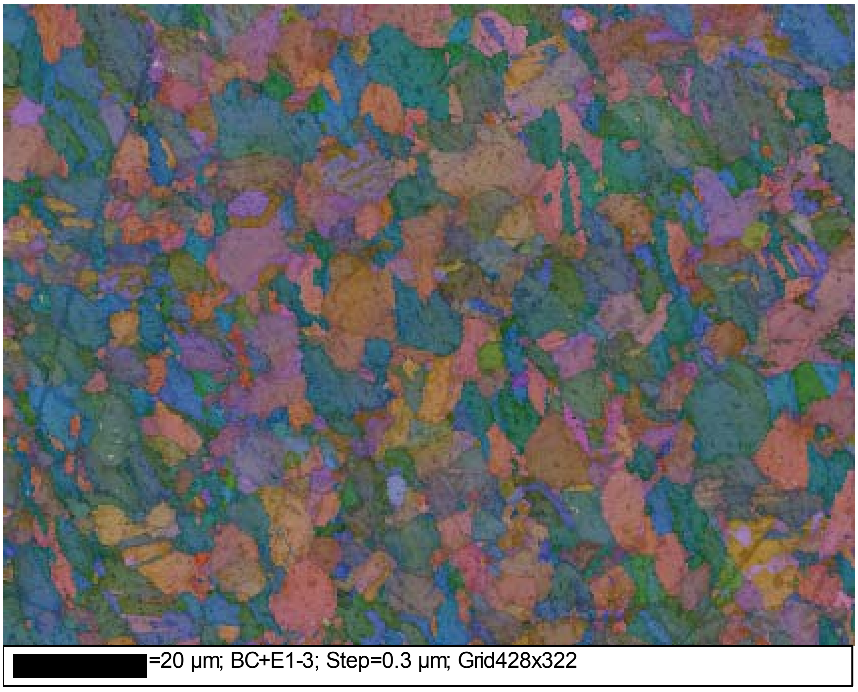

Fig. 5 Surface in backscatter electrons where backscatter electron diffraction pictures were registered

Figure 6 presents pole figures for this area used to calculate the Kernst coefficients

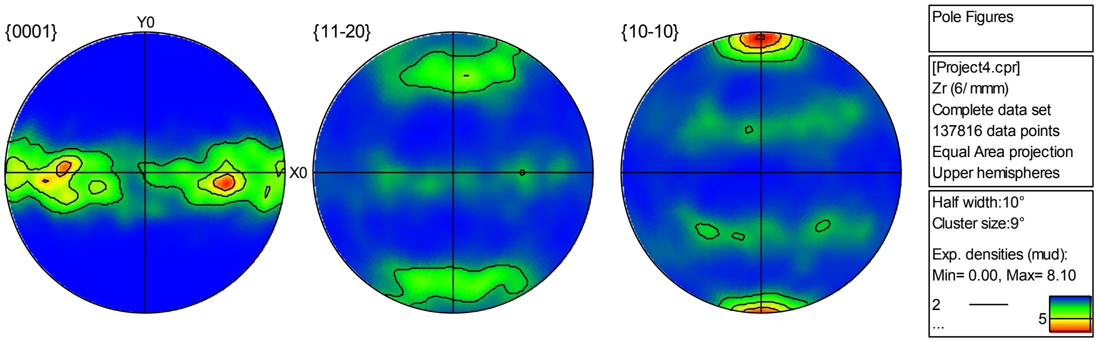

Fig. 6 Straight pole figures for area from Fig.5 and calculated Kernst coefficients.

Fx=0,524	**Fx=0,238**	**Fx=0,238**
Fy=0,039	**Fy=0,480**	**Fy=0,480**
Fz=0,437	**Fz=0,282**	**Fz=0,282**

Since the quality of preparation of surfaces to be examined is not perfect, the identification of the surface structure is about 80%. However, it is enough to plot pole figures and calculate texture coefficients.

Conclusion.

The presented results demonstrate a high capacities of the adapted research unit on the basis of the super-high resolution field-emission electron microscope Zeiss SUPRA55VP, with energy-dispersive spectrometer Inca Energy 350, wave spectrometer Inca Wave 500 and HKL EBSD Premium system for registration and analysis of backscatter electron diffraction to examine irradiated structural materials with an activity of up to 1000μR/s. The HKL EBSD Premium system for registration and analysis of backscatter electron diffraction installed on this electron microscope allows analysis of the texture changes in local areas of spent fuel rod claddings.