Possibilities and Features of Electron Backscatter Diffraction for Reactor Materials Investigation

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Abstract. As a result of tightening safety and economic efficiency requirements imposed on operating and newly developed nuclear power reactors, their active cores have been recently fitted with or proposed to be fitted with elements made from different materials with modified structure (Zr-based alloys with specially developed texture; steels the crystal lattice of which is strengthened by nano-particles; various nano-structured materials) allowing either lifetime extension or improvement of performance of these elements, all other parameters being equal. Therefore, it is necessary to study properties of such materials with complicated structure both before their use in nuclear reactors and after reactor irradiation. In so doing, new research methods which allow the investigation into material structure and composition in small nano-size regions of materials should be used. One of such methods is recording and analysis of electron back-scattered diffraction (EBSD). It allows the identification of crystallographic structure and its orientation in regions up to 25 nm in size. When applying this method together with X-ray spectrum microanalysis, it also becomes possible to identify elemental composition in the same local regions of materials. Combination of these two methods is widely used when studying "standard" non-irradiated materials, however, only X-ray spectrum microanalysis is still extensively applied in reactor material testing, what can not be stated for the EBSD method. RIAR has purchased the Zeiss SUPRA55VP, Carl Zeiss AG field-emission scanning electron microscope of super-high resolution equipped with X-ray microanalysis system (wave and energy spectrometer) and HKL EBSD Premium System. Some examples of reactor material studies using EBSD system only or together with energy-dispersed X-ray spectrometer are presented.

1. INTRODUCTION

As a result of tightened safety and economic efficiency requirements imposed on operating and newly developed nuclear power reactors, their cores have been recently fitted with or proposed to be fitted with components made from different materials with modified structure allowing either lifetime extension or improvement of performance of these components, all other parameters being equal. Therefore, it is necessary to study properties of such materials with complicated structure both before their use in nuclear reactors and after irradiation. In so doing, new research methods should be used, which allow the investigation into material structure and composition in minor nano-size regions. One of such methods is recording and analysis of electron back-scattered diffraction (EBSD).

There is a potential possibility to apply this technique for research of structural materials, which structure is strengthened by nano-particles, for example by yttrium oxide particles. This paper presents one more possibility of this method application that is to study the structure of high strained materials deformed in special way to have nano-structural components.

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2. SAMPLES DETAILS

The backscattering electrons diffraction registration and the analysis system HKL EBSD Premium System was used to study the steel AISI321 structure after intensive plastic deformation by the method of equal channel angular pressing. This steel in this condition may be proposed to be used in core internal as bolts and pins for PWR type reactors.

The structure of the same steel after low plastic deformation was studied by sing the same method for comparison.

3. RESULTS

Fig. 3.1 presents an image of a sample surface prepared for investigation by the secondary electrons method.

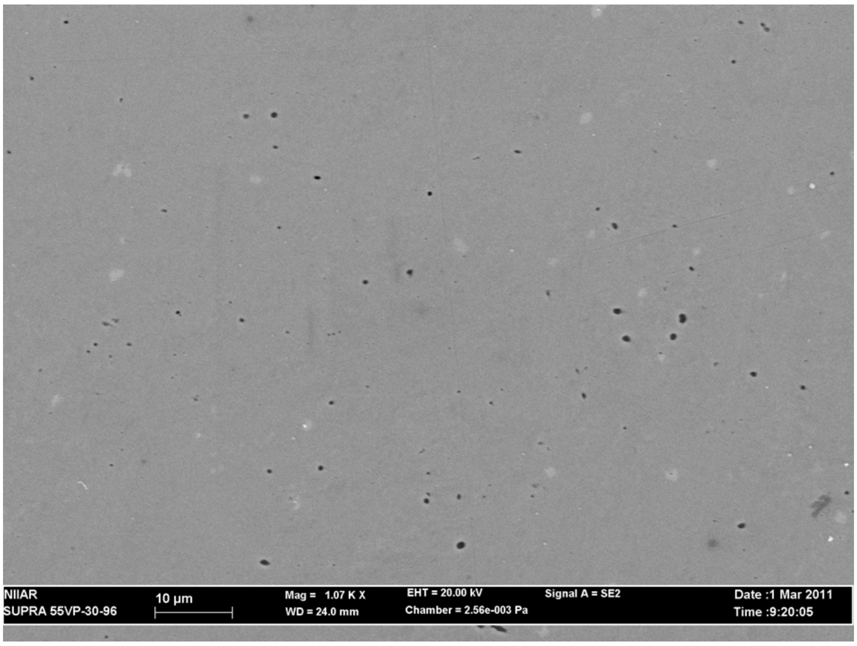

FIG. 3.1. Surface of a sample prepared for investigation.

An important point is that in this case it is difficult to distinguish any peculiarities in the image received in secondary electrons.

Figs 3.2 (Top) and 3.2 (Bottom) present band contrast maps of the slightly-deformed sample (Fig. 3.2 (Top)) and that one after intensive plastic deformation (Fig. 3.2 (Bottom)). When comparing the Figures, one can see a significant difference between the diffraction maps.

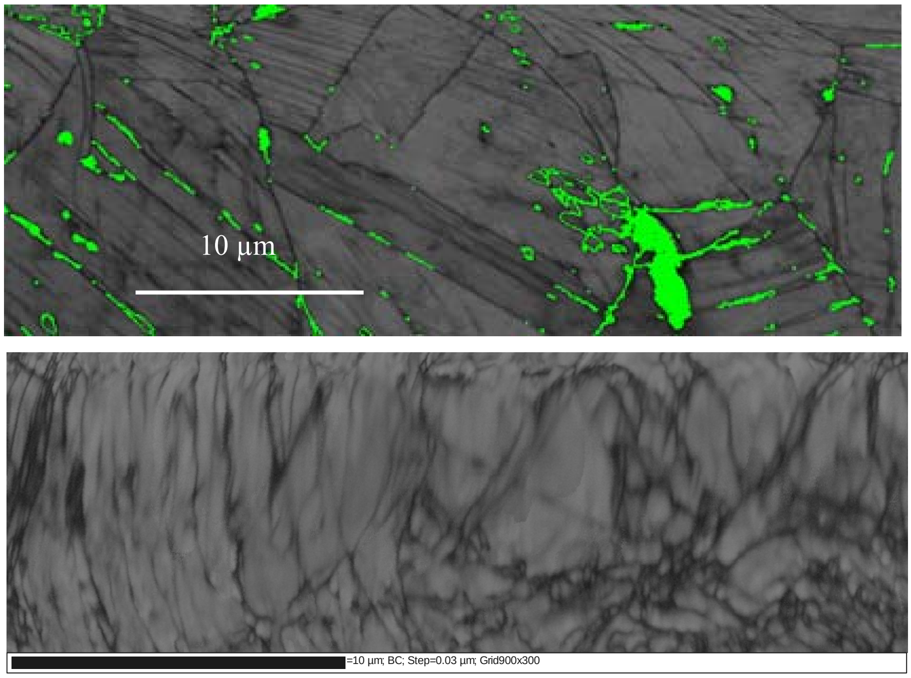

FIG. 3.2. Band contrast map of slightly-deformed steel (Top). In green, there are areas with zero decisions related to low quality of the slightly-deformed sample surface preparation. Band contrast map of steel after intensive plastic deformation by the equal channel angular pressing method (Bottom).

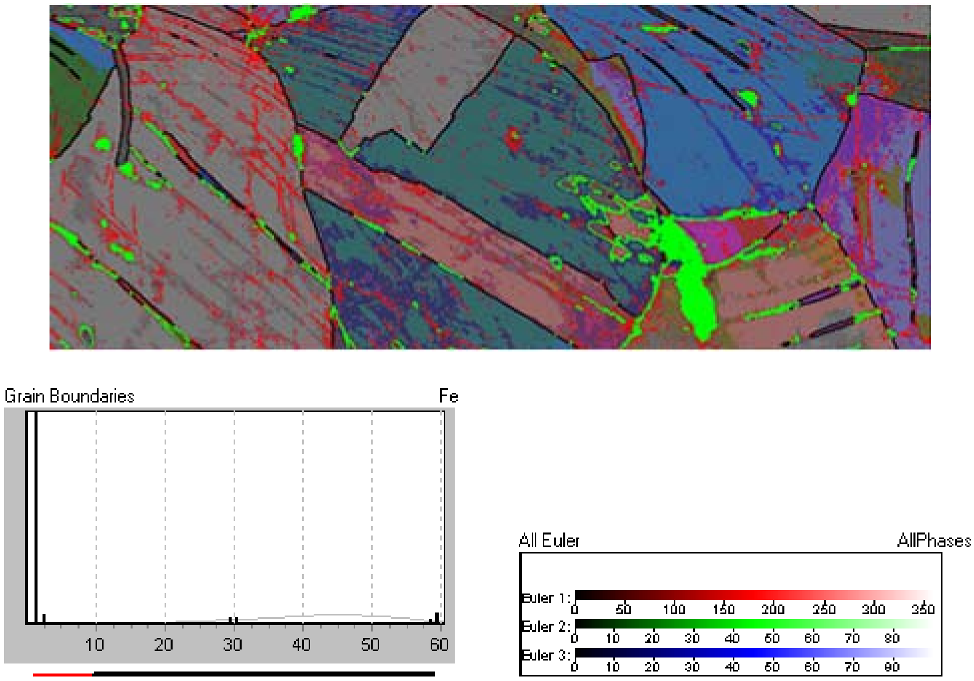

FIG. 3.3. Orientation map of the slightly-deformed steel (Euler angles). Black lines – grain boundaries (›10°), red lines – sub-boundaries (›1°), shades of separate surface areas coloring – intra-granular misorientation (‹1°).

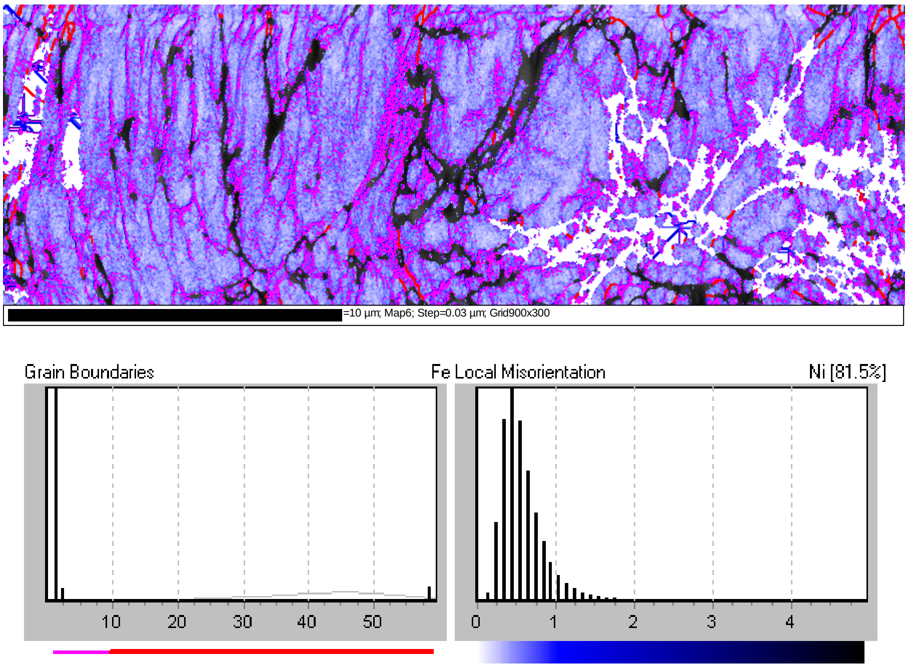

FIG. 3.4. Grain boundary map (red lines), sub-boundaries (purple lines) and intra-granular misorientation (lines colored from white to lilac) for an AISI321 steel sample after intensive plastic deformation by the equal channel angular pressing method. Non indexed regions are colored black.

Fig. 3.3 shows an orientation map of the slightly-deformed sample surface areas, grain boundaries, sub-boundaries and intra-granular misorientation. Fig. 3.4 presents a map of grain boundaries, sub-boundaries and intra-granular misorientation for an AISI321 steel sample after intensive plastic deformation by the equal channel angular pressing method.

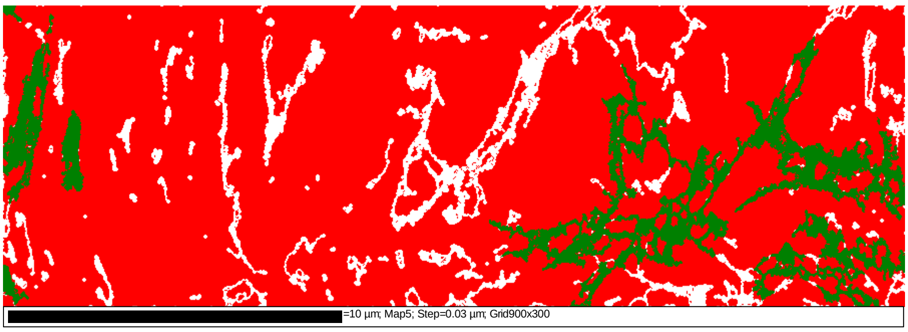

FIG. 3.5. Phase map from the steel after intensive plastic deformation by the method of equal channel angular pressing. Face centered cubic (FCC) lattices – red color, body centered cubic (BCC) lattices – green color. Non indexed regions are colored white.

Fig. 3.5 presents a distribution of regions with FCC and BCC lattices on the surface of a sample after intensive plastic deformation by the equal channel angular pressing method.

When comparing Figures 3.4 and 3.5, one may contend that in the area of a phase with the FCC lattice, there is a misorientation of the neighboring crystalline lattice areas; the average misorientation value is about 0.4–0.6 degrees. At the same time, in the examined area, there are practically no grain boundaries with a misorientation more than 10 degrees and there are a lot of sub-boundaries with a misorientation of 1.5–2 degrees.

In the comparable area of a slightly-deformed sample, one can observe both grain boundaries (misorientation is more that 10 degrees, Fig. 3.3, black lines) and sub-boundaries (Fig. 3.3, red lines). The average value of the local misorientation (inside sub-grains) achieves 0.25–0.3 degrees in the slightly-deformed sample (Figs 3.6–3.7).

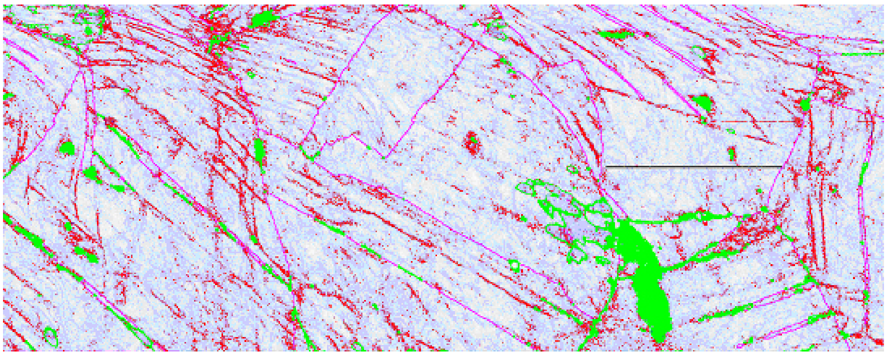

FIG. 3.6. Map of grain boundaries distribution (purple lines), sub-grain boundaries distribution (red lines) and local misorientation distribution (lilac shades) for a slightly-deformed sample. Non indexed regions are colored green. Straight black line is a place for local misorientations profile.

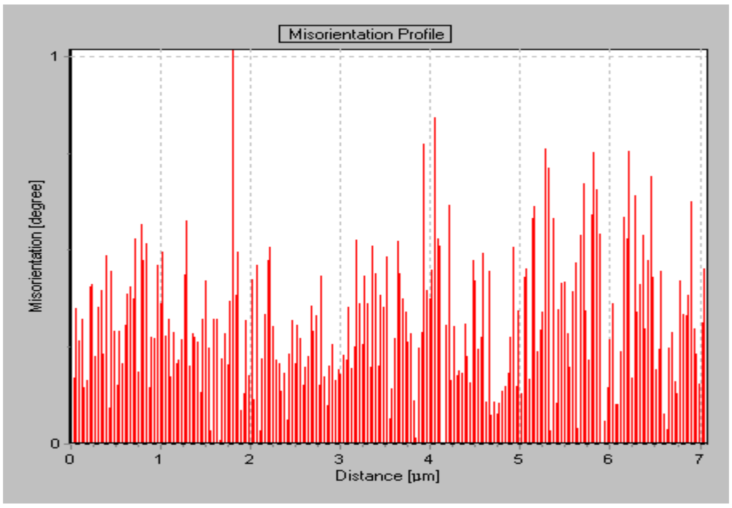

FIG. 3.7. Profile of local misorientations for a slightly-deformed sample.

Fig. 3.8 presents a profile of the local misorientations distribution for an area with the FCC lattice for a sample after intensive plastic deformation by the equal channel angular pressing method (straight line in Fig. 3.7). The average local misorientation achieves 0.4–0.6 degrees.

As for a sample after intensive plastic deformation by the equal channel angular pressing method, there are practically no local misorientations in the BCC lattice phase area (white area in Fig. 3.9).

The comparison of the appearance and distribution of non-indexed areas (a sample after intensive plastic deformation) and areas with FCC lattice allows au to suppose that non-indexed areas show the maximal local misorientation (maximal deformed areas with the FCC lattice), which state is a transition from an FCC to BCC lattice. An insignificant increase of the deformation level of these areas should result in the crystalline lattice conversion.

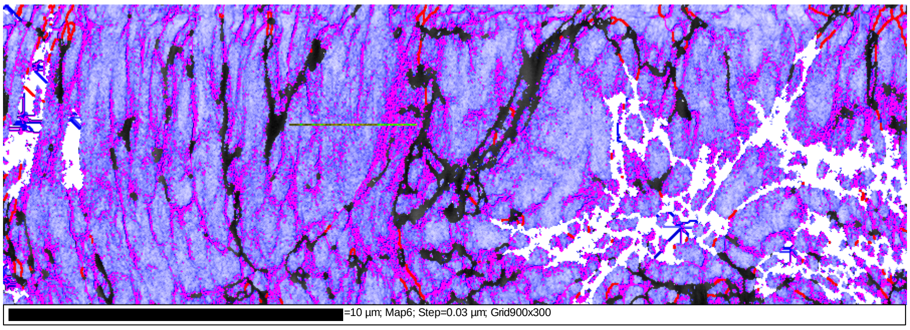

FIG. 3.8. Grain boundaries distribution map (red lines), sub-boundaries (purple lines), local misorientations (lilac shades) for a sample after intensive plastic deformation by the equal channel angular pressing method. Non indexed regions are colored black. A straight line section is local misorientations profile (phase with FCC lattice).

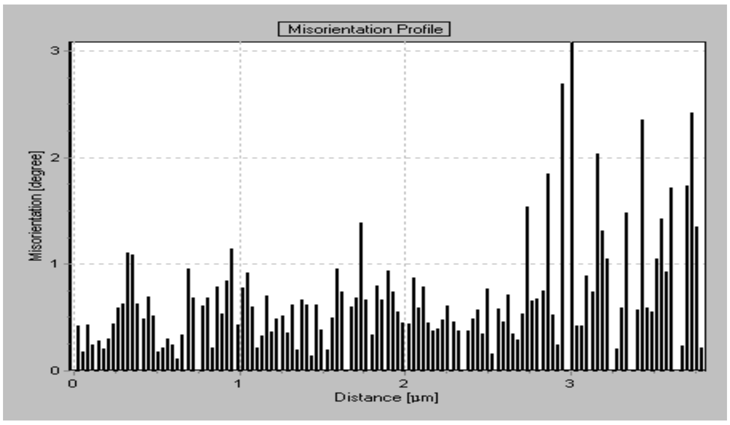

FIG. 3.9. Profile of local misorientations after intensive plastic deformation by the equal channel angular pressing method.

4. CONCLUSION

As a result of examinations of the AISI321 steel structure after intensive plastic deformation by the method of equal channel angular pressing using the electron backscattered diffraction registration and the analysis system HKL EBSD Premium System, it was determined that:

- the technique allows the detection and distribution of the secondary phase in space; the character of distribution of the secondary phase makes it possible to assume that highly deformed (highly stressed) areas of austenite, as well as areas with zero decisions (maximum deformed areas) are places of origin of the secondary phase;
- the technique provides a possibility to define misorientations in a condition of intensive plastic deformation; it is defined that the size of misorientations (stresses) in the secondary phase is significantly less than that of misorientations in the basic phase; in the examined material is found to have no misorientations in the secondary phase areas;
- the technique makes it possible to define the presence of large- and small-angle boundaries in the complex-deformed condition; it is defined that after intensive plastic deformation by a method of equal channel angular pressing, the material is found to have practically no large-angle (intergranular) boundaries, but the density of small-angle boundaries (sub boundaries) is very high.

The obtained results allow considering the EBSD technique as a highly effective procedure for examination of reactor materials in a condition of intensive plastic deformation.