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EBSD Analyses On Metallic Fuel Samples: Tips and Improvements in Sample Preparation

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CONTENTS

FIGURES

TABLES

1. INTRODUCTION

Electron Backscatter Diffraction (EBSD) is an analytic technique used to evaluate the microstructure of a specimen. It can be used to evaluate interesting crystallographic data ranging from grain size to grain orientation, from grain boundaries to stresses. Moreover, in the field of nuclear materials EBSD can aid in estimating properties important for reactor performance such as dislocation and residual strain.[1] EBSD patterns are generated by focusing a stationary electron gun on the specimen and detecting the backscatter electrons that satisfy Bragg conditions, and thus, are diffracted by the crystal planes forming the so called Kikuchi bands (Figure 1). These bands correspond to each of the lattice diffracting crystal planes and are detected on a phosphor screen via a low-light CCD camera.[2] The patterns are generated within a small interaction volume located at a depth of less than 50-100 nm. Because of this, the data quality is extremely sensitive to the integrity of the crystallographic lattice order at the surface of the sample and thus to sample preparation (Figure 1) and to oxidation.[3] While conventional sample preparation methods are widely used in materials science, they often do not lead to quality electron backscatter patterns needed for the characterization of nuclear materials.[4] This has been related by many authors to the challenges associated with handling radiological specimens, their rapid oxidation, and artifacts in such samples (e.g., high number of defects, precipitates and/or porosities).[4,5,6,7,8] EBSD, however, can be an invaluable technique to evaluate microstructural evolution during fabrication and after irradiation at the required mesoscale for model and simulation. It also provides important information on grain structure necessary to understand irradiation effect, such as grain subdivision. Thus, it is of paramount importance to further the application of this technique to nuclear materials and especially irradiated fuels.

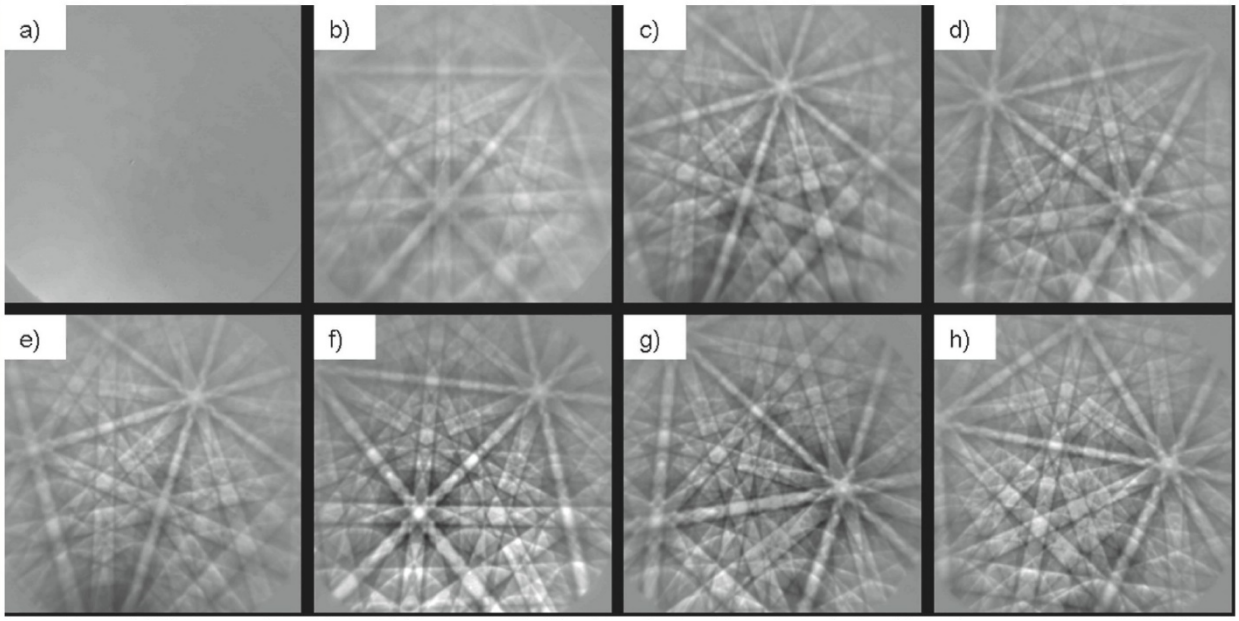

Figure 1. Example of Kikuchi patterns detected on a phosphor screen. These images present increasing quality (from a to h) based on sample preparation technique, from reference 1.

The goal of the described work, performed under the NSUF (Nuclear Science User Facilities) instrument scientist program, was to develop at INL (Idaho National Laboratory) the competencies and procedures for reliable EBSD analyses for reactive metals and, especially, highly radioactive samples, with focus on irradiated metals fuel. Moreover, the role of instrument scientist also entitles the evaluation of new avenues for continuous improvement and the engagement of users in the utilization of new techniques to further the knowledge of nuclear materials behavior. These tasks support the NSUF mission of providing unparalleled research opportunities for nuclear energy and access to world-class nuclear research facilities and technical expertise.

2. EXPERIMENTAL

A review of EBSD mechanisms and available detectors is not the intent of this report. This report aims instead at providing direction for skilled researchers in how to approach EBSD analyses on nuclear material. It is the intent of this report to preserve the knowledge obtained from years of experimental effort and to describe the approach used to progress this technique. This work used a step approach, as described in Figure 2, starting from review of current methodologies and describing the results obtained on nuclear materials. From this review it was evaluated that one limitation for achieving consistent EBSD analyses was related to sample preparation techniques. Thus, focus was posed on the review of current sample preparation techniques in different material science fields. These were compared with the techniques applied in house for radioactive and nuclear materials. This is described in the following paragraphs.

Different EBSD systems are available at the Materials and Fuel Complex at INL for EBSD analyses on such materials. This work was performed using all the available systems and comparing their capabilities, namely:

1. A quanta 3D FEG Dual Beam equipped with the EDAX Hikari EBSD (14 megapixel, 200 pps).
2. The JEOL-IT-500-HR equipped with an Oxford Symmetry Detector (1.2 megapixel, 3000 pps).
3. FEI Helios dual beam plasma focused ion beam/scanning electron microscope (PFIB/SEM) equipped with a Hikari EBSD.

Two other systems (the JEOL-7000 and JEOL-7600) are currently being upgraded with Oxford Symmetry Detector (1.2 megapixel, 3000 pps).

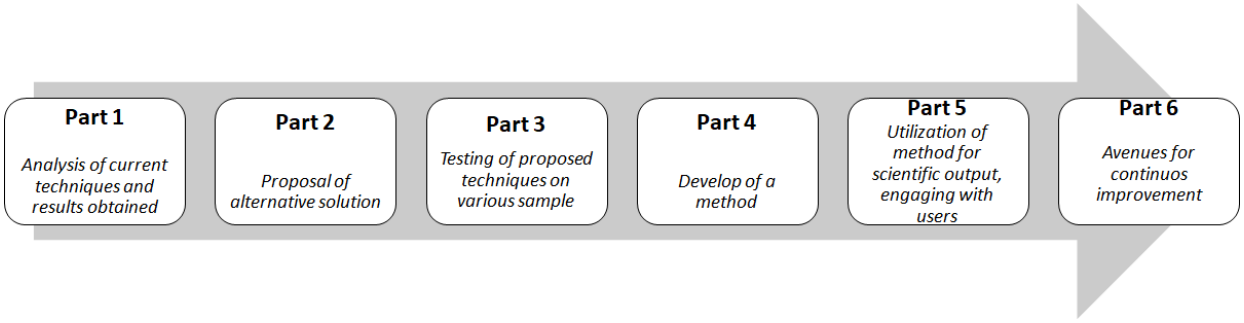

Figure 2. Workflow applied in this project via a step approach.

2.1 Classical Sample Preparation

Various sample preparation techniques are available commercially. The one present in our laboratory has been modified and optimized for use on radioactive specimens. “Metallographic sample preparation has evolved from a black art to the highly precise scientific technique.”[9] Indeed, the preparation of samples requires deep knowledge of material properties and of the process that the specimen was subject to. A literature search before attempting the preparation of a specimen for the first time is suggested to evaluate which technique has already been developed and fine tune it to the application of interest.[9] Different processes are necessary to obtain a specimen analyzable by SEM-EBSD, which are included in sample preparation:

1. **Sectioning.** Sectioning is performed to obtain the specimen of interest from the desired position in the bulk material. The objective during this process is to avoid modifying the microstructure to be analyzed. High speed saws, low speed saws, and shears are available in house to section radioactive specimens. Their application must be considered and the modified layer (mechanically or thermally) during section must be evaluated. This will need to be eliminated during the following steps of sample preparation. It is also important to consider which lubricant to use in saw applications and evaluate its interaction with the specimen of interest and its reaction with the elements present in the

sample. Fire hazard must also be considered, especially when working with metals if small powders are generated while sectioning.

2. **Mounting.** Sectioned samples are usually mounted in an appropriate sample holder compatible with the SEM system and used for further preparation. In our laboratory, epoxy mounts have been used in the past. It has been found that small specimens facilitate positioning in the SEM chamber for EBSD and minimize radioactive dose from the sample. Thus, it has been preferred, whenever possible, to use small, recessed SEM pin stubs (12 mm), a.k.a., mini-mount, rather than the classical metallurgical mounts of over 1 inch diameter. This also minimizes the charging of the sample due to excessive epoxy contained in the mount during analyses, which can render the analyses unreliable. Moreover, having less epoxy also minimizes the coating layer.

3. **Grinding.** The purpose of this step is to remove all sectioning damage and establish a flat surface with a uniform scratch pattern that is suitable for further polishing.[9] Most materials sectioned with a precision saw require a single grinding step only, but some softer materials benefit from multiple steps. Typically, SiC paper is used for this process.[10]

4. **Polishing.** A sample is then polished with progressively smaller abrasive materials to eliminate subsequently smaller deformation induced by abrasion until the desired surface finishing is achieved. Different types of abrasive and suspension mediums are available to perform this task. Diamond polishing compounds have been suitable for nuclear materials. Typically, polishing is begun with a hard cloth with a coarser abrasive and ended with a softer cloth with a finer abrasive.[10] An automatic polisher has usually been applied in this study. This system, called mechanical polishing, has been compared in this work with different techniques. A review of techniques tested in this experimental investigation to achieve the desired surface finishing for EBSD are reviewed and commented on in the following paragraphs. It must be noticed that these techniques are generally applied after this mechanical polishing step is performed.

5. **Coating.** Before SEM analyses, the sample must be coated to ensure no charge builds up during analyses when the electron beam interrogates the samples. As the signal for EBSD comes from a shallow depth in the samples (50-100 nm), coating can reduce the signal and sometimes impede EBSD analyses. Two techniques have been used in this study. The first consists of covering the sample during coating and performing an extensive coating on the epoxy followed by a very light coating on the sample. This ensures conductivity of the specimen while preserving the surface of analyses. The second method consists of using silver paint to cover the epoxy region and the edge of the sample, avoiding coating the area of analyses. Both techniques have been shown to be successful for this application. However, the second has been better suited for bigger mounts, with large epoxy areas to cover.

2.2 Further Sample Preparation Techniques

2.2.1 Vibratory Polishing

Vibratory polishing is based on a horizontal vibratory motion. This is reported to generate a very efficient polishing action with outstanding flatness and superior quality results.[10] It yields less deformation and flatter surfaces and produces a stress-free surface. It can be seen in *Figure 3* the effect of vibratory polishing on a uranium-based material. It can be seen that residual stress is eliminated using vibratory polishing. Moreover, it is noticed that such stresses can strongly influence EBSD results, as texture information was lost due to erroneous sample preparation (*Figure 3*). In our work, vibratory polishing was generally performed using a colloidal SiO_2 solution.

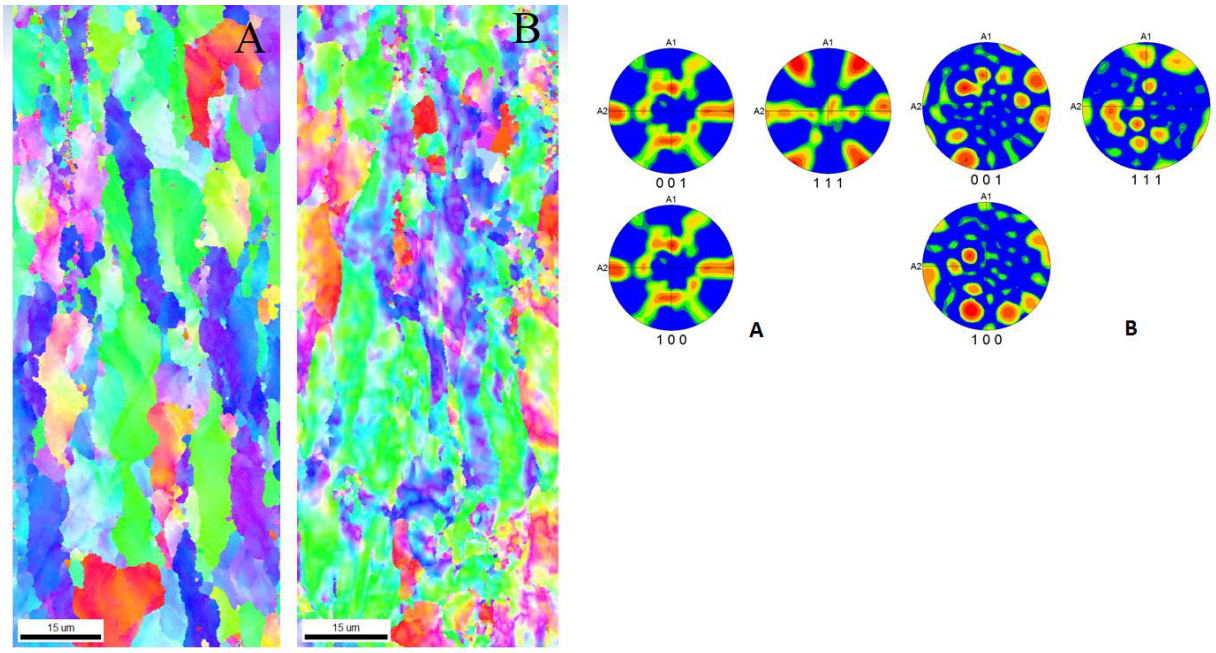

Figure 3. Example of Inverse pole figure maps (left) and orientation distribution function for pole figure (right) for a Uranium based fuel from reference 11. The effect of surface finishing comparing vibratory polishing (A) vs. mechanical polishing finishing at 0.1 µm (B) is shown; both samples were finally cleaned in chamber with the focused ion beam.

2.2.2 Electropolishing

Electropolishing is based on material removal from the sample surface through electrolytic action. Most surface irregularities and any deformation layer on the surface are removed by this method.[10] The selection of the appropriate electrolyte solution is specific to the given material. In the nuclear material application, the necessity of handling material with high contact does and the production of liquid acid radioactive waste generally discourages the use of this technique.[1] Different solution were tested and applied in our study. Table 1 shows a summary of the best results obtained for the different materials tested. Finally, a challenge present by this technique is the presence of precipitates in the desired sample. Indeed, difference in reaction rates may generate inhomogeneity on the surface.

2.2.3 Ion Milling

Ion milling involves the bombarding of the sample surface with an energetic ion beam under vacuum.[10] The bombardment erodes the surface but can damage it by ion implantation that can result in the formation of an amorphous layer. A previous study from our colleagues,[5] reported in *Figure 4*, showed that the amorphization layer can be minimized with the glazing technique. Two different methods have been investigated, both available in our laboratories for work with irradiated specimens:

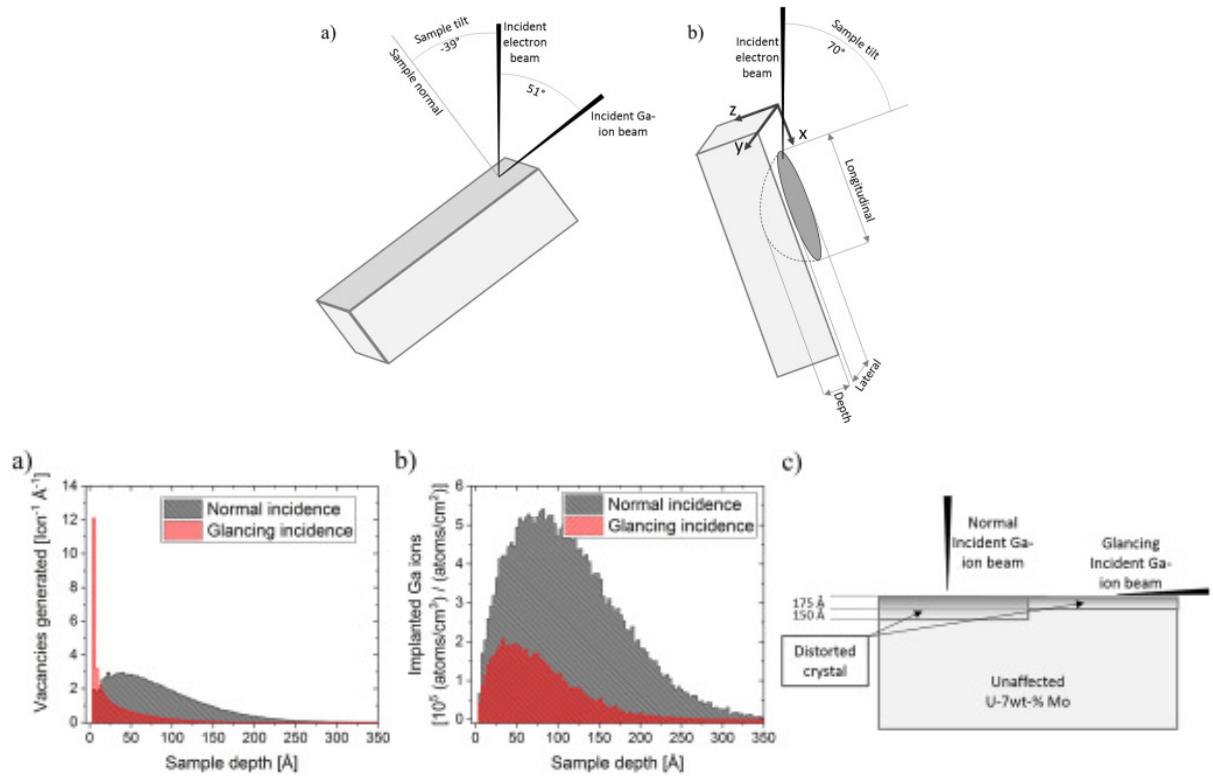

Figure 4. From reference 5: Top figures A) glazing technique, B) EBSD analyzes configurations. Bottom figures show the results of software calculation of damage induced in a U-Mo sample A) and ion implemented region B) with FIB method via incident beam or glazing technique. A schematic of the layer affected is also shown in bottom figure C).

1. Ion milling using the PECS (precision etching coating system). This is a tabletop system that applies an argon milling tool for polishing as well as coating samples. The bombardment is performed on a macroscopic scale; thus, this system provides a large area for analyses. Our investigation was unsuccessful on this system for nuclear material. This was mostly related to cross contamination of the sample. Cross contamination was created using decontamination paint which is required by laboratory radiation control procedures to contain spread of material contamination, fixing it to the sample. However, during milling, paint was vaporized and covered the sample and impeding EBSD analyses. Moreover, the transfer from PECS to SEM chamber required time in air, which may induce oxidation during transfer. Another issued encountered using this system was the preferential etching of the matrix with respect to precipitates in U fuels, as shown in *Figure 5*. This created an uneven surface and shadowing effect impeding EBSD analyses.

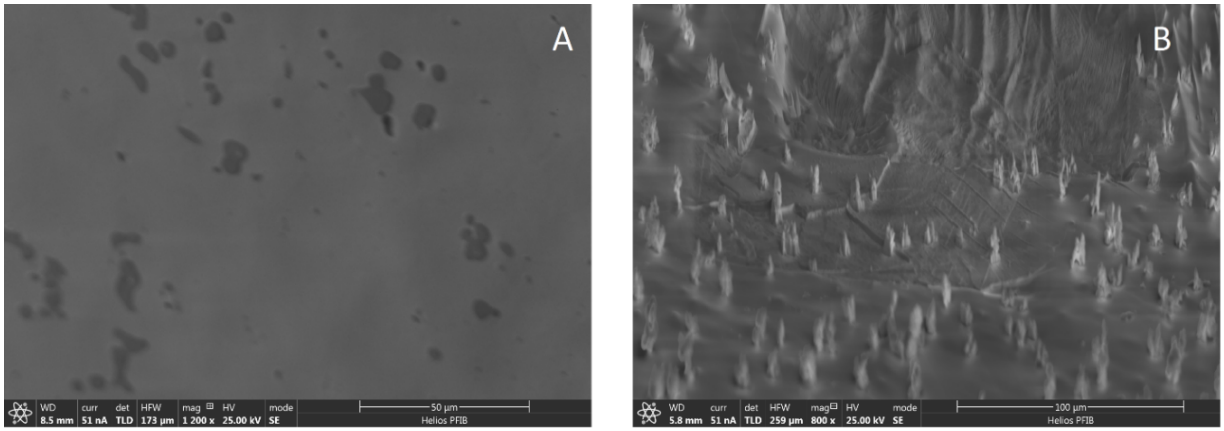

Figure 5. Example of the effect of ion milling by PECS on U (B) compared to electropolishing (A).

2. Focused Ion Beam (FIB) systems operate in the nano-micron scale region. The application of FIB for sample preparation for EBSD has previously been described in depth in references 4,5,7. Two FIB systems have been tested in this investigation: The Helios, operating with Xe ion source, and the Quanta, operating with Ga source. These systems present both pros and con in their application. The Helios can cut larger amount of material with minimal amorphization layer with respect to the Quanta. However, the Xe source can generate large curtaining due to beam broadening that can hinder the results.[12] Two techniques were analyzed for EBSD preparation: 1) glazing technique described in reference 5, and 2) large window lift out, as described in reference 4 and shown in *Figure 6*. Both techniques yield good results. The first permits the analyses of large areas, however porosities and precipitates seem more susceptible to curtaining effect. The second has yielded better results and may be related to the use of platinum cap permit to have a smooth surface and avoid curtain formation. The importance of the use of these systems for EBSD analyses is that at INL for both FIBs, EBSD detectors are mounted on the chamber, thus, the analyses can proceed without the need of sample transfer.

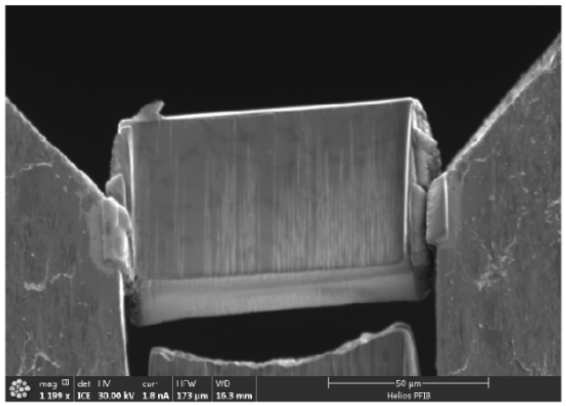

Figure 6. Example of lift out sample for EBSD.

After the review of these techniques, some conclusions on their application are shown in Table 1. These techniques have been following experimental tested in our laboratory and on the materials of interest. Results on the experimental optimization of the parameters for each technique are also presented in the following paragraph. The investigation plan is summarized in Figure 7. This plan was developed based on literature reviewed, progressing from materials in which EBSD was widely reported and considered easy to more complex materials.

Table 1. Analyses of the available techniques in INL laboratory, and their evaluated characteristics.

Technique	Pros	Cons
1. Mechanical polish (down to 0.05 µm)	Large areas, Available expertise, Multiple samples, Fast	Generally, needs of a follow up step
2. Vibratory polish	Large areas, Available expertise, Multiple samples, alkaline effect	Performed in air
3. Electropolish	Large areas, Available expertise, Fast	Performed in air
4. Ion Milling: PECS	Good results obtained in the past, Available expertise, Fast	Spreading of contaminants Transfer in air
5. Ion Milling: FIB/P-FIB	Good finishing, in vacuum chamber with EBSD detector	Long, small areas

2.3 Results

2.3.1 Cladding Materials-Unirradiated

Steel preparation was based on classical mechanical polishing techniques down to 0.05 µm. EBSD results were reliable even when simple mechanical polishing was used and, thus, no following steps were applied for sample preparation. The optimized parameters are reported in Table 2.

Aluminum, which is a soft metal, has been reported to be more difficult to prepare reference 9. For these samples, different mechanical polishing techniques were tested but did not achieve the surface finishing desired. Partial results were obtained with vibratory polishing with colloidal SiO2. Higher CI were, however, obtain using ion milling, as described in Table 2.

Zirconium has also been reported as a difficult metal to prepare for EBSD.[9] Moreover, such material is of importance in nuclear application due to its conventional application in cladding materials (e.g., Zircaloy) and as an alloying element in metallic fuel. Good results were obtained both with the PECS and electropolishing, obtaining higher CI with the electropolishing (presented in Figure 8 and in Table 2.

2.3.2 Metallic Nuclear fuel-Unirradiated

For these materials, various tests were undergone. Difficulties were related to handling nuclear materials and mostly related to contamination/radiation control procedures. Plutonium, especially, requires the use of Personal Protective Equipment and laboratory closure during sample transfer and loading. This may lead to increased cost for these analyses. Improved capabilities at the irradiated material characterization laboratory such as the enclosed shielded glovebox avoid such transfers and procedure, while keeping the samples in a controlled environment. This system, moreover, avoids oxidation, which was one of the goals to obtain successful EBSD analyses on these materials.

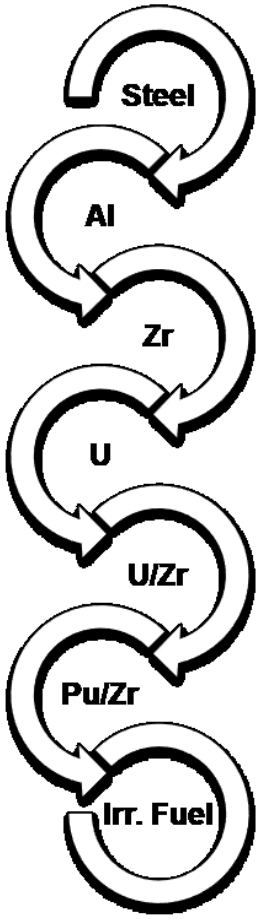

Figure 7. Samples analyzed.

Table 2. Optimized "recipes" proposed from the experimental analyses. DP stands for Diamond Paste, DS indicates Diamond Solution.

Steels	Al	Zr	U	U-alloys (Zr/Mo)	Pu and alloys	Irradiated fuel

Mechanical polishing	Mechanical polishing +PECS	Mechanical polishing + Electropolishing	Mechanical polishing	Mechanical polishing	Mechanical polishing	Mechanical polishing
30 µm DP to plane			Add a 6 µm DP	Add a 6 µm DP	Add a 6 µm DP	Add a 6 µm DP
9 µm DP	1.5 keV 12 µA 1hr	Ethanol+ 7% $HClO_4$	1-Electropolishing 7% $HClO_4$	1-Vibratory polishing	1-Electropolishing 7% $HClO_4$	FIB
3 µm DS	Rocking 25 rpm 80°	12 V 35 mA	38 V 1030 mA	Colloidal SiO_2 0.05 µm**	12 V 93 mA	
1 µm DS		T -20°C Flow 25	T -10°C Flow 30	Diamond 0.05 µm**	T -10°C Flow 30	
0.05µm DS		2-PECS	2-FIB	2-FIB	2-FIB	

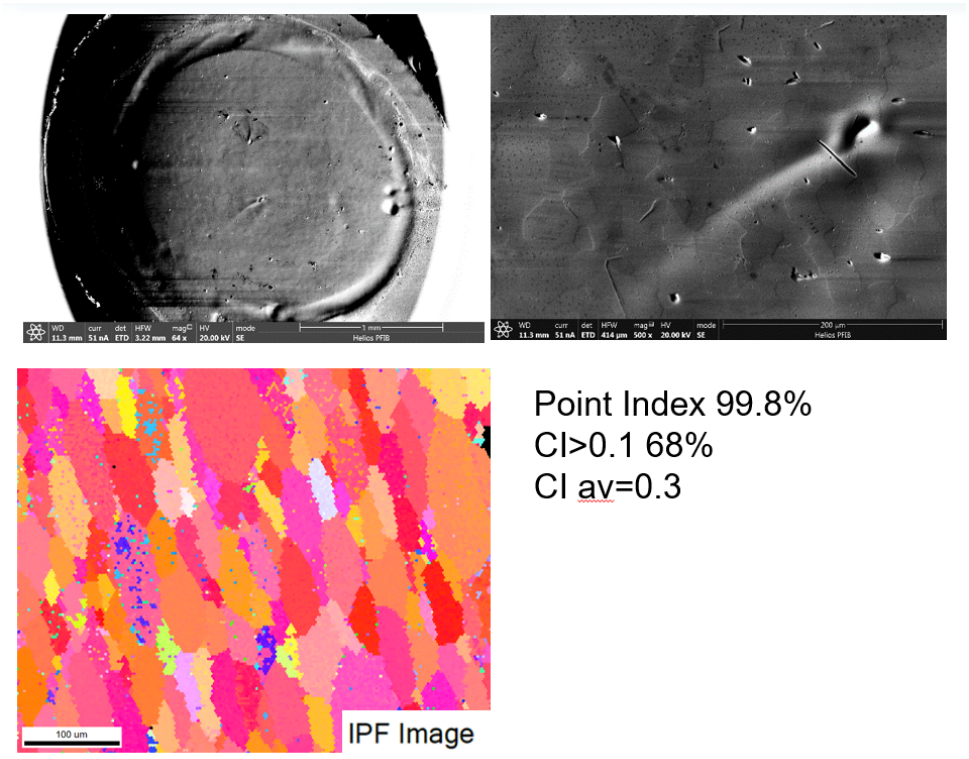

Figure 8. EBSD mapping obtained on Zr, after electropolishing.

Uranium presents a more complex crystal structure at room temperature to be analyzed by EBSD with respect to cubic system of steel (orthorhombic structure). Moreover, uranium has high oxidation rates. This makes EBSD analyses challenging for this specimen. Electropolishing has been reported in the past to provide good results. In these analyses, electropolishing was found to give partial results, while the ion milling with the PECS system was unsuccessful. Two problems, as explained, arose from the use of PECS on these samples: 1) The decontamination paint required by procedure will sputter by the argon beam and thus contaminate the sample. 2) Precipitates in these samples (oxides and carbides) will be preferentially etched (Figure 4), rending the analyses difficult. FIB preparation technique following mechanical polishing leads to reliable EBSD results, with high confidence index even in textured samples (Figure 9). FIB technique, as explained, also minimizes contamination and air transfer. The biggest drawback is the limited area that can be prepared (around 100×100 µm) due to time limitation, thus the area available for analyses can be limited. This can be a problem when analyzing texture if grains are big in the specimen of interest.

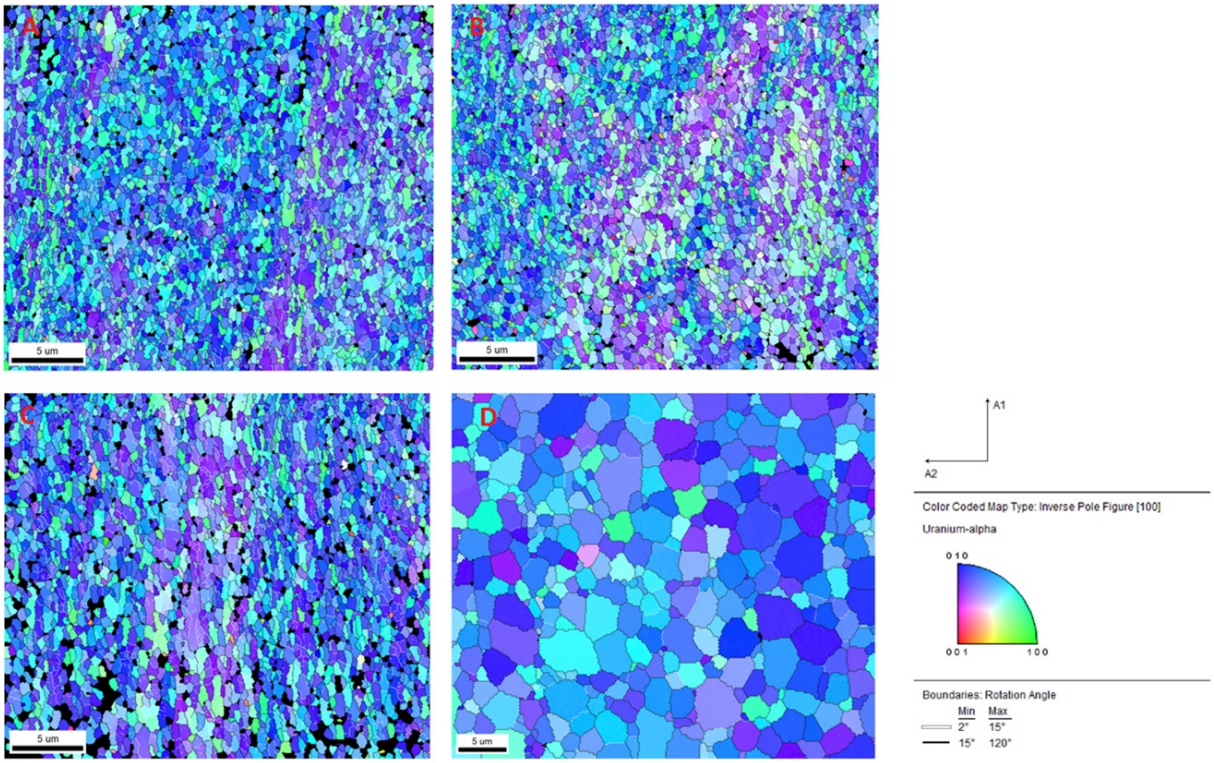

Figure 9. EBSD mapping obtained on textured α-U specimen which underwent different thermal ramps. EBSD was obtained after FIB sample preparation.[12]

Uranium-Zirconium alloys can present different crystal structures based on sample preparation and alloy composition.[13] From the analyses on these specimens, it was noticed that increased Zr content in the sample facilitate the sample preparation and EBSD analyses. This has been related to the fact that Zr enriched phases usually exhibits lower oxidation rates.[14] The sample fabrication process was also shown to influence the results: cast samples were difficult to analyze, probably because of the lamellar microstructure formed. Indeed, the lamellar structure shown in Figure 10 presents very small phases (α/δ), which are under the detection capability of EBSD. Moreover, it is hypothesized that high stresses are presented in this structure, rendering the analyses difficult. When samples were annealed, it was easier to obtain EBSD, and vibratory polishing provided already good results for such samples (Figure 11). The best results were again obtained when FIB preparation was applied. The results from the FIB analyses are not presented here because, due to area limitation on the sample preparation and the large grain size of the annealed sample, only one or two grains could be analyzed with this technique.

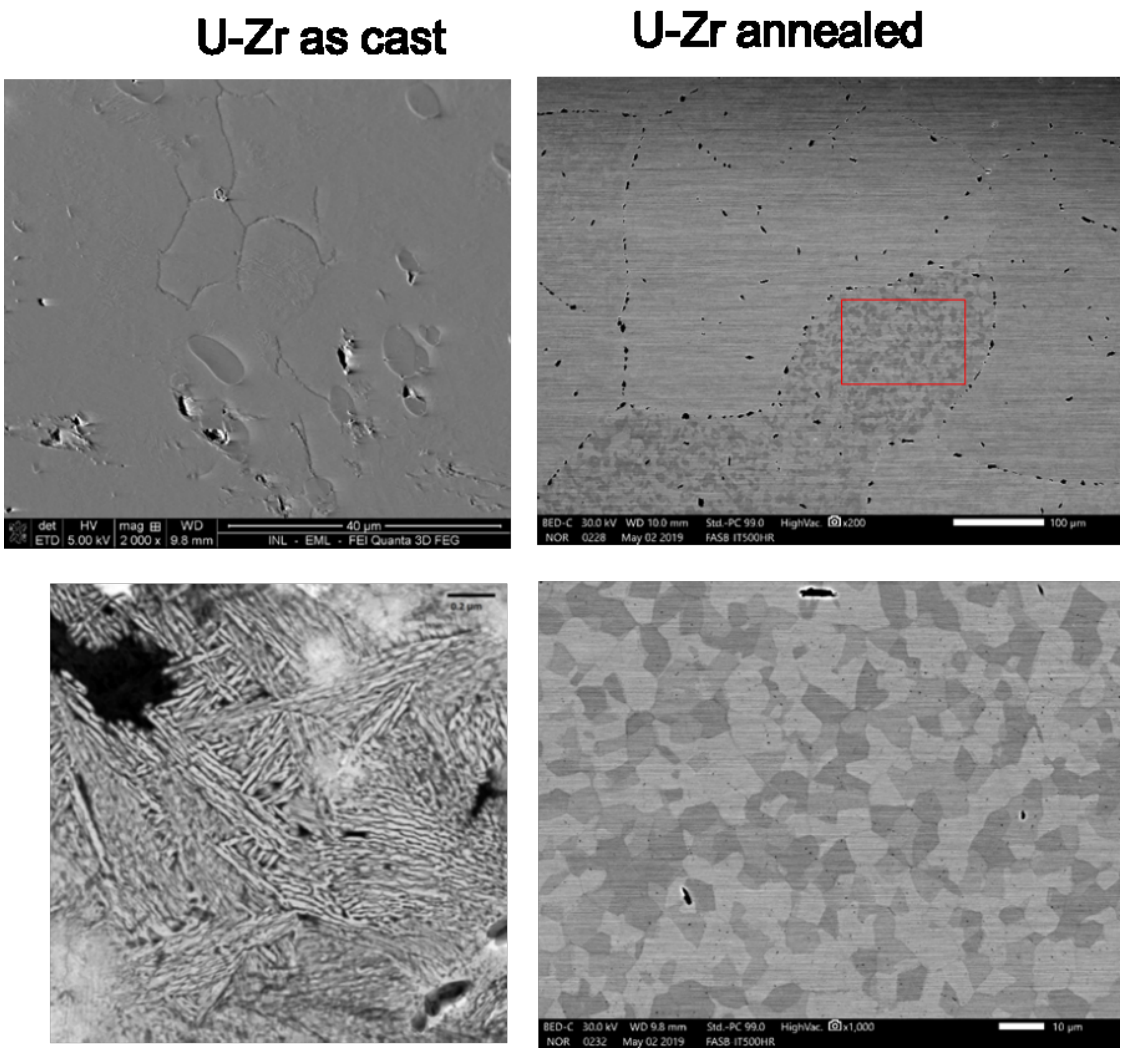

Figure 10. U-Zr showing different microstructure.

Uranium-Molybdenum alloys. These alloys are generally created to stabilize the γ-Uranium cubic structure at room temperature, which provides desired isotropic characteristics. Various techniques have been applied, and similar to Uranium-zirconium alloys, it was found that both mechanical polishing and FIB techniques can provide the surface to adapt to EBSD analyses (Figure 12). It was found that colloidal SiO_2 with a light pressure (3 lb) provided better surface than vibratory polishing. Again, stress induced in the samples during fabrication lead to more difficulties in the analyses and generally required further FIB cleaning after mechanical polish to obtained better indexing (Figure 3). Further details on progress and analyses on U-Mo in our laboratories can be found in reference 15.

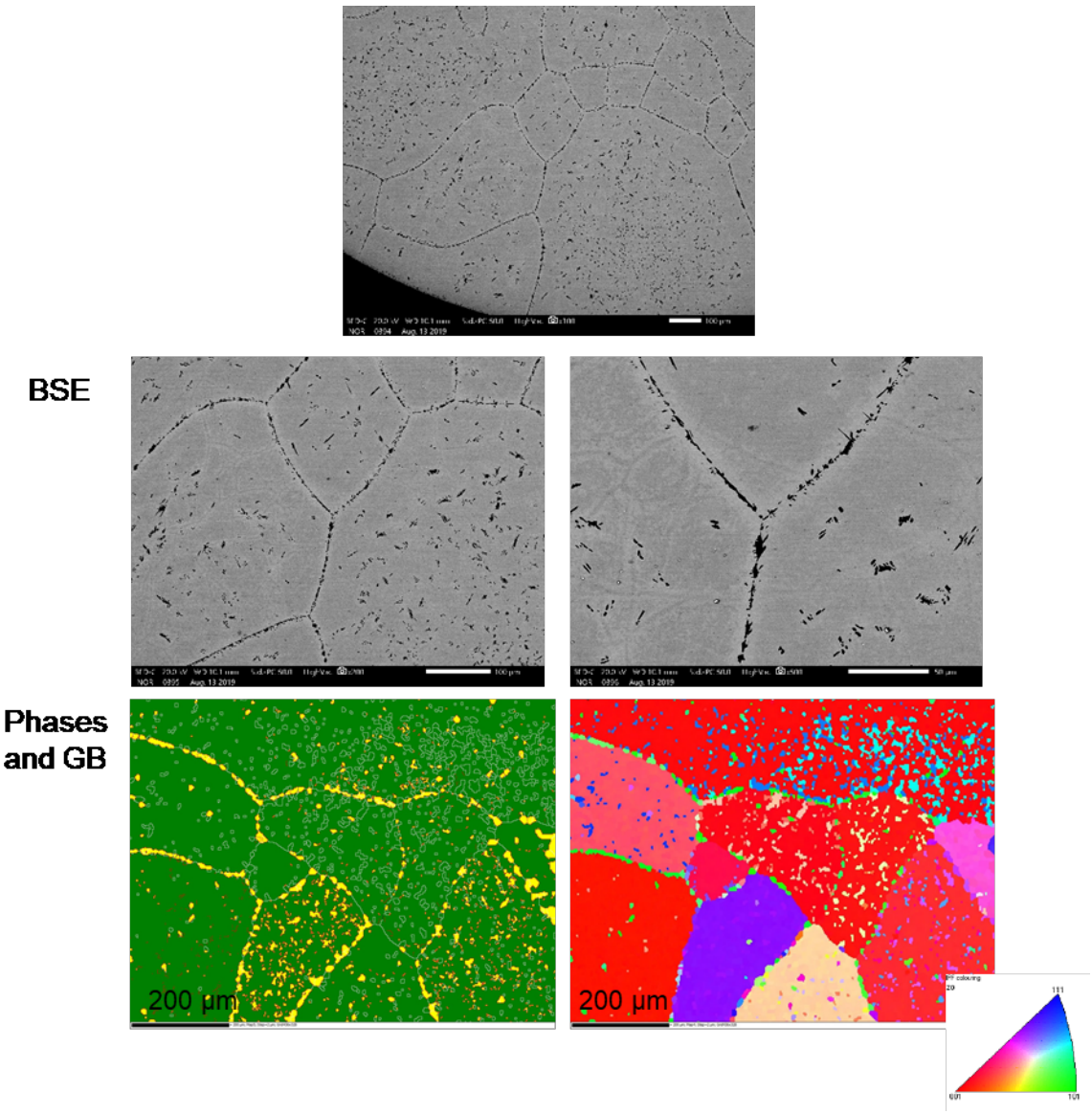

Figure 11. SEM images in Back-Scattered Electron mode (BSE) and EBSD results providing phase analyses and grain orientation on U-Zr alloy.[16]

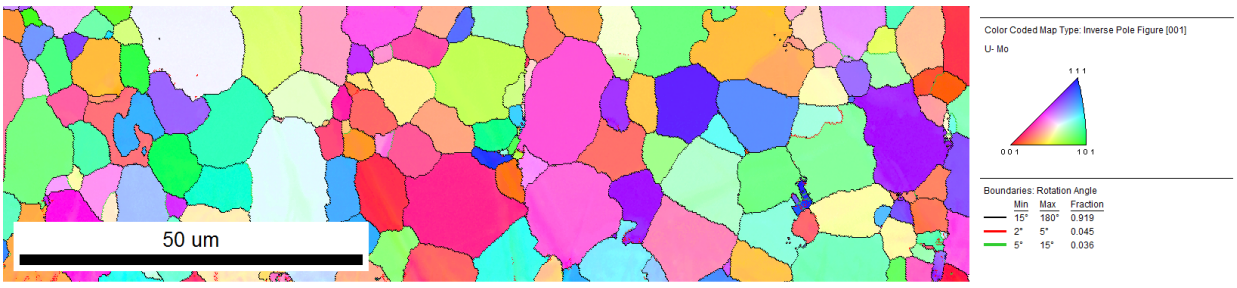

Figure 12. IPF for U-Mo sample, after FIB cleaning.

Plutonium alloys. For these samples, electropolishing was tried with difficulties and did not provide the desired results. It was preferred, due to contamination and successful results in the previous results, to perform final surface cleaning using the FIB after conventional mechanical polishing. For both Pu and it alloy FIB lift outs followed by the glazing technique provided reliable EBSD results (as shown in Figure 13).

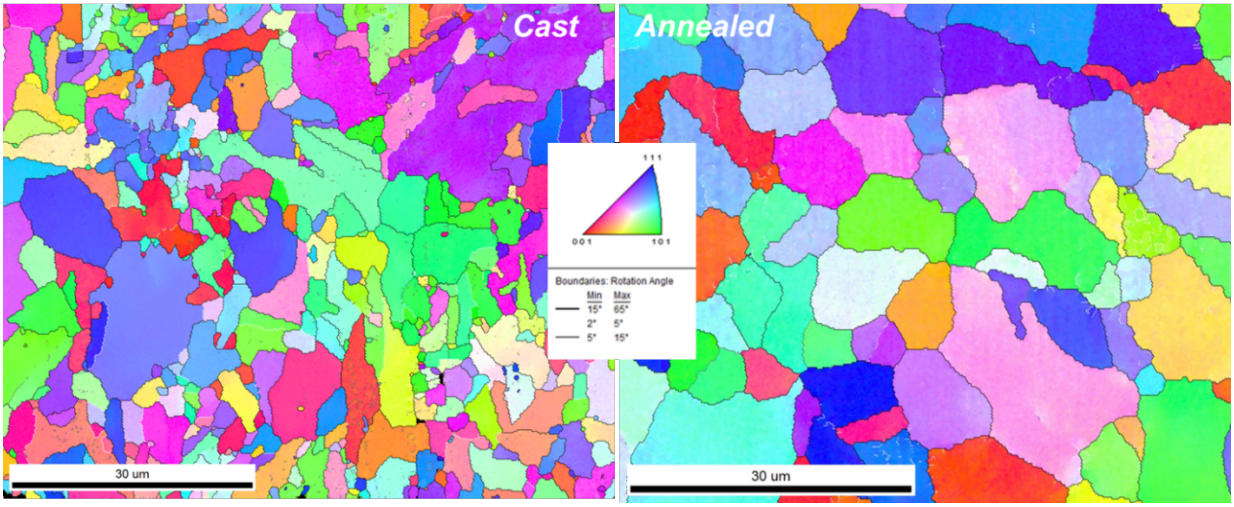

Figure 13. IPF on Pu alloys obtained by FIB, as reported in reference 17.

2.3.3 Irradiated Nuclear Fuel

Irradiated materials represent a complicated system due to multiple phases, small grain structure, and porosities. Similar techniques have been used for the sample preparation. However, being EBSD data quality is extremely sensitive to the integrity of the crystallographic lattice order at the surface of the sample, artifacts such as high number of defects in irradiated fuel have a profound and unavoidable effect on the results. Thus, the results were scarce. Partial results were obtained only when the FIB glazing technique was applied. First, satisfying results were obtained for U-Mo (Figure 14), and following for U-Zr (Figure 15). However, interesting information could be gained such as grain refinement characteristics. In reference 15 it was observed that low angle grain boundaries are formed when the grain subdivided, thus indicating polygonization as the method leading this phenomenon in line with reference 5. For U-Zr, due to the small dimension of the analyzed phases, transmission EBSD was, for the first time, tested, and diffraction patterns were obtained with partial coherent indexing.

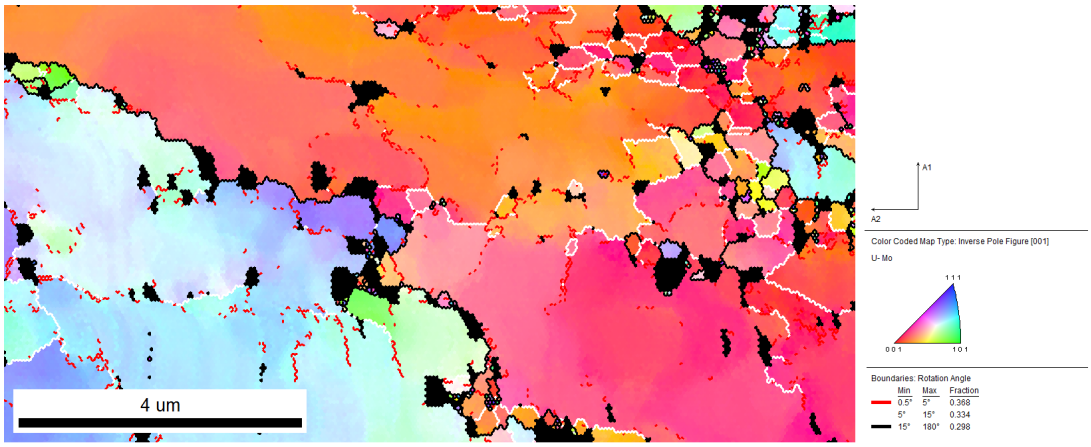

Figure 14. Irradiated sample analyses for U-Mo showing grain refinement.

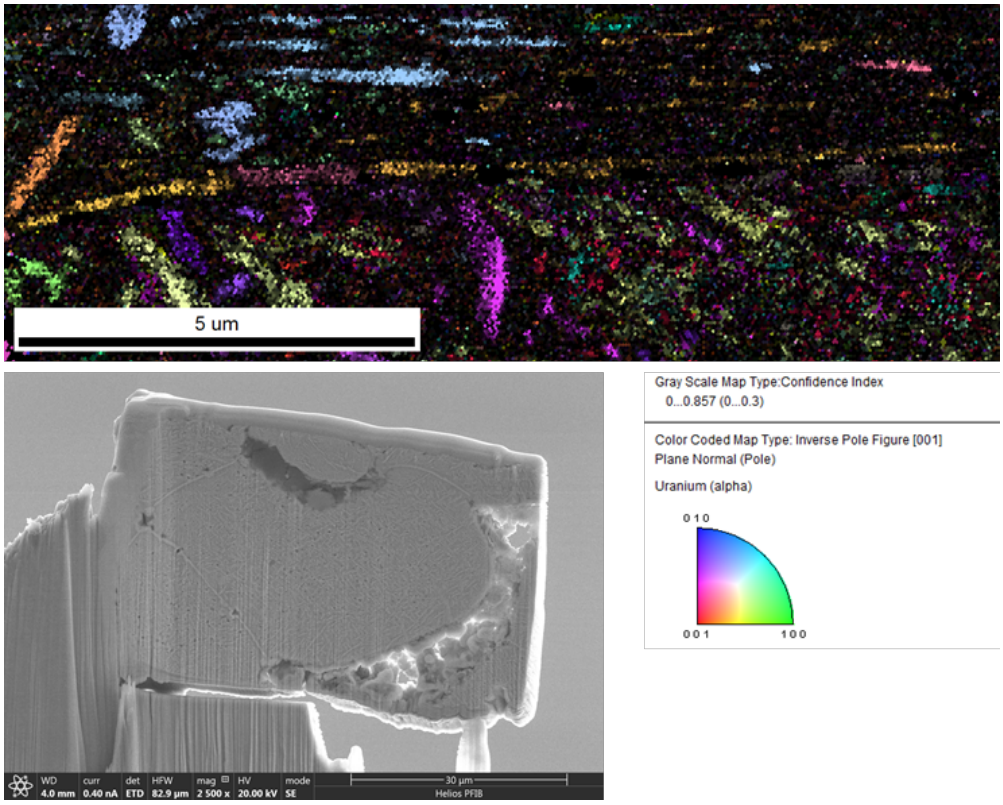

Figure 15. IPF (top) and complex microstructure of an irradiated U-Zr sample.

2.4 Quantitative Analyses Potential and Applications

In the nuclear field it is important to understand the effect of fabrication and irradiation on materials. EBSD analyses has been shown to be useful in obtaining various microstructural parameters relevant to nuclear application. From EBSD analyses, grain size and, thus, the presence of grain refinement and recrystallization process can be observed. Grain boundaries characteristics can be also evaluated. This can confirm the recrystallization process by observing the variation in low angle and high angle grain boundaries. As an example, characteristic grain boundaries abundance was evaluated in four different specimens after different thermal processes (Figure 16). These analyses show that the fourth sample (yellow bar) recrystallized as twin boundaries decreased significantly. EBSD analyses can also provide information on strain in the material by analyzing the grain or kernel average misorientation.

Moreover, understanding, controlling, and monitoring texture evolution during fabrication and irradiation is important to evaluate material properties, as texture can affect material properties up to 20-50% (e.g., Young's modulus, Poisson's ratio, strength ductility, toughness, thermal expansion, and magnetic-electrical properties).[18] This work provides the avenue to also test texture analyses features in the available systems. The different software Channel 5 from Oxford and OIM from EDAX were utilized. While the EDAX presents more flexibility in the visualization of data and control on the cleaning routines applied, Oxford presents interesting 3D features to present the Euler space and the possibility of comparing texture to a standard database (Figure 17). An example of this application can be found in reference 12,15, where texture evolution during fabrication and irradiation was analyzed.

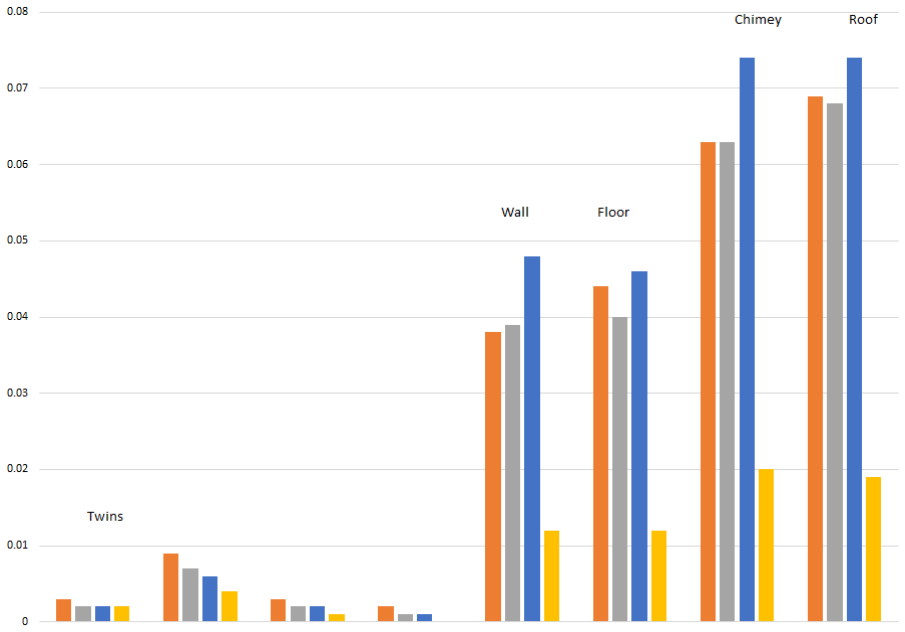

Figure 16. Grain Boundaries evolution for 4 different specimens.

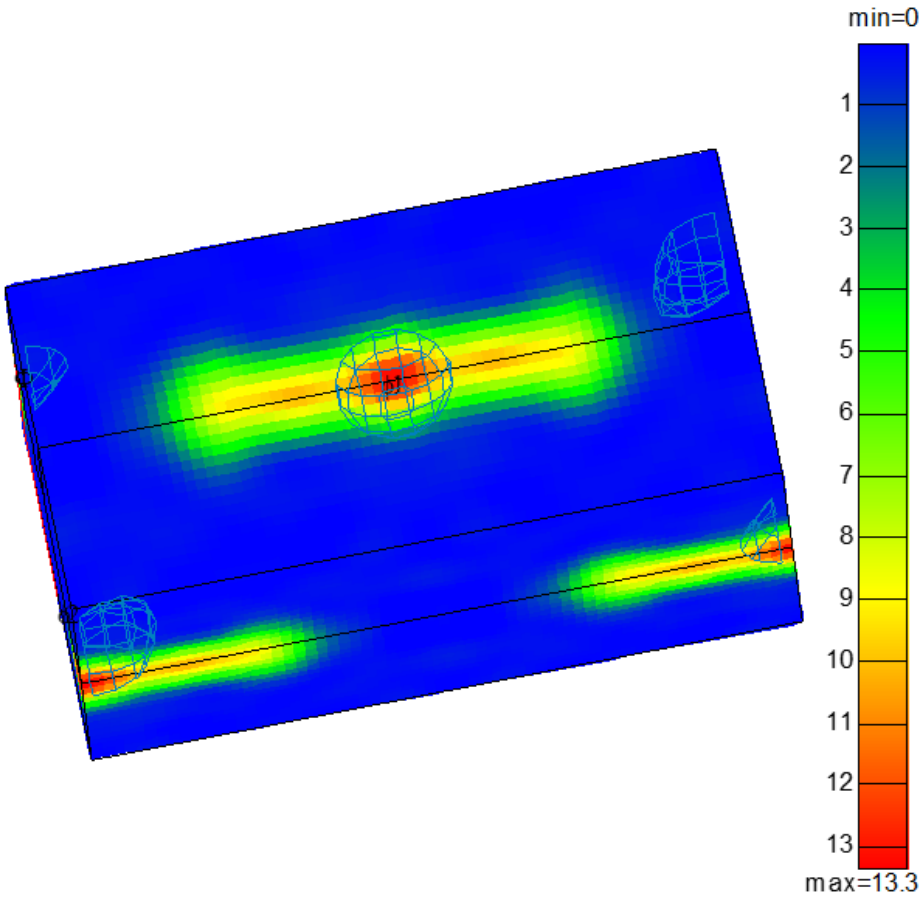

Figure 17. Image of a 3D animation of textured observed in a sample. The lines in light blue show the Gross texture.

3. SUMMARY AND RECOMMENDATION

The report showed improvements and some tips for preparation of metallic fuel specimens. While recognizing that each sample presents different challenges, these guidelines can provide a first step to obtaining the desired EBSD results. EBSD results for fuel specimens, including irradiated samples, were presented. Finally, an overview of EBSD application to analyze different parameters for nuclear fuel performance evaluation was detailed. Further effort should be put into the preparation and analyses of irradiated specimens. Improvement of sample preparation techniques will include electropolishing and vibratory polishing in enclosed environmental controlled atmospheres. Engineered systems to facilitate transfer between different systems is also suggested. This could help with oxidation challenges. Further improvement of current EBSD capabilities could include in situ accessory for a real time analyses of recrystallization, texture, and grain boundaries behavior.

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