

## Applications of Electron Backscatter Diffraction (EBSD) in Materials Research for Nuclear Energy

### Introduction

Increasing demand for energy is one of the most critical challenges facing the world today. Many nations are seeking to reduce their dependence on fossil fuels due to geopolitical instabilities and the inherent problems of pollution and decreasing availability, as well as increasing cost and the risks associated with more advanced extraction technologies. Various energy sources including photovoltaics, wind, hydroelectric, biofuels and nuclear energy are being considered today. In 2008, Baldev Raj, M. Vijayalakshmi, P.R. Vasudeva Rao and K. B. S. Rao of the Indira Gandhi Centre for Atomic Research in India published an excellent article on the subject entitled "Challenges in Materials Research for Sustainable Nuclear Energy". They describe developing technologies in the nuclear industry and the materials challenges that need to be overcome to make these technologies a viable reality for energy production.

The authors outline five long-term targets for making nuclear energy a more attractive energy source: (1) increasing thermal efficiency by shifting to high-temperature reactors; (2) multiple industrial use of high-temperature reactors (i.e. using the heat for commercial purposes such as heating buildings or desalinating water); (3) improved fuels (e.g. thorium) and coolants particularly in fast spectrum reactors; (4) improved safety and reduced nuclear waste through in-situ incineration using accelerator-driven systems; and (5) commercially viable fusion technology. They note increased international cooperation to achieve these objectives and predict commercial fusion energy production in the second half of the 21st century. However, a key step in meeting these objectives lies in overcoming materials challenges posed by these advanced technologies. The general areas of concern are radiation resistance, high-temperature mechanical properties, fuel compatibility, coolant compatibility and fabricability. Generally the core component materials face more severe conditions than the out-of-core materials.

This technical note reviews the materials issues raised in the Raj et al. paper and highlights some of the areas where automated EBSD or Orientation Imaging Microscopy (OIM™) can play an important role in overcoming the materials challenges associated with the next generation of nuclear power. It also briefly reviews some of the research on nuclear materials that has appeared in the scientific literature.

### Current Technology

The objective of much nuclear materials research for current reactor technology is focused on achieving higher "burnup" (energy production per unit quantity of fuel). Table 1 adapted from the Raj *et al* paper shows some of the important components, the current materials used and the major issues involved.

The main consideration in the choice of materials for a thermal reactor is selecting raw materials with low absorption for neutrons. Zirconium meets this requirement. Significant research has been carried out to develop a wide variety of zirconium-based alloys with chemical properties optimized for improved corrosion and irradiation behavior and with minimal tendency toward hydrogen embrittlement as a result of thermomechanical processing.



In order to achieve higher burnup, new materials are needed to overcome issues related to hydriding, irradiation growth and embrittlement.

Reactor Type	Component	Materials	Materials Challenges
thermal	fuel cladding	zirconium-based alloys	neutron economy
thermal	pressure vessels, piping	carbon steels	cost and corrosion
thermal	turbines and steam generators	low alloy steels, 12% Cr steels	cost, corrosion, high temperature and pressure
fast	clad and hexcon	cold worked 316 stainless steel, stainless steel alloy D9 (15% Cr, 15% Ni-Ti stabilized), ferritic steels	void swelling, irradiation creep, irradiation embrittlement, tensile strength, ductility and creep strength, sodium compatibility, fuel & fission products
fast	structural materials	316 stainless steels or 316 L(N) stainless steels	tensile strength, creep low cycle fatigue, creep-fatigue interactions, weldability, fabricability
fast	steam generator	modified 9Cr-10Mo ferritic steels	sodium compatibility, steam corrosion resistance, fretting and wear

Table 1. Materials and material issues for thermal and fast spectrum nuclear reactors for a few critical components.

Fast reactors (or fast neutron reactors) use fast neutrons to sustain the fission chain reaction. Fast reactors can be designed to produce more fissile material than they consume (breeders) or burn the fuel to produce energy and/or transmute long-half-life waste into less troublesome isotopes (burners). Void swelling and irradiation creep are two major materials issues in fast reactors. The dimensional changes and density reduction caused by these factors introduces many engineering problems. In order to achieve higher burnup, these materials are being pushed to their limits. For example, ferritic steels tend to become brittle when exposed to radiation. Research is in progress to overcome this problem through grain boundary engineering (GBE).

Some short term improvements in thermal efficiency for fast reactors are accompanied by an increase in the outlet temperature of the coolant from 823K to about 1,123K. This requires the development of radiation-resistant materials with good high-temperature creep resistance. One promising cladding material is ferritic steel with dispersed nanoparticles of titania and yttria (ODS steel). However, problems with this class of steels include fabricability, deformation texture and anisotropy in mechanical properties.

## New Materials

To achieve higher burnup, advanced radiation resistant materials are being developed including ternary and quaternary

alloys of Zr-Nb. The following statement in Raj et al. spells out the challenges of introducing new materials into the nuclear industry: “A complex interplay of chemistry, microstructure, out-of-core behavior, and in-reactor performance necessitates detailed evaluation and validation of many zirconium-based alloys before acceptance in the nuclear industry.” This certainly applies to any new material, as a candidate material must be validated to make sure it meets the demanding operation conditions existent in nuclear reactors.

A good example in the literature is a study on oxide dispersion strengthened (ODS) steel by Chen, Sridharan, Ukai and Allen (2007). The researchers used a variety of characterization tools including OIM™ and Energy Dispersive Spectroscopy (EDS) to study the corrosion behavior of these materials when exposed to supercritical water, particularly the role of grain boundaries. ODS steels are candidate materials for cladding applications in fast reactors due to their excellent creep properties.

Coupling OIM™ with other analytical techniques such as EDS provides a complete characterization of microstructure – both crystallographic and chemical aspects of the material can be captured. Such information is vital to understanding the “complex interplay” between the chemical and crystallographic aspects of microstructure. As an example, Figure 1 shows simultaneous OIM™/EDS results obtained from an interface between copper oxide and alumina.

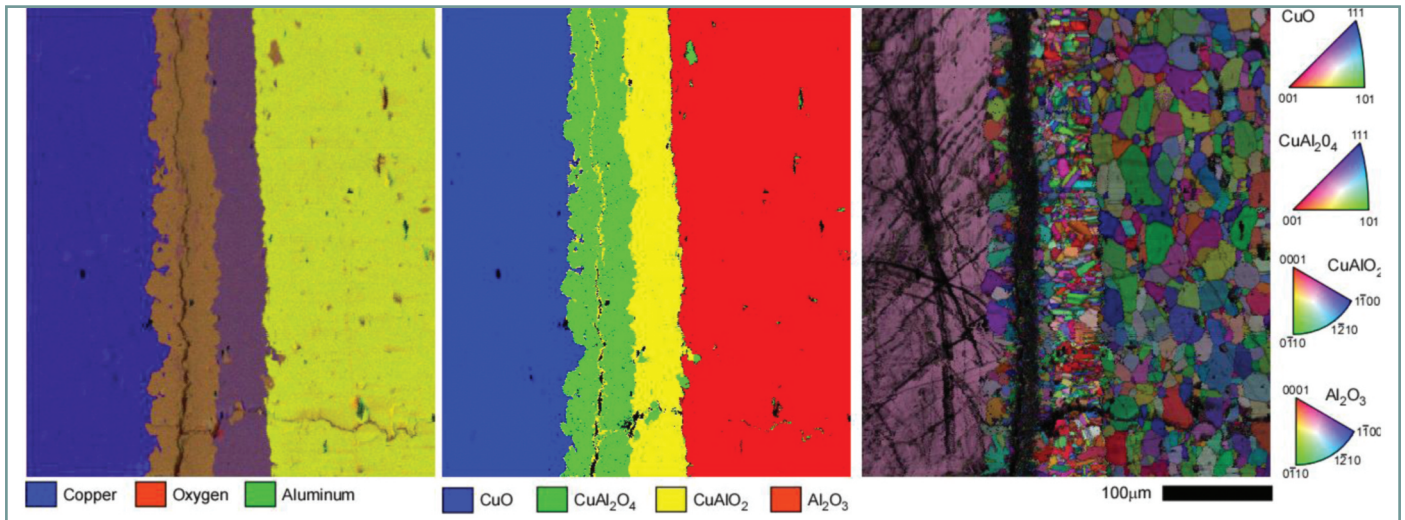


Figure 1. Blended elemental map (left), phase map (center) and orientation map (right) overlaid on a gray scale map based on the quality of individual EBSD patterns at each point in the scan. The maps are constructed from simultaneously collected EBSD and EDS data.

### Improved Material Models

Accurate modeling of material behavior is critical for meeting the material challenges of the nuclear industry. It is often difficult, if not impossible, to replicate the demanding operating conditions present in modern nuclear reactors in the laboratory setting. To predict how a material will behave requires accurate material models which in turn require fundamental understanding of the link between microstructure and properties. With the advent of OIM™ as a practical tool for characterizing the crystallographic orientation, it is now possible to refine the various materials models linking microstructure with properties to include orientation

information for more accurate simulation of microstructural evolution and more precise predictions of material properties under varying conditions. Several good examples have been reported in the literature. For example, Wu, Pan and Stubbins (2007) have shown how information derived from OIM™ data can be used to model the effects of irradiation-induced hardening on 316L stainless steel. They used OIM™ to characterize the crystallography, local misorientation (an indicator of plastic strain), twinning and slip system activity and in turn used this information to improve the finite element modeling (FEM) results. Some sample EBSD and FEM results from this study are shown in Figure 2.

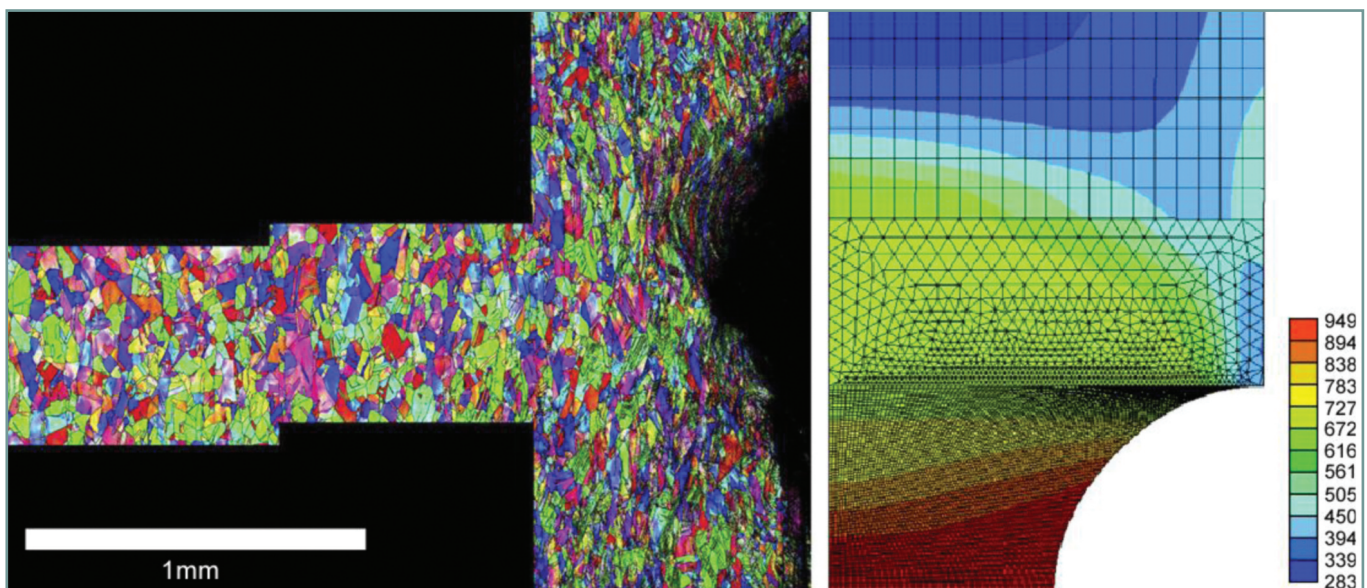


Figure 2. OIM™ orientation map (left) and FEM simulated stress contours (right) for a 316L stainless steel notch sample. (Adapted from Wu, Pan and Stubbins, 2007)

Another example of modeling is described in safety studies by Medevielle, Hugon and Dugne (1999) on the system U, Zr and O (termed the corium system). Corium is of interest because it includes the critical elements involved in the fusion of the tube (Zircaloy) with the nuclear fuel ( $UO_2$ ) during a nuclear incident. EBSD and Wavelength Dispersive Spectroscopy (WDS) were used together to unambiguously identify the complexity of phases present in samples obtained by fusion of the corium elements at varying oxygen concentrations. The crystallographic (via EBSD) and chemical (via WDS) characterization of the resulting oxides enables thermodynamic calculations to predict the corium behavior at high temperatures.

### Grain Boundary Engineering (GBE)

Grain boundary engineering is the use of thermomechanical processing to change the microstructure to favor certain types of grain boundaries over others in order to optimize a grain boundary sensitive material property. For instance, some types of grain boundaries may be resistant to corrosion while others may be more susceptible. Increasing the fraction of the corrosion resistant boundaries at the expense of the corrosion susceptible boundaries is a way of engineering the material to improve its performance. For example, Alexandreanu, Capell and Was (2001) have shown that GBE can improve the corrosion resistance of alloy 600 steam generator tube material. They have shown that improved performance can be achieved by promoting the presence of  $\Sigma$  coincident site lattice (CSL) boundaries. CSL boundaries occur where atoms are shared between the crystallite lattices adjacent to the grain boundary and are postulated to be less resistant to corrosion than random boundaries. These advantageous types of boundaries are introduced into the materials through various thermomechanical treatments. Increasing the CSL boundary fractions through these treatments leads to a reduction in the fraction of boundaries cracked as shown in Figure 3. Tan, Sridharan et al. (2006, 2008) have demonstrated that similar improvements in corrosion resistance can be achieved through GBE in Incoloy 800H and 617 (candidate alloys for Generation IV nuclear power systems).

OIM<sup>TM</sup> is a great tool for GBE research because of its ability to characterize the crystallographic structure of grain boundaries rapidly enough to “facilitate probabilistic analyses where inferences on future component performance and

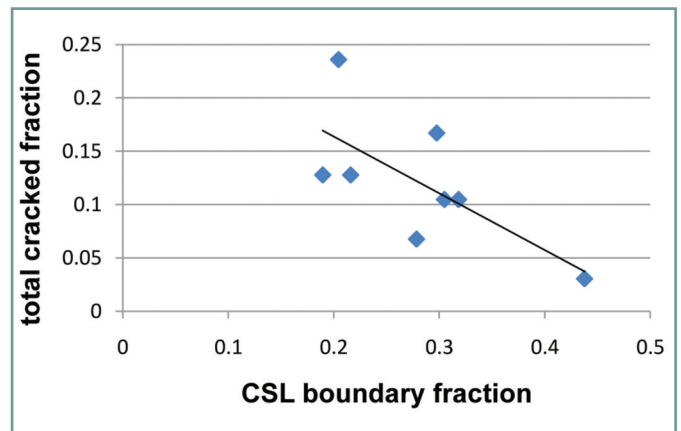


Figure 3. Correlation of total cracked fraction of boundaries at 15% strain with the fraction of CSL boundaries as measured by OIM<sup>TM</sup>. (Adapted from data in Alexandreanu, Capell and Was, 2001)

reliability are possible” (Tan, Sridharan and Allen, 2008).

### Texture

As OIM<sup>TM</sup> measures the crystal orientation at individual points in the microstructure it is an ideal tool for the study of texture and anisotropy. Texture is the statistical distribution of the lattice orientations of the constituent grains in a polycrystalline material. Most materials in their single crystal form exhibit some form of anisotropy (or directionality) of properties with respect to the orientation of crystal. For example, a hexagonal crystal may have a different value in yield strength in a direction parallel to the c-axis or basal pole direction of the crystal relative to a direction perpendicular to the c-axis. Zinc has a Young’s Modulus in the c-axis direction ( $28.7 \times 10^{12} \text{m}^2/\text{N}$ ) over three times larger than in a direction normal to c ( $8.4 \times 10^{12} \text{m}^2/\text{N}$ ) (Nye, 1985). Often it is assumed that in a polycrystal, such directional variations in the constituent crystals are averaged out over the bulk of the polycrystal. However, if the material exhibits texture then it will generally also exhibit anisotropy.

One nuclear application example of these ideas is the effect of texture on delayed hydride cracking (DHC) from deuterium uptake in Zr-Nb pressure tubes (Lehockey et al., 2007). This study found that higher deuterium (D) uptake occurs in microstructures with a broad distribution of basal poles (c-axes) in directions normal to the longitudinal directions of the pressure tube as shown in Figure 4. The authors of this study state that “the correlation between D uptake and texture measured by OIM<sup>TM</sup> may offer one means for optimizing processing/heat treatments during manufacturing to minimize

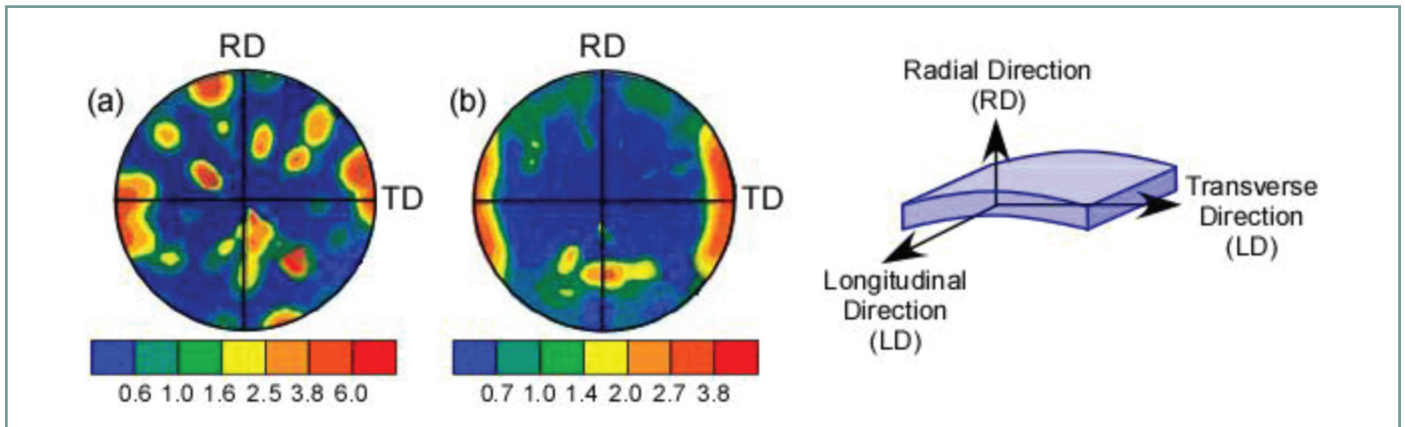


Figure 4. (0001) pole figures showing texture of Zr-Nb tubes with (a) low deuterium uptake and (b) high deuterium uptake. (Adapted from Lehockey, Brennenstuhl, Pagan, Clark and Perovic, 2007)

DHC susceptibility. Alternatively, it may allow a methodology for screening samples to identify installed pressure tubes at risk of DHC.”

Other examples of OIM™ based texture studies on zirconium alloys are as that by Une and Ishimoto (2006) on Zircaloy claddings with different thermomechanical processing, where the emphasis was on the relationship between texture and hydride precipitation and work by Holt and Zhao (2004) on the evolution of texture in extruded Zr-2.5Nb tubes.

## Fuel Cycle

The nuclear fuel cycle is key to the commercial growth of

nuclear power. A closed fuel cycle is preferable because of the costs associated with the nuclear waste stream. Research is ongoing to close the cycle through reprocessing and reuse of spent fuels in reactors. However, until that technology is achieved safe storage of spent fuel in repositories is a critical component of the fuel cycle. Various research studies are ongoing in this arena. Two examples are the research on the storage containers (Lehockey et al., 2007) and on the dissolution of the fuel itself after assumed contact with groundwater (Römer et al., 2003).

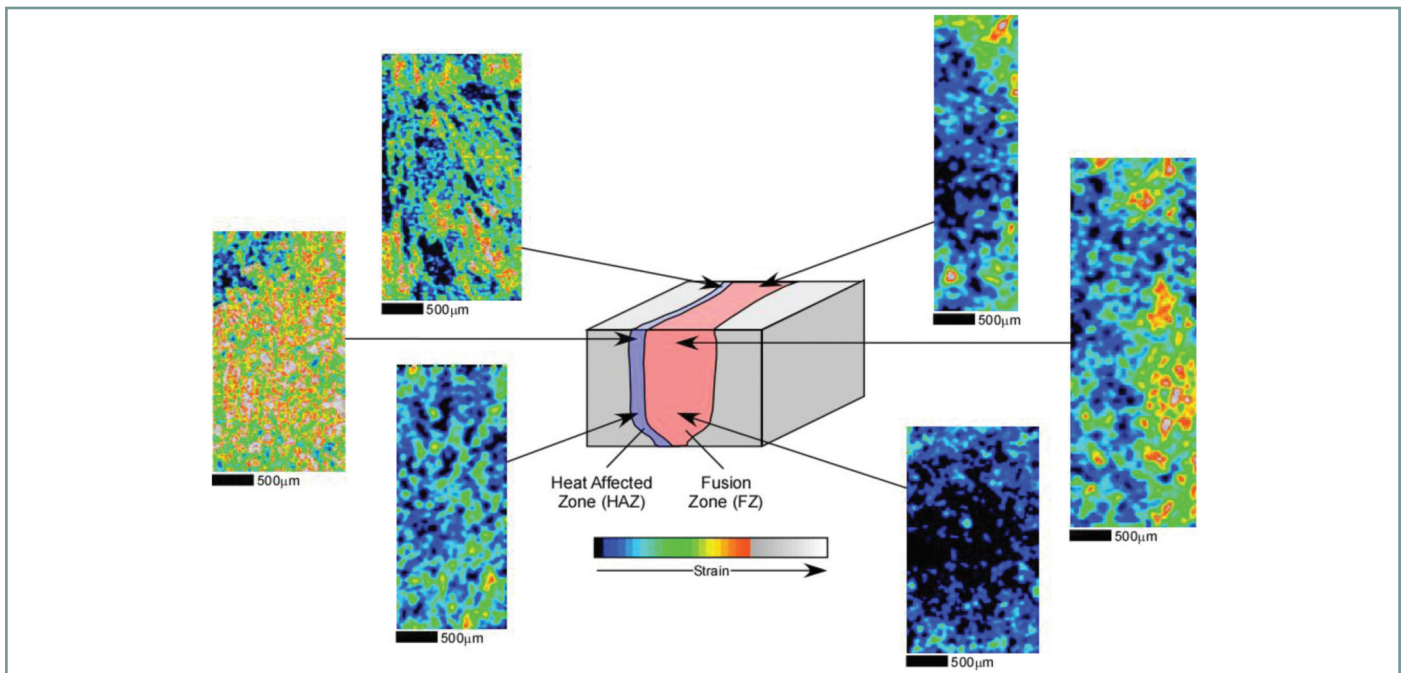


Figure 5. Variation in strain magnitude (as indicated by varying levels of local misorientation) at various locations in a copper waste container weld. (Adapted from Lehockey, Brennenstuhl, Pagan, Clark and Perovic, 2007).

### Waste Container

OIM™ has been used in research to improve the integrity of welds of copper waste containers under development for long-term storage of spent nuclear fuel. OIM™ was used to characterize the local distribution of plastic strain within the weld, as well as the affected zone of material around the weld.

OIM™ is capable of measuring variations in orientation with resolutions of tenths of a degree at the sub-micron scale. Such local variations in orientation give a relative indication of strain in the material. Regions with high local orientation variations are indicative of highly strained regions relative to un-strained regions where the orientation variations are small. Such information can be used, for example, to see the effect of local heating on the microstructure, such as that produced during welding. Lehockey et al., (2007) have applied OIM™ to study weld cracking in copper waste containers under development for long-term storage of nuclear fuel – some sample results are shown in Figure 5. The authors of this work noted that “OIM™ proved useful in recommending development of a more homogeneous heat treatment” to prevent weld cracking. Welding of dissimilar metals also poses challenges that can be addressed using OIM™ (Nelson, Lippold and Mills, 2000).

### UO<sub>2</sub> Dissolution

Römer, Plaschke, Beuchle and Kim (2003) found, using EBSD coupled with electrochemical atomic force microscopy (AFM), that dissolution rates of uranium oxide are strongly anisotropic. Figure 6 shows that the dissolution rate of the grain with a (001) face nearly parallel to the surface (in red) is faster than those

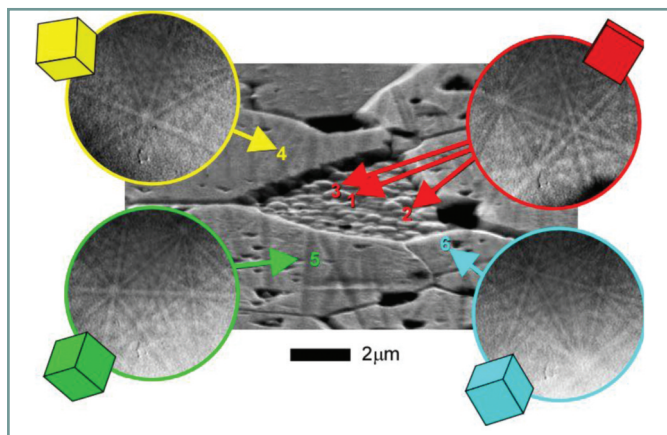


Figure 6. SEM micrograph of a corroded uranium-oxide showing grains of different dissolution levels and the corresponding EBSD patterns and orientation schematics. (Adapted from Römer, Plaschke, Beuchle and Kim, 2003).

with near (111) orientations. Such understanding is critical to ensuring accurate modeling of dissolution as needed for a valid safety assessment of a nuclear waste repository.

### Uranium Hydrides

Metallic uranium alloys are candidate materials for use as fuel. Uranium hydrides can be used as starting materials to create reactive uranium powder. Uranium hydride can be formed by heating uranium metal 250 to 300 °C in the presence of hydrogen. OIM™ has been employed to ascertain the influence of texture on hydride initiation and growth (Bingert, Hanrahan, Field and Dickerson, 2004). Some results from this work are shown in Figure 7.

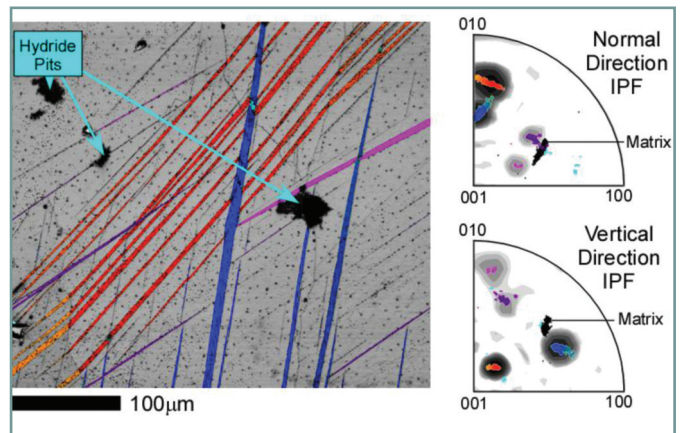


Figure 7. OIM™ orientation map showing deformation twins in hydrided  $\alpha$ -Uranium and bulk textures shown in gray intensities in inverse pole figure form overlaid with the location of key orientation components within the orientation map. (Adapted from Bingert, Hanrahan, Field and Dickerson, 2003).

### Long-Term Developments

All of the following longer term developments require continued research into structural materials for the reactor that can perform under the radiation, strain and thermal conditions that exists in these reactors. However, other materials issues specific to each of these reactor developments must be addressed as well. OIM™ will most assuredly play a part in discovering ways to overcome these issues.

### High Temperature, Multiuse Reactors

High temperature reactor technology needs a range of new materials: refractory alloys, composites and advanced coatings. “Materials behaviors such as microstructural stability; mechanical properties such as creep, fatigue, and toughness; and chemical properties, such as corrosion and compatibility need to be understood in the new domains of higher temperatures and irradiation levels.” (Raj et al., 2008)

Models linking properties with microstructure have been developed for many of these materials at less severe operating conditions. The validity of these models at the more severe service conditions needs to be verified, which requires both properties and microstructural data at data points within these more extreme temperature and irradiation regimes. Including OIM™ data provides a more complete description of the microstructure which can aid in improving these models.

### Fast Reactors

Long term objectives in this development area include alternate fuels such as carbides, nitrides or metal alloys. While the cladding and structural materials for systems with these fuels are similar to those already in service, advanced materials may be required for the reprocessing of the waste products of these fuels since the reprocessing produces highly aggressive environments. (Raj, Ramachandran and Vijalakshmi, 2009). Raj et al. point out the use of some of the technologies already discussed to meet the demands of fast-reactors. This includes the addition of minor elements to improve void swelling behavior, grain boundary engineering to improve intergranular corrosion resistance, and refinement of models for improved predictive capabilities.

One example of research in metal alloy fuels is on the uranium-molybdenum system. Using EBSD measurements, Medevielle, Hugon and Dugne (1999) were able to characterize the solidification structure and observed dendrites, which implied a liquid phase reaction during solidification as shown in Figure 8. Sometimes it is possible to recognize the microstructure prior to the phase transformation from the orientation maps of the post transformation microstructure (Cayron, 2009).

### Thorium Fuel Cycle

Thorium based fuels require the use of relatively high concentrations of fluoride in reprocessing. This motivates a need for extensive research in materials resistant to corrosion in fluoride rich environments. In concert with the development of advanced materials to meet this need, grain boundary engineering is a proven tool for improving corrosion resistance in some systems and may have a role to play in thorium based fuel development as well. With the ability of EDS to measure chemical composition and its spatial distribution in materials, it is an ideal tool for performing research into an advanced

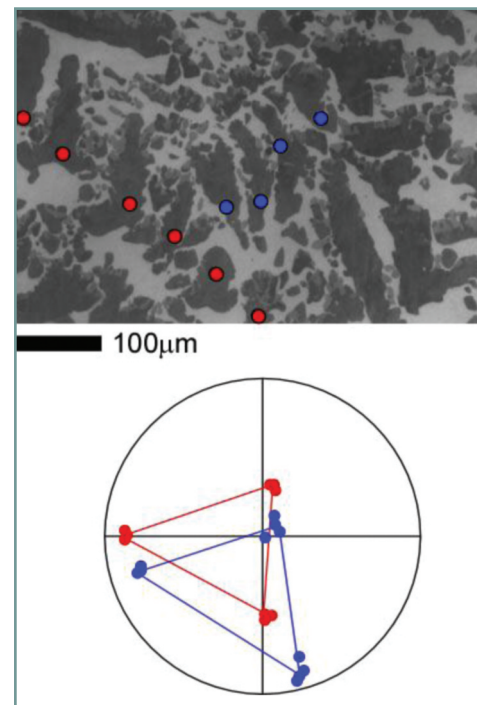


Figure 8. SEM micrograph of a molybdenum solidification structure (top). Corresponding (001) pole figure (bottom). (Adapted from Medevielle, Hugon and Dugne, 1999).

material's performance in a fluoride rich environment and the ability of EBSD to characterize grain boundaries makes it an important part of any grain boundary engineering work.

### Accelerator Driven Systems

The largest materials issue for these systems is the material for the window that separates the reactor from the accelerator. The window material needs to be resistant to irradiation, corrosion and both liquid metal and helium embrittlement, and also to possess good thermo-physical properties. OIM™ can provide information to improve performance through microstructure optimization.

### Fusion

As with all nuclear technologies, further development is needed on structural materials. However, the key area of materials research for fusion is on plasma facing materials. These materials are subjected to high neutron radiation, as well as strong mechanical, thermal and electromagnetic loading. Advanced ferritic steels, vanadium alloys, silicon-carbide composite ceramics and tungsten-based refractory alloys are being developed to meet these rigorous requirements. Materials research has also focused on coating technology to prevent hydrogen embrittlement, which is a particular problem with

the steels and vanadium alloys. Historically, materials behavior has been shown to be very sensitive to exposure conditions. Thus, extrapolation of a material's response in one system to another system is generally inaccurate. This obviates the need for full characterization of material microstructure for more accurate predictive capabilities. OIM™ and EDS play an important role in gaining the insight needed into the effects the

chemical and crystallographic aspects of a material have on material behavior. For example, OIM™ has been used to study the fatigue and creep-fatigue behavior on a P91 martensitic steel (Fournier, Sauzay, Renault, Barcelo, Pineau, 2009). The 9-12% Cr martensitic steels have been selected as candidate materials for structural components in future fusion reactors.

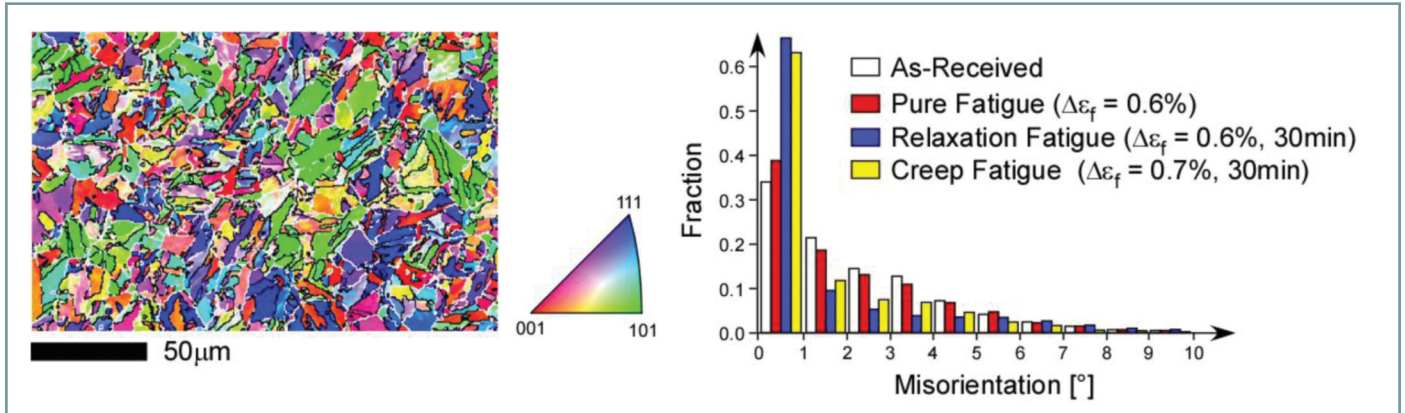


Figure 9. Orientation map (left) of the as-received microstructure and the average mean misorientation per block of laths after various cyclic loadings at 823K (right), where  $\Delta\varepsilon_f$  is the prescribed fatigue strain range. (Adapted from Fournier, Sauzay, Alexandra, Barcelo and Pineau, 2009).

## Conclusions

“Nuclear materials researchers and technologists have gained rich experience studying the behavior of present-generation materials, such as zirconium-based alloys and special steels, in the past three decades. The present trends suggest that these materials will undergo incremental changes in the immediate future, to increase the burnup of the fuel and the lifetime of the reactors. However, sustained R&D on a wide spectrum of materials such as refractory alloys, composites, ceramics, low-activation steels, and coatings and the related processing technologies is essential to meet the demands of emerging nuclear technologies” (Raj et al., 2008). OIM™ can play a key role in these R&D needs – four general areas are:

- Wherever grain boundaries play a role, OIM™ should be considered as an important component of the characterization toolbox. Embrittlement and corrosion damage often happens along grain boundaries. Grain boundary engineering can play a key role in improving the performance of a material in the severe environments associated particularly with in-core applications. OIM™ is a proven tool for characterizing grain boundary distributions in polycrystalline materials and has helped make grain boundary engineering a real possibility for tailoring the microstructure of a material to improve its performance particularly in mitigating corrosion, cracking or intergranular attack at surfaces exposed to aggressive environments inherent to nuclear applications.
- Almost all forming processes impart texture to some degree into materials. Understanding the texture evolution during well-controlled and well-characterized processing is a key input into accurate materials model development. Accurate simulation of material response is key to optimizing a forming process so as to produce a part that with a microstructure specifically tailored to satisfy the in-service demands of the part. The ability of OIM™ to provide statistically relevant and spatially specific orientation data makes it an ideal tool for providing the researcher with key orientation data needed by advanced finite element based modeling.



- In addition to texture, components formed using deformation processes such as forging, rolling or extrusion also contain a degree of residual strain. Sometimes, this is relieved through heat treatment. However, at other times the material is left in the strained state. In addition, the material in a component may also be strained in the operating state. Often the strain is not homogeneously distributed throughout the component but varies locally at the micron scale. OIM™ provides a means of characterizing these local variations in strain through its ability to measure local small variations in orientation. Such information can be invaluable for optimizing thermomechanical forming paths, as well as understanding induced stress-states during service.
- With integrated systems OIM™ and EDS can be used together to provide insight into the interplay of crystallographic structure and chemical composition as new materials or systems of materials are developed. Such a capability can be useful in the characterization of the spatial distribution of different phases within the microstructure of multiphase systems or in the identification of phases. Information on second phase inclusions or bulk phase transformations can provide critical insight into the local conditions these materials face during service in terms of temperatures, strains or irradiation. Such information is critical to accurately model the behavior of these materials in the extreme service environments in which they are placed.

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