Comprehensive Microstructure Characterization of Nuclear Material Databases; Insights from Combined Synchrotron and Machine Learning Experiments

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Background & Motivation

Next-generation fission and fusion nuclear reactors expose various material components to extreme environments, including mixed neutron/gamma radiation fields, high stresses, corrosive media and high-temperatures [1]. To ensure that large safety margins are maintained over the life cycle of these reactors, large sample databases are typically fabricated and subsequently irradiated within test reactors for accelerated material testing programs. These material databases, incorporating both metallurgical (microstructure, composition, product form and heat treatment) and irradiation (neutron flux, fluence, irradiation temperature) variables, can be appreciably large and contain hundreds to thousands of unique specimens. These databases are instrumental in developing statistically relevant data sets so that structural integrity and material performance and predictable operation envelopes can be maintained during reactor operation.

These large material databases require significant investment over multiple year periods. Additional characterization techniques that can yield non-destructive insight into understanding processing-structure-property relationships are thus of high value. Here, we have employed a combination of advanced, non-destructive synchrotron based characterization techniques including X-ray Diffraction (XRD) and Small Angle X-ray Scattering (SAXS) to characterize one such material database. Rapid “model-free” analysis of these large data sets was achieved by performing principal component analysis (PCA) and multidimensional scaling (MDS) in addition to conventional analysis methods to extract quantitative microstructural information. By applying advanced machine learning analysis techniques to these data sets, subtle and not so subtle correlations between alloy content and irradiation conditions were uncovered.

Experimental Methods, Preprocessing and Analysis

High-Throughput Synchrotron Experiments were performed at the X-ray Powder Diffraction beamline at the National Synchrotron Light Source II. High-energy (52 keV) XRD and SAXS patterns were collected for over 100 different combinations of alloy chemistry and neutron irradiation fluence (a total of 200 specimens including unirradiated baselines were measured). Batch software for all preprocessing and analysis steps were developed to reduce the 2D detector images to 1D patterns and organization of metadata (such as alloy chemistry and irradiation variables) that are needed in the analysis [2]. The process from collection to analysis is illustrated below.

Step 1: XRD and SAXS Data Sets collected at XPD

Step 2: Preprocessing datasets

Mask and calibrate

Conversion of the 2D XRD patterns to 1D plots of intensity for analysis

Software: XPDacq, Igor Pro (WaveMetrics)

Step 3: PCA Analysis and Visualization

PCA

Conventional XRD analysis

MDS

Software: Python scripts – Google Collab

Results from Machine Learning and Coupled Characterization

- Rapid analysis of any region in XRD (or SAXS) patterns
- Uncover effects of irradiation on the different crystal phases in the base steels
- Generate contour maps of principal components and visualize with alloy chemistry
- Uncover new nanoprecipitate phase formation (as verified by SAXS)

Summary and Microstructural Insights

Our approach highlights the usefulness of machine learning techniques (such as PCA) to rapidly analyze large data sets collected at synchrotron light sources and to uncover subtle microstructural changes that ensue with irradiation. Our “model-free” approach is particularly useful, as it provides qualitative trends in the response of the different crystallographic phases as functions of alloy chemistry. This approach also guides the user to perform conventional analysis over a smaller region of phase-space to generate quantitative microstructure changes. We highlight that our results have uncovered a significant alloy-dependent response to irradiation, with low Ni and Mn contents being most effected by irradiation. From the combined XRD/PCA and conventional SAXS analysis, we uncovered a composition-dependent trend in the volume fraction of radiation-induced secondary phase precipitates. The chemistry of these intermetallic precipitates is strongly dependent on the initial alloy chemistry. This new capability will provide insights into the design and performance of next-generation reactors.

References


D. Sprouster thanks G.R. Odette and P. Wells from UCSB for providing samples. This project was supported in part by the U.S. Department of Energy, Office of Science, Office of Workforce Development for Teachers and Scientists (WDTS) under the Science Undergraduate Laboratory Internships Program (SULI). Use of the National Synchrotron Light Source II, Brookhaven National Laboratory, was supported by the DOE under Contract No. DE-SC0012704.