



# Analysis and comparison of focused ion beam milling and vibratory polishing sample surface preparation methods for porosity study of U-Mo plate fuel for research and test reactors

Bjorn Westman<sup>a,\*</sup>, Brandon Miller<sup>b</sup>, Jan-Fong Jue<sup>b</sup>, Assel Aitkaliyeva<sup>c</sup>, Dennis Keiser Jr.<sup>b</sup>, James Madden<sup>b</sup>, Julie D. Tucker<sup>a</sup>

<sup>a</sup> Oregon State University, School of Mechanical, Industrial and Manufacturing Engineering, 204 Rogers Hall, Corvallis, OR 97331, United States

<sup>b</sup> Idaho National Laboratory, 1955 N. Fremont Ave., Idaho Falls, ID 83415, United States

<sup>c</sup> University of Florida, Department of Materials Science and Engineering, 156 Rhines Hall, Gainesville, FL 32611, United States

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## ABSTRACT

Uranium-Molybdenum (U-Mo) low enriched uranium (LEU) fuels are a promising candidate for the replacement of high enriched uranium (HEU) fuels currently in use in a high power research and test reactors around the world. Contemporary U-Mo fuel sample preparation uses focused ion beam (FIB) methods for analysis of fission gas porosity. However, FIB possess several drawbacks, including reduced area of analysis, curtaining effects, and increased FIB operation time and cost. Vibratory polishing is a well understood method for preparing large sample surfaces with very high surface quality. In this research, fission gas porosity image analysis results are compared between samples prepared using vibratory polishing and FIB milling to assess the effectiveness of vibratory polishing for irradiated fuel sample preparation. Scanning electron microscopy (SEM) imaging was performed on sections of irradiated U-Mo fuel plates and the micrographs were analyzed using a fission gas pore identification and measurement script written in MatLab. Results showed that the vibratory polishing method is preferentially removing material around the edges of the pores, causing the pores to become larger and more rounded, leading to overestimation of the fission gas porosity size. Whereas, FIB preparation tends to underestimate due to poor micrograph quality and surface damage leading to inaccurate segmentations. Despite the aforementioned drawbacks, vibratory polishing remains a valid method for porosity analysis sample preparation, however, improvements should be made to reduce the preferential removal of material surrounding pores in order to minimize the error in the porosity measurements.

## 1. Introduction

As part of the Reduced Enrichment for Research and Test Reactors (RERTR) program and the Global Threat Reduction Initiative, efforts are being made to convert high power research and test reactors from high enriched uranium (HEU) fuel forms to low enriched uranium (LEU) fuel forms in order to reduce the overall amount of weapons grade material in circulation around the world. The goal of this conversion is to reduce the risk of nuclear and radiological terrorism around the world by reducing the potential for diversion of dangerous nuclear and radiological material. With that goal in mind, the RERTR program is researching new fuels that implement higher uranium density and lower uranium enrichment to reduce those concerns (Wachs, 2007).

Uranium-molybdenum (U-Mo), dispersion and monolithic, plate

fuels have been identified as a promising candidate for a LEU replacement of HEU fuel currently in use around the world (Wachs, 2007). Both monolithic and dispersion fuel plate designs have been investigated and contain between 7 and 10 wt.% Mo with an aluminum (Al)-based cladding, and an Al-based matrix material for dispersion fuel plates. Characterization of U-Mo plate fuels have been performed to better understand its irradiation behavior. Various features have been characterized including phase formation, grain size distribution, and chemical composition (Leenaers et al., 2015; Leenaers et al., 2013; Miller et al., 2015; Keiser et al., 2009; Keiser et al., 2012; Keiser et al., 2014; Gan et al., 2014; Gan et al., 2010; Meyer et al., 2014; Van den Bergh et al., 2008; Kim and Hofman, 2011). Porosity development and its driving forces have been an area of focused study (Collette et al., 2016a; Casella et al., 2017; Kim et al., 2016; Miller et al., 2015; Collette et al., 2016b). Some of the primary causes of failure observed in U-Mo

\* Corresponding author.

E-mail address: [westmanbe@comcast.net](mailto:westmanbe@comcast.net) (B. Westman).

fuel plates have been delamination and pillowing, due to build-up and coalescence of fission gas pores, leading to breaches in the fuel cladding and fission product release (Wachs, 2007). Studies have shown that as fission density increases in fuel, so does porosity. Specifically, at around  $4.5 \times 10^{21}$  fissions/cm<sup>3</sup>, depending on the materials, the fuel starts to undergo a recrystallization process (Aitkaliyeva et al., 2015). Following that recrystallization, porosity grows rapidly, aided by increased grain boundary area providing more sites for fission gas pore nucleation. The result is a microstructure referred to as High Burn-up Structure (HBS) characterized by relatively small grains and increased average pore size and pore density (Aitkaliyeva et al., 2015; Collette et al., 2016a). Therefore, the modeling of formation and growth of fission gas porosity, as well as the characterization of post irradiation porosity, has become a primary concern for the qualification of U-Mo research reactor fuel (Wachs, 2007). To support the characterization of U-Mo plate fuel porosity, a large volume of consistent, high quality, micrographs of irradiated U-Mo plate fuel microstructures must be acquired, and then analyzed, to extract data regarding the pore size distribution from a number of samples of varying plate type, fission density, fission rate, U<sup>235</sup> burn-up, average irradiation temperature, and/or manufacturing parameters.

Research is being conducted to characterize U-Mo plate fuel porosity development and growth employing the generic metallic sample preparation methods along with FIB sectioning and surface preparation (Miller et al., 2012; Aitkaliyeva et al., 2015; Casella et al., 2017). FIB sample preparation techniques possess several drawbacks. These drawbacks include reduced area of analysis and significant beam damage to the sample surface, which makes porosity analysis more difficult, by increasing the number of micrographs required to collect a statistically significant sample and introducing features like FIB curtaining that can be mis-segmented as pores (Aitkaliyeva et al., 2015). Vibratory polishing is a well-known method that has been used in the past to produce sample surfaces with very little surface damage in metallic samples for applications like electron back scatter diffraction analysis (Hofer et al., 2015). Vibratory polishing is a unique polishing procedure in that it combines both mechanical and chemical polishing. While the particulate silica acts as a physical abrasive, the fluid in which it is suspended in is also an etchant. This means that those areas that would be vulnerable to corrosive may also be reduced or removed during the vibratory polishing process. In comparison to FIB surface preparation, vibratory polishing can produce a larger area of prepared surfaces for analysis, which is important for microstructure characterization as it provides larger, more statistically significant, data sets. Some speculation exists that the vibratory polishing process can affect porosity size distribution due to preferential erosion of the material immediately surrounding pores, thus testing is required to determine whether vibratory polishing is a viable surface preparation method for

U-Mo porosity characterization.

In the past, image processing and analysis of nuclear fuel has relied heavily on visual inspection, manual measuring, and segmentation to extract data (Gonzalez and Woods, 2008; Hofer et al., 2015; Gan et al., 2014). When collection relies on manual measurement and visual inspection, data sets tend to be small and less statistically significant. Automating the image analysis process, using computer software programs, has been shown to possess the ability to extract more data, faster than manual analysis, while maintaining better consistency and at least similar accuracy, relative to standard manual counting and measuring procedures (Casella et al., 2017; Collette et al., 2016a; Collette et al., 2016b). In this paper we use a MatLab image analysis program, benchmarked to manual measurements, to analyze fuel samples.

To determine the effectiveness of vibratory polishing as a candidate for the replacement of FIB surface preparation, vibratory polishing has been used to prepare samples that have also been imaged following traditional sample preparation methods in order to evaluate the vibratory polishing results. This paper will outline the methods used to conduct the evaluation of vibratory polishing as a candidate for replacement of FIB sample preparation methods and will report the results of that comparison and discuss what they indicate for the future of LEU U-Mo plate fuel porosity studies. Any trends between irradiation/manufacturing parameters and porosity statistics will also be discussed.

## 2. Methods

### 2.1. Sample irradiation

Samples for this study were taken from three different fuel plates that were irradiated in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL). Samples V5R050-L and V5R050-H were taken from plate V5R050, part of the RERTR-7A experiment, samples 1B5-L and 1B5-H were taken from Plate 1-B5, part of the ATR Full Sized Plate In Center Flux Trap Position (AFIP)-1 experiment, and sample 7ZH-4 was taken from Plate 7 ZH-4, part of the AFIP-7 experiment. (Perez et al., 2011a; Perez et al., 2012; Perez et al., 2011b) Due to the high radioactivity of the fuel plates,  $1.5 \times 1 \times 1$  mm samples were sectioned from the fuel plates in a hot cell using a diamond blade low speed saw. For the V5R050 and AFIP-1B5 plate, two samples were obtained from differing locations in their respective plate to investigate the effects of irradiation parameters independent of fabrication process. Using the plate as-run irradiation parameters, local irradiation parameters were determined for the samples and can be seen Table 1. In the naming of the samples, *L* stands for low fission density, *H* stands for high fission density, *VIB* stands for vibratory prepared, and *FIB* stands for FIB prepared. All the samples in this study will follow this naming convention. In the case of 7ZH-4, where only one sample was taken

**Table 1**  
Local Irradiation Parameters and Fabrication Details for the 5 Samples.

Sample	V5R050-L	V5R050-H	1B5-L	1B5-H	7ZH-4
Fuel Type	Disp.	Disp.	Disp.	Disp.	Mono.
Fuel Material	U-10Mo	U-10Mo	U-7Mo	U-7Mo	U-10Mo
Enrichment (% <sup>235</sup> U)	58	58	19.9	19.9	19.8
Diffusion Barrier	N/A	N/A	N/A	N/A	Zr
Cladding Material	Al-0.5Si	Al-0.5Si	Al-4043	Al-4043	Al-6061
EOL Surface Temperature (°C)	75.7	102	75.9	89.3	
EOL Centerline Temperature (°C)	84.9	139	84.1	121	
EOC Heat Flux (W/cm <sup>2</sup> )	115	284	64.5	135	170
Average Fission Rate (fissions/cm <sup>3</sup> /s) x10 <sup>14</sup>	1.72	3.00	1.30	2.62	
Max. Fission Rate (fissions/cm <sup>3</sup> /s) x10 <sup>14</sup>	1.85	3.22	1.92	3.89	1.16
Fission Density Meat (fissions/cm <sup>3</sup> ) x10 <sup>21</sup>	1.32	2.31	1.76	3.57	
Fission Density Fuel Particle (fissions/cm <sup>3</sup> ) x10 <sup>21</sup>	2.95	4.90	4.05	6.13	2.7
EOC Local <sup>235</sup> U Burn-Up (Ratio)	0.150	0.252	0.380	0.700	
Total Plate Swelling (%)	3.90	6.40	4.20	8.90	
Fuel Swelling (Ratio)	0.160	0.268	0.150	0.290	

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