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Automated Calculation of U-10Mo Fuel and Zr Cladding Thickness

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Prepared for the U.S. Department of Energy
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Summary

Hand calculation and verification of cladding and fuel thickness in a hot-isostatically pressed uranium-molybdenum (U-10Mo) alloy can incur human errors and longer image processing times when analyzing a large set of cross-sectional images. Also, it is difficult to take truly randomized thickness measurements by hand, which introduces a bias in the overall statistical summary. To help alleviate time, cost and errors made using hand measurements, an automated image processing procedure was developed. To do so we utilized Octave, an open-source MATLAB alternative, to repeatedly determine the thickness of Zr and U-10Mo layers. U-10Mo samples with Zr interlayer were imaged using standard secondary electron and backscattered-electron imaging at 250× magnification to capture the various layers present in U-10Mo. Further image processing used pixel-by-pixel calculation of the Zr and U-10Mo layers to gather quantitative statistics on the thickness variations associated with each of the layers.

Acknowledgments

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Acronyms and Abbreviations

DOE	Department of Energy
GIMP	GNU Image Manipulation Program
PNNL	Pacific Northwest National Laboratory
SEM	scanning electron microscopy
U10-Mo	uranium 10wt%-molybdenum
MP2	Mini-plate 2

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1.0 Introduction

Within the Department of Energy's Material Management and Minimization program, the U.S. High Performance Research Reactor Project has been tasked with the development, qualification, and licensing of new fuel systems to convert the five research reactors in the U.S. from highly enriched uranium to low-enriched uranium fuel. Uranium alloyed with 10-weight percent (wt%) molybdenum (U-10Mo) monolithic low-enriched uranium fuel has been selected for this conversion. The fabrication process of the fuel itself is currently being optimized and refined to meet the standards and certification specifications defined for the research reactors[1–13].

The Mini-Plate-2 (MP-2) test is the second in a series of fuel testing campaigns with the purpose of achieving regulatory qualification for the U-10Mo monolithic plate-type fuel system. The objective of MP-2 is to assess performance behavior of fuel plates fabricated by a commercial fuel fabricator (BWX Technologies, Inc.) and make sure that the fuel maintains mechanical integrity and geometric stability and behaves in a stable and predictable manner. Critical to the manufacturing and quality assurance of the fuel plates are the thicknesses of the Zr cladding and U-10Mo fuel meat. During thermomechanical processing of the plates, owing to the rolling reductions, hot rolling can material and thickness and microstructure variation, introduce nonuniform thickness of the cladding and base materials [1, 8–10, 14, 15]. To meet the specifications it is desired that the thickness variations must be within 0.0254 ± 0.0127 mm (1mil ± 0.5 mil) for Zr cladding.

Automated measurements of U-10Mo fuel plate cross sections have the benefit of mitigating the measurement error incurred in typical hand-measurement. Micrographs generated through optical or electron microscopy can capture the full thickness of the plates and can directly be imported through any image processing software to threshold and distinguish the respective zirconium and U-10Mo layers.

The open-source software Octave was used, primarily because its free and open-source nature allows replication of the analysis by those who are familiar with MATLAB formats [16, 17]. The matrix-like nature of Octave/MATLAB allows intuitive pixel-by-pixel calculation along with common image processing techniques that are documented very well in literature.

Proper measurement of the zirconium and U-10Mo fuel thicknesses standardizes the thickness characterization, allowing for insights into the fuel processing pathways and quality control. The code and processes presented can be modified and rewritten, if necessary, for increased user control, speed improvements, or both.

2.0 Sample Prep and Imaging

Specimens of U-10Mo alloy were sectioned such that the transverse (edge-on) view of the plates could be mounted in 1.25” epoxy mounting material. Once cured, the specimens went through a standard metallographic preparation procedure beginning with course 220 grit sand paper, progressively reducing the polishing media size until ending in a colloidal silica vibratory polish. Ensuring a proper clean of the surface is important as any residual silica on the sample surfaces can affect the image quality in the SEM. Typically – a quick rinse with de-ionized water and ethanol will suffice for this removal of residue. A thorough description of the steps for adequately polishing U-10Mo alloys have been documented and reported elsewhere [18].

Once polished, the specimens were then coated in a ~7nm carbon layer to eliminate the charging on the sample during electron microscopy. This step should be done quickly after the cleaning step, as the surface of uranium alloys can oxidize quickly in open air.

Before processing of images, while the sample is undergoing microscopy, users should be cognizant of any electron charging on the sample while imaging. The charging is seen in an SEM as overexposed and white regions, which will alter the pixel calculation. To mitigate charging, proper sample preparation, such as grinding and polishing, should aim to achieve little or no surface relief between specimen and mounting material, and any conductive coating applied to the surface must be thick enough to prevent charging. This is particularly important for secondary electron imaging as charging can greatly affect the overall contrast of the sample features. Backscatter electron images show contrast based on the atomic number with higher Z resulting in more backscatter signal. This imaging mode is particularly important for highlighting the Zr layer for thresholding.

Secondary electron and backscatter image montages were taken of each U-10Mo specimen prepped for the MP-2 experiment. For the large area montages used in this report, only magnifications of 250x were utilized. Stitching of each subsequent image in the mapping were done utilizing Oxford Instruments Aztec© software.

3.0 Image Preprocessing

Images from standard scanning electron microscopy (SEM) software must be modified to fit the analysis code utilized later in this report. A typical .tif image output from the SEM is an $n \times m \times 4$ dataset (horizontal × vertical pixel coordinates × CMYK color code) that needs to be converted through a 16-bit RBG color to gray function and then through a 8-bit black-and-white conversion via thresholding. These series of steps can be done with many free and open-source image processing programs such as ImageJ, Octave, python, or GIMP. Some offer an automated conversion process while others offer more sophisticated processing algorithms. It is up to the user to define which software fits their needs for the image preprocessing. A typical way to perform this in ImageJ is described in Figure 1.

The subsequent steps are fairly homogeneous across computing platforms and involve thresholding and pixel-by-pixel correction algorithms that do not require highly advanced understanding; thus, this procedure can be used for the U-10Mo systems and modified to fit the user’s unique laboratory capabilities.

The first and last 10–15% of the data points were discarded in some cases because the sample had been sheared for mounting considerations and this shearing introduces a significant amount of deformation that artificially increases or decreases the thickness.

The general algorithm pathway is as shown in Figure 1. Starting from “Code Start” to “Noise Remove/Hole Filling” can be done via imageJ or through the Octave code listed in Appendix A. ImageJ was used in this work primarily as a confirmation of the pixel/length ratio of the SEM images which for the electron microscope used in this report at 250x magnification resulted in a ~4.00pixel/micron ratio. The automation of these steps across the multiple samples in MP-2 allows for a rapid determination of thickness variations within the fuels, negating steps and error induced via hand calculations.

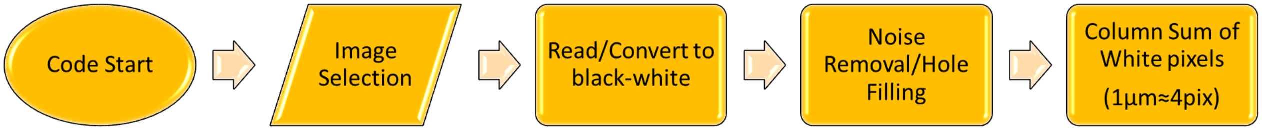
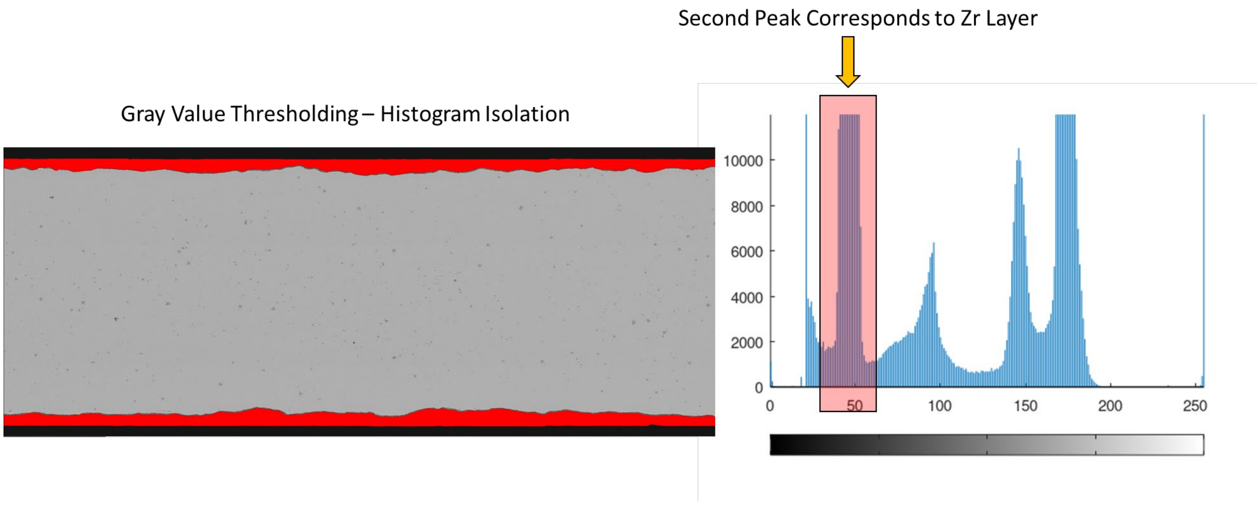


Figure 1. Algorithm flowchart for U-10Mo and Zr thickness calculation

Using Octave as the main language, algorithms were written to incorporate all of the steps in Figure 1 to calculate the thickness of Zr and fuel meat in each specimen for the MP-2 experiment. Details regarding the use of the code is described below.

4.0 Zr Thickness Calculation

Using Octave, the backscatter electron image is selected and loaded into the software. A distribution gray value pixels is then shown exhibiting multiple sharp peaks, each associated with a particular region/piece of the fuel. The Zr layer is then isolated by locating the correct peak in the gray value histogram, Figure 2 shows the correct peak associated with the Zr layer in a grayscale SEM micrograph. The first peak corresponds to the mount material or black areas, while the succeeding Zr layer tends to be separate from the U-10Mo fuel meat in gray values. This second peak will then need to be isolated by the user and is a subjective range: typically, the minima on each tail of the peak are chosen as threshold ends.


Gray Value Thresholding – Histogram Isolation

Second Peak Corresponds to Zr Layer

Figure 2. Highlighting of Zr layer in U-10Mo fuel cross section (left) and histogram of gray values highlighting the peak that represents Zr-layer pixels (right). Isolation of this peak will leave only the Zr visible after thresholding.

After the correct thresholding values are chosen, each Zr layer is then isolated, and the thickness variations are calculated individually for the top and bottom Zr layers (Figure 3). The top and bottom layers are separated in the code to avoid double counting of the Zr-classified pixels. Separating the two Zr layers allows the user to perform further analysis on the top and bottom layers, as necessary, to determine specification conformance or to show regions that may be out of specification. The code to determine the same and implementation is provided in Appendix A and B.

Figure 3. Walk-through visualization of the thresholding process associated with isolating the Zr layer

Calculating the thickness is done by summing the number of white pixels in each column along the *x* direction of the image (Figure 4).

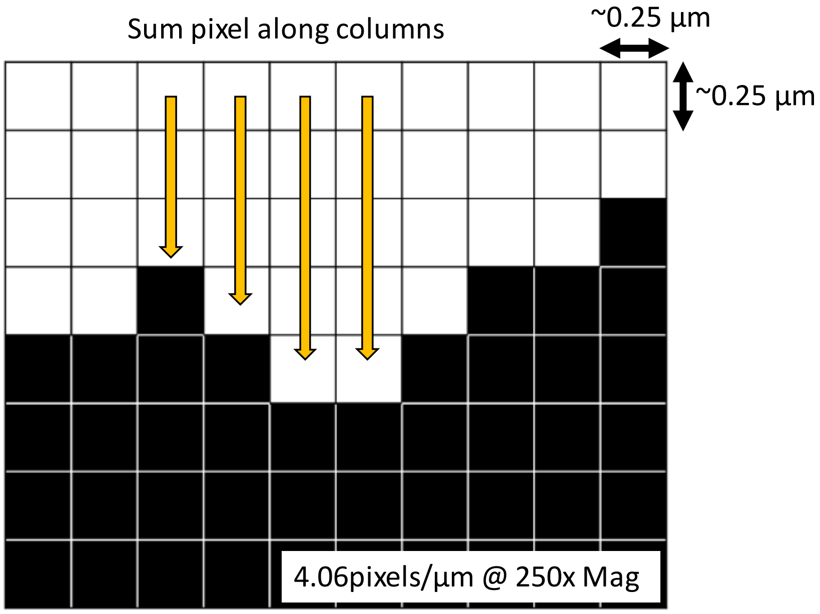


Figure 4. Calculation of U-10Mo and Zr thickness, pixel by pixel. At 250× magnification, each pixel is approximately 0.25 µm on a side, resulting in ~4 pix/µm. Adding the white pixels along columns will gather a column-by-column statistic of layer thickness and variation.

For the SEM 250x magnification montages used in the MP2 test campaign, the scale equates to ~4.06 pixels per micron. A scaling step may needed to determine the correct pixel to micron value depending on the microscope settings - which can be done via any image processing software such as ImageJ – so long as there is a scale bar present in the micrograph. Thus, the number of white pixels is correlated with the local Zr thickness in a given area and the variation can be determined. Using the white pixel values along the full length of the cross section, a full statistical description of the Zr layer thickness can be determined. Previously, this was done by hand via image manipulation software and was subject to human errors and under sampling issues. Figure 5 highlights the large differences in the results of the hand calculation versus the computational methods presented here. When hand calculations were limited to 5–10 measurements the thickness ranges, on average, were higher than the true variation. It is difficult to take truly randomized thickness measurements by hand, which introduces a bias in the overall statistical summary. As the number of hand measurements increases, the thickness values approach those from the computational method.

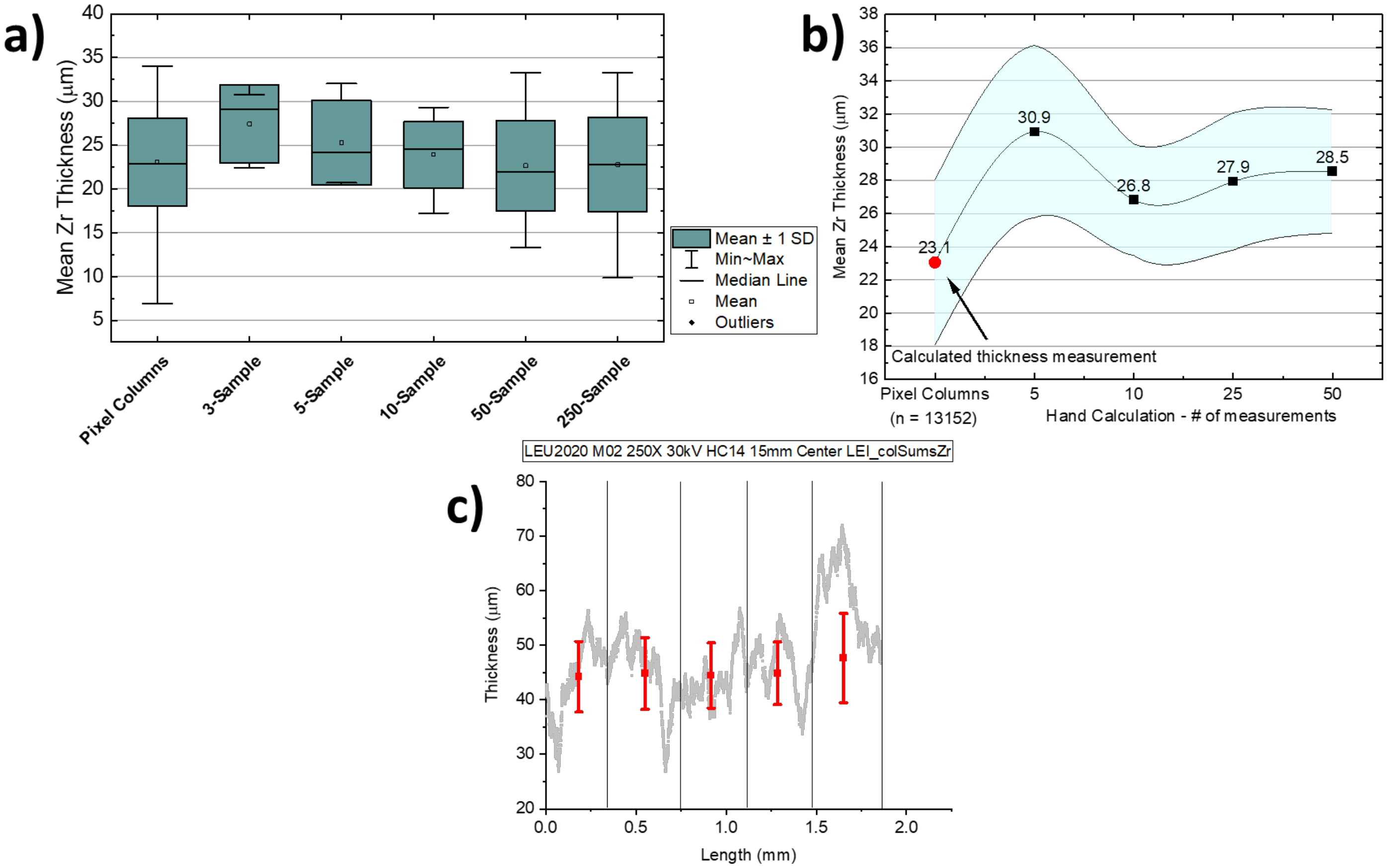


Figure 5. Plots of Zr thickness variation calculated via pixel column calculation with various sub-sampling (a) vs. by hand using ImageJ (b. Under sampling results in a higher estimated mean thickness and approaches true variation values with increase in hand calculations. Blue area on right image indicate +/- one standard deviation. (c)

Thickness variations as a function of total length of material used. Each subsequent mean/standard deviation datapoint incorporates the previous subsets.

5.0 U-10Mo Fuel Meat Calculation

Calculation of the U-10Mo fuel meat thickness is similar to the Zr calculation. The fuel meat has a higher secondary phase inclusion content present in the micrographs compared to the Zr layer, which introduces a significant amount of noise (Figure 6b) that must be removed before an adequate thickness estimate can be made. The gray values associated with the fuel meat are distinct from those for the Zr and unique when imaged under a backscattered-electron condition and can be separated out as such.

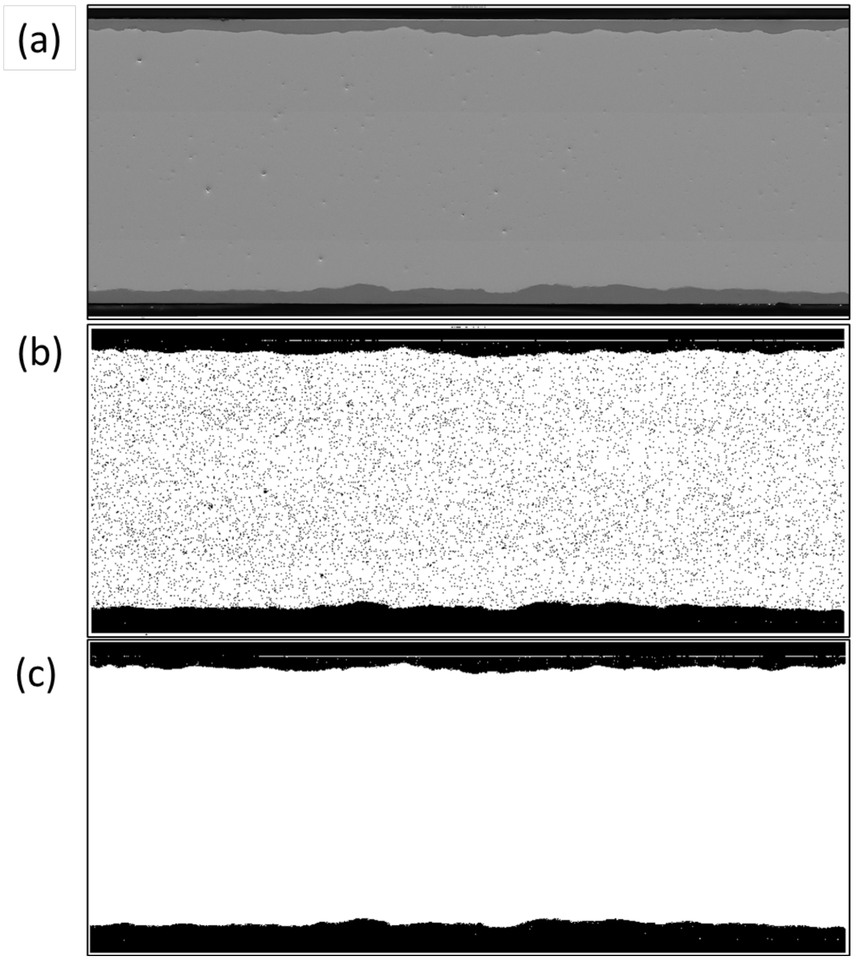


Figure 6. Step-by-step process of isolating U-10Mo fuel meat: (a) raw backscattered-electron image; (b) conversion of gray values to black and white and (c) removal of noise from grains and inclusions present within the fuel meat. Some thresholding artifacts can be present, such as intensity blooming near the edges of the material. This is commonly caused by electron charge accumulation between the specimen and mount material.

Plotting the thickness as a function of the *x* direction in the image shows the range of thickness values. The first and last 10–15% of the data points were discarded in some cases because the sample had been sheared for mounting considerations and this shearing introduces a significant amount of deformation that artificially increases or decreases the thickness. Removing the data points associated with these regions yields a better estimate of the overall plate thickness variation (Figure 7).

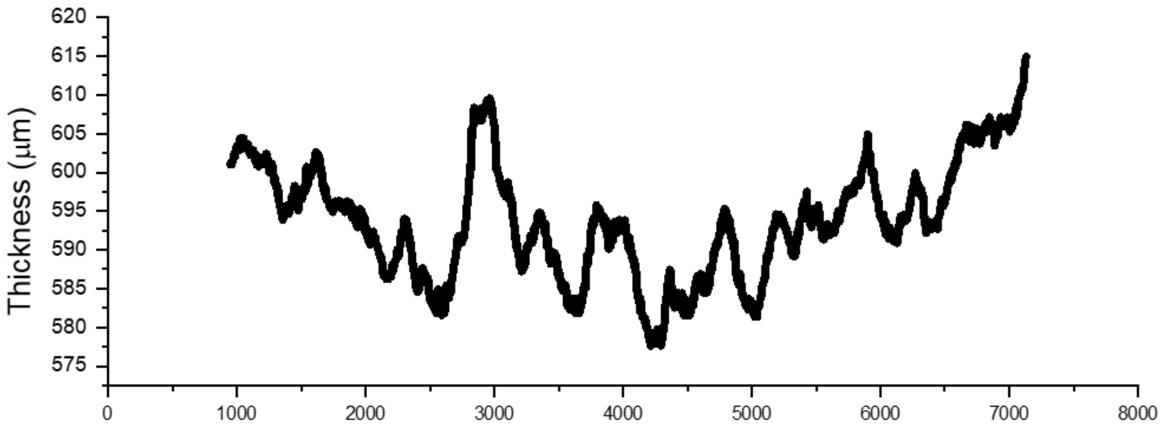


Figure 7. Thickness values overlaid on a black-and-white threshold U-10Mo specimen. The first and last 15% were removed because shearing and preparation of the specimen resulted in deformation which would bias the true thickness statistics. Using the middle 70% of data, a good estimate of thickness can be made. (x-axis associated with pixel columns from left-right)

6.0 Conclusion

An automated method for determining the thickness of Zr cladding and fuel meat thickness in U-10Mo fuel plates were developed and used to optimize the BWXT MP2 test campaign and all the future campaigns for characterization. Image processing algorithms were utilized to segregate the cladding and meat along with steps to remove noise introduced from the secondary phases present in the material. From here, a pixel-by-pixel calculation was done to determine the thickness variations based off of black-white images of each layer. This pixel-by-pixel calculation mitigates the human error of under sampling the thickness by hand and gathers a full statistical description of the thickness across the image.

7.0 Quality Assurance

This work was performed in accordance with the Pacific Northwest National Laboratory (PNNL) Nuclear Quality Assurance Program (NQAP). The NQAP complies with the United States Department of Energy Order 414.1D, Quality Assurance. The NQAP uses NQA 1 2012, Quality Assurance Requirements for Nuclear Facility Application as its consensus standard and NQA 1 2012 Subpart 4.2.1 as the basis for its graded approach to quality.

This work emphasized acquiring new theoretical or experimental knowledge. The information associated with this report should not be used as design input or operating parameters without additional qualification.

APPENDIX A : Source Code

Below, the source codes for the Zr and fuel meat Octave scripts are listed.

U-10Mo_Zr_measurement.m

```
1; %Dummy to start script
pkg load image
pkg load statistics
pkg load io

function [avg, stdev] = getThicknessVariation (data)

  %Sum along columns, equals number of pixels
  sums = sum(data,1);
  %colThickness = numel(imageMask(:,1))-sums;

  avg = mean(sums);
  stdev = std(sums);

endfunction


%% Initiate image import and convert to black and white----------------------
  close all

  % Get image path information
  imagePath = imgetfile()
  [dir, name, ext] = fileparts(imagePath)

  % Load image --> Smooth out noise
  imageData = imread(imagePath);
  imageData = im2uint8(imageData);
  imageData = wiener2(imageData);
  %%
  %imageData = histeq(imageData);
  figure
  imshow(imageData);
  figure
  imhist(imageData);
  threshholdValues = input('Input Estimated Threshold Values ([lower high]): ')

  % Detect Fuel plate
  threshMask = imageData;

  %UPDATE VALUES FROM MANUAL THRESHOLD VIA IMAGEJ
  tmp = threshMask > threshholdValues(1) & threshMask < threshholdValues(2);
  imageBW = im2bw(imageData);
  imageBW = imcomplement(imageBW);
  mask = imageBW.*tmp;
  figure
  imshow(mask)
  mask = bwareaopen(mask,1500); %Remove internal blobs
  mask = imfill(mask,"holes"); %fill in bad points
  figure
```

```
 imshow(mask)
imwrite(mask,[dir '\' name '_MASKED' '.bmp'])

 %% Crop pic in half

 dims = size(mask)
 IS_EVEN = ~mod(dims(1),2); %check if y points are even, if not pad row with
zeros
 if IS_EVEN==0
  fprintf("Adding zero row...");
  zeroRow = zeros(1,dims(2));
  mask = [mask; zeroRow];
 end

 dims = size(mask)
 xdim = dims(2);
 ydim = dims(1);
 % Upper
 maskUpper = imcrop(mask, [1 1 xdim ydim/2]);
 % Lower
 maskLower = imcrop(mask, [1 ydim/2 xdim ydim]);

 maskSidebySide = [maskUpper maskLower];

 %% Thickness Variation Calculation ----------------------------------------

  pixelScale = 4.06 %pixels/micron ADJUST IF NEEDED
  [avgThicknessPixels, devThicknessPixels] =
getThicknessVariation([maskUpper maskLower]);
  %[avgThicknessPixels, devThicknessPixels] =
getThicknessVariation([maskLower]);
  combinedThicknessMicrons = [avgThicknessPixels
devThicknessPixels]/pixelScale

colSumLower = sum(maskLower,1);
colSumUpper = sum(maskUpper,1);
colSum = sum(maskSidebySide,1);
colSumLower = colSumLower';
colSumUpper = colSumUpper';
colSum = colSum';

colSumAll = [colSumLower colSumUpper];
csvwrite(['CHANGE TO DIR OF CHOICE' '\' name '_colSumsZr' '.csv'], colSumAll)
%cell2csv([dir '\' name '_colSumsZr' '.csv'], colSumAll)
```

U-10Mo_Thickness_measurement2.m

```
1; %Dummy to start script
pkg load image
pkg load statistics

%% Define thickness calc function (may not be needed...)---------------------

function [avg, stdev] = getThicknessVariation (imageMask)
```

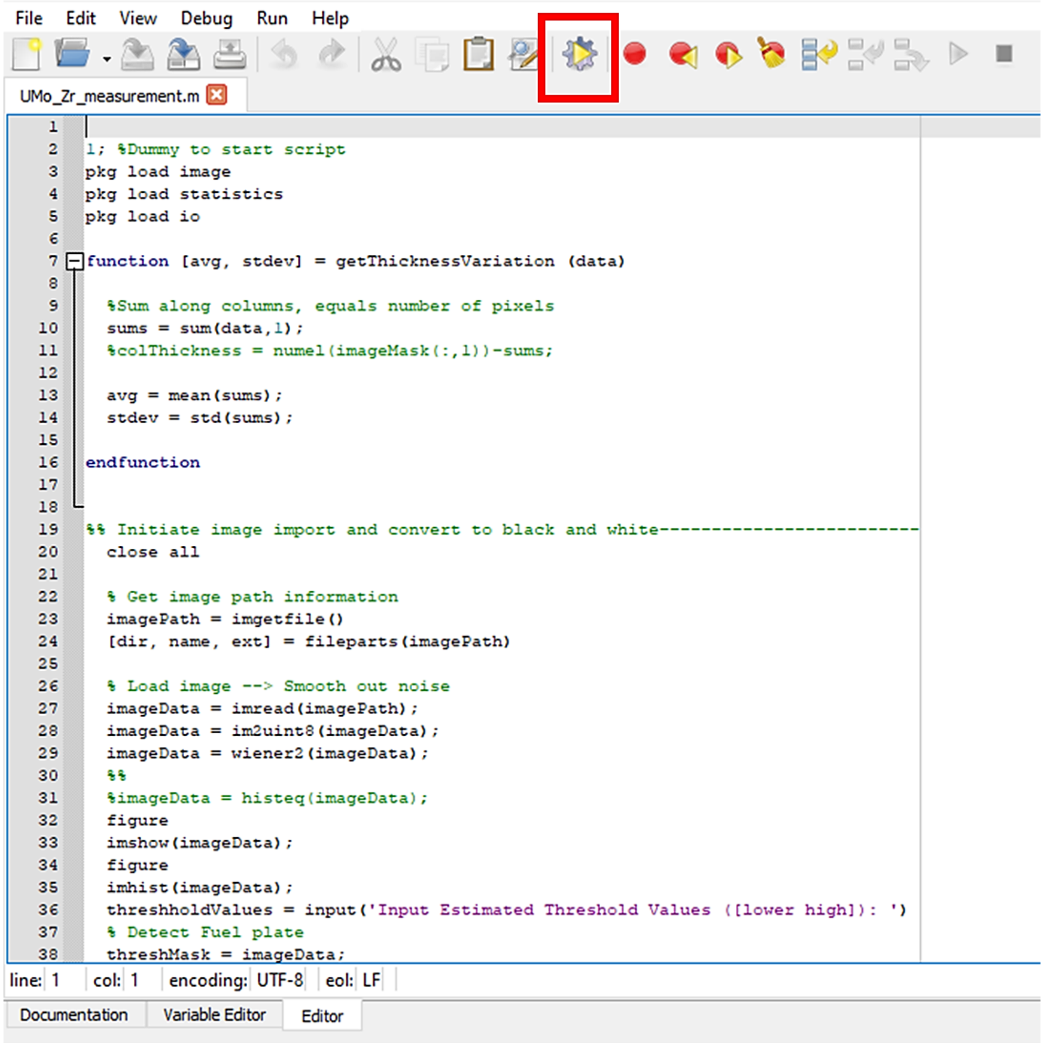
```
%Sum along columns, equals number of pixels
sums = sum(imageMask,1);
%colThickness = numel(imageMask(:,1))-sums;
avg = mean(sums);
stdev = std(sums);
endfunction

%% Initiate image import and convert to black and white----------------------

close all
imagePath = imgetfile() % Get image path information
[dir, name, ext] = fileparts(imagePath)
imageData = imread(imagePath); % Load image --> convert to black and white
imageGrey = im2bw(imageData);

figure()
title = 'bw image';
imshow(imageGrey)

%% pixel Scale Calibration---------------------------------------------------

imageDims = size(imageGrey)
ydim = imageDims(1);
xdim = imageDims(2);
imageCrop = imcrop(imageGrey, [0 ydim-25 1050 25]);
figure
title ='bw image after crop';
imshow(imageCrop)
% UNCOMMON FOR USER PIXEL SCALE DETERMINATION
% [ptx, pty] = ginput(2)
% pixelDist = norm(ptx - pty)
% pixelScale = pixelDist/250 %Define scale length
pixelScale = 4.06 %pixels per um, determined from imageJ

%% Thresholding to remove outliers, blobs, spurs-----------------------------

Mask = imfill(imageGrey,'holes'); %remove blobs LEAVES EDGES-NEED TO SOLVE
##  figure()
##  title ='After fill';
##  imshow(Mask)
Mask([1:21],:)=0; %remove image title and scale bar info
Mask([end-20:end],:)=0;

figure
title = 'Prior to thickness calc';
imshow(Mask)

%% Thickness Variation Calculation ------------------------------------------

[avgThicknessPixels, devThicknessPixels] = getThicknessVariation(Mask)
thicknessMicrons = [avgThicknessPixels devThicknessPixels]/pixelScale

%% Save statistic information -----------------------------------------------

statFile = [dir '\' name '_ThicknessSummary.txt']
dlmwrite(statFile, thicknessMicrons)
```

```
colSum = sum(Mask,1);
colSum = colSum';
csvwrite([dir '\' name '_colSums' '.csv'], colSum)
```

APPENDIX B : Process Walk-Through

U-10Mo_Zr_measurement.m

After loading the image analysis script into Octave, run the code using the button highlighted in Figure 8. The script will then prompt the user for the location of the cross-section montage of choice.



```

1; %Dummy to start script
pkg load image
pkg load statistics
pkg load io

function [avg, stdev] = getThicknessVariation (data)

  %Sum along columns, equals number of pixels
  sums = sum(data,1);
  %colThickness = numel(imageMask(:,1))-sums;

  avg = mean(sums);
  stdev = std(sums);

endfunction


%% Initiate image import and convert to black and white-------------------------
  close all

  % Get image path information
  imagePath = imgetfile()
  [dir, name, ext] = fileparts(imagePath)

  % Load image --> Smooth out noise
  imageData = imread(imagePath);
  imageData = im2uint8(imageData);
  imageData = wiener2(imageData);
  %%
  %imageData = histeq(imageData);
  figure
  imshow(imageData);
  figure
  imhist(imageData);
  threshholdValues = input('Input Estimated Threshold Values ([lower high]): ')
  % Detect Fuel plate
  threshMask = imageData;
```

Figure 8. Octave interface with the Run Script button highlighted (red)

After the image has loaded, the user will be presented with a series of figures showing the uploaded image and a histogram of gray values. Selecting the second peak away from zero (Figure 2) in the histogram will isolate the Zr layers. In the command window, input the edges of the Zr peak *[left-value (space) right-value]*. Wrong input will cause an error and the process will need to restart. Bug fixes and code optimization is ongoing.

Once the thresholds have been set, the process will need no further intervention, because the thickness summary outputs will be placed in the starting image directory. Top and bottom Zr layers will be separated for further analysis.

U-10Mo_Thickness_measurement2.m

Calculation of the U-10Mo fuel meat thickness will be similar to analyzing the Zr measurements except that input of thresholding will not be necessary. The conversion of the gray image to 8-bit black and white will inherently isolate the necessary areas without user intervention as described in section APPENDIX A.

Simply run U-10Mo_thickness_measurement2.m in Octave, locate the montaged image, and select it. The output will be saved in the image directory for further analysis by the user's software of choice.

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