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Electroplating Zr on U-Mo Foils

Process Qualification Report

May 2017

KD Meinhardt
WD Bennett
ME Dahl
MA Catalan

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Richland, Washington 99352

Statement of Work Revision History

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Abstract

Fuel Fabrication identified electroplating of zirconium onto the U-Mo fuel low enriched fuel for high performance reactors as an alternative technology. The Process Qualification was performed under the Process Qualification Plan for Fabrication of MP-1 Zirconium Electroplated Foils, Pacific Northwest National Laboratory (PNNL), 67075-PQP-0001.

On December 14, 2016 PNNL was authorized to begin qualification plating runs effective January 2017 by the Fuel Fabrication Pillar. PNNL began electroplating of the BWX Technologies Nuclear Operation Group provided qualification bare foils. Receipt inspection of foils identified numerous issues with the surface quality of the foils. Zirconium Electroplating of the 12 thick and 12 thin foils was completed in March 2017.

This report summarizes the results of qualification plating runs and provides the documentation necessary to qualify the electroplating process and authorize PNNL to begin plating of the MP-1 foils. Prior to plating of the MP-1 foils, PNNL will be plating demonstration low enriched uranium (LEU).

Acronyms and Abbreviations

B&W	Babcock and Wilcox
BWXT	BWX Technologies Nuclear Operation Group (formerly Babcock and Wilcox)
DU	depleted uranium
DE	Destructive Examination
EDS	energy dispersive X-ray spectroscopy
FSP-1	full-size plate, first campaign
ICP/MS	inductively coupled plasma and mass spectroscopy
IGF	inert gas fusion
INL	Idaho National Laboratory
LANL	Los Alamos National Laboratory
LEU	Low Enriched Uranium
MAQP	Manufacturing and Quality Plan
MP-1	mini-plate-1 experiment, first campaign
NCR	Nonconformance Report
PNNL	Pacific Northwest National Laboratory
PQP	Process Qualification Plan
QA	Quality Assurance
QA-POC	Quality Assurance Point-of-Contact
SEM	scanning electron microscopy
U-Mo	an alloy consisting of uranium and nominally 10 weight percent molybdenum
USHPRR	United States High Performance Research Reactors

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1.0 Introduction

This Process Qualification Report (PQR) documents the activities and criteria necessary to qualify the Fabricator, Pacific Northwest National Laboratory (PNNL), to begin electroplating a zirconium (Zr) diffusion barrier onto uranium-molybdenum (U-Mo) foils used in the production of mini fuel plates. This plan has been prepared for the United States High Performance Research Reactors (USHPRR) fuel conversion program to use mini plates in the Mini-Plate (MP-1) experiment to be performed in the Idaho National Laboratory (INL) Advanced Test Reactor (ATR).

This PQR applies to PNNL's MP-1 project to develop an alternative approach to apply Zr to the surface of U-Mo alloy fuel plates that will be irradiated in the test. Together, the Process Qualification Plan document number 67076-PQP-001, and PNNL Manufacturing and Quality Plan for Fabrication of MP-1 Zirconium Electroplated Foils document number 67075-MAQP-001, describe the work to be performed and are intended to demonstrate compliance with the Fuel Specification for MP-1 and FSP-1, SPC-1691.

In support of the MP-1 test, PNNL will electroplate low enriched uranium (LEU) and high enriched uranium (HEU) U-Mo foils received from BWX Technologies Nuclear Operation Group (BWXT). The foils will be of two different nominal thicknesses: 0.0085 and 0.025 inches. The electroplating process will initially be demonstrated on depleted uranium (DU) foils at Plasma Processes, a PNNL subcontractor, and then the process will be qualified on DU foils at PNNL. Once qualified on DU, the process will be employed on LEUHEU mini-foils for use in the MP-1 test.

The electrolytes used to deposit Zr are mixtures of ZrF_4 and alkali fluorides. The U-Mo foil is used as the cathode and Zr electrode is inserted into the molten salt to serve as the anode. A current density is applied at a preset temperature and the ZrF_4 is disassociated, resulting in the Zr^{4+} ions traveling to the foil where they are deposited in a coating layer. The atmosphere (oxygen and water content) is controlled to ensure the quality of the deposited Zr coating. The detailed electroplating process to be qualified is documented in the Manufacturing and Quality Plan, 67075-MAQP-001.

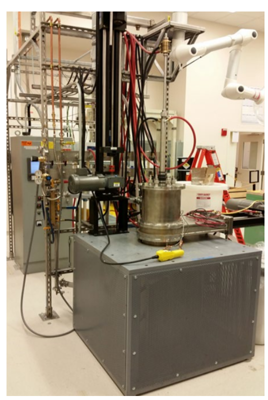

Figure 1. Electroplating Equipment

2.0 Qualification Criteria

2.1 Zirconium Composition

The raw Zr and the acceptable electroplating salts were procured as commercial items with supplier provided certificates of analysis. The Zr composition of the completed coated foils was measured by ATI Specialty Alloys and Components, Analytical Services in Albany, Oregon (ATI) by chemical analysis and reported for informational purposes.

The elements to be measured with the chemical analysis are specified in Table 1of the ASTM B352-11 standard (UNS R60001) and will provide information on the Zr composition. The average result of the sample compositions will be compared to the acceptance criteria. These elemental measurements were performed using industry standard measurement techniques in an analytical laboratory certified in accordance with ISO/IEC 17025:2005 for Chemical Testing of Metals. PNNL Acquisition Quality Support Services verified that ATI was a qualified supplier certified to the ISO/IEC 17025 in the field of Chemical Testing of Metals.

Table 1. Zirconium Composition[1]

Element	ppm	Element	ppm
Al	75	Mn	50
B	0.5	Mo	50
C	270	N	80
Cd	0.5	Ni	70
Co	20	O	1000
Cr	200	S	120
Cu	50	Sn	50
Fe	1500	Ti	50
H	25	U	3.5
Hf	100	W	100
Mg	20		

The average results of the sample compositions will be reported and compared to the limits of ASTM B352-11 (UNS R60001). Because this ASTM standard applies to hot-rolled and cold-rolled products, not plated Zr, these Zr composition results are reported for informational purposes only.

2.2 Zirconium Thickness

Foil shall have a coating thickness of 0.001±0.0005 inch on each side of the foil. Zr thickness variability (taper lengthwise and cross section) is determined by destructive examination coupled with weight

[1] From ASTM B352-11 (UNS R60001)

gain/micrometer method. Eight optical metallography measurements are made at three locations for six foils of each thickness (0.025 and 0.0085 inches) as shown in Figure 2.

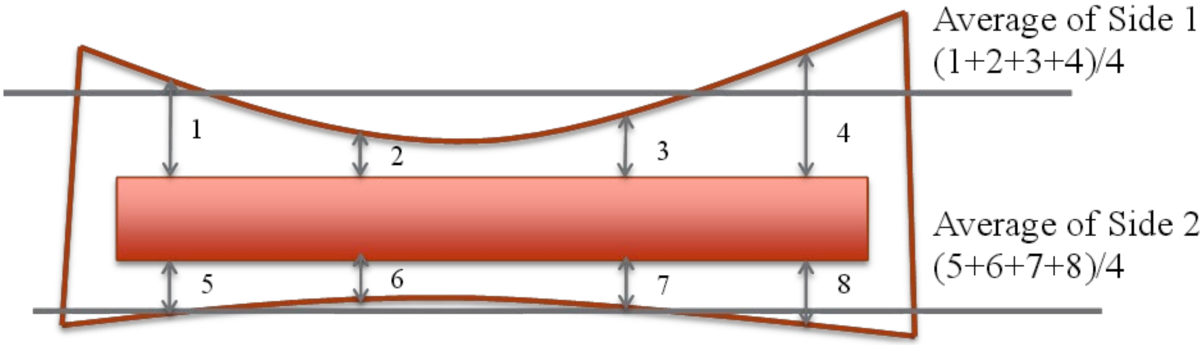

Figure 2. Optical Measurements

The minimum and maximum thickness capability of the electroplating process were determined by destructive examination with optical imaging using the following four steps:

1. Find the minimum average thickness for all six foils of each thickness (2 sides or 12 thickness measurements).
2. Calculate the standard deviation for all thickness measurements (24 total measurements).
3. Subtract two standard deviations from the minimum thickness. This thickness will be the minimum thickness capability of the electroplating process.
4. The analysis of the maximum thickness capability will be performed by adding two standard deviations to the maximum thickness.

2.3 Zirconium Coverage

100% coverage of in-plane surfaces (not the edges) will be confirmed via visual inspection.

2.4 Surface Condition

The foil shall have no surface defects (voids, cracks, scratches, inclusions) having any surface dimension larger than 0.06 inch will be confirmed by visual inspection.

2.5 Identification

Identifier visibly confirmed via visual inspection.

3.0 Electroplating and Qualification Process

3.1 Receipt Inspection

Foils were inspected in accordance with 67075-SOP-0003, *Inspecting Bare U-Mo Foils and Zirconium Electroplate Foils* by a certified inspector qualified to the requirements of 67075-SOP-0009, *Qualification and Certification of Inspection Personnel*. The results of the inspections were documented in the corresponding Data Sheet and Traveler (Appendix B). These inspections occurred upon receipt and post cleaning. Numerous foils held noticeable surface defects and scratches. After receipt inspection, the foils were immediately cleaned and plated. The foils were cleaned following 67075-SOP-0002, *Cleaning of Uncoated U-Mo Foils*. Results of the cleaning were documented on the Data Sheet and Traveler. Mass loss during the cleaning process was recorded as were the dimensions prior to plating and are provided in Table 2. Dimensional measurements in Table 2 are averages.

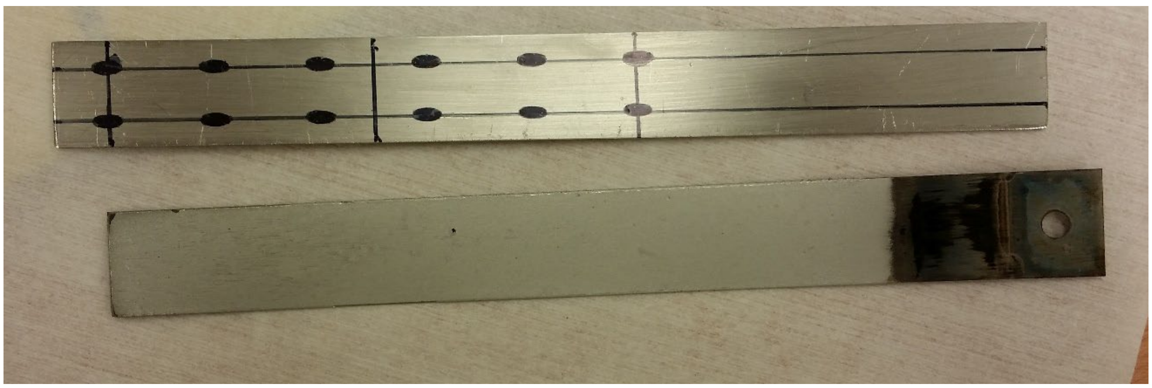

Figure 3. Measurement Locations

Figure 3 is a guide developed to help provide uniformity in measurements. The horizontal and vertical lines identify the two length and three width measurements. The 12 thickness measurement approximate locations are identified as black dots. The average length, width, and thickness measurements are provided in Table 2 along with the mass loss per foil. The average resulting mass loss was 0.2698 grams for all foils with the average thin foil losing 0.1740 and the average thick foil losing 0.3779 grams.

Table 2. Receipt Inspection Measurements[2]

Foil ID	Cleaning Date	Length (in)	Width (in)	Thick (in)	Mass Loss (g)
Q1E012	12/29/2016	6.9998	0.7502	0.0113	0.0736
Q1E013	1/3/2017	7.0005	0.7428	0.0114	0.0662
Q1E014	1/3/2017	6.9788	0.7453	0.0114	0.1056
Q1E016	1/4/2017	7.0043	0.7550	0.0114	0.1200
Q1E017	1/4/2017	7.0075	0.7483	0.0112	0.1237
Q1E021	1/10/2017	6.9958	0.7495	0.0108	0.1531
Q1E024	1/11/2017	6.9823	0.7532	0.0112	0.1270
Q1E026	1/12/2017	7.0073	0.7552	0.0111	0.0983

[2] Dimensional measurements are averageaverages per Figure 3

Foil ID	Cleaning Date	Length (in)	Width (in)	Thick (in)	Mass Loss (g)
Q1E028	1/12/2017	7.0375	0.7438	0.0110	0.0752
Q1E029	1/13/2017	7.0070	0.7497	0.0107	0.0769
Q1E030	1/16/2017	7.0063	0.7605	0.0109	0.0845
Q1E033	1/16/2017	7.0005	0.7440	0.0112	0.1121
Q1E034	1/20/2017	7.0023	0.7535	0.0112	0.0721
Q1E035	1/23/2017	7.0030	0.7562	0.0111	0.0829
Q1E040	1/24/2017	6.9970	0.7530	0.0115	0.0979
Q1E041	1/25/2017	7.0023	0.7497	0.0106	0.0990
Q1E042	1/26/2017	6.9980	0.7485	0.0106	0.1268
Q1E043	2/2/2017	6.9978	0.7548	0.0100	0.1736
Q1E044	2/6/2017	6.9968	0.7555	0.0107	0.0930
Q1E045	2/6/2017	6.9960	0.7487	0.0106	0.0992
Q1E046	2/15/2017	6.9938	0.7477	0.0106	0.1074
Q1E047	2/16/2017	6.9975	0.7570	0.0125	0.0846
Q1E048	2/17/2017	6.9935	0.7485	0.0121	0.0828
Q1E049	2/20/2017	7.0013	0.7463	0.0117	0.1009
Q1E050	2/21/2017	7.0055	0.7557	0.0112	2.1165
Q1E052	2/21/2017	7.0125	0.7498	0.0109	0.1346
Q1E053	3/22/2017	6.9985	0.7565	0.0112	0.0902
Q1E054	3/22/2017	7.0010	0.7500	0.0112	0.0945
Q2E001	2/23/2017	7.0035	0.7432	0.0249	0.6930
Q2E002	2/7/2017	7.0025	0.7528	0.0246	0.1950
Q2E003	2/7/2017	7.0020	0.7518	0.0247	0.2268
Q2E004	2/8/2017	7.0065	0.7453	0.0247	0.1520
Q2E005	2/22/2017	7.0023	0.7525	0.0251	0.1907
Q2E006	2/9/2017	6.9983	0.7487	0.0247	0.2038
Q2E007	2/10/2017	7.0018	0.7438	0.0244	0.1938
Q2E008	2/13/2017	7.0060	0.7532	0.0244	0.2873
Q2E009	2/14/2017	7.0060	0.7198	0.0275	0.3200
Q2E010	2/16/2017	7.0063	0.7510	0.0275	0.1846
Q2E011	2/23/2017	7.0005	0.7437	0.0490	0.2006
Q2E012	2/24/2017	6.9005	0.7522	0.0292	1.4964
Q2E013	2/28/2017	7.0050	0.7498	0.0291	0.4937
Q2E014	3/1/2017	7.0045	0.7530	0.0295	1.3512
Q2E015	3/2/2017	6.9995	0.7473	0.0296	0.7719
Q2E016	3/3/2017	6.9958	0.7515	0.0536	0.1978
Q2E017	3/6/2017	7.0080	0.7548	0.0303	0.2000
Q2E018	3/7/2017	6.9895	0.7503	0.0285	0.1897
Q2E019	3/14/2017	7.0048	0.7535	0.0285	0.1990
Q2E020	3/14/2017	7.0058	0.7504	0.0292	0.2463

Foil ID	Cleaning Date	Length (in)	Width (in)	Thick (in)	Mass Loss (g)
Q2E021	3/16/2017	7.0000	0.7463	0.0280	0.3027
Q2E022	3/17/2017	6.9970	0.7530	0.0283	0.1955
Q2E023	3/21/2017	6.9958	0.7477	0.0288	0.1996

3.1.1 Visual Inspection

As received surface finish defects were not visible prior to cleaning in many cases. Many foils had a noticeable burr and other surface defects. Examples of these foils are provided in Figure 4 through Figure 8. These foils were accepted to be plated for qualification under 67075-NCR-0TS-04107.

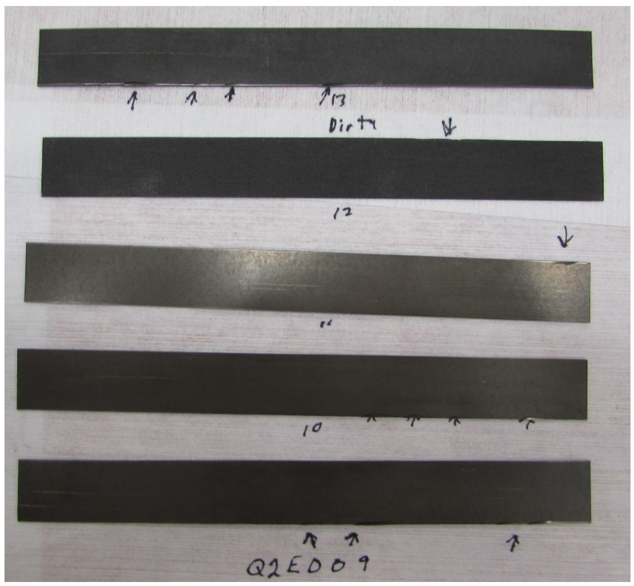

Figure 4. Receipt Inspection (Thick Foils)

Figure 5. Receipt Inspection – Cracked Foil

Figure 6. Receipt Inspection - Scratches

Figure 7. Receipt Inspection - Scratches

Figure 8. Receipt Inspection - Burr and Surface Defect

3.2 Plating

Foils were plated according to 67075-SOP-0005, *Electroplating Zirconium on U-Mo Foils*. The plating process was limited due to the quality of the foils (burrs and surface defects in the base metal negatively affected the plating process). These defects resulted in poor surface adhesion, and in some locations poor coverage. These defects can be seen in Figure 13. Cracking of Foil (Q2E020) through Figure 15**. Q1E024 Showing Burr Effect on Plating**. The plating mass (e.g., thickness) was calculated based on the parameters and plating time.

3.2.1 Foils for Cleaning Studies

Foils Q1E001 through Q1E011 were used to finalize process parameters, including the cleaning procedure. These foils were also used for hot isostatic press studies and adhesion tests. These foils are removed from the net-foil (total foils that passed inspection vs total foils plated for qualification) calculations.

3.2.2 Nonconforming Foils for DE

For both the thick and thin foils, six foils of each type were selected for destructive examination (DE). These foils were selected because they met the mass and dimensional requirements; however, failed visual inspection due to surface defects. These foils were included in the net-foil calculation as failed foils. These nonconforming foils were documented, dispositioned, and approved for use in DE inNonconformance Reports (NCRs) 67075-NCR-OTS-03995, Rev 0, 67075-NCR-OTS-04101, Rev. 0, 67075-NCR-OTS-04102, Rev. 0, and 67075-NCR-0TSOTS-04051, Rev 0.

3.3 Foils Q1E029 and Q1E054

Two foils were plated longer than normal in an effort to obtain the necessary Zr mass for chemical composition sampling. Three samples from Foil Q1E029 were extracted for chemical analysis and two samples from Foil Q1E054 were extracted for radiological release of the Zr and subsequent chemical analysis.

3.4 Post-Plating Inspection

Foils were inspected after plating with 67075-SOP-0003, *Inspecting Bare U-Mo Foils and Zirconium Electroplate Foils* using a certified inspector that met the requirements of 67075-SOP-0009, *Qualification and Certification of Inspection Personnel*. The results of the inspections were documented in the corresponding Data Sheet and Traveler Representative foil images are provided in Figure 9 and Figure 10. Numerous foils held noticeable surface defects and markings. Table 3 provides the acceptable mass gain range.

Table 3. Acceptable Mass Gain

Plated Length (in)	**Thin Foil**		**Thick Foil**	
	Minimum Zr Mass (g)	Maximum Zr Mass (g)	Minimum Zr Mass (g)	Maximum Zr Mass (g)
5.4	0.4491	1.3158	0.4594	1.3467
5.5	0.4574	1.3402	0.4679	1.3716
5.6	0.4657	1.3645	0.4764	1.3965
5.7	0.4741	1.3889	0.4849	1.4213
5.8	0.4824	1.4132	0.4934	1.4462
5.9	0.4907	1.4376	0.5019	1.4711
6.0	0.4990	1.4619	0.5104	1.4960
6.1	0.5073	1.4863	0.5188	1.5209

Looking at strictly mass gain in Figure 9, an indication of plating thickness, all foils met the mass requirements. The targeted mass is roughly one gram of Zr. The rejected foils had issues with coverage and surface defects but not plating thickness.

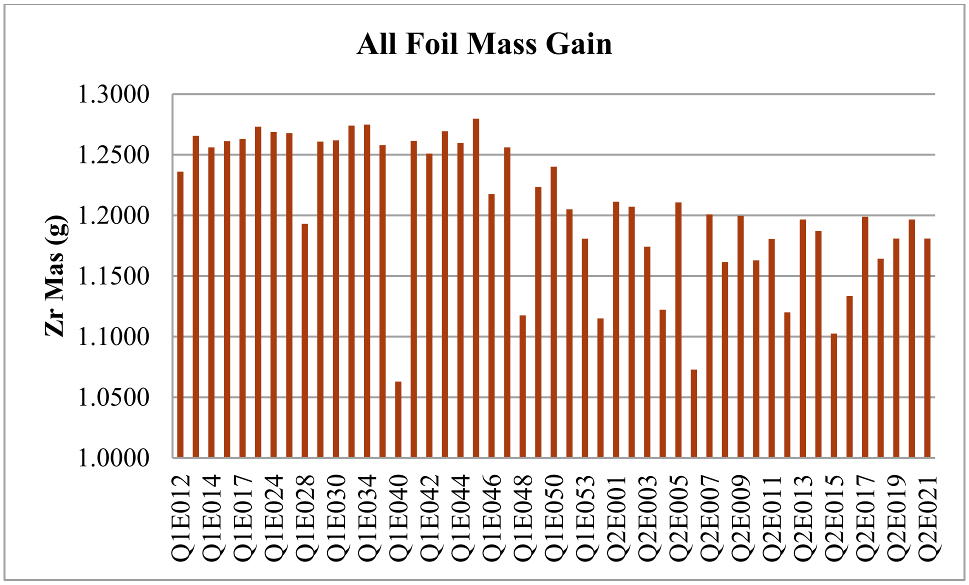

Figure 9. Mass Gain (All Foils)

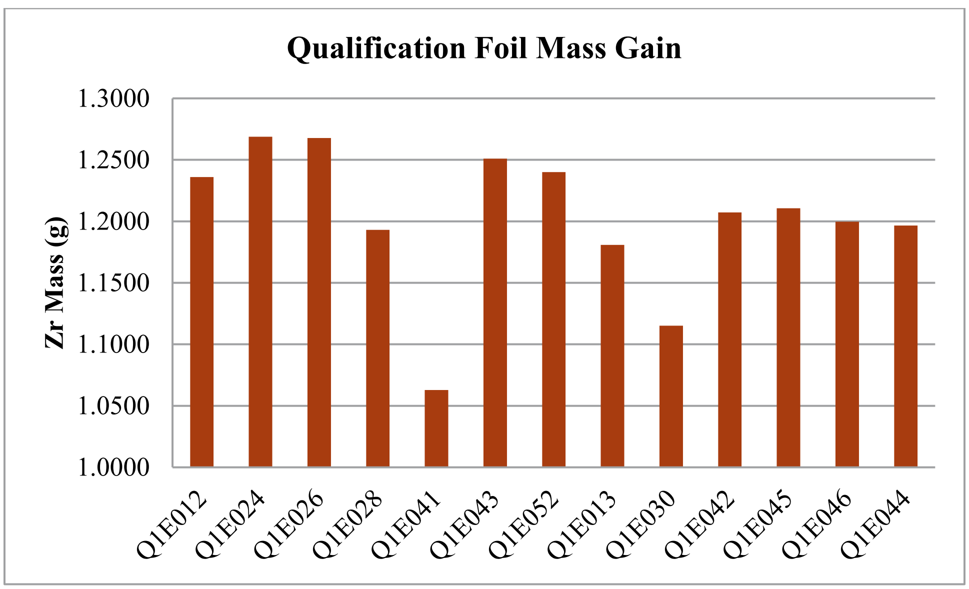

Figure 10. Mass Gain (Passing Foils)

As depicted in Figure 10, the average mass gain for all foils was 1.2059 grams for all the foils and 1.2162 for only the qualification foils. A detailed breakdown of the averages is provided in Table 4.

Table 4. Mass Gain

Foil	Qualification (pass/DE) (g)	All Plated Foils (g)
All Thicknesses	1.2163	1.2059
Thin	1.2397	1.2407
Thick	1.1857	1.1679

4.0 Qualification Data

4.1 Zirconium Composition

The supplier provided certificates of analysis and chemical test reports are included in Appendix A. The results of the chemical analysis are provided in Table 5.

Table 5. Zirconium Composition (in ppm)[3]

Element	ASTM-B352-11	Meets ASTM B352-11	Anode	Q1E029 2/8/17 (bottom)[5]	Q1E029 3/30/17 (middle)	Q1E029 3/30/17 (top)	Q1E054[4] 3/30/17	Q1E054[4] 3/30/17
Al	75	No	50	83	170	120	84	89
B	0.5	Yes	0.4	<.25	<.25	<.25	<.25	<.25
C	270	Yes	140	<20	70	50	<20	<20
Ca		Yes	0	<10	<10	<10	<10	<10
Cd	0.5	Yes	0.4	<.25	<.25	<.25	<.25	<.25
Co	20	Yes	13	<10	<10	<10	<10	<10
Cr	200	Yes	80	<50	<50	<50	<50	<50
Cu	50	Yes	12	<25	<25	<25	<25	<25
Fe	1500	Yes	180	<50	<50	<50	<50	<50
H	25	No	10	63			63	74
Hf	100	Yes	60	<25	<25	<25	<25	<25
Mg	20	Yes	11	<10	<10	<10	<10	<10
Mn	50	Yes	13	<25	<25	<25	<25	<25
Mo	50	Yes	20	<10	83	29	<10	<10
N	80	Yes	30	<20	<20	<20	<20	<20
Nb		Yes		<50	<50	<50	<50	<50
Ni	70	Yes	50	<35	<35	<35	<35	<35
O	1000	No	170	48000	36000	38000	36,000	34000
Si	120	Yes	60	<10	<10	<10	<10	<10
Sn	50	Yes	10	<25	<25	<25	<25	<25
Ti	50	Yes	20	<25	<25	<25	<25	<25
U	3.5	No	1	45	580	210	30	36
W	100	Yes	30	<25	<25	<25	<25	<25

[3] From ASTM B352-11 (UNS R60001)

[4] Sufficient Zr was available in the sample to repeat the analysis.

[5] Q1E029 (Bottom) was analyzed as QUAL-1 by ATI.

As can be seen in Table 5, the electroplated Zr tends to have significantly less trace elements with the noticeable exceptions of aluminum, hydrogen, oxygen, and uranium. The composition of the anode was provided as a reference to show it was not the source of the higher than expected elements. The source of the uranium is likely from the foil.

In addition to the chemical analysis, for informational purposes, microhardness of the plated Zr was taken on Foil Q1E029 at three locations (top, middle, and lower). These are the rough locations where the Zr was taken for LECO oxygen measurements. Qualitatively, the plating from the top of the foil was more difficult to remove than the bottom. The hardness to oxygen content relationship was taken from a plot based on Nakatsuka etal. (1985). Journal of Nuclear Science and Technology 22, pp. 239-241 (March 1985). The oxygen content in the paper only went to about 1000 ppm O2 but a linear fit of the data was extrapolated to higher values and appears to give a reasonable estimate of the oxygen content. The relationship used was $y = 36.51x + 451.21$ where x is the square root of the wt% of oxygen in ppm and y is the Vickers hardness in MPa. The resulting calculated oxygen based on microhardness matched the oxygen levels measured (this is an order of magnitude estimation). This is shown in Table 6.

Table 6. Oxygen/Hardness Q1E029

Location	Vickers Estimate	LECO
Top	48,936	38,000
Middle	36,523	36,000
Bottom	37,854	48,000

4.2 Qualification Foils

The following foils (Table 7) were selected for qualification. This list represents all the passed foils (sheared to length) and the foils with surface defects sheared for DEs.

Table 7. Selected Qualification Foils

Foil ID	Plating Date	Mass Zr (g)	Thickness (in)[6]	Sheared Length (in)
Q1E012	12/29/2016	1.23600	0.00130	5.33775
Q1E024	1/11/2017	1.26880	0.00144	5.73300
Q1E026	1/12/2017	1.26780	0.00125	5.54725
Q1E028	1/12/2017	1.19310	0.00125	5.68100
Q1E041	1/25/2017	1.06290	0.00135	5.67725
Q1E043	2/2/2017	1.25100	0.00148	5.59075
Q1E052	2/21/2017	1.24010	0.00145	5.55148

[6] Micrometer readings

Foil ID	Plating Date	Mass Zr (g)	Thickness (in)[6]	Sheared Length (in)
Q1E013	1/3/2017	1.26560	0.00146	
Q1E030	1/16/2017	1.26090	0.00121	
Q1E042	1/26/2017	1.26140	0.00133	
Q1E045	2/6/2017	1.25970	0.00115	
Q1E046	2/15/2017	1.27970	0.00173	
Q1E044	2/6/2017	1.26950	0.00108	
Q2E001	2/23/2017	1.18080	0.00129	5.55000
Q2E002	2/7/2017	1.11510	0.00125	5.67980
Q2E004	2/8/2017	1.20720	0.00129	5.69225
Q2E007	2/10/2017	1.21070	0.00137	5.69000
Q2E011	2/23/2017	1.19970	0.00120	5.44800
Q2E015	3/2/2017	1.19660	0.00122	5.58100
Q2E003	2/7/2017	1.21130	0.00124	
Q2E005	2/22/2017	1.17420	0.00123	
Q2E012	2/24/2017	1.16290	0.00137	
Q2E019	3/14/2017	1.19880	0.00131	
Q2E020	3/14/2017	1.16430	0.00138	
Q2E023	3/21/2017	1.18090	0.00123	

4.2.1 Zirconium Thickness

Optical measurements were performed on six thin and six thick foils to confirm the relationship between mass gain and plating thickness as required by the PQR. Foils were sectioned according to the PQR and optical measurements performed according to 67075-SOP-0010, *DEs of Zirconium Electroplated U-Mo Foils*. A few representative figures are provided in Figure 12 through Figure 17. All optical measurement images are provided in Appendix B. The results are presented Table 8. Optical measurements were performed in micrometers. The specification required thickness was 0.0010 inches with a minimum of 0.005 inches and a maximum 0.0015.[7]

Figure 11 provides the average Zr thickness per side and is accompanied by Table 9 provides the overall average optical measurements. All the measurements per side were averaged using the Excel Average function providing to get the per side thickness measurements identified in Figure 2 identified in the PQP. All these average foil thickness measurements (Table 9) were then averaged to determine the overall average and standard deviation provided in Table 8. The average of the optical measurements is within the specification as is the two sigma minimum thickness (per the PQR) and maximum thickness. The standard deviation (STD DEV) was calculated by Excel using the STDEV.P function.

[7] The electroplating parameters were set to intentionally bias the plating on the high (thicker) side.

Table 8. All Qualification Foil Average

	Min (in)	Ave (in)	Max (in)
	0.0008	0.0011	0.0014
STD DEV (in)		0.0002	

More detailed information is provided in Table 9, the results of the individual foil optical measurements. Some foils had measurements that indicated Zr coverage outside of specification. When the per side averages were calculated, only one foil (Q1E013) had a thickness outside of specification. The majority of the individual measurements that were outside specification tended to be higher than specification (i.e. the plating thickness was greater than 0.0015 inches).

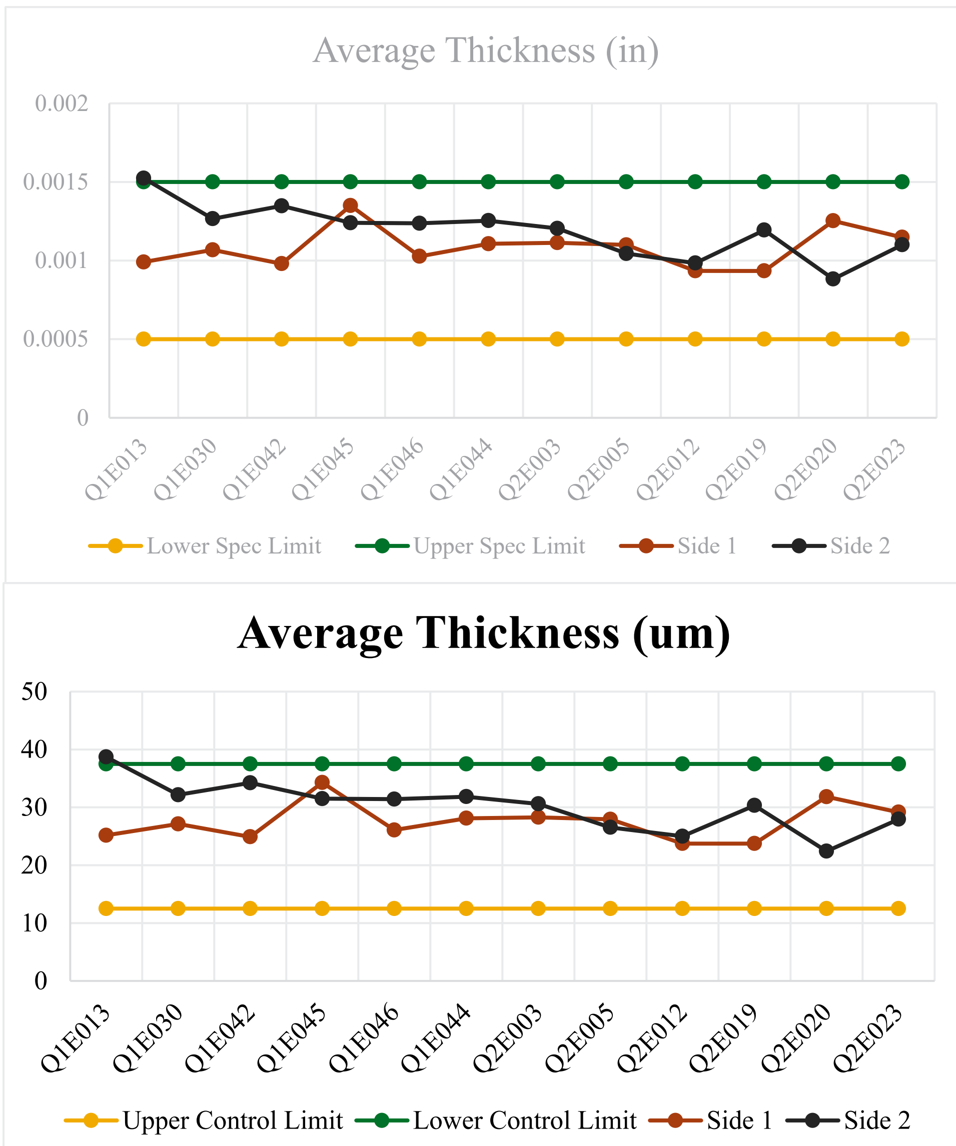

Figure 11. Optical Measurements

Table 9. DE Foil Results

Foil ID	Measured[8] (in)	Mass Gain (g)	DE Average (in)
Q1E013	0.0015	1.2656	0.0013
Q1E030	0.0012	1.2609	0.0012
Q1E042	0.0013	1.2614	0.0012
Q1E045	0.0012	1.2597	0.0013
Q1E046	0.0017	1.2797	0.0011
Q1E044	0.0011	1.2695	0.0012
Q2E003	0.0012	1.2113	0.0012
Q2E005	0.0012	1.1742	0.0011
Q2E012	0.0014	1.1629	0.0010
Q2E019	0.0013	1.1988	0.0011
Q2E020	0.0014	1.1643	0.0011
Q2E023	0.0012	1.1809	0.0011
All	0.0013	1.2241	0.0011

4.2.2 Destructive Examination Optical Images

Selected optical images are provided in Figures Figure 12 through Figure 16. These were chosen to be representative of the foils. All foil images are provided in Appendix B: Optical Images. As can be seen in the images, dark occlusions and surface defects greatly affect plating quality. It is important to note the scale of these defects as many are not visible by the naked eye. Of most concern are the cracks which appeared within the foils. As can be seen in the center section (Figure 12) of Q2E023, the surface defects typically correspond with occlusions at the surface of the foil itself. These issues may be caused by cleaning issues or scratching on the foil itself.

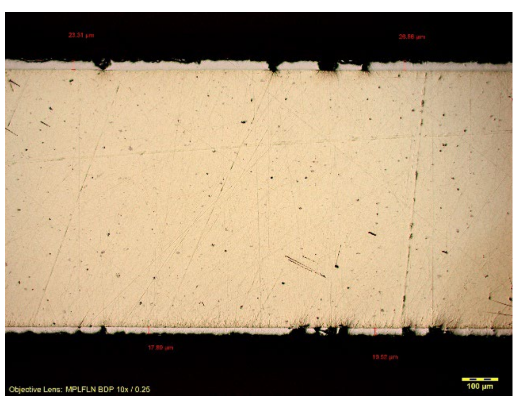

Figure 12. Center Section Q2E023

Another issue that was identified was internal cracking within the foil. This is illustrated in the Figure 13, the large crack leads to generally poor plating conditions. The plating typically followed the surface

[8] Measured by micrometer during inspection.

conditions leading to a fairly uniform coating. The burr that was found on many of the foils resulting in plating challenges (the burr was plated but under high stress leading to bonding issues).

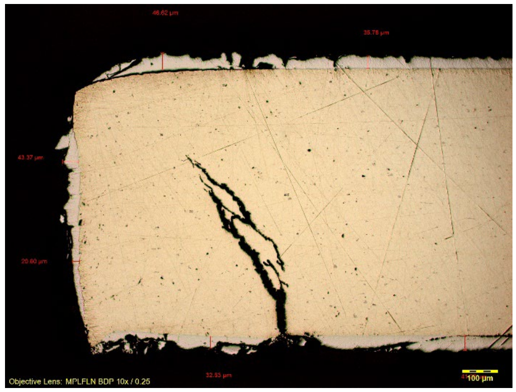

Figure 13. Cracking of Foil (Q2E020)

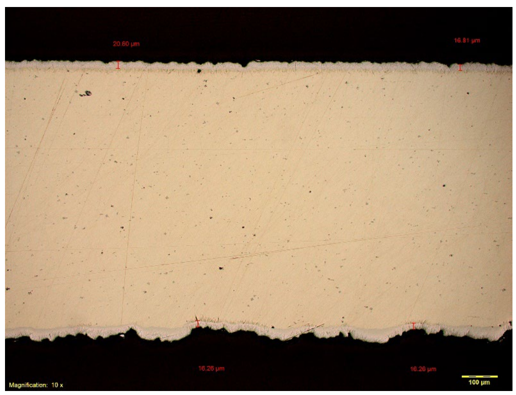

Figure 14. Q2E012 Showing Plating Tracking Surface Conditions

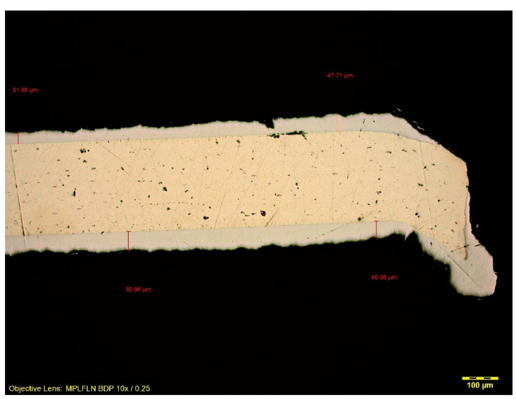

Figure 15. Q1E024 Showing Burr Effect on Plating

4.2.3 SEM Results

In the optical images there are dark occlusions in both the U-Mo base metal and Zr plating. An example of such occlusions in Foil Q1E045 is provided in Figure 16. Many foils had such dark spots. SEM was performed on this foil at the locations of some of the occlusions to determine their chemical composition for information only. SEM analysis was performed according to 67075-SOP-0010. The results are presented in Table 10 and Table 11, as well as shown in Figure 16 through Figure 19, the occlusions looked at tended to have high levels of uranium, molybdenum, with noticeable amounts of Zr, oxygen, and carbon. This indicates that the occlusions are not voids within the plating or substrate.

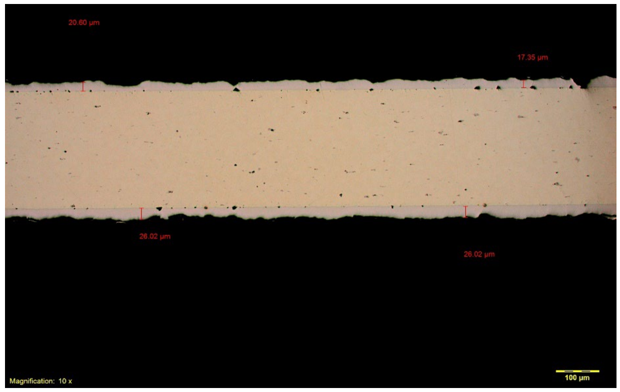

Figure 16. Optical 10x Showing Occlusions

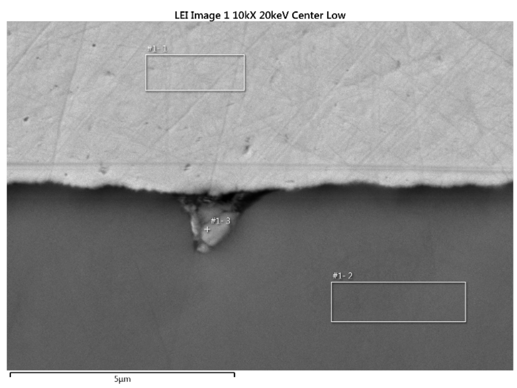

Figure 17. Optical 10x Showing Occlusions

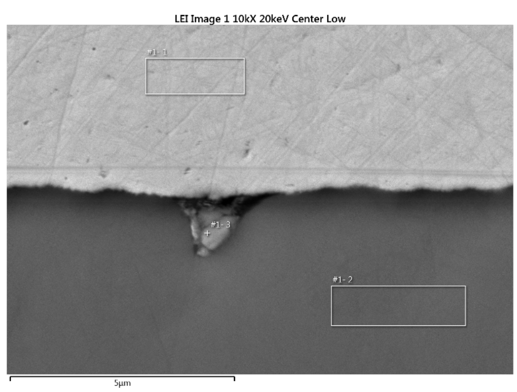

Figure 18. SEM of Foil

Table 10. SEM Results Location 1 Analysis 1

Result Type	Weight %

Spectrum Label	C	O	Zr	Mo	U	Total
#1- 1	3.30	1.52	0.00	9.17	86.01	100.00
#1- 2	0.00	5.59	94.41	0.00	0.00	100.00
#1- 3	6.36	8.51	32.21	5.58	47.35	100.00

Table 11. SEM Results Location 1 Analysis 2

Result Type	**Weight %**				
Spectrum Label	O	Zr	Mo	U	Total
#1- 1	1.56	0.00	9.54	88.91	100.00
#1- 2	5.59	94.41	0.00	0.00	100.00
#1- 3	8.98	34.48	6.02	50.52	100.00

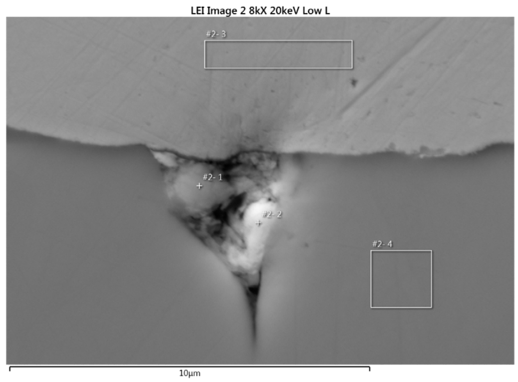

Figure 19. SEM of Occlusion

Table 12. SEM Results Location 2 Analysis 1

Result Type	**Weight %**					
Spectrum Label	C	O	Zr	Mo	U	Total
#2- 1	4.76	8.27	2.01	9.16	75.80	100.00
#2- 2	4.56	6.29	8.85	8.36	71.94	100.00
#2- 3	3.12	1.77	0.00	9.47	85.63	100.00
#2- 4	0.00	5.35	94.65	0.00	0.00	100.00

Table 13. SEM Results Location 2 Analysis 2

Result Type	**Weight %**				
Spectrum Label	O	Zr	Mo	U	Total
#2- 1	8.62	2.14	9.70	79.54	100.00
#2- 2	6.54	9.36	8.82	75.27	100.00
#2- 3	1.81	0.00	9.83	88.36	100.00
#2- 4	5.35	94.65	0.00	0.00	100.00

4.3 Visual Inspection

Three of the requirements were performed by visual inspection of the foils. These include coverage, surface condition, and the identification. Images for the some of the qualification foils are provided in Figure 20. All 12 foils pass these requirements.

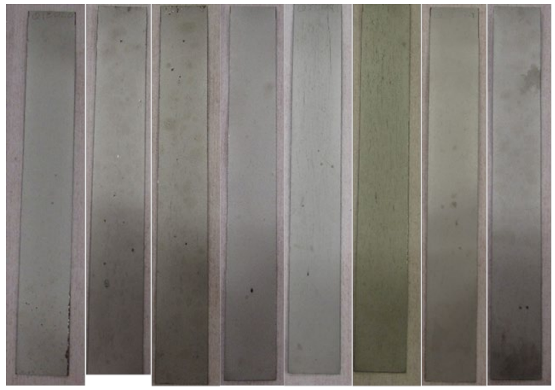

Figure 20. Foil Post-Plating Images

4.3.1 Zirconium Coverage

100% coverage of in-plane surfaces (not the edges) will be confirmed via visual inspection. Most foils achieved 100% coverage.

4.3.2 Surface Condition

The foil shall have no surface defects (voids, cracks, scratches, inclusions) having any surface dimension larger than 0.06 inch will be confirmed via visual inspection. This was the hardest of the criteria to meet. Numerous small and large defects happened. Surface defects were the most common reason why a foil failed inspection. Often the surface defects were in the base metal and propagated through the plated metal. Examples of these defects can be seen in Figure 21 through Figure 25.

Figure 21. Failed Foil (Q1E045 (top), Q1E046 (bottom)) Post-Plating Images

Figure 22. Failed Foil (Q2E010) Post-Plating Images

Figure 23. Foil Failed Foil (Q2E013) Post-Plating Images

Figure 25. Foil Failed Foil (Q1E052) Post-Plating Images

4.3.3 Identification

Identifier visibly confirmed via visual inspection. All foils passed this requirement. The vibratory tool was able to engrave the Zr without penetrating to the base metal.

5.0 Conclusions

The electroplating of Zr on U-Mo fuel meets the requirements identified in the PQP and MAQP. The process provided a Zr coating meeting the thickness and coverage requirements. The optical measurements taken from the DE examined foils indicate that the plating thickness was as expected (the target mass of about 1 gram corresponded to roughly 0.001 inch thick plating). Targeting slightly over 1 gram for the plating mass ensured that the average plating thickness was greater than the minimum required.

The main concern with releasing PNNL for production of low enriched uranium and high enriched uranium fuel is the low number of foils that pass inspection (less than 30%). While it is possible that this low net count is in part due to the poor surface quality and dimensional defects of the qualification foils, this is not known. Oxygen content of the Zr is still high, but has been trending down over time over the past several months.

6.0 Reference Documents

Fuel Specification for MP-1 and FSP-1, SPC-1691 Rev. 2, 3/15/16

FFC Quality Assurance Program Document, FFC-QA-002 Rev. 0, 6/2016

Inspecting Bare U-Mo Foils and Zirconium Electroplated U-Mo Foils, PNNL, 67075-SOP-0003 Rev 3, 3/20/2017

Certificate of Conformance, PNNL, 67075-SOP-0008, Rev 0, 9/21/2016

Control of Nonconforming Items, NQAP-IP-1501. Rev 0, 12/22/2015

Manufacturing and Quality Plan for Fabrication of MP-1 Zirconium Electroplated Foils, PNNL, 67075-MAQP-001, Rev 5, 2/1/2017

Quality Assurance Manual (NQAP), PNNL, NQAP-2012, Rev 1.0, 9/30/2016

Electroplating Zirconium onto U-Mo Foils, PNNL, 67075-SOP-0005, Rev 3 3/20/2017

Process Qualification Plan for Fabrication of MP-1 Zirconium Electroplated Foils, PNNL, 67075-PQP-0001., Rev 5, 2/1/2017

Standard Specification for Zirconium and Zirconium Alloy Sheet, Strip, and Plate for Nuclear Application, ASTM B352-11/B352M-1

Journal of Nuclear Science and Technology 22, pp. 239-241, Nakatsuka etal. (March 1985)

DEs of Zirconium Electroplated U-Mo Foils, PNNL, 67075-SOP-0010., Rev 0 , 5/19/2016

Appendix A: Zirconium Composition

EAGLE ALLOYS
CORPORATION
Global Material Solutions

178 West Park Court • Talbott • TN • 37877
Phone: (423) 586-8738 Fax: (423) 586-7456
www.eaglealloys.com

Customer:	Plasma Processes, Inc.	**Date:**	11/10/2015
PO#:	6056-003-JK-101415	**Eagle Sales Order No.:**	10128
Material:	Zirconium	**Batch No.:**	FM-BN-15100525
Size:	0.500"Thk x 2.00" x 12" Lg	**Specification:**	ASTM-B-352 UNS R60001
Quantity:	2pcs		

Certificate of Analysis

Chemical Properties:

- **Impurity Analysis:**

Impurity Content (MAX PERCENT) Zr Remainder			
Element	**Content**	**Element**	**Content**
O	0.017	Al	0.005
B	0.00004	Cd	0.00004
C	0.014	Cr	0.008
Co	0.0013	Cu	0.0012
Hf	0.006	H	0.001
Fe	0.018	Mg	0.0011
Mn	0.0013	Mo	0.002
Ni	0.005	N	0.003
Si	0.006	Sn	0.001
W	0.003	Ti	0.002
U	0.0001	Zr	balance

Mechanical Properties:

Mechanical Properties			
Direction of Test	Tensile Strength Rm (Mpa)	Yield Strength Rp0.2 (Mpa)	Elongation at Break A (%)
Longitudinal	321	163	20
Transverse	325	228	21

William Bartow QA **Dated: 11/10/2015** The liability of Eagle Alloys Corporation is limited to the cost of the materials in question.

ATI Specialty Alloys & Components

Analytical Services
1600 Old Salem Rd NE
Albany, OR 97321

Office: 541-967-6913
1-888-926-4211 ext
Fax: 541-967-6986

Report of Analysis

Report Id: B-31012017-47423V1 Total Pages: 4

February 08, 2017

Battelle Northwest Laboratories
790 6th Street
Richland, WA 99354

Order No:164381
Order Line:1

MATERIAL IDENTITY:	ZR
PURCHASE ORDER:	311490

J-270040	CUSTOMER ID:	QUAL-1
J-270041	CUSTOMER ID:	5-102516/1
J-270042	CUSTOMER ID:	5-102516/2
J-270043	CUSTOMER ID:	5-102816/1
J-270044	CUSTOMER ID:	5-102816/2
J-270045	CUSTOMER ID:	5-103116/2
J-270046	CUSTOMER ID:	5-101316/2
J-270047	CUSTOMER ID:	5-101916/1
J-270048	CUSTOMER ID:	5-101916/2
J-270049	CUSTOMER ID:	5-110216/2
J-270050	CUSTOMER ID:	5-101316/1
J-270051	CUSTOMER ID:	5-110416/1
J-270052	CUSTOMER ID:	5-110416/2

ATI Sample ID:		J-270040	J-270041	J-270042	J-270043	J-270044	J-270045	J-270046
AL ICAPQ ICP-MS.103 Original	ppm	83	<20	<20	<20	<20	<20	<20
B ICAPQ ICP-MS.103 Original	ppm	<0.25	<0.25	<0.25	0.42	1.2	<0.25	<0.25
C LECO CS-444 Original	ppm	<20	340	160	320	1280	80	40
CA SPECTRO ICP-OES.12 UNIT 2 Original	ppm	<10	10	12	52	105	<10	10
CD SPECTRO ICP-OES.12 UNIT 4 Original	ppm	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25
CO ICAPQ ICP-MS.103 Original	ppm	<10	<10	<10	<10	<10	<10	<10
CR ICAPQ ICP-MS.103 Original	ppm	<50	<50	<50	<50	<50	<50	<50
CU ICAPQ ICP-MS.103 Original	ppm	<25	135	<25	<25	<25	<25	<25
FE SPECTRO ICP-OES.12 UNIT 2 Original	ppm	<50	<50	<50	50	<50	<50	<50
H LECO RH-404 UNIT 1 Original	ppm	63	320	300	340	300	190	200
HF ICAPQ ICP-MS.103 Original	ppm	<25	<25	<25	<25	<25	<25	<25

ATI Specialty Alloys & Components

Analytical Services
1600 Old Salem Rd NE
Albany, OR 97321

Office: 541-967-6913
1-888-926-4211 ext
Fax: 541-967-6986

Report of Analysis

Report Id: B-29032017-52230V1 Total Pages: 3

March 30, 2017

Battelle Northwest Laboratories
790 6th Street
Richland, WA 99354

Order No:164844
Order Line:3

MATERIAL IDENTITY:	ZR
PURCHASE ORDER:	311490

J-270916	CUSTOMER ID:	Q1E054
J-270916	LAB COMMENT:	N check out of range high, results reported for information due to insufficient sample for rerun.

ATI Sample ID:		J-270916
AL ICAPQ ICP-MS.103 Original	ppm	84
AL ICAPQ ICP-MS.103 Rerun	ppm	89
B ICAPQ ICP-MS.103 Original	ppm	<0.25
B ICAPQ ICP-MS.103 Rerun	ppm	<0.25
C LECO CS-444 Original	ppm	<20
C LECO CS-444 Rerun	ppm	<20
CA SPECTRO ICP-OES.12 UNIT 4 Original	ppm	<10
CA SPECTRO ICP-OES.12 UNIT 4 Rerun	ppm	<10
CD SPECTRO ICP-OES.12 UNIT 4 Additional Request	ppm	<0.25
CD SPECTRO ICP-OES.12 UNIT 4 Rerun	ppm	<0.25
CO ICAPQ ICP-MS.103 Original	ppm	<10
CO ICAPQ ICP-MS.103 Rerun	ppm	<10
CR ICAPQ ICP-MS.103 Original	ppm	<50
CR ICAPQ ICP-MS.103 Rerun	ppm	<50
CU ICAPQ ICP-MS.103 Original	ppm	<25

Appendix B: Optical Images


2 mm
Objective Lens: MPLFLN BDP 10x / 0.25
2 mm
Objective Lens: MPLFLN BDP 10x / 0.25
2 mm
Objective Lens: MPLFLN BDP 10x / 0.25
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2 mm
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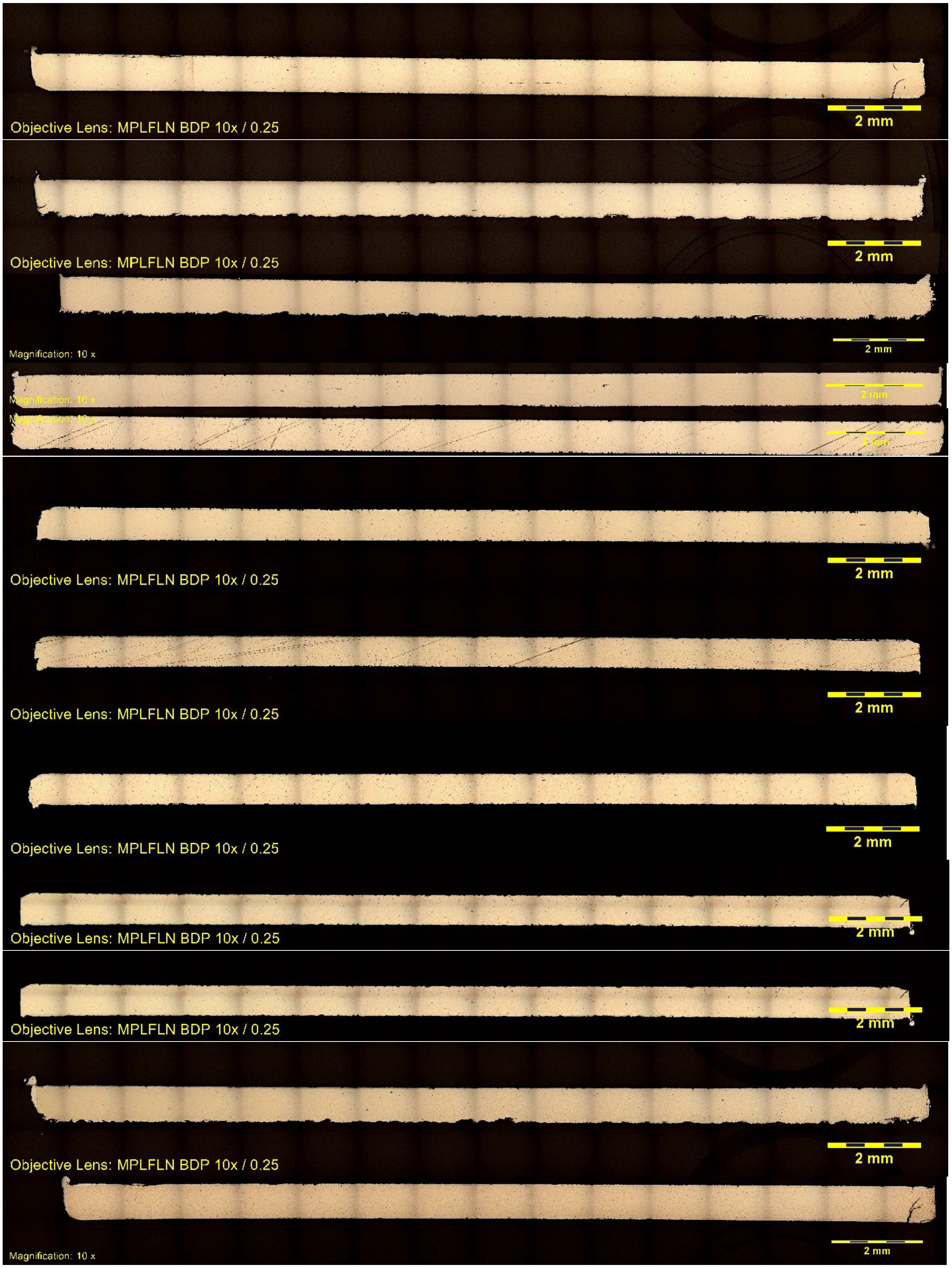
Objective Lens: MPLFLN BDP 10x / 0.25
2 mm
Objective Lens: MPLFLN BDP 10x / 0.25
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Magnification: 10 x
2 mm
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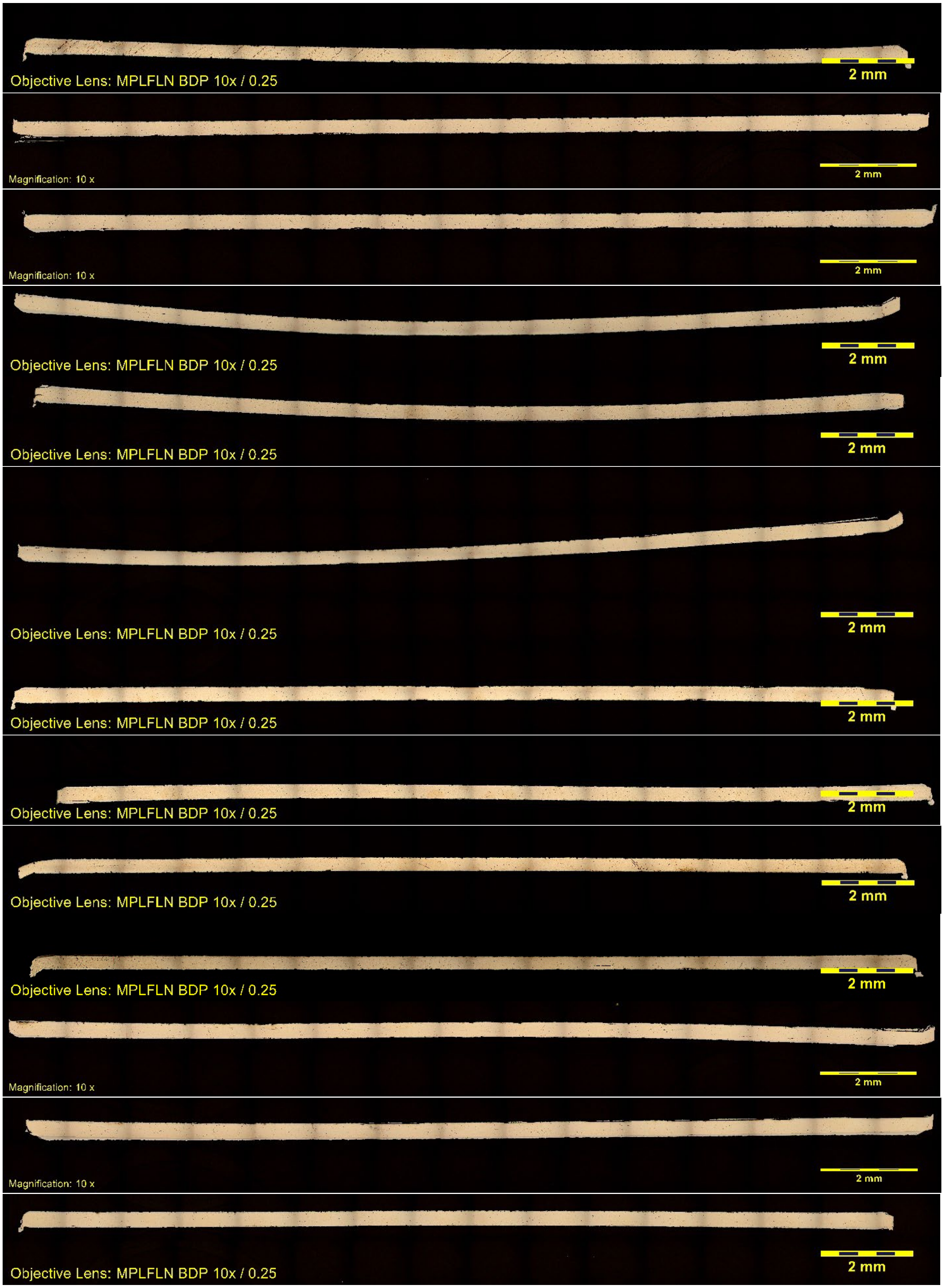
Objective Lens: MPLFLN BDP 10x / 0.25
2 mm
Magnification: 10 x
2 mm


Objective Lens: MPLFLN BDP 10x / 0.25
2 mm
Magnification: 10 x
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Magnification: 10 x
2 mm
Objective Lens: MPLFLN BDP 10x / 0.25
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Objective Lens: MPLFLN BDP 10x / 0.25
2 mm
Magnification: 10 x
2 mm
Magnification: 10 x
2 mm
Objective Lens: MPLFLN BDP 10x / 0.25
2 mm


Magnification: 10 x
2 mm
Objective Description: MPLFLN BDP 10x / 0.25
2 mm
Magnification: 10 x
2 mm
Magnification: 10 x
2 mm

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