

Electropolishing of a Full-Sized U-10Mo Plate

December 2022

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Abstract

Electropolishing is used to remove material from the surface of a metal using electric potential and current. U-10Mo fuel is produced from a low enriched uranium plate alloyed with 10% molybdenum (U-10Mo). Electropolishing is being considered in two steps of the process of fabricating this U-10Mo fuel. First, it could be used as a means initially improving the surface finish of the cast plate while removing smut from the casting process from the surface prior to homogenization. Second, it could be used again after homogenization to remove oxidation and continue to improve the surface finish prior to hot rolling. The purpose of this work is to demonstrate the process on full-size ingots and optimize the process. A depleted uranium U-10Mo plate was electropolished three times: the first two times were prior to homogenization and the third after a homogenization step. The first two polishes decreased the overall roughness, as measured by laser confocal microscopy. After each polish, a smoother and shinier blue-gold surface was left on the plate. Upon homogenization, the surface had a more matte appearance and the surface roughness increased back to the pre-homogenization values obtained prior to the initial polish. After electropolishing the homogenized pieces, the changes in surface roughness increased, as measured by laser confocal microscopy. The final polishing step was not long enough to return the surface roughness to pre-homogenization conditions, implying less polishing is needed before the homogenization versus afterwards. Heat generation during the polish was addressed by circulating the electrolyte solution through an external heat exchanger; however, solubility and anodization challenges require further study.

Summary

Electropolishing is being considered in the fabrication process for high-assay, low-enriched uranium with a 10% molybdenum alloy (U-10Mo) for the U.S. High Performance Research Reactor Program. This program seeks to convert the five US high flux research reactors from high enriched uranium (HEU) fuel to low enriched uranium (LEU). After cast and heat treatments, excessive oxide and smut form on the surface that require removal prior to further processing. Cleaning of uranium (U) metal using nitric acid is a well understood method for removal of oxide and commonly used. However, the uranium metal surface quickly re-oxidizes afterwards and with no improvement to the metal surface finish. An alternative cleaning method being evaluated for the program is electropolishing. Electropolishing has demonstrated significant improvement in surface finish with smaller U-Mo samples showing no re-oxidation after >30 days. However, prior to being considered for implementation, this process requires demonstration on a full-sized ingot. The purpose of this study is to perform that demonstration and optimize the process.

Two depleted uranium U-10Mo (DU-10Mo) plates were cast for this electropolishing study. A series of two, 10-minute electropolishes were carried out on one of the cast plates, followed by homogenization and one final, 10-minute electropolish. The plate was analyzed visually and via laser confocal microscopy between polishes. The results were compared to the other cast DU-10Mo plate etched in nitric acid.

The first polish was carried out on the entire plate for 10 minutes at 5.0 A/in². A blue-black film was formed and remained on the plate prior to the final rinse. Rinsing with deionized water and ethanol easily removed this film prior to laser confocal microscopy (LCM) and further polishing. The surface roughness decreased, and the plate turned a shinier, blue-gold color. It is expected that during this process uranium is preferentially removed leaving behind a blue-gold molybdenum layer. Overall, the surface quality, defined by a reduction in surface roughness, improved following the first polish. The second polish was performed on the bottom half of the plate for 10 minutes at 5.2 A/in² and with similar results. The same, blue-black film was formed during the polish and following the final rinse, the plate was a shinier, blue-gold color. The surface roughness further decreased as a result of this second electropolishing step. A dilute sulfuric solution (0.2M) successfully washed the film off the plate.

Oxidation of the DU-10Mo cast plate during the homogenization process increased the surface roughness of the plate, diminishing gains from the additional polishing step. The post-homogenization electropolish was performed on the bottom of the plate using a current density of 5.2 A/in² using the same 10-minute polishing period. The same blue-black film was formed during the polish. Once the film was rinsed off, a blue sheen remained on the surface and did not re-oxidize even after 9 months. However, the surface was still rougher than the previous step, although lower than the nitric acid cleaning test. The plate was analyzed via laser confocal microscopy again one week later and found the surface quality did not change. This study taught us that electropolishing reduces impacts of surface damage caused from re-oxidation. It also taught us that less electropolishing is required before homogenization and more is required afterwards.

Scaling of the system to a full-size casting found issues not observed from previous testing. While heat generation observed at this scale was easily solved with heat transfer equipment, issues with hydrated film formation on the plate and excessive anodization still need to be addressed.

Acknowledgments

This work was funded by the U.S. Department of Energy and the National Nuclear Security Administration's Office of Material Management and Minimization and performed at Pacific Northwest National Laboratory under contract DE-AC05-76RL01830.

Acronyms and Abbreviations

AFM	Atomic force microscopy
C-DIC	C-laser differential interference contrast
DI	deionized
DU-10Mo	depleted uranium alloyed with 10% molybdenum
HEU	high-enriched uranium
H ₂ SO ₄	sulfuric acid
HNO ₃	nitric acid
LCM	laser confocal microscopy
LEU	low-enriched uranium
LEU-10Mo	low-enriched uranium alloyed with 10% molybdenum
U	uranium
U-10Mo	uranium alloyed with 10% molybdenum
VIM	vacuum induction melter

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1.0 Introduction

1.1 Background

Electropolishing is being considered in the fabrication process for high-assay, low-enriched uranium with a 10 wt% molybdenum alloy (U-10Mo) for the U.S. High Performance Research Reactor Program. This program seeks to convert the five US high flux research reactors from high enriched uranium (HEU) fuel to low enriched uranium (LEU). Through extensive research, it was decided that low-enriched U-10Mo (LEU-10Mo) was the ideal fuel replacement candidate due to its high intrinsic uranium density and good irradiation performance, swelling response, mechanical properties, and corrosion resistance. The fuel itself consists of a LEU-10Mo foil with a zirconium interlayer and aluminum cladding. This is produced by first casting the LEU-10Mo ingot, binding the zirconium foil to the LEU-10Mo ingot through hot rolling, and then applying the cladding through hot isostatic pressing. During hot processing methods, the uranium metal surface can become oxidized that requires its removal. Cleaning before heat-treated is performed to primarily to protect annealing furnaces while cleaning afterwards is performed to improve surface finish prior rolling operations.

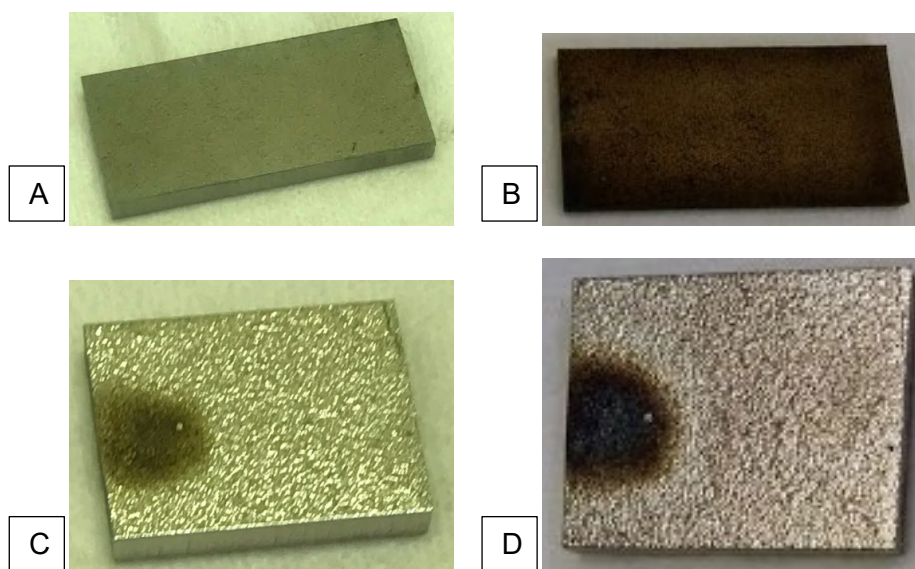
Cleaning with nitric acid (HNO_3) removes oxide and surface smut from uranium (U) metal surfaces effectively and well understood, but it leaves an active surface that re-oxidizes rapidly in air. This is due to how HNO_3 and water both react with the metal surface to convert metal uranium to an oxide prior to the HNO_3 dissolving the oxide on the surface (Katz & Rabinovitch, 1951). This oxidation is accelerated during heat treatment and requires a separate step for the oxidation/smut on the surface to be removed. To minimize re-oxidation of the LEU-10Mo prior hot-working the material, the homogenized plate is cleaned just prior to rolling with minimal water rinsing (Kasper et al. 2018).

PNNL is investigating electropolishing as an alternative cleaning method for LEU-10Mo to help reduce re-oxidation concerns and improve the surface finish of cast plates. Initial studies were performed cleaning 1-inch square coupons of depleted U-10Mo (DU-10Mo) by both nitric acid dissolution and electropolishing with 30 wt% sulfuric acid (H_2SO_4). These studies found a reduction in the re-oxidation rate in the electropolish samples along with improve surface finish (Kasper et al. 2019) as measured with atomic force microscopy (AFM). This study was followed with electropolishing 1-in square coupons from LEU-10Mo center strip using both H_2SO_4 and HNO_3 to evaluate surface finish and re-oxidation (Shimskey et al. 2019). Improved surface finish was seen on the LEU-10Mo samples electropolished in 30 wt% sulfuric as well as a significant decrease in the oxidation rate with no visible sign of re-oxidation after 30 days of being kept dry (Figure 1). While the resulting surfaces were promising, some issues were discovered that implied additional effort would be needed for understanding the system scale-up for larger plates:

- Anodization of surface was visible at the location of where the electrode contact was made (Figure 1c) but could be minimized by how the potential of the power supply was ramped up and down.
- Nickel and platinum plated electrodes were utilized for the testing and both experienced corrosion and found as contamination in the electrolyte solutions, including copper and iron (nickel coated screw on the anode) and copper (underneath the platinum coated cathode).

- Precipitation was observed in the spent sulfuric solutions. The solubility of uranium is rather low in sulfuric acid (Susuki et al. 1990) and inversely proportional to the sulfate ion. As metal dissolution increases with part size, more careful management of the uranium concentration is required.
- Samples were relatively small compared to the electrolyte solution, so heat generation could not be measured.

For these and many other reasons, it became apparent that prior to being considered for implementation, this process requires demonstration on a full-sized ingot and optimization of the electropolishing parameters.



- A) LEU-10Mo ingot directly following electropolishing in 30% HNO_3 .
 B) Sample from A) **following 17 days of storage** in plastic bag in fume hood.
 C) LEU-10Mo ingot directly following electropolishing in 30% H_2SO_4 .
 D) Sample from C) **following 30 days of storage** in plastic bag in fume hood.

Figure 1. Images from electropolishing LEU-10Mo Center Foils (Shimskey et al. 2019)

1.2 Goals of Study and Evaluating Criteria of Polishing

The purpose of this study is to demonstrate the electropolishing process on a full-sized U-10Mo ingot and optimize the process. In this report, a cast DU-10Mo plate is electropolished before and after homogenization and compared with the surface achieved by cleaning another cast DU-10Mo plate with nitric acid instead, as well as results from small-scale testing. There are two areas that are focused on to evaluate the effectiveness of the electropolishing process and how it compares with nitric acid cleaning:

- **Visual Appearance**

- *Description:* While visual appearance is frequently called a cosmetic attribute to cleanliness, the statement “*If it doesn’t look clean, it isn’t clean*” is to evaluate a cleaning/finishing process. Surfaces of cleaned material are expected to be free of smut, manufacturing debris, oil and grease, and uniform in appearance. Visual standards are commonly used for this when other quantification methods are not available. When the cleaned surface does not appear uniform, it can be an indicator of other issues such as irregular oxide growth, surface contamination, or an issue with the finishing operation are present (e.g., abrasive belt wearing out, insufficient rinse).
- *Quantification Method:* The visual appearance before and after cleaning is discussed, documenting this with digital pictures and how the surface oxidation changes over time. Close-up examination of the surface is performed with the Keyence VT laser confocal microscope (LCM) which can capture C-laser differential interference contrast (C-DIC) images at different magnifications to observe changes in surface topography.

- **Surface Roughness**

- *Description:* Surface roughness is the measurement of the surface texture of a material that examines the total spaced irregularities on the surface by measuring the height difference of these irregularities using either a surface profilometer with a contact stylus or using an optical surface method like a laser confocal microscope. This technique is useful to quantify if a surface is considered “rough” or “smooth” for a material. It is also used to measure effectiveness of a finishing process and variability.
- *Quantification Method:* Surface roughness measurements are performed using a Keyence VT laser confocal microscope which measures the surface roughness while imaging the surface. Measurement of samples are performed before and after cleaning to evaluate the change in surface roughness and provide a close-up visual of the surface for comparison. Ra and Rz (average and maximum differences found across a linear span) were measured before and after cleaning/finishing steps, as well as Sa and Sz (average and maximum differences found across an area region).

The subsequent sections will describe how well this process is scaled-up and any issues not experienced during previous testing.

2.0 Materials, Equipment, and Methodology

2.1 Cast Depleted U-10Mo Plates for Electropolishing Study

Depleted uranium with 10% molybdenum alloy (DU-10Mo) plate was cast in a vacuum induction melter (VIM), loaded into a zirconia crucible, and was heated to 1400°C, as described in Huber et al. (2020). Once the metal was molten, it was poured into an yttrium oxide coated graphite mold to produce a cast plate with nominal dimension 7.5 in x 3.6 in x 0.2 in (Length x Width x Depth) once the hot top was removed. An example of a DU-10Mo casting used for this work is shown in Figure 2. Once the working electrode connection was made at the top of the plate, the maximum length that the plate could be polished was 6.7 in of length from the bottom of the cast plate as shown in in Figure 3.



Figure 2. DU-10Mo cast plate with hot top present



Figure 3. DU-10Mo cast plate with hot top removed and attached to working electrode connection prior to electropolishing

To prepare the plate for polishing, it was weighed, and the dimensions were determined based on the intended depth of the sample in the acid solution. The surface area was calculated based on these dimensions and then was utilized to determine the amperage to achieve a target current density for the surface (amp per square inch). Each plate was analyzed via LCM at the 5x and 10x magnification to measure the surface roughness as well as C-DIC images to show physical changes on the surface. Imaging and surface roughness measurements for the cast plate used for electropolishing testing are shown in Figure 4 and Figure 5.

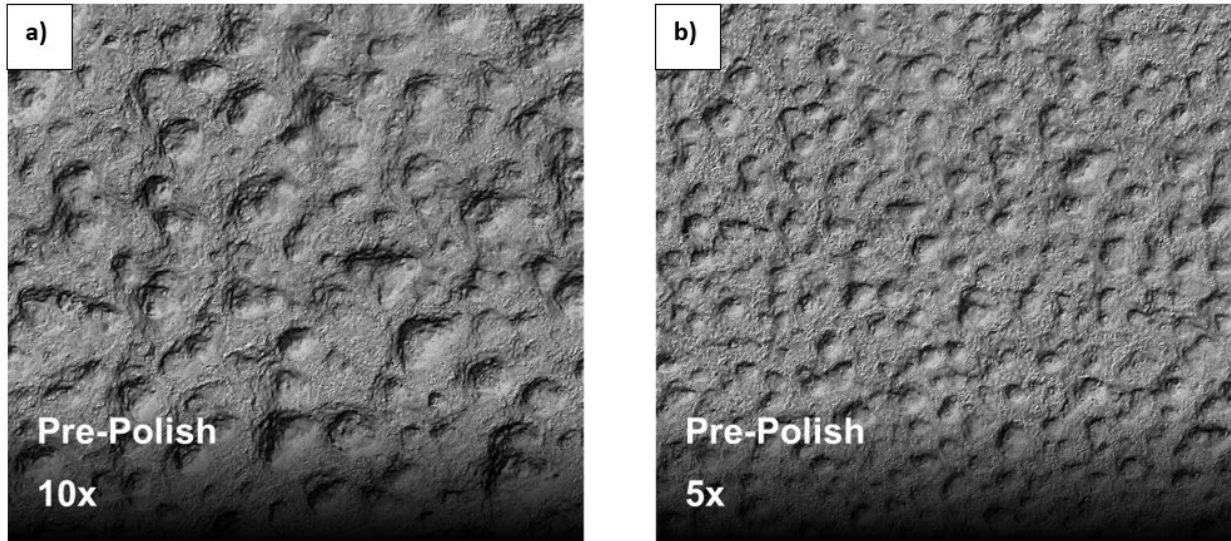


Figure 4. The a) 10x magnification and b) 5x magnification C-DIC images of the Bottom Left section of Side 1 of the DU-10Mo casting used for electropolishing

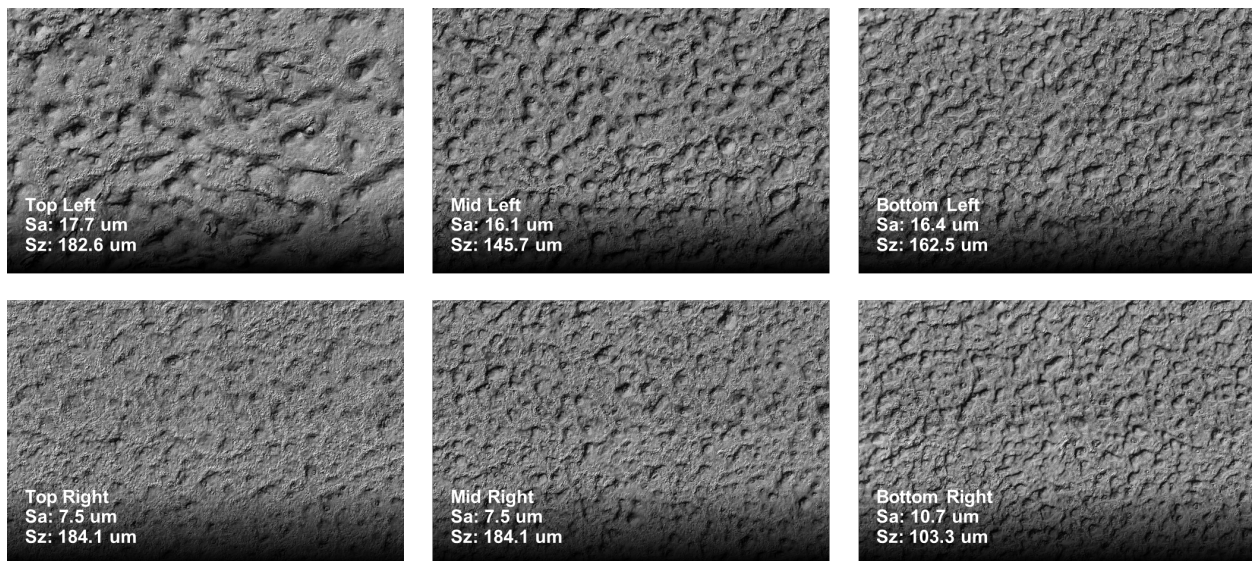


Figure 5. C-DIC images (5x magnification) of Side 1 of the DU-10Mo casting used for electropolishing with correlating surface roughness measurements (Sa and Sz)

2.2 Nitric Acid Cleaning Test Equipment and Method

2.2.1 Nitric Acid Cleaning Equipment

Nitric acid cleaning of DU-10Mo occurred in a 10" Diameter Pyrex container (Figure 6) using 1-L of solution (6M HNO₃) prepared with reagent grade chemicals and deionized (DI) water, along with stainless steel tongs used to hold the plate and spray bottles filled with DI water to rinse. Work occurred within a radiological fume hood.

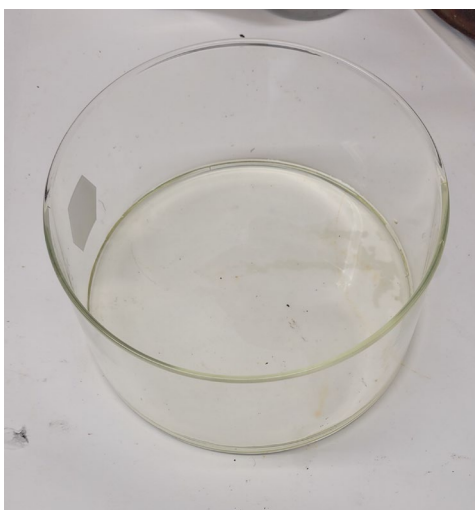


Figure 6. Pyrex pan (10" Diameter) Used for etching DU10Mo cast plate in 6M Nitric Acid

2.2.2 Nitric Acid Cleaning Method

The work occurred by manually immersing the cast plate into the solution at room temperature for 10 minutes. The cast plate was moved using tongs during etching and was flipped completely after 5 minutes. Spray bottles with DI water were used to afterward to rinse the cast plate as it left the solution, and it was patted dry afterwards.

Table 1. Nitric acid cleaning parameters for the DU10Mo cast plate

Parameter	Value
Plate Dimensions	7.5 in x 3.6 in x 0.2 in
Plate Surface Area	58 in ²
Sample Depth	Full
Polishing Area	58 in ²
Acid Type	HNO ₃
Acid Concentration	30 wt% (6 M) HNO ₃
Acid Volume	1L
Target Temperature	Room Temperature
Time Period	10 minutes

2.3 Electropolishing Test Equipment and Method

2.3.1 Electropolishing Test Equipment

There are four main components of the electropolishing system (Figure 7) required to electropolish a metal or alloy:

- Polishing vessel containing electrolyte solution
- DC Power Supply
- Anode (Working Piece)
- Cathode (Platinum Mesh Electrode)

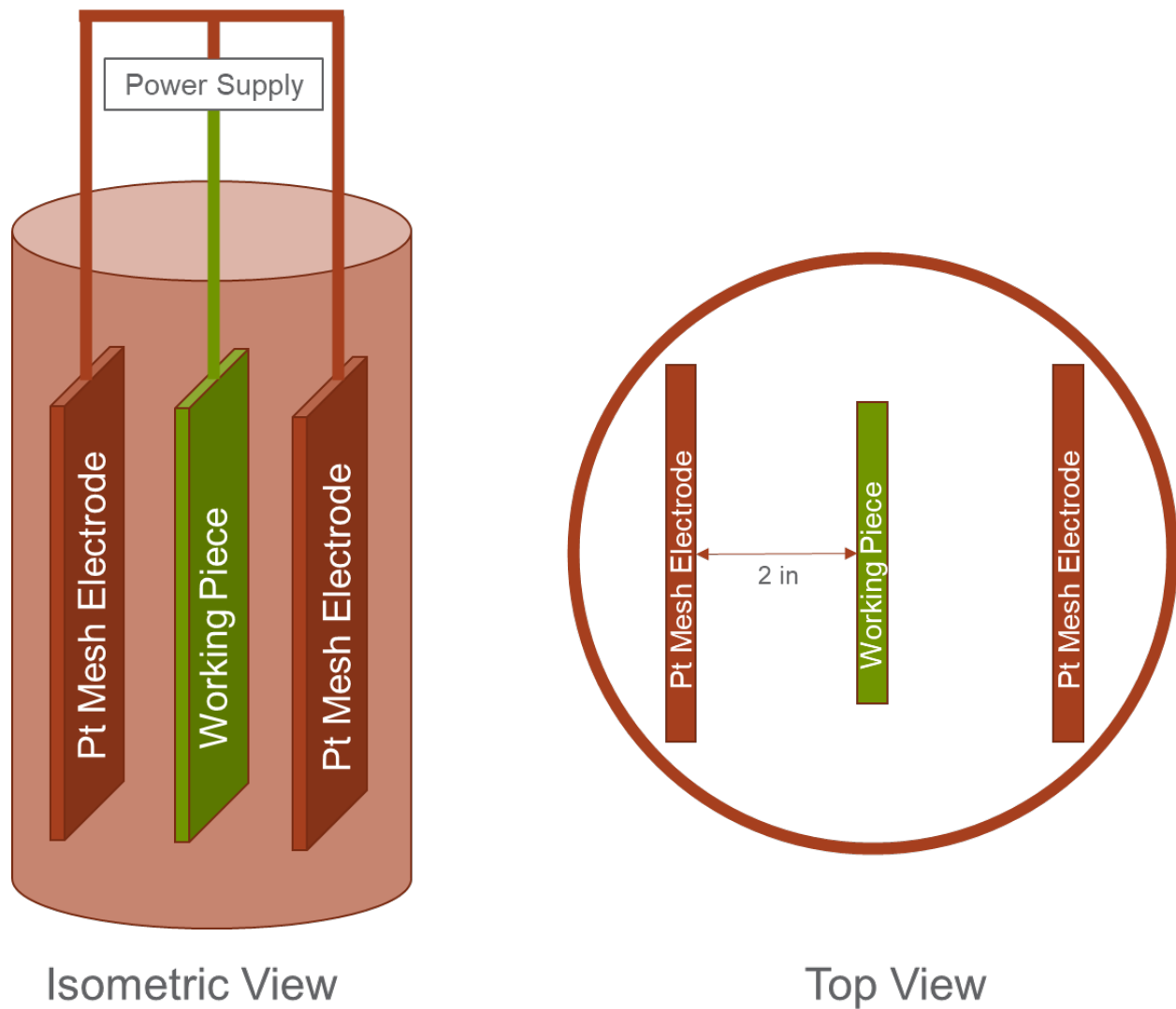


Figure 7. The electropolishing setup for the U-10Mo plate (working anode) and the platinum mesh (cathode)

A custom PTFE cylindrical tank (Figure 8) designed to hold up to 3-L of working electrolyte with the test piece was used as the polishing vessel inside a radiological fume hood. The TDK-Lambda DC power supply (Figure 9) was connected to both the working piece (DU-10Mo cast) and platinum coated mesh electrodes using 2-gauge copper wire. A copper clamp (Figure 10) was fabricated to connect to the top of the DU-10Mo, which is connected to the positive terminal of the power supply. Two custom fabricated niobium coated platinum mesh electrodes (Figure 11) were placed on opposite sides of the DU-10Mo (Figure 7) and connected to the negative terminal of the power supply to function as the cathode (tool electrode). Two PTFE screens separate the working electrode and the two platinum mesh electrodes to prevent contact while the system is energized. The electrolyte solution used was sulfuric acid at a nominal 15 wt% concentration that was made with reagent grade chemical and DI water.

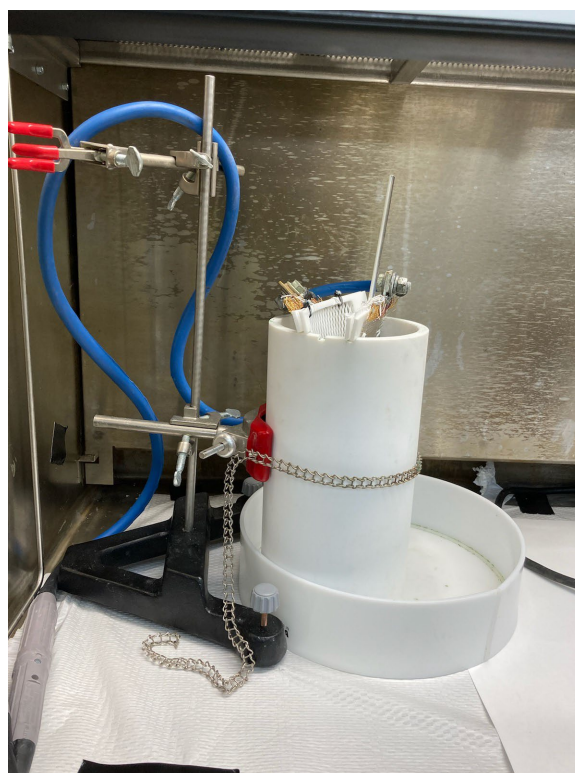


Figure 8. The electropolishing setup containing the polishing vessel and support stand



Figure 9. TDK-Lambda Gen 10-500 DC Power Supply used for Electropolishing



Figure 10. Fabricated Anode Work Piece Connection

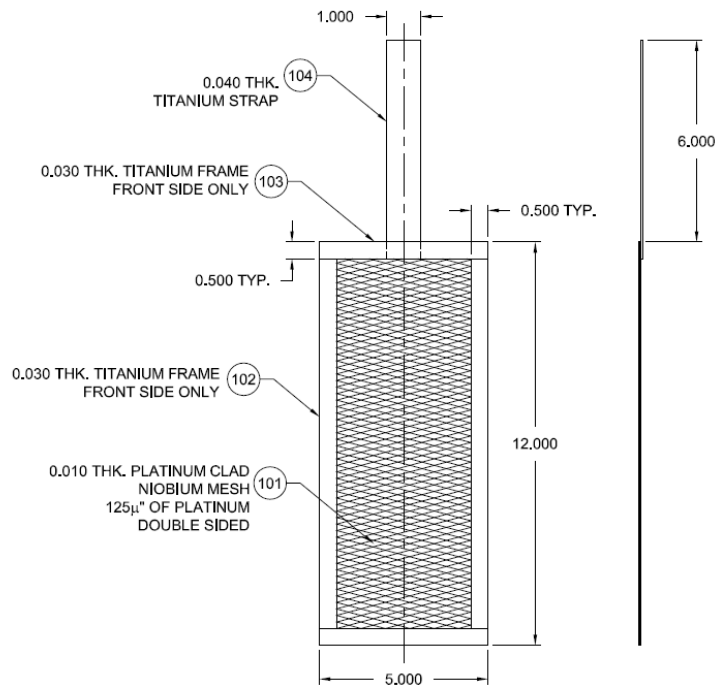
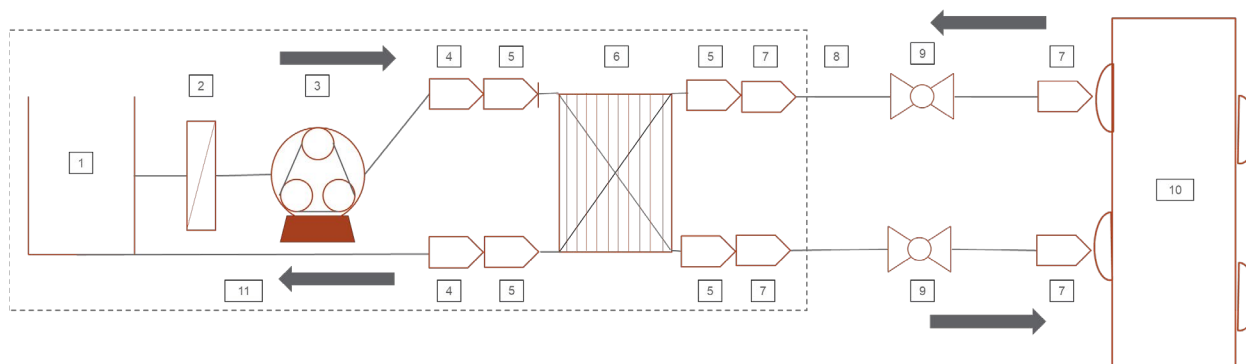


Figure 11. Custom Fabricated Platinum Coated Niobium Mesh Cathode

Initial shakedown testing found significant generation of heat produced from this process electropolishing DU-10Mo roughly 20 times larger than was previously tested. To maintain the temperature between 25-35°C, an external cooling circulating system was added to the system (Figure 12 and Table 2). The polishing vessel was connected to a variable drive peristaltic pump to circulate the sulfuric acid solution through a two-pass stainless steel heat exchanger. Cooling water flowing through a closed loop external chiller flowed through the other side of the heat exchanger and removed heat effectively from the electrolyte solution.



Note: The components contained within the dashed line are found in the fume hood contamination area

Figure 12. The electropolishing solution cooling system diagram

Table 2. The electropolishing solution cooling system parts list

Part #	Description
1	Fabricated Teflon Electropolishing Vessel (3L of 15% H ₂ SO ₄ Solution)
2	¼" Inline Filter
3	Masterflex L/S Peristaltic Tube Pump with Analog Variable Speed Drive
4	½" NPT Male x 3/8" Compression Fitting
5	3/8" NPT Female x 3/8" Compression Fitting
6	Bell & Gossett Stainless Steel Heat Exchanger (BPN400-10 LCA); 60,000 BTU/HR
7	3/8" NPT Male x 3/8" Barbed Tube Fitting
8	Tygon Masterflex L/S Precision 18 Tubing (Tubing ID 0.31")
9	3/8" OD Reinforced PVC Tubing
10	MTI Recirculating Water Chiller (MTI KJ-53000); 7,000 BTU/HR.
11	0.75" OD PTFE Tubing

2.3.2 Electropolishing Testing and Operating Parameters

Three electropolishing tests were performed on a single DU-10Mo cast plate as outline in Figure 13 and tested according to the test parameters listed in Table 3.

- Cast plate characterization.** After the DU-10Mo plate was cast, the initial dimensions and mass were measured for surface area calculations and net mass losses. The surface is also characterized by LCM to image the surface topography with C-DIC and measure the initial surface roughness across the plate.
- Electropolishing of the cast plate.** Using the parameters listed in Table 3, the cast plate was immersed into the electrolyte solution as far as possible (6.7-inch immersion) and electropolished. The net mass change of the plate after the electropolish was measured and the changes in surface topography and surface roughness were measured afterwards to a measured applied voltage and current density. Temperature changes were also measured to evaluate the effectiveness of the cooling system.

- **Repeat electropolishing of the cast plate.** Once the electropolished plate was characterized, a repeat electropolish of the plate, using the parameters listed in Table 3, was performed to see if improved performance would occur. The plate was only immersed 3.75 inches to evaluate how the change in surface area compared to tank volume would impact heat generation and polishing performance.
- **Homogenization anneal of electropolished cast plate and follow-on characterization.** After the second polishing run, the polished cast DU-10Mo plate was wrapped in zirconium foil and placed into a vacuum furnace. The furnace was pulled to high vacuum before heating to 900°C at a rate of 10°C/min. The furnace was held at 900°C for 144 hours under vacuum before cooling to room temperature naturally and opening the furnace. The annealed DU-10Mo plate was analyzed via LCM for surface imaging and surface roughness.
- **Electropolishing of the homogenized plate.** Using the parameters listed in Table 3, the cast plate was immersed into the electrolyte solution for 3.75-inch immersion and electropolished. The net mass change of the plate after the electropolish was again measured and the changes in surface topography and surface roughness were measured afterwards based on the applied voltage and current density. Temperature changes were also measured to evaluate the effectiveness of the cooling system.

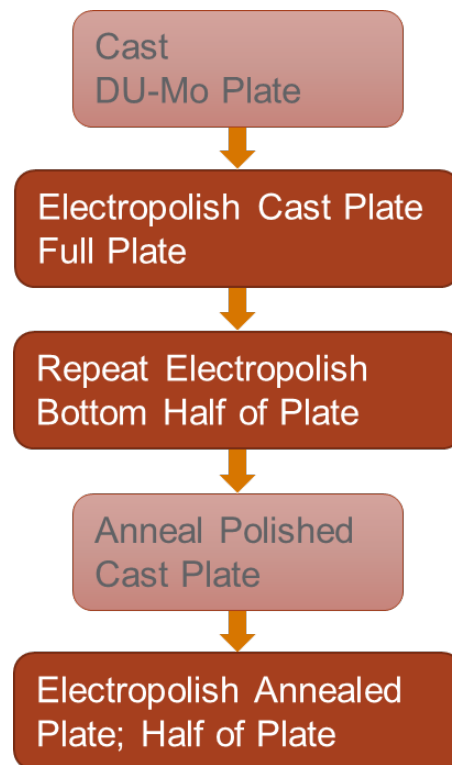


Figure 13. Testing order of the electropolishing of cast DU-10Mo plate before and after homogenization anneal

Table 3. Electropolishing test parameters

Parameter	Value
Plate Mass	1.3-1.5 kg
Plate Dimensions	7.5 in x 3.6 in x 0.2 in
Plate Surface Area	58 in ²
Sample Depth	3.75-6.7 in
Polishing Area	29-52 in ²
Target Current Density	4-6 A/in ²
Ramp Up Time	01:00 min
Hold Time	10:00 min
Ramp Down Time	01:00 min
Acid Type	H ₂ SO ₄
Acid Concentration	15 wt% (1.7 M)
Acid Volume	3 L
Distance between Anode Mesh	2 in
Temperature	15-35°C

3.0 Results and Discussion

3.1 Nitric Acid Cleaning Test Results

A DU-10Mo plate was cast and analyzed via LCM before and after cleaning with 6M HNO₃. Images of the plate after cleaning are shown in Figure 14. The one side was allowed to sit on a wet towel as it dried causing increased oxidation of that side of the plate (Figure 14b). No significant difference was found in the surface roughness of the plate before and after cleaning (Table 4). However, comparison of C-DIC images (Figure 15) of the before and after cleaned surfaces show visible pitting across the surface of the cleaned plate like that seen in the small-scale testing by AFM (Kasper et al. 2019).

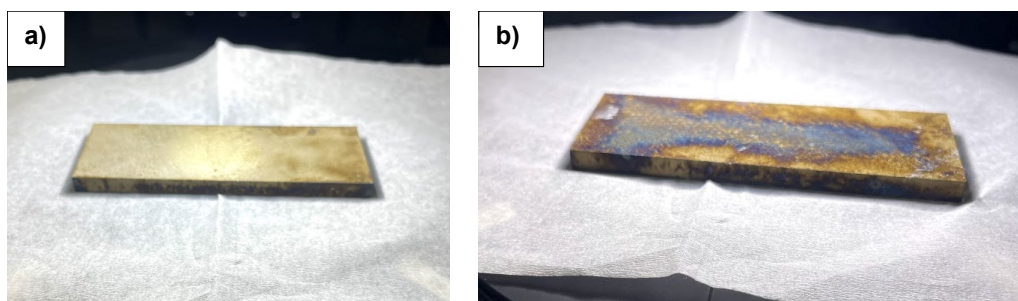


Figure 14. The U-10Mo plate after etching. Image a) shows Side A and Image b) shows Side B. Side B shows significant irregularity in color across the surface resulting from the bottom sitting on a wet towel

Table 4. The surface roughness values before and after etching in 6M HNO₃

Sample	Ra (μm)	Rz (μm)	Sa (μm)	Sz (μm)
Pre-Etch	29.2 (\pm 7.2)	146.3 (\pm 30.5)	30.6	245.4
Post-Etch Side A	23.2 (\pm 5.8)	129.8 (\pm 27.6)	22.5	198.8
Post-Etch Side B	32.9 (\pm 7.2)	155.3 (\pm 31.5)	33.5	252.8

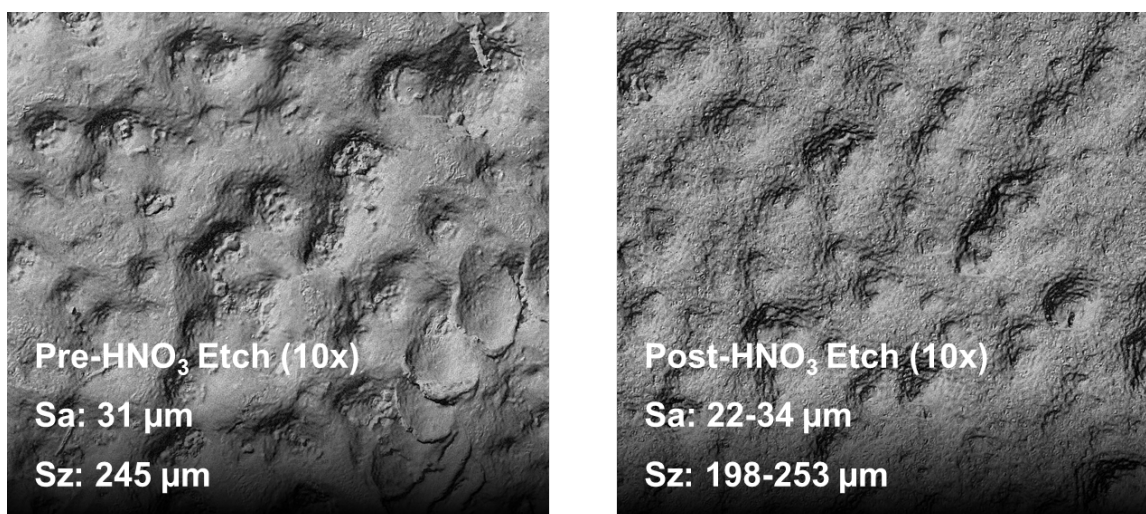


Figure 15. C-DIC Images of DU-10Mo cast surface before and after 6M HNO₃ cleaning.

3.2 Electropolishing Polishing Test of Cast Plate Results

3.2.1 Initial Polishing of Cast Plate (Full Plate)

The first electropolish was ran using the test parameters listed in Table 5 and images of the plate before and after polishing are found in Figure 16. The acid solution turned blue after 3 minutes, and a blue/black film formed on the plate during the polishing process (Figure 16b). While a dilute blue water film was observed on the surface during small scale polishing, this type of film was not experienced with small scale testing or considered an issue (Shimskey et al. 2019).

The substance found on the surface is likely uranium sulfate, as U^{4+} has low solubility in H_2SO_4 (Susuki et al. 1990). This compound has a blue/green tint similar in color to this film on the surface of the plate following polishing. While this film was not seen in the small-scale studies, only mass loss of 1-2 g was experienced for those tests for a similar working solution volume (2-L) while a mass loss of 55 g was seen in this test (3-L). Also, the small-scale testing experience more localized agitation from gas generation from the platinum electrodes. On the large-scale system, plastic mesh was inserted between the working pieces and the platinum electrodes to prevent contact, but it also kept any mixing from the generation of gas away from the working electrode. This combined lack of mixing and issues with solubility likely cause a localized concentration of uranium to occur on the surface of the plate which exceeded the solubility of uranium sulfate and formed this film.

Once the plate was removed from the acid solution, the film was rinsed off with deionized water, and rinsed again with ethanol, and allowed to dry. After rinsing, the surface was free of smut and oxide with a bare metal surface. However, the metal surface finish was tinted with both blue and gold colors (Figure 16c and Figure 16d). This implied that the surface was either re-oxidized with water during the removal of the film with water, or the surface was anodized with a stable oxide from the applied voltage similar to what was seen where the electrode contact was made on the small-scale test pieces shown in Figure 1c. Over time, the surface of the plate did not re-oxidize like the nitric acid cleaning, so the high voltage experienced during the test (7.6 V) likely caused anodization. Reducing the distance between the working and tool electrodes as well as finding better methods to connect to the working piece would reduce the total voltage applied.

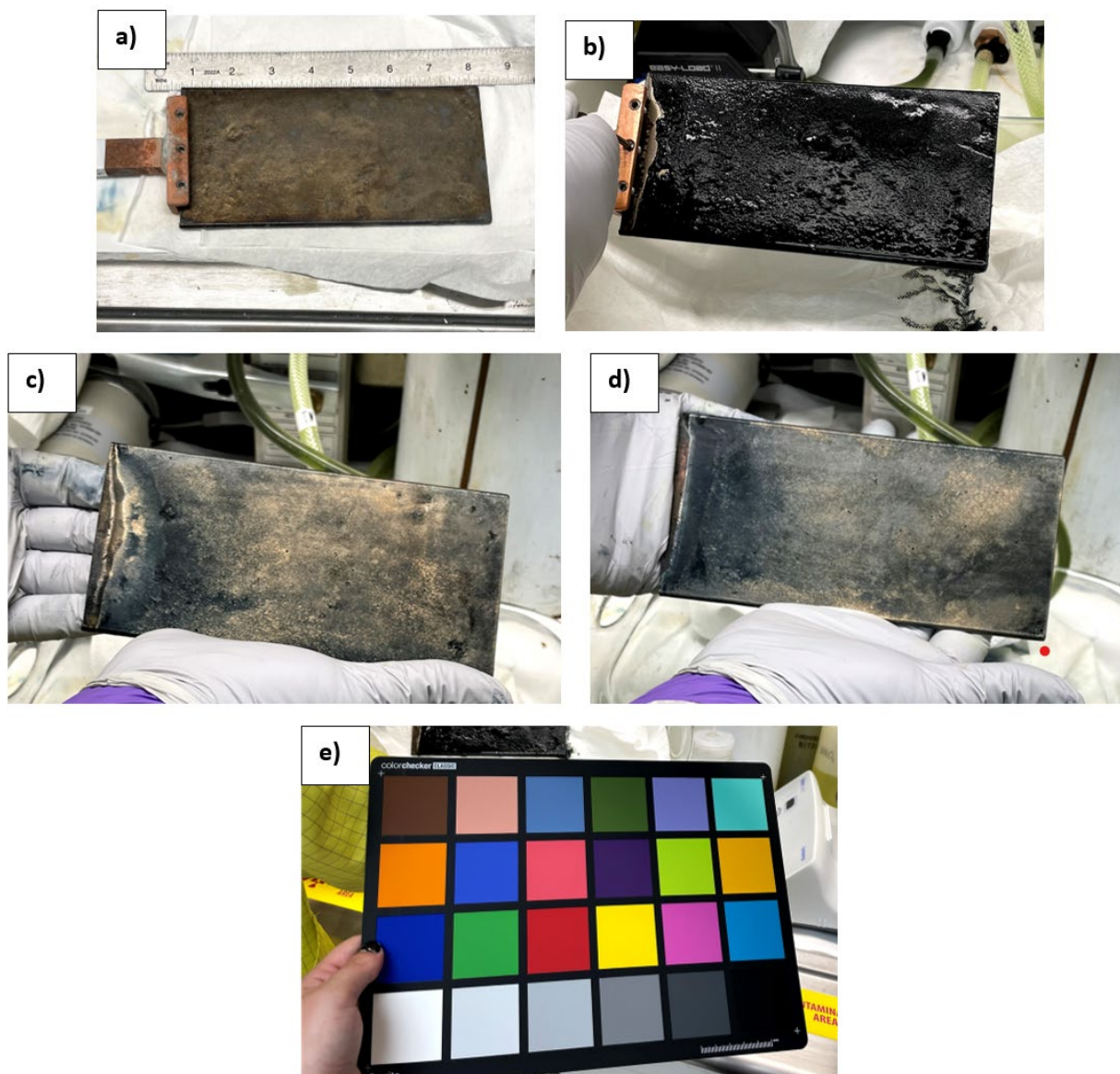
After the surface was dry, it was analyzed via LCM at the 5x and 10x magnifications (Figure 17) and surface roughness measurements taken (Table 6). The measured surface roughness saw 12-15% reduction in Ra and Sa values, showing improvement compared to nitric acid cleaning. Examination of the electropolished surface imaging showed the pitting seen in the original surface (Figure 17) was beginning to be polished out. However, some pitting was still visible in the polished images (Figure 17) which is likely why the Sz values did not change significantly. Since most of the surface removal was oxide and smut from the casting during this surface treatment, more time is likely required to remove these pits from the surface after the oxide is removed.

Temperature measurements before and after testing found that the system increased in temperature during the test between 7-8°C with the cooling system operating. The increase in temperature was rapid within the initial three minutes running the peristaltic pump at 40% of the maximum speed. However, the temperature stabilized once the speed was increased to 70% of the maximum speed to increase the heat transfer rate of the heat exchanger. The temperature increase was not surprising considering the significant mass losses (55 grams into 3L)

measured. However, the increase in temperature did not impact the test significantly and the temperature remained within the operating range of the test.

Table 5. Operation Parameters for Initial Electropolish Run of Cast Plate

Parameter	Value
Plate Initial Mass	1.420 kg
Plate Dimensions	7.5 in x 3.6 in x 0.2 in
Sample Depth in Electrolyte	6.7 in
Polishing Area	52 in ²
Applied Voltage	7.6 V
Power Supply Amperage	260 amp
Target Current Density	5.0 A/in ²
Ramp Up Time	01:00 min
Hold Time	10:00 min
Ramp Down Time	01:00 min
Initial Temperature	24-26°C
Final Temperature	32-33°C
Plate Final Mass	1.365 kg
Removed Mass	55 g
Removed Surface	0.2 g/cm ²



- The plate before electroplating.
- The plate immediately after polishing and before rinsing. A blue/black film appeared on the surface during the polishing process.
- Side 1 of the plate following polishing and rinsing. There were sections with dark blue and copper coloration and the plate was smoother to the touch.
- Side 2 of the plate following polishing and rinsing. There were sections with dark blue and copper coloration and the plate was smoother to the touch.
- A color chart for comparison purposes

Figure 16. The DU-10Mo plate throughout the first electroplating process

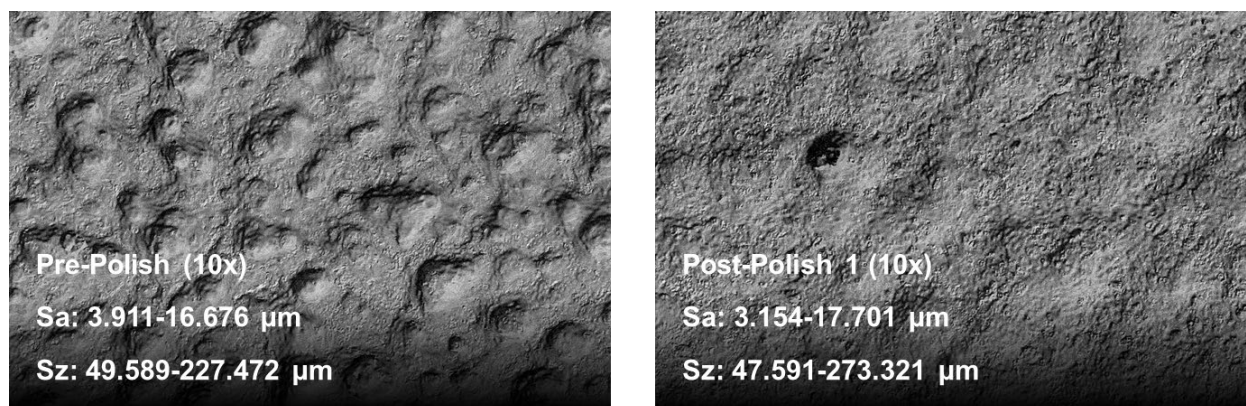


Figure 17. C-DIC Images of DU-10Mo cast surface before and after initial electropolish

Table 6. The surface roughness values before and after electropolishing

	Average Ra (μm)	Average Rz (μm)	Average Sa (μm)	Average Sz (μm)
Pre-Polish	8.8 (\pm 3.7)	57.2 (\pm 21.4)	9.6 (\pm 3.8)	122.7 (\pm 52.4)
Post-Polish	7.6 (\pm 4.1)	48.9 (\pm 33.7)	8.4 (\pm 4.7)	119.8 (\pm 77.0)

3.2.2 Second Polishing Test of Cast Plate (Half Plate)

After characterizing the plate and replacing the electrolyte solution, the initially electropolished DU-10Mo cast plate was polished again using the parameters established in Table 7. Only half of the plate (3.75 in) was immersed for the test using the same run time. The amperage was adjusted to keep the current density the same while the applied voltage across the electrodes did not change significantly. Images of the polish of the bottom half of the plate are found in Figure 18. The mass loss measured was 35 g for an average surface removal of 0.2 g/cm². This was significantly lower than the 0.5 g/cm² from the previous test where oxide/smut from the cast plate was removed as well.

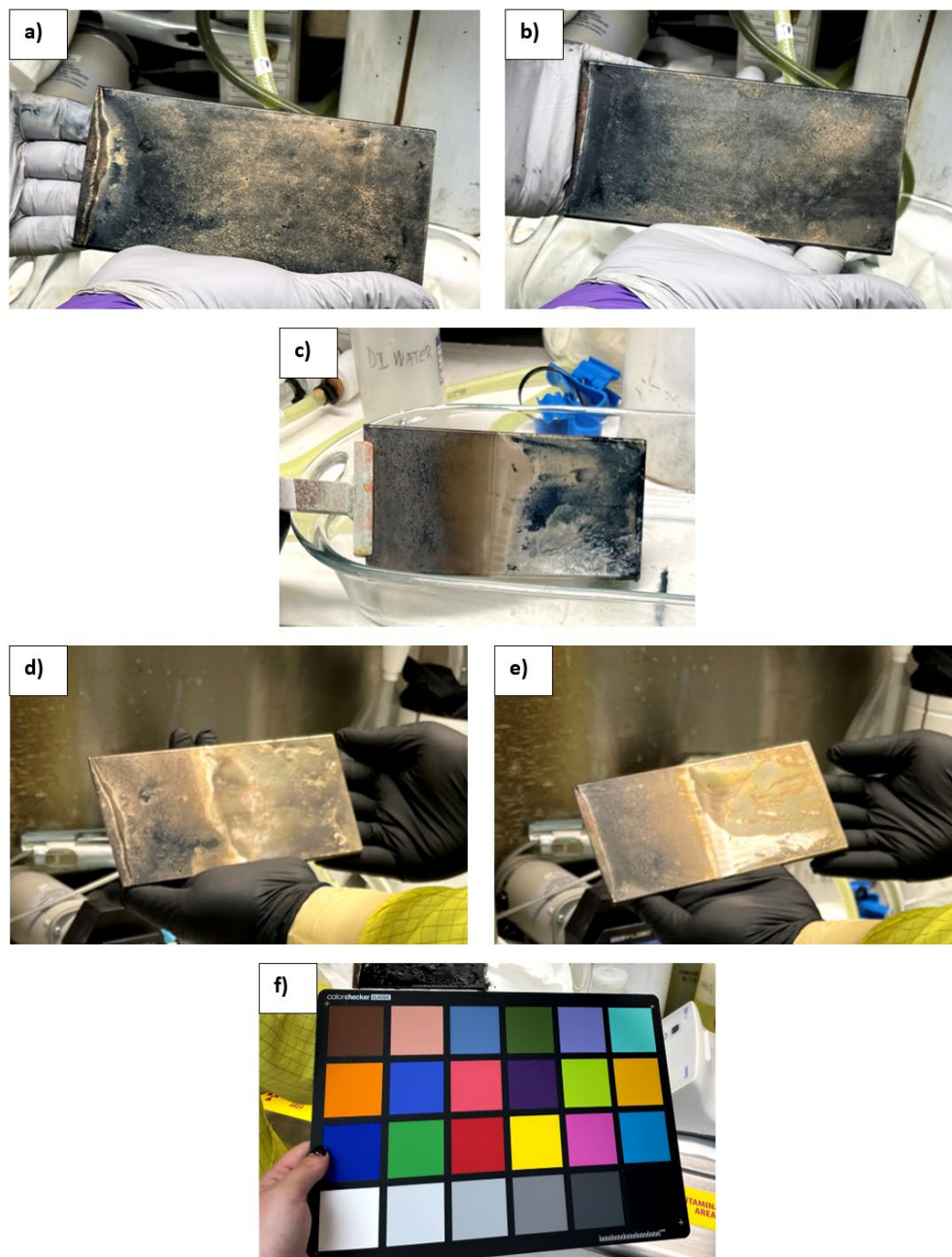
The polishing solution changed to a blue/black color at between 4-5 minutes and a dark blue/green film was present on the surface afterwards (Figure 18c). A dilute solution (0.2M) solution of sulfuric acid was initially used to remove the film from the polished piece. This solution easily redissolved this film into solution allowing the use of less DI water to rinse the surface clean. The newly electropolished surface was tinted a light gold color and lighter than the blue-gold color of the previous surface (Figure 18d and Figure 18e).

The newly electropolished surface was analyzed via LCM at the 20x and 50x magnifications for comparison to the previous surface condition (Figure 19). The images show a smoother surface with less variability was created after the second electropolish. Changes in surface roughness were also measured by LCM and compared to the initial cast and polished values. The overall average Ra/Rz/Sa/Sz values and their standard deviations are plotted in Figure 20. Not only was there a significant reduction in surface roughness (>75%) after the second polish, but a significant decrease in the variability of the measurement. A uniform surface finish was achieved.

Temperature measurements before and after testing found that the system increased in temperature during the test between 5-8°C with the cooling system operating with the peristaltic pump run at 50% of the maximum speed for the entire test. Decreasing the working surface area proportionally decreases the heat generation and load on the cooling system to maintain temperature.

Table 7. Operation Parameters for Second Electropolish Run of Cast Plate

Parameter	Value
Plate Initial Mass	1.365 kg
Plate Dimensions	7.5 in x 3.6 in x 0.2 in
Sample Depth in Electrolyte	3.75 in
Polishing Area	29 in ²
Applied Voltage	7.3 V
Power Supply Amperage	150 A
Target Current Density	5.2 A/in ²
Ramp Up Time	01:00 min
Hold Time	10:00 min
Ramp Down Time	01:00 min
Initial Temperature	16-17°C
Final Temperature	21-25°C
Plate Final Mass	1.330 kg
Removed Mass	35 g
Removed Surface	0.2 g/cm ²



- a) Side 1 of the plate prior to polishing. The surface was smooth with sections of blue and copper coloration.
- b) Side 2 of the plate prior to polishing. The surface was smooth with sections of blue and copper coloration.
- c) The plate after polishing and before rinsing. A dark blue film, likely uranyl sulfate, formed on the surface during the polish. The acid solution (not shown) also turned blue approximately 03:00 minutes into the polish
- d) Side 1 of the plate after polishing and rinsing. The surface is a smoother, shinier, copper color.
- e) Side 2 of the plate after polishing and rinsing. The surface is a smoother, shinier, copper color
- f) A color chart for comparison purposes

Figure 18. The U-10Mo plate throughout the second electropolishing process

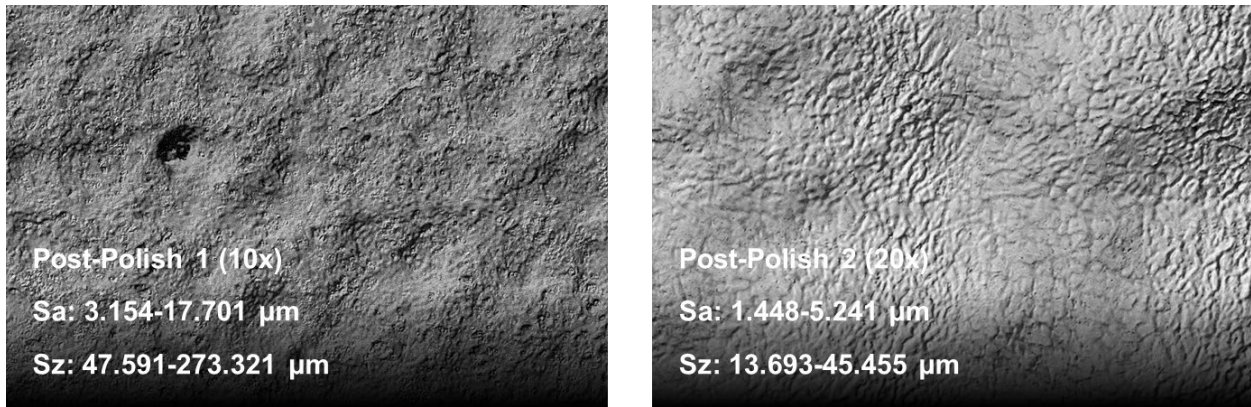


Figure 19. C-DIC images of the DU-10Mo cast surface before (left) and after the second electropolish (right). Note: The magnification of the two images is different.

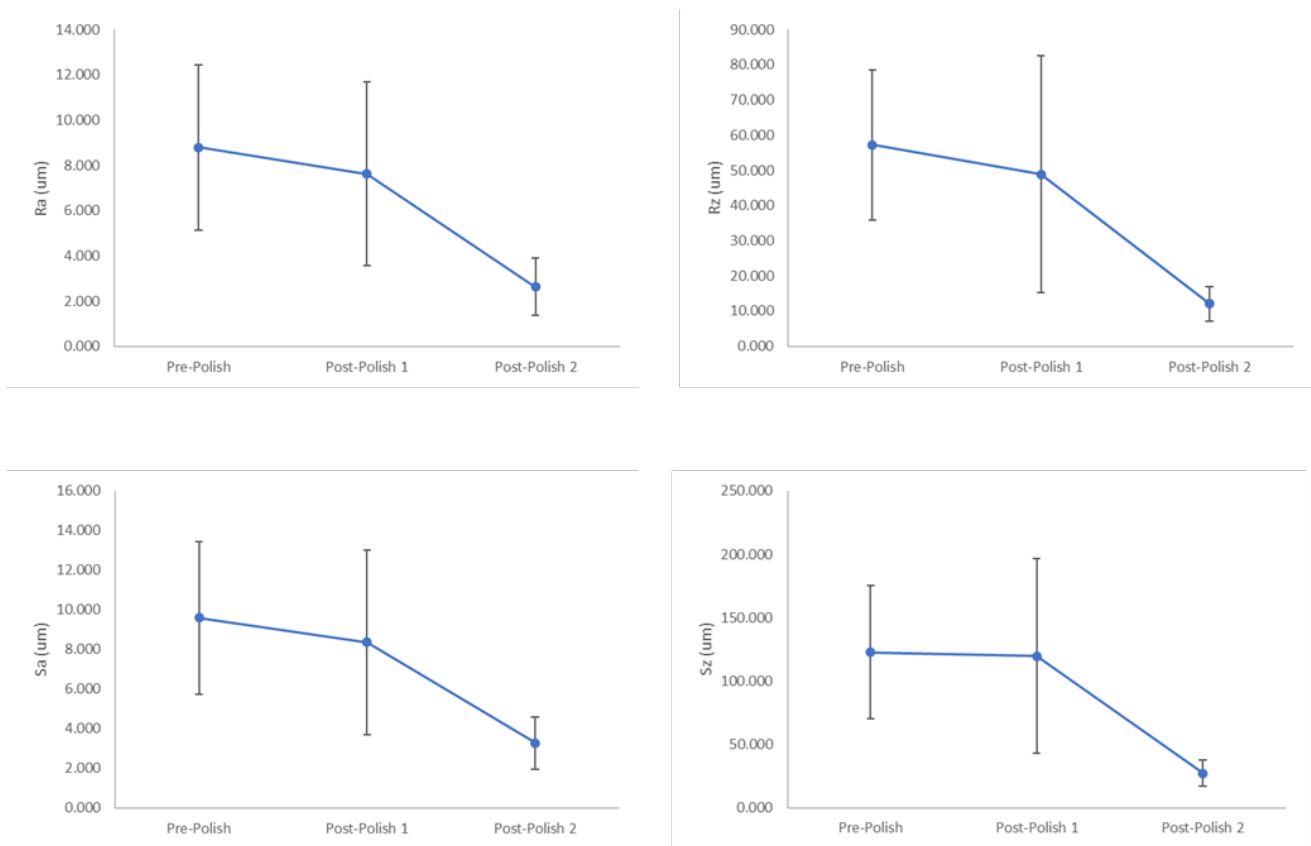
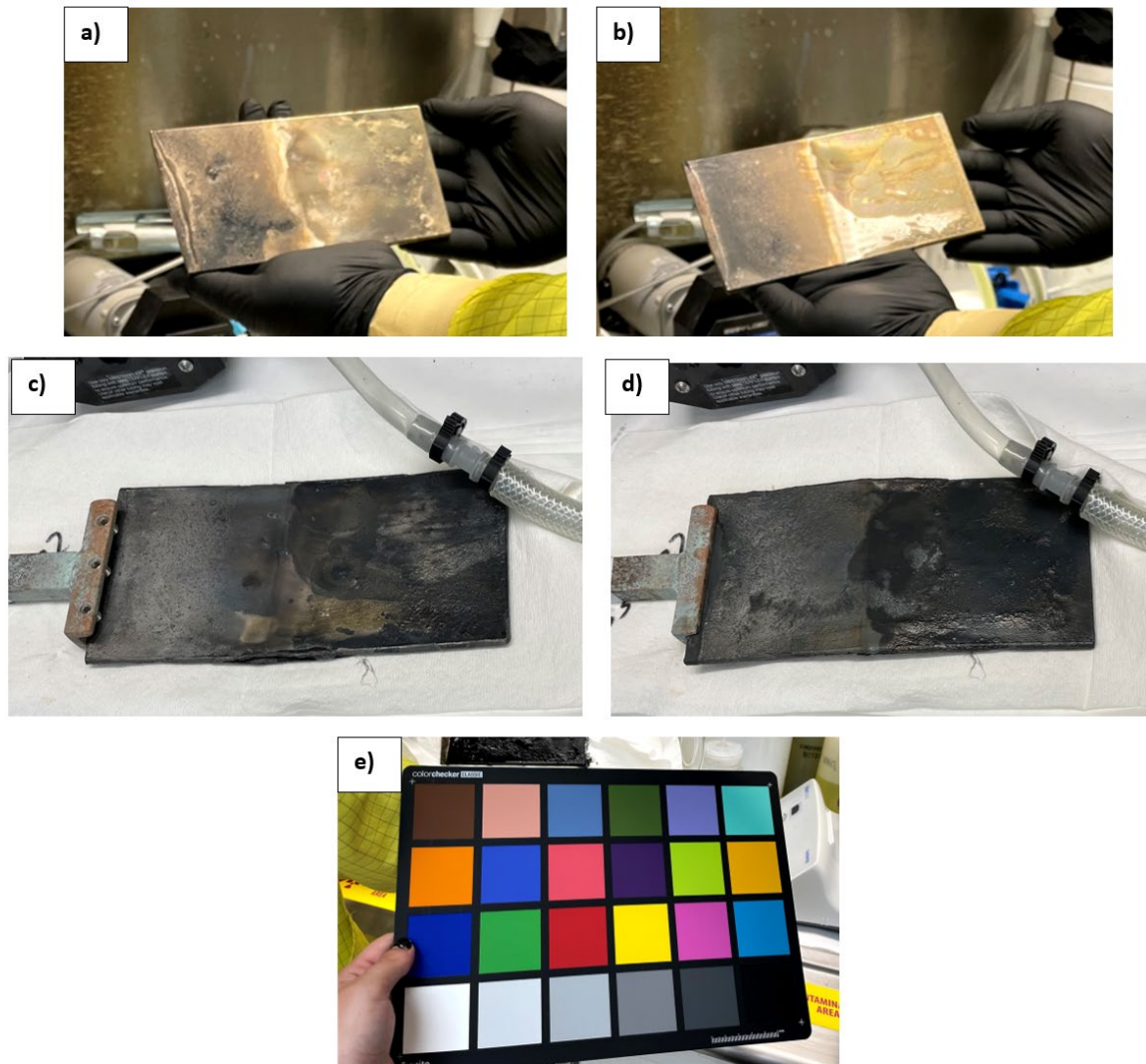


Figure 20. Average Ra, Rz, Sa, and Sz surface roughness values (μm) for the DU-10Mo plate prior to polishing and after the two electropolishes. The error bars represent one standard deviation.

3.3 Electropolishing After Homogenization

3.3.1 Homogenization and Post Surface Analysis

The U-10Mo plate before and after homogenization is shown in Figure 21 and the surface was re-analyzed by LCM afterwards (Figure 22). The average surface roughness increased following homogenization. Variability of surface roughness measurements also increased when compared to the pre-homogenization surface.



- Side 1 of the plate prior to homogenization. The surface is smooth with a shiny copper color.
- Side 2 of the plate prior to homogenization. The surface is smooth with a shiny copper color.
- Side 1 of the plate following homogenization. The surface was gray and more matte with a rougher surface. There is also a slight bend in the plate due to a hole becoming apparent during homogenization.
- Side 2 of the plate following homogenization. The surface was gray and more matte with a rougher surface. There is also a slight bend in the plate due to a hole becoming apparent during homogenization.
- A color chart for comparison purposes

Figure 21. The U-10Mo plate before and after homogenization

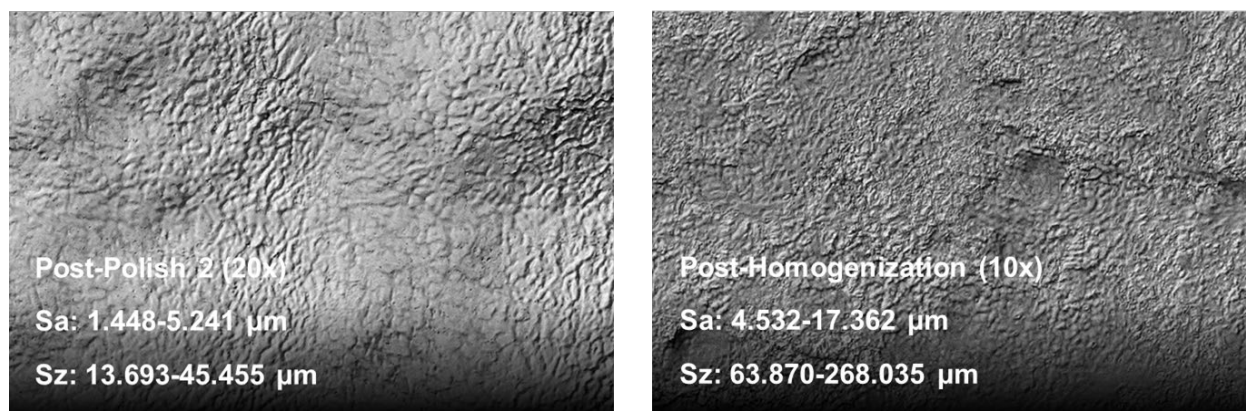


Figure 22. C-DIC images of the DU-10Mo cast surface before (left) and after the homogenization anneal (right). Note: The magnification of the two images is different.

3.3.2 Post-Homogenization Polishing Test

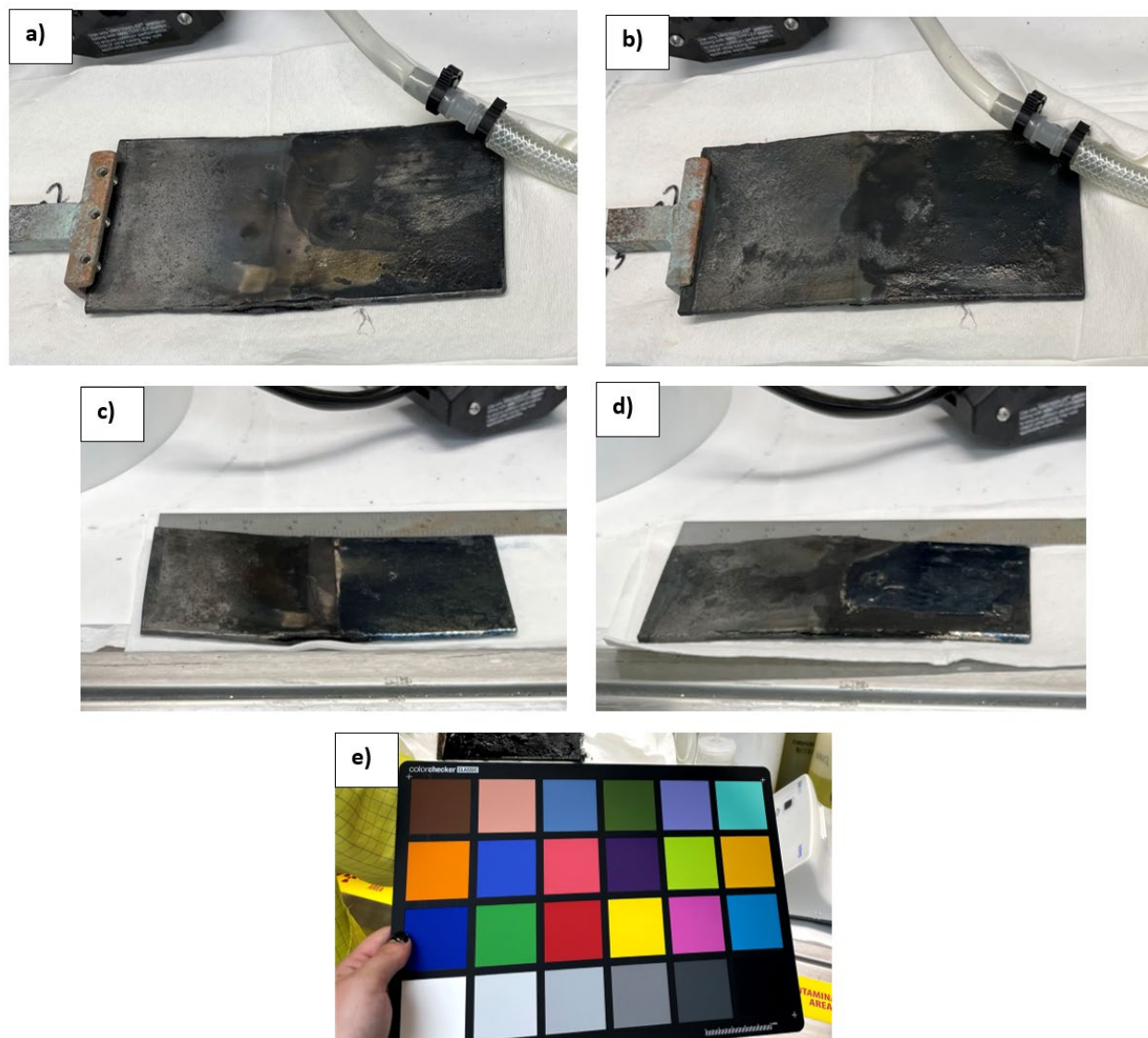
After characterizing the post-homogenized surface, the plate was electropolished using the polishing parameters are found in Table 8. Only half the plate was inserted into the electrolyte solution (3.75 in) and the amperage was kept the same as the previous polish to get a current density of 5.2 A/in². The overall applied voltage decreased to 6.4 V. Images of the post-homogenized plate before and after electropolishing are found in Figure 23. The mass loss measured was 36 grams (0.2 g/cm²) like the electropolishing just before the homogenization using only half the plate surface area. Circulating pump to the heat exchanger was increased from 40% to 70% percent of maximum flow rate to maintain the temperature near room temperature with only a 2-6°C increase in temperature.

The acid solution changed to a blue/black color at ~7 minutes into the polish and a dark blue-green film was present again. DI water and ethanol were used to rinse the surface and it was allowed to dry. The surface of the plate appeared smoother, however the final polish after homogenization resulted in an increase in surface roughness and inconsistency on the surface. The polish also left a blue/gold sheen on edges of the annealed plate.

The dried plate was analyzed via LCM immediately and one week after polishing at the 5x and 10x magnification. Color optical imaging of the plate (Figure 24) show the difference in the surface color from edge of the plate to the center. C-DIC images of the electropolished surface (Figure 25) and average surface roughness measurements of the plate (Figure 26) show an increase in variability of the surface area of the plate after homogenization but the variability did not change after one week and the surface remained free of oxide. The lack of oxidation and changes to the microstructure also appear to have minimized the surface roughness improvement on the pre-homogenized cast plate after the second polishing. This implies that the initial polish prior to this homogenization step should focus on removing the oxidation/smut from the received plate while more polishing is required post-anneal to improve the surface further. The C-DIC images for the Bottom Left for each of process step are shown in Figure 27 for comparison and correlation to the average numerical surface roughness data shown in Figure 26.

Table 8. Test parameters for third electropolishing with cast and homogenized plate

Parameter	Value
Plate Initial Mass	1.330 kg
Plate Dimensions	7.5 in x 3.6 in x 0.2 in
Sample Depth in Electrolyte	3.75 in
Polishing Area	29 in ²
Applied Voltage	6.4 V
Power Supply Amperage	150 A
Target Current Density	5.2 A/in ²
Ramp Up Time	01:00 min
Hold Time	10:00 min
Ramp Down Time	01:00 min
Initial Temperature	19°C
Final Temperature	21-25°C
Plate Final Mass	1.294 kg
Removed Mass	36 g
Removed Surface	0.2 g/cm ²



- a) Side 1 of the plate prior to polishing. The surface is rough, matte, and dark grey.
- b) Side 2 of the plate prior to polishing. The surface is rough, matte, and dark grey.
- c) Side 1 of the plate after the post-homogenization electropolish. The plate had a rough and shiny surface with majority blue coloration.
- d) Side 2 of the plate after the post-homogenization electropolish. The plate had a rough and shiny surface with majority blue coloration.
- e) A color chart for comparison purposes.

Figure 23. The U-10Mo plate throughout the post-homogenization electropolish

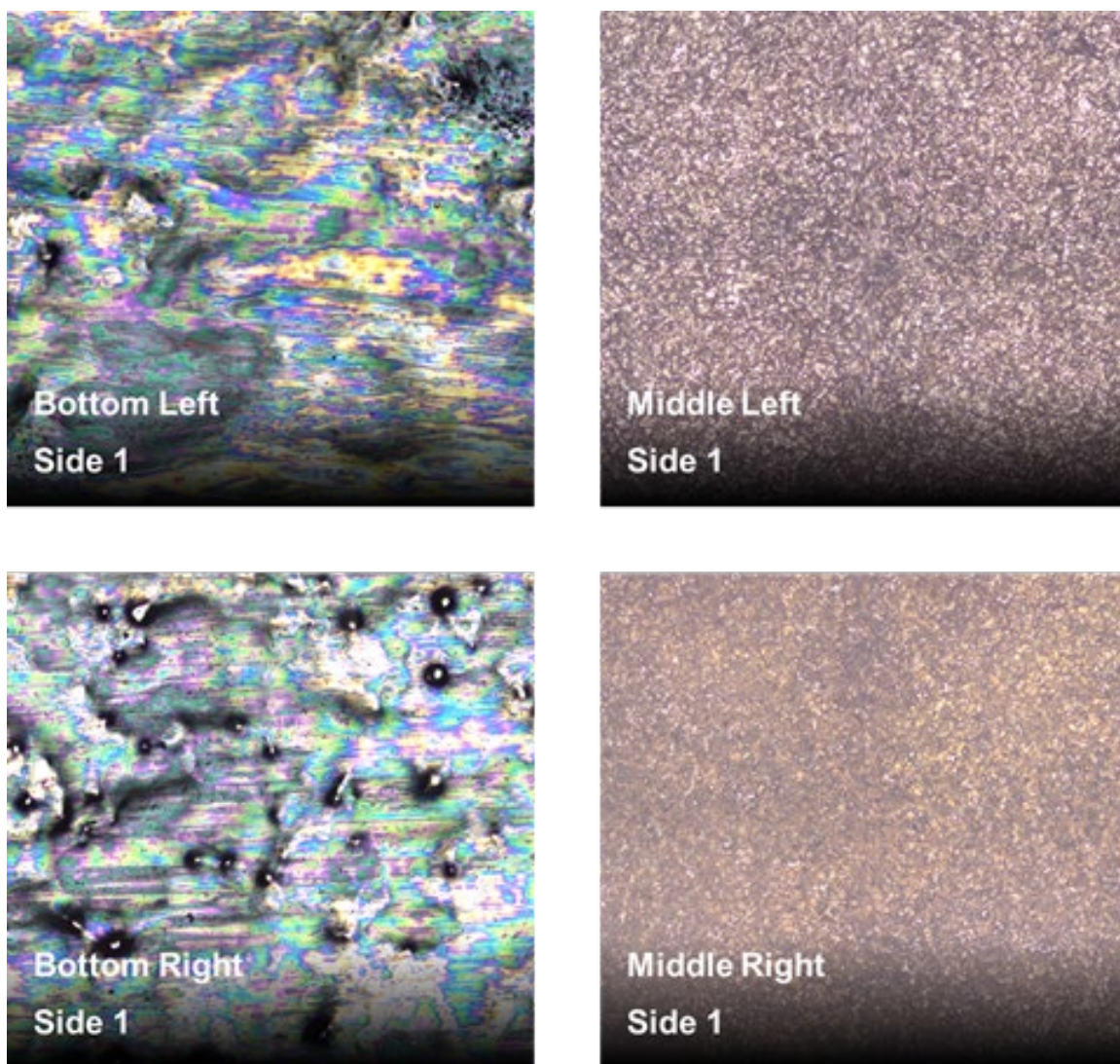


Figure 24. The optical images for each section of Side 1 after the third electropolish. Both bottom sections showed a blue sheen with gold undertone that was not as intense at the center of the plate.

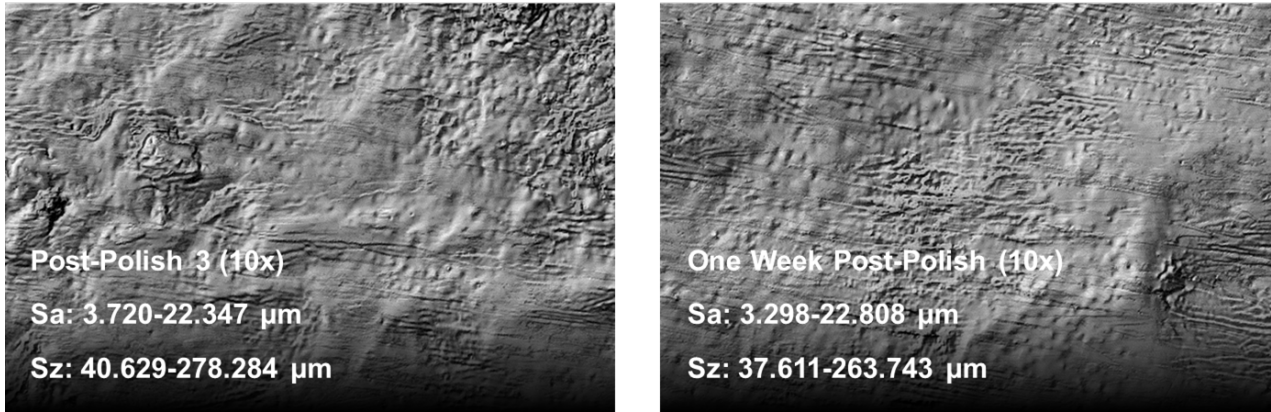


Figure 25. C-DIC images of the annealed DU-10Mo surface after electropolishing (left) and one week later (right)

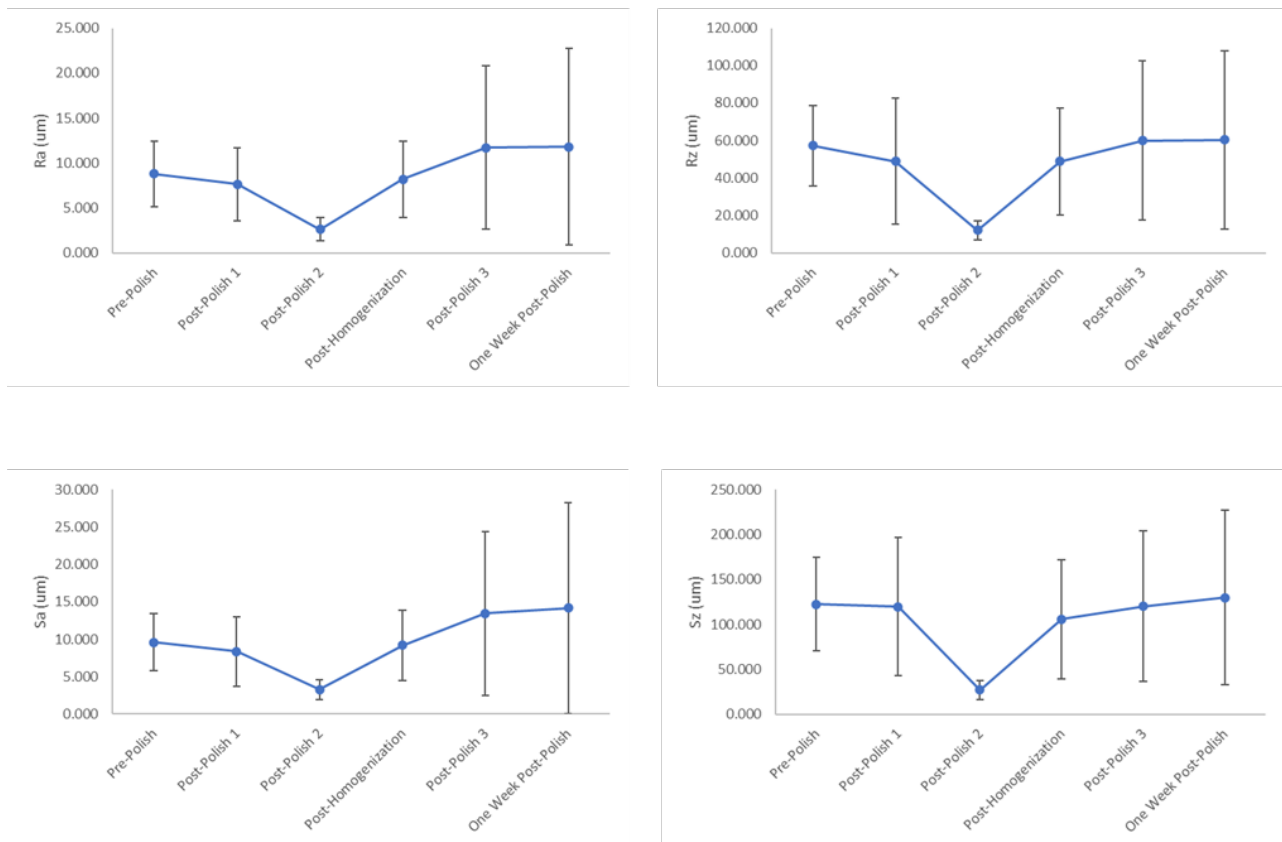
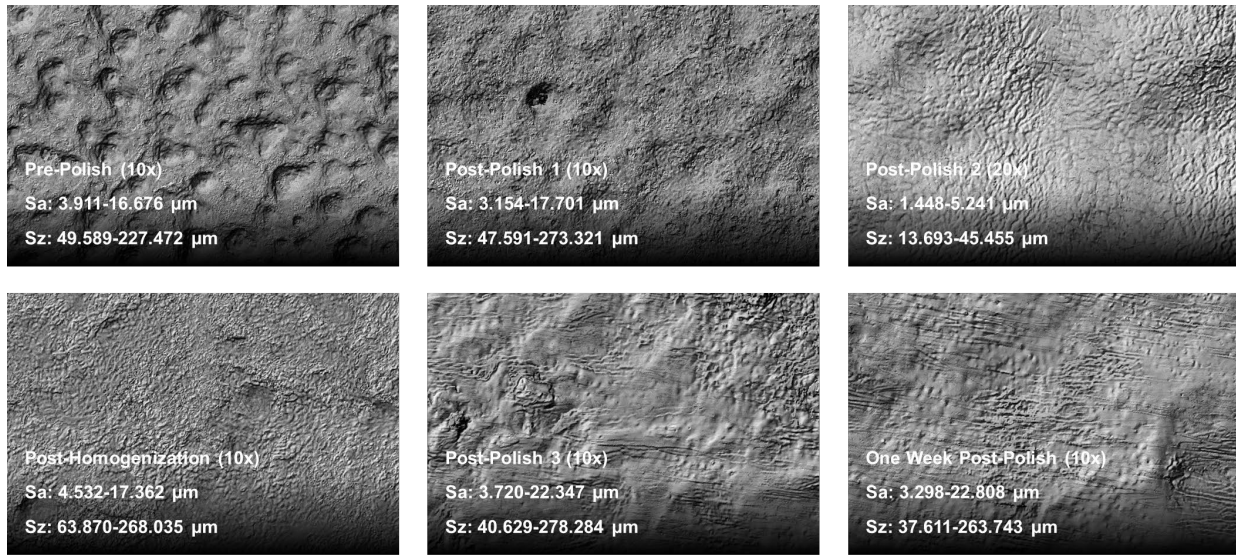


Figure 26. The average Ra, Rz, Sa, and Sz values for the U-10Mo plate from initial casting to one week after the final post-homogenization electropolish. The error bars represent one standard deviation.



9

Figure 27. The 10x magnification C-DIC images for the Bottom Left section of Side 1 throughout the polishing process, ending with the images taken one week after polishing. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes.

3.4 Equipment Limitations and Scale Up Concerns

As expected, issues were experienced in scaling up that were not observed in previous testing. Despite decreasing the concentration of H_2SO_4 from 30 wt% to 15 wt%, the solubility of uranium in solution was still an issue. Because of limited circulation within the electrolyte solution, a hydrated film (likely uranous sulfate) formed on the surface of the plate that was difficult to remove and likely increased the applied voltage on the plate to maintain a desired current density and increased the likelihood the plate surface would anodize. While this film can be removed by with a dilute H_2SO_4 , additional waste generation is not desired. Efforts need to be explored to increase the oxidation state of U^{4+} in solution to the more soluble of U^{6+} using hydrogen peroxide, but this may lead to more oxidation on the U-10Mo plate during electropolishing. Increased agitation is recommended as well as an increase in tank volume to minimize localized areas of high U concentration in solution near the plate. Other electrolyte solutions without these solubility limitations could also be investigated.

Heat generation not observed in the small-scale studies was found to be significant as the system was scaled larger. To maintain temperatures below 35°C , an external heat exchanger and chiller were installed to remove heat from the electrolyte during operations. The hardware used (stainless steel exchanger, poly lines) was resistant to corrosion for this testing. With this system installed, the initial and final temperatures of the acid bath were approximately the same. The system was manual operated but could be automated in a more industrial application.

As with previous testing, the contact point between the work piece and the electrode wiring connections still requires more work. The non-uniform shape of the plate made it difficult to determine a contact location and an irregular voltage was noted. This irregular voltage created anodization on the surface that was seen in both small and large-scale studies. This anodization was more pronounced at the locations where power was applied.

3.5 Final Discussion

Overall, the use of electropolishing was able to smooth the surface of the cast DU-10Mo plate. Images of the plate following each step of the process are shown in Figure 28. The surface became shinier and smoother following the first two polishes; however, it became dull and rough following homogenization and was only slightly improved following a single final polish, suggesting that more effort to reduce the surface roughness should occur after homogenization than before. However, the ability for the electropolish to reduce oxidation was still observed in all test cases, with the surface of the annealed DU-10Mo that was electropolished still free of oxidation after 9 months (Figure 29).

The C-DIC images, S_a , and S_z values for a U-10Mo plate before and after nitric acid cleaning carried out in a different study are shown in Figure 30. For comparison the C-DIC images and surface roughness values before and after each step of the electropolishing process carried out in this study are shown in Figure 30. The use of a multi-step electropolishing process decreased the surface roughness values more than the use of the nitric acid cleaning process.

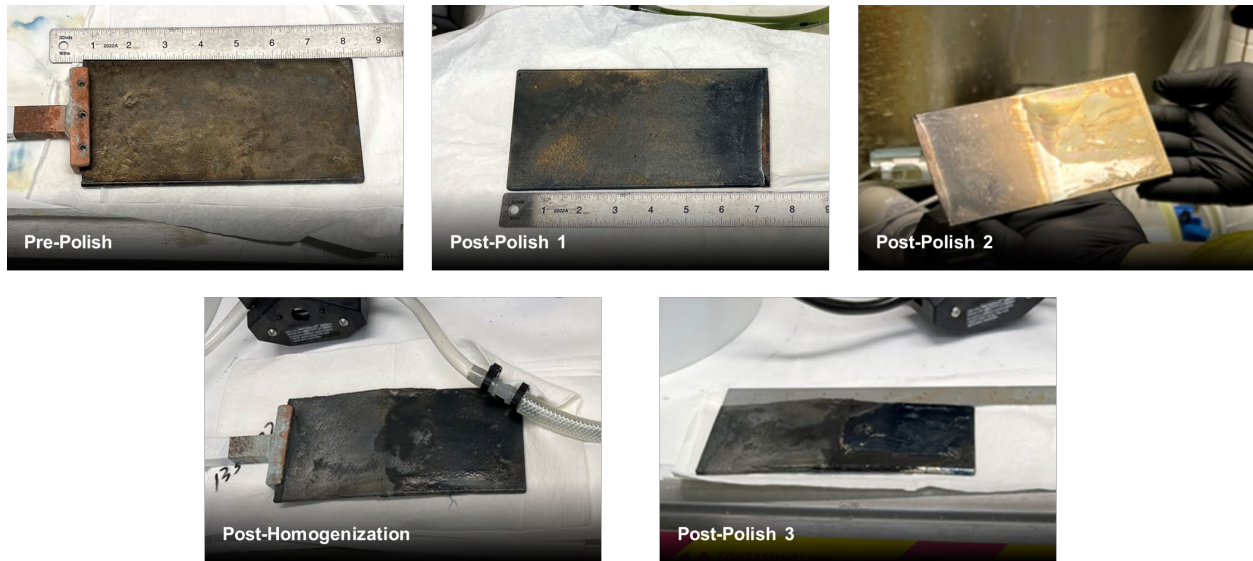


Figure 28. The U-10Mo plate before and after each step of the electropolishing process

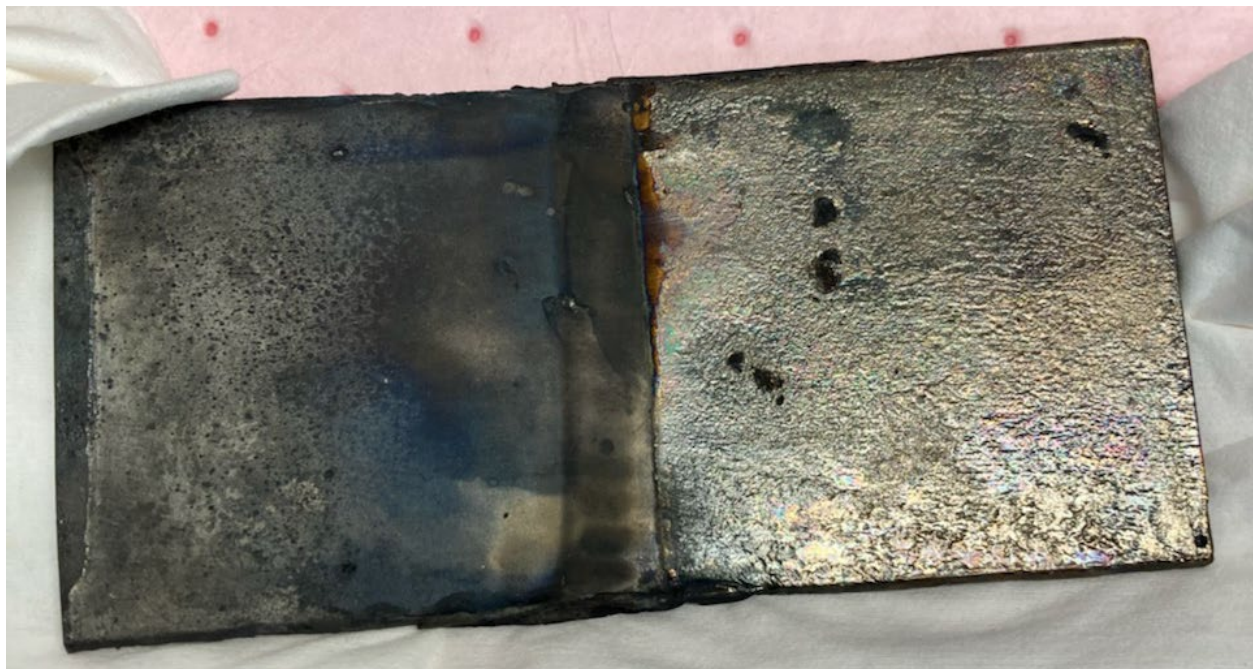


Figure 29. Homogenized DU-10Mo surface ~9 months after the bottom-half (right side) was electropolished

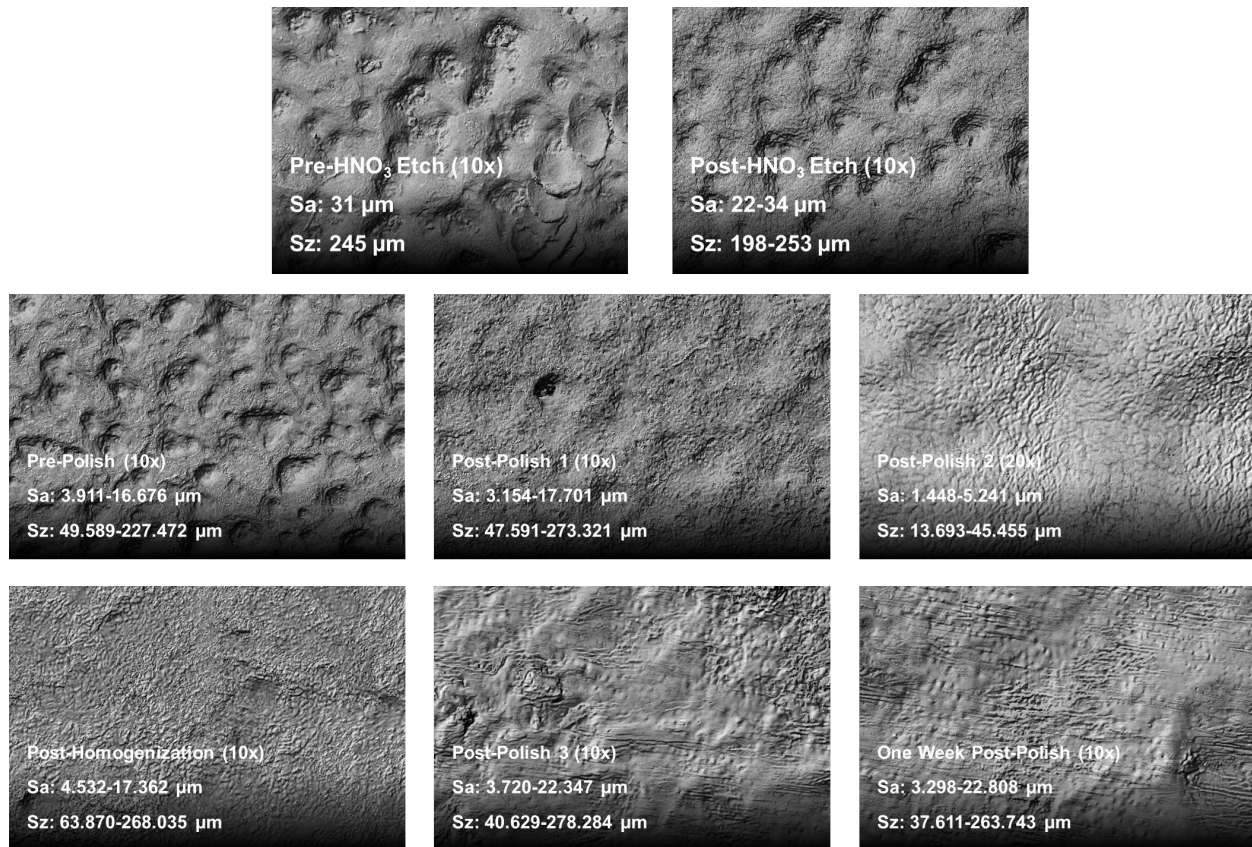


Figure 30. The C-DIC images and surface roughness values for a U-10Mo plate before and after HNO₃ etching and before and after each step of the electro polishing process. The use of electro polishing was able to decrease the Sa and Sz values in each case. These values were also lower than those found in the HNO₃ study.

4.0 Conclusions

Electropolishing is a useful method of surface finishing, passivation, and deburring. It is also capable of removing tarnishes, oxides, surface corrosion, and scratches. In the case of U-10Mo fuel fabrication, it is being investigated as a method of removing oxidation and to smooth the surface prior to initial hot rolling of the anneal cast. A series of three electropolishing trials, with a homogenization step in between the second and third polishes, were carried out. Each polish lasted for approximately 12:00 minutes in 15% H₂SO₄ with a constant current density for each test. The surface roughness was measured using LCM following each step in the process. The roughness decreased after the first and second polishes, however the surface roughness increased during the homogenization minimizing the impacts made by the first two polishes and a single third polish was not long enough to reduce the impact from re-oxidation from the homogenization. The study implies that efforts to clean the surface prior to homogenization should be focused on oxide/smut removal and longer electropolishing times should occur after homogenization to improve surface roughness. However, in all cases with electropolishing, the surface roughness values were lower when compared to the nitric acid cleaning study, and the final electropolished surface was still free of oxide months afterwards.

Scaling up from a small coupon to a full-sized plate presented challenges during testing. Heat generation was more significant than previously observed from small scales tests. This issue was addressed using a heat exchanger. Lack of circulation combined with the low solubility of U⁴⁺ in solution allowed a hydrated film of a uranium compound to build up on the surface of the plate that was difficult to remove and generated additional waste. There was also more anodization observed on the surface of the plate than in previous testing. Additional studies with different electrolytes, tank configurations, and electrode to the U-10Mo plate are needed to resolve some of these outstanding issues.

5.0 Quality Assurance

This work was performed in accordance with the Pacific Northwest National Laboratory 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.

6.0 References

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Appendix A – C-DIC Images

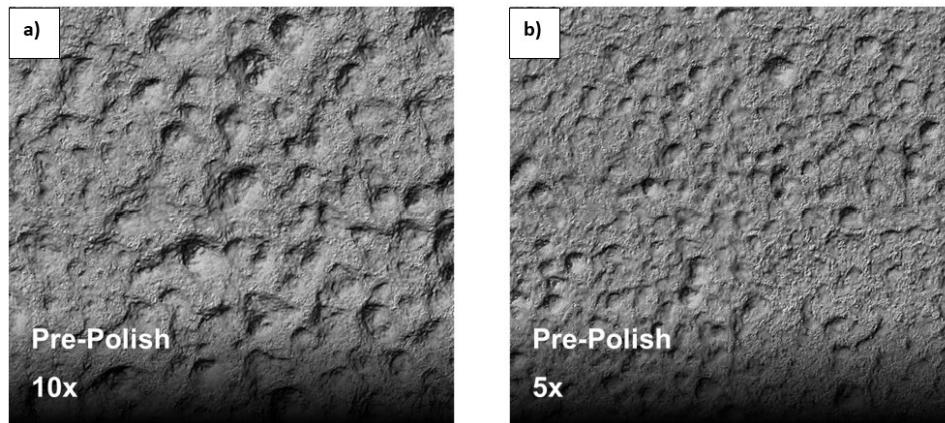


Figure A.1 The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 1 of the U-10Mo plate prior to polishing.

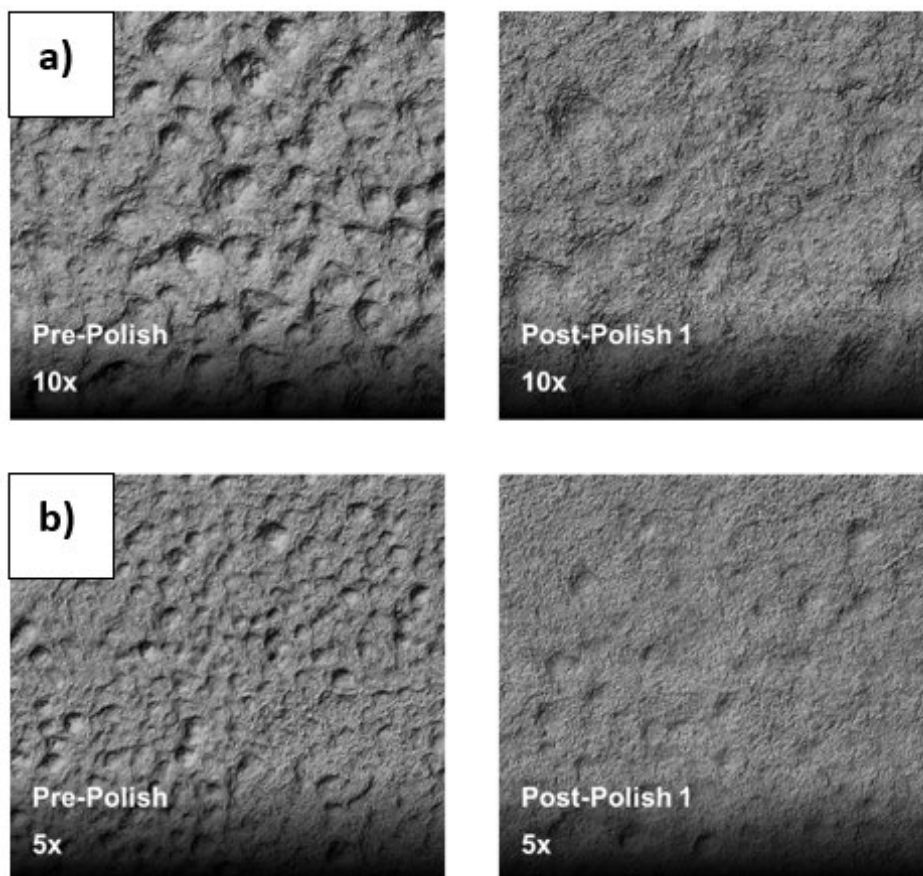


Figure A.2. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 1 of the U-10Mo plate before and after the first electropolish. The surface of the plate was noticeably smoother, which is corroborated by the numerical data.

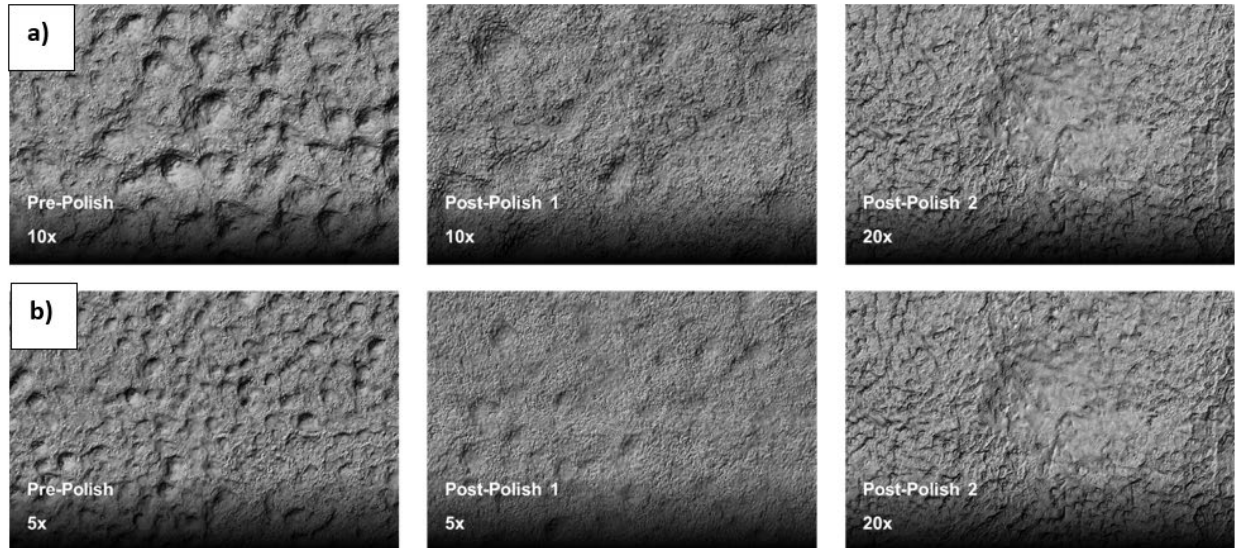


Figure A.3 The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 1 of the U-10Mo plate before and after two polishes. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes; however, the numerical data indicates that the surface roughness decreased following the second polish.

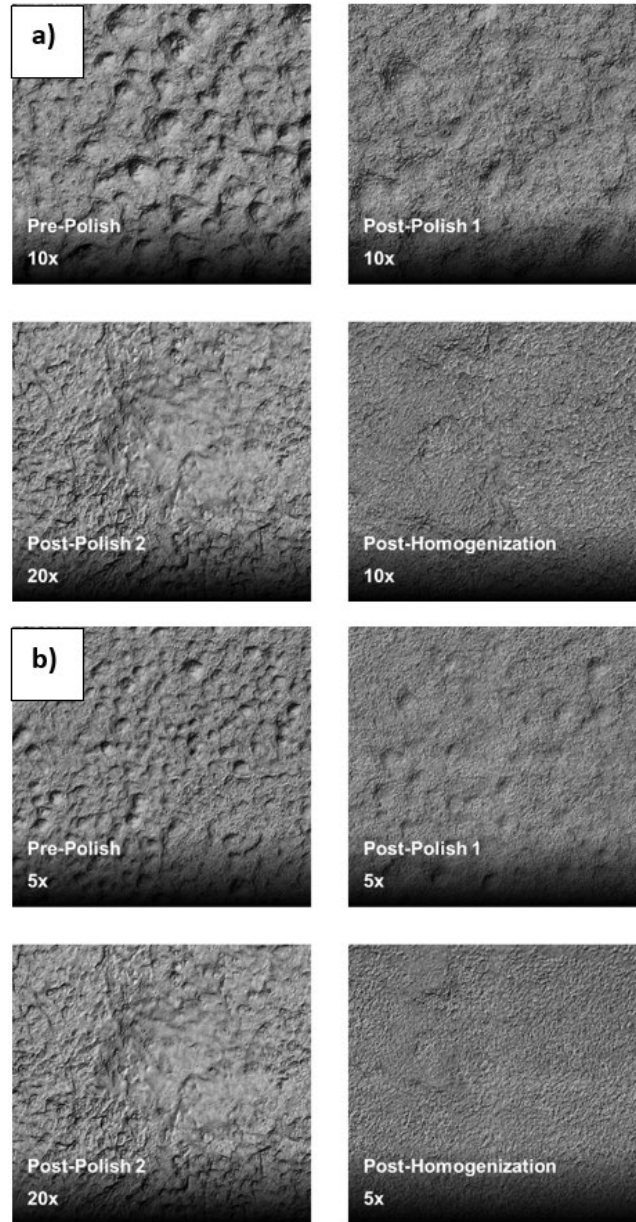


Figure A.4. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 1 of the U-10Mo plate before and after two polishes and a homogenization step. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. Following homogenization, the quality of the surface appeared like that of the surface following the first polish. This is corroborated by the numerical data.

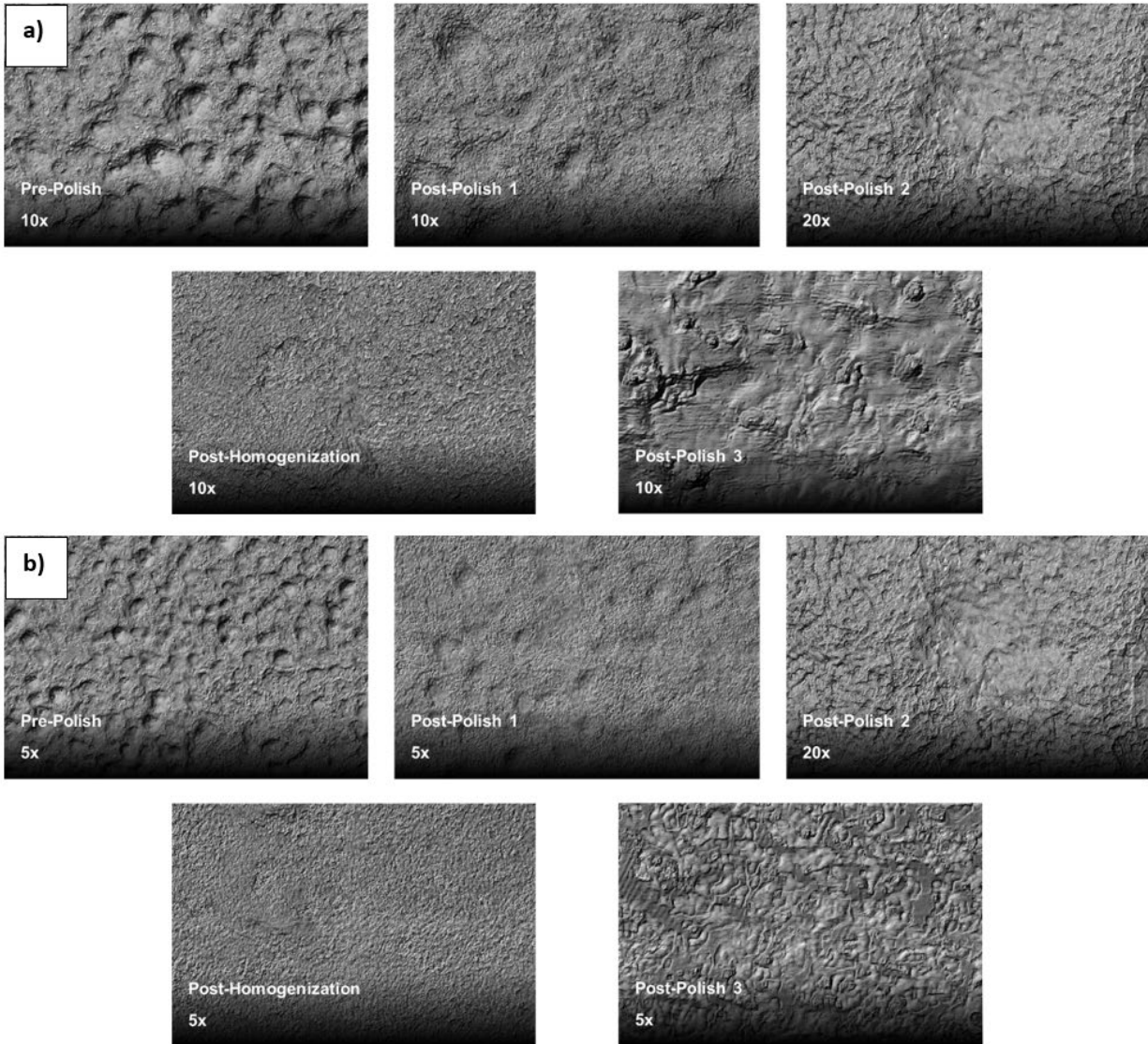


Figure A.5. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 1 of the U-10Mo plate before and after two polishes, a homogenization step, and a third polish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness appeared to slightly increase between homogenization and the final polish. This is supported by the numerical data.

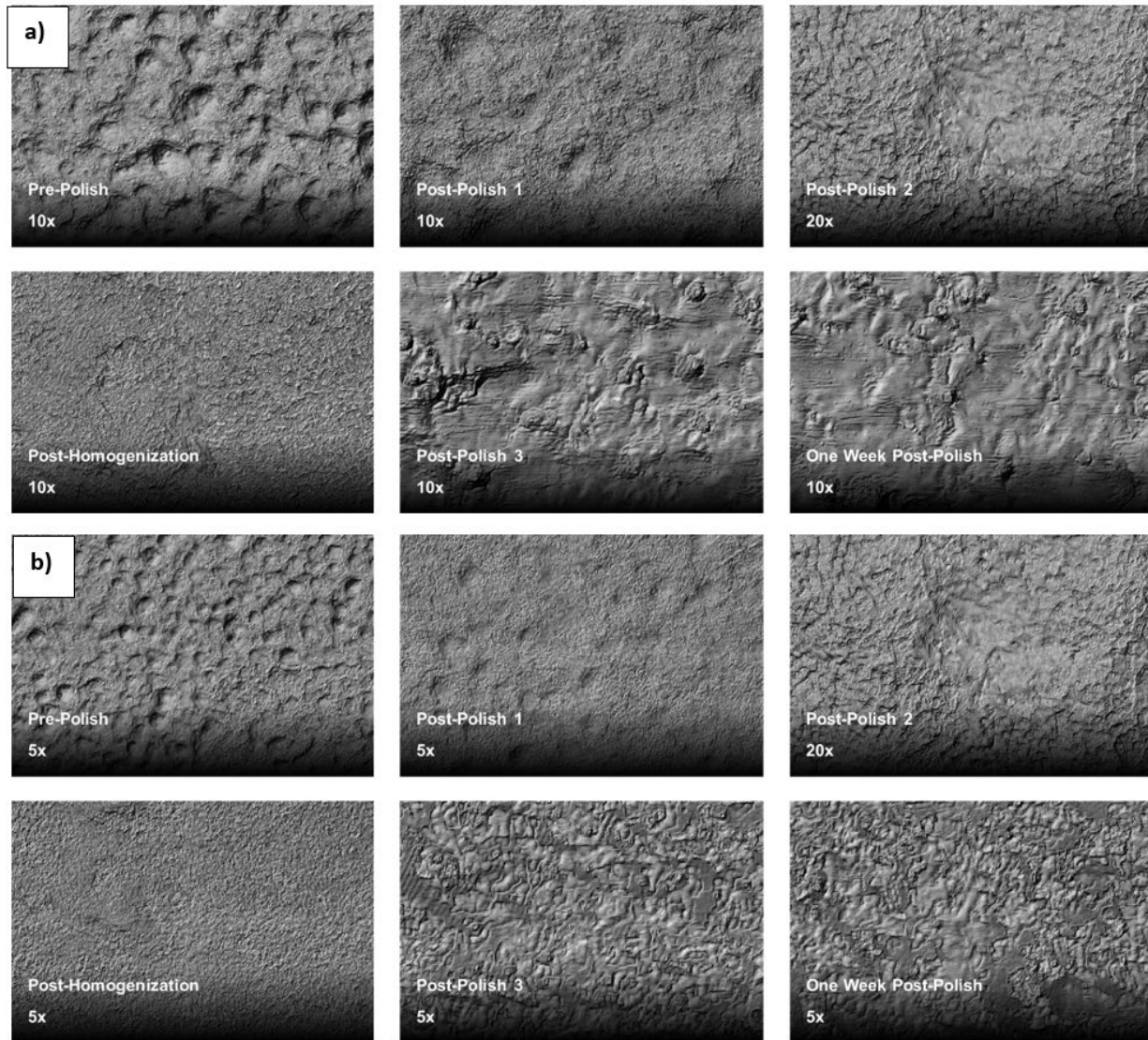


Figure A.6. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 1 of the U-10Mo plate throughout the polishing process and one week after the final polish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface of the plate appeared the same one week after the final polish, which is supported by the surface roughness data.

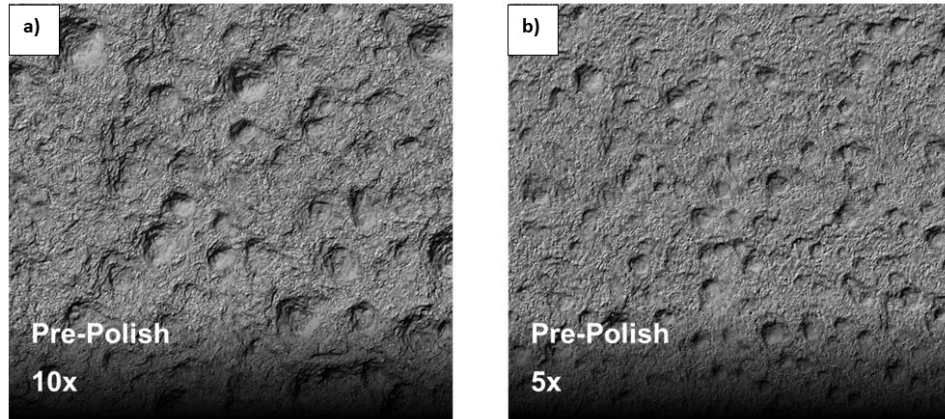


Figure A.7. The a) 10x magnification and b) 5x magnification C-DIC images of the Middle Right section of Side 1 of the U-10Mo plate prior to electropolishing.

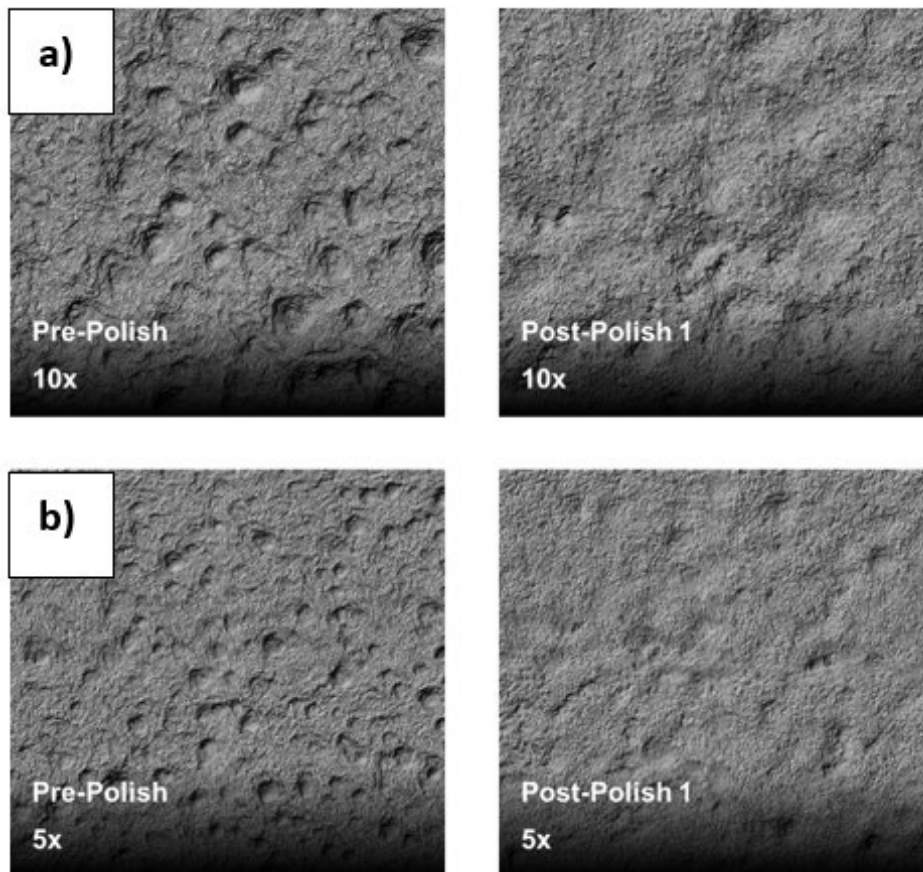


Figure A.8. The a) 10x magnification and b) 5x magnification C-DIC images of the Middle Right section of Side 1 of the U-10Mo plate before and after electropolishing. The surface roughness appeared to decrease following the first polish. This is corroborated by the numerical surface roughness data.

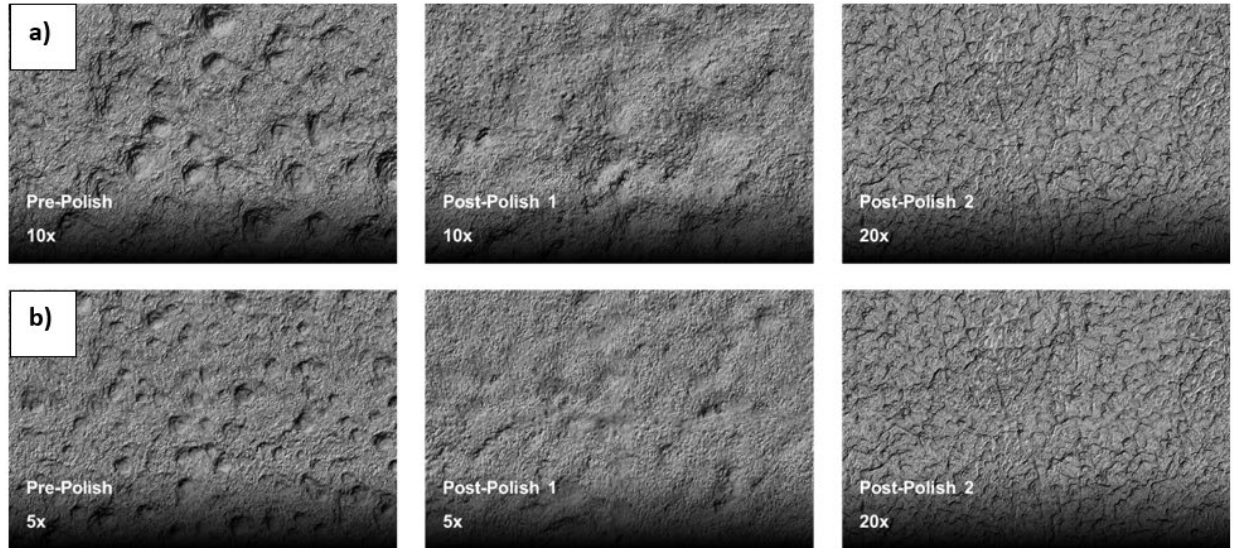


Figure A.9. The a) 10x magnification and b) 5x magnification C-DIC images of the Middle Right section of Side 1 of the U-10Mo plate before and after two electropolishes. The Post-Polish 2 images were taken at the 20x magnification and will not be used for comparison purposes; however, the numerical data indicates a decrease in surface roughness.

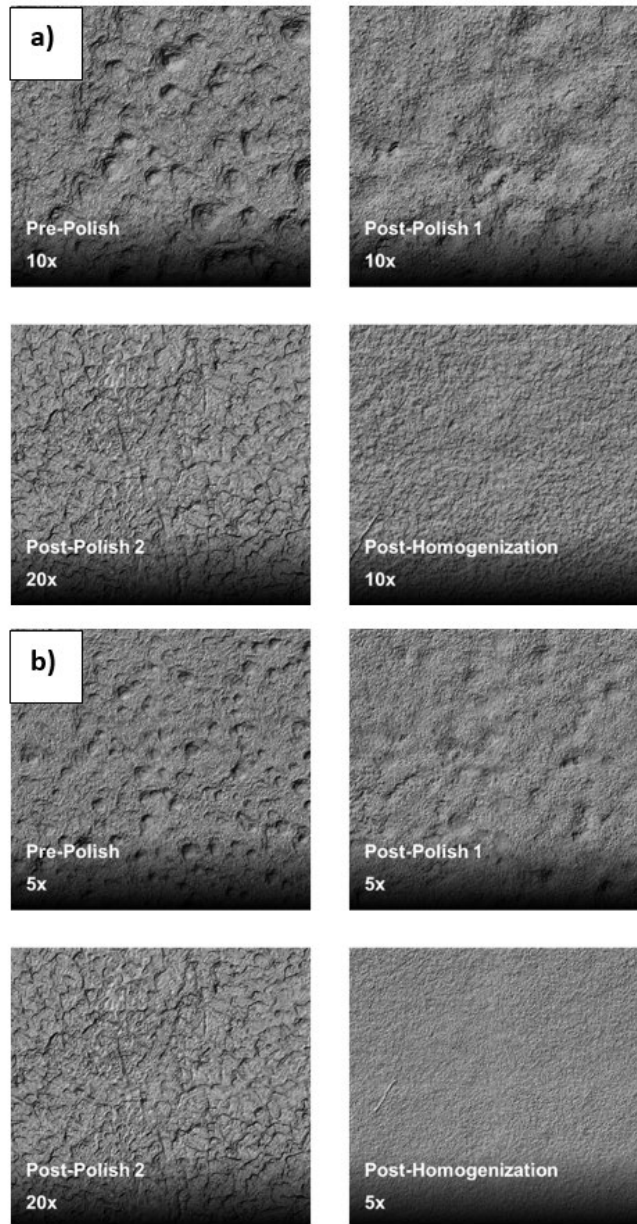


Figure A.10. The a) 10x magnification and b) 5x magnification C-DIC images of the Middle Right section of Side 1 of the U-10Mo plate before and after two electropolishes and a homogenization step. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface of the plate following homogenization had a similar finish to the surface following the first polish. This is supported by the numerical data.

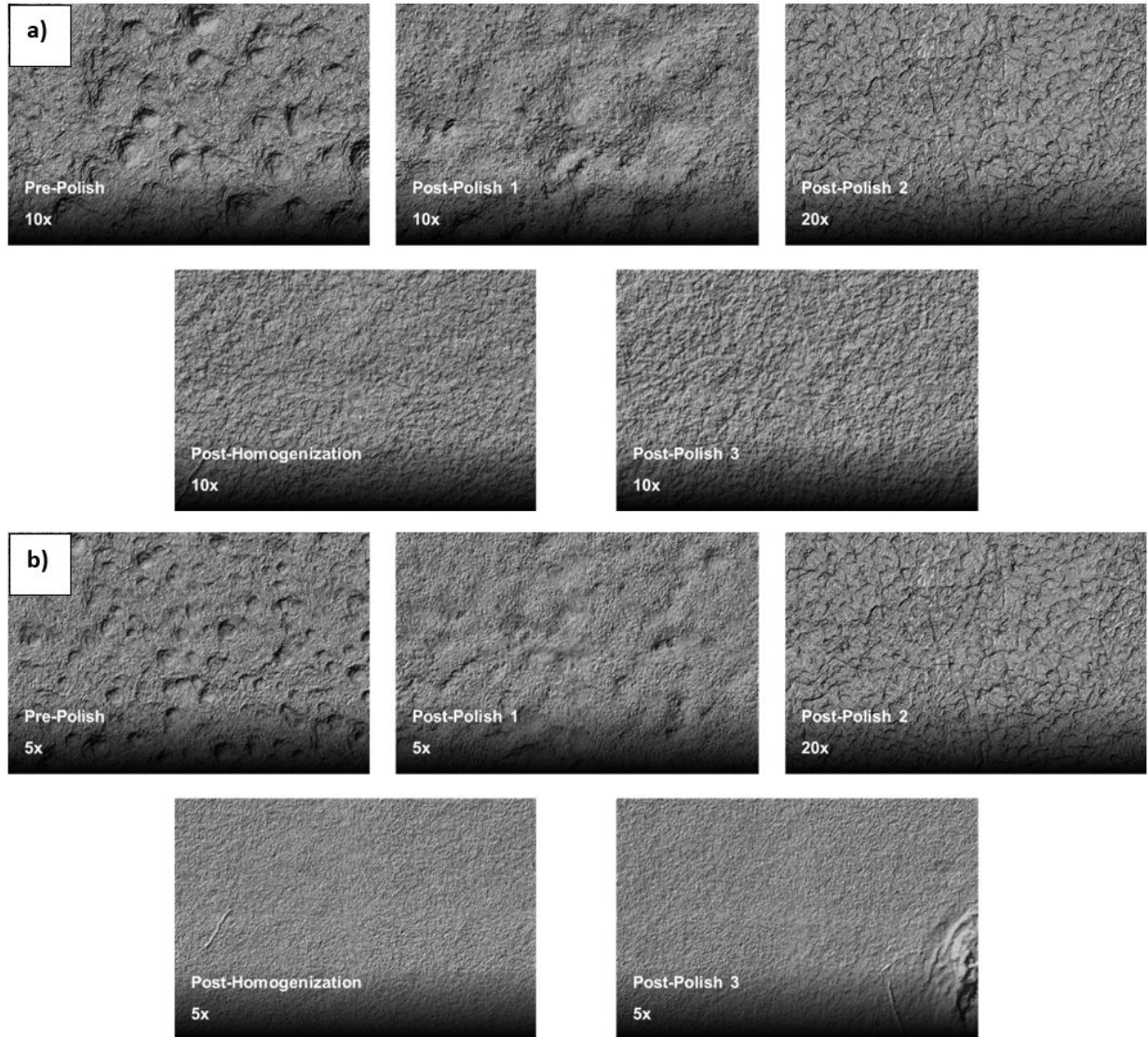


Figure A.11. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Right section of Side 1 of the U-10Mo plate before and after two electropolishes, homogenization, and a third electropolish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for comparison purposes. The surface of the plate remained unchanged after the final polish, which is supported by the numerical data.

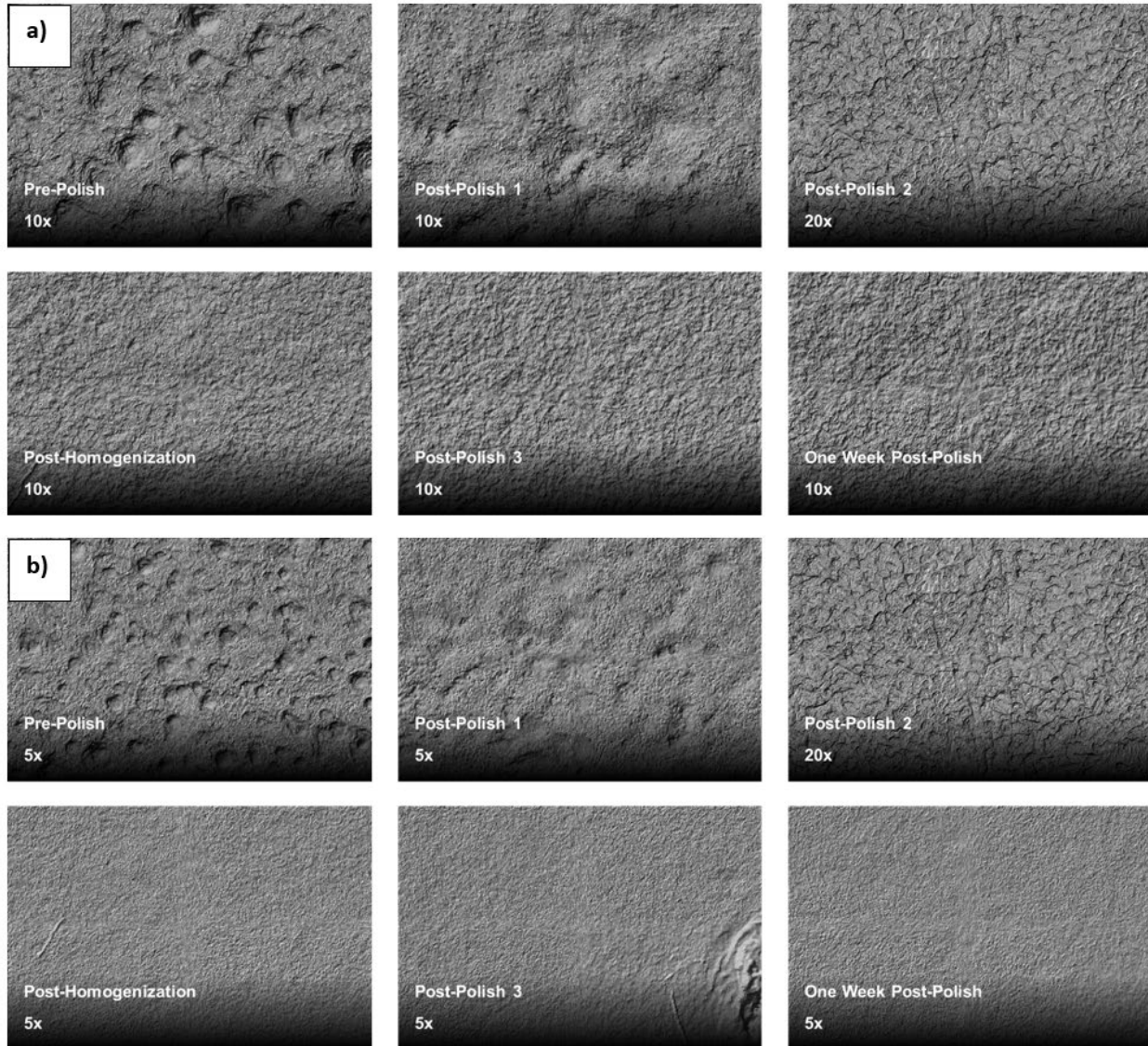


Figure A.12. The a) 10x magnification and b) 5x magnification C-DIC image for the Middle Right section of Side 1 of the U-10Mo plate throughout the electropolishing process and one week after the final polish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for comparison purposes. The surface of the plate remained the same one week after the third polish. This is supported by the surface roughness data.

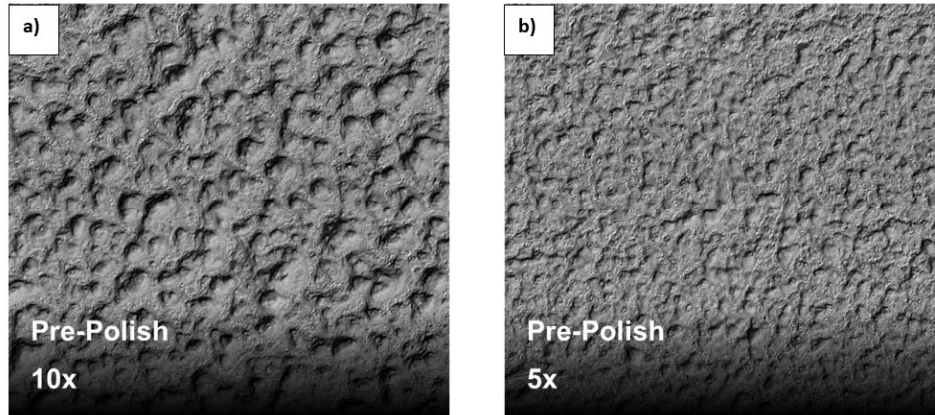


Figure A.13. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Left section of Side 2 of the U-10Mo plate prior to electropolishing.

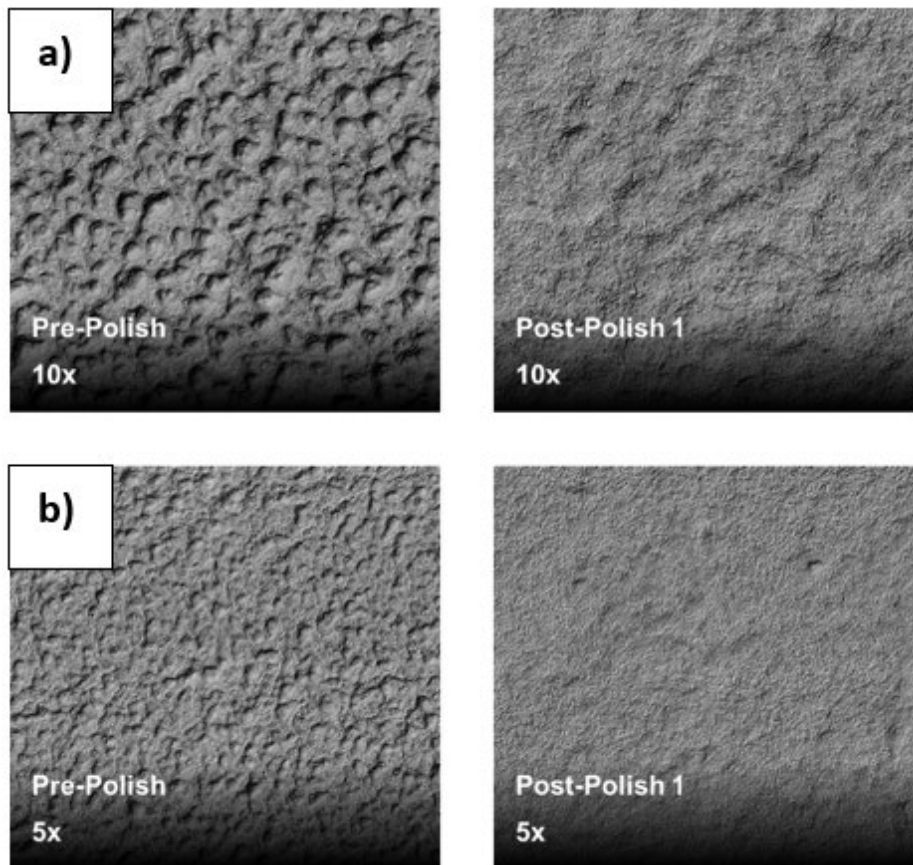


Figure A.14. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Left section of Side 2 of the U-10Mo plate before and after electropolishing. The surface roughness visually decreased following the first electropolish, which is supported by the numerical data.

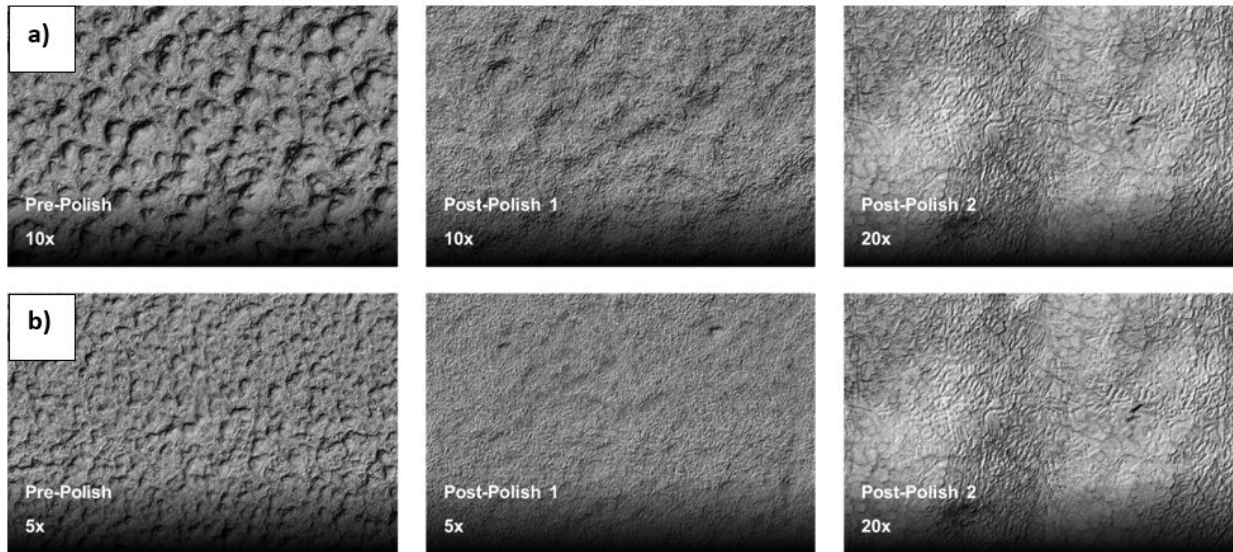


Figure A.15. The a) 10x magnification and b) 5x magnification C-DIC images of the Bottom Left section of Side 2 of the U-10Mo plate before and after two electropolishes. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes; however, numerical data indicates that the surface roughness decreased following the second polish.

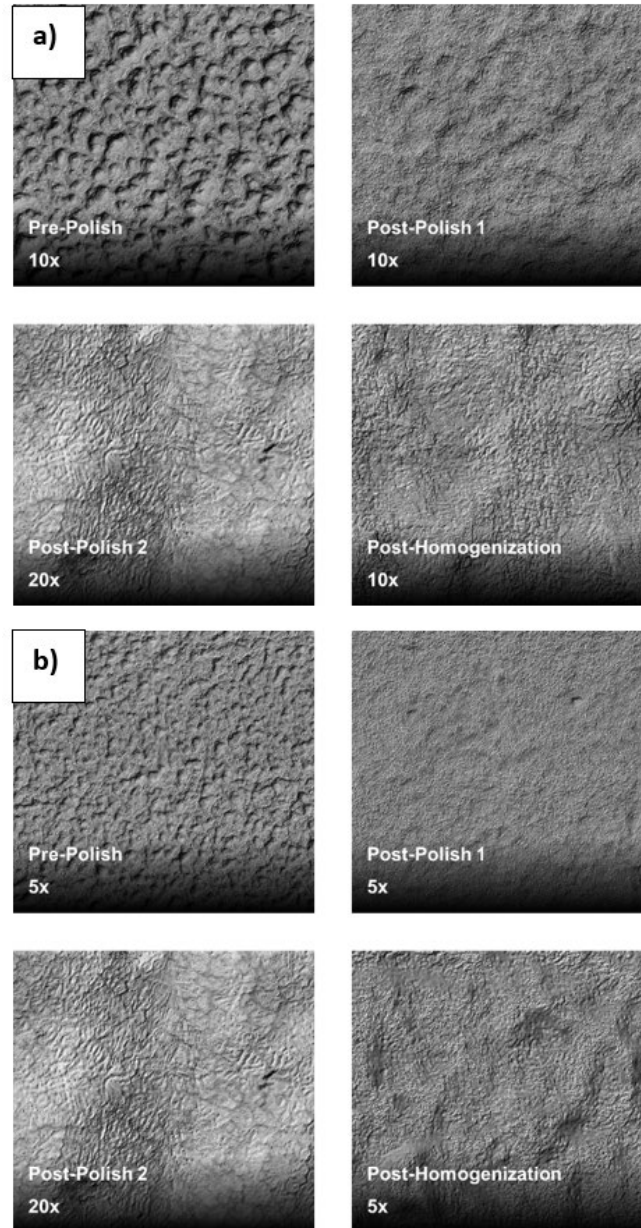


Figure A.16. The a) 10x magnification and b) 5x magnification C-DIC images before and after two electropolishes and homogenization for the Bottom Left section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness of the plate after homogenization appears rougher than that of the plate following the first polish. This is corroborated by the numerical surface roughness data.

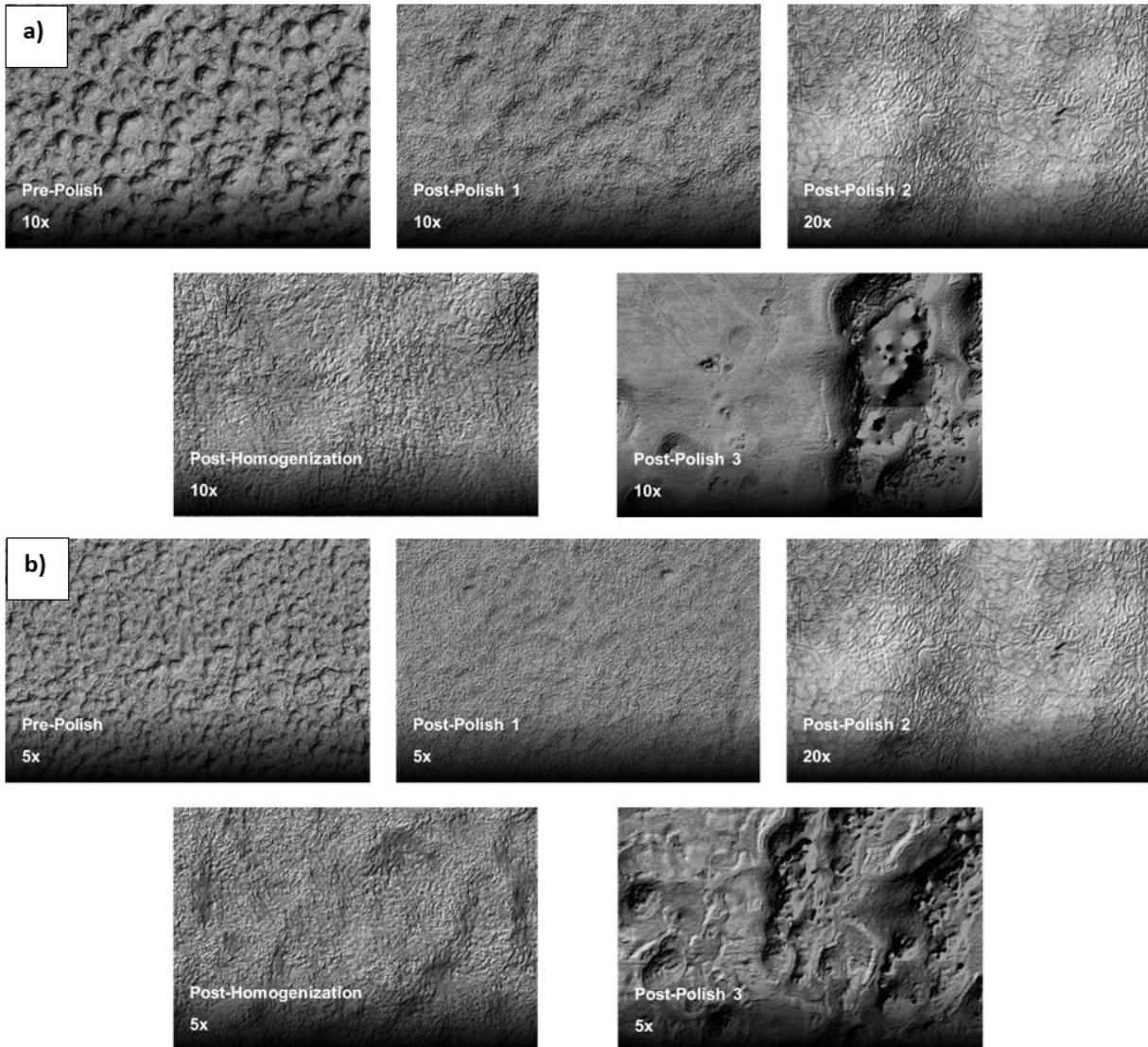


Figure A.17. The a) 10x magnification and b) 5x magnification C-DIC images before and after two electropolishes, homogenization, and a third polish for the Bottom Left section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at 20x and will not be used for visual comparison purposes. The surface of the plate appears rougher following the third polish. This observation is supported by the numerical data.

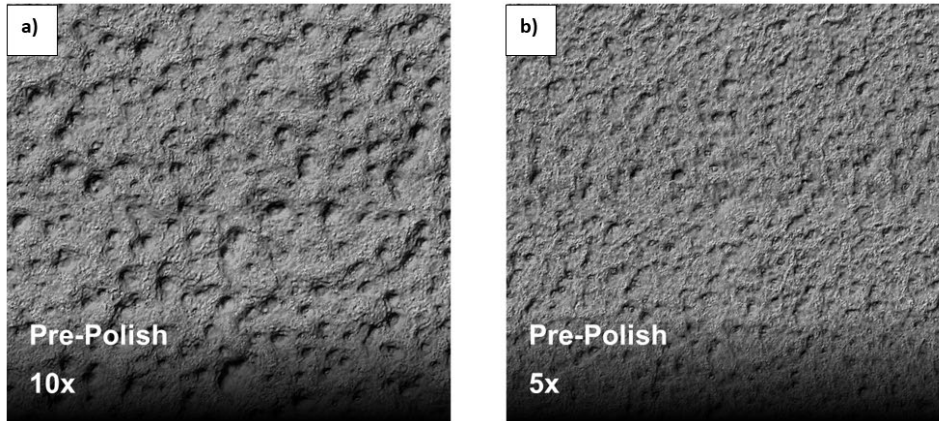


Figure A.18. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Left section of Side 2 of the U-10Mo plate prior to electropolishing.

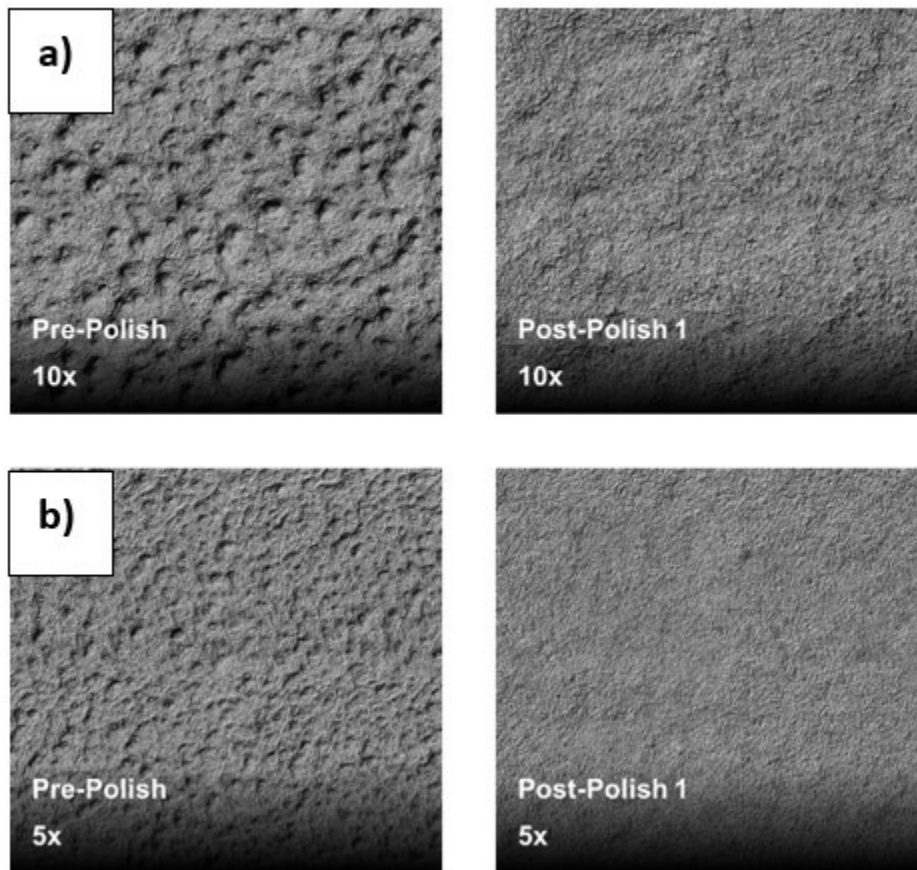


Figure A.19. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Left section of Side 2 of the U-10Mo plate before and after electropolishing. The surface appears visually smoother following the first electropolish. This is corroborated by the numerical data.

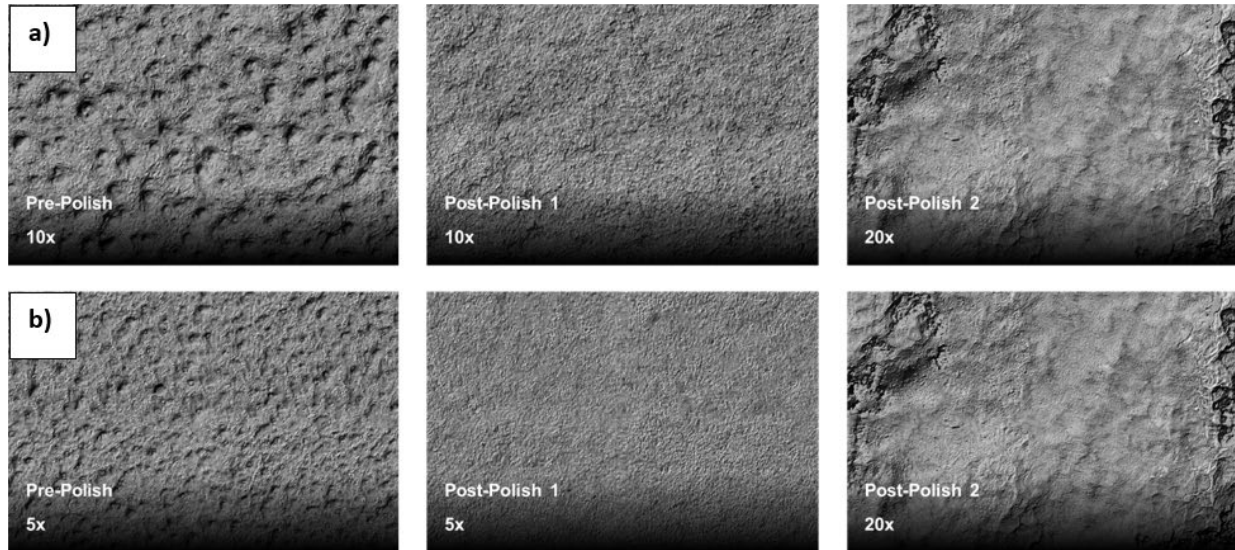


Figure A.20. The a) 10x magnification and b) 5x magnification C-DIC images before and after two electropolishes for the Middle Left section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes; however, the numerical surface roughness data indicates a decrease in surface roughness following the second electropolish.

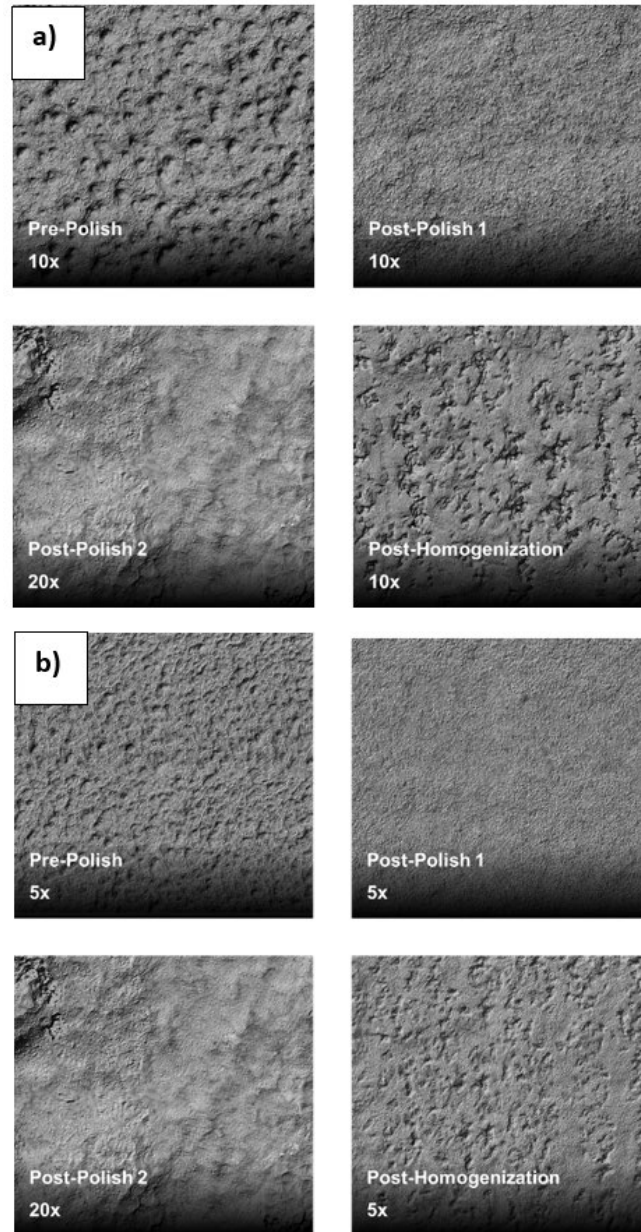


Figure A.21. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Left section of Side 2 of the U-10Mo plate before and after two electropolishes and homogenization. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness visually increased following homogenization. This is corroborated by the numerical surface roughness data.

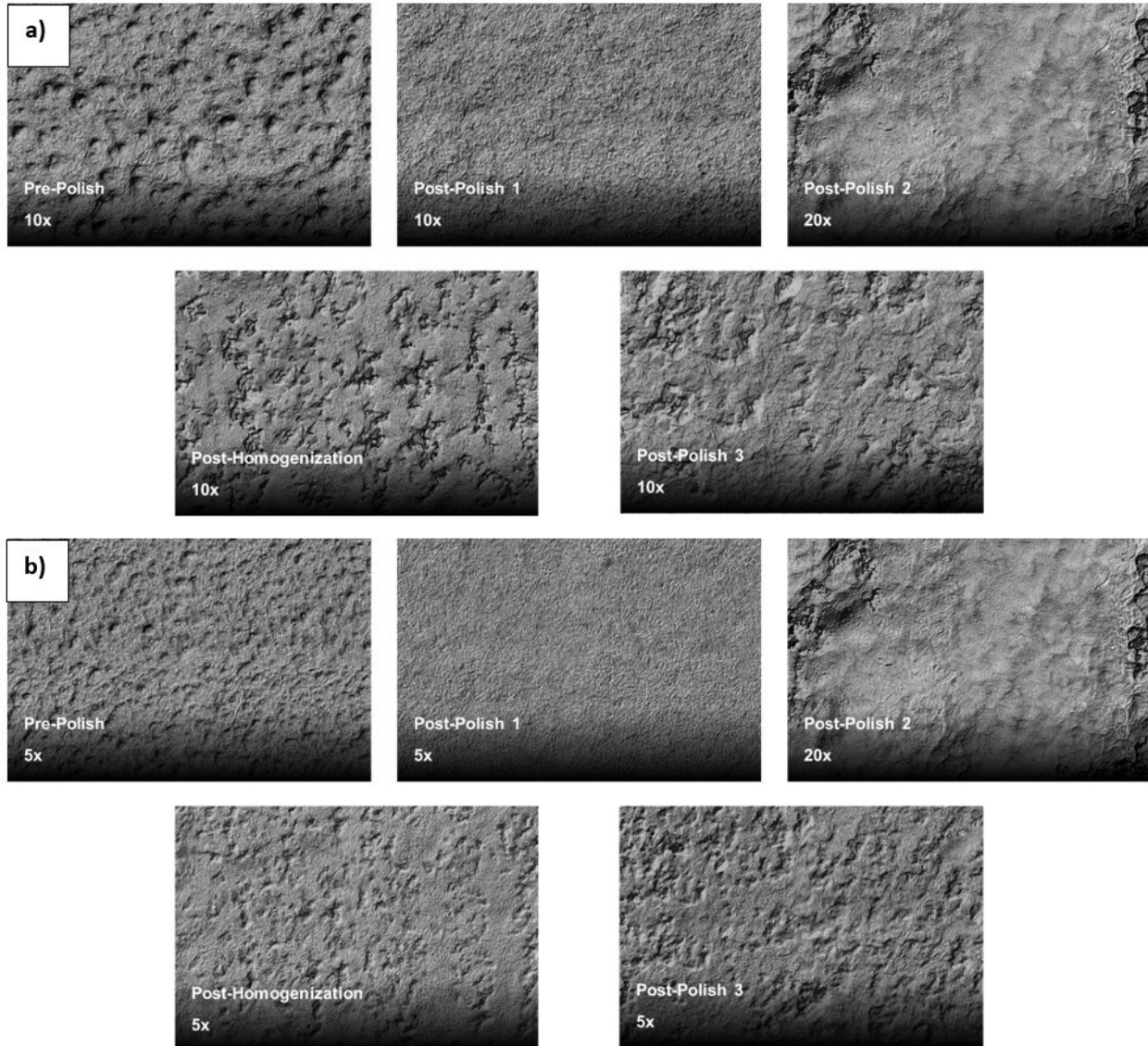


Figure A.22. The a) 10x magnification and b) 5x magnification C-DIC images before and after two electropolishes, homogenization, and a third electropolish for the Middle Left section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface of the plate following the third polish appeared similar to that of the plate after homogenization. This observation is supported by the numerical data.

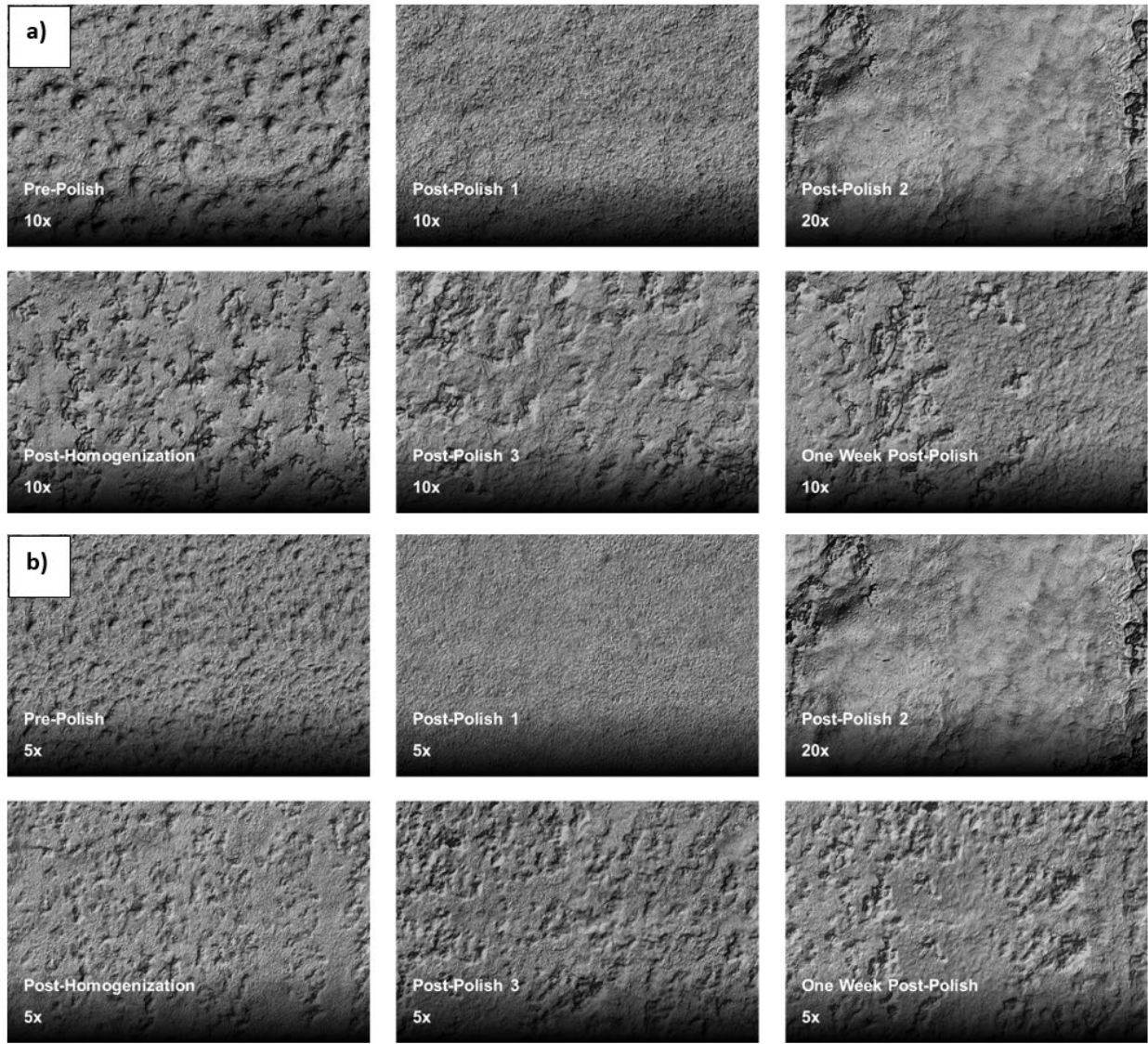


Figure A.23. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Left section of Side 2 of the U-10Mo plate throughout the electropolishing process and one week after the final polish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness appears unchanged one week after polishing. This is corroborated by the surface roughness data.

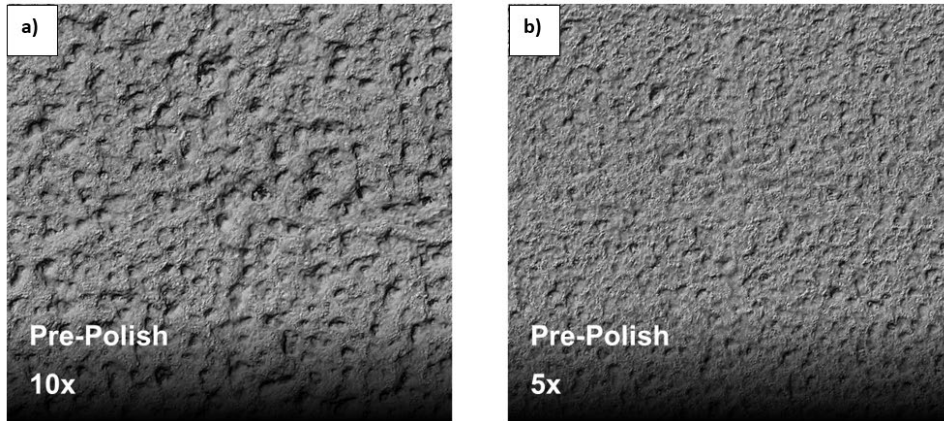


Figure A.24. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 2 of the U-10Mo plate prior to electropolishing.

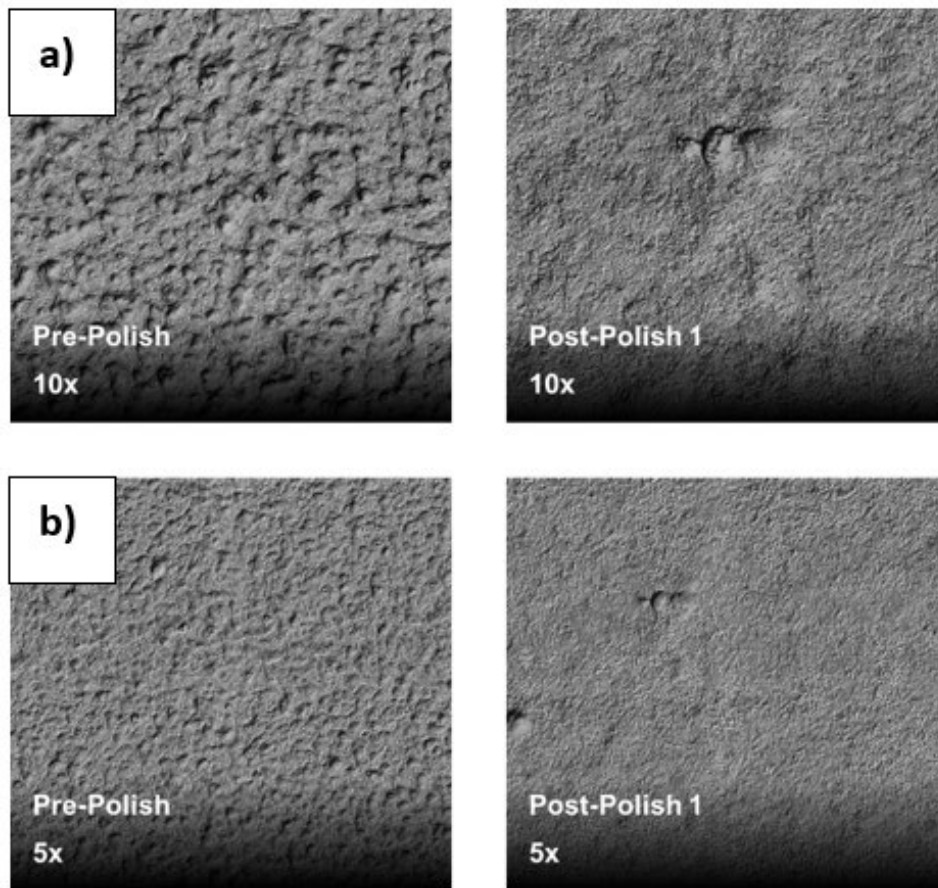


Figure A.25. The a) 10x magnification and b) 5x magnification C-DIC images for the Bottom Right section of Side 2 of the U-10Mo plate before and after electropolishing. The surface roughness appeared to decrease after polishing. This is supported by the numerical data.

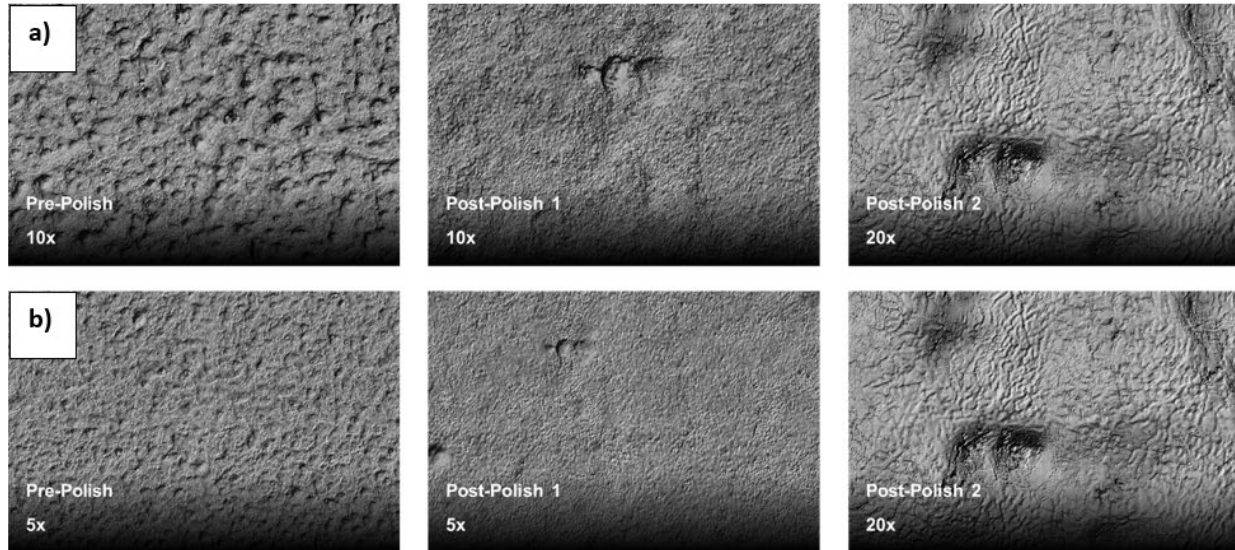


Figure A.26. The a) 10x magnification and b) 5x magnification C-DIC images before and after two electropolishes for the Bottom Right section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes; however the numerical data indicates a decrease in surface roughness following the second polish.

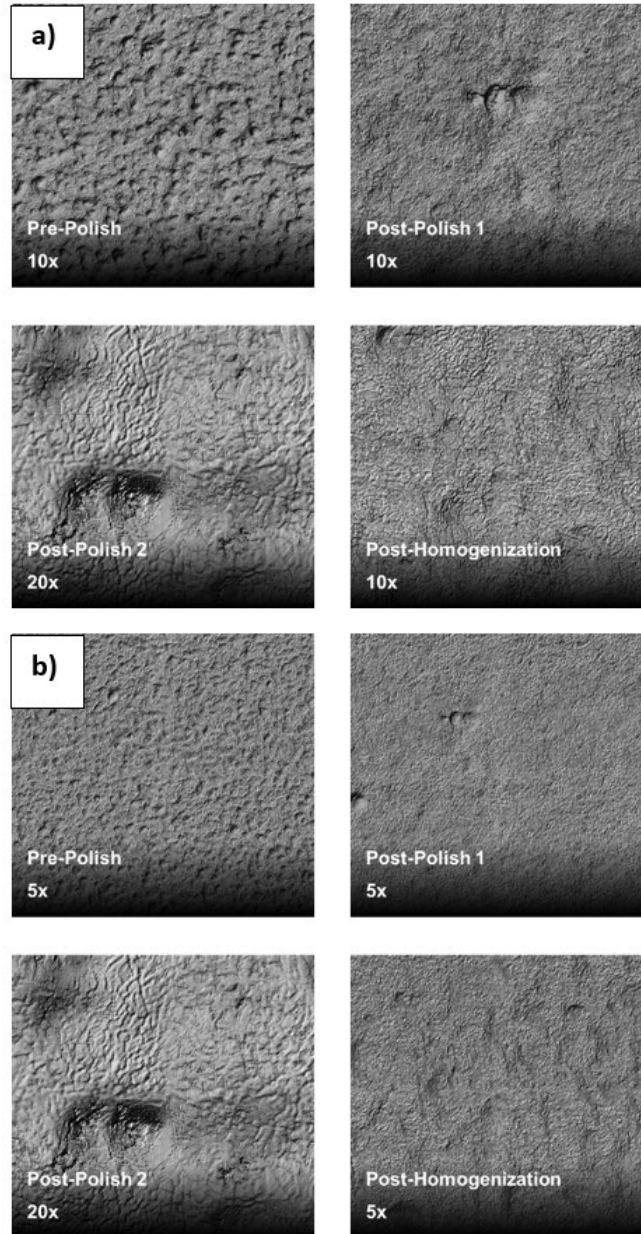


Figure A.27. The a) 10x magnification and b) 5x magnification C-DIC images before and after two electropolishes and homogenization for the Bottom Right section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness of the plate following homogenization appeared like that of the plate following the first polish. This is corroborated by the numerical data.

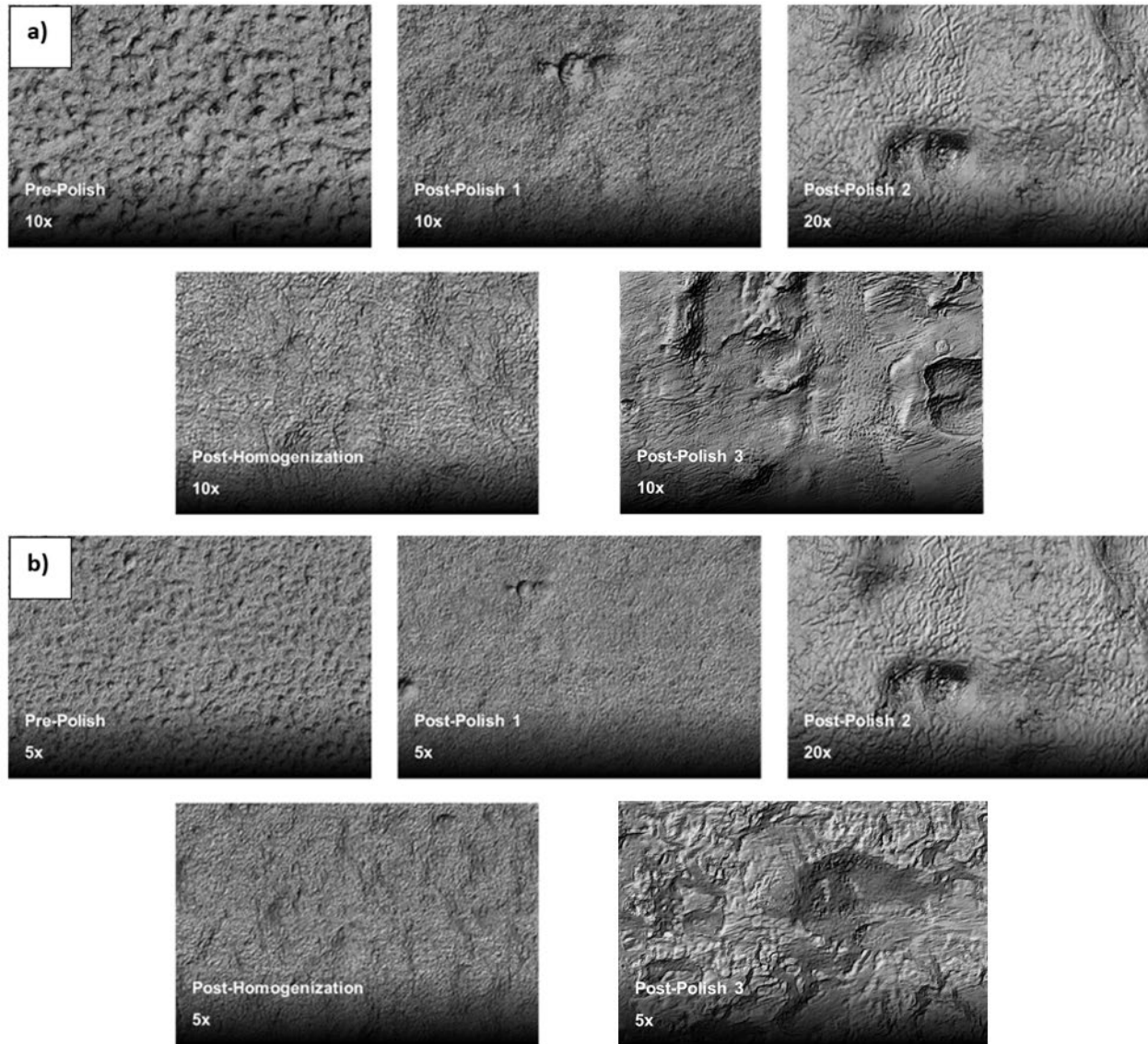


Figure A.28. The a) 10x magnification and b) 5x magnification C-DIC images before and after two polishes, homogenization, and a third polish for the Bottom Right section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness of the plate visually increased following the third polish. This observation is supported by the numerical surface roughness data.

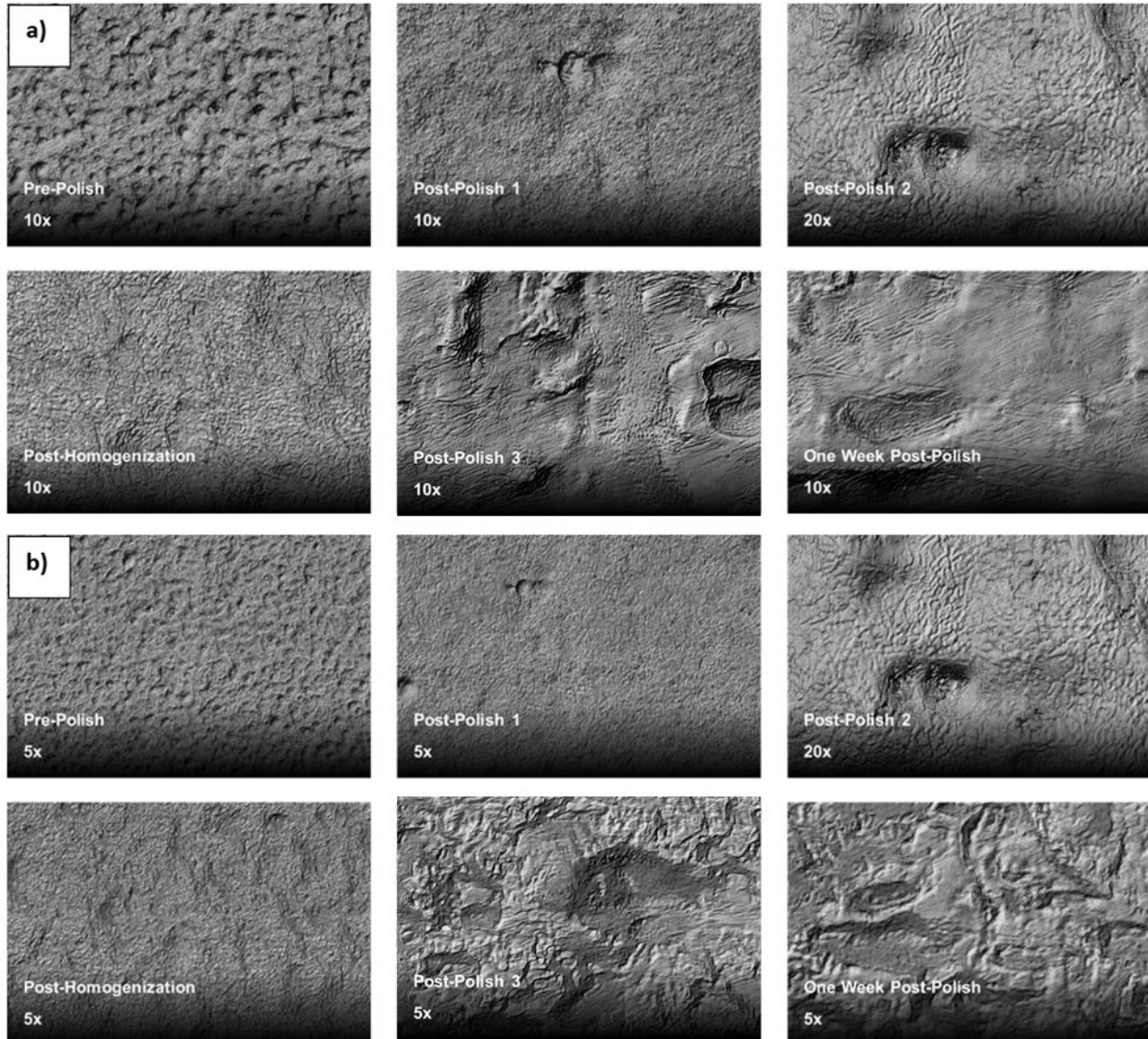


Figure A.29. The a) 10x magnification and b) 5x magnification C-DIC images throughout the polishing process and one week after the final polish for the Bottom Right section of Side 2 of the U-10Mo plate. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface roughness of the plate remained relatively unchanged one week after polishing. This is supported by the numerical data.

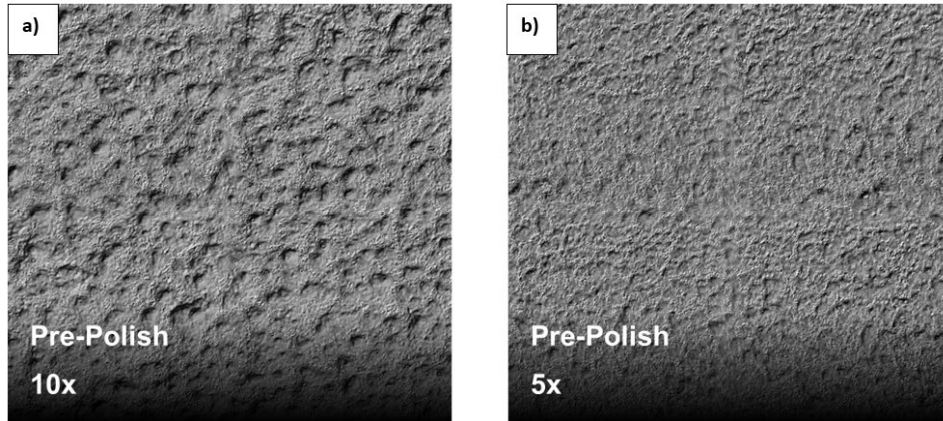


Figure A.30. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Right section of Side 2 of the U-10Mo plate prior to electropolishing.

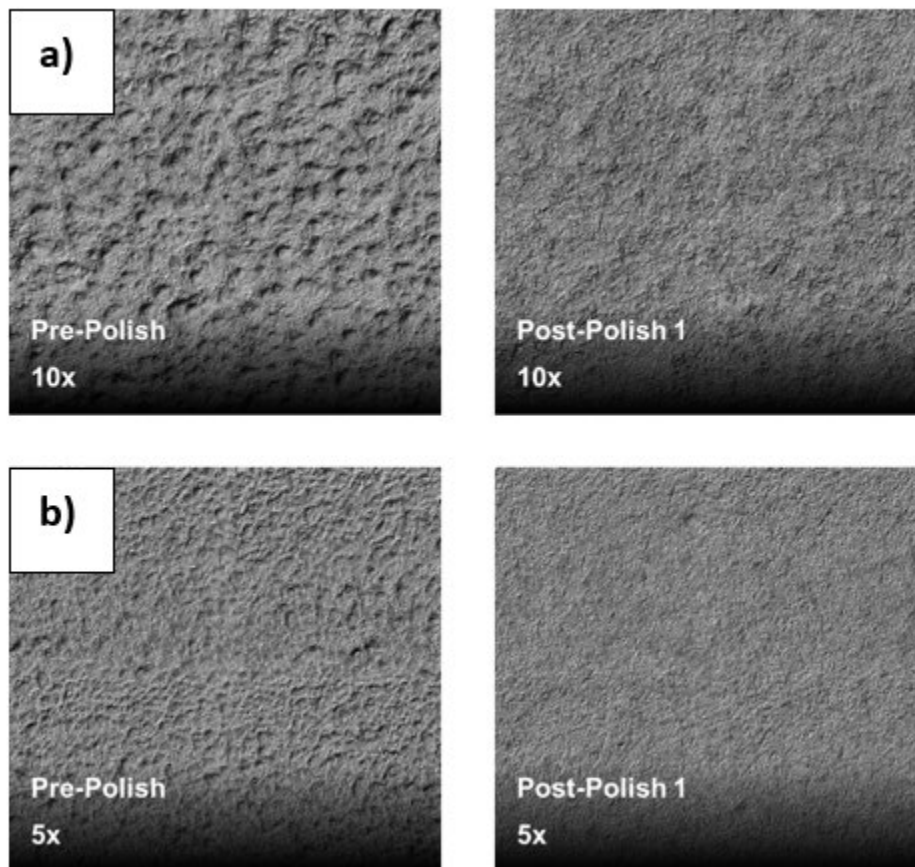


Figure A.31. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Right section of Side 2 of the U-10Mo plate before and after electropolishing. The surface roughness visually decreased following the first polish. This is supported by the numerical data.

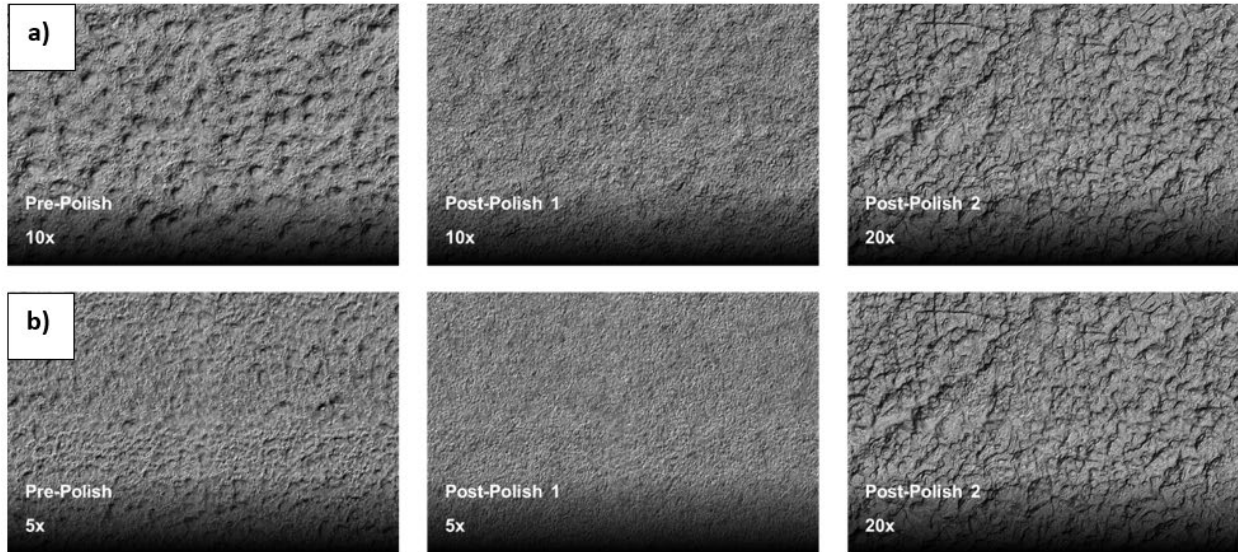


Figure A.32. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Right section of Side 2 of the U-10Mo plate before and after two electropolishes. The Post-Polish 2 images were taken at the 20x magnification and will not be used for comparison purposes; however, the numerical data indicates a decrease in surface roughness following the second polish.

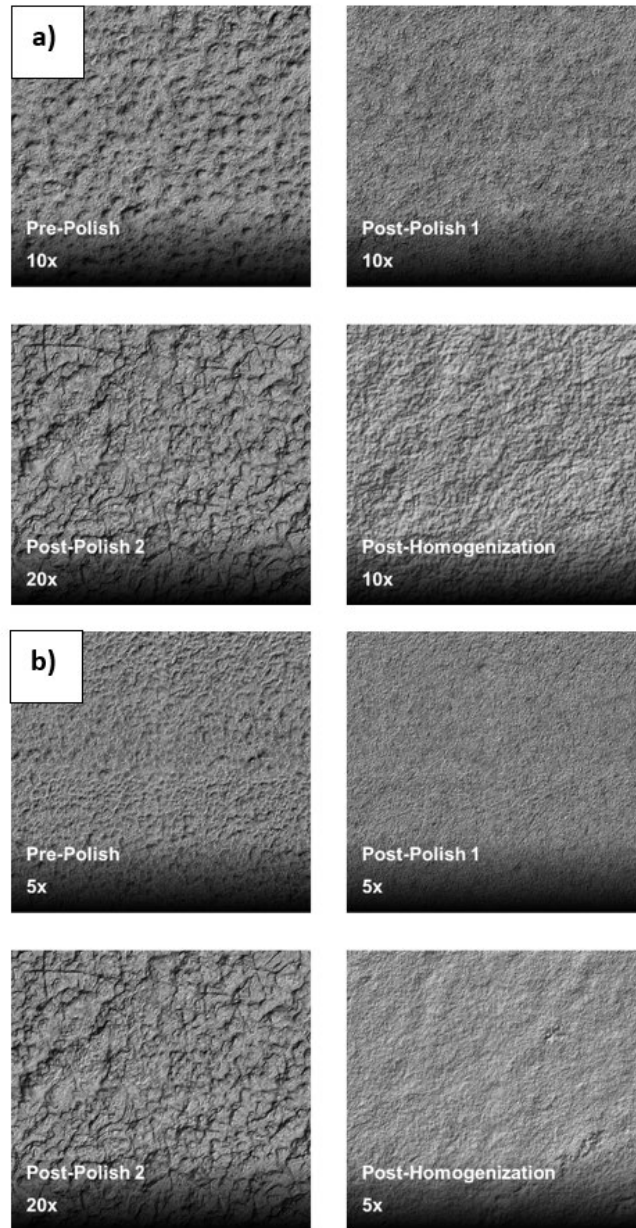


Figure A.33. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Right section of Side 2 of the U-10Mo plate before and after two polishes and homogenization. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface of the plate following homogenization appeared rougher than that of the plate following the first polish. This is supported by the numerical surface roughness data.

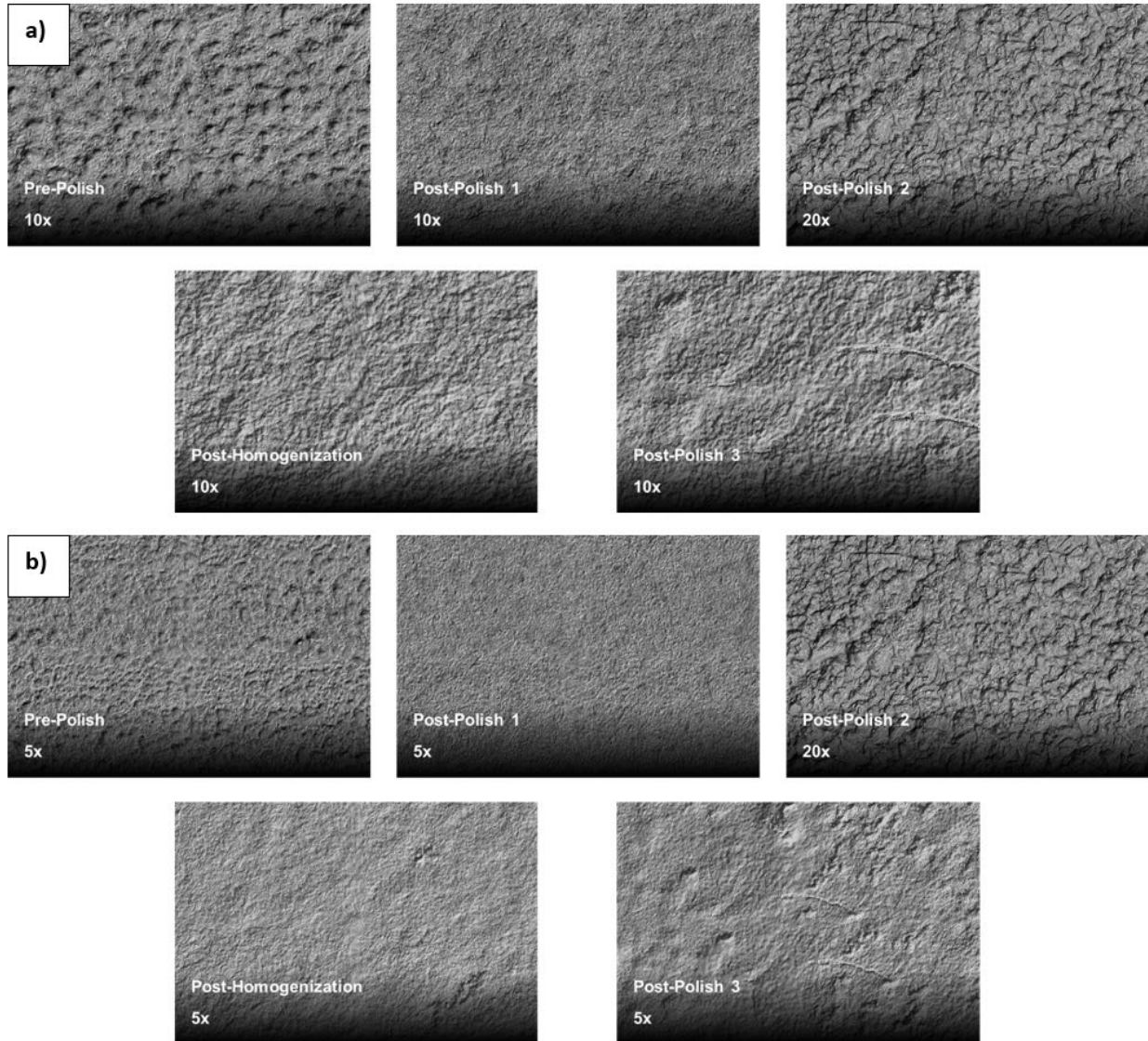


Figure A.34. The a) 10x magnification and b) 5x magnification C-DIC images for the Middle Right section of Side 2 of the U-10Mo plate before and after two polishes, homogenization, and a third polish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for visual comparison purposes. The surface of the plate following the third polish was visually rougher than that of the plate following homogenization. This is corroborated by the numerical data.

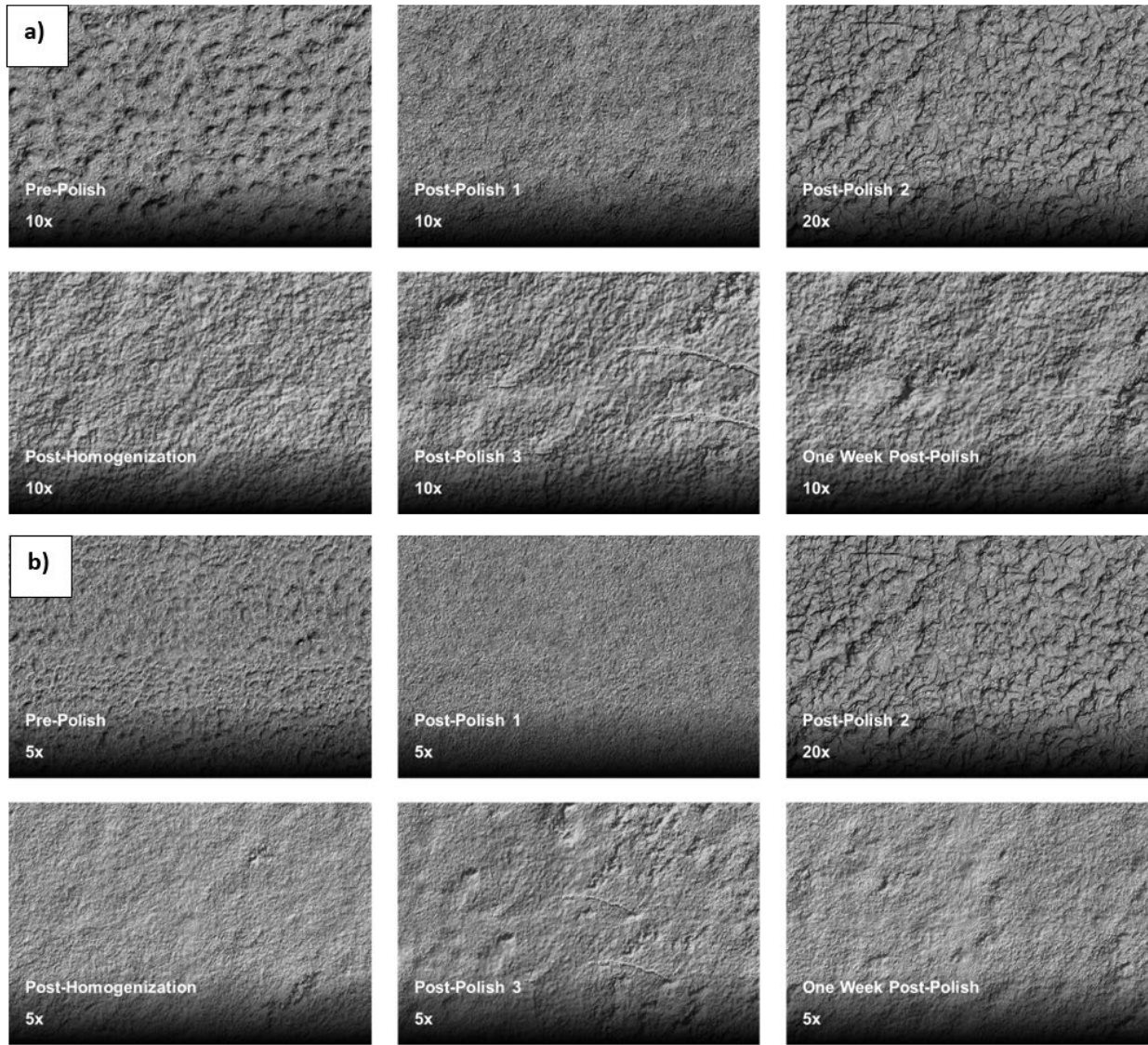


Figure A.35. The a) 10x magnification and b) 5x magnification C-DIC images of the Middle Right section of Side 2 of the U-10Mo plate throughout the polishing process and one week following the final polish. The Post-Polish 2 images were taken at the 20x magnification and will not be used for comparison purposes. The surface of the plate remained relatively unchanged one week after the final polish. This observation is supported by the numerical data.

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