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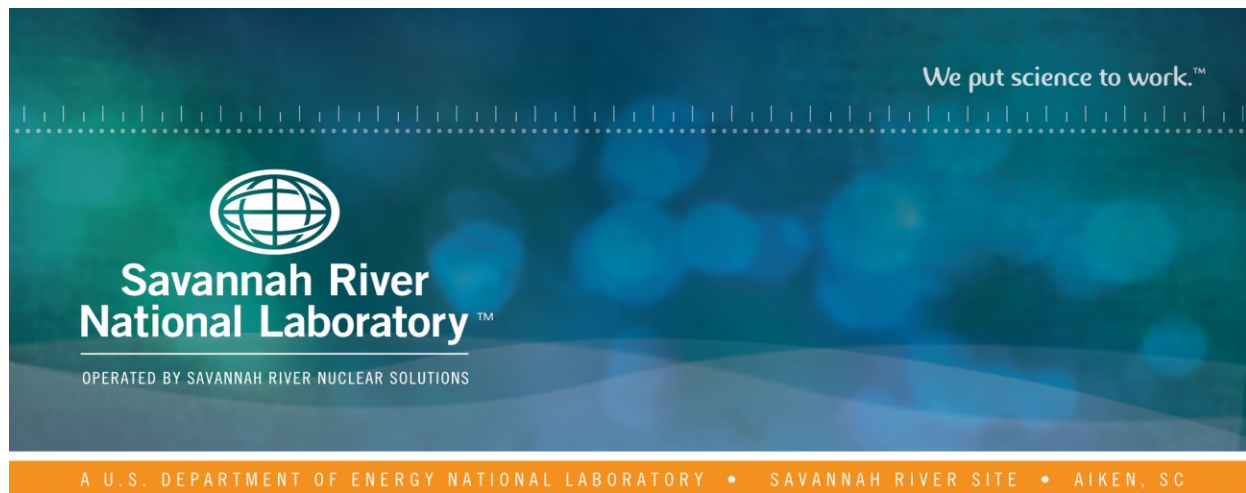
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# **Estimating Effects of Various Factors on Corrosion of Pipeline Grade Carbon Steel Under Disbonded Coating — Progress Report**

**Pavan K. Shukla**

December 2019

SRNL-L5430-2019-00008, Revision 0



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**Retention:** *Permanent*

# Estimating Effects of Various Factors on Corrosion of Pipeline Grade Carbon Steel Under Disbonded Coating — Status Update

Pavan K. Shukla

December 2019

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Prepared for the MESA under  
contract number SPP-2018-020



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## 1.0 Introduction

This report provides description of the activities, associated data and results for a work focused on understanding corrosion of pipeline grade carbon steel under coating disbondment. Specifically, the work reported here was for a pipeline that was installed in 1968 with the following characteristics. The line was coated with polyethylene tape coat (brand Polyken®), and the line joints were electric-flash welded. The line joints are 34-inch outer diameter, and made of API 5L X52 grade carbon steel. The line was initially operated at 55 °F, but subsequently the line temperature increased to 105 °F due to higher fluid flow rate inside the pipe. Due to age and type of the pipeline coating, coating disbondment has occurred at several locations. The increase in operating temperature, coupled with age of the coating, is likely to have resulted in migration in corrosive species from the soil underneath the coating. As a result, the pipeline is experiencing enhanced corrosion. The objective of this study was to evaluate whether the change in operating temperature has affected the corrosion rate of the pipeline external surfaces under areas of disbonded coating.

SNRL is conducting following activities to address the project objectives:

- Measure corrosion properties of API 5L X52 grade carbon steel in soil samples collected from field, and study effect of temperature and bacterial activity on the pipeline steel.
- Develop a corrosion cell experiment to study corrosion under the disbondments, and study effect of temperature increase on corrosion under the disbondments
- Identify the role of temperature on bacterial activity and combined effect of temperature and bacterial activity on corrosion of pipeline material under disbondments
- Conduct several repeat experiments to generate enough data that would statistically bound corrosion rates for a range of temperatures.

Field soil and coating samples were collected. The samples were used to setup various experiments. Three sets of experiments were setup at various temperatures. The experiments included:

- Pipeline grade carbon steel exposed to soil samples
- Pipeline grade carbon steel coupons covered with coating samples, untreated soil water mixture injected in the interspace of the coating and coupons, and moist soil placed on top of the coating
- Pipeline grade carbon steel coupons covered with coating samples, soil water mixture treated to pH 11.5 to simulate cathodic protection, treated soil water mixture injected in the interspace of the coating and coupons, and moist soil placed on top of the coating

The test matrix is detailed in Table A-1 which is in Appendix.

## 2.0 Methods

**Resistivity:** SRNL measured the sand sample resistivities using the four-pin resistivity method in a soil box, as described in ASTM G57 (ASTM International, 2012).

**pH:** SRNL followed EPA Method 9045D (EPA, 2016) to measure the soil sample's pH. As per the method, 20 g of each sand sample was mixed with 20 mL of reagent-grade deionized water and the mixture was stirred for 5 minutes. The mixture was allowed to settle for 1 hour, and then pH of the resulting solution was measured with a pH meter.

**Chloride and sulphate concentrations:** Ion chromatography was used to determine the ionic species concentrations. The leachate solutions developed during pH measurements using the EPA method was used.

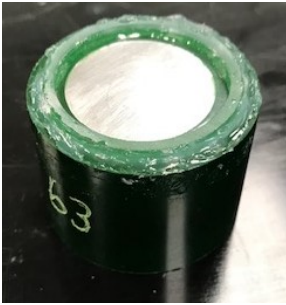
**Bacterial Activity:** Bacterial activity was measured using the BART method which is an alternative to the serial dilution method (NACE International, 2014), and provides same level of information as the dilution method. Details of BART method are available in the book authored by Cullimore (2014) and is available for free download.

**Corrosion of API 5LX52 Carbon Steel Coupons:** Corrosivity of the soil samples towards the carbon steel coupons under coating disbondment was measured by exposing several coupons to the samples. The coupons were made by potting 1 inch diameter disks in a two-part epoxy solution. A holding cup was made for each coupon, with bottom of the cup consisting of the potted coupon. Some coupons' metal surface was covered with the coating samples to simulate coating disbondment; these coupons are referred as coating disbondment coupons hereafter. Soil plus water mixture was injected under the coating in the coating disbondment coupons. Soil samples were placed in each cup, with several coupons at each temperature points. Coupons' details of the are provided in Table A-1. The coupons were placed in several chambers where relative humidities was close to 100%. Images of the coupons and chambers are presented in Figures 1 and 2, respectively.

Exposure duration was six months. The coupons were cleaned of any corrosion products after completions of the tests. Each coupon was analyzed for metal wastage on its surface. The analysis results were used to estimate pitting and surface average corrosion rates for each coupon. Experimental data and results for each sand sample is provided next.

This report only provides data for the coupons where metal was directly exposed to the soil at 22, 28, 33, and 38 °C. The remaining coupons are undergoing exposure and will be extracted in January 2020. This report will be updated after remaining coupons have been extracted and data has been collected.





**(a) Coupon with cavity to place soil-water solution**



**(b) Images of the coupons**

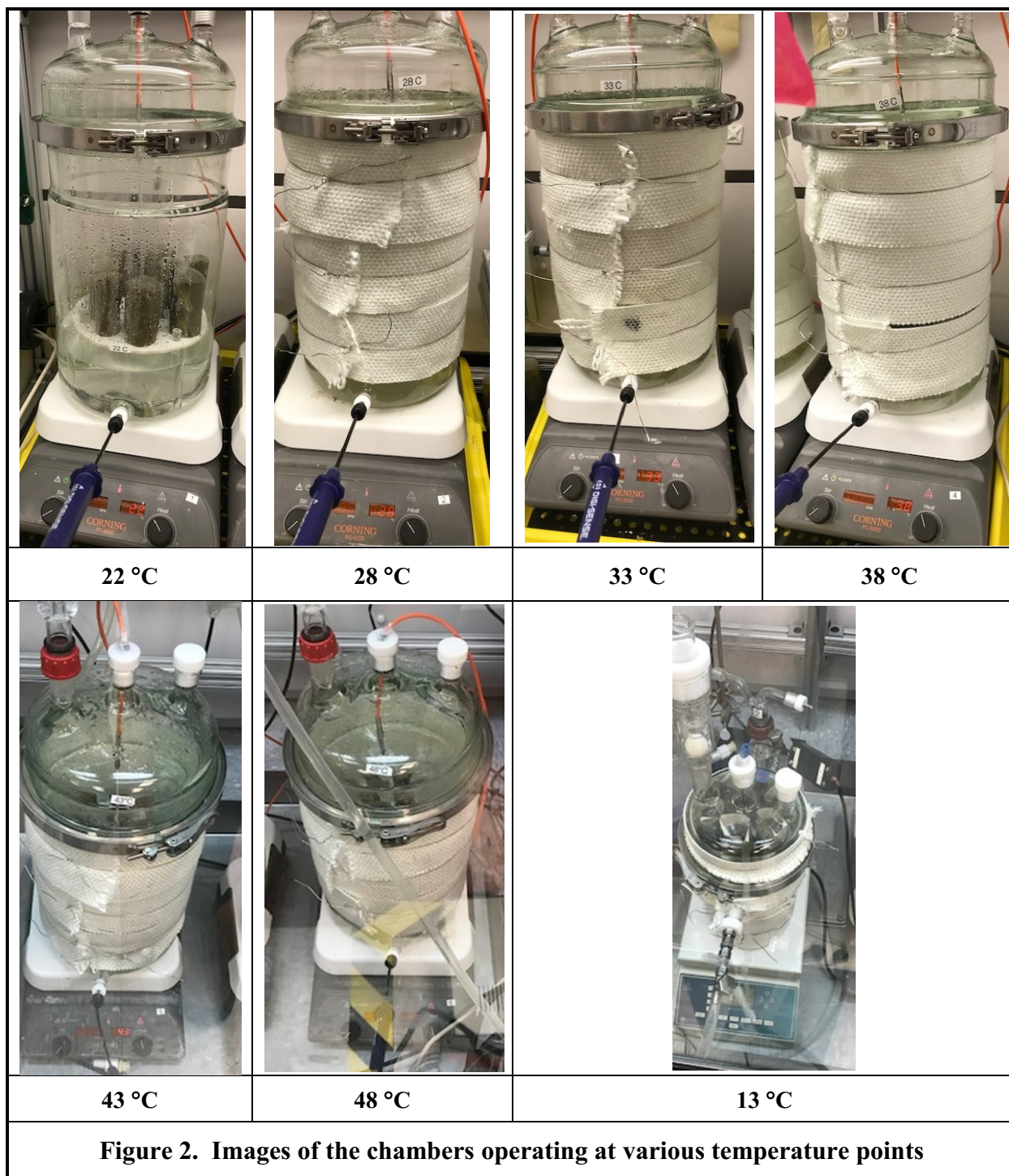


**(c) Polyken tape coat applied to the coupons to simulate a disbonded**



**(d) Coupons placed in a chamber**

**Figure 1. Images of the coupons**



### 3.0 Experimental Data and Results

Resistivity, pH, chloride and sulphate concentrations are listed in Table 1.

<b>Table 1. Resistivity, pH, and chloride and sulphate concentrations in the soil sample</b>					
<b>Sample ID</b>	<b>Resistivity (<math>\Omega</math>-cm)</b>	<b>pH</b>		<b>[Cl<sup>-</sup>] (ppm)</b>	<b>[SO<sub>4</sub><sup>2-</sup>] (ppm)</b>
		<b>Indicator paper</b>	<b>EPA method</b>		
Field Soil Sample	3200	-	7.4	33	23

The bacterial activity levels, i.e., bacteria count in the samples are listed in Table 3.













<b>Table 2. Bacteria count in the sand samples</b>				
<b>Sample ID</b>	<b>Acid Producing Bacteria (CFU/g)</b>	<b>Heterotrophic Aerobic Bacteria (CFU/g)</b>	<b>Sulphate Reducing Bacteria (CFU/g)</b>	<b>Total Bacteria Count (CFU/g)</b>
Field Soil Sample	164000	1150000 (Aerobic)	650 (Anerobic SRB)	1314650

**Corrosion of API 5LX52 Carbon Steel Coupons:** Each coupon's surface was profiled after corrosion product cleaning. Images of the coupons after corrosion product cleaning is in Table 3. Each coupon was also profiled using a visible-light based telescope which processed the profiled data and generated a color map of the surface, with red being the original surface and blue representing the deepest pit. The color map of each coupon's surface is presented in Table 4. The color map of each coupon surface was further processed and adjusted such that color scale was same for all coupons. The adjusted color maps of each coupon are presented in Table 5. Larger size images of the coupons, compared to the ones presented in Tables 3, 4, and 5, are presented in Appendix. The coupons' mass loss and deepest pit depth data were used to estimate surface average and pitting corrosion rates. The coupon data include deepest pit penetration depths and corresponding corrosion rates. The surface average and pitting corrosion rates for each coupon are listed in Tables 6 and 7, respectively. Following observations are made using the listed data in Tables 6 and 7

- Most mass loss occurred in the 33 °C coupons, and surface average corrosion rates are highest for the coupons maintained at 33 °C
- Deepest pits occurred on the coupons maintained at 22 °C. The average pitting corrosion rate for the 22 °C coupons is approximately 22 mpy.



**Table 3. Coupons' Images After Exposure**

Temperature (°C)	Coupons								
22									
28									
33									
38									

**Table 4. Color Map of Coupons' Surfaces with Red Representing Original Surface and Blue Being Deepest Pit**

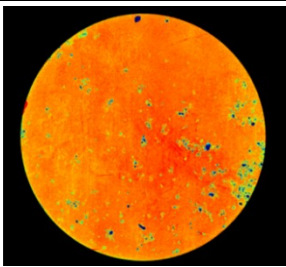
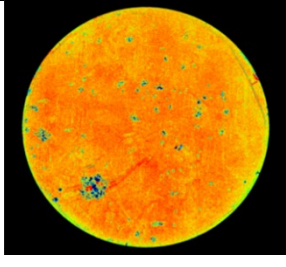
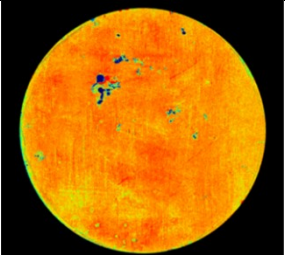
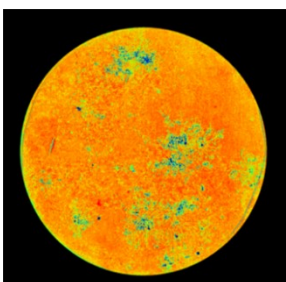
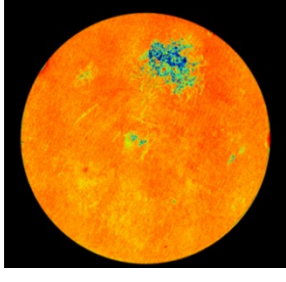
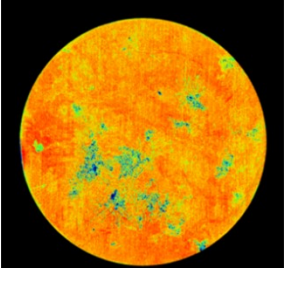
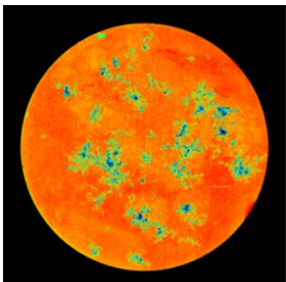
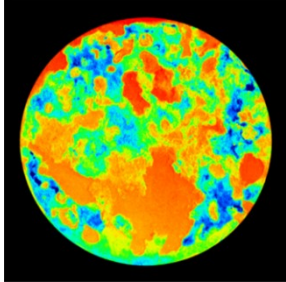
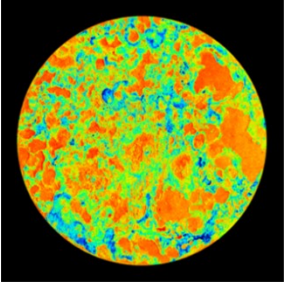
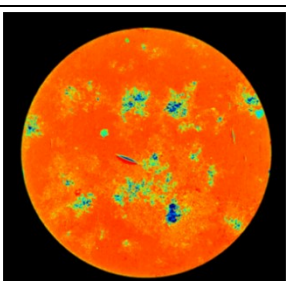
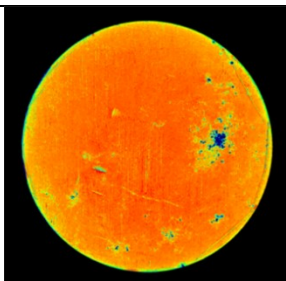
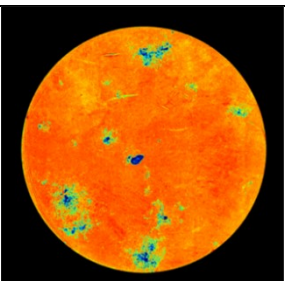



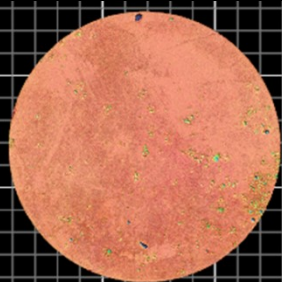
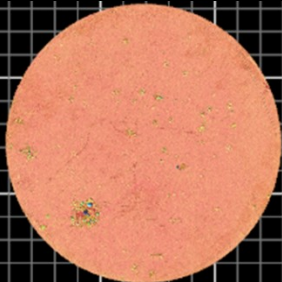
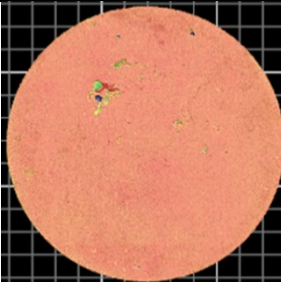
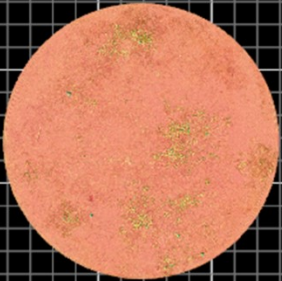
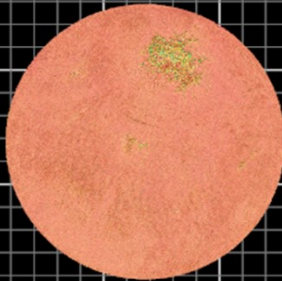
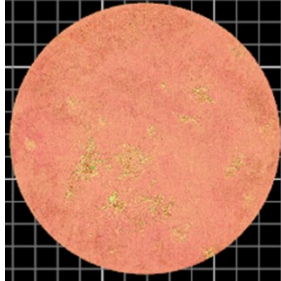
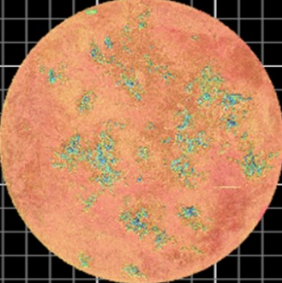
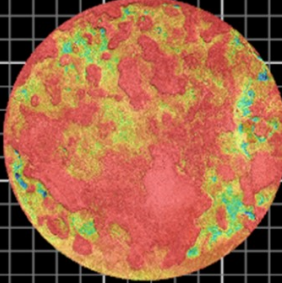
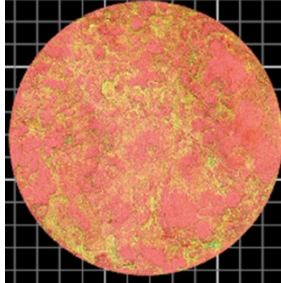
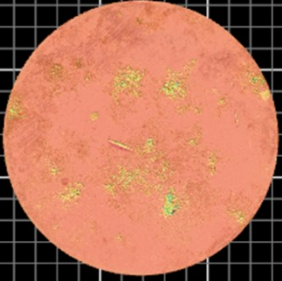
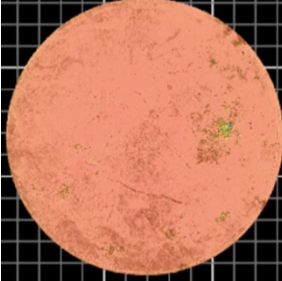
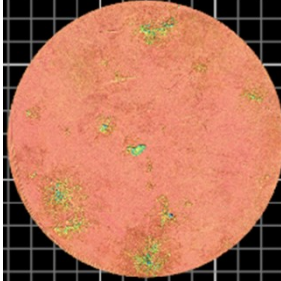
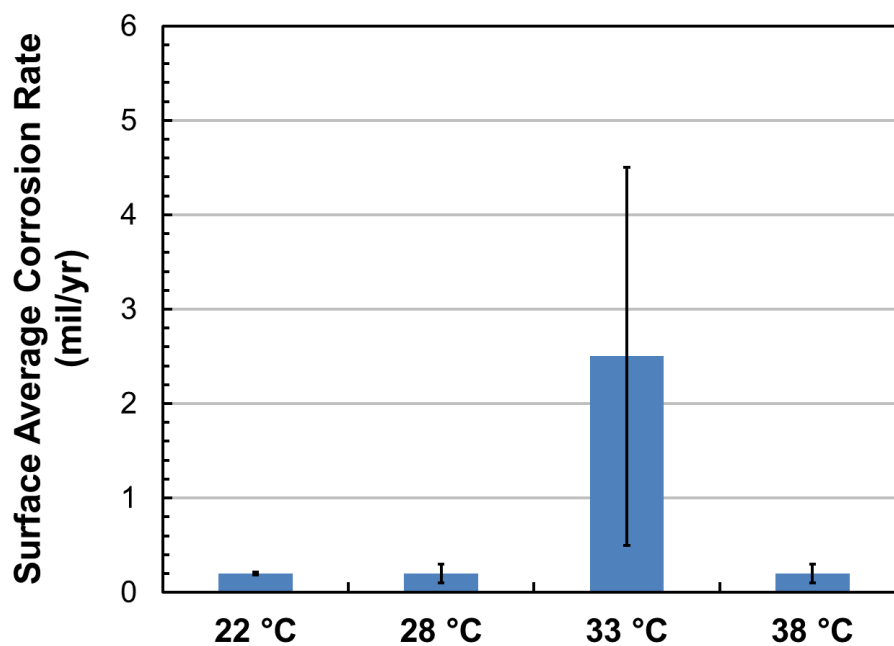
Table 4. Color Map of Coupons' Surfaces with Red Representing Original Surface and Blue Being Deepest Pit						
Temperature (°C)	Coupons					
22						
						
28						
						
33						
38						



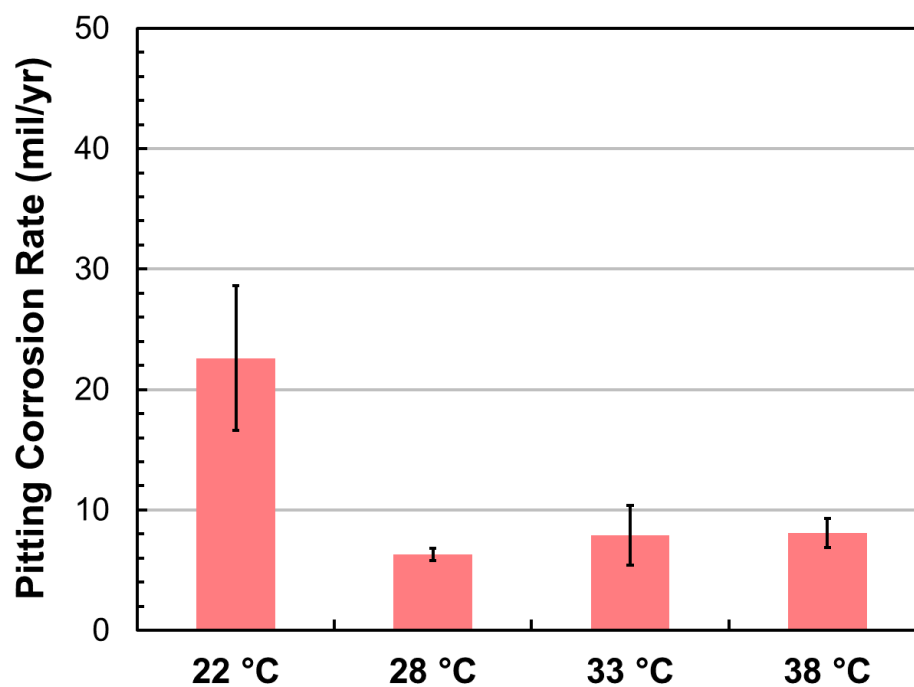
Table 5. Uniform Scaled Color Map of Coupons' Surfaces									
Temperature (°C)	Coupons								
22									
28									
33									
38									

<b>Table 6. Surface Average Corrosion Rates of Coupons</b>								
<b>Coupon ID</b>	<b>Temperature</b>							
	<b>22 °C</b>		<b>28 °C</b>		<b>33 °C</b>		<b>38 °C</b>	
	<b>Mass Loss (mg)</b>	<b>Corrosion Rate (mpy)</b>	<b>Mass Loss (mg)</b>	<b>Corrosion Rate (mpy)</b>	<b>Mass Loss (mg)</b>	<b>Corrosion Rate (mpy)</b>	<b>Mass Loss (mg)</b>	<b>Corrosion Rate (mpy)</b>
<b>Coupon 1</b>	10	0.20	12.2	0.24	16.8	0.33	15.9	0.31
<b>Coupon 2</b>	8.2	0.16	18.0	0.36	214.7	4.23	6.0	0.12
<b>Coupon 3</b>	8.8	0.17	6.7	0.13	141.9	2.80	14.4	0.28
	<b>Average ± std</b>	<b>0.2 ± 0.02</b>	<b>Average ± std</b>	<b>0.2 ± 0.1</b>	<b>Average ± std</b>	<b>2.5 ± 2</b>	<b>Average ± std</b>	<b>0.2 ± 0.1</b>

<b>Table 7. Maximum Pit Depths and Pitting Corrosion Rates of Coupons</b>								
<b>Coupon ID</b>	<b>Temperature</b>							
	<b>22 °C</b>		<b>28 °C</b>		<b>33 °C</b>		<b>38 °C</b>	
	<b>Pit depth (µm)</b>	<b>Corrosion Rate (mpy)</b>	<b>Pit depth (µm)</b>	<b>Corrosion Rate (mpy)</b>	<b>Pit depth (µm)</b>	<b>Corrosion Rate (mpy)</b>	<b>Pit depth (µm)</b>	<b>Corrosion Rate (mpy)</b>
<b>Coupon 1</b>	348	27.3	86	6.8	103	8.1	119	9.3
<b>Coupon 2</b>	203	15.9	80	6.3	131	10.3	88	6.9
<b>Coupon 3</b>	314	24.7	74	5.8	68	5.3	104	8.2
	<b>Average ± std</b>	<b>22.6 ± 6</b>	<b>Average ± std</b>	<b>6.3 ± 0.5</b>	<b>Average ± std</b>	<b>7.9 ± 2.5</b>	<b>Average ± std</b>	<b>8.1 ± 1.2</b>



(a) Surface Average Corrosion Rates



(b) Pitting Corrosion Rates

Figure 3. (a) Surface average, and (b) pitting corrosion rates of the coupons



#### 4.0 Summary

Coupons are being exposed to soil and soil plus water mixture at various temperature points in several chambers. The data so far indicate no specific trend, but it appears that most extensive corrosion occurred at 33 °C, whereas pitting corrosion rates were highest at 22 °C. One plausible explanation is following: at lower temperature of 22 °C, soil moisture level was maintained for longer period than at higher temperatures of 28, 33, and 38 °C, this enabled more intense corrosion to occur for longer period.

Regarding more extensive corrosion at 33 °C, it is well known that corrosion increases with temperature. However, in the case of soil, the competition between the loss of water in soil and temperature-dependent corrosion rate is a plausible reason for the extent of corrosion peaking at 33 °C.

#### 5.0 References

1. ASTM International. "Standard Test Method for Field Measurement of Soil Resistivity Using the Wenner Four-Electrode Method." ASTM G57-06. 2012.
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3. NACE International. "Standard Test Method Field Monitoring of Bacteria Growth in Oil and Gas Systems." TM0194-2014. Houston, Texas: NACE International. 2014.
4. Cullimore, D.R., Standard Methods for BART Testers, 4<sup>th</sup> Ed.,  
<https://www.dbi.ca/StandMeth/StandardMethod-4th.pdf>, 2014.

## **Appendix**

Table A-8. Experimental Test matrix

Table A-8. Experimental Test matrix									
Vessel Temperature (°C)	Treatment	Coupon ID	Weight (g)	Weigh coupon cold mount with tape (g)	Weigh coupon with soil solution (g)	Weigh coupon with soil (g)	Weigh coupon with extra water (g)	Coupon Placement Date	Coupon Extraction Date
13	No treatment	65	76.1790	107.0100	N/A	147.07	149.06	Wednesday, July 24, 2019	Friday, January 24, 2020
		66	76.2223	105.5800	N/A	145.51	147.51	Wednesday, July 24, 2019	Friday, January 24, 2020
		67	76.0879	101.8400	N/A	141.87	143.88	Wednesday, July 24, 2019	Friday, January 24, 2020
	Coating with injected liquid natural soil pH	55	76.0631	107.2700	108.33	148.37	150.36	Wednesday, July 24, 2019	Friday, January 24, 2020
		56	76.2088	104.5700	105.79	145.76	147.75	Wednesday, July 24, 2019	Friday, January 24, 2020
		60	76.2028	106.3800	107.61	147.63	149.66	Wednesday, July 24, 2019	Friday, January 24, 2020
	Coating with injected liquid pH 11.5	61	76.1997	105.3800	106.5	146.59	148.62	Wednesday, July 24, 2019	Friday, January 24, 2020
		62	76.2021	105.8300	106.99	147.02	149.06	Wednesday, July 24, 2019	Friday, January 24, 2020
		64	76.1232	106.1200	107.3	147.33	149.36	Wednesday, July 24, 2019	Friday, January 24, 2020
	22	No treatment	2	76.1795	126.9500	N/A	124.334	125.95	Wednesday, June 12, 2019
73			76.0603	88.3325	N/A	128.38	130.38	Wednesday, June 12, 2019	Thursday, December 12, 2019
3			76.0193	87.2700	N/A	127.24	129.27	Wednesday, June 12, 2019	Thursday, December 12, 2019

Table A-8. Experimental Test matrix

Vessel Temperature (°C)	Treatment	Coupon ID	Weight (g)	Weigh coupon cold mount with tape (g)	Weigh coupon with soil solution (g)	Weigh coupon with soil (g)	Weigh coupon with extra water (g)	Coupon Placement Date	Coupon Extraction Date
	Coating with injected liquid natural soil pH	5	76.0506	103.7500	104.91	144.92	146.94	Wednesday, July 03, 2019	Friday, January 03, 2020
		4	76.1801	104.9300	105.53	145.59	147.48	Wednesday, July 03, 2019	Friday, January 03, 2020
		1	76.2069	107.7600	108.9	143.98	150.97	Wednesday, July 03, 2019	Friday, January 03, 2020
	Coating with injected liquid pH 11.5	6	76.0450	104.9800	106.15	146.14	148.15	Wednesday, July 03, 2019	Friday, January 03, 2020
		7	76.0642	106.9600	108.22	148.23	150.25	Wednesday, July 03, 2019	Friday, January 03, 2020
		8	76.2142	106.0300	107.21	147.18	149.17	Wednesday, July 03, 2019	Friday, January 03, 2020
28	No treatment	11	76.2110	85.0400	N/A	125.04	127.05	Wednesday, June 12, 2019	Thursday, December 12, 2019
		12	76.0888	86.2200	N/A	126.24	128.25	Wednesday, June 12, 2019	Thursday, December 12, 2019
		13	76.2126	85.4000	N/A	125.4	127.41	Wednesday, June 12, 2019	Thursday, December 12, 2019
	Coating with injected liquid natural soil pH	9	76.1721	105.9600	107.03	147.17	149.19	Wednesday, July 03, 2019	Friday, January 03, 2020
		14	76.0480	106.3000	107.43	147.49	149.48	Wednesday, July 03, 2019	Friday, January 03, 2020
		10	76.2106	107.2200	108.35	148.42	150.43	Wednesday, July 03, 2019	Friday, January 03, 2020

Table A-8. Experimental Test matrix

Vessel Temperature (°C)	Treatment	Coupon ID	Weight (g)	Weigh coupon cold mount with tape (g)	Weigh coupon with soil solution (g)	Weigh coupon with soil (g)	Weigh coupon with extra water (g)	Coupon Placement Date	Coupon Extraction Date
	<b>Coating with injected liquid pH 11.5</b>	16	76.2028	107.9900	109.22	149.26	151.33	Wednesday, July 03, 2019	Friday, January 03, 2020
		17	76.1877	105.0000	106.23	146.35	148.34	Wednesday, July 03, 2019	Friday, January 03, 2020
		18	76.0587	104.4800	105.65	145.68	147.72	Wednesday, July 03, 2019	Friday, January 03, 2020
33	<b>No treatment</b>	21	76.2109	86.5000	N/A	126.51	128.53	Wednesday, June 12, 2019	Thursday, December 12, 2019
		22	76.0891	86.0800	N/A	126.06	128.06	Wednesday, June 12, 2019	Thursday, December 12, 2019
		23	76.1963	86.5900	N/A	126.59	128.57	Wednesday, June 12, 2019	Thursday, December 12, 2019
	<b>Coating with injected liquid natural soil pH</b>	19	76.0634	106.3300	107.42	147.39	149.37	Wednesday, July 03, 2019	Friday, January 03, 2020
		20	76.0716	105.7900	106.86	146.88	148.84	Wednesday, July 03, 2019	Friday, January 03, 2020
		24	76.2016	106.6900	107.67	147.63	149.63	Wednesday, July 03, 2019	Friday, January 03, 2020
	<b>Coating with injected liquid pH 11.5</b>	25	76.0407	107.5600	108.78	148.84	150.84	Wednesday, July 03, 2019	Friday, January 03, 2020
		26	76.1993	106.0300	107.2	147.27	149.33	Wednesday, July 03, 2019	Friday, January 03, 2020
		27	76.1994	108.9100	110.12	150.12	152.1	Wednesday, July 03, 2019	Friday, January 03, 2020

Table A-8. Experimental Test matrix

Table A-8. Experimental Test matrix									
Vessel Temperature (°C)	Treatment	Coupon ID	Weight (g)	Weigh coupon cold mount with tape (g)	Weigh coupon with soil solution (g)	Weigh coupon with soil (g)	Weigh coupon with extra water (g)	Coupon Placement Date	Coupon Extraction Date
38	No treatment	30	76.0716	86.1100	N/A	126.14	128.14	Wednesday, June 12, 2019	Thursday, December 12, 2019
		31	76.0446	86.9200	N/A	126.91	128.9	Wednesday, June 12, 2019	Thursday, December 12, 2019
		32	76.0642	87.0400	N/A	127.04	129.05	Wednesday, June 12, 2019	Thursday, December 12, 2019
	Coating with injected liquid natural soil pH	28	76.1998	105.8800	106.93	146.98	148.97	Wednesday, July 03, 2019	Friday, January 03, 2020
		29	76.0645	107.1000	108.21	148.2	150.21	Wednesday, July 03, 2019	Friday, January 03, 2020
		33	76.1992	107.3800	108.49	148.54	150.53	Wednesday, July 03, 2019	Friday, January 03, 2020
	Coating with injected liquid pH 11.5	34	76.2341	106.0700	107.17	147.22	149.24	Wednesday, July 03, 2019	Friday, January 03, 2020
		35	76.2098	105.5700	106.76	146.81	148.87	Wednesday, July 03, 2019	Friday, January 03, 2020
		36	76.2095	105.8100	106.9	147.01	149.02	Wednesday, July 03, 2019	Friday, January 03, 2020
	43	No treatment	39	76.0281	86.9900	N/A	126.98	128.95	Friday, July 12, 2019
40			76.2149	88.5700	N/A	128.57	130.58	Friday, July 12, 2019	Sunday, January 12, 2020
41			76.2543	87.4800	N/A	127.48	129.44	Friday, July 12, 2019	Sunday, January 12, 2020

Table A-8. Experimental Test matrix

Vessel Temperature (°C)	Treatment	Coupon ID	Weight (g)	Weigh coupon cold mount with tape (g)	Weigh coupon with soil solution (g)	Weigh coupon with soil (g)	Weigh coupon with extra water (g)	Coupon Placement Date	Coupon Extraction Date
	Coating with injected liquid natural soil pH	37	76.0670	107.3700	108.52	148.56	150.68	Friday, July 12, 2019	Sunday, January 12, 2020
		38	76.0466	103.6400	107.77	144.76	146.85	Friday, July 12, 2019	Sunday, January 12, 2020
		42	76.2086	104.3100	105.41	145.45	147.43	Friday, July 12, 2019	Sunday, January 12, 2020
	Coating with injected liquid pH 11.5	43	76.0580	109.1800	110.35	150.42	152.42	Friday, July 12, 2019	Sunday, January 12, 2020
		44	76.0494	105.2000	106.38	146.42	148.56	Friday, July 12, 2019	Sunday, January 12, 2020
		45	76.0584	106.5700	107.72	147.73	149.73	Friday, July 12, 2019	Sunday, January 12, 2020
48	No treatment	48	76.2057	87.4400	N/A	127.46	129.41	Friday, July 12, 2019	Sunday, January 12, 2020
		49	76.0362	86.9500	N/A	127.02	129.04	Friday, July 12, 2019	Sunday, January 12, 2020
		50	76.0459	88.0900	N/A	128.06	130.09	Friday, July 12, 2019	Sunday, January 12, 2020
	Coating with injected liquid natural soil pH	46	76.0154	106.2100	107.4	147.45	149.43	Friday, July 12, 2019	Sunday, January 12, 2020
		47	76.2074	106.6900	107.89	147.89	149.91	Friday, July 12, 2019	Sunday, January 12, 2020
		51	76.0705	102.8300	103.95	143.91	145.98	Friday, July 12, 2019	Sunday, January 12, 2020
	Coating with injected	52	76.2195	105.8400	106.98	147.05	149.01	Friday, July 12, 2019	Sunday, January 12, 2020
		53	76.0566	103.6000	104.74	144.75	146.75	Friday, July 12, 2019	Sunday, January 12, 2020

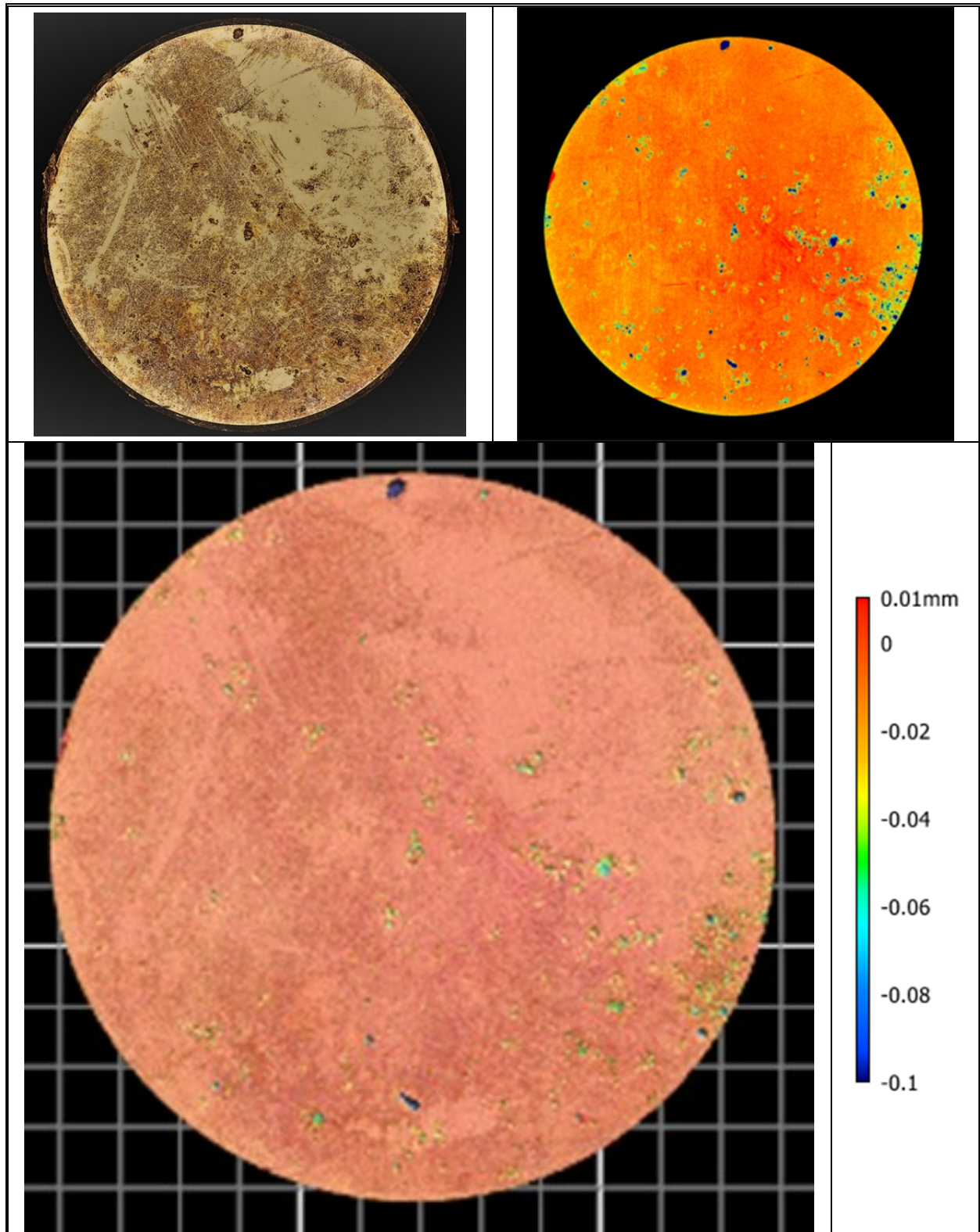
**Table A-8. Experimental Test matrix**

<b>Vessel Temperature (°C)</b>	<b>Treatment</b>	<b>Coupon ID</b>	<b>Weight (g)</b>	<b>Weigh coupon cold mount with tape (g)</b>	<b>Weigh coupon with soil solution (g)</b>	<b>Weigh coupon with soil (g)</b>	<b>Weigh coupon with extra water (g)</b>	<b>Coupon Placement Date</b>	<b>Coupon Extraction Date</b>
	<b>liquid pH 11.5</b>	54	76.2074	103.6600	104.81	144.81	146.89	Friday, July 12, 2019	Sunday, January 12, 2020

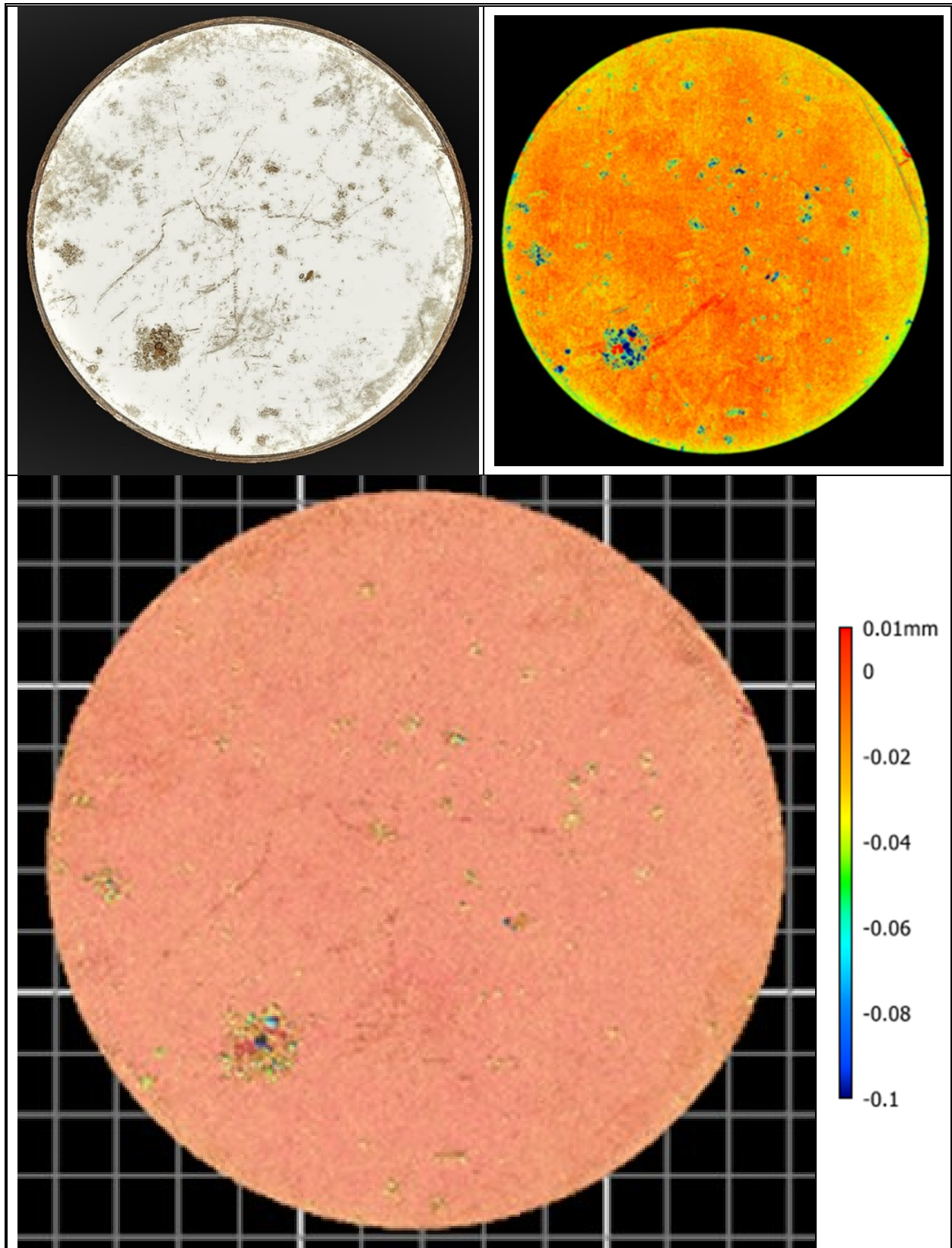


Temperature 22 °C

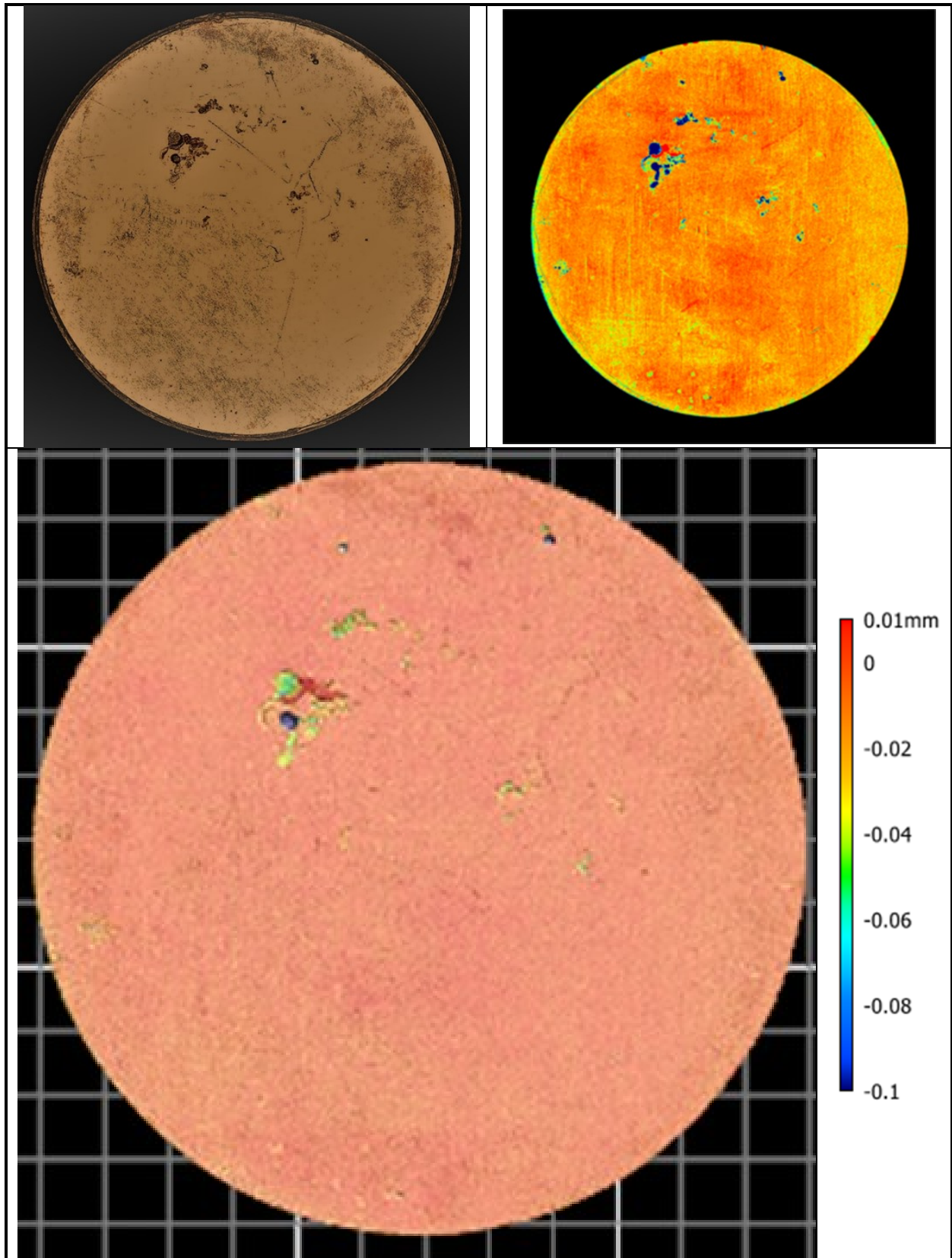
Coupon 1



Coupon 2



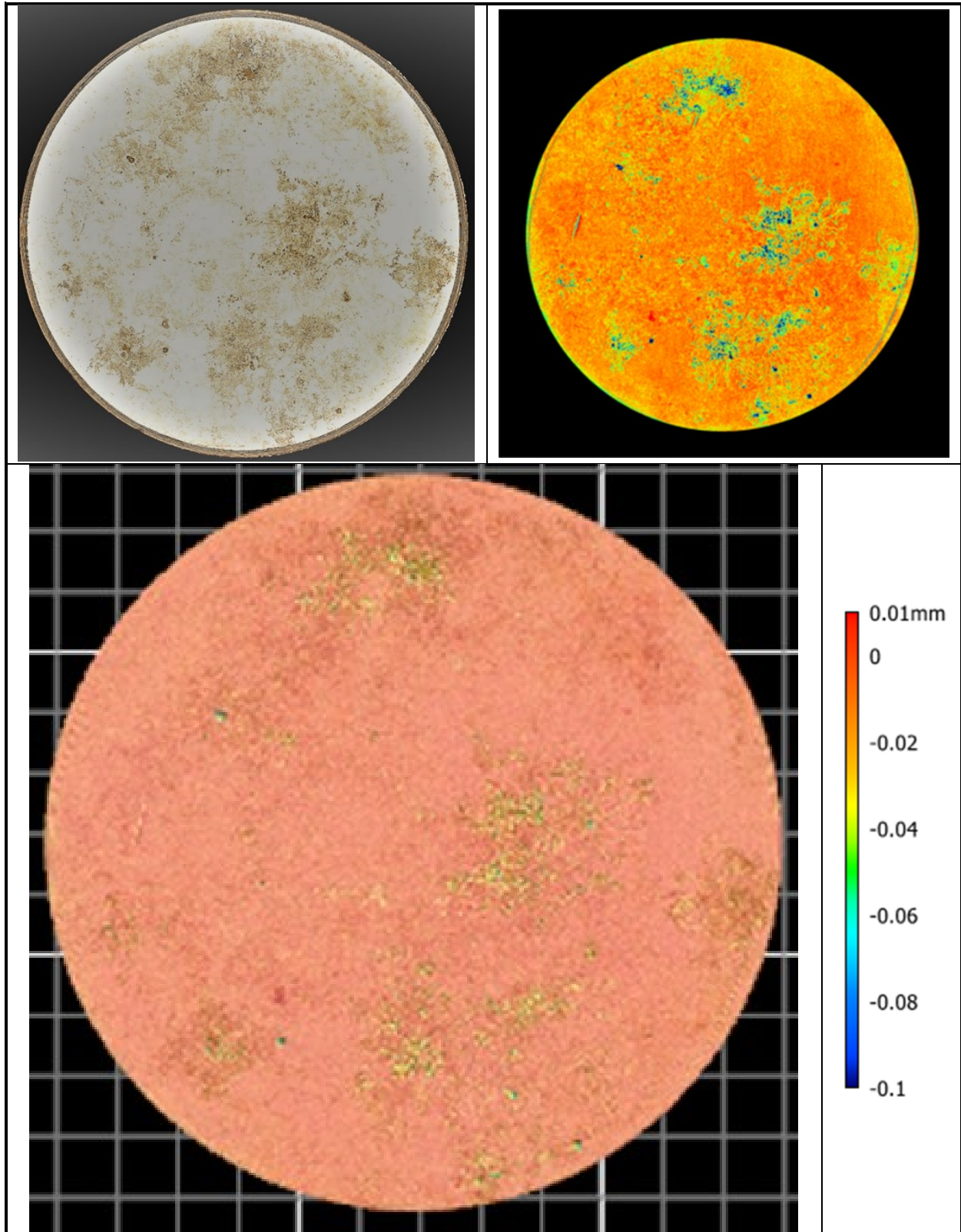
Coupon 3



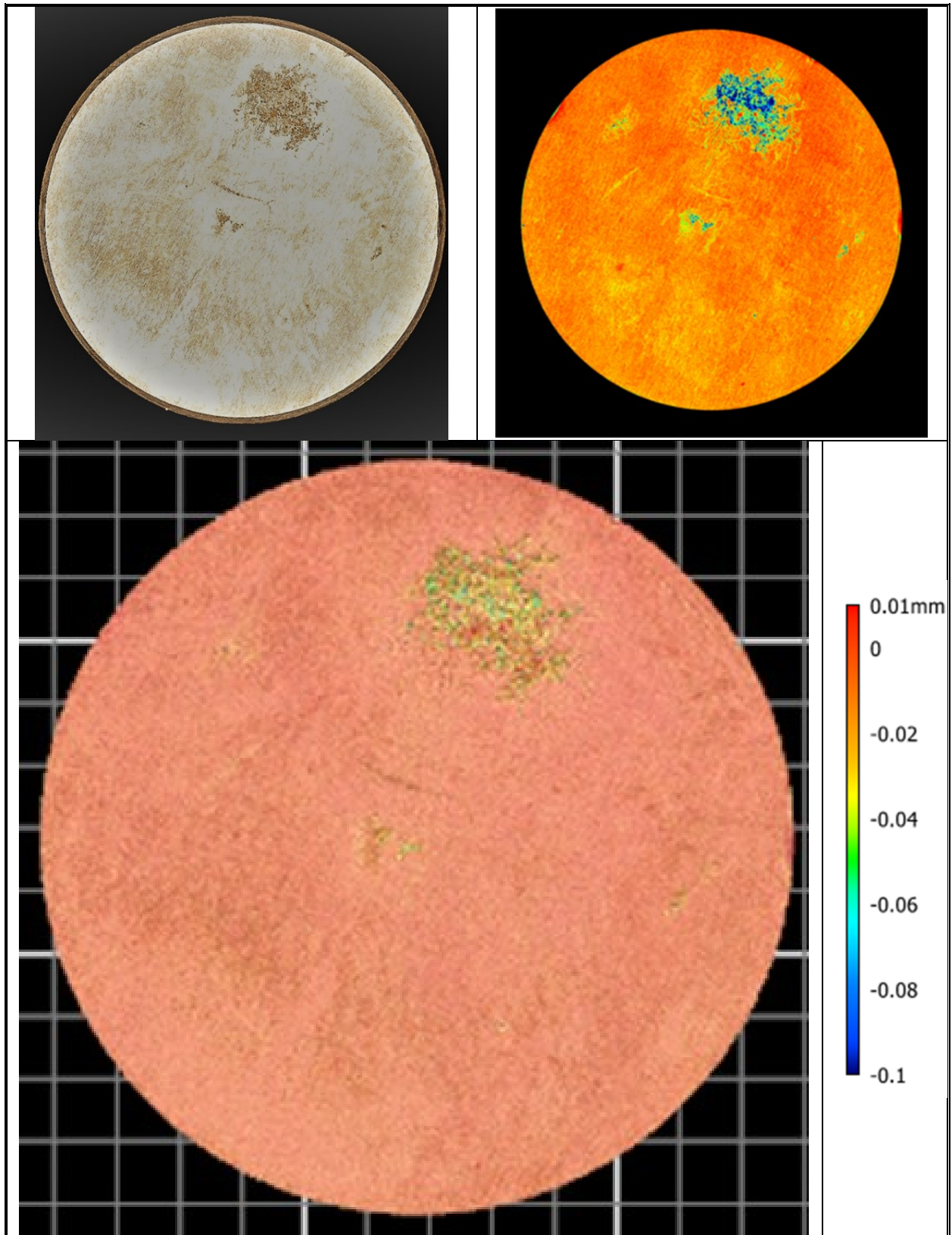


Temperature 28 °C

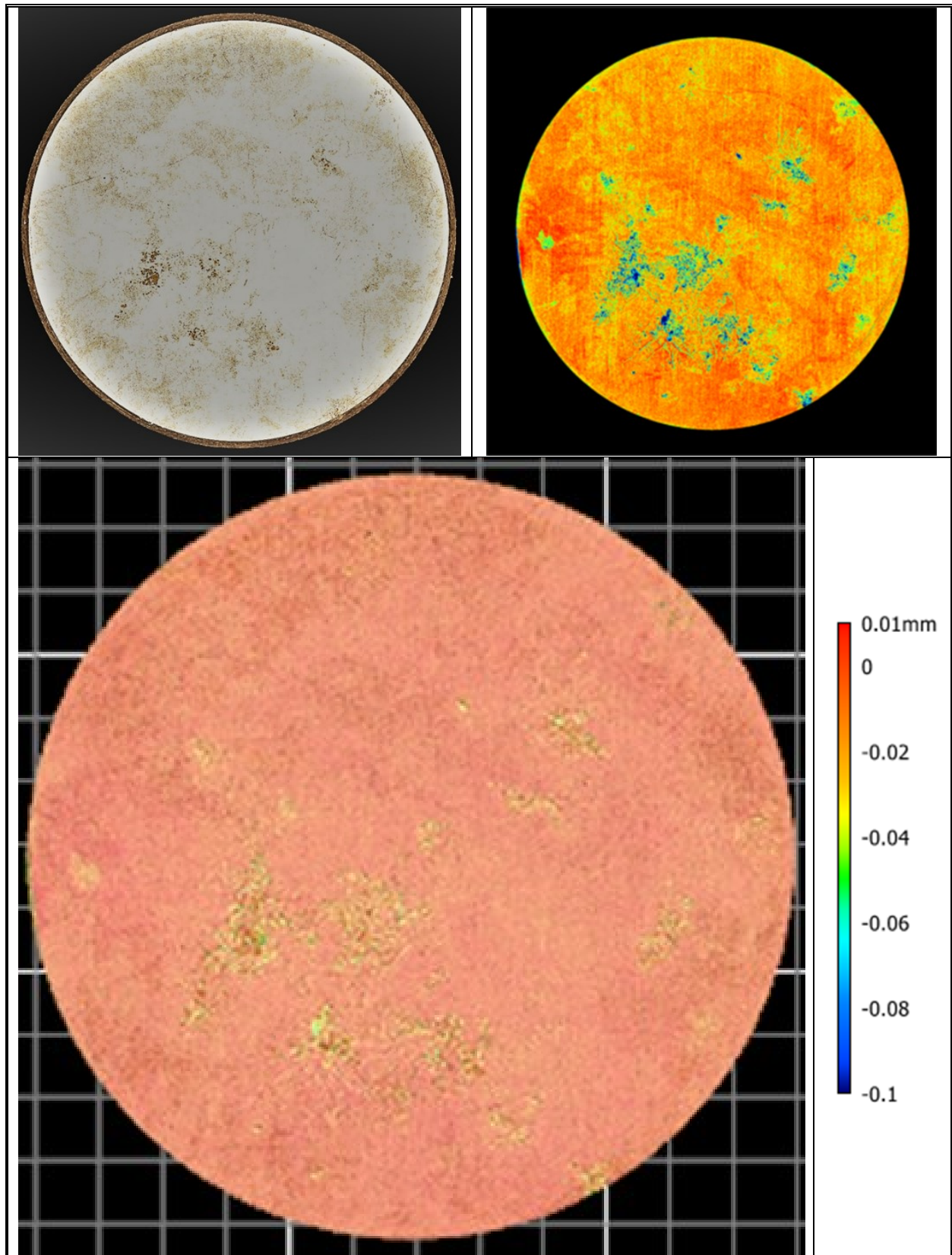
Coupon 1



Coupon 2



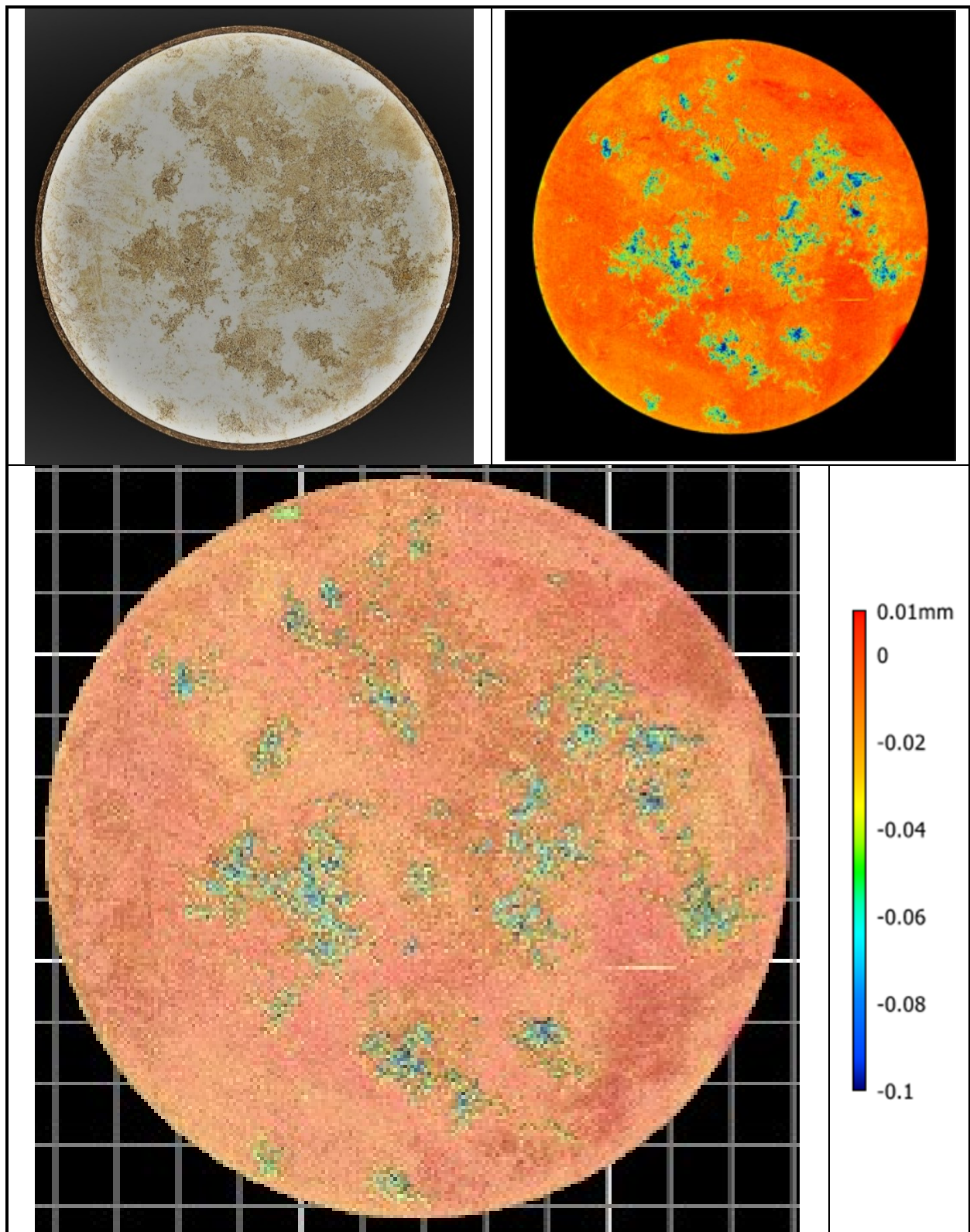
**Coupon 3**



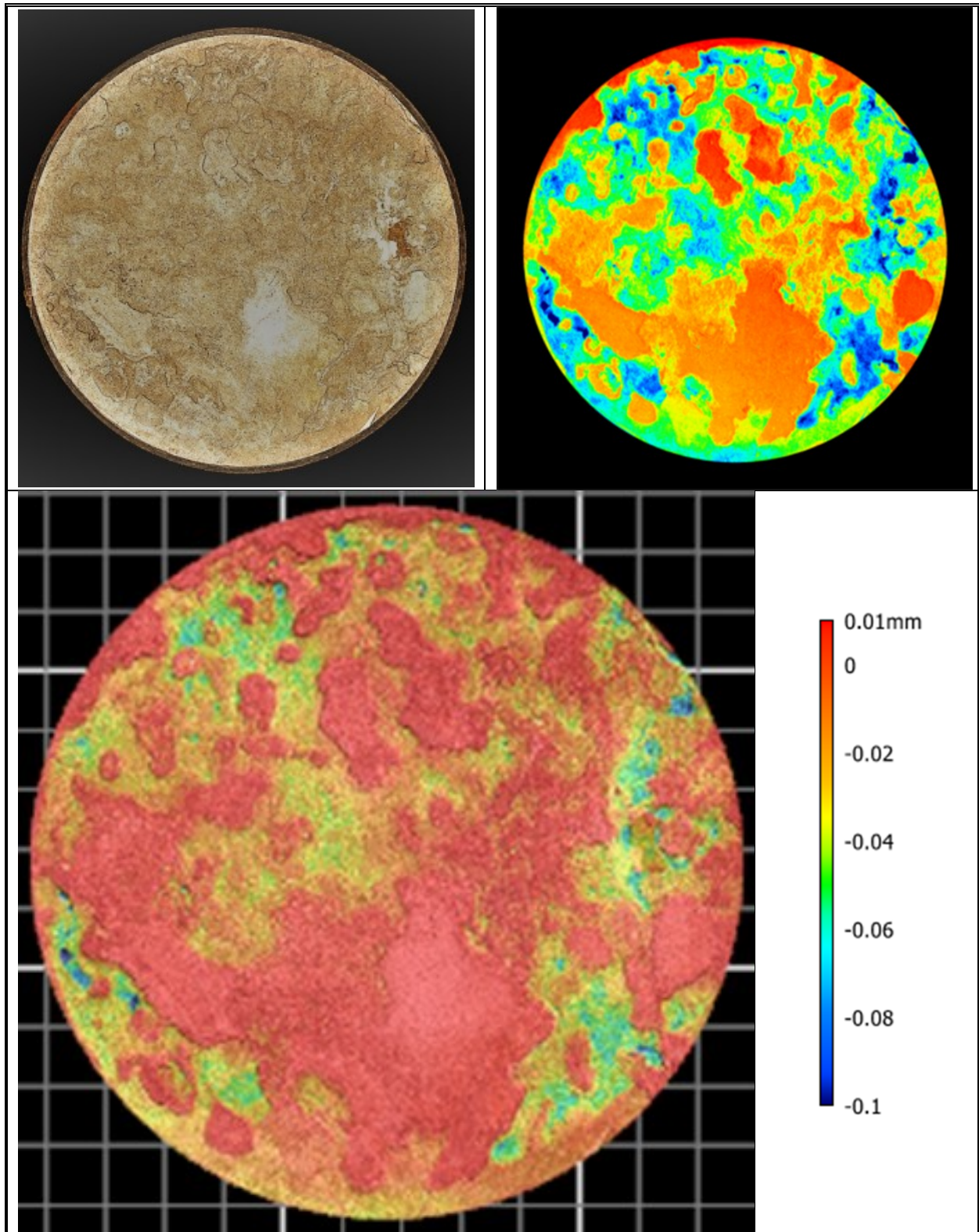


Temperature 33 °C

Coupon 1

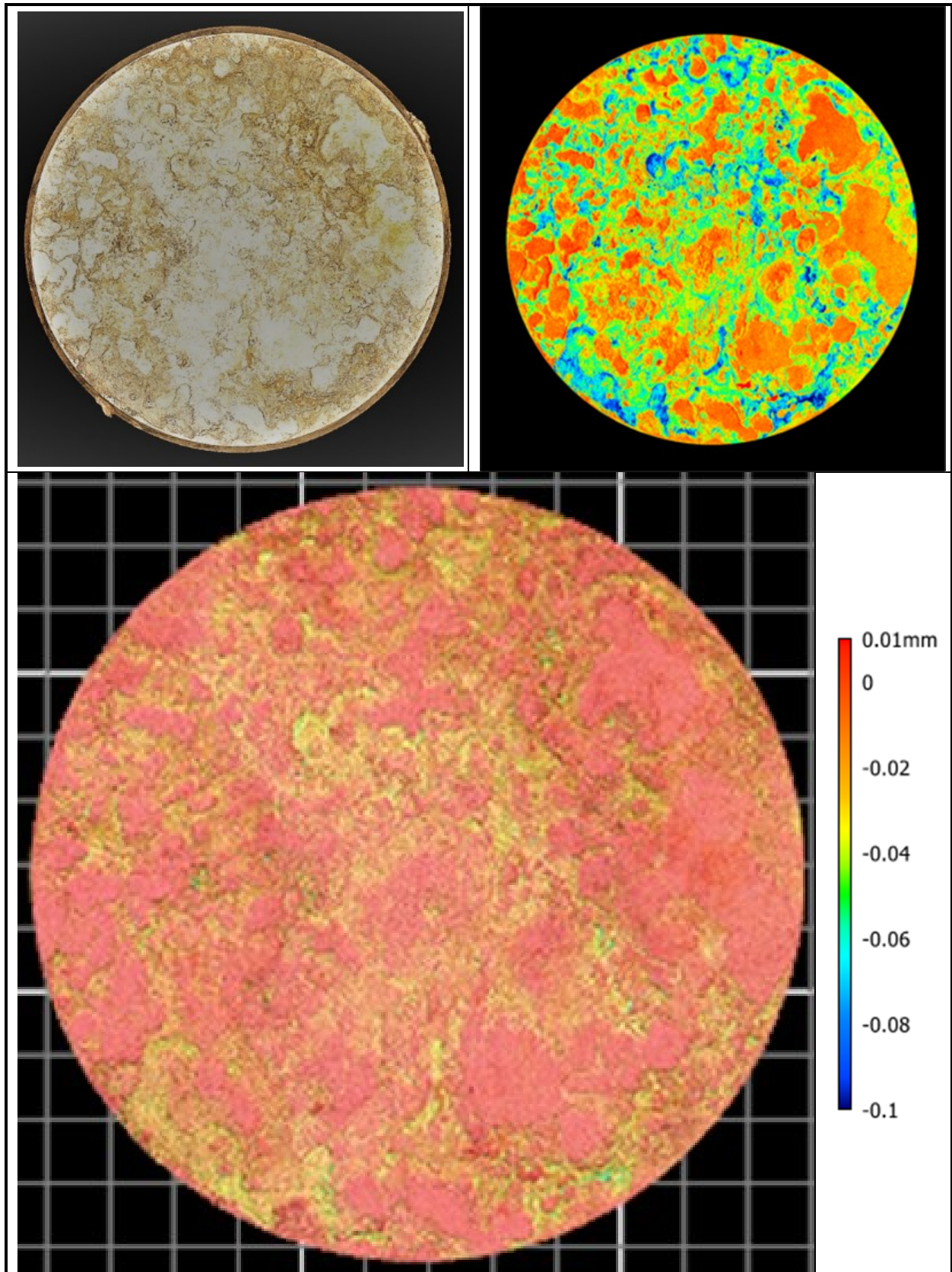


Coupon 2



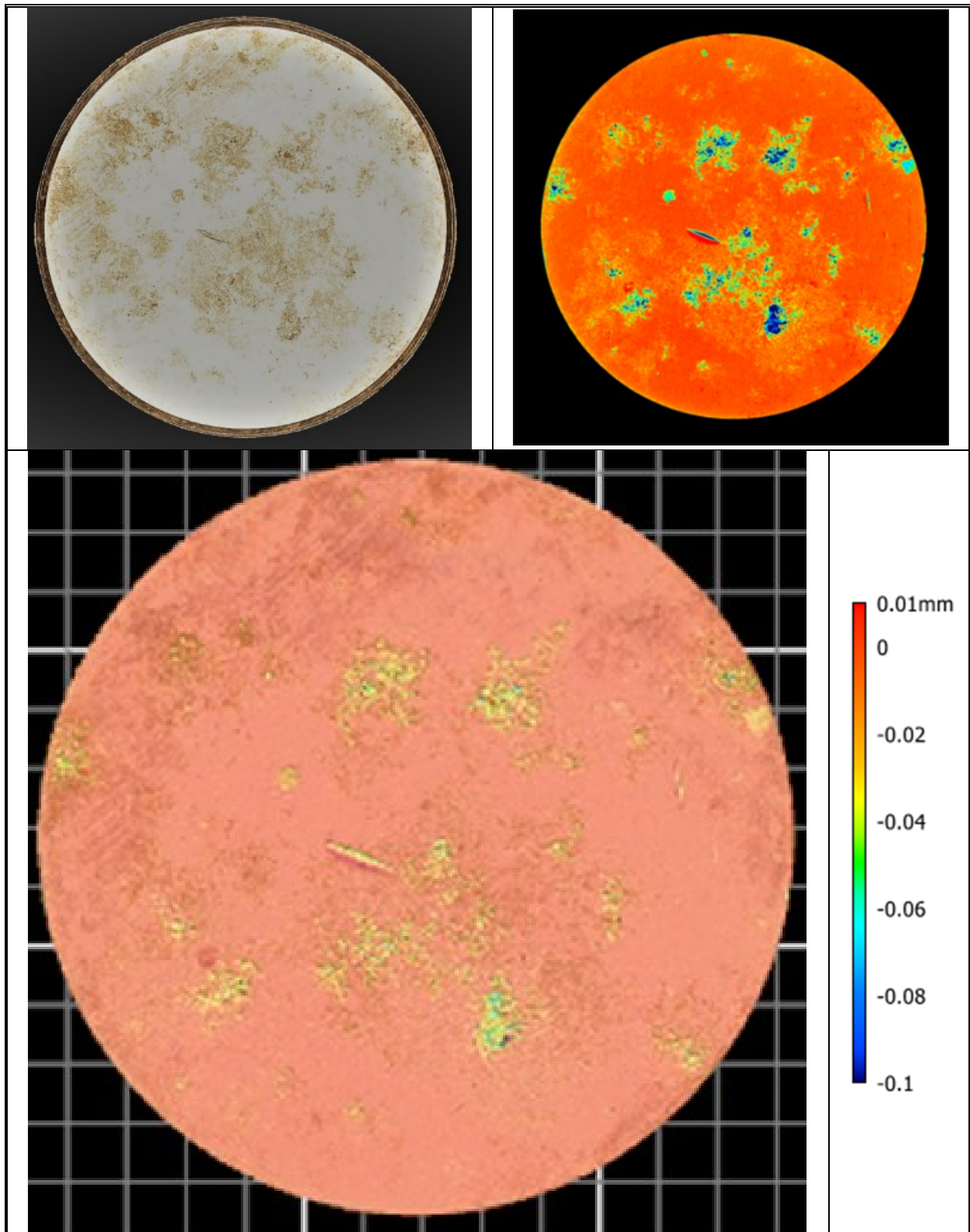


Coupon 3

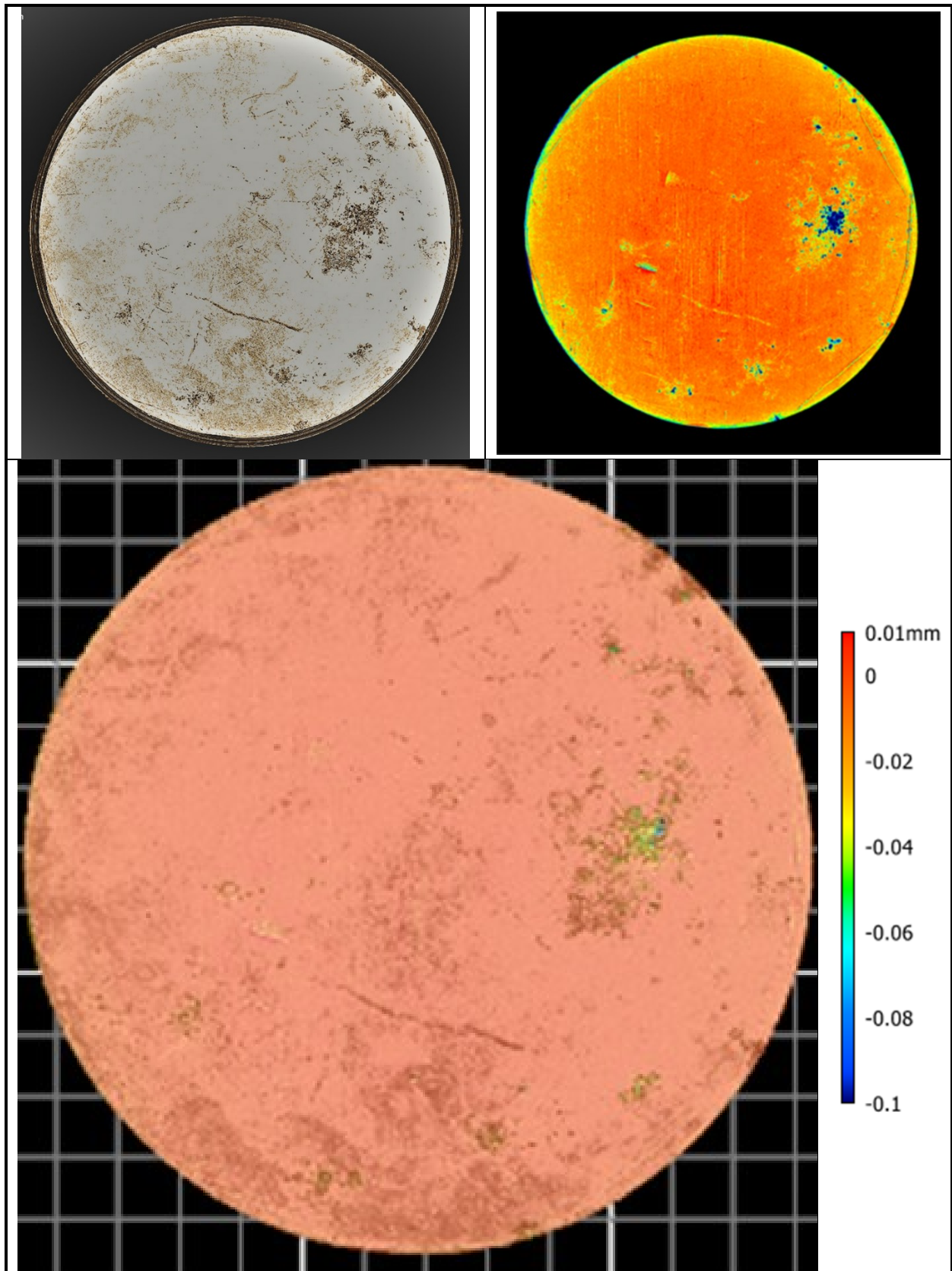


Temperature 38 °C

Coupon 1

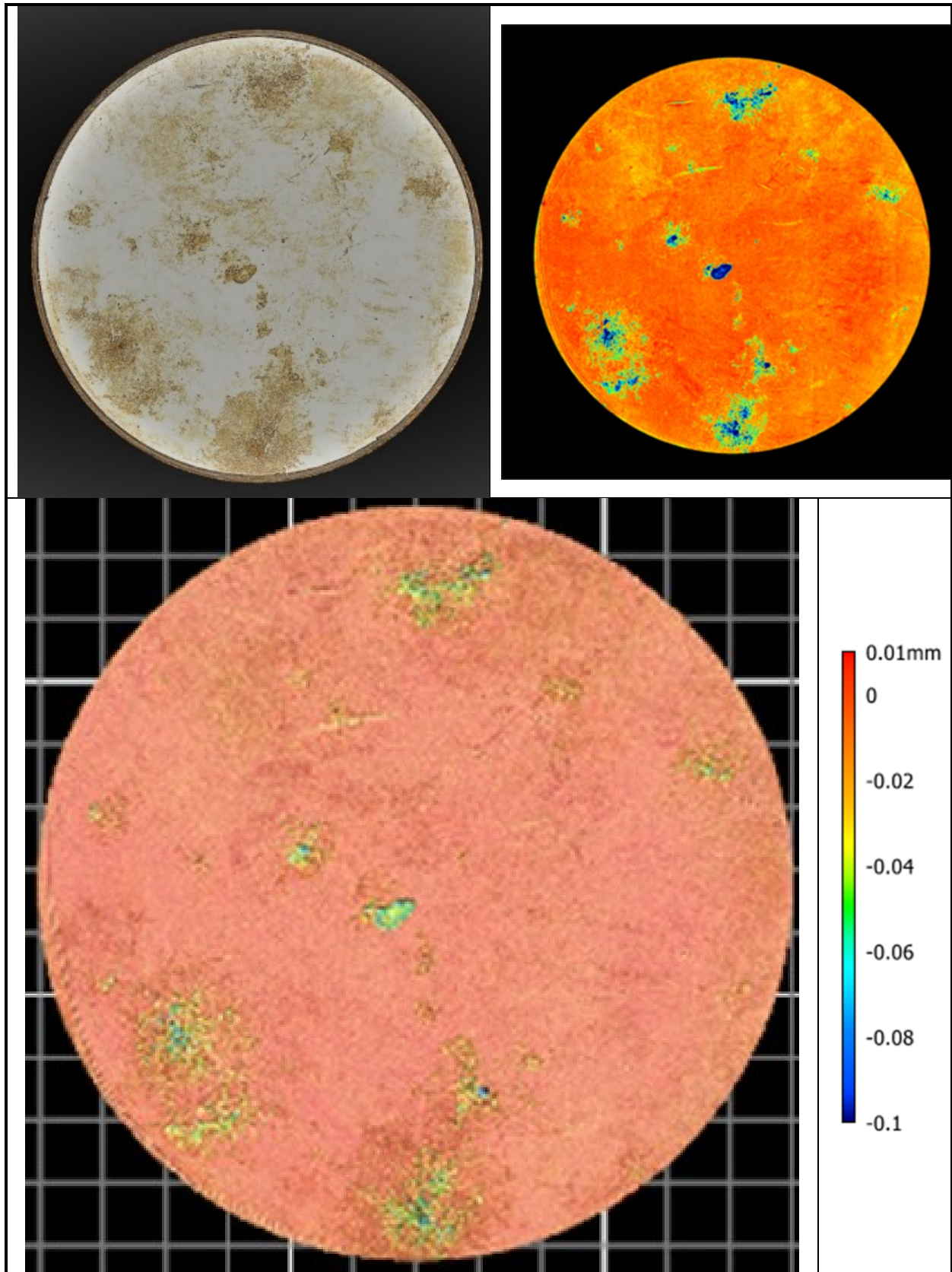


Coupon 2





Coupon 3



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