Investigation of Chloride-induced Stress Corrosion Cracking for Long-Term Storage of Spent Nuclear Fuel in Dry Storage Systems

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Abstract

Chloride-induced stress corrosion cracking (CISCC) has been identified as the main degradation mechanism for spent nuclear fuel dry storage canisters. This type of induced cracking is complex and depends on several factors, such as material composition, exposure temperature, relative humidity, applied tensile stress, and atmospheric salt concentration. An accelerated experiment was designed to simulate marine environments in a controlled fogging chamber to examine 304 and 304L stainless steel U-bend and welded U-bend samples. The samples were exposed to chloride rich and humid fogging in a corrosion chamber at 35℃ continuously for 4 weeks, 8 weeks, and 12 weeks. The same experiment was repeated at 50℃ for 4 weeks, 8 weeks, and 14 weeks to study the sensitivity of CISCC to temperature changes. A qualitative evaluation of optical micrographs from a 3D Surface Profiler was performed for 16 corroded samples and compared with 2 reference samples. Cracking was observed on 12 out of 16 samples exposed to 35℃ and 50℃ for durations ranging from 8 to 14 weeks. Likely cracking observations were noted on 4 out of 16 samples. A quantitative statistical analysis was also performed using surface profile depth (valley) data from corroded and reference samples. The quantitative analysis examined the effect of temperature, welding, exposure duration, and material composition. The quantitative results were compared with the qualitative results and literature published in CISCC.

Investigation of Chloride-induced Stress Corrosion Cracking for Long-Term Storage of Spent Nuclear Fuel in Dry Storage Systems

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General Audience Abstract

Most nuclear power plants are currently using dry storage canisters (DSCs) which are made of a concrete vault and a stainless steel canister that houses the spent nuclear fuel (SNF) assemblies. Multiple conditions must be present simultaneously for chloride-induced stress corrosion cracking (CISCC) to develop, such as the presence of a susceptible alloy, high relative humidity, high temperature, high atmospheric salt concentrations, and applied tensile stresses. DSCs are typically made from 300-series austenitic stainless steels which are susceptible to this type of corrosion during long-term storage near marine environments. Therefore, understanding of the factors leading to CISCC is critically important for proper management and mitigation and to estimate the service life of DSCs for the safe long-term storage of SNF. An accelerated experiment was designed to examine the effects of temperature, exposure duration, and welding on pitting and cracking for 304 and 304L U-bend samples. The experimental results concluded that stainless-steel grades 304 and 304L are susceptible to CISCC when exposed for 8 weeks or longer to fogging at temperatures between 35°C and 50°C, 95% relative humidity, and 5% salt concentration. This study also concluded that increasing exposure duration from 8 to 12 weeks or the temperature from 35°C to 50°C had no significant effect on the acceleration of CISCC. Also, unwelded samples were deemed more susceptible to CISCC than welded samples and the susceptibility of 304 and 304L grades were relatively similar.

Dedication

This work is dedicated to my wonderful parents, Ismail Shakhatreh and Norhan Gharaibeh, my lovely wife Hanoof Qablan, and the joy of my life Nawras Shakhatreh

أهدي هذا العمل لوالدي الرائعين اسماعيل شخاترة ونورهان غرايبة وزوجتي الحبيبة هنوف قبلان وفرحة حياتي نورس شخاترة

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IIHCC destination area is committed to transdisciplinary projects that exist along the interdependencies between humans, community, and infrastructure with the ultimate goal of improving quality of life around the globe. IIHCC brings together and supports teams of faculty and students from across academic disciplines to address particular needs, ranging from large societal problems to industry-specific challenges.

ICTAS is a VT investment institute that provides seed funding to support pilot work to support the efforts of engineering faculty to improve the chance of success of proposals to targeted external research funding opportunities that align with VT Destination Areas.

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Table of Contents

List of Figures

List of Tables

List of Abbreviations

SNF	Spent Nuclear Fuel
DSC	Dry Storage Canister
ISFSI	Independent Spent Fuel Storage Installation
CRS	Congressional Research Services
RH	Relative Humidity
HAZ	Heat Affected Zone
SCC	Stress Corrosion Cracking
CISCC	Chloride-induced Stress Corrosion Cracking
NRC	Nuclear Regulatory Commission
EPRI	Electric Power Research Institute
NPP	Nuclear Power Plant
DOE	Department of Energy
IAEA	International Atomic Energy Agency
QA	Quality Assurance
SNL	Sandia National Laboratory
SWRI	Southwest Research Institute
ASTM	American Society for Testing and Materials
NG	Nuclear Grade
GTAW	Gas Tungsten Arc Weld
DCPD	Direct Current Potential Drop
CFM	Cubic Feet Per Minute
PSI	Pound per Square Inch

W	Welded Designation
L	Low Carbon Designation
LW	Low Carbon Welded Designation
TDS	Total Dissolved Salts
CT	Computed Tomography
ANOVA	Analysis of Variance
SRS	Simple Random Sampling
S_{max}	Larger Standard Deviation Between the Two Tested Groups
S_{min}	Smaller Standard Deviation Between the Two Tested Groups
H_0	Null Hypothesis
H_a	Alternative Hypothesis

1. Chapter 1: Introduction

1.1 Background

Harsh environmental conditions surrounding spent nuclear fuel (SNF) dry storage canisters (DSCs) or often referred to as independent spent fuel storage installations (ISFSIs) can be detrimental to the canister over time. The utilization of DSCs has increased in the United States and around the world in recent years as data shows in the Congressional Research Services (CRS) report for congress [1], many of them are stored near corrosive marine environments. Harsh corrosive conditions are characterized with high atmospheric salt concentrations, high temperatures, and high relative humidity (RH). Moreover, these stainless steel canisters exhibit residual stresses from fabrication, and some are welded resulting in material sensitization around the weld area known as heat affected zone (HAZ) which reduces the canister's resistance to corrosion attacks [2]. Certain types of corrosion attacks can lead to cracking through the steel such as, stress corrosion cracking (SCC) or more specifically chloride-induced stress corrosion cracking (CISCC) resulting from harsh environments with high chloride contents (*e.g.*, sites near sea water). CISCC is a complex phenomenon and only occurs when certain conditions are present simultaneously for extended durations.

Due to the complexity of CISCC, experimental data is desired to better understand the phenomenon itself and to find thresholds for the environmental conditions that lead to pitting corrosion and crack initiation [3]. Furthermore, data on crack propagation through stainless steel is needed to estimate the lifetime of DSCs [4]. This research investigates CISCC in a controlled corrosive environment to characterize pitting and cracking of U-bend and welded U-bend samples made from 304 and 304L austenitic stainless steel at different temperatures.

Furthermore, there are no clear guidelines or specific regulatory guides on the service life of DSCs. The U.S. Nuclear Regulatory Commission (NRC) has entertained the idea of SNF dry storage for up to three-hundred years [1], but concerns regarding long-term storage have been raised by leading research institutions. One example is the research conducted by the Electric Power Research Institute (EPRI) which concluded that CISCC is the most credible threat to DSCs in a Canister Breach Consequence Analysis Study [5]. Currently, DSCs are licensed for 20 years only with the option to renew, some license holders have already extended their dry storage license for another 20 years (*e.g.*, Surrey Nuclear Plant in VA) [6].

This thesis provides the results of two accelerated CISCC experimental campaigns to enhance understanding of the factors leading to CISCC in 304 and 304L U-bend stainless steel samples with applied tensile stress. Experimental results can provide the NRC and commercial nuclear power plants (NPPs) with means to estimate the risk of confinement breach due to CISCC for early intervention to alleviate its effects and prevent premature cracking.

1.2 Spent Nuclear Fuel Storage

Commercial NPPs originally planned to reprocess SNF and re-use it as fresh fuel while by-products such as Plutonium (Pu-239) are repurposed for military applications. In the late 1970s, the U.S. President Jimmy Carter imposed a ban on SNF reprocessing citing proliferation and national security concerns. As a result of this ban, NPPs had no means of storing SNF outside the reactor pools. Reactor pools were originally designed to house SNF assemblies for up to five years to cool them down to a lower activity level suitable for dry storage (*e.g.,* deep geological repository) [7]. Moreover, the U.S. Department of Energy (DOE) has committed to establish a deep geological repository to transfer and consolidate SNF assemblies from operational NPPs after the five-year cooling period [8]. This commitment came as a result of the reprocessing ban, but it has not come to fruition as previous efforts have failed such as the Yucca Mountain Project that was terminated in 2009 by the former president Barack Obama [1] [7].

Due to the lack of action on deep geological repositories for the past several decades, DSC systems have been gaining momentum and popularity and their licenses are being extended from few years to decades according to the International Atomic Energy Agency (IAEA) [9]. Furthermore, IAEA stated that any storage beyond a fifty year term is considered "Long-Term" and now there is not enough data on the integrity of these systems for long-term storage. Investigating the long-term feasibility of DSCs is a necessity now since no plans for a government-sponsored deep geological repository in foresight. Based on the current inventory and rate of generation of SNF, the need for a robust DSC system and a credible regulatory framework will only intensify in the future. Table 1 shows facts regarding SNF in the U.S. from the CRS report [1]. Figure 1 shows the locations and estimated number of ISFSIs in the U.S. as of October 2021 [10].

Table 1. SNF In Brief from the CRS Report for Congress [1]

SNF In Brief
Commercial SNF is composed of metal assemblies about 12'-15' long
SNF contains uranium and elements created in nuclear reaction.
SNF assemblies are removed from reactors after being used to produce power.
Existing reactors generate about 2,000 metric tons per year.
More than 67,000 metric tons of commercial SNF is currently being stored.
Most SNF is stored at 77 sites in 35 states
Some SNF is stored at closed reactors.
Some SNF is stored at DOE facilities.
Only 4% of the SNF in the United States is DOE owned.

SNF In Brief
SNF is stored in wet pools and dry casks.
SNF storage at reactors was intended to be temporary, pending disposal.
No nation operates a disposal site for SNF.
Proposed U.S. disposal site for SNF at Yucca Mountain in Nevada was terminated in 2009.
SNF storage is expected to be needed for more than 100 years.

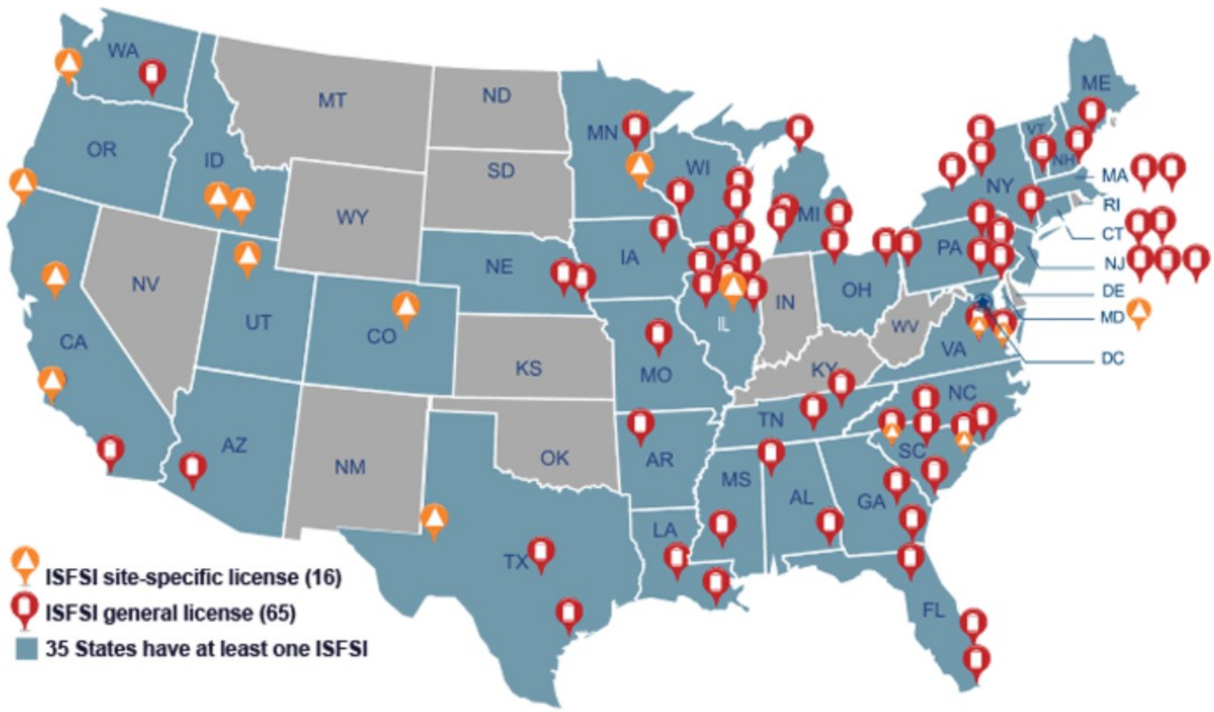

Figure 1. Locations of ISFSIs in the United States as of October 2021 [10]

1.3 Dry Storage Cask Systems

The current fuel cycle in the United States is depicted in Figure 2, this cycle may look different for other countries such as France and Japan where recycling of SNF is permitted [11]. In the U.S., a fresh nuclear fuel bundle, which is shown on the far left of Figure 2, is typically used in the reactor's core for 24-36 months before it's taken out and labeled SNF. Newly removed SNF assemblies are highly radioactive due to the accumulation of fission products from the U-235 fission process during the reactor's operation. Therefore, SNF must be stored in a pool next to the reactor's core for a minimum of five years until they reach a lower activity level suitable for DSCs that are shown on the far right of Figure 2.

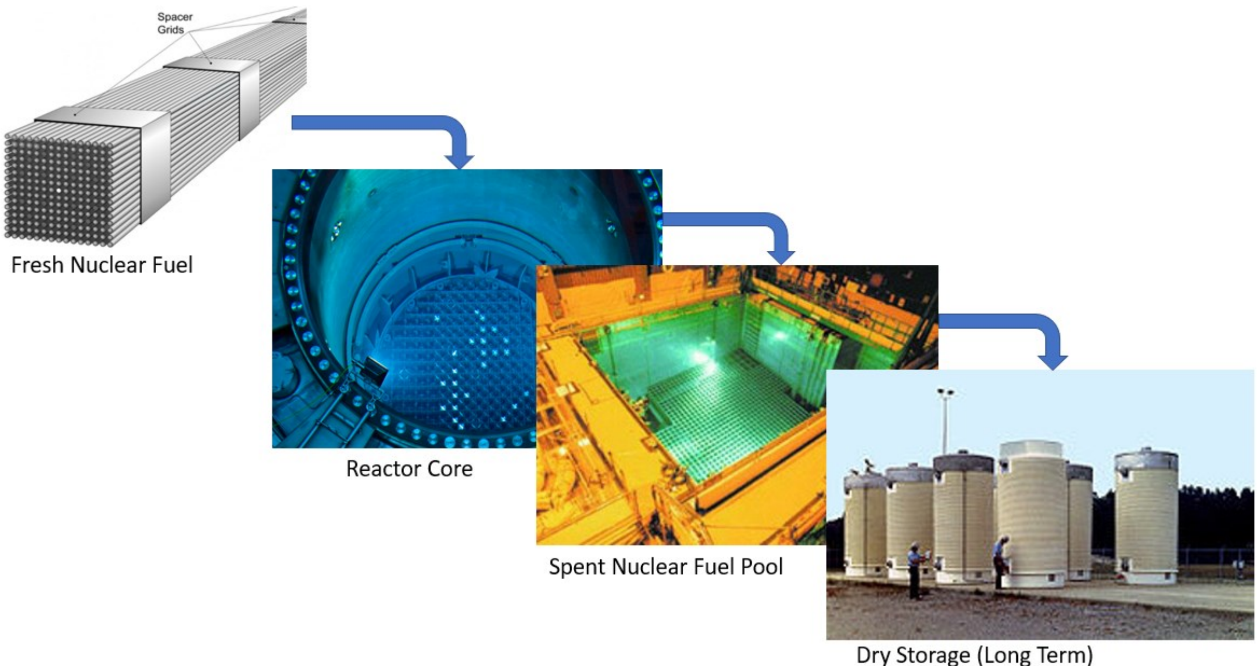

Figure 2. Nuclear Fuel Cycle in the United States

There are two dry storage cask designs that are currently licensed by the NRC, vertical and horizontal systems shown in Figure 3 and Figure 4, respectively. The two designs are similar in their capability of decay heat removal from the SNF by natural convection, but the onsite footprint, construction method, construction material, and their efficiency of heat removal are different.

A typical dry storage cask unit consists of a concrete vault surrounded by an outer metal lining. Inside the concrete vault (shell) is the stainless steel canister that houses the SNF assemblies. The primary construction material of the internal canister is 300-series stainless steel and most commonly austenitic stainless steel 304, 304L, 316, and 316L grades with bolted or welded canister's lid [2]. DSC systems are stored independently outdoors within the nuclear plant's vicinity on a designated concrete pad to allow for natural convection. Vertical DSCs can be stored as independent units, such as the Hi-STORM dry cask shown in Figure 3 which is made by Holtec International [12]. Horizontal DSCs are stored in groups as shown in Figure 4 with a natural convection cycle through the concrete structure to remove decay heat, such as the NUHOMS dry cask made by Orano [13].

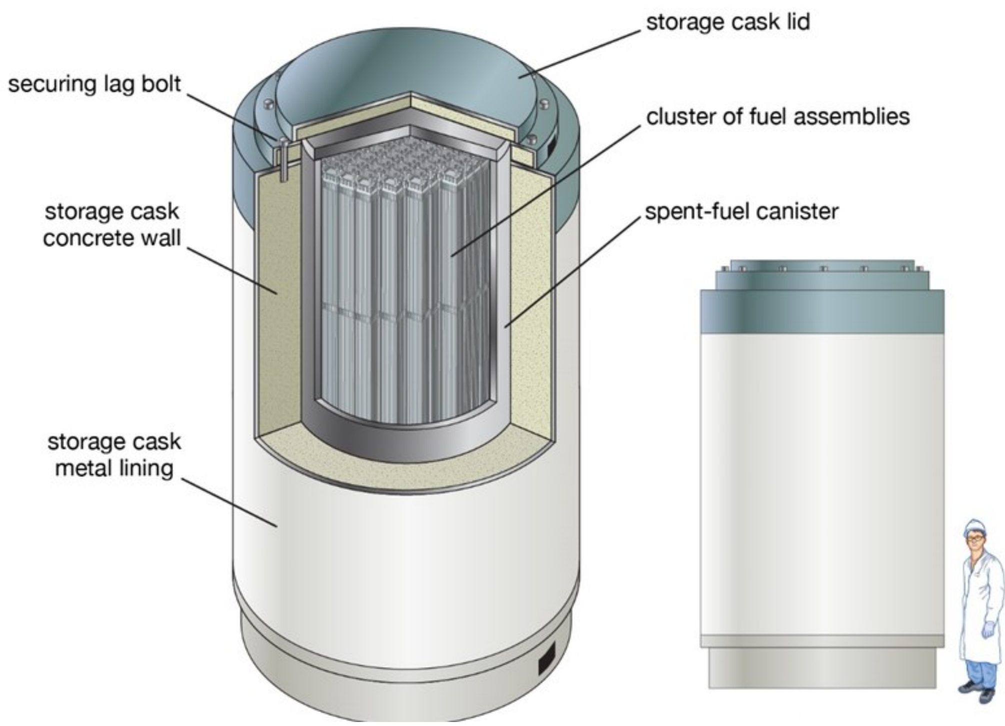

Figure 3. Vertical SNF Dry Storage System (HI-STORM) [12]

Figure 4. Horizontal SNF Dry Storage System (NUHOMS) [13]

1.4 Chloride-induced Stress Corrosion Cracking (CISCC)

SCC is defined as the initiation and propagation of cracks caused by a combination of environmental conditions that are best aligned with those conditions that exist in marine environments. CISCC begins with deposits of chloride salts on the material's surface that absorb moisture from the surrounding air to form a deliquescence salt film that causes pitting or crevice corrosion [14]. The deposition rate of sea salt particles is a function of the geographical area and the surrounding environmental conditions. Current research suggests that the formation of deliquescence salt film is highly dependent on temperature and RH [15] [16]. Additionally, research suggests a high probability of SCC in highly stressed areas such as bent surfaces and heat sensitized areas from welding or heat treatment [2]. However, research suggests that there is no threshold for tensile stress to initiate SCC but increased tensile stress accelerates time to failure. Applied stresses makes the stainless steel's protective oxide layer susceptible to damage which encourages pitting corrosion [17] [18]. Pitting corrosion advances into SCC when the appropriate conditions are present as depicted in Figure 5 [19].

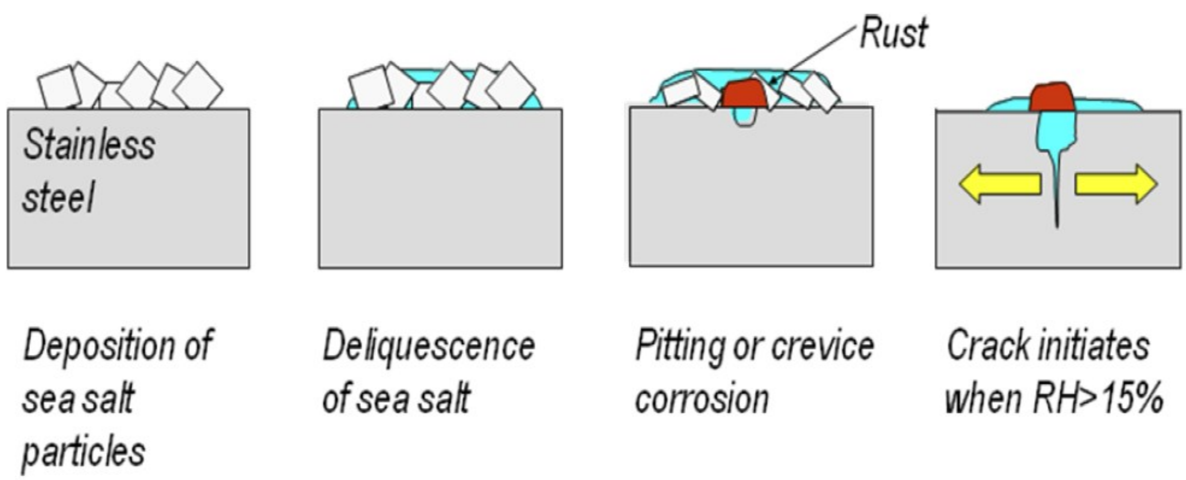

Figure 5. Stages of Stress Corrosion Cracking [19]

1.5 Material Susceptibility to CISCC

Susceptibility to CISCC is highly dependent on the material's chemical composition (*e.g.*, material grade) and stress state such as applied stress, residual stress, and heat sensitization. It has been shown that standard grades of austenitic stainless steel such as 304, 304L, 316, 316L are the most susceptible to CISCC [20]. Corrosion experiments have shown various levels of corrosion, pitting, or crack propagation for dissimilar grades of austenitic stainless steels exposed to the same conditions emphasizing the role of alloy composition in CISCC [16]. Residual stresses from fabrication processes such as bending or applied stress through bolted connections also decreases the material's resistance to CISCC attacks [2].

Additionally, heat sensitization due to welding or heat treatment reduces the material's resistance to corrosion. Welding stainless steel requires heating to 400-800°C resulting in a HAZ where carbon atoms diffuse to the edges of the grain boundaries and react with the available chromium forming chromium carbides ($Cr_{23}C_6$) as shown in Figure 6 [21]. Chromium in stainless steel is the primary protective element as it reacts with oxygen when exposed to the atmosphere to form a protective oxide layer on the material's surface. On the other hand, chromium depletion can be detrimental to the stainless steel's ability to fight corrosive attacks by weakening the grain boundaries leading to intergranular stress corrosion cracking [22]. Low carbon materials such as, 304L or 316L (L indicates a low carbon content) are recommended for welded structures to mitigate the effect of heat sensitization [22] [23]. DSCs are made from rolled stainless steel shells which are likely to have residual stresses from manufacturing and some are welded which further increases its susceptibility to CISCC.

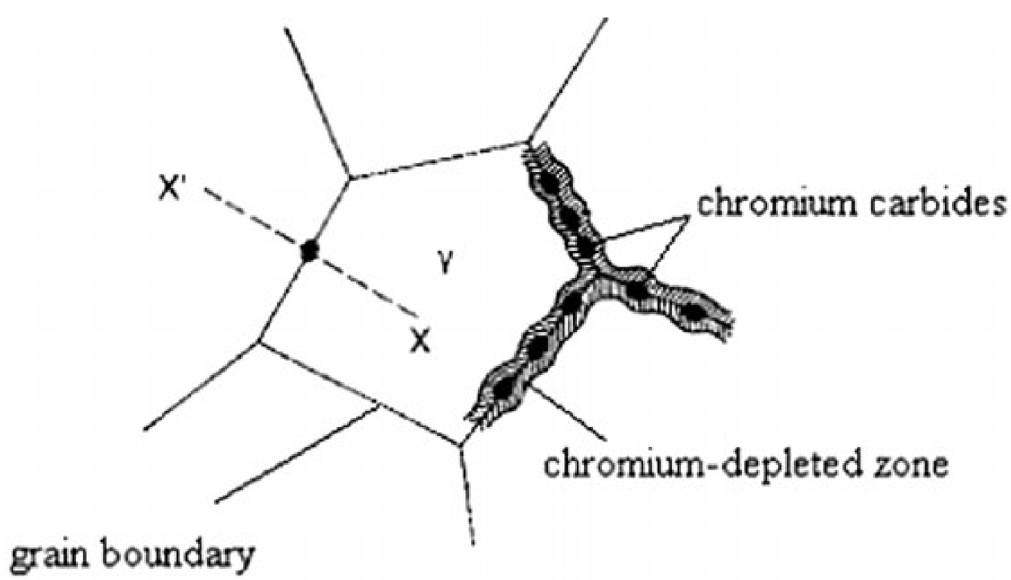

Figure 6. Chromium Carbide Precipitation from Welding [21]

1.6 Environmental Factors Leading to CISCC

The presence of a susceptible alloy, heat sensitization, and tensile stresses without a corrosive environment is not enough to trigger concerns over CISCC. Crack initiation and propagation is also dependent on the environmental conditions surrounding the material. Figure 7 shows the conditions that must be present simultaneously for the initiation of SCC [24]. If one condition is eliminated or avoided, the likelihood of developing SCC will be reduced dramatically. Likewise, exposure duration is an essential variable to the development of SCC although not depicted in Figure 7. SCC is a long-term phenomenon as stated previously, its development takes place over extended exposure given all pre-conditions are met.

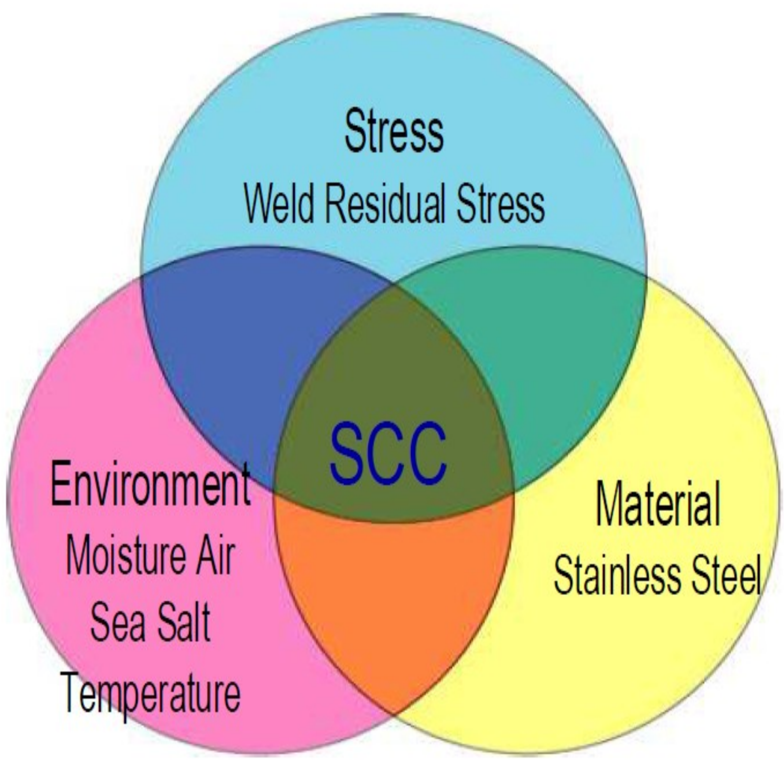

Figure 7. Stress Corrosion Cracking Conditions Map [24]

A typical corrosive environment is best characterized with high concentrations of salts, high atmospheric temperature, and high RH. High RH coupled with high concentration of salts in the atmosphere enables a process known as deliquescence. Deliquescence is the process where solid salt deposits on material surfaces absorb humidity from the surrounding air to form a chloride rich liquid film that eventually dissolves the stainless steel's chromium passive layer leading to the oxidization of the base metal (Fe_2O_3). On the other hand, high temperatures enable efflorescence which is the process of drying the material's surface leaving behind solid salt deposits as shown in Figure 8 [25]. Both deliquescence and efflorescence, which are competing processes, contribute to the material's degradation and lead to corrosion, pitting, and cracking after extended exposure.

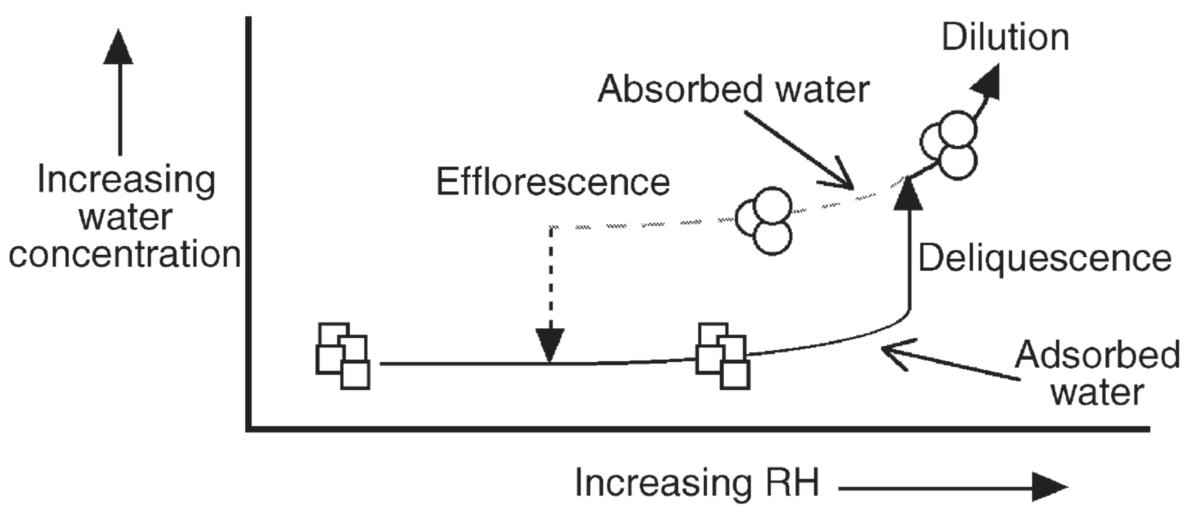

Figure 8. Salt Deliquescence, Efflorescence, and RH Relationship [25]

Research suggests that there is a threshold RH at which the risk of SCC increases which is known as "deliquescence relative humidity". The deliquescence RH threshold is a function of the salt type and is independent of the quantity of deposited salt [26].

The schematic in Figure 9 shows a DSC (*aka.,* ISFSI) unit located near sea water which is the motivation behind this research to examine its service life. NPPs located near marine environments and considering DSCs for long-term storage must be mindful of the CISCC risk within their aging management strategy. Experimental data on pitting and cracking can be useful to the NRC in licensing commercial DSCs for long-term storage beyond the initial 20-year term.

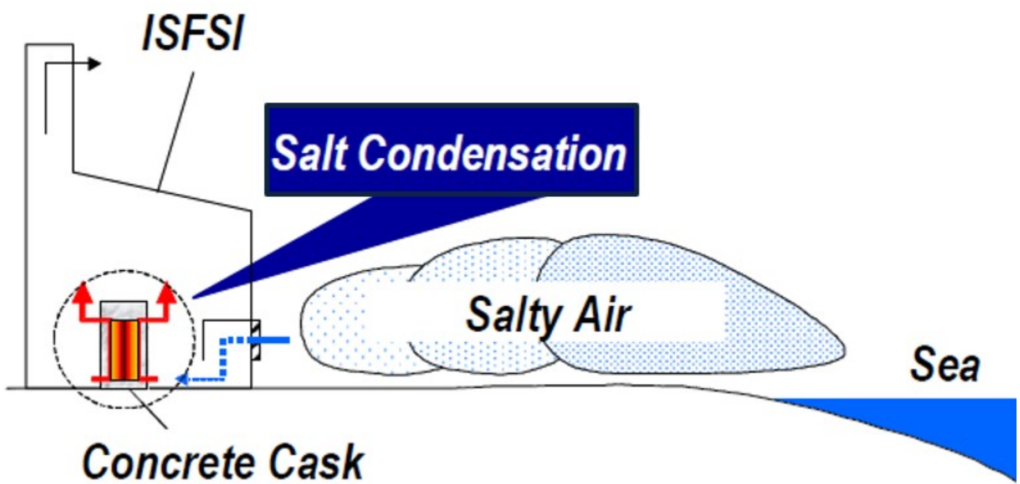

Figure 9. Schematic of ISFSI in a Corrosive Environment [24]

1.7 Thesis Structure

Chapter 2 provides literature review and a summary of the relevant previous work in SCC with emphasis on accelerated CISCC experiments in a controlled fogging chamber. Chapter 3 provides a detailed overview of the experimental design, procedures, data collection, and the quality assurance (QA) procedures implemented to control the experiment's parameters and quality of results. Chapter 4 includes the sample characterization procedures, results, and analysis of results of the two experimental campaigns in terms of susceptibility to CISCC. Finally, Chapter 5 provides conclusions and recommendations for future research in accelerated CISCC.

2. Chapter 2: Literature Review

Extensive research has been done in the field of SCC and CISCC in austenitic stainless steel using natural exposure and accelerated experiments to understand the phenomenon itself and the factors leading to SCC. Many researchers examined SCC for a wide range of applications such as the storage of SNF near coastal areas and to find alternative materials for reactor components that are susceptible to SCC. The mechanisms of pitting, cracking, and crack propagation are not well understood and the data available in this area are limited due to the complexity of the phenomenon and the variety of factors involved [3]. Moreover, SCC experiments are sensitive to the method and approach which adds another layer of complexity, for instance changing the geometry of the experimental setup or the salt composition could lead to unique results. Furthermore, Sandia National Laboratory (SNL) published a report summarizing data available on SCC from multiple experimental work and concluded that "*Available SCC crack growth rate data from corrosion testing under atmospheric conditions is highly scattered, in part due to wide variety of testing methods used to collect the data. Other contributing factors are study-to-study variations in potentially important parameters such as salt load and RH*" [3].

Although knowledge in SCC is relevant to a diverse range of applications, this Chapter is focused on CISCC experiments relative to the storage of SNF in DSC systems. Several prominent research organizations and institutions around the world are actively pursuing academic and industrial research in CISCC such as the NRC, EPRI, DOE, and Southwest Research Institute (SWRI). EPRI performed a consequence analysis scoping study to examine the confinement breach of welded stainless steel DSCs due to CISCC and stated that "*Chloride induced stress corrosion cracking* *leading to canister confinement breach is identified as the most credible aging management degradation mechanism to impact the canister confinement function"* [5].

Most of the existing research on CISCC for nuclear applications is focused on stainless steel grades 304, 304L, 316, and 316L as these grades are known to be susceptible to cracking under corrosive conditions and widely used in the nuclear industry [16]. Papers discussed in this Chapter examined the effect of heat sensitization from welding or heat treatment, RH, temperature, and salt concentration from accelerated SCC experiments. The following sections provide a summary of the state of knowledge in this area with emphasis on the approach utilized in each paper. Table 2 summarizes the relevant papers that will be discussed in this Chapter with high-level details on each paper.

Table 2. Papers Summary on Accelerated Stress Corrosion Cracking

Author (year)	Organization	Experiment Type	Sample Type/ Materials	Experiment Conditions
L. Caseres and T.S. Mintz (2010)	NRC/ SWRI	Accelerated Fogging Chamber/Spray Test	U-bend and Welded U-bend 304, 304L, 316L	Temp.: 25 - 176°C RH: up to 70% Salt Conc.: 5% by weight NaCl
Akio Kosaki (2007)	Central Research Institute of Electric Power Industry	Accelerated Fogging Chamber/Natural Exposure	4 point bent & 3 point bend 304, 304L, 316LNG	Temp.: Ambient/60°C RH: 95% (at 60°C) Salt Conc.: Saturated NaCl
Zakaria et al. (2020)	Centre for Corrosion Research, Universiti Teknologi PETRONAS	Immersion test	U-bend, 304L	Temp.: 28°C/70°C/90°C Salt Conc.: 0.1 wt.%, 1 wt.%, 3.5 wt.% NaCl
Mintz et al. (2013)	SWRI/NRC	Cyclic humidity test (fog chamber)/Static test conditions (constant humidity)	U-bend and Welded U-bend 304 as well as sensitized samples	Temp.: 35°C/45°C/52°C/ 60°C. RH: variable per test Salt Conc.: 0.1, 1, 10 g/m^2

Author (year)	Organization	Experiment Type	Sample Type/ Materials	Experiment Conditions
Cook et al. (2011)	University of Manchester, UK, University of Oxford, UK, CSIRO, Melbourne	Accelerated Experiment/Natural Exposure	U-bend, 304L & 316L	Temp.: 80℃ RH: 30% Salt Conc.: varies (multiple salts and concentrations)
Kim et al. (2021)	Korea Institute of Materials Science	Accelerated Fogging Chamber/Spray Test	Welded U-bend 304L & 316L	Temp.: 60℃/50℃ RH: 30% & 20% Salt Conc.: 3.5 wt.%

2.1 Review of Previous CISCC Accelerated Experiments

The NRC in collaboration with the SWRI performed an accelerated CISCC experiment [16] following a modified version of GM 9540P accelerated test procedure. The NRC used a standard fogging chamber to evaluate U-bend and welded U-bend samples made of 304, 304L, and 316L austenitic stainless steel at temperatures ranging from 25℃ - 176℃ and varying RH. The corrosion chamber used by the NRC (Auto Technology model number CCT-NC-40) has a cyclic capability to maintain a salt layer on the samples. The cyclic feature works by fogging salt for a predetermined duration and then dispersion stops for drying leaving behind salt deposits on the sample's surface. RH was a function of the exposure zone temperature, wet and dry cycles alternated RH from high to low and vice versa but it did not exceed 75% in any of the experiments. The NRC used an optical microscope to examine corroded samples; cracks were observed under 50X magnification with widths ranging from 3 to 5 μm as shown in Figure 10 for the 304 sample exposed to 43°C for 16 weeks.

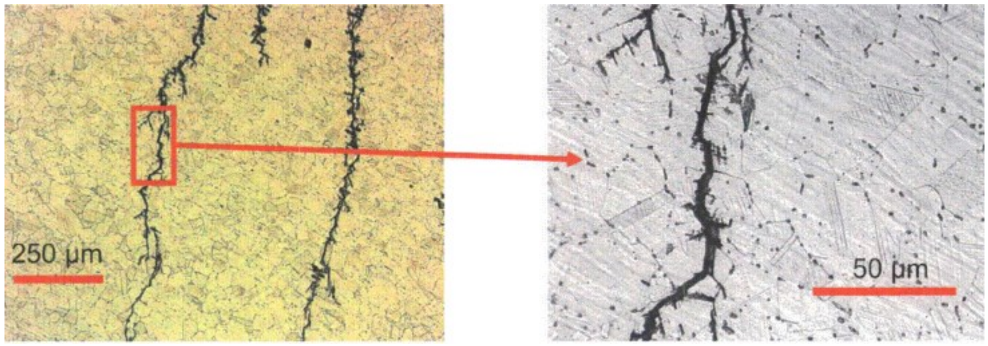

Figure 10. CISCC Observed on 304 Sample Exposed to 43°C for 16 Weeks [16]

The NRC conclusions did not agree with previous published literature [27] relative to the effect of temperature on SCC. The NRC observed cracks at exposure temperature below 43°C for all sample types and exposure durations (4 - 32 weeks). The NRC findings indicate that CISCC does not accelerate at higher temperatures as evident by the plot shown in Figure 11 for 304 samples. Moreover, welded U-bends did not crack between 4 and 16 weeks of exposure at 43°C while at least one unwelded U-bend cracked during this time undermining the effect of sensitization due to welding.

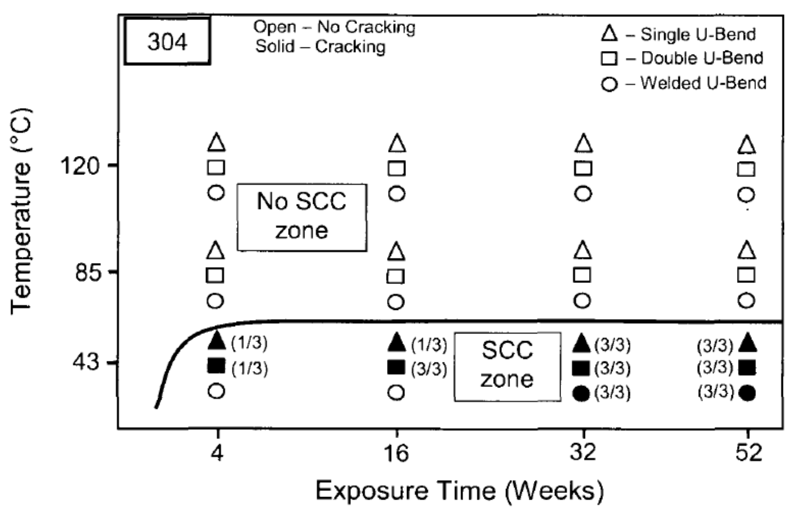

Figure 11. Exposure Time vs. Temperature for 304 Samples [16]

Worth noting the U-bend samples used in the NRC experiment were prepared in accordance with the American Society for Testing and Materials (ASTM) G30 standard (Standard Practice for Making and Using U-Bend SC Test Specimens) [28]. Welded U-bend samples were prepared in accordance with ASTM G58 standard (Standard Practice for Preparation of SC Test Specimens for Weldments) [29].

Kosaki [4] performed two experiments to estimate SCC initiation time and crack growth rate under both natural and accelerated exposure conditions. The SCC initiation experiment was performed using the 4-bend point samples without cracks, the crack growth experiment utilized 3-bend point samples with a pre-existing crack. The Kosaki experiment utilized base metal and welded samples made from 304, 304L, 316LNG (NG is a nuclear grade designation), refer to Figure 12 for the sample's configurations.

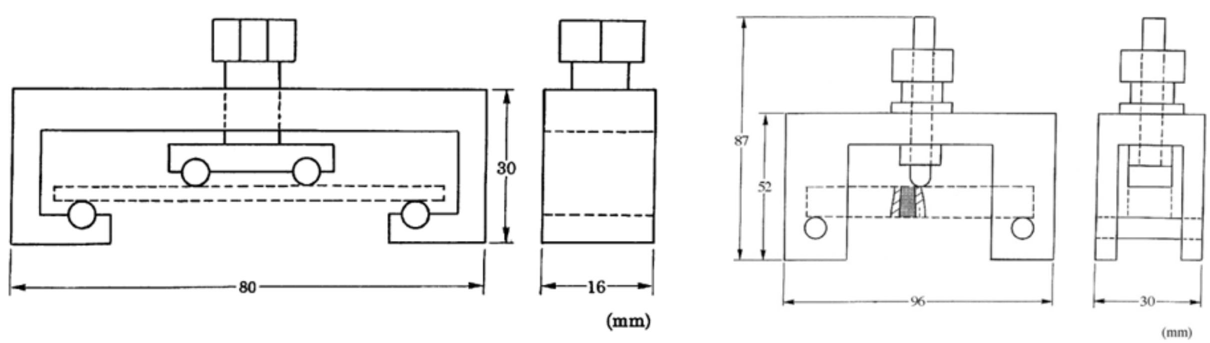

Figure 12. Kosaki Samples, 4-point Bend (Left) and 3-point Bend (Right) [4]

Natural exposure experiments were done at Miyakojima Island (most corrosive location in Japan); and the accelerated experiment was completed in a controlled chamber filled with saturated NaCL mists at 60°C for 5 years. For the natural exposure experiment at ambient conditions, initiation of

SCC was only observed on the 304 weld samples after 2.5 years of the test. Under accelerated conditions, SCC was observed within 30 days on all sample types (304, 304L, 316LNG). However, the findings of the accelerated experiment cannot be attributed to a single parameter since temperature, RH, salt deposition rate or salt concentration were all increased simultaneously and not independently. Results from the crack growth experiments showed higher crack growth rates in specimens subjected to accelerated conditions over those placed in open air.

Zakaria et al. [27] performed an accelerated experiment using 304L U-bend samples prepared in accordance with the ASTM G30 standard [28]. The experiment method was long-term immersion of as-received and heat sensitized samples (600°C for 2 hours). This experiment utilized NaCl salt solutions with 0.1 wt.%, 1 wt.%, and 3.5 wt.% concentrations and 28°C, 70°C, and 90°C exposure temperatures. The samples were immersed for 42 days to examine the effect of heat sensitization, temperature, and salt concentration on the acceleration of CISCC.

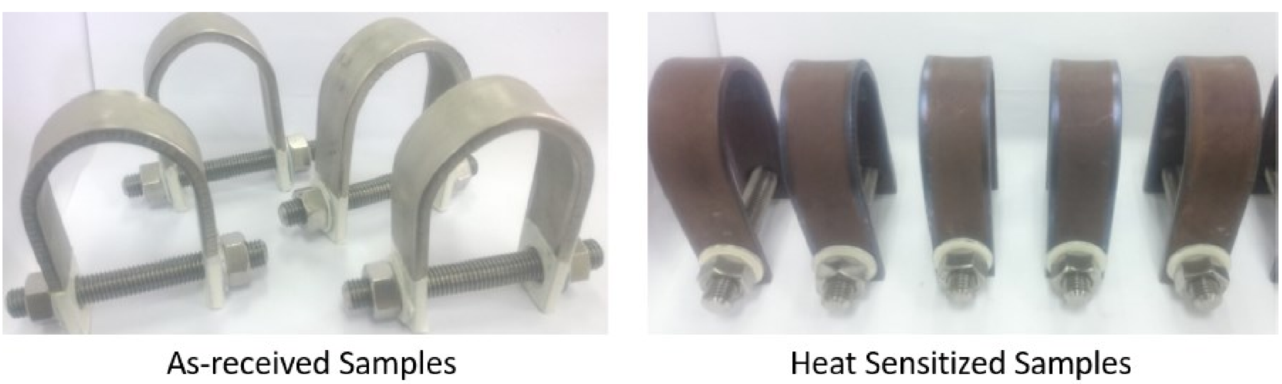

Figure 13. U-bend Samples from Zakaria's Immersion Experiment [27]

None of the as-received and heat sensitized samples exposed to 28°C showed any signs of cracking after the 42 days had elapsed (this was the case for all salt concentrations). At 70°C, general corrosion was observed on the as-received samples placed in the 0.1 wt.% and 1 wt.% containers, pitting started after 21 days on the samples placed in the 3.5 wt.% container and cracking after 42 days. On the sensitized samples at 70°C, pitting and cracking were noted on all samples at all salt concentrations. On the samples exposed to 70°C and 3.5 wt.% salt concentration, cracking was initiated in 28 days rather than 42 days for the as-received samples. At 90°C, the conclusions were similar to the conclusions from the 70°C experiments, but corrosion, pitting, and cracking occurred faster. Time to cracking on the heat sensitized specimens was 7 – 28 days as opposed to 14 – 42 days on the as-received specimens. This paper shows that increasing temperature and salt concentration are directly proportional to the acceleration of CISCC. However, Zakaria's finding regarding the effect of temperature does not agree with the NRC conclusions [16]. Additionally, the experiment showed that heat sensitization increased susceptibility, corrosion rate, and crack propagation rate which was not evident in the NRC study [16]. This paper did not examine the effect of material composition and only tested 304L samples.

Mintz et al. [30] performed two accelerated experiments using 304 base metal, welded, and heat sensitized U-bend samples prepared in accordance with the ASTM G30 standard [28]. The objective of the Mintz experiment is to examine the effects of salt concentration, temperature, and RH on SCC initiation on austenitic stainless steel. The salts used in this experimental work were sodium and magnesium chlorides which have deliquescence points at approximately 70% and 30% RH, respectively. Sensitized samples were heated in air at 650°C for 2 hours to simulate the condition of the weld HAZ. The first experiment was conducted in a corrosion chamber with cyclic humidity test, the samples were placed on cylindrical heaters. The temperatures used in the first experiment were 35, 45, 52, and 60°C and the samples were examined as early as one month. The second experiment was conducted in a corrosion chamber under static environmental conditions (constant temperatures). In the second experiment samples were deposited with salt particles and placed in a corrosion chamber under constant temperatures at 45, 60 and 80°C. A stereomicroscope with 50x to 1000x magnification was used to examine the samples post exposure. Further examination of cracks was conducted once a crack is observed.

The results from the first cyclic experiment showed high dependence between salt concentration and SCC. The samples exposed to 10 g/m^2 deposition density were more susceptible than those exposed to 0.1 and 1 g/m^2 deposition densities and cracked as early as one month of exposure. Refer to Figure 14 and Figure 15 for susceptibility maps for 10 g/m^2 and 0.1 g/m^2 simulated sea salts, respectively. Mintz observed cracks on the samples exposed to 35, 45, and 52°C but not at 60°C. Sensitized specimens showed more cracking than as-received samples and more interestingly welded samples were less susceptible to cracking than heat sensitized and as-received samples similar to the conclusion drawn from the NRC study [16]. Cracks were observed in the cyclic experiment after one month and it was more severe after four months as evident in the susceptibility maps in Figure 14 and Figure 15. On another observation from this experiment, samples exposed to lower simulated sea salts showed increased susceptibility at lower temperatures. This experiment shows that CISCC depends on salt deposition density and temperature (coupled dependence), increasing temperature only did not necessarily correlate with increased susceptibility to SCC in agreement with the NRC conclusions [16].

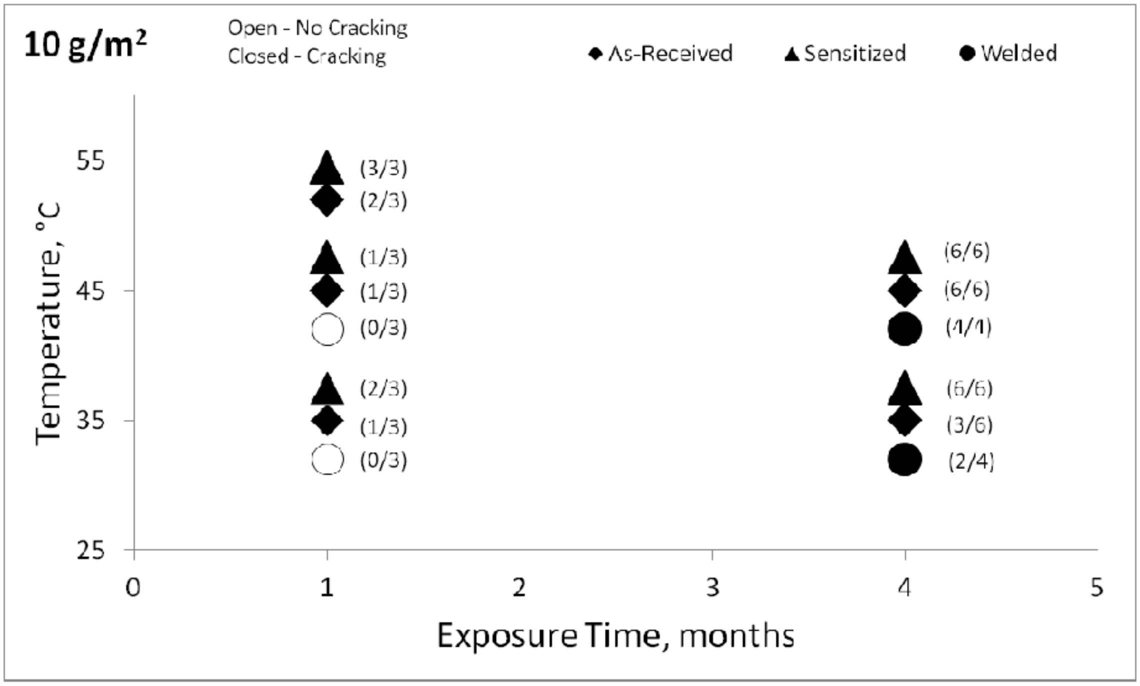

Figure 14. SCC Map for 10 g/m^2 Simulated Sea Salt [30]

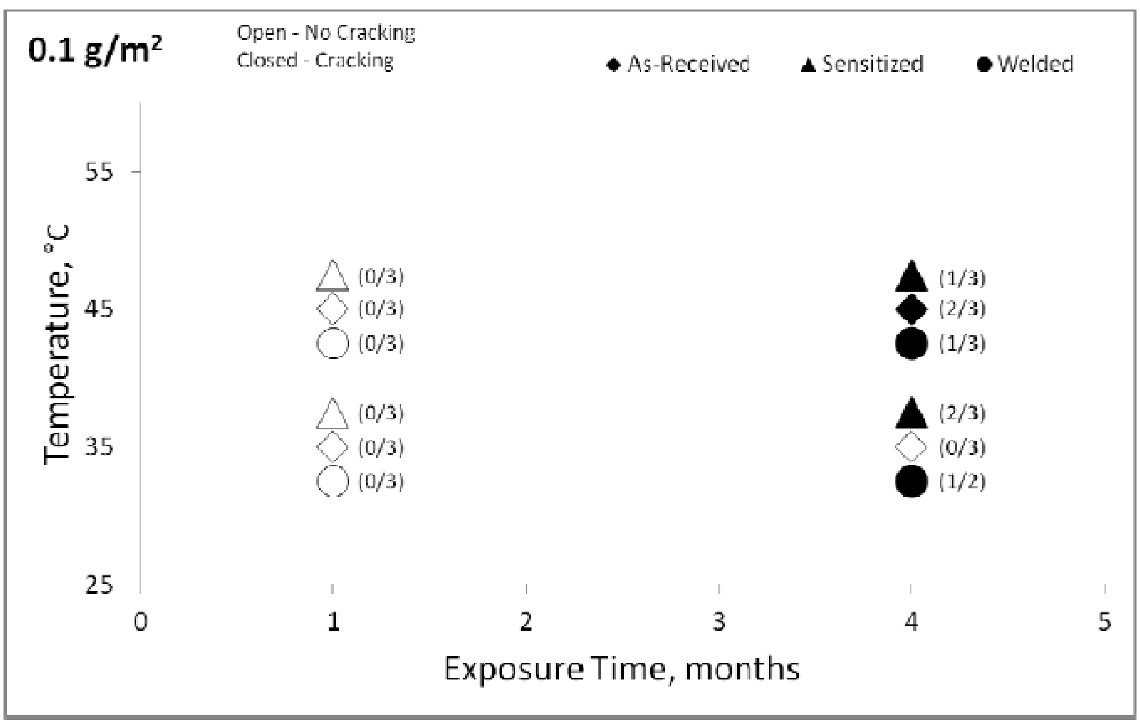

Figure 15. SCC Map for 0.1 g/m^2 Simulated Sea Salt [30]

The results from the second experiment at constant humidity or static environmental conditions were consistent with the results from the cyclic tests. At 60°C of the constant humidity test, crack initiation was observed at above 25% RH and no cracking or pitting were observed below this threshold. For the samples held at 80°C, the RH threshold was determined 35% or higher as illustrated in Figure 16 for both the cyclic and static tests. The experimental work performed by Mintz indicates that crack initiation is a function of RH, temperature, and salt deposition density as shown in Figure 16.

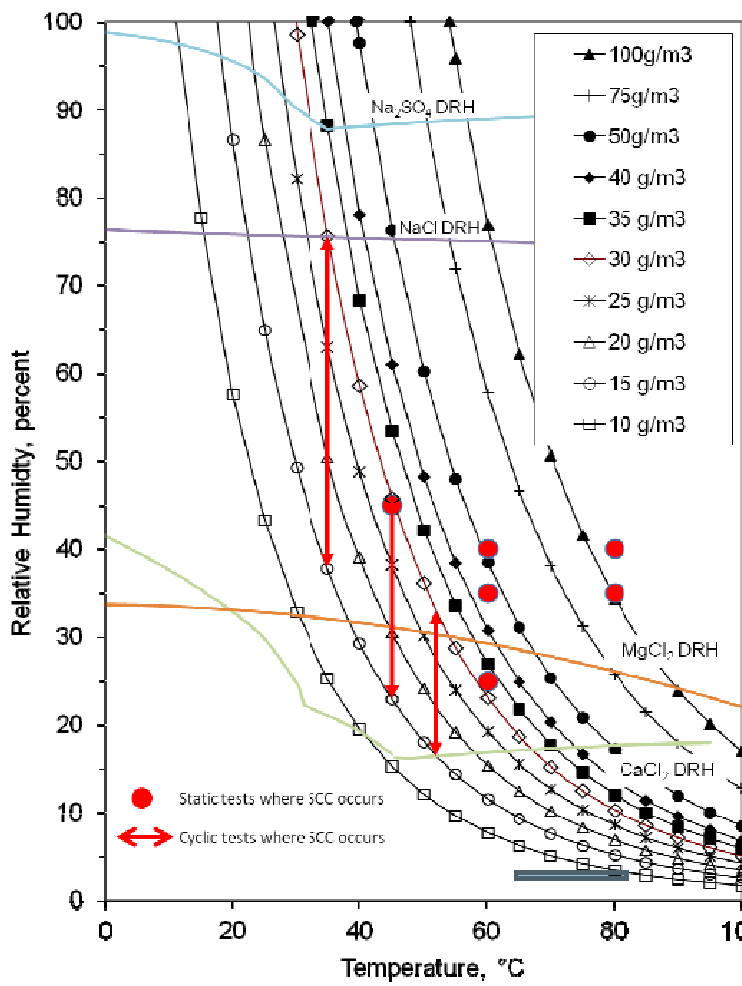

Figure 16. SCC Map for RH vs. Temperature for Cyclic and Static Tests [30]

Cook et al. [31] performed two experiments, an accelerated experiment (*aka.,* laboratory experiment) and field exposure experiment. Only the accelerated experiment with sea salt deposition is discussed from Cook's paper. In the laboratory experiment, exposure regimes were developed to deploy salt deposits to Type 304L and 316L austenitic stainless steel U-bend samples prepared in accordance with ASTM G30 standard [28] prior to the extended exposure to the corrosive environment. Cook's purpose of the laboratory experiment was to simulate natural aerosol deposition. Table 3 shows the results of the laboratory exposure experiments for 1000 hour at 80°C and 32% RH. Regime 1 corresponds to sea water, regime 2 corresponds to x10 diluted sea water, and regime 3 corresponds to x100 diluted sea water. Cook defined the term "complete failure" as a crack through the material's thickness and crack frequency as follows,

$$Failure\ frequency = \frac{No.\,of\ specimens\ in\ deposition\ regime\ having\ undergone\ complete\ failure}{Total\ No.\,of\ specimens\ in\ deposition\ regime}$$

Table 3. Laboratory Experimental Results (32% RH, 80°C, at 1000 hour) [31]

Material	Deposition regime	Average nominal salt deposition density (µg/m^2)	No. of specimens exposed	No. of failures	Cracked but not failed	Failure frequency
304L	1	370	5	5	0	1
304L	2	35	5	4	1	0.8
304L	3	4	5	2	3	0.4
316L	1	363	5	5	0	1
316L	2	34	8	6	2	0.75
316L	3	6	2	0	1	0

Worth noting that salt deposition densities from the Cook's experiment (0.0004 g/m^2) [31] were much lower than the salt deposition density from the experiment described earlier by Mintz (0.1 g/m^2) [30]. However, the results cannot be directly compared due to differences in the exposure durations and temperatures. The results from this experiment show strong correlation between cracking and salt concentration as the failure frequency was higher for regime 1 than both regimes 2 and 3. Also, failure frequency of 316L was lower than 304L emphasizing the effect of alloy composition on the susceptibility to CISCC as seen from Table 3.

Kim et al. [32] performed an accelerated experiment to evaluate welded U-bend samples made from 304L and 316L austenitic stainless steel at different temperatures and RHs. The objective of this experiment was to examine the effect of temperature and RH on welds. The samples were welded using automated gas tungsten arc weld (A-GTAW) method, welding temperature was maintained below 175°C. The samples were instrumented with a direct current potential drop (DCPD) to monitor the initiation of CISCC signal as shown in Figure 17.

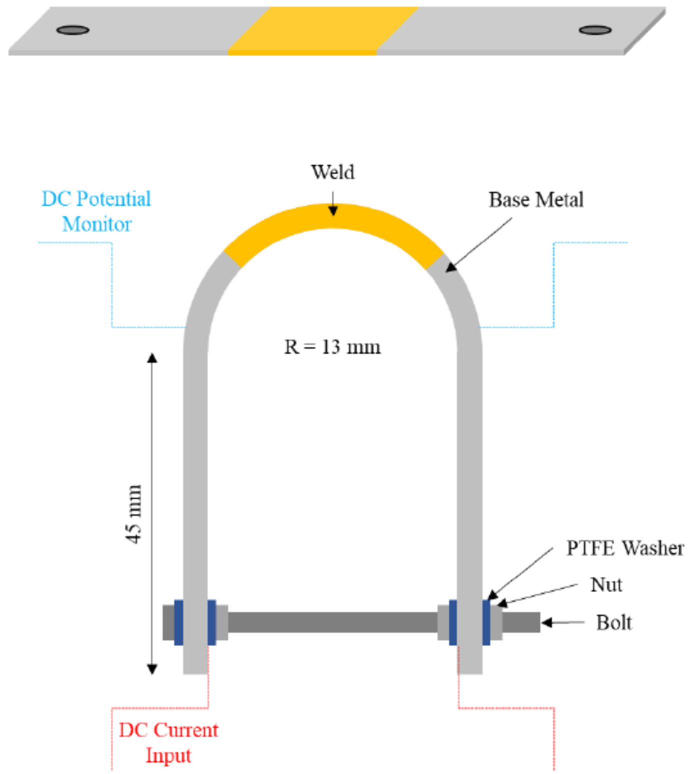

Figure 17. Sample Configuration for CISCC Signal Monitoring [32]

CISCC tests were conducted in a salt-spray chamber where 3.5 wt.% NaCl solution was sprayed every 10 hours for 30 mins. Table 4 shows the test parameters and conditions from Kim's experiment.

Table 4. Experimental Parameters from Kim's Paper [32]

Parameter	Experiment 1	Experiment 2	Experiment 3
Temperature	60°C	50°C	60°C
Relative Humidity	30%	30%	20%
Salt Concentration	3.5 wt.%	3.5 wt. %	3.5 wt. %
Duration	12 weeks	12 weeks	12 weeks
No. Samples/Material	2 samples removed every 3 weeks 304L & 316L	2 samples removed every 3 weeks 304L & 316L	2 samples removed every 3 weeks 304L & 316L

Figure 18 shows the DCPD signal for 304L and 316L samples at 60°C and RH 30%. The graph shows a sharp increase in the signal for 304L after 800 hours of immersion indicating a crack initiation. This voltage increase was not observed on the 316L sample which emphasizes the increased resistance of 316L grades to CISCC over 304L grades.

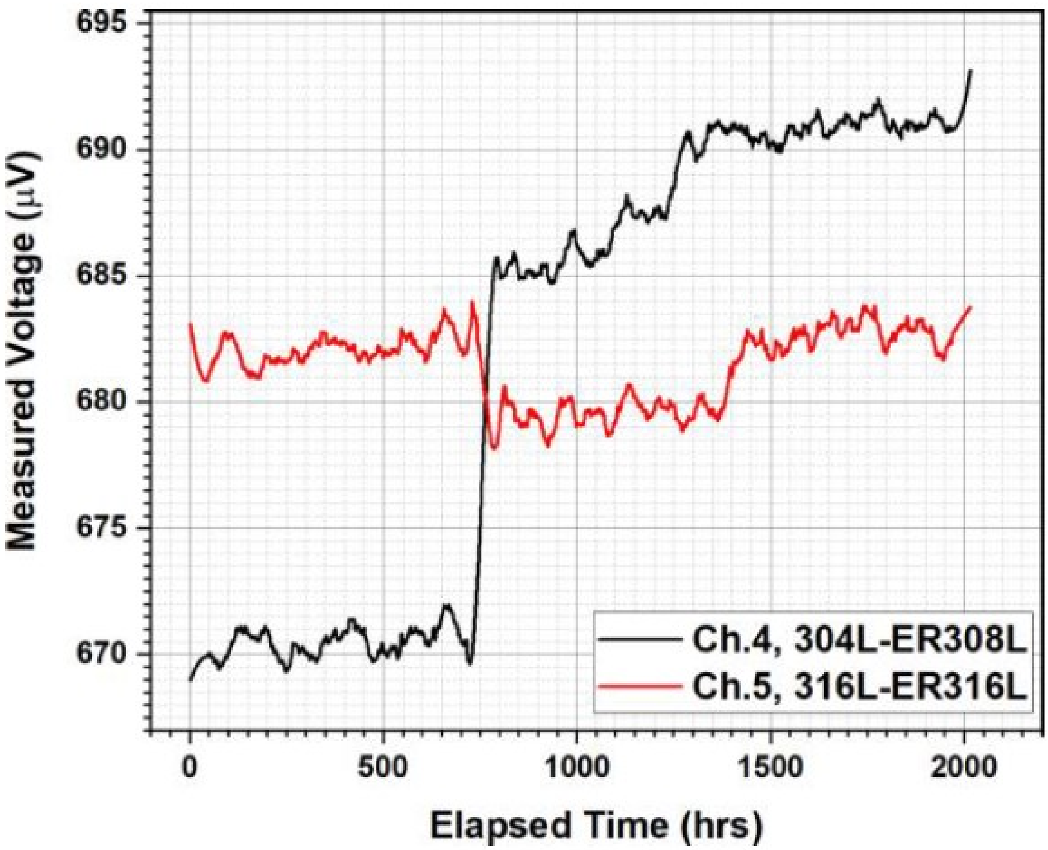

Figure 18. DCPD Results for 304 and 316 at 60°C, RH 30% [32]

Visual analysis of the sample with time elapsed is shown in Figure 19 which clearly illustrate the sharp increase of DCPD signal when cracking was initiated. Pitting was reported after 504 hours of testing, pitting advanced into cracking after the 800-hours mark as shown on the images labeled 1512 hours and 2016 hours. The agreement of the visual analysis with the DCPD signal validates the approach to detect cracking in U-bend samples. This experiment illustrates the mechanism for pitting development and crack propagation through stainless steel and emphasizes the effect of material composition to CISCC susceptibility. This paper didn't report the results of the 50°C at 30% RH or the 60°C at 20% RH experiments to evaluate the effects of RH and temperature.

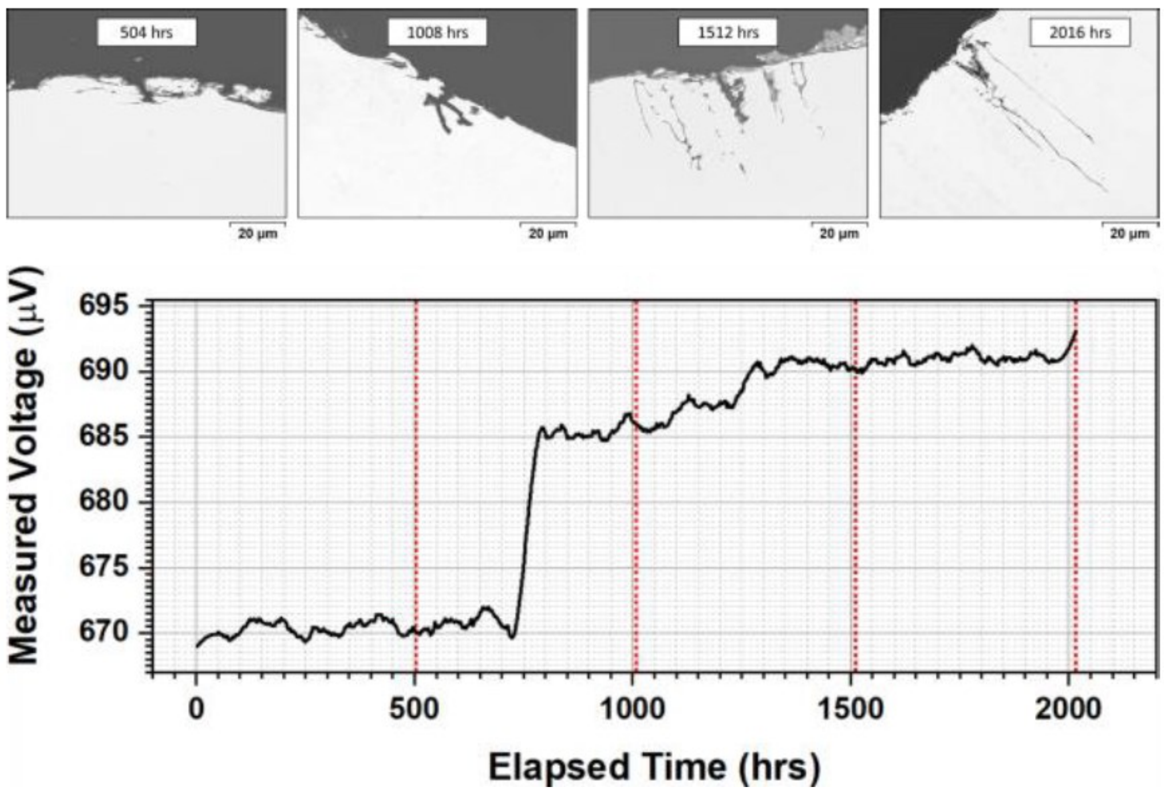

Figure 19. Pits Development and Cracks in 304L Welded Sample [32]

2.2 Material Composition and Susceptibility to CISCC

Numerous researchers studied the effect of material susceptibility and heat sensitization on CISCC. For instance, the NRC experimental results [16] clearly show increased susceptibility of 304 samples over 316L samples for both welded and unwelded configurations as shown in Figure 20 and Figure 21 (number of cracked samples/total samples are in parenthesis). At least one 304 single U-bend sample cracked after 4 and 16 weeks of exposure as shown in Figure 20, while no cracking was observed under the same experimental conditions and temperatures on the 316L U-bend samples as shown in Figure 21. After 32 weeks of exposure, three welded U-bend samples cracked while only two 316L cracked under the same conditions. The number of cracked samples was the same for the two material grades at 52 weeks of exposure indicating increased susceptibility with increased exposure duration.

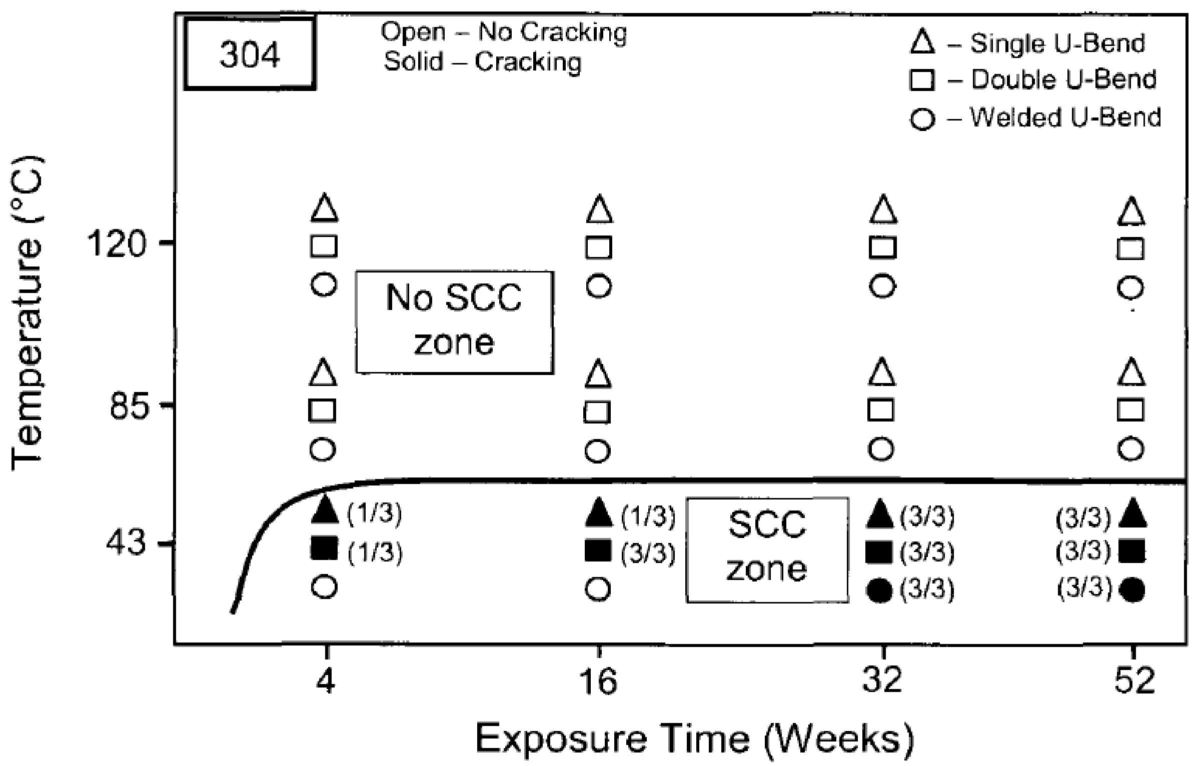

Figure 20. NRC CISCC Susceptibility Map for 304 Stainless Steel [16]

Figure 21 shows the susceptibility of welded and unwelded 316L U-bend samples. Ignore the double U-bend depicted in rectangles as these are not discussed from this paper.

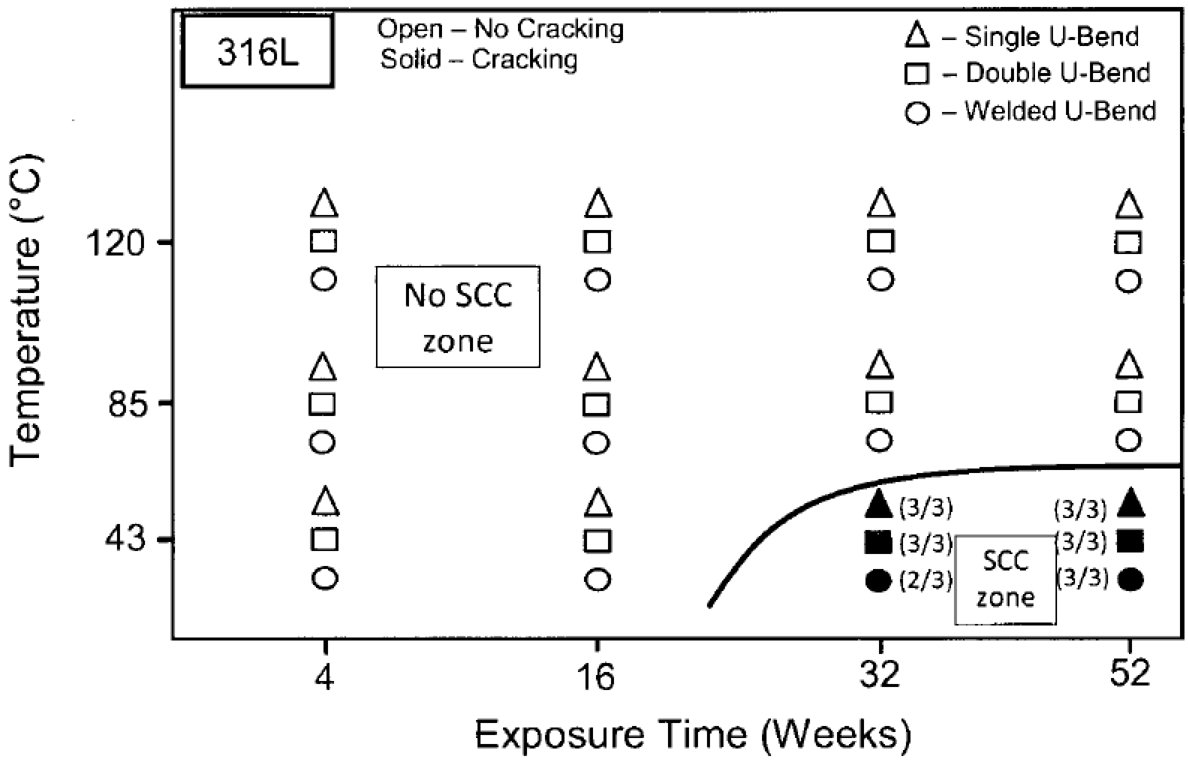

Figure 21. NRC CISCC Susceptibility Map for 316L Stainless Steel [16]

Table 3 from the Cook's experiment [31] shows increased cracking frequency of 304L U-bend samples over 316L U-bend samples for deposition regimes 2 and 3 (low salt deposition density regimes). The cracking frequency of 304L and 316L samples were the same for deposition regime 1 (high salt regime). Cook reported 7-34 $\mu g/m^2$ salt deposition densities for regimes 2 and 3 and 360 $\mu g/m^2$ for regime 3 indicating a potential relationship between material susceptibility and salt deposition density.

2.3 Heat Sensitization Effect on CISCC

Heat sensitization from welding or heat treatment has been proven to weaken the alloy's microstructure due to the depletion of chromium [21]. However, the increased susceptibility of sensitized samples to CISCC from published literature discussed in this Chapter have not been consistent. The NRC experimental results [16] discussed earlier show that U-bends cracked after 4 and 16 weeks of exposure while welded U-bends did not under the same experimental conditions. Furthermore, the results from the experiment performed by Mintz [30] indicate that welded samples were less susceptible to cracking when compared to heat sensitized and as-received samples when tested under the same conditions. Refer to Figure 14 from the Mintz experiment which shows that no cracking was observed on the welded samples after one month of exposure at 35°C and 45°C for the 10g/m^2 deposition density while cracking was observed on the as-received and heat sensitized samples. Furthermore, heat sensitized samples were more susceptible than welded and as-received samples when tested under the same conditions. The cracking frequency of heat sensitized samples was mostly higher than as-received samples for all temperatures and testing durations from the Mintz experiment.

2.4 Temperature Effect on CISCC

CISCC dependence on temperature also has not been consistent from the published literature discussed in this Chapter. The NRC experimental results in Figure 20 [16] show that cracking was only evident at 43°C, and no cracking was observed on any of the samples tested at temperatures below 25°C or above 43°C. Kosaki's experimental results [4] show that cracking was initiated in just under 30 days using an accelerated experiment with NaCl mists at 60°C. Zakaria's experimental results [27] show no evidence of cracking on all of the samples tested for 42 days at 28°C and several salt concentrations in agreement with the NRC finding. However, Zakaria found that cracks developed faster as temperature increased, cracking at 70°C and 90°C occurred in just 28 days or less contrary to the NRC findings where no cracks were observed at temperatures above 43°C. Mintz's experimental results [30] show that cracking was observed after 1 month of exposure at 35°C. Also, Mintz experimental results in Figure 14 clearly show that cracking susceptibility increases as temperature increases, and temperature effect is more prevalent with increased exposure duration.

2.5 Relative Humidity Effect on CISCC

Several research papers examined the RH threshold necessary to increase susceptibility to CISCC. For instance, Mintz [30] established a threshold RH for cracking to initiate, which is a function of exposure temperature and salt deposition density. Mintz concluded that crack initiation was observed at 60°C and 25% RH, and no cracking or pitting were observed below this RH threshold. The threshold RH was determined 35% or higher for the samples held at 80°C as shown in Figure 16. The NRC [16] allowed the relative humidity to vary from 25% to 75% based on the experiment's temperature. The NRC stated that a threshold RH exists based on their extensive experimental work to determine the optimal RH appropriate for salt deliquescence. The NRC also claimed that heated samples to 85°C and above only reached 18% RH which is below the threshold for sodium chloride salt to deliquescence. The NRC concluded that higher temperatures (80-120℃) did not accelerate SCC due to the decreased RH in the air surrounding the samples. The NRC also reported 75% RH on the samples maintained at 43°C which is appropriate for sodium chloride salt to deliquescence and promote SCC. Zapp [26] also reported the risk of SCC increases at a threshold RH known as "deliquescence relative humidity". Zapp also claimed that the deliquescence RH is a function of the salt's chemical composition and not the amount of deposited salt.

2.6 Literature Review Conclusions

Literature discussed in this Chapter shows that each CISCC experiment utilized a different methodology, experimental setup, and parameters. Variations in the experimental procedures and designs in published literature make the study to study comparisons a challenging task. CISCC experiments are also sensitive to changes in the experimental parameters and exposure conditions such as, variations in the concentrations and type of simulated salts, RH, temperature, material composition, and sample preparation methods. However, trends from previous literature clearly show that increased salt concentration coupled with a susceptible material accelerated CISCC. Additionally, data from previous literature is well established on the existence of a deliquescence threshold RH to form a corrosive salt brine on the material's surface. Data on the effects of exposure temperature, heat sensitization, and material composition from the experiments discussed in this Chapter were highly scattered and not conclusive.

Although previous studies provided valuable insights into the CISCC phenomenon, it did not provide conclusive data to establish thresholds for the factors leading to CISCC for a specific material grade. In part this is due to the differences in the experimental approach and the variability of the factors across all experiments discussed in this Chapter. Therefore, there is a need to perform CISCC experiments using a standard procedure to control parameters and eliminate variability and sources of error as practically as possible. Following a standard procedure, such as the ASTM B117, provides a uniform approach that can be reproduced for future extended testing to develop "time to failure" models and accurately estimate DSCs service lifetime. Another advantage of using a standardized procedure is the ability to attribute acceleration in CISCC or crack propagation to a particular parameter. Quality assurance controls and experimental procedures following recognized industry standards were utilized and emphasized in the experimental work discussed in the next Chapter.

3. Chapter 3: Experiment Design and Procedure

3.1 Experimental Setup

A high-level schematic of the accelerated CISCC experimental setup is shown in Figure 22. The main components are the corrosion chamber, compressed air supply, air dryer, Type IV water filtration system, and the exhaust outlet. The experimental setup relies primarily on the corrosion chamber that is depicted by the yellow box. Compressed air passes through an industrial air dryer and is directly connected to the corrosion chamber as depicted in the blue lines. A dedicated water filtration system was installed at the lab to produce Type IV water (red lines). The green exhaust line is a direct connection from the corrosion chamber to the atmosphere to exhaust fogging and to vent the chamber for data collection.

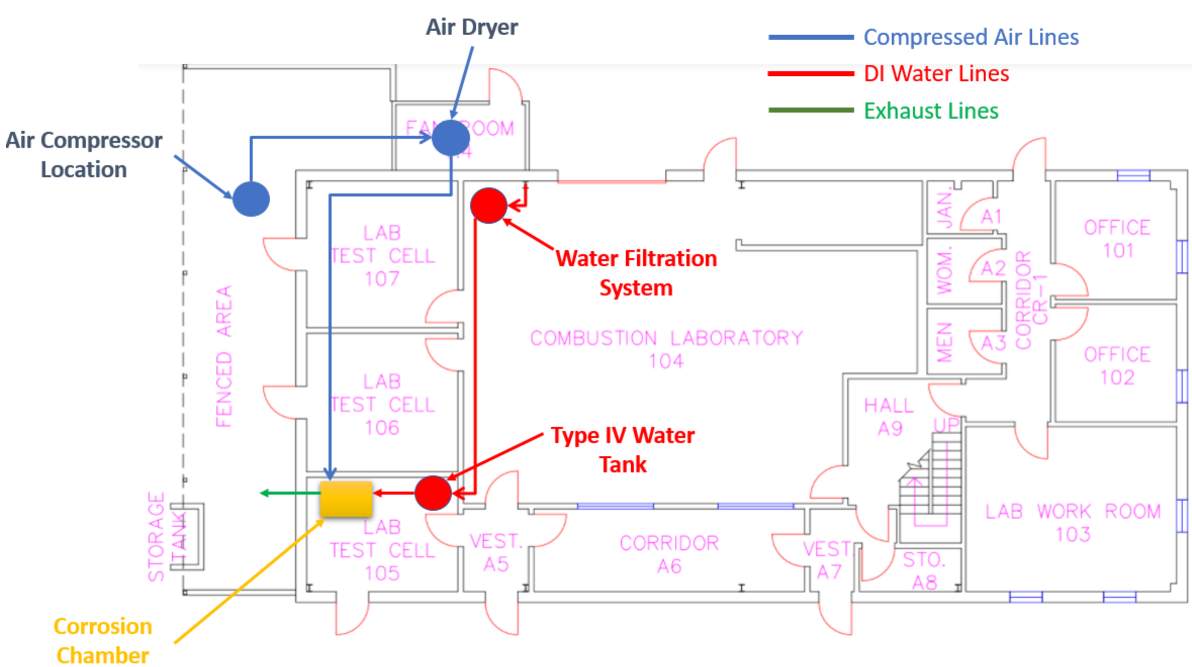

Figure 22. Experimental Setup and Equipment Locations

3.2 Corrosion Fogging Chamber

The corrosion chamber (Singleton Model SCCH22) houses the stainless steel samples and provides the necessary simulated corrosive conditions. The Singleton SCCH22 chamber is designed, built, and calibrated in accordance with the ASTM B117-19 standard (Standard Practice for Operating Salt Spray (Fog) Apparatus) [33]. Calibration and conformity certificates for the SCCH22 chamber are included in Appendix A.

The main components of this chamber are the salt solution tank, level-matic tank, humidifying tower tank, sample racks, and the control system as shown in Figure 23. The chamber's internal dimensions are 48" x 30" x 36" with an internal volume of 30 ft^3. The SCCH22 chamber runs at a constant RH of 100% which cannot be varied during the experiment. The control system displays the exposure zone, dry bulb, and wet bulb temperatures with the ability to vary the exposure zone temperature between ambient and 50C°. The salt is mixed manually and thus salt concentration can be adjusted per the experiment's requirement.

The chamber features a water jacket design with Polypropylene covers, the water jacket ensures a tight seal around the covers during operation and to prevent fogging leakage. Additionally, the chamber has a data logging capability through PLC communication and control software for monitoring the experiment conditions remotely.

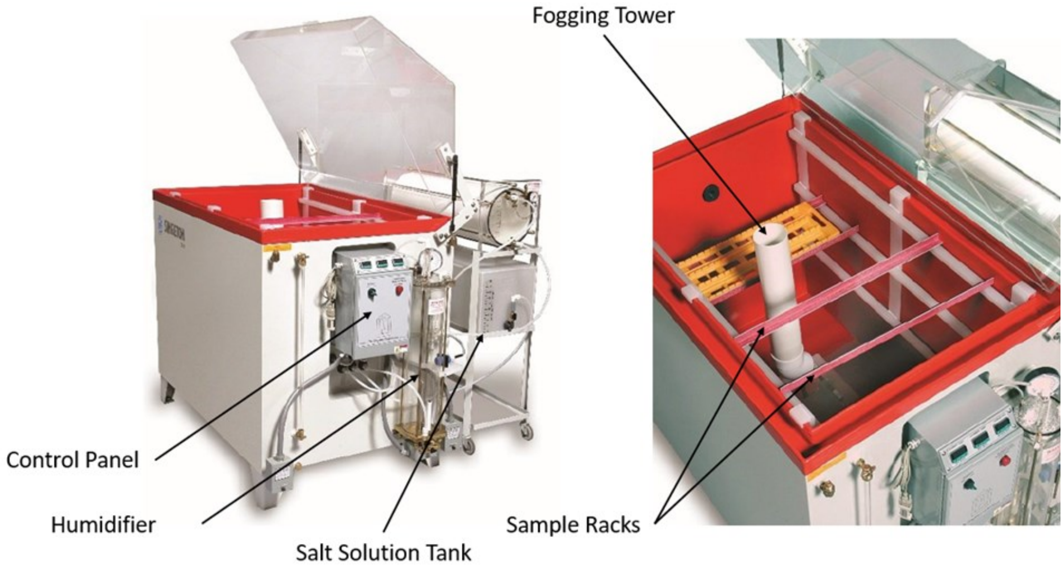

Figure 23. Singleton SCCH22 Corrosion Chamber [34]

Refer to Figure 24 and Figure 25 for a detailed view of chamber's components [34] that could be referenced in this Chapter.

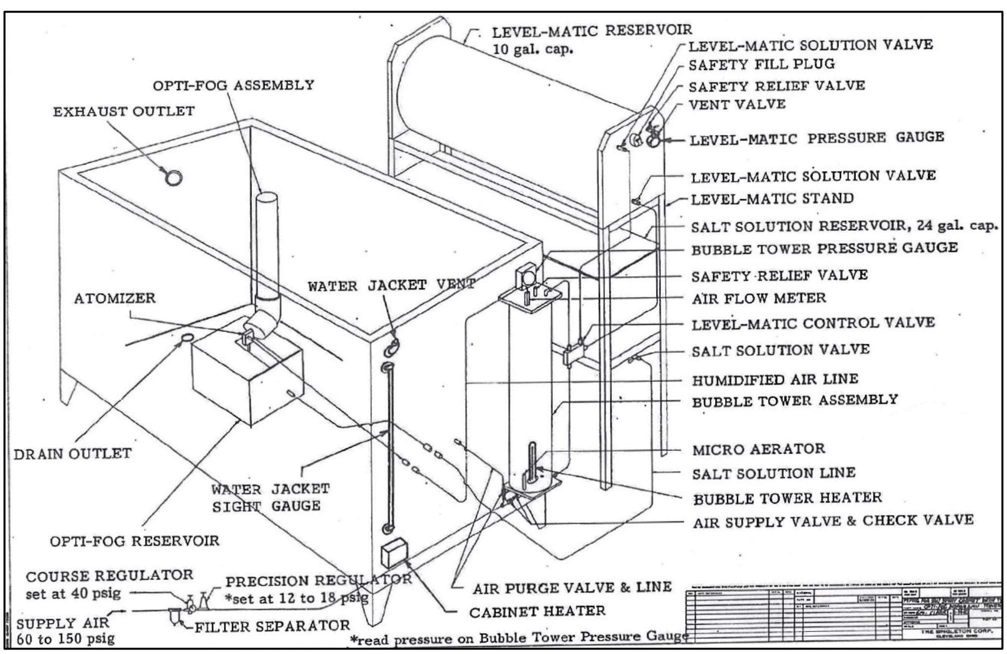

Figure 24. Drawing of Singleton SCCH22 Corrosion Chamber (Manual)

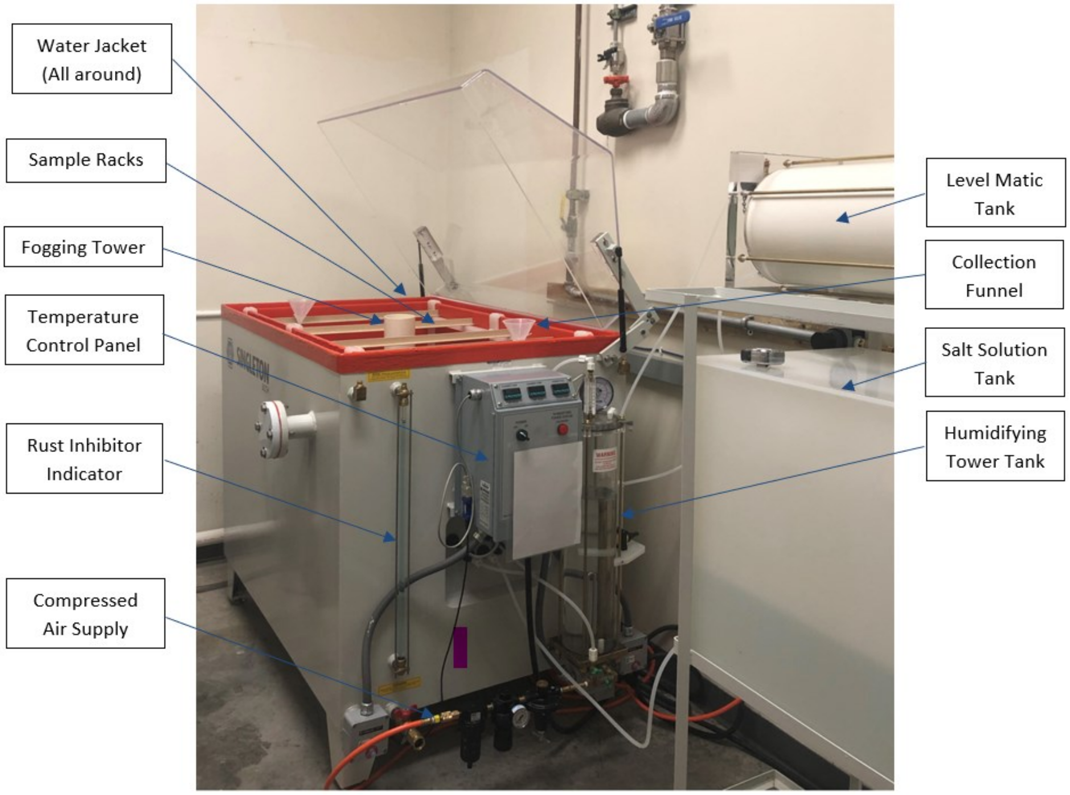

Figure 25. Corrosion Chamber SCCH22 Major Components

3.3 Compressed Air Supply

A 5 HP piston type air compressor was used to provide a constant air supply at 3CFM and 60 PSI which is necessary to operate the corrosion chamber [35]. Compressed air passes through an oil and water separator first and then air dryer to remove humidity before reaching the corrosion chamber. At the corrosion chamber, compressed air passes through another air filter and the regulator assembly shown in Figure 26. The coarse air regulator reduces the pressure of incoming air from 60 to 40 PSI, and the precision regulator further reduces the air pressure from 40 to 15 PSI before reaching the humidifying tower tank. This process ensures only clean and dry air at the appropriate pressure gets to the atomizer, and prevents oil and dirt collections which may cause clogging of the atomizer's fine orifices and affect fogging rate.

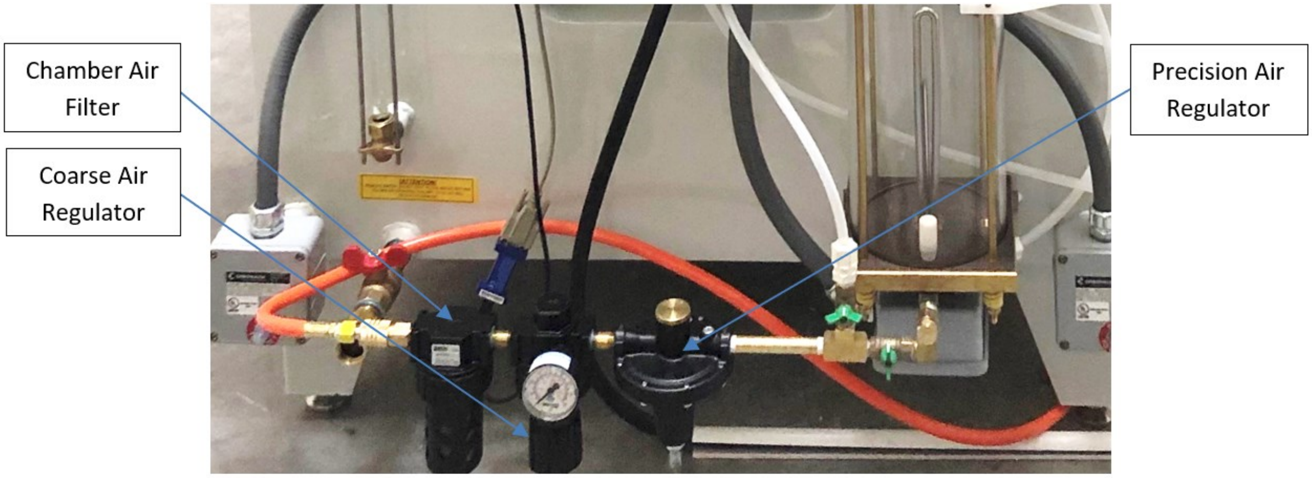

Figure 26. Filter-Regulator Assembly for Compressed Air Supply

3.4 Water Filtration System

Type IV water was purified at the lab through a Culligan LC Series filtration system installed in the laboratory [36]. The filtration system comes with a 55-gallon tank and maintains Type IV water supply that meets the requirements of ASTM D1193 with a conductivity ≤1.0 μS/cm (or resistivity ≥1.0 MΩ·cm) [37]. Type IV water was also used to prepare the 5% salt solution.

3.5 U-bend Samples

The samples used in this experimental work were U-bends and welded U-bends made from 304 and 304L austenitic stainless steel. U-bend samples were fabricated in accordance with the ASTM G30 standard requirements and made from a flat stainless steel strip and then shaped and fixed with a bolt/nut as shown on Figure 27 [28]. Welded U-bend samples were fabricated in accordance with the ASTM G58 standard requirements and made from a welded flat stainless steel strip and then shaped as shown in Figure 28 [29]. Note that welds at the tip of the welded U-bend samples were polished and may not be visible to the naked eye.

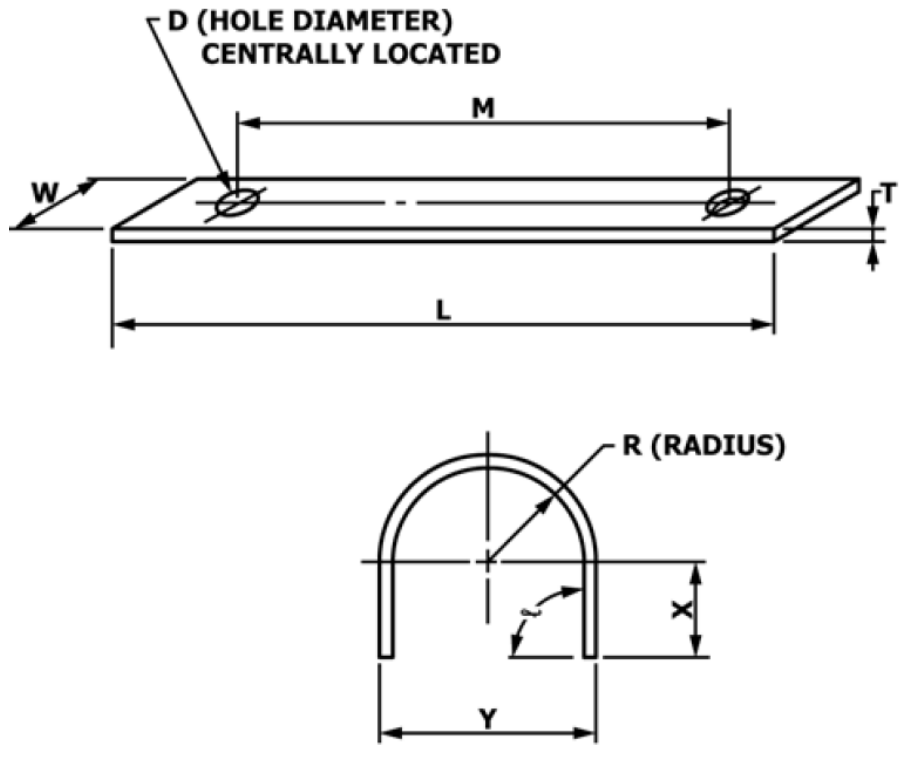

Figure 27. U-bend Sample Configuration [28]

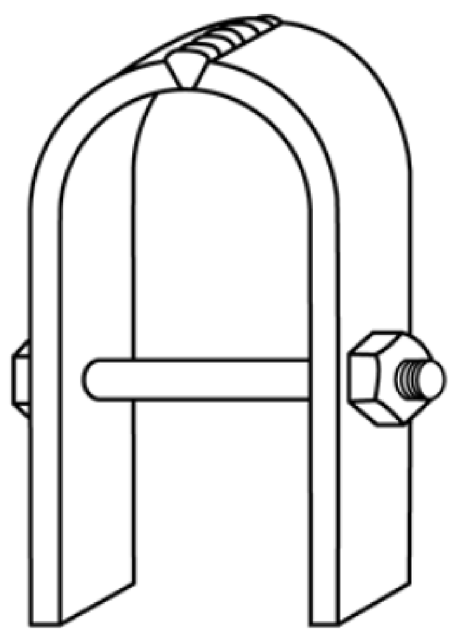

Figure 28. Welded U-bend Sample Configuration [29]

Table 5 shows the dimensions and finish of the U-bend samples.

Table 5. Dimensions of U-bend Sample [28]

Feature	Dimension
Thickness (T)	1.57 mm
Width (W)	19.05 mm
Length (L)	127 mm
Radius (R)	12.7 mm
Finish	120 grit

Methods for stressing U-bend samples from the ASTM G30 standard are shown in Figure 29. These methods introduce applied stress into the samples through a single stage stressing method. For this experiment, the applied stress was considered constant across all samples and was not measured or quantified. A discussion on potential variations in the applied stress among the samples is included in Chapter 4.

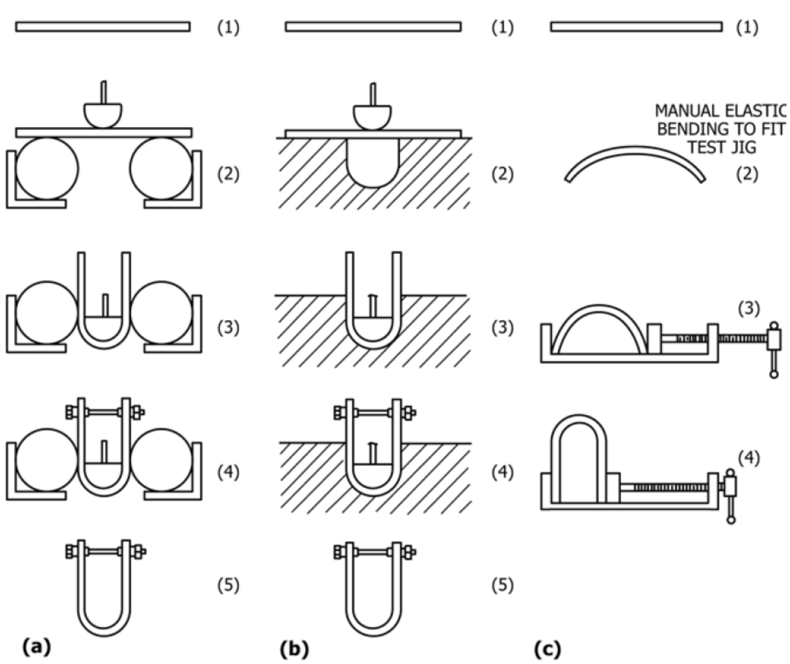

Figure 29. U-bend Sample Stressing per ASTM G30 Standard [28]

Figure 30 shows a picture of the actual samples. Each sample is stamped with the material grade (304 or 304L), sample identification number, and the designation “W” for welded samples (304W or 304LW).

Figure 30. U-bend Sample Picture

A total of 78 U-bend and welded U-bend samples made from stainless steel 304 and 304L grades were used in this experimental work. Chemical composition and mechanical testing certificates of the U-bend samples are included in Appendix B. Below is the breakdown of the 78 U-bend samples used in this experiment,

- 24 - 304 U-bends (stamped with 304 & serial number)
- 18 - 304 Welded U-bends (stamped with 304W & serial number)
- 18 - 304L U-bend Samples (stamped with 304L & serial number)
- 18 - 304L Welded U-bends (stamped with 304LW & serial number)

3.6 Experimental Preparations

This section details the steps required to prepare the SCCH22 chamber for experimentation in accordance with the chamber's operations manual and the ASTM B117-19 standard requirements.

The First step in preparing the chamber for experimentation is mixing the 5% by weight salt solution. Salt solution is prepared manually by mixing 20.74 Ibs of Morton Culinox 999 salt [38] with 50 gallons of Type IV water. Culinox salt has 99.99% sodium chloride as shown on the salt certificates provided in Appendix C. The 5% salt solution is then poured into the salt solution tank shown in Figure 25. One full salt solution tank (50 gallons) lasts for approximately three weeks of continuous operation. Note that the salt solution tank is connected to a small reservoir (*aka.,* Opti-fog reservoir) at the bottom of the fogging tower at the center of the chamber. The valve between the salt solution tank and the Opti-fog reservoir remains open during the experiment to maintain a constant supply of salt solution.

The second step is filling the level-matic and humidifying tower tanks with Type IV water. The humidifying tower tank must be maintained at two-thirds full to ensure a constant supply of humid hot fogging to the chamber. The level-matic tank supplies Type IV water to the humidifying tower tank and automatically maintains it at the appropriate level of two-thirds full. One full level-matic tank lasts for approximately one week of continuous operation. The mobile cart shown in Figure 25 next to the corrosion chamber holds both the level-matic and the salt solution tanks.

The third step is ensuring that the fogging tower is positioned as practically as possible at the center of the corrosion chamber to ensure a uniform fog distribution throughout the corrosion chamber and onto the samples.

The fourth step is placing the two collection funnels (collectors) in the appropriate locations as shown in Figure 31. The collection funnels are used to measure collection rates inside the chamber at two opposite locations. The required collection rate per the ASTM B117 standard must be within 1-2 ml/hour. The chamber was calibrated by the factory for this range as shown on the calibration certificates provided in Appendix A. Higher or lower than normal collections can be corrected by adjusting the atomizer's angle at the bottom of the fogging tower (*aka.,* atomizer tower) or by adjusting the fogging tower's position to the center of the chamber.

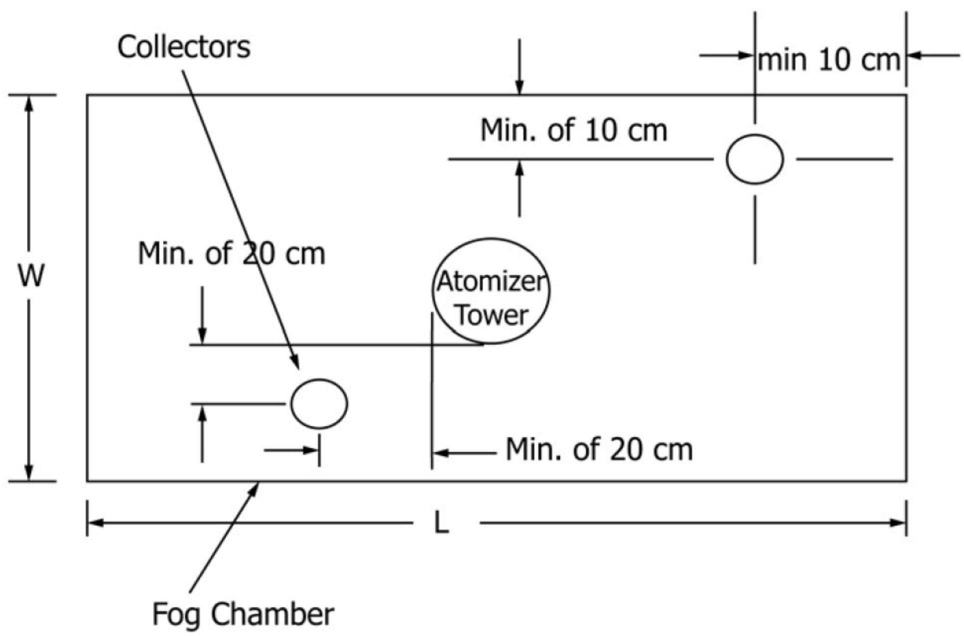

Figure 31. Collection Tube Locations in the Corrosion Chamber [33]

The fifth step is placing the samples on the angled racks inside the chamber as shown in Figure 32, the sample racks are designed angled at 30° per the requirements of the ASTM B117-19 standard. The racks closer to the fogging tower were positioned approximately 5 inches from the center of the tower, and the racks farther from the fogging tower were approximately 10 inches from the center of the fogging tower.

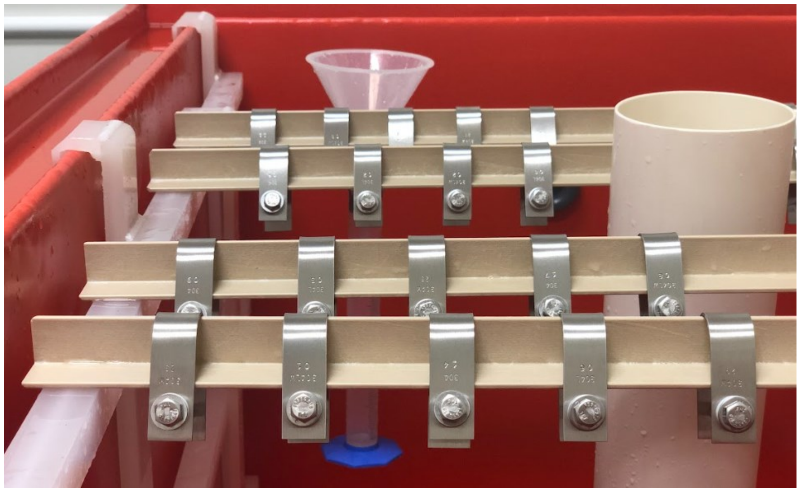

Figure 32. U-bend Samples Positioned on Racks Ready for Testing

3.7 Experimental Campaigns

Two experimental campaigns were performed for this thesis, Table 6 shows the test conditions of each campaign. Each sample was weighed to the nearest tenth of a milligram before starting the experiment, weights are included in Appendix D. Experimental campaign 1 was performed at 35°C as mandated by the ASTM B117-19 standard and experimental campaign 2 was performed at 50°C. In both campaigns, the same set of samples were utilized to compare the results and characterize susceptibility to CISCC.

Table 6. Experimental Campaigns and Test Conditions

Parameter	Experimental Campaign 1	Experimental Campaign 2
Relative Humidity	100%	100%
Exposure Zone Temperature	35°C	50°C
Salt Concentration	5% by weight	5% by weight
Testing Duration	4, 8, 12 weeks	4, 8, 14 weeks
Number of U-bend Samples per testing duration (total of 39 for each experiment)	4x 304 3x 304 Welded 3x 304L 3x 304L Welded	4x 304 3x 304 Welded 3x 304L 3x 304L Welded

Refer to Figure 33 and Figure 34 for experimental campaigns 1 and 2 diagrams, respectively. Sample locations were determined randomly, the stamped sample number is placed in parentheses next to the sample type.

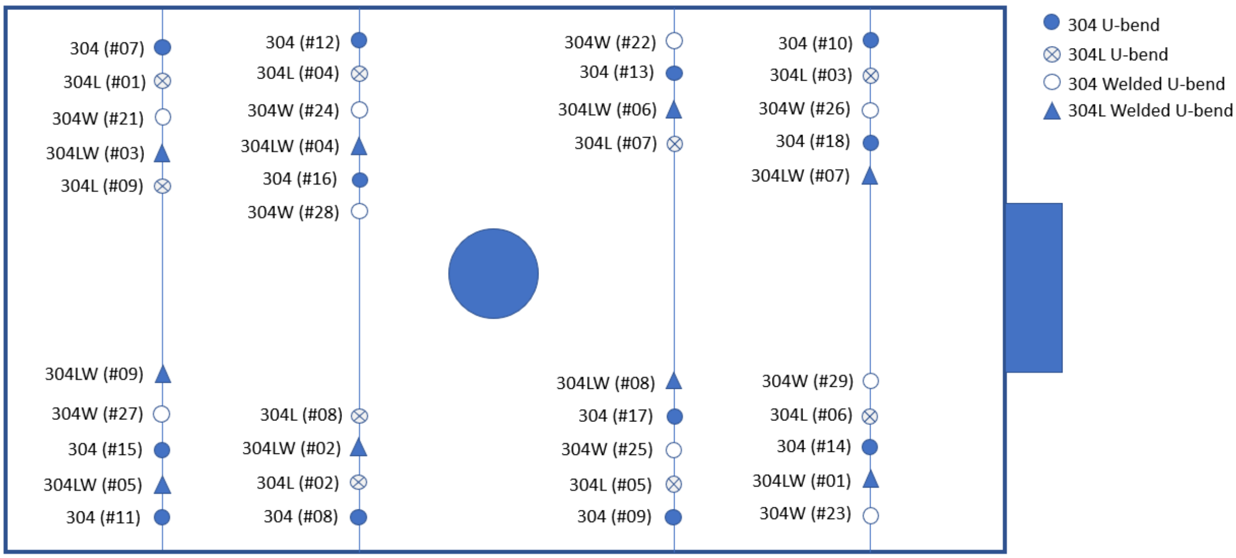

Figure 33. Experimental Campaign 1 Diagram (35°C) – U-bend Sample Positions

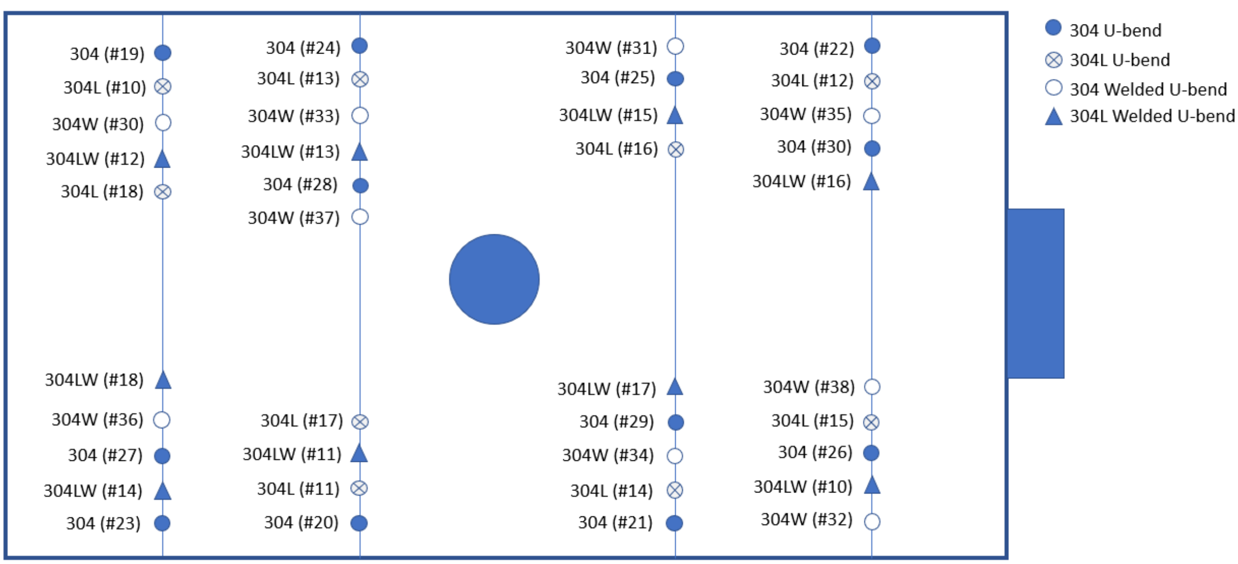

Figure 34. Experimental Campaign 2 Diagram (50°C) – U-bend Sample Positions

Note the sample racks are made from plastic material that does not corrode or impact the sample's corrosion rate. Figure 35 shows a fully loaded chamber with 39 samples ready for experimentation.

Figure 35. Experimental Setup and Sample's Placement in Corrosion Chamber

3.8 Operational Procedure

The level-matic tank supplies Type IV water to the humidifying tower during operation, the humidifying tower heats incoming water then bubbles are generated through a micro aerator located at the bottom of the tower (compressed air is required for this operation). The fine aerosols (fogging) pass through the salt solution in the Opti-fog reservoir to the atomizer which is located directly above the Opti-fog reservoir; and then the salty fogging is channeled into the chamber through the fogging tower. Collected salt solution at the bottom of the chamber drains directly to the main drainage lines in the lab through a drain outlet. The chamber runs continuously at the preset exposure temperature (35°C or 50°C) until it is manually shut down by the experiment's operator to either collect data or remove a set of samples for characterization (after 4, 8, 12, or 14 weeks). Refer Figure 36 shows the corrosion chamber in operation, notice the clouded chamber through the top cover.

Figure 36. SCCH22 Corrosion Chamber in Operation

Regular data collection is mandated by the ASTM B-117 standard to ensure that the experimental conditions are within the required parameters. It is required that no more than 96 hours would elapse between data collections as long as the experiment is running. The fogging chamber was turned off every 40-60 hours typically for data collection. The following required data points were collected each time the chamber was turned off which took 1.5 hours on average,

- Accumulation rate of each collection tube
- Temperature of each collection tube
- pH level of each collection tube
- Exposure zone temperature
- Dry and wet bulb temperatures

Table 7 shows the lower and upper limits of the experimental parameters as specified in the ASTM B117 standard. Deviations from these limits during the experimental work would be corrected immediately to bring the parameters within the required range.

Table 7. Experimental Parameter Limits Per ASTM B117 Standard [33]

Data	Lower Limit	Upper Limit
Accumulation rate of collection tubes (mL/hr.)	1.00	2.00
Temperature of accumulated solution, T (°C)	20.0	26.0
pH level of accumulated solution at T	6.5	7.2
Exposure zone temperature (°C)	35 or 50	
Relative Humidity (%)	95 - 100	

Table 8 shows an example of the data collected every 40-60 hours throughout the experimental work. The complete set of data collected during experimental campaigns 1 and 2 including sample weights before and after exposure are included in Appendix D.

Table 8. Example Data Collected During Experimental Campaign 1 (4 Weeks)

Data Collection																		
Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
11/14/2021	10:30 PM	72.00	11:30 PM	4	Overflow	94	Overflow	1.305	24.7	25.0	6.95	6.91	1.030 - 1.035	1.035	95.2	95.4	119.5
11/16/2021	4:40 PM	41.17	6:40 PM	4	93	65	2.259	1.579	24.0	22.7	6.97	6.99	1.035 - 1.040	1.035 - 1.040	95.7	95.9	118.9
11/18/2021	3:10 PM	44.50	4:40 PM	3	51	71	1.146	1.596	24.5	23.9	6.98	7.02	1.035 - 1.040	-	95.9	96.1	119.0
11/20/2021	12:10 PM	43.50	2:00 PM	4	89	67	2.046	1.540	22.9	23.0	6.90	6.92	1.035 - 1.040	1.035 - 1.040	95.0	95.0	119.3
11/22/2021	10:00 AM	44.00	11:30 AM	3	51	69	1.159	1.568	22.7	23.1	6.98	6.95	1.035 - 1.040	-	95.5	95.7	117.0
11/24/2021	8:30 PM	57.00	10:00 PM	4	53	72	0.930	1.263	23.0	23.5	6.85	6.94	1.035 - 1.040	1.035 - 1.040	95.0	95.0	118.0
11/26/2021	1:00 PM	39.00	2:30 PM	3	55	71	1.410	1.821	23.5	23.0	6.95	6.96	1.035 - 1.040	1.035 - 1.040	95.4	95.7	118.4
11/28/2021	5:30 PM	51.00	7:00 PM	3	59	43	1.157	0.831	25.8	24.4	7.03	7.04	1.040	1.040	95.2	95.7	118.8
11/30/2021	2:30 PM	43.50	4:00 PM	4	108	58	2.483	1.333	23.2	22.7	7.00	6.93	1.035 - 1.040	-	94.6	94.6	117.0
12/2/2021	2:30 PM	46.50	3:45 PM	4	80	50	1.720	1.075	24.7	23.9	6.94	6.98	1.040	-	95.7	96.1	119.1
12/4/2021	10:00 AM	42.25	11:15 AM	3	64	40	0.947	1.515	24.5	23.4	6.98	7.00	1.035 - 1.040	1.035 - 1.040	94.8	94.8	118.2
12/6/2021	10:30 AM	47.25	12:00 PM	3	71	59	1.503	1.249	25.0	24.3	7.04	7.06	1.035	1.035	95.9	96.1	118.6
12/8/2021	3:30 PM	52.50	4:50 PM	4	82	66	1.652	1.257	23.7	23.4	6.76	6.79	1.035 - 1.040	1.035	88.0	87.3	120.2
12/10/2021	12:30 AM	43.67	N/A	4	65	73	1.489	1.672	23.2	22.5	6.83	6.82	1.035	1.040	95.5	95.9	118.0

3.9 Quality Control Data Collection

Prior to the start of experimental campaigns 1 and 2, a trial set of six 304 U-bend samples were tested at 35°C for 3 weeks to adjust the parameters within the desired limits. The trial experiment confirmed the functionality of the chamber, and the limits of all essential variables such as, exposure temperature, RH, and salt concentration. The following data points were collected for quality assurance for all experiments, although these are not essential to the experiment:

- Purity of the Type IV water, this was measured using a TDS meter to measure the total dissolved solids (TDS) in ppm. TDS measurements were collected to ensure that the water filtration system is working as expected and the total dissolved solids are within range for Type IV water (<5 ppm). TDS periodic measurements during the experimental work are included in Appendix D.
- Specific gravity of each collection tube. Specific gravity measurements are included in Appendix D.
- Available salt solution vs. solution consumed
- Available level-matic Type IV water vs. water consumed
- Humidifying tower and level-matic tanks pressures to ensure the compressed air pressure is maintained within the required range of 12-15 PSI.

Additionally, the Type IV water tank was recycled 2-3 times a week for one hour each time during the experimental work to prevent salts settling at the bottom of the tank which could affect the chemistry of Type IV water. Quality data was checked after each instance of data collection to ensure the chamber and the Type IV water filtration system are functioning as expected.

3.10 Post Experiment Procedure

Four weeks after the start of each experimental campaign, one third of the samples (four 304, three 304L, three 304W, and three 304LW) were removed from the chamber. This process was repeated eight and twelve weeks after the start of experimental campaign 1 (35°C) and 8 and 14 weeks after the start of experimental campaign 2 (50°C). Each experimental campaign was concluded with the removal of the final batch of samples. The diagram in Figure 37 shows the samples removed after 4 weeks of testing at 35°C; the complete set of diagrams of removed samples after each exposure duration in both experimental campaigns are included in Appendix E.

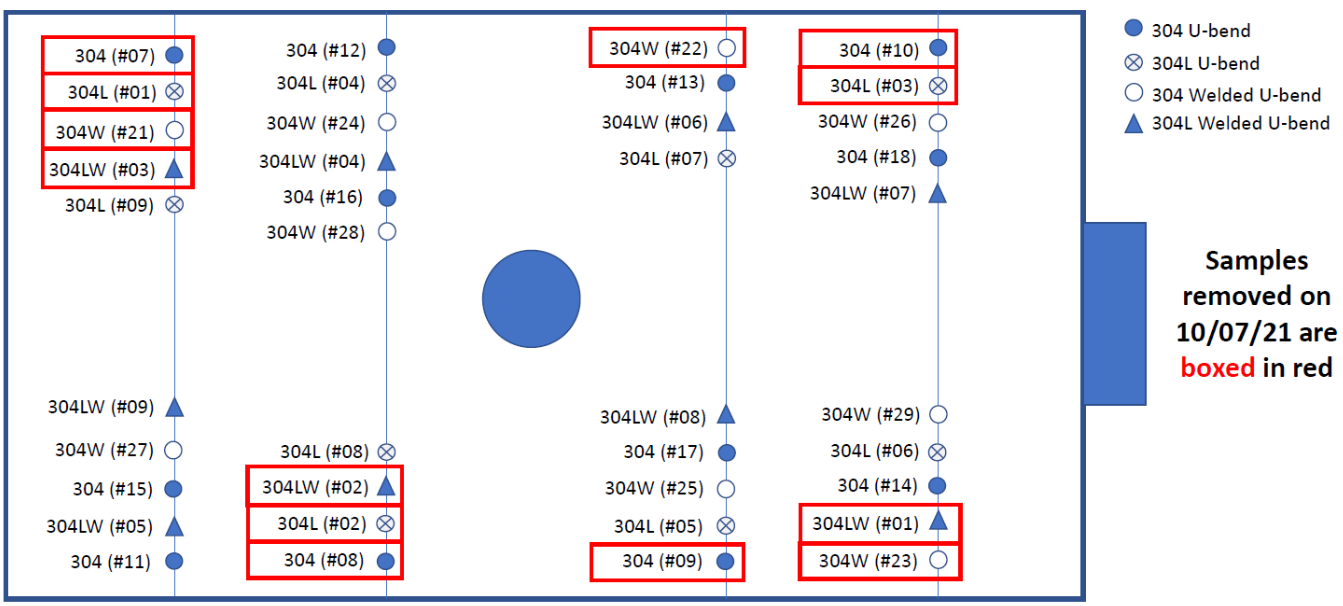

Figure 37. U-bend Samples Extracted After 4 Weeks of Experimental Campaign 1

Table 9 and Table 10 show the complete set of samples removed after each exposure duration for experimental campaigns 1 and 2, respectively (sample number in parentheses).

Table 9. U-bend Samples Removed in Experimental Campaign 1 (35°C)

Exposure Duration	U-bend Samples Removed			
4 weeks	304 (#07) 304 (#08) 304 (#09) 304 (#10)	304L (#01) 304L (#02) 304L (#03)	304W (#21) 304W (#22) 304W (#23)	304LW (#01) 304LW (#02) 304LW (#03)
8 weeks	304 (#11) 304 (#12) 304 (#13) 304 (#14)	304L (#04) 304L (#05) 304L (#06)	304W (#24) 304W (#25) 304W (#26)	304LW (#04) 304LW (#05) 304LW (#06)
12 weeks	304 (#15) 304 (#16) 304 (#17) 304 (#18)	304L (#07) 304L (#08) 304L (#09)	304W (#27) 304W (#28) 304W (#29)	304LW (#07) 304LW (#08) 304LW (#09)

Table 10. U-bend Samples Removed in Experimental Campaign 2 (50°C)

Exposure Duration	U-bend Samples Removed			
4 weeks	304 (#19) 304 (#20) 304 (#21) 304 (#22)	304L (#10) 304L (#11) 304L (#12)	304W (#30) 304W (#31) 304W (#32)	304LW (#10) 304LW (#11) 304LW (#12)
8 weeks	304 (#23) 304 (#24) 304 (#25) 304 (#26)	304L (#13) 304L (#14) 304L (#15)	304W (#33) 304W (#34) 304W (#35)	304LW (#13) 304LW (#14) 304LW (#15)
14 weeks	304 (#27) 304 (#28) 304 (#29) 304 (#30)	304L (#16) 304L (#17) 304L (#18)	304W (#36) 304W (#37) 304W (#38)	304LW (#16) 304LW (#17) 304LW (#18)

At the end of each exposure duration, the 13 removed samples were then washed in Type IV water, dried with compressed air, photographed, weighed, inspected visually for any signs of cracking, and stored in air-tight containers pending microscopic analysis and characterization. Samples were weighed before and after exposure to quantify weight loss which could be an indication of corrosion rate or cracking potentially. Weights of all samples listed in Table 9 and Table 10 before and after exposure are included in Appendix D. Figure 38 shows a sample photograph before and after exposure at 50°C for 14 weeks. Notice the heavy signs of corrosion on the sample post exposure in the corrosion chamber in comparison with the clean sample.

Figure 38. U-bend Sample Before and After Exposure to 50°C for 14 Weeks

Chapter 4 discusses the material characterization methods, pitting and cracking results, and the analysis of results. Additionally, the effects of temperature, heat sensitization (welding), material composition, and exposure duration on CISCC are discussed in Chapter 4.

4. Chapter 4: Characterization Results and Analysis

This Chapter details the sample characterization techniques used to examine the U-bend samples after exposure to the accelerated corrosive conditions, the results, and the analysis of results.

At the end of experimental campaigns 1 and 2, and after washing and drying the samples, an Olympus BH-2 optical microscope was used to scan all samples for signs of pitting and corrosion. The Olympus Microscope did not reveal cracking or pitting due to limitations on the magnification level, but it was used to select samples that showed heavy signs of corrosion for further analysis.

Selected U-bend samples with heavy signs of corrosion were scanned with a Micro Computed Tomography (CT) scanner at the Federal University of Rio De Janeiro in Brazil for crack detection. The scanner model used is SKYSCAN 1273, this scanner features a combination of high energy X-ray source running at higher power with a large format flat-panel detector which provides excellent image quality in seconds. The SKYSCAN 1273 is capable of testing samples up to 500 mm length, 300 mm diameter, and a maximum weight of 20 kg. Refer to Table 11 for the SKYSCAN 1273 specifications and Figure 39 for a picture of the machine used.

Table 11. Specifications of SKYSCAN 1273

Feature	Specification
X-ray source	40-130 kV 39 W
X-ray detector	Active pixel CMOS flat-panel, 6 MP (3072 x 1944)
Object size	250 mm diameter 250 mm height
Power supply	100-240 VAC, 50-60 Hz, 3 A max

Figure 39. SKYSCAN 1273 at LIN (Nuclear Instrumentation Laboratory)

The MicroCT characterization provided scans at small angular increments generating 360° scans of the entire U-bend sample. Additionally, the samples were characterized at small increments along the cross-section starting from the tip of the arch region through the entire length of the sample. Refer to Figure 40 for a schematic of the scanning procedure used. SKYSCAN 1273 is capable of detecting cracks larger than 7μm, but it did not reveal any cracking or pitting on the twelve (12) scanned U-bend samples from both experimental campaigns 1 and 2. If cracking is present, it could potentially be smaller than 7μm. Refer to Figure 41 for a sample MicroCT rotational and cross-sectional scans, additional sample MicroCT scans are included Appendix F.

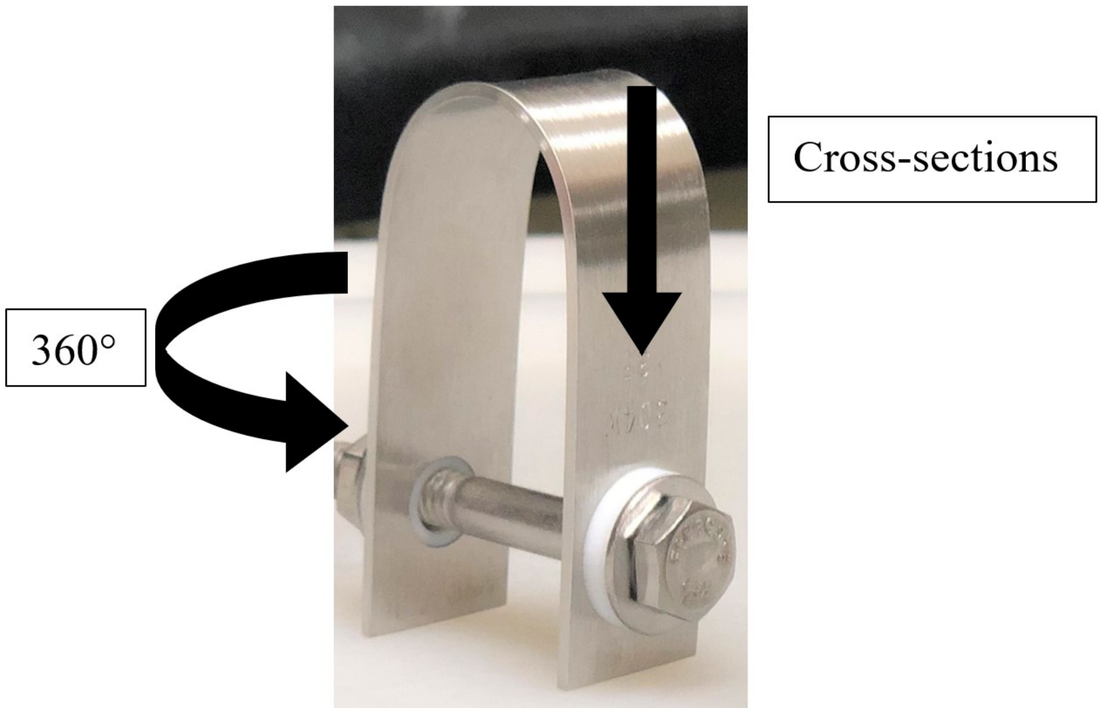

Figure 40. Scanning Procedure with SKYSCAN 1273

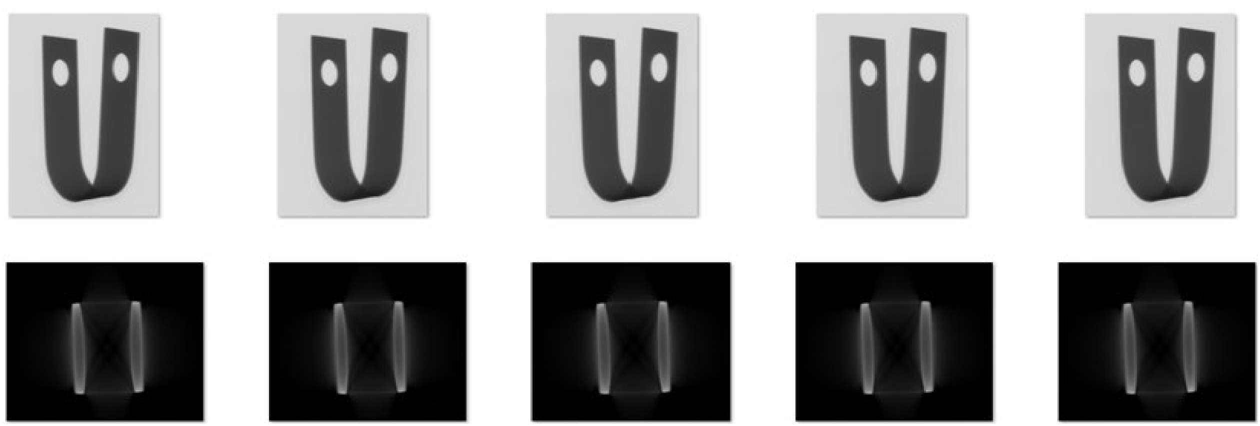

Figure 41. Example MicroCT Scans (SKYSCAN 1273)

The next tool selected for pitting and crack characterization is a 3D Surface Profiler Model VK-X3000 that is shown in Figure 42. This scanner was used to characterize a selected set of samples with the heaviest signs of corrosion based on the Olympus Microscope observations.

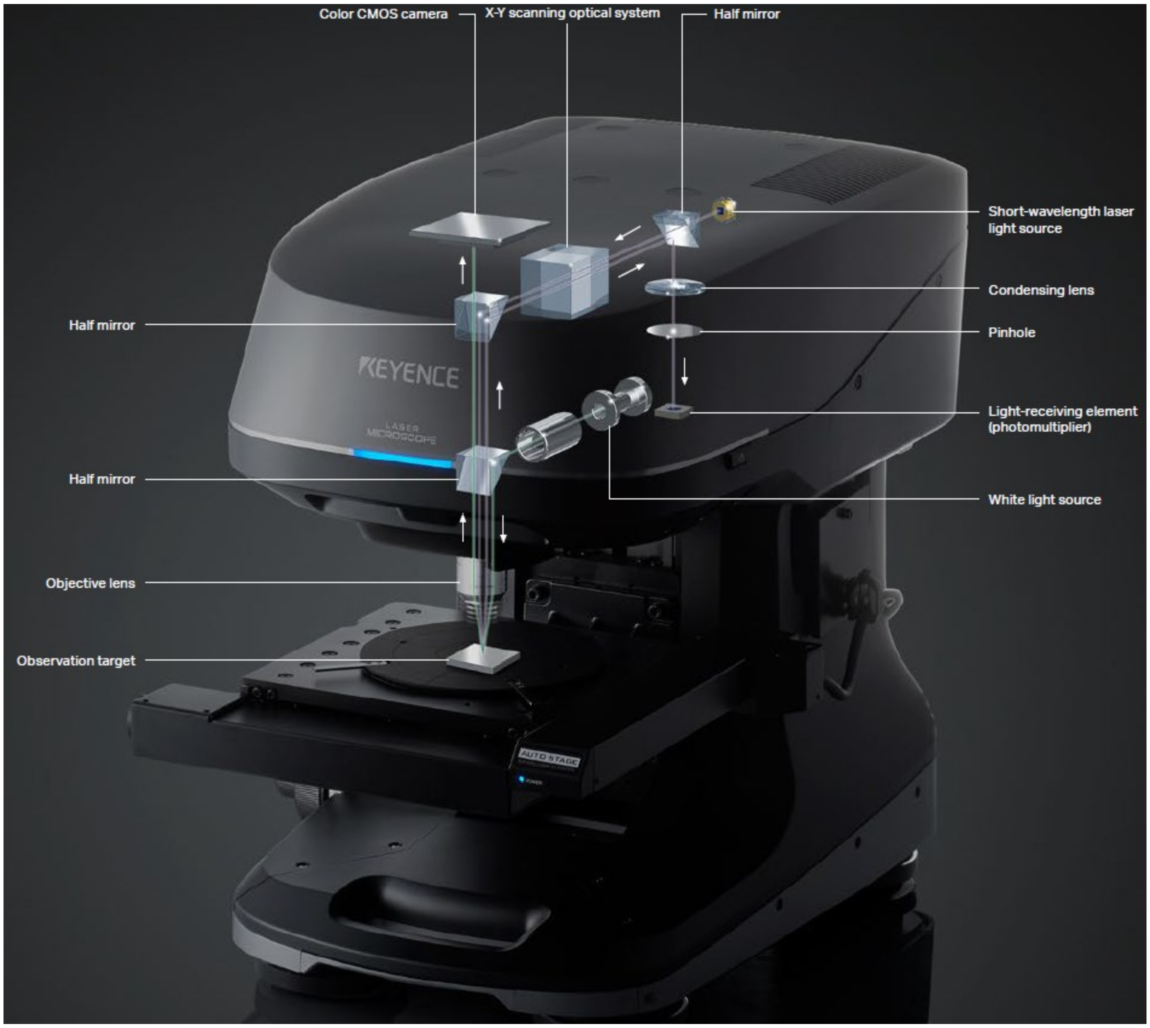

Figure 42. Keyence VK-X3000 3D Surface Profiler

The 3D Surface Profiler provides a magnification level of up to x28,800 with non-contact surface scanning and automatic focusing. The 3D Surface Profiler provides high quality optical micrographs along with a surface profile report in one step. The U-bend samples selected for profile characterization are listed in Table 12. None of the samples tested for 4 weeks from either experimental campaign were analyzed with the 3D Surface Profiler because of the lower level of corrosion observed with the optical microscope, only the samples tested for eight or more weeks.

Table 12. Selected U-bends for Characterization with 3D Surface Profiler

Test Duration	Experimental Campaign 1 (35°C)			
8 weeks	304 (#11)	304L (#06)	304W (#26)	304LW (#06)
12 weeks	304 (#16)	304L (#07)	304W (#27)	304LW (#08)
Test Duration	**Experimental Campaign 2 (50°C)**			
8 weeks	304 (#26)	304L (#14)	304W (#35)	304LW (#13)
14 weeks	304 (#28)	304L (#17)	304W (#38)	304LW (#16)

4.1 3D Surface Profiler Results

Surface profiles were generated only at the arch region of the U-bend samples as depicted in Figure 43. The applied tensile stress at the arch region is the highest and it is the most likely location to show signs of pitting and cracking. Approximately, the same region was scanned on all sixteen (16) U-bend samples to establish surface profiles for a qualitative microscopic characterization and quantitative statistical analysis for comparisons among the samples.

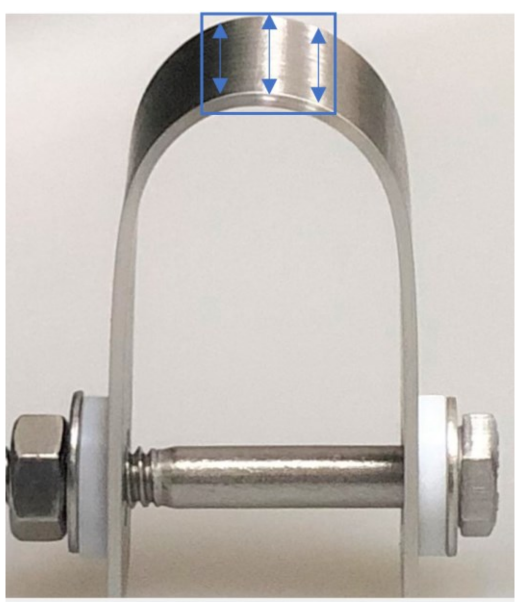

Figure 43. Characterization Region with 3D Surface Profiler

On average, 10 surface profiles were generated for each U-bend sample listed in Table 12. Additionally, two reference samples made from 304 and 304W that had never been exposed to corrosive conditions were scanned. Each surface profile represents an area approximately 300-400 μm^2. Reference U-bend samples were fabricated to the same standard as the samples used in the experimental work and were supplied by the same manufacturer. Therefore, the reference samples were used for comparisons with the corroded samples to determine what is considered a normal surface defect and what is considered abnormal such as pitting or cracking.

For each scanned surface, a micrograph with the associated surface profile report was saved for further visual analysis. Figure 44 shows a micrograph of the reference 304 sample and Figure 45 shows a micrograph of the 304 sample exposed to 50°C for 14 weeks, both at 50x magnification.

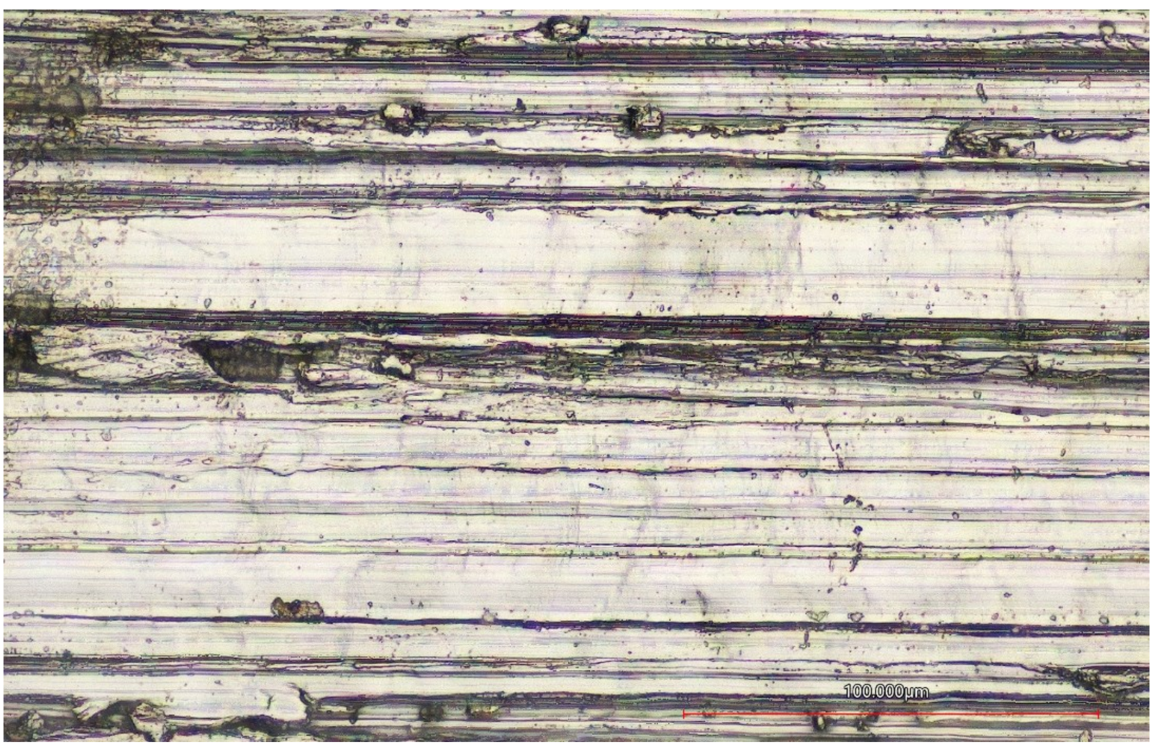

Figure 44. Optical Micrograph at the Arch of Reference 304 U-bend Sample

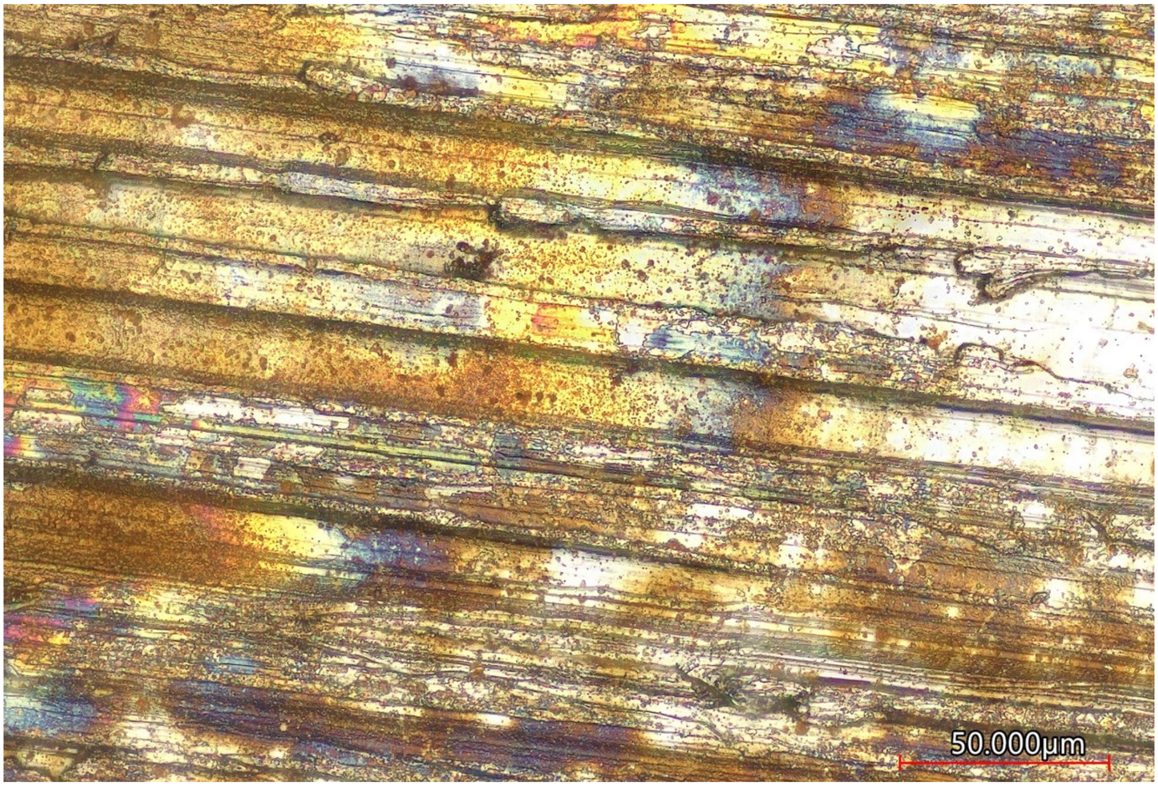

Figure 45. Optical Micrograph at the Arch of 304_50°C_14 Weeks U-bend Sample

Figure 46 shows an example surface profile characterization report. The report shows the optical micrograph on the left and the surface height profile on the right. The heigh profile shows the peaks and valleys (depth) to a reference plane relative to the sample itself, plane correction was done automatically in the 3D Surface Profiler. Beside the profile report, additional micrographs at various magnification levels ranging from 10x to 50x were saved for each U-bend to qualitatively characterize pitting and cracking. The height color bar on the far right of the report shows the height color gradient of the scanned surface and lists the maximum peak's height (+) and the maximum valley depth (-). The maximum valley data of all scanned surfaces were used to quantitatively characterize pitting and cracking. Additional optical micrographs and surface profile reports are included in Appendix G.

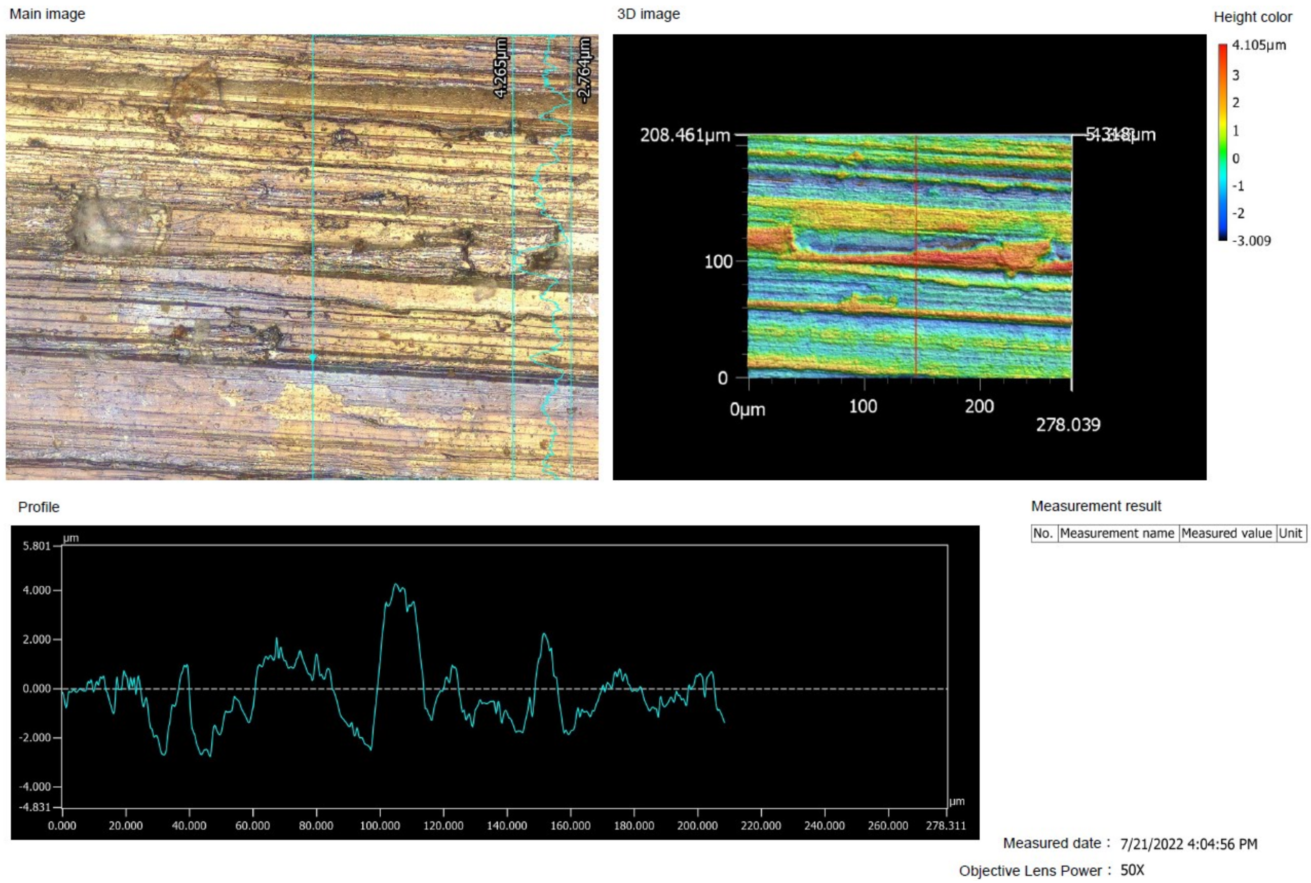

Figure 46. Example 3D Surface Profiler Report

4.2 Qualitative Cracking and Pitting Results

Surface profiles and micrographs were examined and compared with the reference samples to qualitatively characterize pitting and cracking. This is a qualitative comparative analysis and therefore a procedure was established to determine the criteria of normal surface defects based on observations from the reference surface profiles (clean samples). Below are the observations from the reference optical micrographs and a discussion of the approach used for crack characterization. The first criterion is the direction of crack propagation; cracks are expected to be perpendicular to the applied stress as depicted in the blue arrows in Figure 47. To distinguish the direction of cracks on micrographs, cracks must be perpendicular to the material polishing lines.

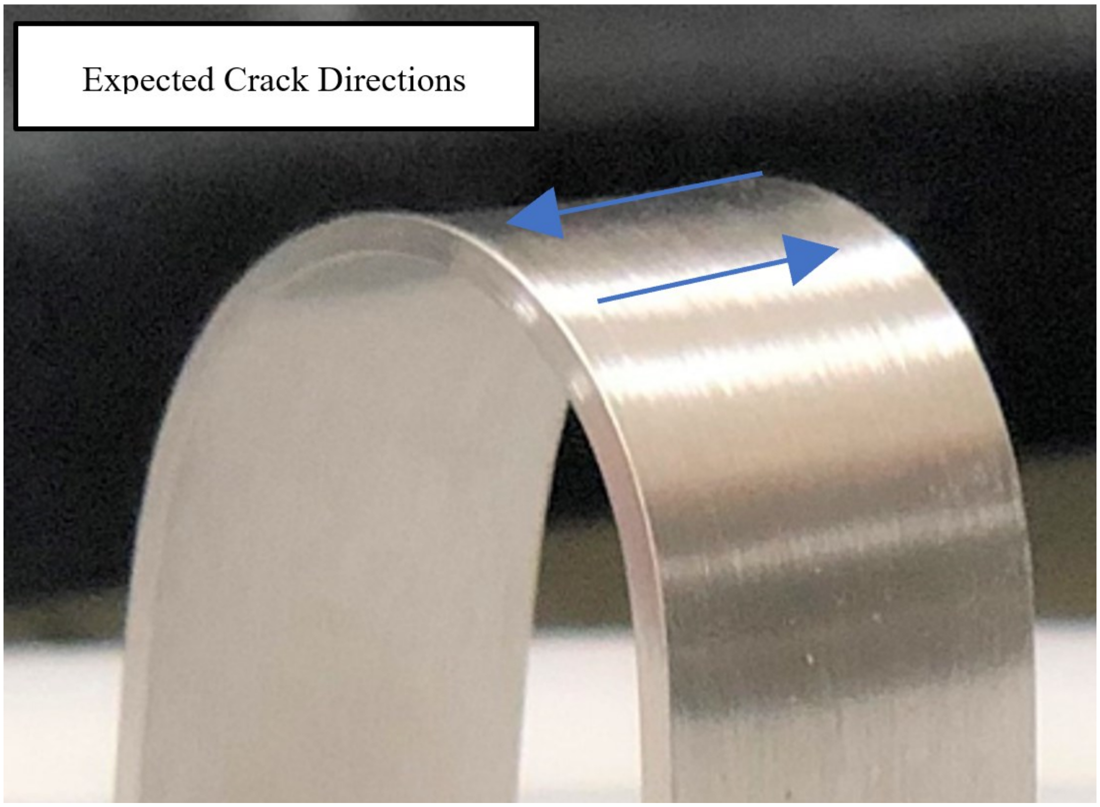

Figure 47. Expected Crack Directions, Perpendicular to Applied Stress

Optical micrographs and surface profiles of the reference 304 and 304W samples were examined to establish additional criteria of normal surface features. Figure 48 shows an optical micrograph of the reference 304 U-bend sample along with the height profile on the right. Peaks are ignored for the purposes of characterizing pitting and cracking. Notice the dark blue lines and valleys that are aligned with the material polishing lines (along the red arrows) and not perpendicular to the applied stress. Furthermore, these lines are straight and do not branch out or wave away perpendicular to the material polishing lines. Such lines were not labeled cracks when observed on corroded samples and were considered normal surface features. Refer to Appendix G for the complete set of surface profiles collected from the reference 304 U-bend sample along with large-scale optical micrographs.

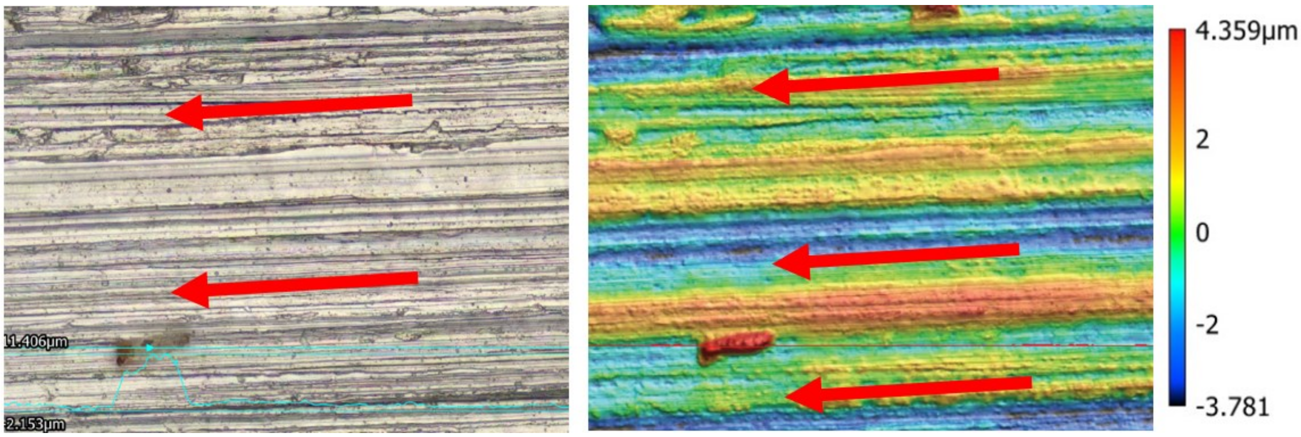

Figure 48. Profile of Reference 304 Sample at 50x Magnification

Additionally, the wide blue horizontal valleys (channels) marked with red arrows on the height profile in Figure 48 were considered normal polishing lines as these were consistently observed on the reference samples. Also, horizontal polishing lines are typically larger than 10 micrometers in width as opposed to a few micrometers which is the expected width of cracks.

Figure 49 shows a micrograph of the welded reference 304W U-bend sample along with the height profile. As stated earlier, peaks are ignored for the purposes of characterizing pitting and cracking. Similar to the previous observations on the 304 reference sample, horizontal blue lines that are aligned with material polishing lines and not perpendicular to the applied tensile stress are considered normal surface features. Additionally, these lines are straight and do not branch out or wave away perpendicular to the applied tensile stress. Another important observation, the edges surrounding peaks such as the one circled in Figure 49 may have the same appearance as cracks on the associated micrographs. Therefore, micrographs and the associated height profiles were compared side to side to distinguish these edges on the height profile appearing in dark blue from cracks. Edge lines were labeled cracks only if significant branching from the edges perpendicular the applied tensile stress is observed.

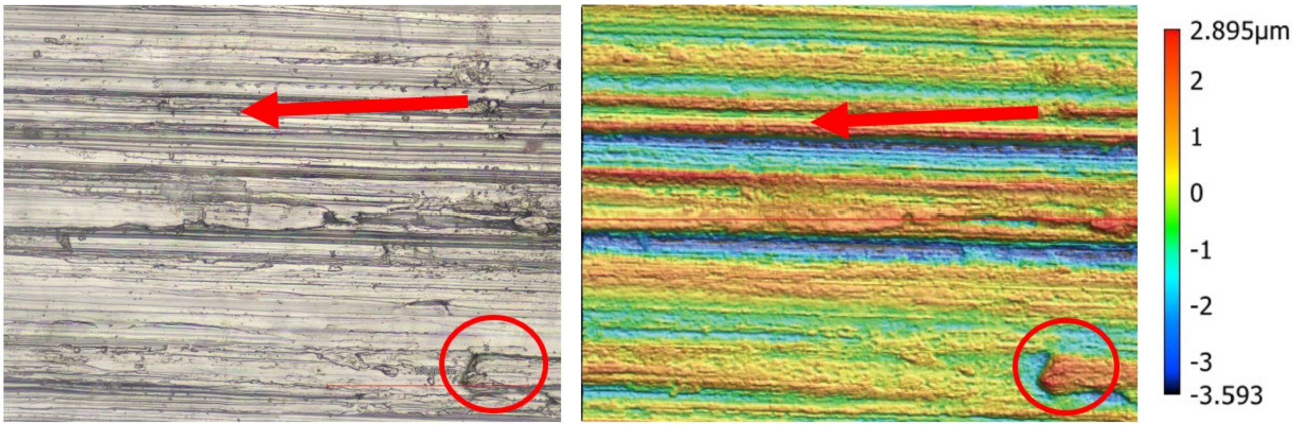

Figure 49. Profile of Reference 304W Sample at 50x Magnification

The micrographs and surface profiles of the sixteen (16) corroded samples were visually analyzed and compared with the reference samples discussed earlier. Approximately, 6-12 profiles on each sample were visually analyzed for signs and patterns consistent with cracking patterns. Note that determination of whether the cracks are intergranular or transgranular was not possible since the characterized samples were not polished, and the grain boundaries were not visible on these micrographs.

One limitation on the 3D Surface Profiler that must be considered while analyzing these profiles is the ability to distinguish small cracks from the surrounding material based on the color profile. The expected cracks are on the order of a few micrometers in width, and the optical light on the 3D Surface Profiler may not be able to penetrate these cracks and thus not differentiate cracks from the surrounding material. However, subtle color gradients for small cracks were noticed on the height profiles and careful examination is required to detect those subtle differences. Table 13 shows the qualitative results of pitting and cracking on the sixteen (16) corroded U-bend samples.

Table 13. Cracking and Pitting Results of 16 Corroded Samples

Test Duration	Experimental Campaign 1 (35°C)			
8 weeks	**304 (#11)**	**304L (#06)**	**304W (#26)**	**304LW (#06)**
	Cracking Light Pitting	Cracking Light Pitting	Likely Cracking Heavy Pitting	Cracking Heavy Pitting
12 weeks	**304 (#16)**	**304L (#07)**	**304W (#27)**	**304LW (#08)**
	Cracking Moderate Pitting	Cracking Moderate Pitting	Cracking Heavy Pitting	Likely Cracking Heavy Pitting
Test Duration	**Experimental Campaign 2 (50°C)**			
8 weeks	**304 (#26)**	**304L (#14)**	**304W (#35)**	**304LW (#13)**
	Cracking Light Pitting	Likely Cracking Light Pitting	Cracking Heavy Pitting	Likely Cracking light Pitting
14 weeks	**304 (#28)**	**304L (#17)**	**304W (#38)**	**304LW (#16)**
	Cracking Heavy Pitting	Cracking Heavy Pitting	Cracking Heavy Pitting	Cracking Heavy Pitting

Figure 50 shows a micrograph of the 304 sample exposed to 35°C for 8 weeks at 50x magnification. A few small cracks circled in blue appear to be developing in the expected direction perpendicular to the applied stress; such lines were not observed on the reference samples. Additionally, the color profile for these lines is different from the surrounding material indicating a deeper surface. Moreover, the observed lines in Figure 50 show signs of branching as opposed to the straight polishing lines on the reference samples. Cracks were also observed on the 304L and 304LW samples exposed to 35°C for 8 weeks as shown in Figure 51 and Figure 53, respectively. Micrographs of the 304W sample exposed to 35°C for 8 weeks did not show cracking lines consistent with the established criteria except for one "likely cracking" observation that is shown in Figure 52.

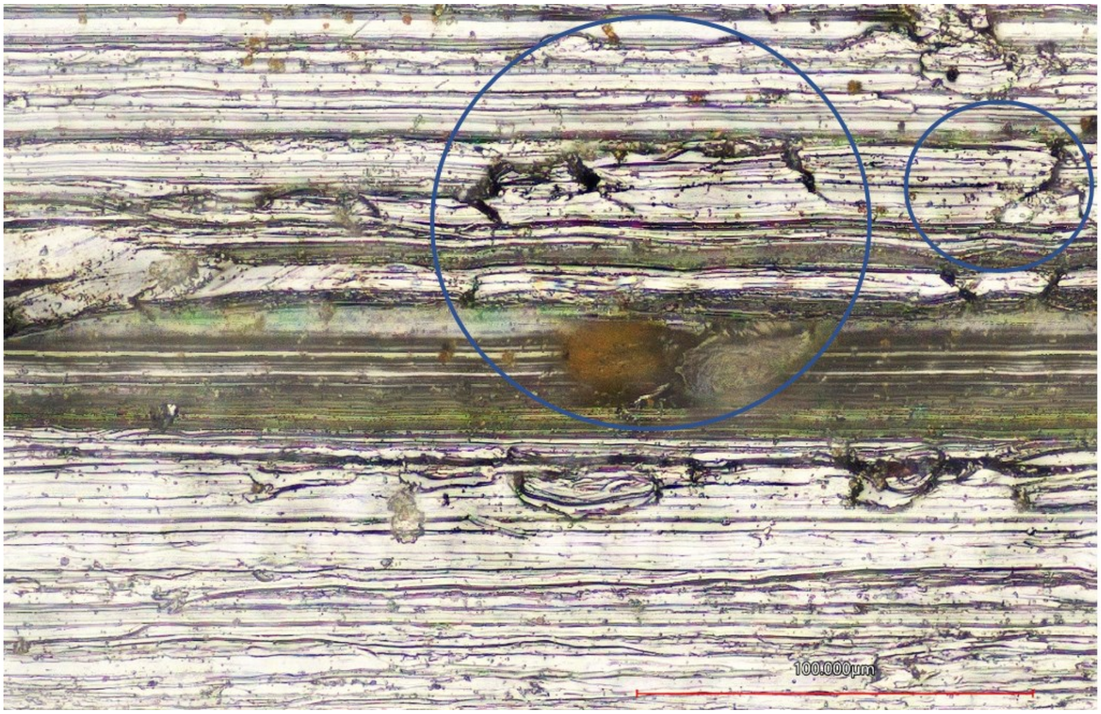

Figure 50. Cracking at the Arch Region on 304 Exposed to 35°C for 8 Weeks

Figure 51. Cracking at the Arch Region on 304L Exposed to 35°C for 8 Weeks

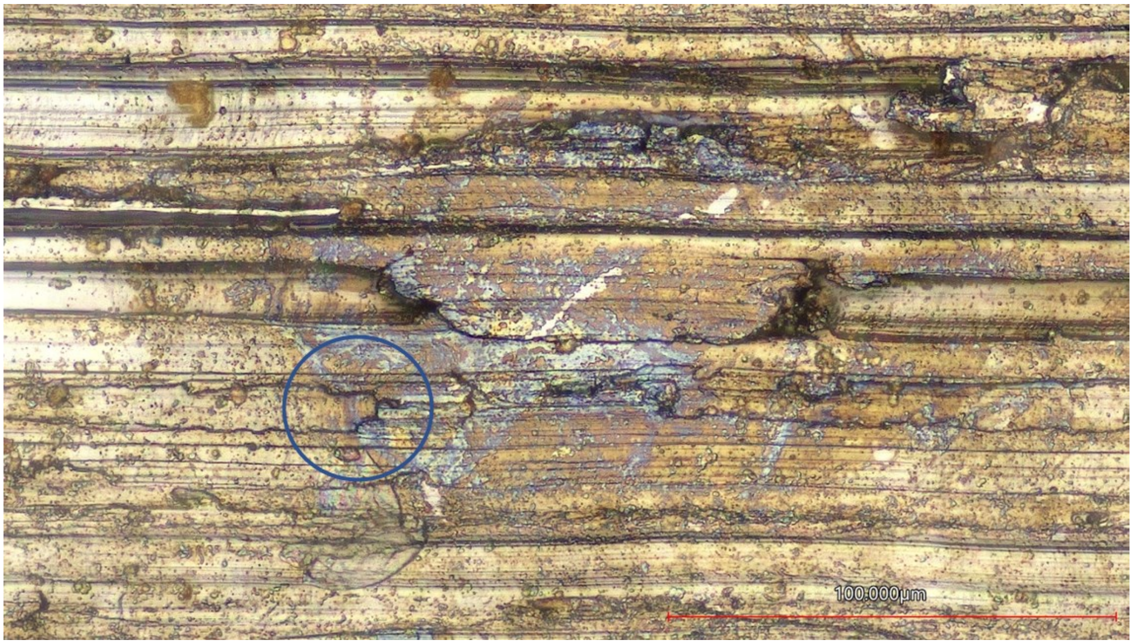

Figure 52. Likely Cracking at the Arch Region on 304W Exposed to 35°C for 8 Weeks

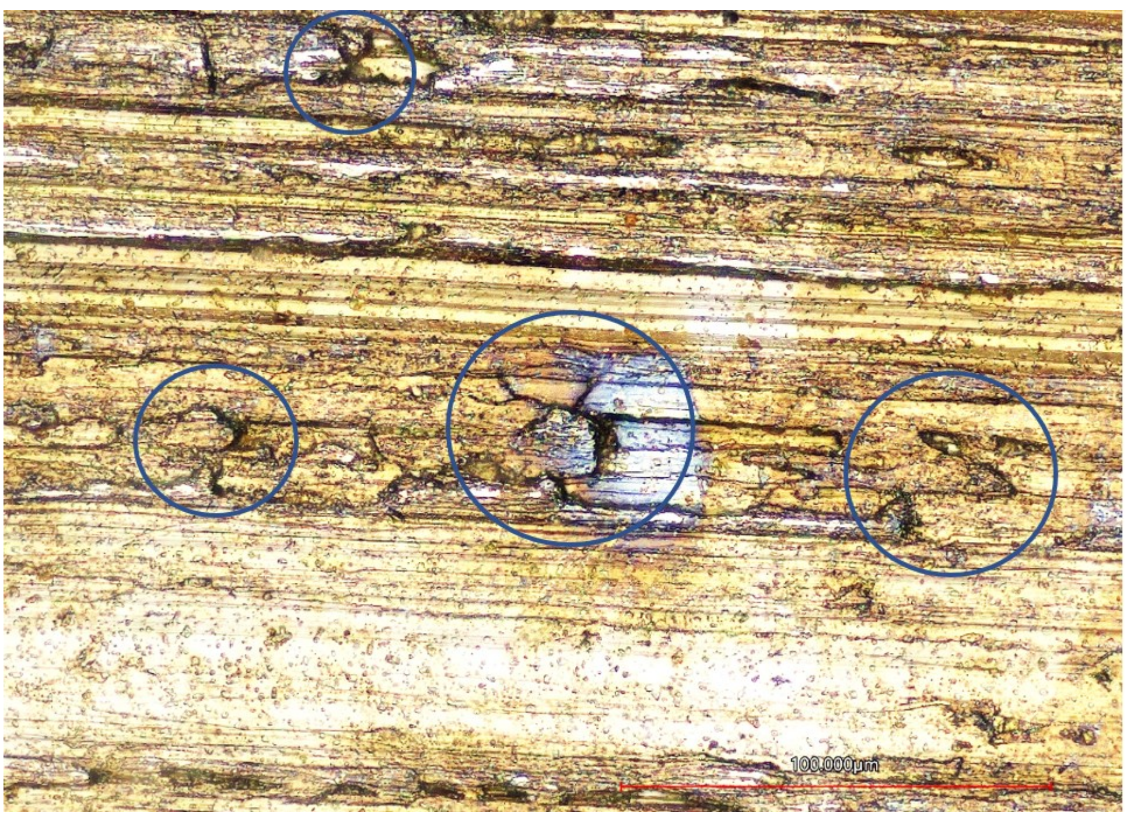

Figure 53. Cracking at the Arch Region on 304LW Exposed to 35°C for 8 Weeks

Figure 54 shows the micrograph for the 304 sample exposed to 35°C for 12 weeks. Samples exposed for 12 weeks showed heavier signs of corrosion and pitting than those exposed for 8 weeks. Pitting was labeled “moderate” on this sample in comparison with the 304 sample tested for 8 weeks. Three cracks circled in blue appear on this sample perpendicular to the polishing lines as expected.

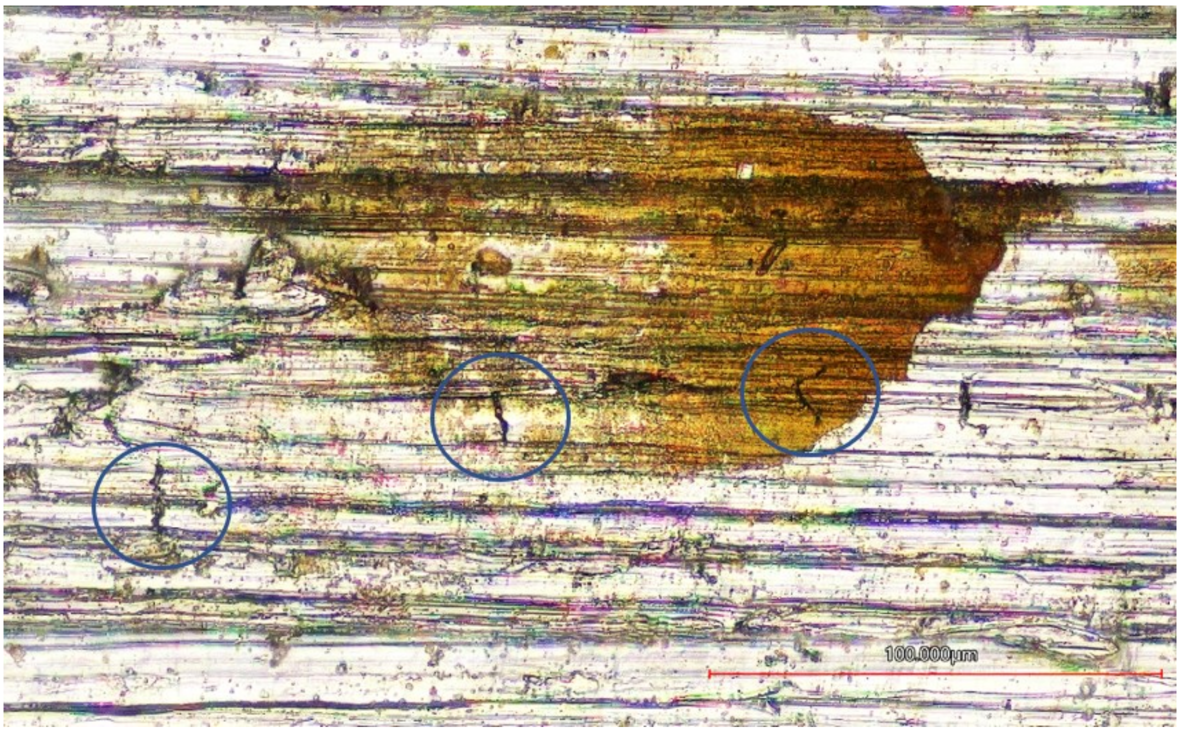

Figure 54. Cracking at the Arch Region on 304 Exposed to 35°C for 12 Weeks

The height profile of the 304 sample exposed to 35°C for 12 weeks (same sample shown in Figure 54) and the height profile of the reference 304 sample are shown in Figure 55. Increased pitting is evident by the scattered dark blue dots circled in blue. Cracks on this sample are circled in red which are propagating in the expected direction; the height profile shows a deeper surface on the three cracks compared with the surrounding material based on the color gradient.

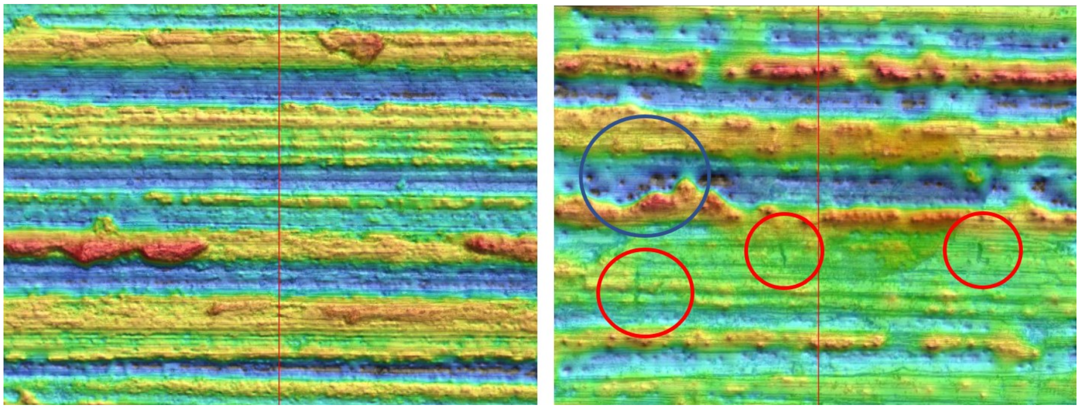

Figure 55. Reference 304 (right) vs. 304 Sample Exposed to 35°C for 12 Weeks (left)

The final micrograph presented in this section is shown in Figure 56 for the 304L sample exposed to 50°C for 14 weeks. More corrosion, pitting, and cracks were noticed on this sample in comparison with the samples exposed to 35°C. Refer to Appendix G for additional micrographs from experimental campaigns 1 and 2.

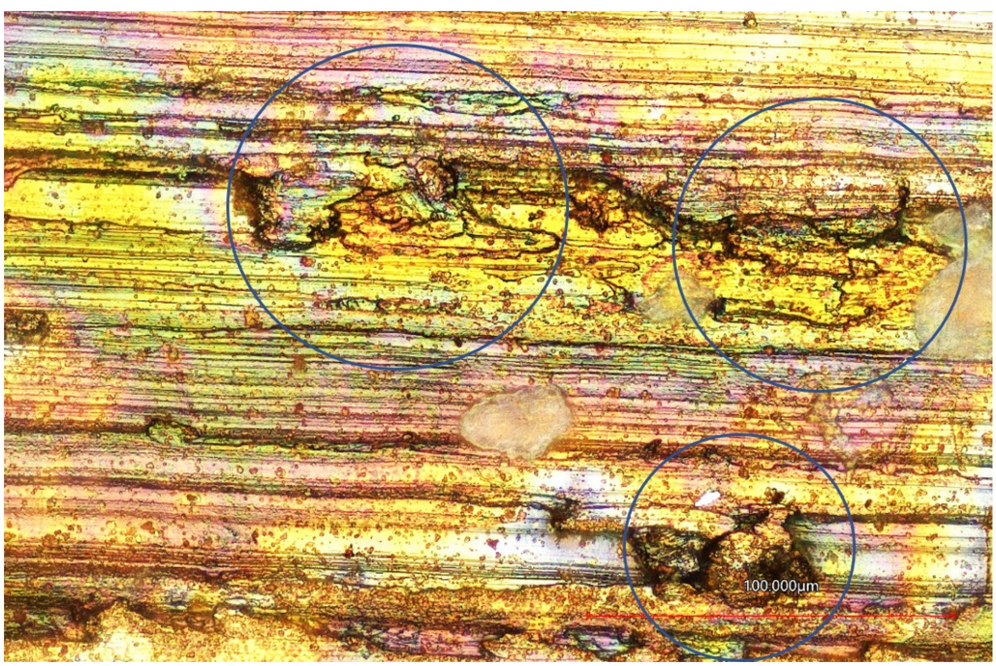

Figure 56. Cracking at the Arch Region on 304L Exposed to 50°C for 14 Weeks

The NRC [16] study observed cracking on 304, 304W, 304L, and 304LW samples exposed to 43°C for 4-16 weeks. The qualitative results shown in Table 13 also conforms with the NRC findings, cracking was identified on the same type of samples exposed to approximately similar temperature and durations. Additionally, the NRC concluded that fewer welded samples cracked in comparison with unwelded samples in agreement with the findings of this research since three "likely cracking" observations were noted on welded samples vs. one "likely cracking" on unwelded samples from both experimental campaigns, while cracks were observed on all other samples. Mintz [30] reported that 304 welded U-bend samples were less susceptible to cracking than unwelded samples in agreement with the NRC and the findings of this research. The reduced susceptibility of welded samples is, however, contrary to the expected behavior since welded samples have a weaker microstructure due to the depletion of chromium when the material surrounding the weld area is heated to 600-800°C. Moreover, all welded samples showed heavier pitting signs in comparison with unwelded samples.

The effect of increasing temperature from 35°C to 50°C cannot be determined based on the qualitative observations since the number of cracked samples at 35°C for both exposure durations is comparable to the number of cracked samples at 50°C for both exposure durations. The NRC also concluded that temperature increase from 43°C to 80°C reduced the susceptibility to CISCC. The comparison between the NRC findings and the findings of this research in terms of temperature effect is not possible due to the differences in the temperature range used. Mintz [30] however reported that SCC was evident on the 304 U-bend samples exposed to 35, 45, and 52°C but not at 60°C in agreement with the NRC and the findings of this research. In this study, temperature increase evidently increased corrosion based on the qualitative observations, but cracking observations were relatively similar for both temperatures (35°C and 50°C). Also, the susceptibility of 304 was comparable to 304L for both temperatures based on the qualitative observations. Figure 57 and Figure 58 show the qualitative cracking and pitting results in a graphical format.

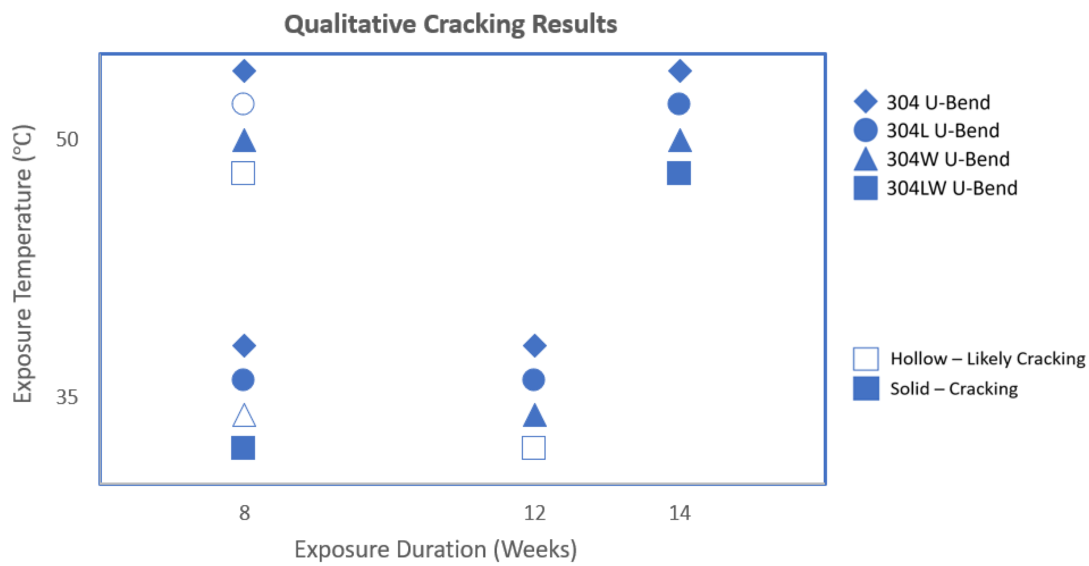

Figure 57. Qualitative Cracking Results of the 16 Corroded Samples

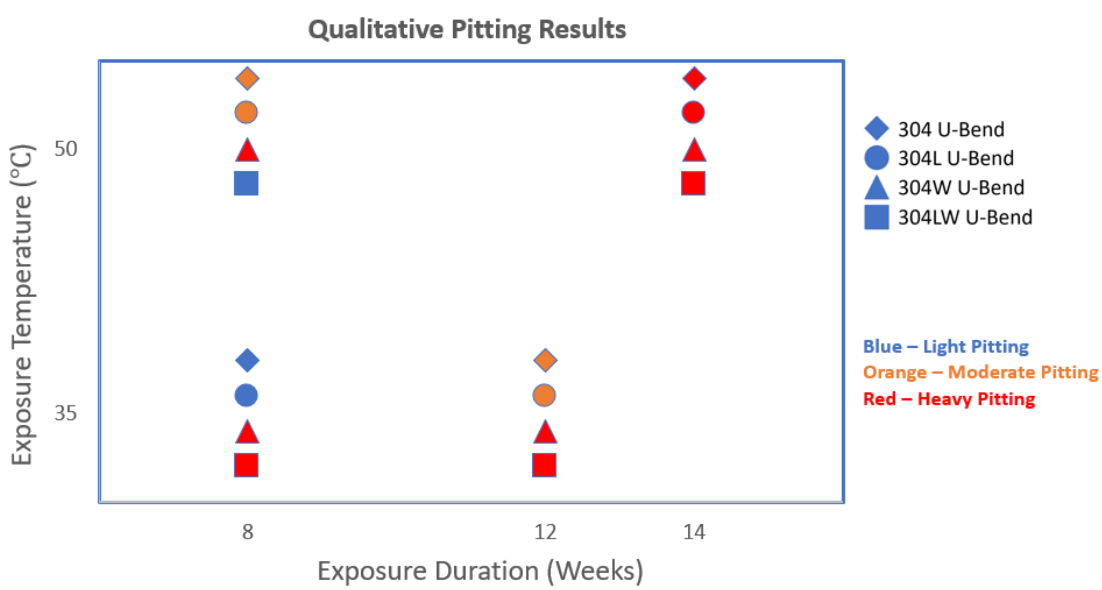

Figure 58. Qualitative Pitting Observations of the 16 Corroded Samples

4.3 Quantitative Cracking and Pitting Results

Surface profiles' data were used to examine the effects of temperature, material composition, welding, and exposure duration on CISCC. Only the surface valleys were used in this comparative analysis, such as the one circled on the example profile shown in Figure 59. Peaks' data was ignored since this data is not relevant to pitting and cracking characterization. This quantitative analysis is then compared with the qualitative results from Section 4.2 and published literature.

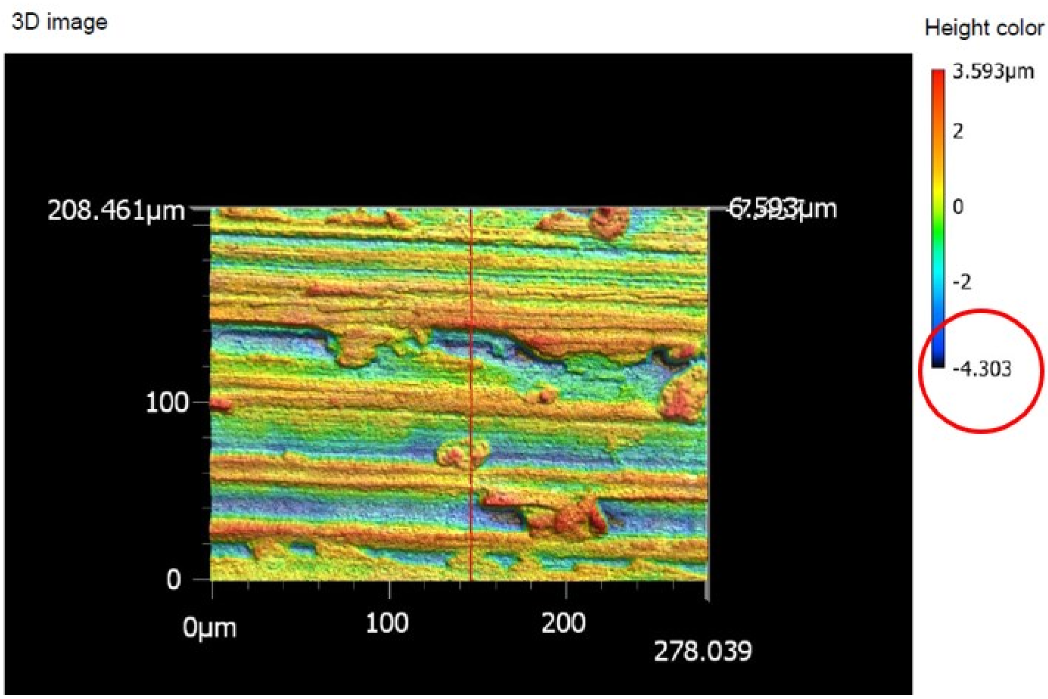

Figure 59. Example Surface Profile Output from the 3D Surface Profiler

Table 14 shows an example summary of the surface valleys from 11 profiles gathered on the 304L sample exposed to 50°C for 14 weeks. Each data point represents the maximum valley depth (negative value) from the scanned area at the arch region of the U-bend sample. The complete datasets for the 16 corroded and 2 reference samples are provided in Appendix H.

Table 14. Surface Profile Data for the 304L Sample Exposed to 50°C for 14 Weeks

Profile No.	Max Valley Depth (µm)
1	-7.074
2	-8.247
3	-7.619
4	-7.084
5	-8.924
6	-8.026
7	-8.644
8	-10.726
9	-6.355
10	-33.183
11	-4.303

The T-test and Analysis of Variance (ANOVA) statistical tools were used in this comparative analysis to test whether a significant statistical difference exists between the surface profiles of the corroded samples in Table 13 and the reference samples. Comparisons were performed between the surface profiles of welded and unwelded samples made from 304 and 304L, and samples tested at different temperatures and exposure durations. The t-test is used to determine whether a significant statistical difference exists between the surface profiles of two samples. For example, the t-test can be used to examine the effect of prolonged exposure on pitting and cracking by comparing the surface profile data of the 304 sample exposed to 35°C for 8 weeks with the 304 sample exposed to 35°C for 12. The ANOVA test is used to determine if there is a significant statistical difference between the surface profiles of multiple samples (3 or more). The ANOVA test can determine if a significant statistical difference exists between at least two of tested groups. For example, the ANOVA test can be used to examine the effect of material composition on CISCC by comparing surface profiles of 304, 304L, 304W, and 304LW exposed to the same temperature for the same exposure duration (*e.g.,* 35°C for 8 weeks or 50°C for 14 weeks). If the ANOVA test confirms that a significant difference exists between the tested samples, then t-tests were performed to test the differences between the samples in groups of 2.

JMP Pro. 16 was used to perform the ANOVA and t-tests. First, ANOVA and t-tests assumptions were examined to determine the appropriateness of these statistical tools for this application. Below are the assumptions and how each is met for this application,

1. Data is collected using a simple random sampling technique (SRS), this is true for this application as the surface profiles are selected randomly on each U-bend sample among a large set of available profiles.

2. Independent samples, this is true since each dataset comes from a different independent U-bend sample that was randomly placed in the corrosion chamber. This is also an assumption for the t-test.

3. Equal variances for each group (homoscedasticity). This is also an assumption for the t-test. This assumption is tested by the ratio of larger to smaller standard deviation as shown in the equation below, note that moderate variations of this assumption are not significant. This assumption can also be tested by checking the Residuals vs. Predicted graph in JMP which shows how the spread may differ across groups. Refer to Figure 60 for an example homoscedasticity test for the surface profiles of 304, 304L, 304W, and 304LW samples exposed to 35°C for 8 weeks.

$$\frac{S_{max}}{S_{min}} \leq 2$$

Smax: is the larger standard deviation between the two tested groups

Smin: is the smaller standard deviation between the two tested groups

4. Normality, approximately normal population distribution for each group. This assumption is true for all datasets. The normality check was performed by checking the Normal Quantile Plot in JMP which is a technique for assessing whether a dataset is approximately normally distributed. Refer to Figure 61 for an example Normal Quantile Plot for the comparisons between surface profiles of 304, 304L, 304W, and 304LW samples exposed to 35°C for 8 weeks.

The example Residuals vs. Predicted graph shown in Figure 60 for the samples exposed to 35°C for 8 weeks shows that the spread is relatively similar for all four materials (304, 304L, 304W, and 304LW). The spread is wider on the 304 surface profiles, especially the data point at the bottom, but the differences in variances do not seem extreme enough to undermine the ANOVA procedures.

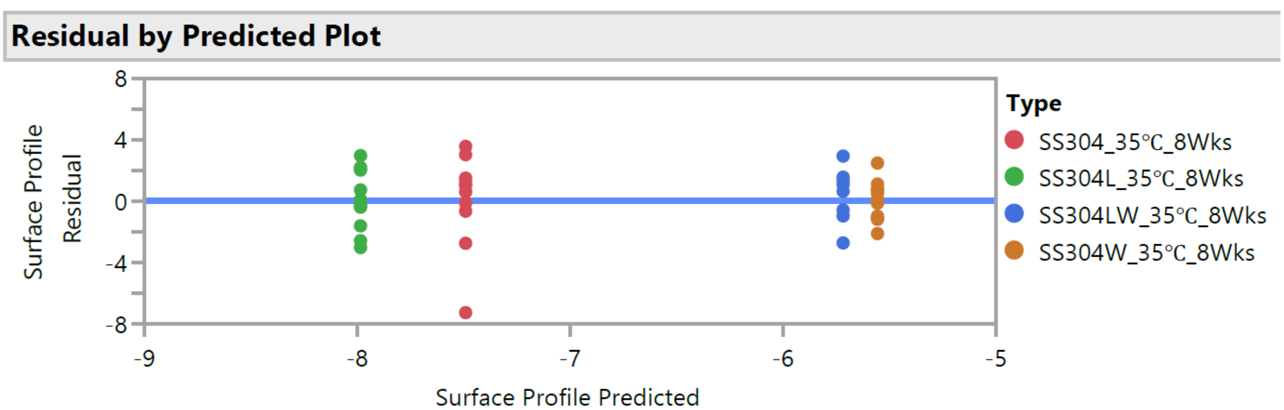

Figure 60. Example Residuals vs. Predicted Graph for Homoscedasticity Check

The Normal Quantile Plot shown in Figure 61 is fairly linear with the exception of two outliers on the bottom left corner, indicating the data is likely normal. A normality problem would be indicated if the points are far from the center line and beyond the dotted lines which are there for guidance.

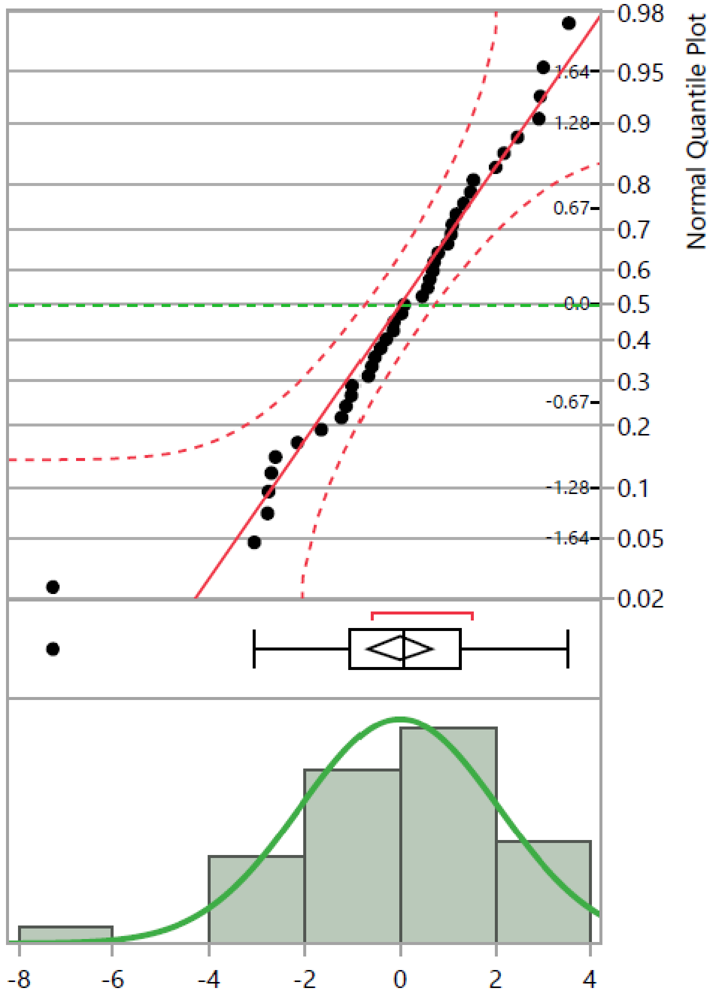

Figure 61. Example Normal Quantile Plot for Normality Check

Based on these analyses, ANOVA and t-test assumptions are considered valid, therefore, the statistical techniques were deemed appropriate for this application. For ANOVA tests, the null hypothesis (H_0) is that the means are equal for the different groups ($\mu 1=\mu 2=\mu 3$, etc.,), the alternative hypothesis (Ha) is that at least two means are different. For t- tests, the null hypothesis (H_0) is that the means are equal for the two samples ($\mu 1=\mu 2$), the alternative hypothesis (Ha) is that means are different ($\mu 1\neq\mu 2$). A significance level (α) of 0.05 was used for all ANOVA and t-tests.

4.3.1 Reference Sample Comparisons

Two ANOVA tests were performed to determine if a significant difference exists between the reference and corroded samples. The first ANOVA test (ANOVA-1) was performed to compare the surface profiles of the reference 304 sample (clean sample) with the corroded 304 samples exposed to different temperatures and durations. The second ANOVA test (ANOVA-2) was performed to compare the surface profiles of the reference 304W sample (clean welded sample) with the corroded 304W samples exposed to different temperatures and durations. Refer to Table 15 for the samples compared in each ANOVA test. JMP output results of ANOVA-1 and ANOVA-2 tests are provided in Appendix I.

Table 15. ANOVA Tests for Reference vs. corroded 304 and 304W Samples

ANOVA-1 (304 Samples)	ANOVA-2 (304W Samples)
304_35°C_8 weeks	304W_35°C_8 weeks
304_35°C_12 weeks	304W_35°C_12 weeks
304_50°C_8 weeks	304W_50°C_8 weeks
304_50°C_14 weeks	304W_50°C_14 weeks
304_Reference	304W_Reference

Figure 62 shows ANOVA-1 analysis plot. Note that the grand mean of all surface profiles is represented by the horizontal gray line (grand mean) and the individual means are represented by the horizontal green line in the middle of the diamond shaped boxes (group mean) as shown in Figure 62. The diamond boxes represent the 95% confidence interval of each sample type. Overlap marks appear as lines above and below the group mean as marked on Figure 62. For groups with equal sample sizes, overlapping marks indicate that the two group means are not significantly different at the given confidence level. Note that the number of surface profiles (sample sizes) are not equal on some of the U-bend samples, this does not impact the ANOVA analysis. The p-value for ANOVA-1 was 0.0294 which is less than 0.05. Therefore, the H_0 is rejected in favor of a significant statistical difference between the surface profiles of 304 samples. Notice the reference sample mean is significantly larger than the mean of all corroded samples, indicating potentially significant pitting and cracking on the corroded samples. Another important observation, the mean of samples exposed to 35°C is larger than the mean of samples exposed to 50°C indicating that temperature increase could potentially have an accelerating effect on CISCC. Generally, ANOVA-1 test indicates that a difference exists between at least two U-bend samples. Further comparisons between the 304 samples were performed using t-tests to examine the significance of increasing temperature and exposure duration.

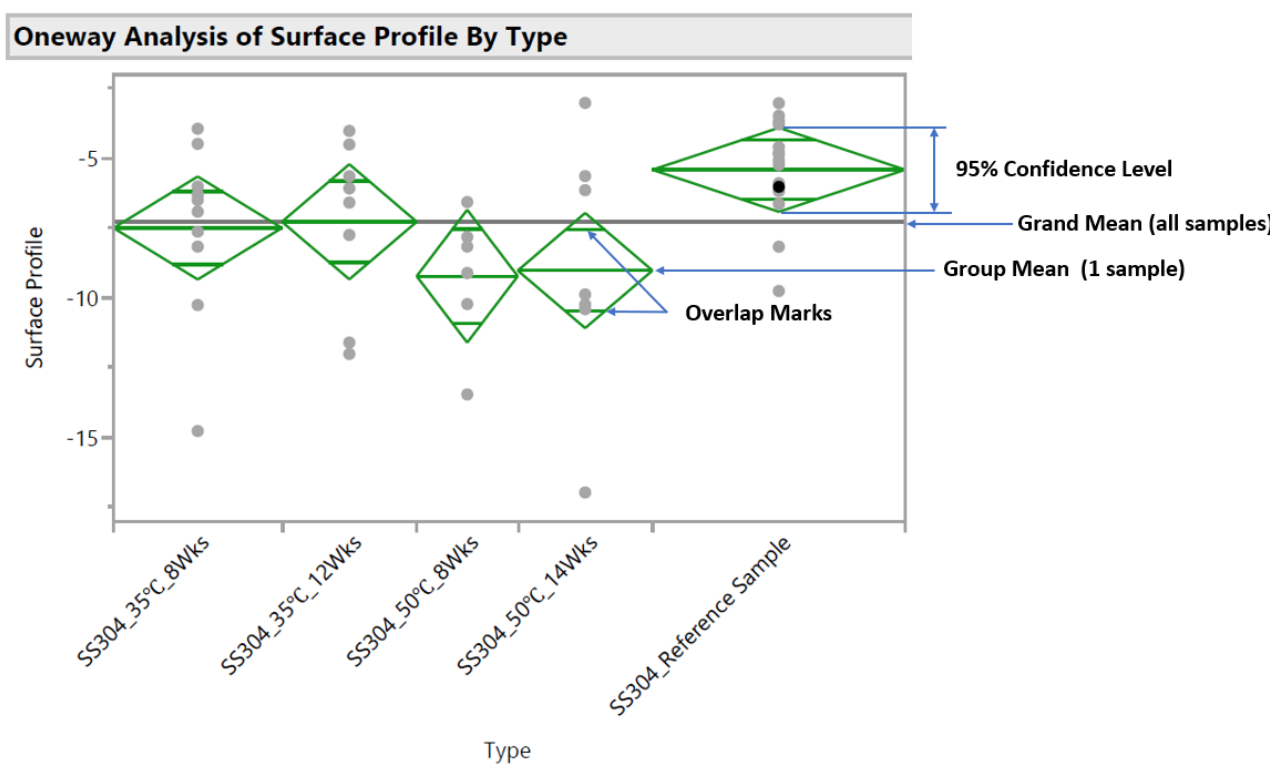

Figure 62. ANOVA-1 Analysis Plot for 304 Samples

The p-value for ANOVA-2 was 0.0519 which is slightly greater than 0.05, refer to Figure 63 for ANOVA-2 analysis results. Therefore, the H_0 is accepted for a significance level of 5% in favor of equal means between the surface profiles of the welded reference sample and the corroded welded samples. ANOVA-2 test indicates that no significant difference exists between the reference sample and the 304W welded samples tested at different temperatures and different exposure durations. One observation on ANOVA-2 is that the mean of samples exposed to 35°C is larger than the mean of samples exposed to 50°C indicating that temperature increase could potentially have an accelerating effect on CISCC as was concluded from ANOVA-1 (to be confirmed by t-tests). ANOVA-2 results can be interpreted as welded samples are less susceptible to CISCC than unwelded samples.

The conclusion regarding susceptibility of welded samples agrees with the NRC [16] and Mintz [30] conclusions as both found that welded samples were less susceptible to CISCC than unwelded samples as described earlier in the qualitative section. Zakaria [27] however concluded that 304L heat sensitized samples were more susceptible to CISCC than base metal samples. However, these samples were heated to 600°C for 2 hours and not welded.

Further comparisons between the welded samples exposed to 35°C and 50°C and different exposure durations with the reference welded sample were performed using t-tests to examine the significance of increasing temperature and exposure duration. The expected outcome of these t-tests is that the differences between all welded samples are insignificant, but these t-tests were performed to compare the surface profile means at different temperatures and exposure durations.

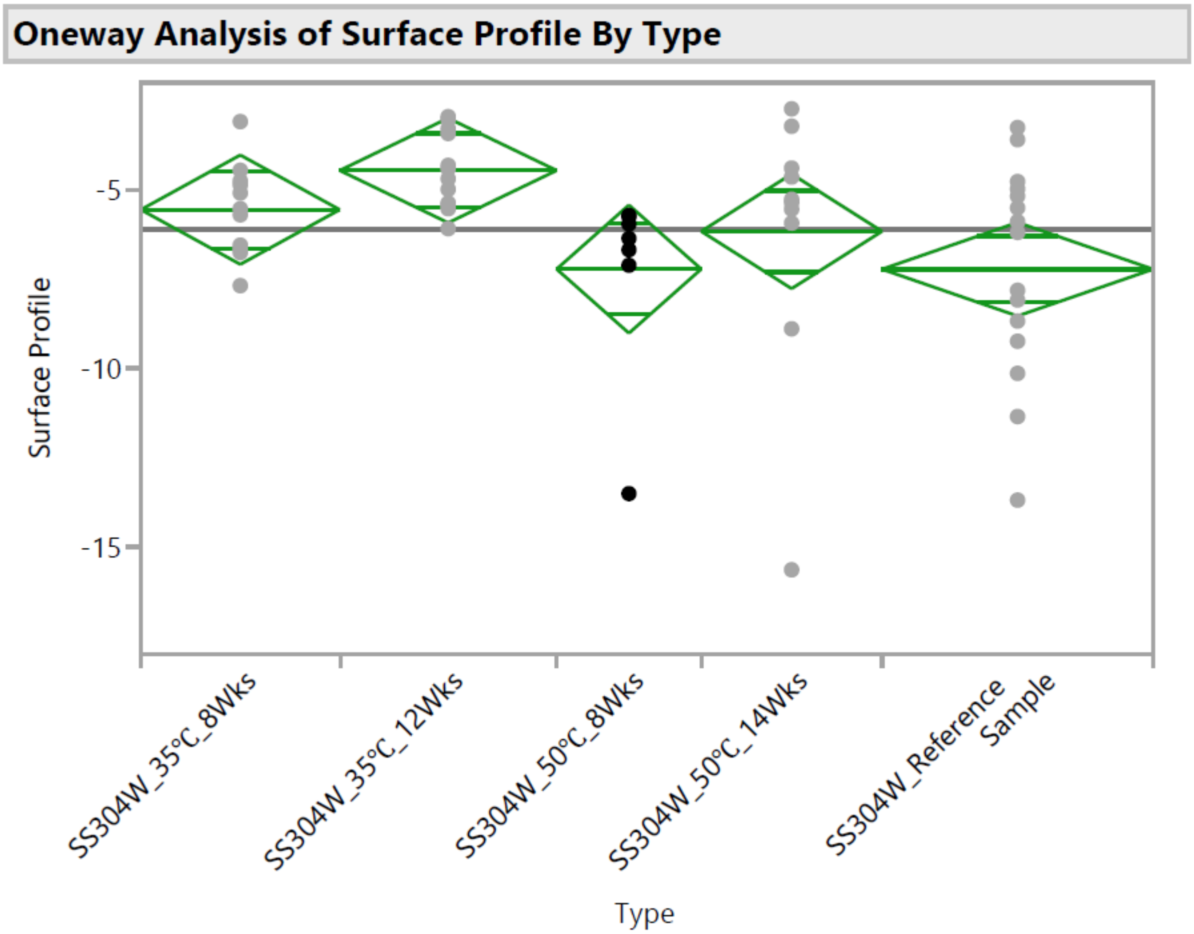

Figure 63. ANOVA-2 Analysis Plot for 304W Samples (Welded)

T-tests were performed to compare the corroded 304 samples with the reference 304 sample and the corroded 304 welded samples with the reference 304 welded sample to evaluate the statistical differences. Refer to Table 16 for the t-tests performed, p-values, and comments on each analysis. JMP output results of the t-tests listed in Table 16 are included in Appendix I.

The t-tests showed no significant differences between surface profiles of 304 welded samples in comparison with the reference 304 welded sample at different temperatures and exposure durations.

Table 16. T-tests for Reference Samples vs. Corroded 304 and 304W

304 U-bend Samples		
T-test	**p-value**	**Result and comments for a significance level of 5%**
304_35°C_8 weeks vs. 304 Reference	0.0392	Significant difference exists in the expected direction[1]
304_35°C_12 weeks vs. 304 Reference	0.0713	Not a significant difference exists, trends in the expected direction
304_50°C_8 weeks vs. 304 Reference	0.0046	Significant difference exists in the expected direction
304_50°C_14 weeks vs. 304 Reference	0.0239	Significant difference exists in the expected direction
304W U-bend Samples (welded)		
T-test	**p-value**	**Result and comments for a significance level of 5%**
304W_35°C_8 weeks vs. 304W Reference	0.0339	Significant difference exists in the **opposite** direction
304W_35°C_12 weeks vs. 304W Reference	0.0018	Significant difference exists in the **opposite** direction
304W_50°C_8 weeks vs. 304W Reference	0.9974	No significant difference exists
304W_50°C_14 weeks vs. 304W Reference	0.4615	No significant difference exists

Refer to Figure 64 and Figure 65 for the t-test plots for 304 samples in comparison with the reference sample at two different temperatures. The center line on the box plot shows the data median, where half of the data is above the center line and half is below. If the data are symmetrical, the median will be in the center of the box. The lines at the bottom and top of the box represent the 1st (25th) and 3rd (75th) quantiles, or percentiles. The lines that extend from the box are called whiskers; whiskers represent the expected variation in the data. If the data do not extend to the end of the whiskers, then the whiskers extend to the minimum and maximum data values.

[1] The expected direction is that reference samples will have larger mean in comparison with the mean of corroded samples.

Any values that fall above or below the end of the whiskers, they are plotted as dots and called outliers.

Notice the mean of the 304 sample exposed to 50°C is smaller than the mean of the sample exposed to 35°C indicating increased pitting and potentially cracking (more valleys on the 50°C scanned surfaces in comparison with the reference sample). Although the t-test on the 304 sample exposed to 35°C for 12 weeks was determined not significant for a significance level of 5%, the p-value was 0.0713 which is slightly greater than 0.05.

Notice the outcome of the t-test for the 304W samples exposed to 35C for 8 and 12 weeks in comparison with the reference 304 welded sample. The differences were significant in the opposite direction indicating that surface profiles' depth mean of the reference sample is smaller than some of the means of corroded welded samples. More surface profiles would be needed on the reference sample to quality this trend.

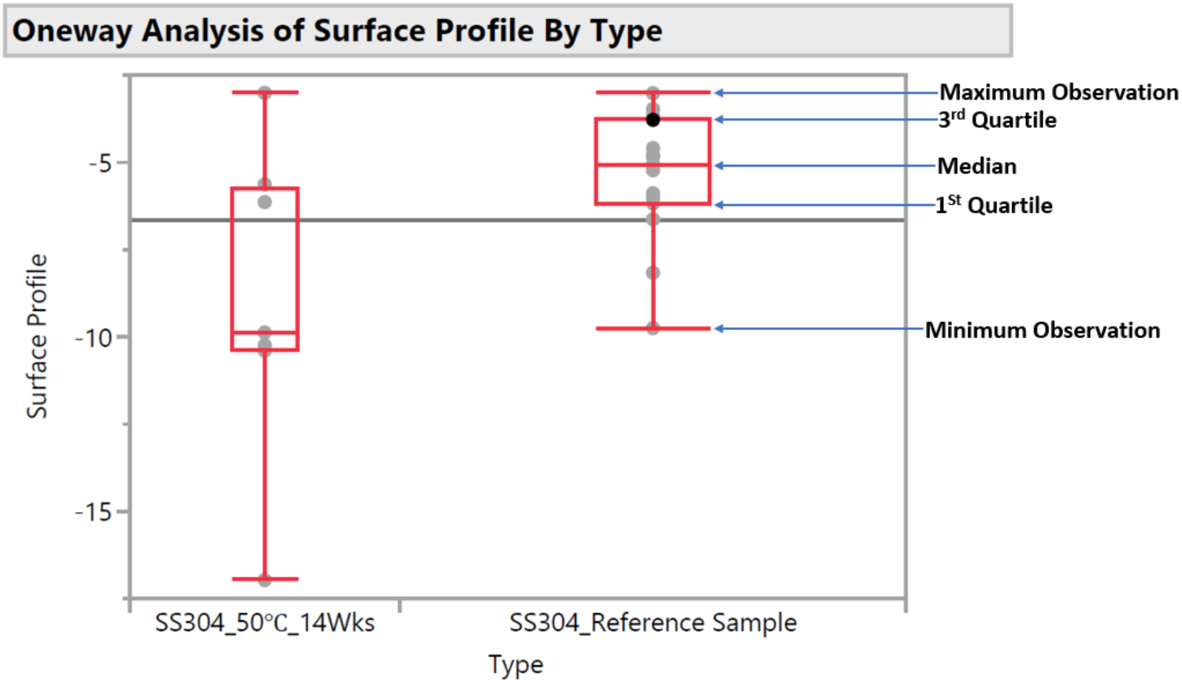

Figure 64. T-test for 304_50°C_14 Weeks vs. Reference Sample

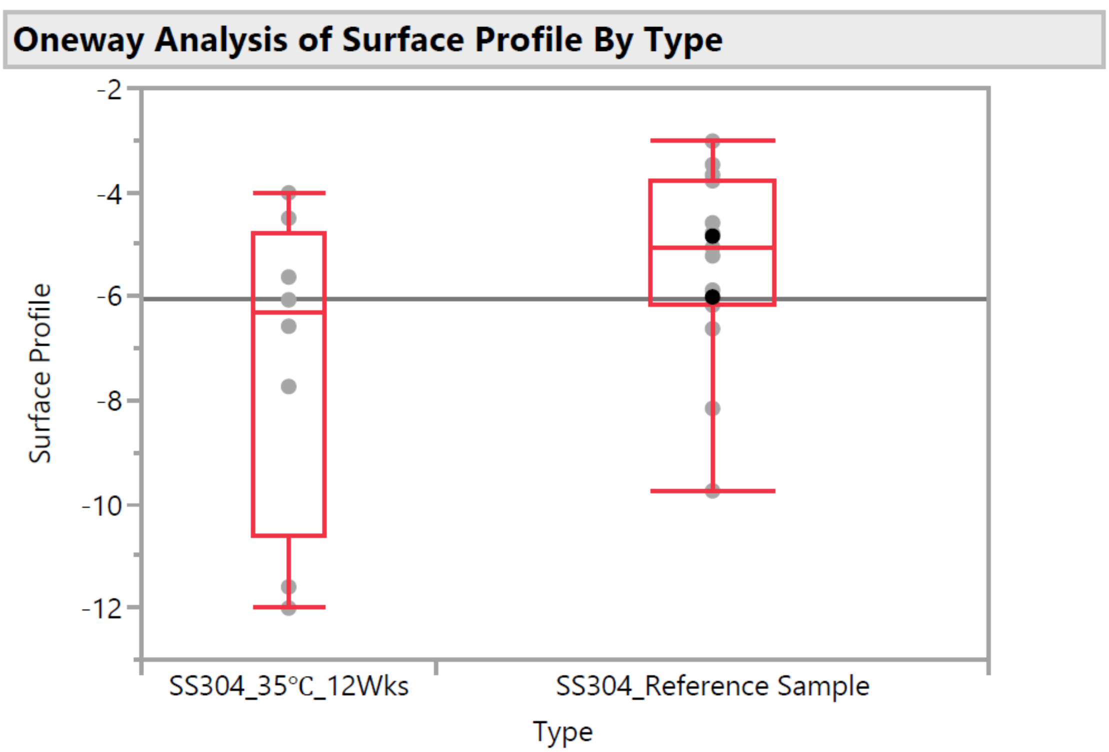

Figure 65. T-test for 304_35°C_12 Weeks vs. Reference Sample

4.3.2 Effect of Material Analysis

Table 17 shows the ANOVA performed to determine if a significant statistical difference exists between the different types of U-bend samples (304, 304L, 304W, 304LW). Surface profile data of 304, 304L, 304W, and 304LW U-bend samples were compared at different temperatures and exposure durations. JMP output results of the ANOVA tests listed in Table 17 are provided in Appendix I.

Table 17. ANOVA Tests for Material and Welding Effect on CISCC

ANOVA Tests	p-value	Result and comments for a significance level of 5%
35°C_8 weeks 304, 304L, 304W, and 304LW	0.0292	Significant difference exists between the different samples
35°C_12 weeks 304, 304L, 304W, and 304LW	0.0180	Significant difference exists between the different samples
50°C_8 weeks 304, 304L, 304W, and 304LW	0.0018	Significant difference exists between the different samples
50°C_14 weeks 304, 304L, 304W, and 304LW	0.361	No significant difference

Figure 66 shows the ANOVA test on material effect for the samples exposed to 35°C for 8 weeks. Surface profile means of 304 and 304L are smaller than the means of 304W/304LW welded samples indicating reduced susceptibility of welded U-bends compared to base metal U-bends as concluded earlier, this finding conforms with the findings from the NRC [16] and Mintz [30] experiments. Additionally, the means of 304 and 304L U-bends appear relatively similar, indicating an insignificant difference between the susceptibility of 304 and 304L samples exposed to 35°C for 8 weeks. The NRC results [16] show a comparable number of cracked 304 and 304L samples at the same exposure duration, for instance the number of cracked 304 samples exposed to 43°C were identical to the number of cracked 304L samples exposed to 43°C for 52 weeks.

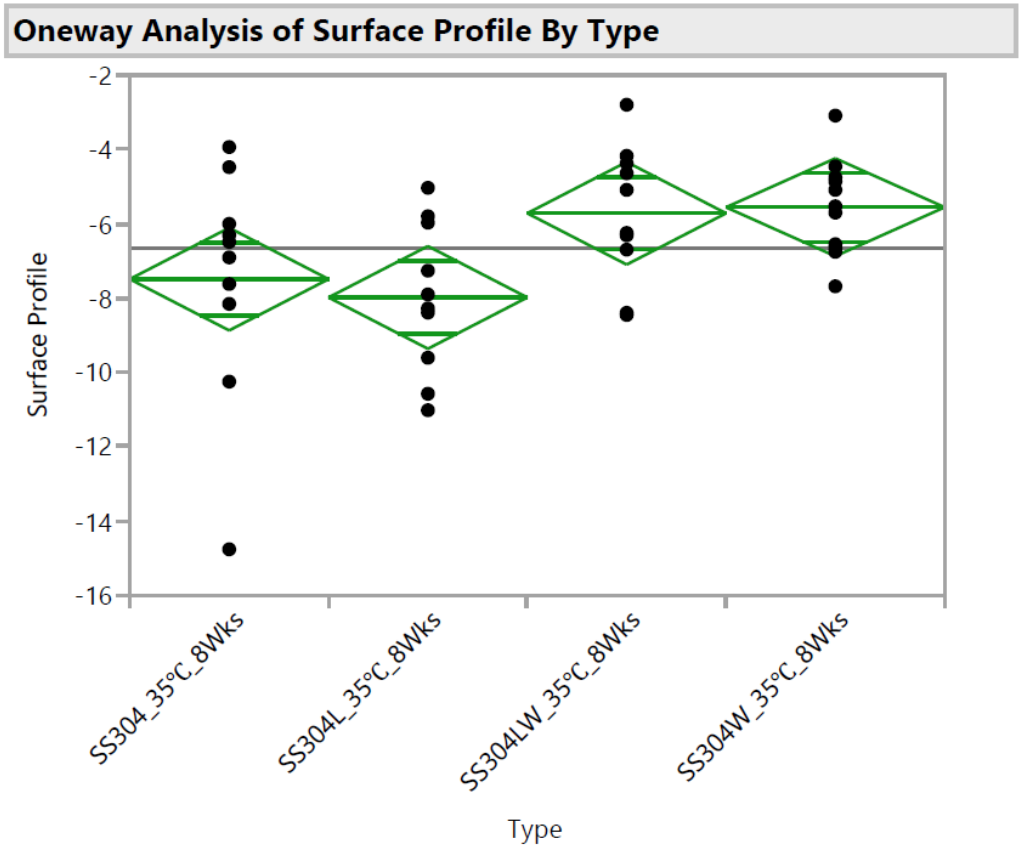

Figure 66. ANOVA Analysis Plot for Different Sample Types (35°C_8 Weeks)

The ANOVA test on material effect for the samples exposed to 35°C for 12 weeks confirms the previous observation of increased susceptibility of 304 U-bends over 304W welded U-bends as the means for these two samples are significantly different. The differences between 304 and 304L samples were not significantly different.

At higher temperature and longer exposure, the samples are showing the same level of corrosion based on the results of the 50°C_14 weeks ANOVA test. The comparisons between different material types exposed to 50°C for 8 weeks confirms the increased susceptibility of unwelded samples over welded samples for both material types 304 and 304L. Additionally, the difference in susceptibility between 304 and 304L samples was insignificant.

Figure 67 shows the t-test result for comparison between 304 exposed to 35°C for 12 weeks and 304W exposed to 35°C for 12 weeks. The p-value of this test is 0.035 which is less than 0.05 and therefore the null hypothesis is rejected in favor of a significant statistical difference between the samples. The t-test between 304L and 304LW exposed to 35°C for 12 weeks did not yield the same conclusion where the two samples were determined not significantly different.

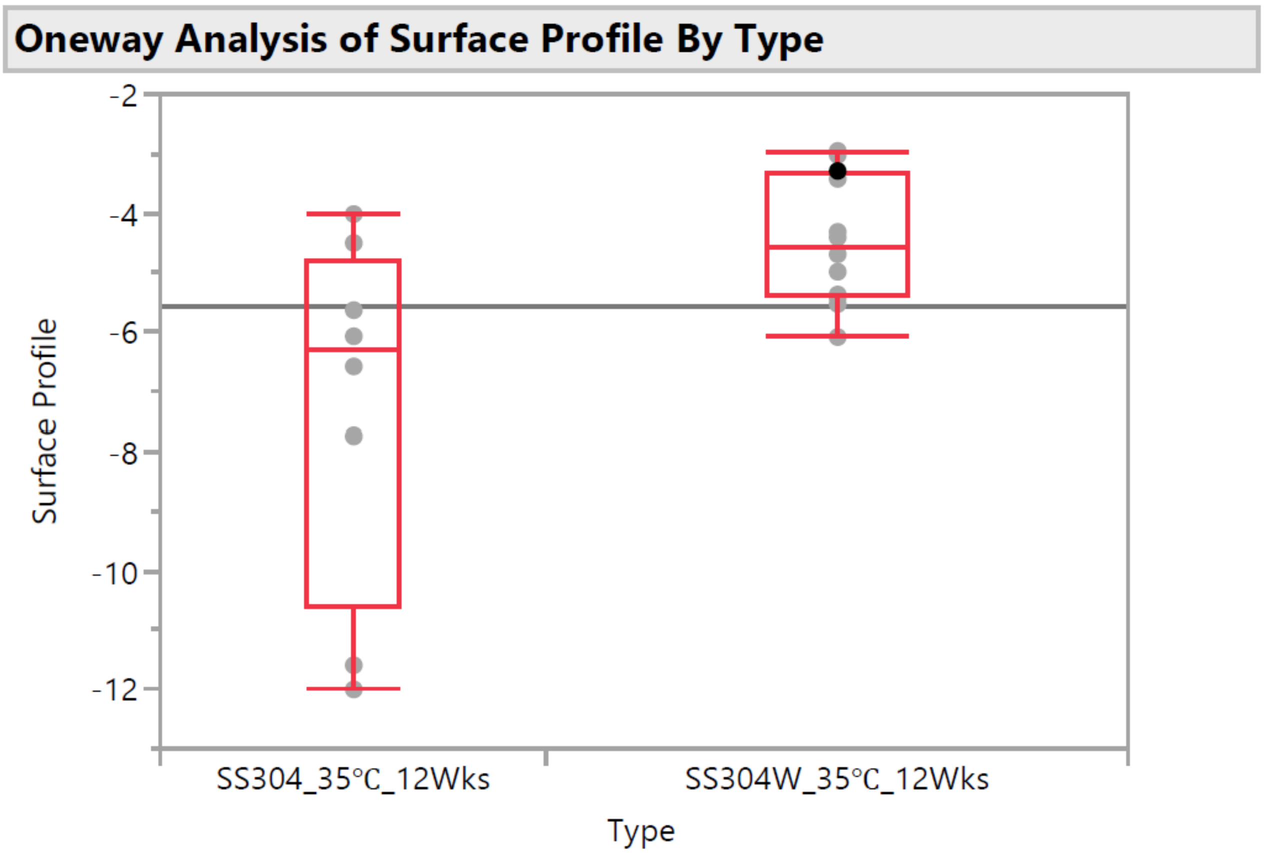

Figure 67. T-test for 304_35°C_12 Weeks vs. 304W_35°C_12 Weeks

4.3.3 Effect of Temperature Analysis

T-tests were performed to determine the significance of increasing temperature on CISCC susceptibility. The surface profiles of the different types of samples (304, 304L, 304W, 304LW) exposed for 8 weeks at two different temperatures were compared. The goal of this test is to determine if changing the increasing temperature from 35℃ to 50℃ will accelerate pitting and cracking based on the surface profile data. Refer to Table 18 for the t-tests performed and the results of each test. JMP output results of the t-tests listed in Table 18 are provided in Appendix I.

Table 18. T-tests for U-bends Exposed to Different Temperatures

T-test	p-value	Result and comments for a significance level of 5%
304_35℃_8 weeks vs. 304_50℃_8 weeks	0.2375	Difference is not significant, trends in the expected direction[2]
304L_35℃_8 weeks vs. 304L_50℃_8 weeks	0.0011	Significant difference exists in the expected direction
304W_35℃_8 weeks vs. 304W_50℃_8 weeks	0.1269	Difference is not significant, trends in the expected direction
304LW_35℃_8 weeks vs. 304LW_50℃_8 weeks	0.0014	Significant difference exists in the expected direction

Figure 68 shows the t-test plot of the comparison between 304 samples exposed to 35℃ and 50℃ for 8 weeks. The t-test concluded that a temperature increase from 35℃ and 50℃ had no effect on the acceleration of CISCC for 304 samples for a significance level of 5%. However, the mean of the 304 sample exposed to 35℃ is larger than the mean of 304 samples exposed to 50℃ as expected indicating increased surface pitting and potentially cracking on the samples exposed to

[2] The expected direction is that samples exposed to 35℃ will have larger mean in comparison with the mean of samples exposed to 50℃.

50°C (deemed not significant per the t-test results). The same conclusions can be made on the 304W samples, the mean was smaller on the sample exposed to 50°C as expected.

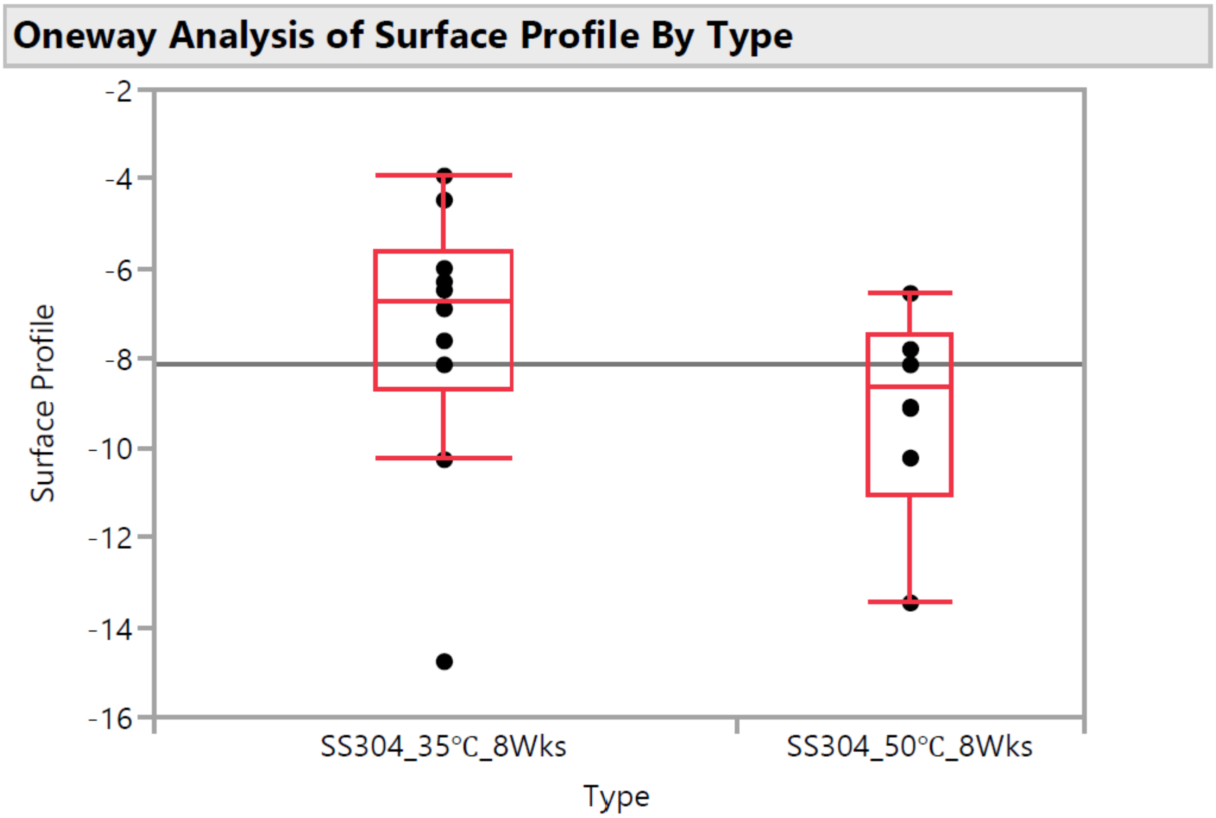

Figure 68. T-test for 304_35°C_8 Weeks vs. 304W_50°C_8 Weeks

Figure 69 shows the t-test for 304L samples exposed to 35°C and 50°C for 8 weeks. The t-test concluded a significant difference between the two temperatures. This means the susceptibility of 304L samples to CISCC increases as temperature increases from 35°C to 50°C. The conclusions on the 304LW t-test were similar to the 304L conclusions. These conclusions indicate that temperature increase has a significant increase in pitting and cracking on 304L and 304LW but not on 304 and 304W samples. The NRC [16] didn't report a correlation between susceptibility of

304L and increased temperature. However, the NRC susceptibility maps show the number of cracked 304L samples increased as exposure duration increased unlike 304 and 316 samples.

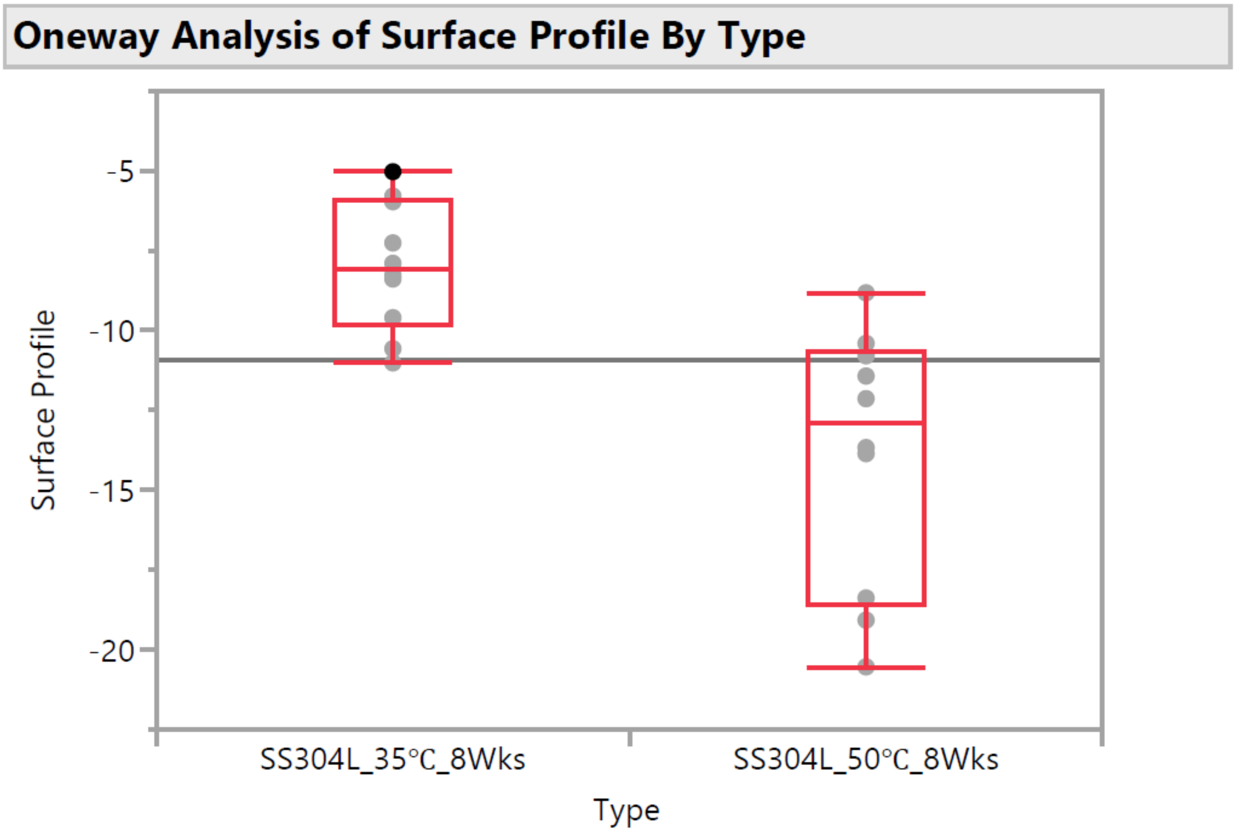

Figure 69. T-test for 304L_35°C_8 Weeks vs. 304L_50°C_8 Weeks

The statistical analysis agrees with the NRC [16] conclusions on the effect of temperature. The NRC observed cracking on all samples (304, 304L, and 316L) at 43°C only and no cracking was observed at 80°C or 120°C. Additionally, Mintz [30] reported that SCC was evident on 304 U-bend samples exposed to 35°C, 45°C, and 52°C but not at 60°C. Additionally, Mintz's results show the number of cracked U-bend samples exposed for 1 and 4 months at 35°C, 45°C, 55°C were comparable on all sample types (as-received, heat-sensitized, and welded).

4.3.4 Effect of Exposure Duration Analysis

Finally, the effect of increasing exposure duration on CISCC was examined. T-tests were performed to determine the significance of increasing the exposure duration for the different U-bend sample types tested at different temperatures. The surface profiles of the different sample types (304, 304L, 304W, 304LW) exposed to 35°C and 50°C were compared for the two different exposure durations. The goal of this test is to determine if changing the exposure duration from 8 weeks to 12 weeks (experimental campaign 1) or 8 weeks to 14 weeks (experimental campaign 2) will significantly accelerate pitting and cracking based on the surface profile data. T-tests showed that no significant statistical difference exists between the two exposure durations at 35°C and 50°C. This could be interpreted as pitting and cracking on samples exposed for 8 weeks were comparable to samples exposed for 12 or 14 weeks. JMP output results of some of the exposure duration t-tests are provided in Appendix I.

These conclusions regarding exposure duration agree with the NRC [16] results as the number of cracked 304 samples after 4 weeks of exposure were comparable to the number of cracked samples after 16 weeks of exposure. For the 316L samples, the NRC results show the number of cracked samples exposed for 32 weeks were comparable to the number of cracked samples exposed for 52 weeks. However, Mintz [30] reported more cracking on the samples exposed for 16 weeks than the samples exposed for 4 weeks. Both the qualitative and the quantitative analyses from this research show comparable results between the samples exposed for 8 weeks and the samples exposed for 12 or 14 weeks at both temperatures. These results indicate that increasing the experiment duration by 4 or 6 weeks did not significantly impact pitting and cracking initiation when tested to 35°C and 50°C.

4.4 Sources of Errors

Sources of error and a discussion of the potential impact on CISCC are discussed in the following sections.

4.4.1 Variability of Applied Tensile Stress

One of the important variables in the development of CISCC is the applied tensile stress. Pitting and cracking are expected to initiate in highly stressed regions such as the arch of the U-bend sample. The applied stress was not measured in this experimental work and was assumed constant across all U-bend samples; this is a safe assumption since all samples were manufactured by the same supplier per the ASTM G30 and G58 standards.

The bolts on some of the 304W samples in experimental campaign 1 were not completely tightened as illustrated in Figure 70 which may have influenced the applied stress on these samples. This was noticed post experimentation and could not be corrected. The expectation that most of the stress in the arch is applied during manufacturing by the single stage stressing process and this may have had a minor impact on the development of pitting and cracking.

The results of the quantitative and qualitative analyses indicate reduced susceptibility of welded over unwelded U-bends samples. This observation was also reported by other researchers with comparable test conditions. However, the expectation is that welded samples would be more susceptible to CISCC due to the depletion of chromium and weakening of the grain boundaries during the welding process. Also, welding introduces residual stresses that could accelerate time to failure and encourage pitting.

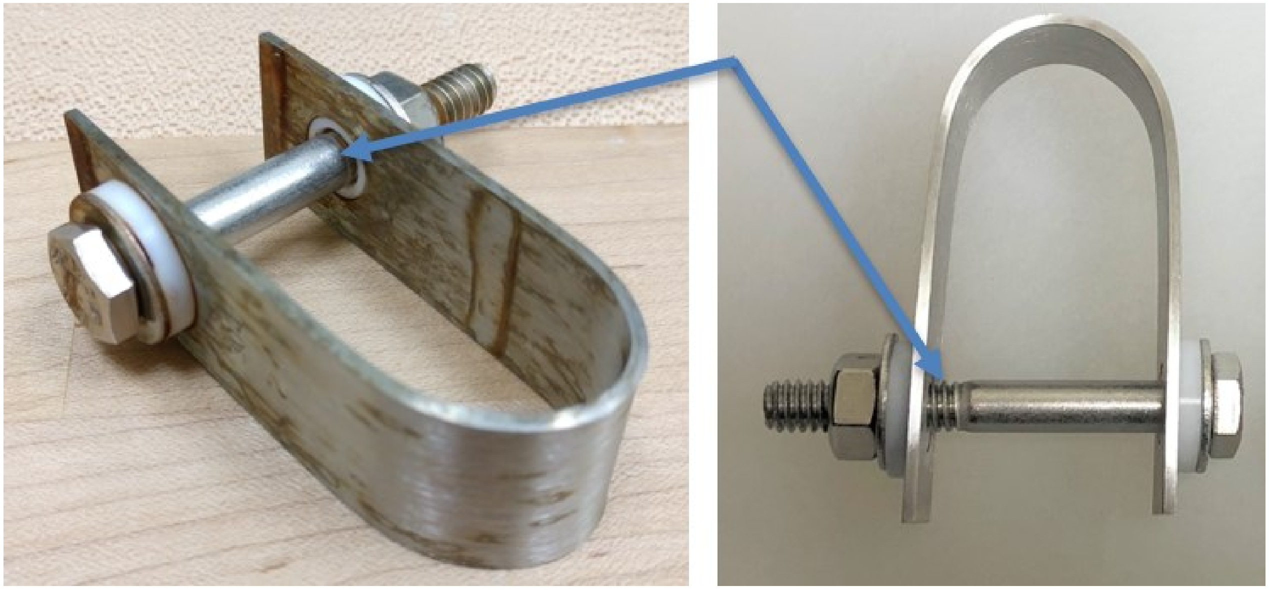

Figure 70. Fully Tightened (left) vs. Partially Tightened U-bend Sample (right)

4.4.2 Limited Surface Profile Data

Data collection on surface profiles is a time consuming process and the statistical analysis is limited by the number of surface profiles gathered for each sample (6-12 profiles each). Each surface profile represents an area 300-400 μm^2 which is a small area in comparison with the sample's size. However, the surface profiles were collected from different areas of the arch region to as accurately as possible obtain datasets that are representative of the entire surface profile. Larger datasets which require gathering more surface profiles could enhance the accuracy of the results and may provide conclusive results on some key aspects such as the effect of exposure duration of CISCC. Moreover, the qualitative analysis is largely based on observations of the optical micrographs and therefore human mistake is a potential source of error.

4.4.3 Collection Rate Variability

During the experimental work, collection rate variability issues were encountered due to the blockage of the fogging atomizer by small particles. For example, experimental campaign 1 was elongated by a week due to lower than normal collection rate (< 1 mL/hr.). Also, the collection rates throughout the chamber were not consistent but were within the expected range of 1-2 ml/hr. For instance, one side of the chamber constantly collected more salt solution than the other side which could have impacted pitting and cracking results. The samples were, however, placed in arbitrary locations inside the chamber to randomize the possible effects on the fog and deposition distribution. Collection rates were within the expected range during most of the experimental work; periods of low collections were accounted for by extending the experiment's duration to compensate for lost collection and to minimize the impact on CISCC.

4.5 Comparison of Qualitative and Quantitative Analyses

A comparison between the qualitative quantitative results is provided in Table 19. Additionally, Table 20 compares thesis conclusions with previous literature in accelerated CISCC.

Table 19. Qualitative vs. Quantitative Analyses

Qualitative Results	Quantitative Results
Effect of Temperature	
The effect of temperature is not significant based on the qualitative observations since the number of cracked samples at 35°C for both exposure durations was comparable to the number of cracked samples at 50°C for both exposure durations. However, pitting was more severe on the samples exposed to 50°C for 14 weeks in comparison with samples exposed to 35°C for 12 weeks on all sample types.	Concluded that increasing temperature from 35°C to 50°C had no significant effect on the acceleration of CISCC for 304 and 304W samples. However, the means of 304 and 304W samples exposed to 35°C were larger than the means of 304 and 304W samples exposed to 50°C indicating increased pitting and potentially cracking as temperature increased. However, a significant temperature effect was concluded for 304L and 304LW samples.

Qualitative Results	Quantitative Results
Effect of Welding	
Welded samples were less susceptible to CISCC than unwelded samples Three “likely cracking” observations were noted on welded samples vs. one “likely cracking” on unwelded samples from both experimental campaigns, while cracks were observed for all other samples.	Welded samples were less susceptible to CISCC than unwelded samples based on several surface profile comparisons. The comparisons between welded 304W samples and the reference welded sample were all deemed not significant statistically.
Effect of Expose Duration	
Increased test duration from 8 to 12 weeks at 35°C and from 8 to 14 weeks at 50°C had no significant effect on the acceleration of pitting and cracking for all sample types. Cracking and likely cracking observations are relatively similar for both durations.	Increased test duration from 8 to 12 weeks at 35°C or from 8 to 14 weeks at 50°C had no significant effect on surface profiles for all sample types. The means of samples exposed for 8 and 12 weeks at 35°C were similar. Also, the means of samples exposed for 8 and 14 weeks at 50°C were similar.
Effect of Material	
The susceptibility of 304 samples compared with 304L is not significant based on the qualitative analysis.	The susceptibility of 304 compared with 304L is not significant based on the surface profile comparisons.

Table 20. Thesis Conclusions vs. Published Literature in CISCC

Thesis Conclusions	Published Literature
Effect of Temperature	
The effect of increasing temperature from 35°C to 50°C for both exposure durations is deemed <u>not significant</u>. However, the statistical analysis indicates increased pitting and potentially cracking.	<u>NRC Conclusions:</u> The NRC observed cracking on all samples at 43°C and no cracking was observed at 80°C or 120°C. <u>Mintz’s Conclusions:</u> SCC was evident on 304 U-bend samples exposed to 35, 45, and 52°C but not at 60°C. Increased temperature did not necessarily correlate with increased SCC susceptibility. The number of cracked samples at 35°C, 45°C, 55°C were comparable on all sample types.

Thesis Conclusions	Published Literature
	Zakaria's Conclusions: Zakaria concluded that as temperature increased from 28°C to 70°C, pitting and cracking increased. However, Zakaria's experiment was an immersion experiment which is different from fogging experiments.
Effect of Welding	
Welded samples are less susceptible to CISCC than unwelded samples from both qualitative and quantitative analyses. This applies to 304 and 304L U-bend samples.	NRC Conclusions: The NRC results show fewer cracked 304 welded samples than unwelded samples exposed for 4 and 16 weeks. Mintz's Conclusions: Mintz concluded that 304 welded samples were less susceptible to cracking than heat sensitized and as-received samples. Zakaria's Conclusions: Zakaria concluded that 304L heat sensitized samples were more susceptible to CISCC than base metal samples. However, these samples were heated to 600°C for 2 hours and not welded.
Effect of Expose Duration	
Increased test duration from 8 to 12 weeks at 35°C and from 8 to 14 weeks at 50°C had no significant effect on the acceleration of pitting and cracking for all sample types based on the qualitative and quantitative analyses.	NRC Conclusions: The number of cracked 304 samples after 4 weeks of exposure were comparable to the number of cracked samples after 16 weeks of exposure. For 316L samples, the number of cracked samples after 32 weeks of exposure was comparable to the number of cracked samples after 52 weeks of exposure.
Effect of Material	
The susceptibility of 304 samples compared with 304L is not significant based on the qualitative and quantitative analyses.	NRC Conclusions: The NRC susceptibility maps show that the number of cracked 304L samples increased as exposure duration increased unlike 304 and 316 samples. Indicating that 304L samples were more susceptible to CISCC than 304 with longer exposure.

5. Chapter 5: Conclusions and Future Work

The experimental design established in this research simulated a controlled corrosive environment to test austenitic stainless-steel U-bend samples. The experimental conditions simulated were relevant to the long-term storage of SNF in DSCs near marine environments (coastal corrosive areas). The objective of this research is to examine the sensitivity of CISCC to welding, increased temperature, and material's composition and to provide recommendations on the susceptibility of DSCs to CISCC degradation.

The effect of increasing the exposure temperature from 35°C to 50°C cannot be determined based on the qualitative observations; the number of cracked samples at 35°C for both exposure durations were comparable to the number of cracked samples at 50°C for both exposure durations. However, pitting was more severe on the samples exposed to 50°C for 14 weeks in comparison with samples exposed to 35°C for 12 weeks on all sample types. The quantitative statistical analysis agreed with the qualitative analysis and showed that temperature increase from 35°C to 50°C had no effect on the acceleration of CISCC for 304 and 304W when tested for a significance level of 5%. However, the mean of surface profiles' depths (or valleys) of the 304 sample exposed to 35°C was larger than the surface profiles' mean of the 304 sample exposed to 50°C as expected. This indicates a potential increase in pitting and cracking on the samples exposed to 50°C, but it was deemed not significant per the t-test results. The same conclusions can be made on the 304W samples, the surface profiles' mean was smaller on the sample exposed to 50°C as expected.

Regarding the effect of welding, welded samples were less susceptible to CISCC than unwelded samples based on the qualitative analysis. Three "likely cracking" observations were noted on the welded samples compared with one "likely cracking" on the unwelded samples from both experimental campaigns 1 (35°C) and 2 (50°C). The statistical analysis also showed that welded samples were less susceptible to CISCC than unwelded samples based on several profile comparisons. The mean of surface profiles' depths of the 304 welded sample exposed to 35°C for 12 weeks was larger than the surface profiles' mean of the unwelded 304 sample exposed to 35°C for 12 weeks indicating reduced susceptibility (fewer valleys caused by corrosion and cracking). However, heavier pitting was observed on all welded samples in comparison with unwelded samples. In theory, welded samples should be more susceptible to pitting and cracking due to the depletion of chromium when the material surrounding the weld area is heated to high temperatures (600-800°C). Further examination of welded vs. unwelded samples is desired in future research to examine specific welding procedures and attributing factors to the reduced susceptibility.

The susceptibility of 304 compared with 304L was determined not significantly different based on the qualitative analysis. The level of cracking and pitting from both experimental campaigns and test durations were comparable for both 304 and 304L except for one instance where the 304 sample was more susceptible than the 304L sample (50°C for 8 weeks). Also, the statistical analysis showed that the susceptibility of 304 samples compared with 304L were not significantly different for a significance level of 5%. The means of surface profiles' depths of 304 and 304L samples were relatively similar.

The effect of increasing exposure duration from 8 to 12 weeks at 35°C and from 8 to 14 weeks at 50°C had no significant effect on the acceleration of pitting and cracking for all sample types based on the qualitative analysis. Cracking and likely cracking observations were relatively similar for both durations. However, increased pitting was observed with increased exposure duration at both exposure temperatures. Also, the statistical analysis showed that increasing the exposure duration had no significant effect on surface profiles for all sample types at both exposure temperatures. The surface profiles' means were comparable for the different exposure durations.

Based on the results of this research, cracking due to chloride-induced stress corrosion cracking is likely to occur at the following combined conditions,

- Exposure temperatures ranging from 35°C to 50°C for 8 weeks or more of exposure
- Materials made from 304 and 304L austenitic stainless steels
- Applied tensile stresses (bent surfaces)
- RH greater than 95%
- NaCl Salt concentration of 5% by weight

Based on the conditions used in this experimental work, the risk of CISCC in SNF dry storage canisters stored near marine environments is very likely. However, the acceleration factor caused by testing the samples at high salt concentration and high RH was not quantified in this research. To estimate the service life of DSCs, laboratory experiments are needed to estimate the acceleration factor and crack growth rates by testing samples at various RH levels and salt concentrations (*e.g.,* 1 - 5% wt.).

The recommendation for future CISCC experimental work is to perform additional accelerated experiments for 304 and 304W U-bend austenitic stainless-steel samples at the following conditions,

- Constant exposure temperature in the range 35°C to 50°C
- Constant tensile stress comparable to DSCs stresses
- Extended exposure durations 3, 6, 9, 12, and 24 months

- Various RHs comparable to the expected RH in marine environments
- Various salt concentrations to estimate the acceleration factor

Furthermore, gathering an extensive and large set of surface profiles using the 3D Surface Profiler before and after exposure is critical for estimating the acceleration factor. Additionally, crack lengths post exposure can be gathered using the 3D Surface Profiler to estimate crack propagation rate for each exposure duration. Moreover, non-destructive examination techniques such as acoustics and ultrasonic techniques should be considered to verify the 3D Surface Profiler's data. As an example, ultrasonic examination could be used first to locate areas of heavy pitting and cracking for further examination by the 3D Surface Profiler. Identified locations of heavy pitting or cracking can be polished before the 3D surface profiler examination to enhance visualization of the cracks. This research would require years of testing and material characterization, but it provides a reasonably accurate time to failure models to estimate the service life of SNF dry storage canisters.

Other factors that could be investigated in future experiments are the effects of varying the applied tensile stress, cyclic effect, and the effect of salt composition. Based on published literature, high applied tensile stresses accelerate time to failure due to CISCC. Since variations in the applied stresses on DSCs are not expected, future experiments should consider tensile stresses comparable to those on DSCs. Additionally, cyclic effects were not investigated in this research. Some corrosion chambers provide the ability to alternate between wet and dry cycles, fogging is distributed for a predetermined duration with dry cycles (hold time) in between to allow fogging to dry leaving behind solid salt deposits on the material's surface. Solid salt deposits then deliquesce on the materials' surface by absorbing moisture from the surrounding air. This could potentially have an accelerating effect on CISCC that could be investigated in future research.

Finally, the effect of salt's composition was not investigated in this research. Future experiments could potentially examine the effect of salt's composition on the acceleration of CISCC using salts representative of those encountered in coastal areas (*e.g.,* $MgCl_2$, KCl, etc.). Future experiments should consider the threshold RH required for each tested salt; salt's ability to deliquesce is a function of the atmospheric RH.

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7. Appendices

[1]	Appendix A	SCCH22 Chamber Calibration Certificates
[2]	Appendix B	U-bend Samples Material Certificates
[3]	Appendix C	Morton Culinox 999 Salt Certificate
[4]	Appendix D	Data Collected During the Experiment
[5]	Appendix E	Diagrams of Sample Extraction
[6]	Appendix F	MicroCT Sample Scans
[7]	Appendix G	3D Surface Profiler Sample Results
[8]	Appendix H	Summary of Surface Profile Data
[9]	Appendix I	ANOVA and T-test Results

SINGLETON
CORPORATION

3280 w. 67th place | cleveland, ohio 44102
216.651.7800 | fax: 216.651.4247 | www.singletoncorp.com

Corrosion Test Chambers

Statement of Certification

for ASTM Standard B117-18

Model#: SCCH22 **Serial#:** 22-57190 **Description:** corrosion test chamber

Electrical: 480 **volts** 3 **phase L1 amps** 12 **L2 amps** N/A **L3 amps** 12

This hereby certifies that the above referenced equipment was operationally tested for temperature, pressure, and collection rate in accordance with ASTM B117-18, calibrated and certified on

11-17-20

Date

Chamber Exposure Temperature: 95

Humidifying Tower Temperature: 118

Humidifying Tower Pressure (psig): 15 **psi**

Atomizer Flow Rate* (scfh): 43 **scfh**

Makeup Solution pH*: 7 **Makeup Solution Specific Gravity (s.g.)*:** ______

Collected Condensate pH: 7 **Collected Condensate Specific Gravity (s.g.):** ______

Collection Rate:

Opti-Fog "A"		Opti Fog "B" (*If Applicable*)	
1.7 ml/hr	1.1 ml/hr	____ ml/hr	____ ml/hr
(near) 1	(far) 2	(near) 3	(far) 4

Technician: [signature] **Date:** 11-17-20

***data not required by ASTM specifications**

Factory Corrosion Test Report

Corrosion Test Report

Total Test Cycle Time: 16 **hours**

No. of cycles: 1

Total Test Time: 16 **hours**

DI water supply pH-uS/cm: ____

Notes: ______________________

Condensate Collection Mapping Diagram – Exposure Zone

Collector 1: 20 total 1.3 ml/hr.
Collector 2: 17 total 1.1 ml/hr

Collector 3: ____ total ____ ml/hr
Collector 4: ____ total ____ ml/hr

4 2

Opti-Fog "A" **Opti-Fog "B"**

1 3

SINGLETON CORPORATION

SCCH Factory Calibration Sheet
Cal-Comm Control System

Form No: SCCHCAL-CALCOMM - 001 REV 8 Date: 1/23/19

Customer

Name: Virginia polytechnic Int and ST Unt

Address: 445 Goodwin hall

635 prices fork Rd.

City: blacksburg State: VA Zip: 24061

Equip. Certified

Model #: SCCH22

Serial #: 22-57190

Description: corrosion test chamber

Contact Info

Name: ______ Phone: ( ) ______ Fax: ( ) ______

Components Calibrated

Description	Manufacturer	Model #
Chamber Temperature Control	Cal-Comm	3300
Tower Temperature Control	Cal-Comm	3300
Chart Recorder Wet/Dry Bulb	Honeywell	DR4300

Calibration Equipment Used

Description	Manufacturer	Model #	Calibrated / Expires
Digital Thermometer	Fluke 51	971600180	1-8-20 / 1-8-21

Calibration Procedure Tolerance Data

Procedure: Compare component data to certified measurement equipment.
Tolerance: Calculated at +/-1°F across entire range

Temperature Controller Calibration

	Cabinet (Dry Bulb)			Wet Bulb			Tower		
	Reference	Before Cal	After Cal	Reference	Before Cal	After Cal	Reference	Before Cal	After Cal
Temperature (°F)	95	97.5	95	95	96.8	95	118	114.5	118
Controller Zero Setting			-2.5			-1.8			-1.5

Chart Recorder Calibration (option O-6 only)

Temperature (°F)	Cabinet (Dry Bulb)			Wet Bulb		
	Reference	Before Cal	After Cal	Reference	Before Cal	After Cal
LOW	N/A			N/A		
HIGH						

Calibrated by: [signature]

Date: 11-17-20

H.J. KIRBY CORP.
29325 CLEMENS ROAD • WESTLAKE, OHIO 44145-1093
TELEPHONE: (440) 835-3440
FAX: (440) 835-0233

Certificate of Calibration

Date: 9/29/20

Owner of Instrument: Singleton Corp

Reference: 16099

The instrument referenced herein has been certified to the accuracy shown below on **WIKA Instrument Test Gauge Serial #HJK30** certified October 10, 2019, which is traceable to the National Institute of Standards and Technology, certified by H J Kirby Corp, on. **Chandler Engineering Type 23-1 Deadweight Tester, Serial Number 1240**, traceable to the National Institute of Standards and Technology. through Ashland Scale Co, Inc , which is an ANSI/NCSL Z540-1 and ISO/IEC 17025 certified lab. Certificate #L2114-1, Calibration dated October 10, 2017.

Instrument Description: WIKA 111.10 4.0" 1/4LM 30 PSI/kPa w/adj ptr

New H.J. Kirby Corp. Test No.: 200927
Prior H.J. Kirby Corp. Test No.: N/A
Serial Number on Socket: 200927

The following table documents our findings that this instrument is currently accurate within a maximum error of 1.7% of full scale, which is within the catalog accuracy of 3.0%-2.0%-3.0% full scale. We do hereby certify these findings to be accurate and true. Readability of this instrument is plus or minus 0.25 PSI or 0.8% full scale.

Temperature: 68F Humidity: 92%

Deadweight Tester	Upscale Reading	Downscale Reading	Maximum Deviation	Percent of Full Scale
5.00	5.00	5.00	0.00	0.0
15.00	14.75	14.75	0.25	0.8
25.00	24.50	24.50	0.50	1.7
30.00	29.50		0.50	1.7

Testing performed by Richard Otter

Signed and Certified ______________________
Richard Otter

Metal Samples
A Division of Alabama Specialty Products, Inc.

Metal Samples Co.
152 Metal Samples Rd.
P.O. Box 8
Munford, AL 36268
Phone: (256) 358-4202
Fax: (256) 358-4515
E-mail: msc@alspi.com
Internet: www.metalsamples.com

November 3, 2020

CERTIFICATE OF CONFORMITY

METAL SAMPLES QUALITY ASSURANCE CERTIFIES AND AFFIRMS THAT ALL PRODUCT SUPPLIED AGAINST **VIRGINIA POLYTECHNIC INSTITUTE P.O. # P3821153** HAS BEEN MANUFACTURED IN ACCORDANCE WITH ALL QUALITY REQUIREMENTS AS SET FORTH IN THE ABOVE-MENTIONED PURCHASE ORDER, **USING MATERIAL LISTED BELOW.**

MATERIALS, WORKMANSHIP, AND DIMENSIONAL INTEGRITY OF THE PRODUCT SUPPLIED WERE VERIFIED IN ACCORDANCE WITH THE REQUIREMENTS OF OUR ISO 9001-2015 CERTIFIED QUALITY SYSTEM; QUALITY MANUAL, REV. 3, DATED JANUARY 16, 2020.

LINE	ALLOY	LOT#
1	304 SS	AZ377
2	304L SS	BA333
3	304 SS (WELDED)	AY941
4	304L SS (WELDED)	BB352

Larry Braden K.T.

LARRY BRADEN
QUALITY MANAGER

*

Request: 5528181
Samuel, Son & Co., LTD.

ALABAMA SPECIALTY PRODUCTS PO# 139090
SSM Order # 1514903-

SSM Order Line#	Job#	Customer Part#	Heat/Lot#	# of tags
1	SS 304 2B Sheet 16 GA X 60" X 120"			
			TE00737	(1)

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT

MTL ID NO. BA333 PO NO. 139090
ALLOY 304L INITIALS CH

NOTICE OF SHIPMENT/ PACKING LIST

A&T STAINLESS

A&T 1000 218

CERTIFICATE OF TEST

CUST ORDER NO. & DATE		CUST CODE	FINISHING LOCATION	SHIPPER NO	PRODUCT CODE	MILL ORDER NUMBER	DATE SHIPPED
018S-7380965	07/16/19	100506	MIDLAND, PA	411972	13020102060064	72-079-101	08/02/19

FORMS DISTRIBUTION: SOLD TO → | SHIP TO → | SPEC →

MG BILL L291069

REPEAT ORDER 11148-0 | DO RATE 25 | PRIORITY

GOV'T CONTRACT

MAT'L 1121 | SHIPPING LOCATION MIDLAND | PA | INVOICE 007686

SOLD TO	PRIME	SEC.	SHIP TO
SAMUEL SON & CO INC	DSO	DSO	SAMUEL SON & CO INC
3635 FRANCIS CIRCLE	10		3635 FRANCIS CIRCLE
ALPHARETTA GA 30004			ALPHARETTA GA 30004

GRADE AND SPECIFICATIONS CARRIER - IDAHO TRUCK LINES INC.

304 STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE A&T STAINLESS (AMS 5513J) (AMSQQS763D-CONDACHEM/MECH COLD F) (ASTM-A-480-18A) (ASME-SA-480 ED 2017) (ASTM-A-262-15 PRA E/SCREEN PRA A) (QQ-S-766D) (NACE MR0175/ISO 15156-3:2015) (ASME-SA-666 ED 2017) (UNS S30400) (DIN EN 10204:2005 3.1 CERTIFICATE) (ASTM-A-666-15) (ASME-SA-240 ED 2017) (ASTM-A-240-16)

ITEM	PCS	DIMENSIONS W/G/L	HEAT #	COIL #	TEST #	GROSS	TARE	NET	THEO	TAG #/ CD SKID #
004A	1	60.046/.059/1878.	TE00737	AAC45313-A	M415341	22310		22310		637248
	1	60.046/.059/1969.	TE00737	AAC45313-B	M415341	23430		23430		637208
C CUST IDENTITY 5136072-JV										
	2	COILS				45740		45740		

CB12

DIST: EMAIL S/N TO ASHLEY.HUNT@SAMUEL.COM EMAIL INV TO ATLANTAAP@SAMUEL.COM

TYPE HEAT/TEST (WT %)	C	MN	P	S	SI	CR	NI	MO	CU	N
HEAT TE00737	.02	1.62	.031	.002	.44	18.29	8.11	.001	.04	.06
TEST LOCATION	TC	TC	TC	TC	TC	TC	TC	TC	TC	TC

ITEM TEST NO	YIELD PSI *	TENSILE PSI	% ELONG IN 2"	% R/A	HARDNESS	BEND	CORROSION A 262 PR E	ASTM GRAIN SIZE	HARDENABILITY
004A M415341				NR	86.HRBW	T PASS	PASS	NR	NR
	T 48100.	99500.	55. #		86.HRBW				
TEST LOCATION	TC	TC	TC		TC		TC		

* Y.S. BY 0.2% OFFSET METHOD
# ELONG AT FRACTURE

NR = DATA NOT REQUIRED

GRADE VERIFICATION WAS CARRIED OUT SPECTROSCOPICALLY

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. BA333 PO NO. 139090
ALLOY 304L INITIALS CK

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PAGE 01 - CONTINUED ON PAGE 02 08/02/19 10:34:57

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Robert Iseman
Sr. Manager, Technology

NOTICE OF SHIPMENT/ PACKING LIST

A&T STAINLESS

A&T 1000 218

CERTIFICATE OF TEST

CUST ORDER NO & DATE		CUST CODE	FINISHING LOCATION	SHIPPER NO	PRODUCT CODE	MILL ORDER NUMBER	DATE SHIPPED
0185-7380965	07/16/19	100506	MIDLAND, PA	411972	1302010206064	72-079-101	08/02/19

FORMS DISTRIBUTION: SOLD TO | SHIP TO | SPEC

MG BILL
L291069

REPEAT ORDER	DO RATE	PRIORITY	GOV'T CONTRACT	MAT'L	SHIPPING LOCATION		INVOICE
11148-0	25			1121	MIDLAND	PA	007686

PRIME SEC.
DSO DSO
10

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. B4333 PO NO. 139090
ALLOY 304L INITIALS CK

SOLD TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

SHIP TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

CARRIER - IDAHO TRUCK LINES INC.

GRADE
304 STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE A&T STAINLESS

ALLEGHENY & TSINGSHAN STAINLESS DOES NOT USE MERCURY IN THE TESTING OR PRODUCTION OF ITS PRODUCTS.

MATERIAL WAS MELTED IN THE REPUBLIC OF INDONESIA. MATERIAL WAS ROLLED AND ANNEALED IN THE USA.

THESE COMMODITIES OR TECHNOLOGY, WHEN EXPORTED FROM THE UNITED STATES, ARE IN ACCORDANCE WITH THE USA EXPORT ADMINISTRATION REGULATIONS. DIVERSION CONTRARY TO U.S. LAW IS PROHIBITED. U.S. LAW ALSO PROHIBITS DISPOSITION OF THESE COMMODITIES/TECHNICAL DATA TO ANY END-USER OR FOR ANY END-USE RELATED TO THE DESIGN, DEVELOPMENT, PRODUCTION, STOCKPILING, OR USE OF CHEMICAL, BIOLOGICAL, OR NUCLEAR WEAPONS, OR MISSILES, WITHOUT THE PROPER APPROVAL OF THE UNITED STATES GOVERNMENT.

DIN EN 10204:2005 - ALLEGHENY AND TSINGSHAN STAINLESS, LLC IS APPROVED AS A MANUFACTURER ACCORDING TO PRESSURE EQUIPMENT DIRECTIVE PED 2014/68/EU.

MATERIAL WAS PRODUCED IN ACCORDANCE WITH ALLEGHENY & TSINGSHAN STAINLESS QUALITY MANUAL DATED MAY 13 2019. CERTIFICATE OF TEST SHALL NOT BE REPRODUCED EXCEPT IN FULL. FEDERAL LAW PROHIBITS THE RECORDING OF FALSE, FICTITIOUS, OR FRAUDULENT STATEMENTS OR ENTRIES ON THE CERTIFICATE AND MAY BE PUNISHABLE AS A FELONY UNDER FEDERAL LAW.

ALLEGHENY & TSINGSHAN STAINLESS, LLC QUALITY SYSTEM CERTIFIED ACCORDING TO EQUIPMENT DIRECTIVE 2014/68/EU , ANNEX I, SECTION 4.3 BY TUV SUD, INDUSTRIE SERVICE GMBH, (NOTIFIED BODY 0036).

ALLEGHENY & TSINGSHAN HAS NO CONTROL OVER CUSTOMER PROCESSING OF MATERIAL; INCLUDING, BUT NOT LIMITED TO, BUFFING OR ELECTROPOLISHING MATERIAL WITH A 2B OR OTHER FINISH, AND DOES NOT REPRESENT OR GUARANTY THAT THE MATERIAL IS SUITABLE FOR ANY PARTICULAR PROCESS OR USE OF CUSTOMER.

EN 10204:2005 3.1 CERTIFICATE.

1

PAGE 02 - CONTINUED ON PAGE 03 08/02/19 10:34:57

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Robert C [signature]
Robert Iseman
Sr. Manager, Technology

NOTICE OF SHIPMENT/ PACKING LIST

A&T STAINLESS

A&T 1000 218

CERTIFICATE OF TEST

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0185-7380965	07/16/19	100506	MIDLAND, PA	411972	13020102060064	72-079-101	08/02/19

FORMS DISTRIBUTION: SOLD TO → | SHIP TO → | SPEC →

MG BILL	REPEAT ORDER	DO RATE	PRIORITY	GOV'T CONTRACT	MAT'L	SHIPPING LOCATION		INVOICE
L291069	11148-0	25			1121	MIDLAND	PA	007686

SOLD TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

PRIME SEC.
DSO DSO
10

SHIP TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

GRADE
304 STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE A&T STAINLESS

CARRIER - IDAHO TRUCK LINES INC.

TESTING WAS PERFORMED AT THE FOLLOWING LOCATIONS:
TC = ATI-FLAT ROLLED PRODUCTS; 1300 PACIFIC AVENUE; NATRONA HEIGHTS, PA 15065

<<<<<<<< FOR ACCESS TO ONLINE CERTIFICATES OF TEST >>>>>>>>
<<<<<<<< REGISTER AT MYATI.ATIMETALS.COM >>>>>>>>

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. BA333 PO NO. 139090
ALLOY 304 L INITIALS CH

PAGE 03 - FINAL PAGE. 08/02/19 10:34:57

1

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Robert C Iseman
Robert Iseman
Sr. Manager, Technology

AL 6168-B 0417

NOTICE OF SHIPMENT/ PACKING LIST

ATI Flat Rolled Products

CERTIFICATE OF TEST

CUST. ORDER NO. & DATE		CUST. CODE	FINISHING LOCATION	SHIPPER NO.	PRODUCT CODE	MILL ORDER NUMBER	DATE SHIPPED
018C-7392542	10/14/19	100506	LOUISVILLE, OH	425150	13020102060000	30-109-231	12/09/19

FORMS DISTRIBUTION: SOLD TO → | SHIP TO → | SPEC

MG BILL L330758

REPEAT ORDER 11874-0 | DO RATE 25 | PRI-ORITY

GOV'T CONTRACT

MAT'L 1124 | SHIPPING LOCATION LOUISVILLE OH | INVOICE 247627

PRIME DSO 10 | SEC. DSO

SOLD TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

SHIP TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

GRADE AND SPECIFICATIONS

CARRIER - IDAHO TRUCK LINES INC.

"ATI 304L" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE (304/304L) (AMS 5511K) (AMS 5513J) (ASME-SA-480 ED 2017) (AMSQQS763D-CONDA-CHEM/MECH COLD F) (ASTM-A-480-18A) (QQ-S-766D) (ASTM-A-262-15 PRA E/SCREEN PRA A) (NACE MR0175/ISO 15156-3:2015) (DIN EN 10204:2005 3.1 CERTIFICATE) (UNS S30400) (UNS S30403 (ASME-SA-666 ED 2017) (ASTM-A-666-15) (ASME-SA-240 ED 2017) (ASTM-A-240-18) (NACE MR0103/ISO 17945:2015)

ITEM	PCS	DIMENSIONS W/G/L	HEAT #	COIL #	TEST #	GROSS	TARE	NET	THEO	TAG #/ CD SKID
001A	1	48./.060/2305.	859283	AAC83580-B	L311431	22360	30	22330		657281
	1	48./.060/2205.	859283	AAC83580-A	L311433	21420	40	21380		657327
C CUST IDENTITY 5142042										
	2	SKIDS	CB5			43780	70	43710		

DIST: EMAIL S/N TO ASHLEY.HUNT@SAMUEL.COM EMAIL INV TO ATLANTAAP@SAMUEL.COM

TYPE HEAT/TEST (WT %)	--C---	--MN--	--P---	--S---	--SI--	--CR--	--NI--	--MO--	--CU--	--N---
HEAT 859283	.024	1.61	.032	.0003	.53	18.09	8.25	.46	.60	.07
TEST LOCATION	BN	BN	BN	BN	BN	BN	BN	BN	BN	BN

ITEM TEST NO	YIELD PSI *	TENSILE PSI	% ELONG IN 2"	% R/A	HARDNESS	BEND	CORROSION A 262 PR E	ASTM GRAIN SIZE	HARDENABILIT
001A L311431				NR	86.HRBW	T PASS	PASS	NR	NR
	T 49300.	93000.	53. #		86.HRBW				
TEST LOCATION	TC	TC	TC		TC	TC	TC		
L311433				NR	85.HRBW	T PASS	PASS	NR	NR
	T 48400.	93000.	54. #		85.HRBW				
TEST LOCATION	TC	TC	TC		TC	TC	TC		

* Y.S. BY 0.2% OFFSET METHOD
\# ELONG AT FRACTURE

PAGE 01 - CONTINUED ON PAGE 02

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPAN
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. BB352 12/09/19 14:14:04 PO NO. #276641
ALLOY 304L INITIALS LA

CONSIGNEE—Please Note—This consignment was turned over to carrier in first class condition, being correctly loaded, at which time our responsibility for loss or damage in shipment ceased. For your protection please examine shipment as it arrives. If any shortage or damage is discovered, have a full description made by transportation agent on waybill before signing.

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The above is a true copy of data on file. The material and test results conform to the sales contract and specification(s) as set forth in ATI Flat Rolled Products' Order Acknowledgement.

Noreen DeFerrari
Sr. Manager Quality, Corporate Quality Assurance

AL 6168-8 0417

NOTICE OF SHIPMENT/ PACKING LIST

ATI Flat Rolled Products

CERTIFICATE OF TEST

CUST ORDER NO. & DATE		CUST CODE	FINISHING LOCATION	SHIPPER NO.	PRODUCT CODE	MILL ORDER NUMBER	DATE SHIPPED
018C-7392542	10/14/19	100506	LOUISVILLE, OH	425150	130201020600000	30-109-231	12/09/19

MG BILL
L330758

REPEAT 11874-0 OD 23 PRI. GRADE PRIORITY
PRIME SEC.
DSO DSO
10

GOV'T CONTRACT

MAT'L 1124 SHIPPING LOCATION LOUISVILLE OH

INVOICE 247627

FORMS DISTRIBUTION SOLD TO → SHIP TO → SPEC

SOLD TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

SHIP TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

CARRIER - IDAHO TRUCK LINES INC.

GRADE
"ATI 304L" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE

NR = DATA NOT REQUIRED
GRADE VERIFICATION WAS CARRIED OUT SPECTROSCOPICALLY

ATI FLAT ROLLED PRODUCTS DOES NOT USE MERCURY IN THE TESTING OR PRODUCTION OF ITS PRODUCTS.

MATERIAL IS OF USA MELT AND MANUFACTURE.

THESE COMMODITIES OR TECHNOLOGY, WHEN EXPORTED FROM THE UNITED STATES, ARE IN ACCORDANCE WITH THE USA EXPORT ADMINISTRATION REGULATIONS. DIVERSION CONTRARY TO U.S. LAW IS PROHIBITED. U.S. LAW ALSO PROHIBITS DISPOSITION OF THESE COMMODITIES/TECHNICAL DATA TO ANY END-USER OR FOR ANY END-USE RELATED TO THE DESIGN, DEVELOPMENT, PRODUCTION, STOCKPILING, OR USE OF CHEMICAL, BIOLOGICAL, OR NUCLEAR WEAPONS, OR MISSILES, WITHOUT THE PROPER APPROVAL OF THE UNITED STATES GOVERNMENT.

EN 10204:2005 - ATI FLAT ROLLED PRODUCTS IS APPROVED AS A MANUFACTURER ACCORDING TO THE PRESSURE EQUIPMENT DIRECTIVE PED 2014/68/EU.

MATERIAL IS COMPLIANT WITH THE EUROPEAN UNION DIRECTIVES 2011/65/EU (ROHS2) AND 2015/863/EU (ROHS3) - RESTRICTION OF HAZARDOUS SUBSTANCES.

MATERIAL MELTED AND ROLLED IN THE UNITED STATES AND COMPLIES WITH DFARS JANUARY 2010 EDITION, SECTION 252.225-7008 AND 252.225-7009, FOR SPECIALTY METALS (JUL 2009).

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. BB352 PO NO. 142641
304L 14
INITIALS

PAGE 02 - CONTINUED ON PAGE 03

12/09/19 14:14:04

1

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AL 6168-8 0417

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018C-7392542 10/14/19	100506	LOUISVILLE, OH	425150	130201020600000	30-109-231	12/09/19

MG BILL
L330758

REPEAT	DO RATING	PRIORITY	GOV'T. CONTRACT	MAT'L	SHIPPING LOCATION		INVOICE
11874-0		25		1124	LOUISVILLE	OH	247627

PRIME SEC.
DSO DSO
10

FORMS DISTRIBUTION: SOLD TO → SHIP TO → SPEC.

SOLD TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

SHIP TO
SAMUEL SON & CO INC
3635 FRANCIS CIRCLE
ALPHARETTA GA 30004

CARRIER - IDAHO TRUCK LINES INC.

GRADE
"ATI 304L" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE

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QUALITY SYSTEM CERTIFIED ACCORDING TO PRESSURE EQUIPMENT DIRECTIVE 2014/68/EU, ANNEX I, 4.3 BY TUV SUD, INDUSTRIE SERVICE GMBH, (NOTIFIED BODY 0036).

ATI FLAT ROLLED PRODUCTS PERFORMS CHEMICAL ANALYSIS BY THE FOLLOWING TECHNIQUES
FOR TESTING LOCATIONS TC, BN, & LO:
C, S BY COMBUSTION/INFRARED
N, O, H BY INERT FUSION/THERMAL CONDUCTIVITY
MN, P, SI, CR, NI, MO, CU, CB, CO, V BY WDXRF
B BY OES
AL AND TI (>=0.10%) BY WDXRF, OTHERWISE BY OES
PB, BI, AG BY GFAA

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES — METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. 8B352 PO NO. 142641
ALLOY 304L INITIALS 4

ATI FLAT ROLLED PRODUCTS HAS NO CONTROL OVER CUSTOMER PROCESSING OF MATERIAL; INCLUDING, BUT NOT LIMITED TO, BUFFING OR ELECTROPOLISHING MATERIAL WITH A 2B OR OTHER FINISH, AND DOES NOT REPRESENT OR GUARANTY THAT THE MATERIAL IS SUITABLE FOR ANY PARTICULAR PROCESS OR USE OF CUSTOMER.

EN 10204:2005 3.1 CERTIFICATE.

PAGE 03 - CONTINUED ON PAGE 04

12/09/19 14:14:04

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PACKING LIST

ATI Flat Rolled Products

AL 6168-8 0417

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018C-7392542	10/14/19 MG BILL	100506	LOUISVILLE, OH	425150	13020102060000	30-109-231	12/09/19

FORMS DISTRIBUTION		REPEAT	DO RATING	PRIORITY	GOV'T CONTRACT	MAT'L	SHIPPING LOCATION		INVOICE
SOLD TO → SHIP TO → SPEC →	L330758	11874-0	23			1124	LOUISVILLE	OH	247627

SOLD TO	PRIME	SEC.	SHIP TO
SAMUEL SON & CO INC	DSO	DSO	SAMUEL SON & CO INC
3635 FRANCIS CIRCLE	10		3635 FRANCIS CIRCLE
ALPHARETTA GA 30004			ALPHARETTA GA 30004

GRADE

CARRIER - IDAHO TRUCK LINES INC.

"ATI 304L" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE

TESTING WAS PERFORMED AT THE FOLLOWING LOCATIONS:
BN = ATI FRP BRACKENRIDGE; 100 RIVER ROAD; BRACKENRIDGE, PA 15014
TC = ATI FRP NATRONA HEIGHTS; 1300 PACIFIC AVENUE; NATRONA HEIGHTS, PA 15065

<<<<<<<< FOR ACCESS TO ONLINE CERTIFICATES OF TEST >>>>>>>>
<<<<<<<< REGISTER AT MYATI.ATIMETALS.COM >>>>>>>>

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. BB352 PO NO. 142641
ALLOY 304L INITIALS LA

PAGE 04 - FINAL PAGE.

12/09/19 14:14:04

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Noreen DeFerrari
Sr. Manager Quality, Corporate Quality Assurance

NAS NORTH AMERICAN STAINLESS

6870 Highway 42 East
Ghent, KY 41045-9615
(502) 347-6000

METALLURGICAL TEST REPORT

Certificate: 433944 01
Customer: 0305 803

Mail To:
PHOENIX METALS COMPANY
4200 EAST LAKE BLVD.
BIRMINGHAM, AL 35217

Ship To:
PHOENIX METALS COMPANY
4200 EAST LAKE BLVD.
BIRMINGHAM, AL 35217

Date: 8/02/2018 Page: 1
Steel: 304
Finish: No 4
Corrosion: ASTM A262/15 A;180Bend-OK

Your Order: 213383
NAS Order: AN 0820816 01

PRODUCT DESCRIPTION:

STLS STL COIL, C.R. ANNEALED & PICKLED; POLISHED;UNS 30400
ASTM A240/17,A480/16b,A666/15;ASME SA240/17,SA480/17,SA666/17
CHEM ONLY ON FOLLOWING ASTM: A276/17,A479/17,A484/16,A312/16
CHEM ONLY ON FOLLOWING ASME: SA312/13,SA479/13
AMS 5513J XMRK; MIL-S-5059D AMEND3 (X CROWN MEAS)
NACE MR0175/ISO 15156-3:2009 A, MR0103/07;QQS766D-A X MAG PERM
MIN. SOLUTION ANNEAL TEMP 1900F, WATER QUENCHED

REMARKS:

Mat'l is Free of Mercury Contamination. No weld repairs.
EN 10204:2004 3.1; RoHS 1 & 2 Compliant
Material is Free of Radioactive Contamination
Steel Making Process: EAF, AOD, & Cont. Casting
Product Mfg.by a Quality Mgt.Sys. in Conf. w/ISO 9001
*Melted & Manufactured in the USA; Mat'l is DFARs Compliant
*Melted & Manufactured in the USA; Mat'l is DFARs Compliant

Product ID #	Coil #	Thickness	Width	Weight	--------Length-------		Mark	Pieces	COMMODITY CODE
03897D C	* 03897D C	.0575	48.0000	18,940	COIL	2049.7	4	1	8494 Low Temp

ANAB, ISO/IEC 17025, Certificate# L2323

CHEMICAL ANALYSIS CM(Country of Melt) ES(Spain) US(United States) ZA(South Africa) JP(Japan)

Chemical Analysis per ASTM A751/14a

HEAT	CM	C %	CR %	CU %	MN %	MO %	N %	NI %	P %	S %
897D	US	.0417	18.1420	.4070	1.7040	.3250	.0629	8.0760	.0330	.0013
		SI %								
		.1950								

MECHANICAL PROPERTIES

Product ID #	Coil #	loc	dir	UTS KSI	20C .2% YS KSI	20C ELONG %-2"	% Hard RB	Tail Hard
03897D C	03897D C	F	T	101.05	51.72	47.95	88.50	86.00

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. AY941 PO NO. 134893
ALLOY 304 INITIALS LA

NAS hereby certifies that the analysis on this certification is correct. Based upon the results and the accuracy of the test methods used, the material meets the specifications stated. These results relate only to the items tested and this report cannot be reproduced, except in its entirety, without the written approval of NAS.

Technical Dept. Mgr. KRIS LARK 8/03/2018

APPLIED TECHNICAL SERVICES, INCORPORATED

1049 Triad Court, Marietta, Georgia 30062 • (770) 423-1400 Fax (770) 424-6415

CHEMICAL TEST REPORT

Ref. 308581 Rev.1* **Date** February 4, 2019 **Page** 1 **of** 1

Customer: Alabama Specialty Products, Inc., 152 Metal Samples Rd., Munford, AL 36268

Attention: Larry Braden

Purchase Order #: 136266 Part #/Name: 3"x3"x1/16" Sample, Lot# AZ377

Material Designation: 304 Stainless Steel

Special Requirement: Customer specified carbon range.

Lab Comment: Analyzed using ASTM E1019-18 and ASTM E572-13 as guides.

Test Results

Composition: Weight %

Identification	C(2)	Mn	Si	Cr	Ni	Mo	P	S	Cu	
Alloy or Spec. Req. (1)	0.050 0.060	2.00 max	1.00 max	18.0 20.0	8.0 10.5	—	0.045 max	0.03 max	—	
Sample	0.058	1.23	0.31	18.3	8.5	0.48	0.031	<0.001	0.46	

* Revised significant figures of carbon specification and result

(1) *ASM Metals Handbook*, Vol. 1, 10th edition

(2) Customer supplied carbon specifications

ISO 9001

Prepared by: R. Crowder
Senior Chemist

Approved by: D. M. McKay
Supervisor

ATS300, 05/2010

TW METALS
O'Neal High-Performance Metals Group

Packing Slip

ORDER NO.: 03383920 FROM: CINC PAGE 2

CUST NUMBER: 7000221 NET WGT: 80.179 REQ.DATE: 01/24/19 CONFIRMED

SOLD TO:
ALABAMA SPECIALTY PROD
P O BOX 8
MUNFORD AL 36268

SHIP TO:
ALABAMA LASER TECHNOLOGIES
55 LASER BLVD
MUNFORD AL 36268

SALESPERSON: TONY LATHAM
TERMS: 1/2% 10 NET 30
F.O.B.: SHIPPING POINT
CUST ORD NO.: 136092
VIA: COMMON CARRIER
FREIGHT: PREPAID
RELEASE NO.:
RECEIVING PHONE:

SHIP BY: 01/22/19
7640 REINHOLD DR
CINCINNATI OH 45237

"TW Metals MSDS data is available on our web site at www.twmetals.com. MSDS data can be found under the Technical Resources Tab, Product Statistics & Data and the TW Metals MSDS's heading. If you do not have web site access you may telephone 610-458-1300 and we will mail or fax a copy of our current MSDS data to your location."

"CERTIFICATE OF CONFORMANCE"

"TW Metals certifies that the material supplied on this purchase order and contained in the heat/lot number referenced above has been manufactured, inspected, and tested in accordance with the material specification. These records are on file at TW Metals. Packaging material for shipments to Europe and China consists of manufactured wood products and complies with the European emergency measures for coniferous non-manufactured wood packing material"

Authorized Test Report Clerk Susan E McDaniel Date: 1/22/19

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT

MTL ID NO. A2377 PO NO. 136092
ALLOY 304 INITIALS K

1

THANK YOU FOR THIS ORDER

TERMS AND CONDITIONS APPLICABLE TO THE SALE OF THESE PRODUCTS ARE SET FORTH ON OUR WEBSITE FOR YOUR CAREFUL REVIEW.
http://www.twmetals.com/invoice-terms.html

AL 0100-8 41/

NOTICE OF SHIPMENT/
PACKING LIST

ATI
Flat Rolled Products

CERTIFICATE OF TEST

CUST. ORD. NO. & DATE		CUST. CODE	FINISHING LOCATION	SHIPPER NO	PRODUCT CODE	MILL ORDER NUMBER	DATE SHIPPED
11710	06/26/18	715813	VANDERGRIFT, PA	570608	13020102060000	30-068-485	06/27/18

FORMS DISTRIBUTION: MG BILL L157666

REPEAT ORDER: 00776-2 DO RATE: 25 PRI-ORITY

GOV'T CONTRACT

MATL: 1121 SHIPPING LOCATION: VANDERGRIFT PA

INVOICE: 660890

SOLD TO
RELIANCE STEEL-PHOENIX METALS
PO BOX 805
4685 BUFORD HIGHWAY
NORCROSS GA 30071

PRIME SEC.
DSO DSO
10

SHIP TO
PHOENIX METALS-CINCINNATI
1211 HOOK DRIVE
MIDDLETOWN OH 45042

ALABAMA SPECIALTY PRODUCTS INC (ASI
ALABAMA LASER TECHNOLOGIES METAL SAMPLES CO
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. A2377 PO NO. 136092
ALLOY 304 INITIALS 54

GRADE AND SPECIFICATIONS

CARRIER - BEEMAC TRUCKING

ATI 304" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE (AMS 5513J) (ASME-SA-480 ED 2017)
ASTM-A-480-17) (NACE MR0175/ISO 15156-3:2015) (ASME-SA-479 ED 2017 CHEM ONLY) (ASTM-A-312-17 CHEM ONLY)
ASTM-A-479-18 CHEM ONLY) (ASME-SA-666 ED 2017) (ASME-SA-312 ED 2017 CHEM ONLY) (ASTM-A-666-15) (NACE
R0103/ISO 17495-1:2016) (ASTM-A-276-17 CHEM ONLY) (ASME-SA-276 ED 2017 CHEM ONLY) (ASME-SA-240 ED 2017) (EN
0028-7:2007-1.4301-CR) (ASTM-A-240-17) (UNS S30400)

TEM	PCS	DIMENSIONS W/G/L	HEAT #	COIL #	TEST #	GROSS	TARE	NET	THEO	TAG #/ CD SKID #
01B	1	48./.0575/1562.	855083	AAC29361-B	8858628	15105		15105		574422
	1	48./.0575/1552.	855083	AAC29362-A	8858629	15075		15075		574424
	1	48./.0575/1553.	855083	AAC29362-B	8858629	15105		15105		574426
C	CUST IDENTITY 3425-ET									
	3 COILS					45285		45285		

DIST: EMAIL INVOICES TO DONNA CHAPMAN AT INVOICES@PHOENIXMETALS.NET EMAIL ALL ORDER ACKNOWLEDGEMENTS AND SHIPPING NOTICES TO JPERRY@PHOENIXMETALS.NET, VREED@PHOENIXMETALS.NET AND MMEISTICKLE@PHOENIXMETALS.NET

TYPE HEAT/TEST (WT %)	C	MN	P	S	SI	CR	NI	MO	CU	N
HEAT 855083	.06	1.18	.031	.0002	.35	18.35	8.25	.48	.48	.07
TEST LOCATION	BN	BN	BN	BN	BN	BN	BN	BN	BN	BN

TEM TEST NO	YIELD PSI *	TENSILE PSI	% ELONG IN 2"	% R/A	HARDNESS	BEND	CORROSION A 262 PR E	ASTM GRAIN SIZE	HARDENABILITY
01B 8858628				NR	86.HRBW	T PASS	PASS	NR	NR
	T 53600.	101100	48. #		88.HRBW				
TEST LOCATION	TC	TC	TC		TC	TC	TC		
8858629				NR	87.HRBW	T PASS	PASS	NR	NR
	T 49000.	96900.	52. #		86.HRBW				

PAGE 01 - CONTINUED ON PAGE 02 06/27/18 11:00:03

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The above is a true copy of data on file. The material and test results conform to the sales contract and specification(s) as set forth in ATI Flat Rolled Products' Order Acknowledgement.

Noreen Deferrari
Sr. Manager Quality, Corporate Quality Assurance

NOTICE OF SHIPMENT/
PACKING LIST

ATI
Flat Rolled Products

CERTIFICATE OF TEST

AL 6168-8 417.

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11710	06/26/18	715813	VANDERGRIFT, PA	570608	13020102060000	30-068-485	06/27/18

FORMS DISTRIBUTION	MG BILL	REPEAT ORDER	DO RATE	PRI-ORITY	GOV'T CONTRACT	MAT'L	SHIPPING LOCATION		INVOICE
	L157666	00776-2	25			1121	VANDERGRIFT	PA	660890

SOLD TO
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NORCROSS GA 30071

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PHOENIX METALS-CINCINNATI
1211 HOOK DRIVE
MIDDLETOWN OH 45042

GRADE
ATI 304" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE

CARRIER - BEEMAC TRUCKING

* Y.S. BY 0.2% OFFSET METHOD

	MELT SOURCE	T-YIELD 1%
TEM TEST NO 01B 8858628	1.	58255.
EST LOCATION		TC
TEM TEST NO 01B 8858629	1.	54179.
EST LOCATION		TC

\# ELONG AT FRACTURE

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. A2377 PO NO. 136092
ALLOY 304 INITIALS LA

NR = DATA NOT REQUIRED
RADE VERIFICATION WAS CARRIED OUT SPECTROSCOPICALLY

ATERIAL IS OF USA MELT AND MANUFACTURE.

HESE COMMODITIES OR TECHNOLOGY, WHEN EXPORTED FROM THE UNITED STATES, ARE IN ACCORDANCE WITH THE USA XPORT ADMINISTRATION REGULATIONS. DIVERSION CONTRARY TO U.S. LAW IS PROHIBITED. U.S. LAW ALSO PROHIBITS ISPOSITION OF THESE COMMODITIES/TECHNICAL DATA TO ANY END-USER OR FOR ANY END-USE RELATED TO THE DESIGN, EVELOPMENT, PRODUCTION, STOCKPILING, OR USE OF CHEMICAL, BIOLOGICAL, OR NUCLEAR WEAPONS, OR MISSILES, ITHOUT THE PROPER APPROVAL OF THE UNITED STATES GOVERNMENT.

ATERIAL WAS SOLUTION ANNEALED AT 1900F (1038C) MINIMUM FOR A TIME COMMENSURATE WITH THICKNESS AND APIDLY COOLED WITH AIR AND WATER.

TW METALS
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PAGE 02 - CONTINUED ON PAGE 03 06/27/18 11:00:03

CONSIGNEE—Please Note—This consignment was turned over to carrier in first class condition, being correctly loaded, at which time our responsibility for loss or damage in shipment ceased. For your protection please examine shipment as it arrives. If any shortage or damage is discovered, have a full description made by transportation agent or waybill before signing.

WARNING
Safety Data Sheets for this product are available on our website, www.ATImetals.com. A thorough review of the Safety Data Sheet for the grade of material purchased shall be performed prior to working with this product and all safety & health considerations outlined must be implemented.

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Noreen Deferrari -
Sr. Manager Quality, Corporate Quality Assurance

AL 6168-8 417

NOTICE OF SHIPMENT/
PACKING LIST

ATI
Flat Rolled Products

CERTIFICATE OF TEST

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11710	06/26/18	715813	VANDERGRIFT, PA	570608	13020102060000	30-068-485	06/27/18

FORMS DISTRIBUTION: SHIP TO / SPEC

MG BILL L157666

REPEAT ORDER 00776-2 | DO RATE 25 | PRIORITY

GOVT CONTRACT

MATL 1121 | SHIPPING LOCATION VANDERGRIFT PA | INVOICE 660890

PRIME SEC.
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10

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RELIANCE STEEL-PHOENIX METALS
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4685 BUFORD HIGHWAY
NORCROSS GA 30071

SHIP TO
PHOENIX METALS-CINCINNATI
1211 HOOK DRIVE
MIDDLETOWN OH 45042

CARRIER - BEEMAC TRUCKING

GRADE
ATI 304" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE

TI FLAT ROLLED PRODUCTS DOES NOT USE MERCURY IN THE TESTING OR PRODUCTION OF ITS PRODUCTS.

O WELDS/WELD REPAIRS PERFORMED.

N 10204:2005 - ATI FLAT ROLLED PRODUCTS IS APPROVED AS A MANUFACTURER ACCORDING TO THE RESSURE EQUIPMENT DIRECTIVE PED 2014/68/EU.

ATERIAL IS COMPLIANT WITH THE EUROPEAN UNION DIRECTIVES 2011/65/EU (ROHS2) AND 2015/863/EU (ROHS3) - ESTRICTION OF HAZARDOUS SUBSTANCES.

ATERIAL MELTED AND ROLLED IN THE UNITED STATES AND COMPLIES WITH DFARS JANUARY 2010 EDITION, ECTION 252.225-7008 AND 252.225-7009, FOR SPECIALTY METALS (JUL 2009).

TI FLAT ROLLED PRODUCTS HAS AN ACTIVE RADIOACTIVE TESTING PROGRAM WHICH MONITORS INCOMING AW MATERIALS AS WELL AS MELT SAMPLES FOR EVIDENCE OF RADIOACTIVITY. RAW MATERIAL OR MELT SHOWING ADIATION LEVELS ABOVE NORMAL BACKGROUND ARE NOT PERMITTED TO BE USED.

HIS CERTIFIED MATERIAL TEST REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL. FEDERAL LAW PROHIBITS THE ECORDING OF FALSE, FICTITIOUS, OR FRAUDULENT STATEMENTS OR ENTRIES ON THE CERTIFICATE AND MAY BE UNISHABLE AS A FELONY UNDER FEDERAL LAW. ATI FRP HOLDS SEVERAL QUALITY CERTIFICATIONS THAT INCLUDE SO-9001, AS9100, NADCAP, AND ISO/IEC 17025. MATERIAL WAS MANUFACTURED IN ACCORDANCE WITH THE ATI FRP UALITY MANUAL REVISION 27 DATED 02/01/2018. FOR THE TESTING SPECIFICATION USED IN THE ANALYSIS OF THE ATERIAL, PLEASE SEE THE SCOPE OF APPROVAL FOR NADCAP AND ISO/IEC 17025 FOR THE LABORATORIES. THE SCOPE F THE APPROVALS CAN BE FOUND AT WWW.ATIMETALS.COM.

UALITY SYSTEM CERTIFIED ACCORDING TO PRESSURE EQUIPMENT DIRECTIVE 2014/68/EU, ANNEX I, 4.3 BY TUV SUD, NDUSTRIE SERVICE GMBH, (NOTIFIED BODY 0036).

PAGE 03 - CONTINUED ON PAGE 04

06/27/18 11:00:03

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES
METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. A2377 PO NO. 136092 INITIALS
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CONSIGNEE - Please Note - This consignment was turned over to carrier in first class condition, being correctly loaded, at which time our responsibility for loss or damage in shipment ceased. For your protection please examine shipment as it arrives. If any shortage or damage is discovered, have a full description made by transportation agent on waybill before signing.

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Koreen Deferrari
Sr. Manager Quality, Corporate Quality Assurance

AL 6168-B 417

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1211 HOOK DRIVE
MIDDLETOWN OH 45042

GRADE
ATI 304" STAINLESS STEEL SHEET C R COILS ANNEALED 2B FIN 3 EDGE

CARRIER - BEEMAC TRUCKING

TI FLAT ROLLED PRODUCTS PERFORMS CHEMICAL ANALYSIS BY THE FOLLOWING TECHNIQUES
OR TESTING LOCATIONS TC, BN, & LO:
, S BY COMBUSTION/INFRARED
, O, H BY INERT FUSION/THERMAL CONDUCTIVITY
N, P, SI, CR, NI, MO, CU, CB, CO, V BY WDXRF
BY OES
L AND TI (>=0.10%) BY WDXRF, OTHERWISE BY OES
B, BI, AG BY GFAA

ALABAMA SPECIALTY PRODUCTS INC (ASPI)
ALABAMA LASER TECHNOLOGIES METAL SAMPLES COMPANY
ALABAMA RESEARCH AND DEVELOPMENT
MTL ID NO. A7877 PO NO. 136092
ALLOY 304 INITIALS 4

HE NUMERIC CODES SHOWN UNDER MELT SOURCE CAN BE INTERPRETED AS FOLLOWS:
1. - MATERIAL MELTED, ROLLED AND TESTED IN THE UNITED STATES.
2. - FOREIGN MELT; ROLLED AND TESTED IN THE UNITED STATES.

TI FLAT ROLLED PRODUCTS HAS NO CONTROL OVER CUSTOMER PROCESSING OF MATERIAL; INCLUDING,
UT NOT LIMITED TO, BUFFING OR ELECTROPOLISHING MATERIAL WITH A 2B OR OTHER FINISH, AND DOES NOT REPRESENT
R GUARANTY THAT THE MATERIAL IS SUITABLE FOR ANY PARTICULAR PROCESS OR USE OF CUSTOMER.

N 10204:2005 3.1 CERTIFICATE.

ESTING WAS PERFORMED AT THE FOLLOWING LOCATIONS:
N = ATI-FLAT ROLLED PRODUCTS; 100 RIVER ROAD; BRACKENRIDGE, PA 15014
C = ATI-FLAT ROLLED PRODUCTS; 1300 PACIFIC AVENUE; NATRONA HEIGHTS, PA 15065

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<<<<<<<< FOR ACCESS TO ONLINE CERTIFICATES OF TEST >>>>>>>>
<<<<<<<< REGISTER AT MYATI.ATIMETALS.COM >>>>>>>>

PAGE 04 - FINAL PAGE. 06/27/18 11:00:03

CONSIGNEE—Please Note—This consignment was turned over to carrier in first class condition, being correctly loaded, at which time our responsibility for loss or damage in shipment ceased. For your protection please examine shipment as it arrives. If any shortage or damage is discovered, have a full description made by transportation agent on waybill before signing.

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The above is a true copy of data on file. The material and test results conform to the sales contract and specification(s) as set forth in ATI Flat Rolled Products' Order Acknowledgement.
Noreen Deferrari - St. Manager Quality, Corporate Quality Assurance

MORTON SALT
Morton Salt, Inc.

Certificate of Analysis

F112840000G 50Lb Culinox 999 Paper

MINNEAPOLIS FLOUR CO
6000 PARK AVE
CLEVELAND OH 44105-4947
USA

03-SEP-2020
Manufacturing Site
RITTMAN
151 Industrial Ave, Rittman
OH, 44270-1593
Brandon Haney
Quality Control

Page 1/1

Manufacturer:	Morton Salt, Inc.	Morton Batch No.:	RI20213006
Morton Order No.:	5102023086	Manufact. Date:	31-JUL-2020
Cust. Order No.:	08192020		
Delivery /-Item No:	5204750743 / 900005		
Quantity:	49 BAG	Shipping date:	03-SEP-2020

General information:
This product meets the tolerances for Food Grade Salt as published in the Food Chemical Codex latest edition. It has been manufactured in compliance with all applicable parts of the Good Manufacturing Practice Regulations for foods as set forth in 21 CFR Part 117 and Canadian Food and Drugs Act and Regulations. Product does not contain any of the eleven major food allergens, glutens, or sulfite >10ppm. Product does not contain genetically modified organisms and is not of animal origin. Salt is chemically stable and does not deteriorate over time. This product does not contain any additives.

Parameter	**Result**
Sodium Chloride	99.99 %
Sulfate	0.007 %
Calcium & Magnesium as Calcium	20 ppm
Moisture - Surface	0.001 %
Insoluble Matter (ppm)	21 ppm
Iron - Free	0.0 ppm
Copper	0.00 ppm
Arsenic	<1.0 ppm
Heavy Metals as Lead	<2.0 ppm
Bulk Density (lb/ft3)	76.0 lb/ft3
USS #20 (850µm) Retained	0 %
USS #30 (600µm) Retained	4 %
USS #40 (425µm) Retained	32 %
USS #50 (300µm) Retained	41 %
USS #70 (212µm) Retained	20 %
USS #100 (150µm) Retained	4 %
USS PAN	0 %
Cumulative Passing USS 70	4 %

Shipping Plant: 151 Industrial Ave , Rittman, OH, 44270-1593

Electronically released by Brandon Haney Quality Assurance Technician on 01-AUG-2020

MORTON SALT, INC.

Safety Data Sheet

Section 1: Identification of the Substance/Mixture and of the Company/Undertaking

1.1 Product identifier

Product Name
- **Common Salt without Additives**

Synonyms
- All Purpose Natural Sea Salt
All Purpose Purex Salt
Bulk Culinox 999 NC
Bulk Extra Coarse Solar Salt Undried NC
Bulk KD Industrial Salt NC
Bulk Purex Salt NC
Bulk Rock Salt NOC 17F NC
Bulk Rock WC Extra Coarse Southern NC
Bulk Rock White Crystal Coarse Southern NC
Bulk Solar Coarse Salt Undried NC
Bulk Solar Industrial Crude Salt NC
Bulk Solar WC Extra Coarse Salt NC
Bulk Solar White Crystal Coarse Salt NC
Bulk Solar White Crystal Medium Salt NC
Bunny Spool (Plain Salt)
California Pure Coarse Sea Salt
California Pure Fine Sea Salt
California Pure Medium Sea Salt
Canning & Pickling Salt
Coarse Sea Salt (F114100000x)
Commercial Grade, Water Softening Pellets
Culinox 999 Chemical Grade Salt
Culinox 999 Fine Salt
Culinox 999 Food Grade Salt;
Evaporated Granulated Salt
Evaporated Salt Pellets
Extra Coarse Sea Salt
Extra Fine 50 Sea Salt
Extra Fine 70 Sea Salt
Feed Mixing Salt
Northern Rock, F & R
Fine Mixing Salt
Hi-Purity Super Soft Salt Extra Coarse Crystals
H.G. Blending Salt
Hay & Stock Salt, F&R
Industrial Crude Solar Salt
ISCO Crystals, Bulk
ISCO Medium, Bulk
ISCO Water Conditioning, Bulk
KD Crude Solar Salt
KD Industrial Salt
Kleer Fine Salt
Kleer Granulated Salt
Medium Sea Salt
Mill Run Salt
Natural Coarse Sea Salt
Northern Fine +20 Rock Salt Plain Salt Block
Plain Salt Brick
Pool Salt
Premium Salt Pellets
Professional's Choice Pool Salt
Pure and Natural Water Softener Crystals
PureSun Culinary Crystals
PureSun Culinary Crystals Coarse
PureSun TFC Culinary Crystals
Purex Salt
Purex Select Salt
Reagent Grade Sodium Chloride
Refined Sea Salt
Rock Pretzel Salt
Rock Salt for Making Ice Cream
Safe-T-Salt (bagged w/o YPS)
Screened Bulk Solar Undried Salt NC
Sea Salt, 50 lb. (F113100000x)
Sea Salt, Tote (F113500000x)
Sea Salt Grinder
Sea Salt Grinder Refill
Select Extra Coarse Rock Salt
Select Sea Salt
Service Pack Salt (all)
Ship n' Shore Rock Salt
Solar Salt Water Softening Crystals
Stock Salt
USP Sodium Chloride
Valu-Soft Solar Salt
Water Softening Salt (Undried) Coarse
Water Softening Salt (Undried) Extra Coarse
White Crystal Brine Block (50 lb.)
White Crystal Rock Salt (all)
White Crystal Solar Salt (all)
White Crystal Water Softening Solar Salt (all)

CAS Number
- 7647-14-5

OSHA HCS 2012

Hazard statements • No label element(s) specifically required

2.3 Other hazards

OSHA HCS 2012 • This product is not considered hazardous under the U.S. OSHA 29 CFR 1910.1200 Hazard Communication Standard.

Canada

According to WHMIS

2.1 Classification of the substance or mixture

WHMIS • Classification criteria not met

2.2 Label elements

WHMIS • No label element(s) specifically required

2.3 Other hazards

WHMIS • In Canada, the product mentioned above is not considered hazardous under the Workplace Hazardous Materials Information System (WHMIS).

2.4 Other information

NFPA

See Section 12 for Ecological Information.

Section 3 - Composition/Information on Ingredients

3.1 Substances

Non-Hazardous Components					
Chemical Name	**Identifiers**	**%(weight)**	**LD50/LC50**	**Classifications According to Regulation/Directive**	**Comments**
Sodium chloride	**CAS**:7647-14-5 **EC Number**:231-598-3	> 99%	Ingestion/Oral-Rat LD50 • 3000 mg/kg	**EU DSD/DPD:** Not Classified - Criteria not met **EU CLP:** Not Classified - Criteria not met **OSHA HCS 2012:** Not Classified - Criteria not met	May contain small quantities of naturally occurring calcium and magnesium salts

3.2 Mixtures

6.4 Reference to other sections

- Refer to Section 8 - Exposure Controls/Personal Protection and Section 13 - Disposal Considerations.

Section 7 - Handling and Storage

7.1 Precautions for safe handling

Handling
- Use good safety and industrial hygiene practices. Wash thoroughly after handling. Keep out of reach of children.

7.2 Conditions for safe storage, including any incompatibilities

Storage
- Avoid storage with strong acids and strong oxidizing agents.

Incompatible Materials or Ignition Sources
- Strong oxidizing agents, strong acids.

7.3 Specific end use(s)

- Refer to Section 1.2 - Relevant identified uses.

Section 8 - Exposure Controls/Personal Protection

8.1 Control parameters

Exposure Limits/Guidelines
- No applicable exposure limits available for product or components.

8.2 Exposure controls

Engineering Measures/Controls
- Adequate ventilation systems as needed to control concentrations of airborne contaminants below applicable threshold limit values.

Personal Protective Equipment

Pictograms
- 

Respiratory
- In case of insufficient ventilation, wear suitable respiratory equipment.

Eye/Face
- Wear safety glasses.

Skin/Body
- Wear appropriate gloves.

General Industrial Hygiene Considerations
- Do not get in eyes or on skin or clothing. Handle in accordance with good industrial hygiene and safety practice.

Environmental Exposure Controls
- Follow best practice for site management and disposal of waste.

Section 9 - Physical and Chemical Properties

9.1 Information on Physical and Chemical Properties

Material Description			
Physical Form	Solid	Appearance/Description	Colorless to white crystalline or compressed block/pellet.
Color	Colorless to White.	Odor	Odorless

Aspiration Hazard	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Carcinogenicity	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Germ Cell Mutagenicity	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Skin corrosion/Irritation	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Skin sensitization	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
STOT-RE	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
STOT-SE	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Toxicity for Reproduction	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Respiratory sensitization	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met
Serious eye damage/Irritation	**EU/CLP**•Classification criteria not met **OSHA HCS 2012**•Classification criteria not met

Potential Health Effects

Inhalation

Acute (Immediate)
- Under normal conditions of use, no health effects are expected. Inhalation of dust may cause mild irritation to mucous membranes, nose and throat. Symptoms may include coughing, dryness and sore throat.

Chronic (Delayed)
- No data available.

Skin

Acute (Immediate)
- Under normal conditions of use, no health effects are expected.

Chronic (Delayed)
- No data available.

Eye

Acute (Immediate)
- Based upon practical use and experience using this product eye irritation is not expected to occur.

Chronic (Delayed)
- No data available.

Ingestion

Acute (Immediate)
- Ingestion may cause the following symptoms - diarrhea.

Chronic (Delayed)
- No data available.

Key to abbreviations
LD = Lethal Dose

Section 12 - Ecological Information

Component	CAS	Canada DSL	Canada NDSL	China	EU EINECS	EU ELNICS
Sodium chloride	7647-14-5	Yes	No	Yes	Yes	No

Inventory (Con't.)

Component	CAS	Japan ENCS	Korea KECL	TSCA
Sodium chloride	7647-14-5	Yes	Yes	Yes

Canada

Labor

Canada - WHMIS - Classifications of Substances

•Sodium chloride 7647-14-5 > 99% Uncontrolled product according to WHMIS classification criteria

Canada - WHMIS - Ingredient Disclosure List

•Sodium chloride 7647-14-5 > 99% Not Listed

Environment

Canada - CEPA - Priority Substances List

•Sodium chloride 7647-14-5 > 99% Not Listed

Europe

Other

EU - CLP (1272/2008) - Annex VI - Table 3.2 - Classification

•Sodium chloride 7647-14-5 > 99% Not Listed

EU - CLP (1272/2008) - Annex VI - Table 3.2 - Concentration Limits

•Sodium chloride 7647-14-5 > 99% Not Listed

EU - CLP (1272/2008) - Annex VI - Table 3.2 - Labelling

•Sodium chloride 7647-14-5 > 99% Not Listed

EU - CLP (1272/2008) - Annex VI - Table 3.2 - Notes - Substances and Preparations

•Sodium chloride 7647-14-5 > 99% Not Listed

EU - CLP (1272/2008) - Annex VI - Table 3.2 - Safety Phrases

•Sodium chloride 7647-14-5 > 99% Not Listed

Mexico

Other

Mexico - Hazard Classifications

•Sodium chloride 7647-14-5 > 99% Not Listed

Mexico - Regulated Substances

•Sodium chloride 7647-14-5 > 99% Not Listed

United States

Labor

U.S. - OSHA - Process Safety Management - Highly Hazardous Chemicals

•Sodium chloride 7647-14-5 > 99% Not Listed

U.S. - OSHA - Specifically Regulated Chemicals

•Sodium chloride 7647-14-5 > 99% Not Listed

Environment

U.S. - CAA (Clean Air Act) - 1990 Hazardous Air Pollutants

•Sodium chloride 7647-14-5 > 99% Not Listed

U.S. - CERCLA/SARA - Hazardous Substances and their Reportable Quantities

•Sodium chloride 7647-14-5 > 99% Not Listed

U.S. - CERCLA/SARA - Radionuclides and Their Reportable Quantities

•Sodium chloride 7647-14-5 > 99% Not Listed

U.S. - CERCLA/SARA - Section 302 Extremely Hazardous Substances EPCRA RQs

•Sodium chloride 7647-14-5 > 99% Not Listed

U.S. - CERCLA/SARA - Section 302 Extremely Hazardous Substances TPQs

•Sodium chloride 7647-14-5 > 99% Not Listed

U.S. - CERCLA/SARA - Section 313 - Emission Reporting

MORTON SALT

January 22, 2019

To Whom It May Concern:

Re: **ASTM B 117-18**

Morton® Culinox® 999® Food Grade Salt, manufactured by Morton Salt, Inc., meets the purity requirements as specified in ASTM B 117-18, Standard Practice for Operating Salt Spray (Fog) Apparatus. In addition, Culinox® 999® Food Grade Salt does not contain added anti-caking agents.

Best Regards,

Lorrie-Ann Fisher
Technical Services Specialist, OSHA SGE
Morton Salt, Inc.
P: +1 312.807.2247
TECHNICALDOCUMENTS@mortonsalt.com

This statement is valid for three (3) years from the date set forth above.

MORTON SALT

January 2, 2019

To Whom It May Concern:

Subject: **Shelf Life of Sodium Chloride**

Morton Salt considers Sodium Chloride to be stable for an infinite period of time. Sodium Chloride is commonly known as a nonreactive chemical that does not break down. In fact, the salt deposits that are mined for many of our products were laid down millions of years ago. Since our process only dissolves this salt in water, and then the salt is recrystallized, the salt is not chemically changed it in any way. Therefore, the salt we produce is as stable as the original deposit.

Stability studies performed to date on Sodium Chloride have also not shown any change over time. For these reasons, no expiration date is applicable for Sodium Chloride. In addition, because the product is stable, no retest date is applicable.

Best Regards,

Lorrie-Ann Fisher

Lorrie-Ann Fisher
Technical Services Specialist, OSHA SGE
Morton Salt, Inc.
P: +1 312.807.2247
TECHNICALDOCUMENTS@mortonsalt.com

This statement is valid for three (3) years from the date set forth above.

PDS 2009
Date Published: 07/05/2017

PDS Version No: 04

Spec. No: 295-02
Date Spec Reviewed: 06/27/2017

MORTON SALT **PRODUCT DATA SHEET**

Morton® Culinox® 999® Food Grade Salt

Description

- This product is high purity, food grade granulated sodium chloride produced in vacuum pans from chemically purified brine. Brine treatment, crystallizing technique, and post-crystallizing washing substantially reduce calcium, magnesium, iron, copper and other heavy metals, sulfate and carbonate impurities.
- The salt crystals are cubic in structure.
- Sodium sulfate is the major impurity with traces of calcium carbonate and magnesium hydroxide.
- There are no additives.
- This product complies with Food Chemicals Codex tolerances and federal CGMP standards.
- This salt is annually certified as Kosher for Passover.

Chemical Properties

Analyte	u/m	Range	Note
Sodium Chloride	%	>=99.95	1
Sulfate	%	<=0.03	
Calcium & Magnesium as Calcium	PPM	<=60	
Moisture (Surface)	%	<=0.1	
Water Insolubles	PPM	<=100	
Copper	PPM	<=0.3	
Free Iron	PPM	<=0.7	
Arsenic	PPM	<=1.0	
Heavy Metals as Lead	PPM	<=2.0	

- Note 1. By difference of impurities, moisture-free basis (ASTM Methods).

Product Ingredient Declaration

- Salt

Physical Properties

- Range loose (pour) bulk density (g/ml): 1.04 - 1.28
- Range loose (pour) bulk density (lbs/cu.ft.): 65 - 80
- Production may be unscreened; however, it receives a coarse scalping of 12 Mesh.

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Chicago, IL 60606-0090

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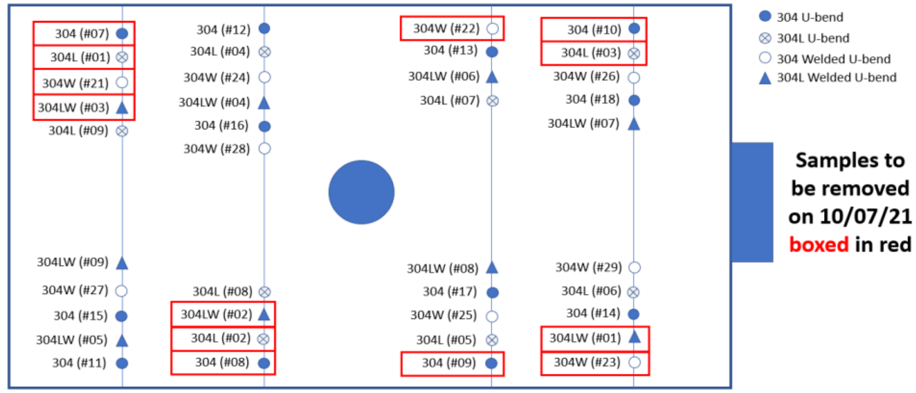
W MortonSalt.com

MORTON SALT, INC.
A K+S GROUP COMPANY

Appendix D: Data Collected During the Experiment

Experiment 1 - 4 weeks
This data for Experiment 1 duriung the first four weeks of testing. The samples removed after 4 weeks are boxed in red on the diagram.



Test Conditions	
Temp. Expoture Zone	35°C ± 2
Relative Humidity	100
Salt Solution	5% (by weight)

Start Date and Time	
Start Date	9/9/2021
Start Time	8:20 PM

Data Collection																	
Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
9/11/2021	2:00 PM	41.70	4:00 PM	4	66	54	1.58	1.27	23.7	22.5	7.41	7.49	1.030 - 1.035	1.035	95.4	95.7	117.0
9/13/2021	6:37 PM	50.60	8:40 PM	5	90	63	1.78	1.22	23.5	23.6	7.17	7.23	1.030 - 1.035	1.030 - 1.035	95.4	95.5	120.0
9/15/2021	7:20 PM	46.70	8:40 PM	4	82	52	1.76	1.11	25.2	24.8	7.15	7.16	1.030 - 1.035	1.030 - 1.035	95.9	96.1	117.9
9/17/2021	7:20 PM	46.67	8:30 PM	4	78	54	1.67	1.16	25.0	24.2	7.10	7.11	1.030 - 1.035	1.030 - 1.035	95.2	95.2	117.9
9/19/2021	3:00 PM	42.50	4:30 PM	4	64	54	1.51	1.27	25.5	25.5	7.00	7.02	1.030 - 1.035	1.030 - 1.035	95.2	95.2	117.5
9/21/2021	3:00 PM	46.50	4:40 PM	4	59	50	1.27	1.08	25.2	25.0	6.96	6.97	1.030 - 1.035	1.030 - 1.035	95.0	95.2	117.3
9/23/2021	6:40 PM	50.00	8:00 PM	5	63	52	1.26	1.04	24.9	24.6	7.05	7.06	1.030 - 1.035	1.030 - 1.035	94.3	94.6	117.2
9/25/2021	5:00 PM	45.00	6:15 PM	5	74	45	1.64	1.00	25.6	24.5	6.90	6.93	1.035	-	95.2	95.5	117.3
9/27/2021	7:15 PM	49.00	8:30 PM	4	60	49	1.22	1.00	25.6	25.0	6.97	6.98	1.035	-	91.1	96.3	118.9
9/29/2021	8:15 PM	47.75	10:15 PM	4	52	45	1.09	0.94	25.4	25.6	7.00	7.01	1.035	1.035	95.4	95.5	118.4
10/1/2021	10:45 AM	26.50	12:45 PM	4	51	47	1.40	1.29	23.9	24.1	6.93	6.95	1.035	1.035	95.4	95.5	119.5
10/3/2021	1:45 PM	49.00	3:00 PM	4	58	56	1.18	1.14	24.3	24.4	6.95	6.95	1.035	1.035	95.5	95.9	119.5
10/5/2021	3:15 PM	48.25	4:45 PM	4	47	46	0.97	0.95	25.2	25.4	6.96	6.98	1.030 - 1.035	1.030 - 1.035	95.7	96.1	118.2
10/7/2021	4:45 PM	48.00	7:55 PM	4	43	39	0.90	0.81	24.8	24.7	7.04	7.07	1.035	1.035	95.9	96.1	119.2

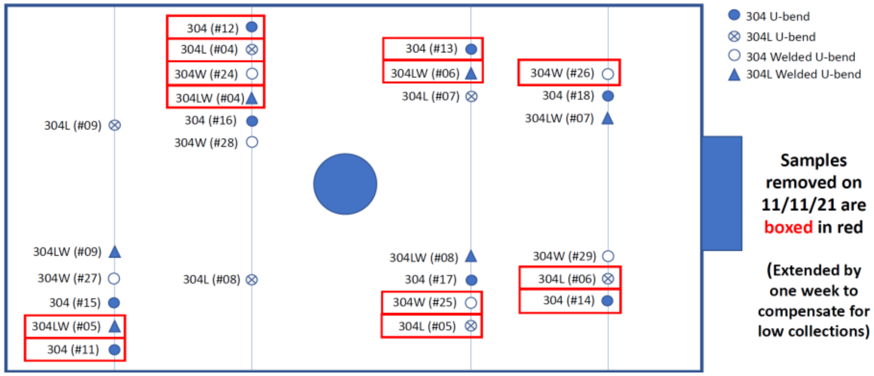
Experiment 1 Sample Weights (35°C) - 4 weeks			
Sample No.	**Weight Before (g)**	**Weight After (g)**	**Difference (g)**
304 (#07)	47.6197	47.6193	0.0004
304 (#08)	48.2223	48.2220	0.0003
304 (#09)	48.0048	48.0051	-0.0003
304 (#10)	47.8839	47.8844	-0.0005
304L (#01)	46.4287	46.4282	0.0005
304L (#02)	46.4399	46.4405	-0.0006
304L (#03)	46.9774	46.9771	0.0003
304W (#21)	47.8918	47.8922	-0.0004
304W (#22)	47.9759	47.9766	-0.0007
304W (#23)	47.9588	47.9587	0.0001
304LW (#01)	47.1503	47.1504	-0.0001
304LW (#02)	47.6073	47.6074	-0.0001
304LW (#03)	47.4557	47.4556	0.0001

Appendix D: Data Collected During the Experiment

Experiment 1 - 8 weeks
This data for Experiment 1 duriung the second four weeks of testing. The samples removed after 8 weeks are boxed in red on the diagram.

Test Conditions	
Temp. Expoture Zone	35°C ± 2
Relative Humidity	100
Salt Solution	5% (by weight)

Start Date and Time	
Start Date	10/7/2021
Start Time	12:25 PM



Data Collection

Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
10/9/2021	12:25 PM	40.50	4:00 PM	5	19	19	0.469	0.469	22.8	22.7	6.96	6.95	1.030 - 1.035	-	94.8	94.8	118.8
10/11/2021	2:15 PM	46.25	3:20 PM	5	13	11	0.281	0.238	24.9	24.8	6.92	6.95	-	-	96.1	96.3	117.5
10/13/2021	7:00 PM	51.67	8:25 PM	4	11	7	0.213	0.135	24.1	23.7	6.95	7.02	-	-	95.2	95.5	119.5
10/15/2021	10:40 AM	38.25	12:10 PM	4	87	59	2.274	1.542	24.1	24.3	7.04	7.04	1.035	1.030 - 1.035	96.1	96.3	119.1
10/17/2021	3:45 PM	51.58	5:15 PM	4	104	70	2.016	1.357	24.8	23.9	7.04	7.06	1.035	1.035	96.1	96.3	119.8
10/19/2021	2:45 PM	45.50	4:15 PM	4	98	60	2.154	1.319	24.7	24.1	6.97	7.00	1.035	1.035	94.6	94.8	118.8
10/21/2021	7:15 PM	51.00	8:45 PM	5		59		1.157		23.6		6.91	-	-	95.9	96.3	118.0
10/23/2021	1:15 PM	40.50	2:45 PM	4	61	52	1.152	1.297	23.6	22.4	6.95	6.94	1.035	-	95.5	95.9	119.3
10/25/2021	4:15 PM	49.50	5:40 PM	4	78	57	1.576	1.152	24.8	24.1	6.94	6.92	1.035	-	96.1	96.4	119.3
10/27/2021	5:00 PM	47.30	6:15 PM	4	71	54	1.500	1.141	24.5	22.4	6.87	6.87	1.035	-	95.7	96.1	118.6
10/29/2021	4:15 PM	46.00	5:20 PM	4	64	50	1.391	1.087	24.7	24.3	6.85	6.88	1.035	-	95.4	95.7	117.3
10/31/2021	12:40 PM	43.33	2:30 PM	3	60	47	1.385	1.084	20.8	20.5	6.87	6.85	1.035	-	94.3	94.6	118.4
11/2/2021	2:30 PM	48.00	4:00 PM	3	58	45	1.208	0.938	20.1	20.7	6.83	6.85	1.035 - 1.040	1.035 - 1.040	94.5	94.8	117.0
11/4/2021	2:45 PM	46.75	4:00 PM	3	91	50	1.947	1.069	23.0	22.3	6.87	6.89	1.035 - 1.040	-	94.5	94.8	118.6
11/6/2021	3:00 PM	47.00	N/A	3	58	45	1.234	0.957	22.8	23.0	6.83	6.80	1.035 - 1.040	1.035 - 1.040	95.4	95.7	117.9
11/9/2021	2:30 PM	43.50	3:40 PM	3	73	61	1.678	1.402	25.3	24.2	6.92	6.90	1.035 - 1.040	1.035 - 1.040	95.5	95.7	116.1
11/11/2021	8:40 PM	53.00	10:30 PM	3	90	74	1.698	1.396	24.5	24.5	7.04	7.05	1.035	1.035	95.0	95.2	118.0

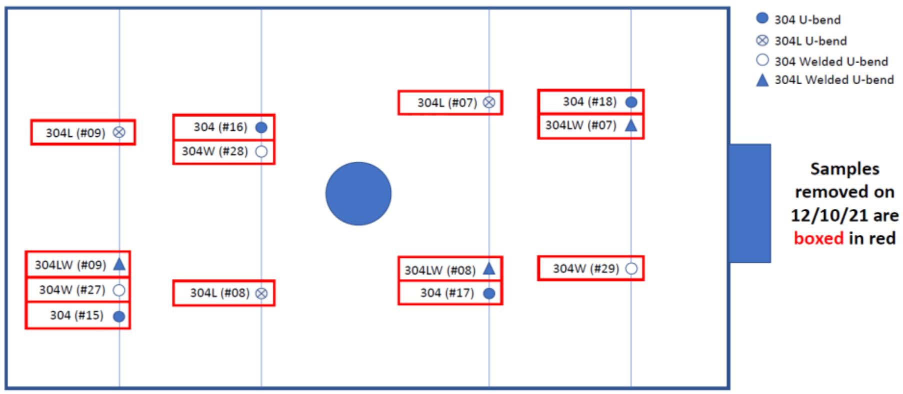
Experiment 1 Sample Weights (35°C) - 8 weeks			
Sample No.	**Weight Before (g)**	**Weight After (g)**	**Difference (g)**
304 (#11)	48.0950	48.1113	-0.0163
304 (#12)	47.8746	47.8907	-0.0161
304 (#13)	47.8337	47.8502	-0.0165
304 (#14)	47.7704	47.7874	-0.0170
304L (#04)	46.7686	46.7852	-0.0166
304L (#05)	47.0133	47.0296	-0.0163
304L (#06)	46.7493	46.7657	-0.0164
304W (#24)	48.0729	48.0914	-0.0185
304W (#25)	47.9981	48.0144	-0.0163
304W (#26)	48.0733	48.0902	-0.0169
304LW (#04)	47.5296	47.5459	-0.0163
304LW (#05)	47.1673	47.1844	-0.0171
304LW (#06)	47.5046	47.5213	-0.0167

Appendix D: Data Collected During the Experiment

Experiment 1 - 12 weeks
This data for Experiment 1 duriung the third four weeks of testing. The samples removed after 12 weeks are boxed in red on the diagram.

Test Conditions	
Temp. Expoture Zone	35°C ± 2
Relative Humidity	100
Salt Solution	5% (by weight)

Start Date and Time	
Start Date	11/11/2021
Start Time	10:30 PM



Data Collection

Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
11/14/2021	10:30 PM	72.00	11:30 PM	4	Overflow	94	Overflow	1.305	24.7	25.0	6.95	6.91	1.030 - 1.035	1.035	95.2	95.4	119.5
11/16/2021	4:40 PM	41.17	6:40 PM	4	93	65	2.259	1.579	24.0	22.7	6.97	6.99	1.035 - 1.040	1.035 - 1.040	95.7	95.9	118.9
11/18/2021	3:10 PM	44.50	4:40 PM	3	51	71	1.146	1.596	24.5	23.9	6.98	7.02	1.035 - 1.040	-	95.9	96.1	119.0
11/20/2021	12:10 PM	43.50	2:00 PM	4	89	67	2.046	1.540	22.9	23.0	6.90	6.92	1.035 - 1.040	1.035 - 1.040	95.0	95.0	119.3
11/22/2021	10:00 AM	44.00	11:30 AM	3	51	69	1.159	1.568	22.7	23.1	6.98	6.95	1.035 - 1.040	-	95.5	95.7	117.0
11/24/2021	8:30 PM	57.00	10:00 PM	4	53	72	0.930	1.263	23.0	23.5	6.85	6.94	1.035 - 1.040	1.035 - 1.040	95.0	95.0	118.0
11/26/2021	1:00 PM	39.00	2:30 PM	3	55	71	1.410	1.821	23.5	23.0	6.95	6.96	1.035 - 1.040	1.035 - 1.040	95.4	95.7	118.4
11/28/2021	5:30 PM	51.00	7:00 PM	3	59	43	1.157	0.831	25.8	24.4	7.03	7.04	1.040	1.040	95.2	95.7	118.8
11/30/2021	2:30 PM	43.50	4:00 PM	4	108	58	2.483	1.333	23.2	22.7	7.00	6.93	1.035 - 1.040	-	94.6	94.6	117.0
12/2/2021	2:30 PM	46.50	3:45 PM	4	80	50	1.720	1.075	24.7	23.9	6.94	6.98	1.040	-	95.7	96.1	119.1
12/4/2021	10:00 AM	42.25	11:15 AM	3	64	40	0.947	1.515	24.5	23.4	6.98	7.00	1.035 - 1.040	1.035 - 1.040	94.8	94.8	118.2
12/6/2021	10:30 AM	47.25	12:00 PM	3	71	59	1.503	1.249	25.0	24.3	7.04	7.06	1.035	1.035	95.9	96.1	118.6
12/8/2021	3:30 PM	52.50	4:50 PM	4	82	66	1.652	1.257	23.7	23.4	6.76	6.79	1.035 - 1.040	1.035	88.0	87.3	120.2
12/10/2021	12:30 AM	43.67	N/A	4	65	73	1.489	1.672	23.2	22.5	6.83	6.82	1.035	1.040	95.5	95.9	118.0

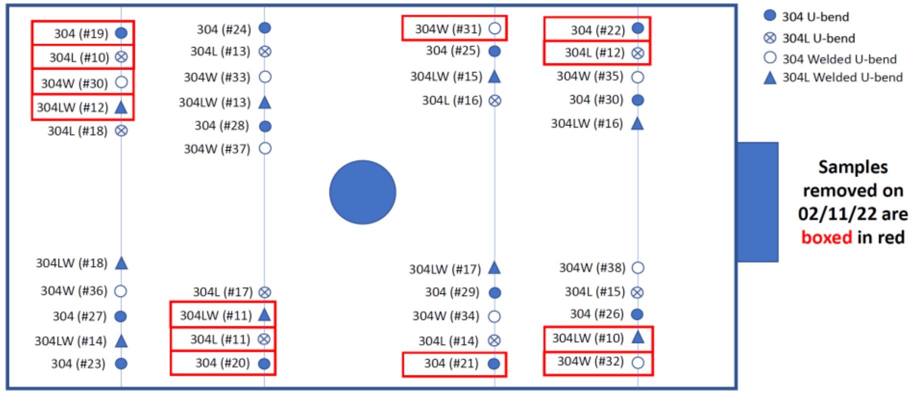
Experiment 1 Sample Weights (35°C) - 12 weeks			
Sample No.	**Weight Before (g)**	**Weight After (g)**	**Difference (g)**
304 (#15)	47.7296	47.7286	0.0010
304 (#16)	48.2841	48.2830	0.0011
304 (#17)	48.0479	48.0469	0.0010
304 (#18)	48.1273	48.1259	0.0014
304L (#07)	46.9758	46.9744	0.0014
304L (#08)	46.6699	46.6685	0.0014
304L (#09)	46.9979	46.9964	0.0015
304W (#27)	48.1337	48.1320	0.0017
304W (#28)	48.0610	48.0500	0.0110
304W (#29)	48.2154	48.2145	0.0009
304LW (#07)	47.4130	47.4113	0.0017
304LW (#08)	47.5719	47.5705	0.0014
304LW (#09)	47.5905	47.5886	0.0019

Appendix D: Data Collected During the Experiment

Experiment 2 - 4 weeks
This data for Experiment 2 duriung the first four weeks of testing. The samples removed after 4 weeks are boxed in red on the diagram.

Test Conditions	
Temp. Expoture Zone	50°C ± 2
Relative Humidity	100
Salt Solution	5% (by weight)

Start Date and Time	
Start Date	1/13/2022
Start Time	7:55 PM



Data Collection																			
Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
1/17/2022	6:30 AM	82.58	8:10 AM	3	Overflow	85	Overflow	1.03	22.2	23.6	7.38	7.35	1.055	1.055	120.4	120.9	117.3
1/19/2022	5:30 PM	57.33	7:20 PM	3	75	54	1.31	0.94	23.0	20.8	7.10	7.09	1.050	-	120.4	121.1	117.9
1/21/2022	6:15 PM	46.92	7:30 PM	4	-	-	-	-	-	-	-	-	-	-	118.80	119.30	119.3
1/23/2022	6:15 PM	46.75	7:40 PM	3	60	55	1.28	1.18	24.7	24.2	7.03	7.01	1.060	-	120.2	120.7	119.1
1/25/2022	2:20 PM	42.67	3:45 PM	3	55	52	1.29	1.22	25.8	25.2	7.04	7.02	1.060	1.060	119.5	120.2	117.1
1/27/2022	2:30 PM	46.75	3:45 PM	3	58	54	1.24	1.16	23.6	24.5	6.95	6.93	1.060	1.060	120.0	120.6	118.0
1/29/2022	3:30 PM	47.75	4:45 PM	3	59	54	1.24	1.13	22.9	24.8	7.02	6.91	1.055	1.055	120.6	120.7	118.8
2/1/2022	6:45 AM	62.00	8:20 AM	4	103	68	1.66	1.10	24.7	21.8	6.96	6.98	1.060	1.055 - 1.060	119.3	119.8	117.3
2/3/2022	7:20 PM	59.00	10:00 PM	3	76	70	1.29	1.19	24.4	24.8	6.89	6.92	1.055	1.055	120.0	120.7	117.7
2/5/2022	4:45 PM	42.75	5:35 PM	3	53	50	1.24	1.17	21.3	22.0	7.00	7.00	1.055 - 1.060	1.055 - 1.060	119.3	119.8	117.9
2/7/2022	5:30 PM	47.92	6:35 PM	4	59	53	1.23	1.11	25.5	24.9	6.91	6.88	1.055	1.055	119.5	120.0	117.9
2/9/2022	1:40 PM	43.08	2:40 PM	4	54	50	1.25	1.16	25.2	25.6	6.80	6.82	1.055	1.055	119.7	120.2	118.2
2/11/2022	12:40 PM	46.00	2:30 PM	4	60	58	1.30	1.26	23.0	23.2	6.91	6.92	1.050	1.050	119.7	120.2	119.3

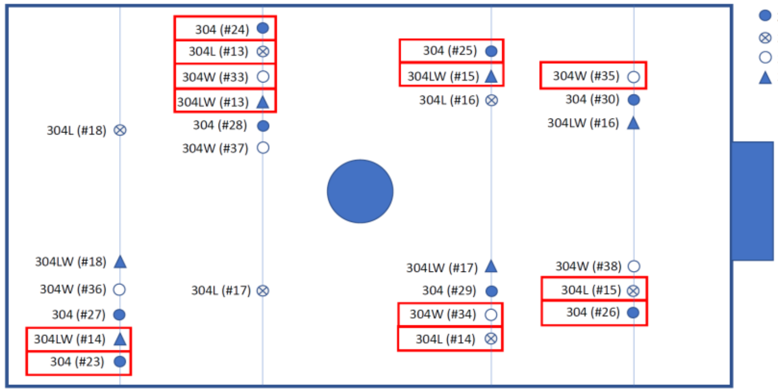
Experiment 2 Sample Weights (50°C) - 4 weeks			
Sample No.	**Weight Before (g)**	**Weight After (g)**	**Difference (g)**
304 (#19)	48.2553	48.2565	-0.0012
304 (#20)	48.2111	48.2120	-0.0009
304 (#21)	47.9018	47.9025	-0.0007
304 (#22)	48.3450	48.3458	-0.0008
304L (#10)	46.6813	46.6818	-0.0005
304L (#11)	47.0194	47.0189	0.0005
304L (#12)	47.0960	47.0965	-0.0005
304W (#30)	48.2467	48.2509	-0.0042
304W (#31)	48.0488	48.0532	-0.0044
304W (#32)	47.9837	47.9856	-0.0019
304LW (#10)	46.9394	46.9406	-0.0012
304LW (#11)	47.0771	47.0783	-0.0012
304LW (#12)	47.2954	47.2993	-0.0039

Appendix D: Data Collected During the Experiment

Experiment 2 - 8 weeks
This data for Experiment 2 duriung the second four weeks of testing. The samples removed after 8 weeks are boxed in red on the diagram.

Test Conditions	
Temp. Expoture Zone	50°C ± 2
Relative Humidity	100
Salt Solution	5% (by weight)

Start Date and Time	
Start Date	2/11/2022
Start Time	2:30 PM



304 U-bend
304L U-bend
304 Welded U-bend
304L Welded U-bend

Samples removed on 03/14/22 are boxed in red

(2 days pause, chamber was not running)

Data Collection																	
Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
2/14/2022	7:30 AM	65.00	9:20 AM	4	83	75	1.277	1.154	20.4	20.0	7.02	7.03	1.050	1.050	120.0	120.4	119.3
2/16/2022	6:00 PM	56.67	7:15 PM	4	72	67	1.271	1.182	25.7	25.3	6.95	6.90	1.055	1.055	120.0	120.6	118.2
2/19/2022	8:00 AM	60.75	9:30 AM	4	79	71	1.300	1.169	21.1	20.5	7.10	7.09	1.050	1.050	120.4	120.4	118.0
2/21/2022	2:00 PM	52.50	4:00 PM	4	67	62	1.273	1.181	25.5	25.0	6.95	6.93	1.050 - 1.055	1.050 - 1.055	119.7	120.4	118.0
2/24/2022	6:30 AM	62.50	8:20 AM	3	85	79	1.360	1.264	25.9	25.9	7.00	7.03	1.045 - 1.050	1.045 - 1.050	119.5	120.2	119.3
2/26/2022	3:45 PM	55.42	5:20 PM	4	73	66	1.317	1.191	25.6	25.8	6.99	7.02	1.045 - 1.050	1.045 - 1.050	120.7	121.1	116.6
3/1/2022	3:00 PM	69.67	4:50 PM	4	93	80	1.335	1.148	26.0	25.5	7.11	7.10	1.050	1.050 - 1.055	120.7	121.3	119.3
3/4/2022	6:33 PM	73.72	8:00 PM	4	102	87	1.384	1.180	22.0	22.1	7.20	7.20	1.050	1.050 - 1.055	120.6	120.9	118.4
3/7/2022	4:43 PM	68.72	6:14 PM	-	99	97	1.441	1.412	20.1	20.2	7.45	7.43	1.050	1.050	120.0	120.4	118.0
3/9/2022	6:56 PM	48.70	8:03 PM	-	98	81	2.012	1.663	23.9	23.7	7.03	7.07	1.050	1.050	120.7	121.1	117.0
3/12/2022	4:30 PM	68.45	5:32 PM	4	Overflow	83	Overflow	1.213	20.1	21.0	6.86	6.84	1.050	1.050	120.2	120.4	117.9
3/14/2022	8:00 PM	50.47	10:00 PM	-	121	85	2.398	1.684	15.2	14.9	6.79	6.79	1.035 - 1.040	1.035 - 1.040	-	-	-

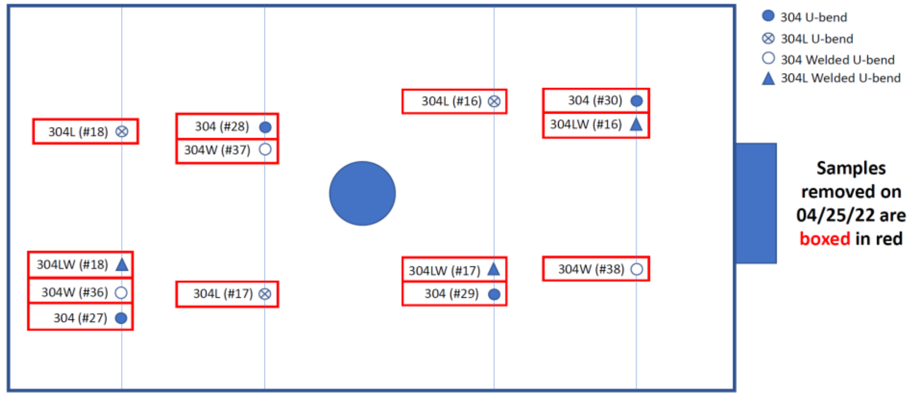
Experiment 2 Sample Weights (50°C) - 8 weeks			
Sample No.	**Weight Before (g)**	**Weight After (g)**	**Difference (g)**
304 (#23)	48.1388	48.1414	-0.0026
304 (#24)	48.2476	48.2474	0.0002
304 (#25)	48.1761	48.1770	-0.0009
304 (#26)	47.7020	47.7014	0.0006
304L (#13)	46.8407	46.8415	-0.0008
304L (#14)	46.5307	46.5292	0.0015
304L (#15)	46.9670	46.9694	-0.0024
304W (#33)	48.1323	48.1341	-0.0018
304W (#34)	47.9676	47.9709	-0.0033
304W (#35)	48.1638	48.1653	-0.0015
304LW (#13)	47.2800	47.2807	-0.0007
304LW (#14)	47.6118	47.6130	-0.0012
304LW (#15)	47.5823	47.5828	-0.0005

Appendix D: Data Collected During the Experiment

Experiment 2 - 14 weeks
This data for Experiment 2 duriung the third four weeks of testing. The samples removed after 14 weeks are boxed in red on the diagram (extended by 2 weeks).

Test Conditions	
Temp. Expoture Zone	50°C ± 2
Relative Humidity	100
Salt Solution	5% (by weight)

Start Date and Time	
Start Date	3/14/2022
Start Time	10:00 PM

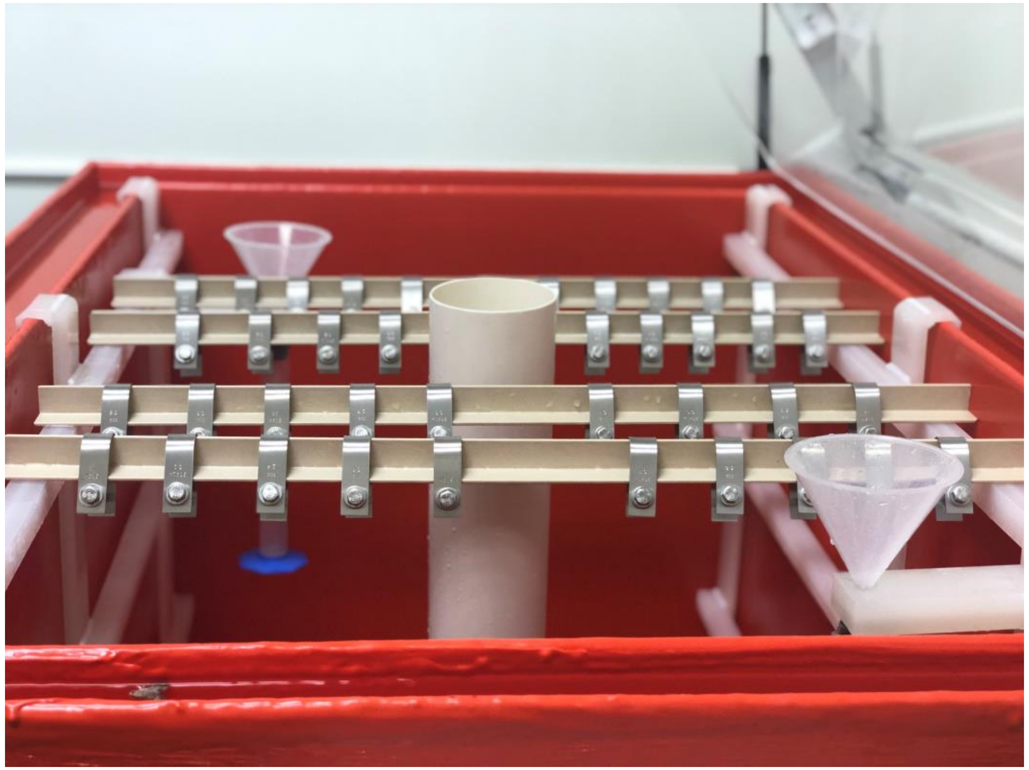


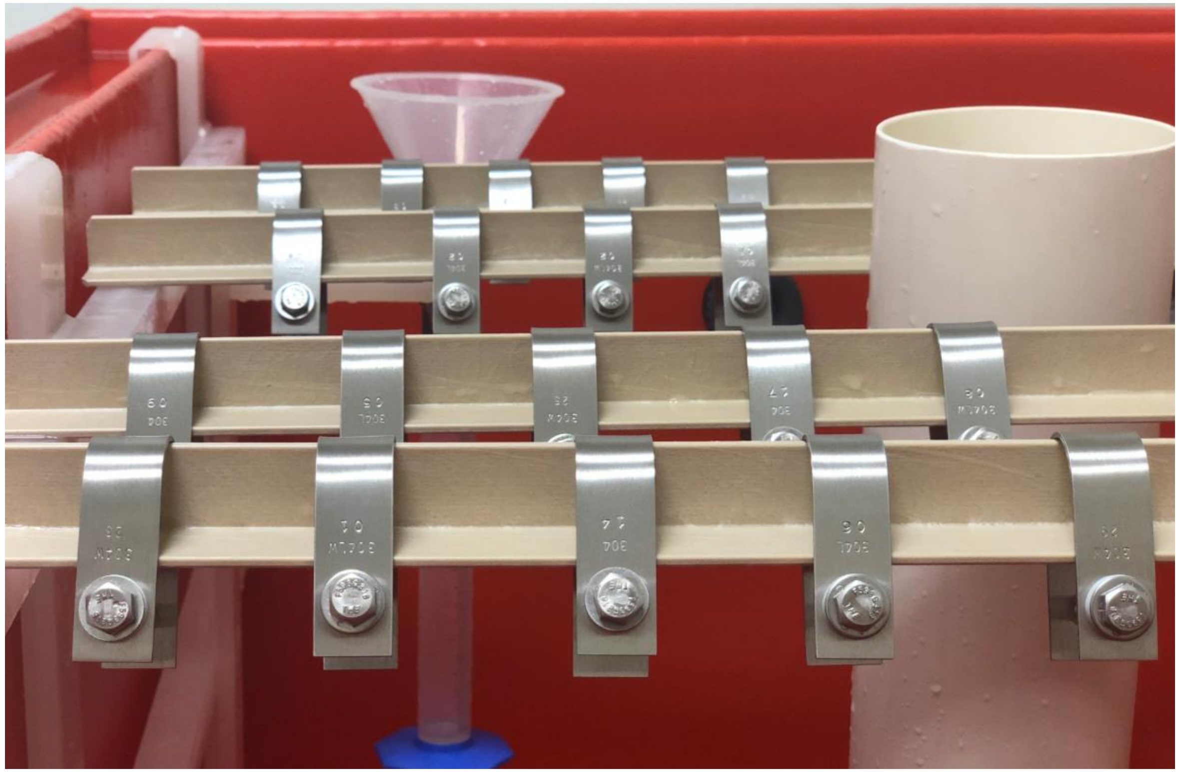
Data Collection																	
Date	Stop Time	Time Elapsed (hrs.)	Experiment Restart Time	TDS Reading	Collection (mL)		Collection Rate (mL/hr.)		Temperature (C)		pH		Specific Gravity		Chamber Temperature (F)		Humidifying Tower Temperature (F)
					Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Dry Bulb	Wet Bulb	
3/17/2022	7:30 AM	57.50	10:15 AM	3	107	73	1.861	1.270	24.3	23.8	6.74	6.71	1.045 - 1.050	1.050	119.5	120.0	117.9
3/19/2022	7:30 PM	57.00	7:42 PM	3	-	-	-	-	-	-	-	-	-	-	120.2	120.0	116.6
3/22/2022	7:26 AM	59.73	9:50 AM	3	Overflow	76	Overflow	1.272	23.9	24.3	6.80	6.80	1.050	1.050 - 1.055	119.8	120.2	117.3
3/24/2022	3:00 PM	53.17	4:30 PM	3	64	58	1.204	1.091	25.9	24.9	6.79	6.77	1.055	1.055 - 1.060	120.6	121.1	119.3
3/26/2022	1:15 PM	44.75	2:55 PM	4	49	51	1.095	1.140	23.8	22.7	6.66	6.66	1.060	1.060	119.8	120.2	116.6
3/29/2022	7:25 AM	64.50	9:00 AM	4	72	58	1.116	0.899	21.0	22.4	6.73	6.76	1.060 - 1.065	1.065	118.9	119.7	119.5
3/31/2022	3:15 PM	54.25	4:33 PM	3	74	58	1.364	1.069	25.1	25.5	6.87	6.82	1.060 - 1.065	1.060 - 1.065	120.2	120.9	118.4
4/3/2022	12:35 PM	67.87	2:20 PM	3	96	73	1.142	1.076	24.4	23.4	6.90	6.87	1.055 - 1.060	1.060	119.1	120.0	119.1
4/5/2022	1:20 PM	47.00	2:45 PM	3	64	49	1.362	1.043	25.6	25.1	6.84	6.80	1.060	1.060	120.7	121.5	119.5
4/7/2022	5:00 PM	50.25	6:40 PM	2	66	54	1.313	1.075	25.4	25.7	6.76	6.85	1.055	-	120.6	121.3	119.7
4/10/2022	2:40 PM	68.00	6:00 PM	3	Overflow	69	Overflow	1.015	21.5	20.8	6.89	6.82	1.060	1.060 - 1.065	120.4	121.1	118.8
4/12/2022	4:25 PM	46.42	6:30 PM	3	64	48	1.379	1.034	25.9	24.9	6.90	6.86	1.065	1.065	119.3	119.8	118.6
4/15/2022	5:30 PM	71.00	7:20 PM	3	116	92	1.634	1.295	25.6	25.4	7.00	6.99	1.050	1.050	120.0	120.0	120.0
4/18/2022	8:45 AM	61.42	11:00 AM	3	104	77	1.693	1.235	21.3	20.8	7.17	7.25	1.050	1.050 - 1.055	120.4	120.9	117.3
4/20/2022	11:30 AM	48.50	1:15 PM	3	80	65	1.649	1.340	22.7	22.0	7.10	7.07	1.055	1.055	119.8	120.6	118.8
4/22/2022	5:45 AM	40.50	6:35 AM	3	69	52	1.704	1.284	25.3	24.0	6.88	6.81	1.050 - 1.055	1.050 - 1.055	119.3	120.0	117.7
4/25/2022	12:30 PM	77.92	NA	3	Overflow	103	Overflow	1.322	23.8	24.3	7.03	7.04	1.050	1.050	120.7	121.5	118.4

Experiment 2 Sample Weights (50°C) - 14 weeks			
Sample No.	**Weight Before (g)**	**Weight After (g)**	**Difference (g)**
304 (#27)	47.7134	47.7129	0.0005
304 (#28)	48.0947	48.0943	0.0004
304 (#29)	47.8902	47.8889	0.0013
304 (#30)	47.8693	47.8693	0.0000
304L (#16)	46.8651	46.8646	0.0005
304L (#17)	46.9667	46.9663	0.0004
304L (#18)	46.9275	46.9244	0.0031
304W (#36)	48.0261	48.0242	0.0019
304W (#37)	47.9273	47.9268	0.0005
304W (#38)	48.0511	48.0497	0.0014
304LW (#16)	47.4364	47.4355	0.0009
304LW (#17)	47.4241	47.4256	-0.0015
304LW (#18)	46.9375	46.9389	-0.0014

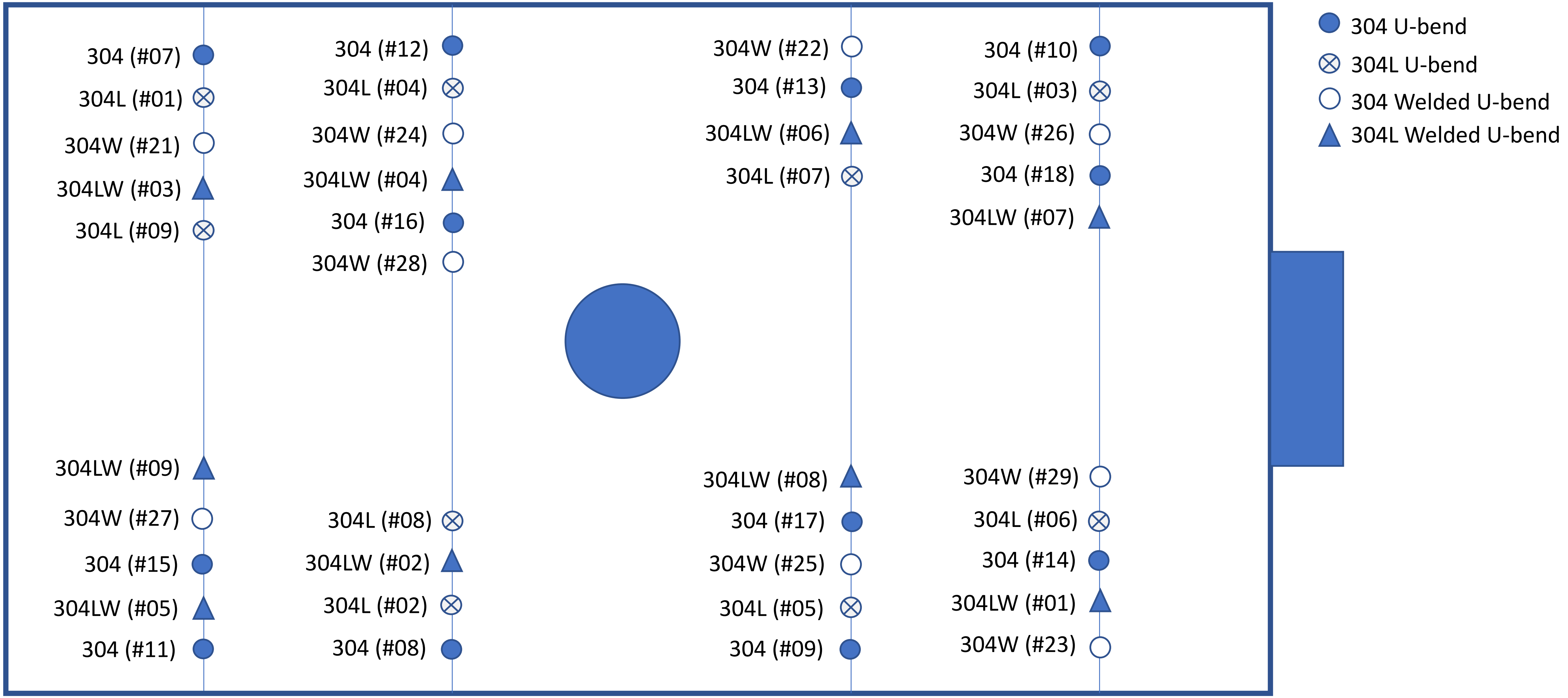
Experiment 1 Diagrams (35°C)

- Experiment 1 was started on 09/09/2021 at 35°C with 39 samples and was concluded on 12/10/2021.

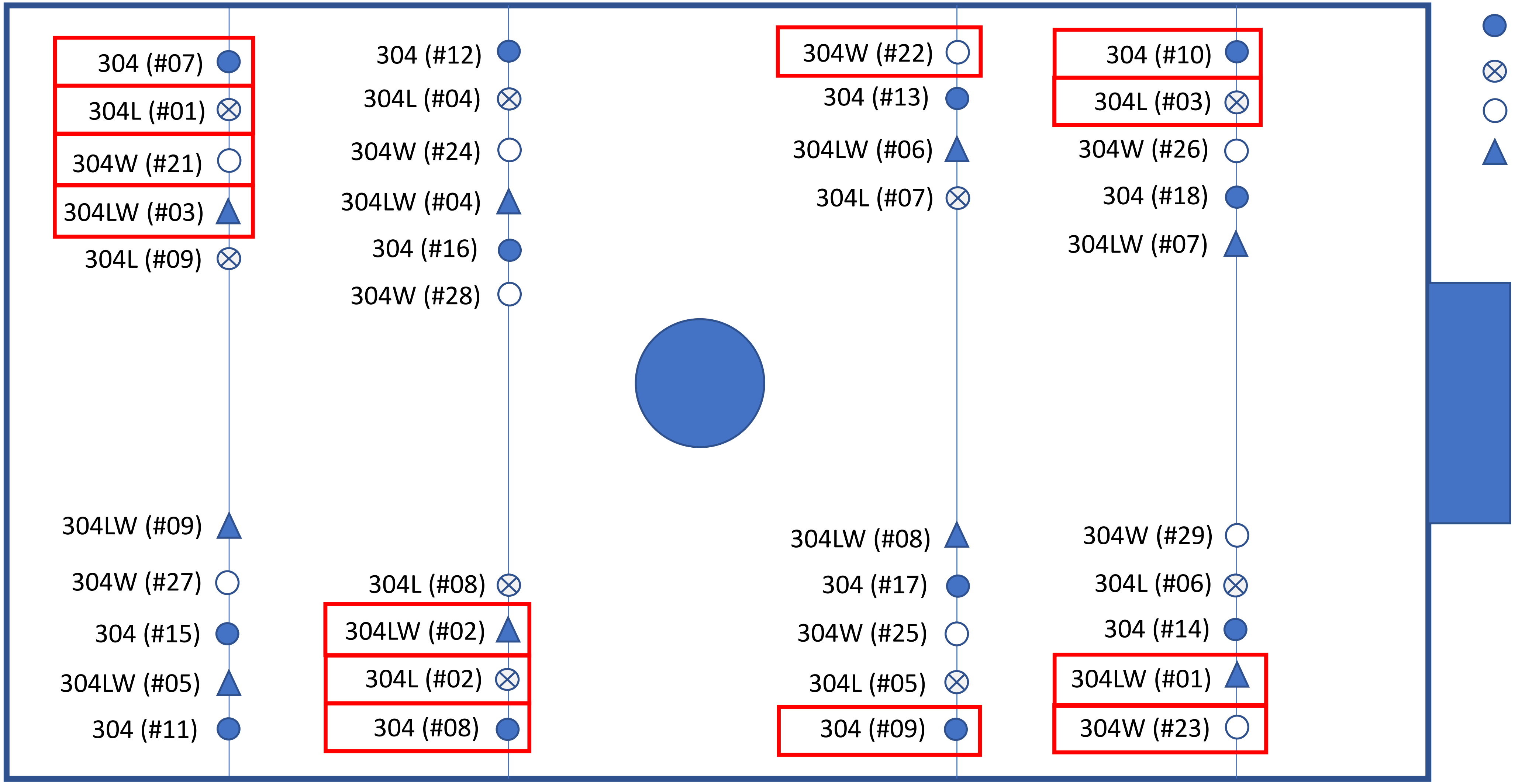




Experiment 1 Diagrams (35°C)



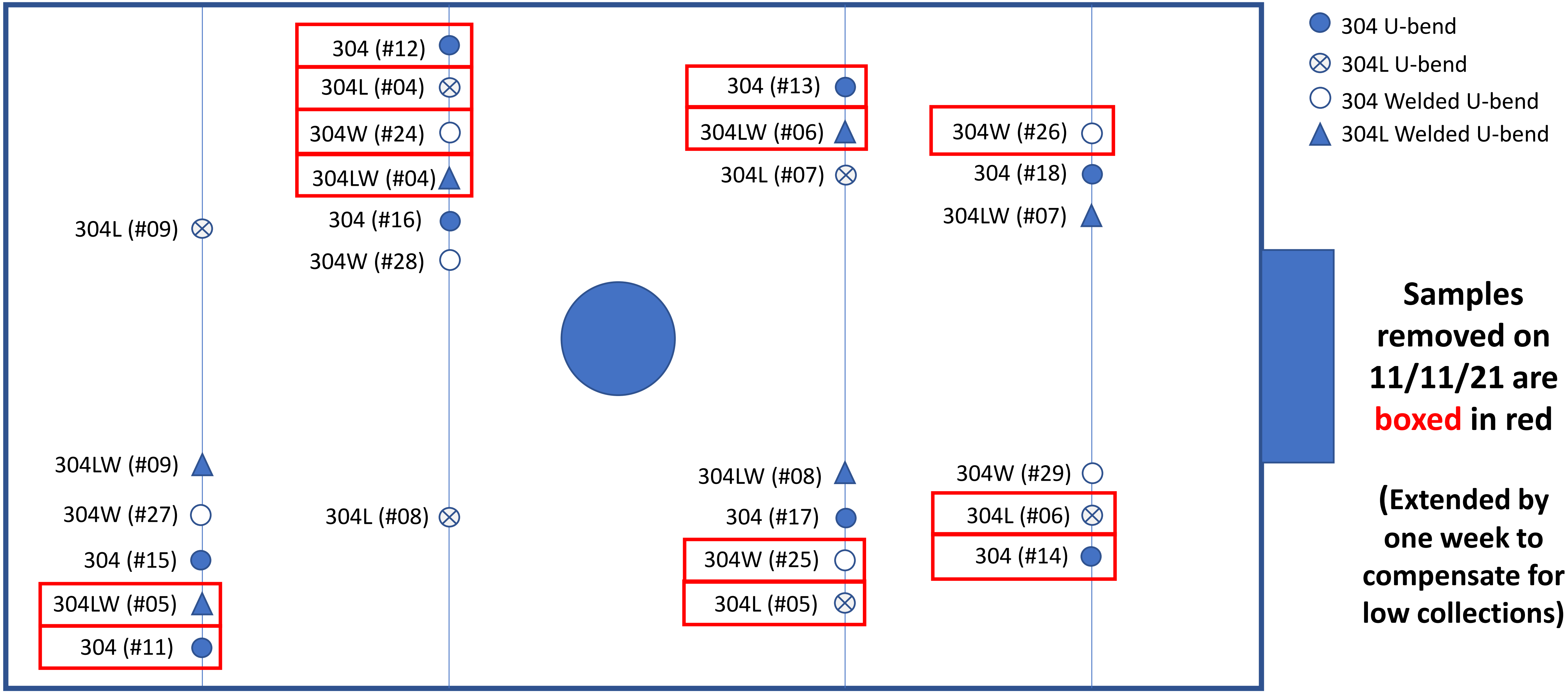
Experiment 1 Diagrams (35°C) – 4 weeks



- ● 304 U-bend
- ⊗ 304L U-bend
- ○ 304 Welded U-bend
- ▲ 304L Welded U-bend

Samples removed on 10/07/21 are boxed in red

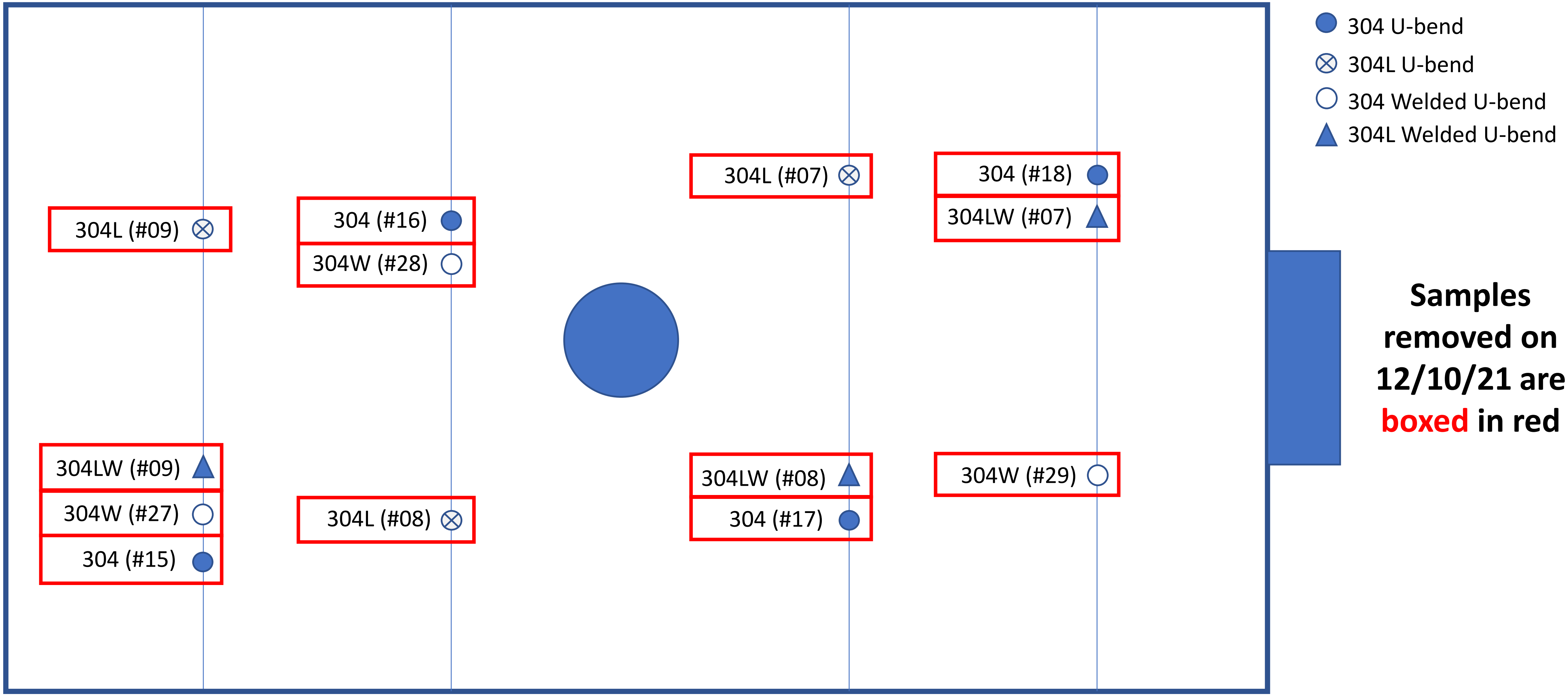
Experiment 1 Diagrams (35°C) – 8 weeks



Samples removed on 11/11/21 are boxed in red

(Extended by one week to compensate for low collections)

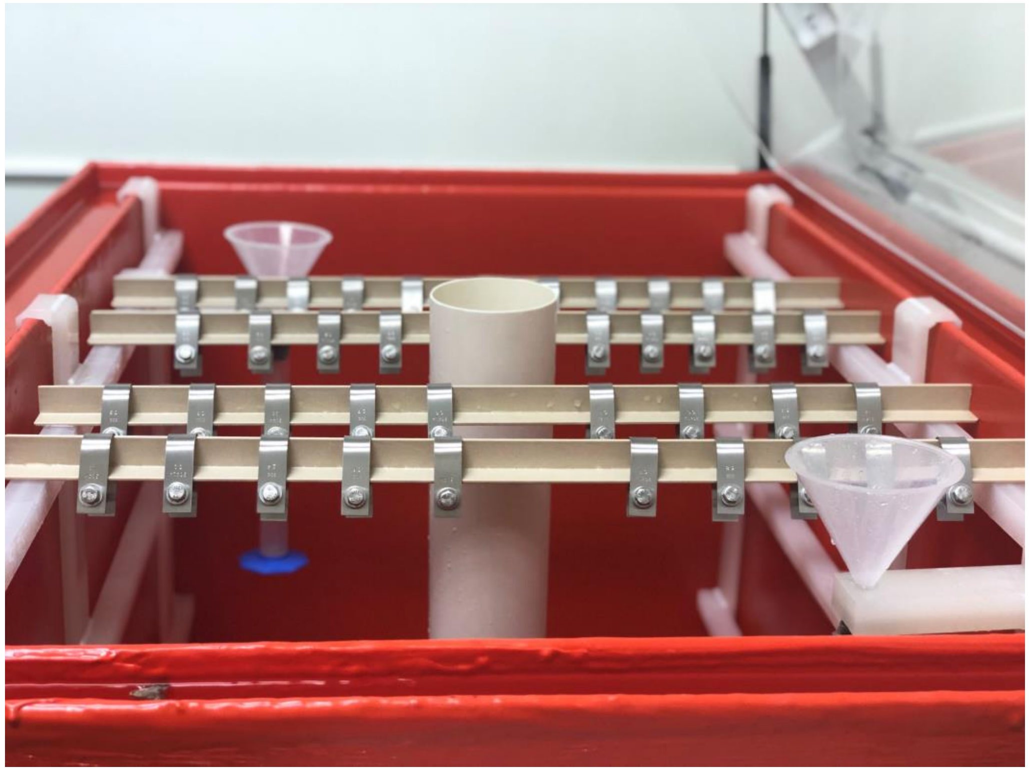
Experiment 1 Diagrams (35°C) – 12 weeks

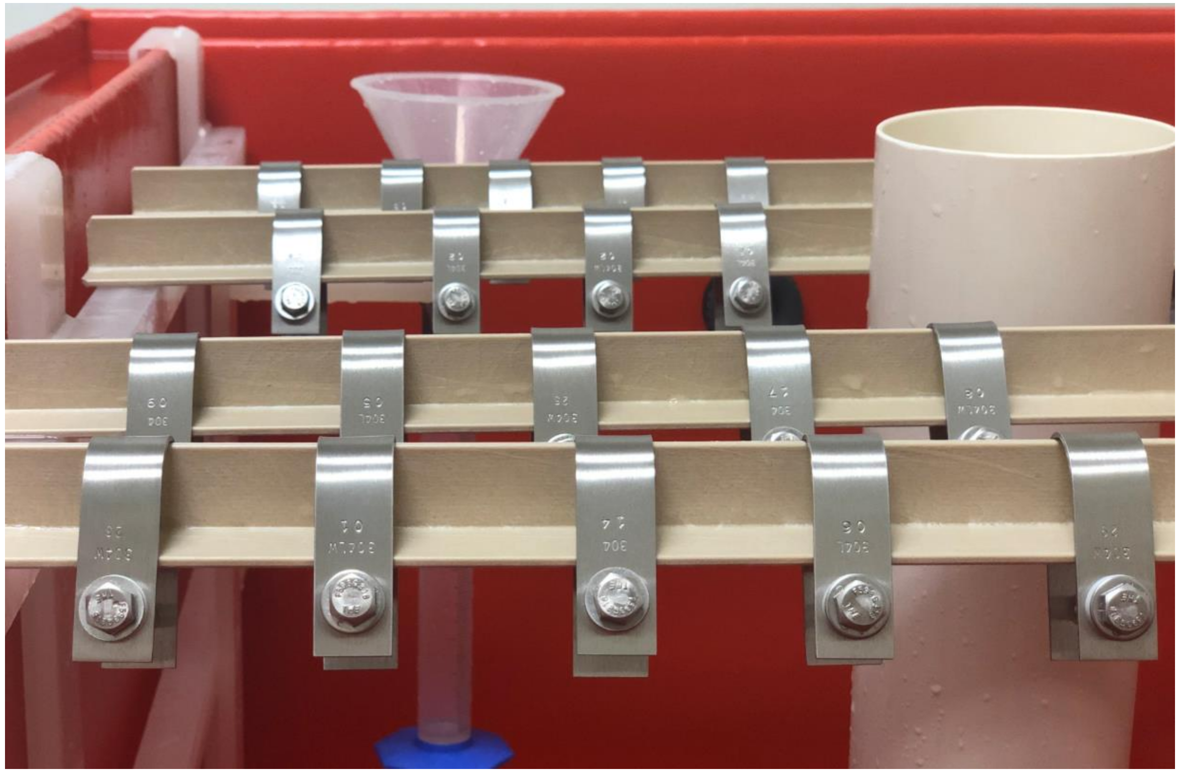


Samples removed on 12/10/21 are boxed in red

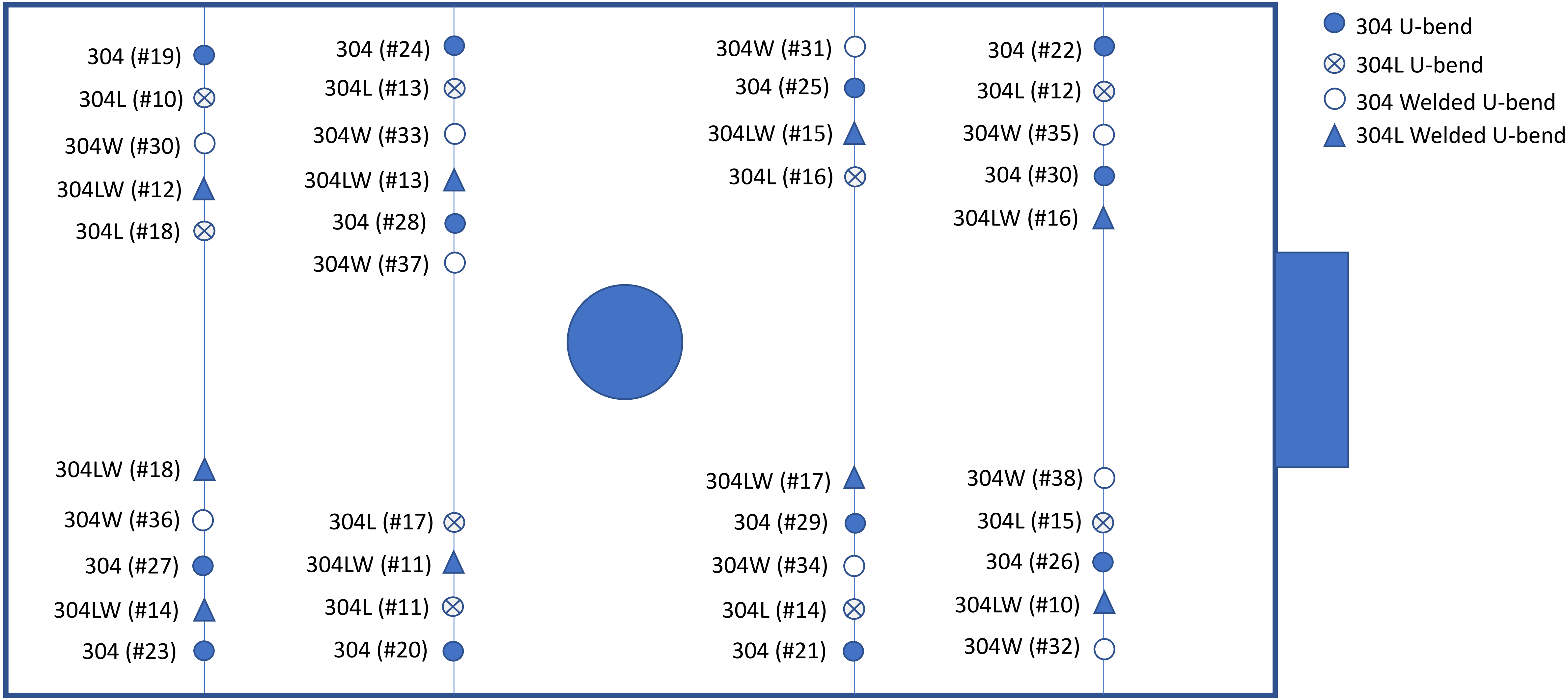
Experiment 2 Diagrams (50°C)

- Experiment 2 was started on 01/13/2022 at 50°C with 39 samples and was concluded on 04/25/2022.

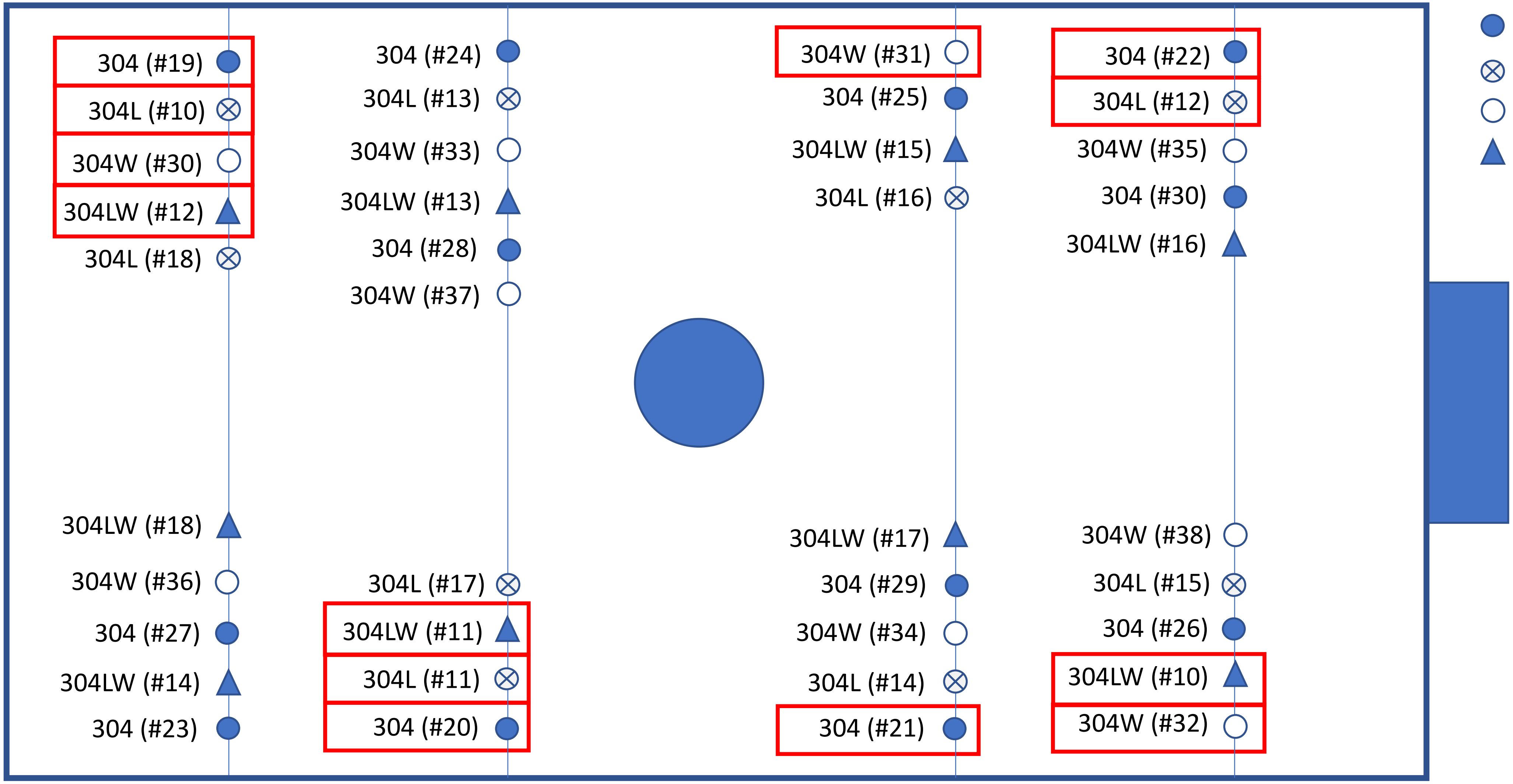




Experiment 2 Diagrams (50°C)



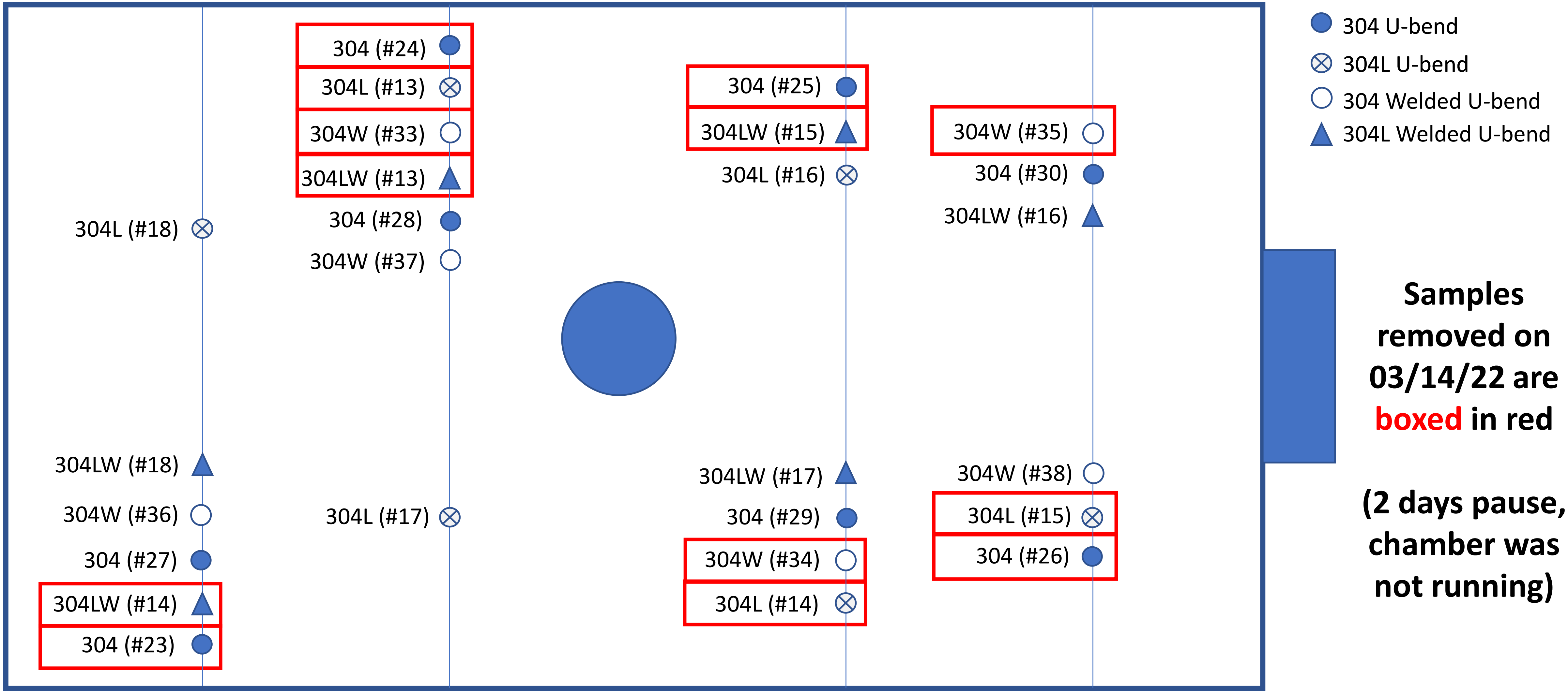
Experiment 2 Diagrams (50°C) – 4 weeks



- 304 U-bend
- 304L U-bend
- 304 Welded U-bend
- 304L Welded U-bend

Samples removed on 02/11/22 are boxed in red

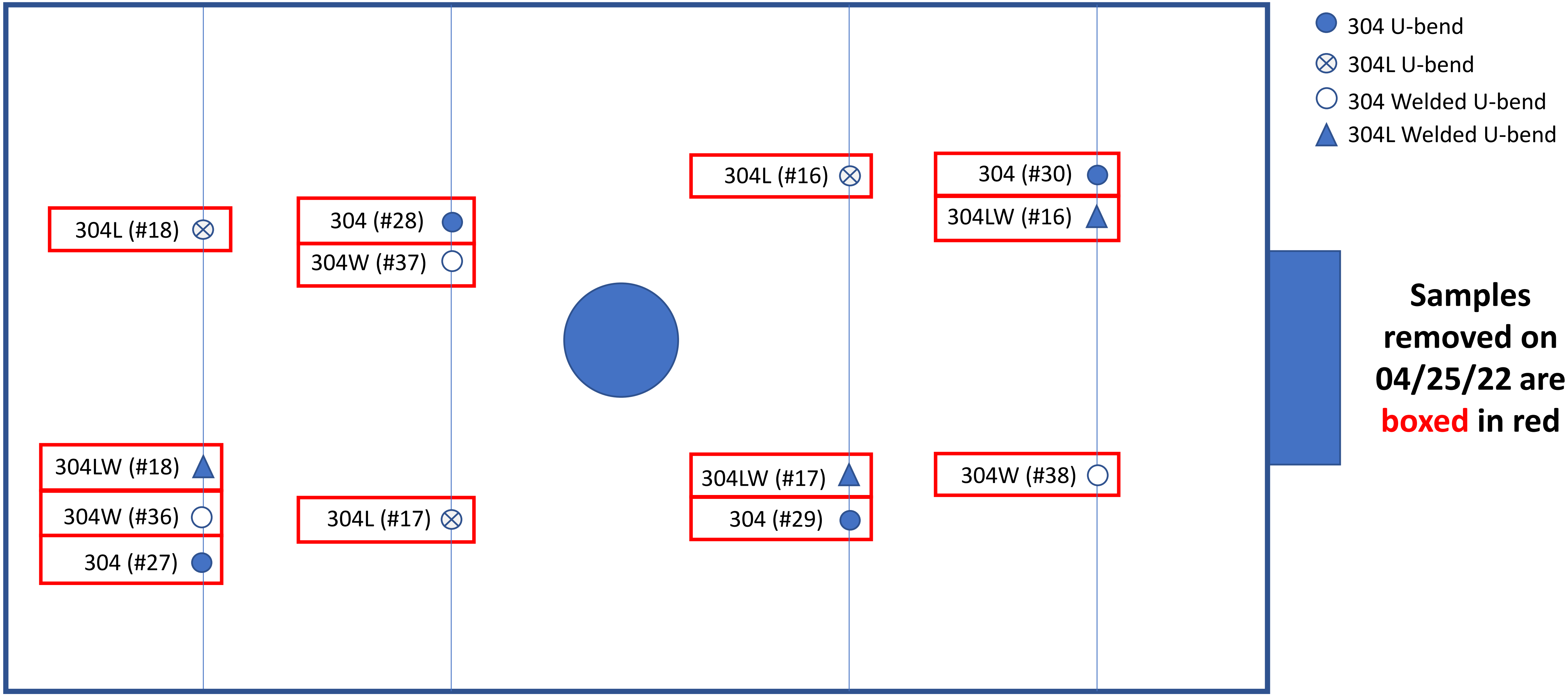
Experiment 2 Diagrams (50°C) – 8 weeks



Samples removed on 03/14/22 are boxed in red

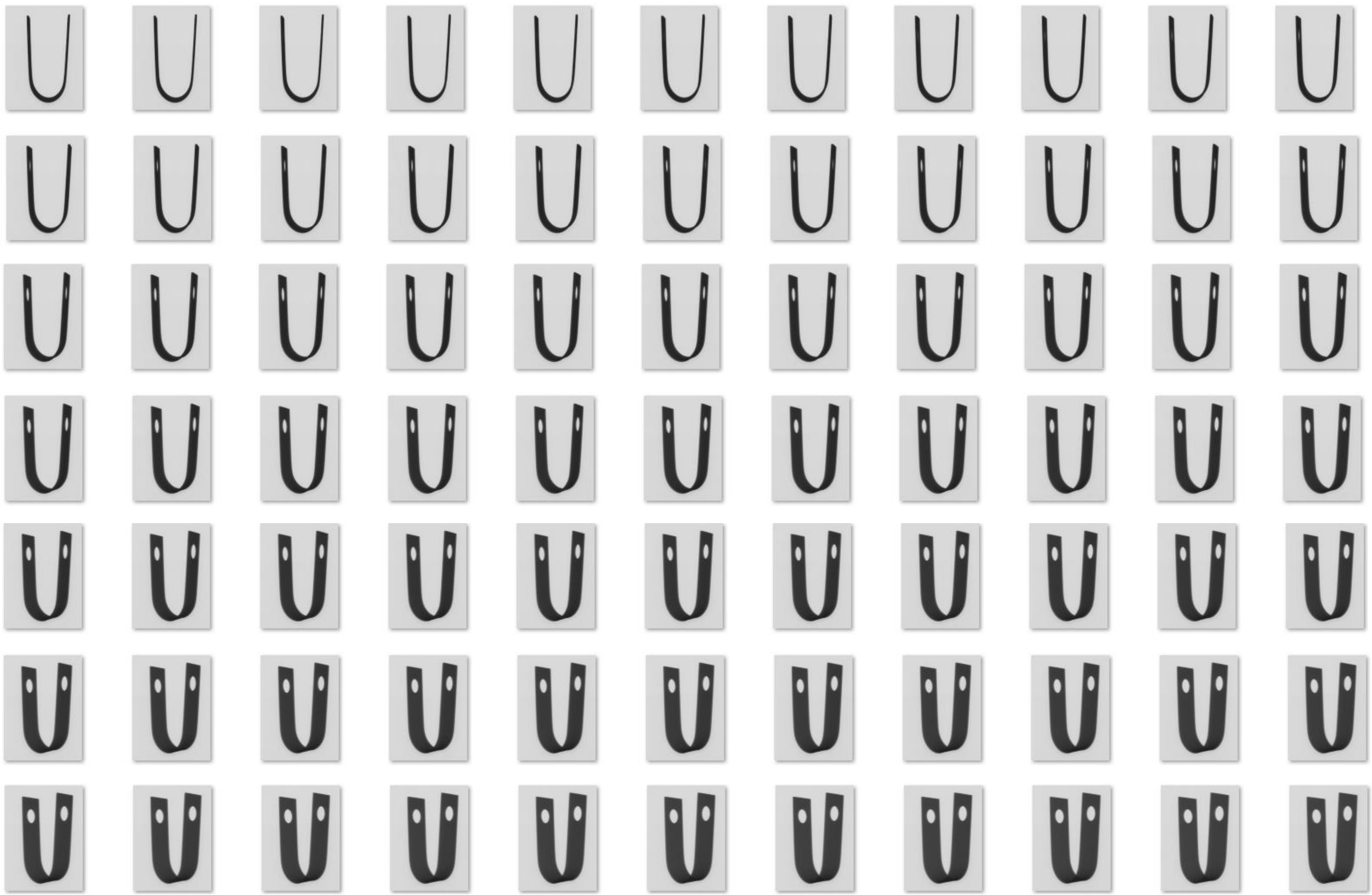
(2 days pause, chamber was not running)

Experiment 2 Diagrams (50°C) – 14 weeks

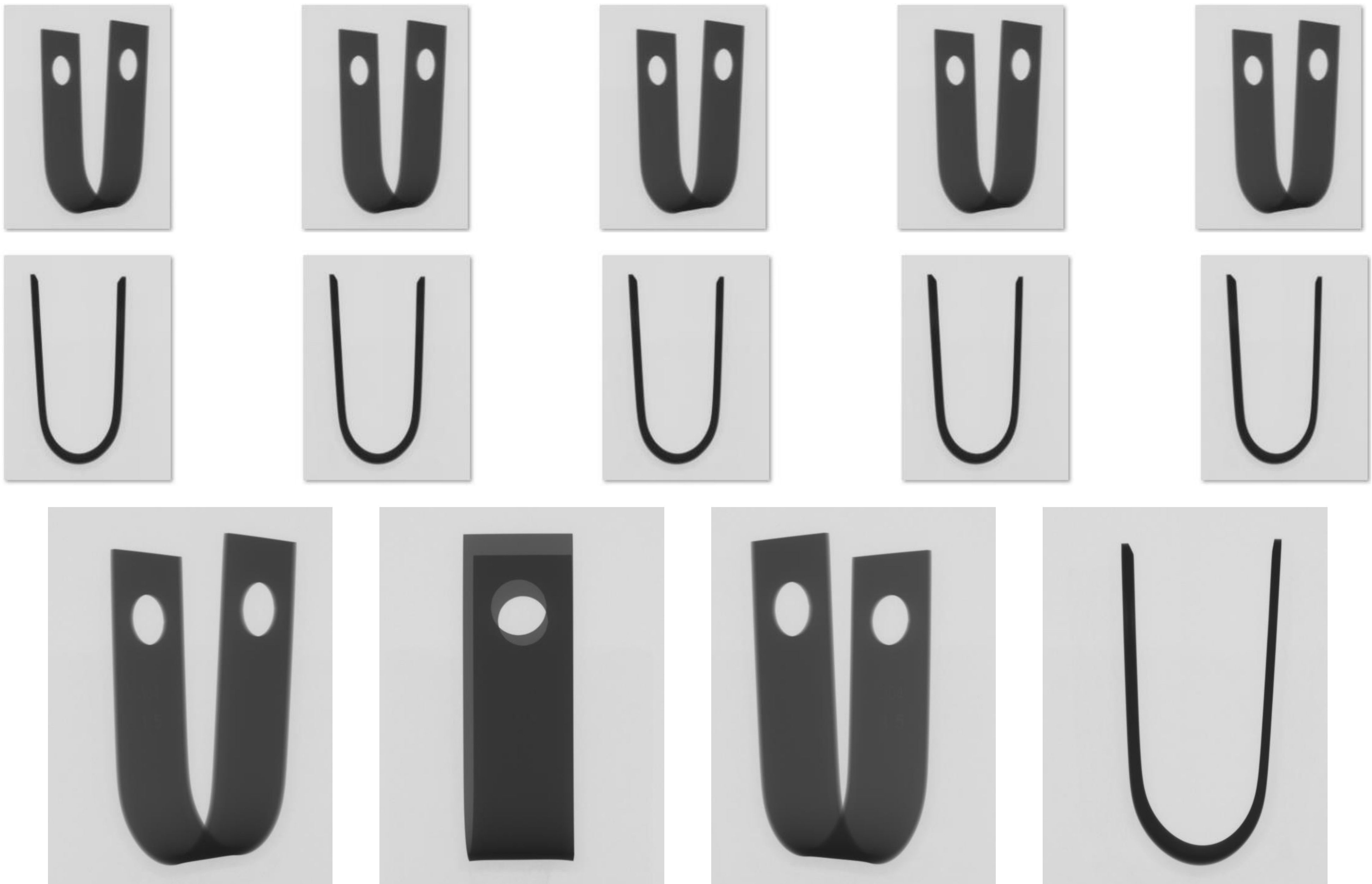


Samples removed on 04/25/22 are boxed in red

MicroCT (SKYSCAN 1273) Rotational Scans for U-bend Sample 304_15_50C_14 weeks

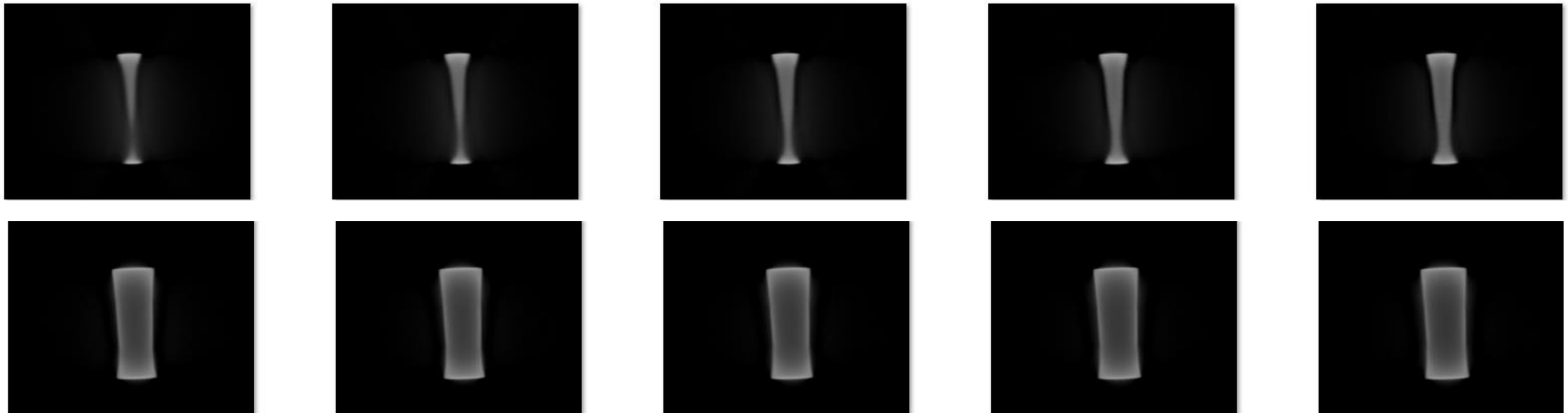


MicroCT (SKYSCAN 1273) Rotational Scans for U-bend Sample 304_15_50C_14 weeks

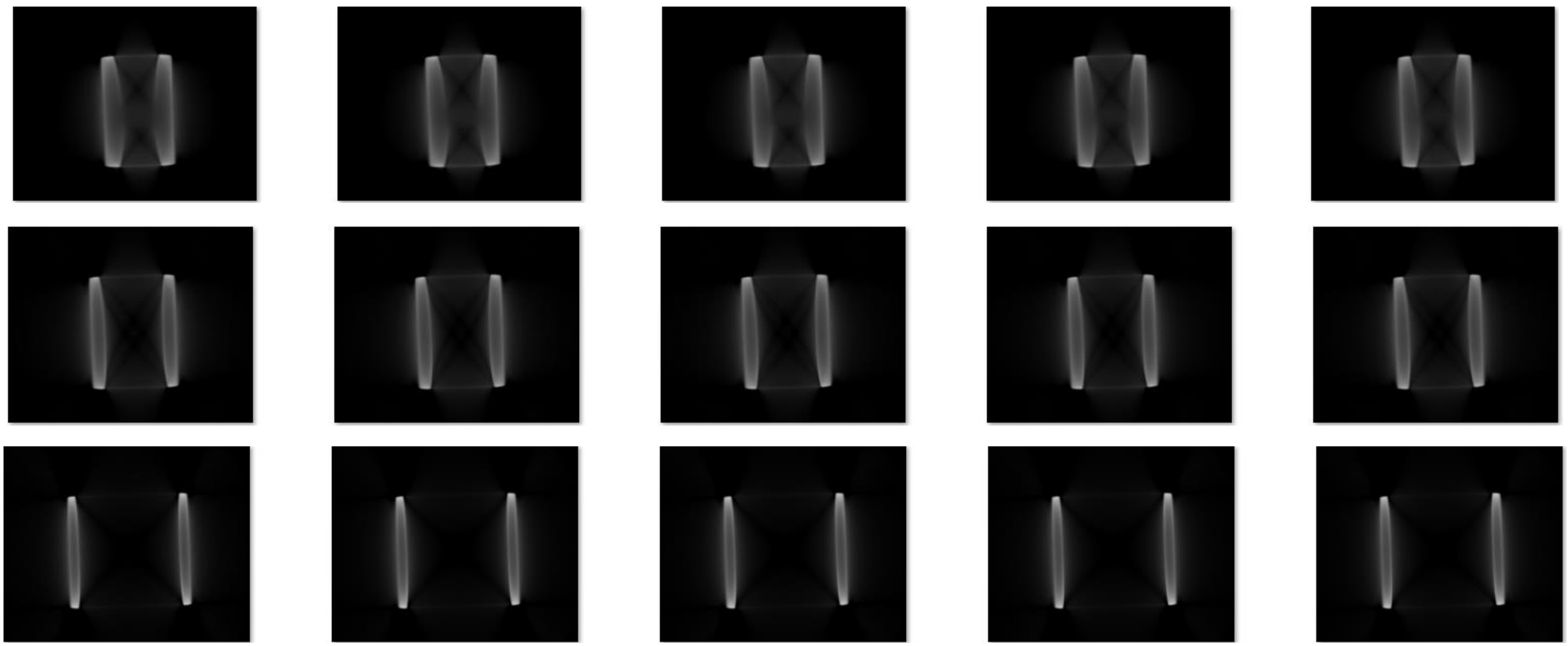


MicroCT (SKYSCAN 1273) Cross-sectional Scans for U-bend Sample 304_15_50C_14 weeks

Arch Cross-sections



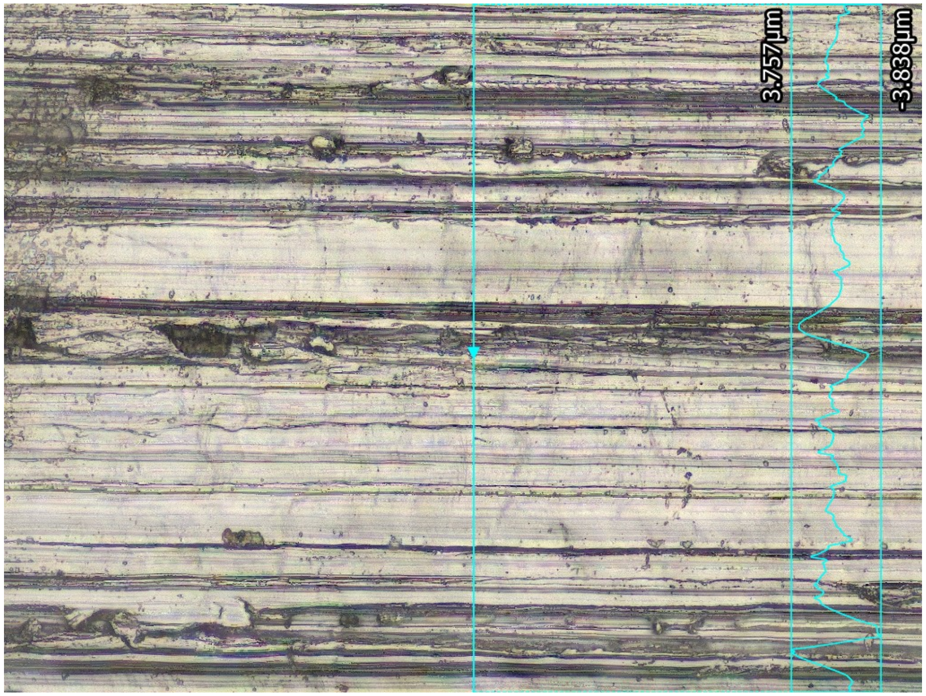
Legs Cross-sections



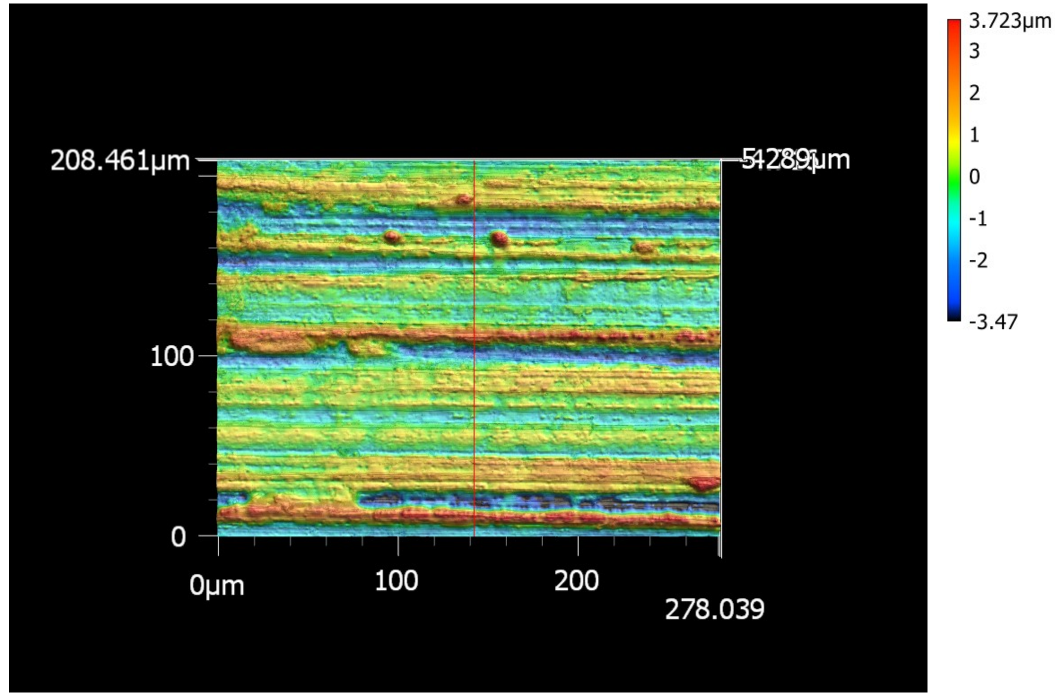
Profile measurement - SS304_No. 25_Reference Sample

KEYENCE VK-X3000 Series

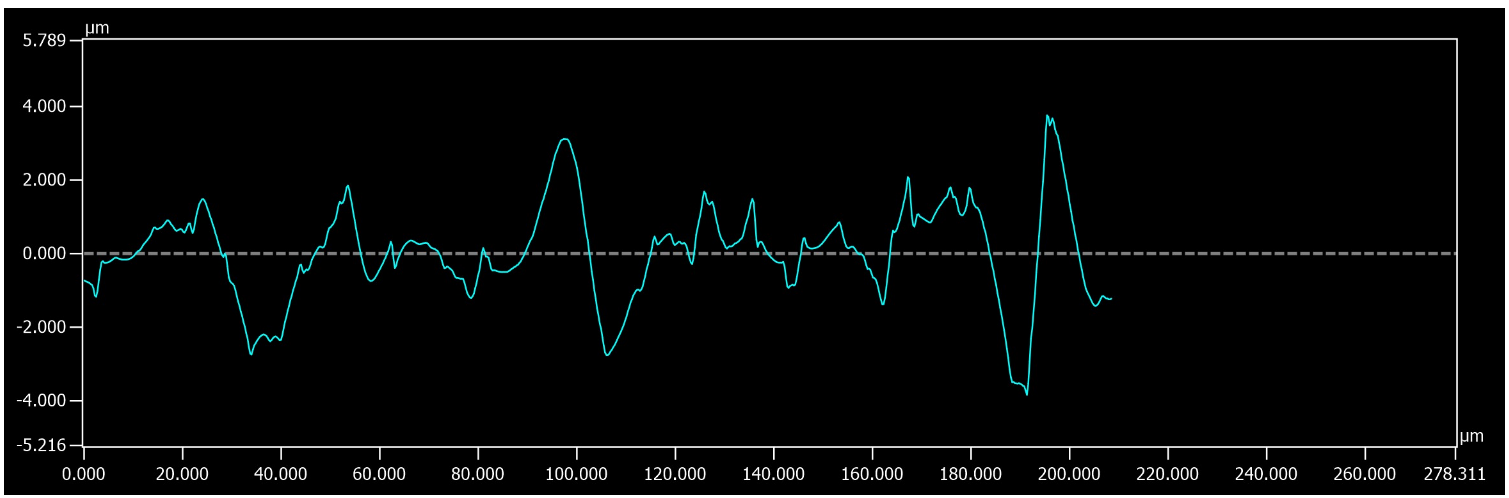
Main image



3D image



Profile

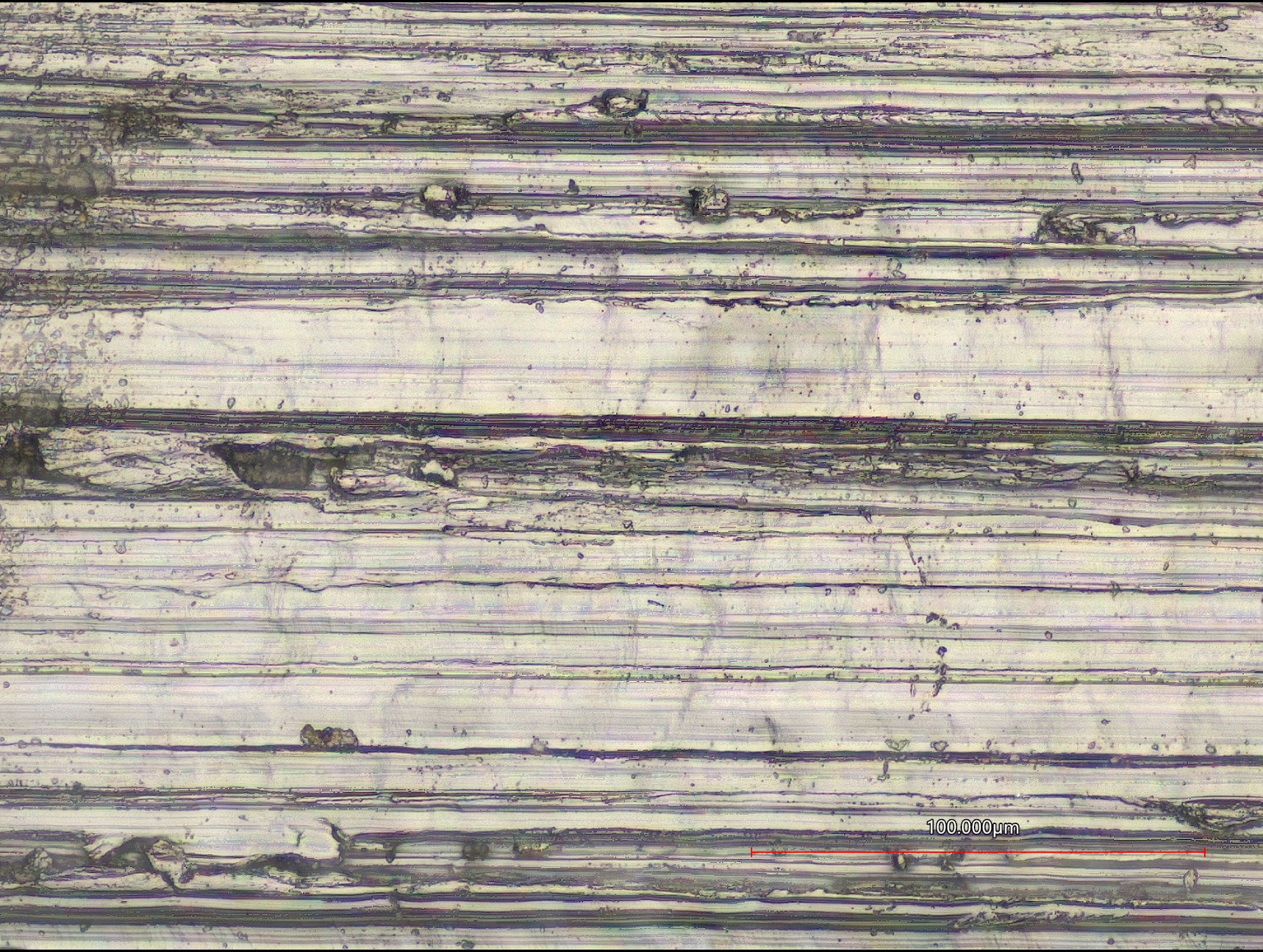


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/21/2022 2:16:17 PM

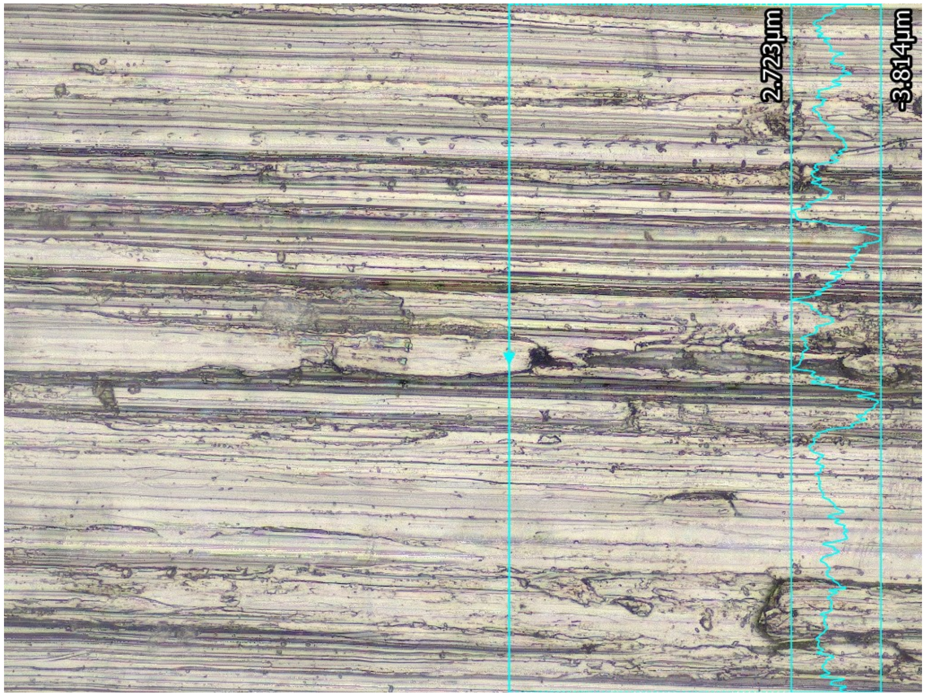
Objective Lens Power： 50X


100.000µm

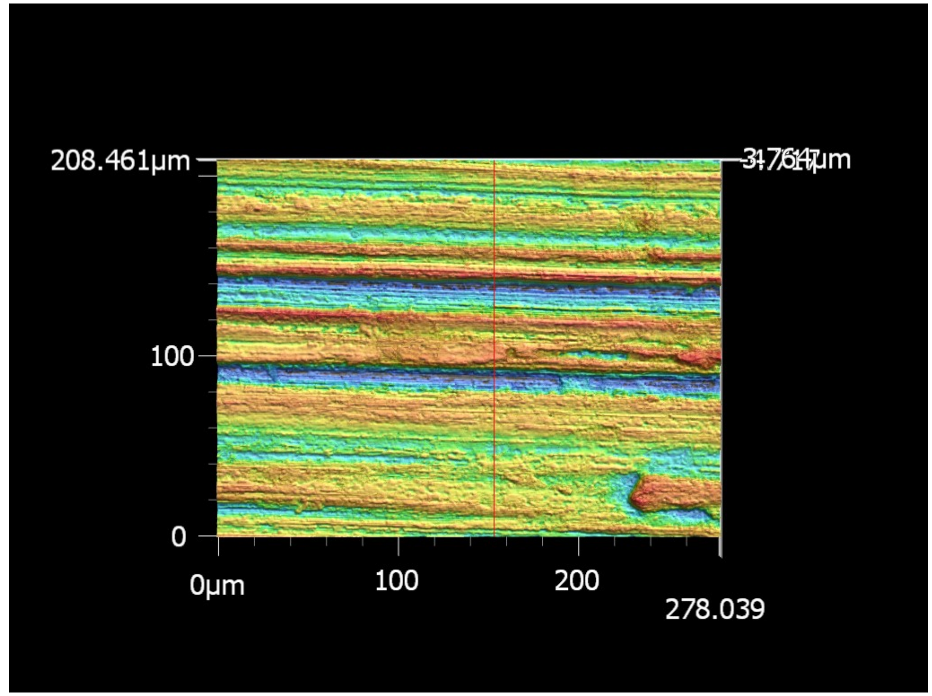
Profile measurement - SS304W_No. 39_Reference Sample

KEYENCE VK-X3000 Series

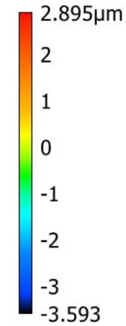
Main image



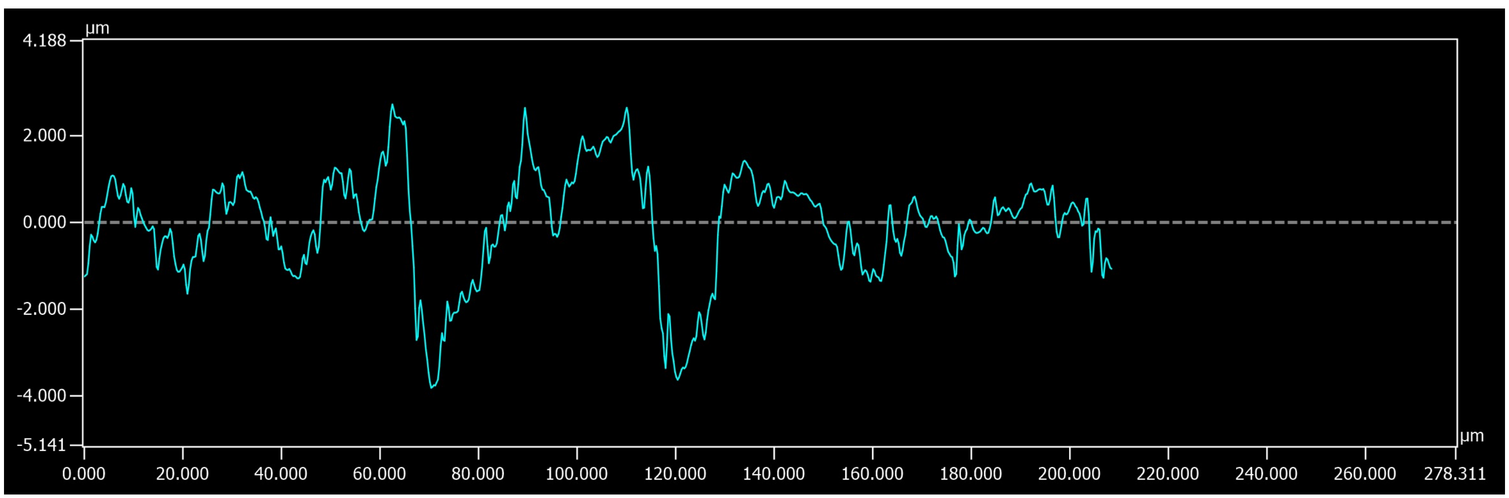
3D image



Height color



Profile

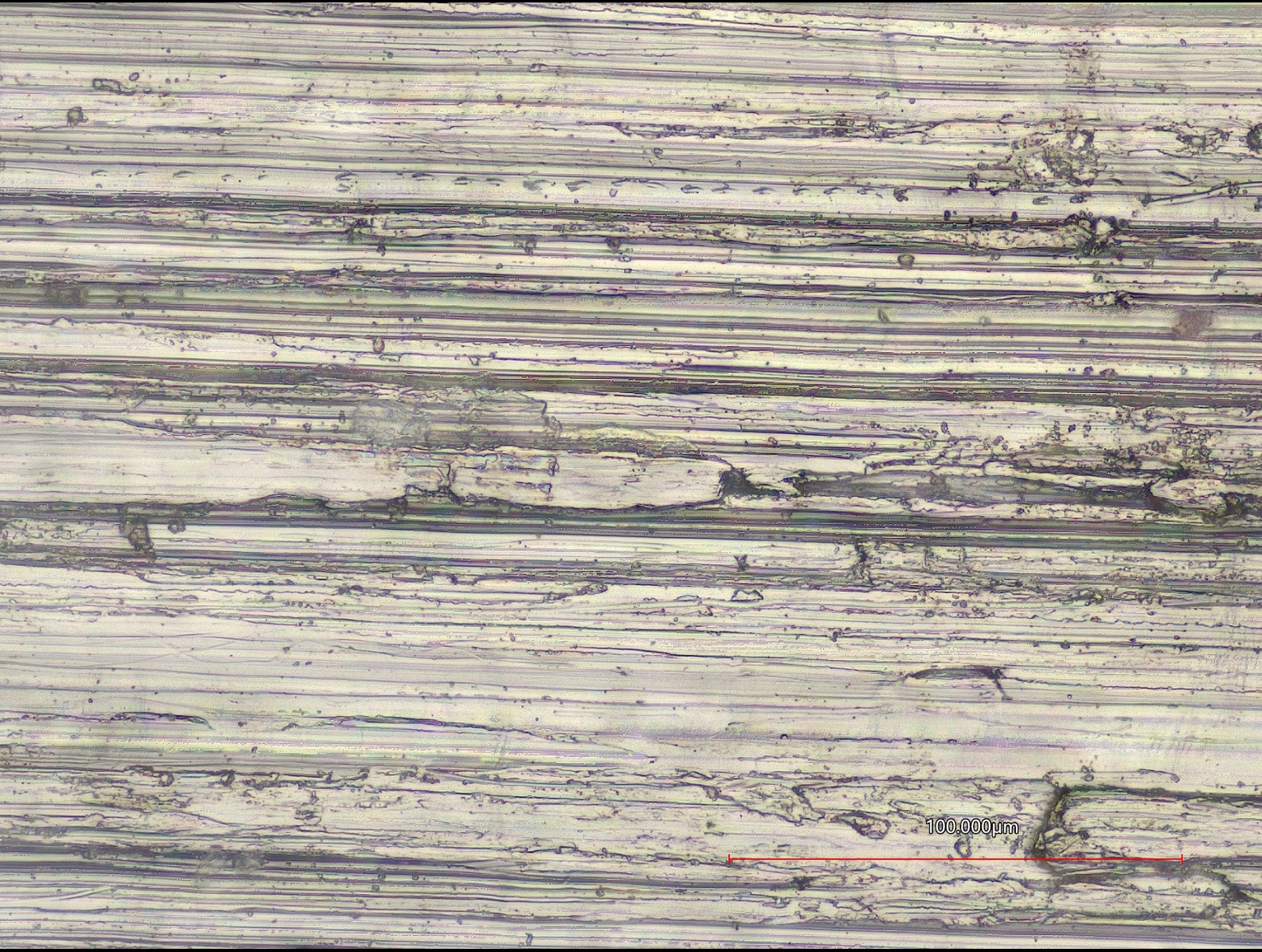


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/21/2022 3:06:48 PM

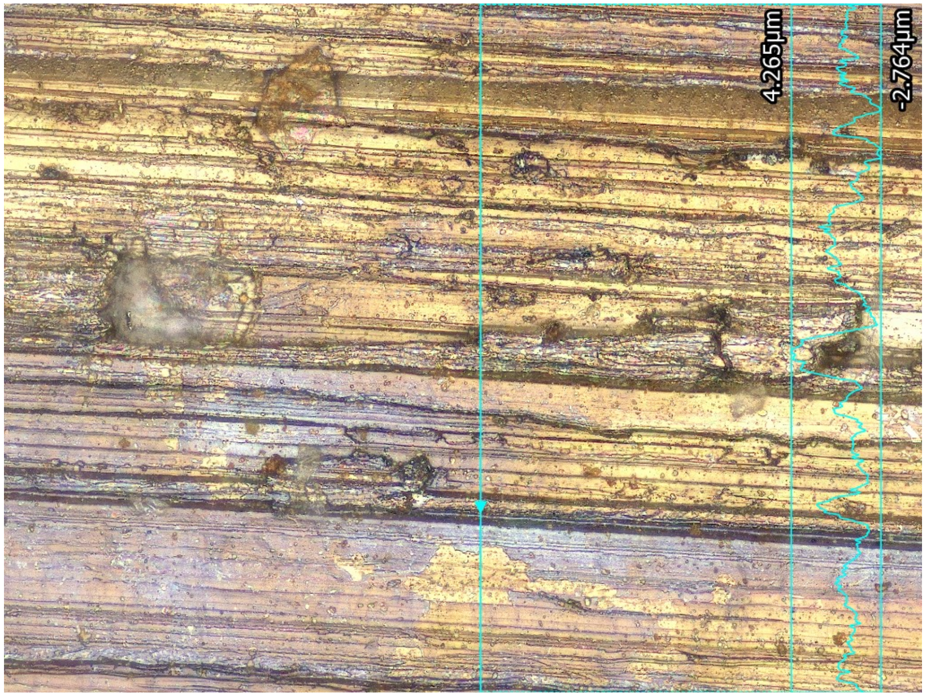
Objective Lens Power： 50X


100.000μm

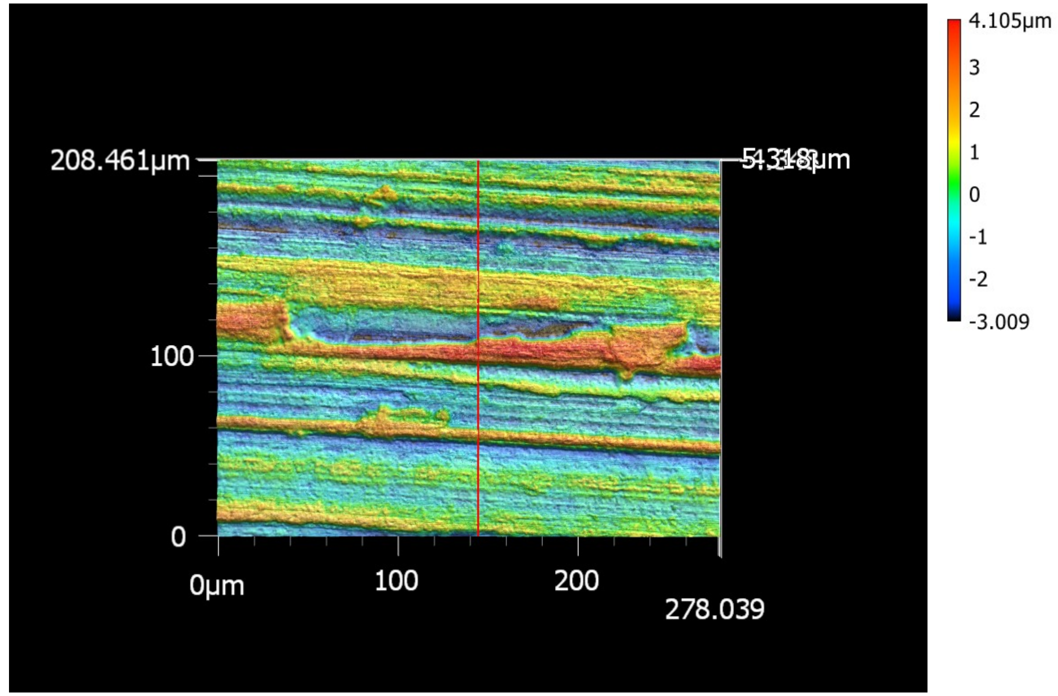
Profile measurement - SS304_No. 28_50°C_14 Weeks

KEYENCE VK-X3000 Series

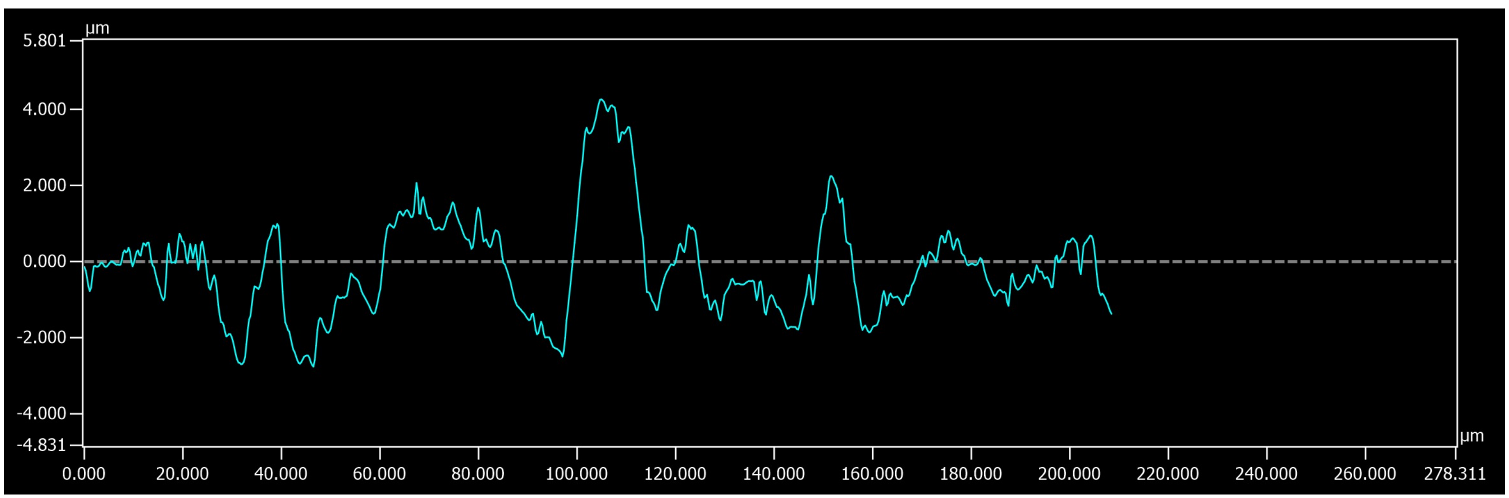
Main image



3D image



Profile

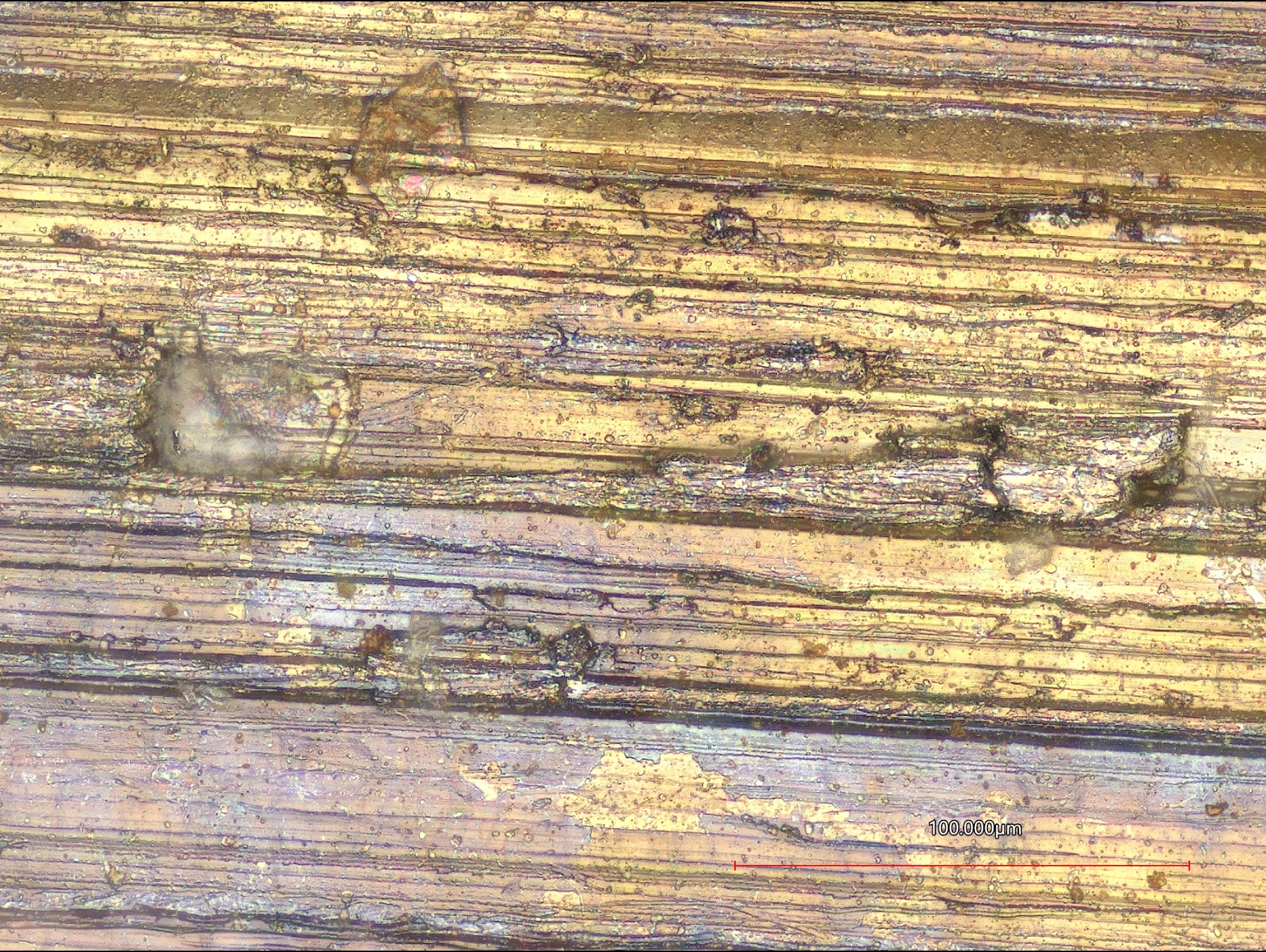


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/21/2022 4:04:56 PM

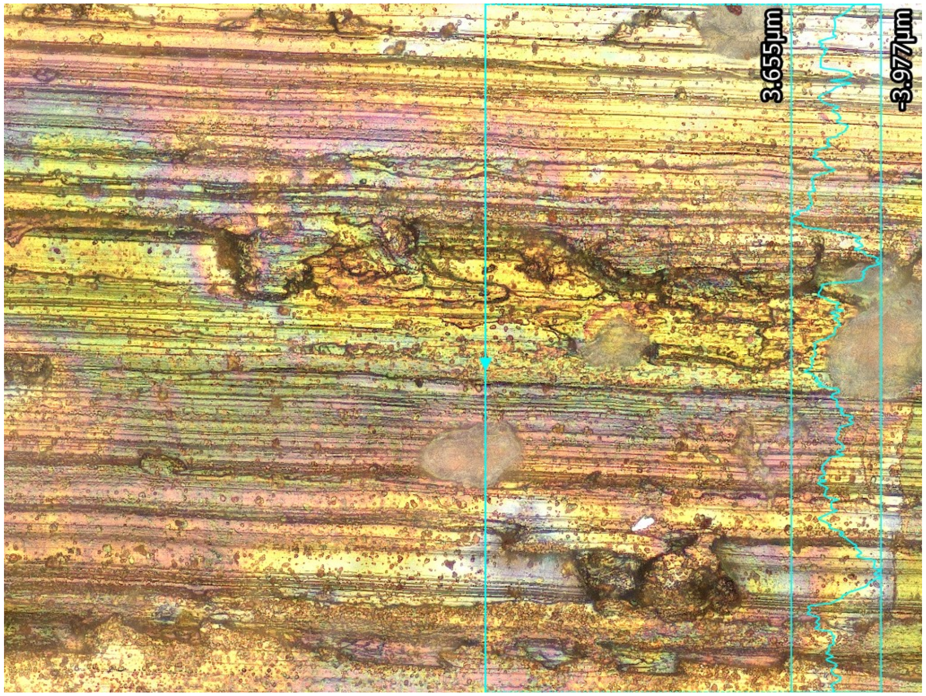
Objective Lens Power： 50X


100.000µm

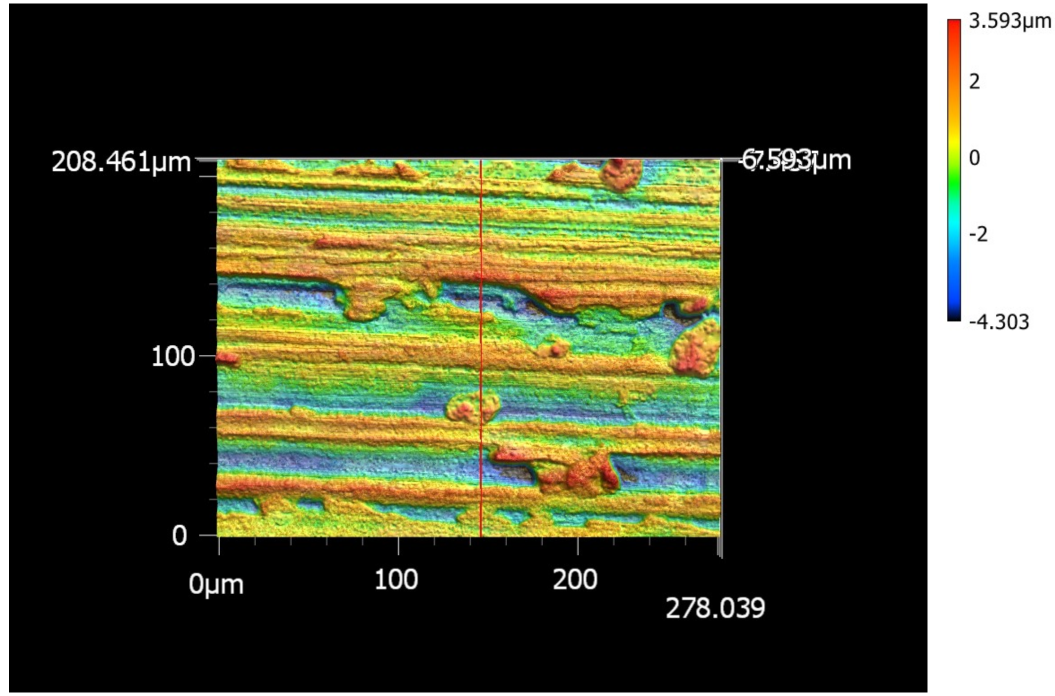
Profile measurement - SS304L_No. 17_50°C_14 Weeks

KEYENCE VK-X3000 Series

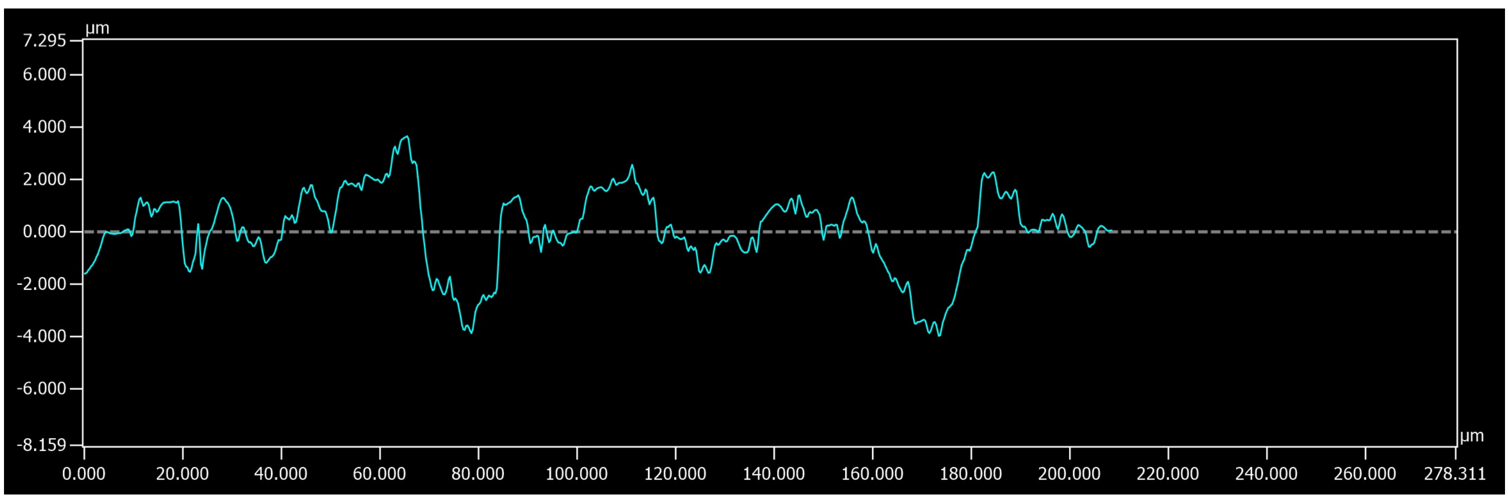
Main image



3D image



Profile

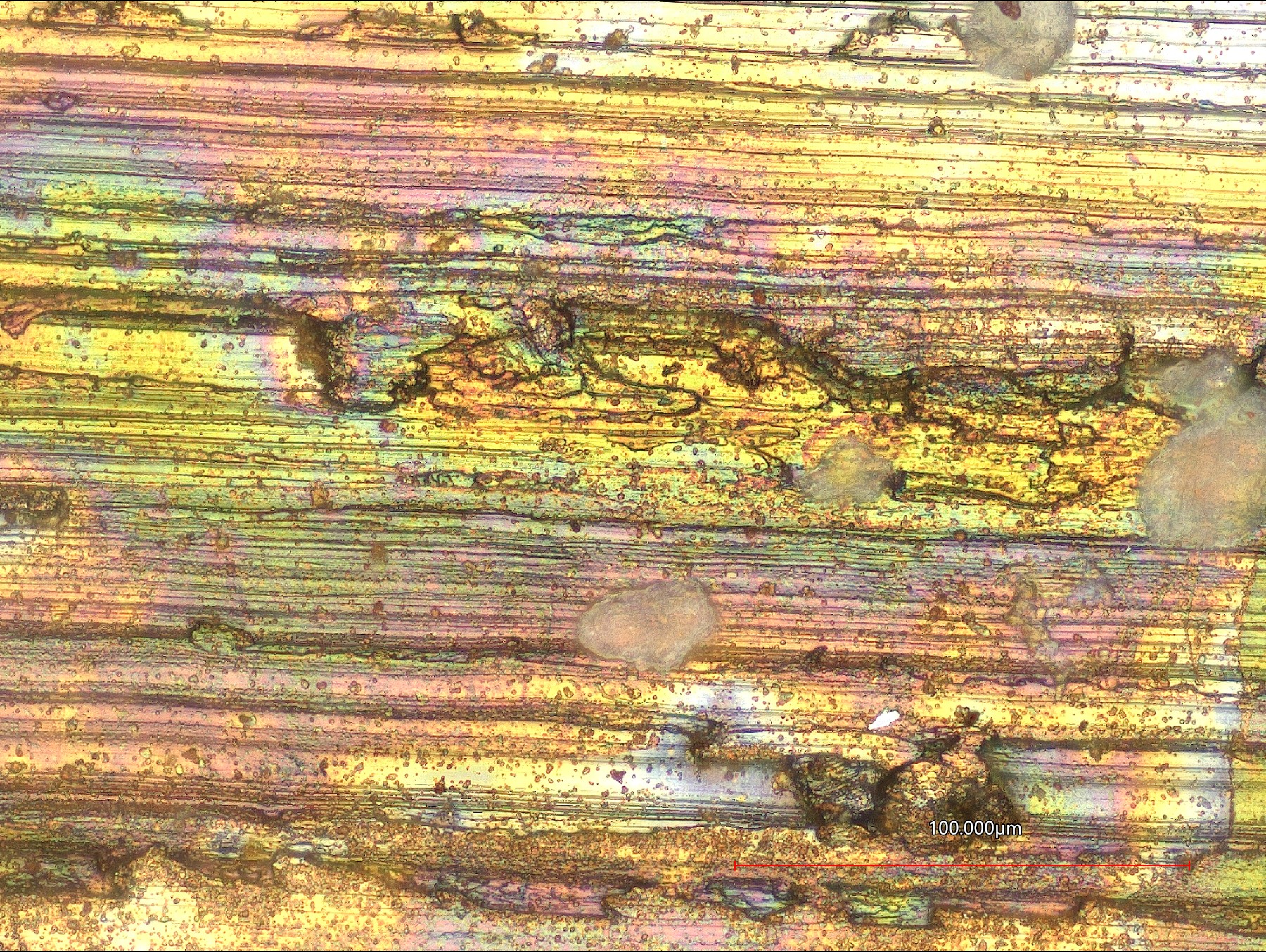


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/21/2022 4:10:30 PM

Objective Lens Power： 50X

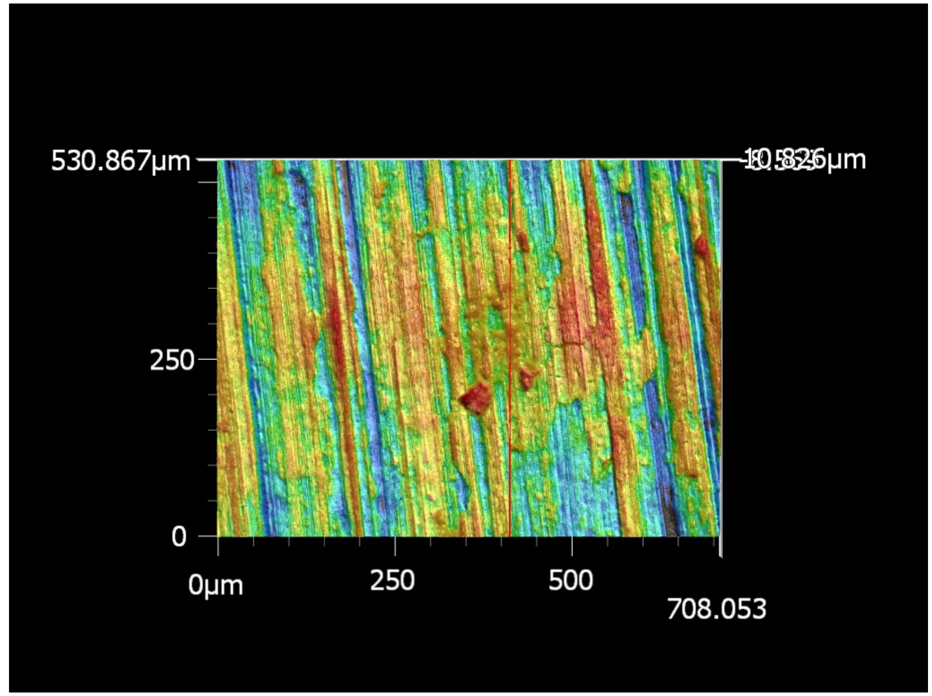

100.000µm

Profile measurement - SS304W_No. 36_50°C_8 Weeks

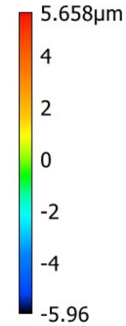
KEYENCE VK-X3000 Series

Main image

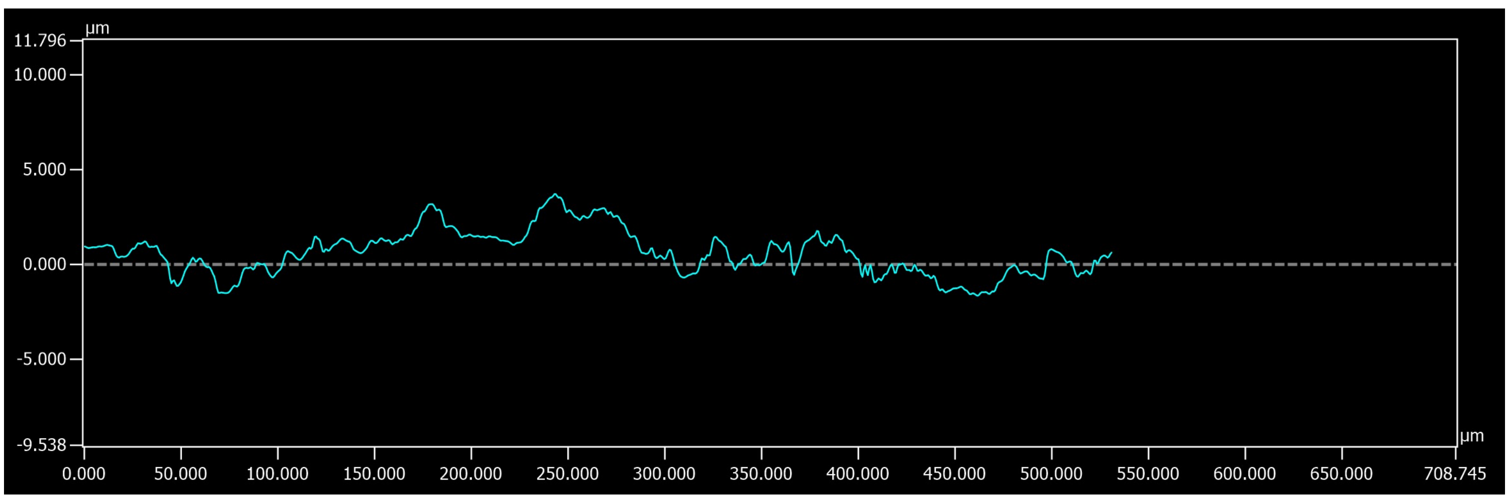
3D image



Height color



Profile



Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/10/2022 5:07:29 PM

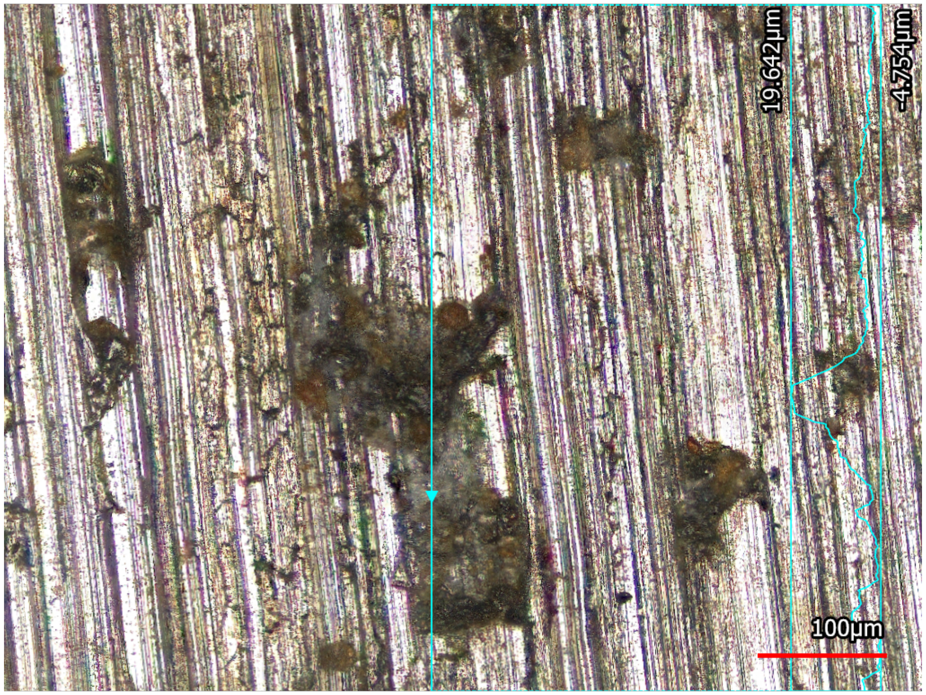
Objective Lens Power： 20X


100µm

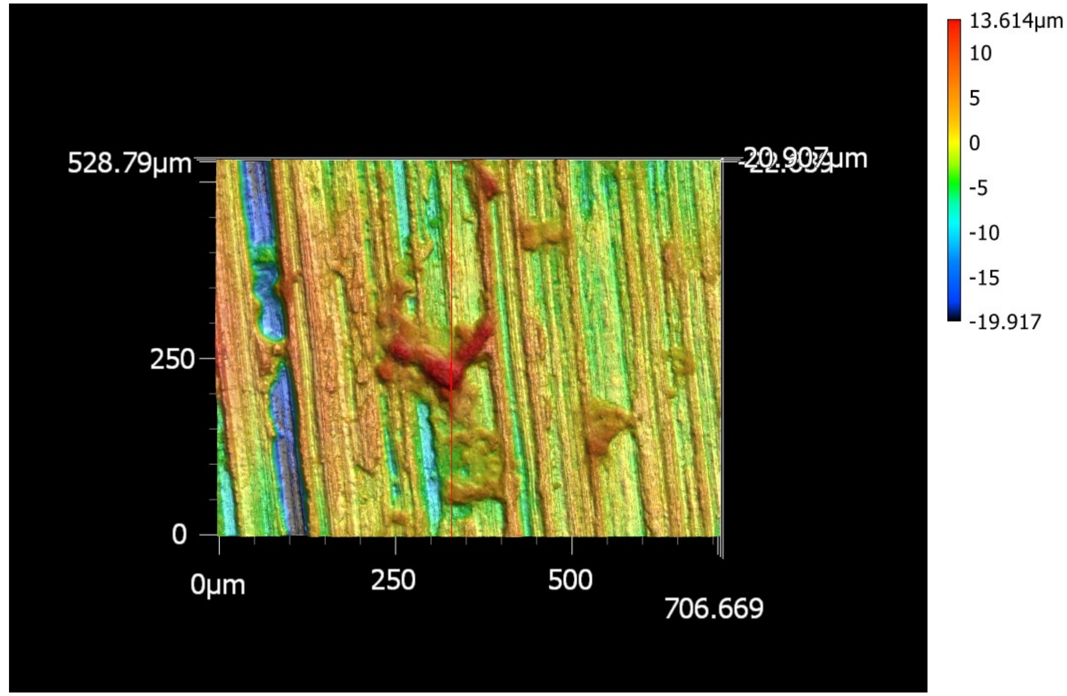
Profile measurement - SS304LW_No. 13_50°C_8 Weeks

KEYENCE VK-X3000 Series

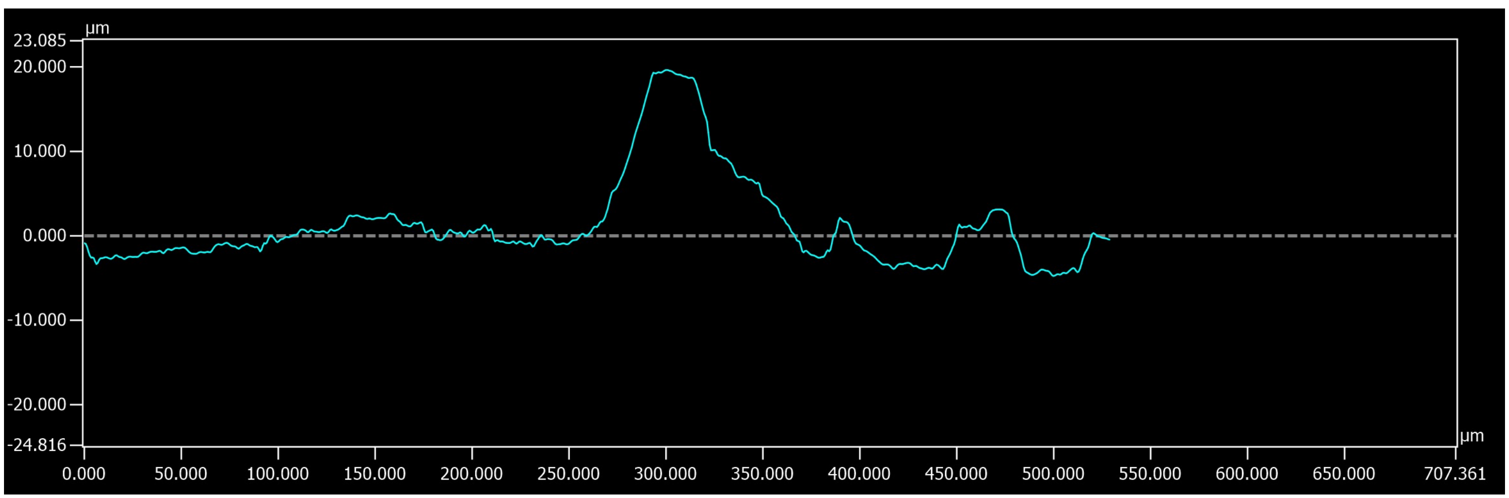
Main image



3D image



Profile



Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/10/2022 5:57:55 PM

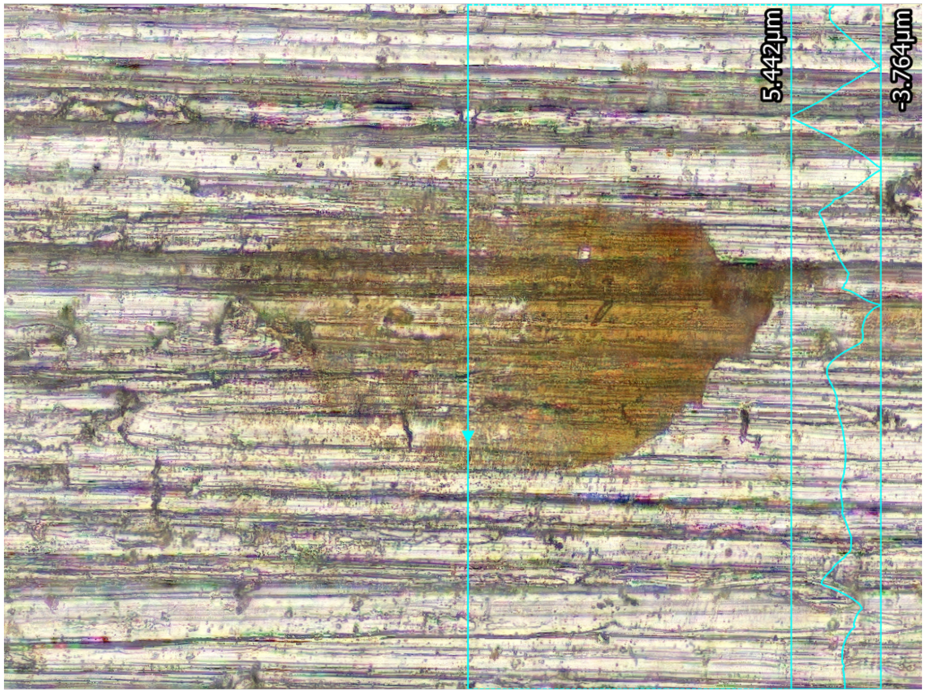
Objective Lens Power： 20X


100µm

Profile measurement - SS304_No. 16_35°C_12 Weeks

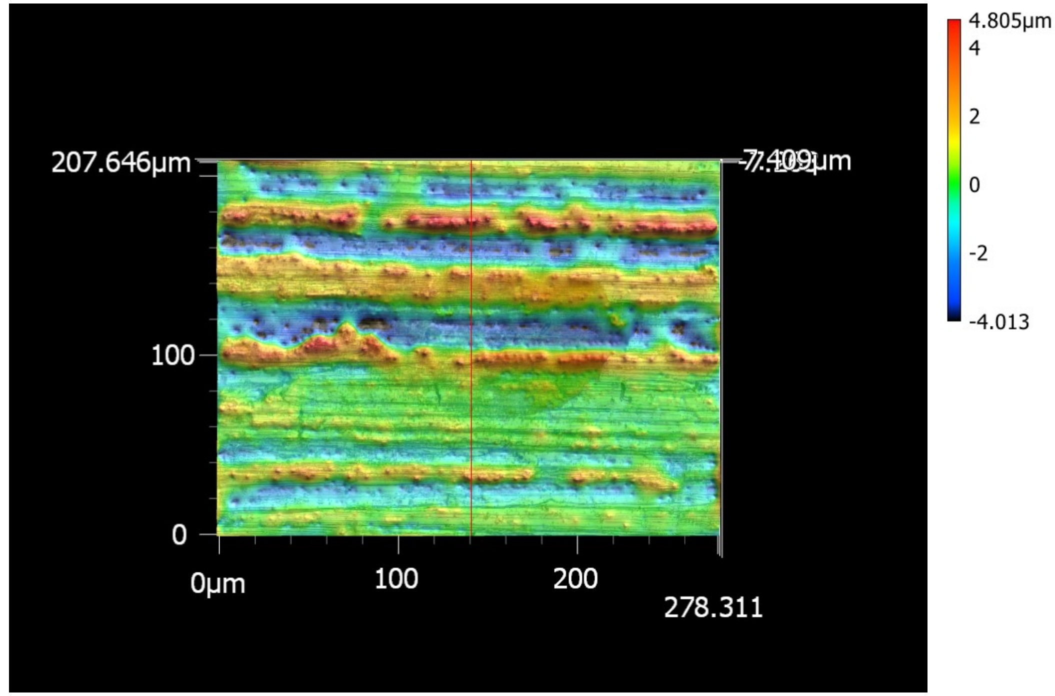
KEYENCE VK-X3000 Series

Main image

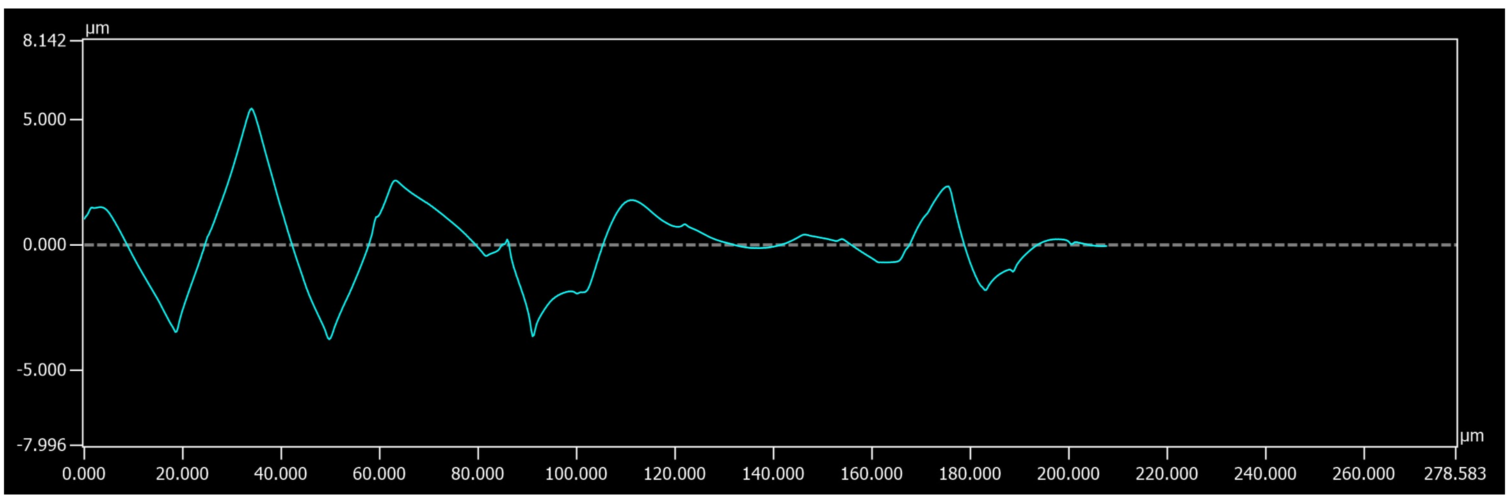


3D image

Height color



Profile

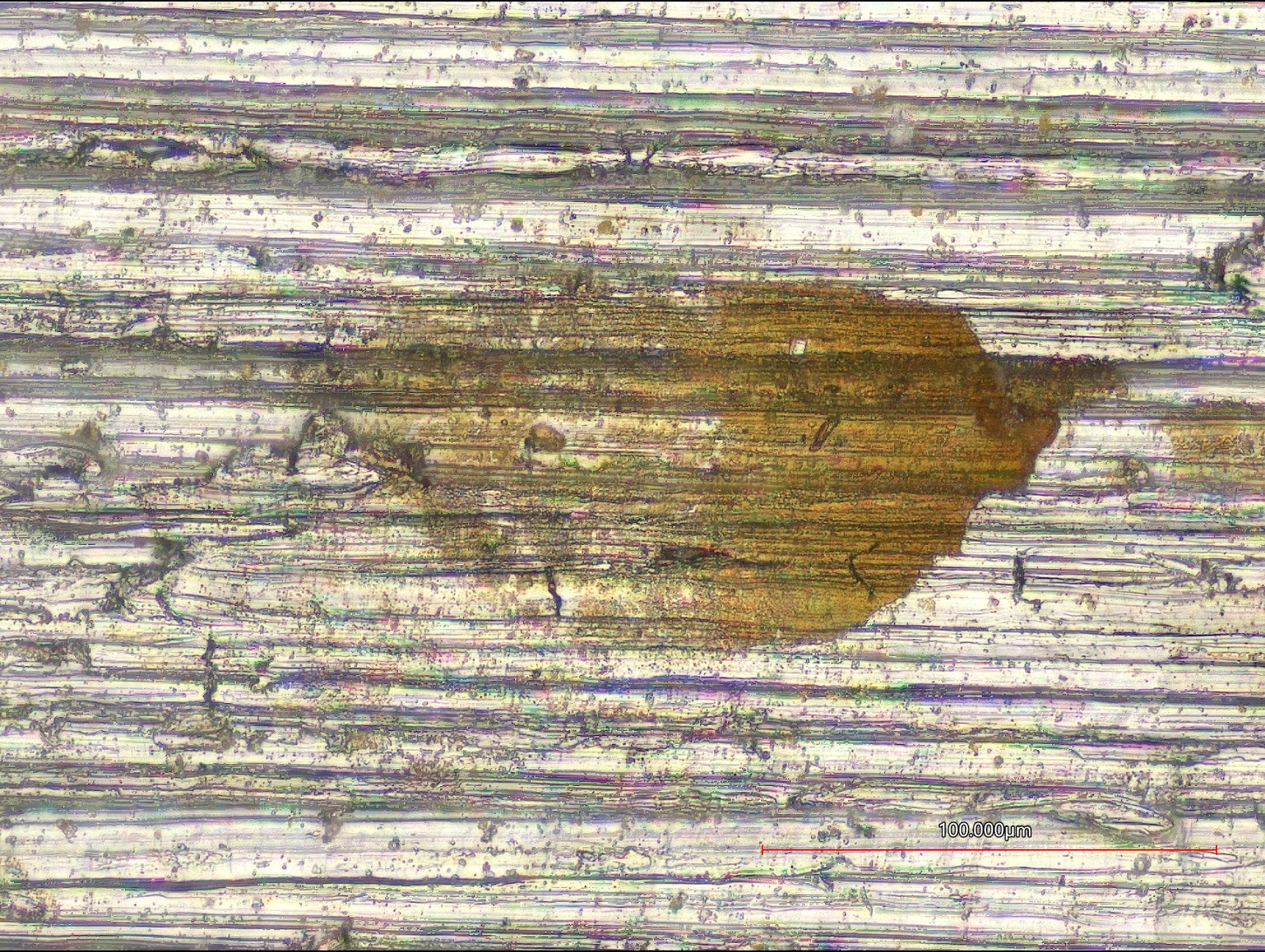


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/11/2022 10:13:47 AM

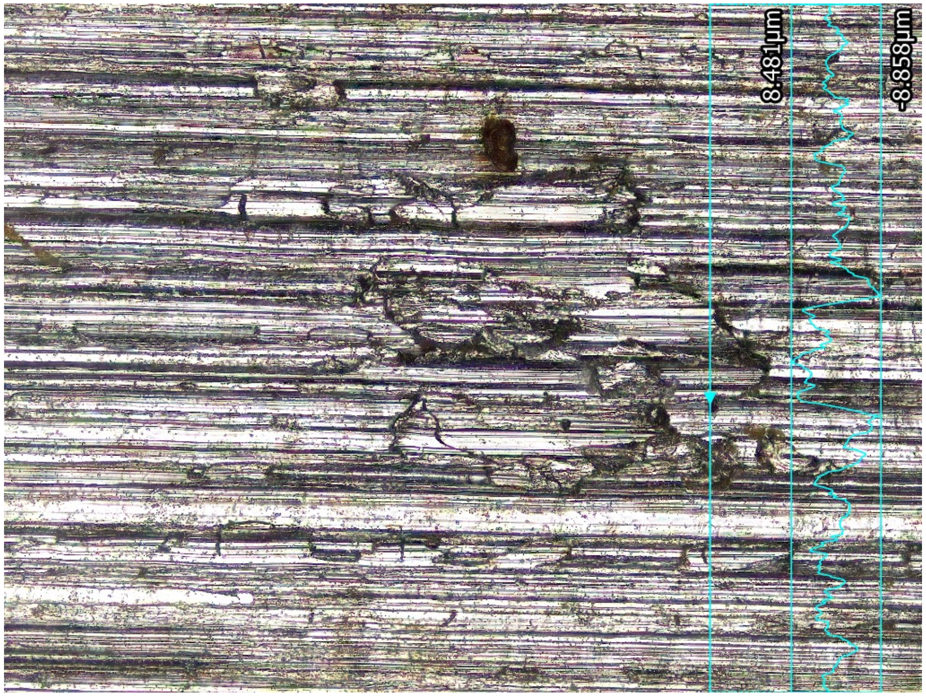
Objective Lens Power： 50X


100.000μm

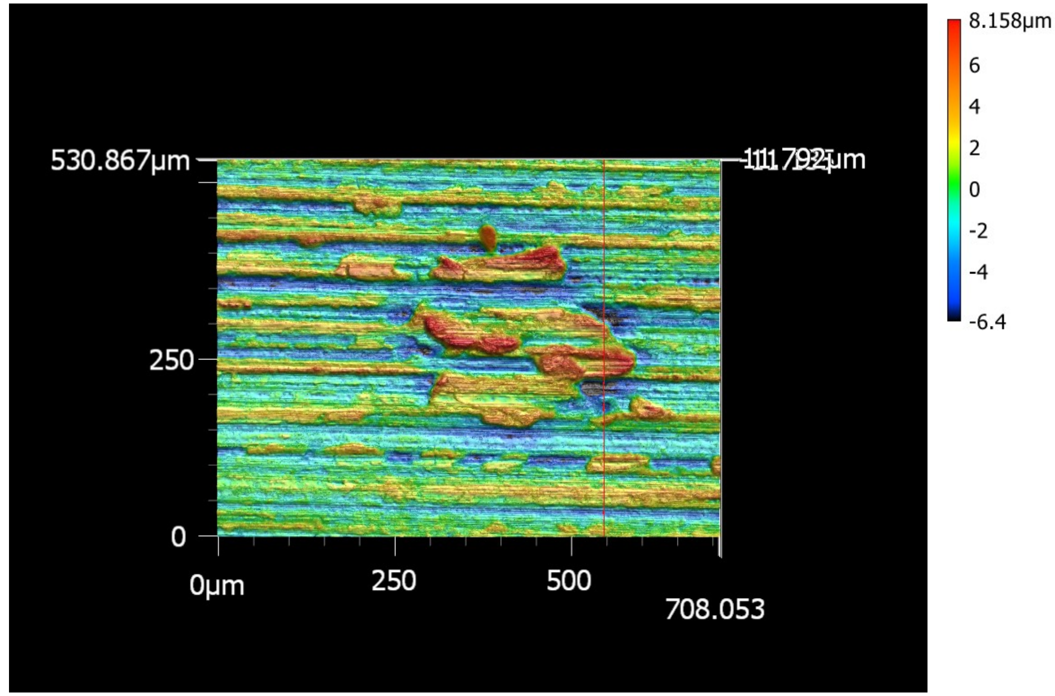
Profile measurement - SS304L_No. 07_35°C_12 Weeks

KEYENCE VK-X3000 Series

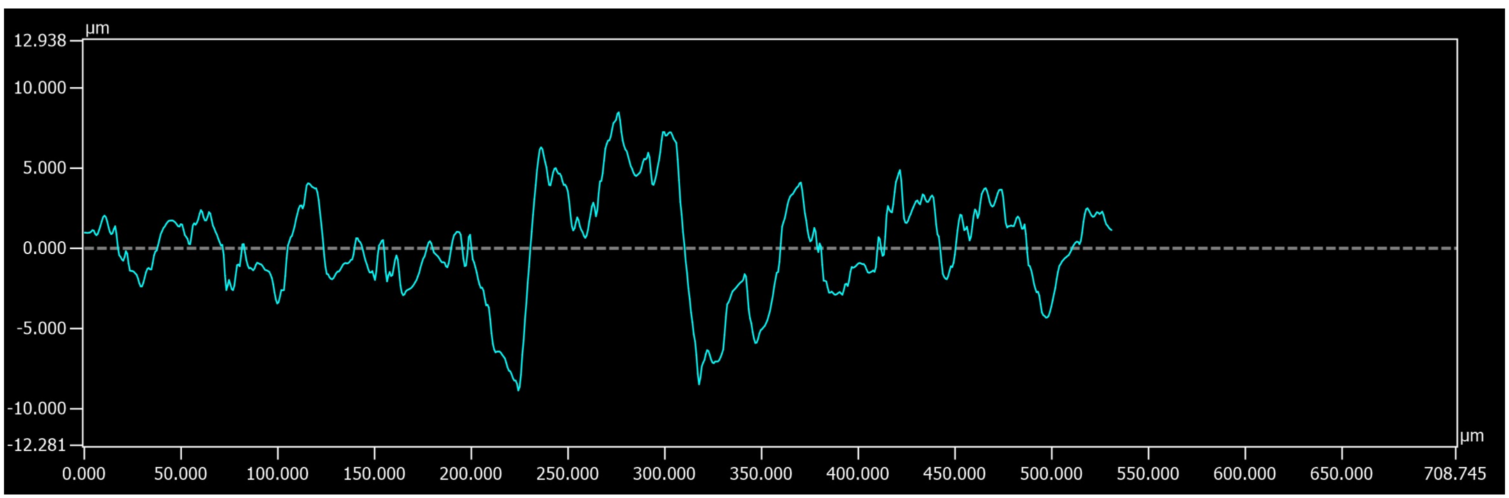
Main image



3D image



Profile

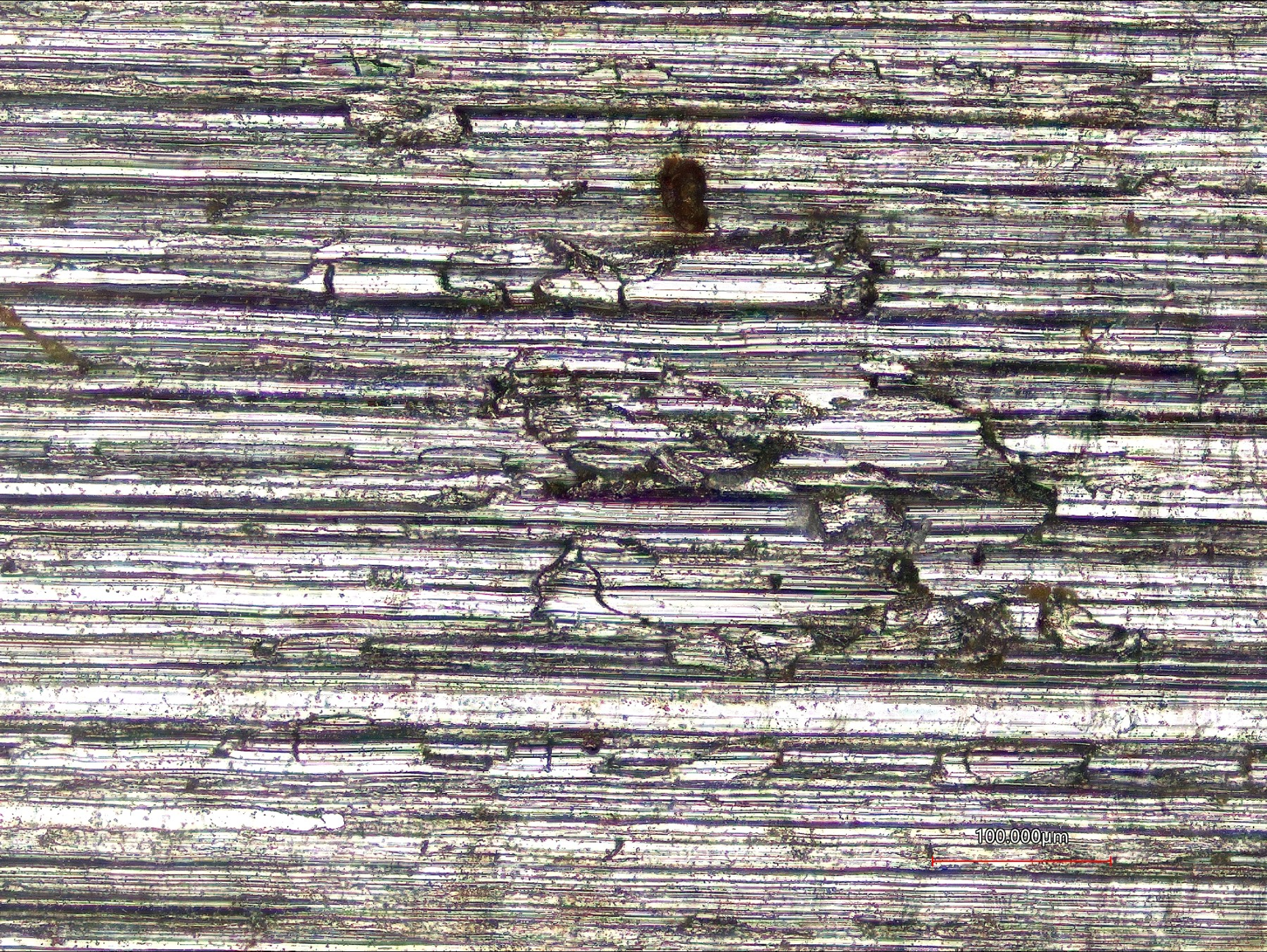


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/11/2022 11:49:40 AM

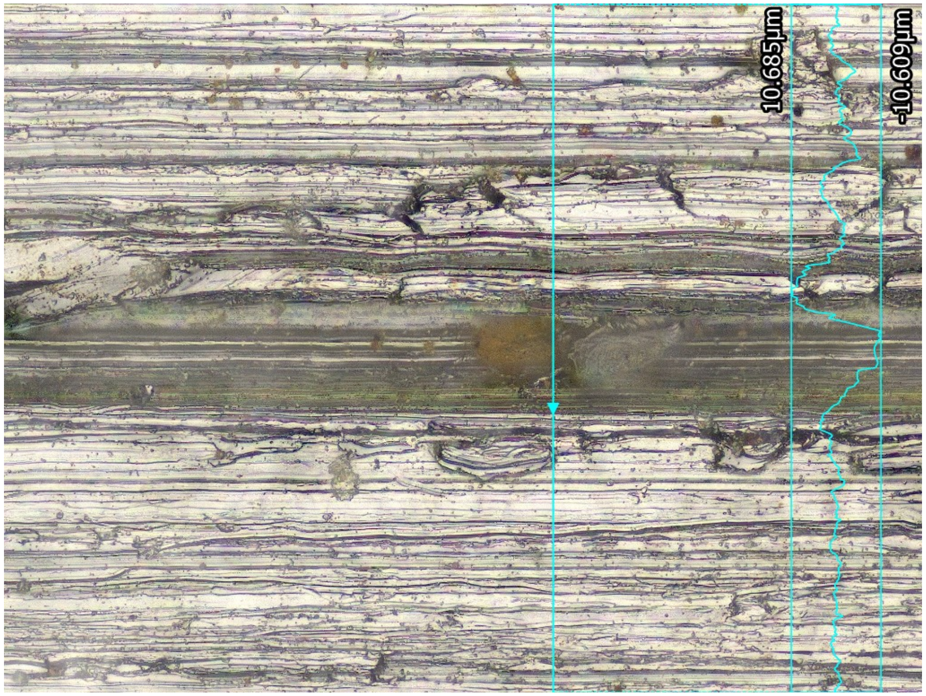
Objective Lens Power： 20X


100.000µm

Profile measurement - SS304_No. 11_35°C_8 Weeks

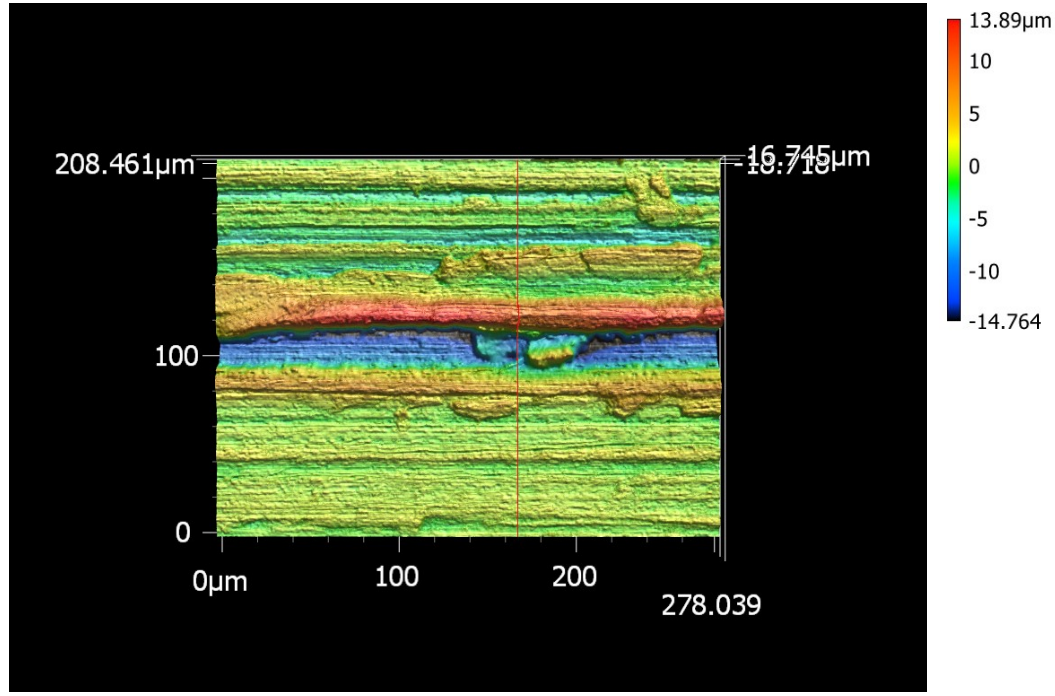
KEYENCE VK-X3000 Series

Main image

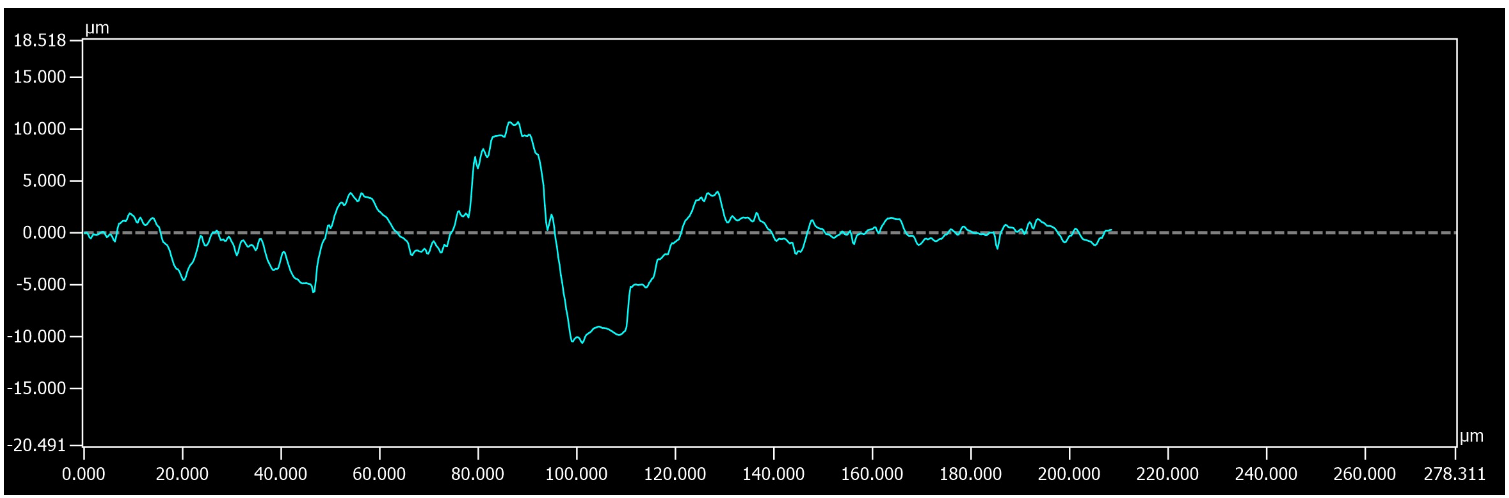


3D image

Height color



Profile

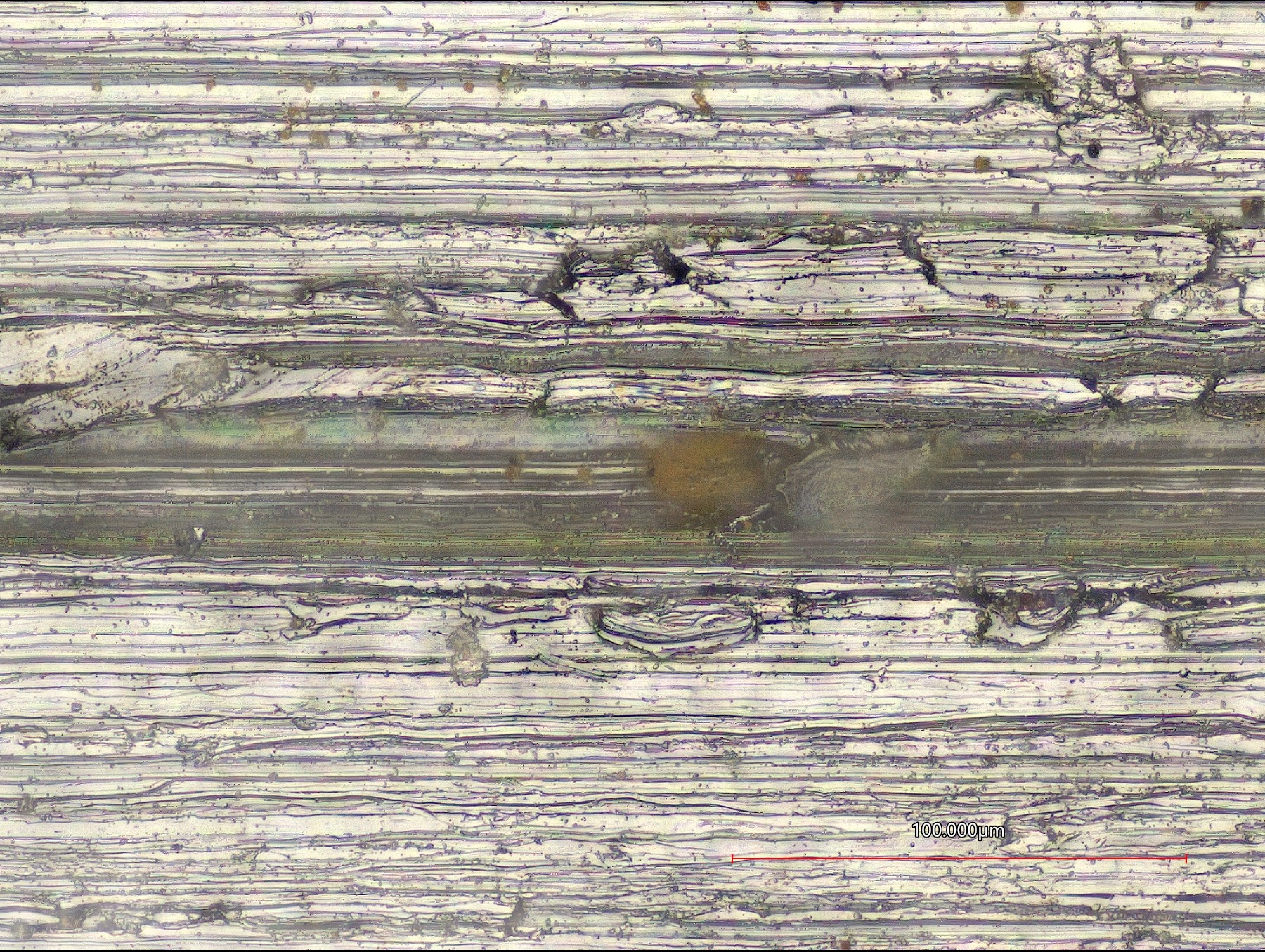


Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/12/2022 11:23:31 AM

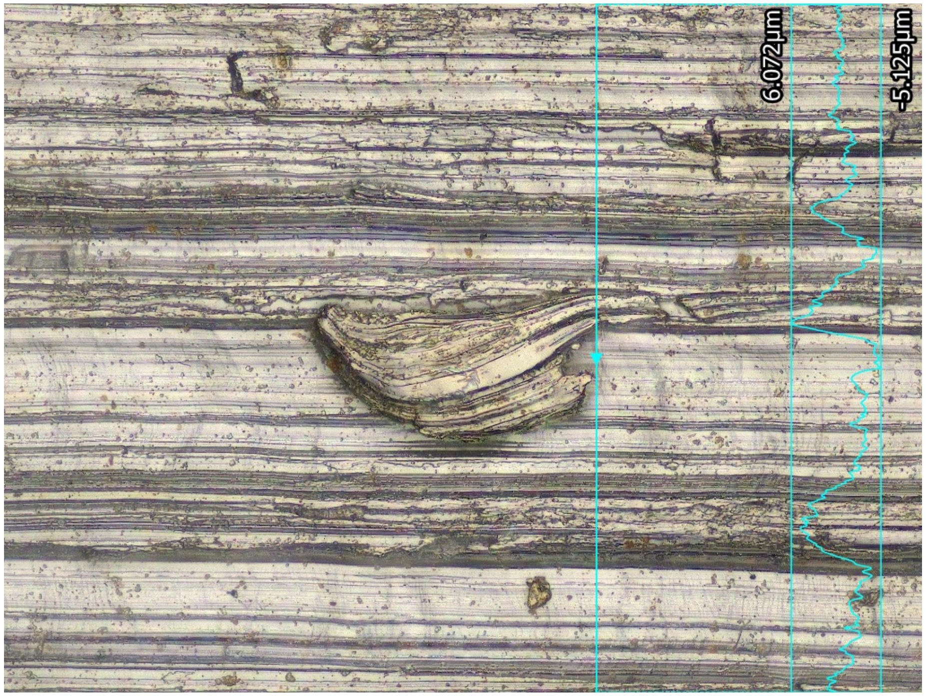
Objective Lens Power： 50X


100.000µm

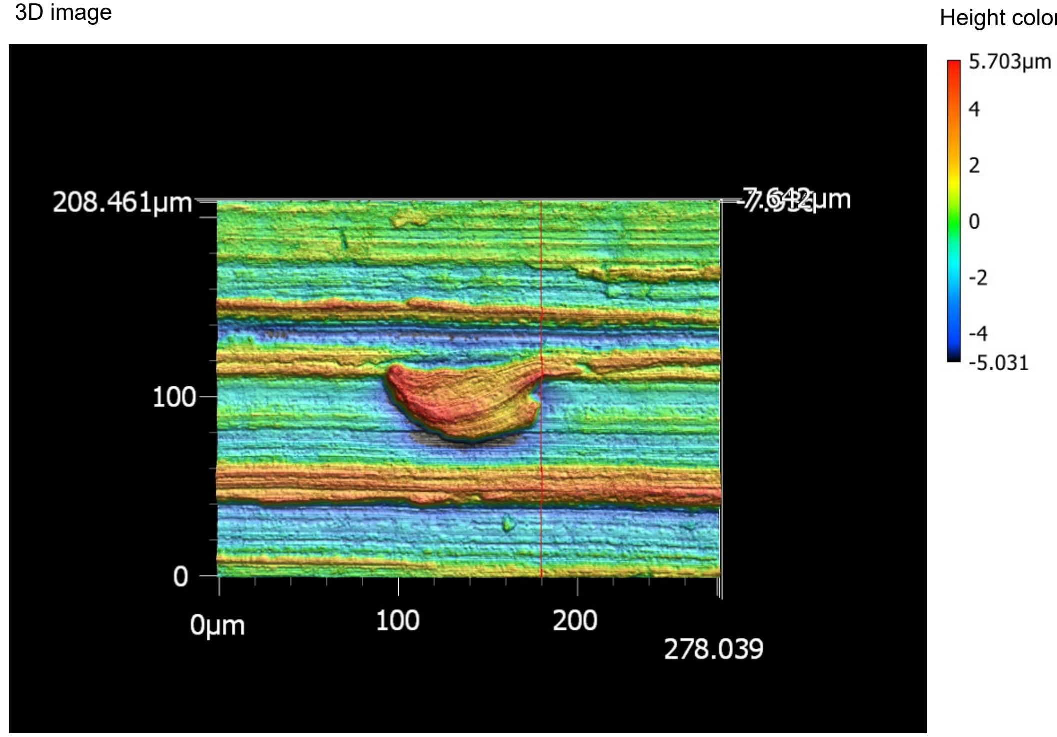
Profile measurement - SS304L_No. 06_35°C_8 Weeks

KEYENCE VK-X3000 Series

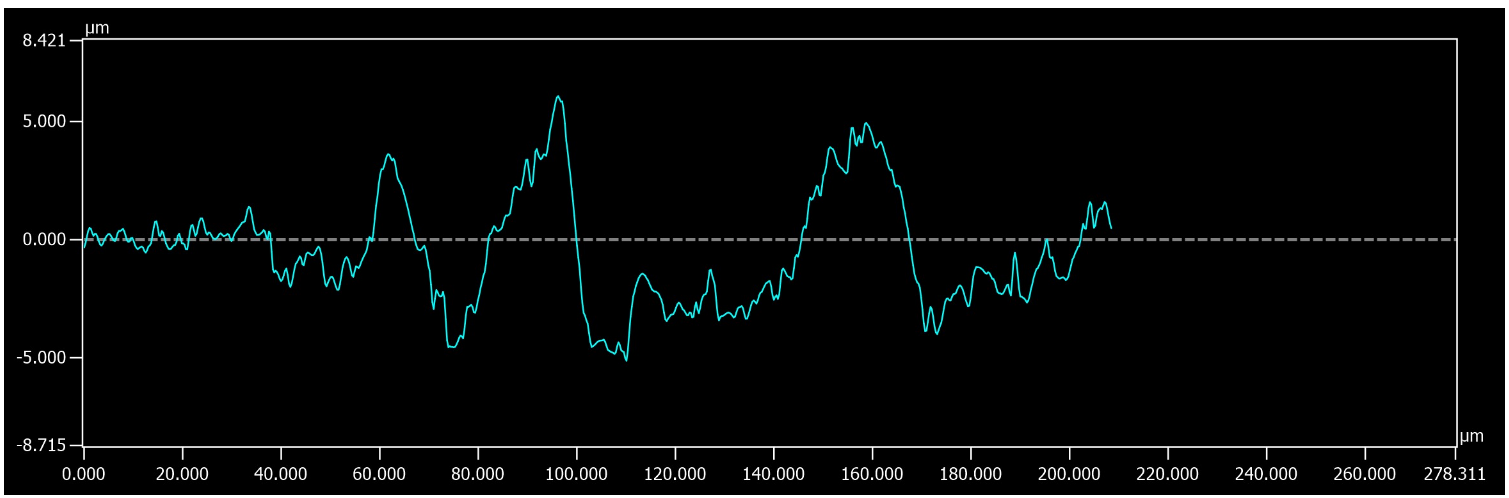
Main image



3D image



Profile



Measurement result

No.	Measurement name	Measured value	Unit

Measured date： 7/12/2022 11:46:38 AM

Objective Lens Power： 50X


100.000µm

Surface Profiles Data of SS304 Samples

SS304_No. 28_50℃_14Wks	
Profile	**Measurement (μm)**
1	-9.866
2	-9.872
3	-10.236
4	-6.135
5	-3.009
Average Dip	**-7.824**
Std. Deviation	**3.171**

SS304_No. 26_50℃_8Wks	
Profile	**Measurement (μm)**
1	-7.813
2	-9.103
3	-8.156
4	-13.452
5	-10.215
6	-6.562
Average Dip	**-9.217**
Std. Deviation	**2.412**

SS304_No. 16_35℃_12Wks	
Profile	**Measurement (μm)**
1	-6.580
2	-12.003
3	-6.072
4	-4.013
5	-11.596
6	-7.740
7	-5.632
8	-4.5
Average Dip	**-7.267**
Std. Deviation	**3.029**

SS304_No. 11_35℃_8Wks	
Profile	**Measurement (μm)**
1	-6.301
2	-7.621
3	-3.934
4	-4.475
5	-6.002
6	-6.905
7	-8.156
8	-6.488
9	-10.249
10	-14.764
Average Dip	**-7.490**
Std. Deviation	**3.121**

Surface Profiles Data of SS304L Samples

SS304L_No. 17_50℃_14Wks	
Profile	**Measurement (μm)**
1	-7.074
2	-8.247
3	-7.619
4	-7.084
5	-8.924
6	-8.026
7	-8.644
8	-10.726
9	-6.355
10	-33.183
11	-4.303
Average Dip	**-10.017**
Std. Deviation	**7.851**

SS304L_No. 14_50℃_8Wks	
Profile	**Measurement (μm)**
1	-12.142
2	-18.386
3	-13.675
4	-19.081
5	-20.543
6	-10.410
7	-11.433
8	-13.863
9	-10.798
10	-8.826
Average Dip	**-13.916**
Std. Deviation	**4.053**

SS304L_No. 07_35℃_12Wks	
Profile	**Measurement (μm)**
1	-7.009
2	-4.298
3	-4.622
4	-5.563
5	-4.008
6	-6.805
7	-6.400
8	-8.216
9	-5.468
Average Dip	**-5.821**
Std. Deviation	**1.400**

SS304L_No. 06_35℃_8Wks	
Profile	**Measurement (μm)**
1	-5.031
2	-5.970
3	-10.576
4	-7.902
5	-11.019
6	-9.606
7	-7.262
8	-8.391
9	-5.797
10	-8.277
Average Dip	**-7.983**
Std. Deviation	**2.025**

Surface Profiles Data of SS304W Samples

SS304W_No. 38_50℃_14Wks	
Profile	**Measurement (μm)**
1	-4.640
2	-8.893
3	-15.645
4	-5.357
5	-5.541
6	-2.730
7	-4.389
8	-5.268
9	-5.930
10	-3.217
Average Dip	**-6.161**
Std. Deviation	**3.730**

SS304W_No. 35_50℃_8Wks	
Profile	**Measurement (μm)**
1	-13.510
2	-6.671
3	-7.112
4	-5.960
5	-6.359
6	-5.715
7	-5.749
8	-6.679
Average Dip	**-7.219**
Std. Deviation	**2.589**

SS304W_No. 27_35℃_12Wks	
Profile	**Measurement (μm)**
1	-5.422
2	-5.528
3	-4.985
4	-4.313
5	-4.690
6	-5.369
7	-2.949
8	-4.405
9	-3.016
10	-6.086
11	-3.42
12	-3.286
Average Dip	**-4.456**
Std. Deviation	**1.075**

SS304W_No. 26_35℃_8Wks	
Profile	**Measurement (μm)**
1	-4.452
2	-7.683
3	-6.754
4	-4.861
5	-5.528
6	-4.749
7	-5.087
8	-5.699
9	-3.087
10	-6.659
11	-6.548
Average Dip	**-5.555**
Std. Deviation	**1.298**

Surface Profiles Data of SS304LW Samples

SS304LW_No. 16_50℃_14Wks	
Profile	**Measurement (œm)**
1	-8.530
2	-7.337
3	-10.057
4	-4.087
5	-9.041
6	-7.879
7	-8.890
8	-8.464
9	-12.818
10	-9.315
11	-5.076
12	-4.711
Average Dip	**-8.017**
Std. Deviation	**2.462**

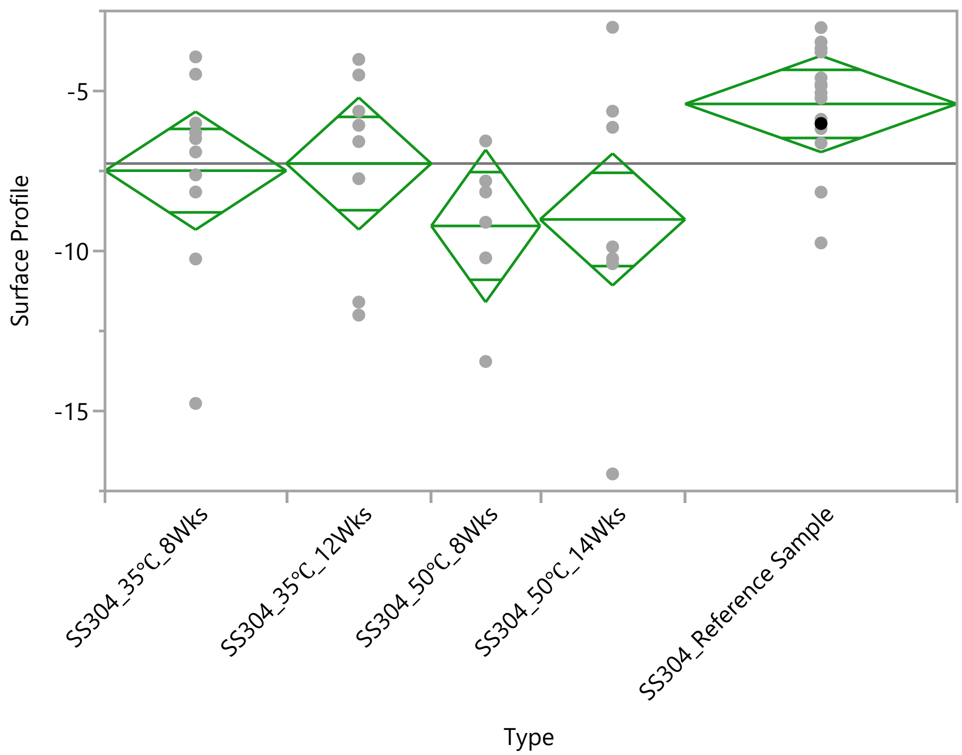
SS304LW_No. 13_50℃_8Wks	
Profile	**Measurement (œm)**
1	-10.474
2	-12.179
3	-10.915
4	-6.854
5	-9.780
6	-10.339
7	-11.059
8	-9.606
9	-19.917
Average Dip	**-11.236**
Std. Deviation	**3.566**

SS304LW_No. 08_35℃_12Wks	
Profile	**Measurement (œm)**
1	-6.221
2	-7.535
3	-6.693
4	-3.856
5	-6.412
6	-6.619
7	-5.847
8	-7.396
9	-2.618
10	-6.828
11	-3.417
Average Dip	**-5.767**
Std. Deviation	**1.680**

SS304LW_No. 06_35℃_8Wks	
Profile	**Measurement (œm)**
1	-8.454
2	-4.640
3	-2.795
4	-4.171
5	-6.695
6	-5.086
7	-4.372
8	-6.246
9	-6.311
10	-8.396
Average Dip	**-5.717**
Std. Deviation	**1.842**

SS304 & Reference Sample Comparison

Oneway Analysis of Surface Profile By Type



Oneway Anova

Summary of Fit

Rsquare	0.221534
Adj Rsquare	0.147394
Root Mean Square Error	2.889434
Mean of Response	-7.26606
Observations (or Sum Wgts)	47

Analysis of Variance

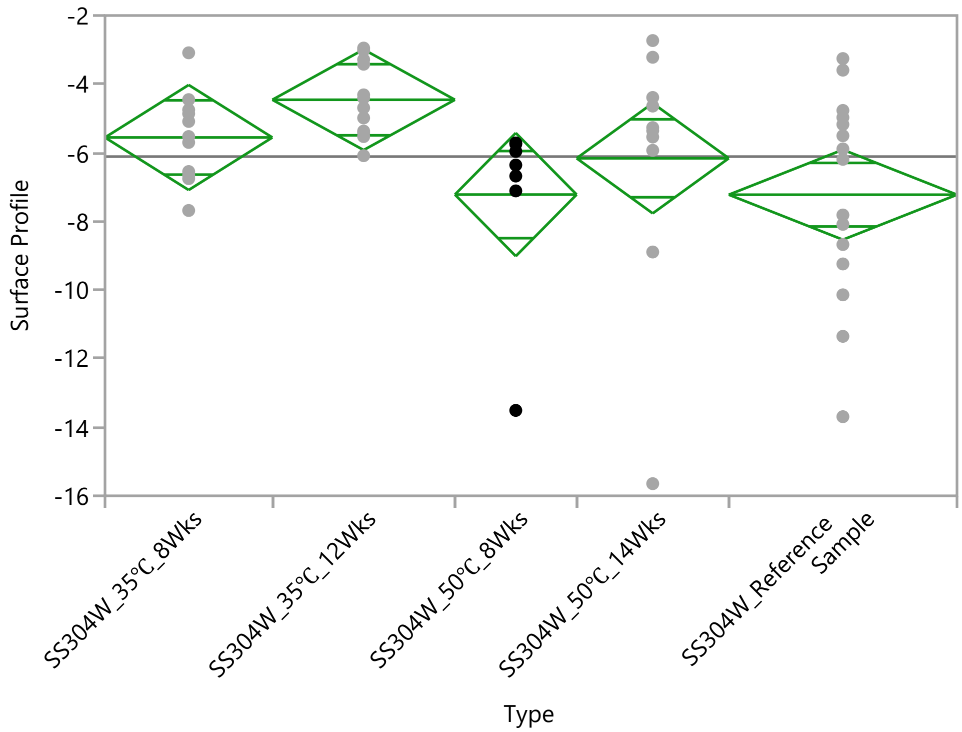
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Type	4	99.78722	24.9468	2.9881	0.0294*
Error	42	350.65077	8.3488		
C. Total	46	450.43800			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
SS304_35°C_8Wks	10	-7.4895	0.9137	-9.33	-5.646
SS304_35°C_12Wks	8	-7.2670	1.0216	-9.33	-5.205
SS304_50°C_8Wks	6	-9.2168	1.1796	-11.60	-6.836
SS304_50°C_14Wks	8	-9.0141	1.0216	-11.08	-6.953
SS304_Reference Sample	15	-5.4040	0.7460	-6.91	-3.898

Std Error uses a pooled estimate of error variance

Oneway Analysis of Surface Profile By Type



Oneway Anova

Summary of Fit

Rsquare	0.165407
Adj Rsquare	0.099949
Root Mean Square Error	2.529776
Mean of Response	-6.11232
Observations (or Sum Wgts)	56

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Type	4	64.68642	16.1716	2.5269	0.0519
Error	51	326.38811	6.3998		
C. Total	55	391.07453			

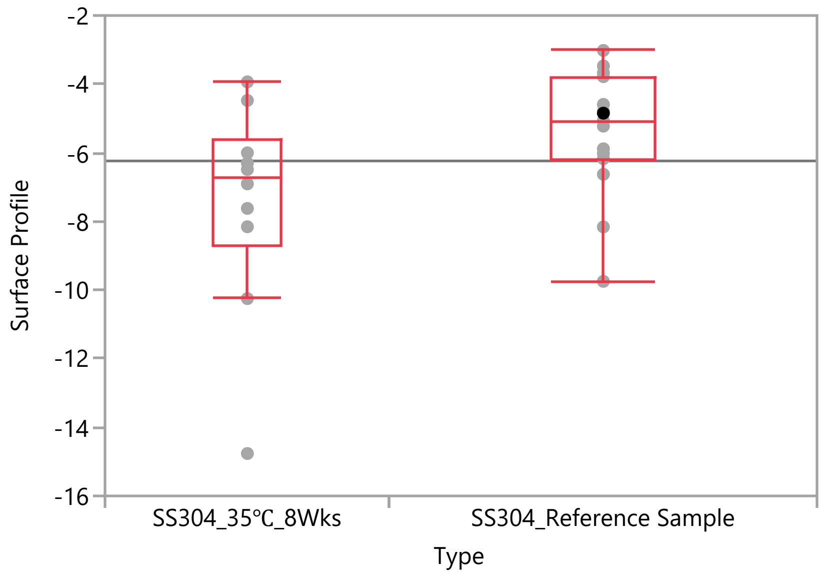
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
SS304W_35°C_8Wks	11	-5.5552	0.76276	-7.086	-4.024
SS304W_35°C_12Wks	12	-4.4558	0.73028	-5.922	-2.990
SS304W_50°C_8Wks	8	-7.2194	0.89441	-9.015	-5.424
SS304W_50°C_14Wks	10	-6.1610	0.79999	-7.767	-4.555
SS304W_Reference Sample	15	-7.2233	0.65319	-8.535	-5.912

Std Error uses a pooled estimate of error variance

SS304 35℃_ 8Wks vs. SS304_Reference

Oneway Analysis of Surface Profile By Type

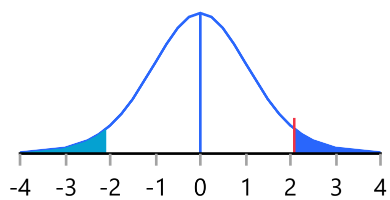


t Test

SS304_Reference Sample-SS304_35°C_8Wks

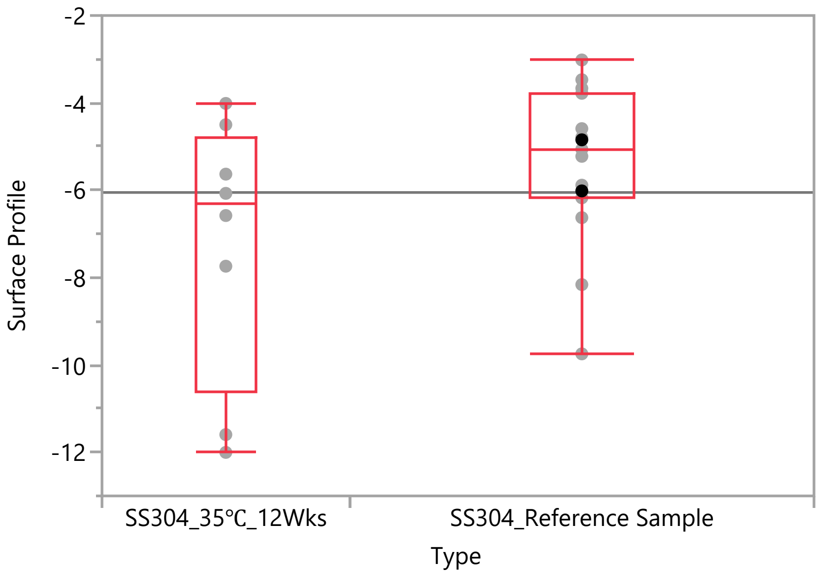
Assuming unequal variances

Difference	2.0855	t Ratio	1.910011
Std Err Dif	1.0919	DF	13.05443
Upper CL Dif	4.4434	Prob > |t|	0.0783
Lower CL Dif	-0.2724	Prob > t	0.0392*
Confidence	0.95	Prob < t	0.9608



SS304 35℃_ 12Wks vs. SS304_Reference

Oneway Analysis of Surface Profile By Type

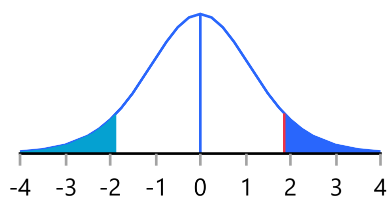


t Test

SS304_Reference Sample-SS304_35°C_12Wks

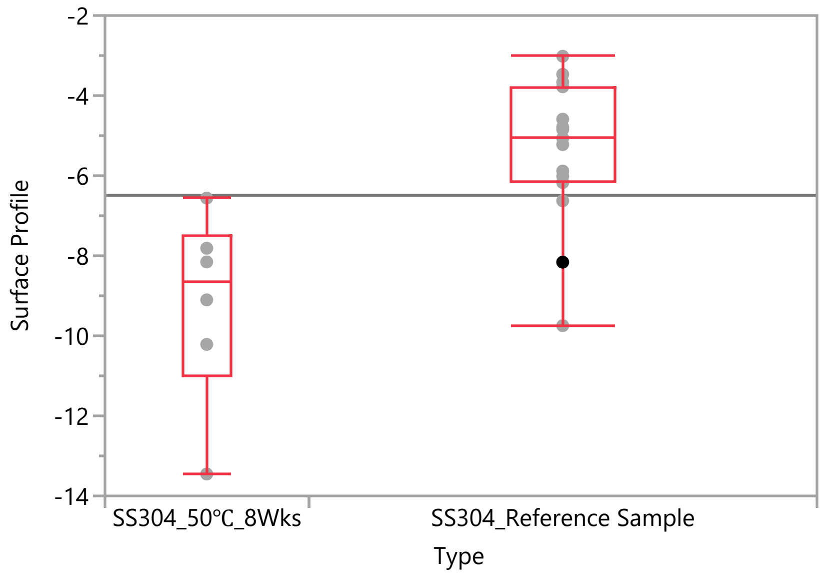
Assuming unequal variances

Difference	1.8630	t Ratio	1.594893
Std Err Dif	1.1681	DF	9.736533
Upper CL Dif	4.4753	Prob > |t|	0.1426
Lower CL Dif	-0.7493	Prob > t	0.0713
Confidence	0.95	Prob < t	0.9287



SS304 50℃_ 8Wks vs. SS304_Reference

Oneway Analysis of Surface Profile By Type

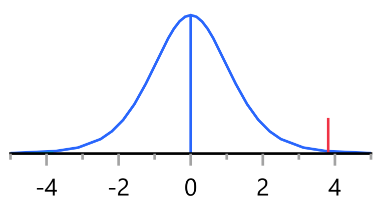


t Test

SS304_Reference Sample-SS304_50℃_8Wks

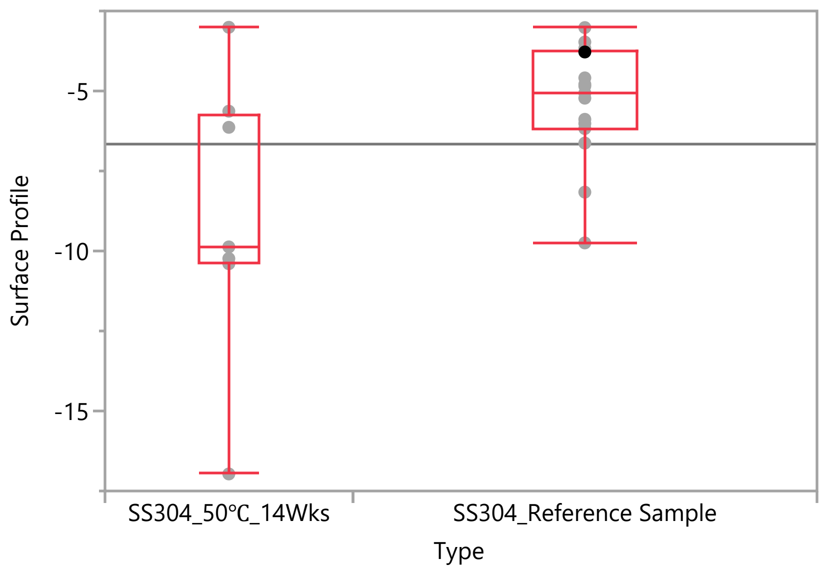
Assuming unequal variances

Difference	3.81283	t Ratio	3.498894
Std Err Dif	1.08973	DF	7.365974
Upper CL Dif	6.36390	Prob > |t|	0.0092*
Lower CL Dif	1.26177	Prob > t	0.0046*
Confidence	0.95	Prob < t	0.9954



SS304 50℃_ 14Wks vs. SS304_Reference

Oneway Analysis of Surface Profile By Type

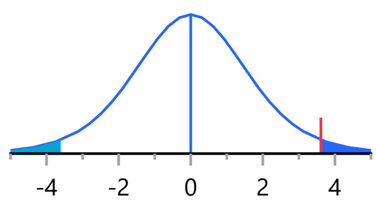


t Test

SS304_Reference Sample-SS304_50°C_14Wks

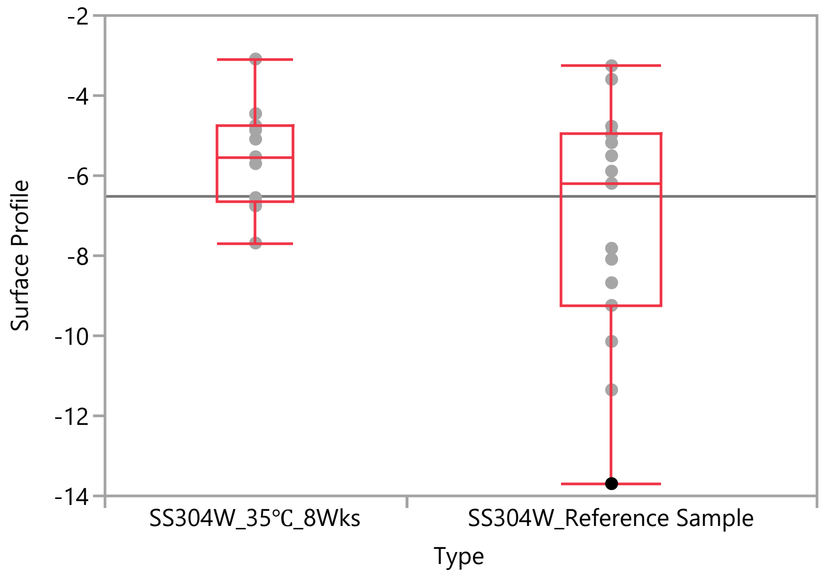
Assuming unequal variances

Difference	3.61013	t Ratio	2.315641
Std Err Dif	1.55902	DF	8.40533
Upper CL Dif	7.17527	Prob > |t|	0.0478*
Lower CL Dif	0.04498	Prob > t	0.0239*
Confidence	0.95	Prob < t	0.9761



SS304W 35℃_ 8Wks vs. SS304W_Reference

Oneway Analysis of Surface Profile By Type

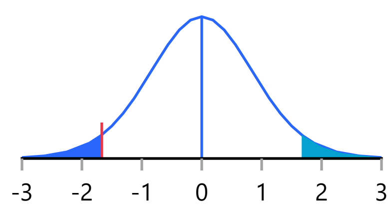


t Test

SS304W_Reference Sample-SS304W_35℃_8Wks

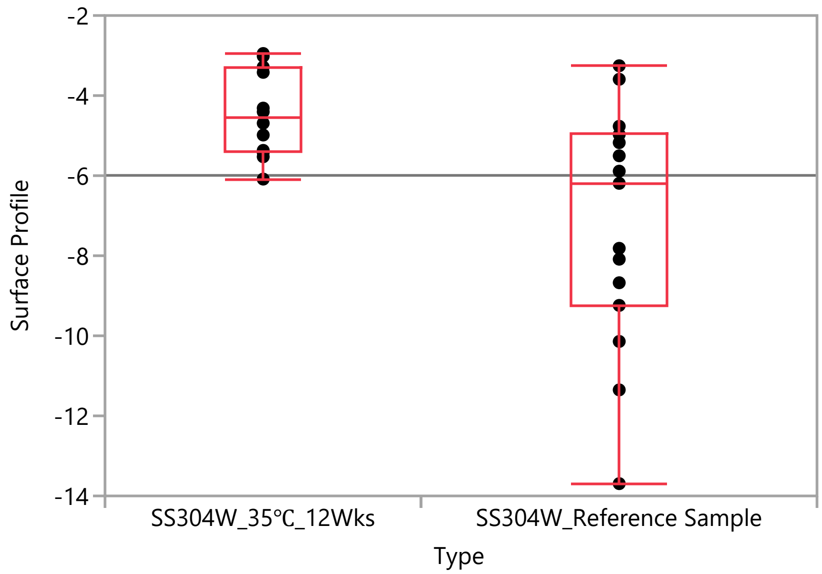
Assuming unequal variances

Difference	-1.6681	t Ratio	-1.92996
Std Err Dif	0.8643	DF	20.26626
Upper CL Dif	0.1333	Prob > |t|	0.0677
Lower CL Dif	-3.4695	Prob > t	0.9661
Confidence	0.95	Prob < t	0.0339*



SS304W 35℃_ 12Wks vs. SS304W_Reference

Oneway Analysis of Surface Profile By Type

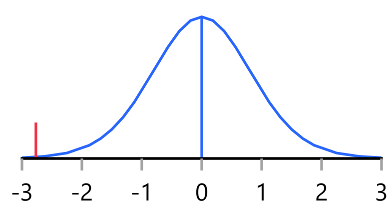


t Test

SS304W_Reference Sample-SS304W_35℃_12Wks

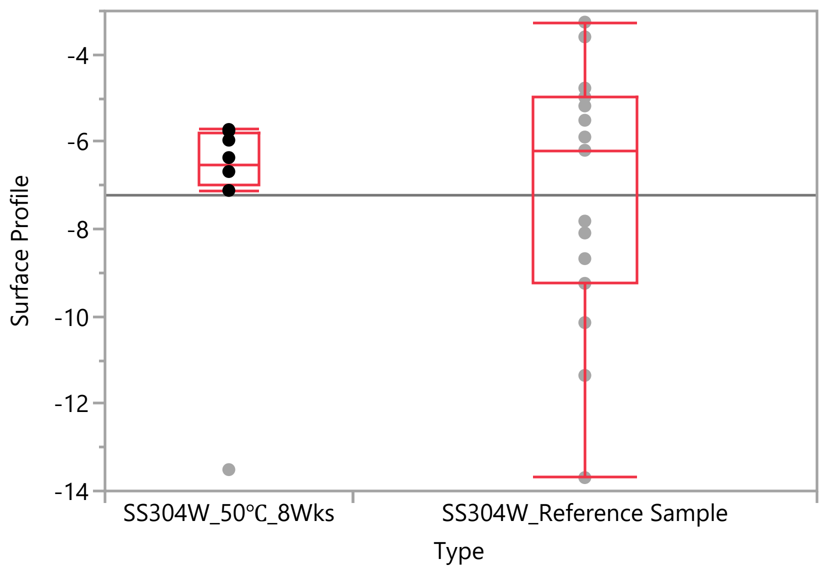
Assuming unequal variances

Difference	-2.7675	t Ratio	-3.33123
Std Err Dif	0.8308	DF	18.29809
Upper CL Dif	-1.0241	Prob > |t|	0.0037*
Lower CL Dif	-4.5109	Prob > t	0.9982
Confidence	0.95	Prob < t	0.0018*



SS304W 50℃_ 8Wks vs. SS304W_Reference

Oneway Analysis of Surface Profile By Type

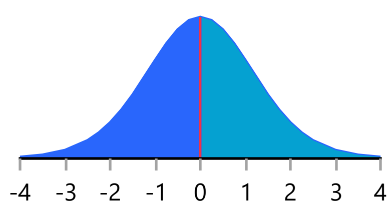


t Test

SS304W_Reference Sample-SS304W_50℃_8Wks

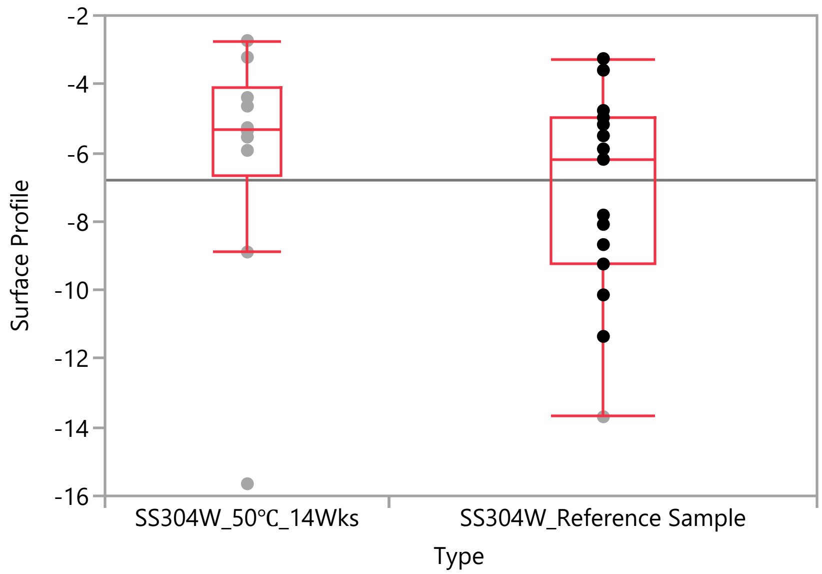
Assuming unequal variances

Difference	-0.0039	t Ratio	-0.00325
Std Err Dif	1.1966	DF	16.33493
Upper CL Dif	2.5286	Prob > |t|	0.9974
Lower CL Dif	-2.5363	Prob > t	0.5013
Confidence	0.95	Prob < t	0.4987



SS304W 50℃_ 14Wks vs. SS304W_Reference

Oneway Analysis of Surface Profile By Type

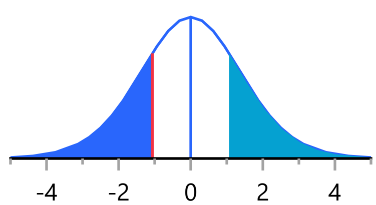


t Test

SS304W_Reference Sample-SS304W_50℃_14Wks

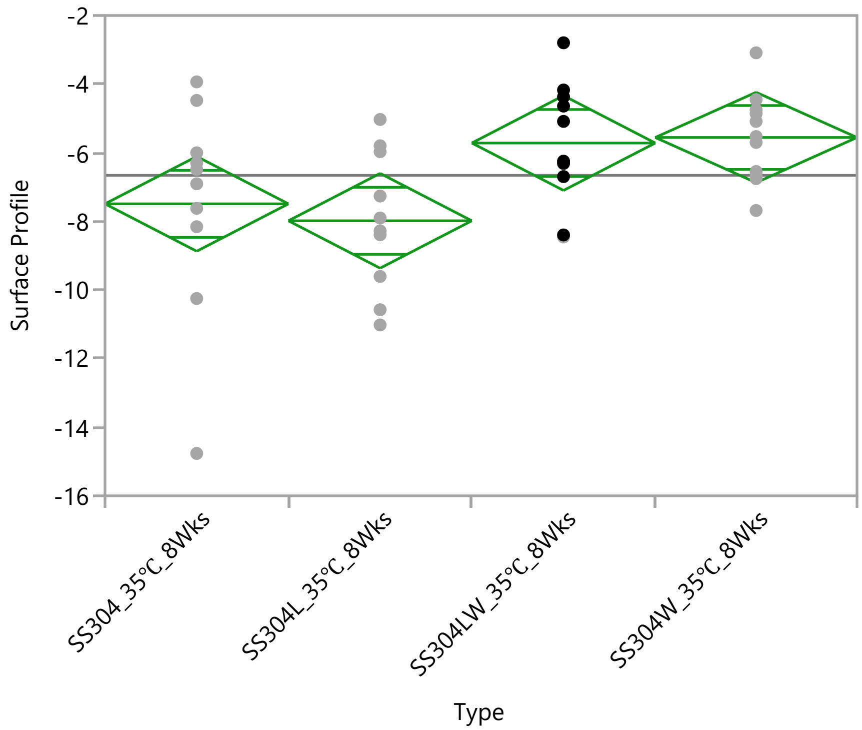
Assuming unequal variances

Difference	-1.0623	t Ratio	-0.75401
Std Err Dif	1.4088	DF	16.40338
Upper CL Dif	1.9183	Prob > |t|	0.4615
Lower CL Dif	-4.0429	Prob > t	0.7692
Confidence	0.95	Prob < t	0.2308



Material Comparisons (35℃_8Wks)

Oneway Analysis of Surface Profile By Type



Oneway Anova

Summary of Fit

Rsquare	0.213587
Adj Rsquare	0.149824
Root Mean Square Error	2.156046
Mean of Response	-6.65851
Observations (or Sum Wgts)	41

Analysis of Variance

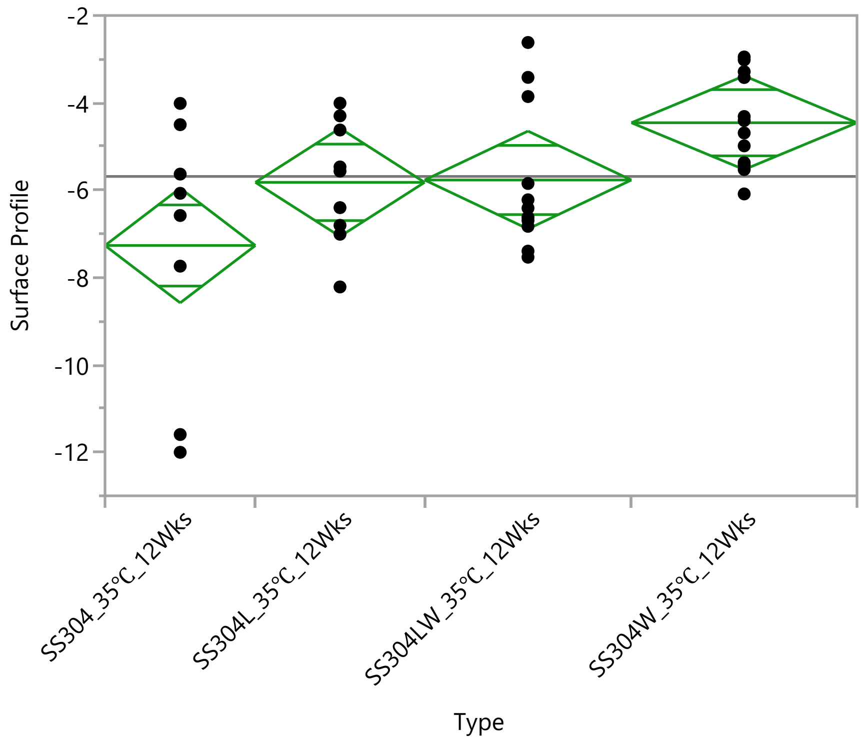
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Type	3	46.71344	15.5711	3.3497	0.0292*
Error	37	171.99570	4.6485		
C. Total	40	218.70914			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
SS304_35°C_8Wks	10	-7.4895	0.68180	-8.871	-6.108
SS304L_35°C_8Wks	10	-7.9831	0.68180	-9.365	-6.602
SS304LW_35°C_8Wks	10	-5.7166	0.68180	-7.098	-4.335
SS304W_35°C_8Wks	11	-5.5552	0.65007	-6.872	-4.238

Std Error uses a pooled estimate of error variance

Oneway Analysis of Surface Profile By Type



Oneway Anova

Summary of Fit

Rsquare	0.241151
Adj Rsquare	0.177913
Root Mean Square Error	1.831991
Mean of Response	-5.6859
Observations (or Sum Wgts)	40

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Type	3	38.39568	12.7986	3.8134	0.0180*
Error	36	120.82285	3.3562		
C. Total	39	159.21853			

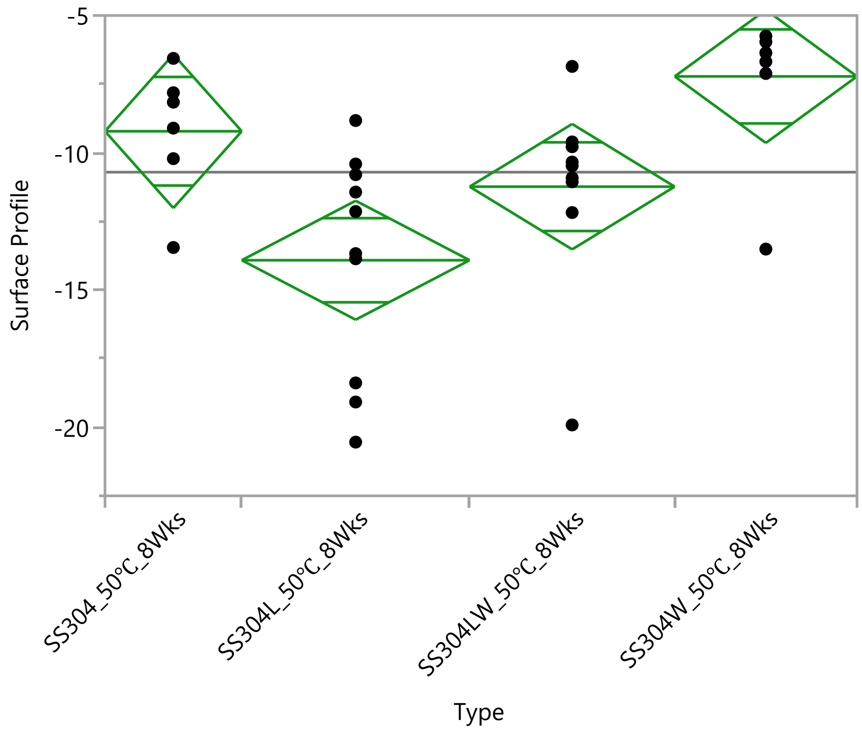
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
SS304_35°C_12Wks	8	-7.2670	0.64771	-8.581	-5.953
SS304L_35°C_12Wks	9	-5.8210	0.61066	-7.059	-4.583
SS304LW_35°C_12Wks	11	-5.7675	0.55237	-6.888	-4.647
SS304W_35°C_12Wks	12	-4.4558	0.52885	-5.528	-3.383

Std Error uses a pooled estimate of error variance

Material Comparisons (50℃_8Wks)

Oneway Analysis of Surface Profile By Type



Oneway Anova

Summary of Fit

Rsquare	0.398911
Adj Rsquare	0.336729
Root Mean Square Error	3.350943
Mean of Response	-10.7072
Observations (or Sum Wgts)	33

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Type	3	216.10687	72.0356	6.4152	0.0018*
Error	29	325.63578	11.2288		
C. Total	32	541.74265			

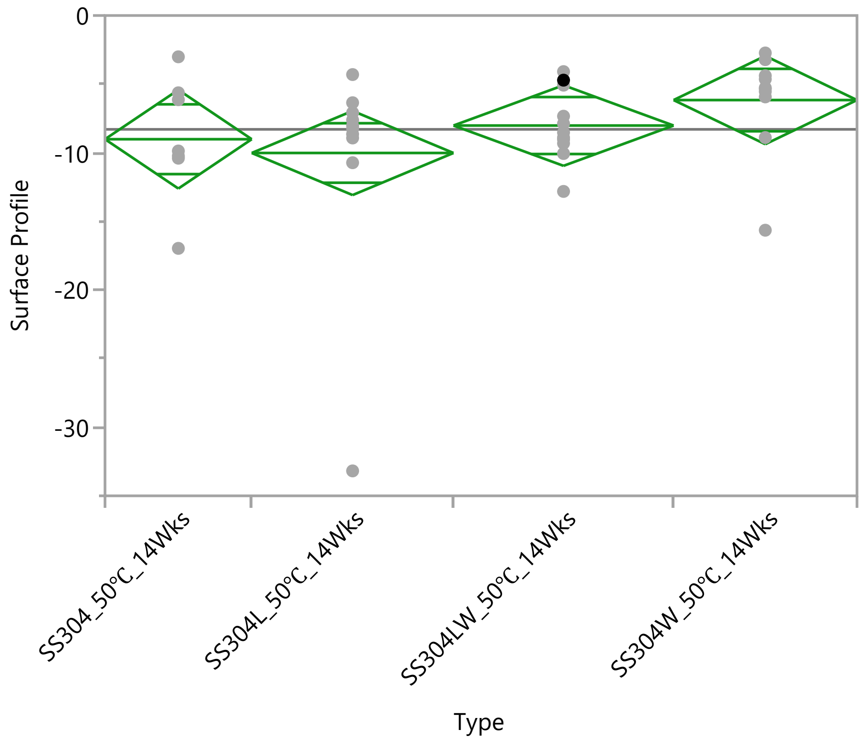
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
SS304_50°C_8Wks	6	-9.217	1.3680	-12.01	-6.42
SS304L_50°C_8Wks	10	-13.916	1.0597	-16.08	-11.75
SS304LW_50°C_8Wks	9	-11.236	1.1170	-13.52	-8.95
SS304W_50°C_8Wks	8	-7.219	1.1847	-9.64	-4.80

Std Error uses a pooled estimate of error variance

Material Comparisons (50℃_14Wks)

Oneway Analysis of Surface Profile By Type



Oneway Anova

Summary of Fit

Rsquare	0.081952
Adj Rsquare	0.007516
Root Mean Square Error	5.019389
Mean of Response	-8.29544
Observations (or Sum Wgts)	41

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Type	3	83.2148	27.7383	1.1010	0.3610
Error	37	932.1880	25.1943		
C. Total	40	1015.4027			

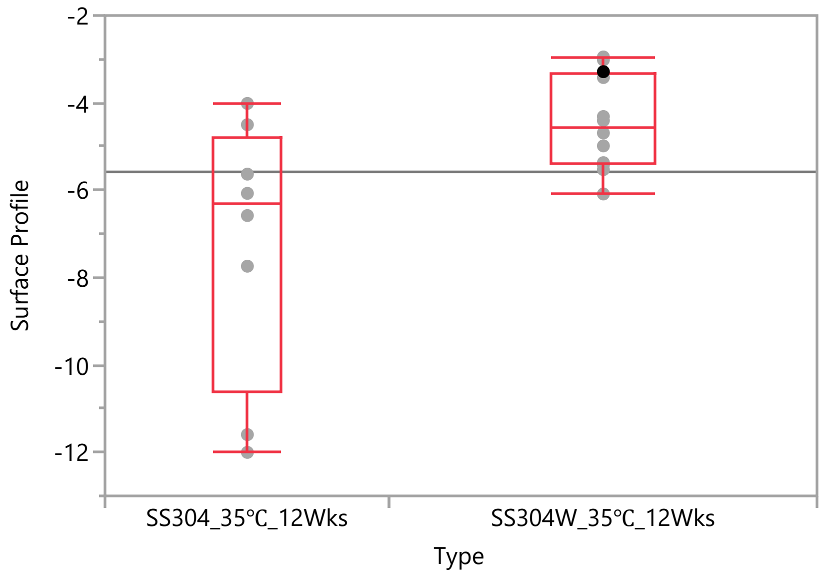
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
SS304_50°C_14Wks	8	-9.014	1.7746	-12.61	-5.418
SS304L_50°C_14Wks	11	-10.017	1.5134	-13.08	-6.950
SS304LW_50°C_14Wks	12	-8.017	1.4490	-10.95	-5.081
SS304W_50°C_14Wks	10	-6.161	1.5873	-9.38	-2.945

Std Error uses a pooled estimate of error variance

304_35℃_12Wks vs. 304W_35℃_12Wks

Oneway Analysis of Surface Profile By Type

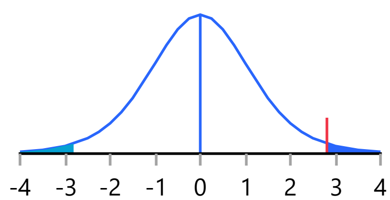


t Test

SS304W_35℃_12Wks-SS304_35℃_12Wks

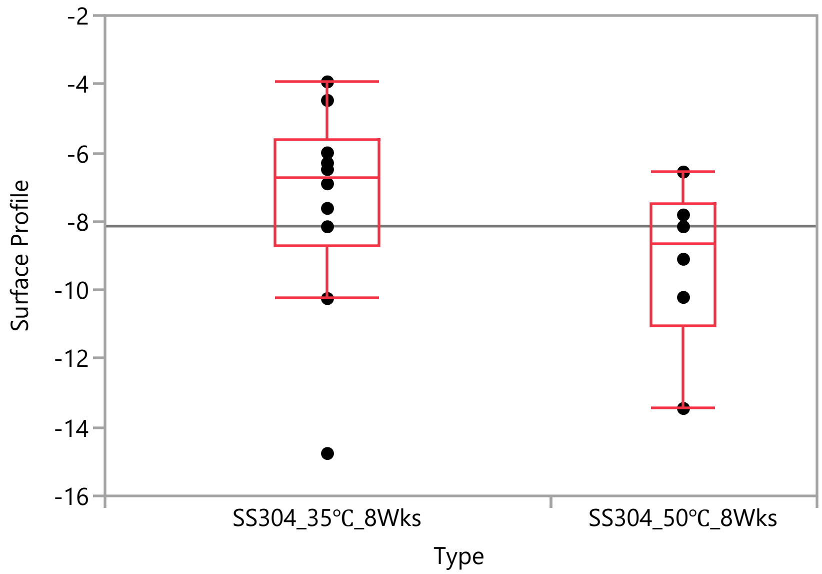
Assuming unequal variances

Difference	2.81125	t Ratio	2.521548
Std Err Dif	1.11489	DF	8.188942
Upper CL Dif	5.37190	Prob > |t|	0.0351*
Lower CL Dif	0.25060	Prob > t	0.0175*
Confidence	0.95	Prob < t	0.9825



SS304 Temperature Effect (8 weeks)

Oneway Analysis of Surface Profile By Type

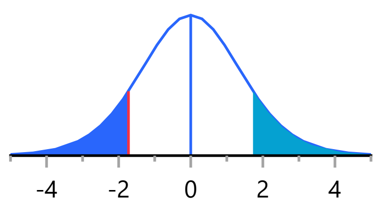


t Test

SS304_50°C_8Wks-SS304_35°C_8Wks

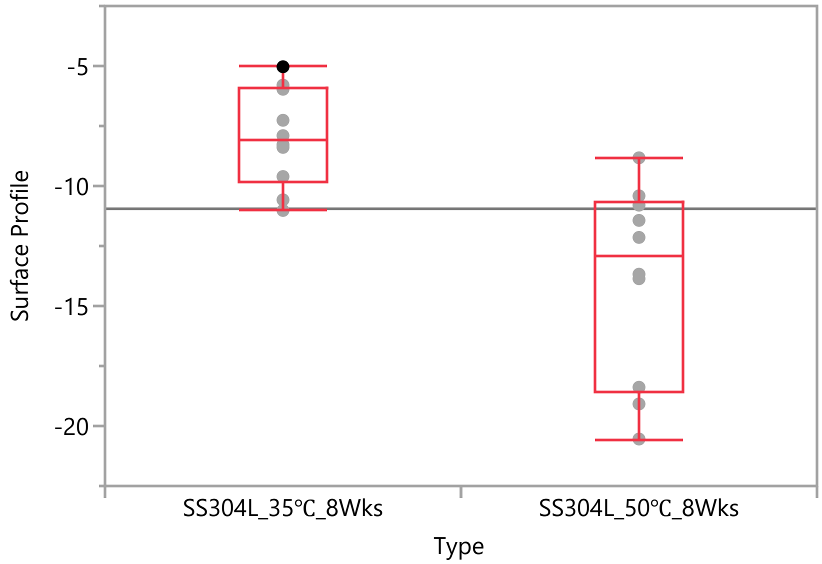
Assuming unequal variances

Difference	-1.7273	t Ratio	-1.23886
Std Err Dif	1.3943	DF	12.87484
Upper CL Dif	1.2878	Prob > |t|	0.2375
Lower CL Dif	-4.7425	Prob > t	0.8812
Confidence	0.95	Prob < t	0.1188



SS304L Temperature Effect (8 weeks)

Oneway Analysis of Surface Profile By Type

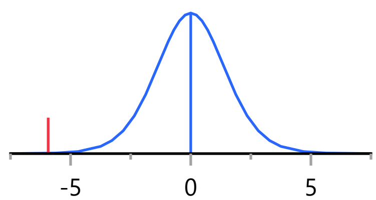


t Test

SS304L_50℃_8Wks-SS304L_35℃_8Wks

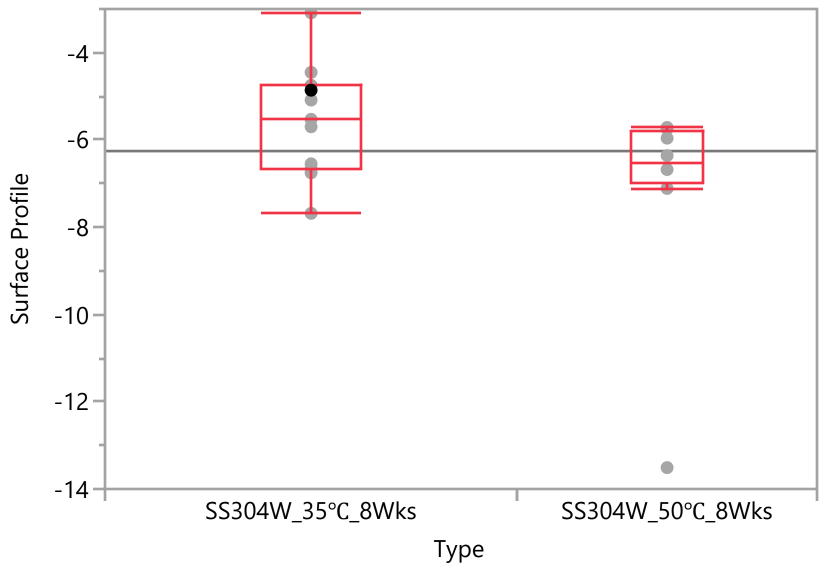
Assuming unequal variances

Difference	-5.9326	t Ratio	-4.14048
Std Err Dif	1.4328	DF	13.2284
Upper CL Dif	-2.8426	Prob > |t|	0.0011*
Lower CL Dif	-9.0226	Prob > t	0.9994
Confidence	0.95	Prob < t	0.0006*



SS304W Temperature Effect (8 weeks)

Oneway Analysis of Surface Profile By Type

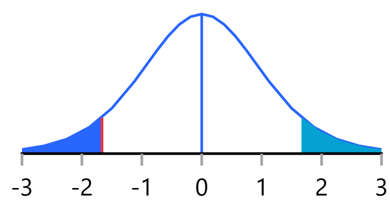


t Test

SS304W_50°C_8Wks-SS304W_35°C_8Wks

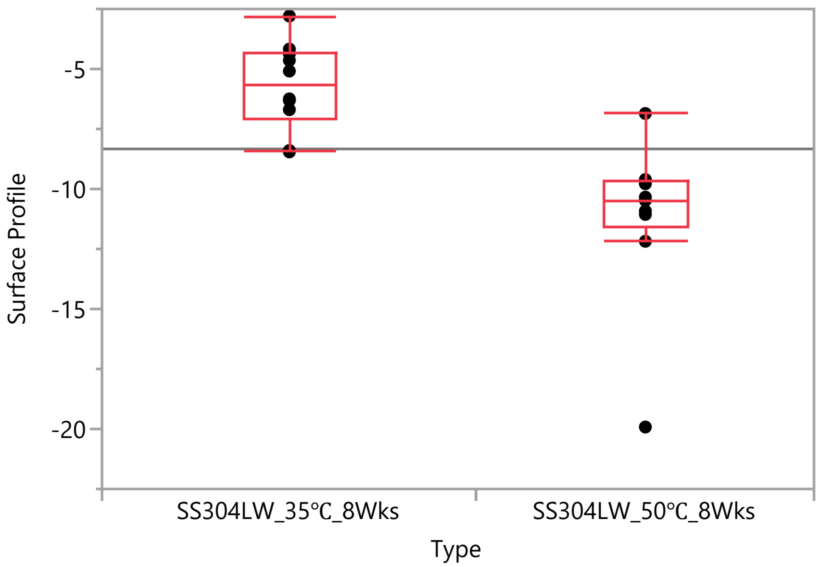
Assuming unequal variances

Difference	-1.6642	t Ratio	-1.67158
Std Err Dif	0.9956	DF	9.569256
Upper CL Dif	0.5677	Prob > |t|	0.1269
Lower CL Dif	-3.8961	Prob > t	0.9365
Confidence	0.95	Prob < t	0.0635



SS304LW Temperature Effect (8 weeks)

Oneway Analysis of Surface Profile By Type

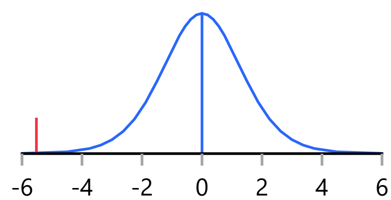


t Test

SS304LW_50°C_8Wks-SS304LW_35°C_8Wks

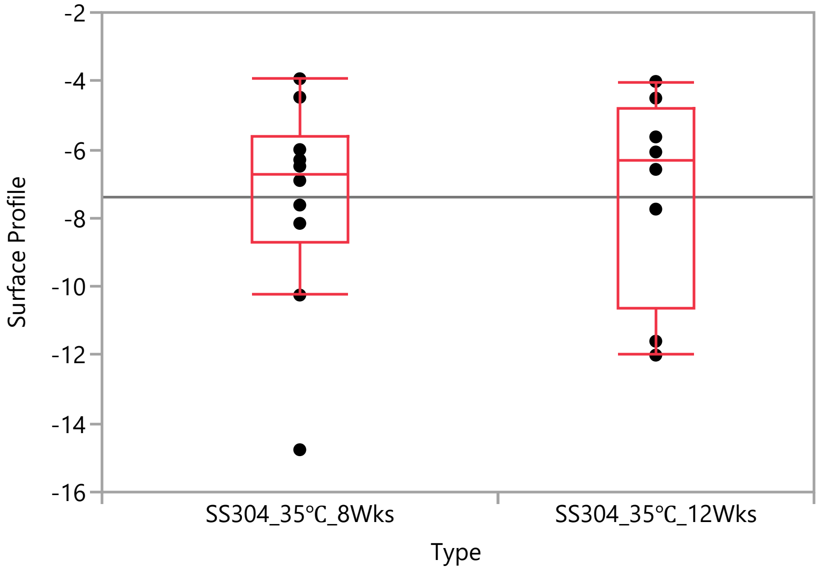
Assuming unequal variances

Difference	-5.5193	t Ratio	-4.16906
Std Err Dif	1.3239	DF	11.70484
Upper CL Dif	-2.6267	Prob > |t|	0.0014*
Lower CL Dif	-8.4118	Prob > t	0.9993
Confidence	0.95	Prob < t	0.0007*



SS304 Exposure Duration Effect (35℃)

Oneway Analysis of Surface Profile By Type

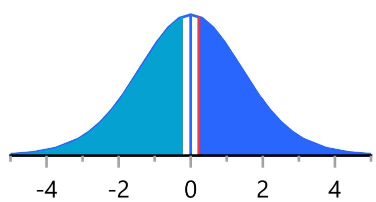


t Test

SS304_35°C_12Wks-SS304_35°C_8Wks

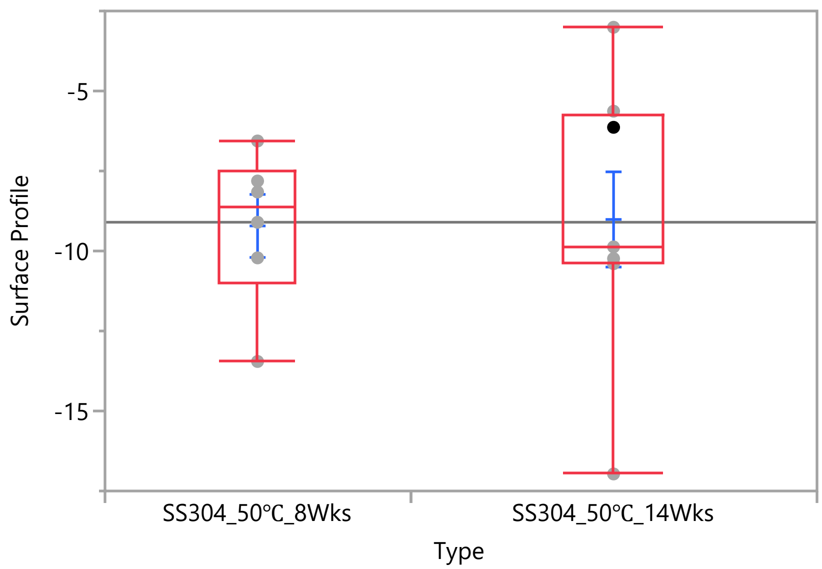
Assuming unequal variances

Difference	0.2225	t Ratio	0.152777
Std Err Dif	1.4564	DF	15.3374
Upper CL Dif	3.3207	Prob > |t|	0.8806
Lower CL Dif	-2.8757	Prob > t	0.4403
Confidence	0.95	Prob < t	0.5597



SS304 Exposure Duration Effect (50℃)

Oneway Analysis of Surface Profile By Type



t Test

SS304_50℃_14Wks-SS304_50℃_8Wks

Assuming unequal variances

Difference	0.2027	t Ratio	0.113631
Std Err Dif	1.7839	DF	11.41142
Upper CL Dif	4.1119	Prob > |t|	0.9115
Lower CL Dif	-3.7065	Prob > t	0.4558
Confidence	0.95	Prob < t	0.5442