LA-UR-25-27800
Approved for public release; distribution is unlimited.

Title: Laser Confocal Microscopy Uncertainty Quantification Study

Author(s): Gigax, Jonathan Gregory
Longoria, Gabriel Rey

Intended for: Report

Issued: 2025-11-08 (rev.2)

Laser Confocal Microscopy Uncertainty Quantification Study

Jonathan G. Gigax and G. R. Longoria

LA-UR-25-27800
09/22/2025

Los Alamos
NATIONAL LABORATORY

Contents

Figures

Tables

INTRODUCTION

At Los Alamos National Laboratory (LANL), the Storage Safety and Engineering (SSE) team completes annual surveillance on a subset of in-use interim nuclear material storage containers in fulfilment of requirements outlined in DOE Manual M 441.1-1 [1]. The containers are selected through several methods, such as subject matter expert judgement, random selection, and trending items. Following these selections, the SSE team has the capacity to complete surveillance on 15-20 containers each fiscal year, composed of a combination of SAVY-4000 and Hagan storage containers. Through previous work, the stainless-steel components of the containers have been identified as life limiting components, with an emphasis on the thin-walled bodies. The team is focused on understanding the extent of general and pitting corrosion, due to observations of extensive corrosion from stored contents and bag-out-bag degradation. Quantifying corrosion effects on the thin-walled stainless steel container bodies, and understanding potential impacts to the respective design release rates and design qualification release rates is paramount to the team.

To date, destructive examination (DE) has proven to be the most insightful method for developing an understanding on the extent of corrosion on used containers. To standardize this process, the SSE team developed a destructive examination guide for analyzing stainless steel components of the containers [2]. Corroded containers of interest are identified during surveillance activities and set aside for sectioning and characterization. Following sectioning, a major step in the DE workflow is the utilization of laser confocal microscopy for scanning corroded samples of interest and extracting data on pits, such as count, depth, and equivalent diameter. Adhering to the techniques outlined in the DE guide, analysis has been completed on two Hagans and one SAVY-4000 container, with the maximum pit depth recorded as 139.1 ± 22.82 µm on a 17.5 year old Hagan [3,4,5]. The findings from the completed destructive examinations will be utilized to support lifetime extension efforts of the SAVY-4000 as the team can better estimate corrosion rates and effects over time based on stored contents and age.

Due to the implications of observing extreme pit depths that approach the nominal container body thickness of .0299 inches (0.759 mm) or minimum container thickness of 0.236” (0.6 mm), high confidence in the LCM measurements is desired. Through testing outlined in [3], it was concluded that the total error ascribed to the 20x objective when conducting large image mapping on the Keyence VK-X3050 laser confocal microscope (LCM) relative to a 50x objective (reference) is 16.4% (± 8.73%). For shallow features on the order of pristine SAVY surface defects (i.e. 5 µm), this uncertainty is appropriate. However, this conservative estimate of total error poses a fundamental concern for pit depths that approach the thickness of the measured samples. That is, with the measurement uncertainty currently employed on all measurements, the LCM would be unable to resolve if a pit with a depth of 515 µm is through wall.

Standard step height samples were procured and used in the present study to assess the resolution and repeatability of height measurements. Understanding the resolution and repeatability of height measurements was the first focus of the team as it relates directly to pit depth, which is of primary concern. Calibration gratings were procured to evaluate the resolution and repeatability of measurements in the X and Y axes of the LCM stage. The results of the depth uncertainty study were conducted first and presented in the subsequent sections. The planar uncertainty study is appended to the depth study with conclusions from both summarized at the end of the report.

EXPERIMENTAL METHODS (DEPTH)

A Keyence VK-3050 laser confocal microscope (LCM) was utilized in this study as this microscope is used exclusively to measure pit depths in corroded container coupons. The VK-3050 features a red laser with variable point densities controlled by a galvanometer. The system is equipped with a motorized turret and holds 6 objective lenses ranging from 5x to 100x. A 20x objective lens (Nikon Apo Plan 20x) was used in this study as this lens provides sufficient resolving power for small corrosion defects (i.e. pits) while balancing characterization durations with a large field of view. Further, this lens is used in all corroded container characterizations as outlined in the destructive examination guide [2].

Metallized silica step height standards from VLSI (ISO-9001 certified) were procured for the study with nominal and actual measurements listed in Table 1. Cr deposition on the surface increases specularity and enables the LCM to resolve features where the base silica variant is transparent to the red laser in the LCM. All VLSI sample standards include an uncertainty quantification report generated using a profilometer calibrated against NIST-traceable standard.

Table 1 - VLSI calibration standard step height values.

Nominal Height (µm)	Actual Height (µm)	Uncertainty (µm)
4.5	4.482	0.059
8	7.999	0.065
19.5	19.358	0.117
50	50.157	0.271

A coarse schematic of the LCM is provided in Fig. 1. Assessment of the repeatability of measurements must enclose all modalities and nominal measurement durations/travel ranges to be made. Principally, this includes two types of measurements: single region and stitching or mapping evaluations. Stage motion, the difference between these two measurement modalities, is one source of uncertainty that will be investigated in this study as a potential influence on the measurement error.

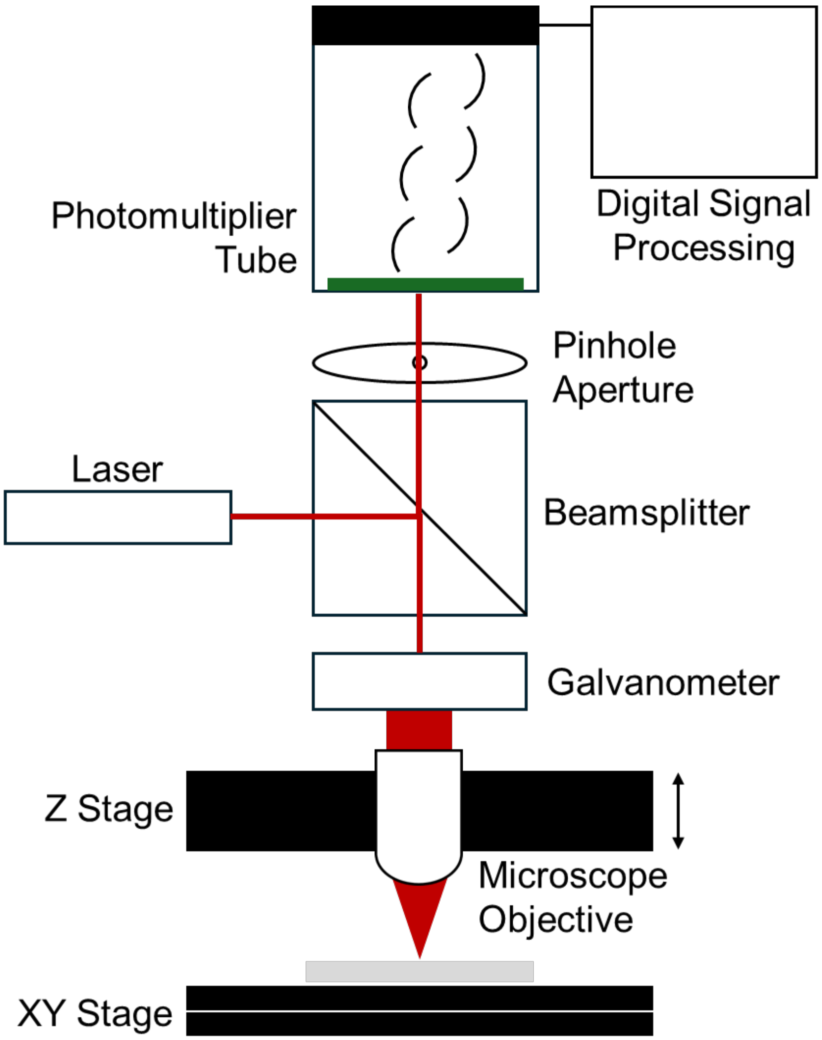


Figure 1 - LCM Schematic Overview

Mapping measurements with a 20x objective for a 25 mm square coupon typically require four separate regions of 10x52 image arrays resulting in 9-15 hours of time to complete per array. Measurement ranges on the coupon may vary relative to the mapping region on a specimen, with shorter durations near the middle of the coupon due to the smaller height difference between the tallest and shortest portions of the region. Uncertainties in the measurements for a range of Z travel distances are evaluated in this study to assess the contribution of this stage's motion.

A single coupon can require 48 hours of measurement run-time. Due to some scans ending during off-hours, most coupon scans require a minimum equipment on-time of 2-3 days. 9 coupons are typically selected from a container and used for analysis. While more may be evaluated, these are often chosen after the first round of characterization and are not considered as part of the machine on-time estimate. If performed efficiently, 9 coupons can be fully characterized within 18-21 working/business days. This translates to total duration of 3.5 – 4.5 weeks. Equipment on-time is another source of uncertainty to evaluate and is a controllable factor in this study.

In short, the following measurements were conducted:

1) Single scans with minimized travel range at a fixed location on each calibration sample, repeated 10 times.

    a. Goal: Assess resolution and single measurement uncertainty of the system.
2) Single scans with varying travel range at a fixed location on only the 4.5 μm calibration sample, repeated 10 times.
    a. Goal: Assess changes in measurement uncertainty associated with Z stage motion.
3) Stitching scans with a 10x50 area with a minimized travel range for the 4.5 μm calibration sample, repeated 5 times.
    a. Goal: Assess changes in measurement uncertainty associated with XY stage motion.
4) Stitching scans with a 10x50 area with extended measurement range, repeated 5 times.
    a. Goal: Assess changes in measurement uncertainty associated with long laser emission times.

There are other factors that are beyond the control of the authors in this study and include relative humidity (RH) and temperature (T) changes as well as the light sensor and processing hardware (i.e. photomultiplier tube and digital signal processing circuitry). The relative humidities and temperatures near the microscope were evaluated at the start of all measurements and also recorded at the end of measurements for durations longer than 5 minutes. Fig. 2 provides an illustrative picture of the location of the RH/T probe and sample configuration on the LCM. A TSI VelociCalc handheld ventilation meter was used to measure the RH/T locally near the LCM. All downstream sensors and electronics contributions to the measurement uncertainty are not resolvable and are a part of the general measurement variability attributed to the instrument as a whole.

All step height sample measurements were evaluated in the Keyence VK Multifile Analyzer tool. Height maps were subject to a linear tilt correction using the bottom left quadrant of the image as the reference for the correction. Step height feature transitions often resulted in sharp feature artifacts at the interface. This required removal via a stylus mode correction that convolves a simulated 10 μm radius tip coupled with a noise filter to the height profile. For the first two measurements (single region scans), 5 line profiles separated by 100 pixels (~71 μm) were used to compute the average step height change. For the second two measurements (stitching scans) only 3 lines were used due to feature constraints on the selected images. Only 80 μm near the shelf or step was used in the analysis to avoid long range distortions arising from lens distortions. We note the similarity of the approach outlined here to a previously published study on a different LCM [6].

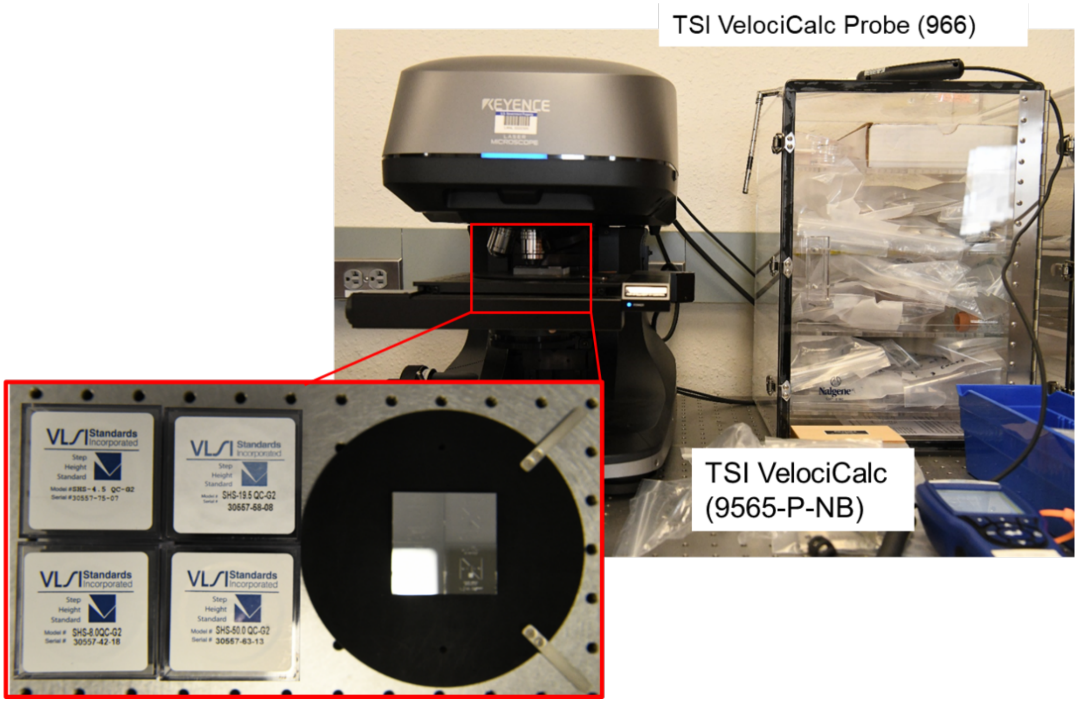


Figure 2 - RH/T probe and calibration sample configuration.

Measurement uncertainty is calculated using the equation

$$\sigma_{meas} = \sqrt{(\sigma_{lcm})^2 + (\sigma_{vlsi})^2}$$

Where σ_{lcm} is the sample standard deviation of the measurement results and σ_{vlsi} is the measurement uncertainty for each respective calibration standard measured.

SINGLE LOCATION MEASUREMENT RESULTS (DEPTH)

A low magnification image of the step height calibration standard (4.5 um) is shown in Fig. 3. For the single region measurements, the region on each sample is located at approximately (10,23) in the image. An example profile of a step feature is shown as an inset in Fig. 3. The loss of straightness on the curve is most likely an artifact of imaging from the LCM on a flat and highly specular surface. Measurements with a contact probe are needed to accurately evaluate the topography of the surface.

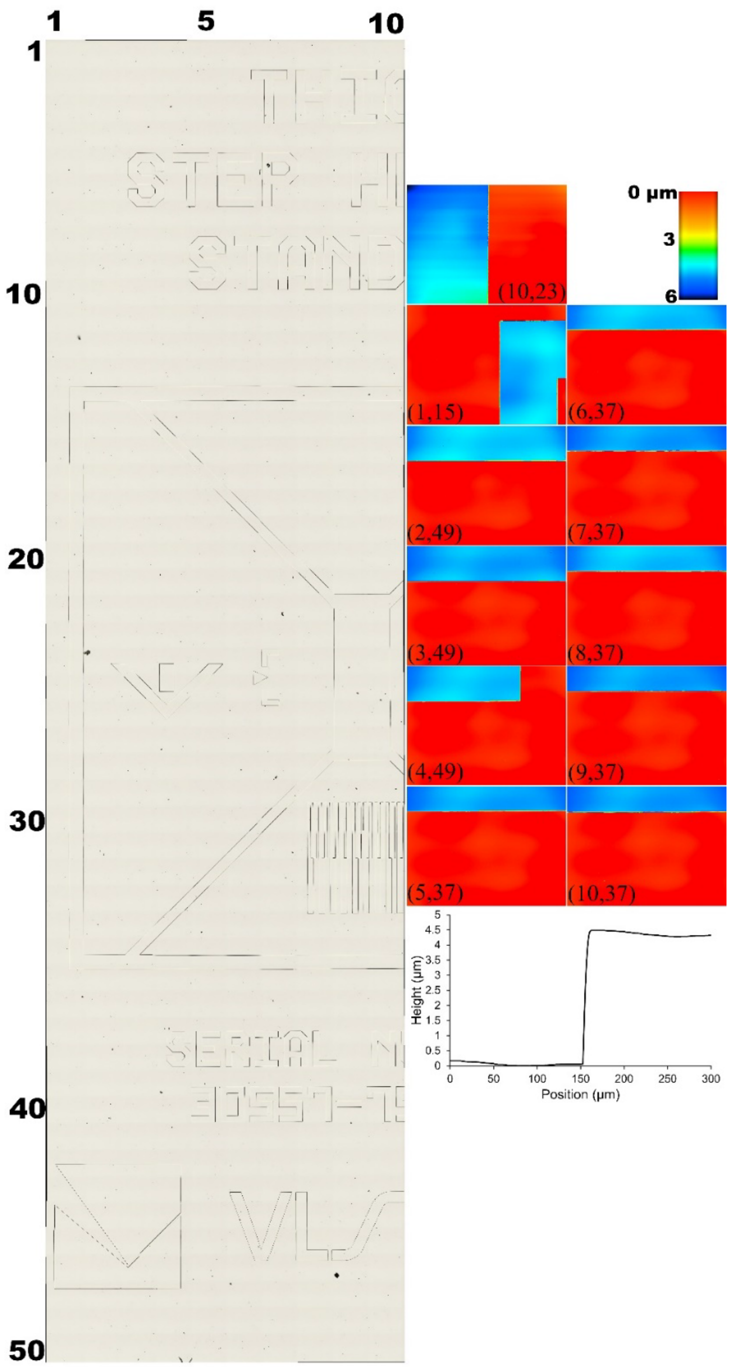


Figure 3 - Calibration sample image and measurements of step height

Fig. 4 shows the results of the single location measurements with measurement types distinguished by different color data points. Black dots represent data collected from a 4.5 µm step height standard with a varying measurement range, and subsequently duration, through four prescribed travel ranges. No significant changes in the measurement difference were observed within the sample measurement variability and height uncertainty from the manufacturer.

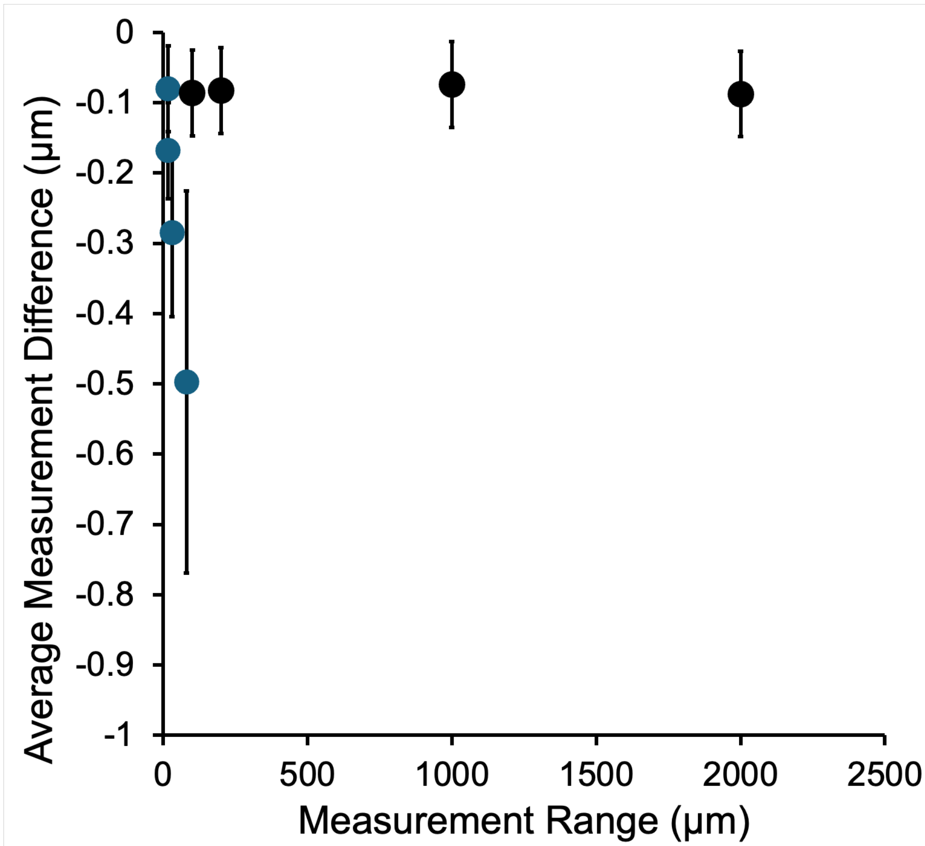


Figure 4 - Comparison of the average measurement difference for different step height samples and travel ranges. Blue data points are from single region measurements with minimized travel. Black data points are from measurements where the travel range was systematically varied.

Blue dots represent data collected from the four step height specimens with minimized travel ranges noted in Table 2. In direct contrast with the measurement results made from varying the travel range on a single sample, the measurement differences for the four specimens shows a significantly different trend with an apparent increase in the difference between the measured step height value and the value reported by VLSI. Combined, the two measurement types indicate that the increase in uncertainty with increasing measurement range is not due to the LCM. Note that in Table 2, the height variation measured by the LCM (middle column) is nearly identical for all measurements, regardless of travel range.

Table 2 - Comparison of step height measurement values, measurement variability, and difference from the actual step height for different specimens and travel ranges.

Nominal Height (µm)	Measurement Z Travel Range (µm)	LCM Measured Height (µm)	Average Measurement Difference (µm)
4.5	16.585	4.562 ± 0.016	-0.080 ± 0.061
8	16.499	8.167 ± 0.021	-0.168 ± 0.068
19.5	30.117	19.64 ± 0.028	-0.285 ± 0.121
50	80.243	50.65 ± 0.022	-0.497 ± 0.272
4.5	100	4.568 ± 0.016	-0.086 ± 0.061
4.5	200	4.565 ± 0.014	-0.083 ± 0.061
4.5	1000	4.556 ± 0.015	-0.074 ± 0.061
4.5	2000	4.570 ± 0.014	-0.088 ± 0.061

Fig. 4 shows the relative fraction of the LCM measurement variability and VLSI measurement uncertainty for each step height calibration specimen. Clearly, the VLSI uncertainty fraction comprises a majority of the calculated uncertainty for each measurement made, with the fraction increasing with increasing step height value. The VLSI calibration standard measurement uncertainty is intrinsic to the sample and cannot be decoupled from the overall measurement uncertainty. For subsequent measurements made in stitching mode, the step height standard with the smallest measurement uncertainty (4.5 µm) was used to assess changes in uncertainties in the LCM under this modality.

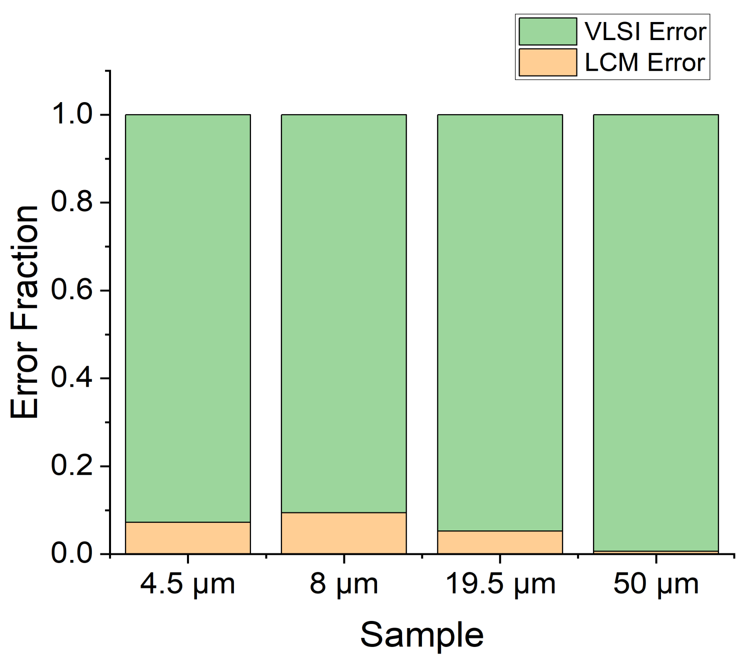


Figure 5 - Comparison of measurement uncertainty fractions for each step height specimen.

STITCHING MODE MEASUREMENT RESULTS (DEPTH)

A small scan range of 30 µm was defined for the first stitching measurement. Scan times for this travel range averaged approximately 4 seconds per region. The LCM uses a comb path for image stitching that involves scanning through all rows of the stitch region in a single column and returning to the first row of the next column when complete. The travel between each row within a column is minimal (~1 second). However, the travel from the last row of the current column to the first row of the next column takes approximately 5 seconds. The scan duration, averaged for all travel, is approximately 5 seconds.

10 image regions were identified with one image in each column of the 10x50 scan, ensuring sufficient spacing between scans to resolve changes arising from the scan time. The 10 locations are noted in Fig. 2 starting with (1,15) and ending with (10,37). Note that equilinear spacing between images was not possible due to the lack of step features in some of the rows. Fig. 6 provides an overview of the measurement difference made between the LCM measured step height and the VLSI reported step height value for each of the 10 regions for all 5 scans.

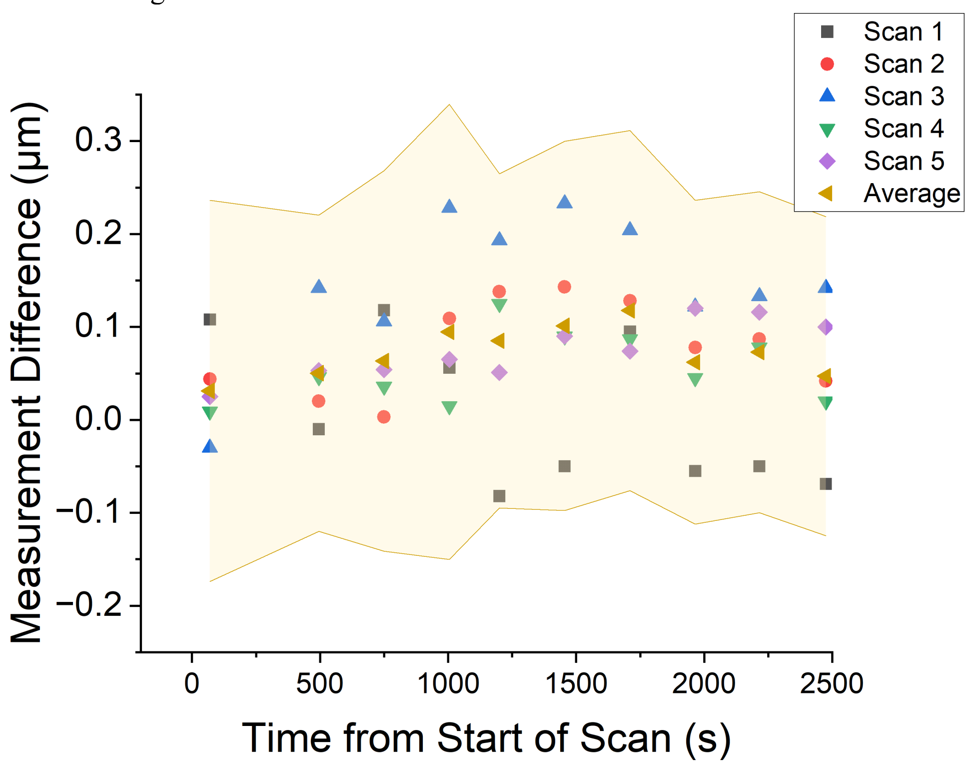


Figure 6 - Comparison of measurement differences made at different locations on the 4.5 µm specimen within a 10x50 array with a small measurement travel range. Outer error band is 6 sigma from the average measurement difference uncertainty.

Notably, the trend show that the measurement difference peaks near the middle of the region with an apparent monotonic increase in the measurement difference with increasing time from the start of the scan. The yellow error band is 6 standard deviations (6σ) from the average uncertainty from all 5 measurements. While not shown in the figure, this error band encapsulates the measurement uncertainty from all measurements as noted in Table 3 with a ±0.2 mm uncertainty on the average.

Table 3 - LCM measured height and difference from actual step height measurement for selected locations within the 10x50 array.

Scan Number	Time From Scan Start (s)	LCM Measured Height (μm)	Average Measurement Difference (μm)
14	75	4.513 ± 0.046	0.031 ± 0.034
98	500	4.532 ± 0.020	0.050 ± 0.028
148	755	4.545 ± 0.043	0.063 ± 0.034
198	1010	4.577 ± 0.062	0.095 ± 0.041
236	1205	4.567 ± 0.031	0.085 ± 0.030
286	1460	4.583 ± 0.037	0.101 ± 0.033
336	1715	4.600 ± 0.037	0.118 ± 0.032
386	1970	4.544 ± 0.027	0.062 ± 0.029
435	2220	4.555 ± 0.026	0.073 ± 0.029
486	2480	4.529 ± 0.023	0.047 ± 0.029

A large scan range of 2000 μm was defined for the second stitching measurement. This measurement, as previously noted, is intended to force both long laser emission times and long measurement times. Fig. 7 provides an overview of the data collected from 5 scans on the same regions identified for the short travel range measurement. Note that the scan duration for a region within the array was more than 2 minutes long, significantly longer than the stage movements. A similar apparent peaking near the middle of the scan was observed with the long travel range. However, the measured difference appeared to have a less well-defined trend. Table 3 provides the average step height measurements and the measurement differences. Similar to the data collected from small travel ranges, the 6σ error band that bounds the measured data is ±0.2 mm from the average.

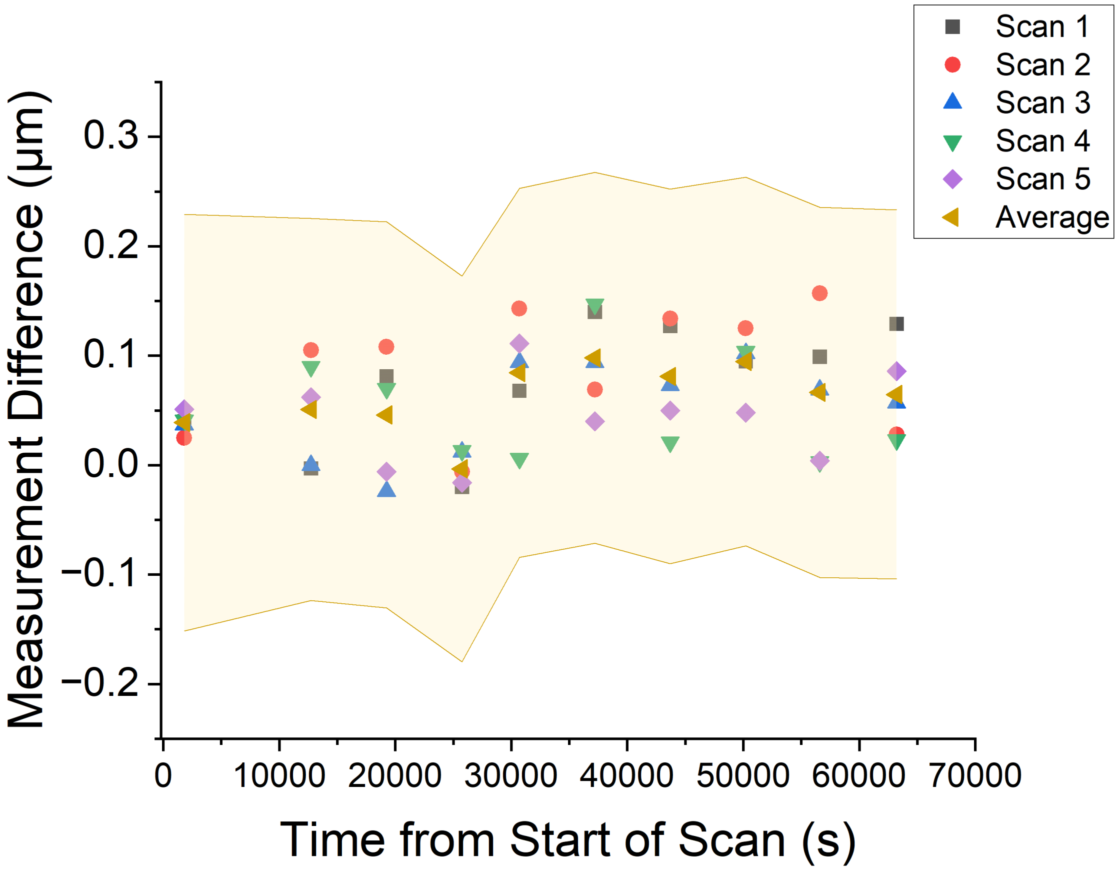


Figure 7 - Comparison of measurement differences made at different locations on the 4.5 µm specimen within a 10x50 array with a large measurement travel range. Outer error band is 6 sigma from the average measurement difference uncertainty.

Table 4 - LCM measured height and difference from actual step height measurement for selected locations within the 10x50 array.

Scan Number	Time From Scan Start (s)	LCM Measured Height (µm)	Average Measurement Difference (µm)
14	1820	4.521 ± 0.038	0.039 ± 0.032
98	12745	4.533 ± 0.027	0.051 ± 0.029
148	19250	4.528 ± 0.029	0.046 ± 0.029
198	25755	4.479 ± 0.028	-0.003 ± 0.029
236	30700	4.566 ± 0.022	0.084 ± 0.028
286	37205	4.580 ± 0.022	0.098 ± 0.028
336	43710	4.563 ± 0.024	0.081 ± 0.029
386	50215	4.579 ± 0.021	0.095 ± 0.028
435	56590	4.548 ± 0.022	0.066 ± 0.028
486	63225	4.547 ± 0.021	0.065 ± 0.028

The presence of a maximum in the measurement difference recorded using different travel ranges/single region scan times implies the trend is related to specimen uniformity. Single region measurements, repeated 10 times, at each of the locations extracted from the stitched image were performed to provide additional support to this conclusion. Fig. 8a and 8b provides a comparison of the step height and measured difference for each of the locations, respectively. All three measurements show a local maximum at scans 237, 287, and 337, with a slight discrepancy with the single region measurement showing a global maximum at scan 14. The consistency of the results indicates that the sample uniformity is the source of the uncertainty variation. However, VLSI did not report a step height uniformity value for the specimen. This, then, requires an incorporation of the uncertainty from these measurements into the final consideration of the bounding error for all measurements made with a 20x objective.

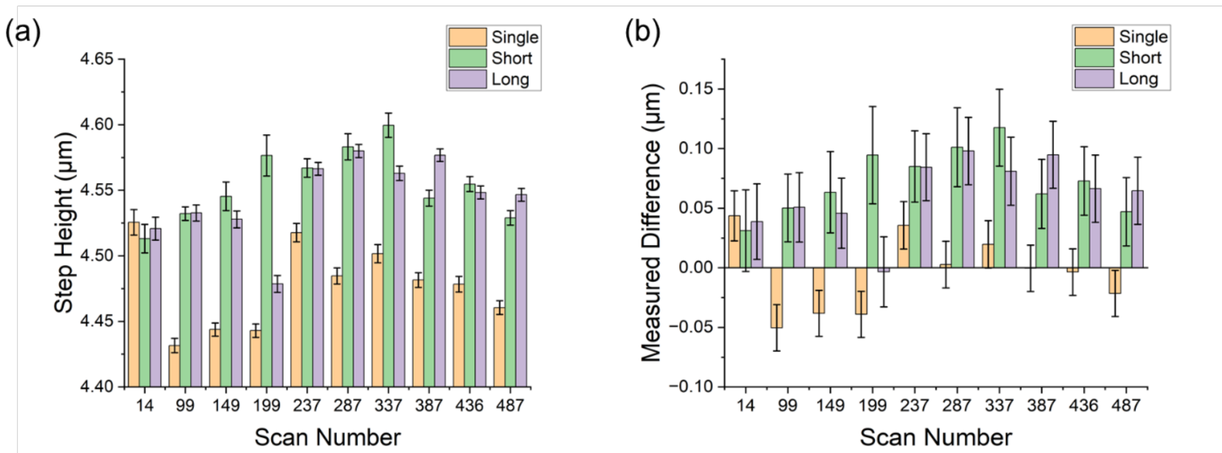


Figure 8 - (a) Comparison of the measured step height value and (b) step height measurement differences for three scan configurations.

EVALUATION OF OTHER FACTORS (DEPTH)

Fig. 8a and 8b provide measurement difference values as a function of measurement range and LCM on time, respectively. These two controllable factors are derived, in part, from measurements made from the preceeding two sections. After all four measurements types were completed, the microscope was then released for characterization on corroded specimen coupons. Periodically, the step height calibration sample was returned to the stage and measurements were made at the first region (1,15). This was repeated until approximately 60 days had elapsed since the start of the first measurement in this study, sufficiently bounding the machine on time required to characterize a corroded storage container. Linear correlations performed on the measurement range and LCM on time datasets resulted in values of -0.055 and 0.129, respectively. These values indicate virtually no relationship between measurement error and these factors.

Relative humidity and temperature were tracked for all measurements and the results are shown in Fig. 9c and 9d, respectively. It is important to note that these factors were not controllable resulting in large spacing between groups of data, especially in the case of relative humidity, owing to the changes in the environment. Linear correlations between the measurement difference and temperature and relative humidity were -0.189 and 0.022, respectively, and indicate no relationship between the measurement error and these factors.

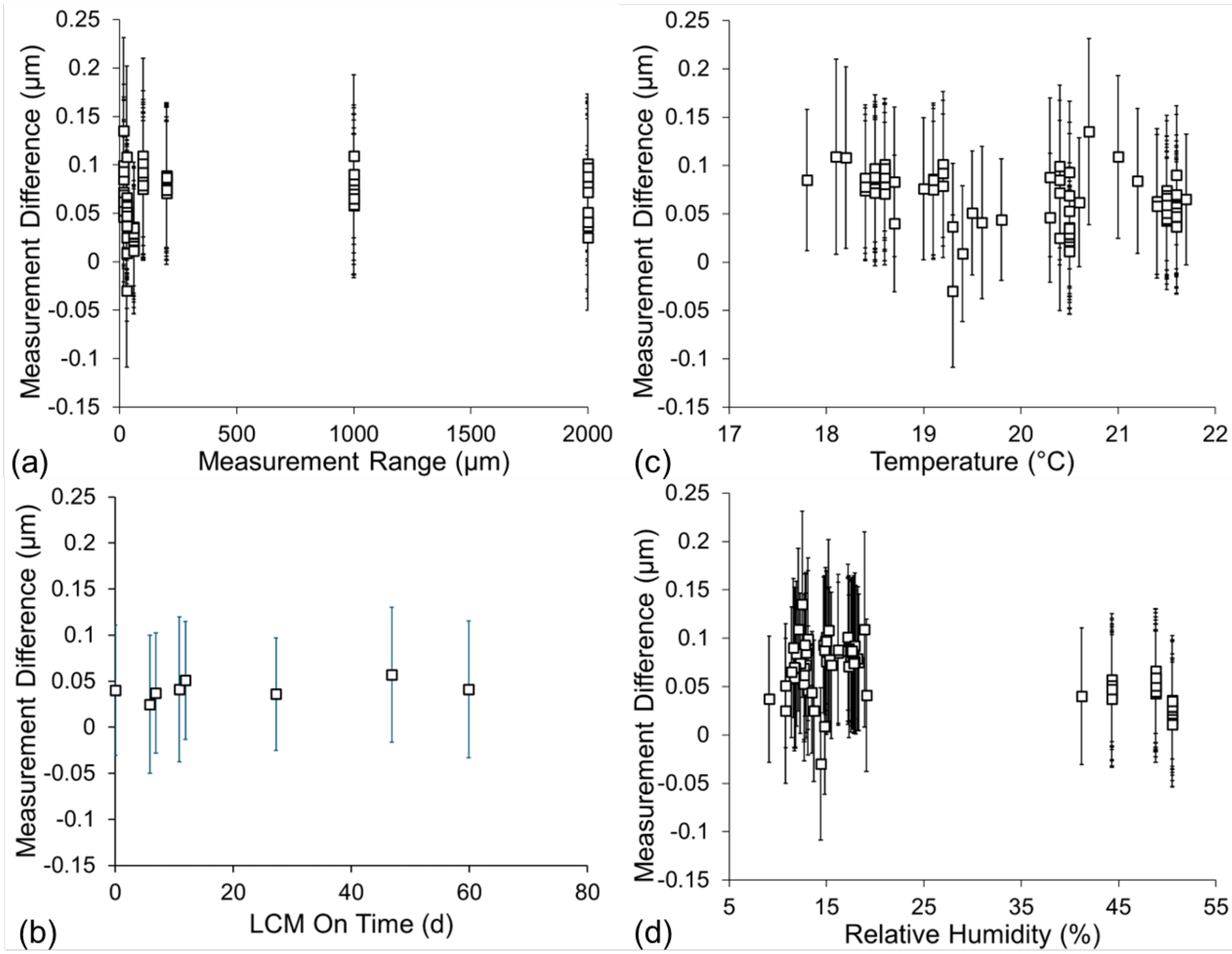


Figure 9 - Measured step height values for the 4.5 µm sample as a function of (a) measurement range, (b) LCM on time, (c) temperature, and (d) relative humidity.

The variation observed in these plots is likely attributable to other components of the LCM (i.e. digital signal processing circuitry). A lack of control over other components translates into a larger error placed on any measurement with the LCM with a 20x objective. That is, the inability to resolve the sources of variation precludes our ability to eliminate or minimize those sources' contribution to the measurement error and uncertainty.

DISCUSSION (DEPTH)

The diffraction limited depth resolution of an LCM is given by:

$$R_z = \frac{1.4\lambda\eta}{(NA^2)}$$

Where λ is the wavelength of the emission source (in this case 661 nm), η is the refractive index of the imaging medium (air; 1), and NA is the numeric aperture of the lens (0.4). Here, the diffraction limited depth resolution of the microscope is ~5.8 um. The Keyence X3000 controller specifies a height/depth resolution of 0.2+0.01*L (L = measurement length) or better. More importantly, the height repeatability specified for a 20x objective is 0.04 um, significantly smaller than the range of measurement uncertainties for all measurements made in this study. Therefore, the measurements made in this study adequately bound the uncertainties specified by the manufacturer.

There are other potential sources of measurement uncertainties to consider including:

- Surface reflectivity
- Sample position on the movement stage
- Data processing

Accuracy of the step height measurements is contingent on the DSP correctly measuring and identifying the maximum light intensity at a given height and location on the specimen. The Cr coating on the surface is highly reflective (specular) and can therefore result in maximum intensity collection at several Z locations within a fraction of the depth of field of the optic, within the noise of the detector and processing circuitry. Additionally, the microscope objectives utilized are not telecentric within the field of view resulting in significant field distortions across the image frame. The approach leveraged in this study does not apply any field corrections to the image in an effort to avoid introducing additional sources of processing error on the specimen. Limiting the analysis to 80 μm near the step height ensures minimizing the influence of aberrations on the measurement.

In this study, the specimen was placed near the center of the sample stage as indicated by Fig. 2. All coupons subject to evaluation under the LCM are approximately the same size and therefore can be placed within the same approximate location on the stage as the calibration standard. Larger deviations in the uncertainty may appear near the extremes of stage travel where differences in stage flatness/straightness may increase.

We note that one operator performed the analysis on all measurement data with concurrence from a second operator. While this does not eliminate reproducibility uncertainty, it does exclude this source of uncertainty from the measurement error considerations. Reproducibility uncertainty will be assessed in a separate study once the procedure for evaluating coupons is finalized.

EXPERIMENTAL METHODS (PLANAR)

Planar uncertainty quantification was conducted following the depth uncertainty quantification on the LCM. Similar equipment and procedures were used with some notable differences for this portion of the study. First, results from the first study show that the largest measurement variation was largely independent on LCM imaging modality, scan time, relatively humidity, or temperature. For planar uncertainty quantification, two scan ranges with a 16.25 µm and 2000 µm scan range, respectively, were used to image a single location on the calibration sample. Scan settings used were identical to the depth quantification, notably the scan resolution of 1024x768 that is standard for all measurements made on the LCM to date.

A low magnification calibration grating (Pelcotec LMS-20) was purchased from Ted Pella, Inc. (fabricated by AISthesis Products, Inc). The smallest separation distance between marks on the surface is a nominal 0.01 mm with a 0.1% nominal uncertainty. Additional information on the calibration grating can be found in Appendix B. A single image near the center of the slide was used for both vertical and horizontal measurements, respectively, as shown in Fig. 10.

Due to the very small variation in surface height on the slide, images acquired from the LCM were post-processed using both linear (tilt) and waveform corrections to produce a flat profile across the entire image. A total of 9 distances between neighboring pairs of tick marks were measured from the tick marks closest to the edge of the image in each of the directions as shown in Fig. 11. A single measurement spanning tick marks over the field of view was made and an average separation distance was computed by dividing the distance by the number of tick marks captured by the measurement. Note that the edge-to-edge distance of tick marks was placed on the same side of the marking to avoid the inclusion of the width of the tick mark.

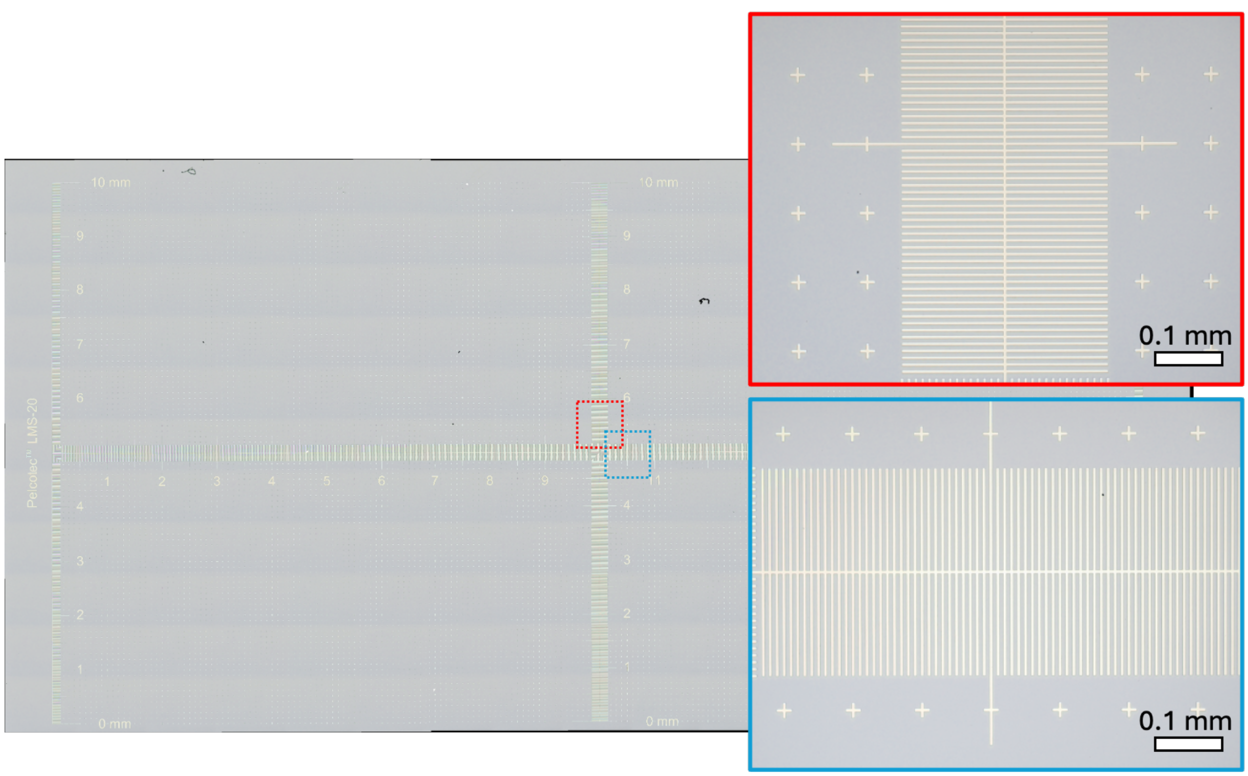


Figure 10 - Location of images taken for the vertical and horizontal uncertainty quantification.

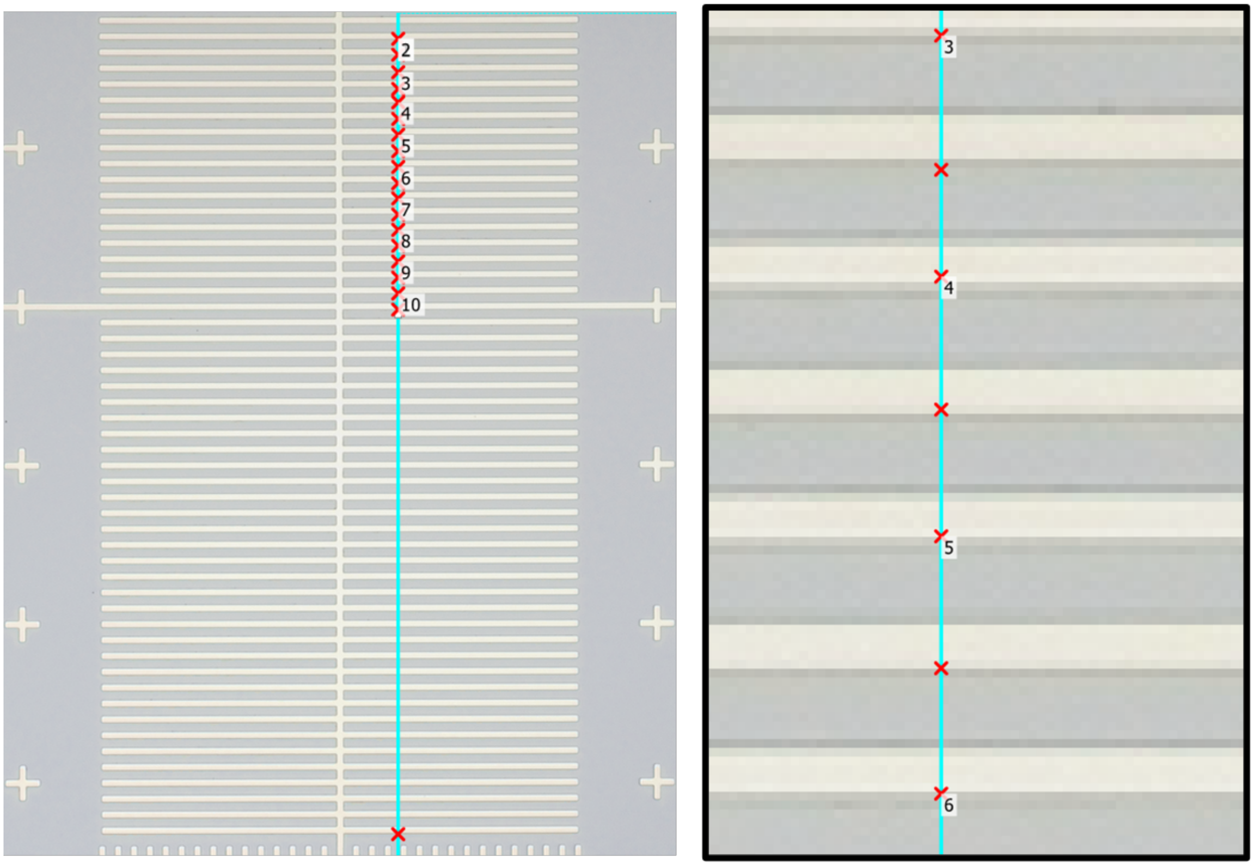


Figure 11 - Location of single tick mark and multiple tick mark measurements.

MEASUREMENT RESULTS AND DISCUSSION (PLANAR)

Table 5 provides all the measurements made in this portion of the study for spacing between multiple tick marks ("multiple") and neighboring tick mark pairs ("pairs"). Due to the 4:3 aspect ratio of the camera, the field of view for the vertical direction is smaller than the horizontal. This results in a fewer number of tick marks included in the field of view for the vertical (50) compared to the horizontal (68). Table 6 provides a comparison of the smallest, largest, and average tick mark separation distances measured for the vertical and horizontal directions, respectively. The average tick mark spacing for pair or multiple measurements is nearly identical with the pair measurements showing significantly greater measurement variability. This is attributed to the relative sensitivity of each measurement at the selected resolution, with pair measurements comprising a few pixels separating features at 20x. Variation in a single pixel can result in large measurement variations.

Short scan durations were used for scans 1-10 and long durations for scans 11-20. For pair measurements in the vertical direction, the average spacing for short and long scan durations was measured to be 9.989 ± 0.152 µm and 10.52 ± 0.291 µm, respectively. For pair measurements in the horizontal direction, the average spacing for the short and long scan durations was measured to be 10.29 ± 0.202 µm and 10.19 ± 0.333 µm, respectively. No statistically significant differences are observed between the short and long scan durations measurements except for the pair measurements in the vertical direction. Therefore, for the data shown in Table 6, short and long scan measurements are averaged together.

Table 5 - Tick spacing measurement results for multiple and pair measurements for all scans conducted in the study. All values are in units of micrometers.

Scan	Vertical										Horizontal									
Number	Multiple	Pair									Multiple	Pair								
1	511.059	9.998	10.076	9.934	10.344	9.897	9.771	9.878	10.028	9.939	697.882	10.233	10.063	9.997	10.344	10.579	10.583	10.121	10.465	10.24
2	511.055	9.988	10.087	9.909	10.341	9.9	9.77	9.916	10.066	9.942	697.89	10.233	10.084	9.993	10.34	10.579	10.584	10.117	10.465	10.238
3	511.056	9.994	10.072	9.93	10.311	9.864	9.751	9.87	10.092	9.937	697.88	10.232	10.093	10.008	10.349	10.578	10.585	10.119	10.467	10.241
4	511.057	9.997	10.076	9.942	10.345	9.905	9.783	9.922	10.062	9.949	697.874	10.227	10.079	10.001	10.346	10.58	10.583	10.137	10.468	10.235
5	511.059	9.99	10.075	9.936	10.33	9.902	9.764	9.847	10.063	9.949	697.875	10.226	10.056	10.005	10.346	10.577	10.584	10.119	10.466	10.238
6	511.057	9.99	10.084	9.934	10.237	9.922	9.75	9.894	10.094	10.001	697.887	10.23	10.086	10.013	10.347	10.579	10.586	10.123	10.465	10.238
7	511.058	9.991	10.091	9.932	10.345	9.912	9.735	9.911	10.04	9.974	697.889	10.232	10.084	10.009	10.344	10.571	10.581	10.123	10.466	10.235
8	511.059	9.994	10.08	9.938	10.342	9.899	9.761	9.876	10.012	9.941	697.879	10.231	10.086	9.998	10.346	10.58	10.593	10.116	10.468	10.242
9	511.059	9.995	10.069	9.934	10.335	9.906	9.753	9.884	10.023	9.981	697.88	10.234	10.037	10.001	10.345	10.576	10.589	10.117	10.467	10.236
10	511.059	9.999	10.08	9.932	10.344	9.901	9.746	9.911	10.093	9.967	697.763	10.21	10.165	10.037	10.357	10.582	10.57	10.147	10.46	10.231
11	512.004	10.214	10.801	10.696	10.613	10.502	10.435	10.241	10.343	10.372	695.837	10.635	9.831	9.974	9.644	10.479	10.512	10.522	10.018	10.216
12	512.004	10.211	10.863	10.696	10.561	10.466	10.416	10.22	10.267	10.467	695.864	10.7	9.829	9.923	9.656	10.482	10.473	10.494	10.035	10.102
13	511.997	10.213	10.961	10.538	10.615	10.814	10.198	10.161	10.379	10.555	695.873	10.634	9.838	9.971	9.646	10.453	10.485	10.495	10.03	10.134
14	512.004	10.212	10.945	10.579	10.524	10.75	10.768	10.137	10.373	10.565	695.86	10.635	9.84	9.972	9.653	10.482	10.47	10.51	10.013	10.106
15	512.004	10.115	10.952	10.371	10.849	10.723	10.529	10.309	10.384	10.582	695.889	10.627	9.861	9.985	9.601	10.428	10.546	10.54	10.041	10.114
16	512.004	10.102	10.96	10.42	10.866	10.717	10.534	10.299	10.404	10.684	695.874	10.62	9.867	9.989	9.631	10.419	10.513	10.546	10.033	10.114
17	511.932	10.049	10.926	10.433	10.909	10.756	10.523	10.291	10.396	10.612	695.887	10.613	9.865	9.988	9.607	10.429	10.508	10.605	10.018	10.106
18	512.004	10.075	10.884	10.563	10.812	10.684	10.521	10.324	10.36	10.732	695.89	10.58	9.866	9.985	9.605	10.411	10.539	10.59	10.013	10.109
19	512.004	10.077	10.879	10.565	10.827	10.674	10.517	10.275	10.406	10.619	695.881	10.597	9.861	9.975	9.618	10.37	10.547	10.548	10.029	10.107
20	512.004	10.073	11.196	10.618	10.785	10.646	10.516	10.282	10.466	10.728	695.891	10.579	9.874	9.975	9.637	10.441	10.497	10.55	10.021	10.109

Table 6 - Minimum, maximum, and average tick spacings for measurements made for vertical and horizontal markings.

Measured Value	Pair		Multiple	
	Vertical	Horizontal	Vertical	Horizontal
Minimum	9.74 μm	9.6 μm		
Maximum	11.2 μm	10.7 μm		
Average	10.3 ± 0.34 μm	10.2 ± 0.28 μm	512 ± 0.49 μm	697 ± 1.02 μm
Measurement Error	2.58% ± 3.42%	2.42% ± 2.79%	2.31% ± 0.14%	2.48% ± 0.18%

Figure 11 shows the results of the planar measurements with uncertainties as error bars in the plot. The largest difference observed from the measurement of tick mark pair separation distances is 0.258 ± 0.342 μm from pair measurements in the vertical direction. The measurement difference in the horizontal direction for pairs is 0.248 ± 0.279, nearly identical to that in the vertical direction. The measurement variability is significantly smaller for the multiple measurement in either the vertical or horizontal directions.

With a 20x objective and a 1024x768 scan resolution, the pixel pitch is 0.707 μm. The measured difference shown in Fig. 11 are both a fraction of this value and, when coupled with the uncertainty in the measurement, are bounded by the pixel pitch. Further, the spatial resolution limit of the system is defined at the Rayleigh limit, $R = \frac{0.61\lambda}{NA}$. For a 20x objective and a 661 nm emission source, the resolution of the system is approximately 1 μm. The pixel pitch, measurement error, and uncertainty are all less than the Rayleigh resolution limit and therefore do not influence the ability of the system to resolve features. This indicates that the measurement accuracy is defined by the scan resolution of the microscope or the resolution limit, whichever is greater. The resolution of the optical system, however, is unable to adequately resolve the tick mark features as these have a knife edge taper significantly smaller than 1 μm.

At first glance, the results indicate that a measurement uncertainty of one pixel would sufficiently bound uncertainties associated with planar measurements. However, larger feature separation distances result in larger deviations from nominal or expected values. For example, the separation distance of tick marks across 50 in the vertical direction should be equal to 500 μm ± 0.5 μm, nominally. The averaged measured value of 512 μm results in a ~12 μm deviation from the nominal value, significantly greater than the sub-micrometer deviation in the pair measurements.

This discrepancy in measurement uncertainty can be resolved by normalizing the measurement difference and associated uncertainty to the nominal measurement distance. Values for the normalized average measurement error are provided in Table 6. All measurements give a similar measurement error ranging between 2.4 – 2.7%, indicating that such an error can be applied for any feature within the field of view. Incorporating the measurement uncertainty to this value gives 6% as a bounding uncertainty on all planar measurements made, except when feature sizes fall below a value as to result in an uncertainty less than the size of one pixel or the resolution of the system (i.e. features less than 16.7 μm). Here, an uncertainty value of the scan resolution of 1 pixel or 0.707 μm should be applied to all feature measurements, whichever is greater.

Measurements differences less than 6% can, in principle, be achieved by increasing the scan resolution on the LCM. The VK-3050 features a scan resolution up to 2048x1536 that increases the resolution by a factor of two in the vertical and horizontal directions. There are a couple of issues with this setting. First, increasing the scan pixel density by a factor of 4 does not change the resolution limit of the system. Second, due to the increased pixel density of the scan, the software limits the number of images to a quarter of that permitted by the standard scan resolution of 1024x768. The higher resolution scan provides minimal benefits to evaluating features of interest with sizes typically on the order of a few micrometers, especially when the duration of the characterization increases by a factor of four. For a typical 25 mm square coupon, this would increase the total LCM characterization time from 2.5 days to more than 10 days.

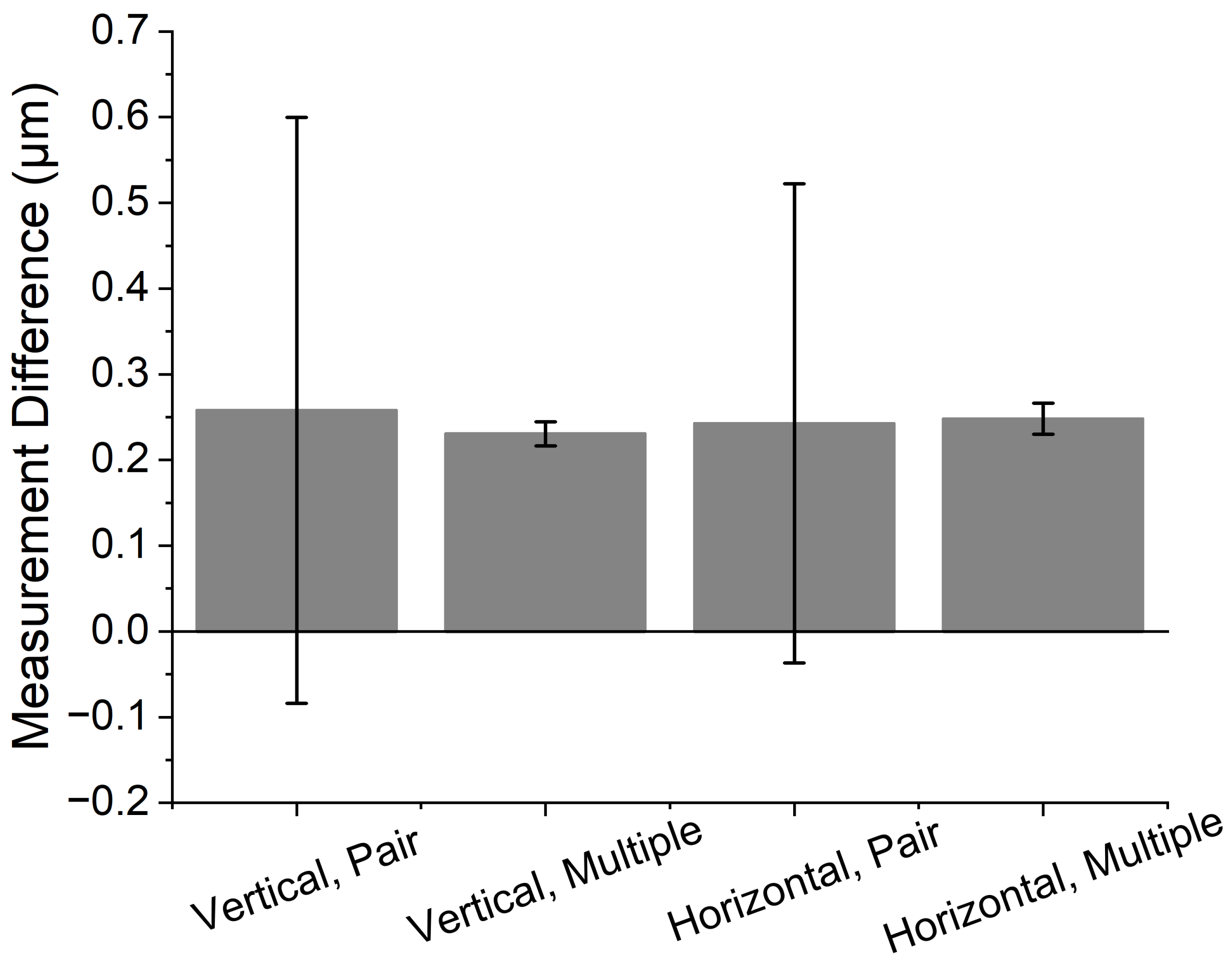


Figure 12 - Measurement differences between the LCM measurement and calibration grating.

CONCLUDING RECOMMENDATIONS FOR UNCERTAINTIES

Depth Uncertainty Quantification

The aggregated results in Fig. 9 clearly show that a combined measurement uncertainty of **± 0.25 µm** adequately captures all error and associated uncertainties with respect to depth in this study. We note that the measurement error is a combination of both the measured difference and the respective measurement uncertainties associated with each value. For instance, a measured difference of 0.1 mm with an associated measurement uncertainty of 0.05 would be adequately captured by a measurement error of 0.15 mm. This naïve approach overestimates the error on the low side of the uncertainty but eliminates the need to perform corrections on all data gathered by the equipment. A measurement error of **±0.25 µm** will then be used in all analyses with the Keyence VK-3050 LCM equipped with a 20x objective.

Sample calibration standard measurements do not change significantly over the course of approximately 2 months of on time with intermittent usage, specifically all-day utilization during work days with idle during the weekends. It is recommended that the destructive examination guide include provisions for incorporating measurements of the step height calibration standard before and after an entire container coupon array is evaluated. This will minimize procedural errors with measuring between coupons (or within coupons), maximizing coupon evaluation efficiency, and ensure that the measurement uncertainty does not exceed the recommended value in this study.

Measurement uncertainties less than ±0.25 µm can be achieved by measuring the calibration standard before and after a specimen measurement. This may be necessary when evaluating differences between features where a small measurement uncertainty is required. For most applications of the system in support of the SAVY lifetime extension effort, this is not needed and the noted uncertainty band derived over a 2-month evaluation period is sufficient to bound measurements where a calibration measurement was not made.

Planar Uncertainty Quantification

The results shown in Fig. 11 indicate that a measurement uncertainty of **±6%** (at 20x) adequately bounds the measurement error, variations, and uncertainties of the LCM. For features smaller than 16.7 µm, a measurement uncertainty of the **scan resolution or 0.707 µm** (for the settings in this study) should be applied. It is important to note that this value applies to feature measurements that span a few pixels in size to the full field of view. The 6% uncertainty can be improved by increasing the scan resolution (up to 2048x1536) but comes at the cost of reducing the total number of images acquired by the LCM during a stitching map by a factor of four.

REFERENCES

1. DOE Manual 441.1-1. Nuclear Material Packaging Manual, Department of Energy (2008).

2. J. G. Gigax, et al., A Destructive Examination Guide for Nuclear Material Storage Container Characterization. Los Alamos National Laboratory (2024), LA-UR-24-29666.

3. J. G. Gigax, et al., Corrosion Analysis of 121103072 (SAVY-4000). Los Alamos National Laboratory (2024), LA-UR-24-29827.

4. G. R. Longoria, et al., Corrosion Analysis of 03-06NMC05395 (Hagan). Los Alamos National Laboratory (2025), LA-UR-25-21723.

5. J. G. Gigax, et al., Corrosion Analysis of 05-99NMC05000-42 (Hagan). Los Alamos National Laboratory (2025), LA-UR-25-21588.

6. O. Guarneros, et al., Uncertainty Estimation for Performance Evaluation of a Confocal. M APAN-Journal of Metrology Society of India 29 (2014) 29-42.

APPENDIX A

VLSI Standards Incorporated

NVLAP® CALIBRATION NVLAP Lab Code 200302-0

CERTIFICATE OF CALIBRATION

STEP HEIGHT STANDARD

Model Number :	SHS - 50.0QC - G2
Serial Number :	30557-63-13
Calibration Date :	March 10, 2025

Step Material: Step etched in quartz
(Chrome-coated)

Scan for Apps notes.

www.vlsistandards.com

CALIBRATED STEP HEIGHT :

Mean Value		Expanded Uncertainty[1]
(50.157	**±**	**0.271) µm**

The certified step height value and uncertainty of measurement is traceable to the International System of Units (SI) using VLSI Standards' master step height standard 4343-23-21 which is certified by the National Institute of Standards and Technology as described in the NIST Test Number 821/261555-99A.[2]

Environmental conditions at the time of measurement:

Temperature:	(20 ± 1) °C
Humidity:	(45 ± 2) %

VLSI Standards, Inc. has been certified to ISO 9001 by DQS under certificate registration No. 10000561 QM15 and is accredited by NVLAP to perform this calibration under Lab Code 200302-0.
This standard is calibrated in compliance with NIST Handbook 150 and ISO/IEC 17025 which supersedes ISO 10012 and ANSI/NCSL Z540-1. Certificate shall not be reproduced, except in full, without specific written approval by VLSI Standards, Inc.

Danna Saechao
Calibration Technician

Tuyet Nguyen
Quality Assurance

[1] Expanded uncertainties are at the 95% confidence level, as defined by the ISO Guide to the Expression of Uncertainty Measurement (GUM).

[2] This certificate does not constitute product endorsement by NVLAP or by any U.S. Government agency.

Shipment Date:

SHIP DATE:MARCH 2025
Made in USA

Certificate Program: 0358308-000_AD

Page 1 of 2

5 Technology Drive, Milpitas, CA 95035-7916 • (408) 428-1800 • Fax (408) 428-9555 • www.vlsistandards.com

STEP HEIGHT STANDARD

Model Number : SHS - 50.0QC - G2
Serial Number : 30557-63-13
Calibration Date : March 10, 2025

Summary of (step height) input and output quantities:

Input quantity:		Mean value
NISTc	NIST reference (NIST report of calibration)	50.0464 µm
NISTm	NIST reference (VLSI observations)	49.8768 µm
Ym	SHS (VLSI uncorrected observations)	49.9873 µm

Output quantity:		Mean value
Yc	SHS (VLSI corrected value)	50.1573 µm

*Yc = NISTc / NISTm * Ym*

Summary of (step height) standard uncertainty components, combined standard uncertainty, degrees of freedom and expanded uncertainty coverage factor :

	Source of uncertainty	Value (µm)	Partial derivative	Combined component (µm)	Degrees of freedom	"Uncertainty of an uncertainty"
u(NISTc) Combined	NIST reference traceability (NIST report of calibration)	0.11574	1.00222	0.11599	8	25%
u(NISTm) Type A	NIST reference reproducibility (VLSI observations)	0.00028	1	0.00028	8	25%
u(resol) Type B	VLSI instrument vertical resolution	3.4E-06	1	3.4E-06	5000	1%
u(NISTmr) Combined	NIST reference reproducibility (VLSI corrected) .... *u(NISTmr) = sqrt{[u(NISTm)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00038	1.00562	0.00038	28	
u(Ym) Type A	SHS step height uniformity (VLSI observations)	0.01695	1	0.01695	8	25%
u(resol') Type B	VLSI instrument precision	2.6E-04	1	2.6E-04	8	
u(Ymr) Combined	SHS step height measurement (VLSI corrected) .... *u(Ymr) = sqrt{[u(Ym)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.01695	1.00340	0.01701	8	

u(Yc) **SHS combined standard uncertainty**

*u(Yc) = sqrt {[Ym / NISTm * u(NISTc)]^2 + [Ym * NISTc / NISTm^2 * u(NISTmr)]^2 +*
*+ [NISTc / NISTm * u(Ymr)]^2}*

u(Yc)		degrees of freedom *ν* (eff)	coverage t - factor
0.11723	µm	**8**	**2.31**

Certificate Program: 0358308-000_AD

VLSI Standards Incorporated

NVLAP® CALIBRATION NVLAP Lab Code 200302-0 ilac-MRA

CERTIFICATE OF CALIBRATION

STEP HEIGHT STANDARD

Model Number : SHS - 19.5QC - G2
Serial Number : 30557-58-08
Calibration Date : March 6, 2025

Step Material: Step etched in quartz
(Chrome-coated)

Scan for Apps notes.
www.vlsistandards.com

CALIBRATED STEP HEIGHT :

Mean Value		Expanded Uncertainty[1]
(**19.358**	**±**	**0.117**) **µm**

The certified step height value and uncertainty of measurement is traceable to the International System of Units (SI) using VLSI Standards' master step height standard 4343-31-05 which is certified by the National Institute of Standards and Technology as described in the NIST Test Number 821/261555-99A.[2]

Environmental conditions at the time of measurement:

Temperature:	(20	±	1	) °C
Humidity:	(46	±	2	) %

VLSI Standards, Inc. has been certified to ISO 9001 by DQS under certificate registration No. 10000561 QM15 and is accredited by NVLAP to perform this calibration under Lab Code 200302-0.
This standard is calibrated in compliance with NIST Handbook 150 and ISO/IEC 17025 which supersedes ISO 10012 and ANSI/NCSL Z540-1. Certificate shall not be reproduced, except in full, without specific written approval by VLSI Standards, Inc.

Danna Saechao
Calibration Technician

Tuyet Nguyen
Quality Assurance

[1] *Expanded uncertainties are at the 95% confidence level, as defined by the ISO Guide to the Expression of Uncertainty Measurement (GUM).*
[2] *This certificate does not constitute product endorsement by NVLAP or by any U.S. Government agency.*

Shipment Date:

SHIP DATE:MARCH 2025
Made in USA

Certificate Program: 0358308-000_AD

Page 1 of 2

5 Technology Drive, Milpitas, CA 95035-7916 • (408) 428-1800 • Fax (408) 428-9555 • www.vlsistandards.com

STEP HEIGHT STANDARD

Model Number : SHS - 19.5QC - G2
Serial Number : 30557-58-08
Calibration Date : March 6, 2025

Summary of (step height) input and output quantities:

Input quantity:		Mean value
NISTc	NIST reference (NIST report of calibration)	19.7195 μm
NISTm	NIST reference (VLSI observations)	19.7518 μm
Ym	SHS (VLSI uncorrected observations)	19.3892 μm

Output quantity:		Mean value
Yc	SHS (VLSI corrected value)	19.3575 μm

*Yc = NISTc / NISTm * Ym*

Summary of (step height) standard uncertainty components, combined standard uncertainty, degrees of freedom and expanded uncertainty coverage factor :

	Source of uncertainty	Value (μm)	Partial derivative	Combined component (μm)	Degrees of freedom	"Uncertainty of an uncertainty"
u(NISTc) Combined	NIST reference traceability (NIST report of calibration)	0.05130	0.98164	0.05035	8	25%
u(NISTm) Type A	NIST reference reproducibility (VLSI observations)	0.00053	1	0.00053	8	25%
u(resol) Type B	VLSI instrument vertical resolution	3.4E-06	1	3.4E-06	5000	1%
u(NISTmr) Combined	NIST reference reproducibility (VLSI corrected) .... *u(NISTmr) = sqrt{[u(NISTm)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00059	0.98004	0.00058	11	
u(Ym) Type A	SHS step height uniformity (VLSI observations)	0.00698	1	0.00698	8	25%
u(resol') Type B	VLSI instrument precision	2.6E-04	1	2.6E-04	8	
u(Ymr) Combined	SHS step height measurement (VLSI corrected) .... *u(Ymr) = sqrt{[u(Ym)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00698	0.99836	0.00697	7	

u(Yc) **SHS combined standard uncertainty**

*u(Yc) = sqrt {[Ym / NISTm * u(NISTc)]^2 + [Ym * NISTc / NISTm^2 * u(NISTmr)]^2 +*
*+ [NISTc / NISTm * u(Ymr)]^2}*

u(Yc)		degrees of freedom ν (eff)	coverage t - factor
0.05084	μm	**8**	**2.31**

Certificate Program: 0358308-000_AD

VLSI Standards Incorporated

NVLAP CALIBRATION NVLAP Lab Code 200302-0 ilac-MRA

CERTIFICATE OF CALIBRATION

STEP HEIGHT STANDARD

Model Number :	SHS - 8.0QC - G2
Serial Number :	30557-42-18
Calibration Date :	March 6, 2025

Scan for Apps notes.

www.vlsistandards.com

Step Material: Step etched in quartz
(Chrome-coated)

CALIBRATED STEP HEIGHT :

Mean Value		Expanded Uncertainty[1]	
(**7.999**	**±**	**0.065**	**) µm**

The certified step height value and uncertainty of measurement is traceable to the International System of Units (SI) using VLSI Standards' master step height standard 4343-26-24 which is certified by the National Institute of Standards and Technology as described in the NIST Test Number 821/261555-99A.[2]

Environmental conditions at the time of measurement:

Temperature:	(20	±	1	) °C
Humidity:	(44	±	2	) %

VLSI Standards, Inc. has been certified to ISO 9001 by DQS under certificate registration No. 10000561 QM15 and is accredited by NVLAP to perform this calibration under Lab Code 200302-0.
This standard is calibrated in compliance with NIST Handbook 150 and ISO/IEC 17025 which supersedes ISO 10012 and ANSI/NCSL Z540-1. Certificate shall not be reproduced, except in full, without specific written approval by VLSI Standards, Inc.

Danna Saechao
Calibration Technician

Tuyet Nguyen
Quality Assurance

[1] *Expanded uncertainties are at the 95% confidence level, as defined by the ISO Guide to the Expression of Uncertainty Measurement (GUM).*

[2] *This certificate does not constitute product endorsement by NVLAP or by any U.S. Government agency.*

Shipment Date:

SHIP DATE:MARCH 2025
Made in USA

Certificate Program: 0358308-000_AD

Page 1 of 2

5 Technology Drive, Milpitas, CA 95035-7916 • (408) 428-1800 • Fax (408) 428-9555 • www.vlsistandards.com

STEP HEIGHT STANDARD

Model Number : SHS - 8.0QC - G2
Serial Number : 30557-42-18
Calibration Date : March 6, 2025

Summary of (step height) input and output quantities:

Input quantity:		Mean value
NISTc	NIST reference (NIST report of calibration)	7.9077 µm
NISTm	NIST reference (VLSI observations)	7.9829 µm
Ym	SHS (VLSI uncorrected observations)	8.0749 µm

Output quantity:		Mean value
Yc	SHS (VLSI corrected value)	7.9989 µm

*Yc = NISTc / NISTm * Ym*

Summary of (step height) standard uncertainty components, combined standard uncertainty, degrees of freedom and expanded uncertainty coverage factor :

	Source of uncertainty	Value (µm)	Partial derivative	Combined component (µm)	Degrees of freedom	"Uncertainty of an uncertainty"
u(NISTc) Combined	NIST reference traceability (NIST report of calibration)	0.02772	1.01152	0.02804	8	25%
u(NISTm) Type A	NIST reference reproducibility (VLSI observations)	0.00034	1	0.00034	8	25%
u(resol) Type B	VLSI instrument vertical resolution	3.4E-06	1	3.4E-06	5000	1%
u(NISTmr) Combined	NIST reference reproducibility (VLSI corrected) .... *u(NISTmr) = sqrt{[u(NISTm)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00043	1.00200	0.00043	20	
u(Ym) Type A	SHS step height uniformity (VLSI observations)	0.00279	1	0.00279	8	25%
u(resol') Type B	VLSI instrument precision	2.6E-04	1	2.6E-04	8	
u(Ymr) Combined	SHS step height measurement (VLSI corrected) .... *u(Ymr) = sqrt{[u(Ym)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00280	0.99058	0.00278	7	

u(Yc) **SHS combined standard uncertainty**

*u(Yc) = sqrt {[Ym / NISTm * u(NISTc)]^2 + [Ym * NISTc / NISTm^2 * u(NISTmr)]^2 +*
*+ [NISTc / NISTm * u(Ymr)]^2}*

u(Yc)		degrees of freedom ν (eff)	coverage t - factor
0.02818	µm	**8**	**2.31**

Certificate Program: 0358308-000_AD

Page 2 of 2

VLSI Standards Incorporated

NVLAP CALIBRATION NVLAP Lab Code 200302-0

CERTIFICATE OF CALIBRATION

STEP HEIGHT STANDARD

Model Number :	SHS - 4.5QC - G2
Serial Number :	30557-75-07
Calibration Date :	March 7, 2025

Step Material: Step etched in quartz *(Chrome-coated)*

Scan for Apps notes.

www.vlsistandards.com

CALIBRATED STEP HEIGHT :

Mean Value		Expanded Uncertainty[1]
(**4.482**	**±**	**0.059) µm**

The certified step height value and uncertainty of measurement is traceable to the International System of Units (SI) using VLSI Standards' master step height standard 4343-12-23 which is certified by the National Institute of Standards and Technology as described in the NIST Test Number 821/261555-99A.[2]

Environmental conditions at the time of measurement:

Temperature:	(20 ± 1) °C
Humidity:	(44 ± 2) %

VLSI Standards, Inc. has been certified to ISO 9001 by DQS under certificate registration No. 10000561 QM15 and is accredited by NVLAP to perform this calibration under Lab Code 200302-0.
This standard is calibrated in compliance with NIST Handbook 150 and ISO/IEC 17025 which supersedes ISO 10012 and ANSI/NCSL Z540-1. Certificate shall not be reproduced, except in full, without specific written approval by VLSI Standards, Inc.

Danna Saechao
Calibration Technician

Tuyet Nguyen
Quality Assurance

[1] *Expanded uncertainties are at the 95% confidence level, as defined by the ISO Guide to the Expression of Uncertainty Measurement (GUM).*

[2] *This certificate does not constitute product endorsement by NVLAP or by any U.S. Government agency.*

Shipment Date:

SHIP DATE:MARCH 2025
Made in USA

Certificate Program: 0358308-000_AD

Page 1 of 2

5 Technology Drive, Milpitas, CA 95035-7916 • (408) 428-1800 • Fax (408) 428-9555 • www.vlsistandards.com

STEP HEIGHT STANDARD

Model Number : SHS - 4.5QC - G2
Serial Number : 30557-75-07
Calibration Date : March 7, 2025

Summary of (step height) input and output quantities:

Input quantity:		Mean value
NISTc	NIST reference (NIST report of calibration)	4.4244 µm
NISTm	NIST reference (VLSI observations)	4.4420 µm
Ym	SHS (VLSI uncorrected observations)	4.5004 µm

Output quantity:		Mean value
Yc	SHS (VLSI corrected value)	4.4824 µm

*Yc = NISTc / NISTm * Ym*

Summary of (step height) standard uncertainty components, combined standard uncertainty, degrees of freedom and expanded uncertainty coverage factor :

	Source of uncertainty	Value (µm)	Partial derivative	Combined component (µm)	Degrees of freedom	"Uncertainty of an uncertainty"
u(NISTc) Combined	NIST reference traceability (NIST report of calibration)	0.02511	1.01313	0.02544	8	25%
u(NISTm) Type A	NIST reference reproducibility (VLSI observations)	0.00055	1	0.00055	8	25%
u(resol) Type B	VLSI instrument vertical resolution	3.4E-06	1	3.4E-06	5000	1%
u(NISTmr) Combined	NIST reference reproducibility (VLSI corrected) .... *u(NISTmr) = sqrt{[u(NISTm)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00061	1.00910	0.00061	12	
u(Ym) Type A	SHS step height uniformity (VLSI observations)	0.00208	1	0.00208	8	25%
u(resol') Type B	VLSI instrument precision	2.6E-04	1	2.6E-04	8	
u(Ymr) Combined	SHS step height measurement (VLSI corrected) .... *u(Ymr) = sqrt{[u(Ym)]^2 + [u(prec)]^2 + [u(resol)]^2}*	0.00210	0.99602	0.00209	8	

u(Yc) **SHS combined standard uncertainty**

*u(Yc) = sqrt {[Ym / NISTm * u(NISTc)]^2 + [Ym * NISTc / NISTm^2 * u(NISTmr)]^2 +*
*+ [NISTc / NISTm * u(Ymr)]^2}*

u(Yc)		degrees of freedom ν (eff)	coverage t - factor
0.02553	µm	**8**	**2.31**

Certificate Program: 0358308-000_AD

Page 2 of 2

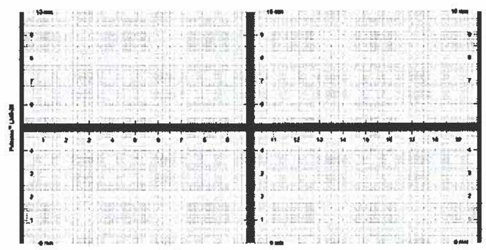
APPENDIX B

AISthesis Products, Inc.

Advanced Imaging Products for Nanotechnology, Engineering and Life Sciences

Certificate of Calibration

Pelcotec™ LMS-20 Low Magnification Silicon Calibration Standard



Product Number: LMS-20C

Product Description: 20 x 10mm Pelcotec™ 1mm Low Magnification Calibration Standard

Product Serial Number: TJ05-033

The accuracy of these products was determined by reference comparison to working standards traceable to the National Institute of Standards and Technology (NIST), Test No. 861/280822-11.

Horizontal

Line length	Certified distance	Standard Deviation (1σ)	Total expanded uncertainty (3σ)
0-1 mm	1.00 mm	±0.10%	±0.30%
0-2 mm	2.00 mm	±0.10%	±0.30%
0-3 mm	3.00 mm	±0.10%	±0.30%
0-4 mm	4.00 mm	±0.10%	±0.30%
0-5 mm	5.00 mm	±0.10%	±0.30%
0-6 mm	6.00 mm	±0.10%	±0.30%
0-7 mm	7.00 mm	±0.10%	±0.30%
0-8 mm	8.00 mm	±0.10%	±0.30%
0-9 mm	9.00 mm	±0.10%	±0.30%
0-10 mm	10.00 mm	±0.10%	±0.30%
0-11 mm	11.00 mm	±0.10%	±0.30%
0-12 mm	12.00 mm	±0.10%	±0.30%
0-13 mm	13.00 mm	±0.10%	±0.30%
0-14 mm	14.00 mm	±0.10%	±0.30%
0-15 mm	15.00 mm	±0.10%	±0.30%
0-16 mm	16.00 mm	±0.10%	±0.30%
0-17 mm	17.00 mm	±0.10%	±0.30%
0-18 mm	18.00 mm	±0.10%	±0.30%
0-19 mm	19.00 mm	±0.10%	±0.30%
0-20 mm	20.00 mm	±0.10%	±0.30%
0-0.1 mm	0.10 mm	±0.10%	±0.30%
0-0.2 mm	0.20 mm	±0.10%	±0.30%
0-0.3 mm	0.30 mm	±0.10%	±0.30%

0-0.4 mm	0.40 mm	±0.10%	±0.30%
0-0.5 mm	0.50 mm	±0.10%	±0.30%
0-0.6 mm	0.60 mm	±0.10%	±0.30%
0-0.7 mm	0.70 mm	±0.10%	±0.30%
0-0. 8mm	0.80 mm	±0.10%	±0.30%
0-0.9 mm	0.90 mm	±0.10%	±0.30%
0-0.01 mm	0.010 mm	±0.43%	±1.30%
0-0.02 mm	0.020 mm	±0.21%	±0.63%
0-0.03 mm	0.030 mm	±0.11%	±0.33%
0-0.04 mm	0.040 mm	±0.10%	±0.30%
0-0.05 mm	0.050 mm	±0.10%	±0.30%
0-0.06 mm	0.060 mm	±0.10%	±0.30%
0-0.07 mm	0.070 mm	±0.10%	±0.30%
0-0.08 mm	0.080 mm	±0.10%	±0.30%
0-0.09 mm	0.090 mm	±0.10%	±0.30%

Vertical (Center Line)

Line length	**Certified distance**	**Standard Deviation (1σ)**	**Total expanded uncertainty (3σ)**
0-1 mm	1.00 mm	±0.10%	±0.30%
0-2 mm	2.00 mm	±0.10%	±0.30%
0-3 mm	3.00 mm	±0.10%	±0.30%
0-4 mm	4.00 mm	±0.10%	±0.30%
0-5 mm	5.00 mm	±0.10%	±0.30%
0-6 mm	6.00 mm	±0.10%	±0.30%
0-7 mm	7.00 mm	±0.10%	±0.30%
0-8 mm	8.00 mm	±0.10%	±0.30%
0-9 mm	9.00 mm	±0.10%	±0.30%
0-10 mm	10.00 mm	±0.10%	±0.30%
0-0.1 mm	0.10 mm	±0.10%	±0.30%
0-0.2 mm	0.20 mm	±0.10%	±0.30%
0-0.3 mm	0.30 mm	±0.10%	±0.30%
0-0.4 mm	0.40 mm	±0.10%	±0.30%
0-0.5 mm	0.50 mm	±0.10%	±0.30%
0-0.6 mm	0.60 mm	±0.10%	±0.30%
0-0.7 mm	0.70 mm	±0.10%	±0.30%
0-0.8 mm	0.80 mm	±0.10%	±0.30%
0-0.9 mm	0.90 mm	±0.10%	±0.30%
0-0.01 mm	0.010 mm	±0.43%	±1.30%
0-0.02 mm	0.020 mm	±0.21%	±0.63%
0-0.03 mm	0.030 mm	±0.11%	±0.33%
0-0.04 mm	0.040 mm	±0.10%	±0.30%
0-0.05 mm	0.050 mm	±0.10%	±0.30%
0-0.06 mm	0.060 mm	±0.10%	±0.30%
0-0.07 mm	0.070 mm	±0.10%	±0.30%
0-0.08 mm	0.080 mm	±0.10%	±0.30%
0-0.09 mm	0.090 mm	±0.10%	±0.30%

The total expanded uncertainty includes both Type A and Type B uncertainties corrected for sample size using an appropriate Student t-factor.

Equipment used:

Instrument	Manufacturer	Serial #	Objective Lenses	NIST Certified CD	Repeatability
Light Microscope	Leitz Type 020-435.034	562 214	10x NPL, 0.2N.A, 20x LL, 0.4N.A. & 50X DF, 0.6N.A.	CD-PG01-0211	0.07%

D. S. Finch
Certified by

Signature

November 1st, 2024
Date

P.O. Box 1950, Clyde, North Carolina 28721 Tel: 828.627.6555 E-mail: CD@aisthesisproducts.com