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Modules of Elasticity and Thermal Expansion Coefficient of ITO Film

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Disclaimer

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Introduction

The purpose of this experiment was to determine the modulus of elasticity (E) and thermal expansion coefficient (α) of RF sputtered Indium Tin Oxide (ITO) as a function of temperature (T), and to collect ITO film stress data.

In order to accomplish that goal, the Toho FLX-2320-S thin film stress measurement machine was used to collect both single stress and stress-temperature data for ITO coated fused silica and sapphire substrates. The stress measurement function of the FLX-2320-S cannot be used to calculate the elastic modulus of the film because the Stoney formula (shown below) incorporates the elastic modulus of the substrate, rather than of the film itself.

$$\sigma_f = \frac{E_s}{1-\nu_s}\left(\frac{h_s^2 k}{6h_f}\right) \tag{1}$$

where σ_f is the film stress, E_s is the elastic modulus of the substrate, ν_s is Poisson's ratio of the substrate, $h_{s,f}$ is the thickness of the substrate and film, and k is the curvature of the wafer. However, the thin film stress measurement tool has an elastic and expansion coefficient function that uses film stress as a function of temperature of two different substrates coated with the same film to calculate and plot the linear thermal expansion coefficient with respect to time, as well as the biaxial modulus of the film. In order to accomplish this, the FLX-2320 substitutes the data collected for each of the two substrates into separate Equation 2's, and then solves the two resulting equations for the film's biaxial modulus $(\frac{E}{1-\nu})$ and coefficient of thermal expansion (α_f).

$$\frac{d\sigma(f)}{dT} = \left(\frac{E}{1-\nu}\right)_f(\alpha_s - \alpha_f) \tag{2}$$

Experimental Results and Discussion

Before coating the fused silica and sapphire substrates with the ITO film, their thickness was measured and blank measurements using the FLX-2320-S were taken, which determined the curvature of the substrate surface. It was found that the fused silica sapphire substrates had thicknesses of approximately 525 µm and 435 µm, respectively. The radius of curvature of the fused silica was -4.97 m, and it was 2.09 m for the sapphire. The samples were then coated with the ITO film at the Vacuum Process Lab (VPL). Both of the samples were coated in the same deposition run, and the average thickness of the ITO film was determined to be 2,157 Ångströms using a witness strip.

Once coated, it was determined using the confocal that the samples were in pristine condition, with no obvious signs of damage or debris. The four point probe was then used to measure the electrical resistivity of the ITO film, which was found to be between 51 and 92 ohms per square for both substrates. The FLX-2320 was then used to collect stress data as a function of temperature for each of the samples. The substrates were heated from room temperature to 350 degrees Celsius in approximately 30 minutes, and then they were immediately allowed to cool. Throughout the heating, stress measurements were collected. This initial annealing run caused the electrical resistivity of the ITO film to increase significantly to values between 281 and 994 ohms per square. It was determined by confocal inspection that the film was not completely annealed on either substrate, which was causing the increase in electrical resistivity. The two substrates were again ramped to 350 degrees Celsius in 30 minutes; however, they were held at that temperature for 20 minutes. Following this run, the electrical resistivity of the film decreased to between 185 and 484 ohms per square, and it was determined using the confocal that the films were completely annealed. In the final temperature-dependent stress experiment, the samples were raised from room temperature to 450 degrees Celsius in 1 hour and 43 minutes, which equates to a ramp of 4.2 degrees Celsius per minute. The electrical resistivity of the ITO film after this final run was approximately 100 ohms per square for the fused silica substrate and 250 ohms per square for the sapphire substrate. Figures 1 and 2 below show the film stress for each sample as a function of temperature for the final FLX-2320 run.

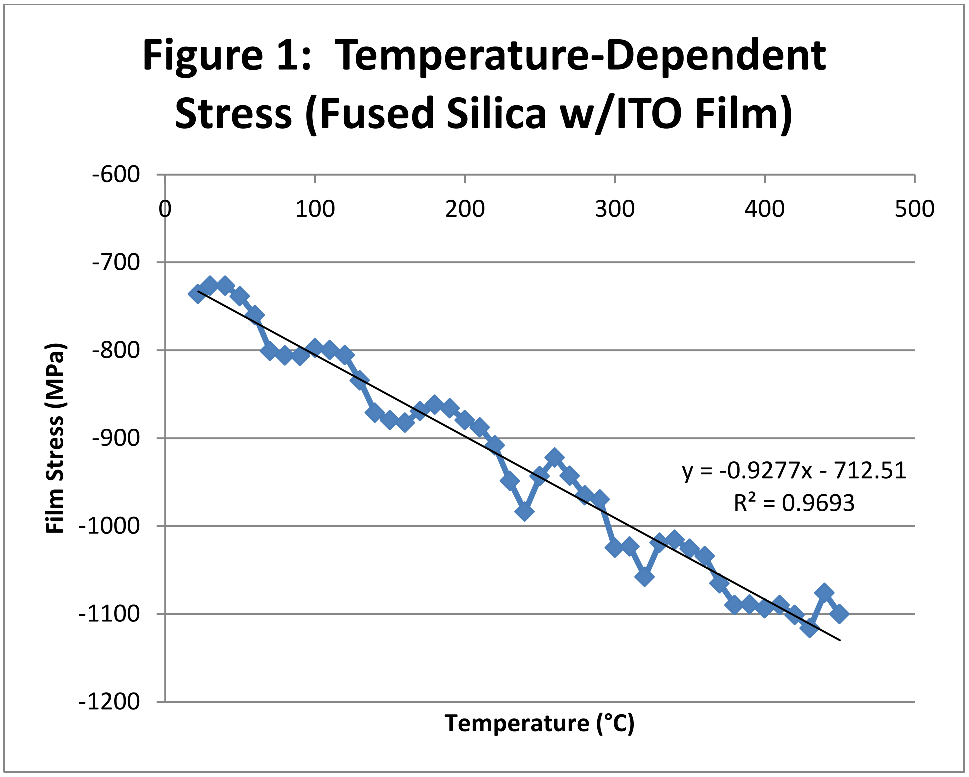


Figure 1: Temperature-Dependent Stress (Fused Silica w/ITO Film)
-600
-700
-800
-900
-1000
-1100
-1200
0
100
200
300
400
500
Film Stress (MPa)
y = -0.9277x - 712.51
R² = 0.9693
Temperature (°C)

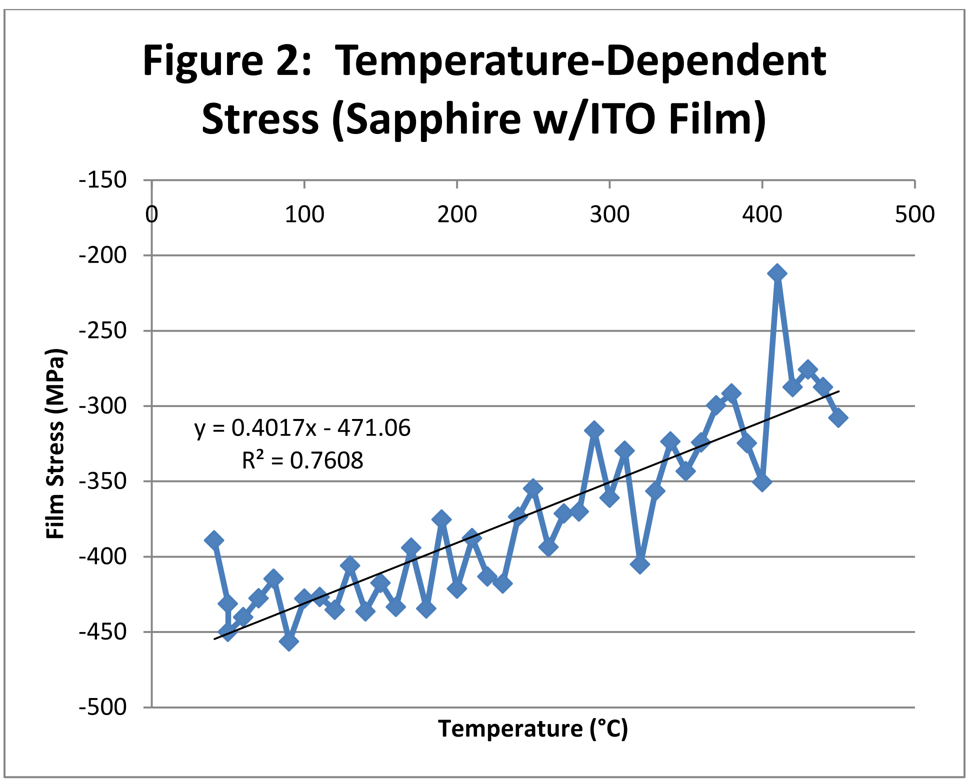


Figure 2: Temperature-Dependent Stress (Sapphire w/ITO Film)
-150
-200
-250
-300
-350
-400
-450
-500
0
100
200
300
400
500
Film Stress (MPa)
y = 0.4017x - 471.06
R² = 0.7608
Temperature (°C)

Data Analysis

Using Equation 2, the elastic and expansion coefficient function in the thin film stress measurement machine graphs the film's linear coefficient of thermal expansion (CTE) with respect to temperature and calculates the CTE equation and the average biaxial modulus of the film.

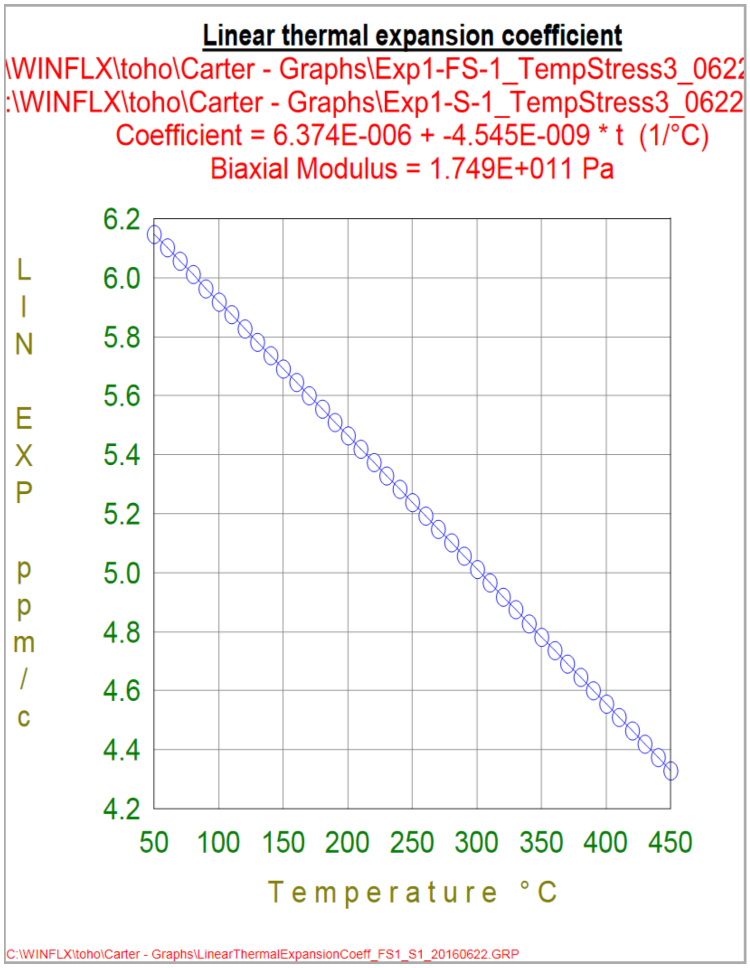


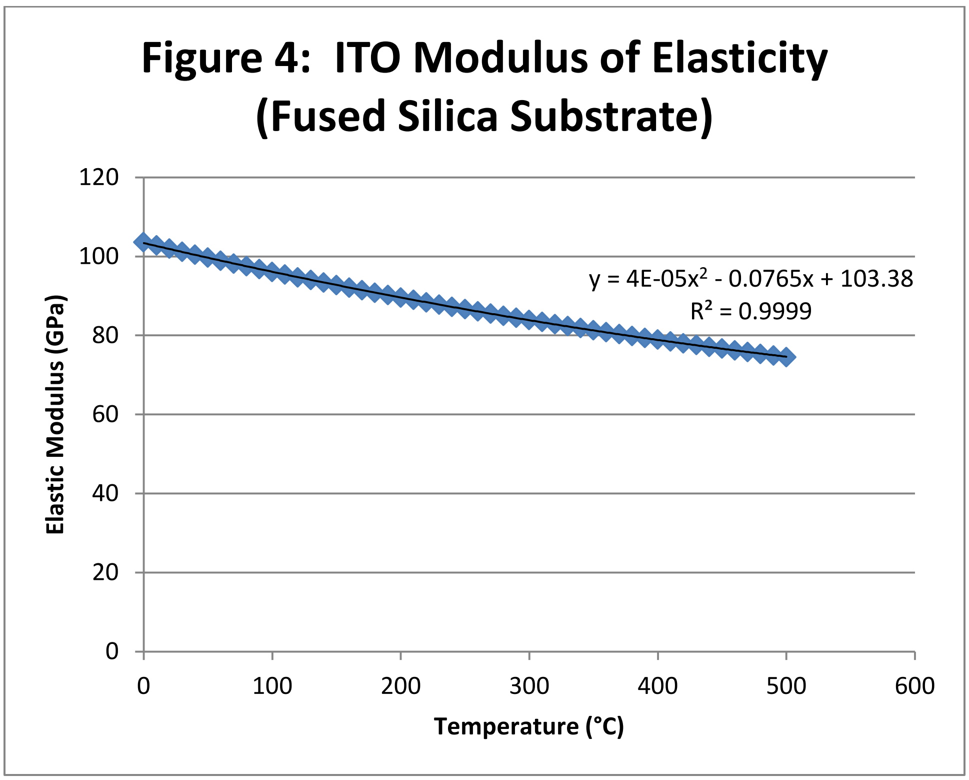
Figure 3: Linear Thermal Expansion Coefficient of ITO

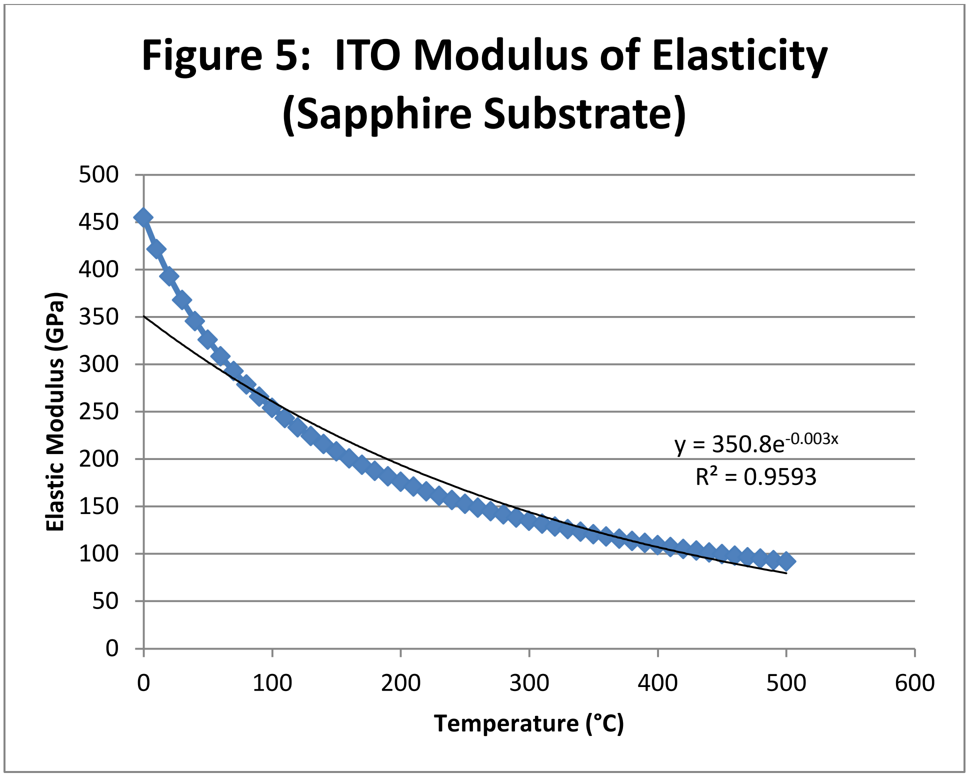
Using the published ITO Poisson ratio of 0.35, it was determined that the average modulus of elasticity of the film over the range of temperatures at which film stress was measured is 113.7 GPa, which is very near the published value of 116 GPa. The first step in calculating the ITO's elastic modulus as a function of temperature was to find an equation that describes the relationship between the film stress and temperature, which was accomplished using a trend line in Excel and can be seen in Figures 1 and 2. This equation was then differentiated and substituted, along with the equation of the ITO's linear CTE, the published substrate CTE, and the published ITO Poisson ratio, into Equation 2. The resulting equation was then solved for the ITO film's elastic modulus (E). Equations 3 and 4 show this equation for the fused silica and sapphire substrates, respectively.

$$E = \frac{-0.9277\ MPa}{(0.55*10^{-6} - 6.374*10^{-6} - 4.545*10^{-9}*T)\frac{1}{°C}}(1 - 0.35) \tag{3}$$

$$E = \frac{0.4017\ MPa}{(5.8*10^{-6} - 6.374*10^{-6} - 4.545*10^{-9}*T)\frac{1}{°C}}(1 - 0.35) \tag{4}$$

Finally, the ITO's elastic modulus was calculated for a range of temperatures between 0 and 500 degrees Celsius using Equations 3 and 4 and graphed (Figures 4 and 5).





Conclusion

The average modulus of elasticity of ITO based on the calculations performed using Equations 3 and 4 is 136.62 GPa. Based on the published elastic modulus of ITO film, the calculated value has an error of 17.78%, and the 113.7 GPa value provided by the thin film stress measurement machine only has a 1.98% error. In order to improve the analysis of the data, the stress versus temperature graphs could be smoothed, which would provide for a better trend line and ultimately more accurate elastic modulus calculations.

Appendix A

Summary of Parameters Used in Analysis:

	Fused Silica	Sapphire	ITO Film
Sample ID	Exp1-FS-1	Exp1-S-1	N/A
CTE	0.55×10^{-6} 1/°C	5.8×10^{-6} 1/°C	$6.374 \times 10^{-6} - 4.545 \times 10^{-9}T$ 1/°C
Poisson Ratio	0.17	0.25	0.35
Published Elastic Modulus	73 GPa	345 GPa	116 GPa
FLX Calculated Elastic Modulus	N/A	N/A	113.7 GPa
Thickness	525 µm	435 µm	215.7 nm

1st FLX Temperature Cycle of Exp1-FS-1 and Diamond Substrate (6-20-16)

Start Temp: 45°C

End Temp: 350°C

Start Time: 1340

End Time: 1415

ID: FS1

Comment: FS1_Temp

Film Thickness: 2157 Å

Orientation: 0°

Laser: 670 nm

Recipe:

Temp (°C)	Time (min.)	Measurements	Ramp (°C/min.)
50	4	5	1.3
100	4	10	12.5
150	4	10	12.5
200	4	10	12.5
250	4	10	12.5
300	4	10	12.5
350	4	10	12.5

1st FLX Temperature Cycle of Exp1-S-1 (6-20-16)

Start Temp: 22°C

End Temp: 352°C

Start Time: 1207

End Time: 1237

ID: S1

Comment: S1_Temp

Film Thickness: 2157 Å

Orientation: 0°

Laser: 780 nm

Recipe:

Temp (°C)	Time (min.)	Measurements	Ramp (°C/min.)
50	4	5	7.3
100	4	10	12.5
150	4	10	12.5
200	4	10	12.5
250	4	10	12.5
300	4	10	12.5
350	4	10	12.5

2nd FLX Temperature Cycle of Exp1-FS-1 and Diamond Substrate (6-21-16)

Start Temp: 24°C

End Temp: 350°C

Start Time: 0953

End Time: 1043

ID: FS1

Comment: FS1_Temp2

Film Thickness: 2157 Å

Orientation: 0°

Laser: 670 nm

Recipe:

Temp (°C)	Time (min.)	Measurements	Ramp (°C/min.)
50	4	5	6.5
100	4	10	12.5
150	4	10	12.5
200	4	10	12.5
250	4	10	12.5
300	4	10	12.5
350	4	10	12.5
350	20	5	0.0

2nd FLX Temperature Cycle of Exp1-S-1 (6-21-16)

Start Temp: 40°C

End Temp: 350°C

Start Time: 1155

End Time: 1245

ID: S1

Comment: S1_Temp2

Film Thickness: 2157 Å

Orientation: 0°

Laser: 670 nm

Recipe:

Temp (°C)	Time (min.)	Measurements	Ramp (°C/min.)
50	4	5	2.5
100	4	10	12.5
150	4	10	12.5
200	4	10	12.5
250	4	10	12.5
300	4	10	12.5
350	4	10	12.5
350	20	5	0.0

3rd FLX Temperature Cycle of Exp1-FS-1 (6-22-16)

Start Temp: 22°C

End Temp: 450°C

Start Time: 1105

End Time: 1250

ID: FS1

Comment: FS1_Temp3

Film Thickness: 2157 Å

Orientation: 0°

Laser: 670 nm

Recipe:

Temp (°C)	Time (min.)	Measurements	Ramp (°C/min.)
50	7	10	4.0
100	12	10	4.2
150	12	10	4.2
200	12	10	4.2
250	12	10	4.2
300	12	10	4.2
350	12	10	4.2
400	12	10	4.2
450	12	10	4.2

3rd FLX Temperature Cycle of Exp1-S-1 (6-22-16)

Start Temp: 41°C

End Temp: 450°C

Start Time: 1411

End Time: 1557

ID: S1

Comment: S1_Temp3

Film Thickness: 2157 Å

Orientation: 0°

Laser: 670 nm

Recipe:

Temp (°C)	Time (min.)	Measurements	Ramp (°C/min.)
50	7	10	1.3
100	12	10	4.2
150	12	10	4.2
200	12	10	4.2
250	12	10	4.2
300	12	10	4.2
350	12	10	4.2
400	12	10	4.2
450	12	10	4.2

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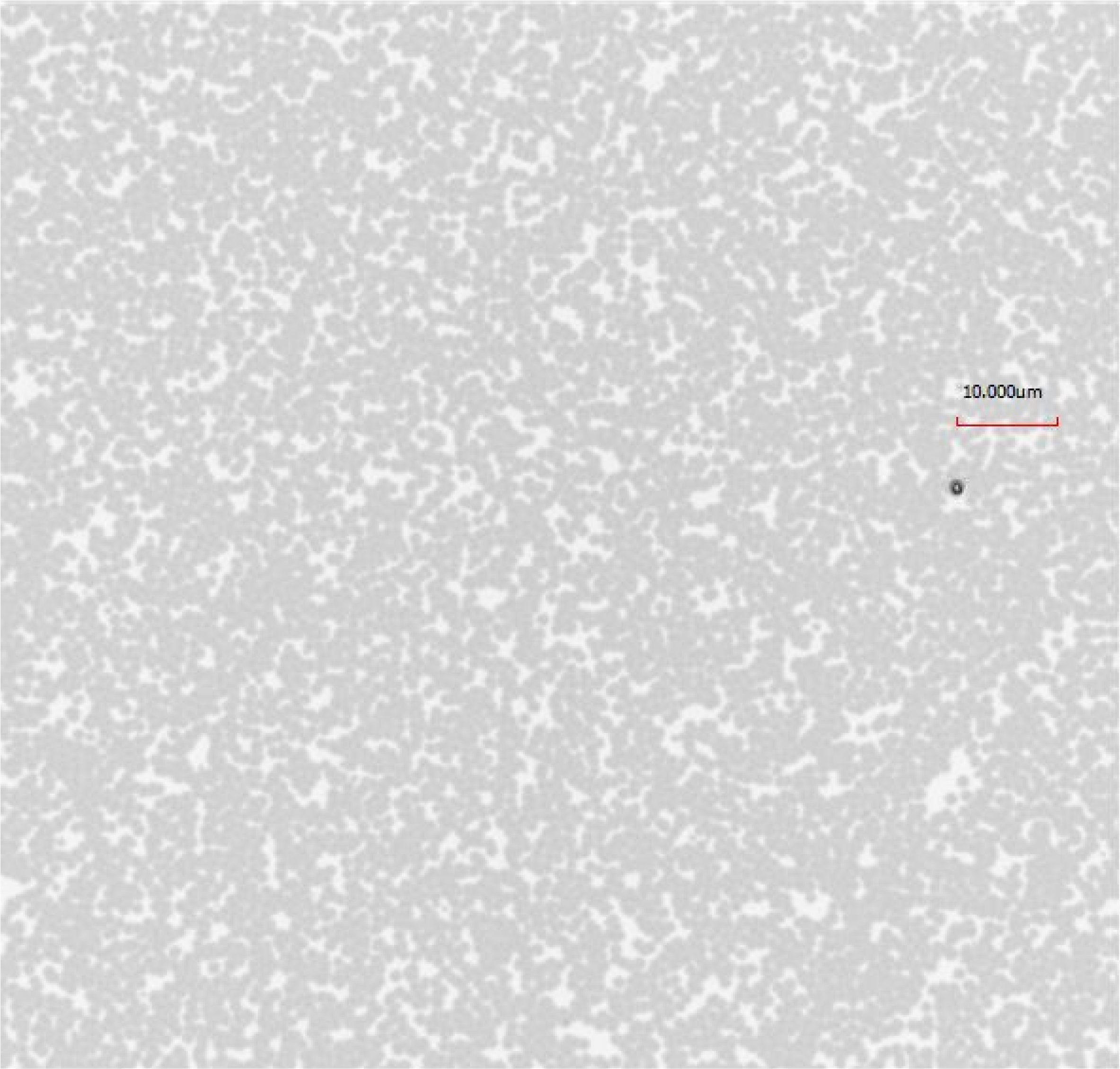
FS1_100x_afterquick350_longer_stay.vk4



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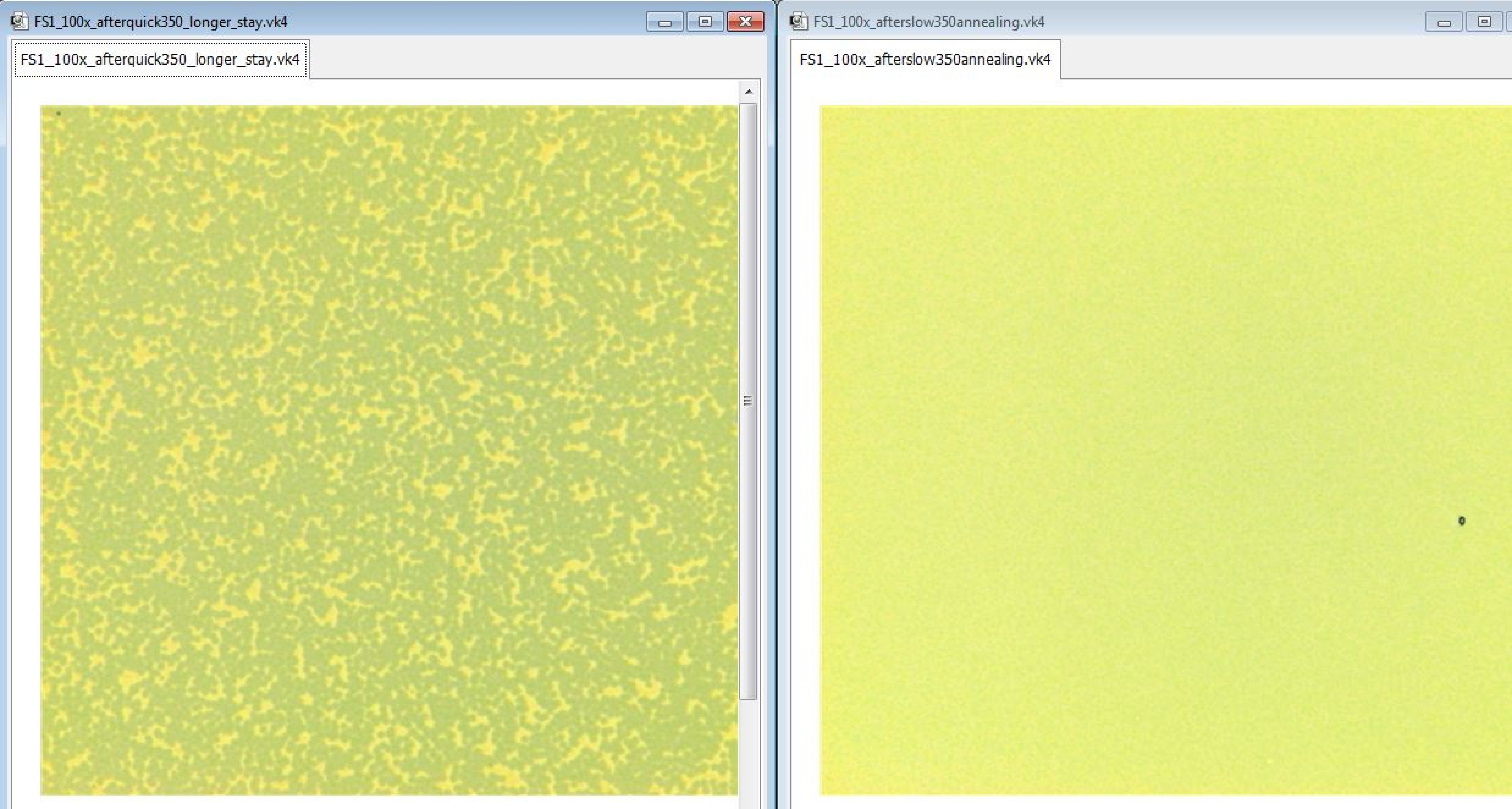
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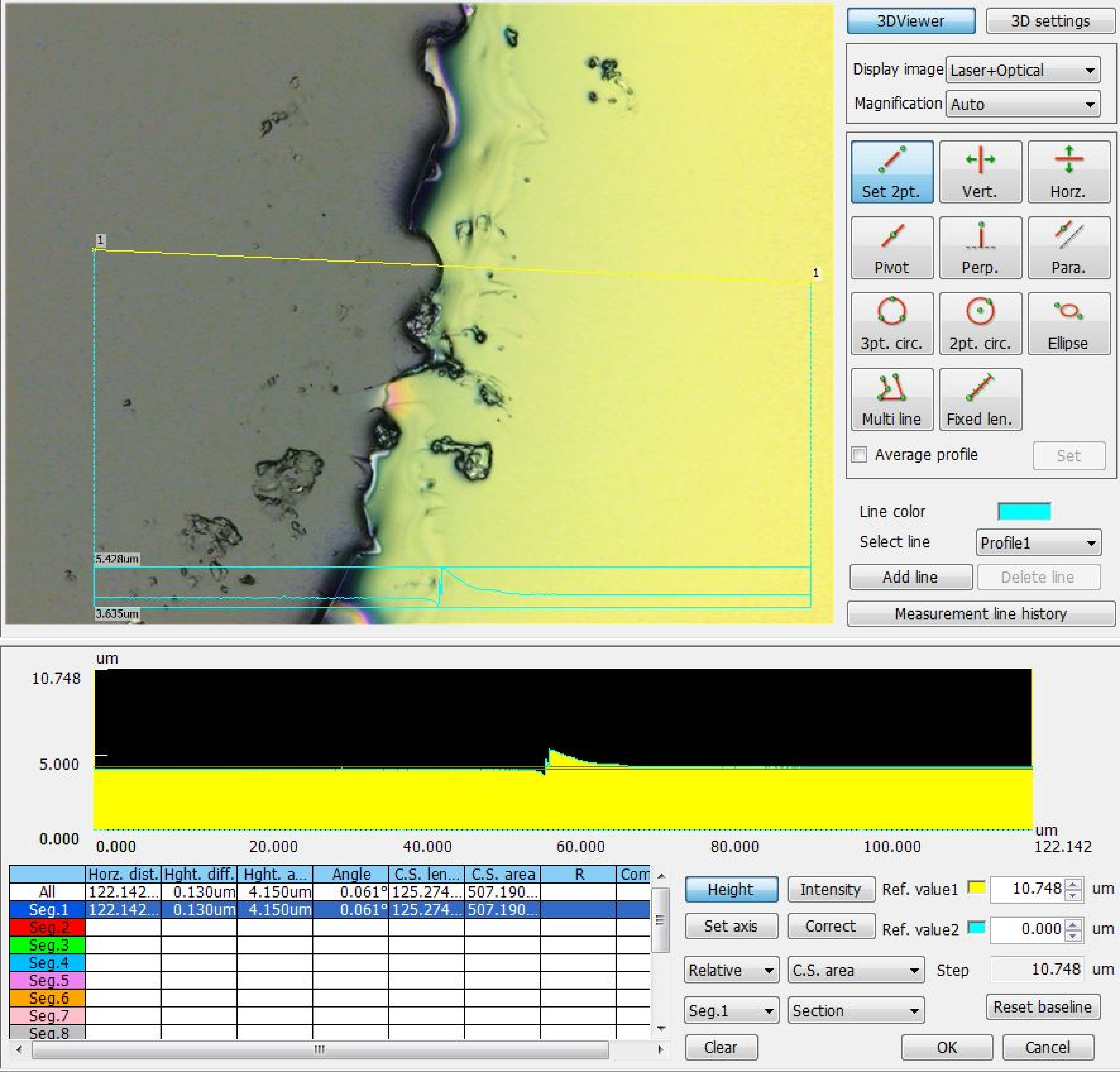

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Display image
Laser+Optical
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Vert.
Horz.
Pivot
Perp.
Para.
3pt. circ.
2pt. circ.
Ellipse
Multi line
Fixed len.
Average profile
Set
Line color
Select line
Profile1
Add line
Delete line
Measurement line history
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3.635um
um
10.748
5.000
0.000
0.000
20.000
40.000
60.000
80.000
100.000
122.142
Horz. dist.
Hght. diff.
Hght. a...
Angle
C.S. len...
C.S. area
R
Com
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Seg.2
Seg.3
Seg.4
Seg.5
Seg.6
Seg.7
Seg.8
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Intensity
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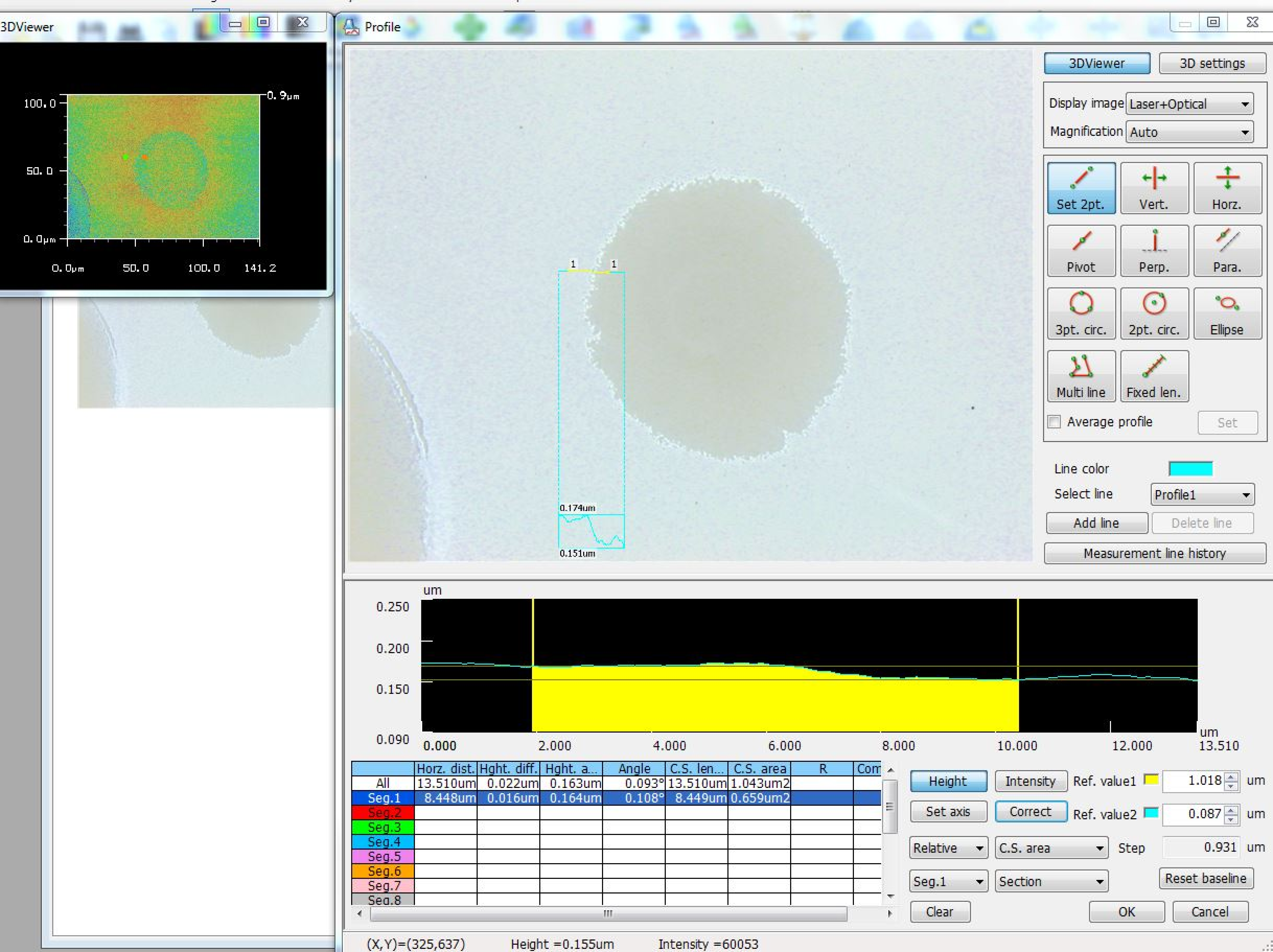
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Ellipse
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Fixed len.
Average profile
Set
Line color
Select line Profile1
Add line
Delete line
Measurement line history
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0.151um
um
0.250
0.200
0.150
0.090
0.000
2.000
4.000
6.000
8.000
10.000
12.000
um 13.510
Horz. dist. | Hght. diff. | Hght. a... | Angle | C.S. len... | C.S. area | R | Com
All 13.510um 0.022um 0.163um 0.093° 13.510um 1.043um2
Seg.1 8.448um 0.016um 0.164um 0.108° 8.449um 0.659um2
Seg.2
Seg.3
Seg.4
Seg.5
Seg.6
Seg.7
Seg.8
Height
Intensity
Ref. value1 1.018 um
Set axis
Correct
Ref. value2 0.087 um
Relative
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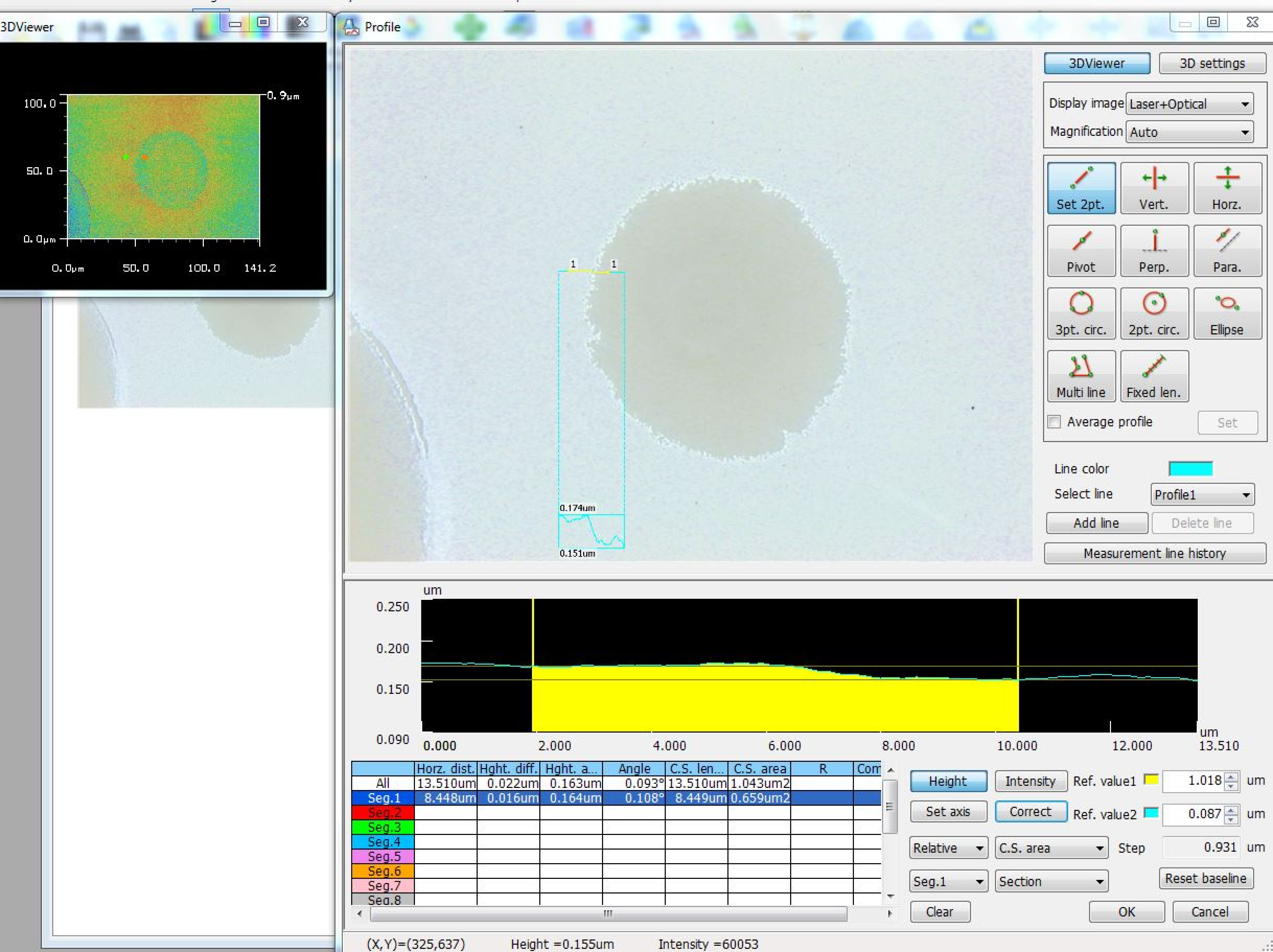
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50.0
100.0
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Magnification
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Vert.
Horz.
Pivot
Perp.
Para.
3pt. circ.
2pt. circ.
Ellipse
Multi line
Fixed len.
Average profile
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Line color
Select line
Profile1
Add line
Delete line
Measurement line history
0.174um
0.151um
um
0.250
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0.000
2.000
4.000
6.000
8.000
10.000
12.000
um
13.510
Horz. dist. | Hght. diff. | Hght. a... | Angle | C.S. len... | C.S. area | R | Com
All | 13.510um | 0.022um | 0.163um | 0.093° | 13.510um | 1.043um2
Seg.1 | 8.448um | 0.016um | 0.164um | 0.108° | 8.449um | 0.659um2
Seg.2
Seg.3
Seg.4
Seg.5
Seg.6
Seg.7
Seg.8
Height
Intensity
Ref. value1
1.018
um
Set axis
Correct
Ref. value2
0.087
um
Relative
C.S. area
Step
0.931
um
Seg.1
Section
Reset baseline
Clear
OK
Cancel
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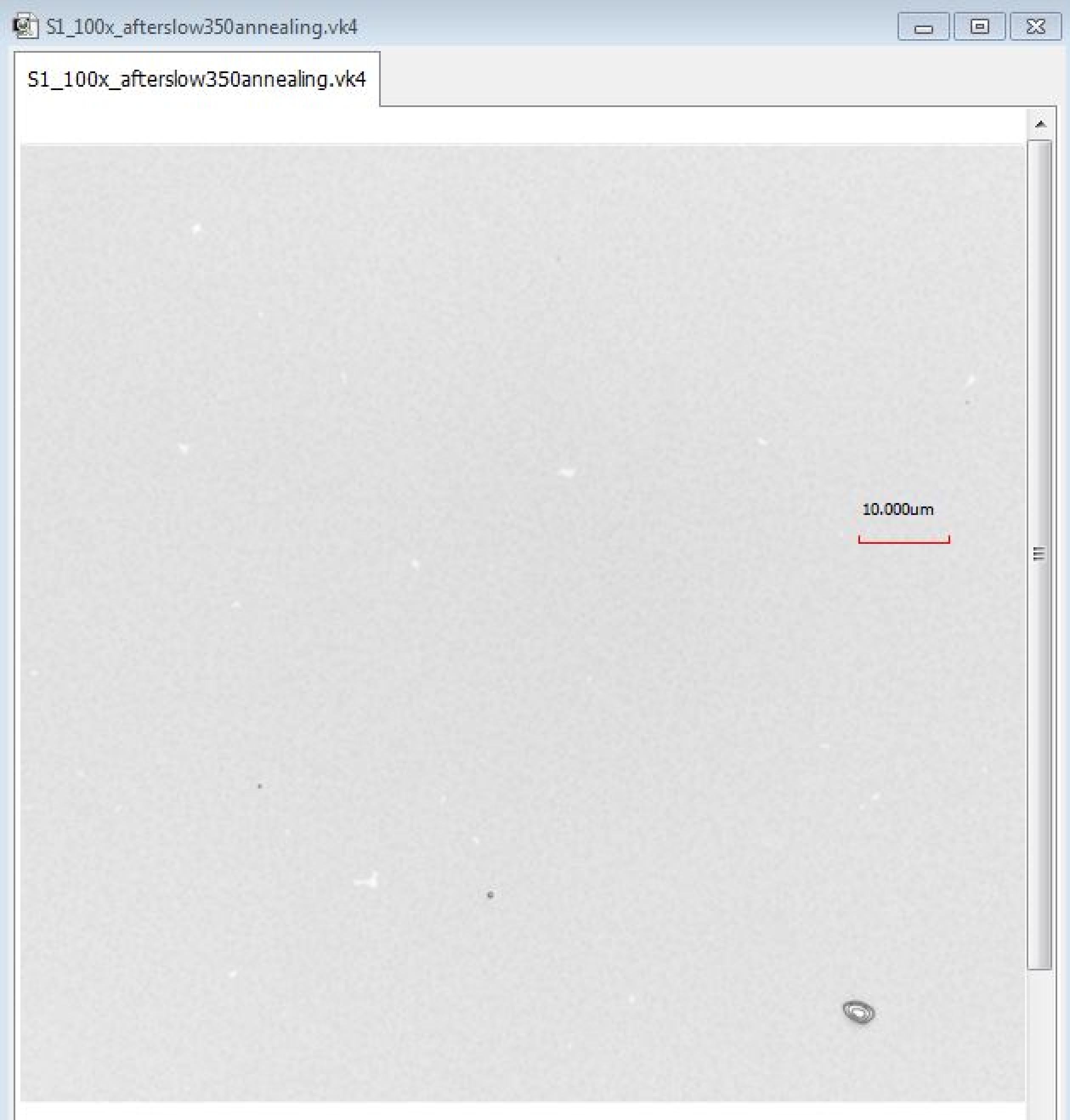
Intensity =60053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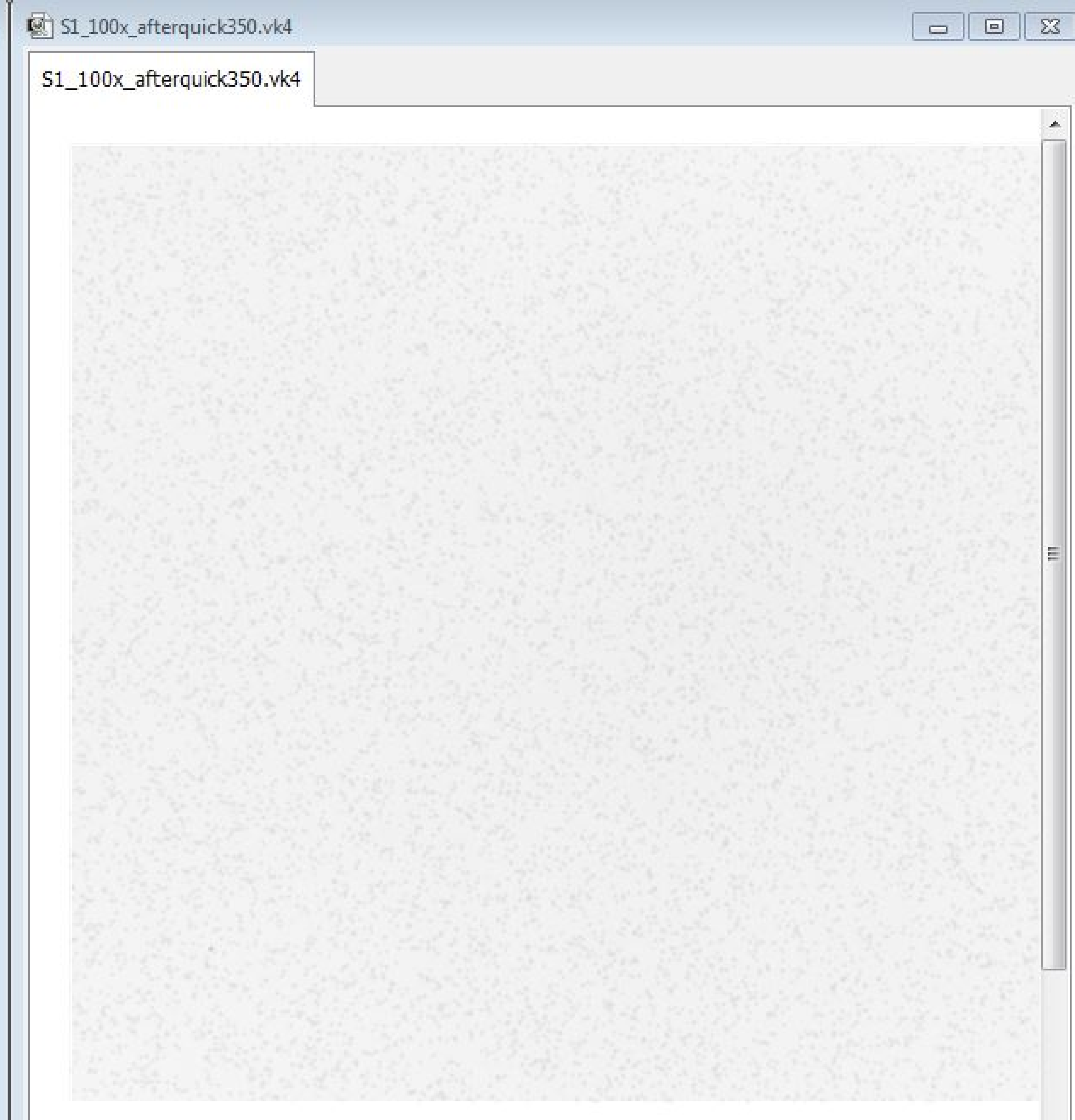
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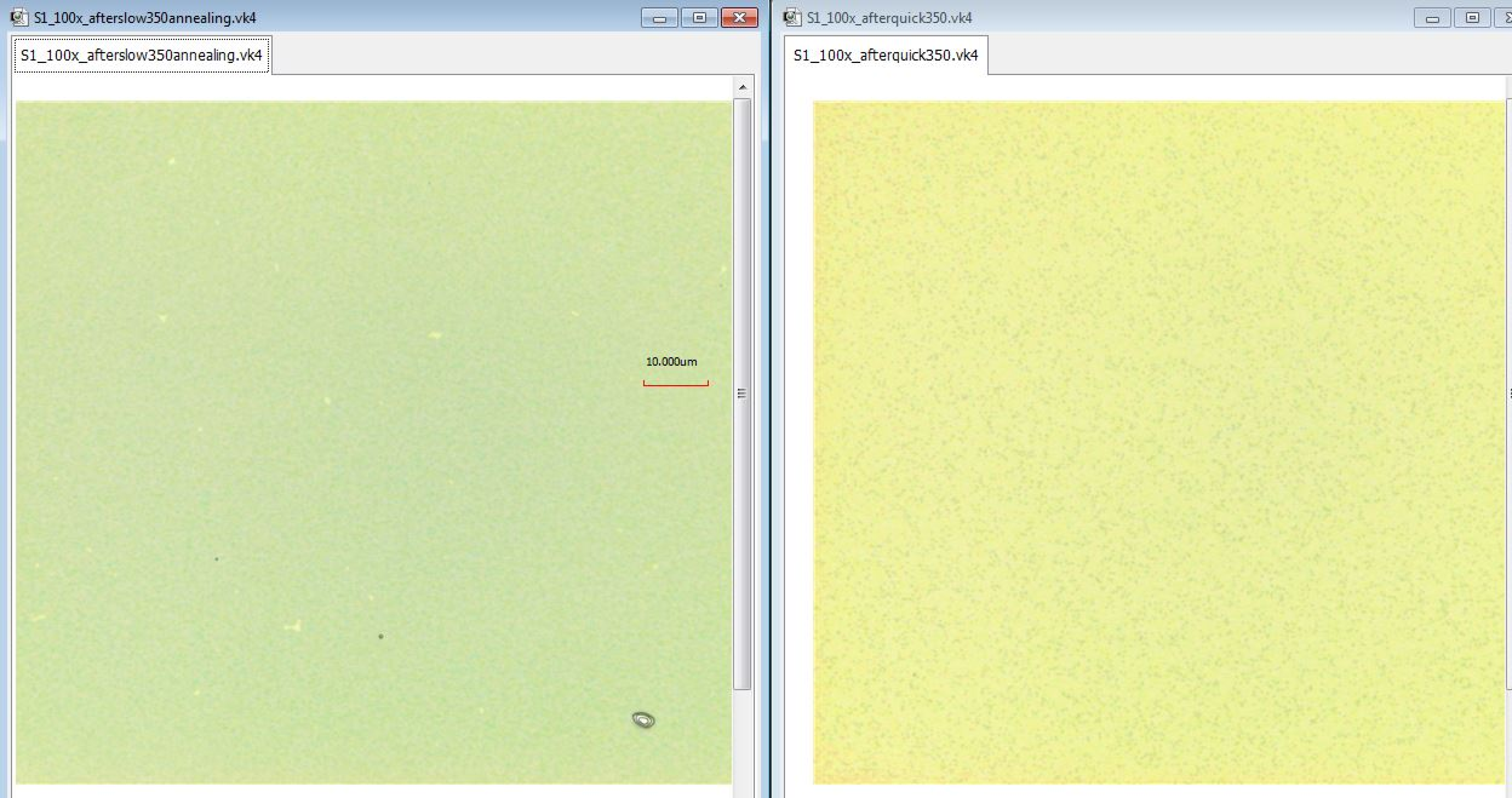
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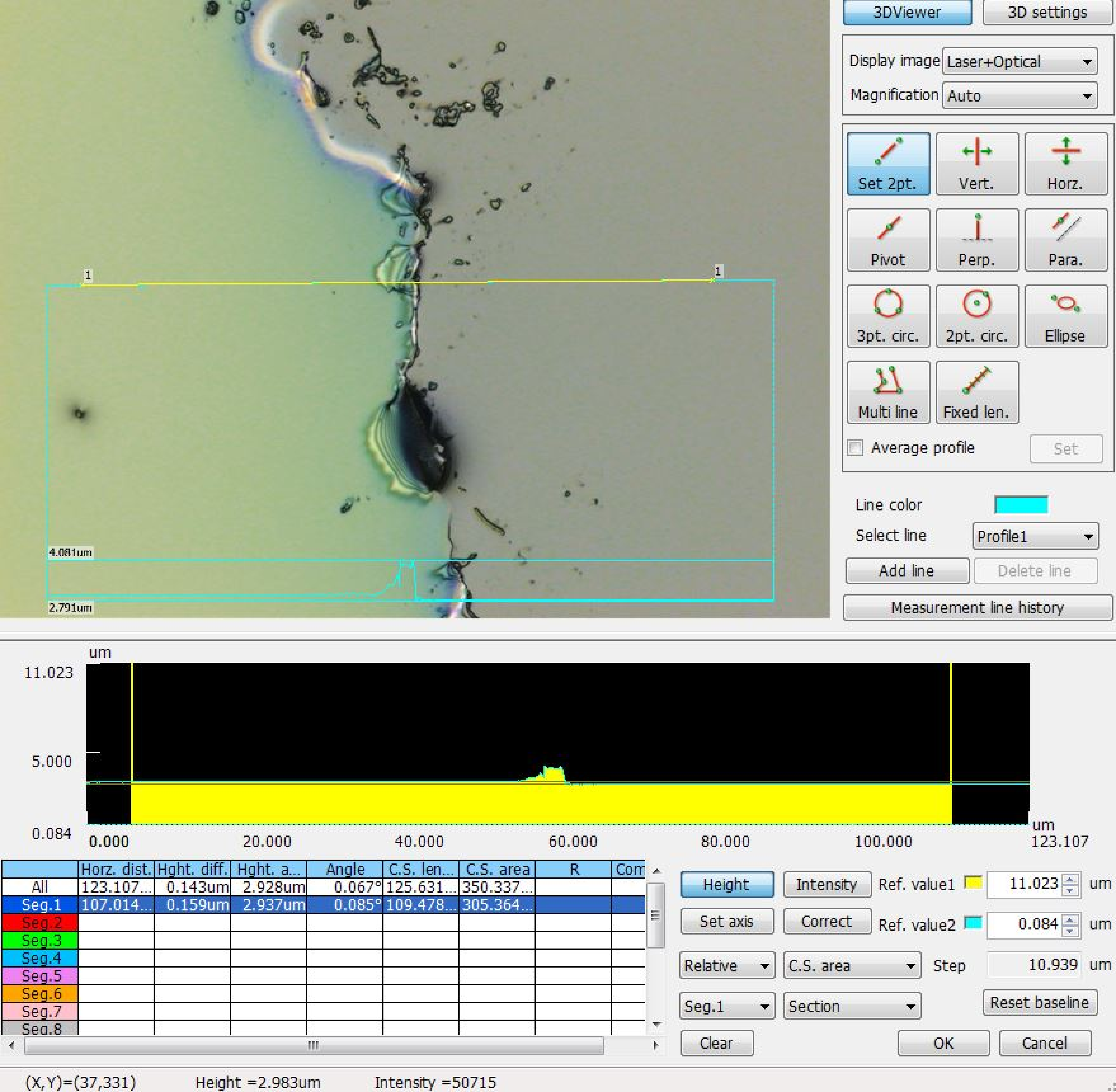
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Ellipse
Multi line
Fixed len.
Average profile
Set
Line color
Select line
Profile1
Add line
Delete line
Measurement line history
4.081um
2.791um
um
11.023
5.000
0.084
0.000
20.000
40.000
60.000
80.000
100.000
um
123.107
Horz. dist. | Hght. diff. | Hght. a... | Angle | C.S. len... | C.S. area | R | Com
All | 123.107... | 0.143um | 2.928um | 0.067° | 125.631... | 350.337...
Seg.1 | 107.014... | 0.159um | 2.937um | 0.085° | 109.478... | 305.364...
Seg.2
Seg.3
Seg.4
Seg.5
Seg.6
Seg.7
Seg.8
Height
Intensity
Ref. value1
11.023
um
Set axis
Correct
Ref. value2
0.084
um
Relative
C.S. area
Step
10.939
um
Seg.1
Section
Reset baseline
Clear
OK
Cancel
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