Updated: November, 2010

MProbe: Measurement Guide Thin-films measurement using Reflectance or/and Transmittance spectroscopy

It is easy to be a measurement expert with MProbe



Distribution in the UK & Ireland



Characterisation, Measurement & Analysis

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The Purpose

This document describes the measurement procedure, basic use of TFCompanion software and set-up instructions for installation/assembly of the MProbe system.

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Fig. 1a Measurement of the thickness of 200nm oxide (SiO2 200nm/Si) with MProbe Vis system (Wavelength measurement range:400-1000nm)



Fig. 1b Measurement of the thickness of 200nm oxide (SiO2 200nm/Si) with MProbe UVVisSR system (Measurement range: 200-1000nm)

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1. Optical measurements.

Optical thin-film measurements are indirect - they, actually, include two steps:

- 1. Measurement of reflectivity/transmittance spectrum of the sample This step includes collecting the signal from spectrometer (raw data) and
 - normalizing it using calibration data.
- 2. Analysis of measured data using optical model of the sample (filmstack). Software compares measured and simulated data and infers the filmstack parameters (calculated thickness and/or optical properties) from the best fit (curve fitting).



TFCompanion software encapsulates details of the measurement process in the *Measurement Recipe* to make it easy and transparent to the user.

There are three types of measurement recipes:

- ✓ Measure ONLY (no calculation). This type of the recipe performs only the first step (as described above) measurement of the normalized reflectance or transmittance. The user can perform the calculation step manually using *Recalculate* button in the main screen. Measure ONLY recipes are typically used for either preliminary measurement on the new samples or to save/export the data for further analysis as later time.
- ✓ Measure & Calculate (no filmstack attached). This is a default type of the recipe that performs two measurement steps automatically. For calculations it uses the current filmstack (filmstack that was set prior to measurement).
- ✓ Measure & Calculate (filmstack attached). This type of the recipe performs two measurement steps automatically, similar to previous recipe type but it also allows specifying the measurement filmstack. When measurement recipe is loaded – the filmstack attached to the recipe is loaded automatically (overrides current filmstack).

Tip. *The type and the details of the recipe can be easily changed in Recipe Dialog (Measure/Review Recipes from the main menu)*

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In most cases, user does not need to create a Measurement recipe explicitly – the default recipe is automatically created when Measure button in pressed.

The Default recipe uses spectrometer and measurement configuration information. The spectrometer information is either read from spectrometer or configuration file and can be edited by the user (see Fig. 2). Measurement configuration is defined in Software Configuration Dialog (Configure/Configure software) and can be modified by the user (see Fig. 3) as well.

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Fig. 2 Hardware configuration dialog. The data is typically read directly from spectrometer or configuration file. User can edit/modify the parameters. After modification, updated configuration can be saved - it will be automatically used the next time software is started.

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Fig 3 Measurement configuration. Defines default measurement conditions and recipe selection options (Configure/Configure Software and select Measurement Configuration tab).

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1.1 Calibration.

The raw intensity signal acquired from spectrometer is a convolution of sample's reflectivity/transmittance and several other factors: spectral radiation of the light source, transmittance of optics, spectral sensitivity of the detector, etc. Calibration is required to extract absolute reflectivity/transmittance of the sample from the raw signal.

Calibration is performed in a two steps process:

1. Measurement of the reference sample.

In case of transmittance calibration – the transmittance of the air (no sample) is used (100% transmittance)

In case of reflectance calibration – a bare Si reference sample is used for UVV is and V is systems and Al sample for NIR systems. (Appropriate calibration sample is supplied with the instrument) It is possible to use other reference samples, with the well known and stable reflectivity, as well.

2. Measurement of dark current signal.

Measurement of the signal with illumination blocked.

Note. For reflectance calibration it is recommended to use an absorbing black sample (provided with instrument). This method works both in the case of a standard reflectance probe and reflectance probe with attached lens. Blocking the light source (e.g. with shutter) will work only in the case of the standard probe since it does not account for the back reflectance from the lens.

A Calibration recipe (a special type of the measurement recipe) manages calibration. In almost all the cases, it is **NOT necessary** to create a calibration recipe manually - it is created automatically.

User is prompted to performed calibration before the first measurement (to force recalibration – select Measure/*Clear Calibration* from the main menu). Calibration recipe is created automatically, on a fly, to match current measurement recipe.

Note. *There are two special cases when calibration recipe needs to be created explicitly:*

✓ Non standard reference sample used for reflectance calibration (standard sample is Si)
 ✓ Two separate calibration recipes are used: one for reference sample, another for dark current measurement. (this is typically used when software is controlled by the external program)

The result of calibration is the mapping between known reflectivity and the raw signal intensity (in units of ADC counts) – an example of calibration result is shown on Fig. 4

Tip. To review current calibration, select Measure/Review Calibration (Reflectance) or Measure/Review Calibration (Transmittance)

The best way to check the calibration is to measure the reference sample (used in calibration) or empty space in case of Transmittance. Known reflectivity of reference sample and measured data should match (Fig. 5). If there is a shift or discrepancy between the data – one needs to recalibrate.



Fig. 4 Results of reflectance calibration. Red line – known reflectivity of the Si wafer LHS axis, blue line – raw signal from spectrometer RHS axis (ADC counts)



Fig. 5 Calibration test – **measurement of Si reference sample**. The blue line is theoretical (simulated) reflectance of the reference sample; the red line is the measured reflectance. A good match verifies that calibration was done well.

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1.2 Practical measurement procedure.

The measurement workflow is presented on Fig. 6.

- 1. Select Measure button
- 2. If no measurement recipe is found the option defined in Measurement configuration is used
- 3. Calibration is checked: if there is no calibration or calibration is not consistent with the measurement user is prompted to calibrate (calibration recipe is created automatically to match current measurement recipe)
- 4. Measurement is taken
- 5. If measurement recipe includes calculation the calculation is performed and results displayed.
- 6. If calculation is not included normalized reflectivity or transmittance spectrum is displayed. User can do calculation manually using Recalculate button.



Fig. 6 Workflow of the measurement process

Salient points:

- ✓ No need to prepare measurement recipe before the start of measurement, in most cases (unless one needs to use advance features)
- ✓ *Calibration recipe is created automatically*

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Calibration Tips.

Tip 1 Calibration needs to be performed at the same conditions as the measurement.

This means that the distance between the end of reflectance probe and the sample should be the same for calibration and measurement sample. If the distance is different – reflectance measurement will be offset - not properly normalized.

Sensitivity to the distance difference depends on particular configuration but, typically, difference <0.1mm does not cause noticeable problems.

There are several options to mitigate this problem:

- 1. Use the reference of exact same thickness as the measured sample.
- 2. Use "scale" correction in software. The "scale" (reflectance) offset parameter is added to fit/calculated values. Additional calculated parameter can reduce the measurement precision: one approach is to measure "scale" factor using a "simple" sample and fix it for use of sample of the same thickness.
- 3. Use a "face-down" measurement configuration (this requires a different configuration set-up: additional table and SMA terminated reflectance probe). In this configuration, both reference and measured samples are placed "face-down" on the table and reflectance probe is placed underneath the sample. This set-up guarantees that the distance to reference sample is always the same as to measured sample independent of sample thickness. This configuration is also very convenient for thin metal tape and other samples that "don't lay flat"
- Tip 2 Reflectivity of the black target (pad) is very low but it is not zero... Depending on sensitivity of the spectrometer and intensity of the light source – this can cause a problem. To mitigate this problem, one can hold the black pad tilted, so that reflected light does not return back to reflectance probe. Check the reflectivity of the black pad using Signal Monitor mode in the software. Tilt the pad to see the effect on the signal.

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Inside Measurement Recipe.

Each measurement recipe has several components:

- ✓ Spectra (Spectrum definition and Measurement conditions)
- ✓ Attributes (recipe type, target hardware, etc)

Spectrum definition includes:

- ✓ Spectral range: it should be within the range of the currently connected spectrometer. (Spectrometer does not have to be physically connected). Optionally, one can selected in the target hardware - this allows software to test that recipe is matching the spectrometer.
- ✓ Number of points/steps
- Measured parameter: Reflectance or Transmittance
 Excluded spectral region

Measurement Conditions:

- ✓ Channel: the spectrometer channel as defined by spectrometer driver
- \checkmark Integration time: fixed or variable
- ✓ Signal adjustment range (% of ADC)
- ✓ Average defines the number of signal averages performed in spectrometer driver
- ✓ Repeat measurement has the same averaging effect (as Average above) but it also calculates actual standard deviation and add it to the measured data
- \checkmark Repeat recipe similar to Repeat measurement but, in case of variable integration time, time is adjusted for each measurement.
- ✓ Resolution number of pixels read independently, e.g. 2 mean that each 2 pixels will be averaged
- ✓ Boxcar smoothing signal smoothing performed by spectrometer driver

Integration time: fixed or variable?

Variable integration time (default) allows software to adjust time to achieve \sim 80-90% of the maximum signal (ADC range). In this case, measurement of different samples or different point on the sample can be done using best signal/noise.

Note. Acceptable ADC % range can be set in the recipe (as of build 02032010). This is particularly useful for short integration time when using spectrometers that support only millisecond integration time adjustment. In spectrometers that support µs granularity of the integration time, practically any range can be satisfied.

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Calculation

Calculation is a process of inferring physical properties of the sample i.e. thickness and/or optical properties based on measured reflectance and/or transmittance. Nonlinear minimization/curve fitting requires that the starting/nominal values of the parameters be relatively close to actual values. For thickness, the difference between starting and measured value is typically <500A (for oxides). If the starting value is too far away – calculation may not converge and will be "trapped" in local minimum.

In some cases, the thickness of the layer is not known a priori and/or can vary in a wide range.

There are two methods that help solving this problem:

- \checkmark The grid search (global search)
- ✓ Power Spectrum Density/ FFT analysis

Grid Search

The grid search defines a grid of starting thickness values to check – the calculation is done for all these values and the best-fit result presented as a solution. For example, if the layer can be in 100nm-1000nm and 19 point grid is selected- calculation will be performed for thicknesses 100nm, 150nm, 200nm, etc. and the best result will be selected. The grid search is working well but has a problem – for a wide range of thicknesses calculation can be time consuming.

FFT analysis

FFT method can be applied only to thick dielectric or lightly absorbing films. It is based on analysis of "fringes" (constructive/deconstructive interference) of the spectrum. This method requires that the measured spectral range include fair amount of oscillation periods. In the visible range, this method is, typically, applicable for layers more than 3μ m thick.

FFT analysis can be used as an independent method for determining thickness (this is a preferred method for more thick films >10um) or used in combination with the curve fitting.

Grid Search can be defined for specific layer(s) of the optical model (filmstack) and is a part of the filmstack.

The type of the calculation: Fit (Marquardt-Levenberg), FFT, FFT+Fit can be defined in the main screen or in Software configuration dialog.

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There are several more advanced calculation options that can be used:

- ✓ Multiple try calculation strategy (defined several models and trigger fit level) this allows to speed up calculation of complex filmstacks
- ✓ Linked layers/materials allows reduction of the number of calculated parameters in cases of multilayer filmstacks with repeated layers (e.g. some anti-reflection coating and heterostructures)
- ✓ Signal scaling allows to correct intensity/ambient light variation in long running in-situ measurements.
- ✓ Multiple samples data analysis. Simultaneously analyze data from several samples. For example, one can use several similar samples with different thickness of one layer to improve the quality of the optical constants calculation (of the specific material)
- ✓ Batch processing of the data. Allows offline analysis of a series of measurements e.g. online measurements, etc.

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1.3 Optimizing measurement precision

Variable integration time (default) in the measurement recipe allows utilizing maximum dynamic range of the electronics (ADC) by adjusting the signal to the 85-95% of the maximum signal.

To adjust integration time properly software needs to know "the real"/experimental maximum signal – this is the signal corresponding to a saturation level. The default maximum signal is defined by the ADC bits e.g. 16 bits correspond to 65536 counts, 14 bits -> 16384 counts. The saturation level signal can be different from the "theoretical" value due to offset in electrical circuit e.g. 16 bits ADC signal may have from 62000 to 66000 maximum counts (for different system) instead of 65536 counts. This offset needs to be corrected.

- 1. Set reflective sample e.g. Si sample
- 2. Start signal monitor (Measure/Signal monitor) from the main menu
- 3. Select Properties/Measurements Condition from the Monitor window menu and adjust integration time to saturate the signal (flat top)
- 4. Select Correct/Saturation Offset from the main menu. The dialog (Fig. 7) will appear
- 5. Select Set Offset current spectrometer configuration will be updated



Fig. 7 Offset correction.

This dialog shows the real maximum signal (saturation level) and calculates the offset (in this case: -3256) that needs to be applied

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1.4 Using Advanced features of measurement recipes

In most cases of simple measurements it is not necessary to create recipes manually - it is only required when using advanced features or to change the default behavior.

Range exclusion.

Specific region in the spectral range can be excluded from measurement. This is especially useful in case of a sharp peak, in particular, deuterium lamp D-alpha line. Excluding the sharp D-alpha peak allows to adjust integration time and achieve best measurement accuracy.

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Fig. 8 Measurement recipe with 653-660nm region of the spectrum excluded from measurement

Spectrum balance.

Measurement of a large spectral range with significant variation of intensity using one spectrometer channel may limit accuracy. For example, it is not uncommon to have a significant difference in measured signal intensity between UV and Vis portions of the 200-1000nm range. Many factors are contributing to this imbalance: light sources, reflectivity of the sample, efficiency of the spectrometer, transmittance of the fiber, etc. The result is that one portion of the spectrum is measured with less precision because integration time is adjusted to avoid saturation in the other, higher intensity, portion of the spectrum.

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One can improve the balance by breaking the spectrum into two parts/spectra: for example 200-400 and 400-1000nm. Both spectra are measured by the same spectrometer, sequentially, but software is able to adjust integration time to achieve best precision.

In the measurement recipe, one can simply add two separate spectra (the spectrometer channel # should be the same). The result of the measurement will be continuous spectrum stitched from two parts.

Adjustment range.

To achieve maximum accuracy one needs to use the full range of the ADC. When "variable integration time" is selected, software tries to adjust integration time to fit it in the specified range, typically 85% to 95% of the ADC. Adjusting integration to fit signal in this narrow range does not cause a problem in most cases. However, some spectrometers may only support 1ms granularity of the integration time, if this is the case and signal is strong (nominal integration time <5ms) - software may not be able to adjust integration time. To solve this problem, one can relax the acceptable range e.g. 85-95 % of ADC



Fig. 9 Measurement recipe with signal adjustment range set to default 85%-95% (of ADC)

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2. Measurement Examples.

In the beginning of this document we discussed that data acquisition and modeling are two separate steps of the measurement process. Depending on selected type of the measurement recipe they can be performed either automatically together or separately. For new/unknown or complex samples, it is better to collect the data (Measure Only recipe type) and develop the model and analyze results offline. Once the model is developed, one can use the "Measure & Calculate" type recipe for future measurements

2.1 Measurement of oxide (Thickness measurement)

This is a basic measurement: we selected oxide2000 filmstack, selected thickness as a calculated parameter and use the default: "Measure and Calculate" type recipe. Results of the measurement and fit are presented on fig. 1 (beginning of the document)



Fig. 10 Full screen zoomed results of the Fig. 1a. Shows a good fit between measured and calculated data.

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2.2 Dynamic measurement

In process monitoring applications (inline, in-situ) one needs continuous measurements. A dynamic measurement plugin option enables this functionality and allows selecting different measurement and calculation strategies.

Dynamic measurement plugin supports:

- 1. Simple sequential measurement (defined by number of measurement, interval or continuously (default).
- 2. Integration with a position sensor (measurement results are saved as a function of the position)
- 3. Multi-pass sequential measurements (require position sensor). This options allows to make multiple measurement using results of the previous measurement e.g. after deposition of the new layer. In this case results of the previous measurement (and position information) are used to limit the number of measured parameters to the top layer.
- 4. Control of the external system (Horiba/Uvisel spectroscopic ellipsometer) to perform sequential measurement and analyze the results (additional plugin is required)

In this example we will select 100 sequential measurements to check repeatability. The measurements are done on the same 2000A oxide sample (as above) using the same measurement recipe.

To select Dynamic measurement we use: Actions/Dynamic Measurement from the main menu; select filmstack, measurement recipe and measurement strategy (we use a simple sequential measurement) in the displayed dialog and start the measurement.

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Fig. 11 Dynamic measurement dialog. Select measurement recipe and filmstack from the list, select results directory and file name.

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Fig. 12 Results of dynamic measurement.

Trend chart shows results of every measurement and statistics table shows $\sim 0.4 A$ standard deviation.

Results of the measurement are displayed as a trend chart(s), statistics table shows s.d., min/max and average for each calculated parameter and are continuously updated. Measurement report and/or individual measurement can be saved to file(s).

2.3. Thickness and n, k measurement (nitride example).

Spectral reflectance/transmittance measures optical thickness $-n^*T$, where n- is reflective index and T is physical thickness of the layer. For dielectric materials (non-absorbing i.e. k=0) it is impossible to measure n & T independently (they are strongly correlated). This means that one need to know optical dispersion of the material in order to determine the thickness. If material is absorbing (k>0) it is possible to decouple n & T and independently measure T, n, k by representing optical dispersion using parameterized approximation: Tauc-Lorentz or other oscillator approximation.

Following example shows results of 700A nitride measurement in 200-1000nm range. The data was analyzed using two different approaches:

- 1. Use standard Si3N4 material data and calculate only thickness.(only T case). Measured thickness: 725A
- 2. Represent nitride dispersion using Tauc-Lorentz approximation and determine T, n, k simultaneously (T,n,k case). Measured thickness: 738A

Only T case, gives good fit in the visible range (Fig. 13) where nitride is dielectric (k=0) and T,n cannot be determine independently anyway. But UV part shows discrepancy that indicate that used optical properties are not accurate (at least in UV range)



Fig. 13 Fit of measured and calculated data for 700A nitride. Using standard nitride optical constants (only T case). UV part show some discrepancy but visible spectrum has a good fit.



Fig. 14 Fit of measured and calculated data for 700A nitride (T,n,k measurement). Nitride optical dispersion is represented using Tauc-Lorentz approximation (see Fig. 15 for nitride dispersion)

Optical Metrology Company Updated: November, 2010 View Print/Save SiN.tos \ Profile **Dispersion Spectrum** 💕 О.К. 2.00 × Cancel 2.90 Save 0.800 😰 Help 2.800 2.70 0.700 Actions Create Material Create .600 Refractive Index. N Coeff...K 2.400 Extinction (2.300 2.20 Material Library 0.300 5/3N4.mat 2.10 2.000 1.90 0.100 Navelength, nn Si3N4.mat(N) - Si3N4.mat(K) - SiN.tos(N) ---- SiN.tos(K)

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Fig. 15 Comparison of the standard nitride optical dispersion and measured dispersion (represented with TL approximation).

2. 4. Thick polymers measurement.

Measurement of the very thick films presents unique challenges. The standard curve fitting is becoming difficult because:

a). The optical phase cycle is < 200nm for most polymers. This means that fitting algorithm has difficulty adjusting outside this range ("trapped in local minimum"). Global grid search can be used to examine wider range of thicknesses but it make calculation very slow. 200nm is a small value compared with the thickness of the polymer (~ 100um).

b). Optical properties of the polymers are not accurately known. For example there maybe small absorption but because of the large thickness of the layer the effect is significant.

The better approach is to use Thick film algorithm that is based on Fourier analysis of the measured data.

Updated: November, 2010 Example 1: Free standing thick polymer film



Fig. 16 Reflectance measurement of the freestanding polymer (~ 80um). Spectrometer range: 700-1100nm, resolution: 0.2nm

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Fig, 17. Results of using thick-film algorithm (FFT) to analyze measured reflectance data for free standing polymer (Fig. 16). Peak indicates thickness of 81.3um

Updated: November, 2010



Example 2. Free standing Polymer with adhesive layer

Fig. 15 Reflectance measurement of the polymer (~ 26um) with a thin adhesive layer (~ 1um)



Fig. 16. Polymer measurement – thickness calculation results. Two peaks indicate that there are two layers: 26.41μm and ~1.26μm (peaks positions: 26.41 and 27.67)

Updated: November, 2010

3. System Installation.

Standard MProbe Thin-film Measurement system includes:

- Main unit (Light source and Spectrometer(s))
- Sample stage for reflectance
- Reflection probe fiber optics assembly
- USB dongle (software license) and software CD, USB cable, power adapter and power cords
- Additional options (focusing lens, transmittance, etc.).
- Computer system configured with all necessary software

The system may arrive packed in several boxes. After unpacking all the components start assembling the system as follows:

3.1 Software installation (skip this step if computer was included with your system)

Supported OS: Windows 2000, Windows XP, Windows Vista, Windows 7 (32 bit). MProbe Vis and MProbe UVVis can also support Linux and Mac computers (please ask for custom installer).

Computer requirements: Minimum RAM 512MB (1 GG recommended), 1 GHz CPU (dual core is recommended), 100MB free HD space, USB2.0 port (NIR spectrometer does not support USB 1.1, other spectrometers can be used with USB 1.0 or 1.1)

a). Install TFCompanion software. There are two versions of the software: Standalone and Hardware-integrated: use Hardware integrated version (it will install the spectrometer driver for MProbe system). Connect spectrometer to USB port. You should see "Found new Hardware dialog" – let Windows find and install the driver automatically (it is already copied in the systems directory).

Note 1. If Windows cannot find/install the spectrometer driver, point it to **/OODriver** directory on CD.

Note 2. Windows installs driver for each USB port separately.

Note 3. Some USB port hubs may not provide adequate power – it is best to connect spectrometer directly to the computer's USB port

b). Connect USB dongle (license key) and start the software. You will be asked to login: select user: Admin_hw and password: psw (you can create other users later on). You should see signal monitor window that shows the signal from spectrometer.

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3.2 Hardware assembly

a). Connect the power adapter cord to Main unit and use the power switch. Both power socket and power switch are located on the back panel.

MProbe UVVis and UVVisNIR use two light sources that can be switched separately (or together) using two switches on the front panel.

Use only the Adapter provided with the unit to avoid damage to the system.

Never look directly in the light source (Illuminator) or in the fiber connected to the light source. This is especially dangerous with UVVis system. Powerful UV light source can cause eye damage – use UV protective glasses.

b). Setting the stage

b1). SS100/300 Reflectance Stage:

Set the sample stage: release the thumbscrew and move the fiber probe holder up the post.

b2). SS100/300 Reflectance stage (face down configuration)

Release the thumb screw and move the table up the post and tighten the screw. Screw-in adapter in the central threaded hole in the center of the table.

b3). T100/300 Transmittance fixture Perform a) and b) above

c). Connect reflectance probe. The probe has two fiber optics legs terminated by SMA connectors: one leg has a 6 fiber bundle (to light source), another has 1 fiber (to spectrometer). The legs are marked "Read"(single fiber leg) and "Illuminator". Connect "Read" fiber spectrometer SMA connector and "Illuminator" led to light source SMA connector.

Insert the reflectance probe (stainless ferrule) in the probe holder and tighten the thumbscrew to keep it in place.

In the software Signal monitor, set number of averages to 1 to make adjustment easier. Set a reference Si wafer on the stage and adjust the height of the probe by moving the probe holder up/down the post. Typically, the distance ~ 10 mm between the probe and the sample gives optimal signal. For visible range, signal integration time

 ~ 10 ms to 30ms, typically, gives full range signal.

c2) SS100/300 Reflectance stage (face down configuration)

Attach common SMA terminated end of the reflectance probe to the adapter underneath the table. Put reference sample on the table face down and check Signal monitor.

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c3). T100/300.

Attach fiber optics patch cables to the adapter underneath the table and in the probe holder above the table. Align adapters so that the face each over (they have collimator lenses).

Balancing the light sources.

Visible light source and UV light source have different intensity. The balance of signal response can also change depending on the type of the sample. Your system is aligned to have optimal signal balance for Si reference sample. You may need to adjust reflectance probe to find optimal position. If you cannot find a reasonable intensity balance, there several options:

1. If your unit has intensity regulator (potentiometer at the back panel of main unit), use a flat screw-driver to adjust the intensity of the Vis light source.

2. Balance on recipe level See section 1.4 Using Advanced features of the measurement recipe

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