

# Performance & Typical Measurements of FR-tools













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## INTRODUCTION

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**FR-tools** are compact turn-key solutions for the characterization of thin & thick, single layer & multilayer, supported & free-standing, transparent & semi-transparent, uniform & non-uniform films. With FRtools the following film properties can be measured:

- Film thickness
- Refractive index (both real and imaginary parts)
- Uniformity
- Roughness
- Color
- o Reflectance
- Transmittance
- Absorbance

The **Film thickness** range that can be measured with FR-tools is  $1nm - 1000\mu m$  depending on the tool configuration and the number of layers. For the measurement of ultra-thin layers, FR-tools operating in the UV/VIS range are employed. The numbers of film thicknesses that can be measured depend on the refractive index contrast and the optical path of the particular layer stack. In certain refractive index contrast cases, the thicknesses of even six layers can be measured simultaneously.

Both parts of **refractive index** can be measured independently of film thickness for both transparent and semi-transparent films. The measurement of the refractive index of a film is performed with the Cauchy and Lorenz models. The minimum thickness for the simultaneous measurement of the thickness and the refractive index is 50nm for FR-tools operating in the UV/VIS range and 100nm for FR-tools operating in the VIS/NIR spectral range.

The **film Uniformity** is measured in terms of film thickness variation over the probing area (circle with typical diameter of  $300-400\mu$ m).

The **Surface Roughness** of the upper film is measured in terms of RMS over the probing area (circle with typical diameter of  $300-400\mu m$ 

The **Colour** of any surface is characterized and the standard parameters L\*, a\*, b\* are calculated.

The standard **Reflectance, Transmittance and Absorbance** parameters of a coating are measured with FR-tools. In the case of Reflectance, both specular and diffuse reflectance can be measured with the use of the appropriate integration sphere. For the transmittance and absorbance measurements the samples are mounted in the FR- Film/Cuvette holder and these parameters are measured within the whole spectral range supported by the particular FR-tool.

In addition to single measurements, all the parameters above are also measured in **real-time** allowing the monitoring of either slow or fast dynamic phenomena.

### **REFLECTANCE MEASUREMENTS**

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### **MEASUREMENT OF THE THICKNESS OF THIN & THICK SUPPORTED FILMS**

With FR-tools, the thickness of thin & thick, single layer & multi-layer, supported & free-standing, transparent & semi-transparent, uniform & non-uniform films is measured in the 1nm to 1000µm range depending on the actual FR-tool configuration. For the measurement of film thickness in the lower part of the thickness range an FR-Basic tool with spectrometer tuned to operate in UV/VIS is employed. *In certain cases, when an ultra-thin layer is deposited on a transparent layer of known thickness, then the thickness of this ultra-thin layer can be measured even if it is <1nm thick.* In the following screen-shots, representative measurements of single films applied on Si wafer (semi-reflective) and glass (transparent) substrates are illustrated for film thickness in the range of 3 nm to 200 µm.



#### Ultra-Thin Films (<10nm)





SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness: 3.0 nm.



 $Al_2O_3$  on Ge wafer.  $Al_2O_3$  film thickness: 5.8 nm.

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Si<sub>3</sub>N<sub>4</sub> on Si wafer. Si<sub>3</sub>N<sub>4</sub> film thickness: 6.5 nm.



Pt on Si wafer. Pt film thickness: 7.3 nm.



Si<sub>3</sub>N<sub>4</sub> on Si wafer. Si<sub>3</sub>N<sub>4</sub> film thickness: 8.0 nm.

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Al<sub>2</sub>O<sub>3</sub> on Si wafer. Al<sub>2</sub>O<sub>3</sub> film thickness: 8.5 nm.



 $Al_2O_3$  on Ge wafer.  $Al_2O_3$  film thickness: 9.0 nm.

### Thin films (10nm- 100nm)

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#### SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness: 12.0 nm.



Si<sub>3</sub>N<sub>4</sub> on Si wafer. Si<sub>3</sub>N<sub>4</sub> film thickness: 14.0 nm.



Si<sub>3</sub>N<sub>4</sub> on Si wafer. Si<sub>3</sub>N<sub>4</sub> film thickness: 19.1 nm.



SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness: 20.1 nm.

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#### ZrO<sub>2</sub> on Si wafer. ZrO<sub>2</sub> film thickness: 23.2 nm.



SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness: 30.6 nm.



PMMA on Si wafer. PMMA film thickness: 40.3 nm.

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SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness: 41.9 nm.



Porous Al<sub>2</sub>O<sub>3</sub> on Si wafer. Porous Al<sub>2</sub>O<sub>3</sub> film thickness: 73.2nm



TiO<sub>2</sub> on microscope glass. TiO<sub>2</sub> film thickness: 84.0 nm

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ITO on Fused Silica substrate. ITO film thickness: 88.8 nm

### Conventional films (100nm-1000nm)

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PMMA -1000 rpm spin coating on microscope glass. PMMA film thickness: 173.7 nm



Si $_3N_4$  on Si wafer. Si $_3N_4$  film thickness: 266.4 nm



AR-P 6200 on Si wafer. AR-P 6200 film thickness: 397.7 nm

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#### GaAs on Si wafer. GaAs film thickness: 545.8nm



SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness: 696.7 nm



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UV5 resist on microscope glass wafer. UV5 film thickness: 825.6 nm



#### UV5 on Si wafer. UV5 film thickness: 826.4 nm



HSQ resist on microscope glass wafer. HSQ film thickness: 875.6 nm

### Thick films (1µm-10µm)

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#### SiO\_2 on Si wafer. SiO\_2 film thickness: 1.01 $\mu m$



Parylene coating on Stainless Steel. Parylene C film thickness: 1.04 µm



Porous Si on Silicon wafer. Porous Si film thickness: 1.05 µm



Porous Si on Silicon wafer. Porous Si film thickness: 2.05 µm

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#### TEOS on Si wafer. TEOS film thickness: 2.46 μm



AZ5214 on microscope glass. AZ5214 film thickness: 2.9 µm





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#### Parylene coating on Stainless Steel. Parylene C thickness: 4.11 $\mu$ m



PMMA on microscope glass substrate. PMMA thickness: 4.83 µm



Porous Si on Silicon wafer. Porous Si film thickness: 5.71 µm

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#### Parylene coating on Stainless Steel. Parylene C thickness: 6.74 $\mu m$



SU-8 on Si wafer. SU-8 film thickness: 9.7 µm



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PMMA on microscope glass substrate. PMMA thickness: 9.94  $\mu m$ 

#### Very Thick films (10µm-100µm)

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Parylene coating on Stainless Steel. Parylene C thickness: 14.19 µm



#### SU-8 on Si wafer. SU-8 film thickness: 14.2 $\mu$ m



SU-8 on microscope glass wafer. SU-8 film thickness: 31.8  $\mu m$ 



#### SU-8 on Si wafer. SU-8 film thickness: 50 µm

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SU-8 on Si wafer. SU-8 film thickness: 86.75  $\mu m$ 

### Ultra Thick films (>100µm)

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Cover slip No.1 from Corning. Thickness: 148.6µm



Fischer Scientific cover slip. Thickness: 210.8µm

### **MEASUREMENT OF THE THICKNESS OF UN-SUPPORTED FILMS**

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In addition to the standard thickness measurements of films coated on substrates, FR-Tools are able to analyze the reflectance spectra of un-supported samples and to measure film thickness and all other optical properties (refractive index, transmittance, reflectance....).



Thickness measurement of a "Soapy" membrane standing on a ring of a holder. Thickness: 2.03µm



Si membrane prepared by Silicon wafer bonding technique and sacrificial silicon dioxide layer. Thickness: 5.09µm.



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Thickness measurement of a standard Stretch Wrap. Thickness: 22.7  $\mu$ m.



Simultaneous thickness measurement of a PET film and its coating ("TEST-A") on top. Thickness of PET measured to be 50.1  $\mu$ m, and coating 23.6  $\mu$ m.

### **MEASUREMENT OF THICKNESS IN MULTI-LAYER FILMS**

In addition, to the case of single films, FR-tools are extensively used for the simultaneous measurement of film thicknesses of stack of films. The maximum number of films that can be characterized simultaneously, depends on the optical properties of the individual films and in particular on the refractive index contrast, i.e. the difference between the refractive index between adjacent films. In the following graphs characteristic cases of the characterization of various multilayer films are illustrated:

• Two layers stack (two dielectric layers)

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- Three layers stack (two dielectric layers, one semiconductor)
- Five layers stack



ZrO<sub>2</sub> (19.2 nm)/Al<sub>2</sub>O<sub>3</sub> (29.7 nm) on Si wafer.



PEDOT (5.1nm)/ITO (104.6nm) on Fused Silica wafer.



GaAs (91.86 nm)/Al<sub>0.804</sub>Ga<sub>0.196</sub>As (191.17 nm) on GaAs wafer.

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One film of  $Si_3N_4$  (99.9 nm) on thermal  $SiO_2$  (1086.7 nm) on Si wafer.



poly-Si (115.1nm)/Si<sub>3</sub>N<sub>4</sub> (148.2nm)/SiO<sub>2</sub> (569.4 nm) on Si wafer.



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PMMA (1292.0nm)/Si<sub>3</sub>N<sub>4</sub> (111.3nm)/SiO<sub>2</sub> (3037.4nm) on Si wafer.

In the case of multilayers of repeating films the option of linked layers can be activated. In this option the film thickness is considered constant for the selected layers and the software finds deviation in the estimated film thickness. In the following screenshot, the case of five layers stack of ITO/polyimide/LC/polyimide/ITO on BK7 glass is illustrated. The fitting process was applied for the three layers (polyimide/LC/polyimide) while the thickness of ITO is fixed at 10 nm.



5 layers stack of ITO/polyimide/LC/polyimide/ITO on BK7 glass.

layers	Thickness
BK7	Infinite
ІТО	10 nm (fixed)
Polyimide	3064.2 nm
LC	691.3 nm
Polyimide	3147.0 nm
ΙΤΟ	10 nm (fixed)
BK7	infinite

### NIR measurements (900-1700nm, wavelength)

For the thickness measurement of conventional and ultra-thick films in the 100nm to 1000 $\mu$ m range, an FR-pRo tool with spectrometer tuned to operate in NIR is employed. In the following screen-shots, representative measurements of single and multi-layer films are illustrated for film thickness in the range of 100nm to 100  $\mu$ m. Also the measurement of thickness of Si wafer is also demonstrated.



#### Conventional films (100nm-1000nm)

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#### Si<sub>3</sub>N<sub>4</sub> on Si wafer. Si<sub>3</sub>N<sub>4</sub> film thickness=115.5 nm



TEOS on Si wafer. TEOS film thickness=626.1 nm

### Thick films (1µm-10µm)

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 $SiO_2$  on Si wafer.  $SiO_2$  film thickness=2.45 $\mu$ m.



 $Al_2O_3$  on Al wafer.  $Al_2O_3$  film thickness=5.85 $\mu$ m.



 $Al_2O_3$  on Al substrate.  $Al_2O_3$  film thickness=7.70 $\mu$ m.

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SU-8 on Si wafer. SU-8 film thickness=9.75µm

### Very Thick films (10um-100um)

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#### SU-8 on Si wafer. SU-8 film thickness=14.3 $\mu$ m



SU-8 on Si wafer. SU-8 film thickness=53.3µm



SU-8 on Si wafer. SU-8 film thickness=84.3µm

#### **Multi-layer films**

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3-layer thickness measurement: Acrylic on top of a layer of Ink, on top of a PET film. Acrylic layer thickness = 0.9μm

Ink layer thickness = 1.36µm

PET layer thickness = 25.3µm

#### Substrate thickness



#### Si wafer. Si thickness=497.4µm

### **MEASUREMENTS OF THE THICKNESS USING FOCUSING MODULE**

#### Thin films (10nm- 100nm)

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SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness=20.97 nm

#### Conventional films (100nm-1000nm)



 $Si_3N_4$  on Si wafer.  $Si_3N_4$  film thickness=115.4 nm





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SiO<sub>2</sub> on Si wafer. SiO<sub>2</sub> film thickness =726.6 nm

#### Thick films (1µm-10µm)



AZ5214 on microscope glass. AZ5214 film thickness: 1.09 µm



SU-8 on Si wafer. SU-8 film thickness =9.9 $\mu$ m

#### Very Thick films (10µm-100µm)

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SU-8 on Si wafer. SU-8 film thickness =50.3µm

#### **Multilayer films**



Si<sub>3</sub>N<sub>4</sub> (102.4nm) film on SiO<sub>2</sub> (1083.1 nm) film on Si substrate

### **MEASUREMENTS OF THE THICKNESS USING FR-uProbe**



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UV5 resist (1520.6nm) film on Si<sub>3</sub>N<sub>4</sub> (202.2nm) film on SiO<sub>2</sub> (3090.7 nm) film on Si substrate





SOI area on a MEMS pressure sensor. Si (5320.1nm) film on SiO<sub>2</sub> (759.2 nm) film on Si substrate





Suspended Silicon area on a MEMS pressure sensor. Si (5329.1nm) film on SiO<sub>2</sub> (759.2 nm) film on Si substrate

### **COLOUR ANALYSIS**

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The colour of any surface can be characterized by FR-tools. The reflectance signal is processed by FR-Monitor software and the standard colour parameters L\*, a\*, b\* are calculated and converted to RGB as well. In the following screenshots, the colour measurements of two surfaces coated with different paints are presented.



A series of colour measurements were performed for a wide range of paints. The L\*, a\*, b\* values are presented in the following table in comparison with the colour analysis of a Tintometer. Any small deviation between the two instruments is own to the different method of measurement.

	Tintometer			FR-tool		
	L*	a*	b*	L*	a*	b*
sample 1	89.65	-13.36	0.99	87.40	-14.66	1.27
sample 2	37.72	-0.83	-3.82	35.22	0.67	-6.56
sample 3	56.6	8.12	-24.86	54.9	9.54	-25.14
sample 4	42.38	48.72	19.9	44.38	51.42	17.43
sample 5	92.97	-2.38	41.03	91.77	-2.29	43.19
	85.64	0.53	74.06	87.53	1.29	75.14
sample 7	80.66	0.64	47.01	80.99	1.72	48.02
sample 8	60.16	45.76	10.24	60.59	50.91	11.03

### **REFRACTIVE INDEX CALCULATIONS**

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With FR-tools, the refractive index of a film is measured independently from the thickness of the film. Both parts (real and imaginary) of the refractive index are calculated for transparent and semi-transparent films by Cauchy, Lorentz and Sellmeier models. In the following figures, the experimental and fitted spectra for SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> films on Si are illustrated. In the first column, the film thickness is measured with the refractive index values from the database and in the others columns the thickness and the refractive index are calculated simultaneously. In all examined cases the calculated Cauchy, Lorentz and Sellmeier parameter values, represent refractive index (both real and imaginary parts) values very close to the database ones.



### **PRECISION, MEASUREMENT STABILITY**

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Thanks to the robust design and high quality of optical components FR-tools provide with measurements with very high precision and measurement stability. In the following plots the precision and measurement stability are illustrated for:

- $\circ$  1 $\mu$ m thick thermal silicon dioxide film on Si wafer
- $\circ$  265nm thick Si<sub>3</sub>N<sub>4</sub> layer deposited via LPCVD on Si wafer.

All measurements were performed during at an exhibition on a standard table without any precautions on environmental light and temperature stability.



300 600 900 1200 1500 1800 2100 2400 2700 3000 3300 3600

Time (sec)

SiO<sub>2</sub> film thickness measurement over a period of 30mins. Mean SiO<sub>2</sub> film thickness value is:

1008.44nm ±0.05nm.

 $Si_3N_4$  film thickness measurement over a period of 60min. Mean  $Si_3N_4$ film thickness value is:

269.70nm ±0.01nm.

269.055 +

Furthermore the long-term stability over a period of 15 sequential days was studied in the case of  $SiO_2$  layer on Si wafer. These measurements were acquired in a standard laboratory environment. In the following figures the related stability results are presented for the  $SiO_2$  layer. Similar results were recorded for the  $Si_3N_4$  case.



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Histogram showing the SiO<sub>2</sub> thickness measurements over a period of 3hours



Mean thickness values for a period of 15 sequential days. Each point is the thickness value from 360 measurements



Standard deviation values for the thickness measurements of the  $SiO_2$  layer

In the following table the results related to the mean thickness and standard deviations are listed.

	SiO <sub>2</sub> on Si wafer
Mean value over 3 hours (typical)	1012.91-1013.00nm
Standard deviation of the mean value over 3 hours (max)	0.08nm
Mean value over 15 days	1012.95nm
Average of standard deviation of mean value over 15 days	0.06nm
2*Standard-Deviation of daily average over 15 days	0.06nm

### ACCURACY

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Film thickness calculation accuracy has been evaluated against independent measurements from spectroscopic ellipsometry and electrical (capacitive) measurements. In both cases the accuracy was better than 1nm. In the following screenshots the measurements from FR-tools and calibrated spectroscopic ellipsometers are compared for  $Si_3N_4$  and PMMA films on Si wafer. In both cases, the difference between the two measurements is less than 0.5nm for nominal thicknesses of 577nm and 857nm respectively.



Sample: Si<sub>3</sub>N<sub>4</sub> deposited by LPCVD on Si



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Sample: PMMA spin coated on Si.

#### Comparison study for thin SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> layers on Si wafer

Sample	Ellipsometry	FR-pOrtable
1	12.0nm	11.8nm
2	10.2nm	10.0nm
3	8.2nm	8.0nm
4	6.3nm	6.5nm
T1	50.0nm	50.1nm
T2	41.7nm	41.9nm
Т3	30.5nm	30.6nm
Т4	20.0nm	20.1nm
T5	15.9nm	16.1nm

### **CHARACTERIZATION OF NON-UNIFORM FILMS**

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In certain cases, depending on the fabrication methodology, the film thickness is not uniform over the probed area. This film thickness non-uniformity, reduces the amplitude of the interference. The measurement of film thickness non-uniformity is illustrated in a wide range of representative cases in the following screenshots.

In the first case, the non-uniformity of a commercial PET foil is investigated. The black curve is the reflectance signal from a suspended thick PET foil. By assuming uniform film and applying the fitting procedure, the PET foil thickness is calculated but the max-min amplitude is constant throughout the whole spectral region. However, there are clearly beats at ~715nm and 850nm respectively. These beats are due to foil thickness non-uniformity.



#### Commercial uncoated PET foil thickness assuming a uniform film.

By applying the non-uniformity option in the FR-Monitor software the standard deviation of the foil thickness distribution is calculated assuming Gaussian function. The results are illustrated in the following figure. The foil thickness was measured to be 50.5microns and the standard deviation of foil thickness uniformity found to be 710nm.



Commercial uncoated PET foil thickness and uniformity distribution.

The non-uniformity of a commercial cover glass is also investigated. By assuming uniform film and applying the fitting procedure the cover glass thickness is calculated but a divergence between the fitting and the experimental curve is observed at higher wavelengths. By applying the non-uniformity option in the FR-Monitor software the standard deviation of the cover glass thickness distribution assuming Gaussian function is calculated. The results are illustrated in the following figure. The cover glass thickness was measured to be 145.8 microns and the standard deviation of foil thickness uniformity found to be 151 nm.

# FRMonit	or - Off-line mo	de (ClProgram Files)Theta Methiss/Data_measurements/Data/17-3-2015_coversilp_reflection/coversilp_1_5ms.es2)	- 9 2
Fie Ed	t View Acqu	Isition Processing Utilities Help	
8	$\times M$	٨ 🗠 🞊 🙉 🕟 👄 🔍 🔍 RS DS S S-D A T R % 🛱 🐣 A <sup>+</sup> A <sup>-</sup>	
Off-line Reflectance	0.3	Filted Spectrum	Title
	800	0 850.0 900.0 950.0 1000.0 Wawlength (nm)	-
Toolbax	Inter Cash Line	meteodia, Antonia I. & Barda Onan Cantral I. Tenenatura Cantral I. Inn	• 0 ×
Layers Cate 1 Misc 2 Glass 3 Misc	layer Stack W gory es v	Menergin 18 Mark Mag Called Themalducceller (199) Menergin 29 Mark Mark Mark Mark Mark Mark Mark Mark	

Commercial cover glass thickness assuming a uniform film.

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Commercial cover glass thickness and uniformity distribution.

In the following screenshots, the characterization in terms of film thickness uniformity of thick  $SiO_2$ and SU-8 films on Si wafers is illustrated. In the case of the thermally grown  $SiO_2$ , the non-uniformity is zero and practically the film thickness is the same over the probed area (circle with diameter of ~ 350µm). On the other hand, in the case of SU-8 the film thickness non-uniformity increases with film thickness due to the spin coating conditions.



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Experimental and Fitted reflectance spectra from a ~3µm SiO<sub>2</sub> film. Zero non-uniformity is calculated.



Experimental and Fitted reflectance spectra from a ~10 µm SU-8 film. Film thickness variation 12nm.



Experimental and Fitted reflectance spectra from the  ${\sim}50\mu m$  SU-8 film. Film thickness variation 131nm.

### **DYNAMIC REAL-TIME & POST-PROCESS MEASUREMENTS**

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FR-Monitor includes, amongst numerous features, the capability of real-time measurement. This feature allows for the monitoring of dynamic phenomena and the storage of the reflectance spectra during a process and subsequent off-line post-processing. Post processing feature is a very useful feature allowing for extraction of important parameters without the pressure of fast dynamic phenomena. Furthermore FR-Monitor provides the feature of producing video files for better demonstration of results.

In the following images, the screenshot of a polymeric film at the beginning of a thermal processing is illustrated followed by screenshots of film thickness changes during the processing. The determination of glass transition temperature of polymeric films through the real time monitoring of thickness and refractive index of the film was conducted.



One film of PMMA (78.5 nm) on thermal SiO<sub>2</sub> (717.0 nm) on Si wafer



Time evolution of PMMA thickness spin coated on thermal  $SiO_2$  (717.0 nm) on Si wafer in 0-350 sec period.

PMMA film Thickness increases versus temperature. T<sub>g</sub> determination.

In the following screenshots, the thickness evolution of a "soapy membrane" holding by a ring is presenting. The "soapy layer" becomes thinner with time and after a certain time the layer breaks. The soapy layer thickness evolution with time is also illustrated in the graph below.



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t=35sec



t=40sec

t=70sec

"Soapy membrane" thickness evolution with time. For the fitting the refractive index was considered to be 1.33.



Soapy layer thickness decreases with the time.

#### MAPPING

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For the fast and automated mapping of film thickness a dedicated FR-tool configuration (FR-Scanner) is employed. In this case, fast and accurate measurement of film thickness and all other parameters on predefined points over large areas is performed through a user-friendly GUI. In the following figures the mapping of thick SU-8 spin coated on 3-inch Si wafer and SiO<sub>2</sub> thermally grown on 8-inch Si wafer are illustrated as shown in the FR-monitor software.



Film thickness mapping of a polycrystalline silicon layer deposited on a 8 inch Si wafer



FR-Scanner scans the samples under test in very high speed by rotating the stage and by moving linearly the optical head on top (polar scanning) and without bending of the reflection probe. The pattern can be generated through FR-Monitor. For example, in the following image a pattern generated for 300mm wafers can be shown, that includes 625points in (R,theta) positions. Each full scan over the 300 mm wafer, lasts approximately 1min and 30sec.



Left: FR-Monitor's pattern generator utility. Right: Graph of the generated pattern showing the sequence of the measurements performed (first measurement is on the center of the wafer).

For the calculation of the statistical parameters and the representation of the point distribution, some points can be excluded according the statistics calculation parameters. These excluded points are considered as "extreme" points, which can alter the statistics in the unwanted way, so the user can exclude them.

In the image bellow, the thickness mapping of the Si wafer coated with MIR703 coating is illustrated taking into account the a) 0.2% and b) 0.1% as acceptance variation from the central value (median), considering that the qualified points have a maximum difference of  $\pm 0.2\%$  and 0.1% accordingly to the median thickness. The black areas illustrate the rejected points.



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Figure 12a: Thickness mapping, statistical results and Gaussian plot of the qualified points of "MIR703" wafer taking into account 0.2% and of the central value.

Figure 12b: Thickness mapping, statistical results and Gaussian plot of the qualified points of "MIR703" wafer taking into account 0.1% and of the central value.

### **TRANSMITTANCE MEASUREMENTS**

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With FR-tools, transmittance measurements of semi-transparent and transparent samples can be performed as well as, of supported and un-supported (suspended) films.

If the absorption in the film and the substrate is very small or even negligible, film thickness may also be determined from transmittance. Such measurements are shown below.

### **MEASUREMENT OF THE THICKNESS OF SUPPORTED FILMS**



TiO<sub>2</sub> on BK7 glass. TiO<sub>2</sub> film thickness: 258.1 nm.



AZ5214 resist on microscope slide. AZ5214 film thickness: 1.1  $\mu$ m.

### PRECISION, MEASUREMENT STABILITY ON THICKNESS MEASUREMENT

Measurement stability has been tested for a 258.1nm TiO<sub>2</sub> thin film on BK7 glass. In the following plots the precision and measurement stability are illustrated. All measurements were performed during at an exhibition on a standard table without any precautions on environmental light and temperature stability.



TiO<sub>2</sub> film thickness measurement over a period of 2hours (x-axis is in seconds).

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### ACCURACY ON TRANSMITTANCE SIGNAL

#### Transmittance measurements of colour filters.

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Transmittance measurements on #01 (Light Bastard Amber) and #04 (Medium Bastard Amber) colour filters made by <u>Rosco Laboratories</u> were performed using a FR-pOrtable, operating in 370-1020nm spectral range. Additional transmittance measurements were taken on both samples using a Perkin Elmer Lambda 40 spectrometer, a high precision table spectrometer. The obtained results were compared with the Spectral Energy Distribution (SED) curves given by the manufacturer.

#### **Results:**

The measurements of transmittance (%) as a function of wavelength (nm) for colour filters #01 and #04 are summarized in the following figures, left and right, respectively. It should be noted that the reference values (Rosco Label) were obtained after digitization of manufacturer's SED curves, in order to acquire data that are more accurate.



### **PRECICION, STABILITY ON TRANSMITTANCE SIGNAL**

#### Stability over an hour

The stability of transmittance measurements (following figure– top) was tested by taking measurements of a glass sample coated (named B10) with AR coating for one hour, with a rate of 1min. The standard deviation (following figure - bottom) of the transmission values acquired for each pixel of the spectrometer for each measurement, express the noise of the system, and was found to be 0.10%. High noise values found only in wavelengths related with the <u>high peaks of Deuterium lamp</u> (e.g. 486nm, 656nm), which can be avoided in case there is no requirement of measurements in the UV region, using an hybrid LED - Halogen light source.



Transmittance values (%) acquired in 1hour and standard deviation obtained on sample B10.

#### Stability of 15 measurements over a second

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Stability measurements were also performed for the condition of 15 measurements per second. The examined sample was again a glass with AR coating by taking as reference a sample of the same glass without the AR coating. The transmittance data and the standard deviation for each case are shown in the following plots.



Transmittance values (%) and standard deviation of 16measurements/sec on sample B10.

### **DETERMNINATION OF SUBSTANCE CONCENTRATION IN SOLUTIONS**

FR-pRo, offers a wide range of accessories to be used for numerous applications. For example, FR-pRo is combined with the FR-Film/Cuvette holder operate as a standard spectrometer for the measurement of the absorption of solutions in standard quartz/glass/plastic cuvettes. In the following graphs such measurements in the VIS/NIR spectral range (recorded by an FR-pRoc VIS/NIR) are illustrated. Methyl orange and Methylene blue aqueous solutions were examined.

**Methyl orange** is a pH indicator and has a pKa of 3.47 in water at 25°C. The absorption spectra as they recorded through FR-Monitor are illustrated in the figure below for four different concentrations. As the hydrogen ion is lost or gained there is a shift in the exact nature of the delocalisation in the molecule, and that causes a shift in the wavelength of light absorbed resulting in a colour change. Dilution in deionized water, where equal amounts of the red and yellow forms are present and so methyl orange looks orange, doesn't affect its initial chromophore. The examined concentration range, below 10<sup>-3</sup>M, is following the Beer-Lambert Law where Absorbance is a linear function of the molar concentration (A= $\epsilon$ bC). Thus, calculation of the molar absorptivity (or molar absorption coefficient,  $\epsilon$ ) of the examined chemical compound is attainable. The measurements are performed at 507nm according to Sigma-Aldrich specifications and the results are in good agreement with the literature.



**Methylene blue** aqueous solutions show absorbance in visible region due to involving of  $\pi$ - $\pi$ \*and n- $\pi$ \* transitions present in molecules. The concentration range is following the Beer-Lambert law where Absorbance is a linear function of the molar concentration (A= $\epsilon$ bC). Thus, calculation of the molar absorptivity (or molar absorption coefficient,  $\epsilon$ ) of the examined chemical compound is attainable. The absorption measurements are performed at 668nm according to bibliography and the results are in very good agreement with the literature.

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**Measurement Set-up** 

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Absorbance calibration curve of Methylene Blue at 668nm at 25°C.

### **SPECIAL FEATURES**

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FR-Monitor includes a long database with >580 materials organized in various categories for easy handling. User is able to further update the materials database by adding new materials. FR-Monitor has been designed to facilitate the user and provides all the necessary functions in order to set-up the film stack for the calculations. Furthermore, layers can be linked to each other in order to decrease the number of fitting parameters. This feature is very useful in case of multilayer stack of repeating layers e.g. 1-D photonic crystals.

FR-Monitor in addition to the measurement of thickness and optical characteristics of films allows for the prediction of the optical properties of any stack of films. This way the user could predict the properties of the stack he design and modify the properties of individual layers in order to meet the desired optical characteristics

Certain functions such as signal scaling, optical dark correction are included as standard.

Optical properties from various samples of the same material can be analyzed in order to obtain more accurate results.

## Appendix: Materials database (More than 580 materials are included)

#### DIELECTRICS

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Al <sub>2</sub> O <sub>3</sub>	$Fe_2O_3$	Mg doped LiTaO₃	SiON	$V_2O_5$
Al₂TiO₅	GeO <sub>2</sub>	MgF <sub>2</sub>	SiO <sub>x</sub>	VO <sub>2</sub>
BaF <sub>2</sub>	GeOx	MgO	SrTiO₃	Y <sub>2</sub> O <sub>3</sub>
BaTiO₃	HfO <sub>2</sub>	MnO₃	Ta₂O₅	ZnO
CaCO₃	HfSiO₄	Nb <sub>2</sub> O <sub>5</sub>	TeO <sub>2</sub>	ZrO <sub>2</sub>
CaF <sub>2</sub>	InSbO	NbN	TEOS	
CeO <sub>2</sub>	InSe	Si <sub>3</sub> N <sub>4</sub>	TiN	
Cr <sub>2</sub> O <sub>3</sub>	LinbO₃	SiO	TiO <sub>2</sub>	
Cu₂O	LiTaO₃ congruent	SiO <sub>2</sub>	TiO₂ b	
CuO	LiTaO₃ stoichiometric	SiO₂ (therm)	TiSi <sub>2</sub>	

#### GLASSES

2.1% fluorine fused silica	BK7	CR-39	Fused Germania	HOYA BAC4
2.6% fluorine fused silica	BSG (Boron silica glass)	D263	Fused Silica	HOYA BACD11
3.7% fluorine fused silica	CORNING 7059	FTO	НОҮА	HOYA BACD14

HOYA BACD15	HOYA E-BAF8	HOYA E-FD8	HOYA FCD1B	HOYA LAC12
HOYA BACD16	HOYA E-C3	HOYA E-FD80	HOYA FCD505	HOYA LAC13
HOYA BACD18	HOYA E-CF6	HOYA E-FDS1	HOYA FCD515	HOYA LAC14
HOYA BACD2	HOYA E-F1	HOYA E-FDS2	HOYA FCD600	HOYA LAC7
HOYA BACD4	HOYA E-F2	HOYA E-FDS3	HOYA FCD705	HOYA LAC8
HOYA BACD5	HOYA E-F3	HOYA E-FEL1	HOYA FD110	HOYA LAC9
HOYA BACED5	HOYA E-F5	HOYA E-FEL2	HOYA FD140	HOYA LACL60
HOYA BAF10	HOYA E-F8	HOYA E-FEL6	HOYA FD225	HOYA LAF2
HOYA BAF11	HOYA E-FD1	HOYA E-FL5	HOYA FD60	HOYA LAF3
HOYA BAFD7	HOYA E-FD10	HOYA E-FL6	HOYA FDS16-W	HOYA LBC3N
HOYA BAFD8	HOYA E-FD13	HOYA E-LAF7	HOYA FDS18	HOYA M-BACD12
HOYA BSC7	HOYA E-FD15	HOYA FC5	HOYA FDS20-W	HOYA M-BACD15
HOYA E-ADF10	HOYA E-FD2	HOYA FCD1	HOYA FDS24	HOYA M-BACD5N
HOYA E-ADF50	HOYA E-FD4	HOYA FCD10	HOYA FDS90	HOYA MC-NBF1
HOYA E-BACD10	HOYA E-FD5	HOYA FCD100	HOYA FF8	HOYA MC-TAF1
HOYA E-BACED20	HOYA E-FD7	HOYA FCD10A	HOYA LAC10	HOYA M-FCD1
HOYA M-FCD500	HOYA M-TAC80	HOYA NBFD29	HOYA TAFD25	N-BK9

HOYA M-FD80	HOYA M-TAF101	HOYA NBFD3	HOYA TAFD30	N-FK5
HOYA M-FDS2	HOYA M-TAF105	HOYA NBFD30	HOYA TAFD32	N-LAF2
HOYA M-FDS910	HOYA M-TAF31	HOYA NBFD32	HOYA TAFD33	SCHOTT BASF51
HOYA M-LAC130	HOYA M-TAF401	HOYA PCD4	HOYA TAFD35	SCHOTT F2
HOYA M-LAC14	HOYA M-TAFD305	HOYA PCD40	HOYA TAFD37	SCHOTT F4
HOYA M-LAC8	HOYA M-TAFD307	HOYA PCD51	HOYA TAFD40	SCHOTT F5
HOYA M-NBFD10	HOYA M-TAFD405	ΗΟΥΑ ΤΑC2	HOYA TAFD45	SCHOTT K10
HOYA M- NBFD130	HOYA M-TAFD51	ΗΟΥΑ ΤΑC4	HOYA TAFD55	SCHOTT К7
HOYA M-PCD4	HOYA NBF1	ΗΟΥΑ ΤΑϹϬ	HOYA TAFD5F	SCHOTT KZFSN5
HOYA M-PCD51	HOYA NBFD10	ΗΟΥΑ ΤΑC8	HOYA TAFD5G	SCHOTT LAFN7
HOYA M- PCD55AR	HOYA NBFD11	HOYA TAF1	HOYA TAFD65	SCHOTT LAK L12
HOYA MP-FDS1	HOYA NBFD12	HOYA TAF2	LAFN7	SCHOTT LAK N13
HOYA MP-LAC8- 30	HOYA NBFD13	HOYA TAF3	Microscope slide	SCHOTT LASFN9
HOYA MP-LAF81	HOYA NBFD15	HOYA TAF4	N-BAF10	SCHOTT LF5
HOYA M-TAC60	HOYA NBFD25	HOYA TAF5	N-BK10	SCHOTT LLF1
SCHOTT N-BAF10	SCHOTT N-BAF4	Sodalime		
SCHOTT N-BAF3	SCHOTT N-BAF51	ZERODUR		

#### METALS

 $\theta$ metrisis

Ag	Au_Yakubovsky (25nm)	Fe	Ni	Та
AI	Au_Yakubovsky (50nm)	Hg	NiP	Ti
Al_Lehmuskero	Ве	lr	Pd	V
Al_sopra	Со	Li	Pt_palik	W
Au_Johnson	Cr	Мо	Rh	Zn
Au-palik	Cu	Nb	Stainless Steel	

#### MISC

1,2-Dichlorethane	1-Propanol	Air	Cauchy sublayer (2-4nm)	Diamond
1,4-Dioxane	4-Cyano-4'- pentylbiphenyl	AlSi	CCl <sub>4</sub>	DLC (Diamond Like Carbon)
1,5-Pentadiol	5PCH – Liquid Crystal	Anthracene	CH₃NH₃PbBr₃ perovskite	DNA - CTMA
1-Butanol	Acetone	Anthracene b	CH₃NH₃PbI₃ Perovskite	Ethanol
1-Pentanol	Acetonitrile	C Amorphous	Cyclohexane	Graphene
Inq₃ - Tris(8- hydroxyquinoline)	ITO [75nm]	MoS <sub>2</sub>	ThF₄ (Thorium tetrafluoride)	WO <sub>2.6</sub> N <sub>0.6</sub>
Isoamyl Alcohol	KCl (Potassium Chloride)	MoSe2	TiC (Titanium carbide)	WO <sub>2.8</sub> N <sub>0.5</sub>
Isobutanol	KDP	NCI	Toluene	WO <sub>3</sub>

ITO	LC (Liquid Crystal)	NCD (NanoCrystalline	VC - Vanadium carbide	ZnOAl
ITO [15nm]	Methanol	PCE (Tetrachloroethene)	VN - Vanadium Nitride	
ITO [25nm]	MoO <sub>3</sub>	PZT	Water	
ITO [50nm]	MoOx	RTC	WO <sub>2.5</sub> N <sub>0.9</sub>	

#### POLYMERS

 $\theta$ metrisis

Acrylic [Plexiglass]	NAS-21	PDMS	PHEMA	Poly Acrylate
CD dye	Optorez 1330	PE (Polyethylene)	PHEMA-TPS	PP (Polypropylene)
Cellulose	P₃HT	PEDOT:PSS	Pl (Polyimide)	PS (Polystyrene)
СҮТОР	Parylene C - Thick	PEMA	PLA	PS [2] (Polystyrene)
EPR (Epoxy Resist)	Parylene C - Thin	PEN	PMMA	PTFE [AF1300]
EPR x-linked	Parylene D	PET	PMMA 495	PTFE [AF1601]
Ethylene-vinyl acetate (EVA)	Parylene N	PFHE	PMMA 950	PTFE [AF2400]
Gelatin	PC (Polycarbonate)	PFN	PNIPAM	PVA (Polyvinyl alcohol)
PVA cured	PVP (Polyvinylpyrrolidone)	Styrene	Styrene - acrylonitrile	Sylgard 184 (Silicon)

#### Resists

APEX-E AR-N 7700 no crosslinked	AR-P 8100	AZ 1500 bleached	AZ 6112
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AR 2600DUV	AR-N 7720 crosslinked	AR-PC 500	AZ 1500 unbleached	AZ 6200 bleached
AR-BR5460	AR-N 7720 no crosslinked	AR-PC 503	AZ 1518	AZ 6200 unbleached
AR-N 2220	AR-P 1220	AR-PC 504	AZ 15nXT unbleached	AZ 6210B
AR-N 4240 exp	AR-P 3100	AR-U 4000 exp	AZ 2400 exp	AZ 6270 DX
AR-N 4240 unexp	AR-P 3210	AR-U 4000 unexp	AZ 2400 unexp	AZ 6600 bleached
AR-N 4340 exp	AR-P 3540T	AZ 111 exp	AZ 3027 bleached	AZ 6600 unbleached
AR-N 4340 unexp	AR-P 3740	AZ 111 unexp	AZ 3027 unbleached	AZ 7209
AR-N 4400	AR-P 5300	AZ 125nXT unbleached	AZ 40XT unbleached	AZ 7510
AR-N 4600	AR-P 617	AZ 1350H exp	AZ 4500	AZ 7700 bleached
AR-N 750	AR-P 6200	AZ 1350J exp	AZ 4620	AZ 7700 unbleached
AR-N 7500	AR-P 631-679	AZ 1350J unexp	AZ 520D unbleached	AZ 9200 bleached
AR-N 7520	AR-P 6510	AZ 1450 exp	AZ 5214E bleached	AZ 9200 unbleached
AR-N 7700 crosslinked	AR-P 7400	AZ 1450 unexp	AZ 5214E unbleached	AZ Aquatar
AZ BARLi	HD-8820 cured	Kodak 809 unexp	ma-N 490 exp	mr-T 85
AZ MiR 701 bleached	HD-8820 developed	Kodak 820 exp	ma-N 490 unexp	mr-UVCur06
AZ MiR 701 unbleached	HPR-204 exp	Kodak 820 unexp	ma-P 1275 G unexp	mr-UVCur21
AZ MiR 703 exp	HPR-204 unexp	KTFR Kodak thin film resist exp	ma-P 1275 HV unexp	mr-UVCur21SF

AZ MiR 703 unexp	HPR-206 exp	KTFR Kodak thin film resist unexp	MegaPosit SPR220	mr-UVL 6000 exp
AZ NOVA 2071	HPR-206 unexp	LOR-A	MegaPosit SPR3600	mr-UVL 6000 unexp
AZ P4110	InkOrmo hardbake	LOR-B	Microposit S1800	OFPR-800 Dynachem
CE7000	InkOrmo no hardbake	M82_Medusa	mr-DWL exp	OrmoClad
COP E.B exp	IP3600- E	ma-N 1400 exp	mr-DWL unexp	OrmoClear
COP unexp	IP3600- UE	ma-N 1400 unexp	mr-EBL 6000 exp	OrmoComp hardbake
EL 2015 Ultramac	IP-Dip	ma-N 2400 exp	mr-EBL 6000 unexp	OrmoComp no hardbake
EpoClad exp	IP-G	ma-N 2400 unexp	mr-I 7000	OrmoCore
EpoClad unexp	IP-L	ma-N 405 exp	mr-l 8000	OrmoStamp
EpoCore exp	IP-S	ma-N 405 unexp	mr-l 9000 M	PermiNex 1000
EpoCore unexp	KMPR 1000	ma-N 440 exp	mr-l PMMA	PermiNex 2000
GKRS	Kodak 809 exp	ma-N 440 unexp	mr-NIL 6000 E	PFI88A5- E
PFI88A5- UE	SPR2FX13JL	SPR700	SU-8	UV3DUV
PW1000T	SPR2FX13JM	SPR850	SU-8 3000	UV5
Sipol	SPR3000	SPR900	SU-8 Gersteltec	UV5DUV
SML	SPR3500	SPR950	SYSTEM8	UV6DUV
SPR2FX13	SPR500A	SPR955-CM	UV2HSDUV	ZEP520A

#### SEMICONDUCTORS

AIN	AIAs_sopra	GaAn	InGaAs <sub>0.24</sub> P <sub>0.76</sub>	RsB
Al <sub>0.099</sub> Ga <sub>0.901</sub> As	AlCu	GaAs	InGaAs <sub>0.42</sub> P <sub>0.58</sub>	Si
Al <sub>0.135</sub> Ga <sub>0.685</sub> As	AlGaAs	GaInP	InGaAs <sub>0.55</sub> P <sub>0.45</sub>	Si (100)
Al <sub>0.198</sub> Ga <sub>0.802</sub> As	AlSb	GaP	InGaAs <sub>0.82</sub> P <sub>0.18</sub>	Si (110)
Al <sub>0.419</sub> Ga <sub>0.581</sub> As	As <sub>2</sub> Se <sub>3</sub>	GaSb	InP	Si Amorphous
Al <sub>0.491</sub> Ga <sub>0.909</sub> As	Bi <sub>2</sub> Se <sub>3</sub>	Ge	InSb	Si Poly
Al <sub>0.59</sub> Ga <sub>0.41</sub> As	CdS	Ge Amorphous	IZO amorphous	Si Porous
Al <sub>0.7</sub> Ga <sub>0.3</sub> As	CdSe	HqTe	MgF2	SiC
Al <sub>0.804</sub> Ga <sub>0.196</sub> As	CdTe	InAs	PbS	ZnS
AlAs_palik	CIGS – Cu[In(1x),Ga(x)]Se <sub>2</sub>	InGaAs	PbSe	ZnSe
ZnTe				





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