PROGRESS IN SPECTROSCOPIC ELLIPSOMETRY FOR THE IN-SITU REAL-TIME INVESTIGATION OF ATOMIC LAYER DEPOSITIONS

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OUTLINE

1. Introduction
2. Experimental setup
3. Results
4. Conclusion
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MOTIVATION

ALD benefits for nanoscale film manufacturing
- atomic-level thickness control
- conformality in 3D structures
- uniformity on large substrates

Metrology issues
- film thickness & quality
- ALD growth behavior
- reaction mechanisms

M. Knaut et al.: JVST A 30, 01A151 (2012).


Ch. Wenger et al.: 18th WoDiM (Kinsale Co Cork, 2014). – submitted
ATOMIC LAYER DEPOSITION

half-reaction A

purge or evacuation

half-reaction B

ATOMIC LAYER DEPOSITION

\[ \sim (1 - e^{-k_A t}) \]

adsorbed precursor

\[ \sim e^{-k_B t} \]

eliminated ligands

condensed / deposited film

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Industry-oriented ALD

- class 10 cleanroom
- capability for 300 mm wafers
- rapid thermal annealing (RTA) up to 1000 °C in vacuo
- current processes: Al₂O₃; Ta₂O₅; TaNₓ(O,C); Ru(C); RuO₂
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In-situ, on-site & In-vacuo metrology

- Spectroscopic Ellipsometry
- Quadrupole Mass Spectrometry
- Photoelectron Spectroscopy (X-ray, Ultra Violet)
- Scanning Probe Microscopy (Atomic Force Microscopy, Scanning Tunneling Microscopy)
- Quartz Crystal Microbalancing
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   a) in-situ real-time SE algorithm
   b) ALD growth behavior of $\text{Al}_2\text{O}_3$
   c) ALD growth behavior of $\text{TaN}_x$

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IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

Ψ, Δ spectrum after 100 Al₂O₃ ALD cycles at 350 °C

spectroscopic
- Ψ, Δ = f(wavelength)

in situ
- at the place

real-time
- Ψ, Δ = f(time)
### in-situ real-time Spectroscopic Ellipsometry (irtSE) algorithm

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>Fine correct experiment-specific deviations</td>
</tr>
<tr>
<td>2</td>
<td>Correct inaccuracies due to the fastest possible data acquisition</td>
</tr>
<tr>
<td>3</td>
<td>Acquire ellipsometric spectra in situ and in real-time with the fastest possible sampling rate</td>
</tr>
<tr>
<td>4</td>
<td>Construct an optical model</td>
</tr>
<tr>
<td>5</td>
<td>Extract the ALD characteristic curve for the linear growth region</td>
</tr>
<tr>
<td>6</td>
<td>Evaluate the acquired ellipsometric spectra time slice by time slice</td>
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<tr>
<td>7</td>
<td>Post-process the evaluated time-resolved result</td>
</tr>
</tbody>
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#### Flowchart

1. **Uncalibrated or suspicious hardware**
   - Calibrate the rotating compensator ellipsometer’s basic components on the ex-situ stage

2. **Calibrate additional in-situ elements in the ellipsometer’s light path at the vacuum reactor**

3. **New sample of interest**
   - Acquire ellipsometric spectra in situ and in real-time with the fastest possible sampling rate

4. **Start of ALD processing sequence**
   - Construct an optical model

5. **Fit modeled to experimental data time slice by time slice**
   - MSE ok?
     - Yes
     - No

6. **End of ALD processing sequence**

7. **Extract the ALD characteristic curve for the linear growth region**

8. **Reveal process parameter (inter)dependencies of characteristic ALD growth values**
SE FOR THE IN-SITU REAL-TIME INVESTIGATION OF ALD

additional in-situ elements in the ellipsometer’s light path at the vacuum reactor
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

timewise mean absolute deviation of $\Psi, \Delta$ at 450 nm in dependence on effective acquisition time comparing fast acquisition and high accuracy mode

$\Psi, \Delta$ at 450 nm over time comparing fast acquisition and high accuracy mode
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

timewise mean absolute deviation of optical layer thickness in dependence on effective acquisition time comparing fast acquisition and high accuracy mode, respectively.

![Graph showing mean absolute deviation of optical layer thickness in nm against effective acquisition time per ellipsometric spectrum in s. The graph compares fast acquisition and high accuracy modes.](image-url)
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

MSE vs. effective acquisition time
comparing fast and high accuracy mode

\[
MSE = \sqrt{\frac{1}{2N-M} \sum_{i=1}^{N} \left( \frac{\Psi_{i}^{\text{Mod}} - \Psi_{i}^{\text{Exp}}}{\sigma_{\Psi, i}^{\text{Exp}}} \right)^2 + \left( \frac{\Delta_{i}^{\text{Mod}} - \Delta_{i}^{\text{Exp}}}{\sigma_{\Delta, i}^{\text{Exp}}} \right)^2}
\]

MSE vs. effective acquisition time comparing fast and high accuracy mode

timewise mean $\Psi, \Delta$ error vs. effective acq. time comparing fast and high accuracy mode
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

spectral $\Psi, \Delta$ difference (experiment – model) in fast acquisition mode

spectral $\Psi, \Delta$ difference (experiment – model) in high accuracy mode

![Graph showing spectral $\Psi, \Delta$ difference in fast acquisition mode](image)

![Graph showing spectral $\Psi, \Delta$ difference in high accuracy mode](image)
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

spectral mean absolute deviation of $\Psi, \Delta$ difference (experiment – model) vs. effective acquisition time comparing fast and high accuracy mode

![Graph showing spectral mean absolute deviation of $\Psi, \Delta$ difference vs. effective acquisition time]

- fast acquisition $\Psi$
- fast acquisition $\Delta$
- high accuracy $\Psi$
- high accuracy $\Delta$
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOOMETRY

- Optical layer thickness (nm)
  - Increment by 1 whenever a threshold value is exceeded
- Growth rate (nm/s)
- ALD cycle index
- ALD process time (min)

1st derivative
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

$\text{Al}_2\text{O}_3$ optical layer thickness in the course of one ALD cycle before averaging

$\text{Al}_2\text{O}_3$ optical layer thickness after averaging over 10 cycles
IN-SITU REAL-TIME SPECTROSCOPIC ELLIPSOMETRY

timewise mean absolute deviation of averaged optical layer thickness in dependence on the number of ALD cycles involved into averaging
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ALD GROWTH BEHAVIOR OF Al₂O₃

averaged optical layer thickness in the course of one Al₂O₃ ALD cycle at varied deposition temperatures

V. Sharma: Student Research Project (Technische Universität Dresden, Dresden, 2014). – manuscript
ALD GROWTH BEHAVIOR OF $\text{Al}_2\text{O}_3$

extracted ALD characteristic values in dependence on the actual deposition temperature

![Graph showing growth per cycle (Å), adsorbed precursor, removed ligands, and loss during purge 2 as functions of actual Si surface temperature (°C).]
ALD GROWTH BEHAVIOR OF TaN$_x$

averaged optical layer thickness in the course of one TaN$_x$ ALD cycle at varied deposition temperatures
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CONCLUSION

- in-situ real-time Spectroscopic Ellipsometry with a sampling rate of 0.8 s per spectrum and a thickness MAD below 0.01 nm

- ALD characteristic curve in the homogeneous growth region containing both saturation curves

- detailed insight into the ALD growth behavior of Al₂O₃ and TaNₓ in dependence on deposition temperature
ACKNOWLEDGMENT

✓ assistance at scientific work, especially
Marion Geidel, Martin Knaut, Christoph Hossbach, Christoph Kubasch, Tino Hoffmann, Keith B. Rodenhausen, Stefan Schöche, Daniela Seiffert (geb. Schmidt), Steffen Strehle, Thomas Wagner, Greg Pribil, Eugene Irene, Maria Losurdo, Kurt Hingerl, ThomasHingst, Tillmann F. Walther, Ralf Tanner

✓ financial support

The PhD project of Marcel Junige is funded by the European Social Fund (ESF) and the Free State of Saxony of the Federal Republic of Germany 2011-2014 (contract number: 100077335).