Optical & Structural Properties Measurements of Material(s) in EUV spectral Range

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EXTATIC Welcome Week 2017
CTU, Prague, Czech Republic
22-24th September, 2017
EXTATIC EMJD Detail

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Outline

• PhD Research Project
• PhD Work Breakdown
• Research Activities
  – Selection of material & motivation
  – Literature review (materials)
  – Theoretical modeling/simulations
  – Experimentation and data analysis
• Research Outcomes First Year
• Future Planed Activities
• Acknowlegments
PhD Research Project
Proposed Research Topic

• **Topic:**

  Optical and structural properties measurements of material(s)/compounds in Extreme Ultraviolet Spectral Range

• **Abstract:**

  The aim of the thesis is to characterize the optical properties of element(s)/compounds, not yet well known in EUV spectral band, applying different diagnostic techniques, based on reflection, absorption and polarimetric measurements, taking into account the polarization of radiation.

  Suitable composite structures will be studied in order to emphasize the effect due to the coupling of different materials like stress and interface characteristics.
PhD Breakdown
PhD Breakdown

PHD
Breakdown

Course Work
- University Requirement
- EXTATIC Requirement

Research Work
- Home University
- Host University
Course Work Progress
Course Work Progress

• Enrolled in the Doctorate Program for 3 years at Department of Information Engineering, University of Padova, Italy

As a fulfillment requirement (20 Credits) for the PhD studies in Padova University and EXTATIC I have attended following courses during the first year:

1. Foundation Module (EXTATIC + DEI Program)
2. EUV Optics (EXTATIC + DEI Program)
3. Statistical Methods (DEI Program)
4. Physics and operation of heterostructure-based electronic and optoelectronic devices. (DEI Program)
5. Italian Language module (EXTATIC)
Conferences/ Seminars

• Seminars
  – Functional materials for astronomical instrumentation by Dr Andrea Bianco (INAF – Osservatorio Astronomico di Brera)

• International Conferences
  – EXTATIC WELCOME WEEK (ICTP, Trieste, Italy)
  – Graphene 2017 (March 28-30), Barcelona, Spain
    (Largest European Conference on Graphene & 2D Materials)
Research Activities First Year
Research Activities

• Scientific background and materials selection

• Modeling and simulation of materials optical throughputs in the EUV spectral range (IMD software).

• Implementation and validation of a table top polarimetric facility for the EUV spectral range EUV ellipsometric studies of SiO$_2$/Si.

• EUV ellipsometric studies of graphene/SiO$_2$/Si monolayer and trilayer. Experiments were performed, the analysis is in process.

• Proposals submission at BEAR beam line (ELETTRA synchrotron) and Bessy II beamline, Berlin for optical characterization of materials of interest in the complete EUV spectral range. Proposal has been accepted as a top ranked proposal and the beam time allotted for experiments in October 2017.

• Research article writing is in process based on the above studies.
Literature Review & Material Selection
Material Selection

2D MoS₂, Graphene and SiO₂ has been selected as potential materials for study particularly in EUV spectral band based on the literature review.

Motivation

– To date the optical properties of 2D MoS₂ & Graphene in EUV and soft X-ray region not well known.

– There are some studies related to graphene but only in the framework defects induced by the EUV radiation.

– Knowledge of optical properties of mono/ few layer MoS₂, & Graphene in the EUV region many potential applications can be suggested for different technological domains e.g.

  • Space optical components
  • Lithography masks, pellicles, protective layers etc.
  • Enhanced optical elements for free electron lasers & fourth generation light sources.
Properties of Materials

**Graphene** is the best known among the 2D materials with following

- Unique electrical properties, e.g.
  - high conductivity,
  - zero band gap
  - semi metallic behavior,
  - massless Dirac fermions, ballistic transport \[1, 2\],
- Chemically inert
- Thermal & chemical stability in harsh environments,
- High mechanical strength
- Impermeability to ion diffusion \[3, 4\].
- Impermeable to gases as small as Helium \[5\]
- Oxidation resistance \[6\].

**2D MoS\textsubscript{2}** is most promising materials for flexible, transparent electronic device components. \[7,8\].

- Ultrahigh photoresponsivity \(\sim 6\) times that of graphene,
- Direct bandgap (sensitive to most of the visible light.
- Optical Bandgap is tunable by thickness control \[9\].
- High mechanical stiffness (Young's modulus of \(270 \pm 100\) Gpa)
- Breaking stress of \(22 \pm 4\) GPa. \[10,11\].
- Protective against oxidation and corrosion. \[12\].
Research Activities

Theoretical Modeling
Theoretical Modeling: MoS\textsubscript{2}

- Following is some comparison of the reflectance spectra of MoS\textsubscript{2}/SiO\textsubscript{2}/Si and SiO\textsubscript{2}/Si at wavelength 40nm obtained by IMD simulation tool.

Fig. Reflectance spectra (1) $R_p$ of MoS\textsubscript{2}/SiO\textsubscript{2}/Si and SiO\textsubscript{2}/Si (2) $R_s$ of MoS\textsubscript{2}/SiO\textsubscript{2}/Si and SiO\textsubscript{2}/Si (3) Difference $R_p$ of MoS\textsubscript{2}/SiO\textsubscript{2}/Si and SiO\textsubscript{2}/Si; angle [5, 85°] @40nm.
Theoretical Modeling: MoS$_2$

- Following is some comparison of the reflectance spectra of MoS$_2$ (mono, bi, trilayer)/SiO$_2$/Si and SiO$_2$/Si at angle 70° by IMD simulation tool.

**Fig. Reflectance spectra**

1. $R_p$ of MoS$_2$ (mono, bi, trilayer)/SiO$_2$/Si (mono, bi, trilayer MoS2) and SiO$_2$/Si ($R_p$ of MoS$_2$/SiO$_2$/Si and SiO$_2$/Si; $R_p$ of MoS$_2$/SiO$_2$/Si and SiO$_2$/Si; angle [5, 85°] @40nm.)

2. $R_s$ of MoS$_2$/SiO$_2$/Si and SiO$_2$/Si ($R_s$ of MoS$_2$/70deg)

3. Difference $R_p$ of MoS$_2$/SiO$_2$/Si and SiO$_2$/Si; angle [5, 85°] @40nm.
Theoretical Modeling: Carbon/graphene

- Following is some comparison of the reflectance spectra of C/SiO$_2$/Si at different wavelength 40, 30, 13.5nm obtained by IMD simulation tool.

**Fig. Reflectance spectra (left) $R$ of C/SiO$_2$/Si at fixed wavelength (right) Difference $R_p$ of C/SiO$_2$/Si and SiO$_2$/Si; angle [5, 85°] @40nm.**
Experimental Setup for Reflectometry & Polarimetric Measurement at IFN, Padova
Setup for Reflectometry and Polarimetric studies
Experimental Setup

Real view of the FUV and EUV normal incidence reflectometer at CNR-IFN Padova.
Fig. (a) Design of four reflecting mirrors polarizer (b) all mirrors consisting of 200 nm layer of Au on Si substrate (c) Overall shape of four reflection polarizer attached to the rotation stage. (courtesy of AHMED)
Brief Theoretical Background
Theoretical Background

Light Beam can be characterized in terms of the Stokes parameters that are the components of the Stokes vector $S = (S_0, S_1, S_2, S_3)$. The effect of simple and complex optical elements can be described by the Mueller matrix $M(4 \times 4)$ associated to the system.

$$ S' = M \cdot S $$

Muller matrix of four reflection polarizer is

$$ M_{\text{FRP}} = \begin{pmatrix}
\frac{|r_s|^2 + |r_p|^2}{2} & \frac{|r_s|^2 - |r_p|^2}{2} & 0 & 0 \\
\frac{|r_s|^2 - |r_p|^2}{2} & \frac{|r_s|^2 + |r_p|^2}{2} & 0 & 0 \\
0 & 0 & |r_s|^2 & |r_p|^2 \\
0 & 0 & |r_s|^2 & |r_p|^2
\end{pmatrix} $$

$r_s^2$ and $r_p^2$ are the electric field amplitude of the reflection of gold mirror.

Mathematical representation of rotated polarizer

$$ \begin{pmatrix}
\frac{|r_s|^2 + |r_p|^2}{2} & \frac{|r_s|^2 - |r_p|^2}{2} & \cos 2\theta & 0 \\
\frac{|r_s|^2 - |r_p|^2}{2} & \frac{|r_s|^2 + |r_p|^2}{2} & \cos 2\theta + r_s^4 r_p^4 \sin^2 2\theta & 0 \\
\frac{|r_s|^2 - |r_p|^2}{2} & \frac{|r_s|^2 + |r_p|^2}{2} & \sin 2\theta \cos 2\theta - r_s^4 r_p^4 \cos 2\theta \sin 2\theta & 0 \\
0 & 0 & 0 & r_s^4 r_p^4
\end{pmatrix} $$
Theoretical Background

Then the output intensity of the beam impinging the sample and passing through the analyzer

\[ S' = R(-\theta) \times M(FRP) \times R(\theta) \times M(WR) \times S \]

\[
S' = \frac{1}{4} \left[ \left( \left| r_s \right|^8 + \left| r_p \right|^8 \right) \left( \left| r_s \right|_R^2 + \left| r_p \right|_R^2 \right) + \left( \left| r_s \right|^8 - \left| r_p \right|^8 \right) \left( \left| r_s \right|_R^2 - \left| r_p \right|_R^2 \right) \cos 2\theta \right] S_0 \\
+ \left( \left| r_s \right|^8 + \left| r_p \right|^8 \right) \left( \left| r_s \right|_R^2 - \left| r_p \right|_R^2 \right) + \left( \left| r_s \right|^8 - \left| r_p \right|^8 \right) \left( \left| r_s \right|_R^2 + \left| r_p \right|_R^2 \right) \cos 2\theta \right] S_1 \\
+ 2 \left( \left| r_s \right|^8 - \left| r_p \right|^8 \right) r_s^R r_p^R \cos \varphi \sin 2\theta \right] S_2 \\
+ 2 \left( \left| r_s \right|^8 - \left| r_p \right|^8 \right) r_s^R r_p^R \sin \varphi \sin 2\theta \right] S_3
\]

Output of the light beam at the detector after reflecting from the sample and passing through the analyzer.

**Ellipsometric Parameters:**

\[ \rho = \frac{r_p}{r_p} = \tan(\Psi) e^{i\Delta} \]

\[ \Delta \text{ relative phase change} \]

\[ \Psi \text{ relative amplitude change} \]
Experimental Results
Polarimetric Results: SiO$_2$

Counts (Arbitrary Units)

Analyzer Angle (Degree)

50 Degree @121.6nm
60 Degree @121.6nm
65 Degree @121.6nm
70 Degree @121.6nm
75 Degree @121.6nm
80 Degree @121.6nm
Reflectometry Data: SiO2

Reflectance (Arbitrary units) vs. Incidence angle (Degrees)

Ratio = \frac{R_{p} \text{SiO}_2/\text{Si}}{R_{s} \text{SiO}_2/\text{Si}} = (\sqrt{\frac{R_p}{R_s}})

Ratio SiO2/Si vs. Incidence angle (Degrees)
Polarimetric Results: SiO$_2$

Fig: Polarization measurements: experimental (black) and fitted (red) data against the incidence angle (blue) shows direct signal acquired without the sample and by rotating the polarizer.

Fig. Phase difference retrieved by using a Matlab code based on the experimental data (black) and phase difference by IMD simulations (red).
Experimental Data SiO2

*Fig. Ratio with error bars: experimental ratio (black+), Matlab fitted ratio (green+) and IMD based modeling ratio (blue+) against the incidence angle (50°- 80°).*
## Summary of polarimetric data SiO$_2$

<table>
<thead>
<tr>
<th>Incidence angle</th>
<th>Ratio (Matlab code)</th>
<th>Ratio (exp)</th>
<th>Ratio (IMD) @121.6nm</th>
<th>Phase (Matlab code)</th>
<th>Phase (IMD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>0.46</td>
<td>0.45</td>
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<tr>
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<td>0.41</td>
<td>0.43</td>
<td>0.40</td>
<td>147</td>
<td>157</td>
</tr>
</tbody>
</table>
Polarimetric Data: Graphene

Counts (Arbitrary Units) vs. Analyzer Angle (Degree)

- 50 Degree @121.6nm
- 60 Degree @121.6nm
- 65 Degree @121.6nm
- 70 Degree @121.6nm
- 75 Degree @121.6nm
- 80 Degree @121.6nm
Reflectometry Data Graphene

Reflectance vs. Incidence Angle

Reflectance ($R_s$) and Reflectance ($R_p$) vs. Incidence Angle (Degree)

Ratio $\frac{R_p}{R_s}$ vs. Incidence Angle (Degree)

$\frac{R_p}{R_s} = \sqrt{\frac{R_{down}}{R_{up}}}$
Research outcome_first year

- Successful execution of planned activities
- Validation and successful Implementation of a table top polarimetric facility for EUV ellipsometric studies of SiO$_2$/Si and Graphene/ SiO$_2$/Si.
- Research article writing is in process based on the above studies.
- Experimental proposal for beam time at Elettra synchrotron facility is accepted as a top ranked proposal and beam time allotted for experiments in October 2017.
- Poster Presentation accepted at the PTB's 304. Seminar "VUV and EUV Metrology". October 19, 2017.
- **Publications:**
  - 02 conference publications (SPIE)
  - 01 article is submitted in an international journal
Future Planned Activity

• Continue writing of manuscript(s) based on the data collected first year.
• Experimentation for data collection at Elettra synchrotron facility
• Sample preparation/ acquisition for future possible experiments at synchrotron facilities at Berlin and Trieste.
• Data analysis and possible research publication(s)
• Remaining EXTATIC modules of courses will be taken
• Mobility will be planned
References

Acknowledgment

I would like to express my sincere thanks and gratitude to:

• The Education, Audiovisual and Culture Executive Agency (EACEA) Erasmus Mundus Joint Doctorate Program and EXTATIC management.

• I would like to express my deepest gratitude to Prof. Piergiorgio Nicolosi and Dr. Paola Zuppella for their guidance, and great cooperation.
THANK YOU FOR YOUR ATTENTION