SAM formation of 11–MUA on gold

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Motivation

- Alkanethiols: model system for SAMs

- Acid end group can easily be replaced by other functionalized groups
Content

- **Substrates and substrate preparation**: Au(glas), Au(111)/mica, Au(foil)

- **SAM formation**

- **SAM characterization**: XPS, AFM, XRD, Contact angle, TDS

- **Outlook**
Substrates and substrate preparation
Au(glas)

- Polycrystalline, tends to have a dominant (111) texture

- Roughness: 6-7 nm
Au(111)/mica

- strongly oriented (111) texture
- linear plateau borders which include angles of 60°

LEED pattern at 206 V
Sputtered Au(111)/mica
Recrystallized Au(foil)

After annealing at 700K for 24h in vacuum

Etching in aqua regia (nitric acid and hydrochloric acid)

LEED pattern at 26.2 V
AFM on etched recrystallized Au(foil)
Cleaning procedures (annealed Au(foil))

subsequent 20 min Ar⁺ - sputtering

2h cleaning in piranha solution

untreated gold foil
How long does the surface stay clean?

cleaned gold(foil)
under ambient conditions for 10min
SAM formation
Preparation of the SAM

- Cleaning of the substrate: Piranha solution, Ar\(^+\)-sputtering
- Immersion in 1 - 4 mM ethanolic solution of 11-MUA for at least 24h
- Removing out of solution, rinsing with Ethanol, drying with CO\(_2\) - Spray
SAM characterization
XPS: X-ray photoelectron spectroscopy

11-MUA on gold(foil)

S 2p
162.1 eV

XPS - signal / a.u.

BE / eV
Additional C-peak at 288.9 eV
11-MUA/Au(glas)

No differences in XPS spectra for different substrates
Calculation of the I.M.F.P. using the attenuation of the Au$4f_{7/2}$ signal

\[ \text{Au}_1 = \text{Au}_0 \ e^{-d/\lambda} \]

I.M.F.P. of $e^-$ in undecanethiol: 35Å
1 ML of 11-MUA: 13 Å

Determination of I.M.F.P. of electrons in 11-MUA: 19Å
**XRD of 11-MUA**

- Crystalline
- Crystallite size in the range of 500 Å
- Lattice plane distance: 27 Å
**Contact angle measurements**

- **Contact angle**: The angle formed between the solid/liquid interface and the liquid/vapor interface.

- **Hydrophilic surface**: Water droplet spreads – low contact angle.

- **Hydrophobic surface**: Water droplet rests on the surface without wetting it – high contact angle.
Measurement setup to determine the contact angle
Contact angle measurements - results

- **H$_2$O:**
  - Au(111)/mica: (87 ± 2)$^\circ$
  - MUA on Au(111)/mica: (77 ± 1)$^\circ$

- **CH$_2$J$_2$:**
  - Au(111)/mica: (20 ± 1)$^\circ$
  - MUA on Au(111)/mica: (42 ± 1)$^\circ$
AFM: atomic force microscopy

11-MUA on sputtered Au(111)/mica

AFM: atomic force microscopy

11-MUA on sputtered Au(111)/mica
11-MUA on Au(111)/mica, cleaned only with Ethanol
Heating the sample up to 900K

Detection of the expected masses by QMS:

- $\text{H}_2$, $\text{H}_2\text{O}$, CO, CO$_2$
- COOH, H$_2$S
- 11-MUA ($\text{C}_{11}\text{H}_{22}\text{O}_2\text{S}$)
Conclusion and Outlook
**SAM formation**
- SAM preparation of 11-MUA on Au(glas), recrystallized Au(foil) and Au(111)/mica

**SAM characterization**
- **XPS:** S2p at 162 eV and additional C peak at 288 eV. Determination of IMPF for electrons in 11-MUA
- **Contact angle**
  - H₂O
    - Au(111)/mica: (87 ± 2)°
    - 11-MUA on Au(111)/mica: (77 ± 1)°
  - CH₂I₂
    - Au(111)/mica: (20 ± 1)°
    - 11-MUA on Au(111)/mica: (42 ± 1)°
- **AFM:** no difference in surface structure
- **TDS:** CO, CO₂, H₂, H₂O, H₂S, COOH
Outlook

- STM images by Prof. Widdra in Halle

- Further IR measurements

- Replacing of the acid end group

\[ \text{HO} - \text{CH}_2 - \text{\text{\textcircled{}} - CH}_2 - \text{SCN} \]
Acqunowledgment

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