Desorption dynamics of oxide nanostructures fabricated by local anodic oxidation

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Outline

- A brief introduction to spectromicroscopy
- Local Anodic Oxidation (LAO) of GaAs
Motivation

Why XPS?
- chemical state information
- surface sensitive
- ease of quantification
- (in general) nondestructive

Why spectromicroscopy?
- semicond. nanostructures: self-organized islands (dots)
- surface faceting (wires)
- carbon nanotubes
- catalysis, chemical waves
- surface magnetism (XMCD)

Scanning vs. direct imaging type

Photon optics is demagnifying the beam:

**Scanning Instrument**
- Whole power of XPS in a small spot mode
- Flexibility for adding different detectors
- Rough surfaces can be measured
- Limited use for fast dynamic processes
- Lower lateral resolution than imaging instruments

Electron optics to magnify irradiated area:

**Imaging Instrument**
- High lateral resolution (20 nm)
- Multi-method instrument (XPEEM/PED)
- Excellent for monitoring dynamic processes
- Poorer spectroscopic ability
- Sensitive to rough surfaces
The SPELEEM at Elettra

Spectroscopic photoemission and low energy electron microscope

The SPELEEM at ELETTRA
XPEEM: Spectroscopic Microscopy

- Images from a Field Effect Transistor (FET) at different binding energies.
- Photon energy 131.3 eV.

XPEEM: Core Level Spectroscopy

- Pb/W(110), Pb 5d core level, hv = 80 eV
- Best energy resolution: 250 meV
Imaging of Dispersive Plane

- W\{110\} clean surface
- W 4f core level
- $h\nu = 131$ eV
- Resolution 130 meV
XMCD-PEEM

MnAs on GaAs(001)

Co on pre-patterned Si

0.6 µm

1.0 µm
Lateral resolution in LEEM mode

- Pb/W(110), E = 11 eV
- Lateral resolution: 10 nm

![Image]

Experimental Data

Fit

100%
85%
15%
0%

9.6 nm
3.3 pixels

1 pixel = 2.9 nm
Lateral resolution in XPEEM mode

- Pb/W(110), Pb 5d$_{5/2}$, hv = 80 eV, KE = 58.2 eV
- Lateral resolution: 27 nm
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Motivation

- Lithography for fabrication of state-of-the-art semiconductor nanostructures
- Basic research and quantum device applications

Approaches:
- Traditional lithography
- Proximal probes (STM or AFM)

Local Anodic Oxidation (LAO)

- Water electrolysis
  \[ \text{H}_2\text{O} \rightarrow \text{H}^+ + \text{OH}^- \. \]
- \text{OH}^- \text{ groups migrate towards the sample.}
- Oxide penetration induced by the intense local electric field.

- Versatile tool at relatively low cost
- High lateral resolution but small area
Local Anodic Oxidation (LAO)

After oxidation.

Oxide removal with HF 10%, 30 s.

The penetration depth is 1.0-1.5 times the oxide height.
LAO on GaAs/AlGaAs

Before oxidation:

Surface states

2DEG
GaAs/AlGaAs heterostructure

After oxidation:

Surface states
oxide

2DEG
GaAs/AlGaAs heterostructure
Quantum Point Contact

Setup for Lithography on GaAs

- voltage source
- Hygrometer
- AFM camera
- Plexiglass hood
- AFM
- \( \text{N}_2 + \text{H}_2\text{O} \)
- Thermomicroscope Microscope CP-Resource
- water bottle
GaAs LAO-Oxide: Photon Exposure

AFM before: height 18nm

AFM after: height 13nm

Images taken with secondary electrons
- Photon energy: 125 eV
- Kinetic energy: 4 eV
- Field of view: 10 µm

GaAs LAO-Oxide: Desorption

Before photon exposure

After hours of exposure

hv = 130eV

We observe a linear relation between exposure time and height reduction.

A dependence on other oxidation parameters (bias, writing speed) could not be detected.

The Knotek-Feibelman mechanism

- This Auger decay leads to a final state with two vacancies in the valence band weakening the bond between Ga or As and O.

Spectra From GaAs LAO-Oxide

Time resolved spectroscopy with SPELEEM using Dispersive Plane \((\text{hv} = 130 \text{ eV})\)

- Sample S03B
- Hole \((3,2)\)
- Writing voltage 15 V
- Structure height 3 nm
- Image taken with secondary electrons:
  - Photon energy: 130 eV
  - Kinetic energy: 0.3 eV
  - Field of view: 10 \(\mu\text{m}\)

The Ga 3d signal remains unchanged.
The As-oxides signal disappears with time.
Ga-oxides mainly Ga$_2$O with small contribution of Ga$_2$O$_3$. 
90% of LAO-oxide are Ga-oxides, only 10% As-oxides.

Also Ga-oxides must desorb.

GaAs signal in Ga 3d and As 3d approximately equal.
As 3d DP-Spectra From LAO and Native Oxide

- LAO: GaAs component too strong for substrate emission
- Evidence for the presence of unoxidized GaAs in the LAO-oxide.

XPEEM-Spectra From GaAs LAO-Oxide

- Ga 3d: Peak shift and intensity difference
- As 3d: Background difference

Line Shape Analysis of Ga 3d

- Higher relative contribution of GaAs in native oxide explains observed peak shift

Line Shape Analysis of As 3d

LAO-oxide: after 160 minutes of exposure basically only GaAs component observed.

Why this intensity difference?

- Different chemistry?
  - Ga 3d: Ga$_2$O
  - As 3d: GaAs
- Surface contamination?
- Surface roughness?

Thermal stability

Each annealing step: 10 minutes in N₂ atmosphere

Summary

- Photon assisted partial desorption of the AFM-grown oxide was observed.
- The AFM-oxide is mainly composed of Ga$_2$O, with a small fraction of Ga$_2$O$_3$ and As-oxides.
- The shape of the Ga peak does not change with exposure time (early stage of desorption).
- All As-oxides desorb completely.
- Also Ga-oxides desorb.
- Evidence for the presence of unoxidized GaAs in the LAO-oxide.
- Not even traces of As-oxides detected in scanning Auger microscopy.
- The As-oxides are located only at the surface.