Thin films behaving badly

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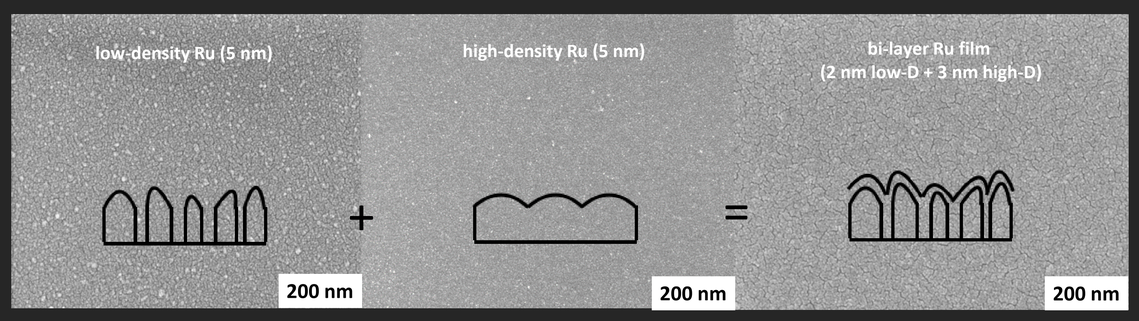
Thin metallic films are of great importance in a wide variety of practical applications. This poster focuses on the growth and evolution of thin Mo and Ru films, sputter-deposited on Si(100) substrates with a native oxide. We use a combination of scanning electron microscopy (SEM) and atomic force microscopy (AFM) for imaging and detailed measurements of the surface topography (roughness) plus x-ray photoelectron spectroscopy (XPS) for elemental and chemical characterization. The structures that we observe after deposition, reflect the polycrystalline nature of even the thinnest films. Upon heating, we find grooving of grain boundaries and, at sufficiently high temperatures, dewetting and other degradation mechanisms.

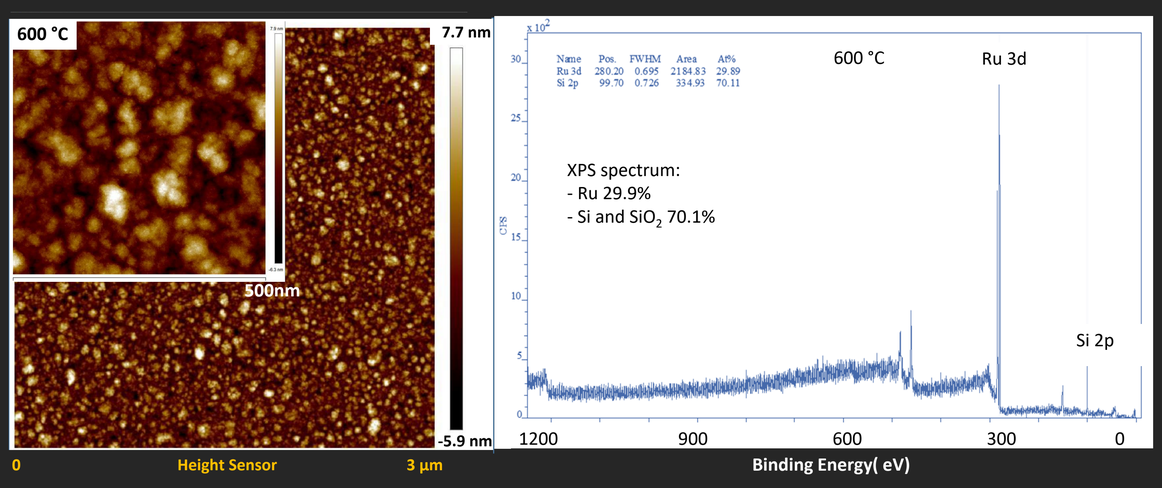
5 nm Ru on Si (with native oxide)



- Porous film
- Dense continuous film
- Combined structure

- DC sputter deposition at different Ar pressures, leading to:
  - low Ru density at 20 mTorr; film not fully closed
  - high Ru density at 200 mTorr; continuous film
- All Ru films are polycrystalline

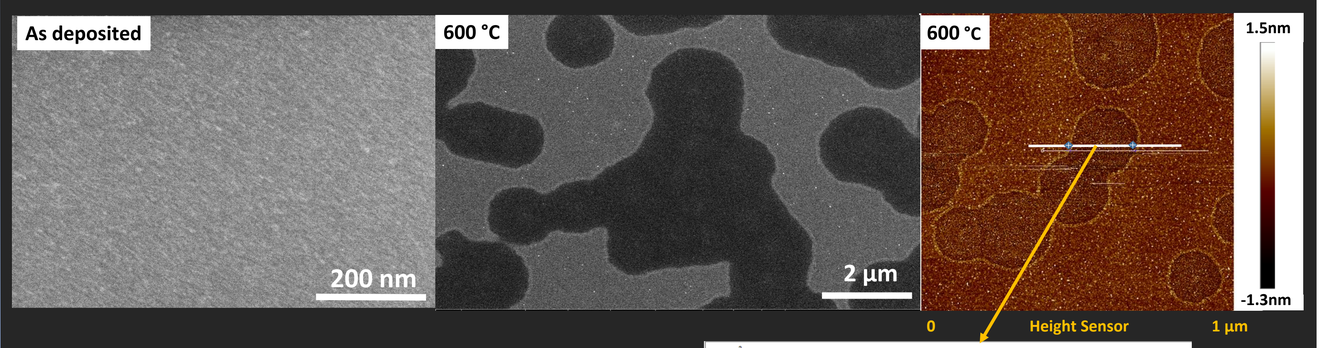
High-density Ru heated at 600 °C



- AFM: roughness increased from 0.34 nm to 2.0 nm (rms)
- Remarkable growth of grain size

- XPS: annealed surface mostly consist of Si:
- *Dewetting*

5 nm Mo on Si (with native oxide)

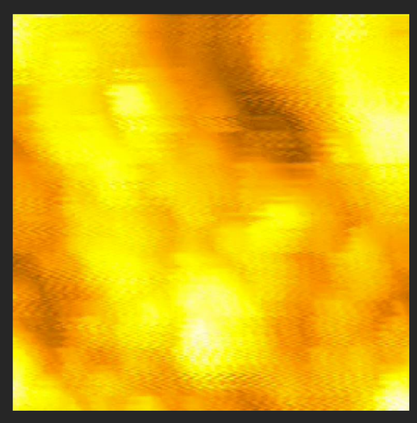


- No remarkable change in morphology up to 600°C
- Circular structures at 600 °C
- XPS: Si (and less O) on surface after annealing
- AFM and XPS suggest silicide formation at 600℃.

AFM + SEM:
- almost no height difference!
- very high SEM-contrast: different material

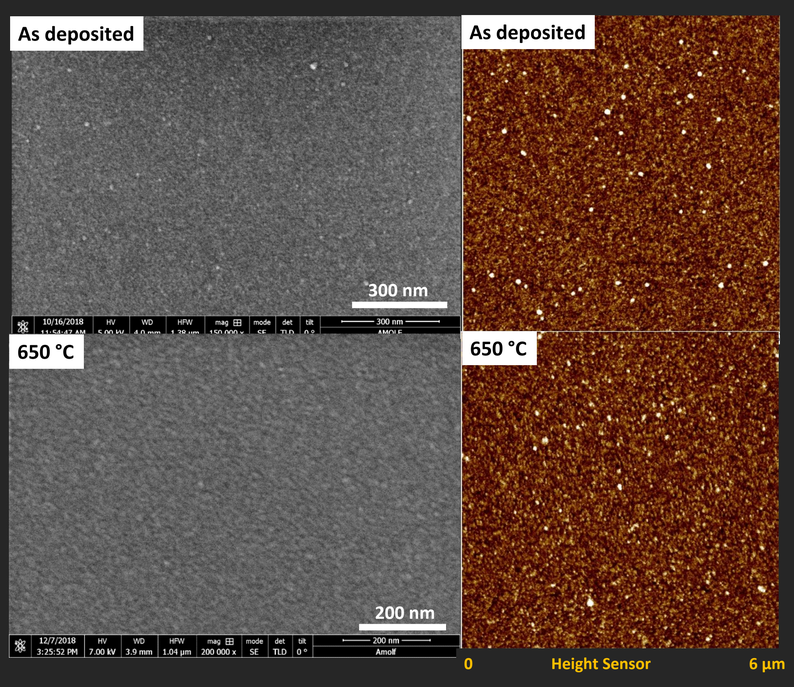
Live STM: Ru deposition on Si (without native oxide)

STM-movie during deposition:
- e-beam evaporation of Ru on Si (without native oxide)
- Rate: 6 nm in 122 minutes (≈ 0.05 nm/min)
- Pressure during deposition below 5 e^{-9} mbar
- Image size = 100 nm x 100 nm



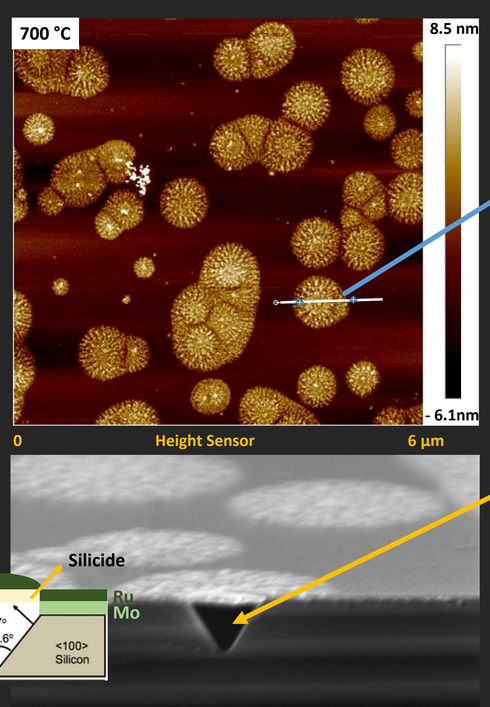
3nm Mo + 3nm Ru bilayer on Si (with native oxide)

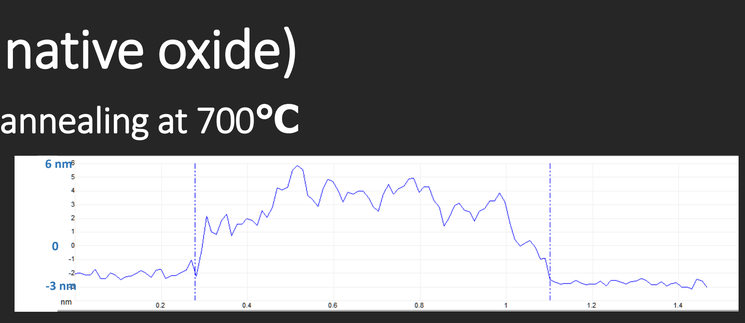
Bilayer thin film: as deposited + heated to 650**°C**

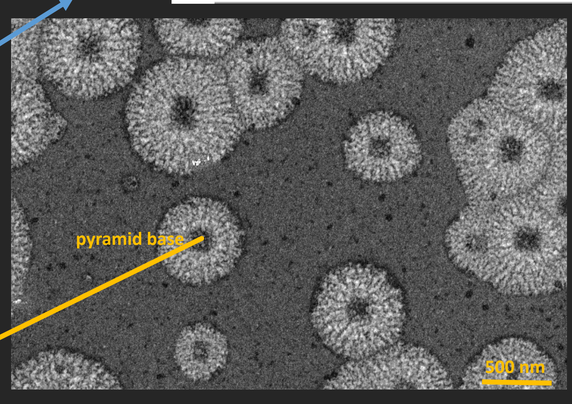


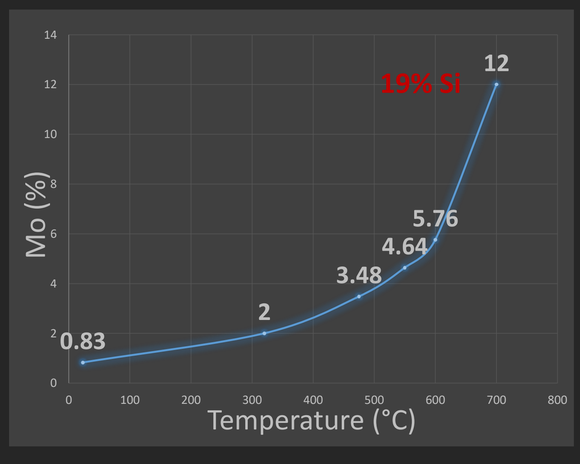
- SEM and AFM show grains: polycrystalline structure
- No remarkable change until 700**°C**
- Surface roughness (rms) : 0.34 nm as deposited
  : 0.41 nm at 650**°C**
- XPS shows the surface consist fully of Ru
- Bilayer showing improved thermal stability. Still closed film!

Bilayer after annealing at 700**°C**









XPS:
- Mo fraction at surface increases with increasing temperature
- Si surface signal starts at 700℃

- Annealing at 700°C results in nano-flowers
- Combination SEM and AFM: square structures in the middle of the nano- flowers reside *below* the surface
- EDX: Ru film still fully closed
- Cross-section SEM: each square structure is the base of an inverted pyramid under a nano-flower
- The facet angles of the pyramids are approximately 54.7°: corresponds to the Si(111) planes, the natural 'etching' planes of Si
- Volumes of flowers and their corresponding pyramids are identical!
- Interpretation: removed Si forms silicide with Mo in the shape of a flower
- Further annealing at 800°C: full surface coverage of nano-flowers

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