$GaPO_4$ Sensors for Gravimetric Monitoring During Atomic Layer Deposition at High Temperatures

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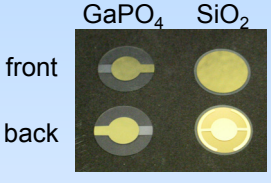
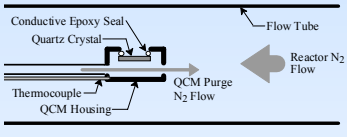
Abstract

The quartz crystal microbalance (QCM) is extremely useful for *in situ* measurements during ALD in a viscous flow environment. Unfortunately, microbalance measurements utilizing typical AT-cut quartz sensors are limited to ALD growth temperatures <~ 300 °C as a result of the extreme sensitivity of the quartz oscillation frequency to temperature fluctuations. Gallium orthophosphate ($GaPO_4$) has much greater frequency stability at higher temperatures than conventional AT-quartz. We demonstrate in this poster that $GaPO_4$ sensor crystals yield precise *in situ* growth measurements during the ALD of Al_2O_3 and TiO_2 films at temperatures as high as 450 °C. $GaPO_4$ sensors show great promise to extend the range of allowable *in situ* ALD measurements to high temperature processes.

Experimental

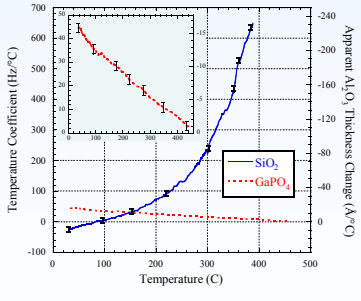


	GaPO4	SiO2
Phase Transition	970 °C	573° C
Coupling Coefficient	16%	9%
Corrosion Resistance	Fair	Excellent



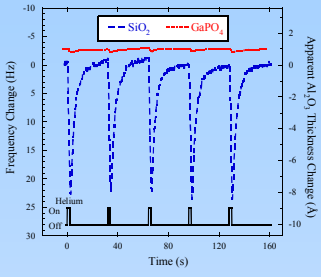
- Sensor is sealed using high temperature, conducting epoxy
- Back surface of sensor purged using N_2
- Maxtek BSH-150 bakeable sensor head (425 °C maximum temperature)
- N_2 purge through water lines, thermocouple near sensor

Sensitivity to Temperature



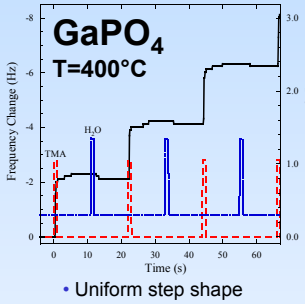
- SiO_2 temperature coefficient increases sharply with temperature
- SiO_2 sensor virtually unusable above ~300°C
- $GaPO_4$ temperature coefficient *decreases* with temperature

Sensitivity to Gas Pulses

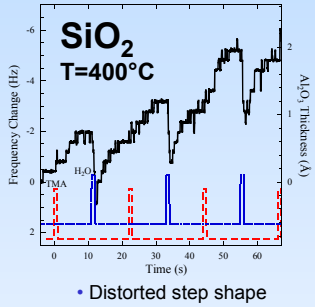


- Helium: large thermal conductivity, no adsorption
- T=400 °C, 2s helium pulses, ΔP=1.8 Torr
- Helium pulses cool sensors by ~0.04°C
- Temperature change perturbs sensor frequency
- Very large perturbation using SiO_2 sensor
- Negligible perturbation using $GaPO_4$ sensor

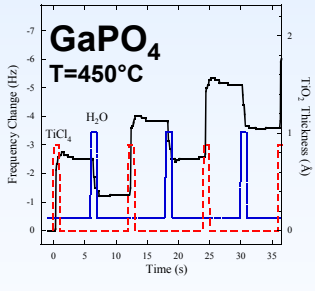
ALD using $GaPO_4$ and SiO_2



- Uniform step shape
- Flat regions during purges
- No transients during dosing



- Distorted step shape
- No flat regions during purges
- Large transients during dosing



- Uniform step shape
- Flat regions during purges
- Maximum temperature limited by sensor head

Future Work

- Construct high-temperature sensor head
- Study high temperature ALD processes (e.g.: SiC ALD, T=800°C)

Reference: *Anal. Chem.* 2005, **77**,3531-3535
GaPO4 Sensors: AVL List GmbH, Part# TXKGAPO00A.A1