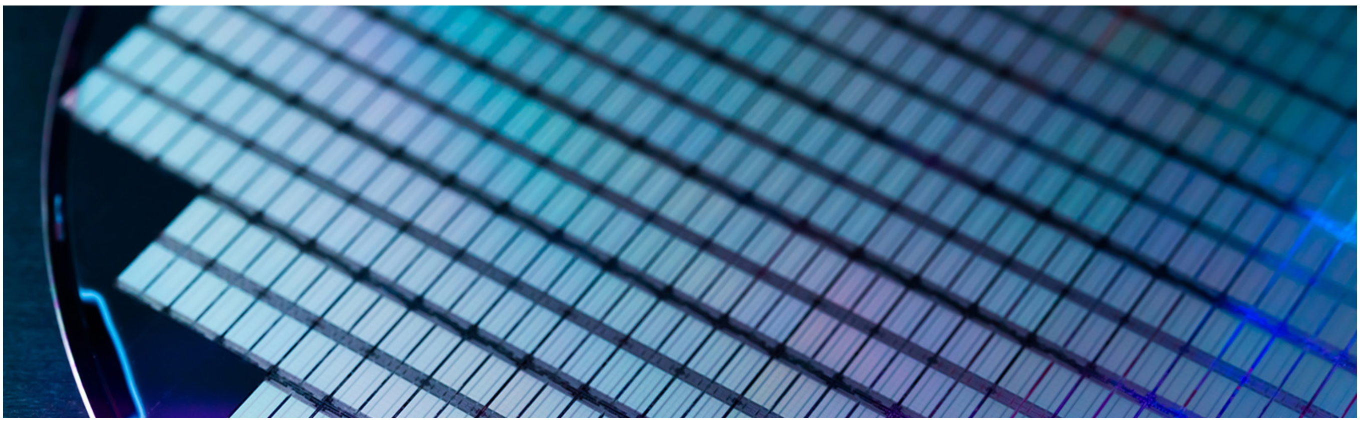
# Overview

# QSense analysis in CMP

## Slurry development, customization and optimization



The CMP (Chemical Mechanical Planarization) method relies on several nanoscale surface interaction processes, and so does a successful CMP result. Irrespective of whether your ambition is to develop and customize high performing slurries, to improve contamination control, or to achieve reliable CMP protocols, it all starts with an understanding, and control of, the surface-molecule interactions involved.

In this overview, we present how QSense® QCM-D (Quartz Crystal Microbalance with Dissipation monitoring) technology can be used in molecule-surface interaction analysis and optimization of CMP related processes. We also demonstrate what information QSense measurements offer.

**The overview covers analysis of:**

- Additive - surface interaction
- Residue removal
- Surface etching

## Characterize and optimize the chemical part of your CMP process

The chemical part of CMP represents a key aspect of the method. It is therefore important that this is optimized for the end use. Both in the development of new slurries or protocols, as well as in the improvement of existing ones, it is important to understand the chemistry and the surface interactions involved. With QSense technology you can study surface processes related to, for example, slurry additives and their interaction with the surface. Questions that could be answered are for example:

- How does this slurry additive interact with the target surface?
- How does the effect of the additive vary with concentration? Which concentration is optimal?
- How does the slurry etch this surface material?
- What is the additive removal rate?
- How efficiently does this post CMP cleaner remove the residue?

**Use QSense QCM-D to understand the chemical part of the CMP process**

- Measure selectivity by varying the surface material of the CMP process
- Measure post CMP cleaning – determine if extra steps are needed to remove residue
- Ability to measure point of use additives and their effect on etching or passivation
- Quantify slurry chemical removal or etch rates
- Prove etch stop behavior with multiple layers, for ex $Si_3N_4$ on $SiO_2$

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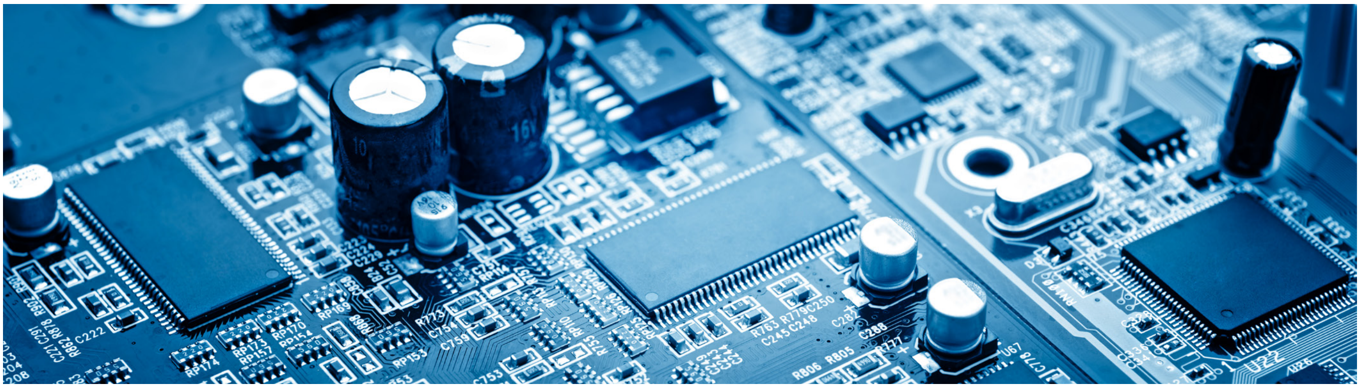


## Analyze material uptake and removal to get the full picture of the surface interactions

QSense is a surface sensitive technology that gives you a new set of eyes in the study of surface interaction processes. It is based on the Quartz Crystal Microbalance with Dissipation monitoring technology (QCM-D) which allows you to study nanoscale mass, thickness and viscoelastic changes of your surface material or coating. The QCM-D method is time-resolved, which means that you can follow the interaction processes in real-time. Interactions and events that can be analyzed are for example adsorption, desorption, molecular binding, and structural changes such as swelling and crosslinking.

### The layer structure provides unique insights

QSense QCM-D senses hydrated mass, i.e. the mass of both the molecules adhering to the surface and the coupled solvent. This makes it an excellent technology to analyze systems where the layer conformation (i.e. structure) and degree of hydration varies with the measurement conditions.

### Study real-life conditions

Surface interaction processes are highly dependent on the conditions where the take place. Therefore, when working with a real-life system, it is important to mimic these conditions in the experimental set up. QSense allows you to vary all the key parameters in your process. Running QSense analysis at relevant conditions, the surface interaction behavior of the additive, additive mixtures or slurries can be characterized and optimized.

Vary for example:

- Surface material/chemistry
- Solution pH
- Temperature
- Chemical additive
- Additive concentration

**QSense QCM-D** is a surface sensitive real-time technology for label free analysis of surface interactions and layer properties at the nanoscale. Monitoring changes in resonance frequency, *f*, and dissipation, *D*, of a quartz crystal, surface interactions and layer properties can be charactreized and quantified in terms of mass, thickness and viscoelastic properties.

### Events that you can analyze:

- Adsorption/desorption
- Layer structure and changes thereof.
- Residue removal
- Etching

### How to interpret the data

**Frequency changes:** The frequency, *f*, provides information about mass changes at the surface. A decrease in *f* indicates a mass uptake and vice versa

**Dissipation changes:** The dissipation, *D*, provides information about the layer softness. As a rule of thumb, the higher the *D*, the softer and/or thicker the layer

| Your question | information provided by QSense QCM-D |
|---|---|
| Will the interaction take place? | Yes/No |
| How fast? | Rate of change |
| How much? | Amounts of mass, thickness and mechanical (viscoelastic) properties (structure) |
| What process am I looking at? | Time-resolved changes in mass, thickness and mechanical properties |
| What is the molecular arrangement? | Thickness and mechanical properties, rigid or soft layer |

**Table 1.** Overview of information related to molecule-surface interactions that QSense QCM-D can extract

## What does QSense analysis of a CMP process look like?

Below we show examples of what typical data could look like in the different cases. We also discuss what information you could extract from the respective measurement and what questions you could typically answer.

### 1. A general example – monitor mass uptake and mass loss

The concept of the analysis method is straightforward. In brief, QCM-D technology monitors mass change as a function of time, i.e. it all comes down to monitoring mass uptake or mass loss from the surface when this is exposed to various samples and chemicals. A typical analysis could for example be that of molecular adsorption and desorption to and from the sensor surface, Fig. 1. The time resolved measurement of the mass changes reveals adsorption and desorption rates as well as the adsorbed amount.

The QCM-D measurement is based on monitoring of the two parameters, $\Delta f$ and $\Delta D$, Fi.g 1, where $\Delta f$ reflects the mass change and $\Delta D$ reflects the viscoelasticity of the layer. Combined, the captured raw a data can be is used to quantify the layer mass, Fig. 1, as well as the layer thickness, and viscoelastic properties (not shown).

As all key conditions can be varied, the amount of information that can be extracted from this seemingly simple analysis is immense.

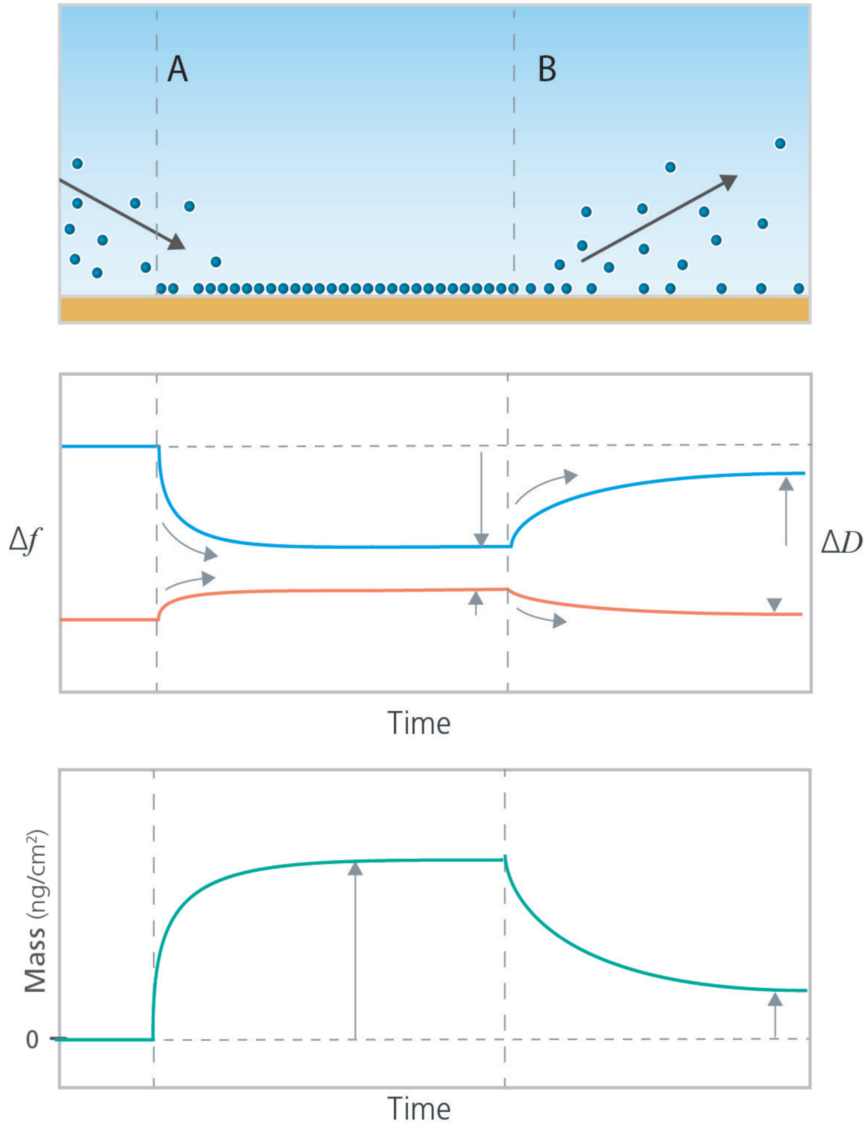



**Figure 1.** Schematic illustration (not to scale) of (top panel) (A) molecule adsorption and (B) desorption, characterized by QSense QCM-D (middle panel). The $\Delta f$ and $\Delta D$ data reflect mass change and layer softness respectively. As indicated by the grey arrows in the schematic graphs, the time-resolved data makes it possible to follow the adsorption and desorption processes, how fast they are, how much material that is added to and lost from the surface. The amount adsorbed to, and desorbed from, the surface can also be analyzed via quantification of the time-resolved layer mass (bottom panel) or layer thickness as well as viscoelastic properties (not shown).

**Use QCM-D for example to:**

- Assess whether a molecule of interest adsorbs to a specific surface material at relevant conditions
- Explore the molecule adsorption dynamics
- Quantify the adsorption rate and total adsorbed amount
- Characterize the molecular arrangement at the surface
- Identify conditions that prevent adsorption
- Optimize the surface or solvent conditions to minimize or maximize adsorption

**Vary meaurement conditions such as**

- Surface material
- Sample concentration
- Temperature
- Solvent
- pH
- Ionic strength

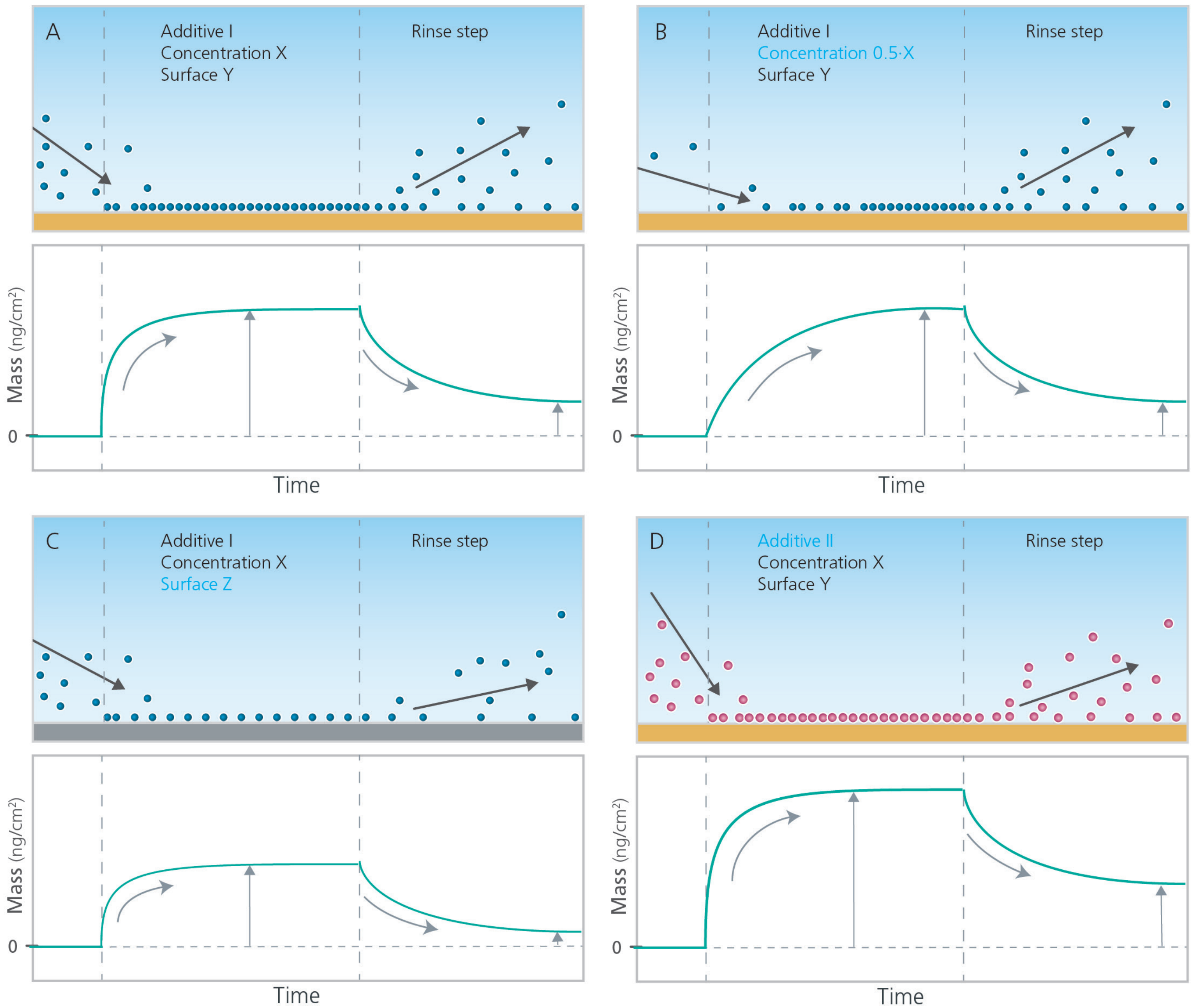



**Figure 2.** Schematic illustration (not to scale) of how the performance and behavior of different additives can be compared. The example shows four different measurements where the parameter settings, in blue text, are varied relative to the measurement conditions in (A). In (B), the additive concentration is reduced, in (C) the surface material is changed, and in (D) a different additive is used. The time-resolved mass curves reveal the additive - surface interaction rate and the additive net mass uptake in the respective measurement.

## 2. Characterize additive - surface interaction

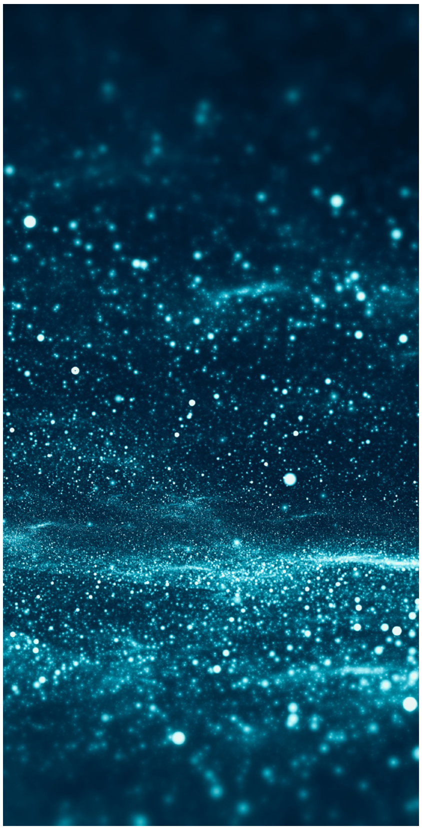
Via the time resolved QSense measurement, it is possible to monitor how additives, additive mixtures, and abrasives interact with the surface of interest. The adsorption and desorption processes can be assessed in terms of how fast they are, and how much material that adsorbs to, or desorbs from, the surface as a function of the chemicals, surface and solvent conditions, Fig. 2. As QSense technology also senses the mechanical properties of the surface-adhering layer, it is possible to analyze the arrangement of the adsorbed molecules via quantification of layer thickness and viscoelastic properties.

**Use QCM-D for example to:**

- Assess how different slurry additives or additive components interact with the target surface
- Compare effects of different additives
- Compare effects of different additive concentrations
- Explore effects of additives at various pH
- Quantify additive adsorption rate to different surface chemistries

### Vary surface material, for example

- Metals: Cu, Co, W, Al, Sn
- Non-metals: Si, $SiO_2$, $Si_3N_4$, SiC, TEOS, polySi, $Al_2O_3$, CuO, $SnO_2$, NiP
- Spin on Organics: PTFE, polyimide
- Custom sensors available upon request



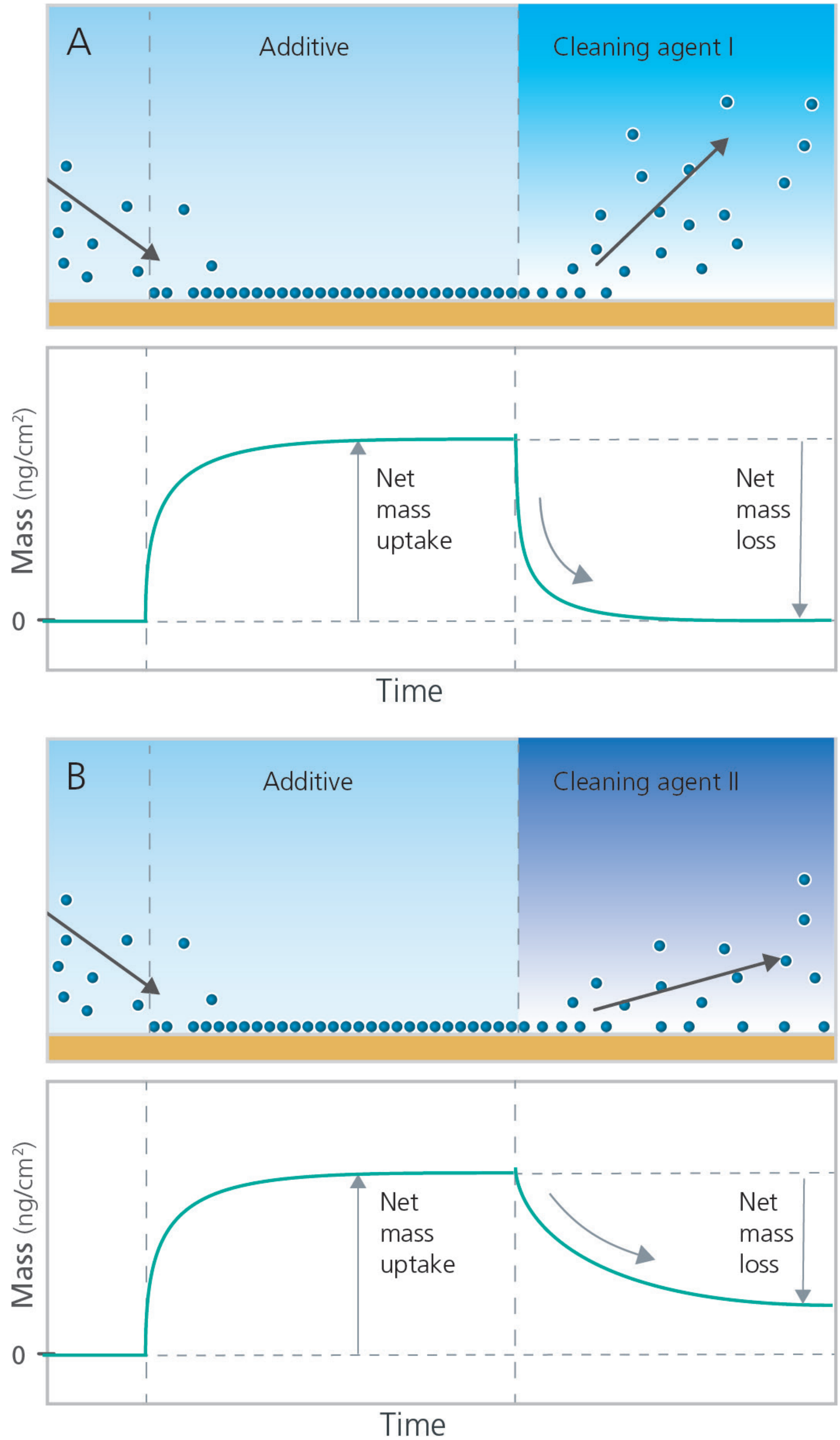


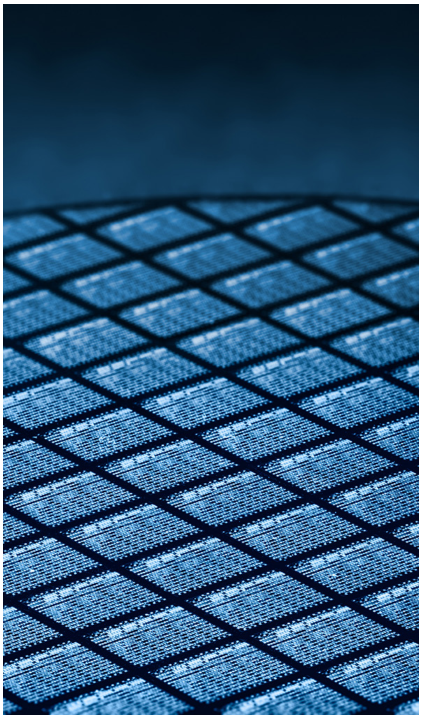

**Figure 3.** Schematic illustration (not to scale) of how post CMP cleaning and residue removal can be analyzed with QSense QCM-D. The surface of interest is exposed to the slurry, or slurry additive, to build up a layer at the surface. Thereafter, the cleaning agent is introduced and the removal rate and removed amount is monitored as a function of time. Via comparison of the removal rates and net mass loss, the efficiency of different cleaning agents can be compared. For example, in the schematic illustration here shown, cleaning agent I would be both faster and more efficient than cleaning agent II

## 3. Analyze post CMP cleaning and residue removal

QSense technology can be used to explore how to best remove post-CMP residue. In this case, the cleaning efficiency and residue removal is analyzed by looking at the mass loss, i.e. the mass removal rate and total amount removed. To run such an analysis, the chemical to be analyzed is first added to build up a layer at the surface, Fig. 2. Next, the surface adhering layer is exposed to the cleaning agent of interest and the mass removal is monitored.

**Use QCM-D for example to:**

- Investigate the removal of residues by post CMP cleaners
- Quantify the removal rates
- Quantify the amount removed from, or remaining at, the surface after the wash step
- Compare the efficiency of different cleaning agents
- Identify the optimal concentration of the cleaning agent



## 4. Assessment of surface etching

In the previous section, we talked about QSense analysis of residue removal via monitoring the mass loss from the surface. Another process that would show up as surface mass loss is that of etching. Exposing the sensor surface to the slurry, or etching solution, of interest, the etching process can be monitored as a function of time, Fig. 4. In the schematic example, the etch behavior of two different slurries, A and B, are compared. Slurry A is etching faster than Slurry B, and the net material loss is higher for slurry A when the experiment ends.

**Use QCM-D for example to:**

- Analyze how the slurry etches different materials
- Investigate how the additive concentration affects the etching behavior
- Explore if the etching process is complex, i.e. if the slurry component first adsorbs and then etches
- Assess if how fast the etching stops when the slurry is removed

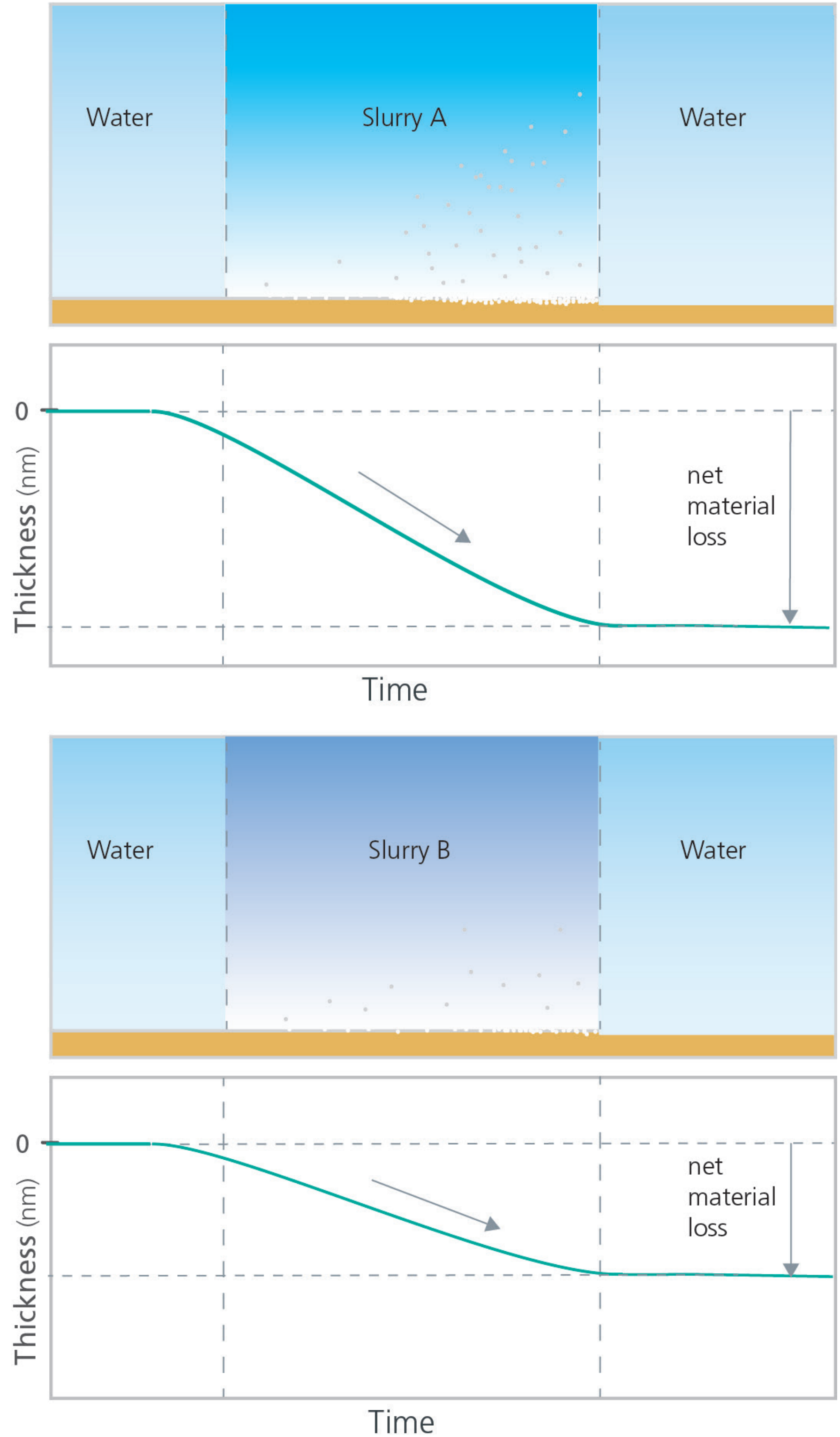



**Figure 4.** Schematic illustration (not to scale) of what QSense etching measurements of two different slurries could look like. The measurements start with bare surfaces in a non-etching solution to establish stable baselines. Thereafter, the slurries, or etching solutions to be studied, are introduced and the sensor material loss is monitored as a function of time. Finally, the measurements end with a rinse step to remove the slurries, and the net material loss can be quantified and compared