Technical Note

# High Precision Sensor Crystal Line

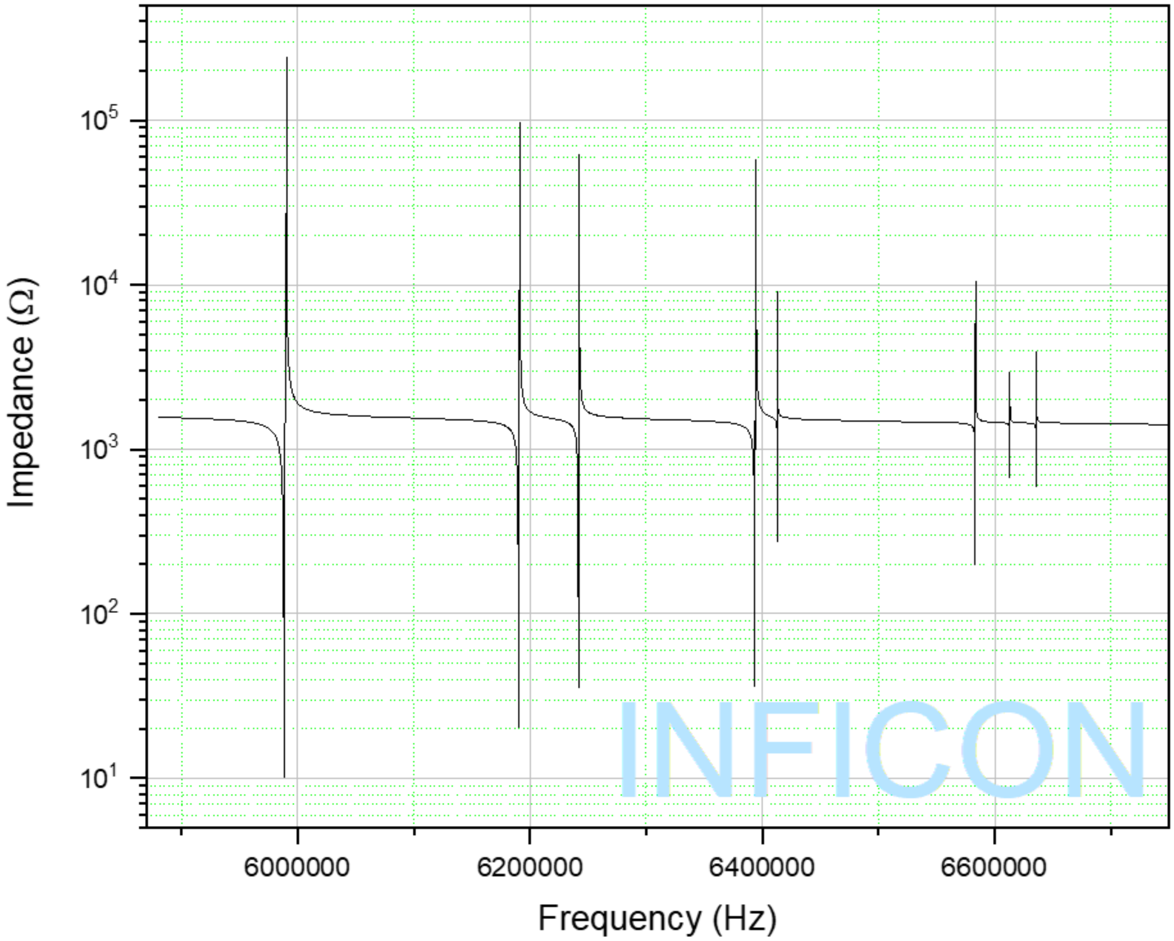
## Background

Quartz crystal microbalance (QCM) is one of the most important INFICON products for organic light-emitting diode (OLED) and optical device fabrication, as well as for many other applications. The change in the QCM's resonance frequency due to mass loading by thin films allows it to be used as a surrogate for correlating the rate of the film thickness accumulation on a substrate.

Many OLED manufacturers currently use standard INFICON gold crystals to monitor and control the deposition of OLED materials. In certain OLED processes, it was seen that INFICON crystals were prone to activity drops (instability in the activity signal) during material deposition. This prompted a design change and the development of new crystals with improved activity stability, leading to less activity dips in terms of the number of occurrences as well as the magnitude of activity dip, and improved deposition rate stability.


## Crystal Activity Dips

The following graph shows the fundamental resonance mode used to monitor material deposition. The resonance peaks to the right show the spurious modes.




These spurious modes are unwanted modes. An active oscillator circuit resonates at the mode with the least impedance, typically the fundamental mode shown in the mode-spectrum. Therefore, the frequency of active oscillators can hop from the fundamental mode to a spurious mode when the impedance of the spurious mode decreases below the fundamental mode impedance. These are called mode-hops and are detrimental to rate monitoring and controlling. For this reason, and for the many other benefits, such as ultra-high rate resolution, lower rate noise, and faster rate updates compared to active oscillators, OLED customers use the INFICON Cygnus®2 with mode-lock technology. Cygnus2 is connected to a QCM via a crystal interface unit (XIU). Using this technology, the crystal is passively driven and its oscillation is locked to the fundamental mode. Although mode-lock prevents the crystal from mode-hopping it cannot prevent the resonance frequency of a spurious mode from overlapping with the resonance frequency of the fundamental mode due to external conditions such as temperature or stress, causing crystal activity dips.

Activity is a function used in Cygnus 2 and IC6 (INFICON thin film deposition controllers) and should not be confused with the crystal activity discussed above. The activity in Cygnus 2 and IC6 is used to monitor the total health of the crystals, including the various electrical connections between the controller and the crystal face. The crystal activity in Cygnus 2 and IC6 does not depend only on the crystal quality, but on many other factors including various cables used to connect crystals to the controllers, XIUs, sensor heads, feedthrough connections, and other sources of electrical connectivity and influence. Prior to looking to the crystal as the cause of activity dips, all electrical connections and components connected to the crystals must be verified as not causing the activity dips or degradation.

When the activity dip happens, the excitation of the unwanted mode results in extra energy dissipation in the resonator, which increases the resistance and makes an unexpected change in the frequency of the resonator. The complete elimination of the activity dip from a crystal is a major challenge and sometimes it is nearly impossible. However, the activity dips can be reduced or controlled significantly by proper design and manufacturing processes of the crystals.

## Crystal Fabrication

To develop the INFICON High Precision Sensor Crystal Line, a detailed investigation was performed into all components of crystal manufacturing and many process parameters including the shape and finish of the crystal, electrode thickness, electrode materials, and the coating rate of the electrode. The materials and process parameters were optimized by fabricating a wide variety of crystals, testing them, and analyzing the results thoroughly. The most important parameter to improve activity stability of the crystals is the final crystal resistance. Using the optimized materials and process parameters, many batches of INFICON High Precision Sensor Crystals were fabricated at INFICON EDC in Kansas, USA.

The crystal manufacturing processes, process parameters, and optimization steps are not discussed in more detail because this information is the intellectual property of INFICON.

## Experimental Methods

After fabricating the crystals, a series of controlled experiments to characterize and evaluate the crystals were conducted. All the characterizations and depositions tests were performed in the two INFICON facilities for verification of consistency. The electrical properties such as resistance and frequency were measured using an impedance analyzer. The quartz blank surface and finished crystals were characterized using various metrology tools including SEM and white light interferometer.

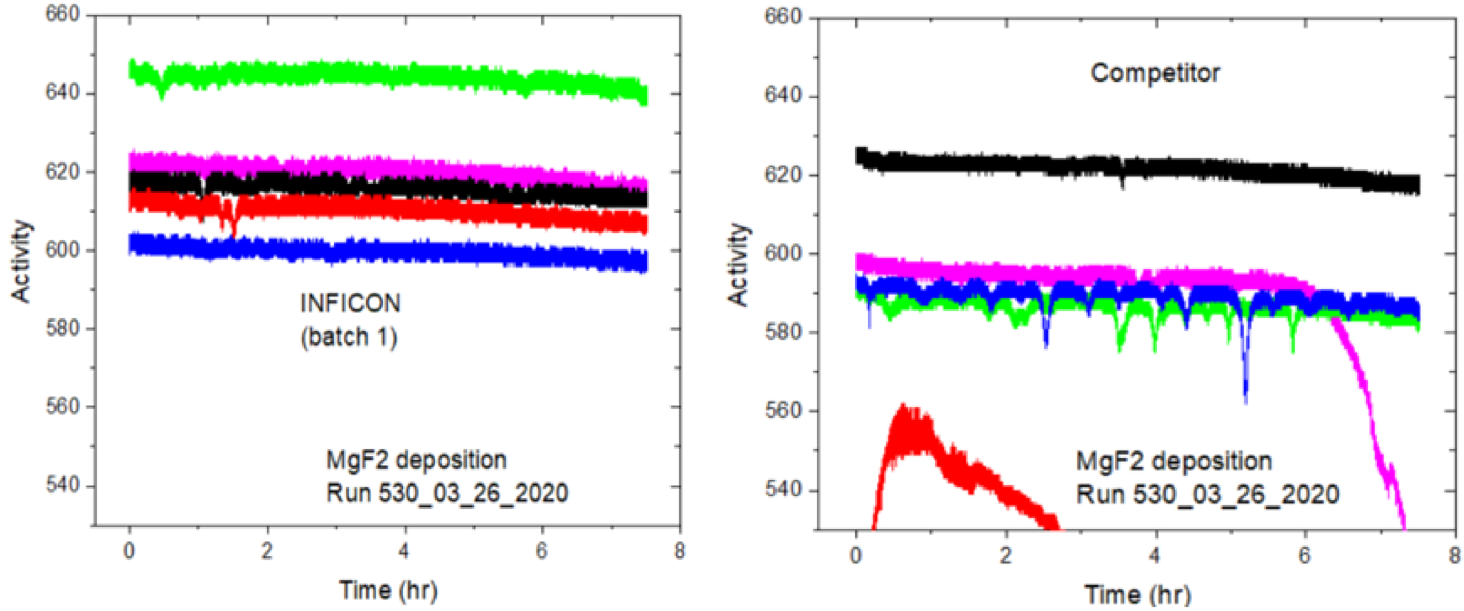
After performing the initial characterization of the crystals, deposition tests were performed to evaluate the activity stability and rate stability of INFICON High Precision Sensor Crystals. An e-beam deposition method to deposit magnesium fluoride ($MgF_2$) and thermal deposition to deposit aluminum (Al) was used. The deposition time, frequency, raw rate, thickness, and activity data were recorded every 100 ms using IC6 Thin Film Deposition Controllers.

## Results and Discussion

The resistance and thermal shock of three batches of INFICON High Precision Sensor Crystals and a competitor's crystals were compared. The summary of these comparisons is shown in the table below. It was found that the resistance of INFICON High Precision Sensor Crystals is lower than the resistance of the competitor's crystals. The thermal shock of INFICON High Precision Sensor Crystals is also significantly lower than that of the competitor's crystals.

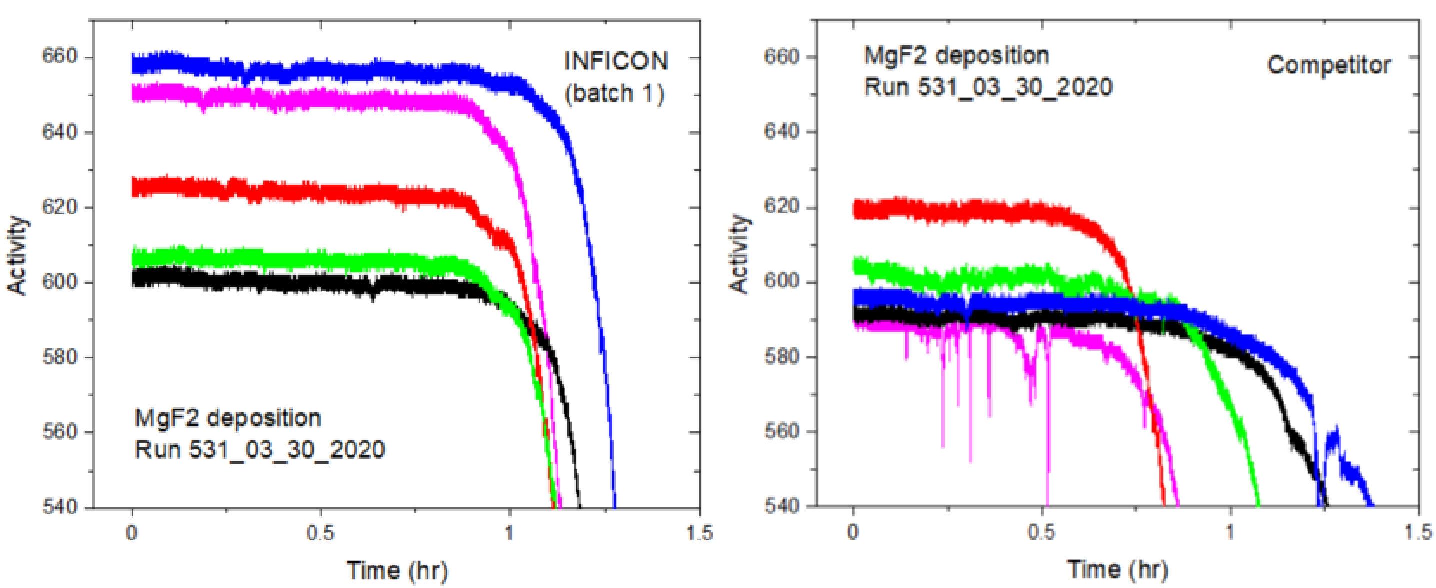
| Crystal | Average Thermal Shock (Hz) | Average Resistance (Ohms) |
|---|---|---|
| High Precision Sensor Crystal (Batch 1) | 72.3 | 7.6 |
| High Precision Sensor Crystal (Batch 2) | 71.0 | 7.8 |
| High Precision Sensor Crystal (Batch 3) | 78.0 | 8.2 |
| Competitor Crystal | 128.8 | 9.0 |

To compare the activity stability and deposition rate stability of INFICON High Precision Sensor Crystals and the competitor's crystals, the deposition test data were analyzed. The figure below shows the plots of the crystal activity versus the deposition time of INFICON High Precision Sensor Crystals (batch 1) and the competitor's crystals. In this test, $MgF_2$ was deposited on INFICON High Precision Crystals and the competitor's crystals in the same deposition run. The PID controlled deposition rate of 0.5 Å/s was used for this test. It is clearly seen that INFICON High Precision Sensor Crystals show excellent activity stability. All INFICON High Precision Sensor Crystals show a lower occurrence and a smaller magnitude of activity dips (both the number of occurrences of activity dips and the change of activity value at the dips are small). However, many of the competitor's crystals (three out of five crystals) show large instability in activity, meaning that the competitor's crystals show a higher number of occurrences of activity dips as well as a larger change in the activity dip signals.

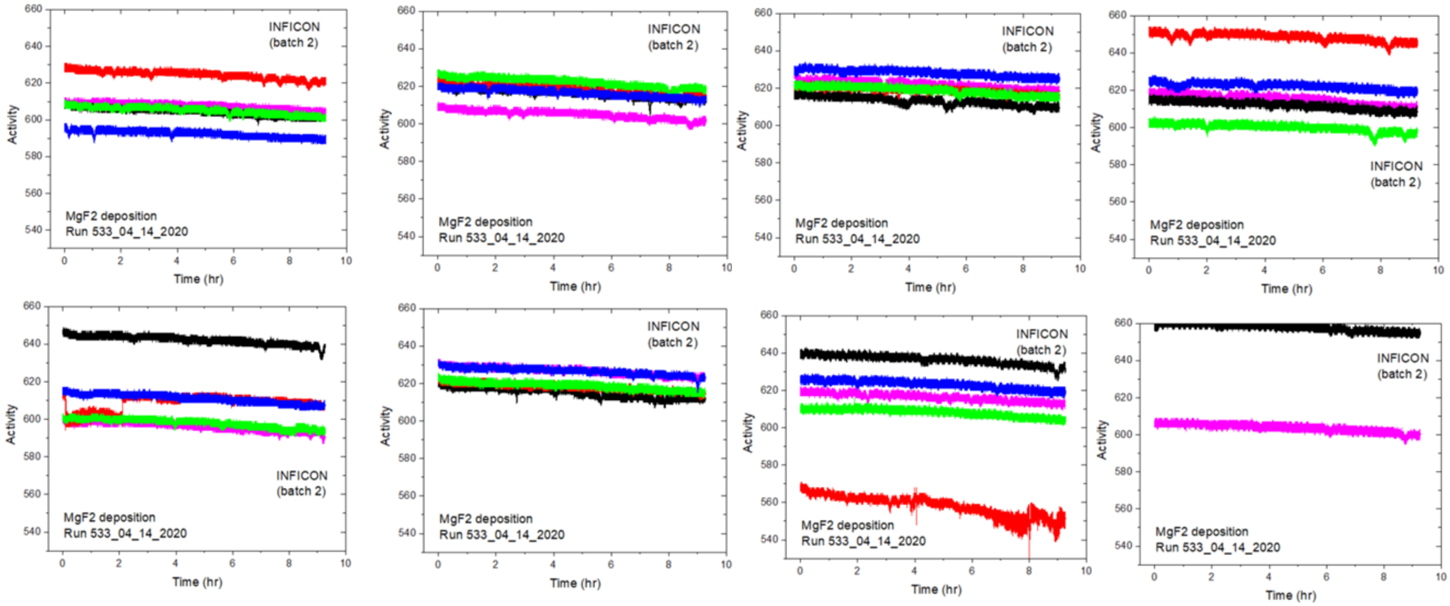



Small activity dips are seen in the activity signals of INFICON High Precision Sensor Crystals. Modified XIUs were used in the tests to reveal small activity dips. When using standard XIUs, these small activity dips may not be visible.

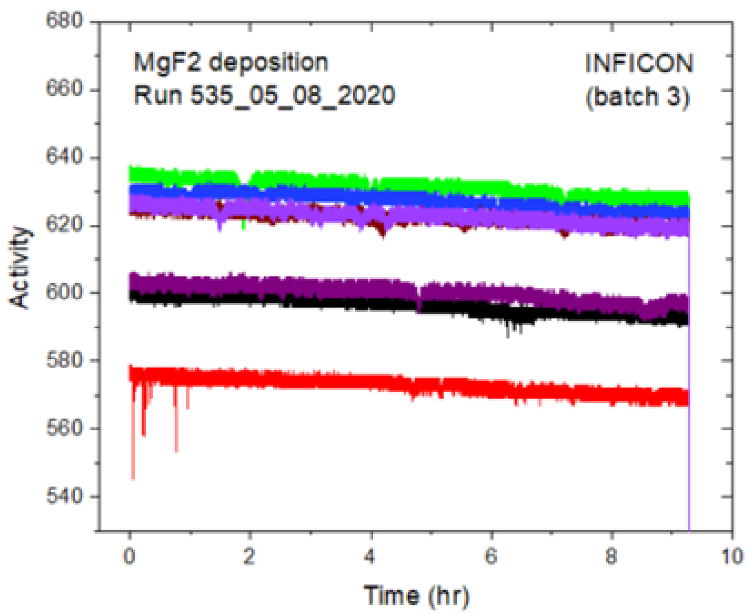
To verify that the quality, mainly the activity stability, of INFICON High Precision Sensor Crystals does not depend on the deposition rate, a deposition test was performed at the higher rate of 8.5 Å/s. The plots of the crystal activity versus the deposition time of INFICON High Precision Sensor Crystals (batch 1) and the competitor's crystals for the PID-controlled deposition rate of 8.5 Å/s are shown in the figure below. This test confirmed that all INFICON High Precision Sensor Crystals tested have better activity stability compared to the competitor's crystals. Moreover, the crystal-to-crystal variation in the activity-roll-off-thickness values for INFICON High Precision Sensor Crystals is lower than that for the competitor's crystals.

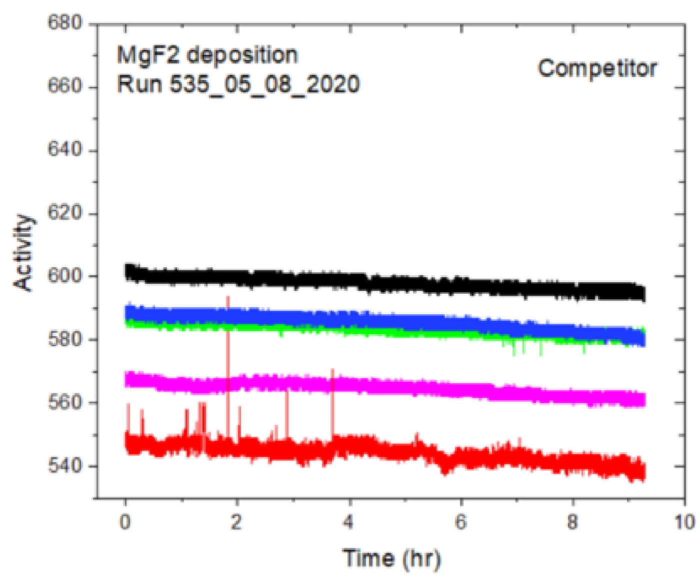


To confirm the reliability and consistency of INFICON High Precision Sensor Crystals, randomly picked crystals from a single manufacturing batch (batch 2) were deposited with $MgF_2$ at a controlled deposition rate of 0.5 Å/s in the same deposition test. It was found that almost all INFICON High Precision Sensor Crystals show excellent stability in the activity signals. A small instability in the activity signal is observed for a few crystals. This activity instability is not caused by the crystals, but is due to electrical contacts to the crystal sensors.

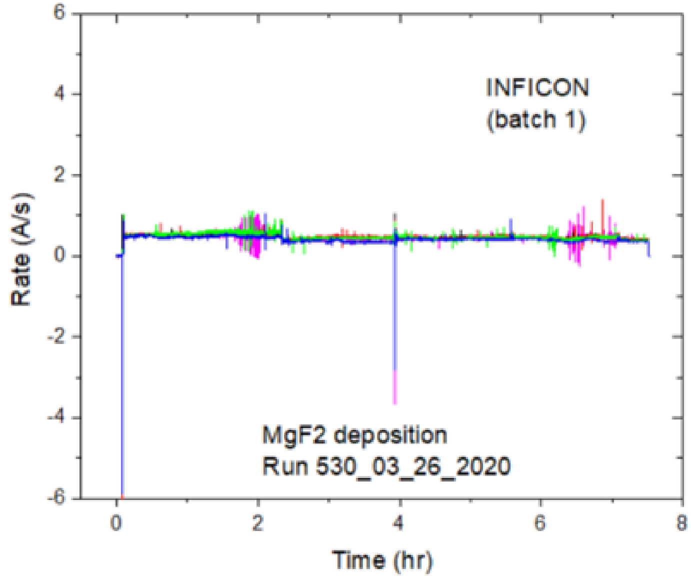


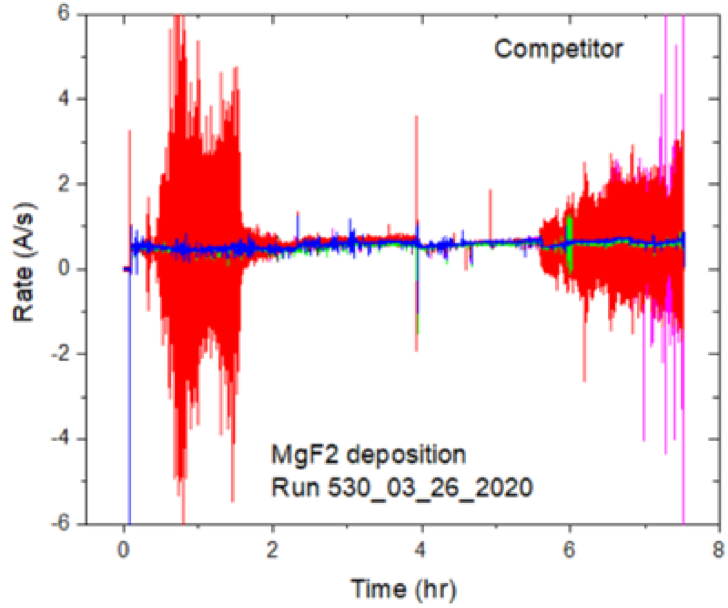

To ensure the batch-to-batch reproducibility of INFICON High Precision Sensor Crystals manufactured, batches of crystals were manufactured on different days and also tested in different depositions. The figure below shows the plot of the crystal activity versus deposition time for the last batch of INFICON High Precision Sensor Crystals (batch 3), and the competitor's crystals. The activity stability of INFICON High Precision Sensor Crystals is similar or better than that of the competitor's crystals. From the previous figures, it can be seen that INFICON High Precision Sensor Crystals from different batches show consistent results in the activity stability, indicating an excellent batch-to-batch consistency in our manufacturing processes.

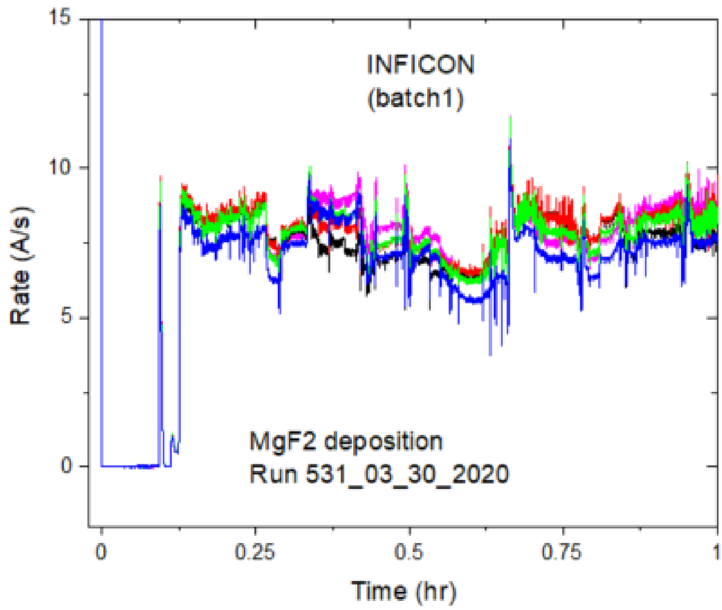


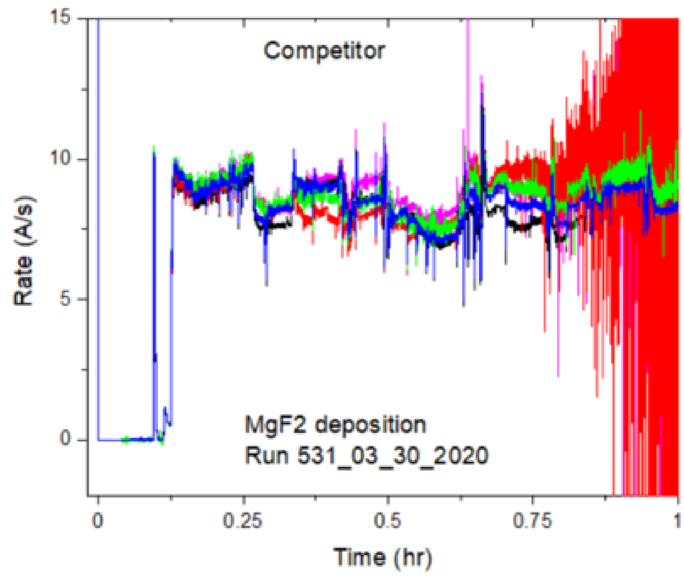



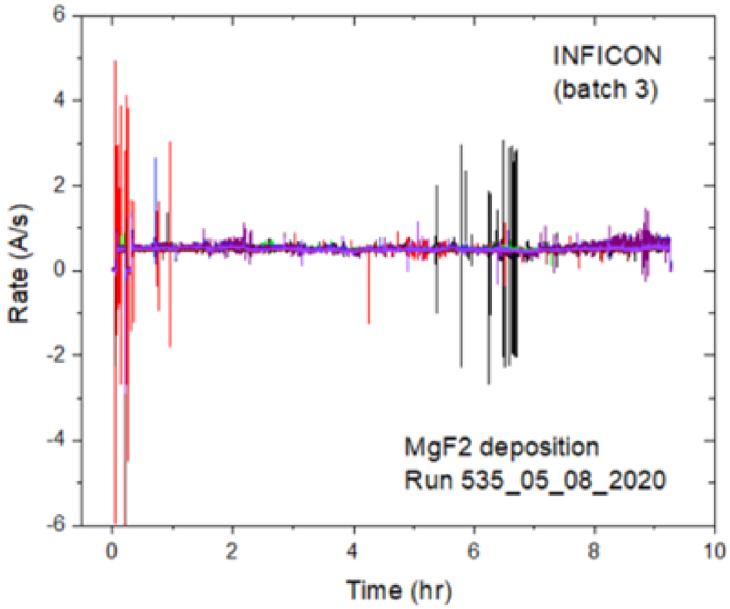

Besides the improvement of the activity stability, another main goal for us is to increase the rate stability for INFICON High Precision Sensor Crystals. The deposition rate versus deposition time for INFICON High Precision Sensor Crystals and the competitor's crystals is shown in the figures below for Batch 1 INFICON High Precision Sensor Crystals (controlled rate of 0.5 Å/s), Batch 1 INFICON High Precision Sensor Crystals (controlled rate of 8.5 Å/s), and Batch 3 INFICON High Precision Sensor Crystals (controlled rate of 0.5 Å/s), respectively. As these figures show, compared to the competitor's crystals, all INFICON High Precision Sensor Crystals show better or similar rate stability. Some common spikes (large rate deviation) in the rate signals are observed for both INFICON High Precision Sensor Crystals and the competitor's crystals. Since these spikes are common for both sets of crystals, they are not generated by the crystals. These spikes may have been generated by deposition anomalies.

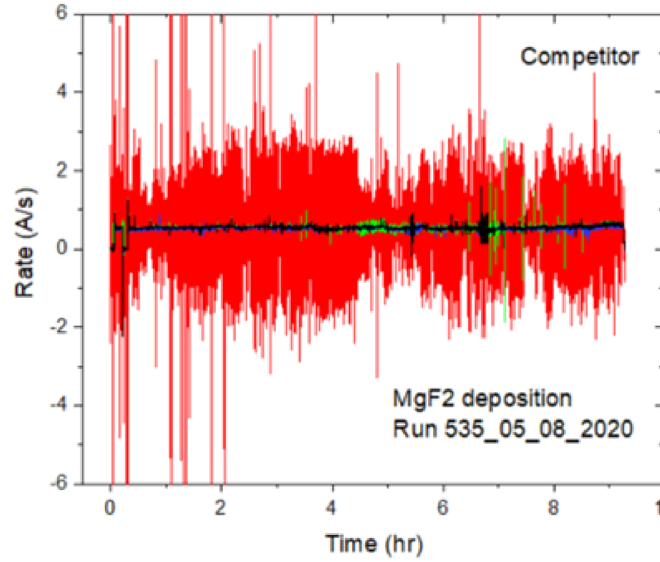





## Summary

In order to manufacture and supply crystals with higher activity stability, we performed a detailed investigation into all components of crystal manufacturing and many process parameters.

INFICON developed new crystals (INFICON High Precision Sensor Crystals) by optimizing many process steps, parameters, and materials. A wide range of experiments were performed to evaluate and compare the performance of INFICON High Precision Crystals and the competitor's crystals.

It was found that:

- The activity stability of INFICON High Precision Sensor Crystals is higher than that of the competitor's crystals.
- The rate stability of INFICON High Precision Sensor Crystals is higher or similar to the competitor's crystals.
- The activity behavior of INFICON High Precision Sensor Crystals is more consistent than the competitor's crystals.
- The manufacturing process of INFICON High Precision Sensor Crystals is highly reproducible and batch-to-batch results are consistent.