ONEIDA RESEARCH SERVICES, INC.
INSPECTION • ANALYSIS • TESTING

Ultra-High Vacuum Evolved Gas Analysis (UHV-EGA) Application Note 3: Investigating the Origin of Organic Contamination in Ceramic UB Substrates

Introduction

Ultra-High Vacuum Evolved Gas Analyzer (UHV-EGA), model 510, is a mass spectrometer system that has been developed by Oneida Research Services, Inc. to identify gases evolved from any type of solid or semi-solid sample (1) under ultra-high vacuum conditions and (2) over a temperature range. This method is particularly valuable in the microelectronics industry where hermetically sealed packages are manufactured using variety of components including adhesives, metal and/or ceramic substrates and lids. Prior to sealing these components in hermetic packages, baking of these components at a specific temperature for a fixed time is critical. Improper baking will result in outgassing that could cause the presence of unwanted gases inside the package and lead to moisture failures per MIL-STD 883, TM 1018 - also known as internal gas analysis (IGA) testing.

Principle of Operation

UHV-EGA involves heating a sample in an ultra-high vacuum environment (< 5E-8 Torr) to temperatures up to 1200°C. The gases released during this process are dynamically identified and characterized by a quadrupole mass spectrometer with a mass range of 1-300 amu. The specially designed ultra-high vacuum heater can achieve ramp rates from 0.1°C to 15°C per minute.

EVACS™ : Superior Operation and Data Processing

The UHV-EGA system runs on the dedicated EVACS™ software platform that provides user friendly procedure set up and operation, to conduct a seamless analysis. Developed by highly experienced programmers at ORS, EVACS™ facilitates precise control of the heating protocols along with mass spectrometer tuning and vacuum parameters, ensuring accurate and reproducible results. EVACS™ also provides multiple data processing methods to understand the acquired results. It features robust data processing capabilities, including real-time monitoring, automated peak identification, and comprehensive data analysis tools including matching unknown mass spectra with NIST database. The user-friendly interface streamlines the operation, making it accessible for researchers and engineers to achieve high-quality data with ease.

Applications

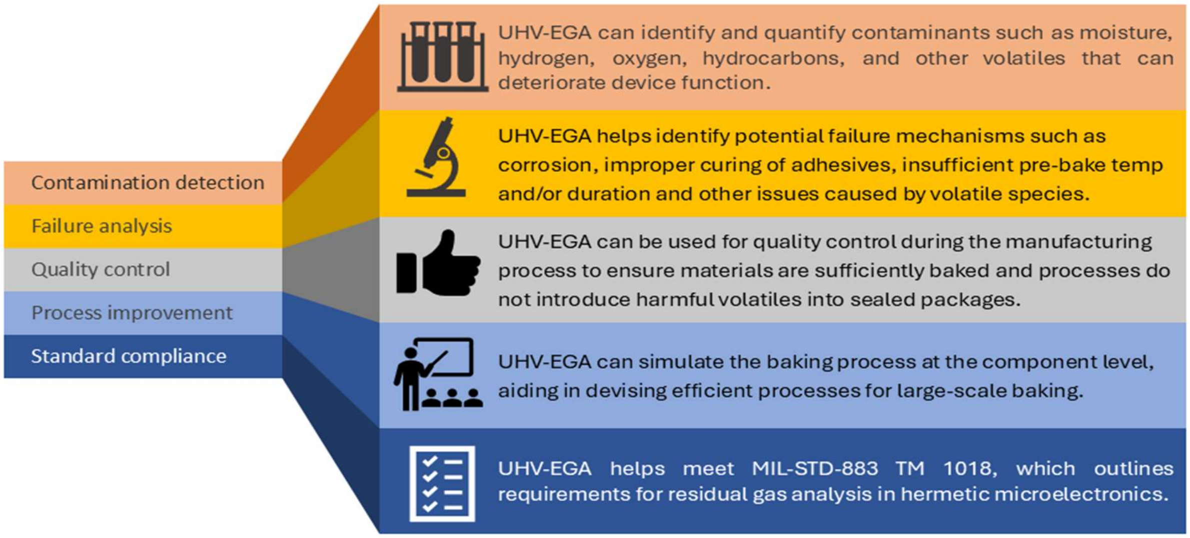


Fig. 1: *Common applications for UHV-EGA*

Case Study: Origin of contamination in ceramic components used for UB packaging

Ceramic Universal Base (UB) packages in microelectronics are protective housings for electronic components, especially integrated circuits (ICs), offering a combination of electrical insulation, thermal management, and mechanical protection. Ceramic UB packages are commonly used in high-end applications, such as military, aerospace, and medical electronics, where reliability and performance are critical. They are often used in applications requiring high reliability and performance, such as high-frequency devices and power electronics.

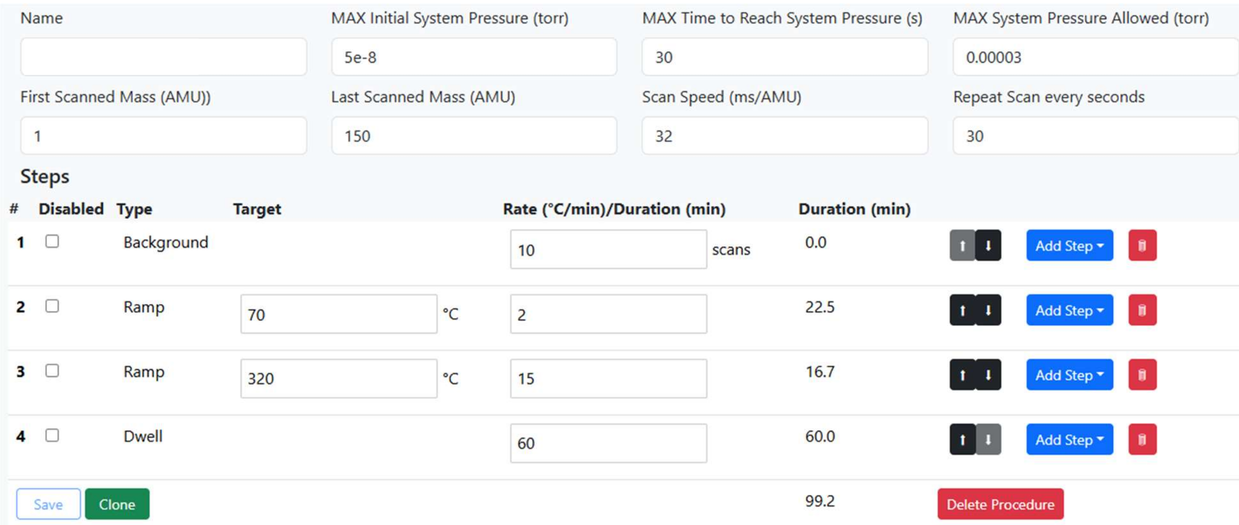
Using clean ceramic components in UB packaging is crucial for maintaining the performance, reliability, and longevity of electronic devices. Clean ceramic components ensure that no residual contaminants, such as organic compounds or moisture, are present. These contaminants can outgas during device operation, leading to the release of unwanted particles or gases. Such outgassing can impact device performance. For example; in applications requiring vacuum or hermetic sealing, such as aerospace, outgassing can compromise the integrity of the sealed environment, affecting device functionality. By ensuring ceramic components are thoroughly cleaned and free from impurities, manufacturers can mitigate these risks and enhance the overall performance and reliability of their devices.

The UHV-EGA (Ultra-High Vacuum Evolved Gas Analysis) technique enables the investigation of impurities in solid materials, including ceramic materials commonly used in UB packaging. This case study demonstrates the analysis of impurities in ceramic UB packages and explores the process of identifying and tracing the source of contamination back to the supplier.

Sample preparation

Small (0.093" x 0.105"x 0.03") UB ceramic substrates were received from a user who have been using these substrates for hermetic UB packaging. The samples were retrieved in clean laboratory environment. Total 150 substrates were packed in Ultra High Vacuum (UHV) grade aluminum boat. The UHV grade aluminum foil is free from contamination and residual rolling oils and certified to ASTM B-479 specifications. Packed samples are then placed on a heater in the UHV-EGA sample chamber to carry out outgassing analysis.

UHV-EGA test procedure



Name	MAX Initial System Pressure (torr)	MAX Time to Reach System Pressure (s)	MAX System Pressure Allowed (torr)
	5e-8	30	0.00003
First Scanned Mass (AMU))	**Last Scanned Mass (AMU)**	**Scan Speed (ms/AMU)**	**Repeat Scan every seconds**
1	150	32	30

Steps

#	Disabled	Type	Target	Rate (°C/min)/Duration (min)	Duration (min)	
1	☐	Background		10 scans	0.0	Add Step
2	☐	Ramp	70 °C	2	22.5	Add Step
3	☐	Ramp	320 °C	15	16.7	Add Step
4	☐	Dwell		60	60.0	Add Step
Save Clone					99.2	Delete Procedure

Fig. 1: A screen capture of the UI in EVACS™ used to define heating protocol for the analysis. MAX initial system pressure (Torr) defines the maximum value for system pressure required to start the analysis. This means pressure has to below 5E-8 torr to initiate the analysis. During analysis the max system pressure allowed defines the value over which MS will shut off the filament and stop the analysis. This limit is essential to protect ion source filament from high gas pressure. First and last scanned mass gives the mass range over which the analysis will be performed. For example, 1 to 150 amu scan were taken for this study. Scan speed is scan time per amu. For example, when a scan is done at 32 ms/amu speed, to complete the scan over 1-150 amu, the total scan time will be 32 ms * 150 = 4,800 ms = 4.8 s. Scans were repeated every 30 seconds.

Table lists steps for heating schedule. The first step is for collecting 10 background scans without any change in the temperature. Second step is a ramp type step, where heating is started from standby heater temperature (i.e., 30°C) value to 70°C, at the rate of 2°C/min. "Duration" lists the time estimated to achieve this step. The next step is set with higher heating rate (i.e., 15°C/min) to heat up to 320°C. The fourth step is dwell type, where the temperature of 320°C is maintained for 60 mins. The analysis will be stopped after completing all the four steps, which is estimated to be completed in 99.2 mins.

Results

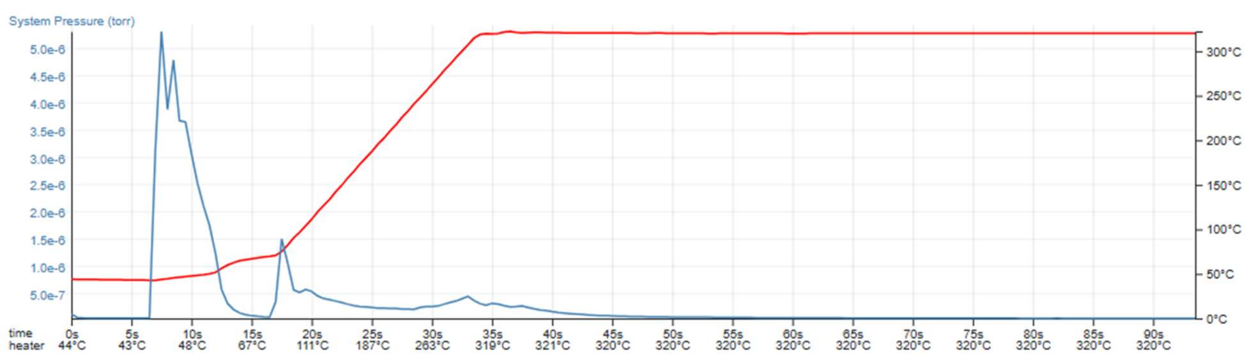


Fig. 2. Pressure graphs: total gas release (torr) with respect to sample temperature and time.

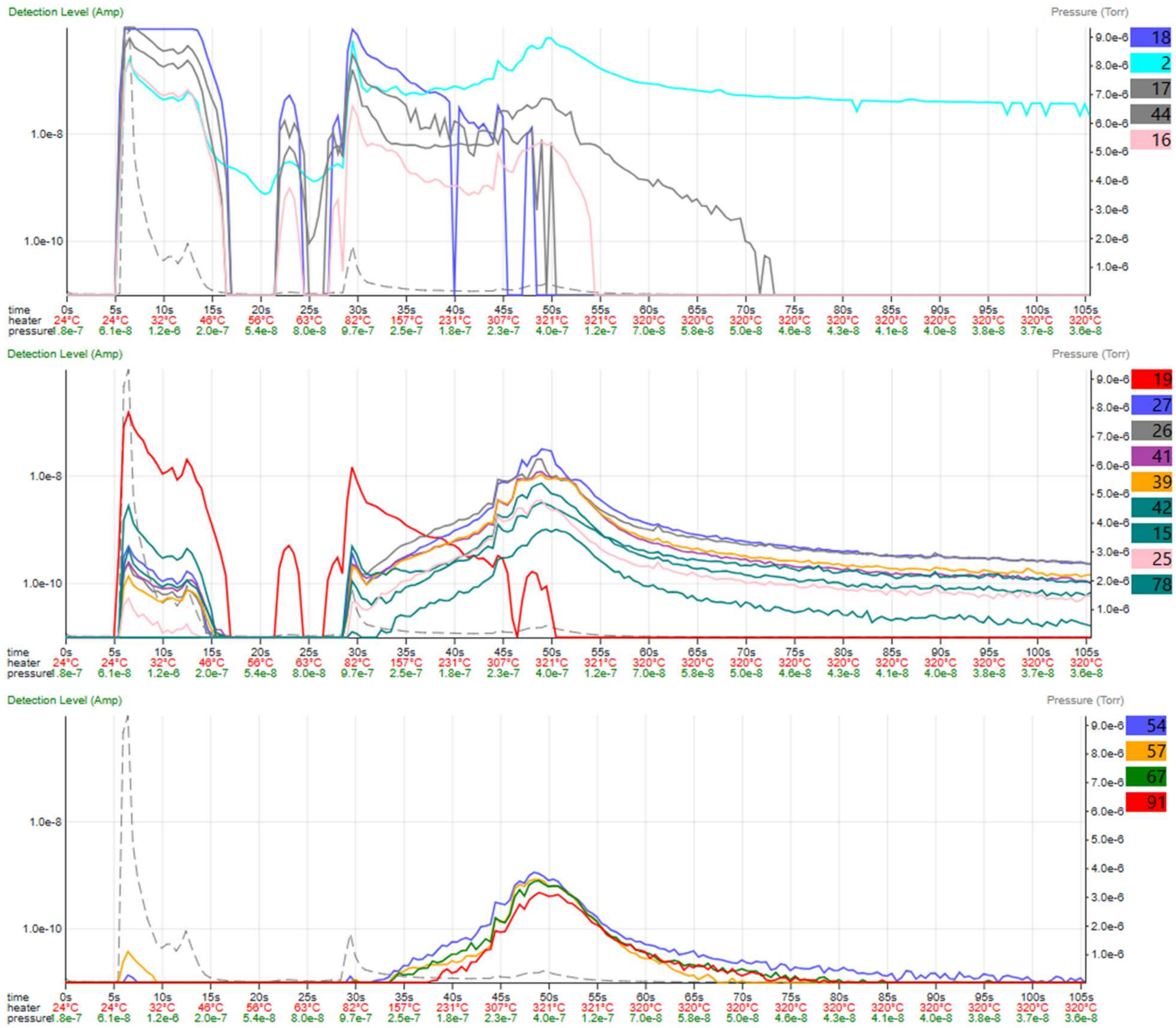
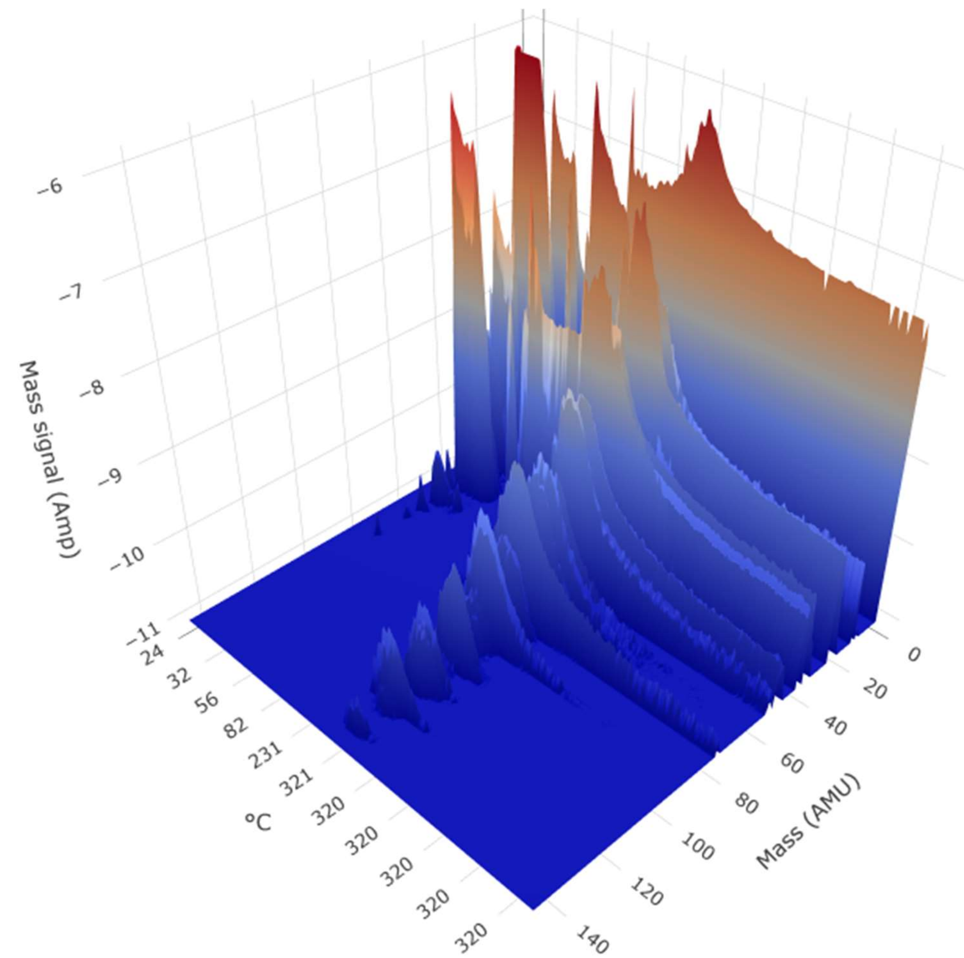


Fig. 3. Plots showing outgassing of various gases with respect to sample temperature. The top plot shows mass signals for top 5 masses (likely to be 18(Moisture), 2(Hydrogen), 17(Moisture fragment (OH ion)), 44(CO_2), 16(Moisture fragment (O ion)). The rest of plots show variety of mass fragments usually associated with outgassing from organic residue. Mass 19 may represent release of Fluorine, could result of hydrogen fluoride (HF). Presence of masses like 54, 57, 67 and 91 indicate fragments released due to hydrocarbon species, could also be contributed by organic residue present on these samples.

3D representation of gas release

The dedicated software, EVACS™, which powers the UHV-EGA system, offers robust data processing capabilities and enables comprehensive data analysis. Among its advanced features is the ability to generate three-dimensional representations of gas release, providing invaluable insights into outgassing behavior. The plot visualizes the relationship between three critical variables: (1) mass signal or signal intensity measured for each mass as ion current, (2) mass, expressed in atomic mass units (amu), and (3) the sample's temperature during heating.

This three-dimensional representation delivers a holistic overview of the entire dataset, plotting outgassing profiles for each mass across the range of heating temperatures. The visualization not only highlights patterns and anomalies in gas evolution but also facilitates the identification of specific masses associated with particular thermal events. By leveraging such detailed and intuitive data presentation, EVACS™ enhances the



accuracy and depth of impurity analysis in materials, further advancing investigative capabilities for materials designed for ultra-high vacuum environments.

Fig. 4: The 3D plot above compares mass signals for each mass with respect to sample temperature. The ceramic UB substrates certainly shows the presence of organic residue primarily released around 300°C. The plot also suggests that higher masses (>80 amu) are reduced to background when substrates are subjected to baking at 320°C for 60 mins.

The results clearly indicate that ceramic UB substrates are contaminated by organic residues which are outgassing in ultra-high vacuum at elevated temperature. Using these substrates for UB packaging could compromise device functionality and result in operational failures.

The findings prompted further investigation into the source of the contamination. Discussions with users revealed that the processes employed by the supplier in manufacturing these substrates could be contributing factors. For instance, the presence of mass 19 can be attributed to the H3O ion, which may originate from acids used in certain processes. Similarly, mass 26 could be associated with the CN ion, as chemicals like KCN or NaCN are commonly utilized in plating or cleaning procedures.

However, some observed masses could not be explained by the chemicals used during manufacturing processes. One potential source of contamination identified was the wax applied during the dicing process. Although steps are typically undertaken to remove organic residues from the wax, the effectiveness of these cleaning measures are not well established. Inefficient cleaning could leave behind organic residues, which may subsequently contribute to contamination.

To ascertain whether the wax is the source of the impurities in ceramic UB substrates, a direct UHV-EGA analysis was planned for the wax material.

Sample Preparation

Small amount of wax applied on an alumina substrate was obtained from ceramic substrate supplier. This sample is then placed on a heater in the UHV-EGA sample chamber to carry out outgassing analysis.

UHV-EGA test procedure

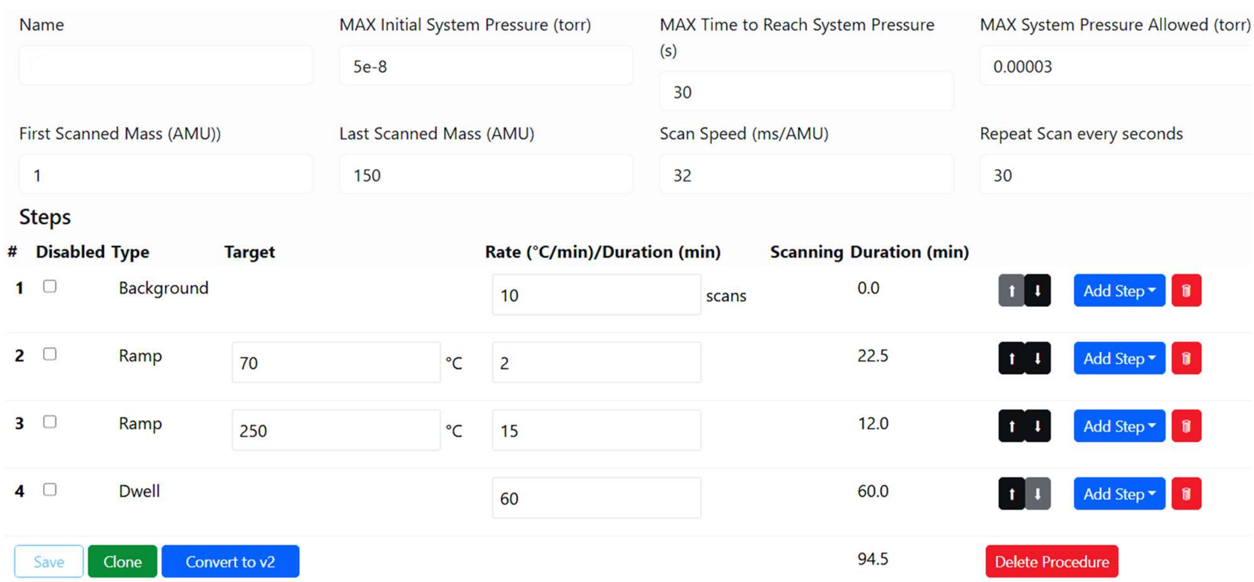


Fig. 5: A screen capture of the UI in EVACS™ used to define heating protocol for the analysis of the wax sample. The maximum temperature of the heating was set to 250°C with a dwell time of 60 mins. All other parameters are kept similar to the parameters set for the ceramic UB substrates analysis.

Results

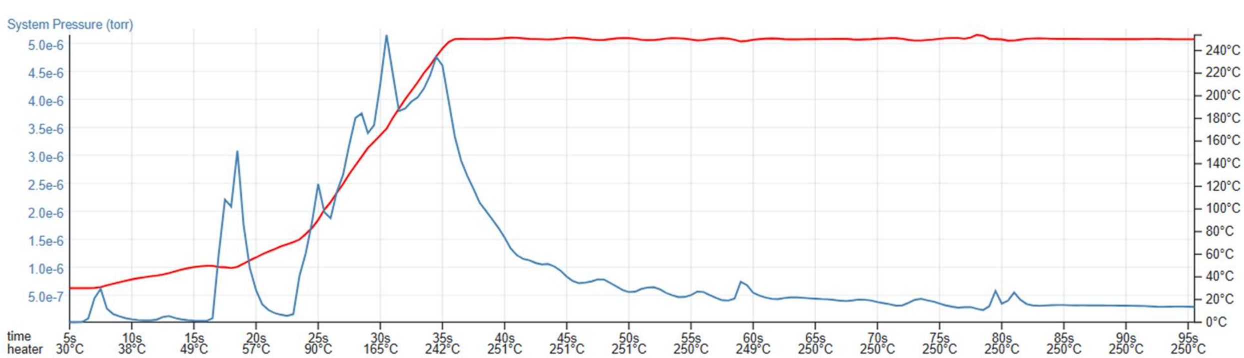


Fig. 6. Pressure graphs: total gas release (torr) with respect to sample temperature and time.

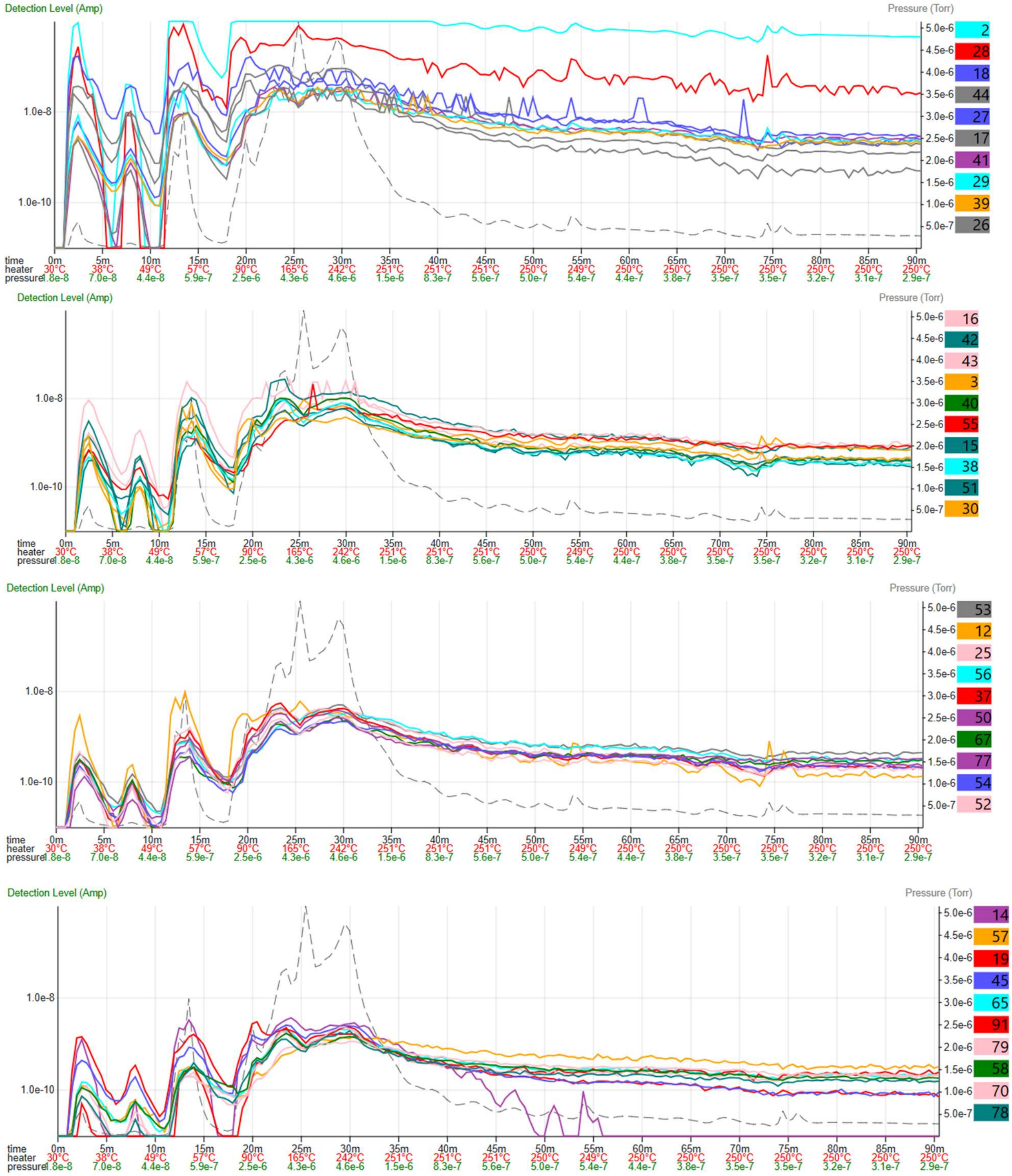


Fig. 7. Plots showing outgassing of various gases with respect to wax sample temperature. The plots show all the masses that could come from wax residue when subjected to heating. The major release occurs at temperature ~165°C.

3D representation of gas release

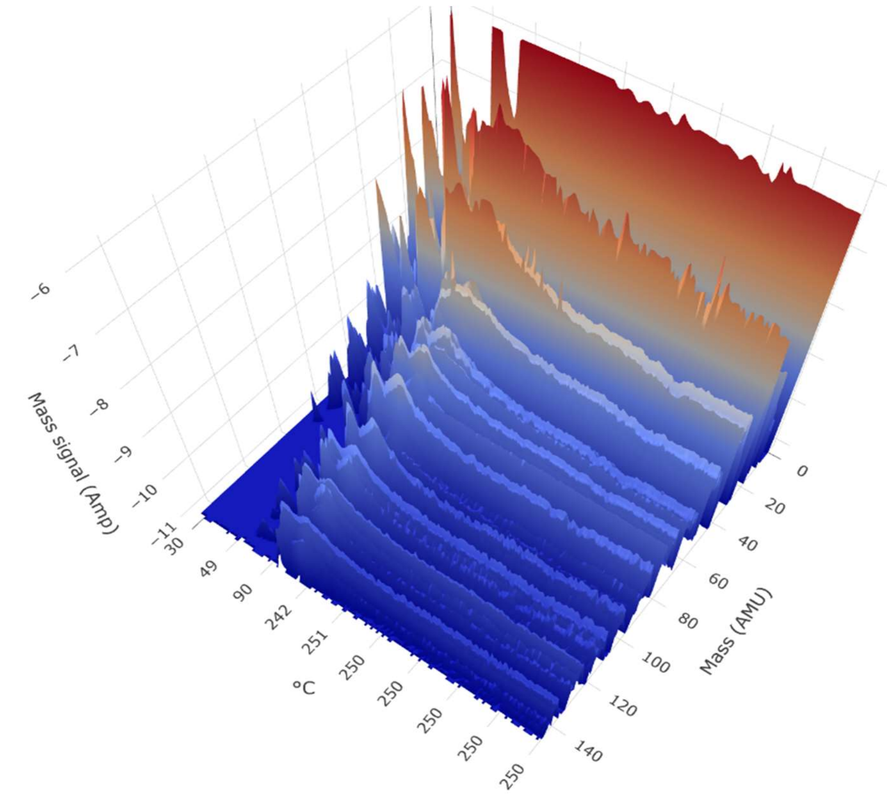


Fig. 8: The 3D plot above compares mass signals for each mass with respect to sample temperature. The wax sample shows how decomposition of wax sample can release range of organic fragments that can evaporate when subjected to higher temperature.

Conclusion

The case study highlights that strengths and capability of UHV-EGA technology in characterizing contaminant and track down the contaminant to the source. UHV-EGA technology can be used to find the baking parameters that can effectively remove wax residue through heating treatment when chemical cleaning is not sufficient. For the case study following conclusions can be made:

- Ceramic UB substrates:
    - UHV-EGA can find and characterize organic residues on the substrates.
    - The test reveal that chemical processes used by manufacturer to remove wax residue is not efficient.
    - The 3D representation, shows that contamination from ceramic substrates can be removed by baking (i.e., Fig. 4)
- Wax sample:
    - UHV-EGA can identify each mass that can release from the wax when subjected to heating.
    - Masses released from the wax indeed matches with the masses observed in ceramic substrates.
    - The 3D representation, shows that the outgassing from wax sample continue during the entire heating schedule, however tends to reduction (Fig. 8). This suggests that if manufacturer introduces a baking step in addition to chemical cleaning, ceramic substrates can be free of wax residue.

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