Ultra-High Vacuum Evolved Gas Analysis (UHV-EGA)
Application Note 2:
Comparing Baking Processes for Adhesive Material

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

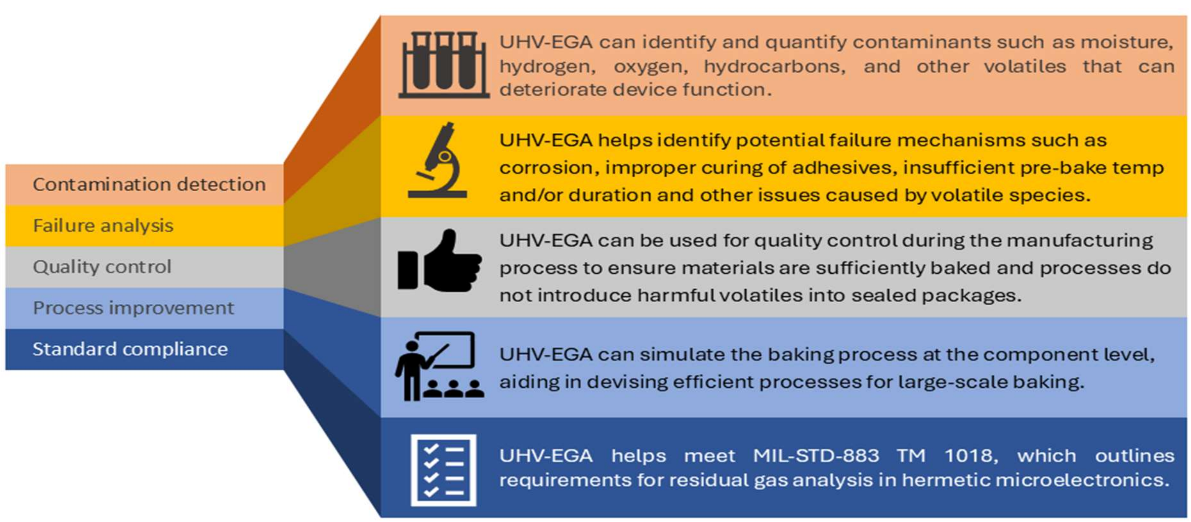

Fig. 1: *Common applications for UHV-EGA*

Case Study

Adhesive materials like Sylgard^{TM} 567 play a critical role in microelectronics hermetic packages by providing robust sealing and mechanical stability. These adhesives ensure that sensitive electronic components are protected from environmental factors such as moisture, contaminants, and temperature fluctuations, thereby extending the longevity and reliability of the device. Sylgard^{TM} 567, known for its strong adhesive properties and thermal stability, is often utilized due to its compatibility with microelectronics and ability to maintain a tight seal under varied conditions. However, improper baking of the adhesive during the curing process can result in a compromised product. If not baked at the specified temperature or duration, the adhesive may not fully cure, leading post sealing outgassing. Such outgassing in internal volume can promote further chemical reaction resulting in presence of moisture and volatiles above the allowed limit. Proper baking is thus essential to ensure the adhesive achieves optimal properties and fulfills its intended purpose.

UHV-EGA has the capability to perform a comparative study between the baking processes. In this case study we compare two baking process to understand behavior of the adhesive during the baking. The study characterizes outgassing species when same material is exposed to two different heating schedules. We extend our case study by repeating a baking process on the same sample to investigate if the material needed baking at a higher temperature.

Baking process -1

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	15

Steps

#	Disabled	Type	Target	Rate (°C/min)/Duration (min)	Scanning	Duration (min)
1	☐	Background		10 scans		0.0
2	☐	Ramp	40 °C	2		7.5
3	☐	Ramp	175 °C	5		27.0
4	☐	Dwell		120		120.0
						154.5

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Baking process -2

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	15

Steps

#	Disabled	Type	Target	Rate (°C/min)/Duration (min)	Scanning	Duration (min)
1	☐	Background		10 scans		0.0
2	☐	Ramp	40 °C	2		7.5
3	☐	Ramp	150 °C	5		22.0
4	☐	Dwell		1440		1440.0
						1469.5

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Baking process -3

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	15

Steps

#	Disabled	Type	Target	Rate (°C/min)/Duration (min)	Scanning	Duration (min)
1	☐	Background		10 scans		0.0
2	☐	Ramp	40 °C	2		7.5
3	☐	Ramp	150 °C	5		22.0
4	☐	Dwell		30		30.0
5	☐	Ramp	175 °C	5		5.0
6	☐	Dwell		120		120.0
7	☐	Ramp	200 °C	5		5.0
8	☐	Dwell		240		240.0
						429.5

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Fig. 1: A screen capture of the UI in EVACS[TM] used to define heating protocol to simulate three different baking processes for the adhesive material. MAX initial system pressure (Torr) defines the maximum value for system pressure required to start the analysis. This means pressure has to be 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 15 seconds.

Table lists steps for heating schedule. For example, for baking process-1, 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 40°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., 5°C/min) to heat up to 150°C. The fourth step is dwell type, where the temperature of 150°C is maintained for 120 mins. The analysis will be stopped after completing all the four steps, which is estimated to completed in 154.5 mins.

Sample strategy

For the analysis, two same samples, each has ~0.2 gm of cured adhesive material are used. For baking process-1, first sample was used. The second sample was analysed using baking process-2 and the same second sample is then exposed to baking process-3 without removing the second sample from UHV-EGA.

Results

The comparison between baking processes reveals distinct outgassing profiles for the adhesive material. During Baking Process-1, when the adhesive material is heated to 175°C and maintained at this temperature for 2 hours, the outgassing for mass 2 and mass 27 remains constant throughout the heating schedule. Outgassing of mass 18 is observed only until the material exceeds 150°C. A significant release is noted for mass 28, which may correspond to nitrogen at lower temperatures, while above 150°C, mass 28 is likely indicative of CO.

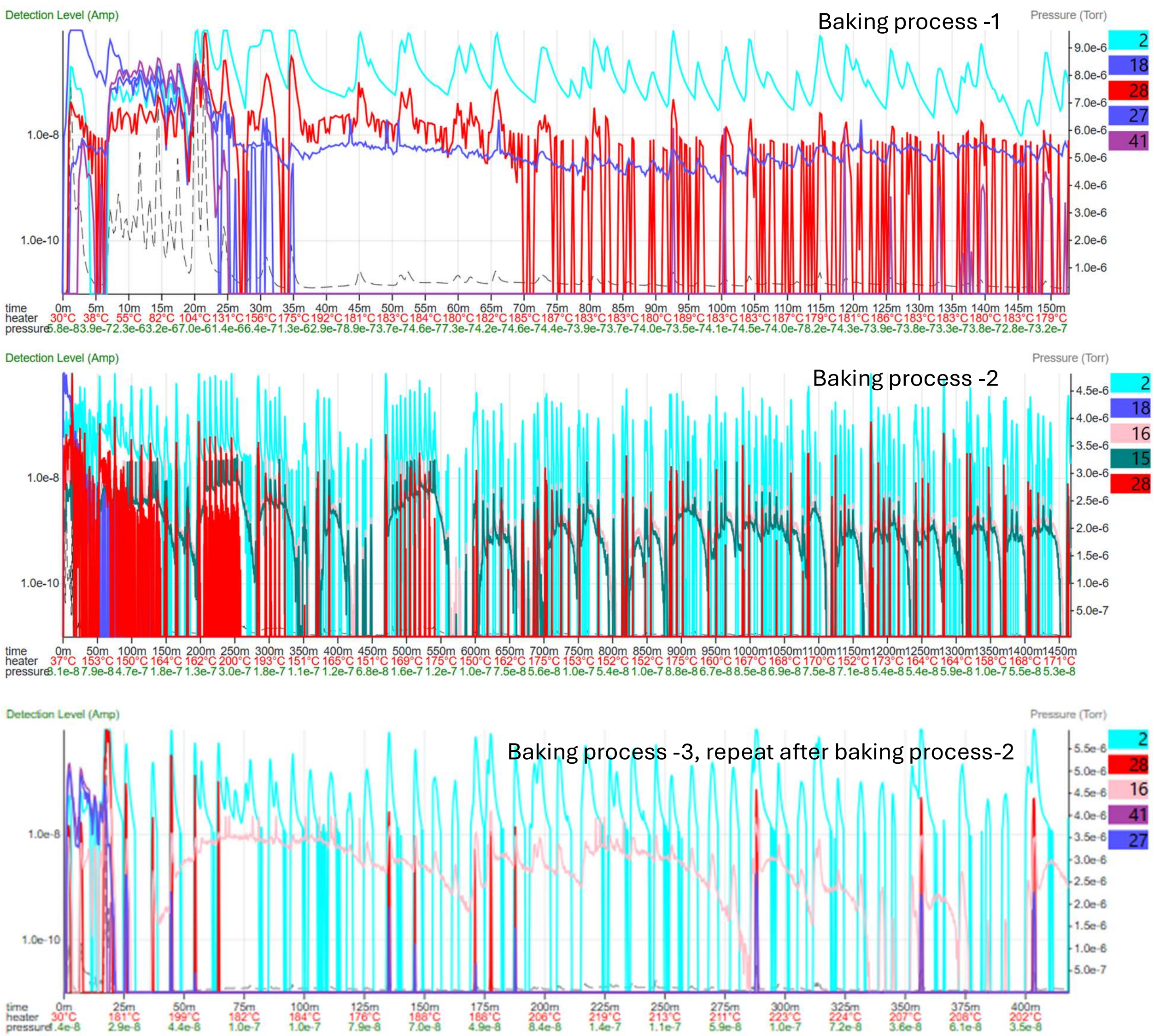

Fig. 2. Plots to compare three baking process. The plots compare mass signals for top 5 masses for each baking process.

In Baking Process-2, when the same material undergoes a 24-hour dwell period, all observed masses decrease to lower values, with spikes indicating outgassing occurring in bursts. Additionally, over the prolonged duration, outgassing of other masses, such as mass 15, is detected. This suggests that extended exposure to elevated temperatures facilitates the diffusion of other gases, which are subsequently released.

Baking Process-3 is conducted on the material previously subjected to Baking Process-2. As expected, very minimal outgassing is observed during this stage.

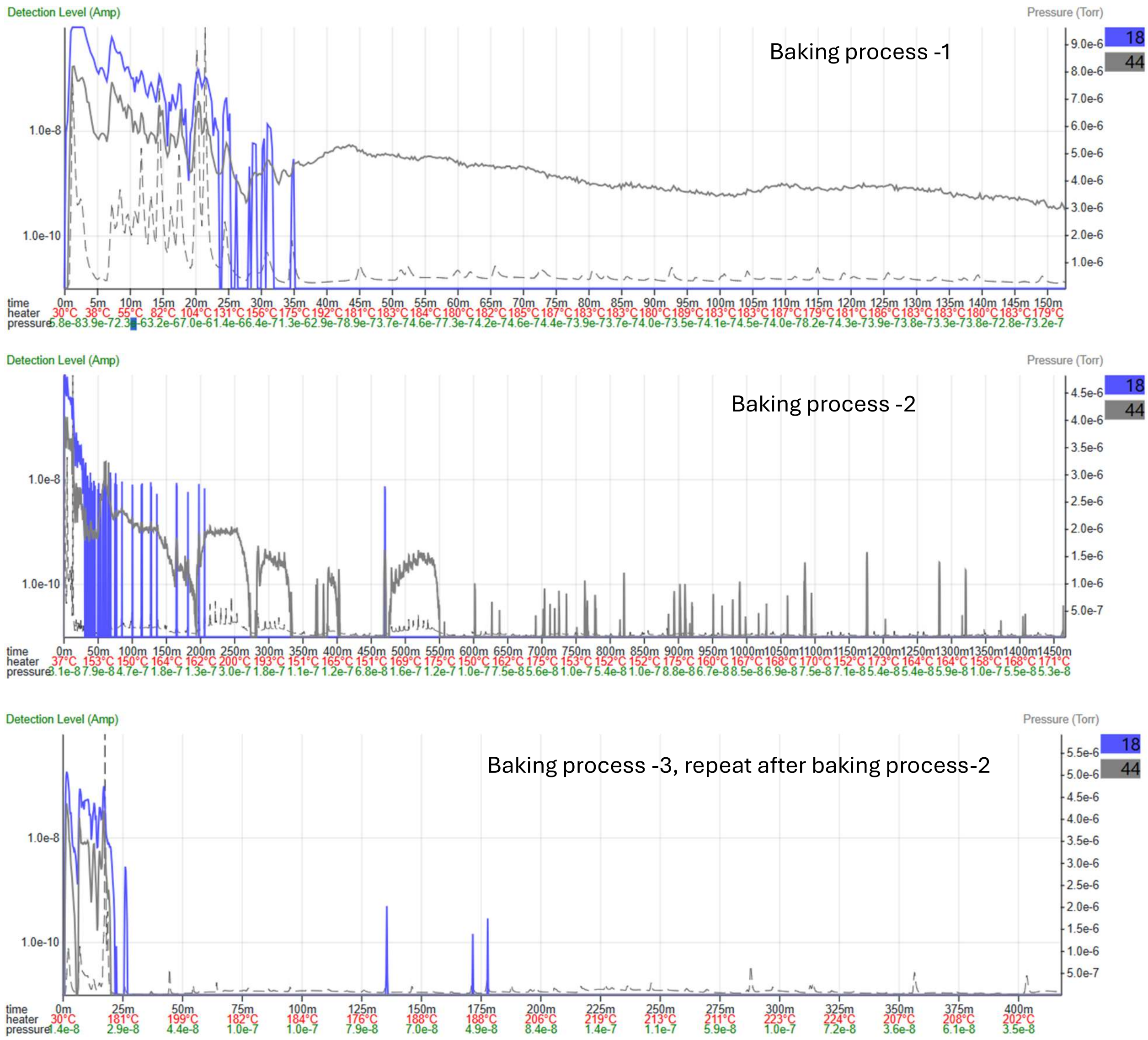

Fig. 4: The plot above compares ion current for mass 18 (i.e., moisture) and mass 44 (i.e., CO_2). The effect of three baking processes is clearly seen in this outgassing pattern. Re-baking step (Baking proces-3) confirms that almost no outgassing happens for the repeat baking process. Difference in outgassing efficiency is evident when comparing baking process-1 and baking process-2. Mass 44 continues to outgas in baking process-1, while longer baking process-2 eventually removes mass 44 from the material and accomplish complete outgassing.

Conclusion

The case study highlights that UHV-EGA technology provides a unique opportunity to accomplish a comparative study for different baking processes for the adhesive material. This technology proves that it can be easily used to verify response of baking processes on outgassing characteristics of adhesive material. For the case study following conclusions can be made:

- Baking process-1:
  - Demonstrates insufficient baking efficiency.
  - Does not achieve adequate outgassing when compared to other baking processes.
- Baking process -2:
  - Provides significantly improved baking efficiency.
  - A repeat baking step (baking process -3) performed after baking-2 confirms its sufficiency, as no outgassing is observed during baking process-3.
- Comparison of Baking Processes:
  - Moisture and CO_2 outgassing show clear differences between baking processes.
  - Highlights the importance of optimizing the baking process for efficient outgassing.

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