WHITE PAPER

How Clean is Your Reliability Chamber?

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INTRODUCTION

Stress testing of electronic devices and systems under specific heat and humidity conditions is required by industry, government and military organizations.[1] These tests are typically performed in environmental chambers, where the temperature and relative humidity can be easily controlled and varied.[2] One concern in this type of testing is the potential incidental contamination of valuable devices by a poorly maintained or poorly cleaned chamber itself.

There are several reasons why test chamber cleanliness is of great importance. Contaminants could inhibit desired electrical connections or facilitate electrical conduction where it is not desired, as well as obscure optical pathways and lead to esthetic degradation of samples. These degrading changes are beyond those that the elevated temperature and humidity are designed to induce. For example, organic and/or inorganic materials that are non-conductive could coat the surface of the device during the test. This could interfere with the electrical testing of the device resulting in perceived failures unrelated to the temperature/humidity stress test. Furthermore, this contaminant could be transferred onto the socket or probes used by ATE (automatic test equipment) and ultimately require the cleaning of that tool, which could significantly slow down the overall testing. This may also require the samples themselves to be cleaned and retested. Alternatively, if a conductive material is deposited onto certain parts, shorting and/or leakage paths could be generated, resulting in failures that are, again, unrelated to the actual reliability test. This may cause delays in qualifying the electronic device and require repeat testing. Thus, it is critical to understand what types of contaminants are released during damp heat, temperature cycling and other chamber-based testing.

In this paper, we determine the level of contamination in reliability stress chambers by placing a silicon witness wafer inside of the chamber to capture any foreign materials. A common damp heat reliability test (called “85/85”) is then performed where the chamber is heated to 85 °C with a relative humidity of 85% for 1000 hours. To obtain a representative sampling of different chamber environments, we evaluate five different chambers from three laboratories labeled A, B and C. A control wafer is also examined, which is not exposed to the chamber conditions but instead kept in a plastic wafer carrier for the same duration of 1000 hours.

Following the damp heat test, the surface of the wafers is analyzed by X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR). Both techniques provide chemical bonding information, but the former uses vibrational spectroscopy to determine the molecular structure, whereas the latter uses X-rays to identify elements and functional groups based on their binding energies. The two techniques also vary in their sampling depth: XPS probes the top 5-10 nm of a surface, whereas FTIR generally provides information about the entire thickness of the contaminant (bulk analysis). Finally, there is a notable difference in the sensitivity of the two techniques: FTIR can only detect species present at 5-20 wt%, whereas XPS is more sensitive can detect trace elements at ~0.1-1 wt%. Together, the two techniques can be used in combination to understand the surface composition.

The insight into the composition of contaminants provided by this paper can be used to develop and optimize cleaning processes for these chambers. We also demonstrate the effectiveness of using a witness wafer to determine the cleanliness of reliability chambers.

SAMPLE PREPARATION:

Six 100 mm Si wafers were obtained for the study. Three different labs (A, B and C) received a witness wafer and instructions regarding how to conduct the test. Lab A had three different chambers and Labs B and C had one chamber each. One wafer remained inside the plastic container for the duration of the test, to serve as the control. After the 85/85 stress test, the wafers were returned and analyzed by optical inspection, XPS and FTIR to determine the level of surface contamination present on each sample. In the cases where contamination was visible, analysis focused on the visibly contaminated areas of the wafer. When contamination was not visible, analysis was performed in the center of the wafer.

EXPERIMENTAL:

The sample wafers were examined by optical microscopy to determine if gross contamination was observed. Optical microscopy is very good at detecting particles, films and staining/roughening of the samples. A Keyence VHX-5000 microscope was used to look for defects and residues on the wafer surfaces.

Fourier transform infrared (FTIR) analysis was performed at different locations on the wafer using a Thermo-Nicolet 6700 spectrometer equipped with a Continuum microscope,. The analytical spot size was approximately 100 µm x 100 µm. The control wafer was used as the instrument blank as no residues were observed on its surface.

X-ray photoelectron spectroscopy (XPS) analysis was performed using a PHI Quantum 2000 spectrometer equipped with a monochromated Al Kα source. The analyses are performed at different locations and the analytical spot size is 1400 µm × 300 µm. The experimental data were then processed with MultiPak V8.1B.

RESULTS AND DISCUSSION:

Optical imaging was used to inspect the surface of each wafer and to obtain images of any contaminants. Figure 1 shows optical images of contaminants from Chamber A1.

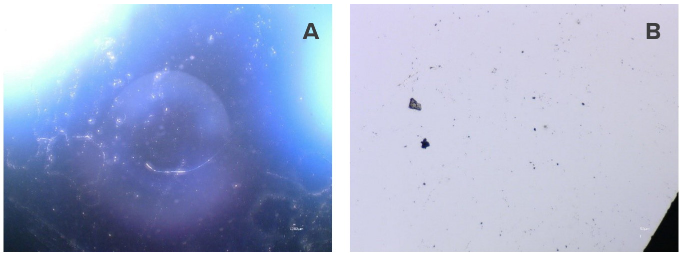

Figure 1 – Optical image of contamination on the wafer from Chamber A1, dark and bright field images. Different locations.

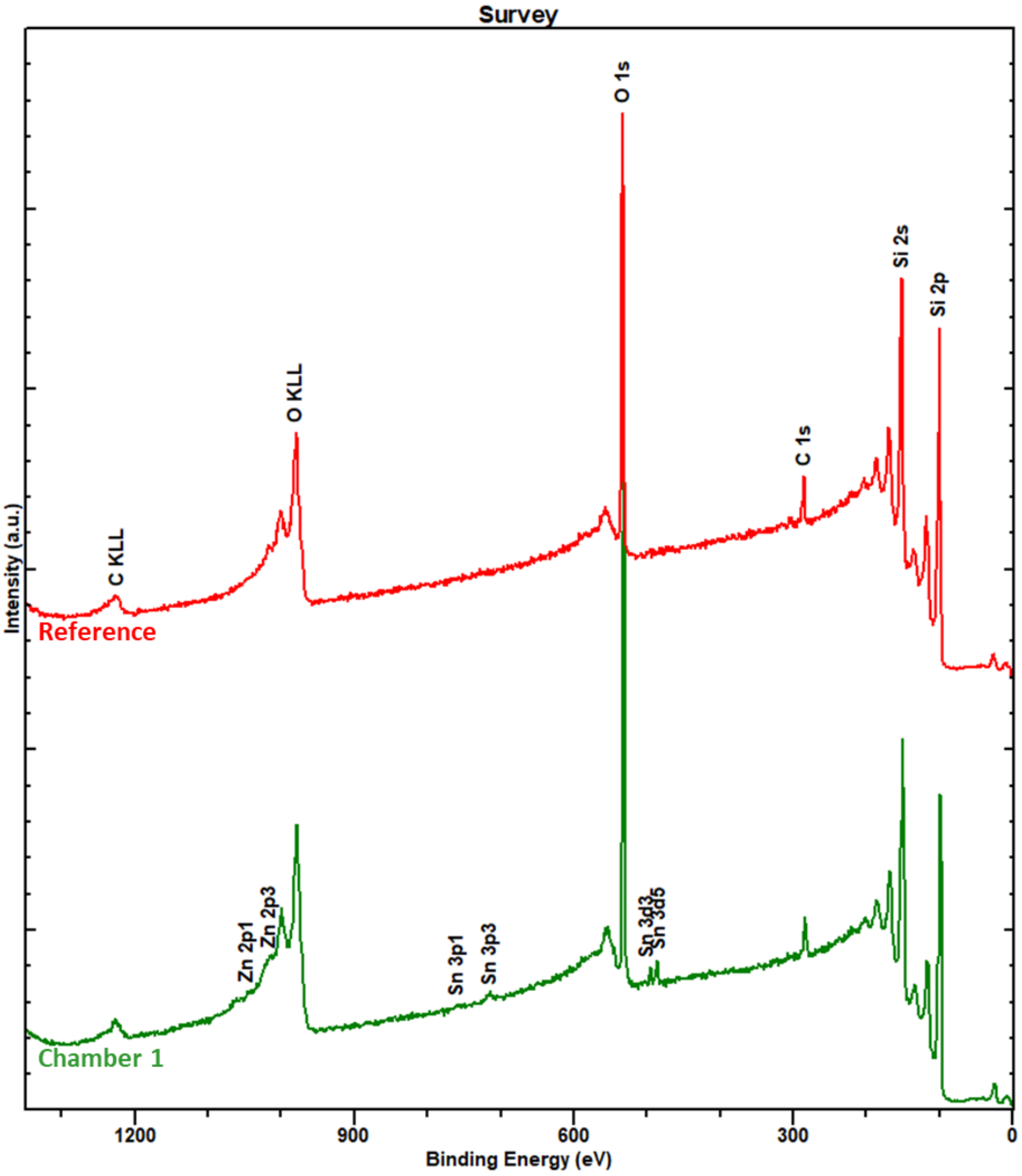

Figure 2: XPS of the reference wafer versus contamination on Chamber A1.

Figure 1 contains images from the same area that were obtained using dark field (A) and the bright field (B) microscopy. The two methods are useful for detecting different types of contaminants: bright field is better for residues, whereas dark field can show particles and fibers. Similar images were obtained of the other chambers, but they did not exhibit any obvious visible residues or contaminants.

XPS Results

The XPS results are summarized in Table 1. Figure 2 provides two representative XPS spectra, comparing Chamber 1A to the reference wafer. As shown in Table 1, the reference wafer contained a low level of adventitious/environmental carbon (10%) in addition to the oxidized silicon surface. This result is very typical for a clean Si wafer surface. Three different testing chambers were evaluated at Site A, and trace levels of Zn and Sn were detected on the surface of witness wafers from all three chambers (A1, A8 and A9), with the highest amount of carbon detected on the wafer from Chamber A9 compared to the other samples. The detection of Sn on the surface of the wafer from Chamber A1 is notable because this element is known to cause leakage and shorting, which could cause false failures in electrical components.

Chamber B1 had high levels of C and Na with trace Cl and N species. These elements may be associated with biological contamination possibly from handling. Chamber C1 was found to be similar to the reference wafer, but it showed the highest oxygen content of all samples and also higher N contamination compared to chamber B1.

Table 1. Summary of XPS results (Atomic%)

Sample	C	O	Si	N	Na	Cl	Zn	Sn
Reference wafer	9.6	36.0	54.4	-	-	-	-	-
Chamber A1	7.9	42.5	49.3	-	-	-	<0.1	0.2
Chamber A8	8.1	43.9	48.0	-	-	-	-	-
Chamber A9	11.6	42.9	45.6	-	-	-	-	-
Chamber B1	56.5	29.6	7.9	0.2	5.5	0.3	-	-
Chamber C1	10.6	44.1	44.5	0.8	-	-	-	-

FTIR Results

FTIR results are summarized in Table 2. Representative FTIR spectral overlays between the contaminants and library references are shown in Figures 3-6. As observed by XPS, Chamber A1 was found to be the most contaminated of all witness wafers. Figures

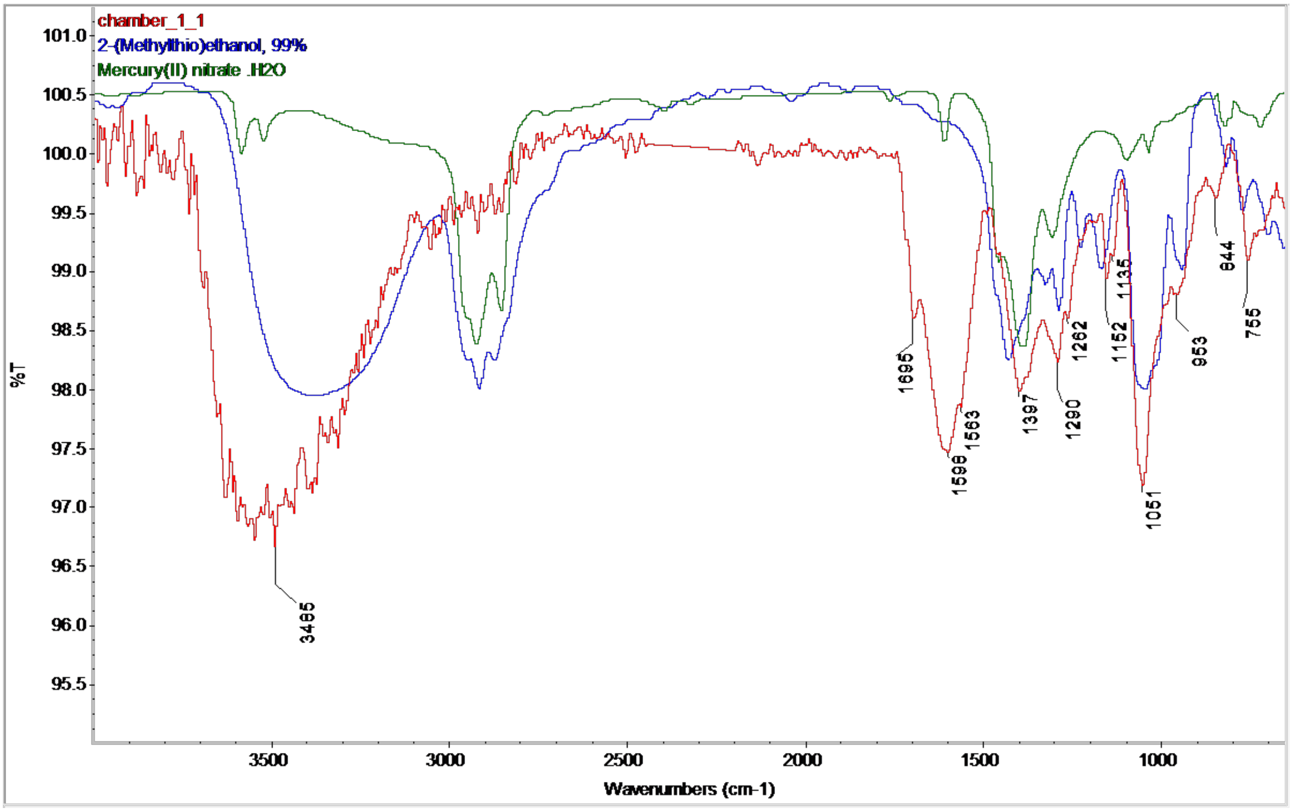

Figure 3. Overlay of Chamber A1 measurement 1

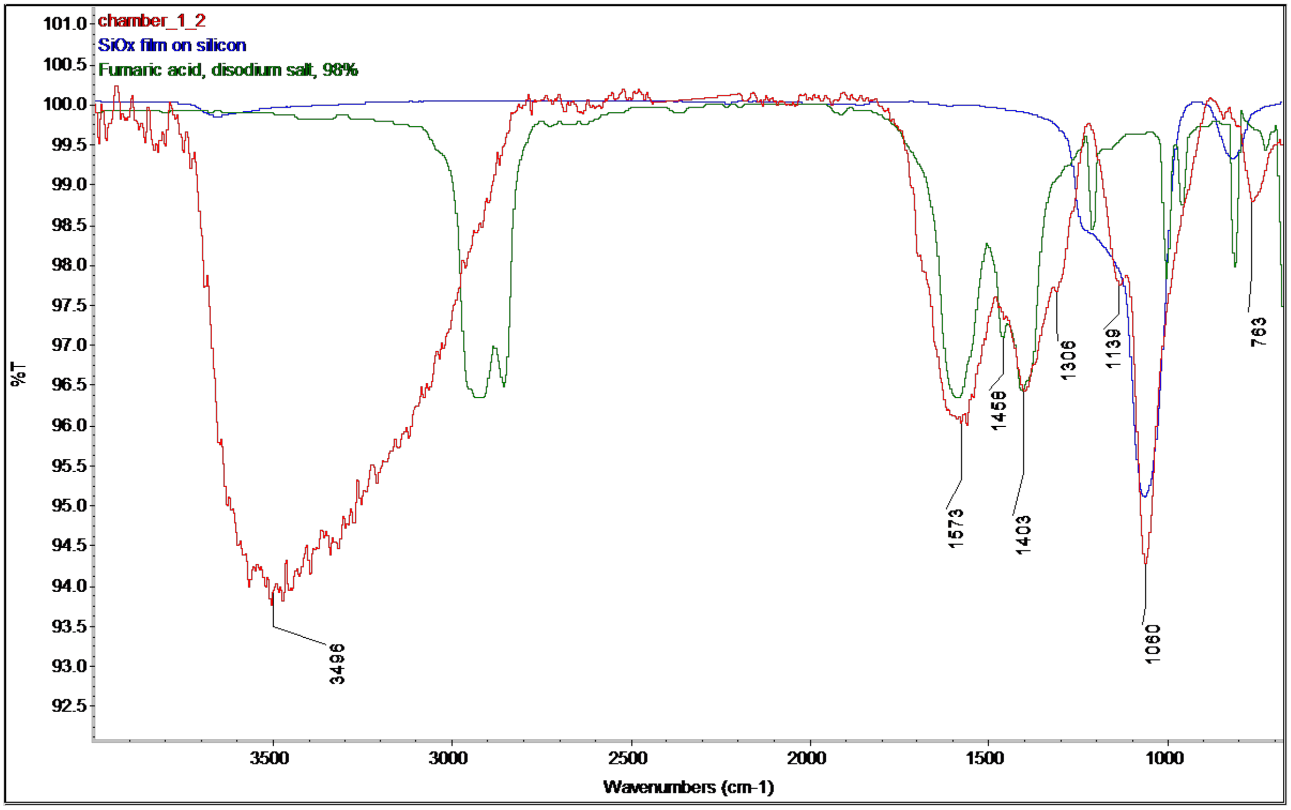

Figure 4. Overlay of Chamber A1 measurement 2

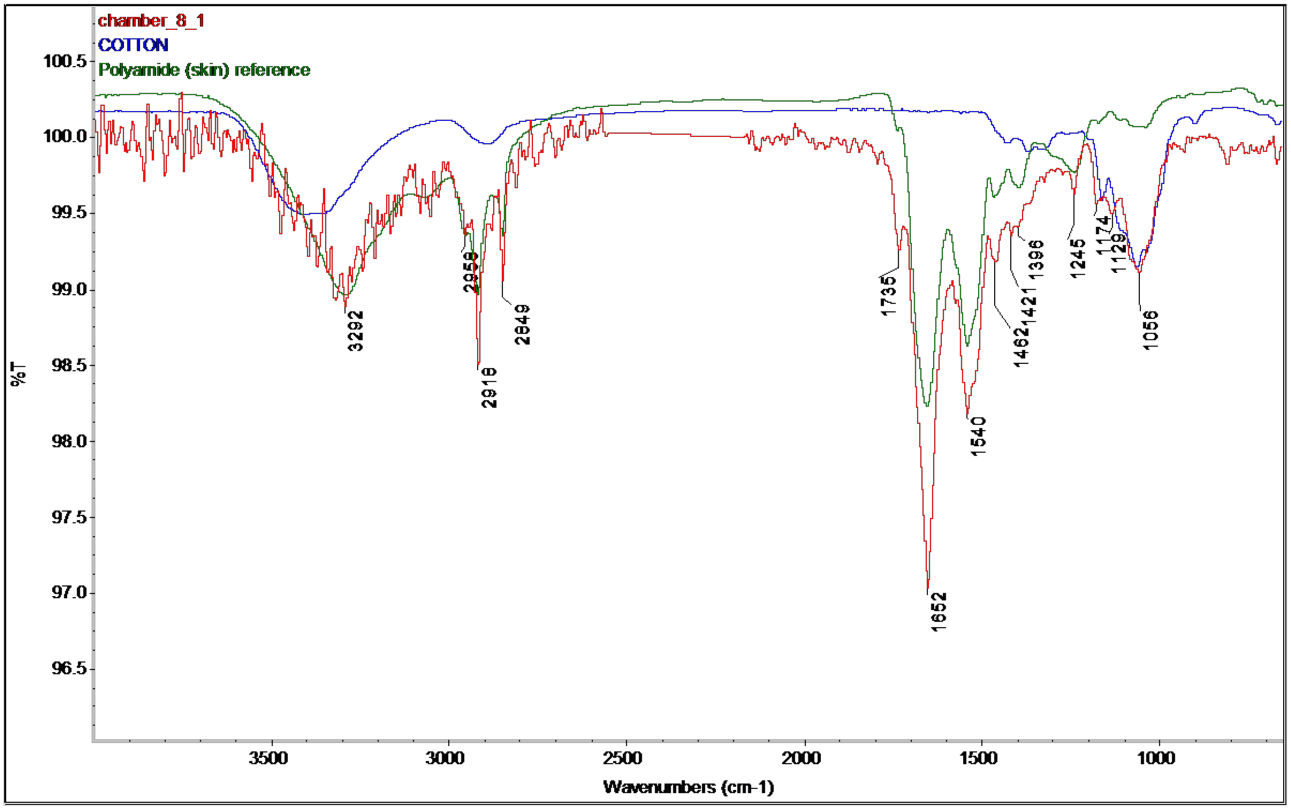

Figure 5. Overlay of chamber A8

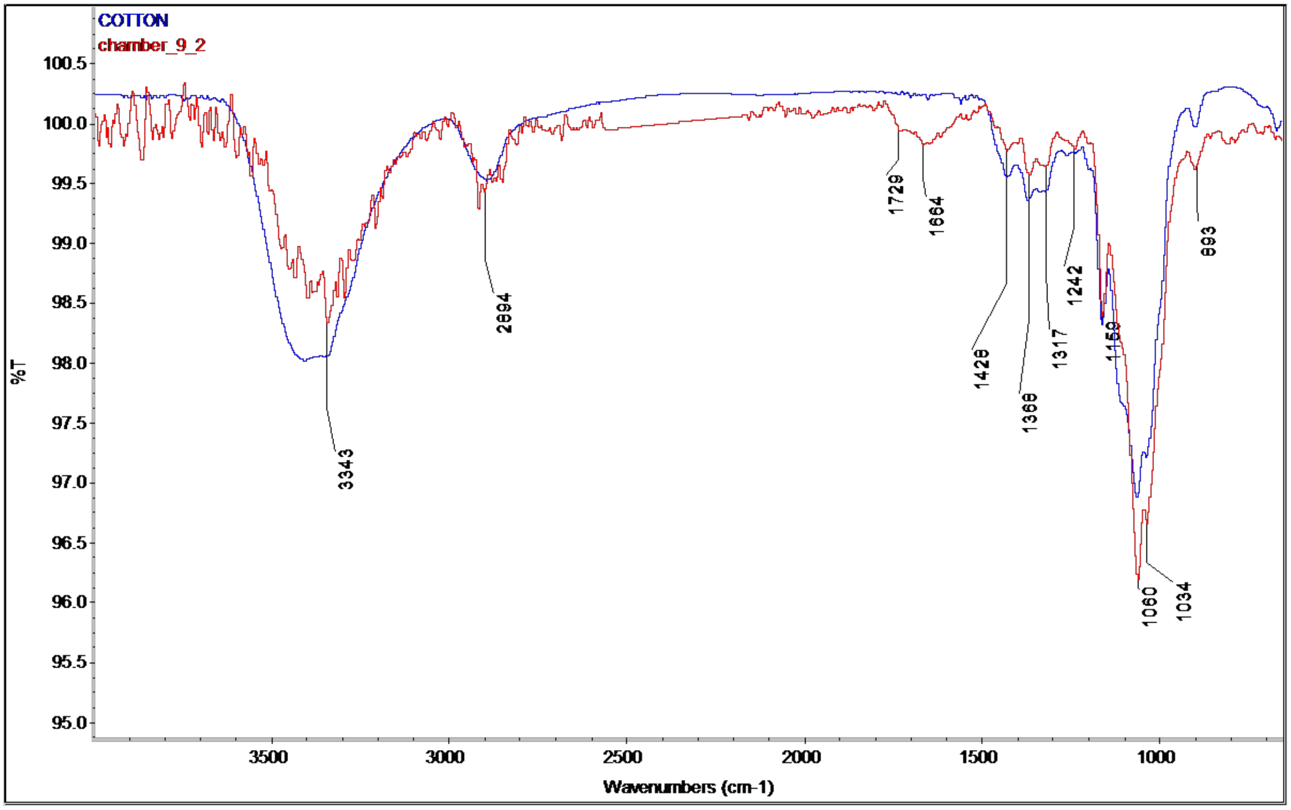

Figure 6. Overlay of chamber A9

3 and 4 demonstrate that the surface of this wafer is a mixture of nitrate species, organic acid, organic acid salt(s), possibly sulfur-containing species, water, silicon dioxide and possibly a saccharide similar to maltotriose. These results are consistent with the XPS finding of elevated oxygen levels as well as detection of Zn, which could be a counter ion for the organic acid salt. The detection of organic acid and acid salts is notable because organic acids are corrosive and can also create conductive paths.

In contrast to significant contamination in Chamber A1, Chambers A8 and A9 only showed contaminants associated with handling, such as polyamides, which may come from skin and cellulose, which may come from cotton. Chamber A8 also contained hydrocarbon species. Similarly, Chamber B1 was fairly clean and only contained silicate and amide species. The presence of amide species complemented the detection of trace N by XPS.

Chamber C1 was more contaminated, containing an interesting mixture of a polyamide similar to skin, possibly aromatic or amide species, ammonium species similar to ammonium sulfate and possibly barium sulfate. The lack of Ba and S and the presence of N in the XPS analysis is helpful in interpreting these results. High oxygen content detected by XPS as well as relatively higher N content support the presence of nitrate species.

Table 2

Description	**Principal FTIR observations**
Chamber A1	Mixture of possibly nitrate species, organic acid, organic acid salt, sulfur-containing species, water, silicon dioxide and possibly a saccharide.
Chamber A8	Polyamide similar to skin, trace ester and hydrocarbon species
Chamber A9	Polyamide similar to skin, cellulose similar to cotton, trace ester and possibly amide
Chamber B1	Silicate possibly similar to and amide species
Chamber C1	Polyamide similar to skin, possibly aromatic or amide species, ammonium species similar to ammonium sulfate and possibly barium sulfate

SUMMARY

It is important to ensure that reliability stress chambers do not introduce foreign contaminants onto devices from previous runs. However, few of these systems are ever tested and many are not adequately or routinely cleaned between different tests or runs. The presence of contaminants on devices after environmental testing is rare. The authors have observed this to be an issue a handful of times in thirty years. The proper maintenance of chambers and the inspection and cleaning of samples prior to placement in the ovens is the primary preventative measure required to avoid these issues. The use of witness samples as describe herein helps to check the health of the chambers as well as the issuance of materials from the samples themselves.

However, on the few occasions that contamination has been observed it has been relatively serious and included: printed circuit boards exiting reliability testing covered in a white haze; or with visible contamination in the form of black spots. Likewise, oily residues on chips and thin films have been detected on electronic components. These contamination events ruined the devices and made the results of reliability testing either questionable or unusable. These devices required cleaning prior to additional testing in a new chamber. Occasionally, components are so contaminated that a new set of devices has to be re-analyzed. This can be an expensive issue and more importantly result in significant time delays in the product development process.

Accordingly, it is critical to understand the materials being deposited onto the samples under testing conditions. Once the composition and/or identity of the contaminants is determined, it is possible to generate a cleaning procedure and verify cleanliness using the witness technique. Herein we have described the development of a process to check the chamber output. The need for such testing is evident from the materials found on these samples. Notable contaminants, including Sn and organic acids, could alter the electrical behavior of the devices. The presence of any of these contaminants on optical or optoelectronic components could reduce light output, transmission or reflection even if it does not interfere with the electrical operation of the units.

The cost of a failed environmental tests is significant due to both time delays and money spent on the original and repeated tests. Thus, we strongly recommend that these chambers are tested routinely between runs to ensure they do not contaminate the samples in future runs.

EAG is implementing a comprehensive chamber witness program to enable cleanliness checks on environmental chambers. The program provides a witness wafer sampling kit as well as analytical techniques described in this paper. In addition, TOF-SIMS is available for the trace contamination detection.

REFERENCES

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