ANALYSIS OF A STRUCTURAL ADHESIVE DETACHMENT

CUSTOMER CASE STUDY

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INTRODUCTION

In microelectronics, there is sometimes a tendency to focus on the most visible parts: the circuitry, the soldering, the processing power for example. However, it would be a mistake to forget the behind-the-scenes actors! Here we are talking about adhesives, polymers, tapes, fibers, paints and other less central elements of the complex assemblies in the industry. To illustrate this principle, we are sharing a professional anecdote.

When a customer returns several thermostat control units because they detached on their own and are hanging by their electronic ribbon, an investigation must be conducted even if these units are still perfectly functional. The investigation shows that the electronics are not faulty, and suspicions naturally fall onto the adhesives.

It is therefore relevant to examine adhesives from the large family of acrylates, ubiquitous among instant adhesives. We will perform analysis with an infrared spectrometer (FTIR) to learn more about its properties and side chains, and by scanning electron microscopy (SEM) to observe the texture and presence of foreign bodies. We will thus track our main suspects.

- **Infrared spectroscopy** allows identification of the molecular bonds present in the sample, mainly organic bonds.
- **The electron microscope** allows observation of the sample at very diverse magnification scales, and identification of the atomic elements present (the heavier the element, the brighter it appears).

TO UNDERSTAND THE SITUATION, IT SEEMS NECESSARY TO PRESENT ALL THE PROTAGONISTS HERE:

- The customer: The company where the units detached.
- The requester: The company that sold and partially assembled the units, and mandated C2MI to investigate this detachment.
- The suppliers: The companies that provided a pre-assembled sub-part of the unit.

FAILURE DESCRIPTION

1. After a few weeks at the customer's site under normal environmental conditions, the screen detaches from its housing and remains suspended by its wiring.
2. The detachment systematically occurs between the ABS of the housing and the acrylate adhesive.
3. Screens from a certain supplier (supplier no. 1) were the only screens to detach in this manner.
4. More specifically, the requester had identified a series of screen-adhesive assembly batches as problematic, manufactured at the supplier during a specific period.

DEVICE DESCRIPTION

1. The requester receives the housings and screen-adhesive assemblies separately, supplied by different partners, according to identical technical specifications.
2. At the end of electronic assembly, the requester uses the second face of the adhesive tape to attach the screen to the housing.
3. The housing is made of ABS (acrylonitrile-butadiene-styrene), a thermoplastic polymer widely used for its robustness and impact resistance.
4. The adhesive is an acrylic foam, double-sided. It is applied by the supplier on the glass bordering the screen, then by the requester, attaching the second side to the ABS surface of the housing.

ANALYZED SAMPLES

- Sample 1: complete device detached at customer site (supplier no.1).
- Sample 2: complete device, assembled with screen-adhesive assembly from supplier no.1.
- Sample 3: complete device, assembled with screen-adhesive assembly from supplier no.2.
- Sample 4: screen-adhesive assembly (suppliers no.1 and no.2).

INVESTIGATION AXES

1. Housing surface analysis: visible anomalies that could explain detachment.
2. Adhesive surface inspection: presence of contaminants or interfacial films.
3. Adhesive characterization: comparison of chemical composition between batches.

We will examine two different situations, and discuss the following aspects:

1. The surface of the housings to which the adhesive is bonded, then detaches. Can we detect or see something that would explain this detachment?
2. The surface of the problematic adhesive. Is there a contaminant, a film, particles, something that could cause suboptimal adhesion by physically occupying the interface?
3. The nature of the adhesive itself. Is it the same adhesive on all screen-adhesive assemblies?

RESULTS AND OBSERVATIONS - SITUATION 1

COMPLETE DEVICE, DETACHED AT CUSTOMER SITE

1. SEM Imaging

Although we did not retain images of the housing surface, no notable difference was observed between the housings of the "good" and "bad" samples.

Many particles could be observed and identified (see Figure 1): gypsum, cardboard, hair... nothing unexpected for a sample that spent some time in a normal environment, attached to a normal wall.

More precisely, we could not determine whether the particles were partly responsible for the adhesion failure, or whether they were symptoms, adhering to the adhesive after it detached from the housing.

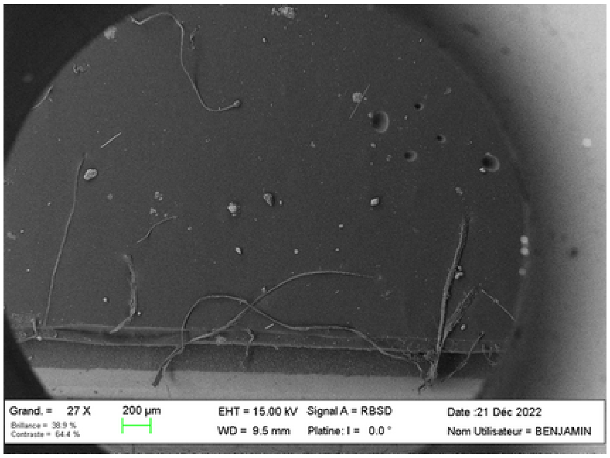

Figure 1: Adhesive detached at a customer site, sample 1 (SEM imaging).

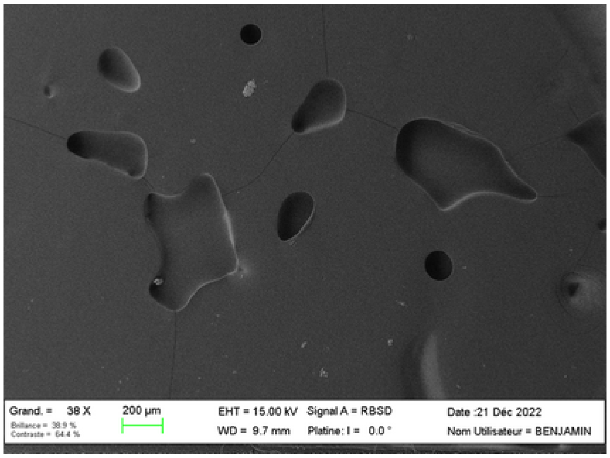

Figure 2: Adhesive detachment at requester site, sample 2 (SEM imaging).

Sample 2 also showed adhesion failure between the acrylate foam and the ABS housing, but was never sent to a customer, because the requester noticed the poor adhesion. It was slightly cleaner (see Figure 2) but showed a similar variety of particles: mineral dust, gypsum, cardboard and hair.

Samples 3 and 4 (next page) were kept at the manufacturer and show no adhesion problems. Furthermore, the acrylate foam shows more intense stretching structures (see Figure 3) after being detached in the laboratory. It is nevertheless possible that these structures resorbed or were flattened after their formation for the detached samples. At this stage, we cannot draw conclusions.

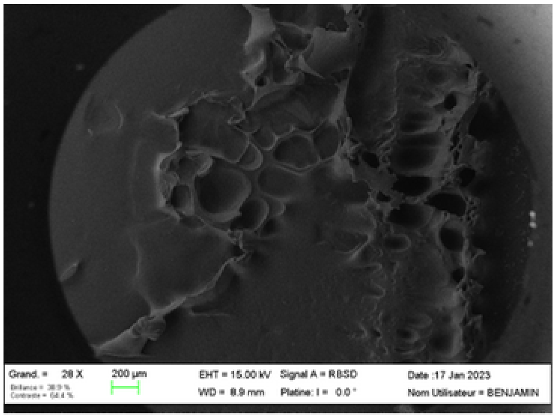

Figure 3: Sample 3, different supplier, no sign of detachment (SEM imaging).

What does SEM imaging tell us?

- No unexpected contaminants.
- The observed particles are rather omnipresent. Some of them might even have deposited on the sample in the laboratory.
- The stretching marks observed on sample 3, the "good sample," indicate better adhesion of this adhesive, assuming these marks do not resorb over time.

Having found no obvious cause, let's move to the next step: FTIR spectroscopy, to examine the adhesive composition.

Fourier Transform Infrared Spectroscopy (FTIR)

Adhesion is eminently sensitive to interface phenomena; FTIR is an imperfect tool for this part of the investigation, as the penetration depth (between < 1 µm and 10 µm approximately, depending on the technique used) may be too high and miss the finest details of surface composition. But FTIR can nevertheless be useful to communicate molecular information about the actual composition of the acrylic adhesive, and allow us to spot certain potential causes, such as composition differences, or signs of chemical alteration (oxidation, water absorption, heat damage, ...).

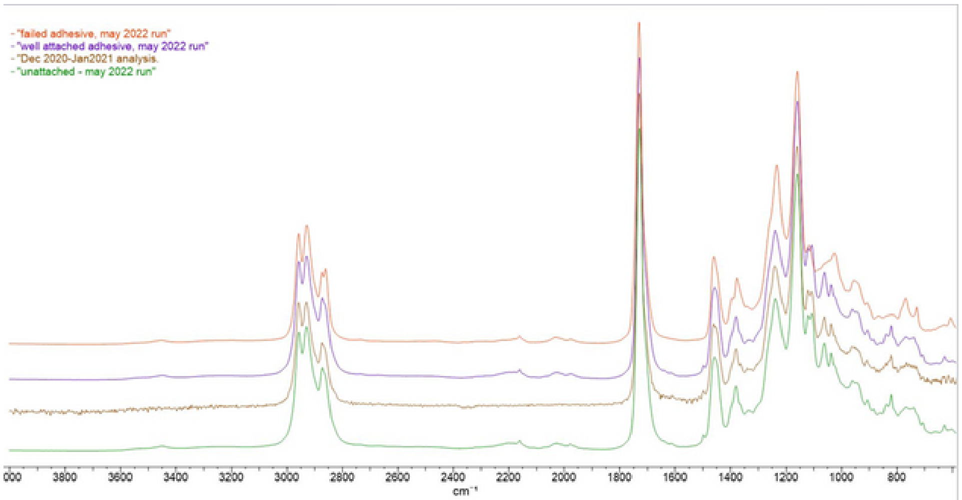

Figure 4: ATR-FTIR spectra of (from top to bottom): detached adhesive from sample 1 (orange), adhesive from sample 3 (violet), adhesive from a batch from the previous year (brown), adhesive from sample 4 (green).

Using our micro-ATR with Germanium, we collected spectra at the surface of various samples (see Figure 4). The general appearance of the spectra corresponds to what was expected: they are all acrylic polymers. However, slight differences exist between sample 1 and all the others, suggesting a probable difference in composition and/or relative abundance of side chains.

More specifically:

- By looking at the peaks around 2850-2880 cm-1, we observe a more marked separation in our problematic sample (see Figure 5).
- The peak around 1230 cm-1 is higher for our problematic sample and slightly shifted toward lower wavenumbers (see Figure 6).
- The peaks between 1150 cm-1 and 1000 cm-1 are very different for our problematic sample (see Figure 6).
- There is no peak at 820 cm-1, and two peaks at 770 and 730 cm-1 (see Figure 6).

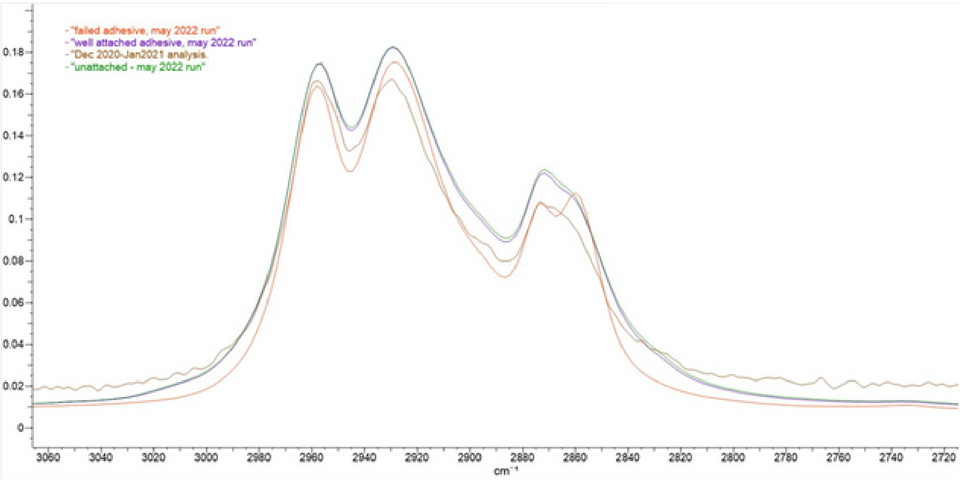

Figure 5: Hydrocarbon region. The spectra have been normalized to facilitate comparison. Detached adhesive from sample 1 (orange), adhesive from sample 3 (violet), adhesive from a batch from the previous year (brown), adhesive from sample 4 (green).

If we consider the peaks around 2860 cm-1 and the presence of two small peaks around 750 cm-1, this could indicate that our problematic acrylate has longer hydrocarbon side chains.

The absence of distinct peaks between 1150 and 1000 cm-1 in our problematic sample could indicate that the C-O bonds are less varied, so that the composition and/or proportion of side chains could be different, but this is more hypothesis than conclusion.

The other differences are more difficult to interpret, as acrylic polymers often have several types of side chains, each with their specific peaks, often close together, which makes any hypothesis risky without some other source of information.

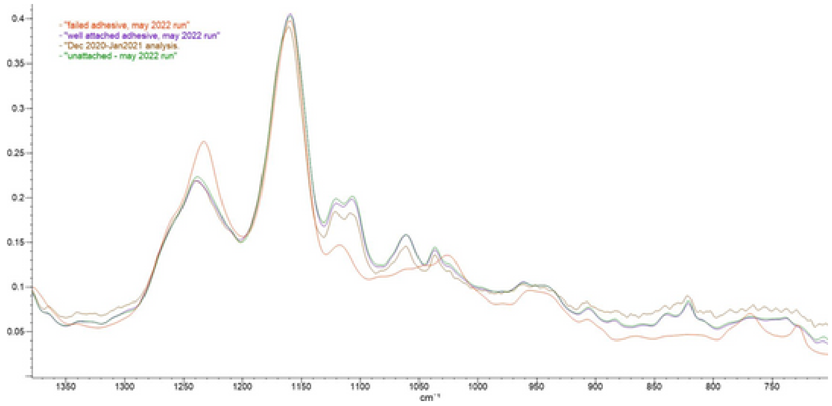

Figure 6: "Fingerprint" region. The spectra have been normalized to facilitate comparison. Detached adhesive from sample 1 (orange), adhesive from sample 3 (violet), adhesive from a batch from the previous year (brown), adhesive from sample 4 (green).

What does infrared spectroscopy tell us?

All adhesives with good adhesion, stable over time, are identical in terms of composition. The problematic adhesive from samples 1 and 2 appears to also be a polyacrylate, but with different side chains.

Other Considerations

Many other factors have been identified and discussed during this investigation, concerning the environmental conditions of the factory where the adhesive is attached to the housing, at the requester's facility.

- How much time elapsed between removal of the protective film and placement of the screen on the housing?
- What were the humidity and temperature conditions in the assembly line premises?
- Was there a change in cleaning products?
- COVID-19 was very much a concern at the time of screen-adhesive assembly; there must have been disinfectant somewhere?
- Was there a change in storage or shipping conditions for adhesive rolls or pre-bonded screens, on the supplier side?

All these questions could lead to one (or several!) cause of alteration of the adhesive properties of the acrylate, often too subtle to be detected by FTIR or SEM.

FIRST CONCLUSION

Unfortunately, there is no irrefutable proof. We cannot determine whether the particles were present before the screen was attached to the housing. The nature of the various particles is not unexpected, and there is no major difference or error in the composition of the housing and adhesive. There is a difference in the adhesive composition, and a difference in the stretching marks left on the adhesive after screen detachment between samples 2 and 3.

A reasonable hypothesis, and the provisional conclusion we reached based on the information available to us, was a difference in the mixing and/or composition of the side chains of the acrylate in the problematic adhesive, which could result in suboptimal adhesion to the housing surface (ABS) and, ultimately, detachment at the customer site.

RESULTS AND OBSERVATIONS - SITUATION 2

IMMEDIATE POOR ADHESION

Here, the problem was more easily spotted and investigated: the requester observed poor adhesion of housings with adhesive from one supplier (supplier "A"), but no problems with that from another supplier (supplier "B").

The requester suspected the protective film, as supplier A reported having removed the original protective film during assembly of the adhesive to the screen and installed another one, to cover the face of the adhesive that would be used at the requester's facility to attach the screen to the housing, while supplier B had not modified the original protective film.

1. Adhesive Composition

The adhesive itself is identical for both suppliers, their composition verified by ATR-FTIR, and by comparing the size of the different adhesive layers.

2. Protective Film Analysis

For the protective films, the situation was quite different. Verified by ATR-FTIR, supplier B's original protective film consisted of a PE (polyethylene) sheet covered with a thick white non-adhesive PDMS (polydimethylsiloxane) layer providing good coverage. The PDMS layer showed some imperfections, but nothing major (see Figure 7). Supplier A's second protective film was, let's say, of inferior quality. The PDMS layer is so thin that it causes the iridescent effect visible in Figure 7, right.

We again used our Germanium crystal for ATR-FTIR analysis, due to its very low penetration depth compared to other ATR materials (between 0.3 and 1.5 µm for Germanium crystals, depending on the sample).

Despite this low penetration depth, PDMS was not the main contributor to the FTIR spectra for supplier A's film.

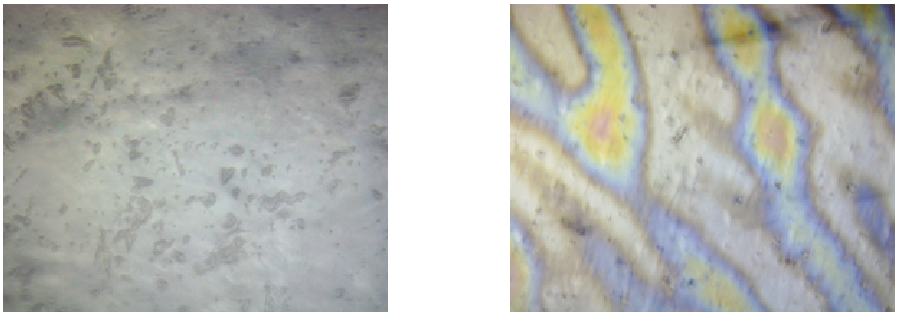

Figure 7: Inner face of protective films, on the adhesive side. Left: supplier B's original protective film, covered with a thick, relatively uniform PDMS layer. The PE core sheet is barely visible in the holes of the PDMS layer. Right: supplier A's second protective film. There is practically no PDMS, the PET core sheet is detectable everywhere.

As shown in Figure 8 below, the PDMS signal is generally weak, sometimes almost non-existent. The simplest way to track the presence of PDMS in this sample is to observe the Si-C peak around 795 cm-1; it is barely visible in the brown spectrum, indicating that it represents about 10% of the signal. Generally (with exceptions), a composition difference of less than 5% will be (very) difficult to detect by FTIR.

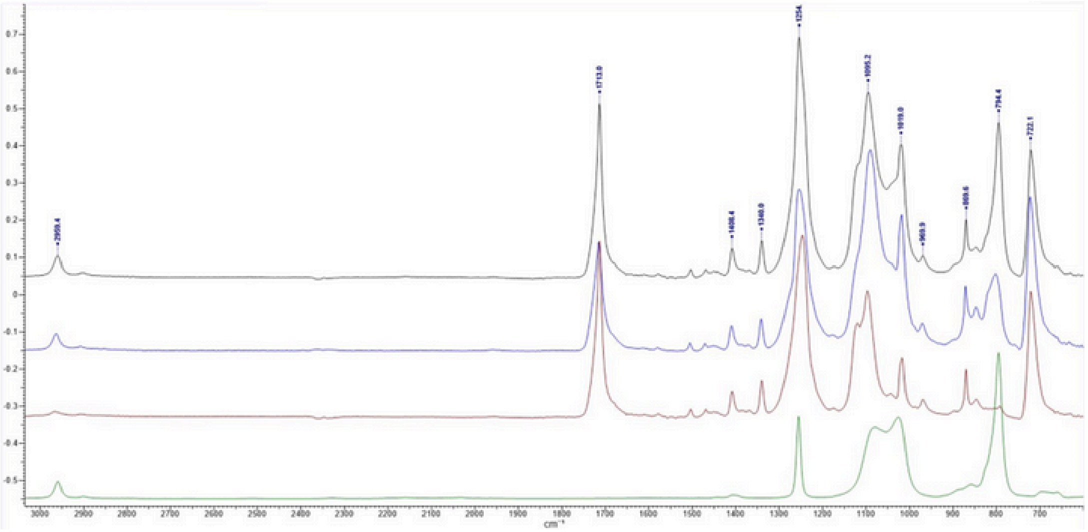

Figure 8: ATR-FTIR spectra of supplier A's protective film. Black: Reconstruction of a PDMS and PET mixture spectrum. Blue: spectrum acquired in the blue zone of Figure 7 (right). Brown: spectrum acquired in the brown zone of Figure 7 (right). Green: generic PDMS spectrum.

This second protective film will therefore not be very effective, as the PDMS layer, which is responsible for covering the acrylate with the least possible interactions, is very thin. We did not investigate whether there were actual holes in this layer, but we can already observe the large variance in this layer's thickness, so it's not impossible. Could the acrylate foam adhere to the PET (polyethylene terephthalate) core in places? It's possible.

Let's propose 2 hypotheses:

1. The adhesion properties of the acrylic adhesive depend on the chemistry and organization of its surface. Now, exposing the adhesive surface to ambient air, even briefly, then replacing a protective film will have the effect of reducing the final adhesion strength. Perhaps not by much, it must be acknowledged.
2. The affinity of acrylate for PDMS is very low, so adhesion between these two materials will be very weak, unlike PET, for which acrylate has greater affinity, as it is more polar than PDMS. If the acrylate adheres locally to the PET core sheet, removal of this protective film will pull on these points, and disturb the structure of the acrylate foam, and reduce the final adhesion strength.

GENERAL CONCLUSION

It is certain that removing the protective film long before the adhesive is attached to the housing is not good practice. Installing another protective film of lower quality is also not good practice.

As the requester indeed had adhesion problems, we proposed these 2 hypotheses, based on observations and analysis results of the protective films.

Here we are confronted with a simple adhesive, which led a customer to return perfectly functional but unusable devices. The problem is not serious, but rather annoying.

These two studies allow us to emphasize the fundamental importance of surface chemistry in adhesion science, and its sensitivity to numerous factors, some more obvious than others: temperature and humidity, presence of volatile organic compounds (VOCs), interaction between matrix composition and surface adhesion among others.

Even with many questions remaining unanswered, the requester now has enough concrete elements, assumptions and informed hypotheses to engage in serious dialogue with their suppliers.

By combining cutting-edge analytical equipment such as SEM and FTIR with our team's expertise, C2MI's chemical characterization laboratory plays a decisive role in optimizing microelectronic products. These analyses not only help prevent costly failures but also accelerate innovation by providing companies with reliable and actionable data. By choosing our laboratory, industry players ensure better control of the quality of their materials and processes.

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