

# The value of material characterization in product development

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TECHNIEKHUYS  
VELDHOVEN

***PLOT CONFERENTIE***  
***TOMORROW'S RELIABILITY***



## Local partner, global reach

>200 companies served yearly

- Reliability testing
- 35 analytical techniques
- Experts in material science & testing
- Consultancy
- Always there to help you



High Tech Campus  
The Netherlands

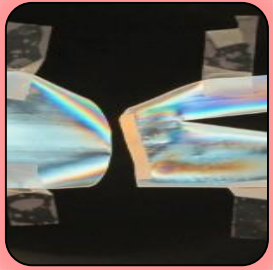
**For general questions related  
to Philips Innovation labs  
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# Agenda

- Material characterization tools
- Examples material characterization in product development  
SEM-EDX, GC-MS, TOF-SIMS, FT-IR,  $\mu$ XRF
- Strenght of combining reliability testing & material characterization

# Overview material characterization tools



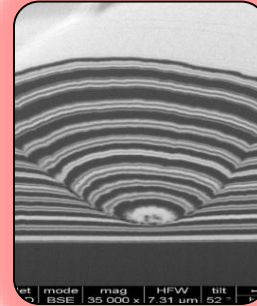
*Ellipsometry,  
ToF-SIMS, XPS,  
XRD, Auger*

**Surface and thin-film**



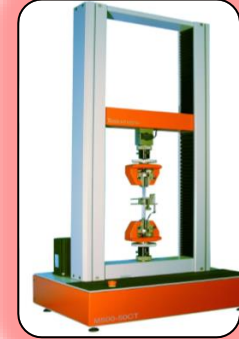
*Atomic Spectroscopy,  
Elemental Analysis  
FTIR, Raman, GC, LC,  
NMR, XRF*

**Compositional analysis**



*SEM, TEM, FIB, XRT,  
AFM, profilometry,  
thermography*

**Advanced imaging**



*Particle size,  
BET, thermal  
analysis,  
rheology,  
mechanical  
testing*

**Physical characterization**



*Hot / Cold Storage,  
Damp Heat,  
Temperature Shock,  
Slow / Fast Cycling,  
Vacuum, UV  
Simulation*

**Environmental testing**



*Lifetime setup,  
Monitor (I/V/R vs  
time), Automated  
Power Supplies,  
Event Detection,  
PTC*

**Insitu Testing**



*HALT,  
MEOST*

**Reliability**

Some examples of material characterization in product development

# Interconnect thickness and composition control – electrical resistance increase

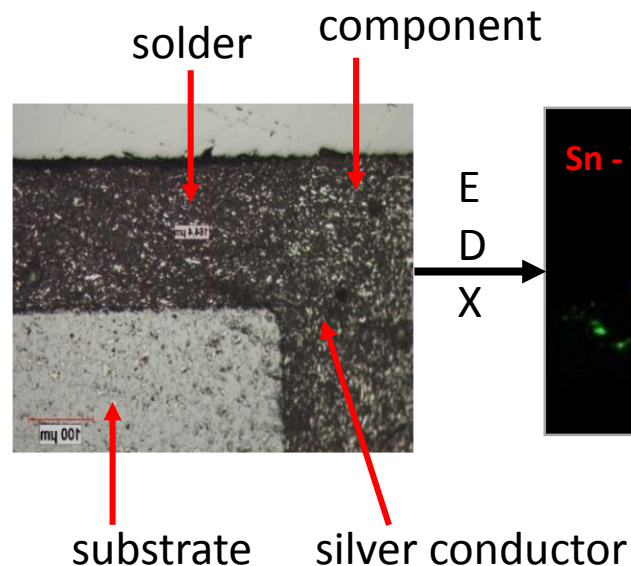
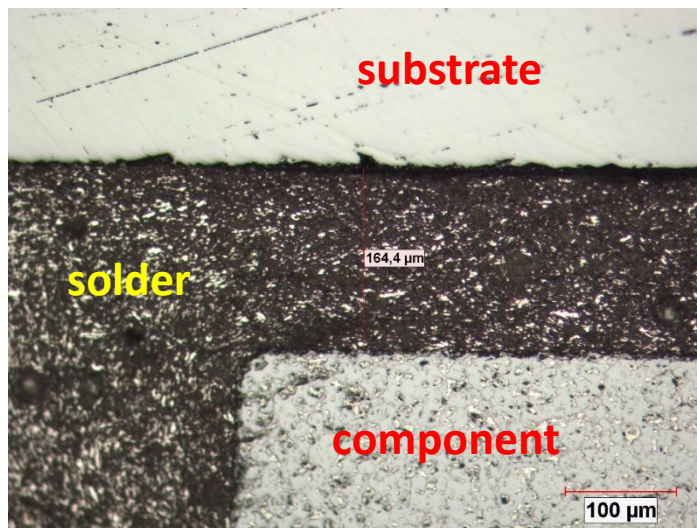
SEM-EDX (Scanning Electron Microscope)

- Compositional info (identification of elements & atomic %)
- Geometrical info (dimensions,..)
- Visual info (crack, voids, ...)
- Magnification >100.000x

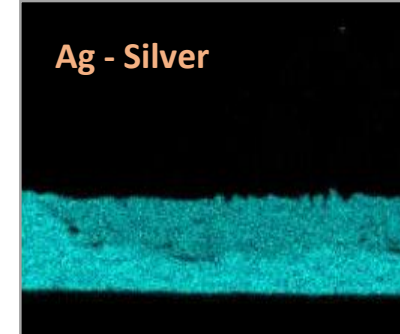
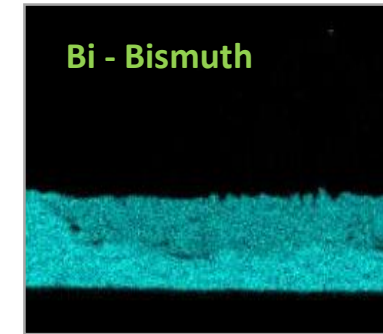
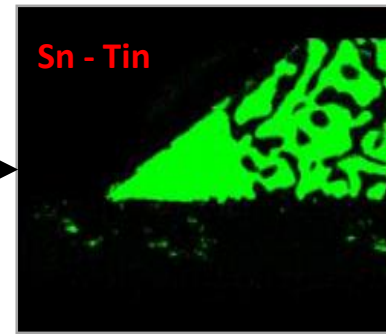
**Backscattered electrons** produce images with contrast that carries information on the differences in atomic mass

**Secondary electrons** give topographic information

**X-rays** are a “fingerprint” of each element; used to identify the elements that exist in a sample



E  
D  
X

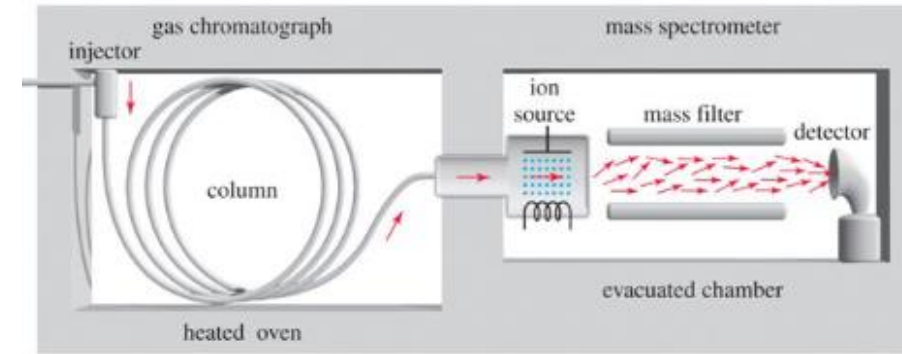


# Polymer outgassing – unwanted deposition inside product

GC-MS

Gas Chromatography - Mass Spectrometry can separate, **identify** and **quantify** complex mixtures of chemicals

Degassed components during glue curing or product life can be measured



Outgassing of polymer at 25°C

Very low outgassing glue at 25°C, only traces of silicon containing compounds (siloxanes) were detected.

Outgassing of polymer at 100°C

The outgassing of the glue at 100°C:

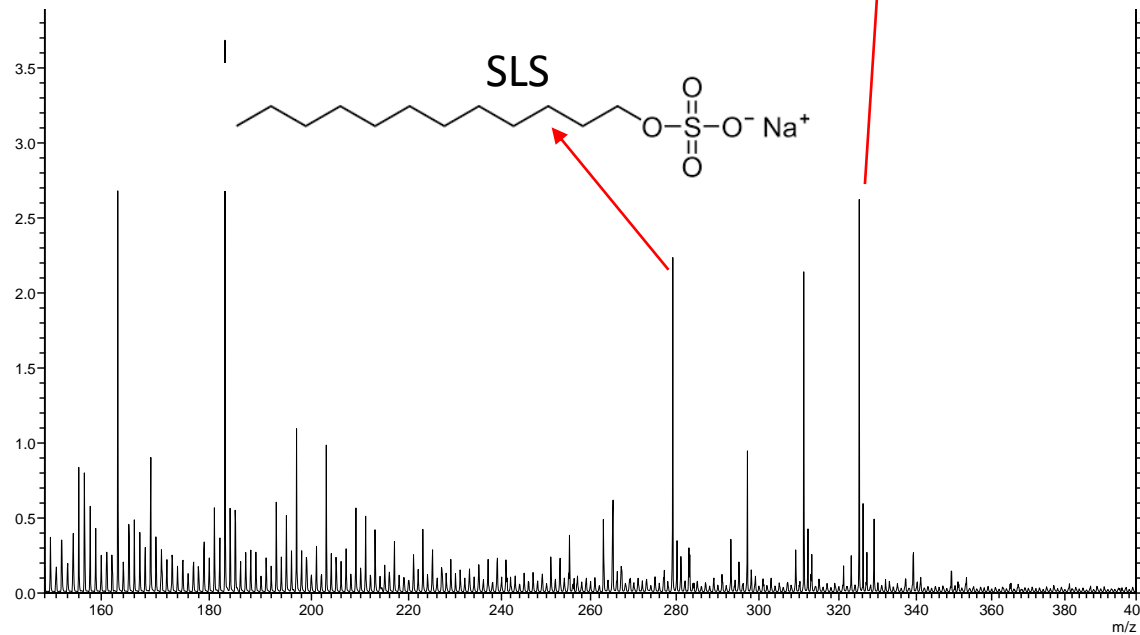
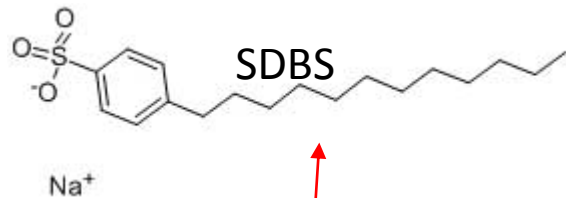
- solvents (e.g. 2-(2-ethoxyethoxy)ethanol)
- triethylphosphate (a flame retardant)

# Surface contamination – adhesion problem even after cleaning

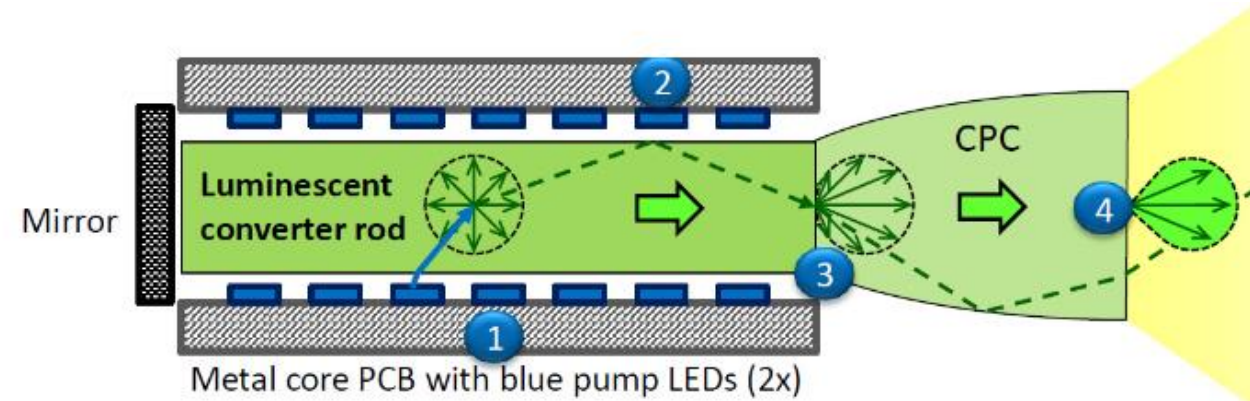
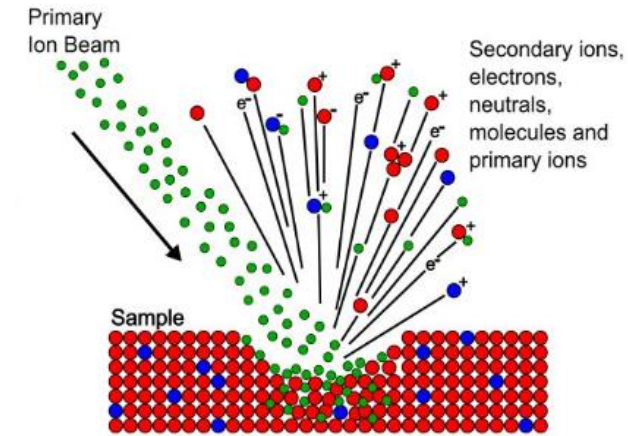
## TOF-SIMS

TOF-SIMS provides molecular information from surfaces of solid materials

- Most sensitive surface analysis technique (ppm/ppb)
- Organic & inorganic contamination



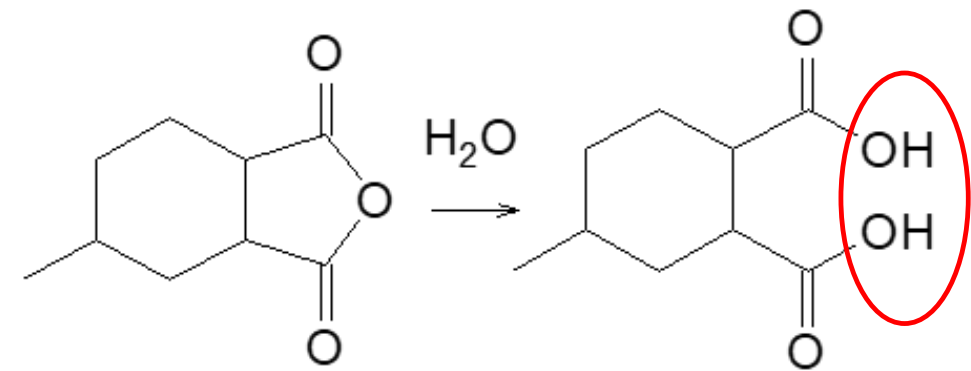
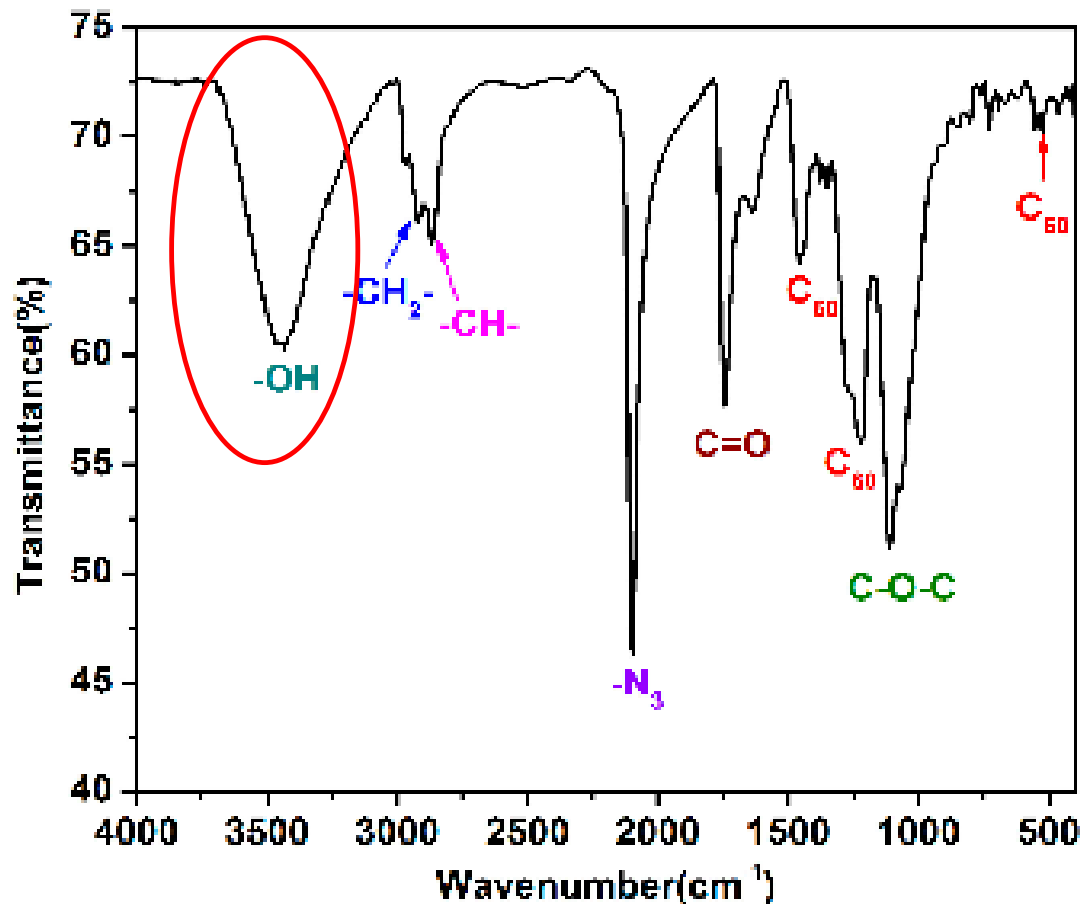
SDBS & SLS are surfactants present on cleaning wipes



# FTIR

## Fourier Transformed Infra Red spectroscopy

**IR spectroscopy** is the spectroscopic technique which uses the [Infrared light](#) and studies its [interaction with the molecules](#). The spectra generated can give a lot of information about the molecule, especially about the functional groups associated.



*Epoxy degradation reaction*

# Black electrical insulator layer – degradation @ high%RH

FT-IR

**Functional parameter:** Break Down Voltage (kV/mm)

**Disadvantage :** BDV test is a destructive test (large nr of samples needed / expensive)



It was discovered that the FTIR peak height at 1500cm<sup>-1</sup> correlates to decreasing break down voltage

## Sensitivity of %RH investigation with a damp-heat storage test two levels

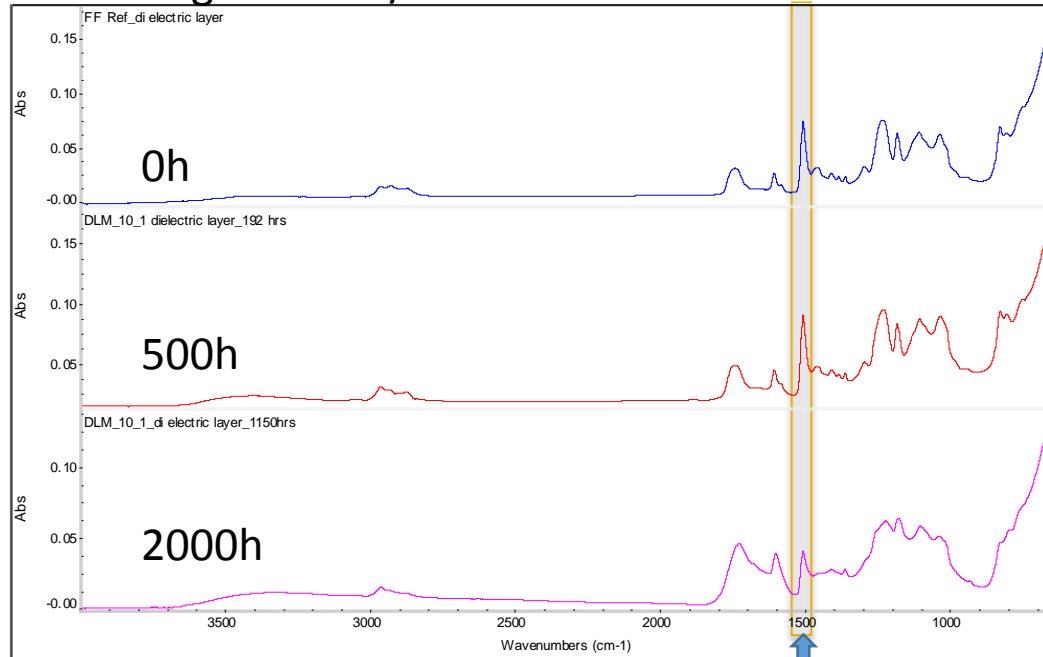
**Test condition A:** Storage at 85°C/15%RH

**Test condition B:** Storage at 85°C/85%RH

# Black electrical insulator layer – degradation @ high %RH

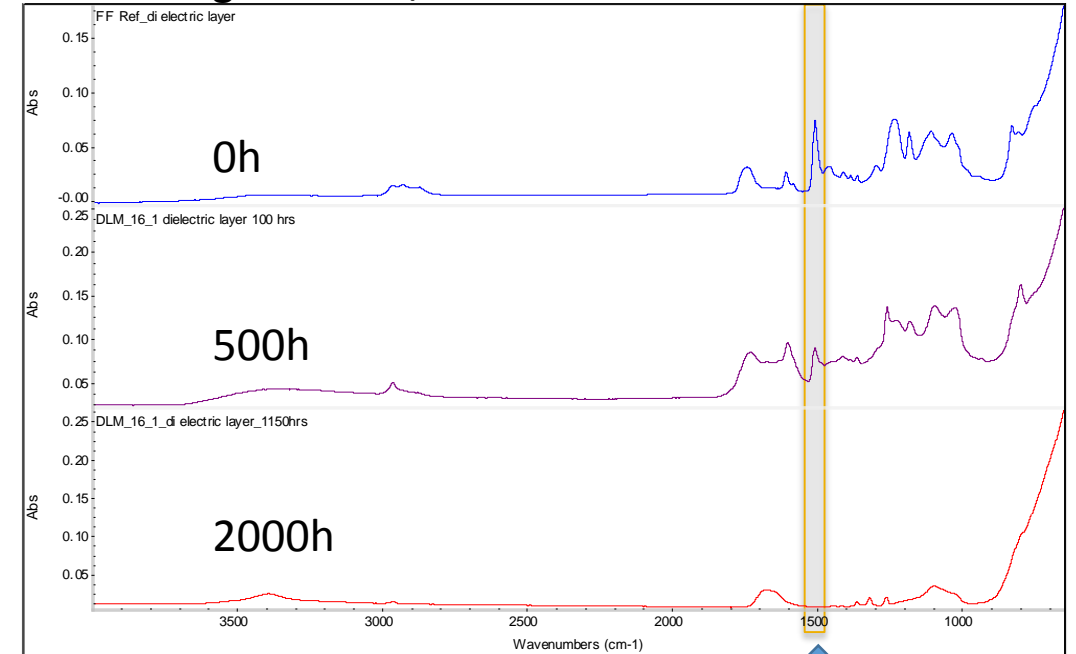
FT-IR

A: Storage at 85°C/15%RH



1500cm-1

B: Storage at 85°C/85%RH



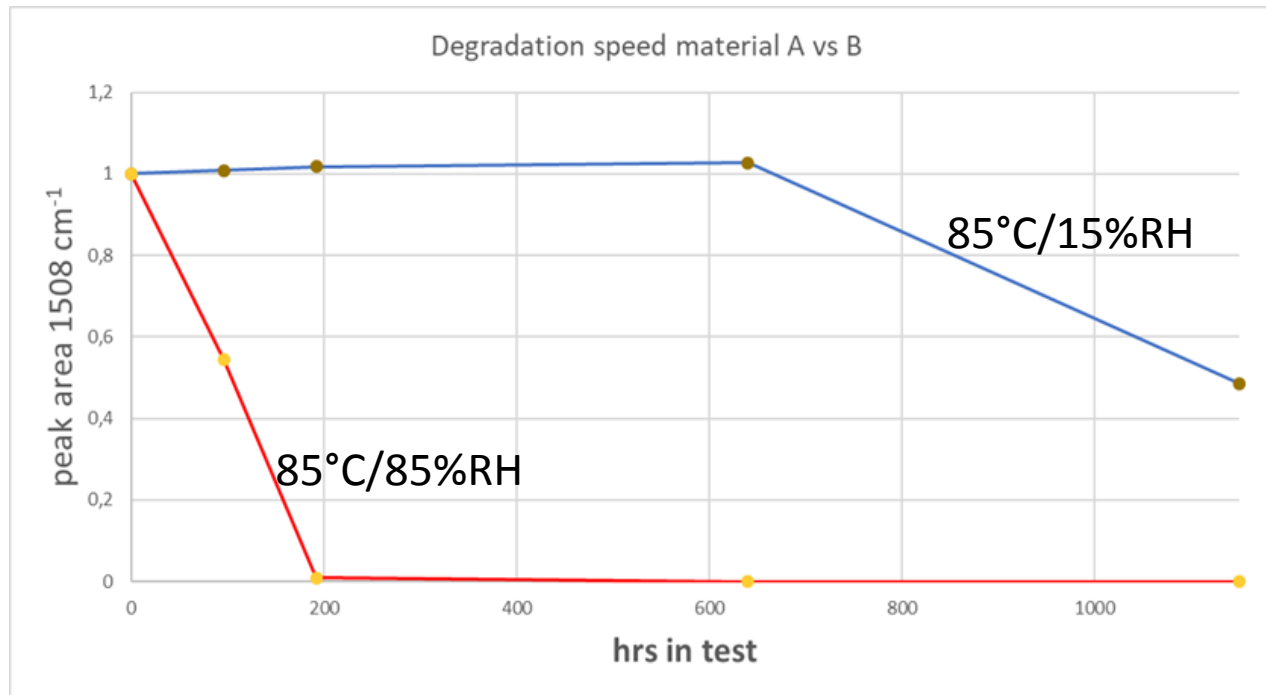
1500cm-1

- Peak height at 1500cm-1 decreases in time (= correlated to decreasing break down voltage)

# Black electrical insulator layer – degradation @ high %RH

FT-IR

Plotting peak height 1508cm<sup>-1</sup> against test duration



- FTIR / Raman can make degradation visible in an early stage
- Molecular understanding of chemistry leading to failure
- Input for modelling the application window / determination of acceleration factors

# Micro-XRF

## X-ray fluorescence

- Elemental analysis technique
- No / minimal sample preparation needed
- Non destructive



|         |          |          |          |           |           |           |           |           |           |           |           |           |            |            |            |            |            |            |
|---------|----------|----------|----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|------------|------------|------------|------------|------------|------------|
| Group→  | 1        | 2        | 3        | 4         | 5         | 6         | 7         | 8         | 9         | 10        | 11        | 12        | 13         | 14         | 15         | 16         | 17         | 18         |
| ↓Period |          |          |          |           |           |           |           |           |           |           |           |           |            |            |            |            |            |            |
| 1       | 1<br>H   |          |          |           |           |           |           |           |           |           |           |           |            |            |            |            |            | 2<br>He    |
| 2       | 3<br>Li  | 4<br>Be  |          |           |           |           |           |           |           |           |           |           | 5<br>B     | 6<br>C     | 7<br>N     | 8<br>O     | 9<br>F     | 10<br>Ne   |
| 3       | 11<br>Na | 12<br>Mg |          |           |           |           |           |           |           |           |           |           | 13<br>Al   | 14<br>Si   | 15<br>P    | 16<br>S    | 17<br>Cl   | 18<br>Ar   |
| 4       | 19<br>K  | 20<br>Ca | 21<br>Sc | 22<br>Ti  | 23<br>V   | 24<br>Cr  | 25<br>Mn  | 26<br>Fe  | 27<br>Co  | 28<br>Ni  | 29<br>Cu  | 30<br>Zn  | 31<br>Ga   | 32<br>Ge   | 33<br>As   | 34<br>Se   | 35<br>Br   | 36<br>Kr   |
| 5       | 37<br>Rb | 38<br>Sr | 39<br>Y  | 40<br>Zr  | 41<br>Nb  | 42<br>Mo  | 43<br>Tc  | 44<br>Ru  | 45<br>Rh  | 46<br>Pd  | 47<br>Ag  | 48<br>Cd  | 49<br>In   | 50<br>Sn   | 51<br>Sb   | 52<br>Te   | 53<br>I    | 54<br>Xe   |
| 6       | 55<br>Cs | 56<br>Ba |          | 72<br>Hf  | 73<br>Ta  | 74<br>W   | 75<br>Re  | 76<br>Os  | 77<br>Ir  | 78<br>Pt  | 79<br>Au  | 80<br>Hg  | 81<br>Tl   | 82<br>Pb   | 83<br>Bi   | 84<br>Po   | 85<br>At   | 86<br>Rn   |
| 7       | 87<br>Fr | 88<br>Ra |          | 104<br>Rf | 105<br>Db | 106<br>Sg | 107<br>Bh | 108<br>Hs | 109<br>Mt | 110<br>Ds | 111<br>Rg | 112<br>Cn | 113<br>Uut | 114<br>Uuq | 115<br>Uup | 116<br>Uuh | 117<br>Uus | 118<br>Uuo |
|         |          |          |          | 57<br>La  | 58<br>Ce  | 59<br>Pr  | 60<br>Nd  | 61<br>Pm  | 62<br>Sm  | 63<br>Eu  | 64<br>Gd  | 65<br>Tb  | 66<br>Dy   | 67<br>Ho   | 68<br>Er   | 69<br>Tm   | 70<br>Yb   | 71<br>Lu   |
|         |          |          |          | 89<br>Ac  | 90<br>Th  | 91<br>Pa  | 92<br>U   | 93<br>Np  | 94<br>Pu  | 95<br>Am  | 96<br>Cm  | 97<br>Bk  | 98<br>Cf   | 99<br>Es   | 100<br>Fm  | 101<br>Md  | 102<br>No  | 103<br>Lr  |

In air

Only in vacuum

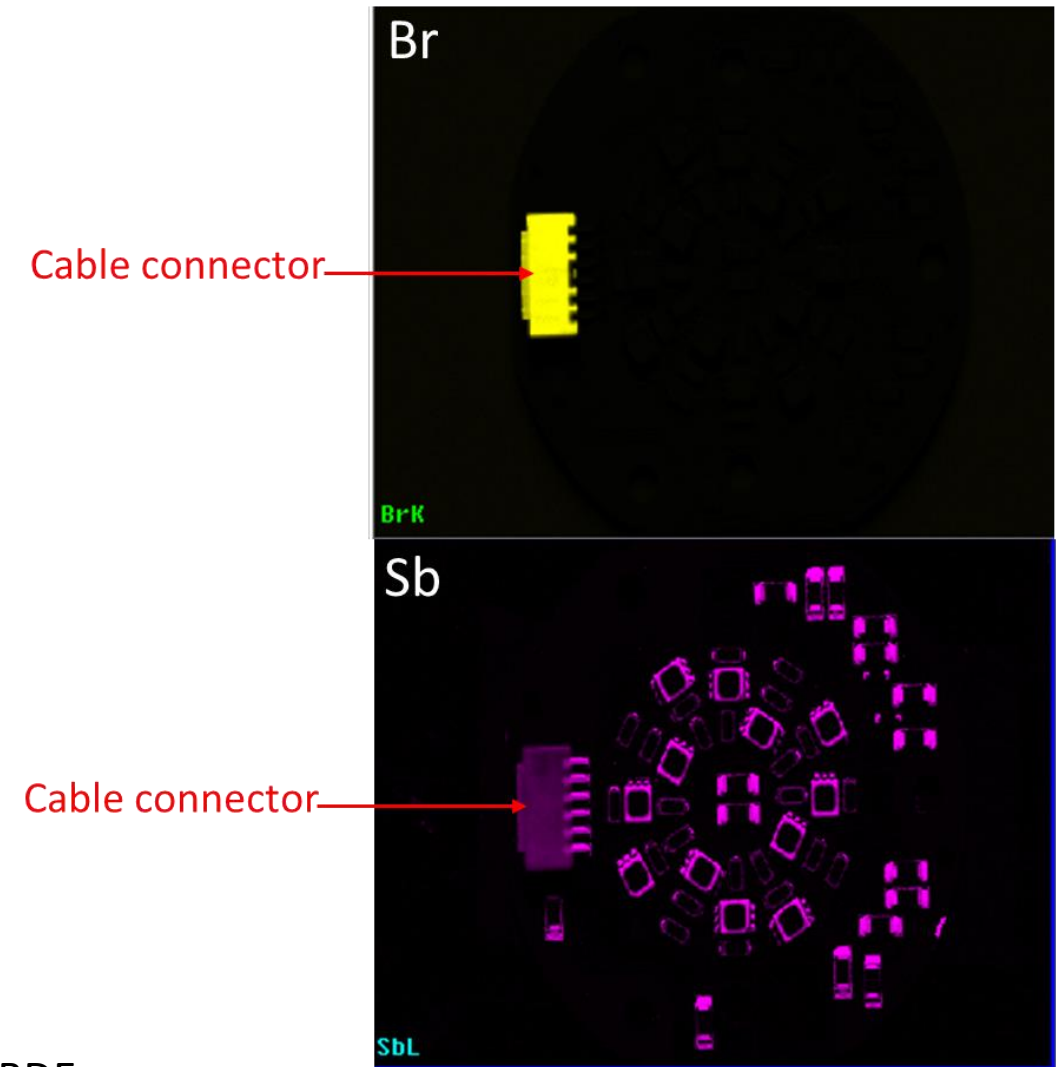
# Restriction of Hazardous Substances (RoHS) – Check sourced BOM parts

## Micro-XRF

The *Restriction of Hazardous Substances Directive* restricts the use of 6 hazardous materials in electronic equipment

| RoHS restricted substances           | Limits   |
|--------------------------------------|----------|
| Lead (Pb)                            | 1000 ppm |
| Mercury (Hg)                         | 1000 ppm |
| Cadmium (Cd)                         | 100 ppm  |
| Hexavalent chromium (Cr6+)           | 1000 ppm |
| Polybrominated biphenyl (PBB)        | 1000 ppm |
| Polybrominated diphenyl ether (PBDE) | 1000 ppm |

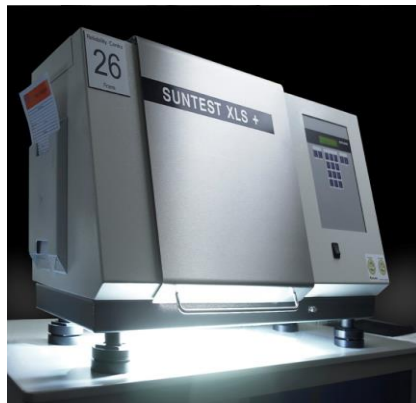
PBB and PBDE are flame retardants and contain 50-85w% Br.  
Br in combination with Sb is an indication for the presence of PBB or PBDE



PCB mapping of RoHS elements (Br and Sb)

# Reliability tests & material characterization

- Feedback loop to material characterization enables identification of failure mode & to follow the degradation process in time (*determination of acceleration factor using multi level tests*)
- Prediction model for degradation speed



- Damp-heat test
- Temperature shock test
- Temperature cycle test
- UV / light radiation
- HALT
- Salt-mist test
- Humidity-freeze test
- IP testing
- Chemical resistance test

**operational & non operational**

# The value of material characterization in product development

## **Lower risk for:**

- Delay in market introduction / product release
- Product quality & reliability issues
- Last minute BOM changes
- Cost uplift
- Supplier “issues”
- .....

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