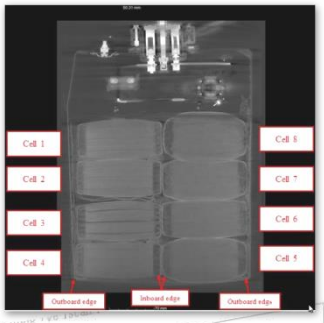
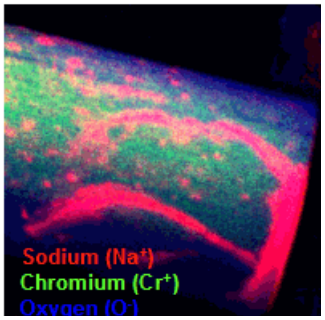
LPD Lab Services





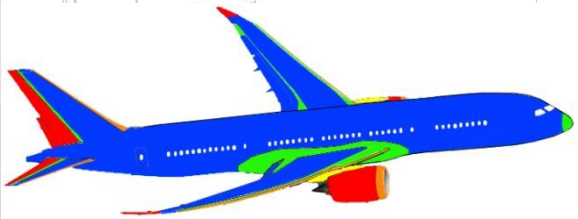
RAPRA
Rubber & Plastics Research Association
Preferred Provider

Industrial Problem Solving and Failure Investigation of Modern Materials

Dr Simon Romani – Technical Director

Aerospace / Advanced Engineering Show 2016 (NEC) - 2nd November 2016

RAPRA Session Invited Speaker



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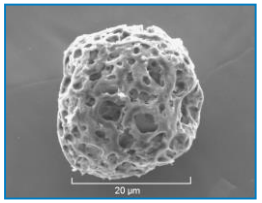
LPD Lab Services

Who are LPD Lab Services?

Filter Failure - Pollen Grain



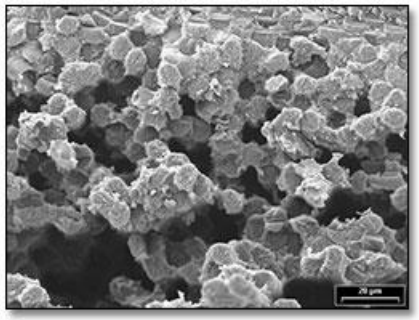
- LPD Labs grew from a buyout of the analytical activities of LG-Philips Displays and is still situated on the ex-Philips site near Blackburn.

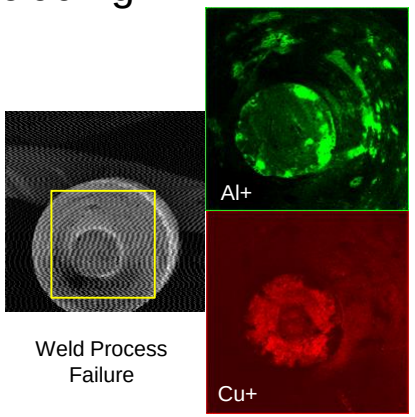
Hydrogen-reduction furnace

- Currently 10 technical staff, including:
  - Industrial chemists
  - Physicists
  - Materials scientists
  - Engineers

- Extensive manufacturing knowledge across diverse materials systems & processes, including:
  - Electronics & Semiconductor
  - Metals
  - Ceramics
  - Polymers
  - Composites

CFRP Fracture Surface





Weld Process Failure

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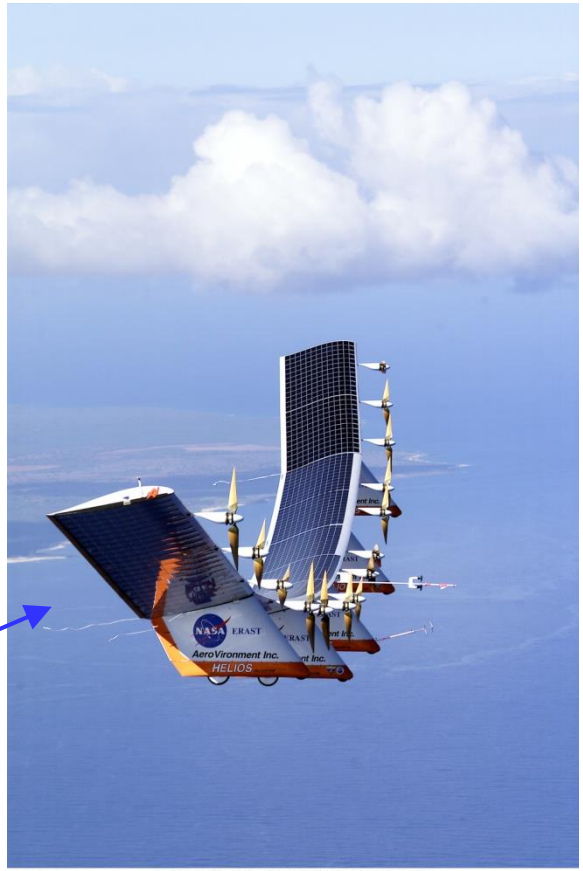
Growing Use of Polymers & Composites

Use of composites & polymers:

- NASA & Military Prototypes – early 70's
- Commercial (mainly non-structural) – mid 80's
  - A310-300 Fin, Rudder Elevators
  - A320 flaps, ailerons (late 80's)
- Commercial (selected air frame & more flight surfaces) – came in through 90's
  - B777-200 tail & floor beams
  - A330/340 Fuel Tank
- Wide spread commercial ~ 2005 onwards..
  - B787 CFRP wing, tail, fuselage
  - A380 Fuselage, Wing ribs & Centre-box

Lightweighting for solar powered Wing on NASA's Helios, very high bending wing back in 2001.

Today similar flexing is commonly seen in commercial aircraft: Airbus A350 wing excursion ~5.2m from taxi to flight...



NASA Dryden Flight Research Center Photo Collection
http://www.dfrc.nasa.gov/gallery/photo/index.html
NASA Photo: ED01-0209-7 Date: July 14, 2001 Photo by: Nick Galante/PMRF
The Helios Prototype flying wing is shown near the Hawaiian island of Niihau during its first test flight on solar power from the U.S. Navy's Pacific Missile Range Facility.

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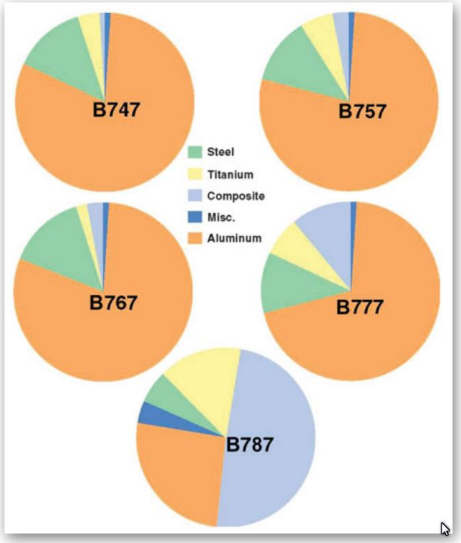
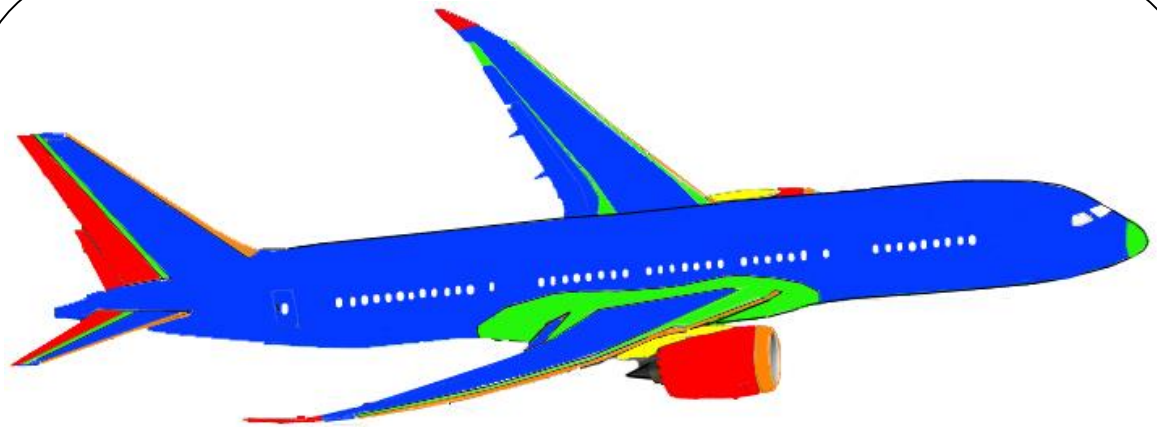
Growing Use of Polymers & Composites: Boeing



{Source: Boyer et al, MRS Bulletin, ,40(12), 2015, pp 1055-1066, with permission from author}



Boeing 787:

50% Composites

- Carbon Laminate
- Carbon Sandwich
- Fibreglass
- Aluminium
- Steel/Titanium/Aluminium

...compare with 25% for Airbus A380 and 53% on latest A350

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Polymer Degradation & Failure

Weak Bonding/ Delamination {SIMS, XPS}...

Stabiliser Issues... {GC-MS, HPLC, SIMS, XPS}...

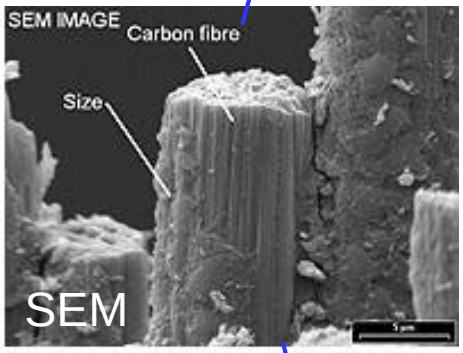
Hydrolysis...

Fibre Reinforcement Issues
- Type, size, orientation, volume fraction {TGA}...

Leaching... {ICP-MS, GC-MS, SIMS, XPS}...

Oxidation... {SIMS, XPS}...

Free radical attack (e.g. UV)



Carbon Fibre Composite Fracture Surface

Embrittlement (Halides)... {IC, SIMS, XPS}...

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Delamination in Composites

Delamination:

- Incomplete Resin Curing? {FTIR, GCMS, TGA, DSC}
- Weak boundary layers – Residual release agents?, interfacial or particle contamination? {XPS, FTIR, SEM}
- Poor surface treatments? (e.g. Flame/Corona) {FTIR, XPS, SIMS}
- Coatings cured too fast during application or mix wrong? {FTIR, GC-MS, HPLC}
- Poor cleaning? {FTIR, XPS, SIMS}
- Water Ingress? {FTIR, XPS}
- Corrosion Issues? {EDX, XPS, SIMS}

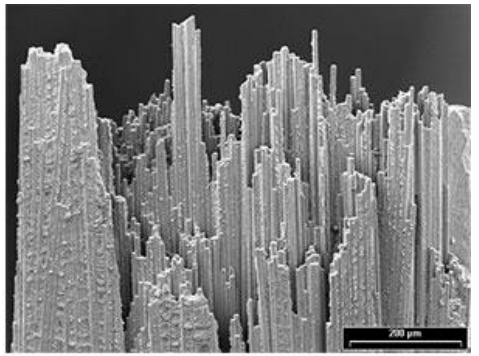

Figure 1 - Carbon Fibre Fracture Surface

e.g. SIMS can show plasticisers segregating to surface (shown here..), or residual mould release agents, etc..

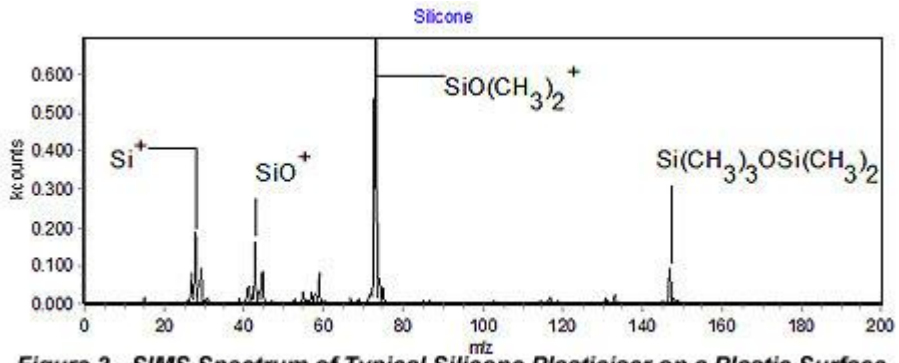

Figure 2 - SIMS Spectrum of Typical Silicone Plasticiser on a Plastic Surface

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Approaching a Failure Analysis...

Failure in new materials requires rapid analysis.....

No matter what the issue, similar approach:

- What does the customer want/request?
- What does the customer actually need?
  - Is sufficient information available (disclosure)?
  - What are the priorities?
  - What techniques are required?
  - What preparation is needed?
  - Is the analysis economical (cost)?

On to a relevant example.....

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AA587 (A300 series) - Crashed minutes after leaving JFK in November 2001...

- "Customer's" Priorities (NTSB, FBI & Airbus):
  - Terrorist Attack? (2 months after 9/11)
  - Structural Failure?
  - Pilot Error?
  - Design Flaws?

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AA587 (A300 series) - Crashed minutes after leaving JFK in November 2001...

- "Customer's" Priorities (NTSB, FBI & Airbus):
  - Terrorist Attack? (2 months after 9/11) → Explosive residues? Swabs collected at crash site in Queens, NY, before boxing... {FTIR/GC-MS, HPLC} – None Found...
  - Structural Failure?
  - Pilot Error?
  - Design Flaw?

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Failure Analysis: Air Crash Example...

AA587 (A300 series) - Crashed minutes after leaving JFK in November 2001...

- "Customer's" Priorities (NTSB, FBI & Airbus):
  - Terrorist Attack? (2 months after 9/11)
  - Structural Failure? → Flight recorder: rudder issues & early loss of stabiliser (found miles from crash site) pointed to possible failure of composite material... Delamination was noted in build notes, requiring repair..
  - Pilot Error?
  - Design Flaw?

{image from NTSB public report}

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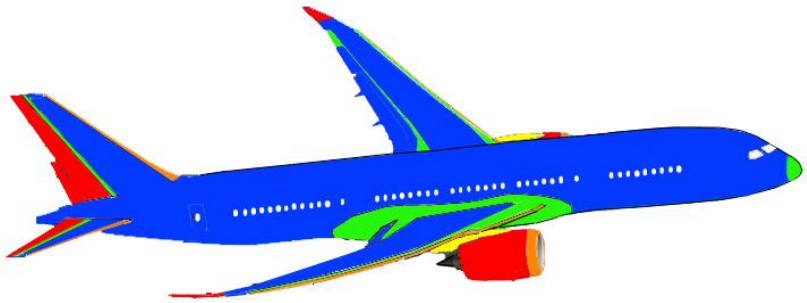
Failure Analysis: Air Crash Example...



Boeing 787:

50% Composites

- Carbon Laminate
- Carbon Sandwich
- Fibreglass
- Aluminium
- Steel/Titanium/Aluminium

Not just Airbus worried about possibility of composites failure!!....

B787 under construction and due for roll-out a few years later...

{eventually rolled-out July 2007...}

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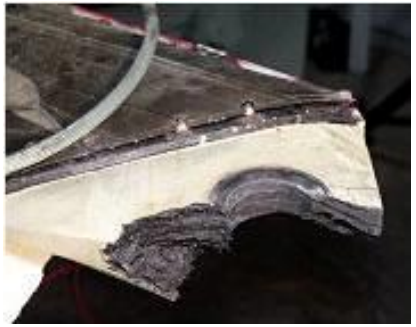
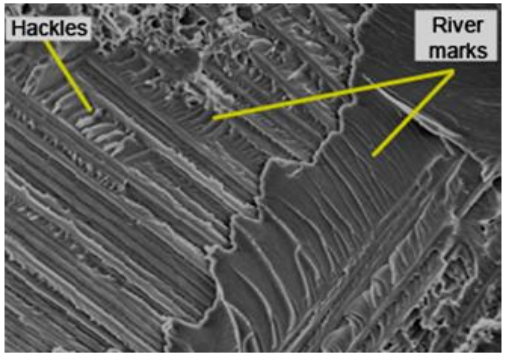
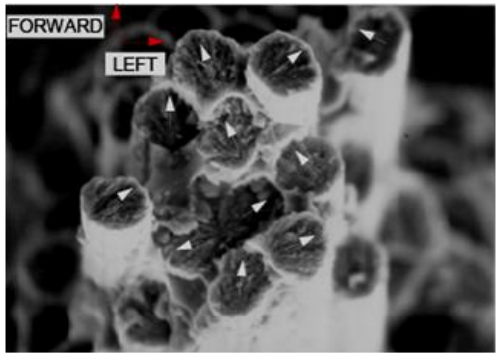


Fractured strut on AA587 stabiliser





Fracture surfaces
{images from NTSB public report}...

SEM & XSEM of fracture surfaces of 3 failed fittings and nearby lamination (100 layer GFRP composite) – No pre-existing faults found (fatigue, etc..)...

Appeared to be over-stress failure...

....Could this be a design flaw?
....Composite not strong enough?

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- "Customer's" Priorities (NTSB, FBI & Airbus):
  - Terrorist Attack? (2 months after 9/11)
  - Structural Failure?
  - Pilot Error?
  - Design Flaw?

Composite strong enough?

Tensile testing at Airbus showed fittings good to 200,000 lbs load, design was to 100,000 lbs...

Tensile test (smaller scale at LPD!...)

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  - Structural Failure?
  - Pilot Error?
  - Design Flaw?

Large rudder excursions of +/- 11° (full-lock positions), many times over just a few seconds.

NASA Simulation showed this could generate stabiliser loads above 200,000 lbs...
Exceeding maximum loading for fracture.

Eventual root-cause: Error in pilot training highlighted... (unrealistic flight scenario, leading to gross over-reaction if ever used in normal flight situations...)

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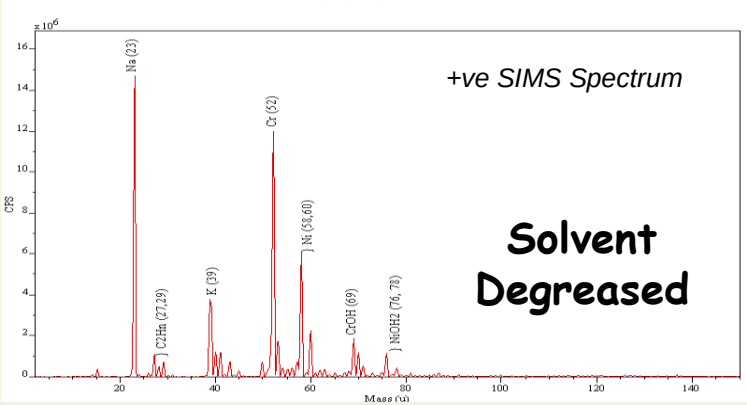
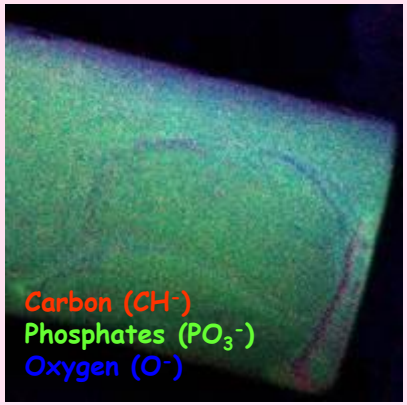
Issues with Degreasing Revealed by SIMS

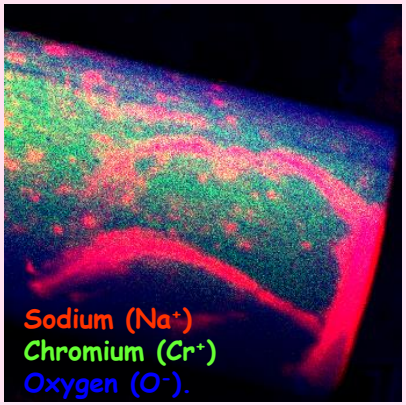
Industry under pressure to replace solvent–based cleaning/degreasing processes with aqueous-based processes (Environmental and H&S reasons).



- Removed hydrocarbon
- Underlying Ni and Cr exposed.
- Some Na and K visible.

➔ Clean surface





Aqueous Degreased (2.5mm FoV)

- Most of hydrocarbon removed
- Underlying Ni and Cr exposed
- High levels of Na, Si, K, Ca, Cl, Phosphates, Silicates and Nitrates from the aqueous degreasing solution.

➢The solvent process is a better cleaning process.
➢Aqueous process leaves more remnants behind, causing functional problems.

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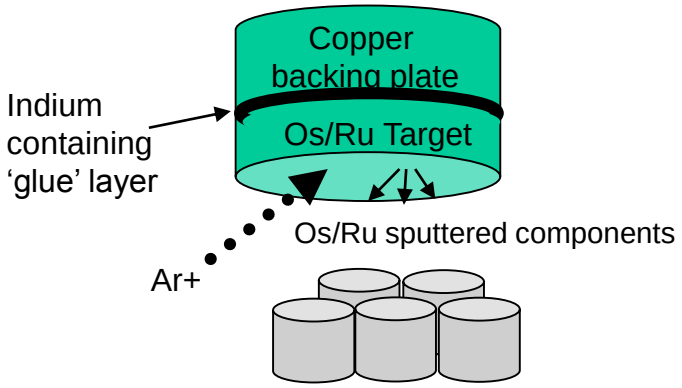
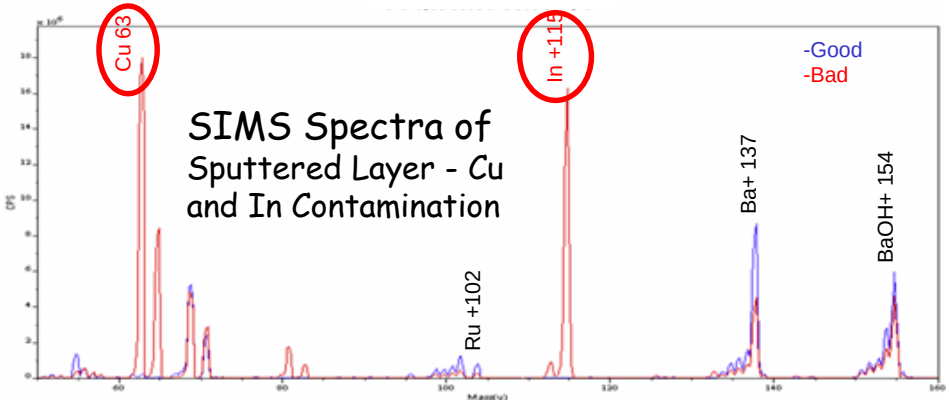
Sputtering Target End of Life





Cathode emission performance tests & production data showed a manufacturing Quality change....

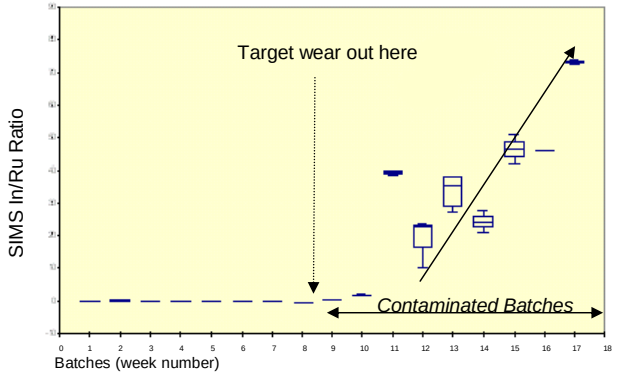
SIMS → Root cause analysis
SIMS → Problem Containment

Cu or In on Pellet surface reduced cathode emission.

Undetected software glitch had reset the target counter life so target worn through to backing plate.

SIMS sensitivity and sample rapid throughput allowed quarantine of finished & unfinished batches across production time frame and to release rest of production.

In/Ru



Production Time / Batch

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Summary & Final Comments

Effective Problem Solving Needs…

RAPRA Rubber & Plastics Research Association Preferred Provider

- True partnership between customer and analytical staff.
- Sufficient disclosure
  - Analyst can also then act as extra R&D manpower for customer
  - Can advise on remedial actions for manufacturing.
- Experienced analytical staff that recognise failure mechanisms.
- A combination of multiple techniques, applied appropriately.
- Adaptable approach and ability to react fast.

Any Questions?

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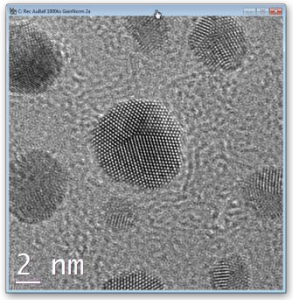
More about LPD.... Summary of Techniques Available

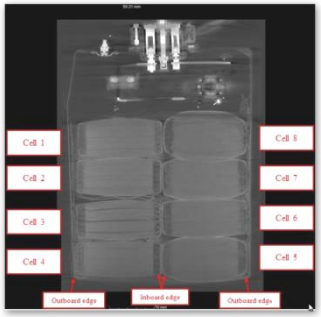
- Diverse in-house laboratory equipment, includes:

  - Extensive Optical and multiple SEMs - essential for FA
  - Surface Analysis – SIMS, XPS, Surface Tension
  - Organics Analyses – TGA, FTIR, GC-MS/FID, HPLC
  - Elemental and Anion/Cation Analysis:
    - EDX, XRF, ICP-MS, AAS, IC
  - Mechanical Test (VH, RH, Tensile, Viscosity)

- Access to additional capabilities at partner labs, includes:



  - TEM, FIB, Raman, AFM, NMR, µCT X-Ray



APU Battery Failure: UPS
Airbus A300-600 #1354
{NTSB, CT X-Ray)

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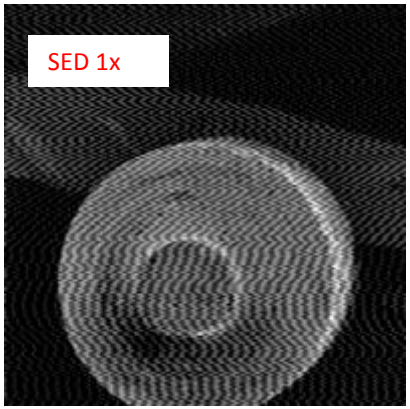
Additional Example1: Spot Welding Process Stability Investigations

Problem

Spot welding - weld strength varying per weld shot.

- Weld pull strength drift vs time.
- Power / Force / Spot and HAZ size unstable over electrode life.
- DoE process trials could not optimise process.



Investigations

SEM/EDX and OM showed:

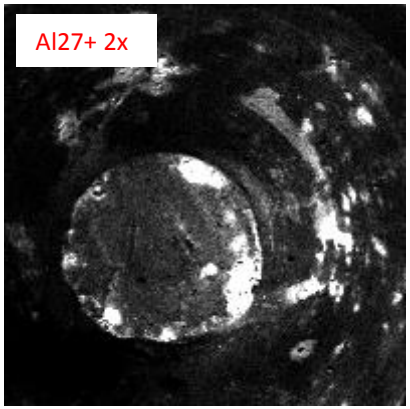
- Electrode edges rounding.
- Copper Alloy with Alumina particles for hard wearing property throughout.

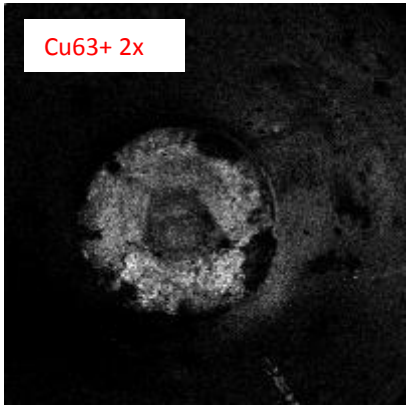
SIMS showed:

- Build up of thin layers of alumina at edge.
- Quantity of residual alumina changed electrode resistance so power per unit area and force varied over time.
- Weld power set too low to stop alumina build up.

Higher Power and Force Solved Problem.

SIMS Images of Bad Electrode....





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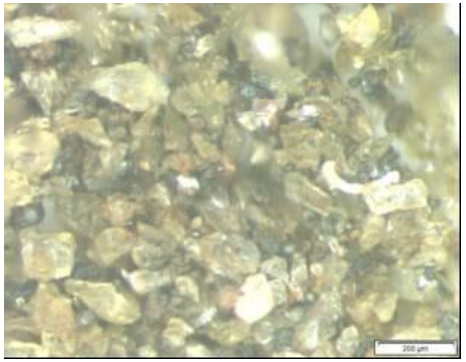
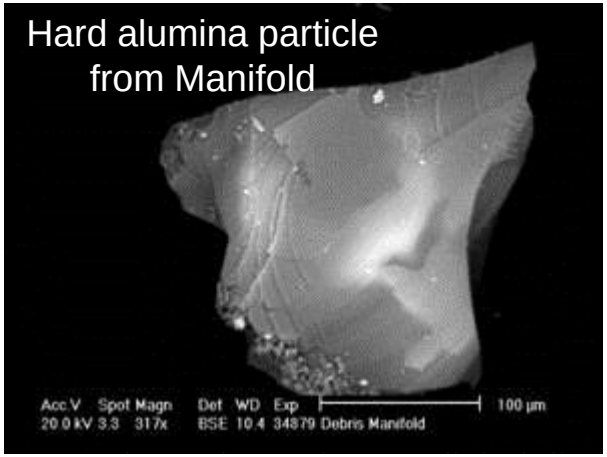
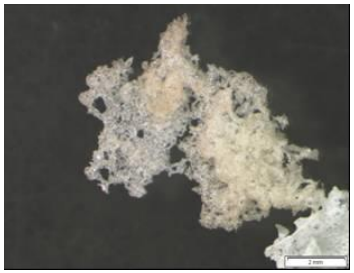
Additional Example2: Deposits Wearing a Water Pump Interior - Early life failure

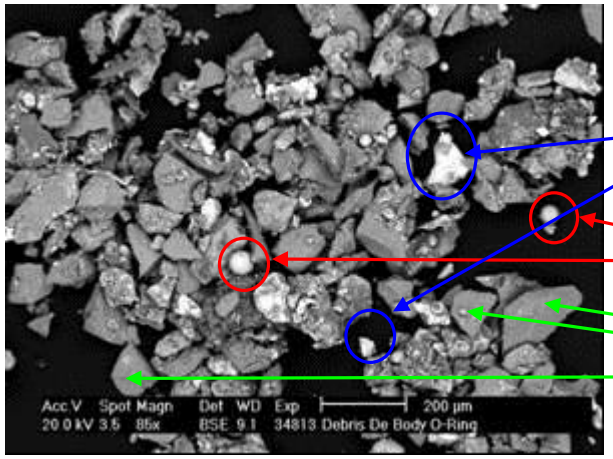
- Characterise the deposits from inside the pump, likely cause and implications for pump.
- Particle sizes and shapes are characteristic of source (optical microscopy and SEM/EDX)
- Source of hard particles critical to cause – Degradation or External contamination?







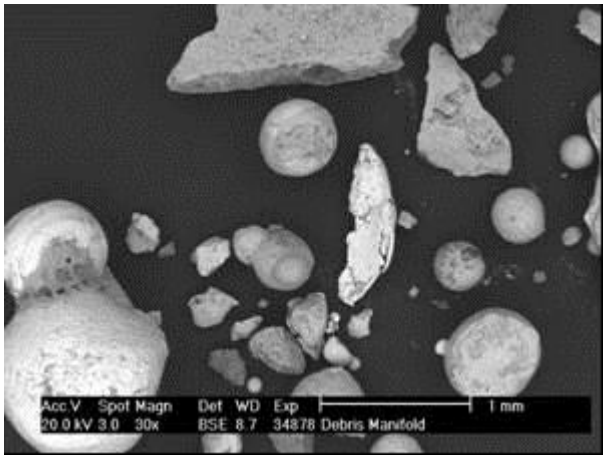
Packing material – Polyurethane prepolymer - Not fully cured
- Not a source of inorganic debris



Irregular titanium rich probably metallic flakes

Iron Oxide Coated Spheroidal Particles – Weld Splatter

Common Aluminosilicate Glass Particles / Fragments



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Additional Example3: Particle Contamination Identification and Elimination

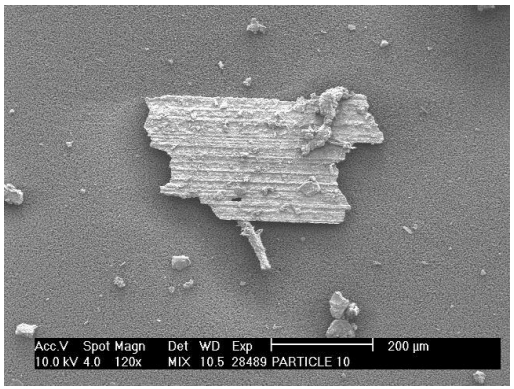
- Particle contamination is problematic for many industries.
- Isolating and characterising the particles (with Optical Microscopy, SEM / EDX, FTIR and SIMS) can yield source.

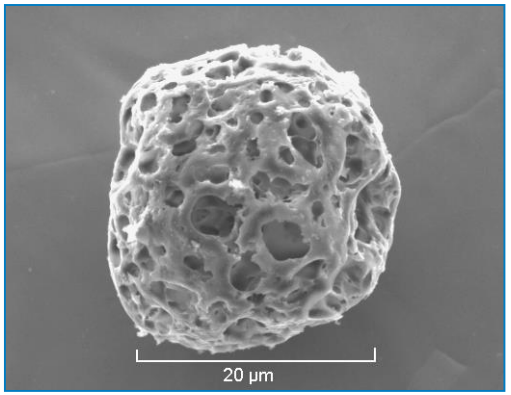
Can perform this work on virtually any type of sample, including:

- Liquid samples – suspended particles (in raw materials or process chemicals).
- On Filters / Membranes.
- Product surfaces and under transparent polymer layers

  e.g. Pre-preg surfaces, in laminated structures.

Metal oxide particle with characteristic machine marks





Charred pollen grain found to block a filter.

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