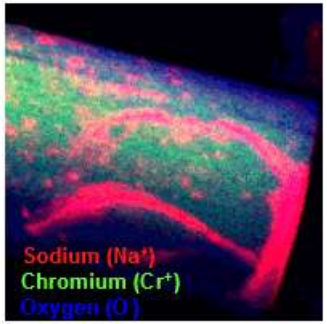

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Technical Industrial Problem Solving and Failure Investigation at LPD Lab Services

Dr Stephen Jenkins – Managing Director / Principal Scientist

Composites / Advanced Engineering Show 2013 (NEC) - 13th November 2013

RAPRA Session Invited Speaker

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LPD Lab Services

Who are LPD Lab Services?

- Mix of analytical industrial chemists, materials scientists / engineers, physicists (Over 180 man-years experience – 9 technical staff)
- Access to Consultants and Trusted Partner Laboratories
- Diverse product and process manufacturing knowledge.
- Experienced and pragmatic problem solvers.
- Used to providing quick and effective solutions to deal with unusual problems
- Diverse laboratory equipment – right tools for job!
- Skilled in bespoke sample preparation without interfering with physical and chemical structures.
- Flexible / proactive approach to scope of work.

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LPD Lab Services

Introduction to Specialities of LPD Lab Services

Specialties:-

- Physical Analysis
- Chemicals Analysis.
- Materials analysis and materials engineering
- Surface analysis
- Bespoke tests and measurements
- Problem Solving
- Consultancy
- Reverse Engineering / Deformulation
- Product and process development.

Key Factors –

Competent, Experienced, Fast, Adaptable and communicative

Turn **complex data** into **understandable practical information.**

Accreditations –

ISO 17025:2005 (laboratory)

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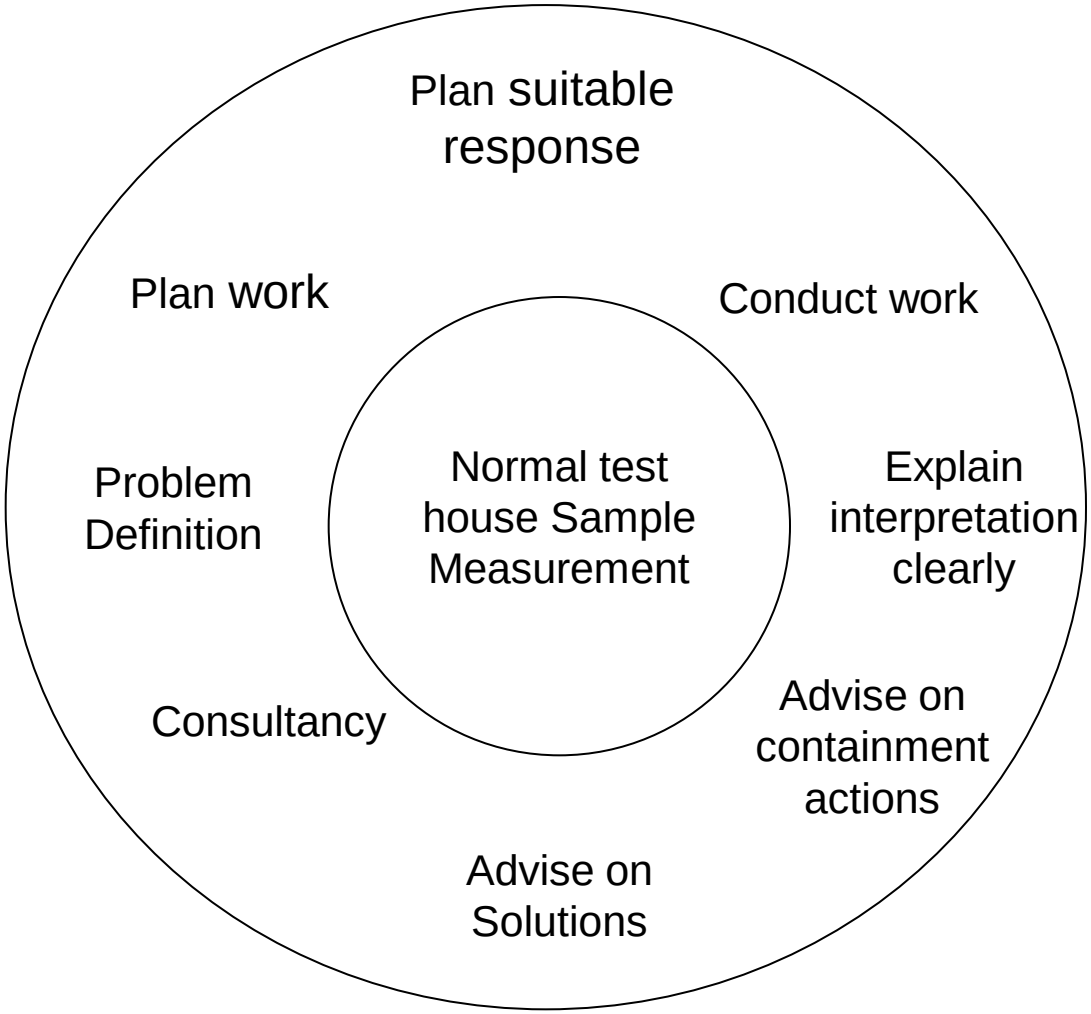
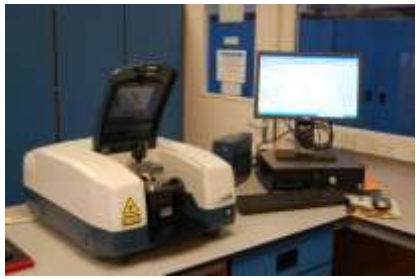
Problem Solving Approach – Beyond a Test House





Support specialising in:-

- Product development.
- Competitor benchmarking.
- Industrialisation and Ramp up
- Production Rejects
- Field Failure – Customer return.

Problem Solving Methodology:-

- 6 Sigma
- 8D
- Kepner Tregoe (KT)

LPD Lab Services

In House Laboratory Techniques

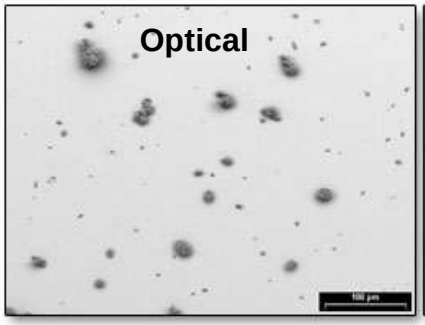
Surface Analysis / Surface Science.
•XPS, SIMS, AES, Contact Angle.

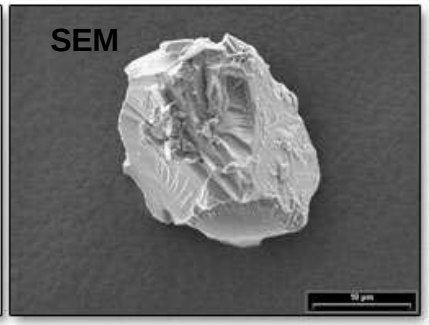
Scanning Electron Microscopy (SEM) / EDX

Optical Microscopy / Image Analysis

Chemical Analysis
•FTIR, AAS, XRF, ICP-MS GC, GC-MS, HPLC, IC, UV/Vis-(NIR) TGA, Wet Chemical

Mechanical Properties
•Tensile and hardness testing





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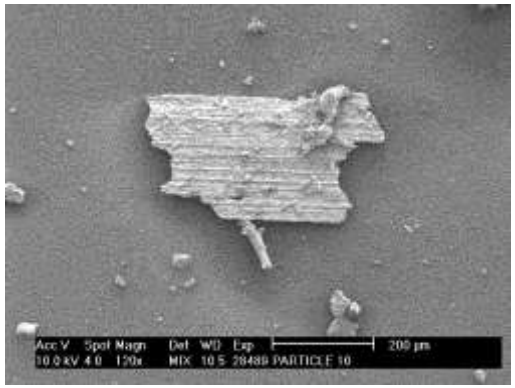
Particle Contamination Identification and Elimination

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

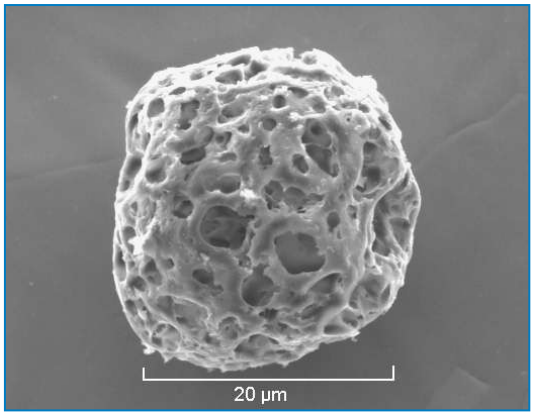
Perform this work on virtually any type of sample, including:

- Liquid samples – suspended particles (in raw materials or process chemicals).
- Filters / Membranes.
- Product surfaces and under transparent polymer layers
  eg Pre-preg surfaces, in laminated structures.



Metal oxide particle with characteristic machine marks

Charred pollen grain found to block a filter.



Buried Particles in Laminate

Particle identification, isolation, characterisation and type matching in a laminate.

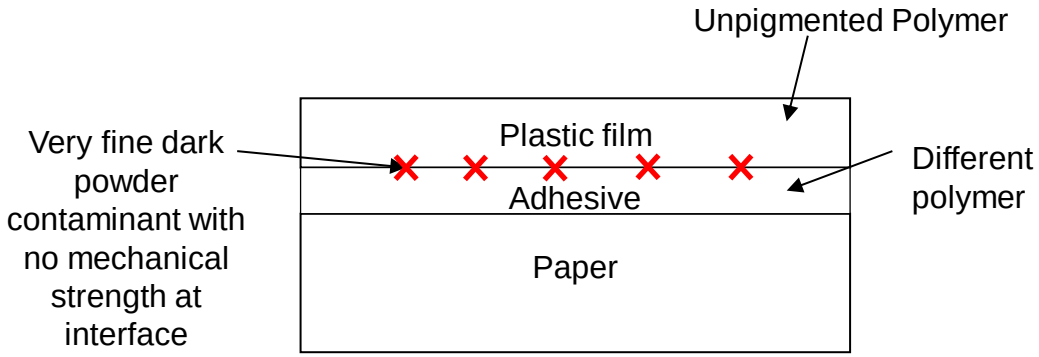
150µm diameter particle

- Dismantle laminate under microscope.

OR

- Solvent removal of over-layers without disturbing physical and chemical structure so causal link can be proved.

SEM/EDX showed particles in raw and finished laminate materials chemically and physically similar (iron oxide + other elements) to confirm supplier was source.



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Materials Problems with Composites, Plastics and Coatings?

Plastic Moulding Cracking Over Life

Moulding conditions can leave residual stress:
Relieved over life giving cracking (Environmental Stress Cracking - ESC)

- Solvent or cleaning agents give polymer chains mobility – Cracks.
- Affected by filler contents, filler types, size shape and distributions (Optical microscopy, SEM, PSD)
- Polymer crystallinity.

Shrinkage

- Mobile species like plasticisers or low molecular weight polymer can leach out under heat.
- Change mechanical properties.
- Cause delamination.

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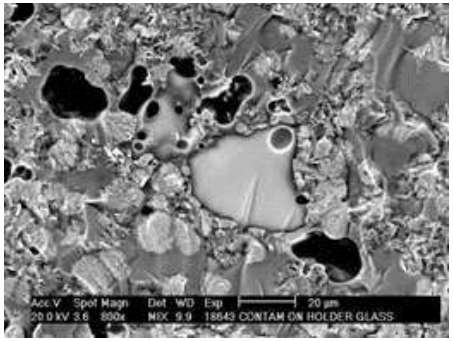
Materials Problems with Plastics and Coatings?



Depolymerisation / Degradation

- Stabiliser concentration problems – Material less robust.
- Hydrolysis (FTIR).
- Oxidation (FTIR).
- Free radical attack (FTIR).
- Attack by mobile metallic ions (AAS).
- Chlorine embrittllement in Polyacetal (IC, SEM/EDX, SIMS)..

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Mechanical Property or Moulding Distortion Issues

- Wrong Volume fraction of reinforcement fibres (TGA).
- Insufficient ‘Size’ applied - Poor matrix – fibre load transfer.
- Moulding conditions left short fibre incorrectly distributed.
- Excess mould release agent.
- Plasticisers leaching out to surface – (XPS or SIMS).
- Contaminants at Pre-preg interface (SEM, FTIR or Surface Analysis).
- Depolymerisation – Chain Scission.
- Environmental Stress Cracking (ESC).
  - Hydrolysis Moisture attack so polymer changes - FTIR, XPS, SIMS, TGA.
  - Discolouration and UV radiation attack FTIR, SIMS.
  - ‘Size’ effects diffusion rates as fast path to interior.
  - Temperature and humidity affects

Carbon Fibre Composite Fracture Surface

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Delamination in Products

Delamination:

- Incomplete Resin Curing.
- Weak boundary layers – Residual release agents, interfacial or particle contamination.
- Poor surface treatments (eg Corona discharge)
- Coatings cured too fast during application or mix wrong (FTIR/XPS and look at recipe and process.)
- XPS, FTIR and SEM to check for residual release films.
- Poor cleaning.
- Water Ingress.
- Corrosion Issues.

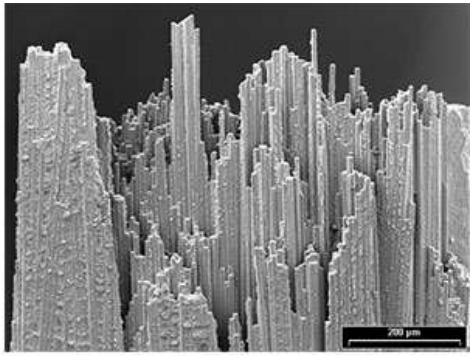

Figure 1 - Carbon Fibre Fracture Surface

SIMS or XPS determination of residual mould release agents or plasticisers surface segregating giving delamination problems

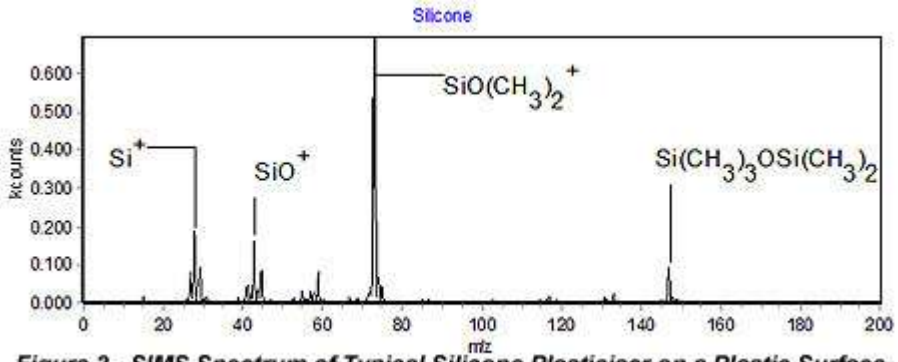

Figure 2 - SIMS Spectrum of Typical Silicone Plasticiser on a Plastic Surface

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Summary and Final Comments

RAPRA
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Effective Problem Solving Needs…

- Experienced analytical staff to recognise failure mechanisms.
- Combine interpretation from multiple techniques.
- Act as extra R&D manpower / consultants for customer.
- Actively input into product and process modifications.
- Add value input when analysts have close understanding of the product and design requirements.
- Adaptable and react fast.

Any Questions?

LPD Lab Services Ltd

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