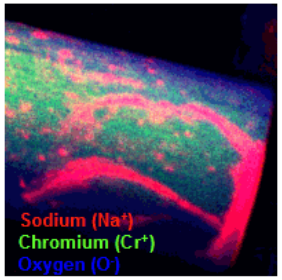



# Technical Industrial Problem Solving, Failure Investigation and Product Development Support at LPD Lab Services

## (Commercial Multi-technique Industrial Problem Solving)

## UKSAF Meeting - 6th January 2016

Dr Stephen Jenkins – Managing Director

RAPRA
Research & Development
Preferred Provider

Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

# **LPD** Lab Services

## Who are LPD Lab Services?

- **Mix of very experienced analytical industrial chemists, materials scientists / engineers, physicists**
- **10 technical staff, many ex-Philips product and process development.**
- **LPD started external services in 2003, independent in Oct 2009**
- **Access to Consultants and Trusted Partner Laboratories**
- **Diverse product and process manufacturing knowledge (Materials, chemicals, products and processing techniques).**
- **Experienced and pragmatic problem solvers backed by 6 Sigma expertise.**
- **Used to providing quick and effective solutions to deal with unusual problems.**
- **Diverse laboratory equipment.**
- **Skilled in bespoke sample preparation without interfering with physical and chemical structures.**
- **Flexible / proactive approach to scope of work.**
- **Customers:- 1-man bands, through SMEs to multinationals.**

Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

# **LPD** Lab Services

## Introduction to Specialities of LPD Lab Services

**Specialties:-**

- Physical Analysis
- Chemicals Analysis.
- Materials analysis and materials engineering
- Surface analysis
- Bespoke test development and measurement
- Problem Solving
- Consultancy
- Reverse Engineering / Deformulation
- Competitor benchmarking
- Product and process development.

**Key Factors:–**

**Competent, Fast, Adaptable and communicative**
Turn **complex data** into **understandable practical information.**

**Accreditations:–**
ISO 17025:2005 (laboratory)

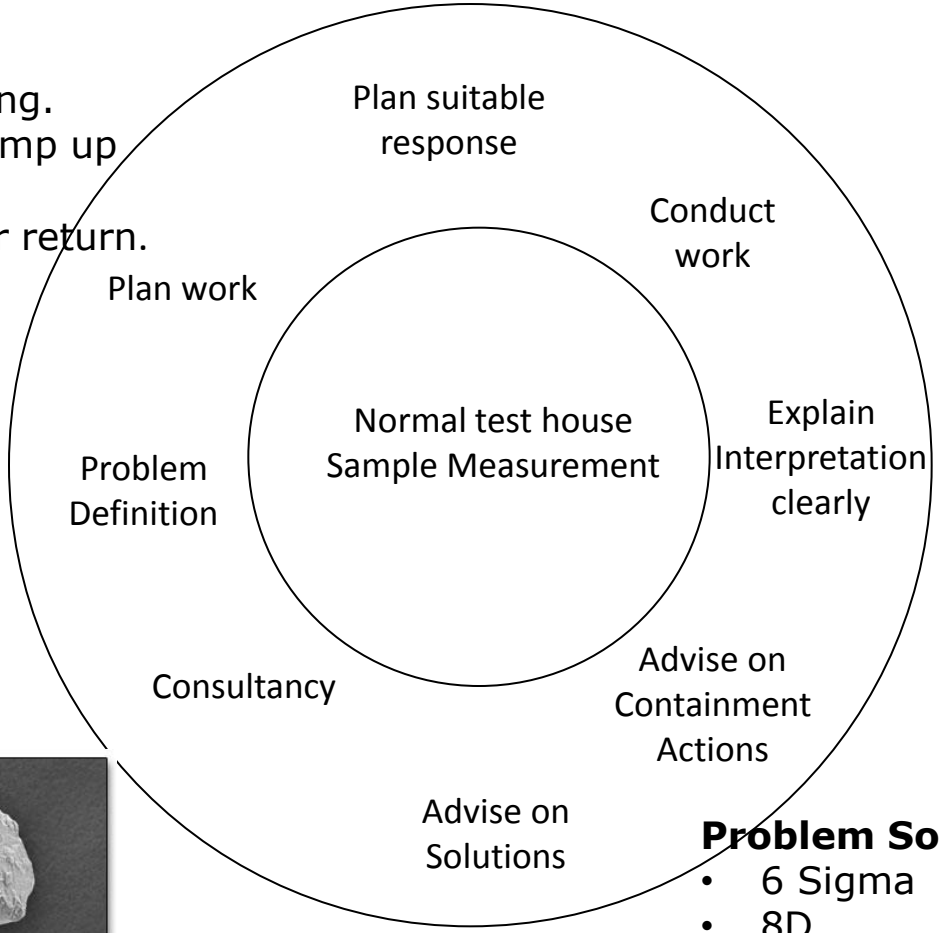
UKAS TESTING
2766

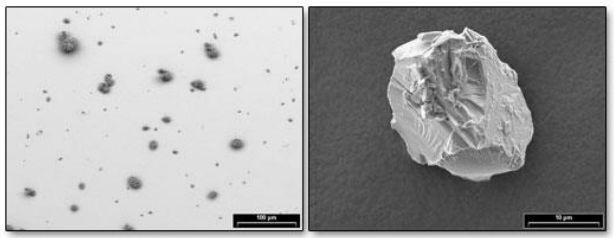
Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

## 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)

Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

# **LPD** Lab Services

## In-house Laboratory Techniques

**Surface Analysis / Surface Science.**
- XPS, SIMS, AES, Wetting / 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

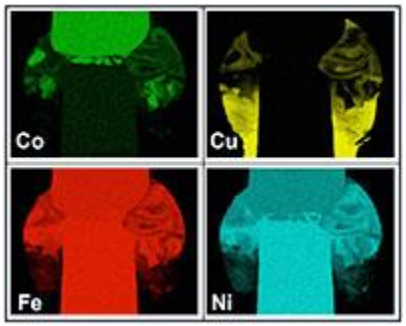
**Mechanical Properties**
- Tensile and hardness testing

Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

## Materials and Failure Analysis

**Why?**

- Generate improved performance and quality of products
- Cost reduction
- Identify, track and remove contaminants.




**Involves:-**

- Materials Analysis and Failure Investigation
- Physical, Structural and Microstructure Analysis
- Chemical Analysis.

**Materials analysis instrumentation:-.**

- **Optical Microscopy, SEM/EDX, FTIR, XPS, SIMS, XRF, AAS, GC-MS**

Cost and Time efficiency:-

- Failure analysis allows skillful dismantling products and components to solve problems - **Determine material and product shortcomings.**
- Reverse engineering **benchmarking of competitors** products to reveal the production methods and materials - **Drive product development.**

# **LPD** Lab Services

## Physical, Structural and Microstructural Analysis

Physical properties, mechanical properties, structure and microstructure of:-

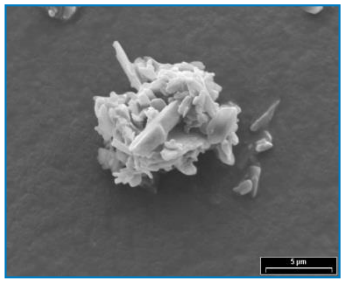
- Chemicals
- Materials
- Assembled products.

Including:

- Top down inspection – Optical Microscopy and SEM
- Cross-sectional analysis – Optical Microscopy and SEM
- Image analysis

- Chemical compatibility and wetting (contact angles) - DCAT/OCAT

- Mechanical Tests and Hardness
- Viscosity
- Particle size, size distribution and shape.

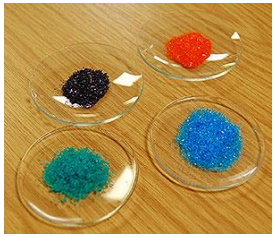


Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk



7

## Chemical Analysis



- **Gases, Liquid** or **Solids**
- Bespoke **Method Development or Routine analysis**
- Wide **array of analytical techniques**:-
  - FTIR, XRF, AAS, ICP-MS, GC-MS, GC-FID, HPLC, IC, UV/Vis Colorimetric, Karl Fisher, Titration, Back-extraction, TGA.
- Practical **Context-orientated interpretation**

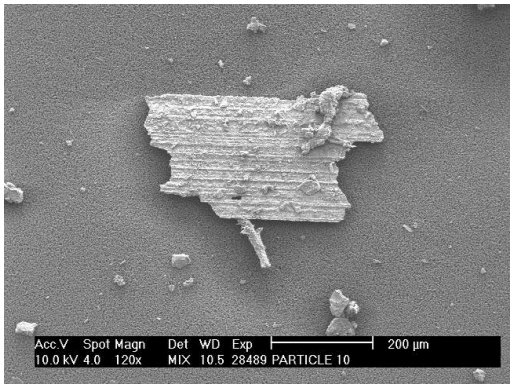
**Example work**:-

- Product / process control and validation
- Identification and elimination of contaminants
- Chemical Product Deformulation and Reformulation
- Adhesives, coatings, adhesion promoters and coupling agents
- Analysis of binders and fillers
- Determination of plastic / polymer types
- Identification of unknown chemicals and detergents
- Trace metal or organic chemical analysis.

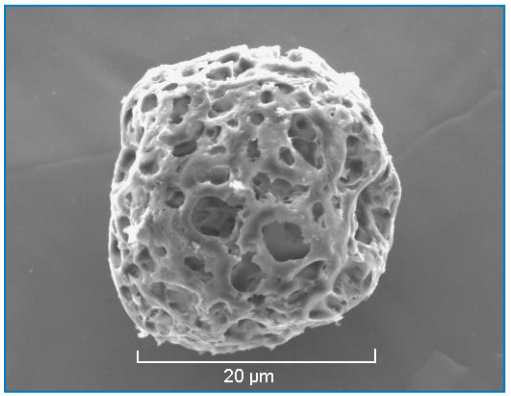
## Particle Contamination Identification and Elimination

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

- **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 polymer layers.



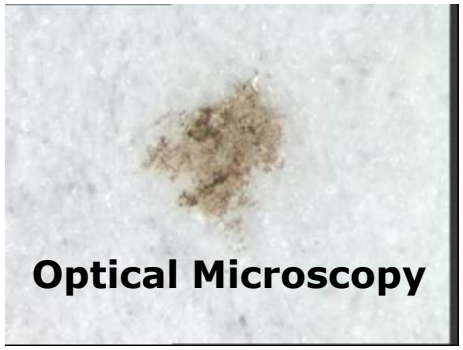

Metal oxide particle with characteristic machine marks




Charred pollen grain found to block a filter.

## Buried Particles in Laminate

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



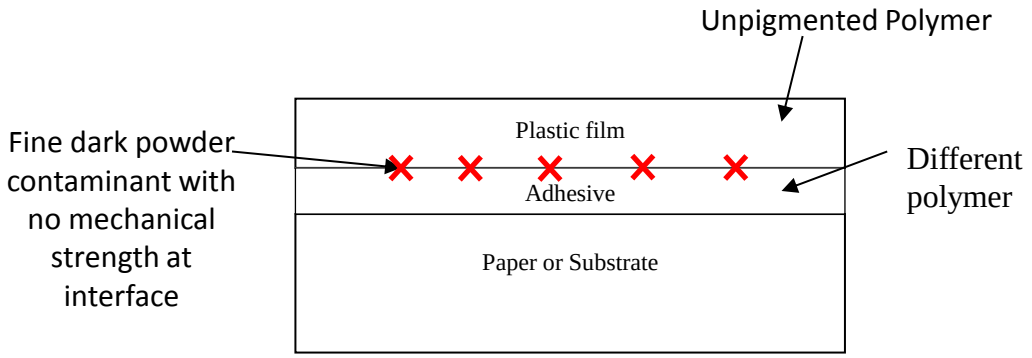

150µm diameter particle

Dismantle laminate under microscope

*OR*

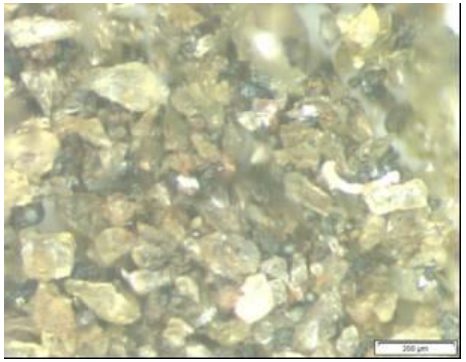
Solvent removal of over-layers without disturbing physical and chemical structure so causal link can be proved

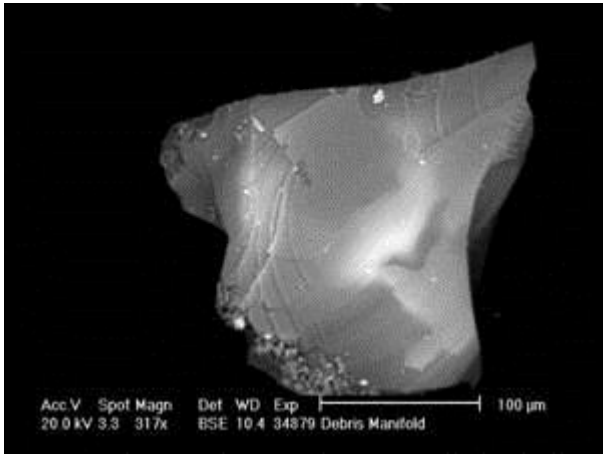
- SEM/EDX showed particles in raw and finished laminate materials chemically and physically similar (iron oxide + other elements) to confirm supplier was source.

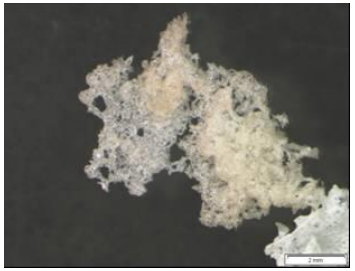



## Deposits Wearing a Water Pump Interior - Early life failure

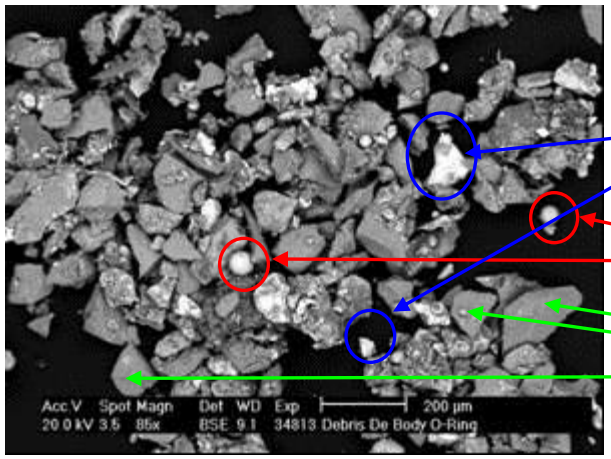
- Characterise the deposits from inside the pump, likely cause and implications for pump.
- Particle sizes and shapes are characteristic of source (optical microscopy and SEM/EDX)
- Source of hard particles critical to cause – Degradation or External contamination?





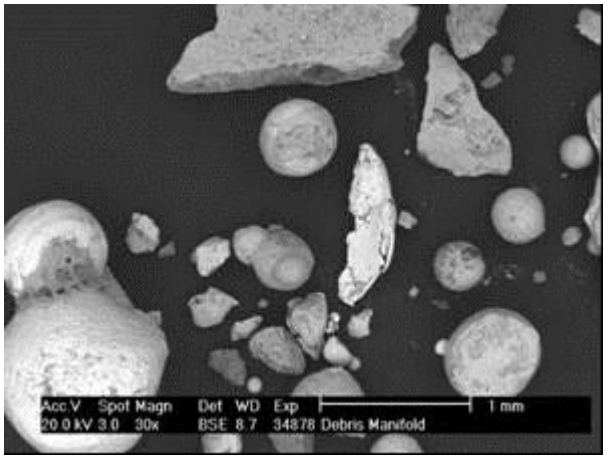
Packing material – Polyurethane prepolymer - Not fully cured but not a source of inorganic debris




Irregular titanium rich probably metallic flakes

Iron Oxide Coated Spheroidal Particles – Weld Splatter

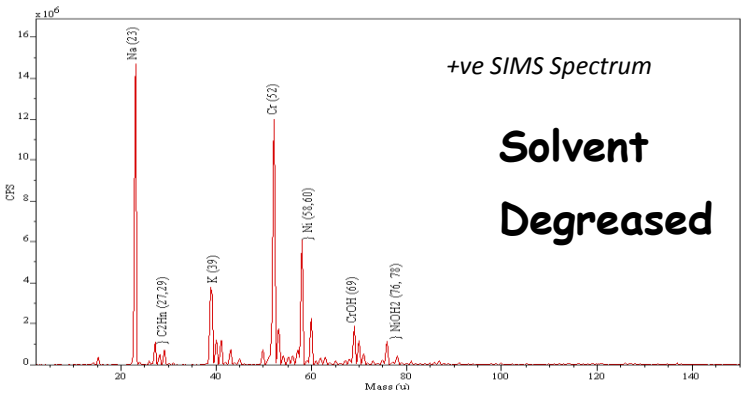
Common Aluminosilicate Glass Particles / Fragments




Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

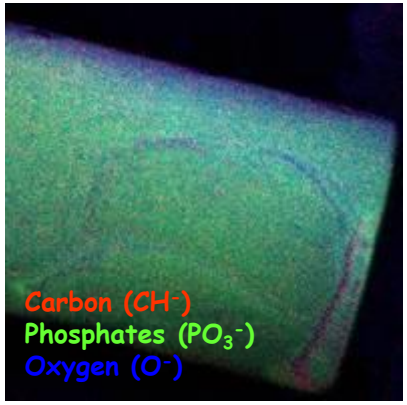
## Issues with Degreasing Remnants on Metal Parts Revealed by SIMS

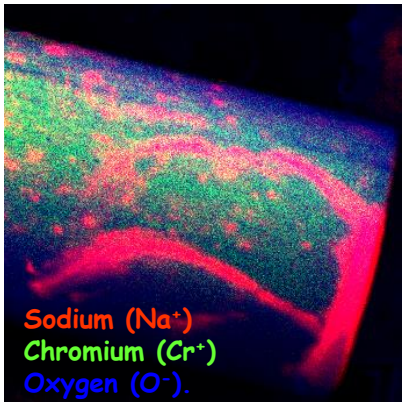
Industry under pressure to replace solvent–based cleaning/degreasing processes with aqueous-based processes (Environmental and H&S reasons).




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

➔ Clean surface




**Aqueous Degreased (2.5mm FoV)**

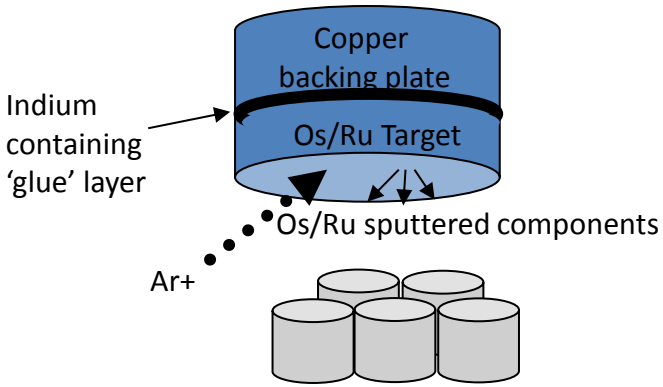
- Most of hydrocarbon removed
- Underlying Ni and Cr exposed
- High levels of Na, Si, K, Ca Cl, Phosphates, Silicates and Nitrates from the aqueous degreasing solution.

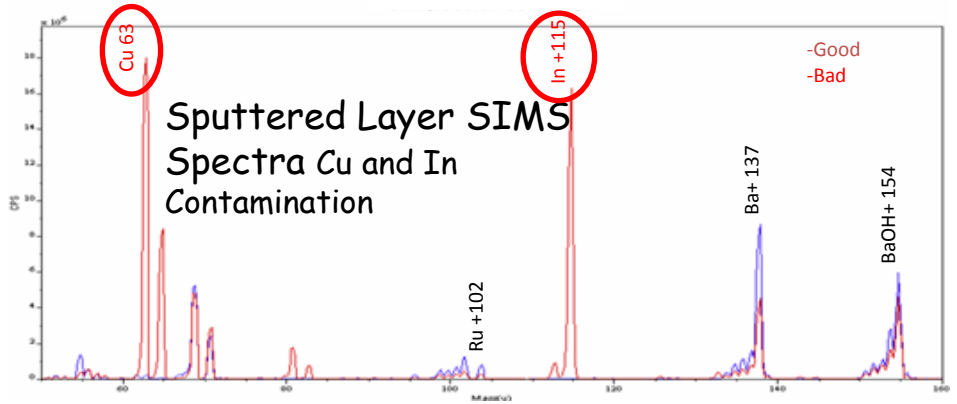
➢ The solvent process is a better cleaning process.

➢ Aqueous process leaves more remnants behind potentially causing functional problems.

# LPD Lab Services

## Sputtering Target End of Life Problem SIMS Investigation of Deposition Products




Cathode emission performance tests & CUSUM data -> showed manufacturing Quality change.

SIMS → Root cause analysis

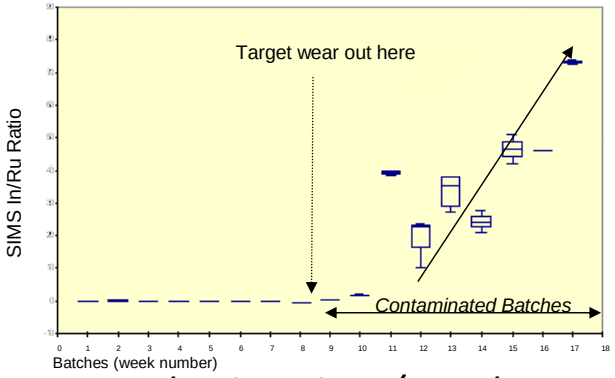
SIMS → Problem Containment

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*

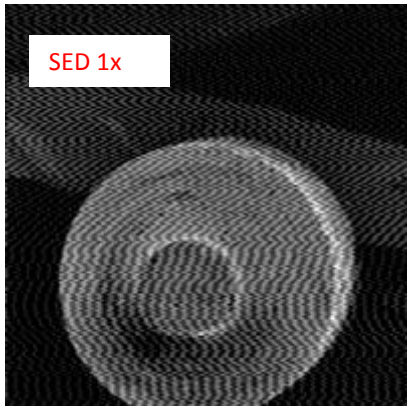
Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk

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.

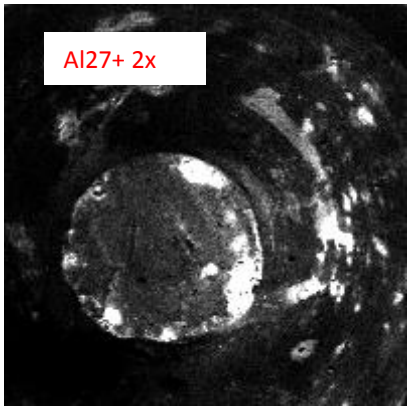
SIMS showed:

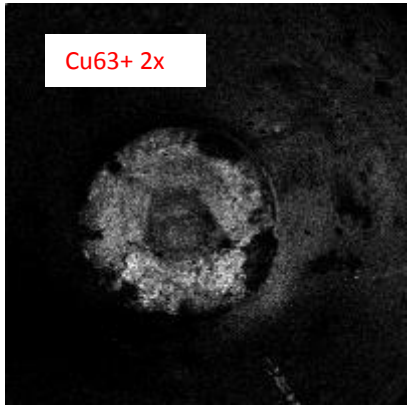
- Build up of thin layers of alumina at edge.

- **Quantity of residual alumina changed electrode resistance so power per unit area and force varied over time.**
- **Weld power set to low to stop alumina build up.**

**Higher Power and Force Solved Problem.**

SIMS Images of Bad Electrode



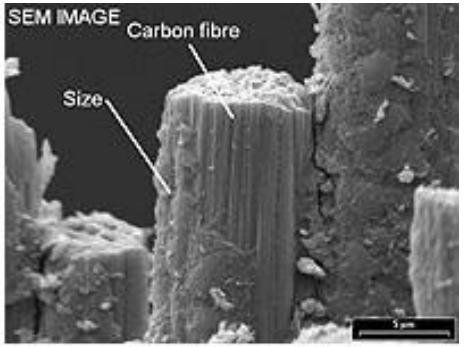



## Summary and Final Comments

- **Effective Problem Solving Needs…**

- Experienced analytical staff to recognise failure mechanisms and solutions.
- Combine interpretation from multiple analytical 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?



Carbon Fibre Composite Fracture Surface

Philips Road Blackburn Lancashire BB1 5RZ UK
Tel: +44 (0)1254 676074 Fax: +44 (0)1254 278845
www.lpdlabservices.co.uk