



The University of
Nottingham

Thermally sprayed coatings: *Strategies for improved reliability and performance*

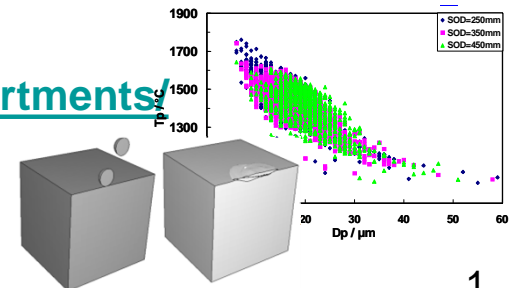
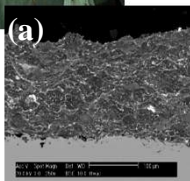
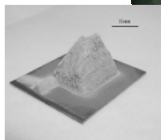
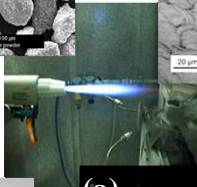
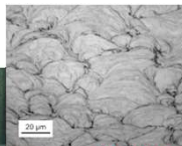
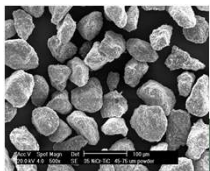
Prof Graham McCartney

Thermal Spray Research Group

Faculty of Engineering

University of Nottingham

<http://www.nottingham.ac.uk/engineering/departments/m3/people/graham.mccartney>



This presentation will cover the following:

- Introduction to thermal spraying at Nottingham.
- What options are available for improving coating reliability and performance?
- Case study 1: Developing appropriate reliability measurements for WC-Co cermet systems.
- Case study 2: Process monitoring of thermal spraying and data interpretation.
- Summary and conclusions.



Introduction – Thermal Spray at Nottingham

- **Academic Staff**
 - Graham McCartney, Philip Shipway, Katy Voisey
- **Processing Facilities**
 - Thermal Spray: Liquid fuel and gas fuel spray systems (MetJet and Top Gun) also cold spray equipment
 - Particle Diagnostic Equipment: DPV 2000 (thermal spray) and Oxford Lasers PIV system (cold spray)
 - Powder production: Sieving, milling, mechanical alloying
- **Coating testing**
 - Characterisation of coatings (XRD, OM, SEM, TEM);
 - sliding and fretting wear behaviour;
 - electrochemical and salt spray corrosion behaviour;
 - high temperature oxidation and creep testing;
 - Coating fracture and ductility by four point bending

Introduction – Thermal Spray at Nottingham

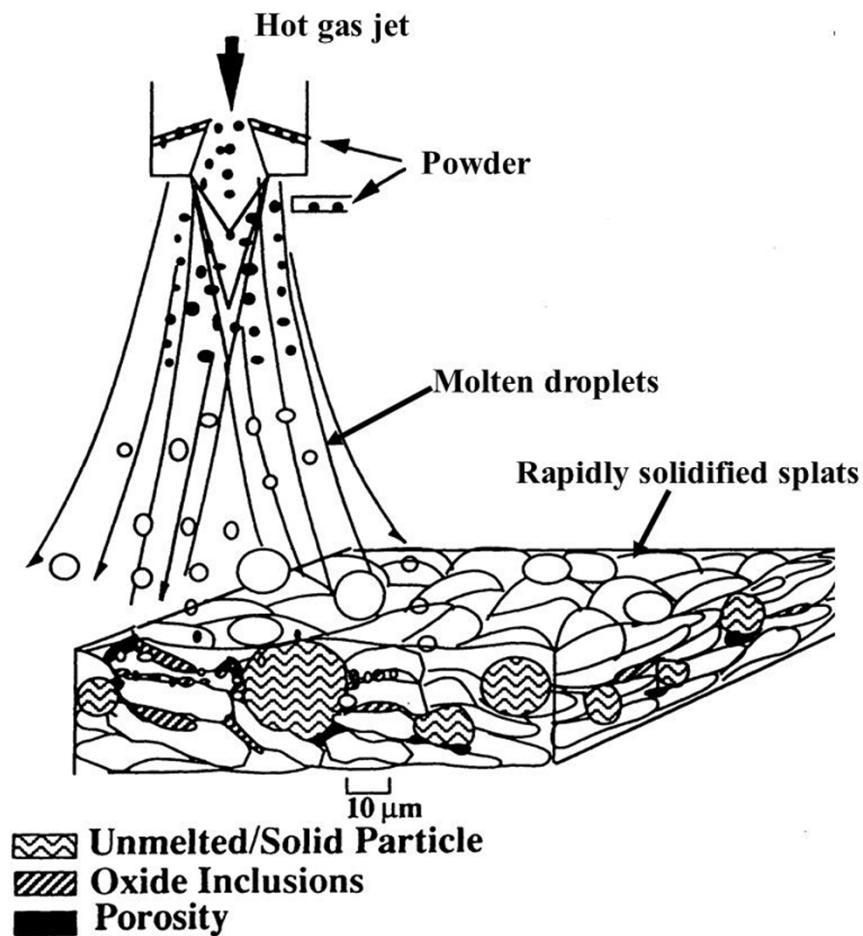
- **Coating systems**
 - Improved cermets (WC-Co, WC-Ni, Cr_3C_2 -Ni(Cr), TiC-Ni(Cr))
 - Improved performance of corrosion resistant alloys (eg IN625)
 - Novel amorphous/nanocrystalline alloys (Nanosteel, Armacor etc)
 - New and improved MCrAlYs for bond coats
 - Cold spray materials: Cu, Ti, multilayers, Al-Si alloys, composites
 - Novel thermal spray applications (Al-Sn journal bearings, pyrophoric materials)
- **Technical support staff**
 - Rory Screaton
- **Research students**
 - Currently 5 PhDs on thermal spraying, over 15 PhDs graduated in thermal spray since 1995.

Improving reliability and performance (1)

- Metallurgists and materials engineers focus on process – structure – property relationships in engineering materials - thermally sprayed coatings are no exception.
- BUT process-property links not well understood in coatings :
 - Thermal spraying is a stochastic process i.e. coating is built up from the deposition of many thousands of individual particles and each particle can have its own individual structural characteristics
 - Thermal spraying has many different process parameters that need to be controlled e.g. powder feedstock, input rates of gases/fuels, torch motion relative to substrate, substrate characteristics
- Additionally, what do we want to measure about the coating? Ideally a *relevant* property or performance but this may not be easy.
- So optimising properties for a particular application is a major challenge.



Thermal spray deposition



Improving reliability and performance (2)

Ultimately we can employ one or other of the following approaches:

- 1. Design of Experiments (DOE/Taguchi) methods**
- 2. Open loop/closed loop process monitoring and control**

However, both have limitations in their capability to achieve consistent, reliable and optimised performance.

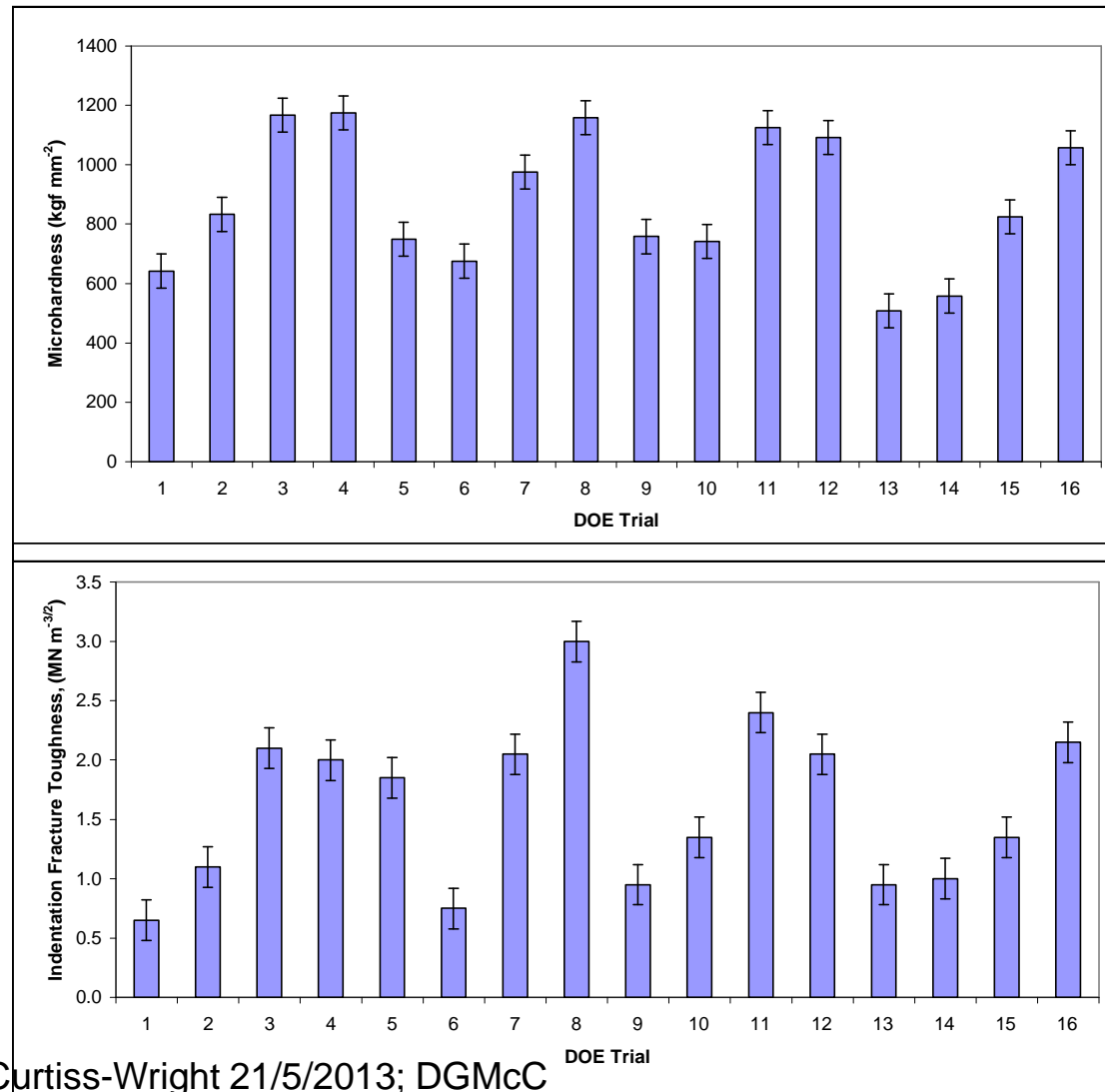
Also we should be asking, “By how much does control improve the economics of the thermal spray industry and what does it cost?”

Case study 1: Improved performance of WC-Co in a safety critical application

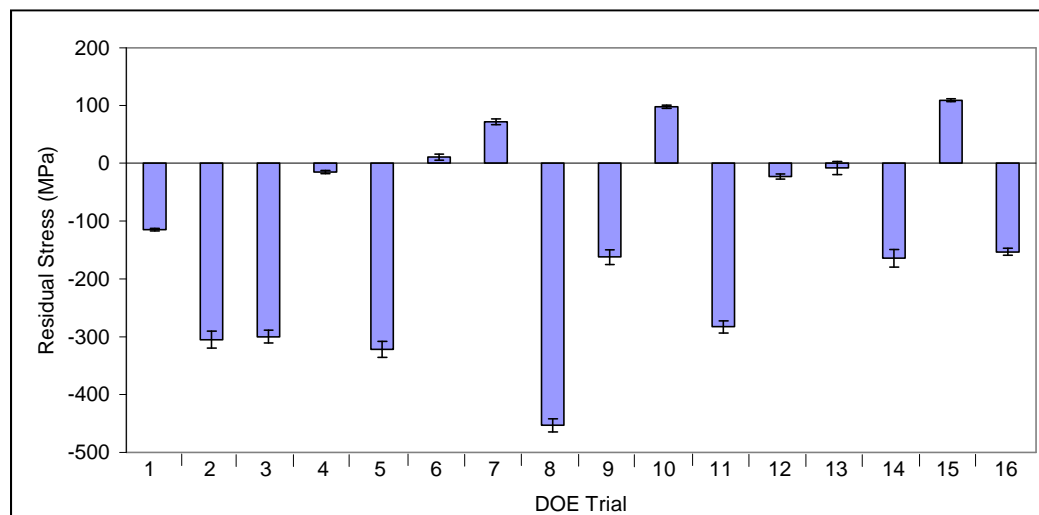
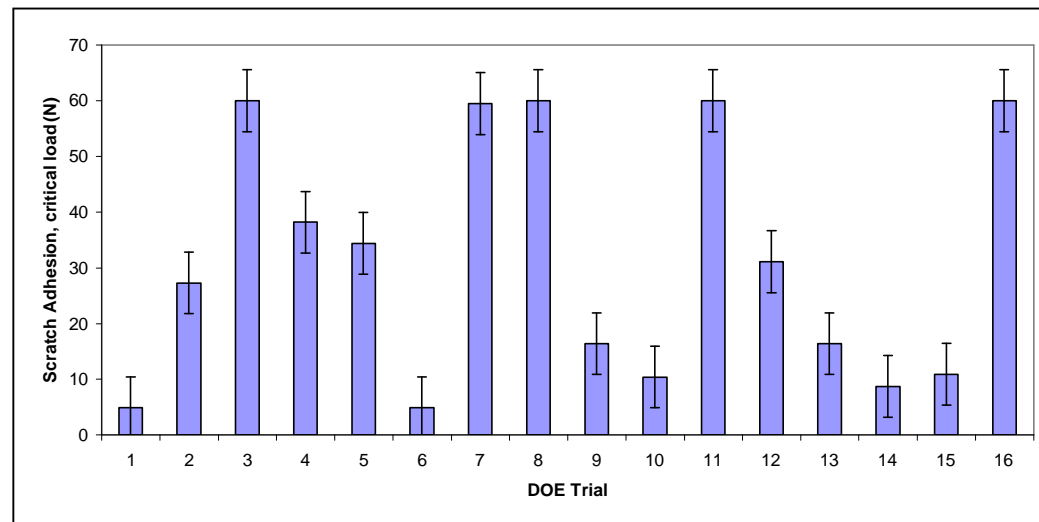
OBJECTIVE

Use experimental design approach with a fractional factorial design (DoE) to optimise performance

- First, identify the ideal outcome (i.e. what is the specification of the coating?) and secondly identify all operator controllable variables and set appropriate levels.
- Secondly, define a number of simple tests to assess the ideal outcome commonly: microhardness, indentation fracture toughness, porosity, microstructural analysis etc; all involve local area/volume analysis.
- Thirdly, plot average response graphs and use analysis of variance (ANOVA) methods to predict spray parameters needed for 'ideal' behaviour.



EXAMPLE Results of DoE trials – hardness and toughness

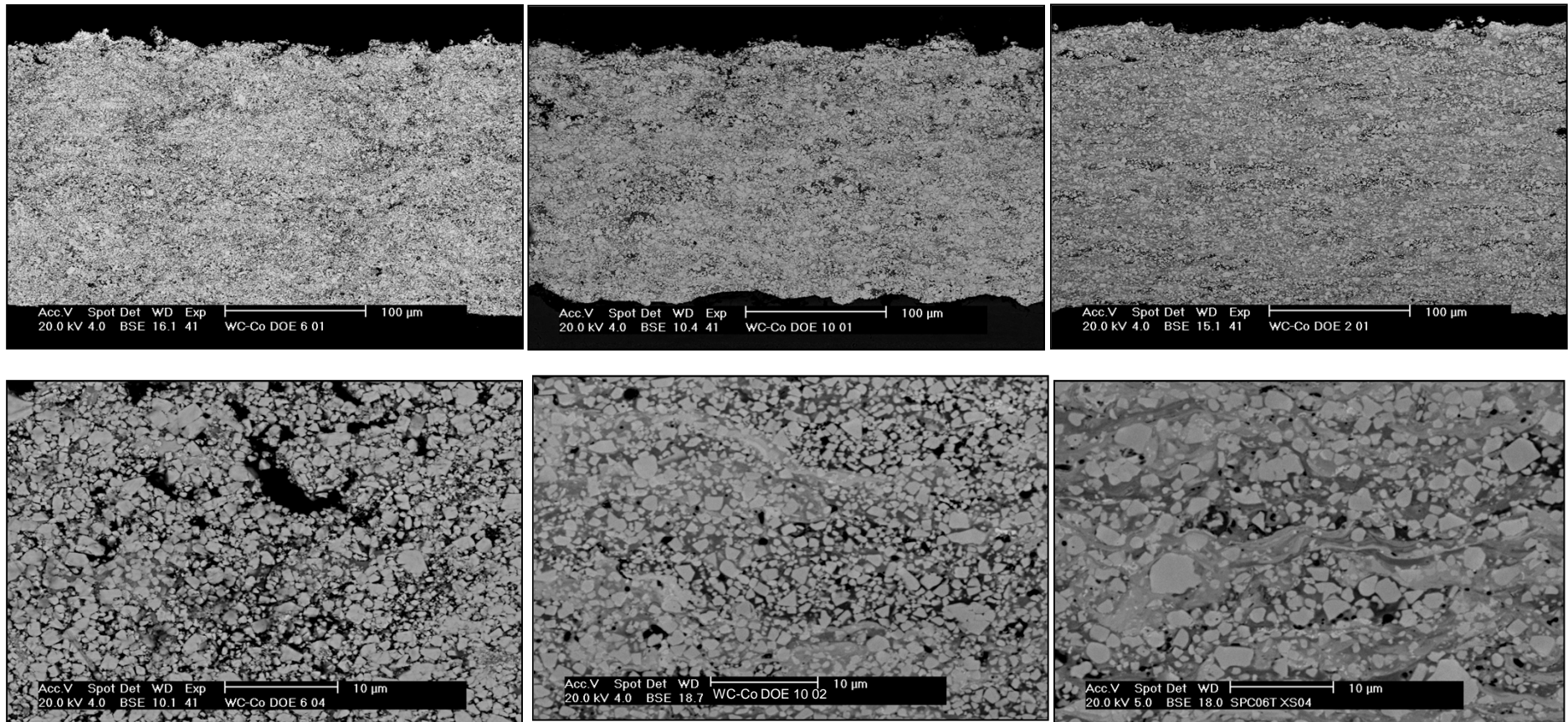


Curtiss-Wright 21/5/2013; DGMcC

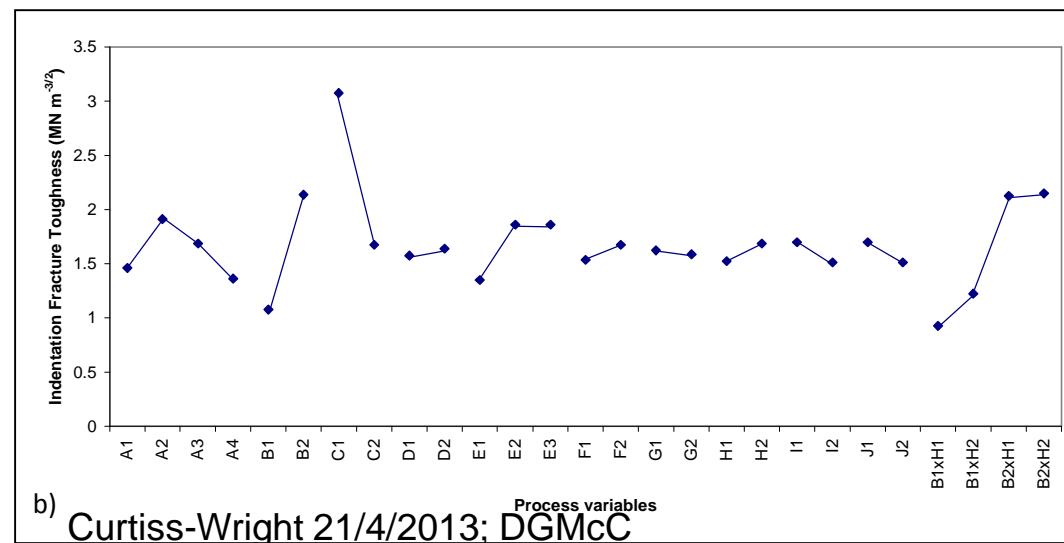
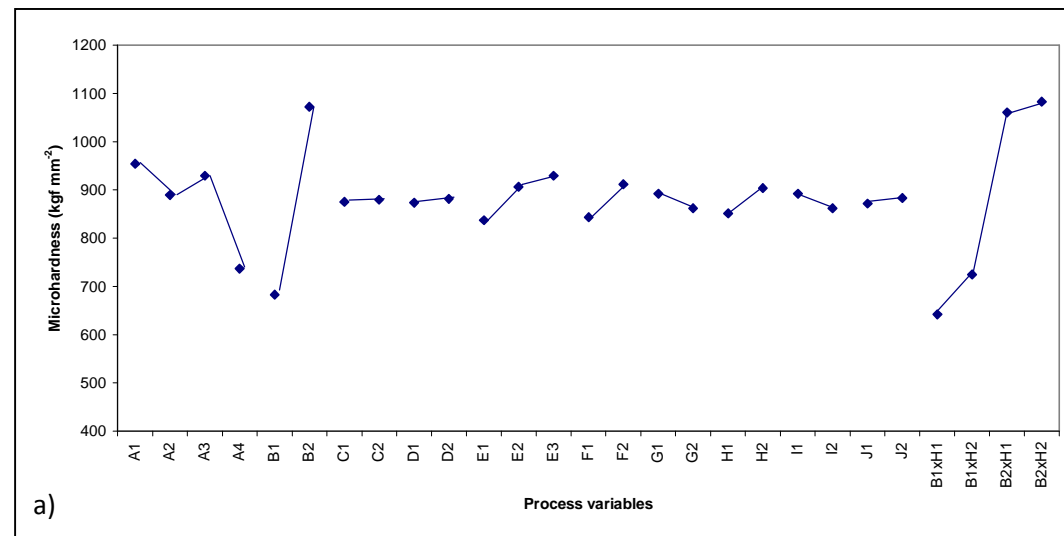
EXAMPLE Results of DoE trials – adhesion and residual stress



Results of initial DoE trials - microstructure



For WC-Co coatings, measure ratio of WC to W_2C



Results of DoE trials

- Plot response graphs (examples shown)
- Use ANOVA to obtain optimum parameters for “ideal coating”

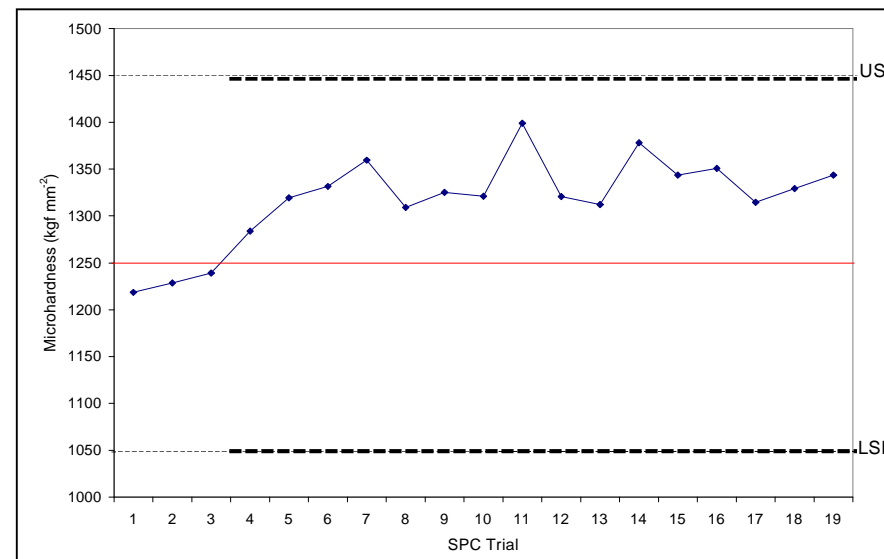
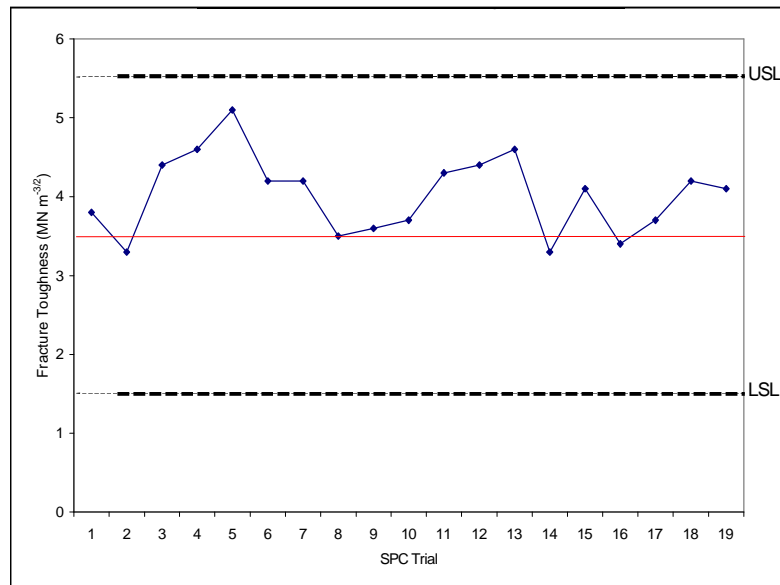
Case study 1: How well does this approach work?

- Monitor process reliability and repeatability in production through statistical process control charts (SPC approach).
- Measure the same “properties” as used in the experimental design to monitor process stability
- Examples are shown on the next slide for 19 spray runs with the “optimised” process parameters



Statistical process control charts for

- Fracture toughness
- Microhardness



Production data lie comfortably within set limits BUT..

Case study 1: How well does this approach work?

- Results look promising
- BUT
 - Have we captured everything?
 - Is this the whole story?
 - Are we measuring the right set of properties?

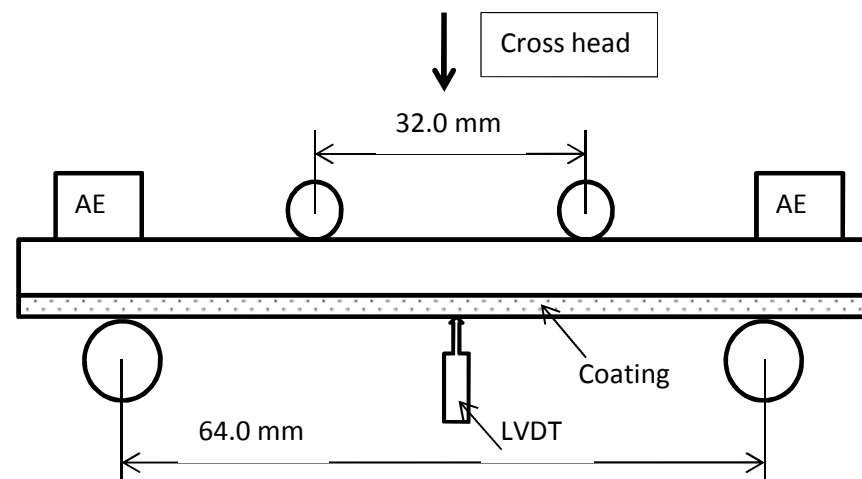
In this example we had collected additional property data which I've not shown you yet on **fracture strain of the coating**. Loosely we can call this the coating “**ductility**”. This shines some new light on the story and highlights two important points

- Simple properties like hardness are not necessarily the right ones to measure
- Very localised property measurements like hardness can give misleading data on Process Control

Case study 1: The Post Script – Measuring Ductility

METHOD

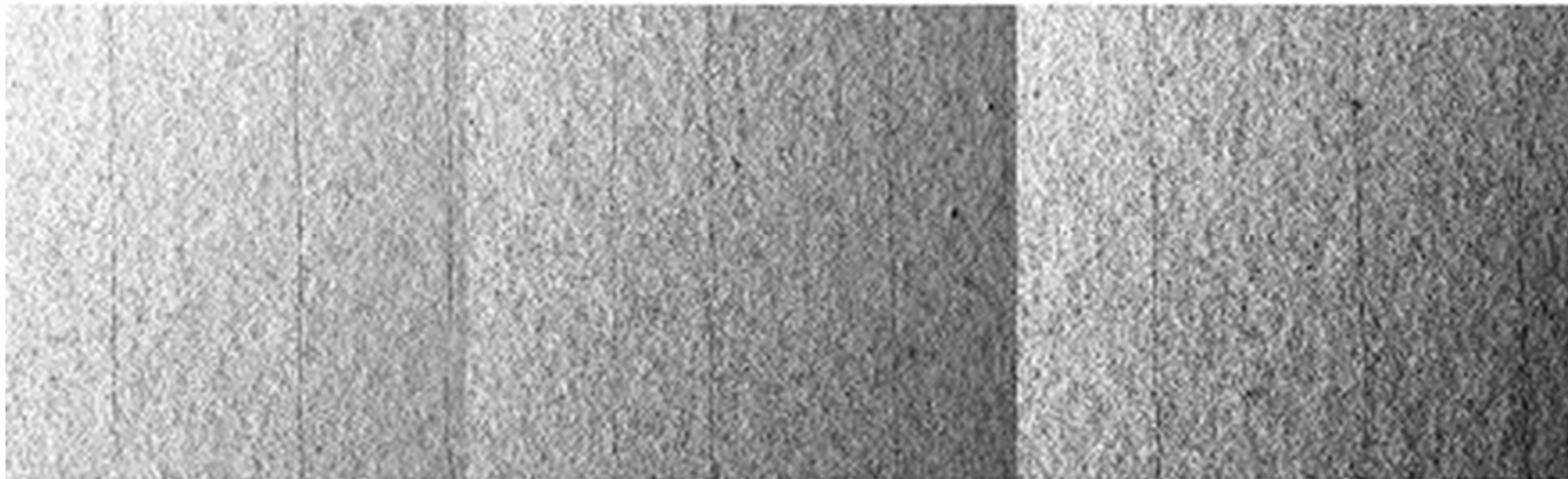
- Four point bend testing with acoustic emission (AE)
- AE sensors detect cracking onset at a critical strain
- AE sensors and associated software compute cumulative strain energy released during bending





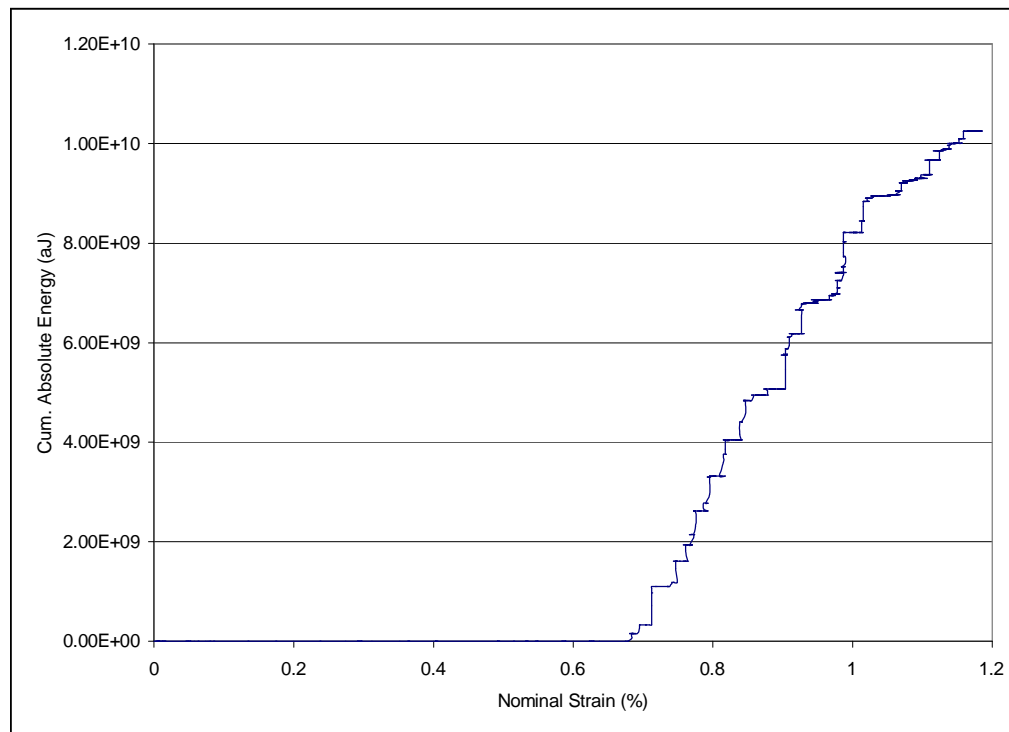
Case study 1: The Post Script – Measuring Ductility

On bending the sample we get multiple cracking of the WC-Co coating as seen in the SEM image of the coating surface. Network of cracks of near uniform spacing





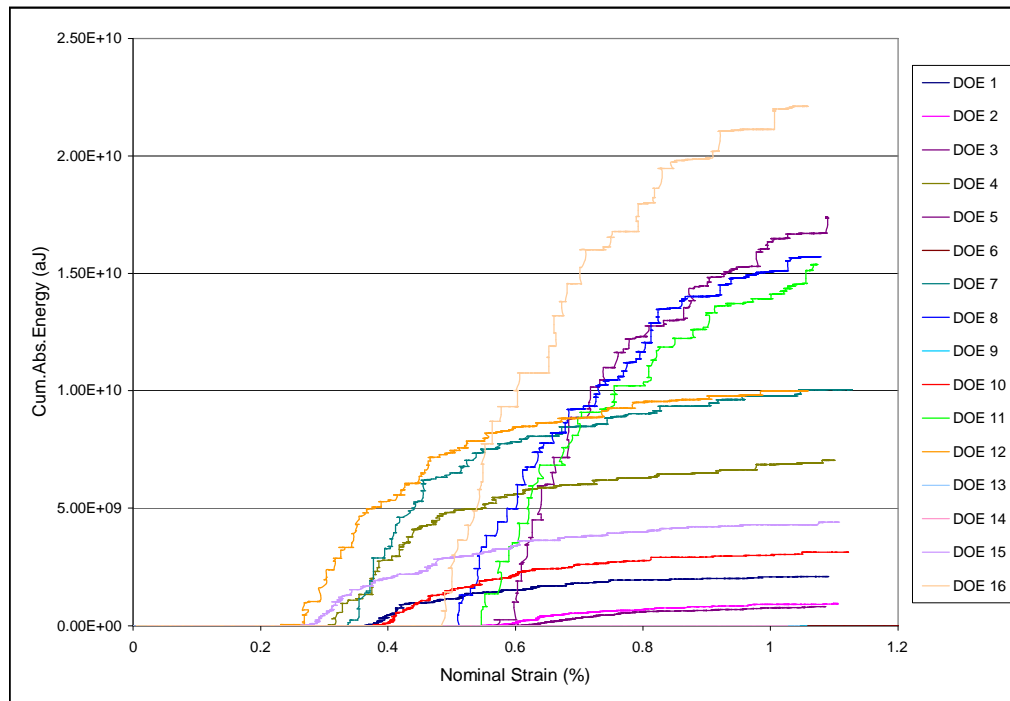
Case study 1: The Post Script – Measuring Ductility



Plot of cumulative energy detected versus nominal strain on the coating surface for a typical HVOF WC-Co coating.



Case study 1: The Post Script – Measuring Ductility



Results of DoE trials new information on:

- strain when cracking begins
- total energy emitted

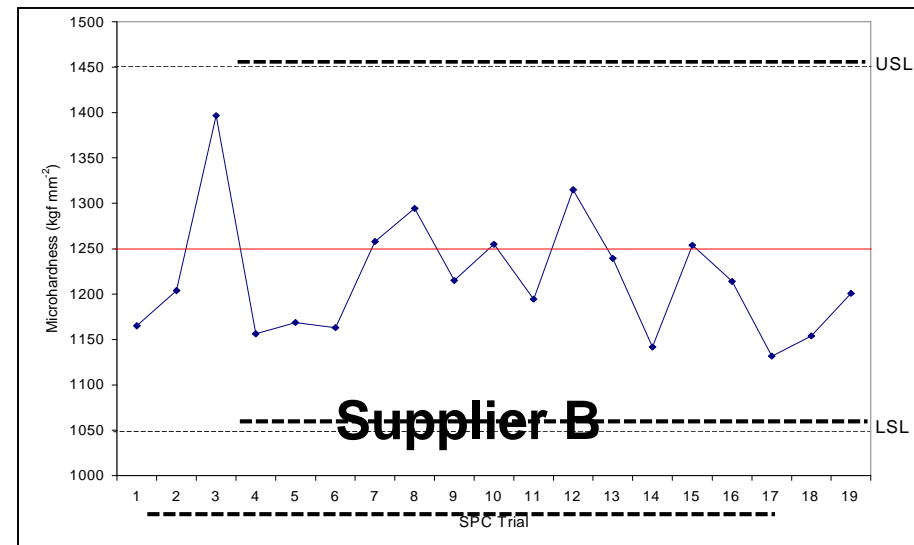
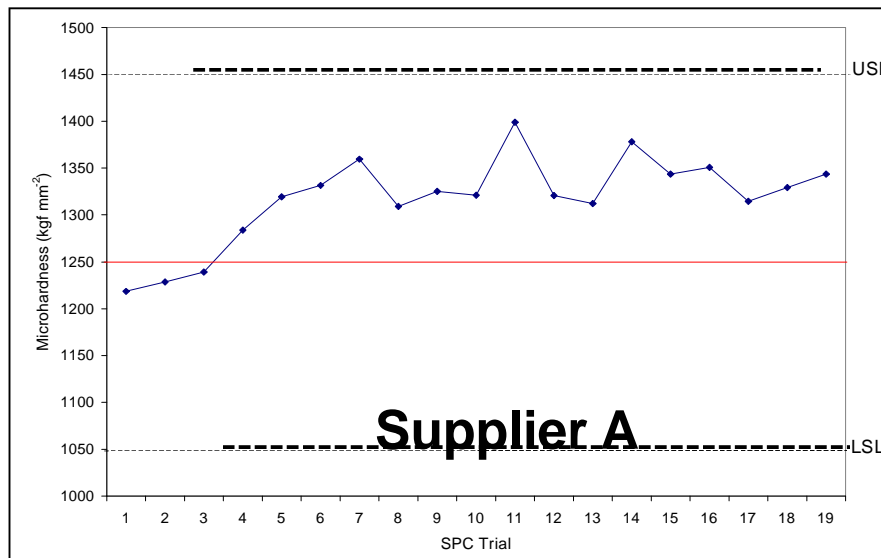
This will change the ANOVA output and influence what we regard as optimised settings.

Case study 1: The Post Script – Measuring Ductility

- A further benefit of measuring the fracture behaviour of coatings in this way is that we are sampling a much larger volume of material than conventional analyses such as microhardness or a microstructural feature
- This is seen in the following slide where we compare four point bend test results as a SPC tool.
- Suppliers A and B look identical from a SPC chart of microhardness measurements but look very different when four point bend test data are examined



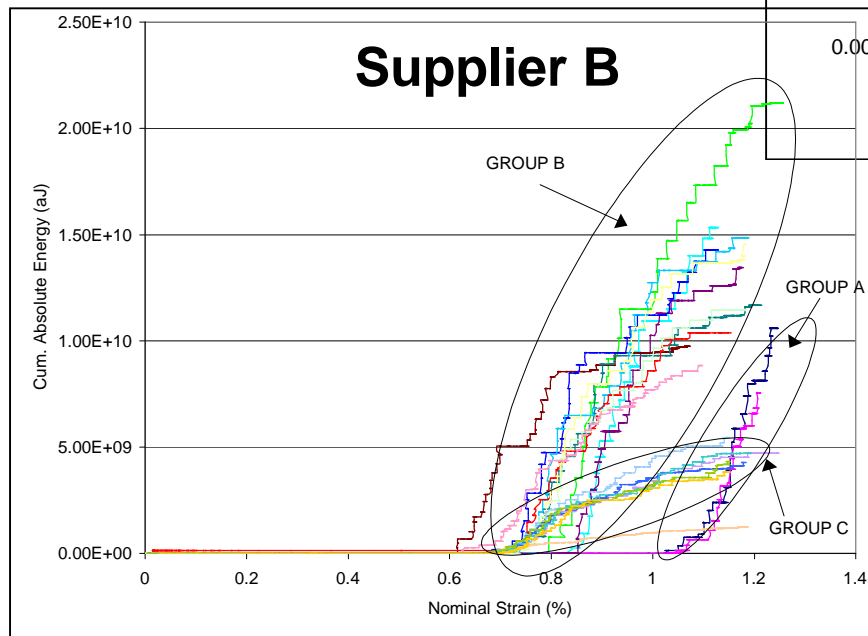
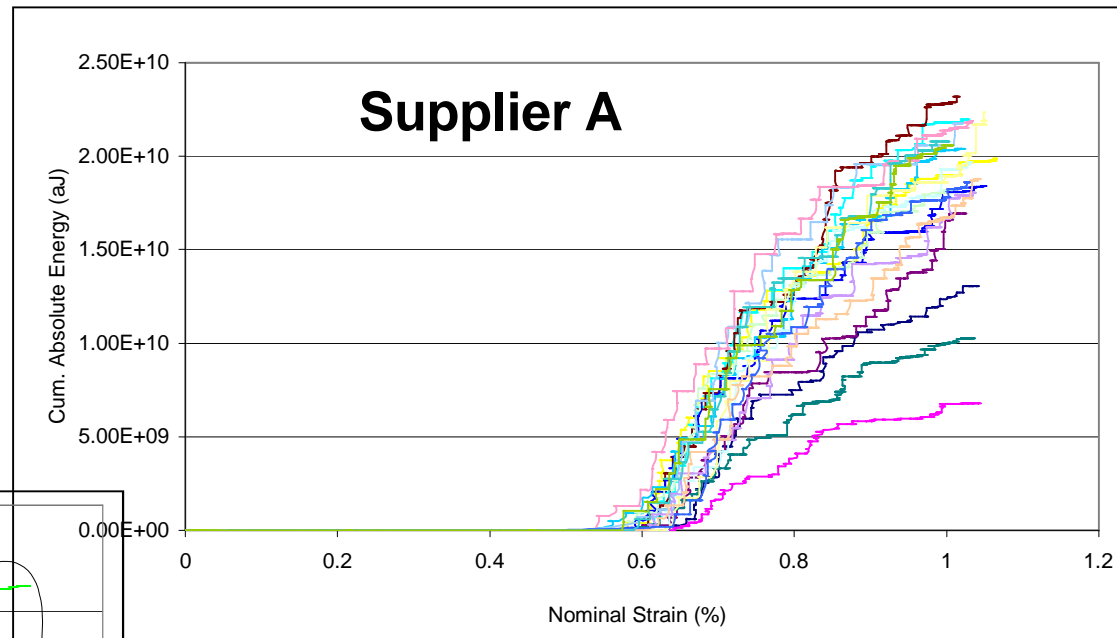
Case study 1: The Post Script – Measuring Ductility



SPC charts based on microhardness. Both suppliers meet the requirements



SPC charts based on
microhardness.
Supplier B has much greater
variability



Case study 1: Improved performance of WC-Co in a safety critical application

CONCLUSIONS

- DoE methods can be successfully used to achieve desired performance characteristics in HVOF sprayed WC-Co coatings
- Selecting appropriate properties for measurement can present a challenge
- Traditional test methods such as microhardness, indentation fracture toughness etc usually involve small localised measurement volumes
- Four point bend testing with in-situ AE is a reliable method for determining onset of fracture in WC-Co coatings.
- This test samples a large volume and can detect variabilities arising from subtle changes in process conditions which are not detected in other testing procedures

Case study 2: Process monitoring of thermal spraying using spray diagnostics

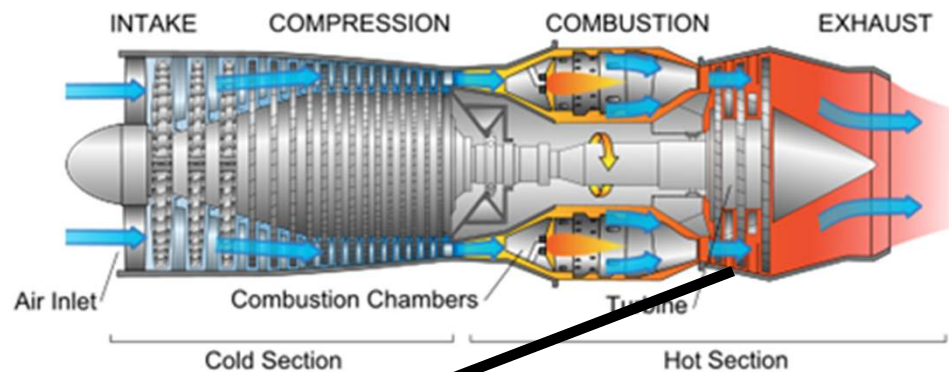
OBJECTIVE

Measure individual particle temperature, velocity and size during spraying and relate to process parameters

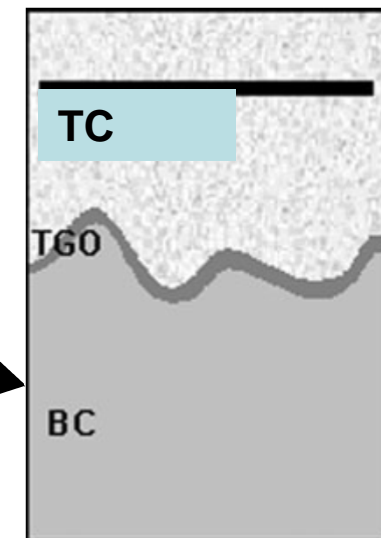
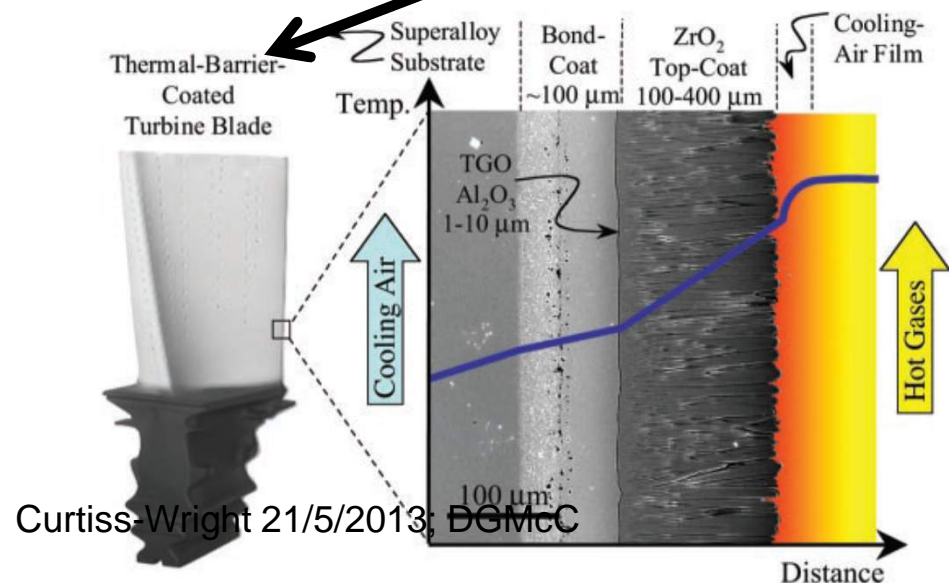
- Methodology is applied to optimising the liquid fuel HVOF spraying of NiCoCrAlY (MCrAlY) coatings used as bond coats in a thermal barrier coating system on superalloy gas turbine blades
- Employs the Tecnar DPV 2000 system to measure particle temp (T), velocity (V) and diameter (D) simultaneously
- Investigates how selected process parameters affect the above particle properties and influence coating features



MCrAlY Bond Coats in gas turbine components



BC = bond coat
TC = ceramic
top coat
TGO =
thermally grown
oxide





Process monitoring of thermal spraying

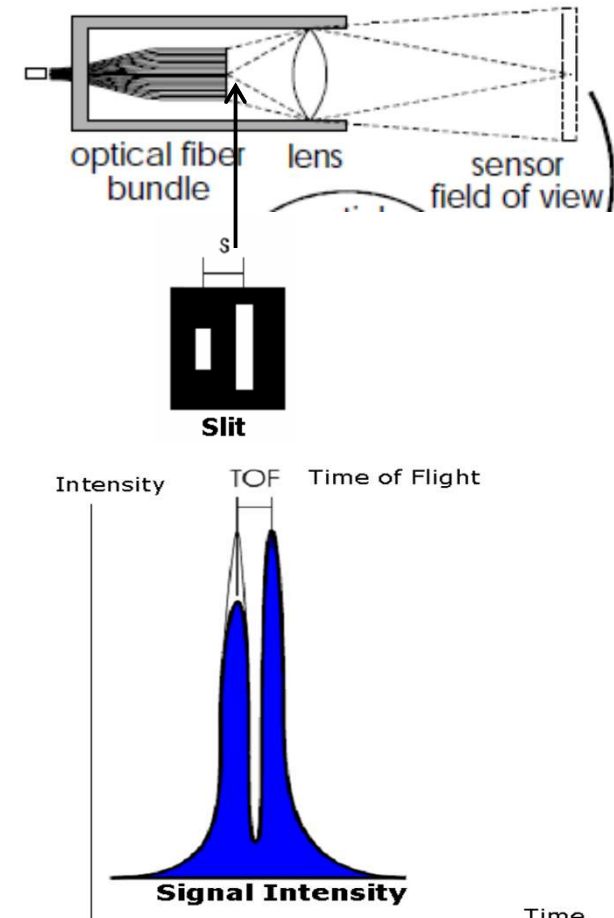
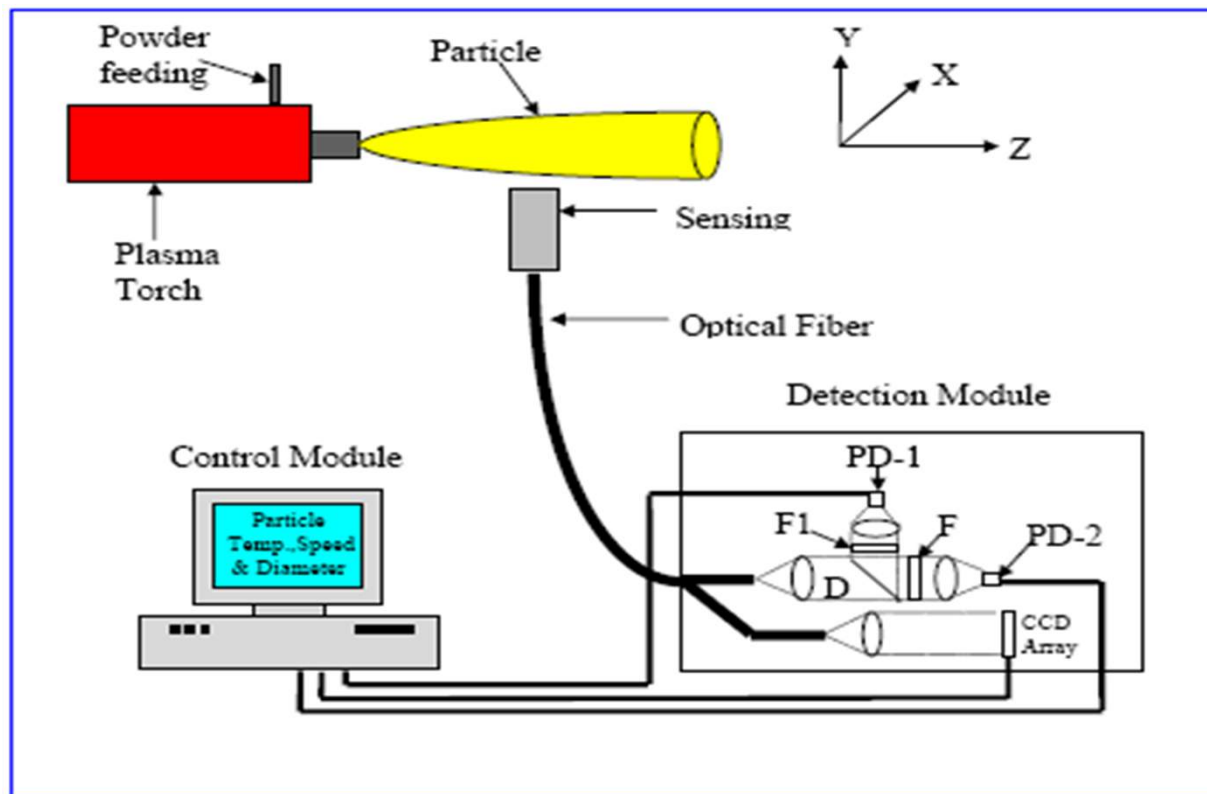
Key bond coat properties:

- **Low oxygen content of coating < 0.5 wt% O**
- **Low porosity of coating**
- Good bonding of “splats”, and coating well bonded to substrate
- Bond coat forms a slowly growing oxide (TGO) when exposed to high temperature (750 to 950 °C).
Excessive TGO growth leads to spallation failure of ceramic top coat

Aim to replace LPPS with HVOF spraying for lower cost with at least equivalent properties



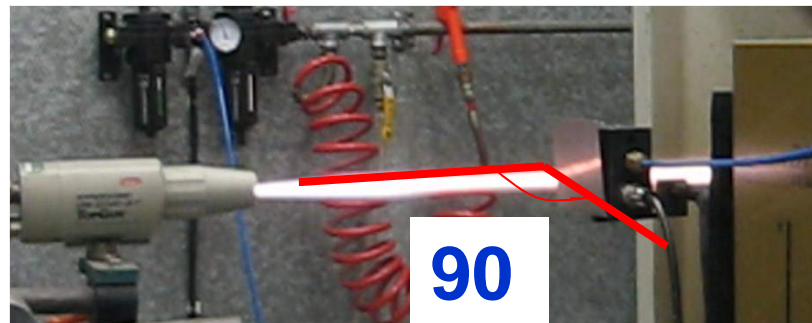
DPV-2000 System





DPV-2000 System

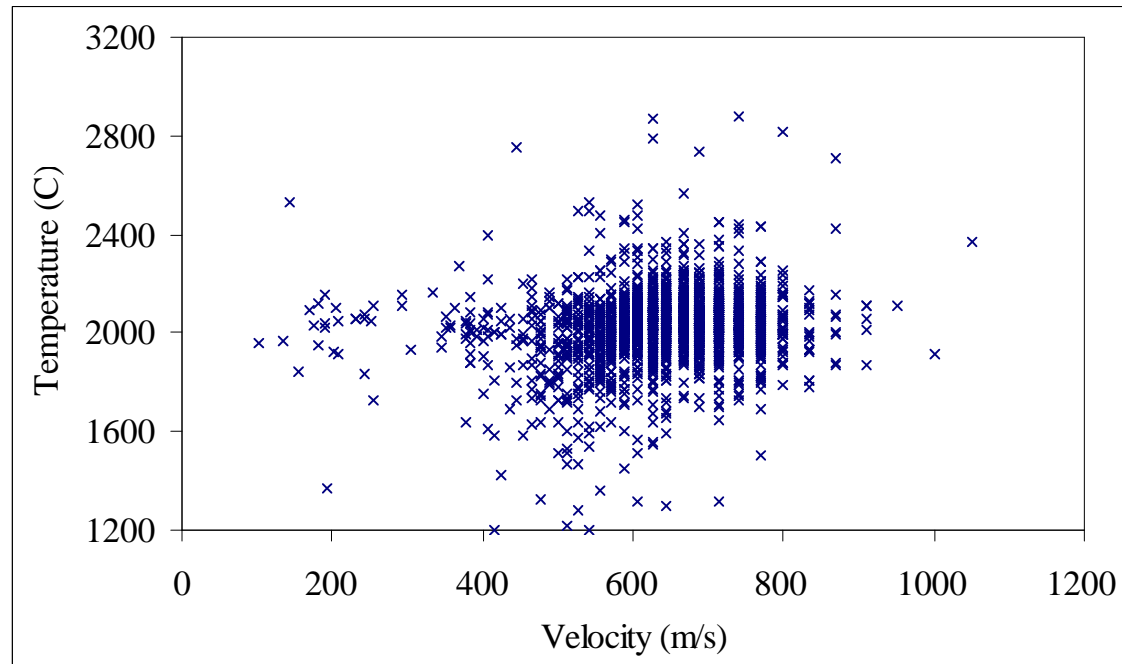
Sensing head





Process monitoring of thermal spraying

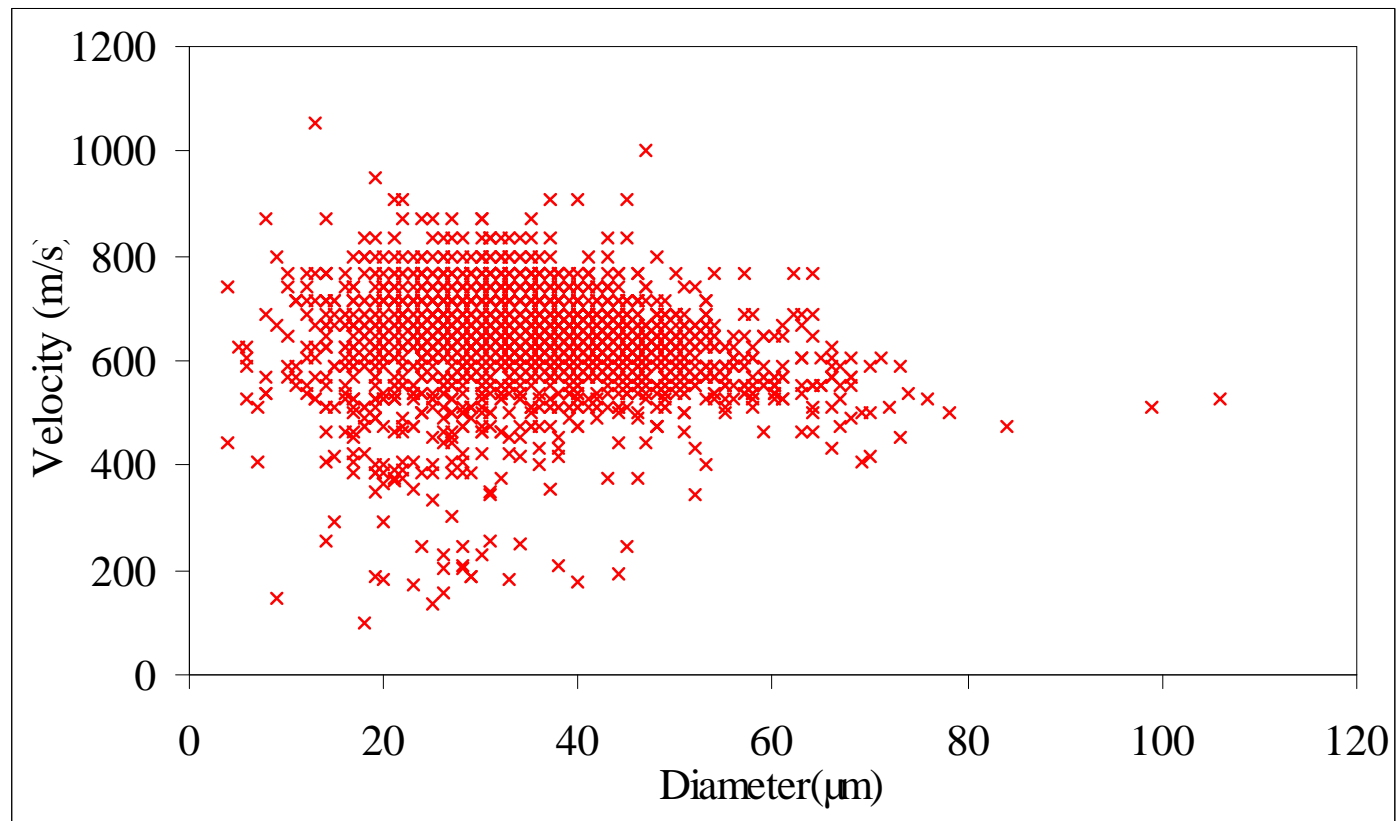
Typical DPV-2000 data: Temperature – Velocity at 300 mm stand-off





Process monitoring of thermal spraying

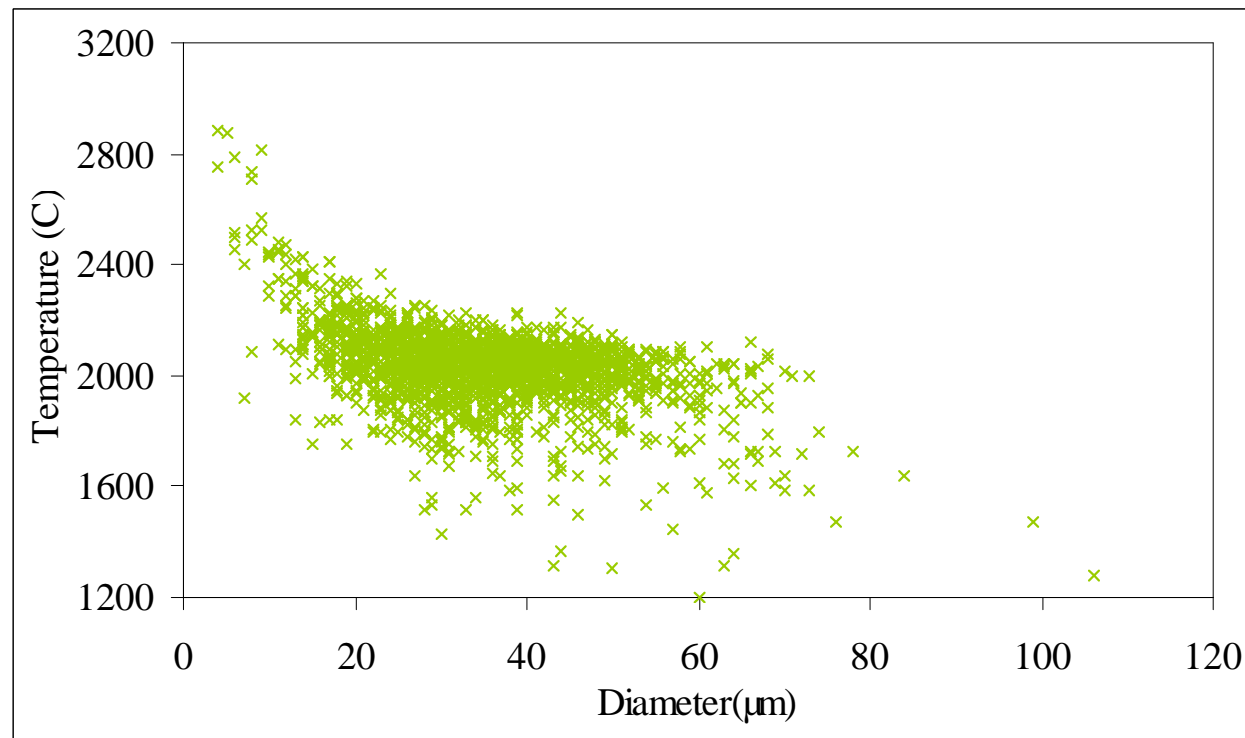
Typical DPV-2000 data: velocity - diameter at 300 mm stand-off





Process monitoring of thermal spraying

Typical DPV-2000 data: temperature – diameter at 300 mm stand-off



Process monitoring of thermal spraying

Can we use the DPV data to help us make coatings better,
faster, cheaper?

Questions we need to answer

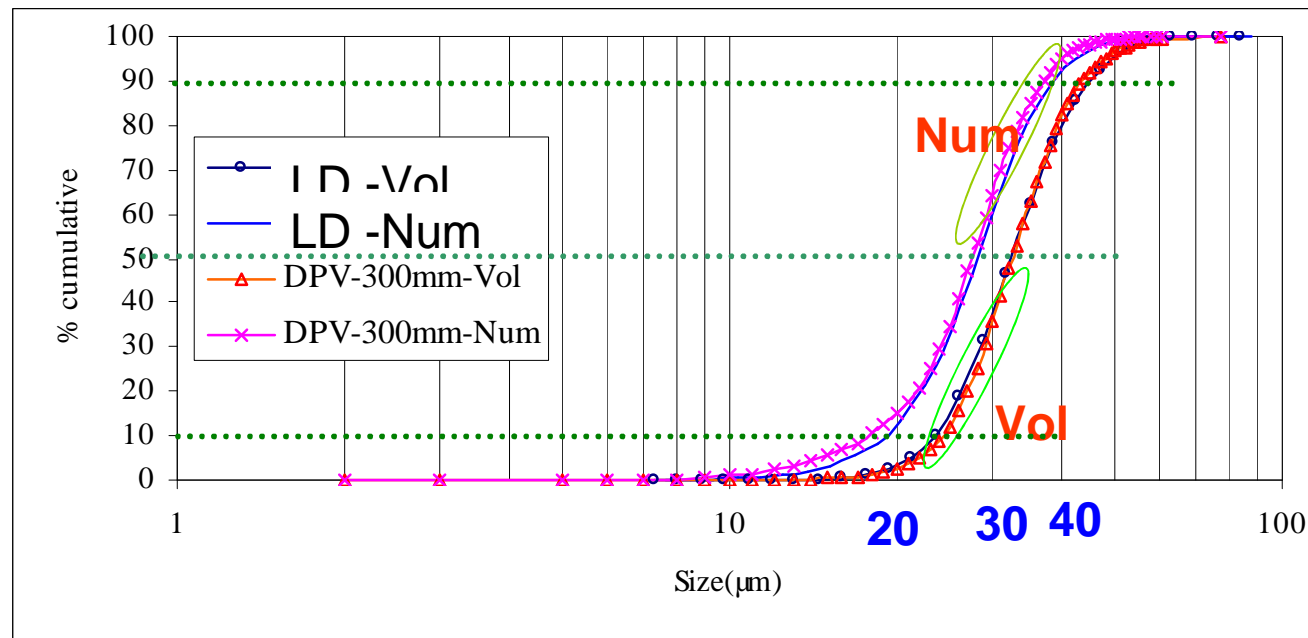
- Is it precise (ie close to correct values)
- Is it accurate (ie reproducible)
- Is it responsive to parameter changes
- How do we relate data to coating formation and ultimately coating properties

Following slides illustrate at least some partial answers



Precision

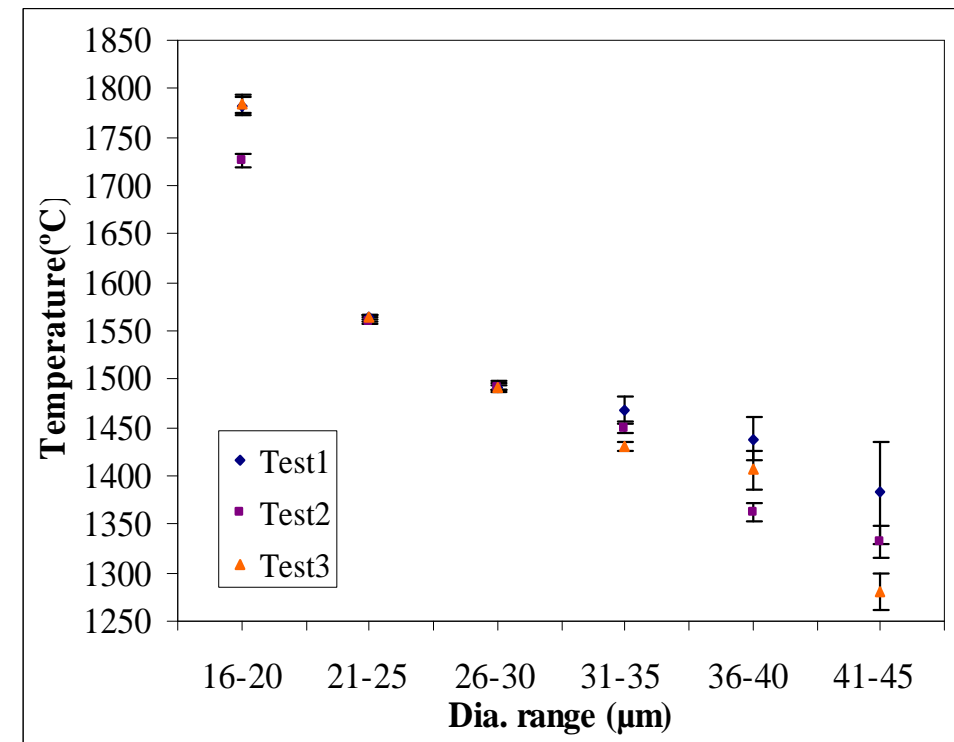
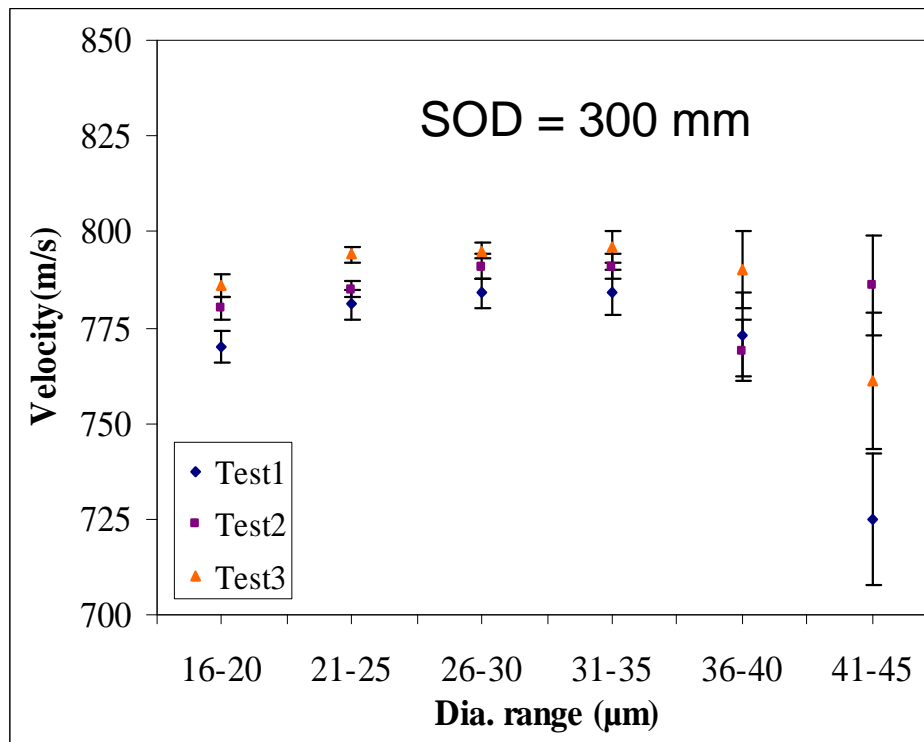
Measurements of particle size distribution in the with the Tecnar in-flight instrument agree well with the size distribution of the original powder measured by laser diffractometry (LD)





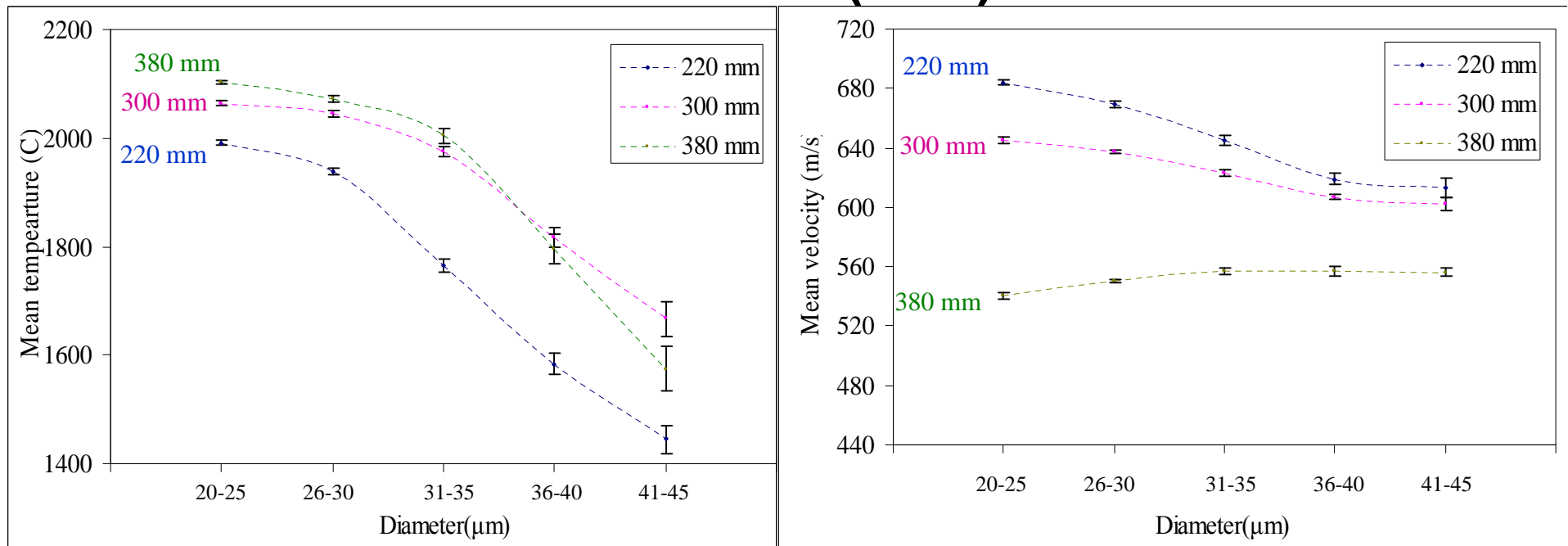
Reproducibility

When data are collected into “bins” of different particle diameter they show reasonable agreement on repeated tests. Trend of falling temperature with increasing diameter fit well with what is expected





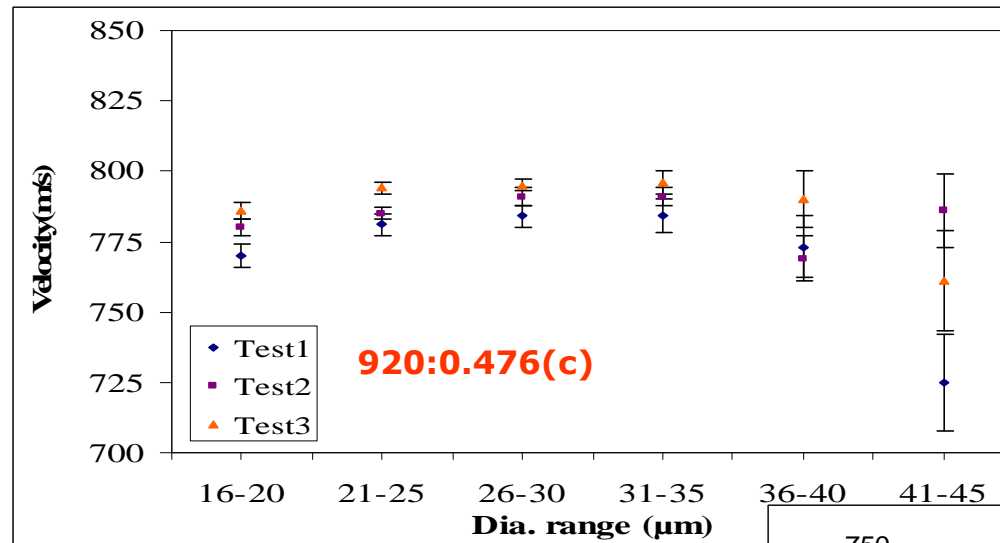
Response to parameter changes – Stand off distance (SOD)



- **Velocity very sensitive to SOD**
- **Velocity not very sensitive to powder particle size**
- **Temperature critically depends on size and but less on SOD**

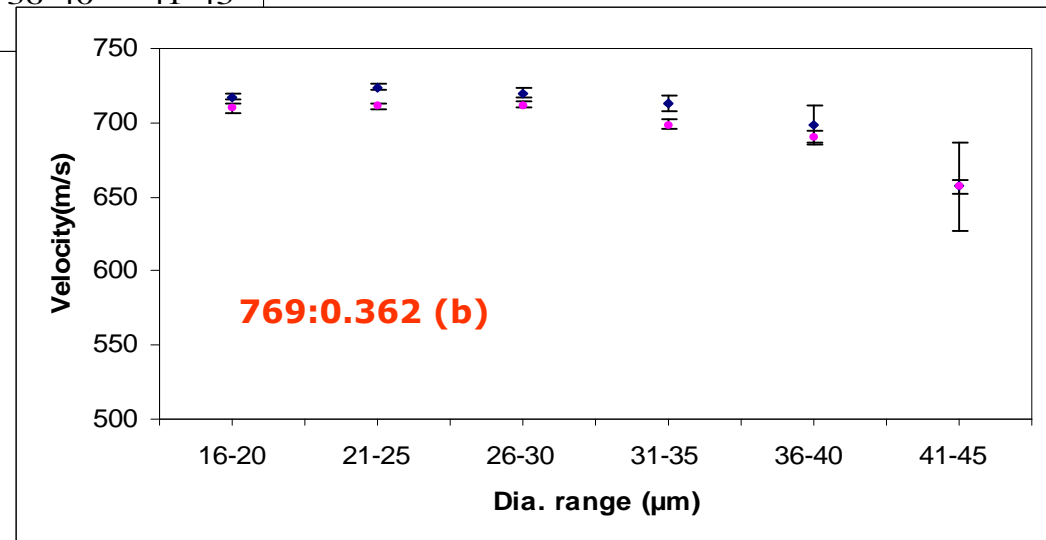


Response to parameter changes – oxygen to kerosene ratios



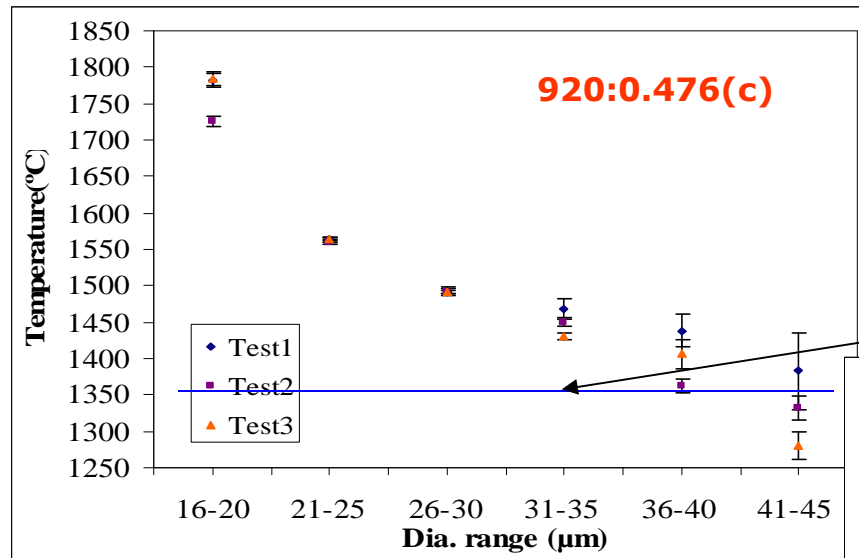
Effect on particle
velocity

Trend is to have lower
velocity in case (b)



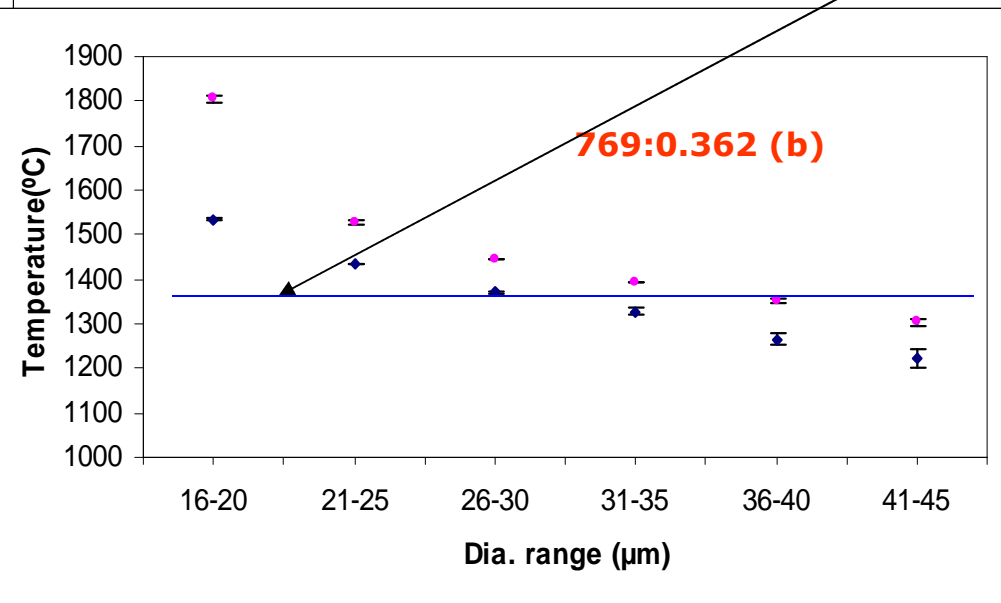


Response to parameter changes – oxygen to kerosene ratios



Effect on particle temperature

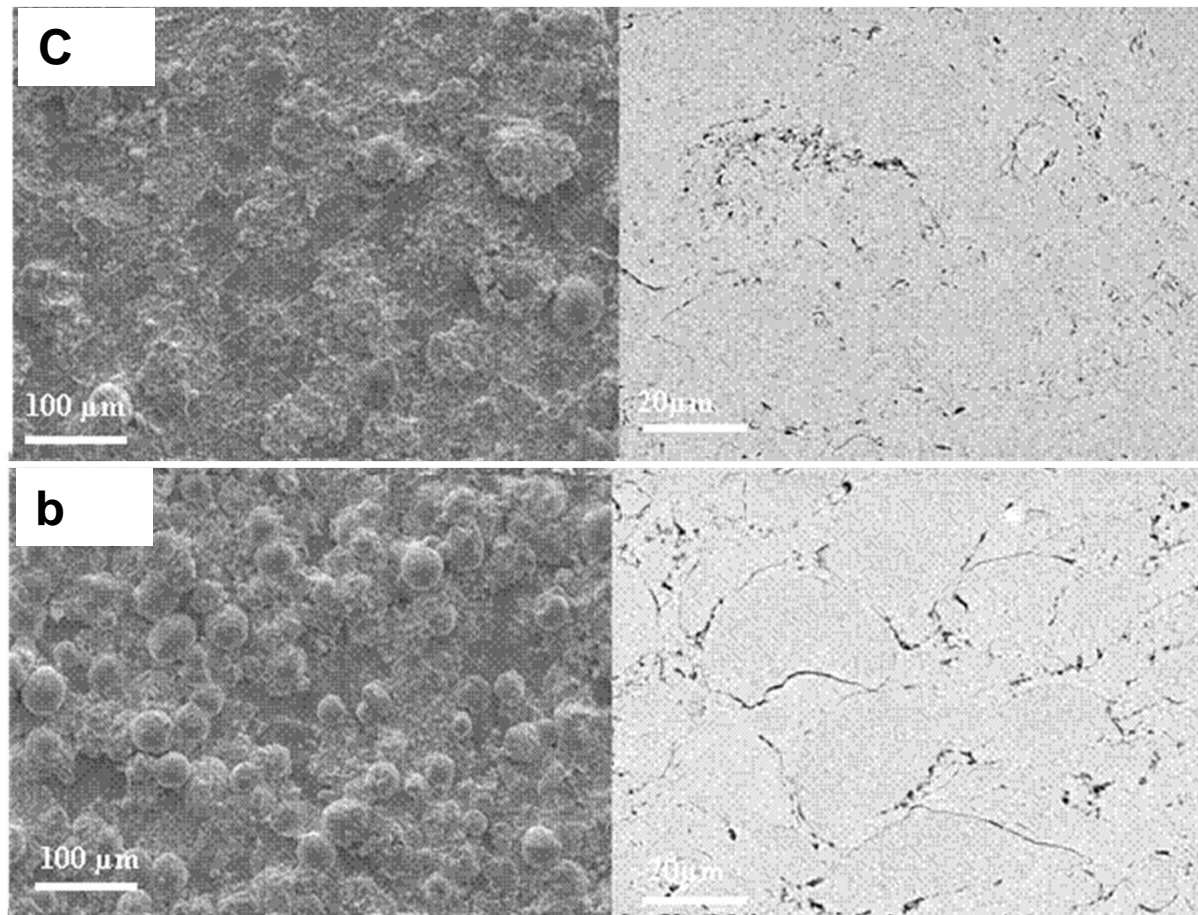
Approx melting
temperature of
alloy.



More un-melted particles in case (b)



Response to parameter changes – oxygen to kerosene ratios



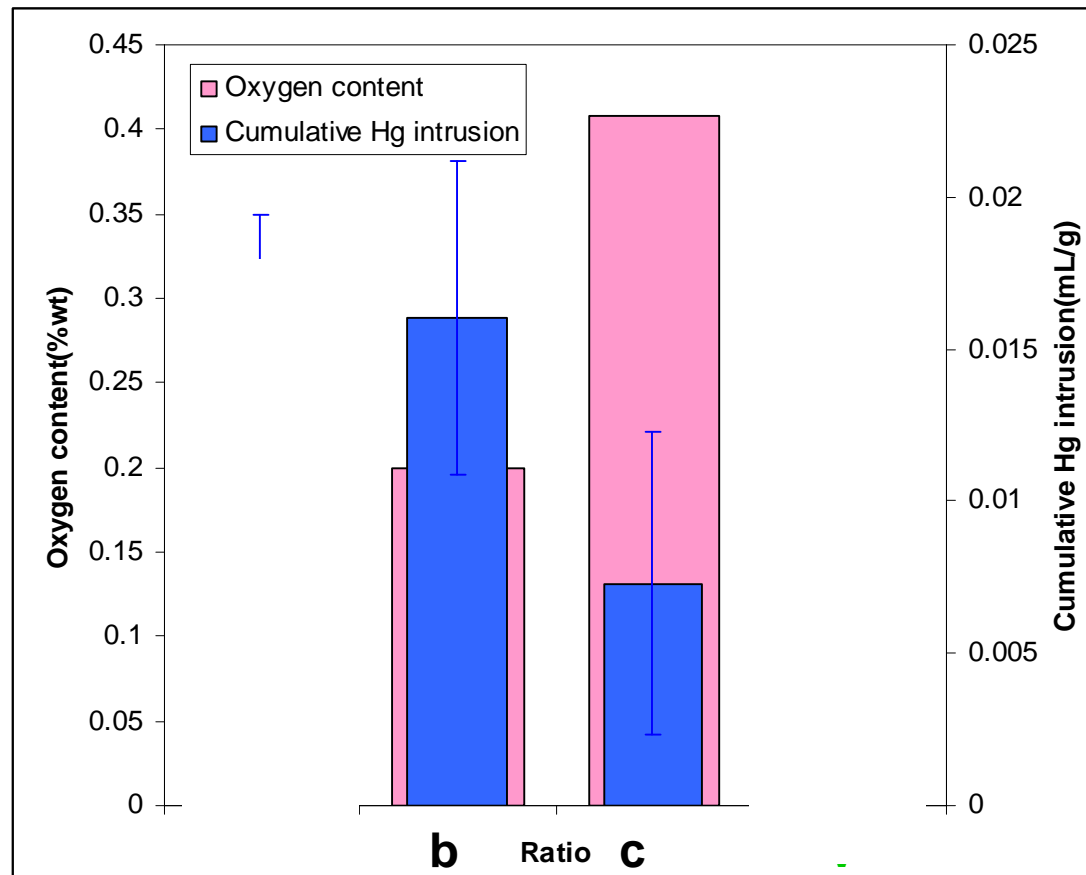
**Effect on
microstructure**

Hotter, higher velocity
powder particles

Colder, lower velocity
powder particles



What parameters are best for this application?



Put simply **b** = colder particles with lower velocity.
So least porosity and most structurally sound coating needs setting **c**

HOWEVER, there is a TRADE-OFF with the other important property which is degree of oxidation during spraying

Case study 2: Process monitoring of thermal spraying using spray diagnostics

CONCLUSIONS

- The link between particle properties and coating microstructures has been clearly demonstrated for a (Ni,Co)CrAlY alloy.
- The particle diagnostics approach provides direct information on how individual parameters affect particle properties (eg fuel, oxygen and powder flows) which DoE does not provide.
- The Tecnar DPV-2000 accurately captures size, temperature and velocity data of individual particles; it requires careful set up and is not, in itself, an industrial control tool.
- The DPV-2000 is not a full field of view instrument so it is time consuming to capture data at various stand-off distances.

OVERALL SUMMARY

- The objective of developing advanced controls is to produce coatings having the same properties day after day or whose properties are within a range of values acceptable for a specific application.
- To reach this goal one needs: reliable spray equipment; consistent feed materials; efficient controllers; properly designed and applied sensing and evaluation techniques.
- A good understanding of the physical and metallurgical processes involved in thermal spraying is essential to implement an adequate control strategy

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