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ARC TRAINING CENTRE
Surface Engineering for Advanced Materials

Australian Government
Australian Research Council

Beautiful Microstructures: Case Histories for Designing the Face.

TRL2 to TRL8: Science Leads to Industrial Applications!

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SWINBURNE UNIVERSITY OF TECHNOLOGY

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IMAT THE VIRTUAL EDITION
INTERNATIONAL MATERIALS APPLICATIONS & TECHNOLOGIES

October 26-28, 2020

Acknowledgement of Country

We respectfully acknowledge the Wurundjeri People, and their Elders past and present, who are the Traditional Owners of the land on which Swinburne's Australian campuses are located in Melbourne's east and outer-east.

We are honoured to recognise our connection to Wurundjeri Country, history, culture and spirituality through these locations, and strive to ensure that we operate in a manner that respects and honours the Elders and Ancestors of these lands.

We also acknowledge the Traditional Owners of lands across Australia, their Elders, Ancestors, cultures and heritage.

2

DEDICATION

Health Care Staff and Essential Service Personnel have demonstrated their commitment and self-sacrifice.

They are working at the front line of the 2020 global pandemic during a crucial time of need.

Their devotion and dedication is inspirational.

We are all stronger when we work together.

THANK YOU!

Image courtesy of GoGraph

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Beautiful Microstructures: Case Histories for Designing the Face.

TRL2-3: Science

Surface Engineering for Advanced Materials

1. **"Beauty is in the eye of the beholder" (Plato).**
2. "What you see is what you get" Really?
3. Will the real customer stand up? What does the customer want?
4. What's the Risk?
5. Who do we work for? Crystal balling (connecting dots)

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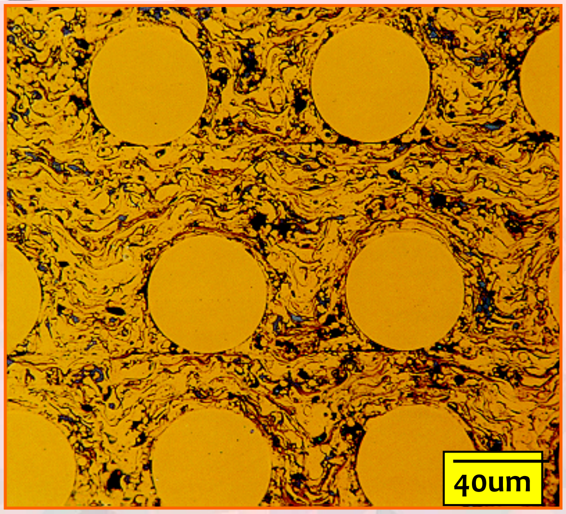
CrNi Fibers in a NiCrAlSi Matrix



Ref: H. Kern, R. Kaczmarek and J. Janczak, "Thermally Sprayed Fiber-Reinforced MMCs", JTST, 1[2] (1992) 137-145.

Courtesy of Prof. H-D. Steffens, University of Dortmund.

Why Use Composites?

- Create special functional properties
- Increased stiffness and dimensional stability
- Increased heat distortion temperature.
- Modified dielectric properties
- Tribological response

Thermal spray can deposit composite coatings and free-standing components

The 'Swiss army knife' of surface engineering

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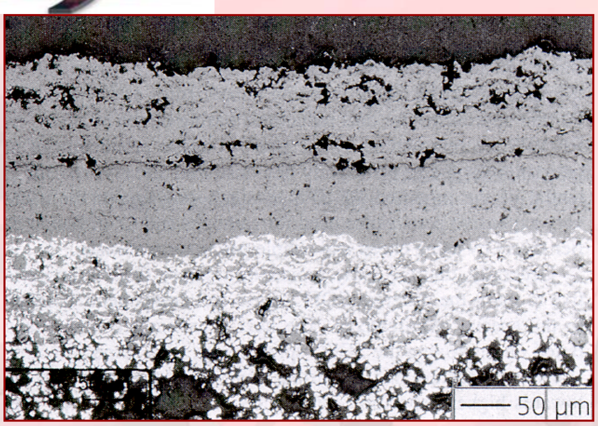
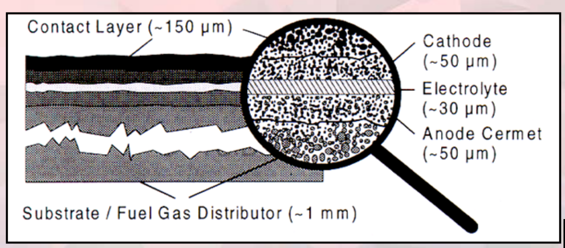
Solid Oxide Fuel Cell Microstructure



Perovskite cathode (top layer)

YSZ electrolyte (middle)

Ni/YSZ anode (bottom)



Ref: "SOFC Components Production – An Interesting Challenge for DC_ and RF-Plasma Spraying", R. Henne, G.Schiller, V. Borck, M. Mueller, M. Lang and R. Ruckdaschel, P. 933-938 of "Thermal Spray Meeting the Challenges of the 21st Century, Ed. C. Coddet, Pub. ASM, 1998, OH-USA.

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Beautiful Microstructures: Case Histories for Designing the Face.

TRL4, 5: Science Leading to Technology

Surface Engineering for Advanced Materials

1. "Beauty is in the eye of the beholder" (Plato).
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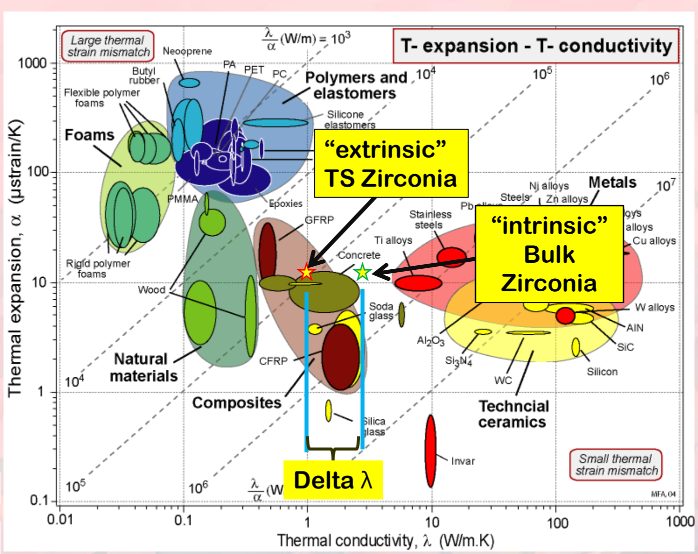
Thermal Spray Properties are Extrinsic



GRANTA

UNIVERSITY OF CAMBRIDGE

New approaches to Materials Education - a course authored by Mike Ashby and David Cebon, Cambridge, UK, 2007

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The Beauty of Non-Equilibrium Phases

- **Deposits experience rapid solidification**
  - Formation of metastable phases is common.
  - Supersaturation effects, change in crystal structure, amorphicity etc.
  - e.g. gamma- Al_2O_3, supersaturation in NiCr eutectic, amorphous phase in hydroxyapatite, etc.
- **Characterization of deposits is challenging**
  - Conventional and synchrotron based diffraction.
  - Microdiffraction for phases within a single splat.
  - TEM, including convergent beam techniques.
  - Neutron diffraction experiments.
  - Mechanical testing.
  - Corrosion studies.

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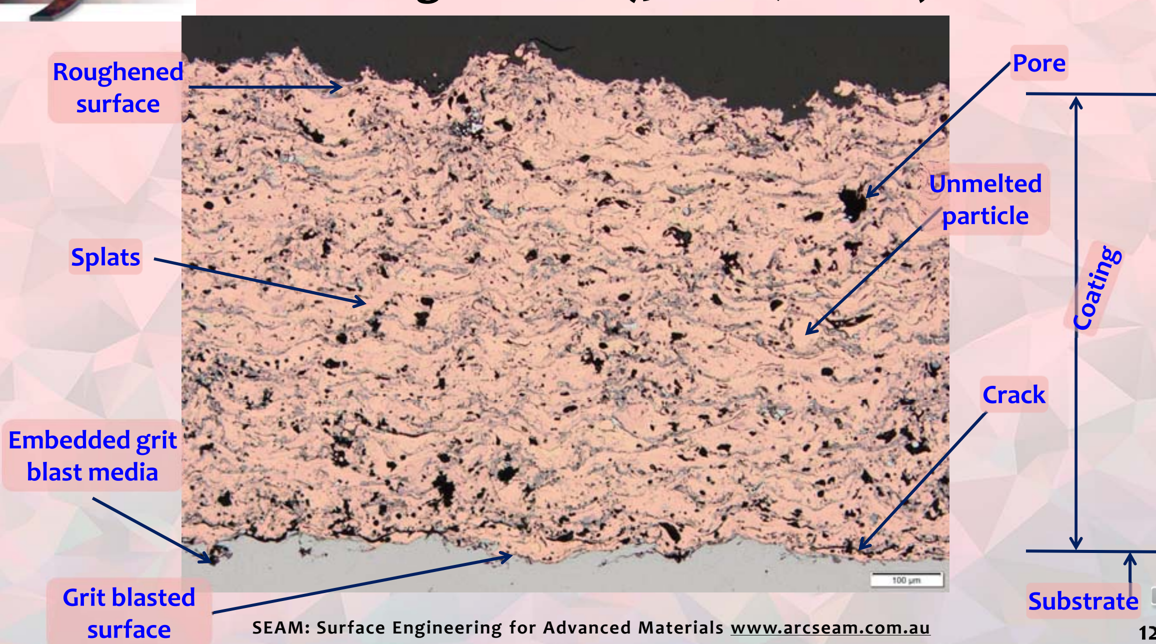
Coating: Bronze (90% Cu, 10% Al)



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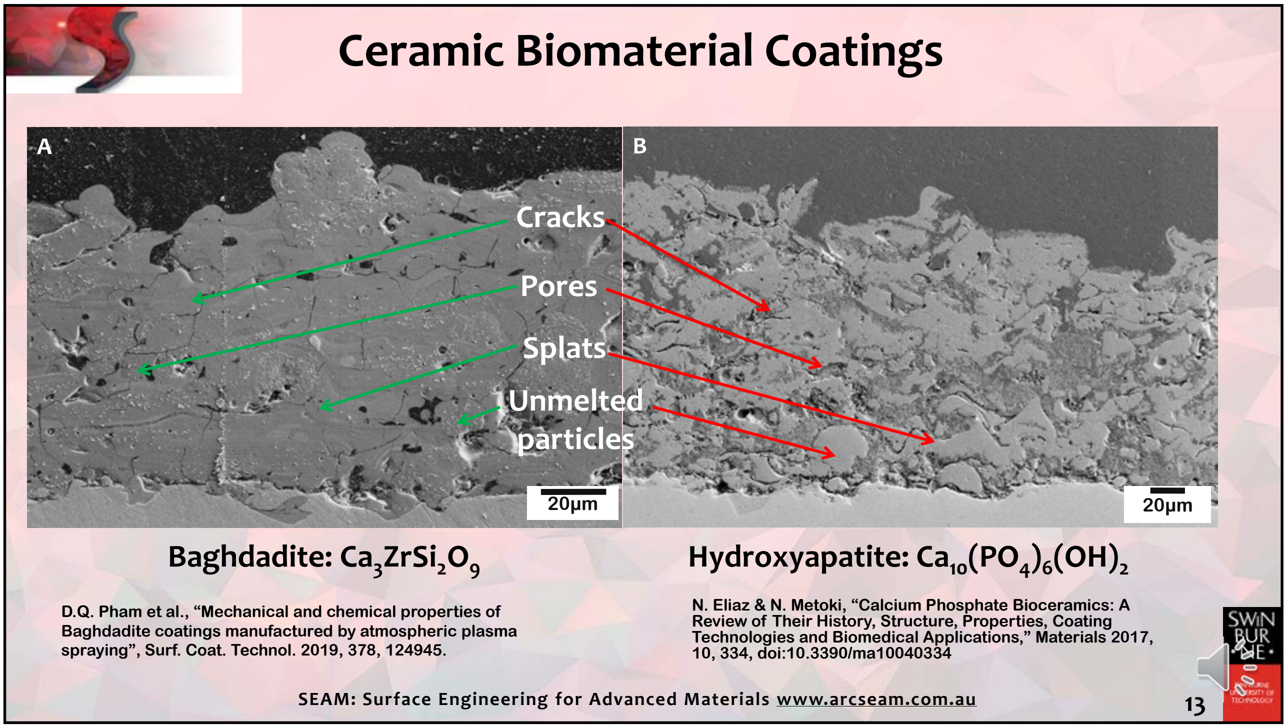

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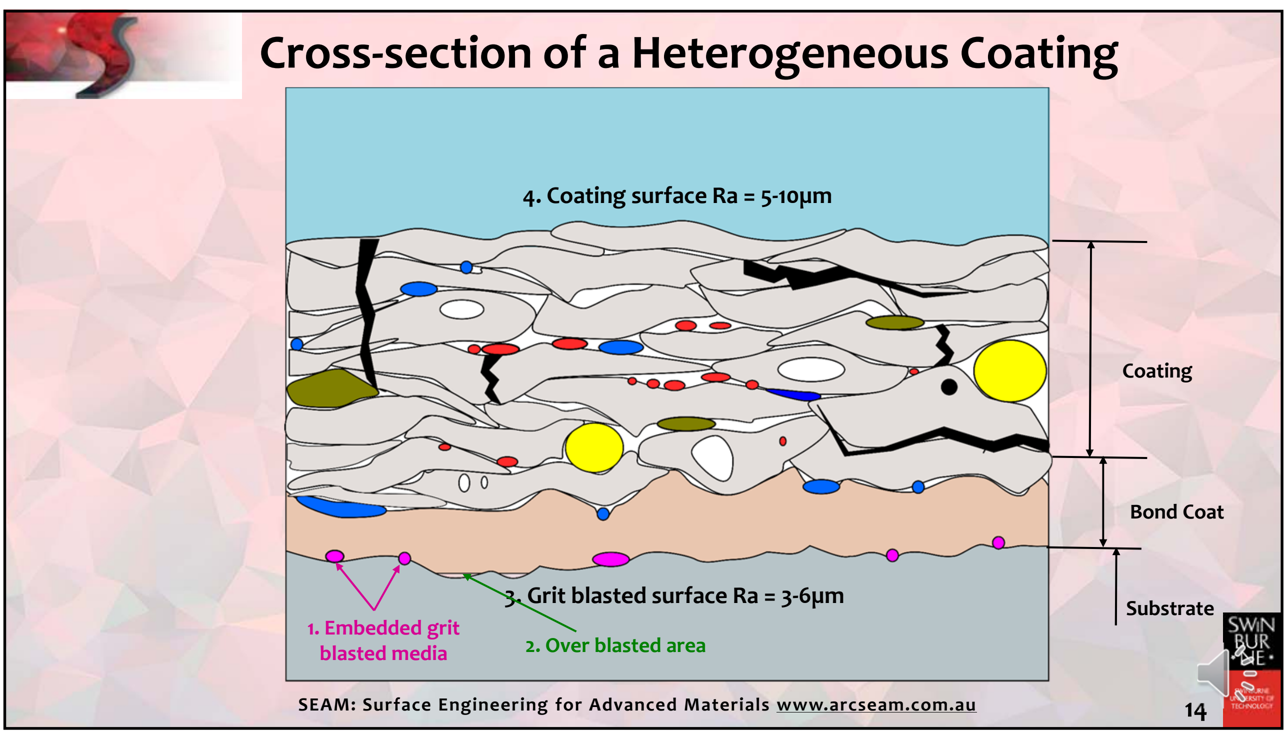

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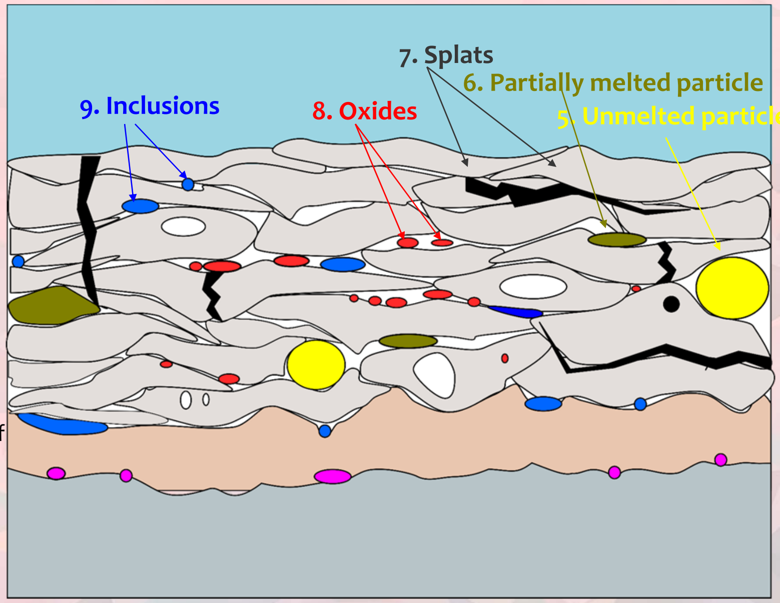
Defect Taxonomy: Degree of Melting



J. Madejski, 'Solidification of droplets on a cold surface', International Journal of Heat and Mass Transfer 1976;19:1009.

A.S.M. Ang et al., 'Modeling the Coverage of Splat Areas Arising from Thermal Spray Processes', J. AM. Ceram. Soc., 95 (2012) 15672-1580

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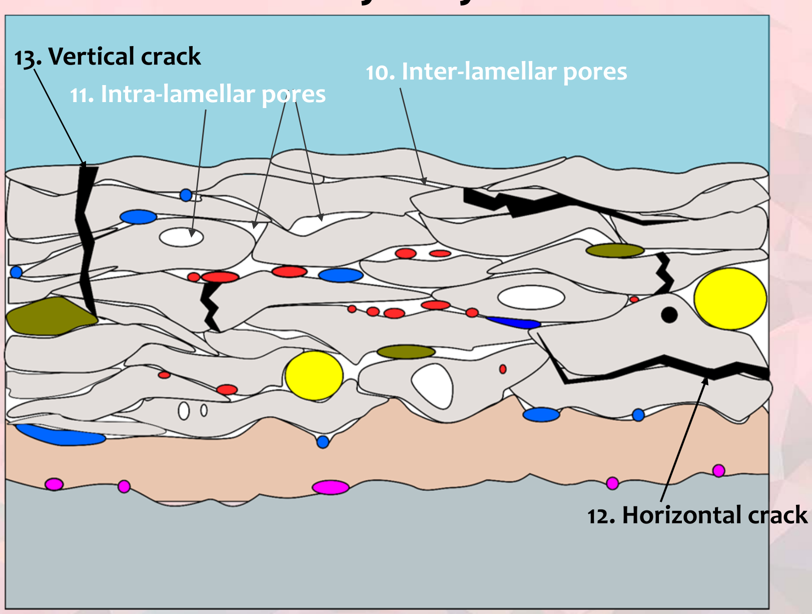
Defect Taxonomy: Physical Interactions



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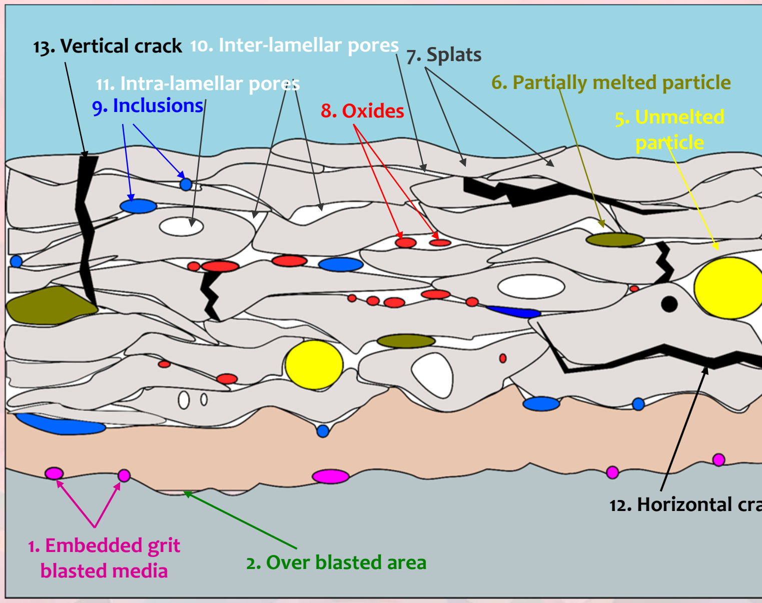
Chaotic Microstructures



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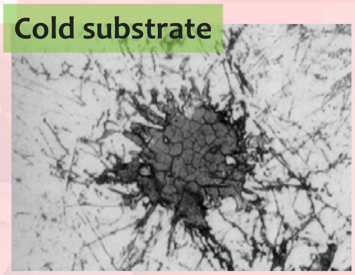
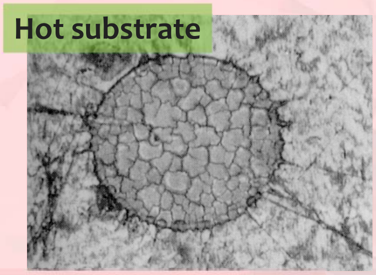
Splat Microstructures

Cold substrate

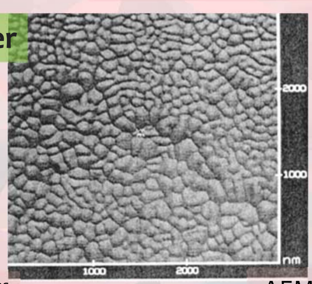


Hot substrate



Effect of substrate temperature on splat morphology (Alumina)

Center



Periphery

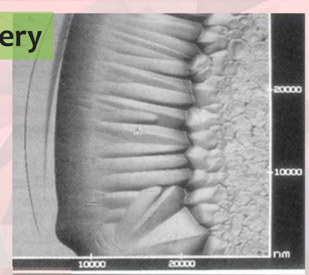


Photo courtesy U. Limoges

AFM analysis of splats (Alumina)

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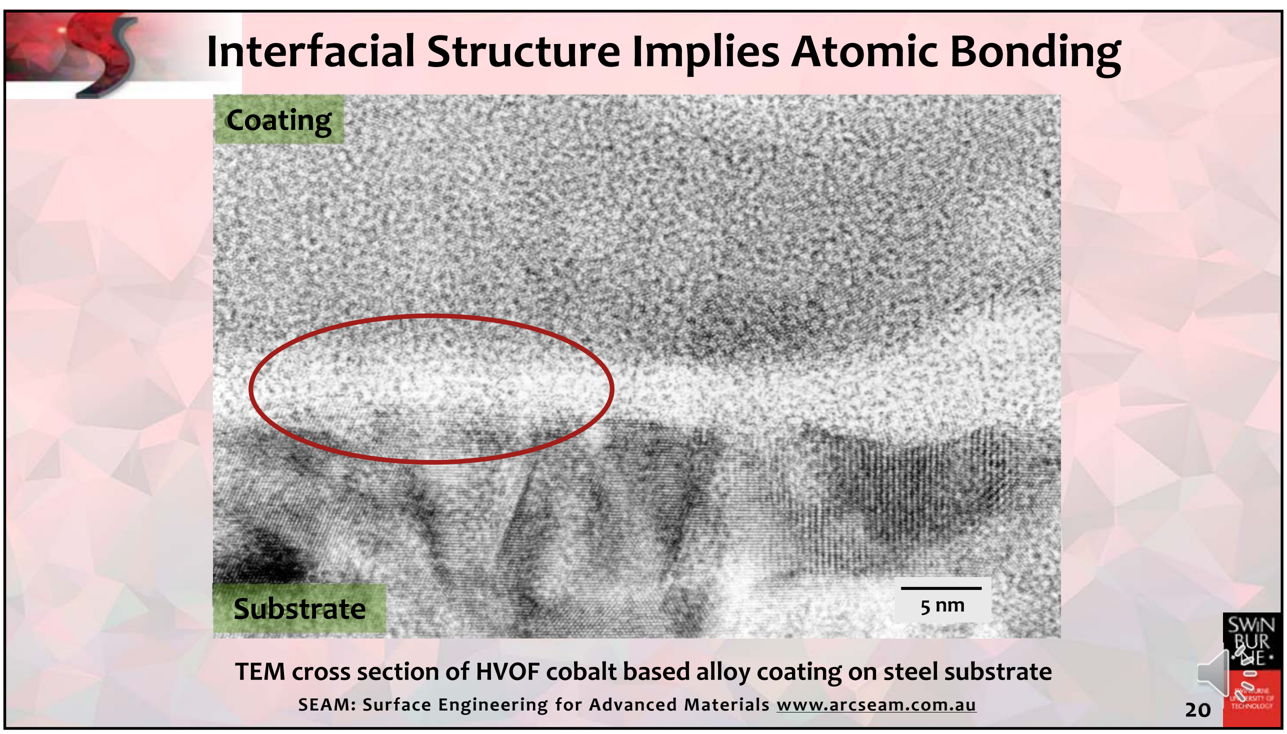

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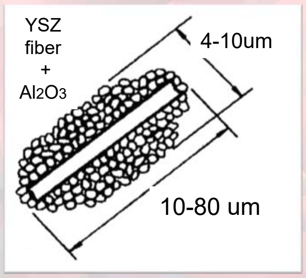
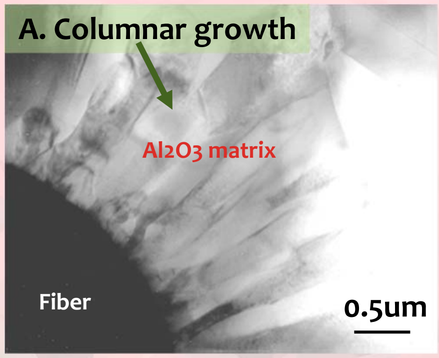
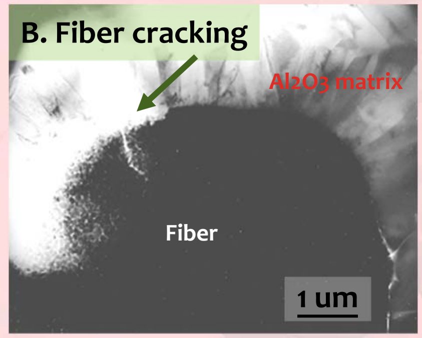
Al_2O_3 Matrix & Yttria Stabilized Zirconia Fiber

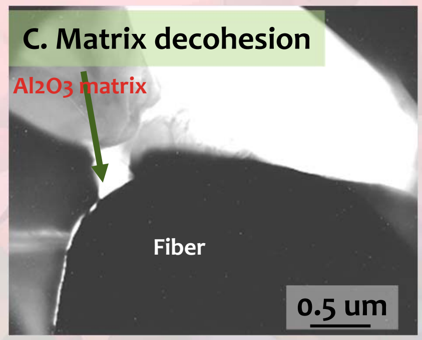
C.C. Berndt, "Composite Plasma Sprayed Coatings", 12th ITSC, 1989, Paper 2, Vol, 1, Pub. TWI, London.

C.C. Berndt, "Toughening of Thermally Sprayed Coatings", Materials Sci. Forum, 34-36 (1988) 469-474.









Local microstructural influences cooling conditions experienced by the matrix.

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An Interim Summary: We are Explorers

1. TRL2&3 (science) is a fertile area.
2. Processing conditions are critical
3. Control of the process is imprecise (at best)
4. Thermal spray coatings are defective

HOWEVER

1. How do we transition into technology & engineering?
2. Who is the real customer?
3. What does the customer want? (TRL7&8)

PS: The microstructures are very, very beautiful!

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Beautiful Microstructures: Case Histories for Designing the Face.

TRL2 to TRL8: Science Leads to Industrial Applications!

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1. "Beauty is in the eye of the beholder" (Plato).
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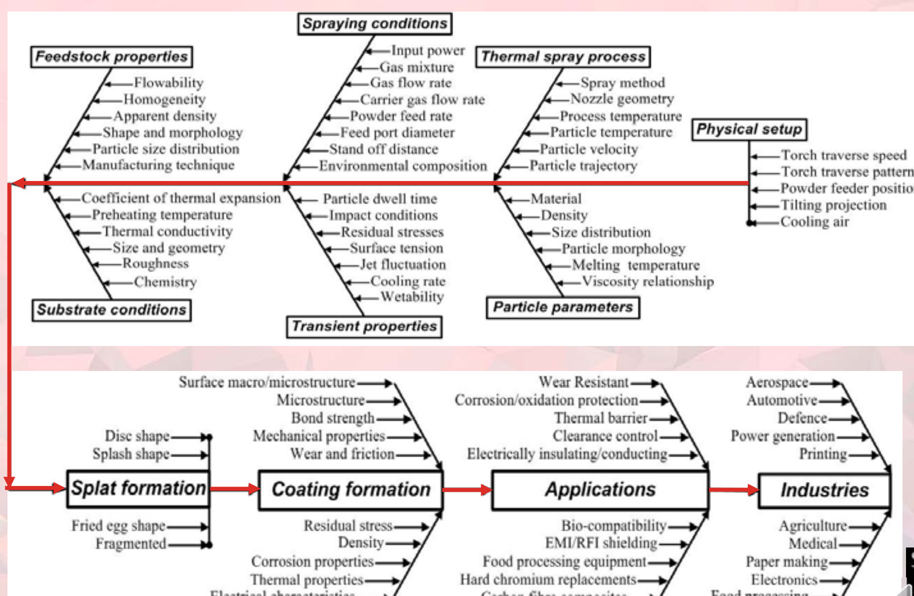
There are too many variables.….



…. Influencing Coating Quality and Applications

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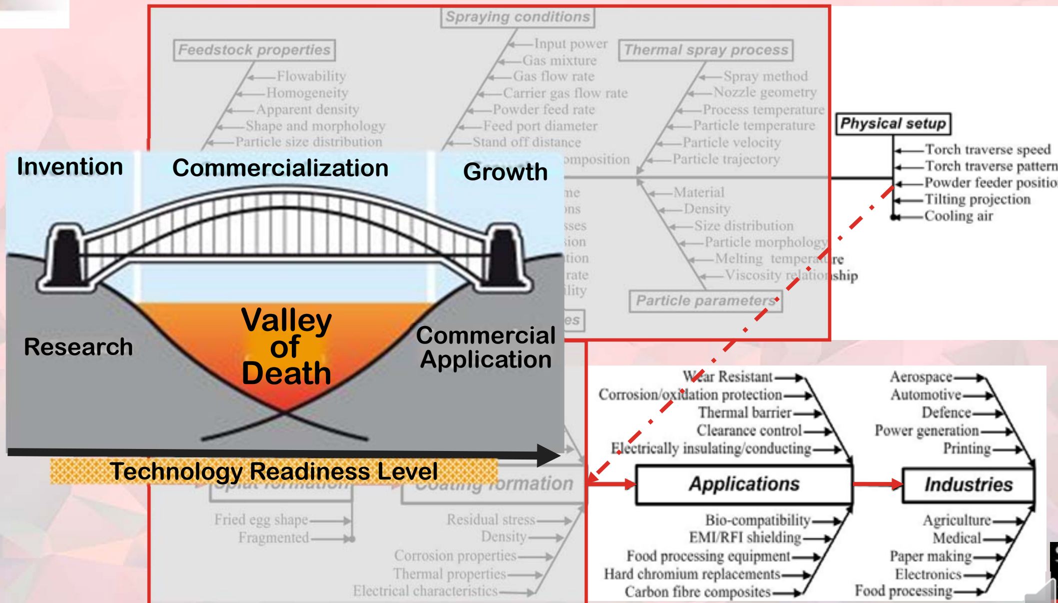
How to Jump this VALLEY of DEATH!



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The influence of Key Thermal Spray Variables on the Microstructure

The 'Swiss army knife' of surface engineering

	CAUSE	EFFECT
ID	Process Tool	Influence on coating microstructure
1	Particle size	Direct relationship to microstructural sizes
2	Particle velocity	High velocity = fine microstructure & high density
3	Substrate temperature	Higher temperature = flatter splats
4	Substrate thermal conductivity	Conductive substrate enhance nucleation = finer microstructure
5	Coefficient thermal expansion (CTE	Thermal residual stresses due to CTE mismatch between coating and substrate
6	Stand off distance	Influences temperature and velocity, which controls the splat cohesion
7	Cooling rate	Types and proportions of phases
∞	… what tool is best?	.. what microstructure presents the desirable property?

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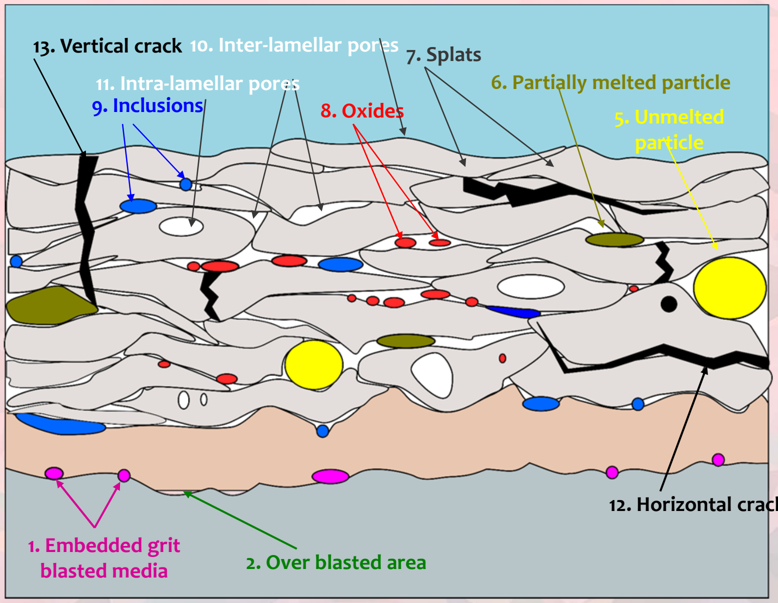
Microstructures that are Rich with Features



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The Exquisite Palette of Microstructures for Engineers

	TAXOMONY	TOOL	OUTCOME
ID	Microstructural Artefact	Impact of the Processing Tool	Effect on Material Properties
	Substrate roughness	Grit blasting pressure and technique	Adhesion strength
1	Over blasted area	Grit blasting pressure and technique	Lower roughness & poor adhesion
3	Embedded grit blast media	Grit blasting pressure and technique	Surface contamination, corrosion sites, poor adhesion
	Coating roughness	Feedstock size	
5	Unmelted particles	Thermal spray parameters (too cold)	Variable physical properties
6	Partially melted particles	Thermal spray parameters (too cold)	Variable physical properties
8	Oxide particles	Thermal spray parameters (too hot)	Micro-hardness increase
9	Inclusions	Thermal spray parameters, feedstock quality	Local corrosion
10, 11	Porosity	Standoff distance	Modulus decreases
13	Vertical cracks	Compressive residual stresses	Hardness decreases
12	Horizontal cracks & lamellar	Tensile residual stresses	delamination or separation
	……….. etc.		

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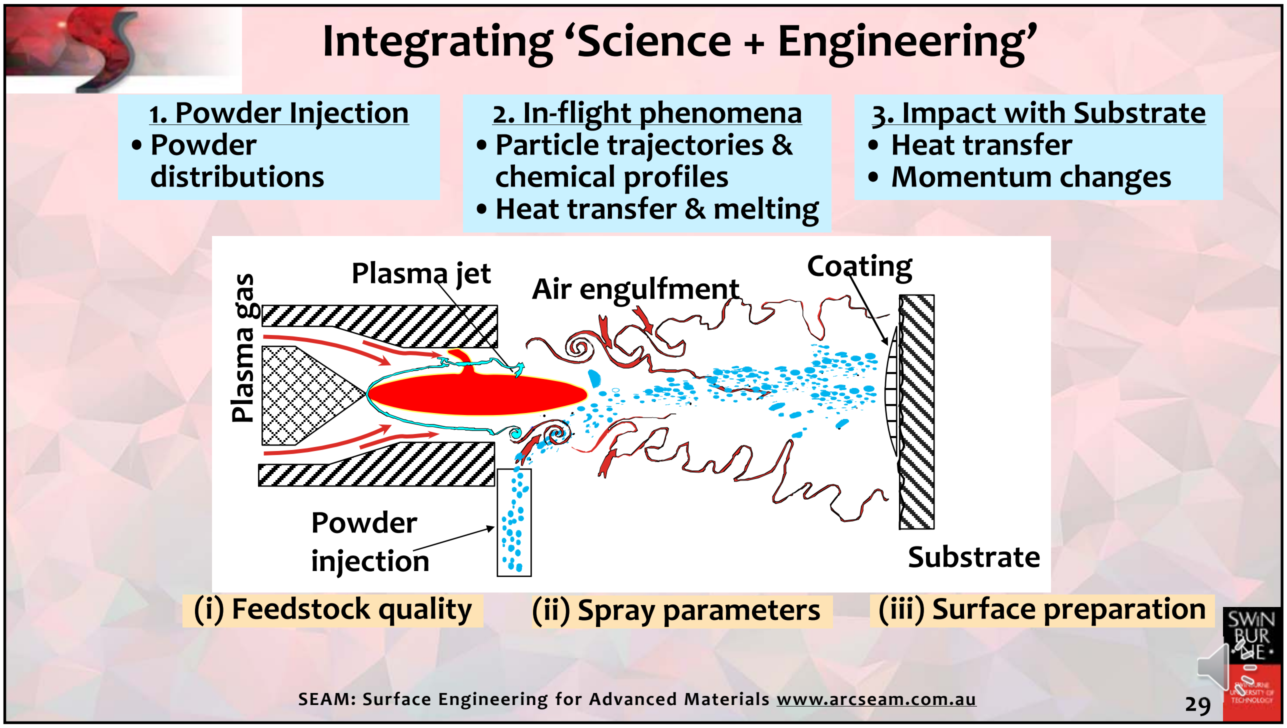

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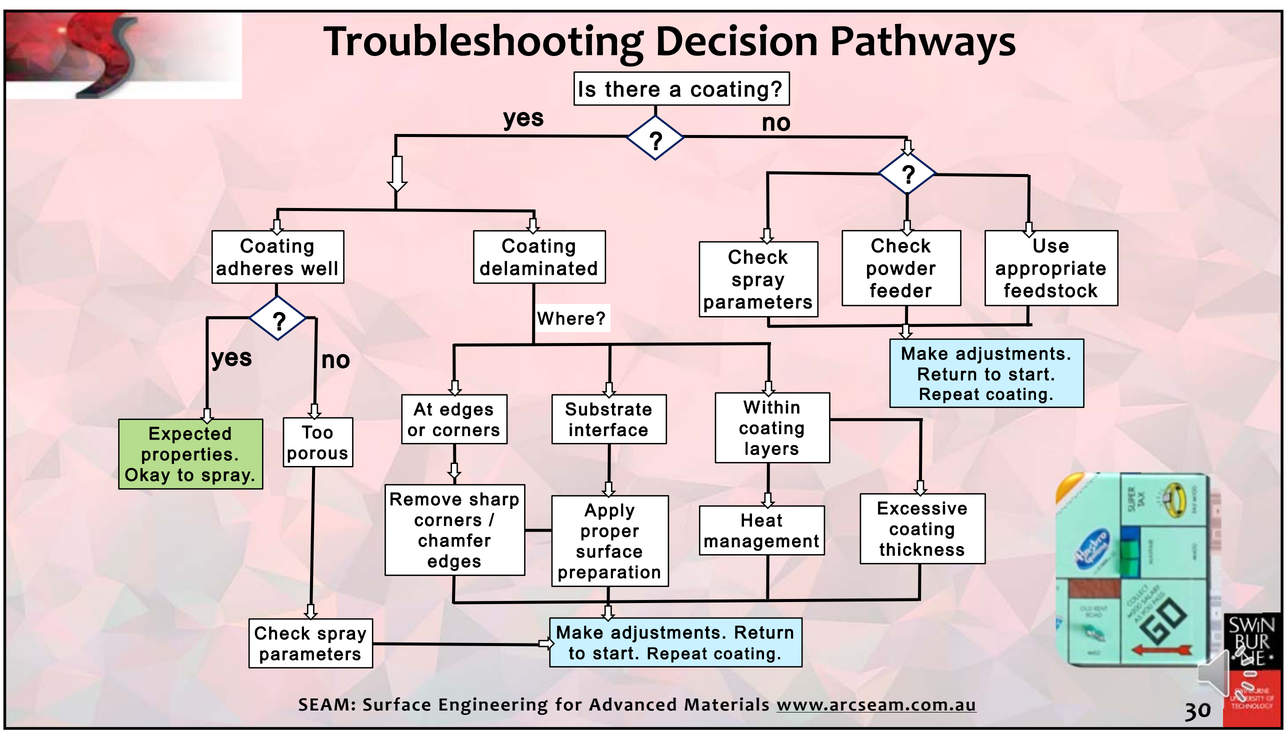

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Beautiful Microstructures: Case Histories for Designing the Face.

TRL5 to TRL6: Science Leads to Industrial Applications!

Surface Engineering for Advanced Materials

1. “Beauty is in the eye of the beholder” (Plato).
2. “What you see is what you get” Really?
3. What does the real customer want?
4. **New Materials … What’s the Risk?**
5. Connecting the dots to the future? Crystal balling.

“A ship is always safe at the shore - but that is NOT what it is built for.
Albert Einstein

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High Entropy Alloys: Displacing traditional bond coats

Attributes of HEAs

1. Less weight
2. Excellent high temperature properties
3. Low cost constituents

A.S.M. Ang et al., ‘Plasma-sprayed high entropy alloys: microstructure and properties of AlCoCrFeNi and MnCoCrFeNi’, Met. Mater. Trans. A, 46A (2015) 791

A. Anupam et al.; ‘Understanding the Microstructural Evolution of High Entropy Alloy Coatings Manufactured by Atmospheric Plasma Spray Processing’, App. Surface Sci., (October 2019), 505:144117 DOI: 10.1016/j.apsusc.2019.144117

B.S. Murty, J.W. Yeh, S. Ranganathan, P.P. Bhattaharjee, High-Entropy Alloys, 2nd edition, Elsevier (2019)

A. Meghwal et al.; ‘Thermal Spray High-Entropy Alloy Coatings: A Review’, JTST 29 (2020) 857-893

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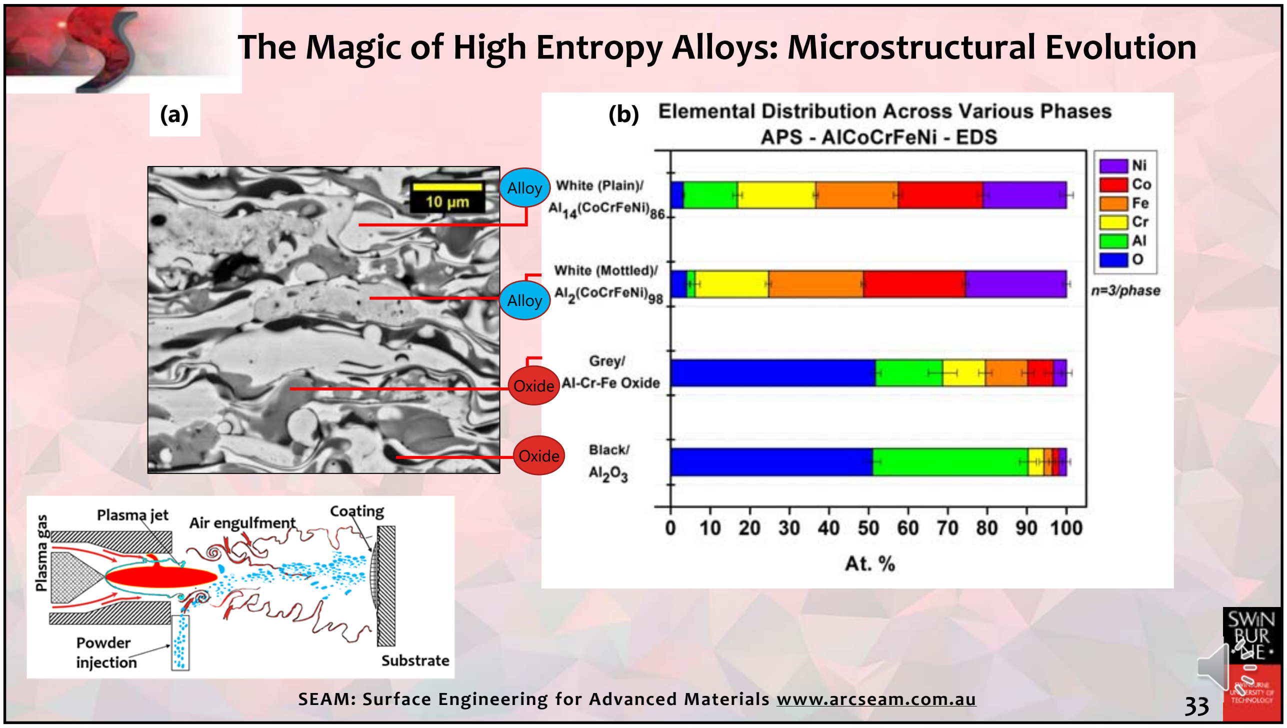

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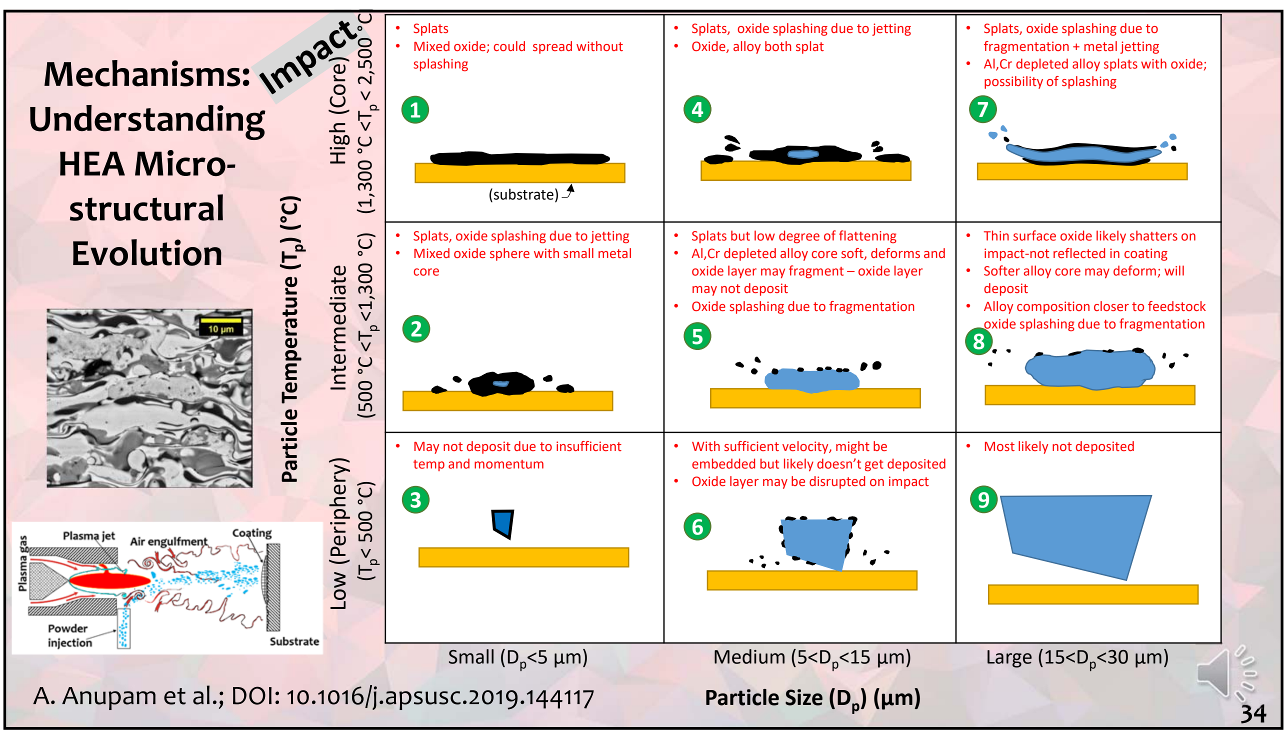

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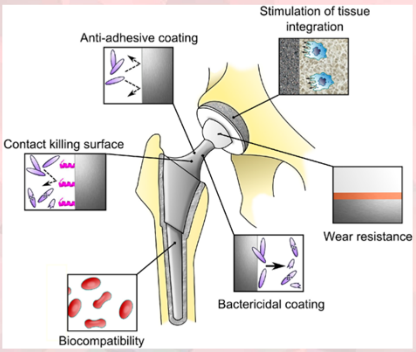
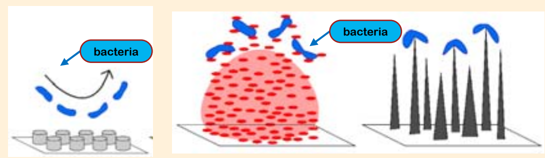
Antimicrobial Coatings: More Beautiful Microstructures

Issues of current implants:
- Insufficient mechanical properties.
- Risks of infection & low wound healing rate.

Outcomes: New coatings for implants to …
- Improve mechanical properties of the coating
- Enhance biocompatibility, bone cell proliferation and osseointegration
- Possess antimicrobial properties



Strategies for antibacterial coatings



Surface Topography | Biocide Releasing | Contact Killing

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J. Gallo et al., Int J Molecular Sciences 15(8) (2014) 13849-13880.
S. Kurtz et al., JBJS 89(4) (2007) 780-785.

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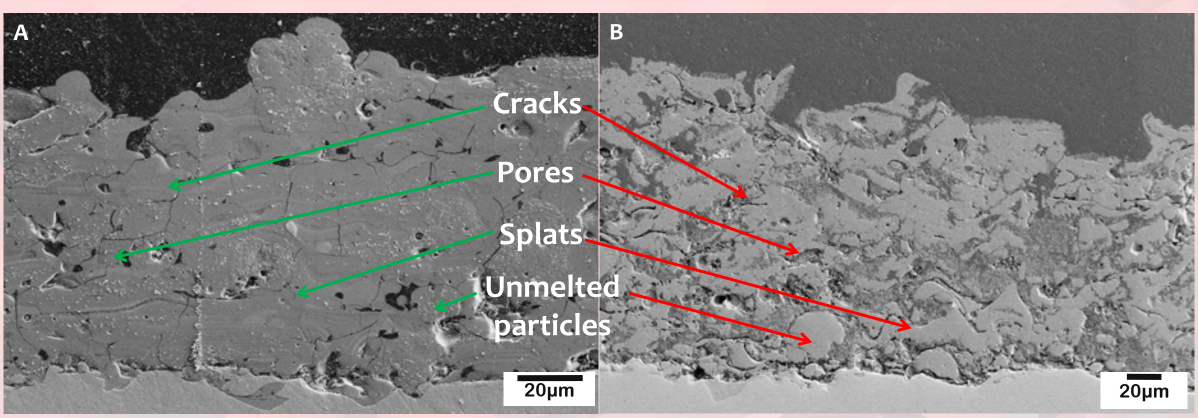
Ceramic Biomaterial Coatings



Baghdadite: $Ca_3ZrSi_2O_9$

Hydroxyapatite: $Ca_{10}(PO_4)_6(OH)_2$

D.Q. Pham et al., "Mechanical and chemical properties of Baghdadite coatings manufactured by atmospheric plasma spraying", Surf. Coat. Technol. 2019, 378, 124945.

N. Eliaz & N. Metoki, "Calcium Phosphate Bioceramics: A Review of Their History, Structure, Properties, Coating Technologies and Biomedical Applications," Materials 2017, 10, 334, doi:10.3390/ma10040334

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Baghdadite vs HAp-Nanoindentation

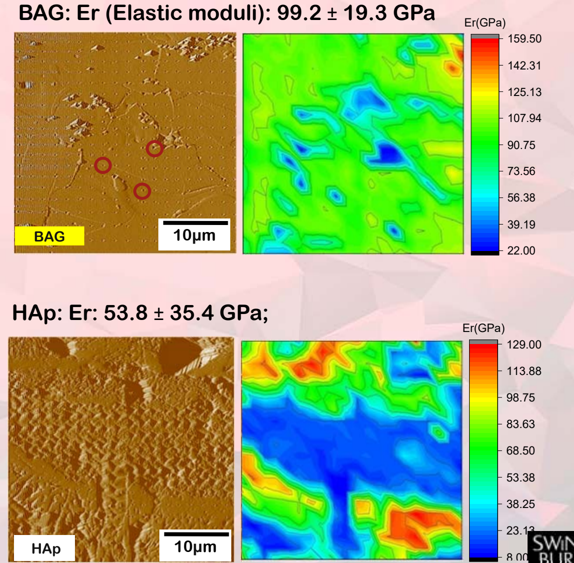
- Baghdadite coating: Uniform structure with more consistent distribution of elastic moduli.
- Baghdadite coating: Significantly higher results of elastic moduli.

Method: XPM (extreme property mapping) to study the distribution of hardness and elastic modulus.

Load 1000µN, 1.5um gap between indents, 400 indents.

BAG: Er (Elastic moduli): 99.2 ± 19.3 GPa

HAp: Er: 53.8 ± 35.4 GPa;



D.Q. Pham et al., “Mechanical and chemical properties of Baghdadite coatings manufactured by atmospheric plasma spraying”, Surf. Coat. Technol. 2019, 378, 124945.

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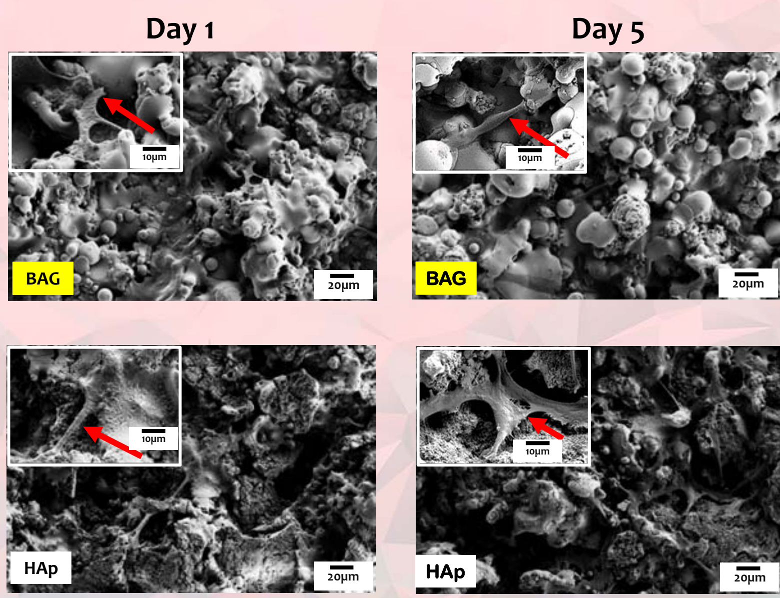
Baghdadite vs HAp-Cell Attachment and Proliferation (MG-63)

- MG-63 cells successfully colonized and proliferate onto the coating surfaces.
- Red arrows indicated the MG-63 cells penetrated into microstructures of BAG and HAp.



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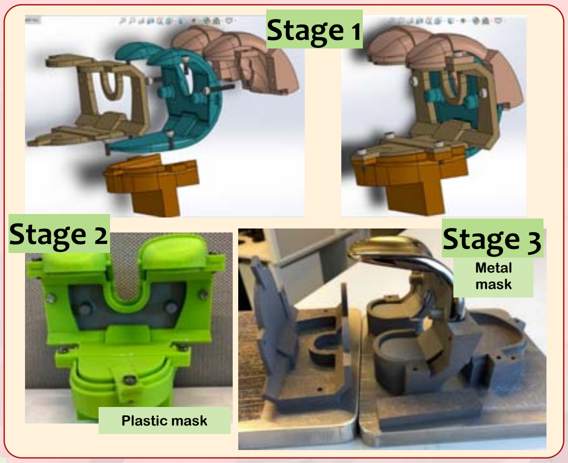
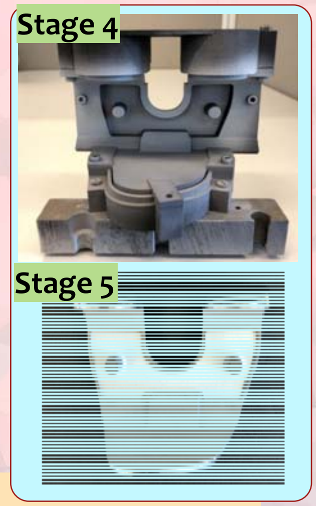
Coating of Knee Implant

ARC Training Centre for **Innovative BioEngineering**

Allegra ORTHOPAEDICS Human Movement Technology

PETER BREHM Die Präzision in Titan für den Menschen





Outcomes: New coatings for implants to …

- Improve mechanical properties of the coating
- Enhance biocompatibility, bone cell proliferation and osseointegration
- Possess antibacterial properties

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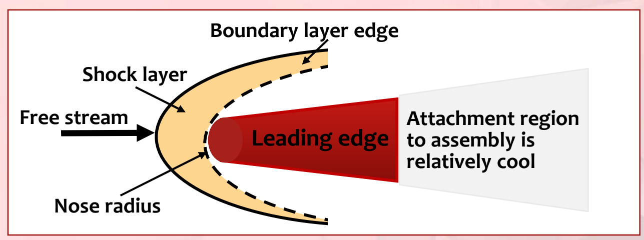
Thermal Protection System (TPS)

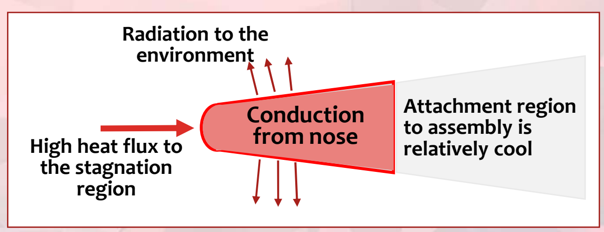
At flight speeds > Mach 5:

- A high enthalpy bow shock forms at the leading edge regions (LERs) of hypersonic aircraft
- Convection and chemical heating produces surface temperatures >2000 °C



Strategy: Thermal protection system (TPS) surface coatings

- Materials with high melting temperatures (>3000 °C) and high thermal conductivity
- High thermal conductivity to bleed heat from at-risk sharp profile LERs



Note: Design criteria for TPS is different than for a Thermal Barrier Coating

AFSOR Award Number FA2386-18-1-4119

T. Squire ET AL., J. European Ceramic Soc, vol. 30, pp. 2239-2251.

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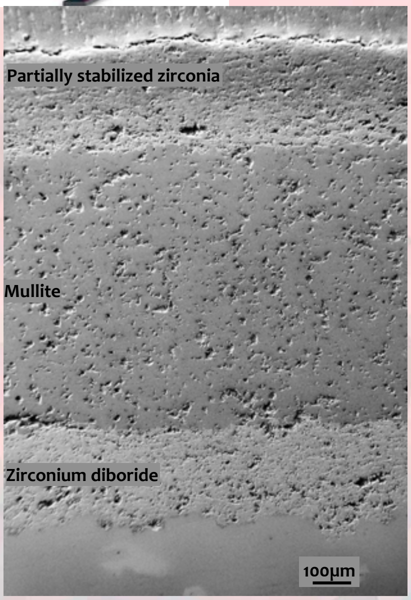
Challenges for Thermal Protection System (TPS)



- Modifying HfB_2 and ZrB_2 powders for thermal spray; i.e., powder flow, particle size distribution, and morphology.
- Augmenting processing conditions for an inert environment.
- Understanding how metastable phase equilibria relationships influence the coating and its properties.

	ZrB_2	$3Al_2O_3$-$2SiO_2$	ZrO_2-$8Y_2O_3$
Mean	801	523	539
Standard Deviation	146	45	128
Coefficient of Variance (%)	18.2	8.6	23.7

- M. Tulii et al., SCT 205 (2010) 1065-1060. (MoSi2 additions)
- C. Bartuli et al., SCT 155 (2002) 260-273 (SiC additions)
- W.G. Fahrenholtz et al., *'Ultra-high temperature ceramics: materials for extreme environment applications.'*, Chemical Engineering Progress, 2015. **111**(2): p. 1-451

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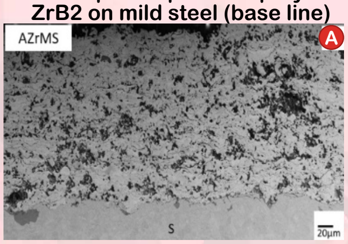
INTERNATIONAL MATERIALS APPLICATIONS & TECHNOLOGIES — IMAT THE VIRTUAL EDITION

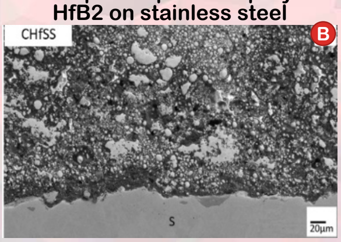
October 26-28, 2020

Microstructure of a Thermal Protection System (TPS)

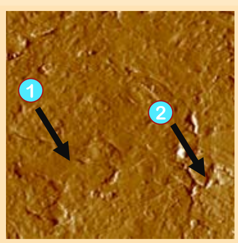
Atmospheric plasma spray of ZrB2 on mild steel (base line)



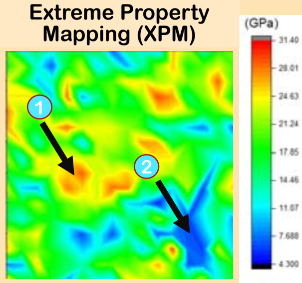
Atmospheric plasma spray of HfB2 on stainless steel

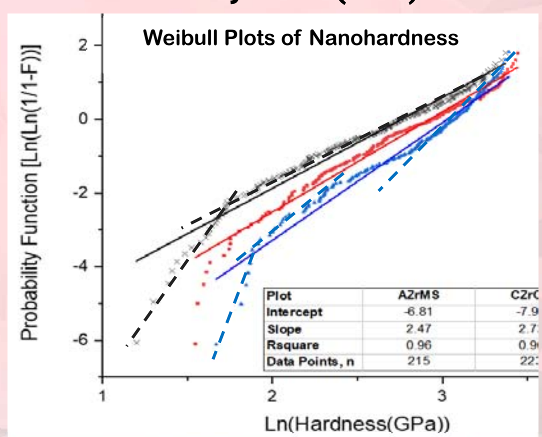


Atmospheric plasma spray of ZrB2 on stainless steel



Extreme Property Mapping (XPM)





- Distribution analysis finds >2 families within the populations.
- Understanding of microstructure / property relationships.

AFSOR Award Number FA2386-18-1-4119

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Beautiful Microstructures: Case Histories for Designing the Face.

TRL2: Science Leads to Sustainability!

Surface Engineering for Advanced Materials

1. "Beauty is in the eye of the beholder" (Plato).
2. "What you see is what you get" Really?
3. What's the Risk?
4. Will the real customer stand up? What does the customer want?
5. **Who do we work for? Crystal balling (connecting dots)**

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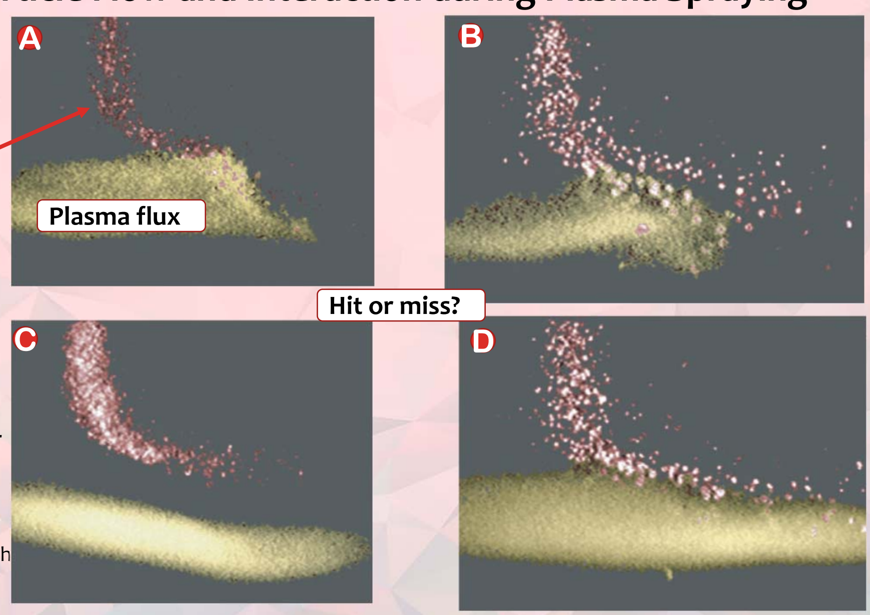
October 26-28, 2020

Particle Flow and Interaction during Plasma Spraying

Molybdenum particles injected into plasma jets (Control Vision with a laser flash duration of 5 µs).



Arc root fluctuations in the restrike mode (3 kHz). Arc root attachment is 30 - 150 µs (~0.1 ms).

Z. Duan and J. Heberlein, "Arc Instabilities in a Plasma Spray Torch JTST, 11[1] (2002) 44-51.

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Deploying our Tool Box & our Exquisite Palette of Micro Features

Structure + Process = Property → • Functionality • Applications • $'s

No.	Microstructural Artefact	Impact of the Processing Tool	Effect on Material Properties
	Substrate roughness	Grit blasting pressure and technique	Adhesion strength
1	Over blasted area	Grit blasting pressure and technique	Lower roughness & poor adhesion
3	Embedded grit blast media	Grit blasting pressure and technique	Surface contamination, corrosion sites, poor adhesion
	Coating roughness	Feedstock size	
5	Unmelted particles	Thermal spray parameters (too cold)	Variable physical properties
6	Partially melted particles	Thermal spray parameters (too cold)	Variable physical properties
8	Oxide particles	Thermal spray parameters (too hot)	Micro-hardness increase
9	Inclusions	Thermal spray parameters, feedstock quality	Local corrosion
10, 11	Porosity	Standoff distance	Modulus decreases
13	Vertical cracks	Compressive residual stresses	Hardness decreases
12	Horizontal cracks & lamellar	Tensile residual stresses	delamination or separation
	……….. etc.		

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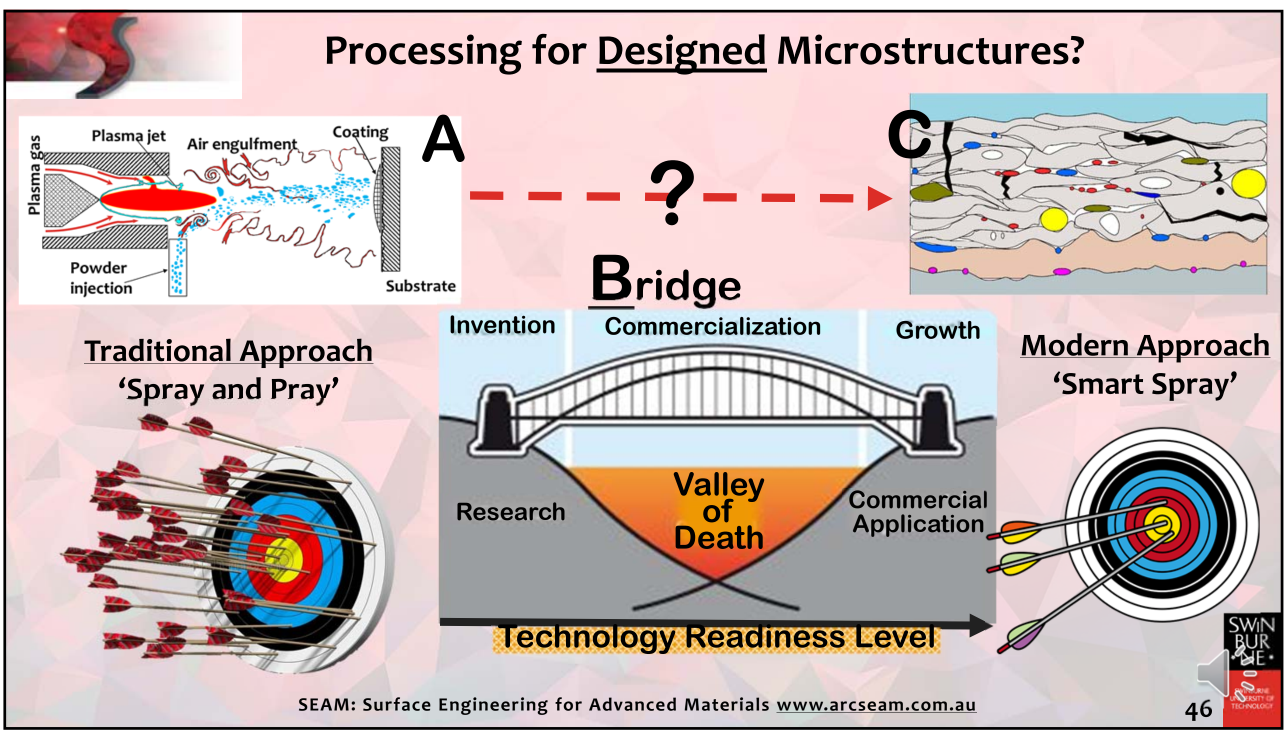

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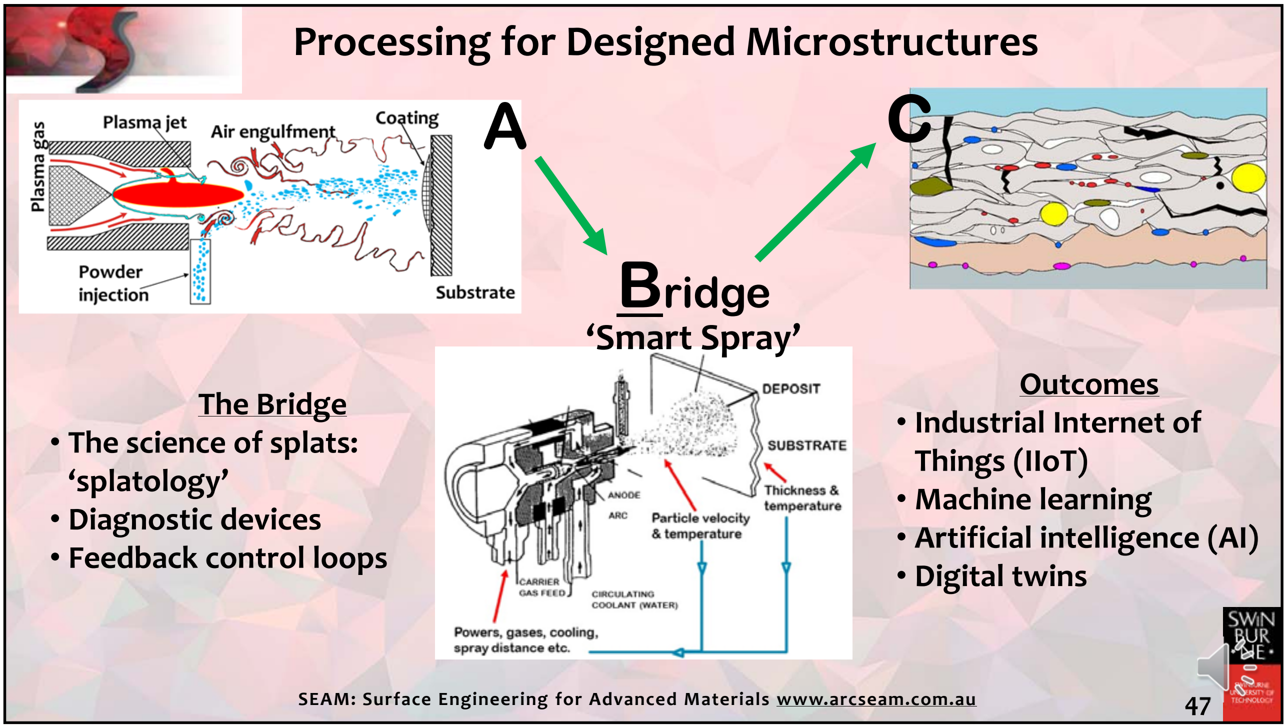

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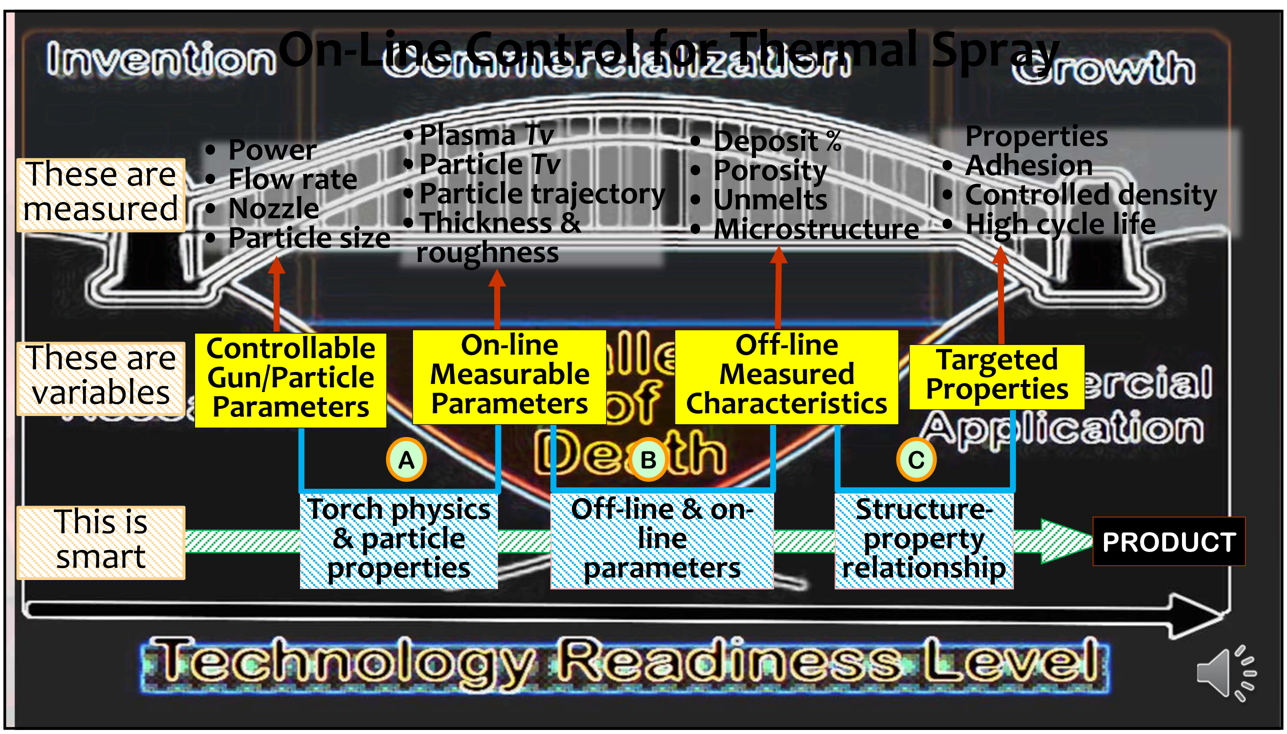

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Vale: In Memory of Past Thermal Sprayers …

Rajan Bamola
M. Bradley Beardsley
David Browne
Philip Cheang
Paula Didiere
Tony Farmer
Scott Goodspeed
Gerry Haddad
Douglas Harris

Joachim V.R. Heberlein
Reginald McPherson
Keith Moore
Richard Moore
Emil Pfender
Walter Riggs
Elliot Sampson
Merle Thorpe
Dongming Zhu

…… & Encouraging Future Generations!

Dr Andrew Ang, Dr Samuel Pinches, Duy Q. Pham, Ameey Anupam, Ashok Meghwal, Bruno Kahl, Malkeet Singh

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Summary and the Pathway Forward

1. What do we know?
A lot about technology. Not so much about science. We are explorers.

2. Discriminate the thermal spray technologies
Thermal spray equipment is versatile

3. Design the microstructure to suit the need
The microstructure is a palette of artefacts to be leveraged

4. Hitting the target
Processing conditions can be controlled

5. Intelligent design (Spray Smart)
Digital twins to accelerate manufacturing.

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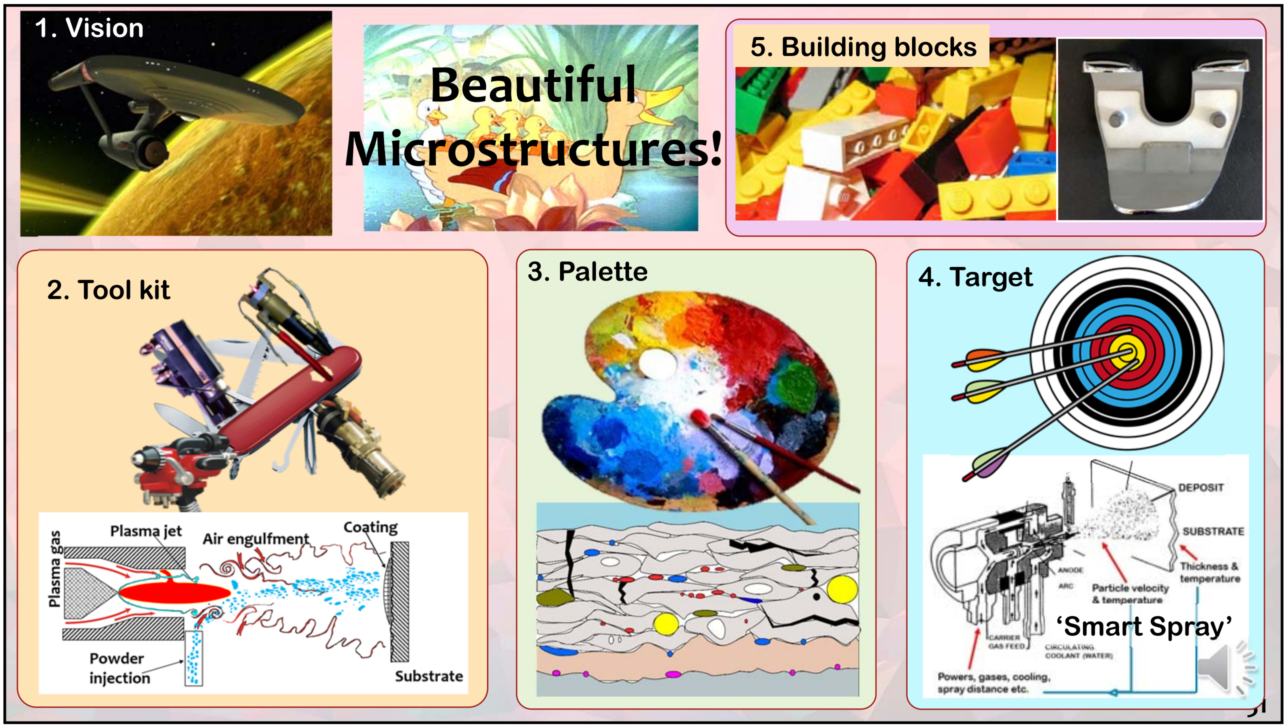

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October 26-28, 2020

ARC TRAINING CENTRE
Surface Engineering for Advanced Materials

Australian Government
Australian Research Council

Acknowledgement: This work has been supported by the Australian Research Council (ARC). The ARC Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005. Additional support has derived from industrial, university and other partners.

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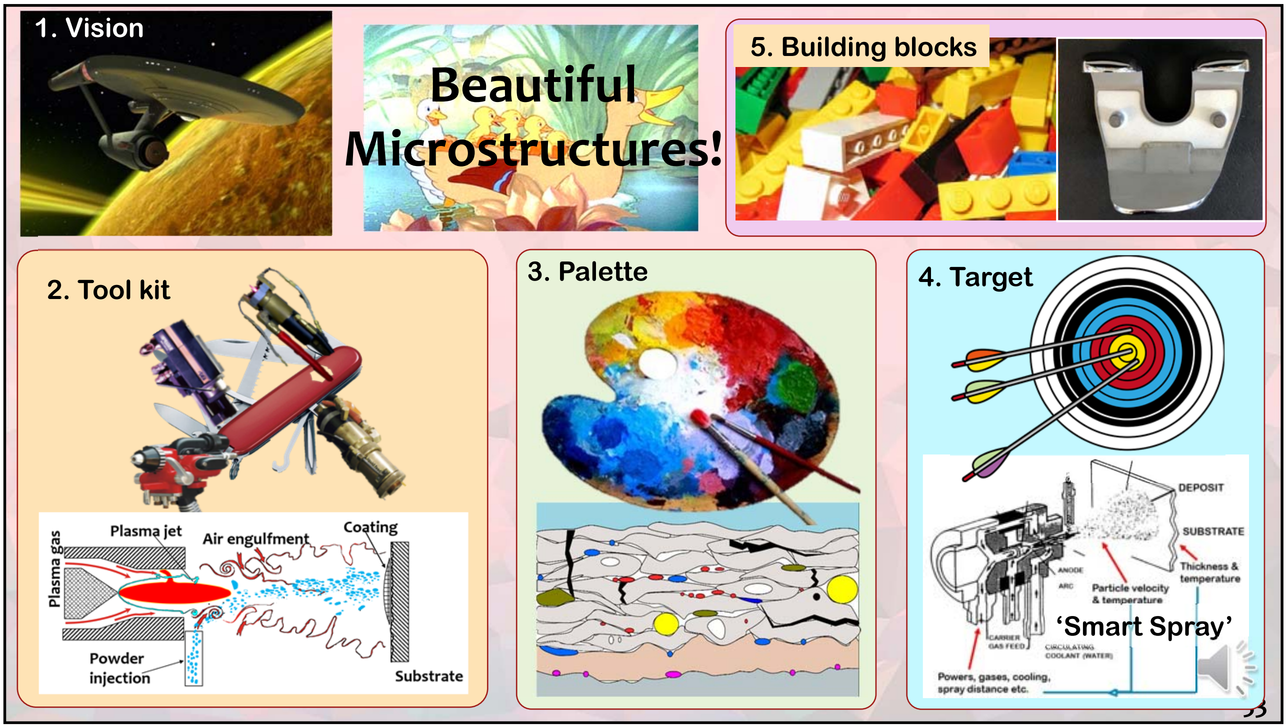
1. Vision
Beautiful
Microstructures!
5. Building blocks
2. Tool kit
Plasma gas
Plasma jet
Air engulfment
Coating
Powder
injection
Substrate
3. Palette
4. Target
DEPOSIT
SUBSTRATE
Thickness & temperature
Particle velocity & temperature
'Smart Spray'
Powers, gases, cooling, spray distance etc.

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Beautiful Microstructures: Case Histories for Design the Face

Christopher C. Berndt
Surface Engineering for Advanced Materials (SEAM)
Swinburne University of Technology, Melbourne, AUSTRALIA
cberndt@swin.edu.au

Slide 1. Beautiful Microstructures: Case Histories for Designing the Face.

Mr Chairman, and thank you to the IMAT co-Chairs and Program Committee for the honour of delivering this Key Note talk. I intend to hit some hot buttons. I will use this platform to push opinions and bring about an informed discussion.

Slide 2. Acknowledgement of Country

Before we go to far, it is usual in Australia to acknowledge the original people of our wonderful country.

We respectfully acknowledge the Wurundjeri People, and their Elders past and present, who are the Traditional Owners of the land on which Swinburne's Australian campuses are located in Melbourne's east and outer-east.

We are honoured to recognise our connection to Wurundjeri Country, history, culture and spirituality through these locations, and strive to ensure that we operate in a manner that respects and honours the Elders and Ancestors of these lands.

Slide 3. DEDICATION

The world is experiencing a pandemic. This presentation is dedicated to the Health Care Staff and Essential Service Personnel who are working at the front line of this 2020 global pandemic.

Their devotion and dedication is inspirational.

We are all stronger when we work together.

THANK YOU!

Slide 4. Case Histories for Designing the Face. TRL2-4: Science

Like any organized story (or thesis) there are chapters. The sequencing of my slides is a progression through Technical Readiness Levels, TRLs, that go from 2 to 8. In brief, a low TRL number represents a science focus, and as we progress through the

Acknowledgement: The Australian Research Council (ARC) Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005.

numbers to TRL8, there is been a transition from science to real-world applications. That is: at TRL8 a company can send out invoices for their manufactured product.

Slide 5. George Vander Voort

The stage is set, so let us start:

"Beauty is in the eye of the beholder", attributed to Plato, and a statement that can be argued intensely.

Here are some truly beautiful microstructures from the web site and calendars of George F. Vander Voort. Wonderful images for anyone, regardless of your background.

For the materials scientist the beauty goes much deeper because you can explore phase transitions, grain boundary behaviour and a host of amazing physical metallurgy artifacts. I encourage you to ask your work mates, and even your children, to explore webs sites such as those of George. This would be their entry into the fascinating world of materials science where we begin to understand deep science.

I also unashamedly give a plug for the CAMS 2021 conference that will be held in Melbourne in December 2021. Escape your northern hemisphere and visit us.

Slide 6. Thermal Spray Coatings: An Ugly Duckling

One of the hot buttons for TS is 'microstructure' where the adjective 'ugly' is often used rather than 'beautiful'.

Here is an important note if you are chasing money. An ugly microstructure does not resonate well with funding agencies. We all experience presenting micrographs to a potential sponsor who is new to thermal spray. We need to lock the doors before the presentation so that they do not flee in terror when they see a material full of defects.

The main point is that these complex microstructures are employed in a host of low and high performance applications. And this list is growing every day.

These micrographs (Slide 6) are of functional coatings. They exhibit, a lamellar morphology, a rugged surface, a rugged interface against the substrate, porosity, cracks and many other artifacts.

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They are the 'ugly duckling' of materials engineering but as you become more mature in understanding their exotic microstructure, then the world of beautiful functionalities is revealed.

I have two more examples.

Slide 7. CrNi Fibers in a NiCrAlSi Matrix

This is a beautiful slide according to anyone's tastes. It shows CrNi fibers embedded in a NiCrAlSi matrix that has been sequentially thermal sprayed. The typical lamellae structure of the matrix material is quite distinctive.

The technological beauty is the near net shape manufacturing of metal matrix composites. The engineering beauty is that we can create special functional properties by using thermal spray processing variables, just like the tool box on a Swiss army knife.

Slide 8. Solid Oxide Fuel Cell Microstructure

A further example is attributed to the complex structures manufactured by using thermal spray to form solid oxide fuel cells. This is additive manufacturing in its most pure form. In fact, thermal spray has been ahead of additive manufacturing before additive manufacturing became a buzz word.

~~And, by the way, thermal spray has lead the way for:~~

- ~~composite technology~~
- ~~nanomaterials~~
- ~~quasi-crystals~~
- ~~low temperature superconductors~~
- ~~3-D printing~~
- ~~anti-microbial coatings~~
- ~~passive coatings that actively clean pollutants from the atmosphere~~
- ~~osteophilic bioengineered surfaces~~
- ~~… and the list goes on.~~

I was tempted during my preparation for this talk to provide a list of applications with images. Such an approach is not my intent. The purpose of this speaking platform is to convince you that there is a better way to design every coating to make it 'fit for purpose'.

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Slide 9. TRL4, 5: Science Leading to Technology

The science of thermal spray is multi-faceted and becomes deep in specialized disciplines. "What you think you are getting is not what you actually produce."

Let me explain. The physical properties of thermal spray coatings are 'extrinsic' in nature. To state this another way: You cannot go to a data book, find the modulus or thermal conductivity of a material (say alumina) and expect the thermal spray coating to exhibit the same properties.

Slide 10. Thermal Spray Properties are Extrinsic

This point is best illustrated by reference to the well known Ashby Diagram of Thermal Conductivity with respect to Thermal Expansion. Sprayed zirconia has a thermal conductivity of 1 W/m.K whereas bulk zirconia has a value of 2 W/m.K. The thermal conductivity of zirconia has been halved … just because we have created unique, extrinsic, microstructure. This singular data point has spawned a multi-billion industry in thermal barrier coatings.

So "What you see, may not be what you get in terms of performance". Because:

Slide 11. The Beauty of Non-Equilibrium Phases

….there is beauty in forming non-equilibrium phases where we can question the Gibb's Phase Rules. The rapid solidification gives rise to a host of thermodynamic behaviours that require sophisticated measurements.

And I am compelled to point you towards our Journal .. the Journal of Thermal Spray Technology … that champions all areas of thermal spray. Have this on your coffee table or in your iPad.

Slide 12. Coating: Bronze (90% Cu, 10% Al)

Thermal spray is often focussed on mimicking the microstructure of bulk materials. This is a mistake … because the material properties of the thermal spray coating can be designed to be unlike those of the original feedstock. This opens the door for materials that have a bespoke architecture that displays specific attributes.

This bronze coating illustrates some of these artifacts. These are typical for the majority of thermal spray coatings manufactured by two wire arc; plasma and flame spray; as well as some HVOF processes.

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Some thermal spray processes typically produce dense coatings where the microstructure is more akin to the bulk material; with the additional feature of residual stress effects.

Slide 13. Ceramic biomaterial coatings

Coating structures are complex, and here are two bioceramic coatings for orthopedic implants.

First of all two cautions: 1. These are 2-D images of a 3-D structure. and 2. Metallographic preparation is an art where the preparation influences what you see.

Here is a question: “Which of these two coatings are most desirable for the application of being located on a metallic implant?” So now the bioengineer needs to specify design criteria and this is the initial stumbling block. The ASTM standard specification F1609 08(2014) provides guidelines for chemical requirements, physical characterization and mechanical characterization.

- ~~Chemical requirements includes purity and environmental stability.~~
- ~~Physical characterization includes coverage of the substrate, thickness, porosity, color, surface topography, and density.~~
- ~~Mechanical characterization includes tensile bond strength, shear strength, and fatigue strength.~~

The porosity indicated for these two coatings are attributes they enable good interactions between the coating and physiological media within a 3-D environment.

The different shades of grey represent different phases, which exhibit different dissolution rates within physiological media. Hence, the biological interaction between the implant and body can be controlled for the optimum patient outcome.

Slide 14. Cross-section of a heterogeneous coating

A generic cartoon of a thermal spray coating sets the initial base line that illustrates the substrate, metal bond coat and ceramic overlay. A bond coat is usually of 50 to 100 μm thick and the overlay ceramic can be 200 to 300 μm thick.

It is indicated that grit can become embedded into the substrate surface and that over grit blasting is detrimental. These are the features labelled as 1 and 2 and some typical roughness values for the grit blasted substrate and coating surface are indicated.

Acknowledgement: The Australian Research Council (ARC) Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005.

Slide 15. Defect taxonomy: Degree of melting

I would now like to introduce the phrase ‘defect taxonomy’. Taxonomy refers to the science of naming, describing and classifying classes or objects; for example the taxonomy of plants, insects, and mammals.

Ii is convenient to use this to now classify the defect structure of thermal spray coatings. This is a convenient instrument to describe a host of features.

Slide 15 shows features that arise from feedstock particles that can be fully melted to form splats (7), be partially melted (6), or do not melt at all (5). The degree of melting is known as the Madejski criteria and is defined as the splat aspect ratio. Thus we now have solid metrics that can be assigned to these taxonomic features.

Or some feedstock particles may experience over-heating and form oxides (feature 8), and in some cases bounce-up particles resolidify and become inclusions. Feature 9.

Slide 16. Defect taxonomy: Physical interactions

Other defects arise due to packing of particles and physical interactions, much in the same manner as squeezing grapes in a jar. For instance inter-lamellar pores can form, feature 10. Or gas evolution from a super-saturated solid solution to form intra-lamellar pores, feature 11. And cracks evolve during and after the spray process due to high energy impacts and stress relief, features 12 & 13.

Slide 17. Chaotic Microstructures

The beauty of thermal spray coatings lies in their rich microstructure. I describe these microstructures as being ‘chaotic’; which is a mathematically more rigorous description than ‘defective artifacts’.

It becomes apparent, that it can be rightly claimed that thermal spray coatings are the original ‘nanomaterial’, the original ‘composite material’, a ‘functionally graded material’, the original example of ‘additive manufacturing’; as well as many other materials engineering firsts.

Thermal spray provides opportunities regarding the special microstructure. ~~include: (i) layered microstructures, (ii) composites that establish tailored material properties, and (iii) rapid cooling rates to create unusual alloys with extended solubility~~. I ask “Why are modern technologists missing these opportunities?”

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You will see this slide a few more times later on in this presentation. The main take home messages are: (i) the microstructure of thermal spray coating is complex, and that (ii) the components of the microstructure can be classified in taxonomy jargon. The missing link is putting numbers and morphologies behind these microstructural features, and this is a grand challenge for our community.

Slide 18. Splat Microstructures

The fine detail of splat microstructures, documented at Limoges and other universities, indicates that macroscopic splat behaviour and microscopic grain growth is determined by the processing environment.

Note that I used the passive expression '**determined** by the processing environment' and not the more active phrase '**controlled** by the processing environment'. Ultimately we must be quite aggressive in **controlling** the structure that we want; and I will address this topic in a little while.

Slide 19. Splat cartoons

And 'yes' splats give us a great deal of enjoyment! And fun!

Slide 20. Interfacial Structure implies atomic bonding

Exacting microscopy has verified that metallurgical bonding does arise across the interface in certain situations. Scientific results such as these allow links between processing and properties to become better understood.

Slide 21. Al2O3 matrix around YSZ fiber

Let me briefly consider the co-spraying yttria stabilized zirconia fibers in conjunction with alumina powders. The objective is to form a reinforced ceramic composite.

It all starts with feedstock processing and the cartoon on the lower left corner shows the yttria stabilized fibers encased in the lower melting point alumina so that a football shaped feedstock can be fed to the processing tool.

Under high magnification we can detect columnar growth of the alumina matrix, YSZ fiber cracking, and decohesion of the alumina matrix.

As a side note this example points to the fact that all thermal sprayers eventually turn into powder technologists since this is the foundation of thermal spray.

Acknowledgement: The Australian Research Council (ARC) Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005.

Slide 22. An Interim summary

So far I have been addressing the science; TRL2&3. This interim summary sets the scene for transitions towards high TRLs where there are revenue-producing applications.

For many of you I have reinforced prior knowledge. But I have also aimed at opening an extra dimension concerning the processing needs and control. Therefore, we must understand the 'how and why' of the defect taxonomy of thermal spray coatings.

We know that the science is fascinating and the great unknown. We are exploring new frontiers just like Captain Kirk and Mr Spock.

I also hope that you are convinced that Thermal spray coating microstructures are very, very beautiful

However, we need to ask some serious questions if we want to head towards TRL7&8.

Slide 23. TRL2 to TRL8: Science Leads to Industrial Applications!

For instance, who is the real customer and what do they want?

Let me back up just a little. Some of you may recall a robust discussion that was initiated by the late Doug Harris during his Plenary talk at an International Thermal Spray Conference in Montreal in the previous century? He challenged the interactions and business relationships between the equipment and feedstock suppliers and their customers. Doug, as a customer, expressed his strong opinions about what he perceived as a one-way relationship.

I am now going down a similar path, but from a different angle. My direction is 'How do scientists at TRL 2-4 relate to manufacturing industries who must operate ate TRL 7-8? There is the so-called 'valley of death' at TRL 5 & 6 during prototyping and pilot studies.

And, by the way, the theme of beautiful microstructures carries over as you will see. Hence, the question is, again for the second time, 'Who is the real customer and what do they want?'

Acknowledgement: The Australian Research Council (ARC) Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005.

Slide 24. There are too many variables…..

The answer is simple .. industry must be sustainable, which is a polite way of saying 'must make money'. A consumer such as a large manufacturing company is not compelled to use any surface engineering method. Often it is perceived as too complex and adding cost. And there are too many variables, as this complex fish bone diagram shows for thermal spray manufacturing where there are 8 major steps indicated.

I am not going to go through all of these many variables that influence coating quality and applications.

Slide 25. There are too many variables….. How is to jump this valley of death?

The reality is that:

(i) industry must progress from the torch to the application ASAP, and
(ii) the manufacturer needs to know the value proposition.

My experience is that there is limited interest from industry in the process, the structure, or the science; they are more focused on pragmatic outcomes.

So how can thermal spray, or in fact any surface engineering technology, jump this technological valley of death?

The answer is that industry should be very interested in the fine detail of the process ….

Slide 26. Influence of chaotic microstructures on extrinsic physical properties

…. because all of the beautiful microstructural artefacts, if controlled, add tremendous value.

The table lists some 'cause-effect' relationships; where the cause is the 'process tool' and the effect is the 'influence on coating microstructure'. The microstructure is now a feature that a materials scientist can deploy to create unique properties. Thermal spray processing is the Swiss army knife of surface engineering.

A simple example is that for ID #2, an increase in velocity, leads to a more fine microstructure and higher density. And ID#6 indicates that the stand off distance influences temperature and velocity, and hence splat cohesion. This is not rocket science.

Acknowledgement: The Australian Research Council (ARC) Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005.

A small footnote is that the generic description of a thermal spray torch can now we expressed as the positive phrase of ‘process tool’.

This reasoning can now be referenced to the blue cells in this table. Now we define the most desirable microstructure and **then** select the best tool for its creation.

Slide 27. Microstructures that are rich with features

So let us recall a previous slide, where the rich structure of coatings was presented.

Slide 28. The exquisite palette of microstructures for engineers

These microstructural artefacts can now be assigned to processing conditions. And these control material properties.

This is the ‘exquisite palette’ that we have available as materials engineers to design the architecture of coatings.

The features in the microstructure are identified and assigned to a classification according to taxonomy principles.

The root cause for the formation of the feature is embedded within the tool and there is an outcome with regards to the material properties.

Slide 29. Integrating ‘science + engineering’

One of the first big picture views is, therefore, to integrate the science with the technology for an engineering outcome.

The science in the blue boxes at the top is defined within the 3 regions that can be controlled: (1) before spraying, (2) during spraying, and (3) after spraying.

These 3 process regions are integrated with 3 engineering criteria shown in the orange boxes at the bottom: (i) the feedstock quality, (ii) the thermal spray parameters, and (iii) the surface preparation of the component.

Slide 30. Troubleshooting decision pathways

Within a production environment these concepts are embedded in technical manuals and are often described as ‘spray tables’. These document the state-of-art and are highly confidential.

Acknowledgement: The Australian Research Council (ARC) Training Centre in Surface Engineering for Advanced Materials, SEAM, has been funded under Award IC180100005.

If there are issues on the shop floor then the technician and engineer will use their experiences to trouble shoot their way out of the problem.

You will never see this type of flow chart within the procedures manual of any company because it oversimplifies a complex array of instrument interactions.

Focus, though, on the blue boxes where we ‘go back to go’, just like a monopoly board game. These iterations cost money. If these are reduced, then the profit margin increases.

Keep the following thought in mind ‘How can science optimize the learning process for spray tables?’ ‘How can we make short cuts?’. I will address these questions in a little while.

Slide 31. New Materials … What’s the Risk?

Let’s now refocus and talk about the future. Einstein said ‘A ship is always safe at the shore – but that is NOT what it was built for.’

Let me paraphrase this quote as ‘If you want 100% assurance of the outcome, then do nothing.’

And the cartoon states ‘Innovate? No – we already tried that once. It didn’t work out’.

Of course: there is risk in exploring new materials (or new drugs in our current climate) but the outcomes of success can be enormous. I would point you towards bond coats, thermal barrier coatings, electronic applications and biomedical surfaces .. .all of which have multi-billion dollar markets.

Remember that it can take 10-15 years for innovation to gain traction from initial conception to consumer uptake. This is a serious commitment that requires continual pushing, trust, faith and ‘resources’. Resources is the polite form of saying ‘cold hard cash’.

Slide 32. High Entropy Alloys: Displacing traditional bond coats

My first example concerns high entropy alloys, HEAs. HEAs consist of a cocktail of at least 5 elements that are in approximately equal molar proportions. For example the elements Al, Co, Cr, Fe, and Ni.

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There are for key references shown that present the back ground material. I am proud to note that Swinburne were among the first to focus on the thermal spraying of HEAs in 2014 in collaboration with colleagues at the Indian Institute of Technology in Madras.

The attributes of HEAs, in summary, are they weigh less, exhibit excellent high temperature properties while retaining good mechanical properties and the constituent materials cost less.

I refer you to these formal manuscripts that expand on the technical and science topics.

Slide 33. The Magic of High Entropy Alloys: Microstructural Evolution

Recall the theme of this presentation: 'beautiful microstructures'. Is this microstructure beautiful?

I now remind you of previous slide in the lower left corner. The process environment creates a composite of splats that exhibit distinct chemistries. The splats range from oxides, indicated as the grey and black phases; to alloys that are whitish in contrast.

This effort is at a TRL 4 where we are navigating the valley of death.

These splat structures reflect the dynamic splat behaviour and the complex chemistries within HEAs. These will now be interrogated to expand our knowledge and understanding.

Slide 34. Mechanisms: Understanding HEA Micro-structural Evolution

The pictorial table lists the mechanisms of splat formation at **impact** under two controls:

(i) particle size on the x-axis, and
(ii) temperature range on the y-axis. And again, I reference the fact that we are looking at the mechanism of phase formation at impact.

All of the details on this table are presented in the recent manuscript by Anupam et al. I will present the overarching knowledge and understanding.

The cell indicated as a green circle 3 indicates that small particles at low temperature do not deposit but may form surface oxides. The tendency to oxidize and adhere

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increases with temperature .. there is a progression from cells 3, to 2 and then to 1. On the other hand large particles tend to retain the HEA phase structure as shown in cells 7, 8 and 9.

The point is that the beautiful microstructure can be explained in terms of thermal spray processing, which I have described as our Swiss army knife.

Slide 35. Antimicrobial Coatings: More Beautiful Microstructures

Another exciting area revolves around antimicrobial coatings. Implants can suffer from poor mechanical properties and high risk of infection, as indicated in the green text box. Can the surface of these implants be engineered to repel microbes or kill them outright?

Thus, the cartoons in the bottom right hand orange panel provide some guidance. For example, how can the surface of a thermal spray coating can be designed to repel microbes or destroy microbes by spearing them?

Or can the microstructure incorporate pores that hold an agent to improve cell growth and release a biocide that kills bacteria?

So the desirable outcomes are improved mechanical properties, enhanced biocompatibility and exhibiting and intrinsic antimicrobial property.

Slide 36. Ceramic biomaterial coatings

A previous slide demonstrated two ceramic biomaterials, one based on a zirconium silicate and the other on a phosphate. Both of these beautiful microstructures are functional bioceramics, but only hydroxyapatite is currently in use.

Some more details are now provided.

Slide 37. Baghdadite vs HAp-Nanoindentation

We have carried out extreme property mapping by nanoindentation on the cross section of these coatings. The 20 x 20 matrix of indents allows contour maps of the elastic modulus to be overlaid onto the microstructure.

Three of these indents are identified as red circles .. but there are 400 of these for each material.

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The Bagdadite coating exhibits a greater modulus with less variability. As well, the mechanical measurements are correlated to the phases within the microstructure, as can be identified by the blue rendition for the hydroxyapatite coating that reveals a low modulus phase.

Slide 38. Baghdadite vs HAp-Cell attachment and Proliferation (MG-63)

Both of these surfaces are inductive to cell attachment and proliferation .. which again demonstrates the relationship between surface texture and nature to bioactive functionality.

The red arrows indicate cells bridging lamellae features and is indicative of a good short term outcome.

These are all positive results and indicate that the sub-units within the microstructure are performing as they are designed.

Slide 39. Coating of knee implant

Finally, the coating must be applied to an orthopedic device. Remember from a previous slide that we have instigated a comprehensive troubleshooting pathway to get to this phase, as repeated in the green box on the bottom right corner. There are 5 stages.

- Stage 1: Measuring up the implant and inputting this data into a graphics program.
- Stage 2: Making a plastic mock up of a clam-shell mask so that the coating is deposited onto only specified surfaces
- Stage 3: Making up a metals clam shell mash by conventional additive manufacturing.
- Stage 4: Masking up the implant.
- Stage 5: Spray the bioceramic coating onto the implant.

Therefore, the desirable microstructure has been precisely located on the orthopedic appliance to manufacture the functional coating. This is an achievement at of TRL6.

Slide 40. Thermal Protection System (TPS)

The next example explores coatings for high speed flights in excess of Mach 5, about 6000 km an hour (or 3800 miles per hour); that is, less than 1 hour to travel from London to New York. At this speed the leading edge of the vehicle gets extremely

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hot due to the high enthalpy bow shock and the surface temperature exceeds 2,000 C (or 3,600 F).

The thermal protection system (TPS) must survive high temperatures and, unlike a thermal barrier coating, must conduct heat away from the leading edge. I repeat, the engineering specification is <u>high</u> thermal conductivity, which implies that the microstructural design be a dense coating.

Thus, design criteria for a TPS is very different from that of a thermal barrier coating (TBC). A TBC requires low thermal conductivity and be porous, which is converse to the requirements of a TPS.

The preferable materials of choice for a TPS are restricted to high conductivity and high melting point materials; which leads is towards zirconium and hafnium diborides.

Slide 41. Challenges for Thermal Protection System (TPS)

There are many challenges to form boride-based coatings; both technical and scientific. One is to modify existing feedstocks so that they can be sprayed. The second hurdle is to have processing conditions that exclude oxygen. And finally, the phase equilibria is complex and difficult to resolve.

Some of our early coatings consisted of a 3-layer design with zirconium diboride being overlaid with mullite and then a top coat of partially stabilized zirconia. ~~Note that this coating system would be removed from the substrate. Thus, the material system incorporates the zirconium diboride as a functional surface interacting with the environment. The mullite acts as an EBC and the PSZ as a traditional TBC.~~

The hardness data provided some confidence that coatings of some quality were being manufactured. But the remaining issue of excessive porosity still remains.

Our work can be discriminated from that of other groups since we are working with pure diboride materials rather than using a composite strategy that incorporates either moly disilicide or silicon carbide.

Slide 42. Microstructure of a Thermal Protection System (TPS)

More recent work has focused on moving to hafnium diborides sprayed onto carbon-carbon substrates. This has been much more challenging and the microstructures

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while not especially pretty … still demonstrate great promise. Our practise, in common with all universities, is to use simple substrates initially, for example mild steel or stainless as shown in figures A and B.

Then, when we feel comfortable with the range of spray parameters, we work towards more complex substrates of carbon-carbon and more difficult feedstocks of hafnium diboride as shown in figure B.

The extreme property mapping in the orange center panel shows how hard and soft regions can be identified and then related to the microstructure. Hence a composite structural analysis can be carried out.

The Weibull plots on the right hand panel show that these coatings consist of several families within the larger population. This approach integrates the microstructural data with the phase analysis and mechanical properties.

Slide 43. Who do we work for? Crystal balling (connecting dots)

My remaining 8 slides have two objectives:

First: To tie together the preceding discussion.

Second: To connect these dots and forecast the future.

Slide 44. Particle flow and interaction during plasma spraying

The reality of the thermal spray process is that it is either ‘hit or miss’ since the processing envelope is highly variable in terms of temperature and velocity. And, as well, this technology deals with particle size distributions.

These frames from a high speed video recorder show moly particles being frozen in time. These particles either enter the plasma flux, as shown in A and D, or miss it entirely, as observed in frames B and C. Thus, the molten state will mirror this ‘hit or miss’ behaviour.

Slide 45. Deploying the ‘Swiss Army Knife’ Tool Box and our ‘Exquisite Palette of Micro Features’ to Design Beautiful Microstructures

Remember the Taxonomy table that was described as ‘an exquisite palette’ where we can use ‘our Swiss army knife tool kit’? This is shown again here, but now focus on the top orange box which summarises the inter-relationships.

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The structure evolves from the micro-features. The processing tool presents the traditional spray tables. Together these establish the materials properties that lead to special functionalities, applications and revenue.

Let me put it this way: 'Structure + Process = Property; which together provide economic drivers.

Slide 46. Processing for designed microstructures

So thermal spray, as the original additive technology, has strong credentials as a disruptive technology.

But the short coming is how to get from A to C and survive the 'valley of death'? The valley of death can be thought of as the progression from TRL 1 to 9.

The traditional 'spray & pray' approach extends the valley of death. It is a trial & error approach that this highly inefficient as indicated by the scattered arrows on the target.

The modern approach is to use the emerging tools to hit the target with focus. I am terming this as 'Smart Spray'.

Slide 47. Design needs for a thermal spray device

The bridge between TRL 3 to TRL 7 is now replaced by 'Smart Spray'. Where we have on-line control. On-line control is not new approach in manufacturing. It has not been implemented for thermal spray due to the many stochastic variables.

The bridge needs 3 prime inputs: (i) a fundamental understanding of splats, (ii) diagnostic devices, and (iii) feedback control loops.

The outcomes are all derived by high level integration with the 'Industrial Internet of Things' to create digital twins of the spray process.

The next slide puts these elements together.

Slide 48. Control Feedback Loops for Thermal Spray

The future is a dream that is gathering substance and momentum. The top two rows are achievable right now … but the cost is prohibitive.

All of the physical inputs and outputs can be measured. This is the top row.

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The process variables in the middle row can also be measured.

The challenge is in the bottom row. Here, the objective is to integrate all of the knowledge and understanding so that ‘This is smart’.

The blue hatched text boxes describe the deep knowledge needed: (i) torch physics and particle properties, (ii) off-line and on-line parameters, and (iii) taxonomy – property relationship data bases.

And you can just see that the Valley of Death is always lurking in the background.

Slide 49. Vale: In Memory of Past Thermal Sprayers …

I do have two summary slides. But first, I would like to use this once-in-a-lifetime opportunity where I am presenting to a global audience to give thanks to the many people who have contributed to my professional growth.

I only wish that these colleagues were still here so that they were aware of their strong influence on me.

…. and, I want to encourage future generations of surface engineers to mimic the high standards of these individuals who have passed away.

Slide 50. Summary and the Pathway Forward

1. We know a lot about technology. We are explorers on new frontiers of science.

2. Thermal spray equipment is versatile and is a processing tool, just like a Swiss army knife.

3. The microstructure can be designed to suit the need. The microstructure is a palette of artefacts that can be mixed and matched for functionality.

4. Processing conditions can be controlled to hit the microstructural target.

5. Intelligent design or ‘Spray Smart’ will work hand-in-hand with digital twins to accelerate manufacturing.

<u>In summary</u>: The traditional ‘Spray & Pray’ conservative mindset must be set aside. Future generations of surface engineering will be propelled by key learnings that are based on understanding the deep science and technology.

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Slide 51. Summary and the pathway forward

The final slide is a pictorial summary.

One: We have a far-reaching vision that is exploring new frontiers.

Two: We have a tool kit to process materials.

Three: We have a microstructural palette.

Four: We have a ‘Spray Smart’ target.

Five: The microstructural elements are the building blocks that enable creativity.

Finally: Thermal spray is proud to be the ugly duckling of manufacturing because we create very, very beautiful and special microstructures.

--- Thank you ---

Slide 52. Acknowledgement

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