25th Feb 2020

James Grant

EngD Student

London Steel Symposium

Swansea University

Tata Steel

Academic Supervisor: Dr Amit Das

Industrial Supervisor: Dr Chris Owen

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TATA Steel's Building and Services application; High Frequency Induction Welded Hot-Finished Low Carbon Steel Conveyance Tubes

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TATA Steel's Building and Services application; High Frequency Induction Welded Hot-Finished Low Carbon Steel Conveyance Tubes

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High Frequency Induction Welding

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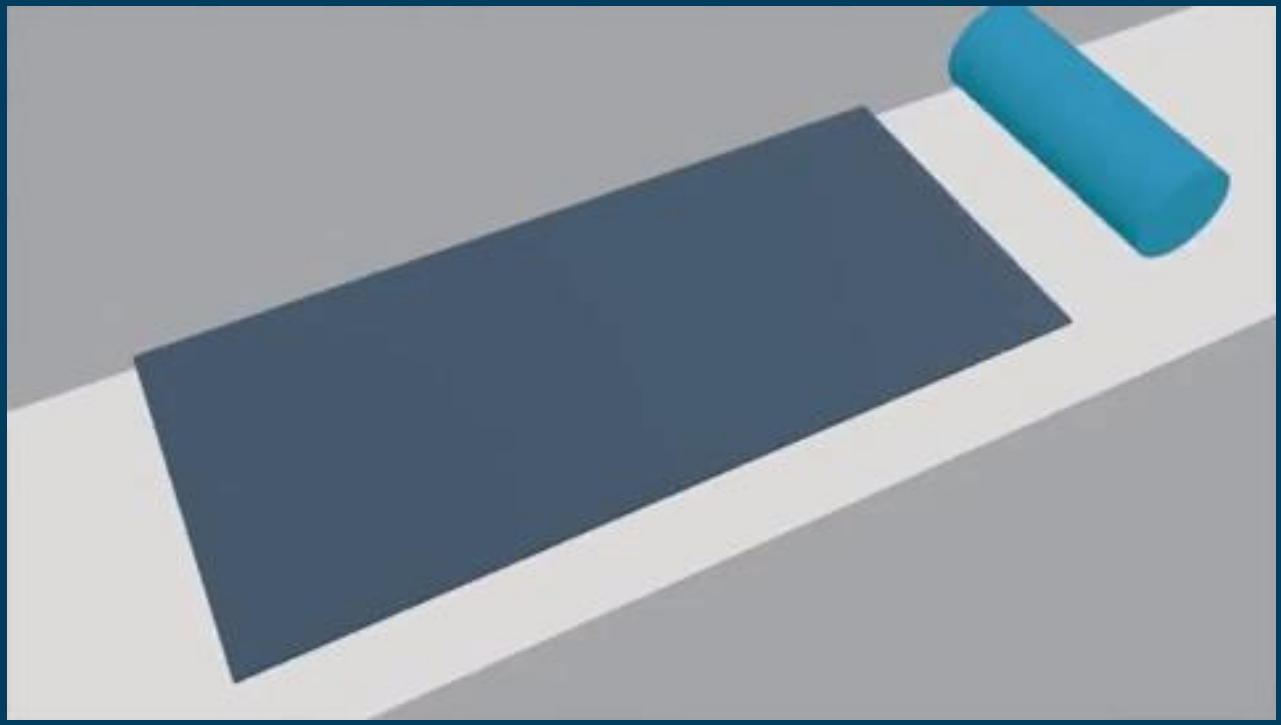
Forming Rolls

HFI Welding



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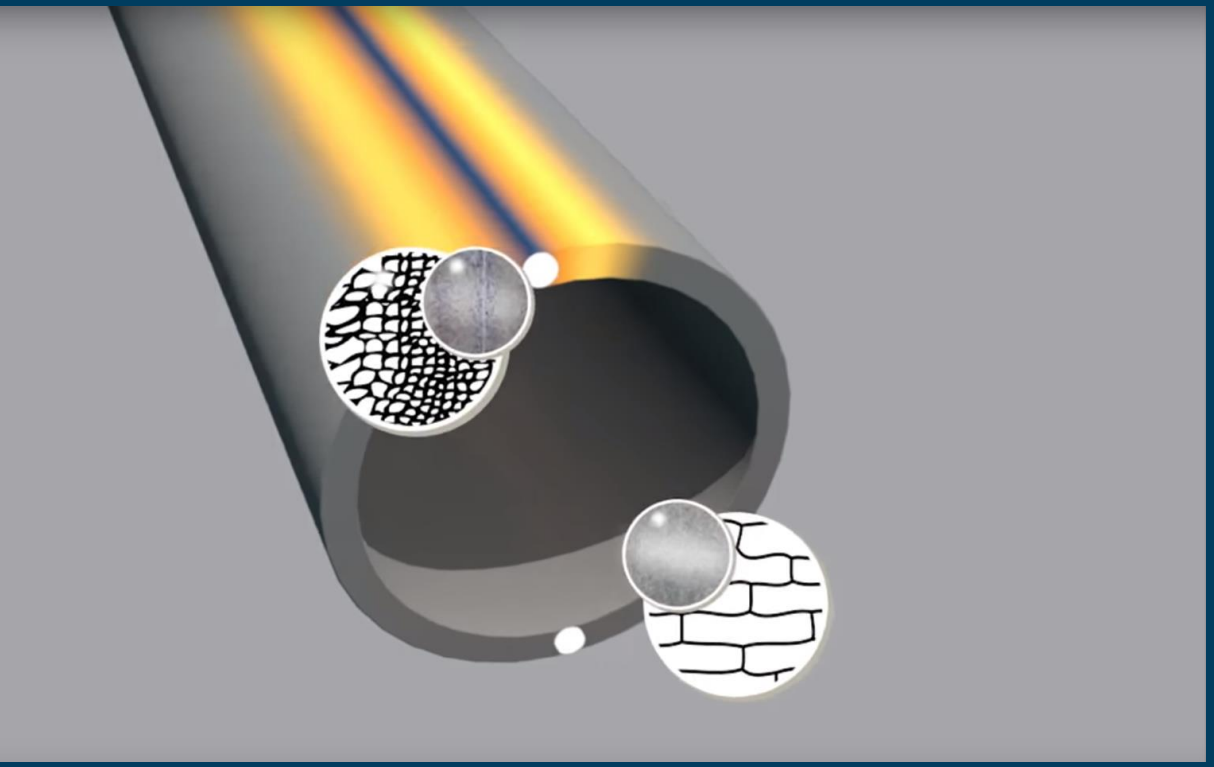
The Heat Affected Zone (HAZ)



- Bulk Microstructure ≠ Weld Microstructure
- Structurally weaker along the Weld Line
- More susceptible to Intergranular Corrosion
- Inability to Bend effectively
- Increased Risk of Splitting
- Lower Strength and Toughness Characteristics

How do you fix this HAZ?

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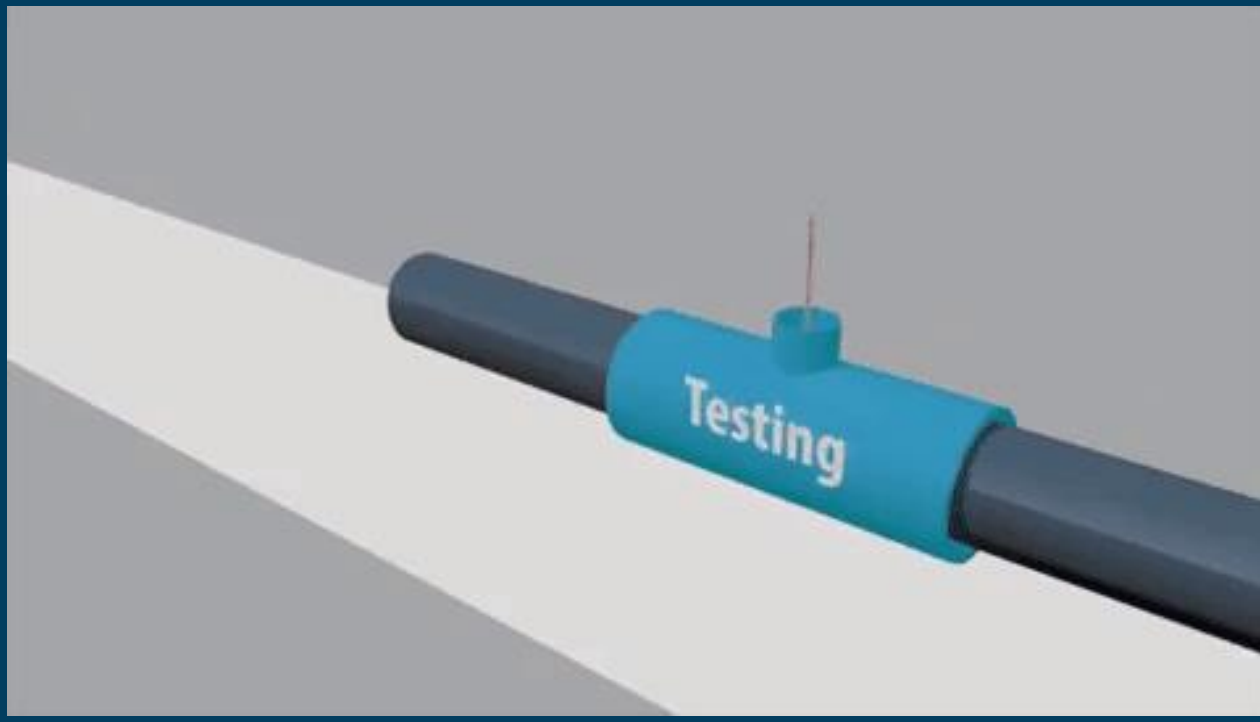
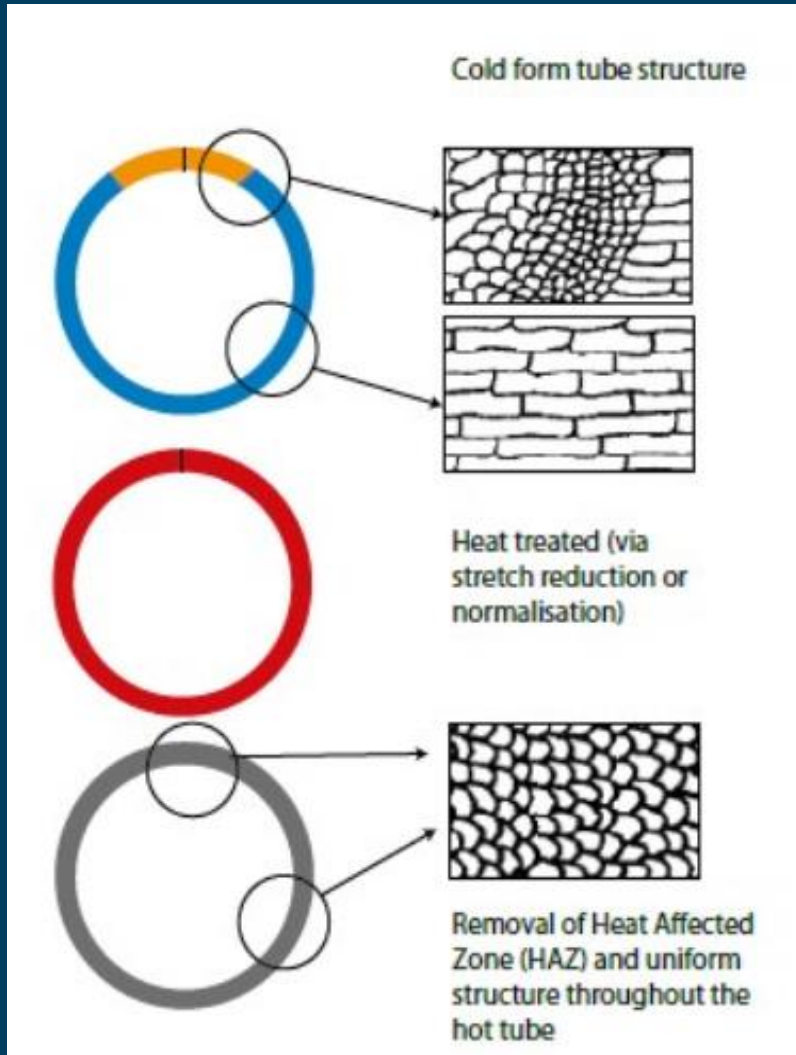
Normalisation at ~ 900°c

30 minute Soak Time

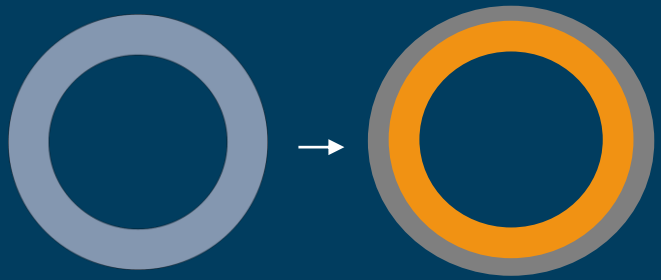


- ✓ Realign and apply homogeneity to the crystal structure
- ✓ Relieve the internal stresses applied during tube forming
- ✓ Increased Toughness and Pressure Integrity



Ah, but we have a problem…

Steel Tubes sent through natural gas furnaces at 950°C



Surface Oxidation

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The Pit

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Surface Condition

Seamless Tubes	Hot – Finished HFI Welded	Cold Formed HFI Welded
3	1	2

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Design a coating which inhibits scale growth during reheating

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Three Key Questions

1. What oxide are we dealing with?
2. How exactly does it grow?
3. Can you prevent it's formation?

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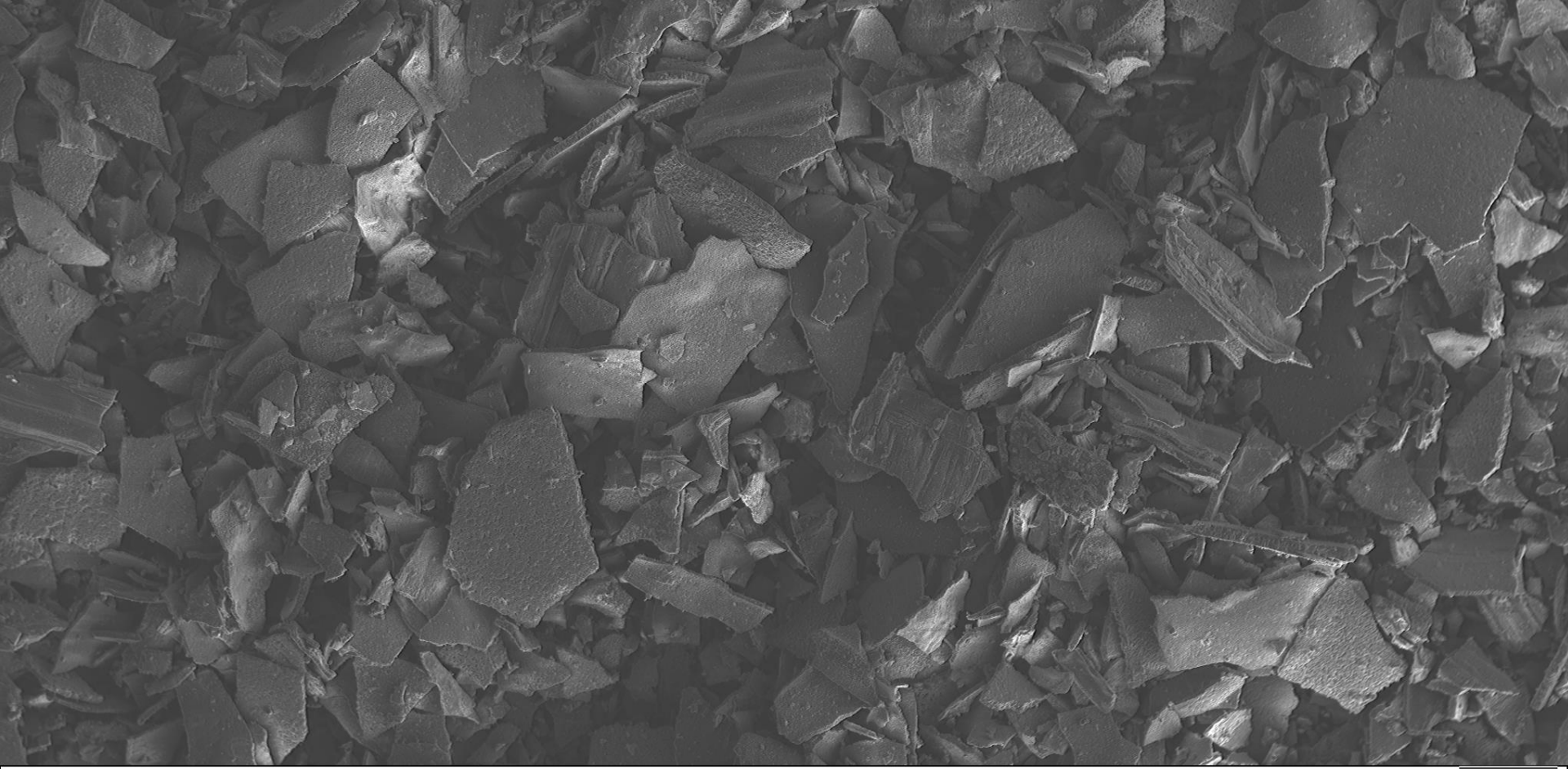
Oxide Characterisation



SEM

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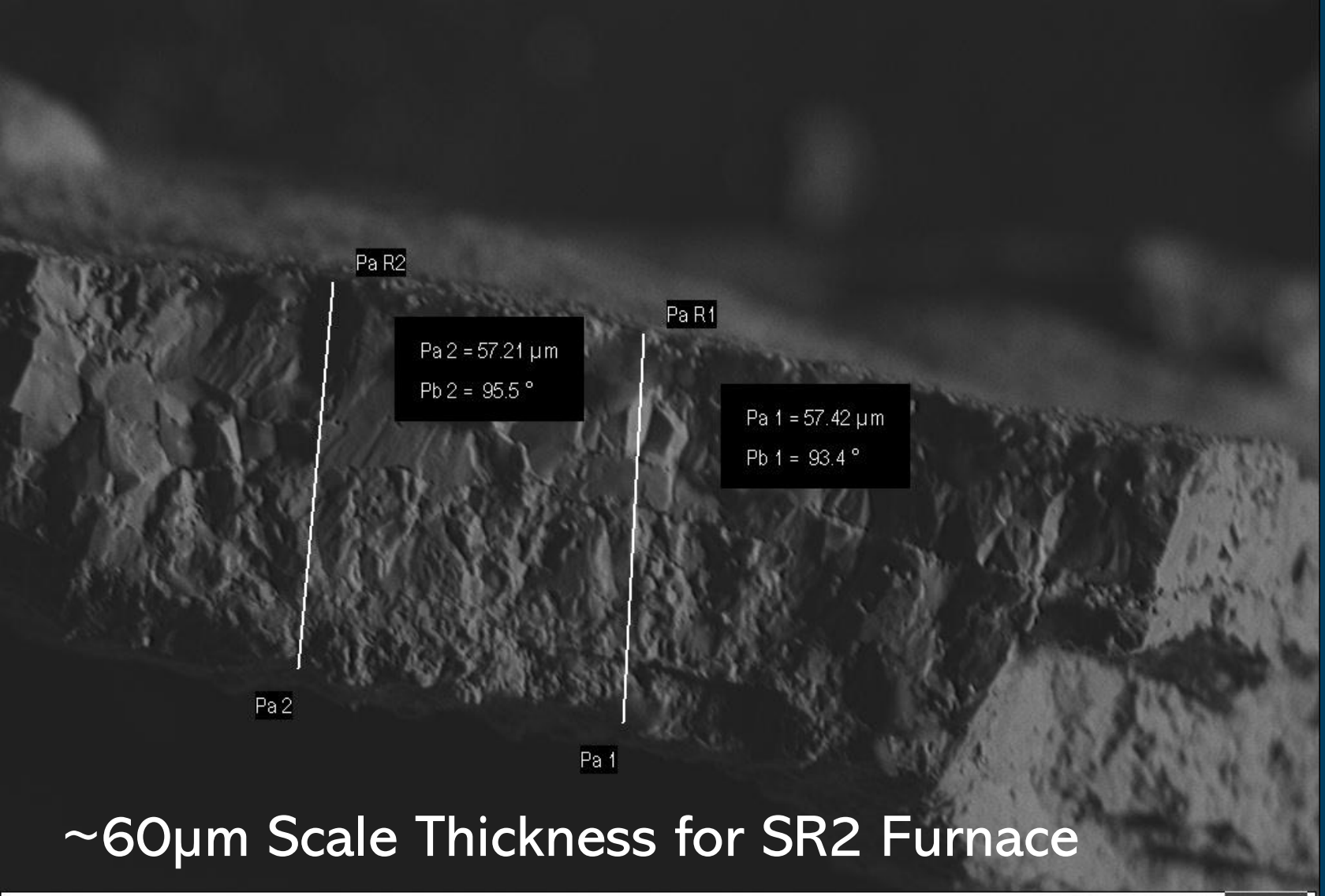
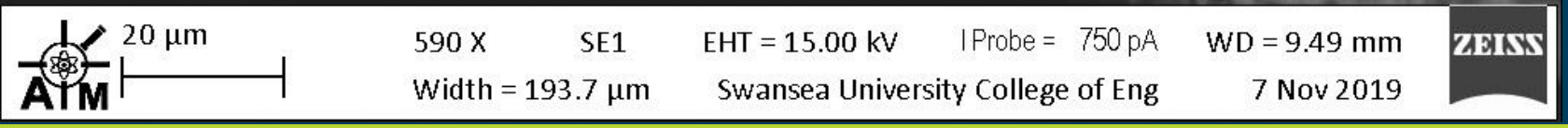


~60µm Scale Thickness for SR2 Furnace



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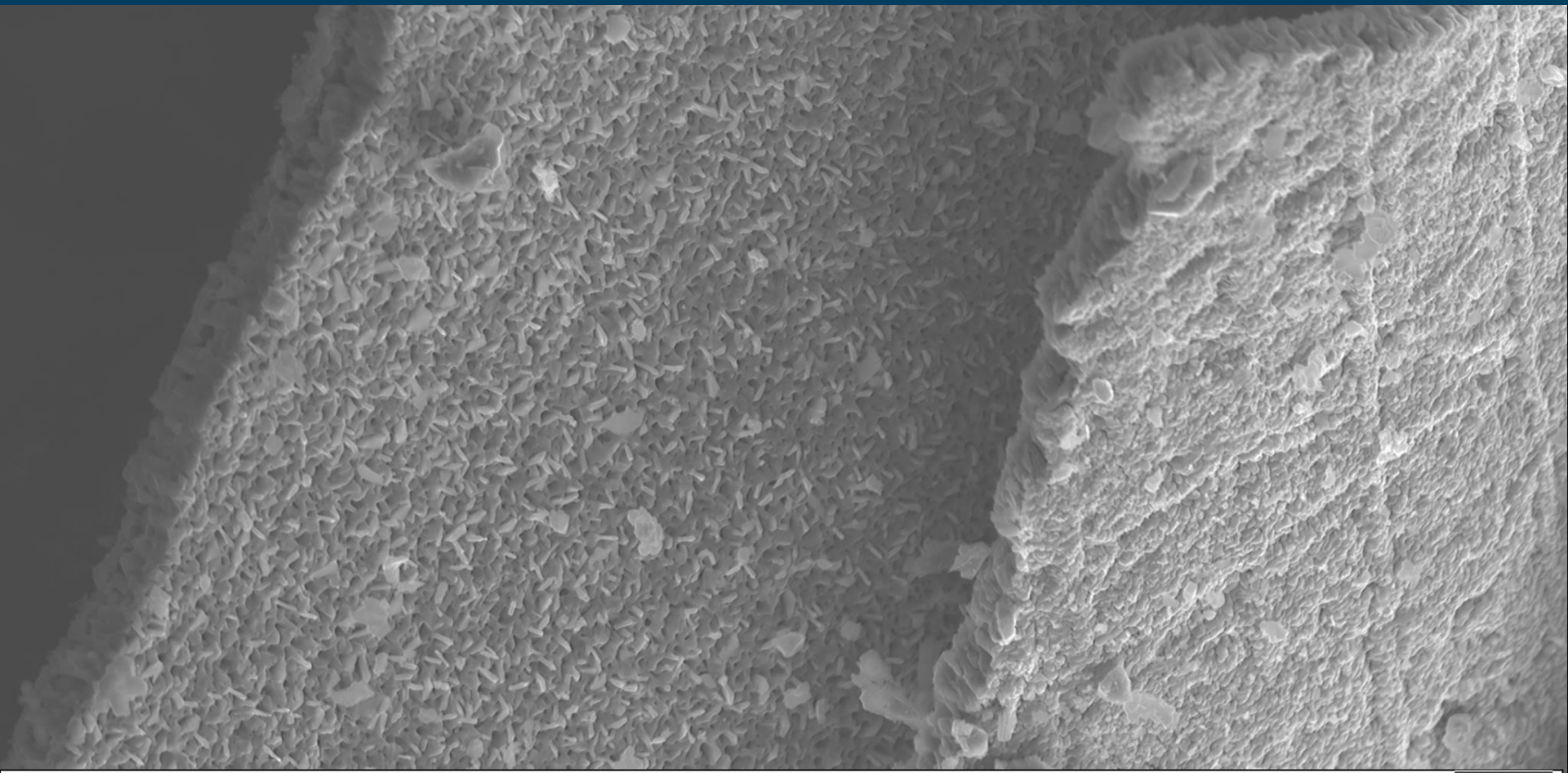

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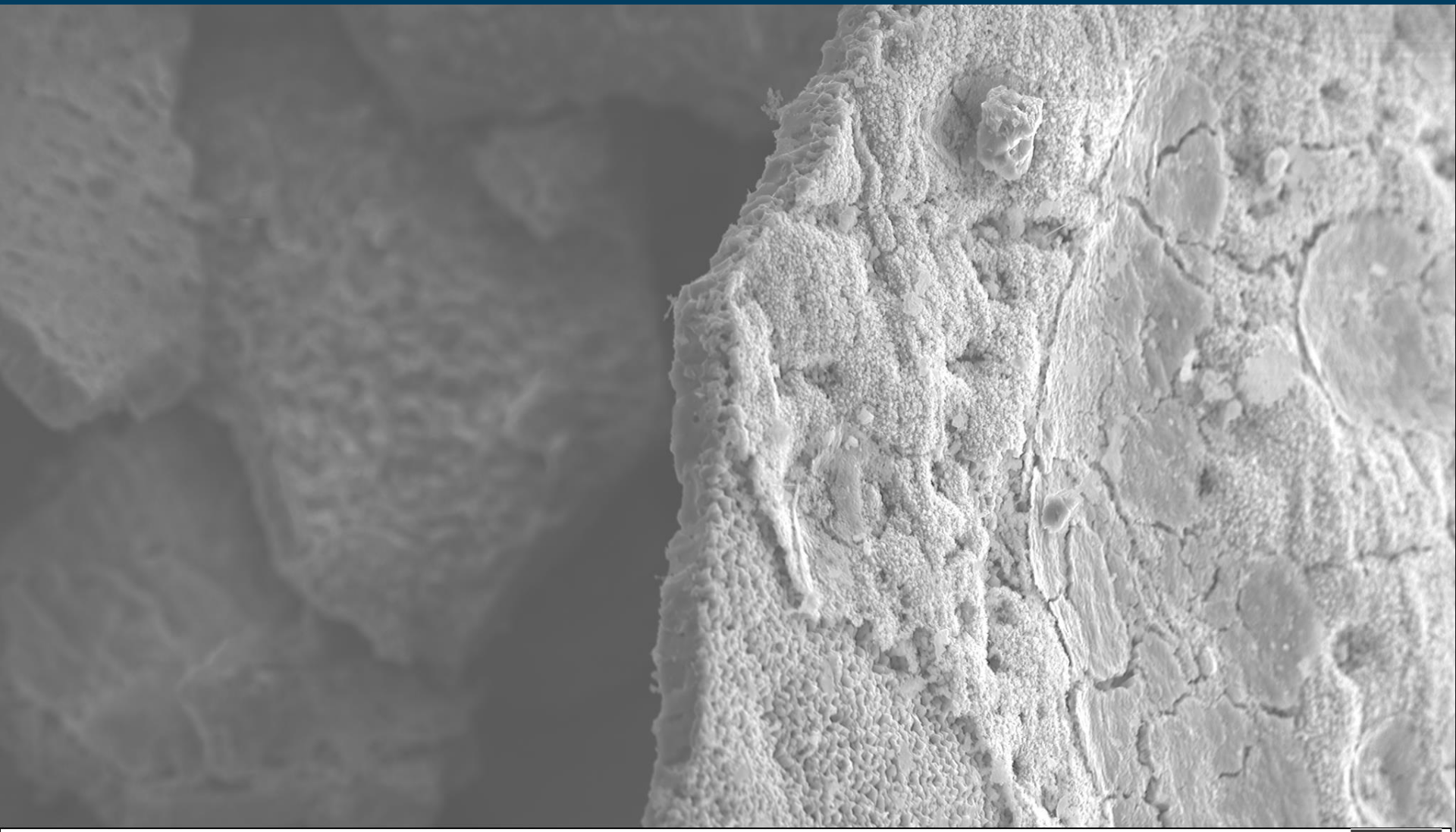

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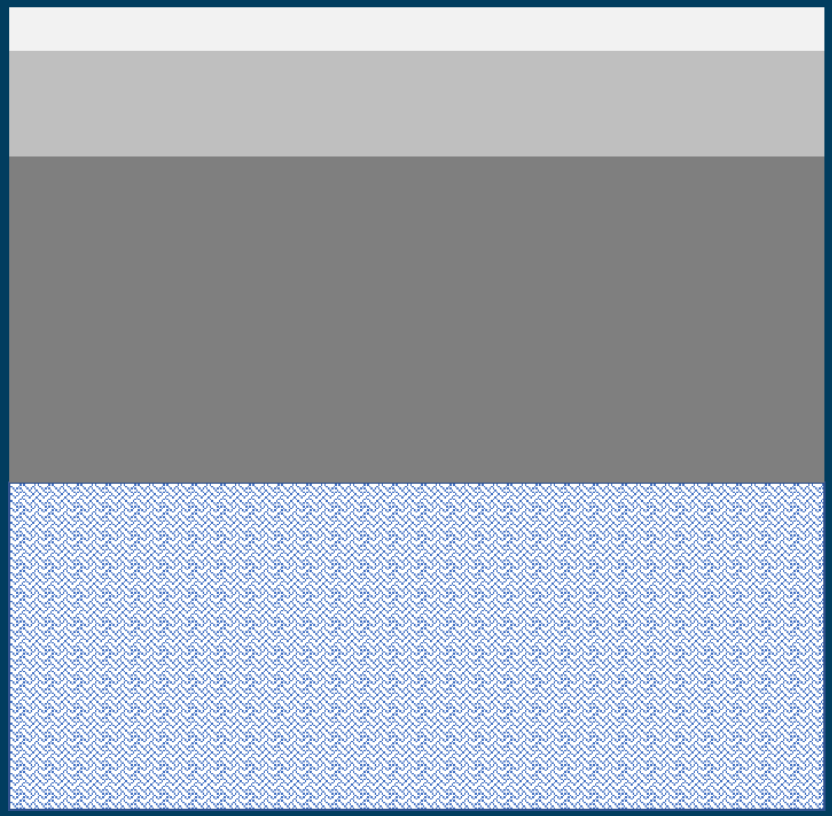

Haemetite Fe_2O_3 1%

Magnetite Fe_3O_4 4%

Wustite FeO 95%

Steel

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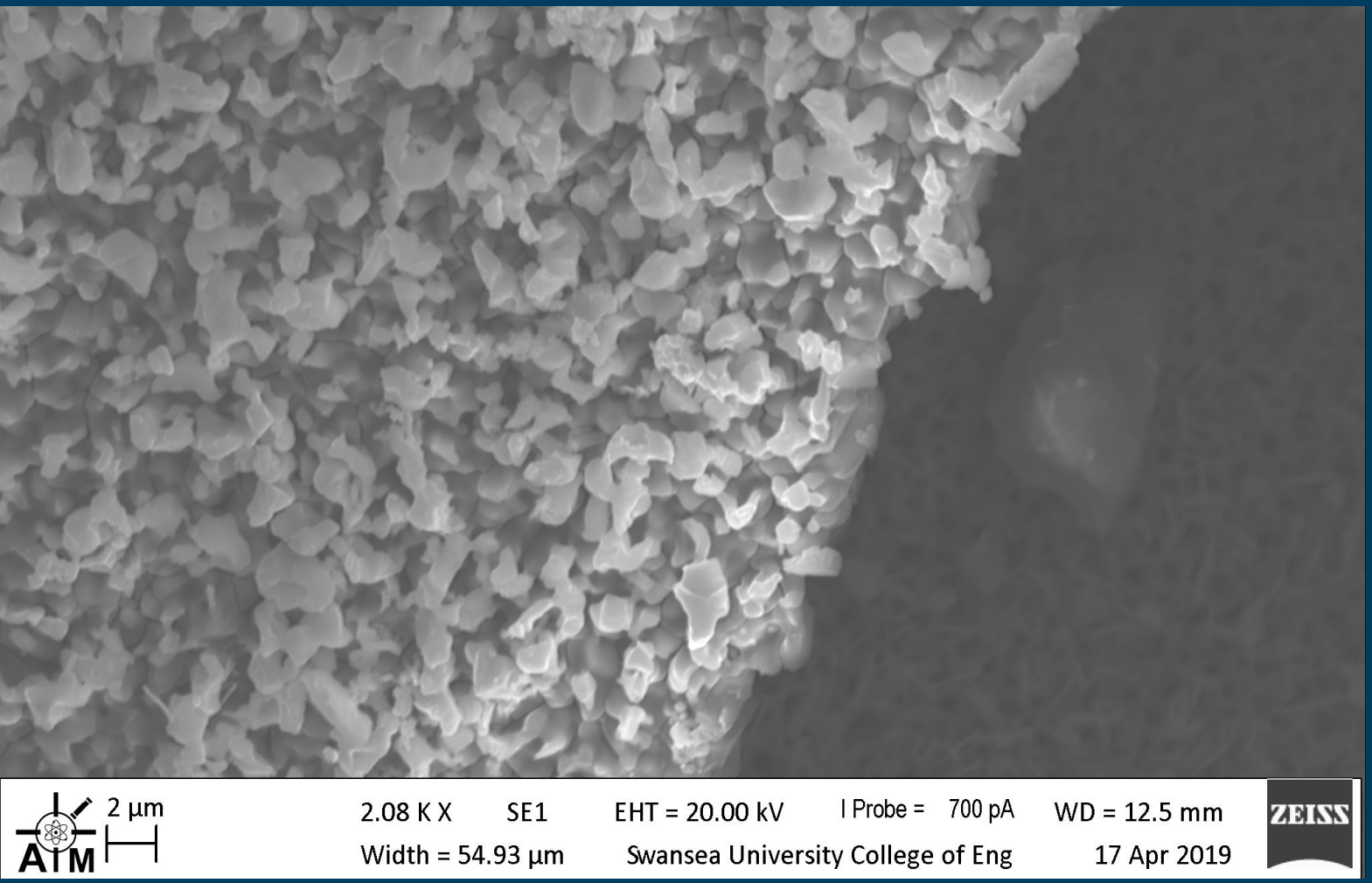

FFC 'Cubic Lattice' Structure

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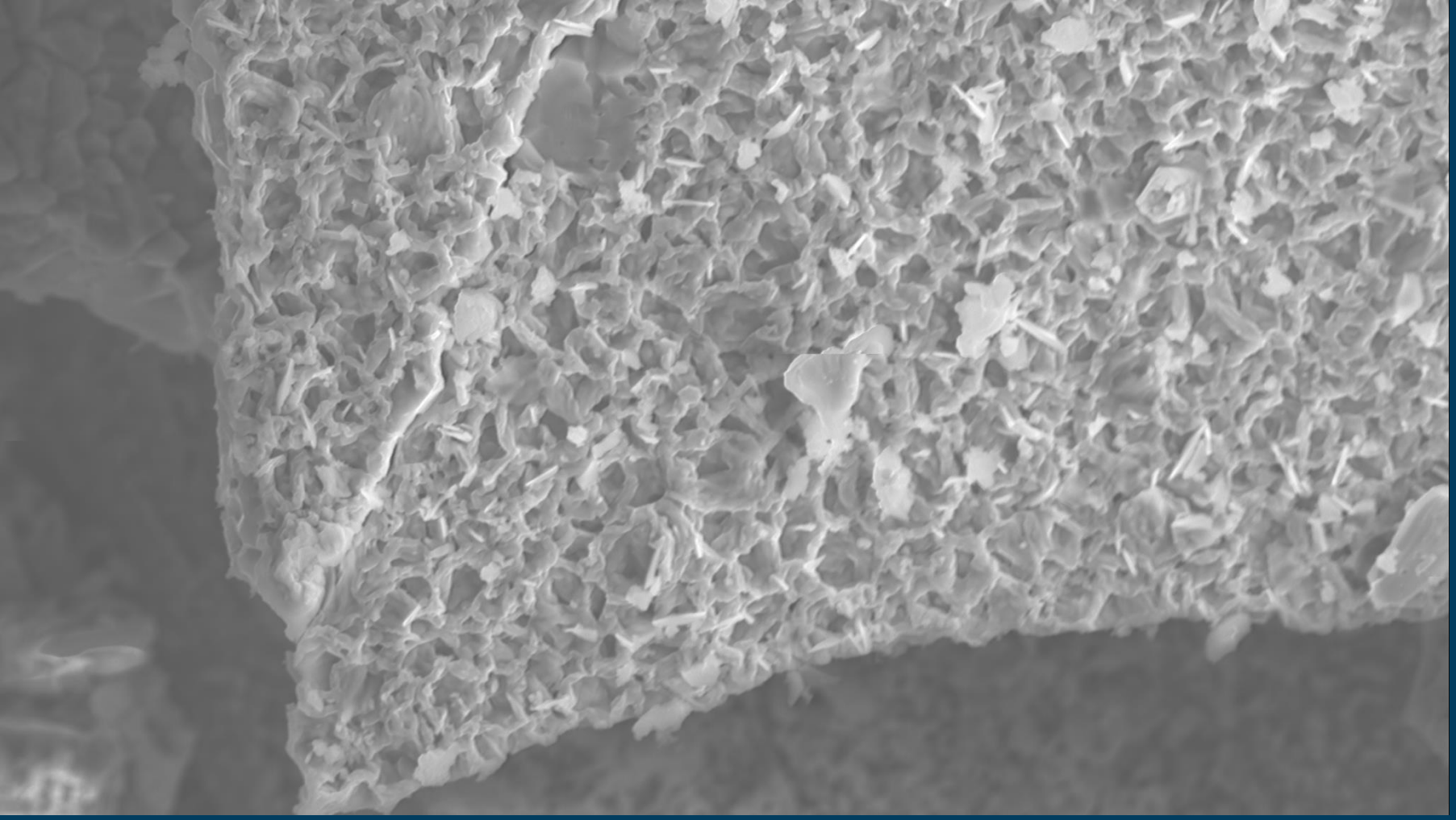

Inverse Spinel Structure

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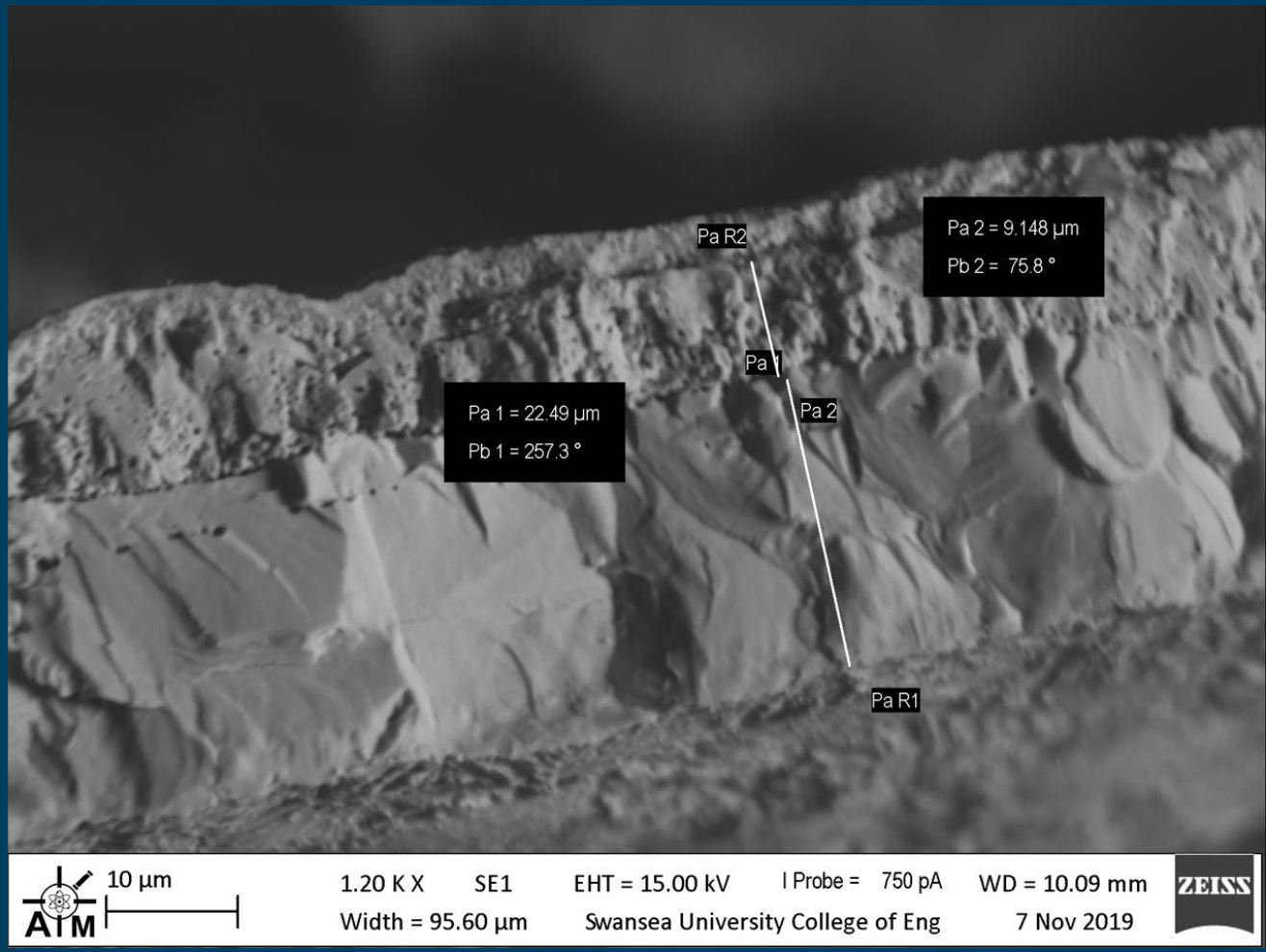
?

Haemetite Fe_2O_3 + Magnetite Fe_3O_4

Magnetite Fe_3O_4

Wustite FeO



Qualitative methods in oxide phase determination

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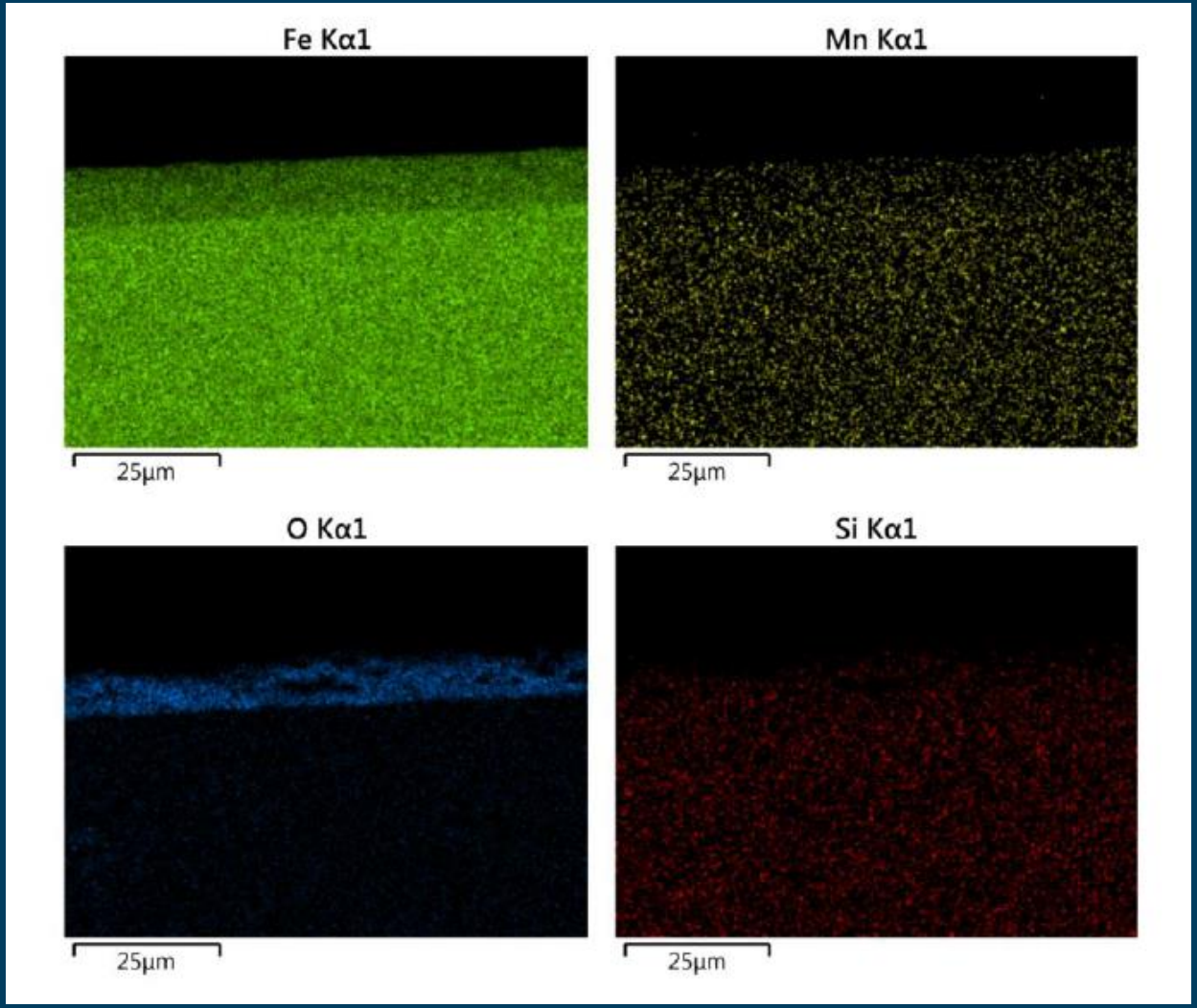
EDS



1.Outline the boundary of oxide phases – not what is present

2. Indicates a phase change has occurred between the oxide layers

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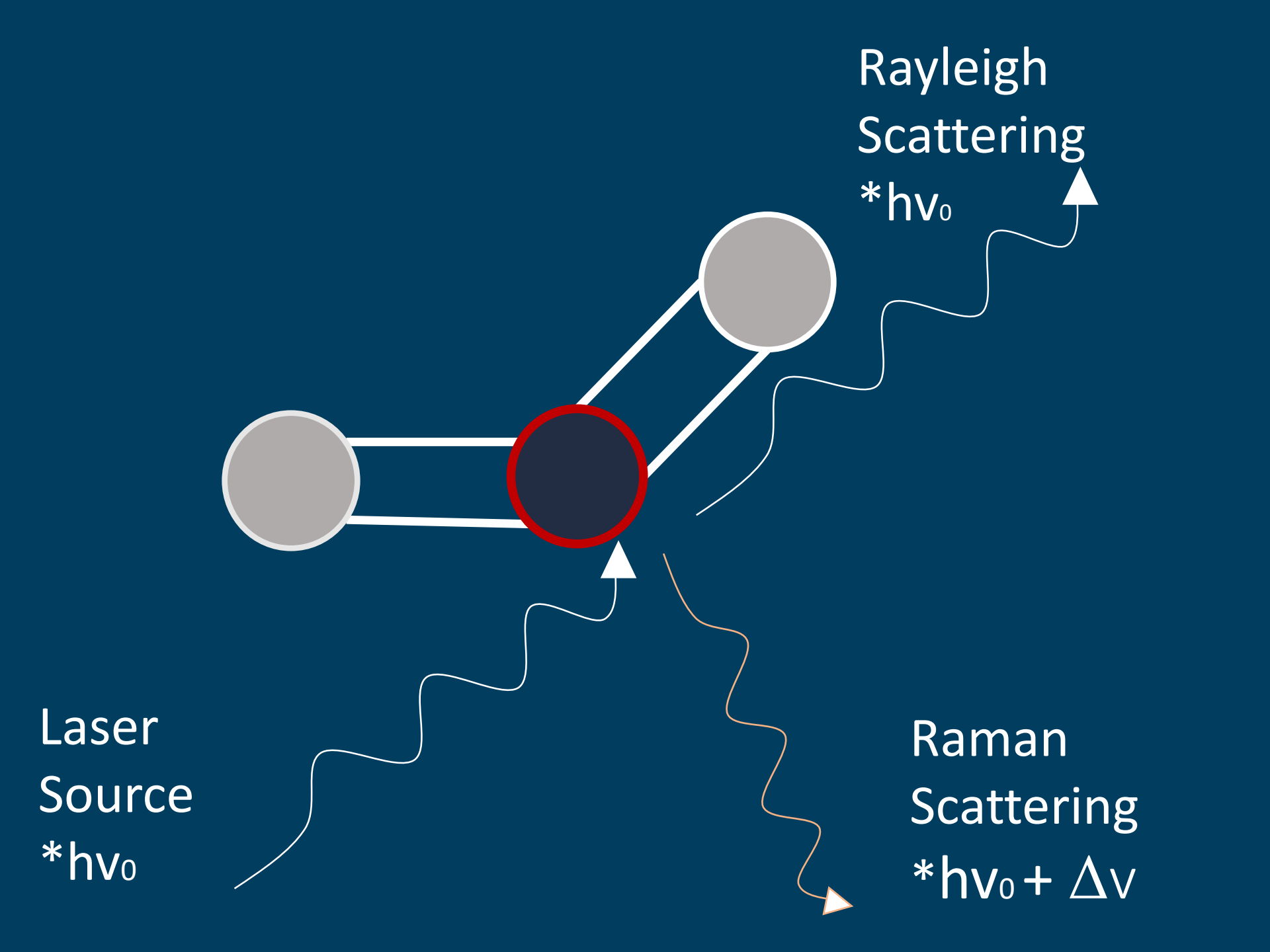
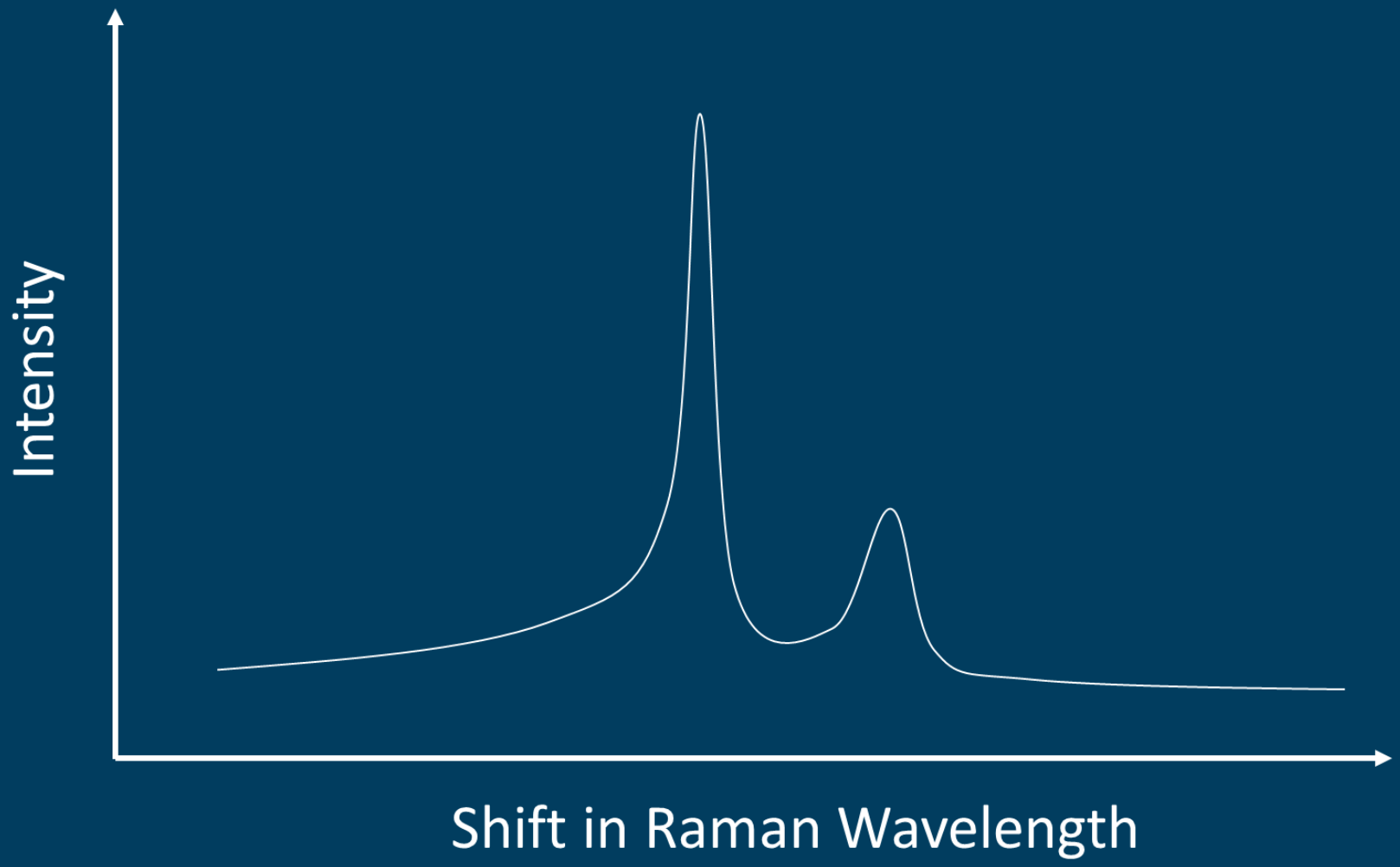
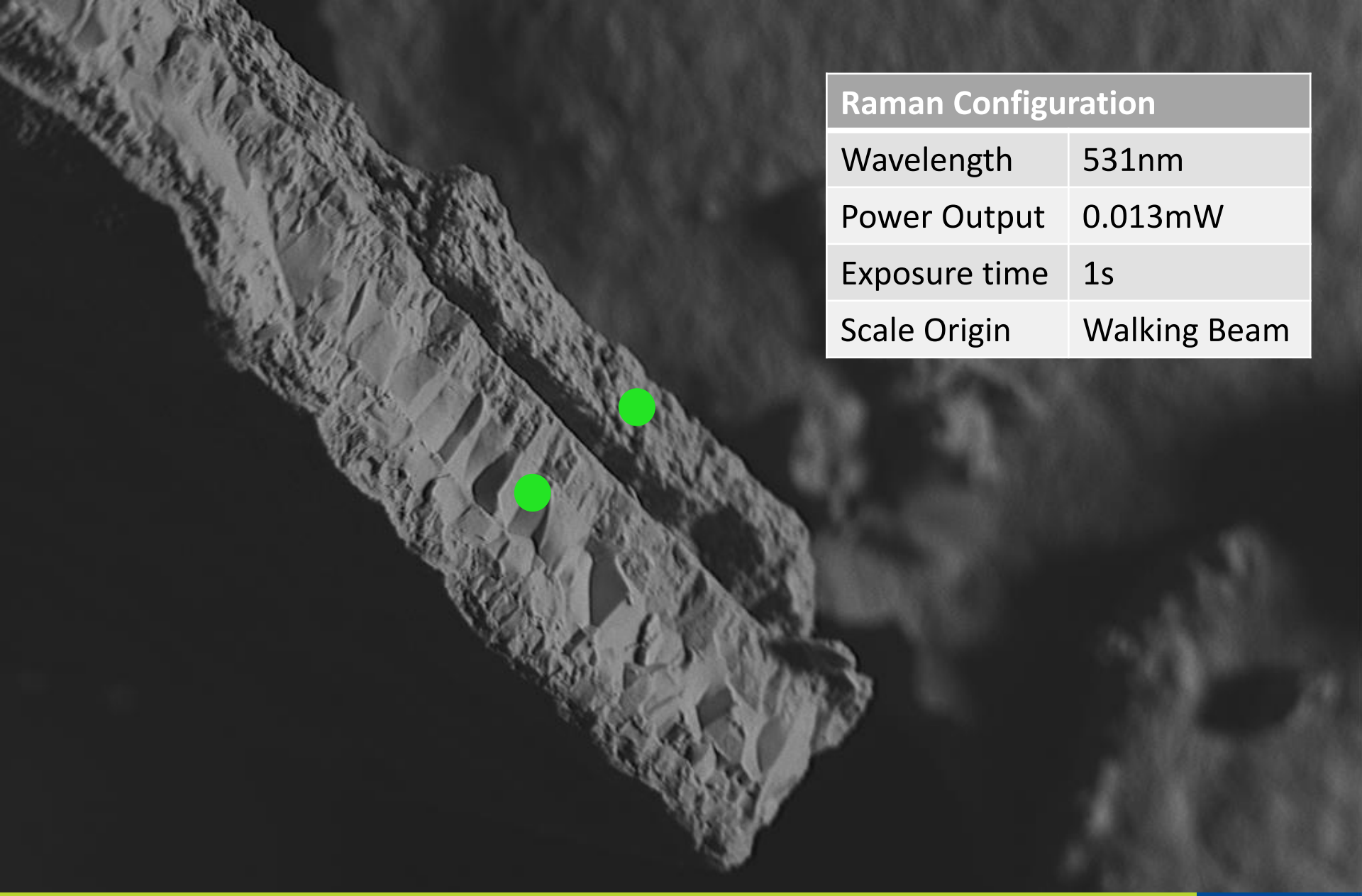
Raman Spectroscopy


Rayleigh
Scattering
*hv0
Laser
Source
*hv0
Raman
Scattering
*hv0 + Δv


Intensity
Shift in Raman Wavelength



Raman Configuration	
Wavelength	531nm
Power Output	0.013mW
Exposure time	1s
Scale Origin	Walking Beam

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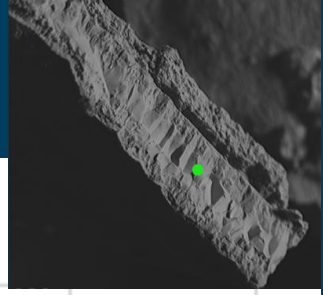
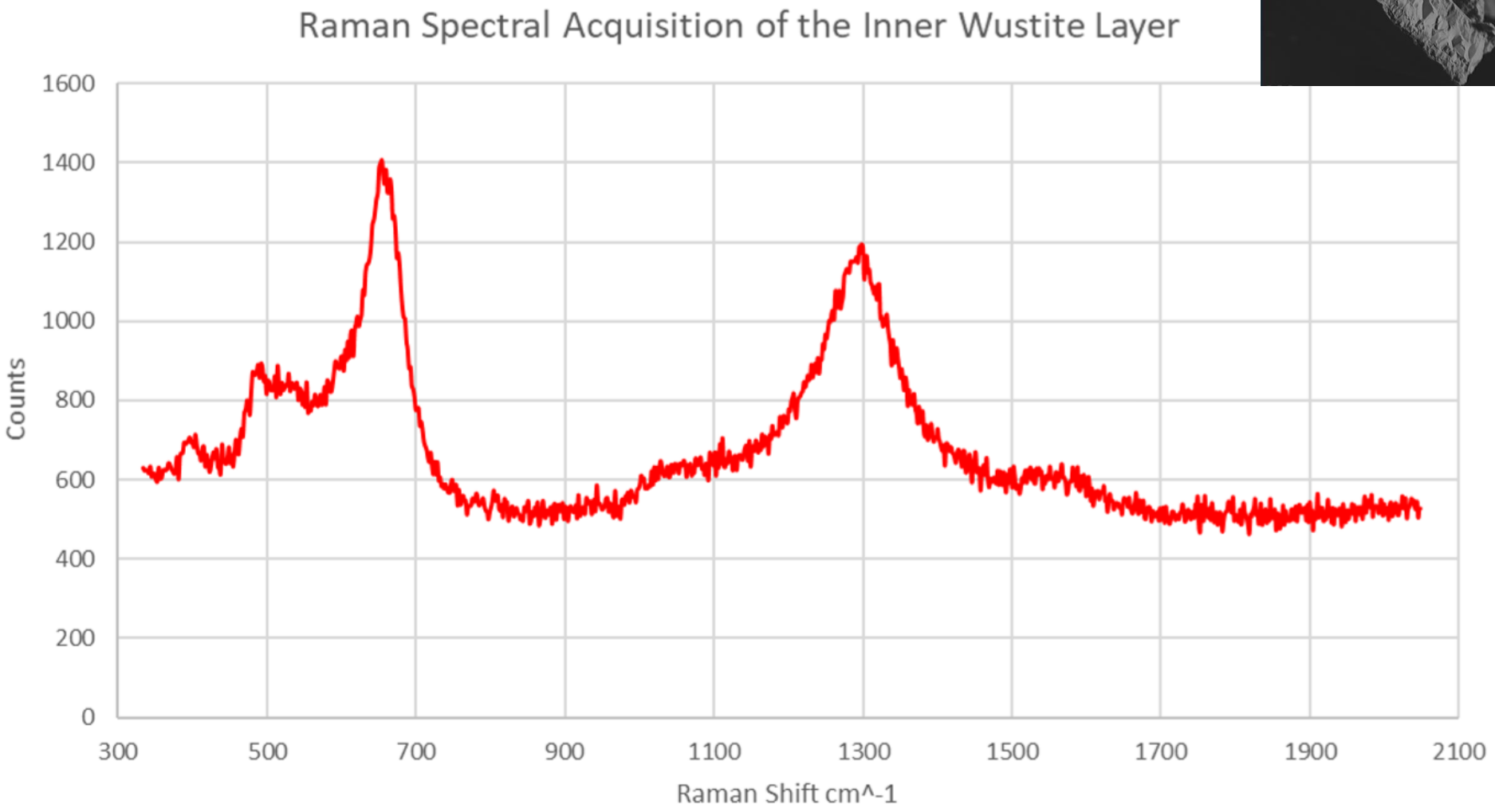
531nm laser source at the Inner Layer of Scale





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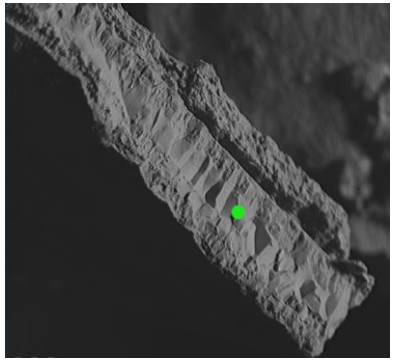
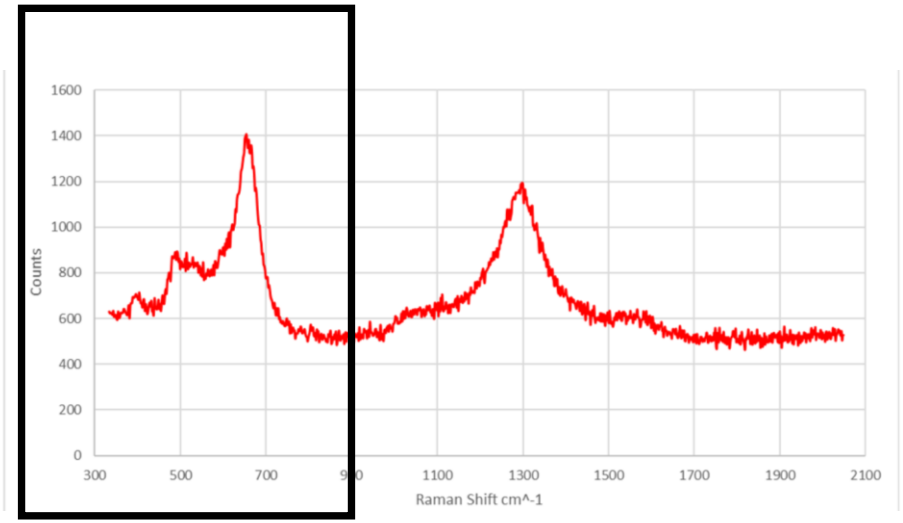
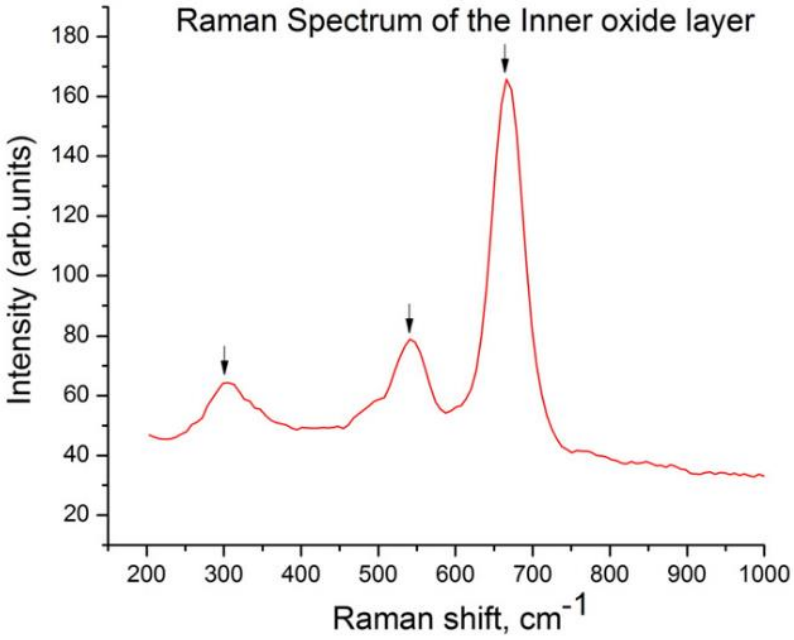


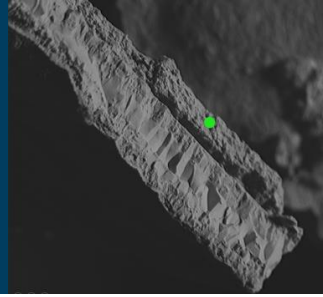
"Mechanical properties and phases determination of low carbon steel oxide scales formed at 1200 °C in air"
Zambrano et al 2015.

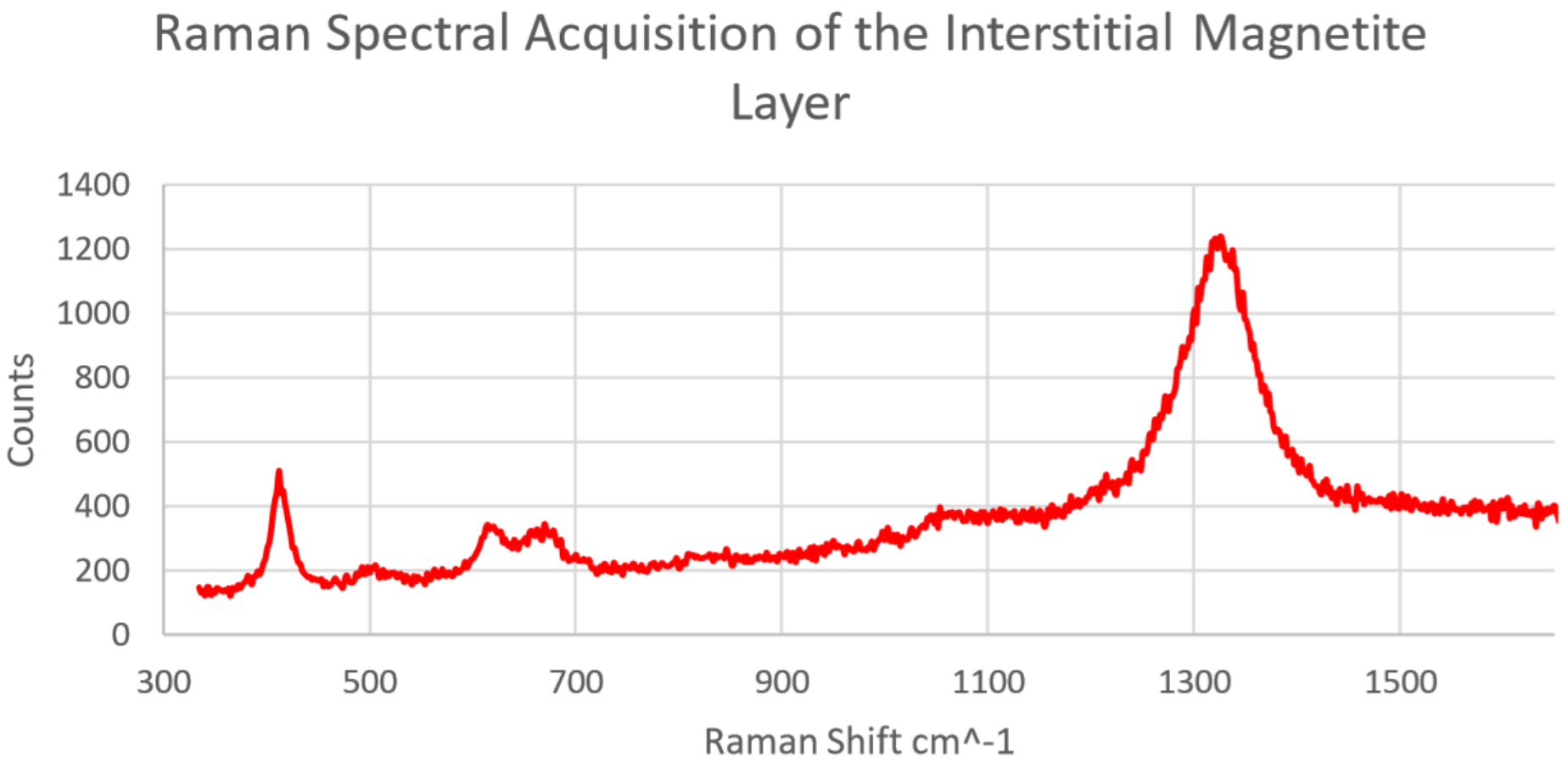


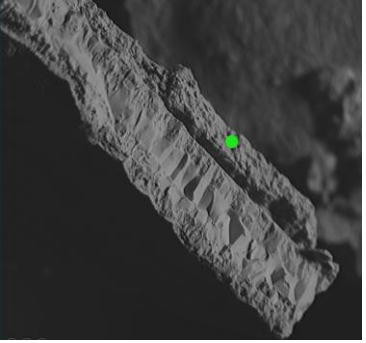


Indicative of Wüstite

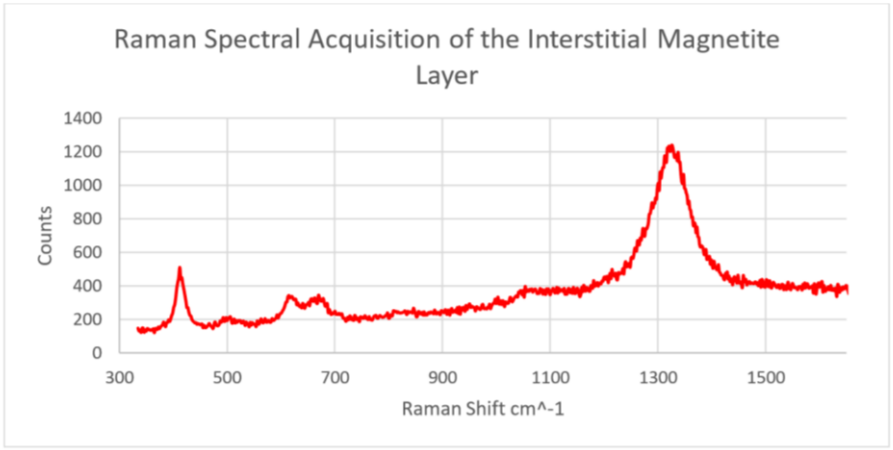
531nm laser source at the Outer Layer of Scale

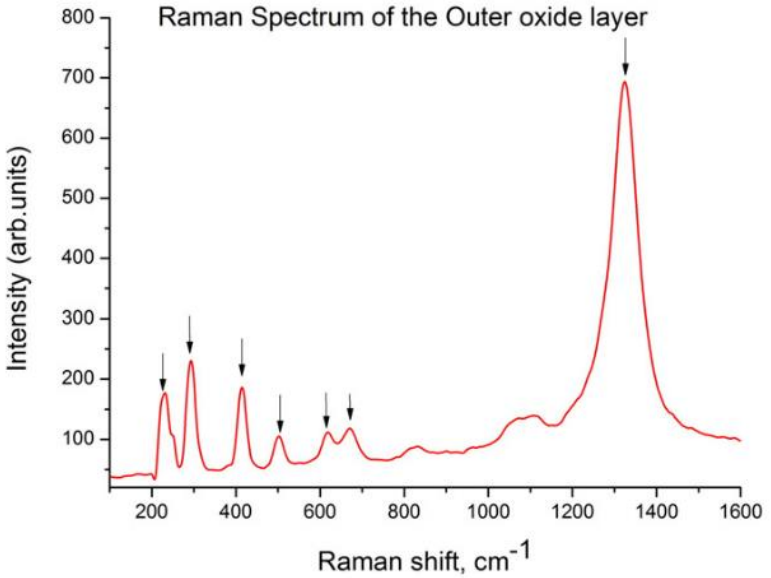






"Mechanical properties and phases determination of low carbon steel oxide scales formed at 1200 °C in air" Zambrano et al 2015.





Indicative of Haematite/Magnetite

1. What oxide are we dealing with?
2. How exactly does it grow?
3. Can you prevent it's formation?

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Oxide Kinetics

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Simultaneous Thermal Analysis

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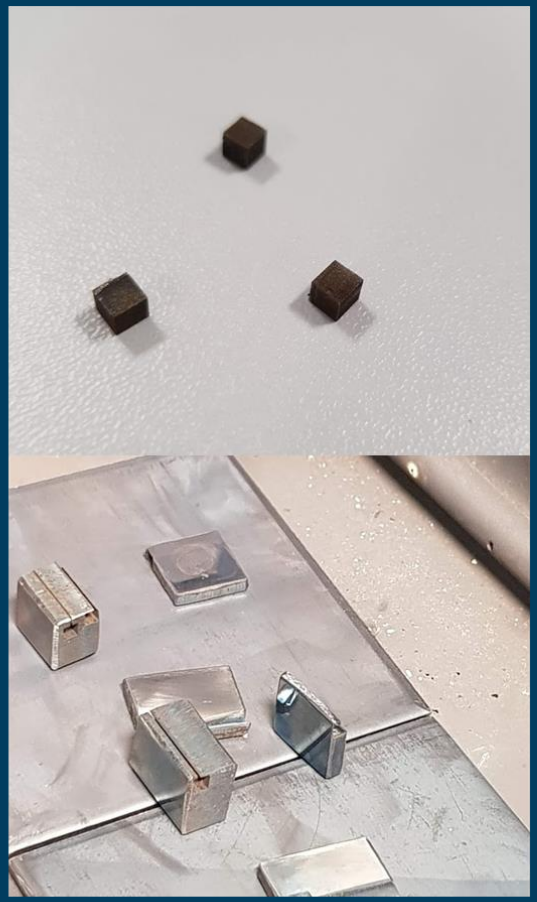

TGA + DSC

3mm² Cubic Samples

10% O_2 flow

Thermal Cycle –
Set Ramp Rates

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Thermogravimetric Analysis

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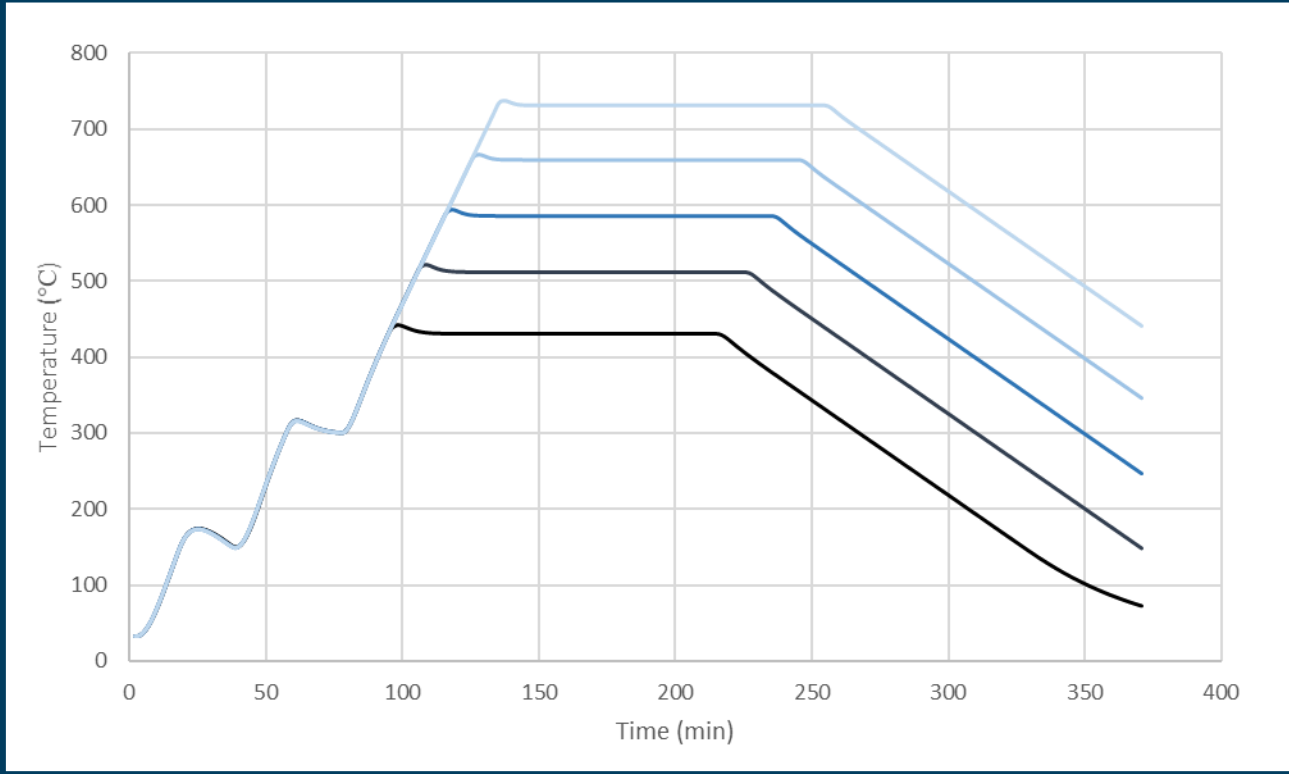
Incremental increase of 50°C

Isothermal Soak at set Temperature for 2 hours

Fixed cooling rate



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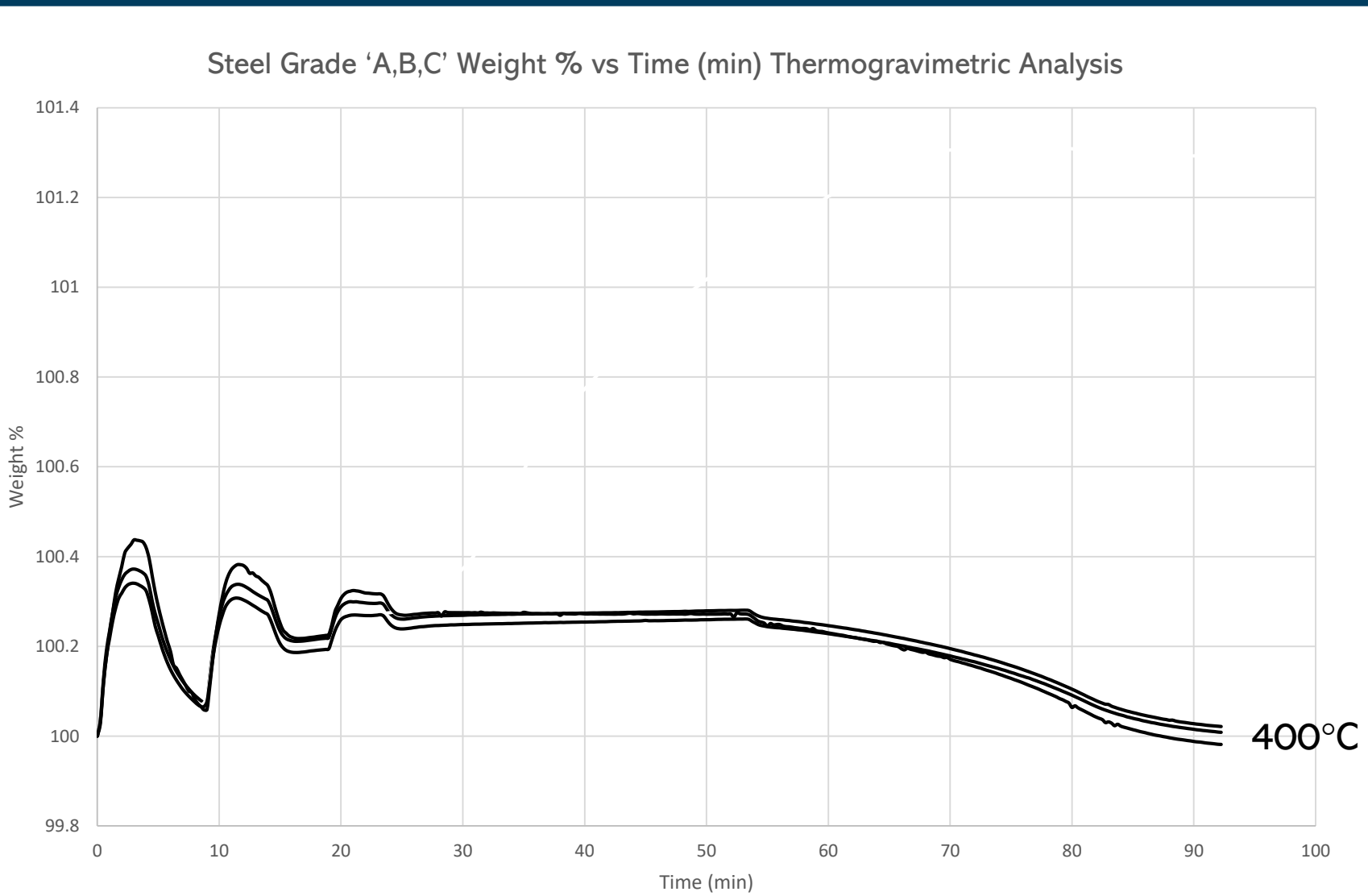
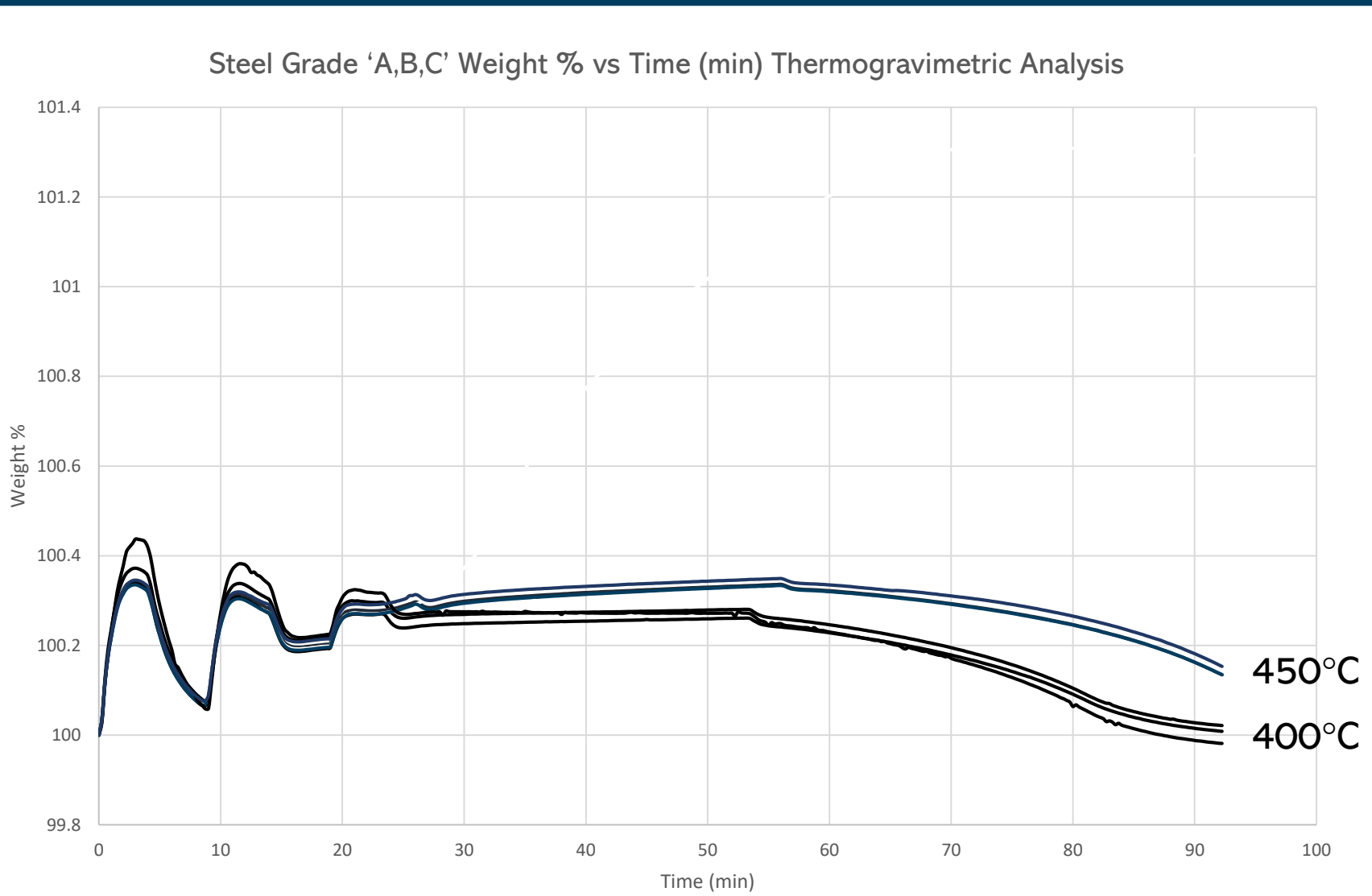

Steel Grade 'A,B,C' Weight % vs Time (min) Thermogravimetric Analysis
101.4
101.2
101
100.8
100.6
100.4
100.2
100
99.8
Weight %
0
10
20
30
40
50
60
70
80
90
100
Time (min)
400°C



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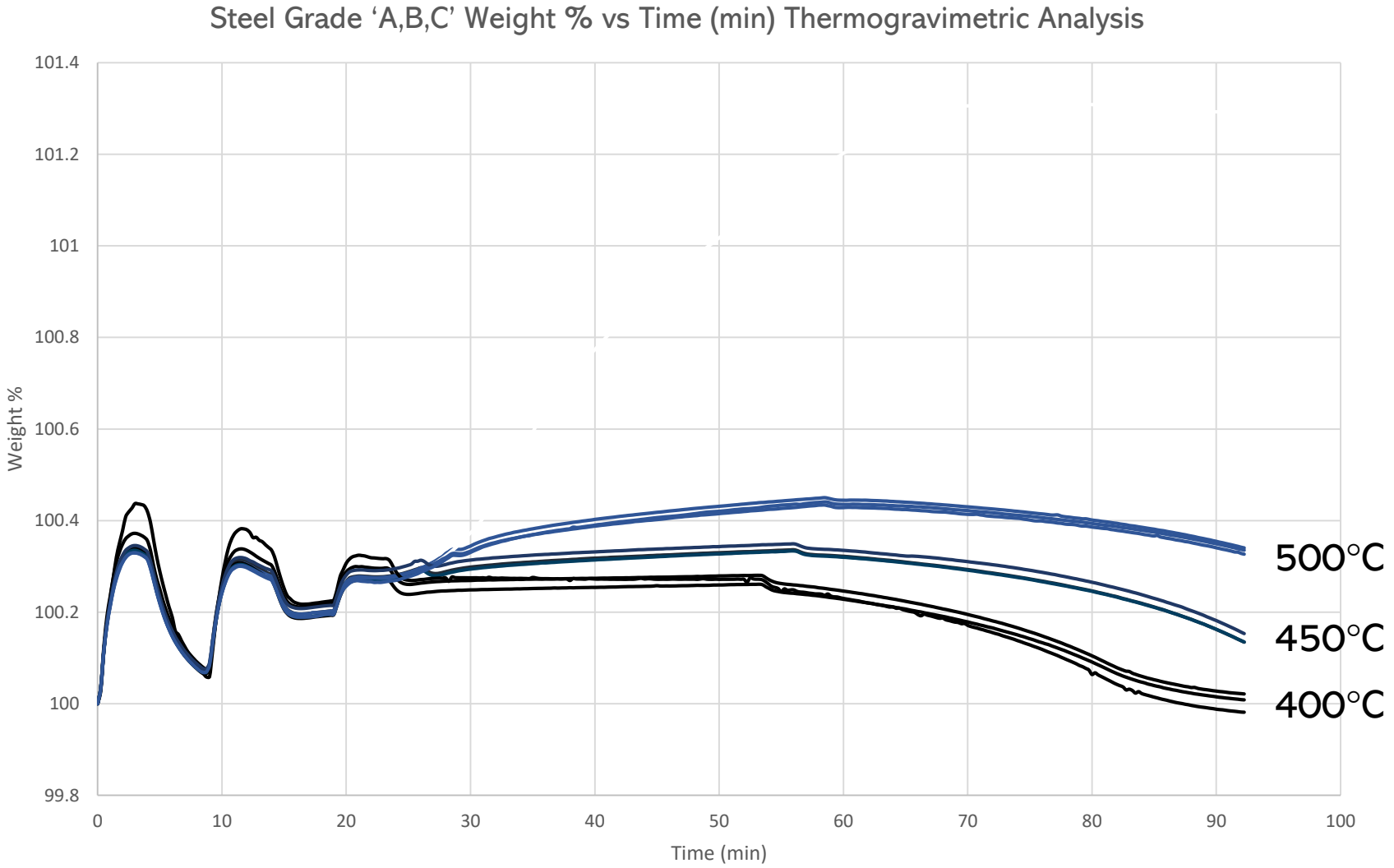

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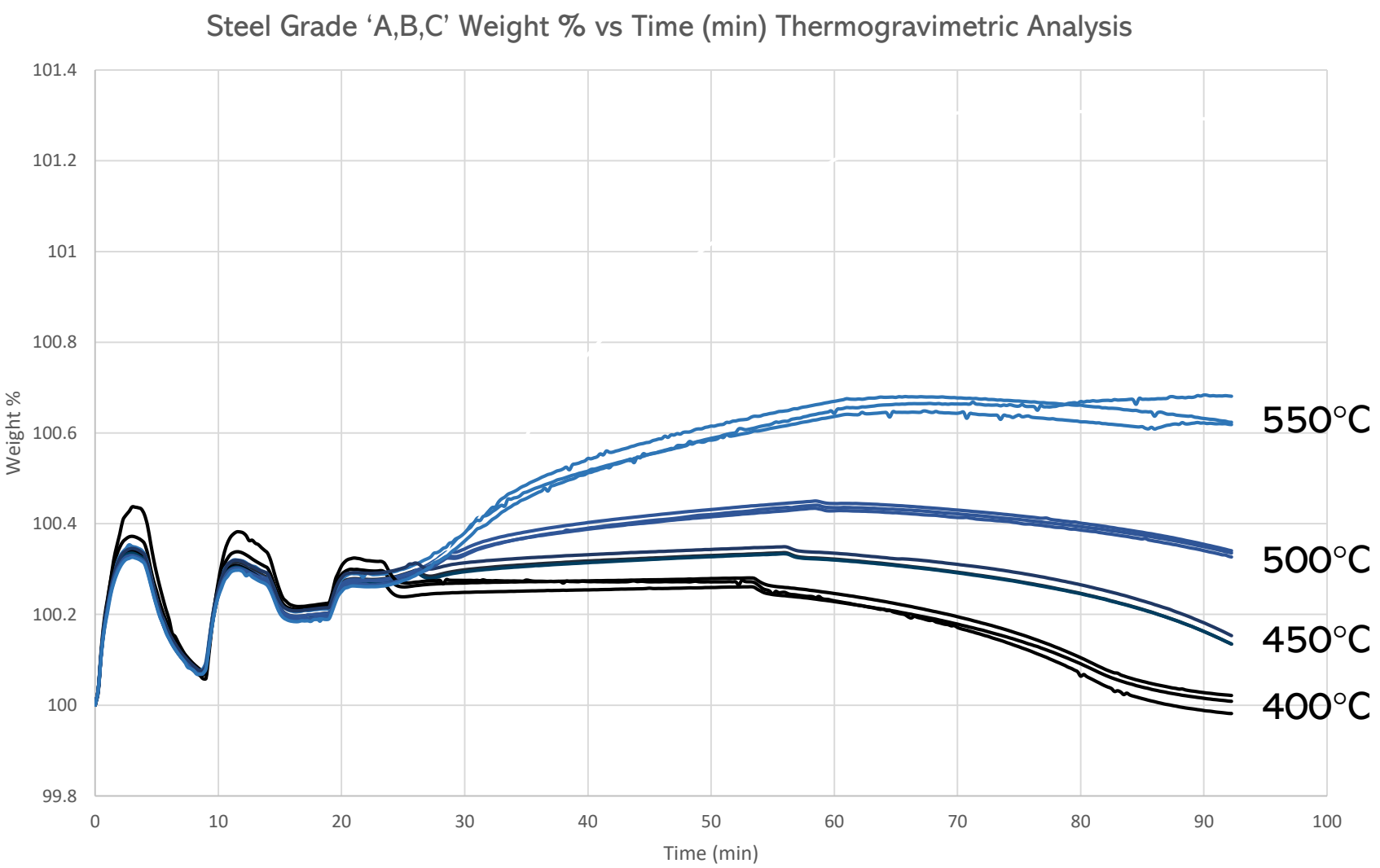

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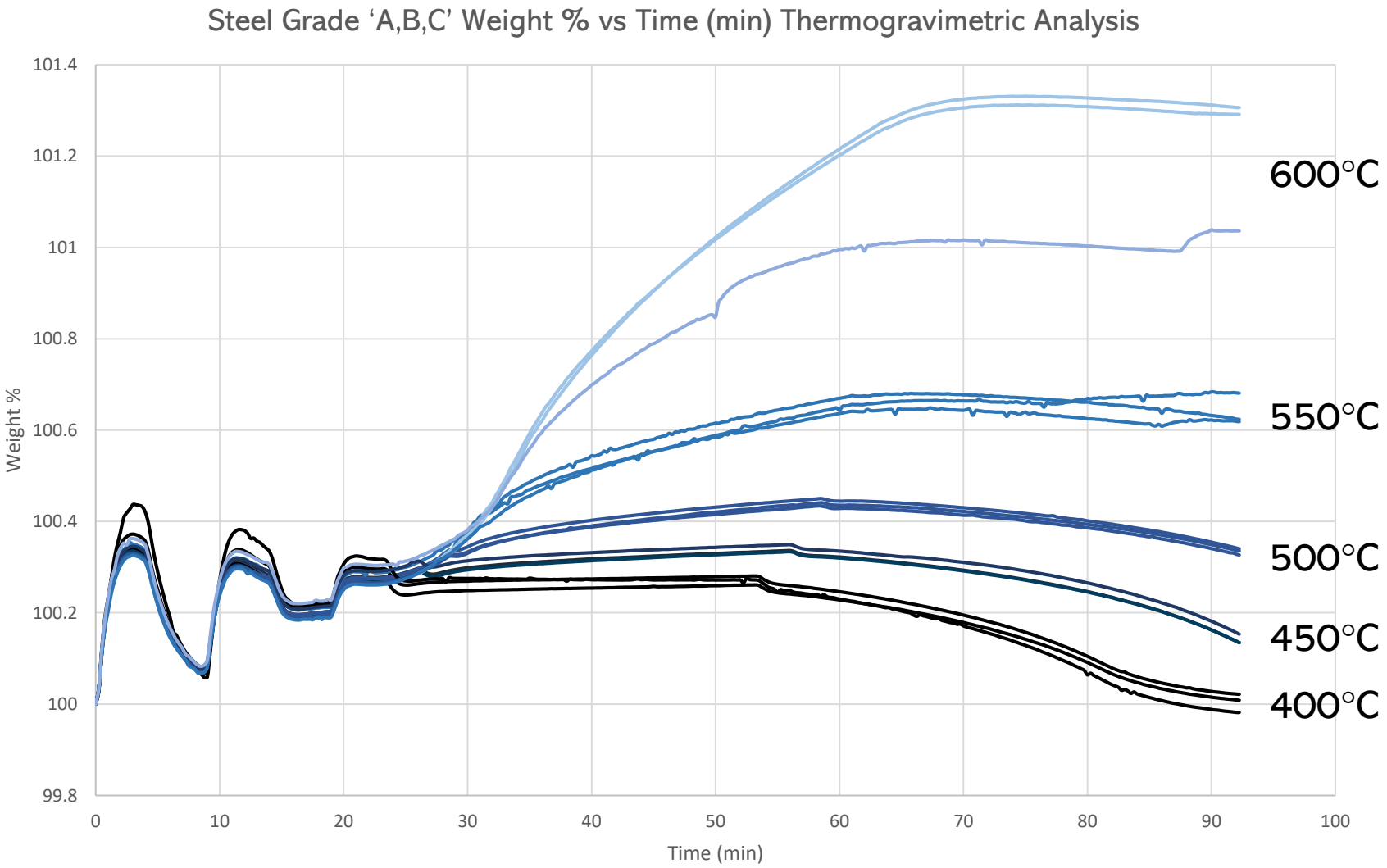

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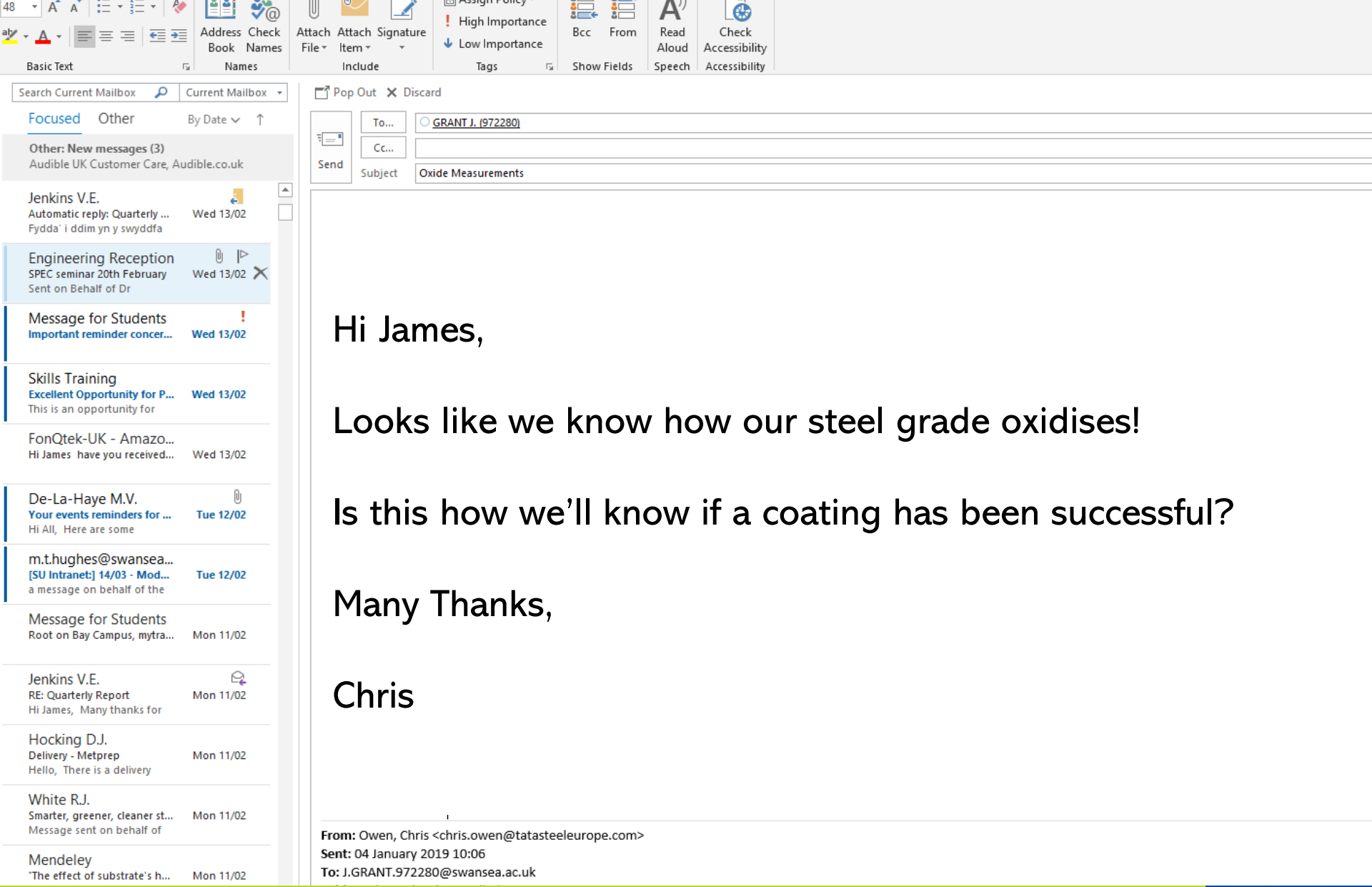


Hi James,

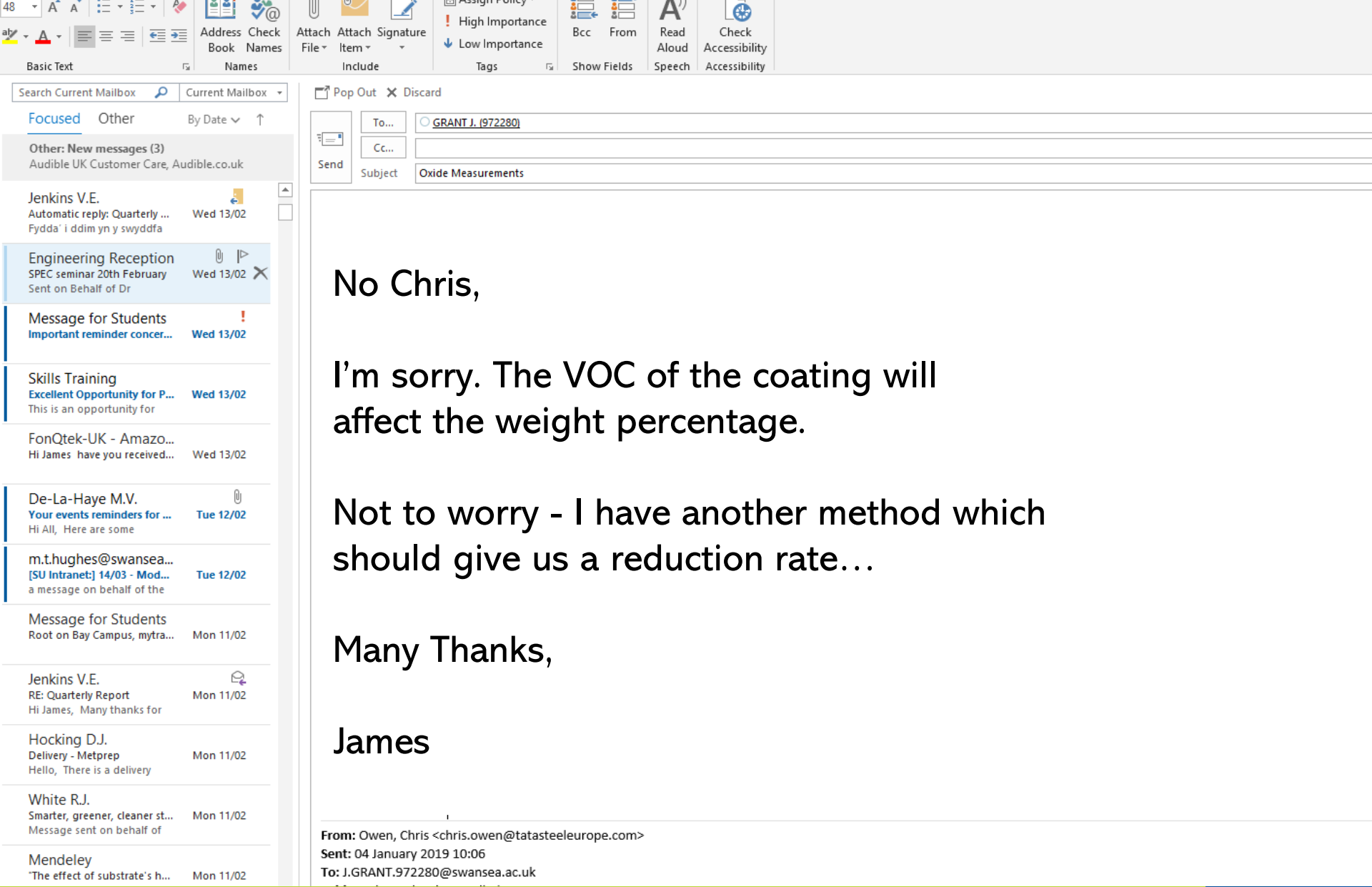
Looks like we know how our steel grade oxidises!

Is this how we'll know if a coating has been successful?

Many Thanks,

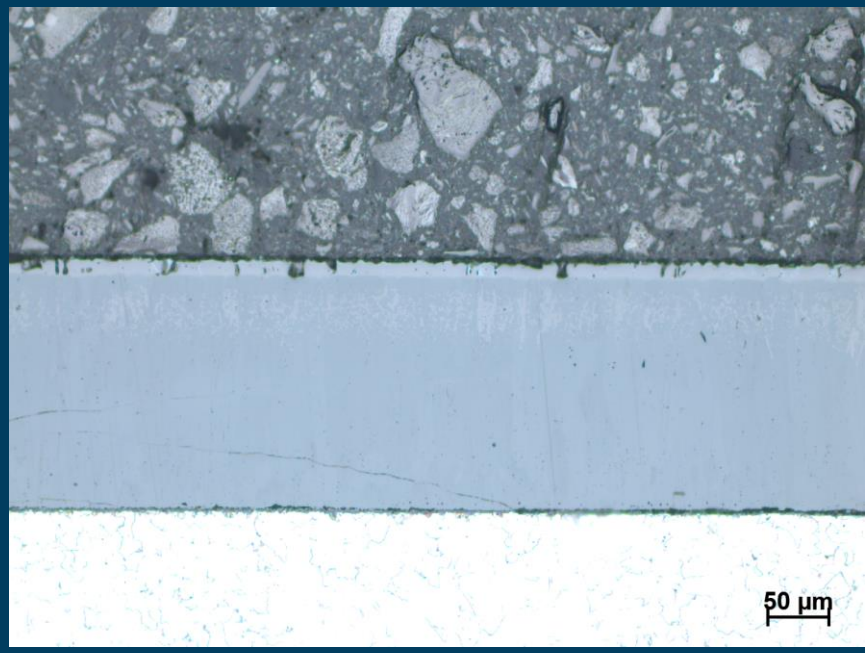
Chris

From: Owen, Chris <chris.owen@tatasteeleurope.com>
Sent: 04 January 2019 10:06
To: J.GRANT.972280@swansea.ac.uk


Address Book
Check Names
Attach File
Attach Item
Signature
High Importance
Low Importance
Bcc
From
Read Aloud
Check Accessibility
Basic Text
Names
Include
Tags
Show Fields
Speech
Accessibility
Search Current Mailbox
Current Mailbox
Focused
Other
By Date
Other: New messages (3)
Audible UK Customer Care, Audible.co.uk
Jenkins V.E.
Automatic reply: Quarterly ...
Wed 13/02
Fydda' i ddim yn y swyddfa
Engineering Reception
SPEC seminar 20th February
Wed 13/02
Sent on Behalf of Dr
Message for Students
Important reminder concer...
Wed 13/02
Skills Training
Excellent Opportunity for P...
Wed 13/02
This is an opportunity for
FonQtek-UK - Amazo...
Hi James have you received...
Wed 13/02
De-La-Haye M.V.
Your events reminders for ...
Tue 12/02
Hi All, Here are some
m.t.hughes@swansea...
[SU Intranet:] 14/03 - Mod...
Tue 12/02
a message on behalf of the
Message for Students
Root on Bay Campus, mytra...
Mon 11/02
Jenkins V.E.
RE: Quarterly Report
Mon 11/02
Hi James, Many thanks for
Hocking D.J.
Delivery - Metprep
Mon 11/02
Hello, There is a delivery
White R.J.
Smarter, greener, cleaner st...
Mon 11/02
Message sent on behalf of
Mendeley
'The effect of substrate's h...
Mon 11/02
Pop Out
Discard
Send
To...
GRANT J. (972280)
Cc...
Subject
Oxide Measurements
No Chris,
I'm sorry. The VOC of the coating will affect the weight percentage.
Not to worry - I have another method which should give us a reduction rate…
Many Thanks,
James
From: Owen, Chris <chris.owen@tatasteeleurope.com>
Sent: 04 January 2019 10:06
To: J.GRANT.972280@swansea.ac.uk

Cross – Sectional Analysis – Advanced Light Microscopy

Quantifying oxide growth through 2D observations.



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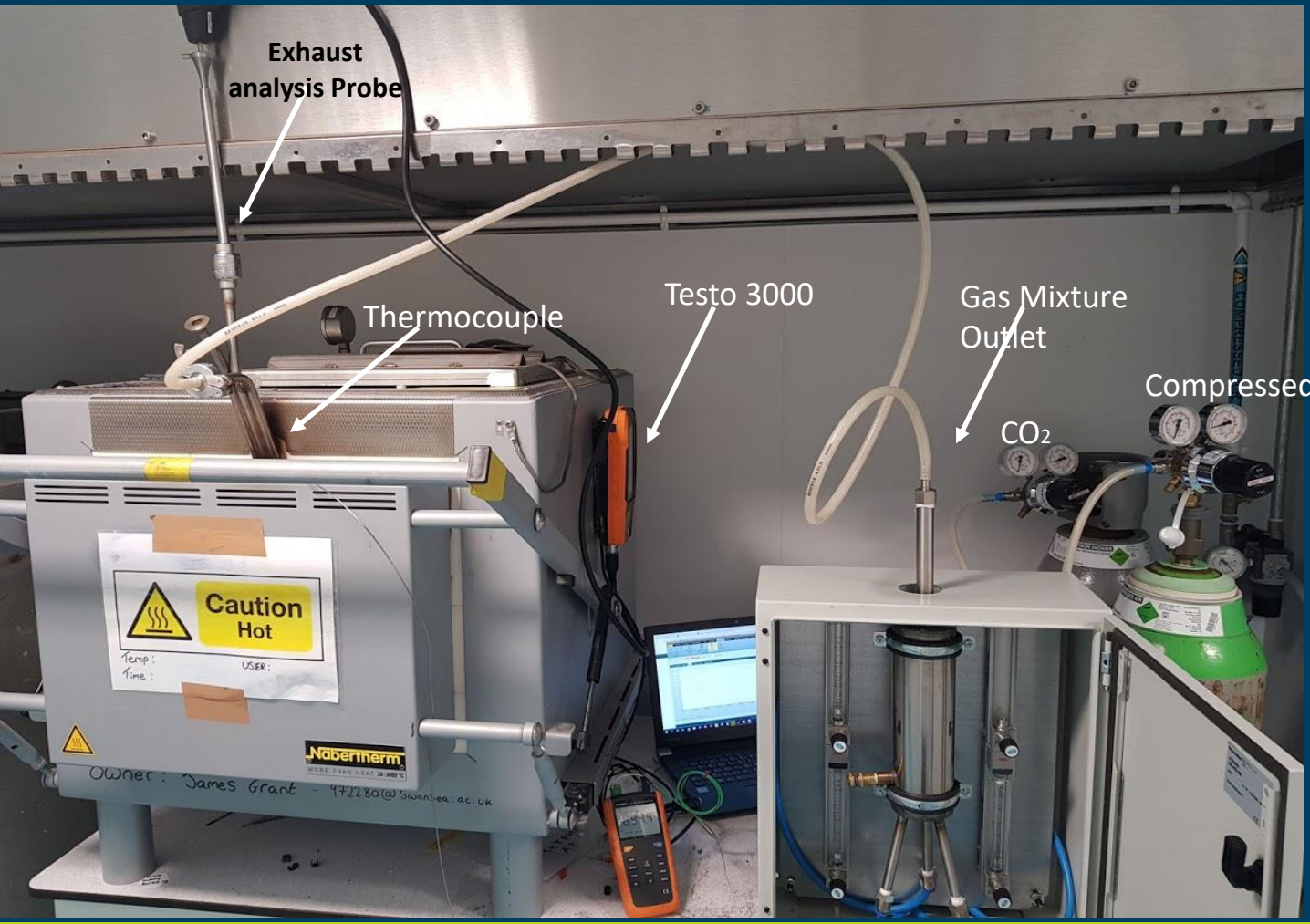
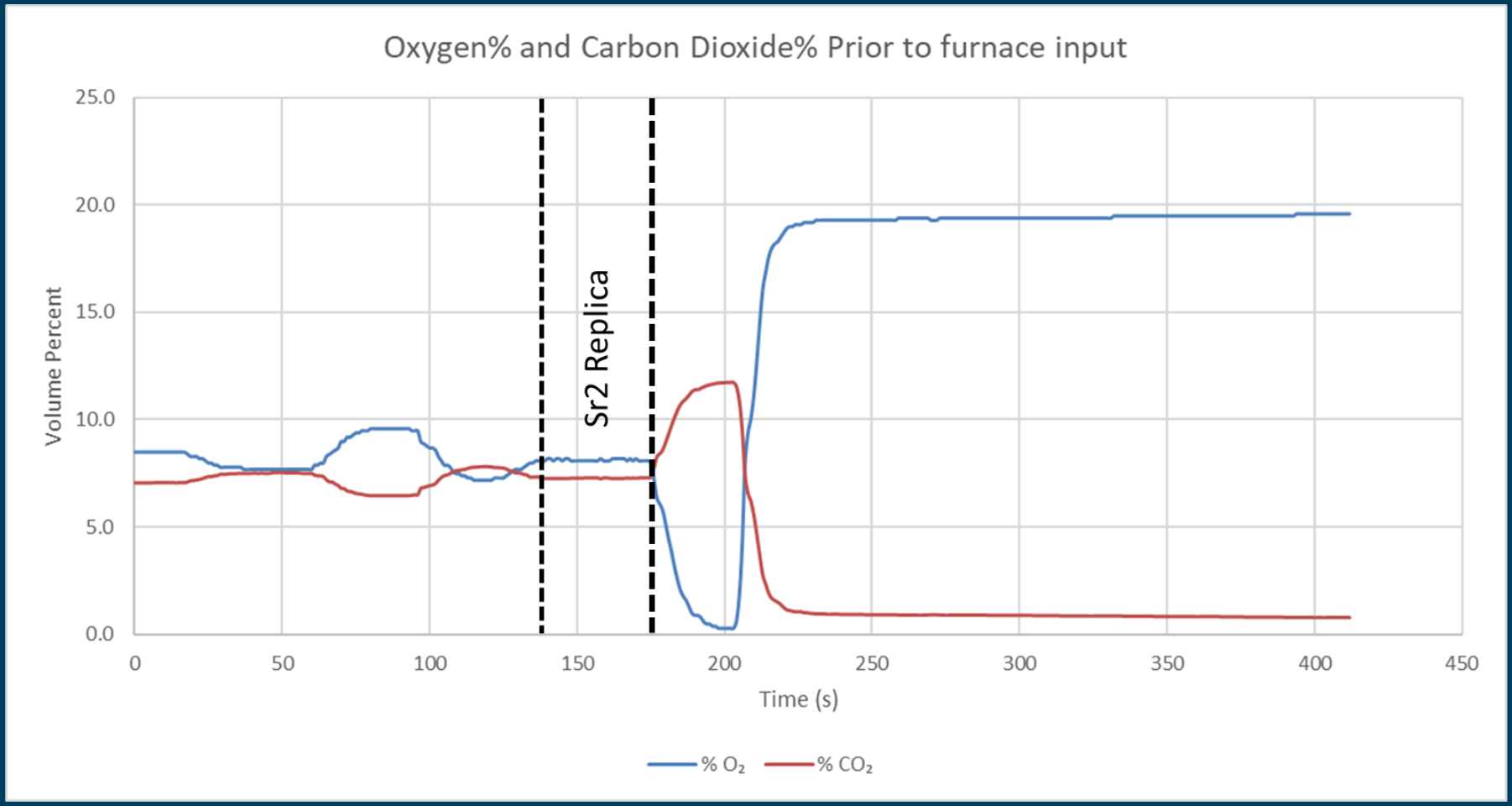


Atmospheric Furnace

Vary the % of each element in the atmosphere

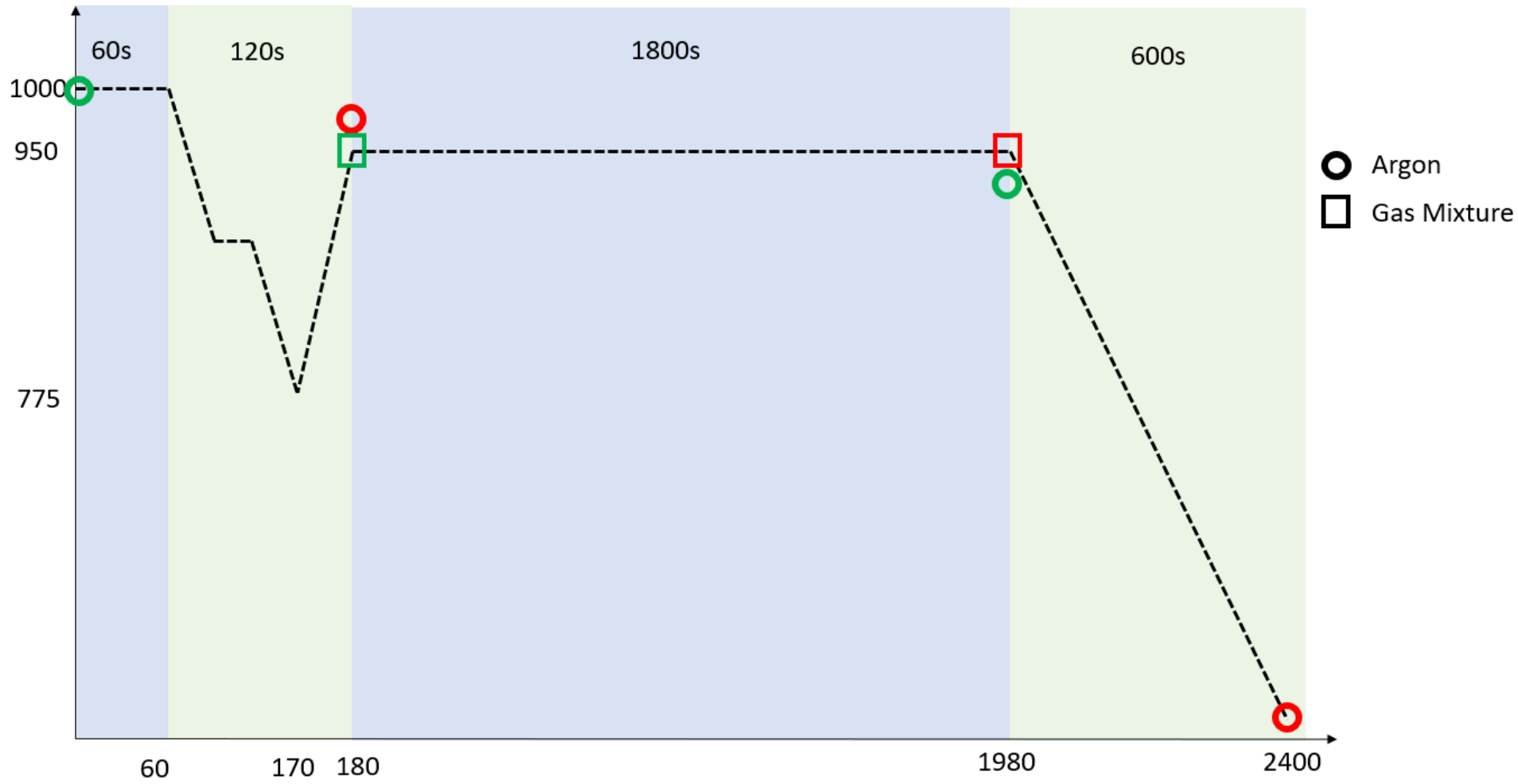


Flowing at 30 L/min

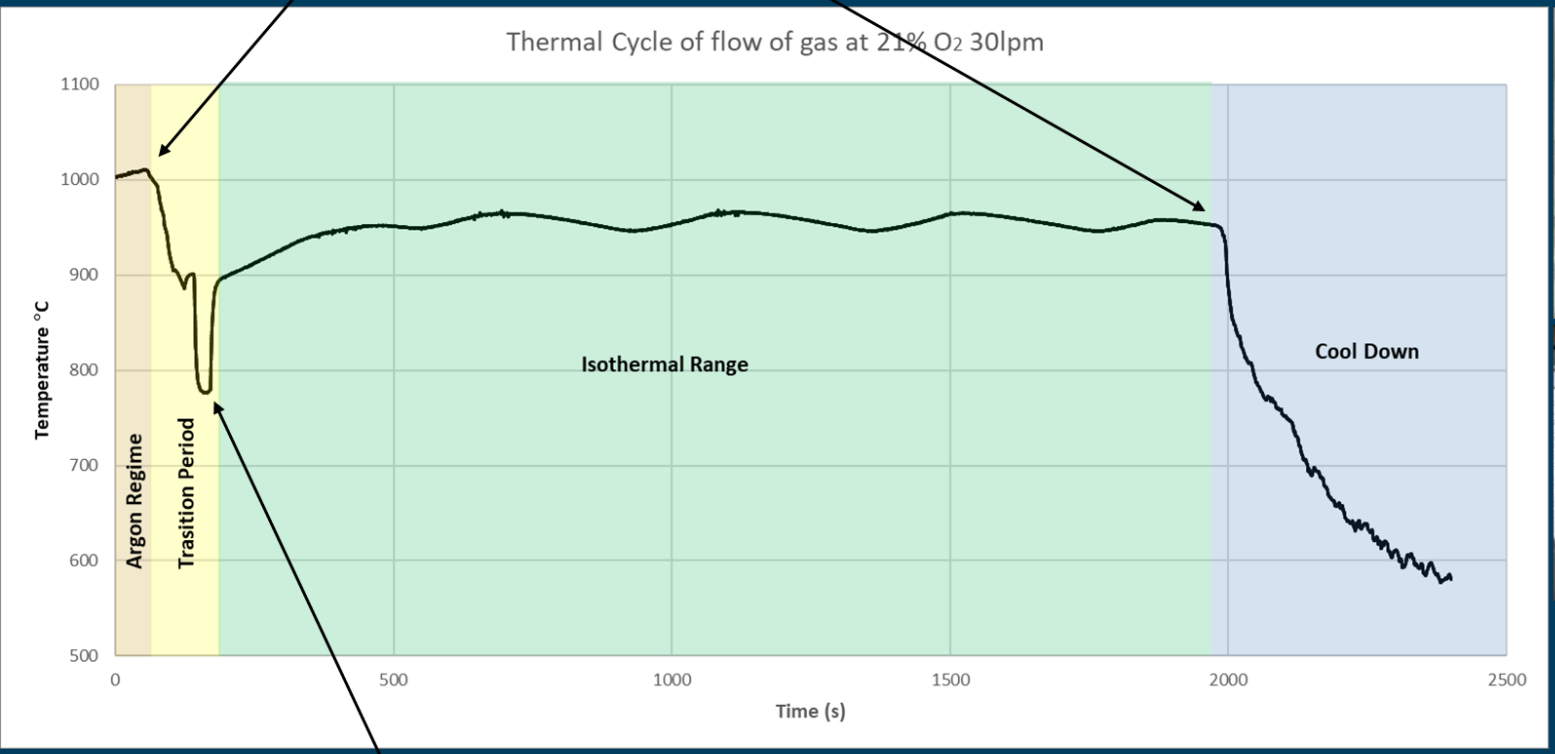
RP2 SR2

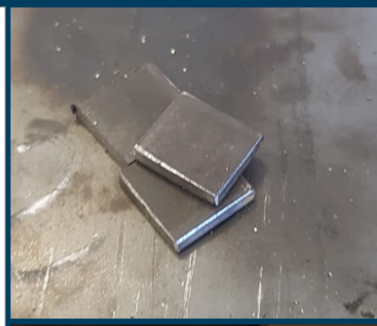
Gas	Carbon Dioxide	Oxygen	Flow Rate L/min	T Stack °C	NO ppm
Weight %	7.5	8	0.98	690	125

Benchmark Thermal Cycle



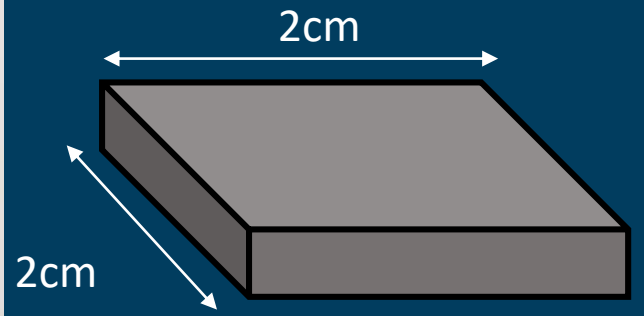
Furnace Opened

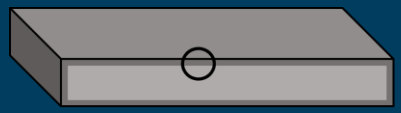


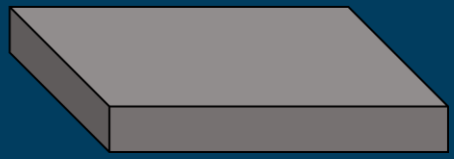


Furnace Closed

Sample A	
Grade	E1
Max Temp	951°C
O_2 %	21%
Flow	30 L/min







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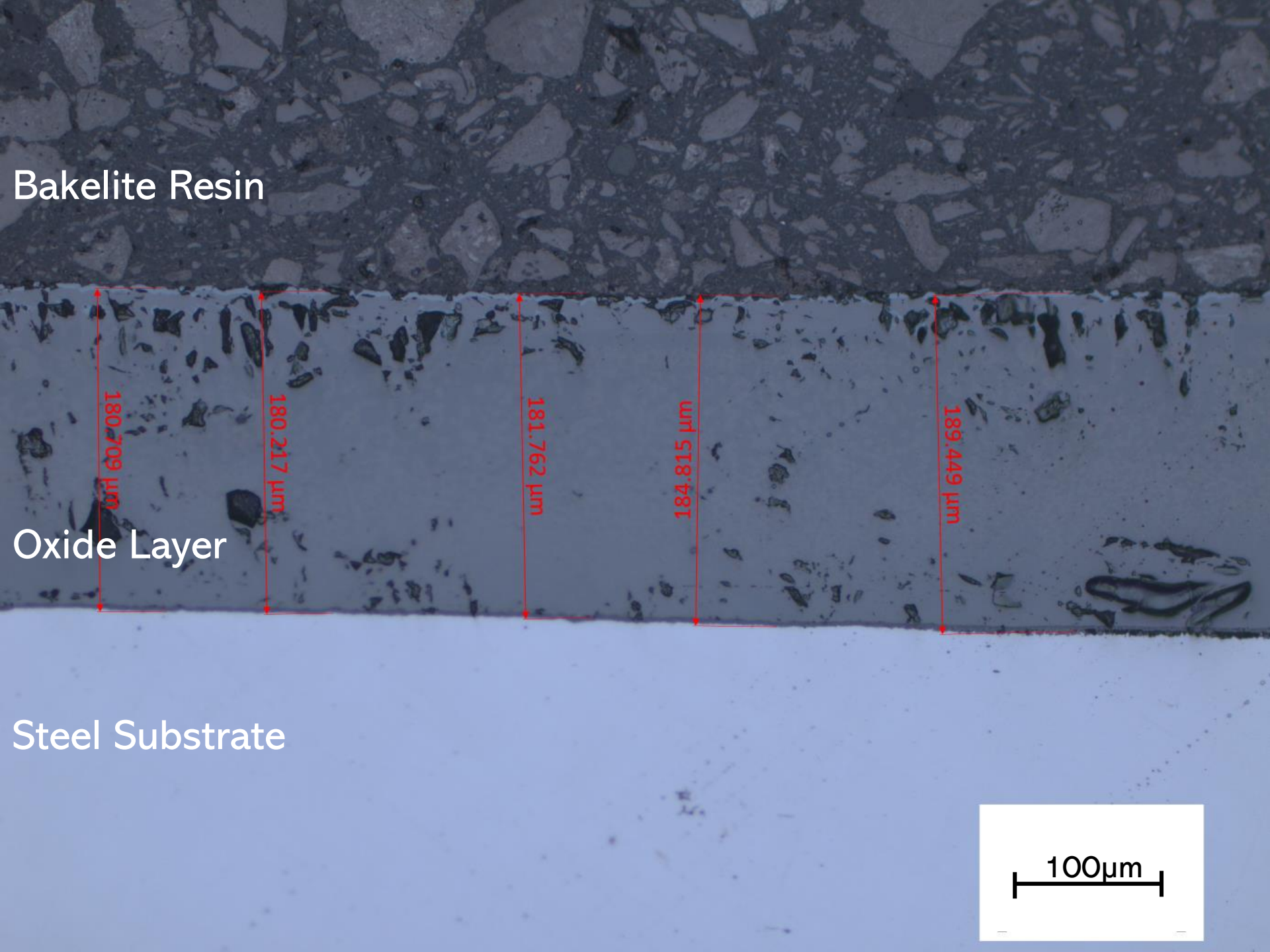
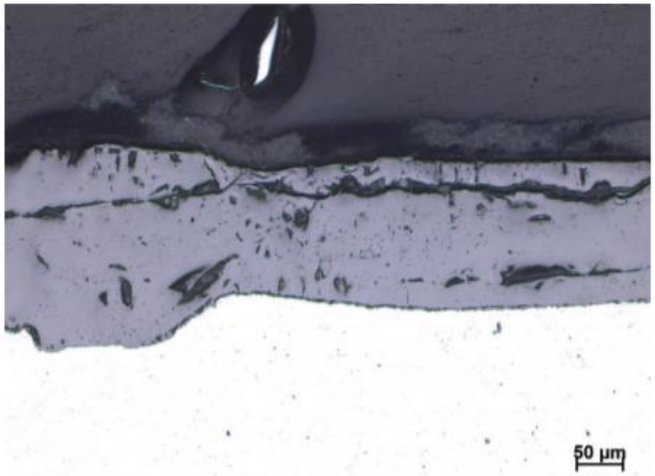
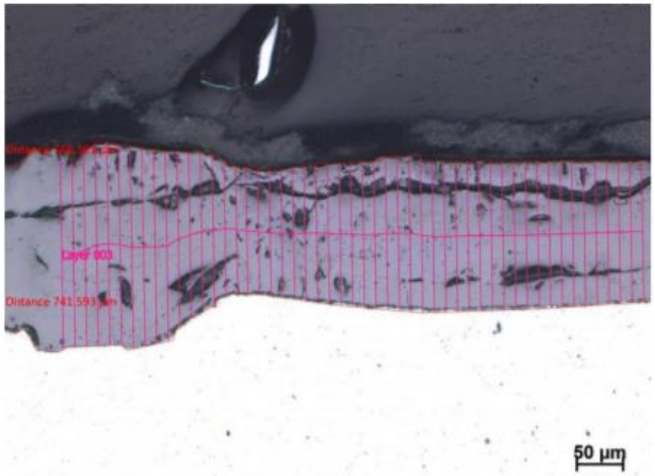

Bakelite Resin
Oxide Layer
Steel Substrate
180.709 µm
180.217 µm
181.762 µm
184.815 µm
189.449 µm
100µm

Automatic Layer Detection Software





Sample A	
Grade	E1
Max Temp	951°C
O_2 %	21%
Flow	30 L/min

Oxide Layer Thickness	
Min Layer Thickness	201.7µm
Max Layer Thickness	221.7µm
Average Oxide Thickness	**214.5 µm ± 6.1µm**

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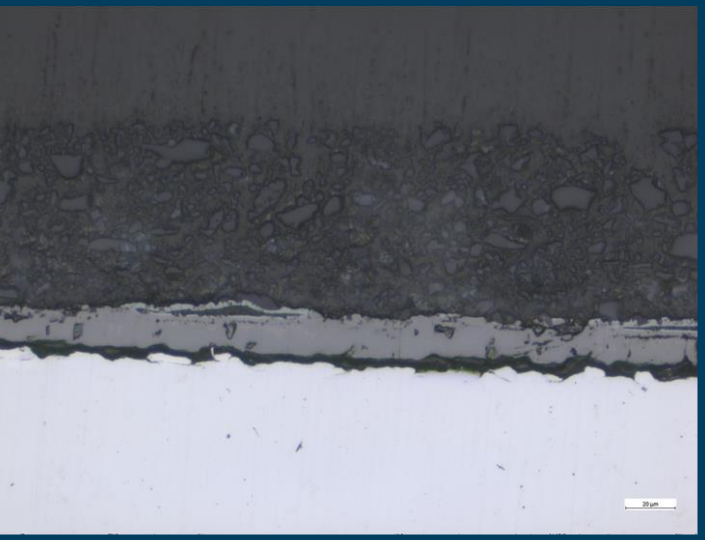
Why is this measurement so useful?

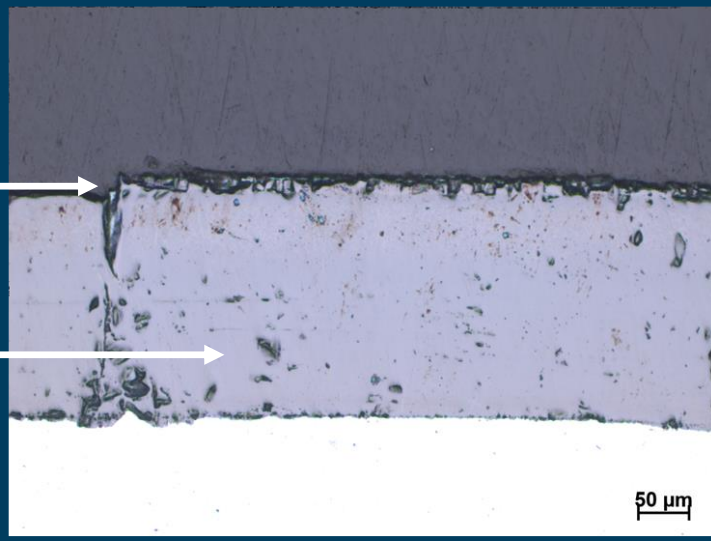
Observe the effect on oxide growth due to:



Coatings

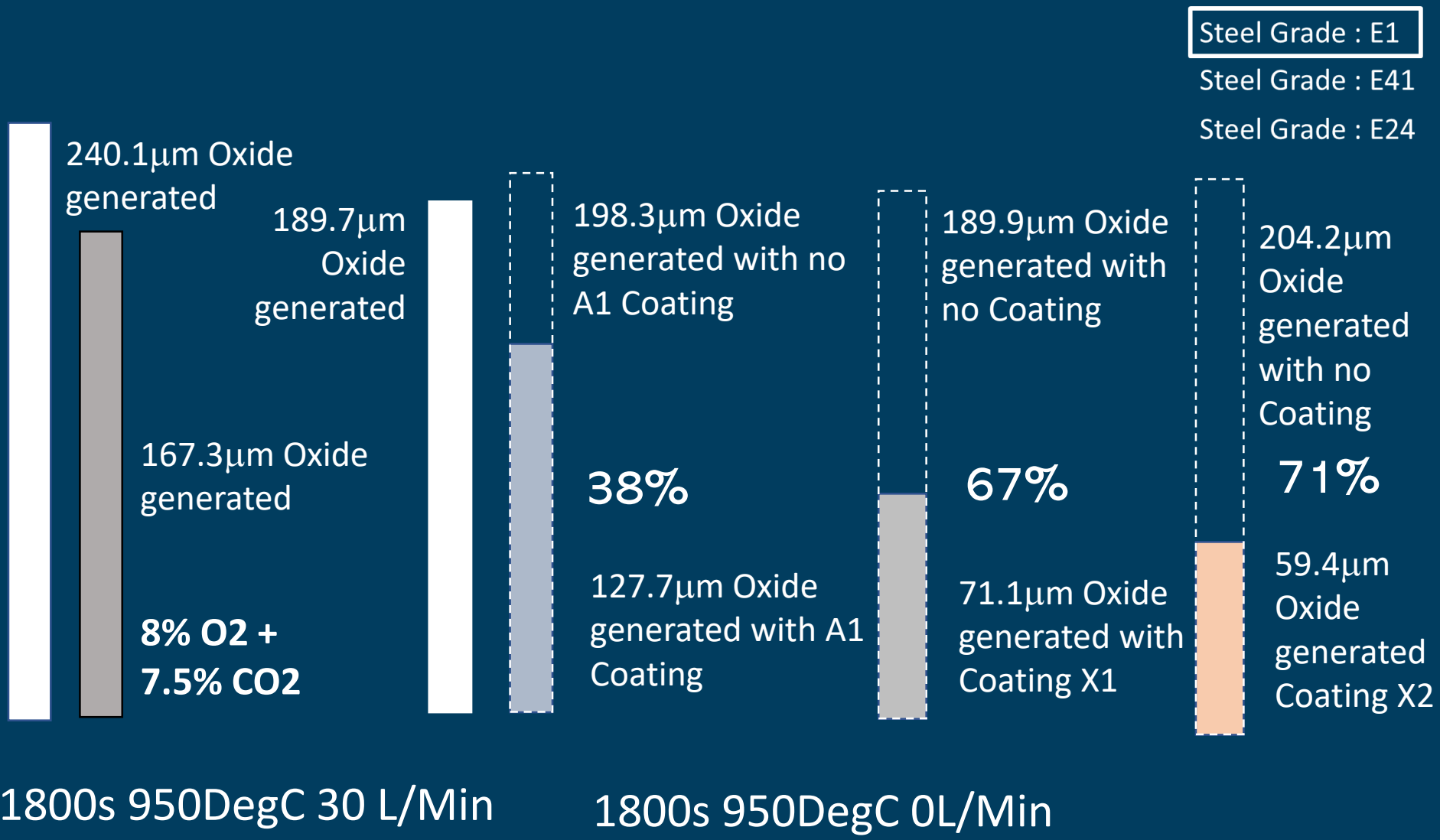
Reduction in Magnetite Layer

Variation in Oxide Porosity



Atmospheric Conditions

Reduction in oxide thickness due to conditional changes during reheating



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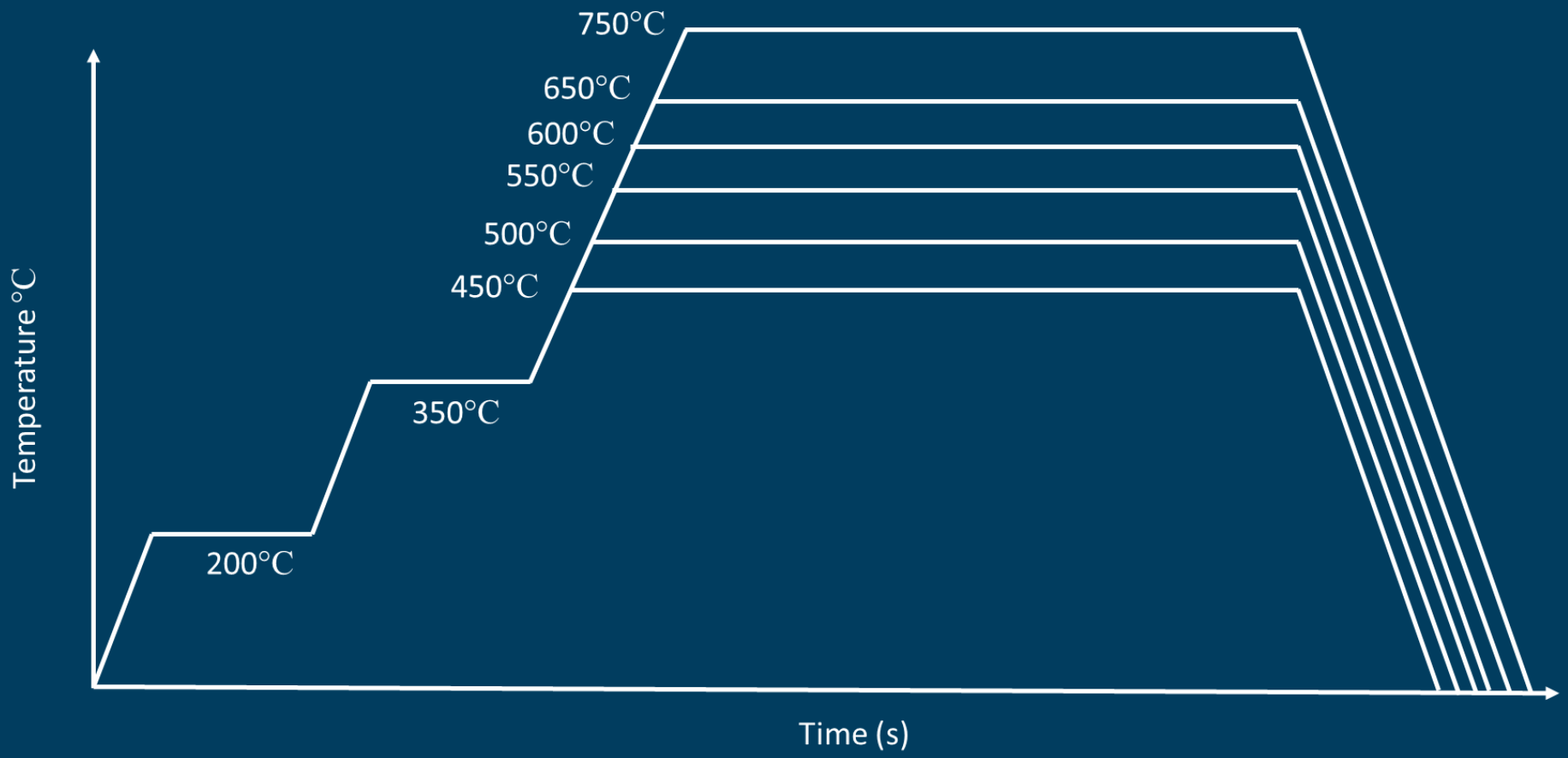
Programmed Thermal Cycle for Oxidation Kinetic Investigation



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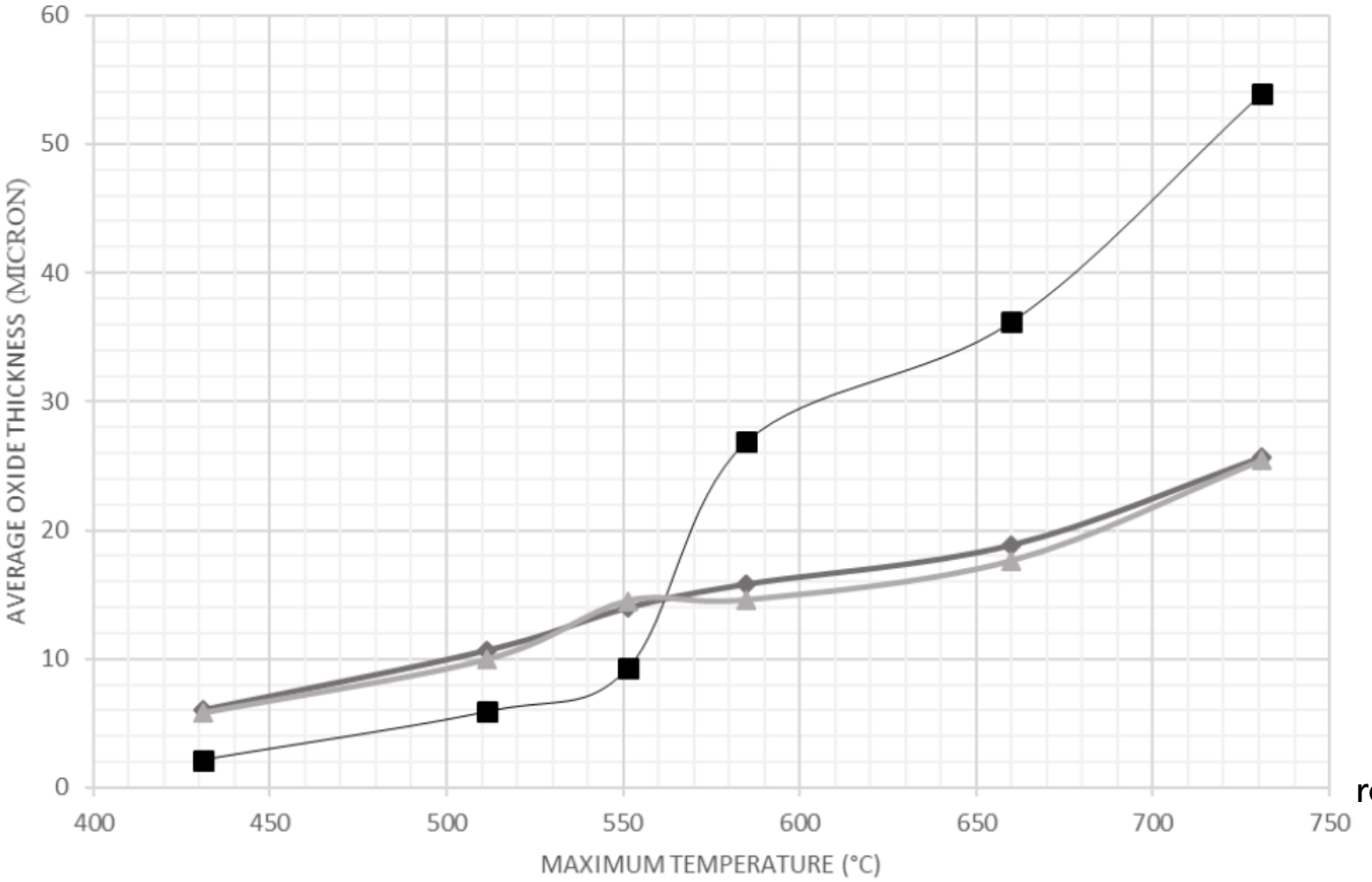
VARIATION IN OXIDE THICKNESS DUE TO MAXIMUM SOAK TEMPERATURE



1. Accelerated E24 oxidation past 550°C in line with STA Observation

2. Coating properties identical for E1,E41 – adjustment required for E24

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1. What oxide are we dealing with?
2. How exactly does it grow?
3. Can you prevent it's formation?

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Protective Coatings

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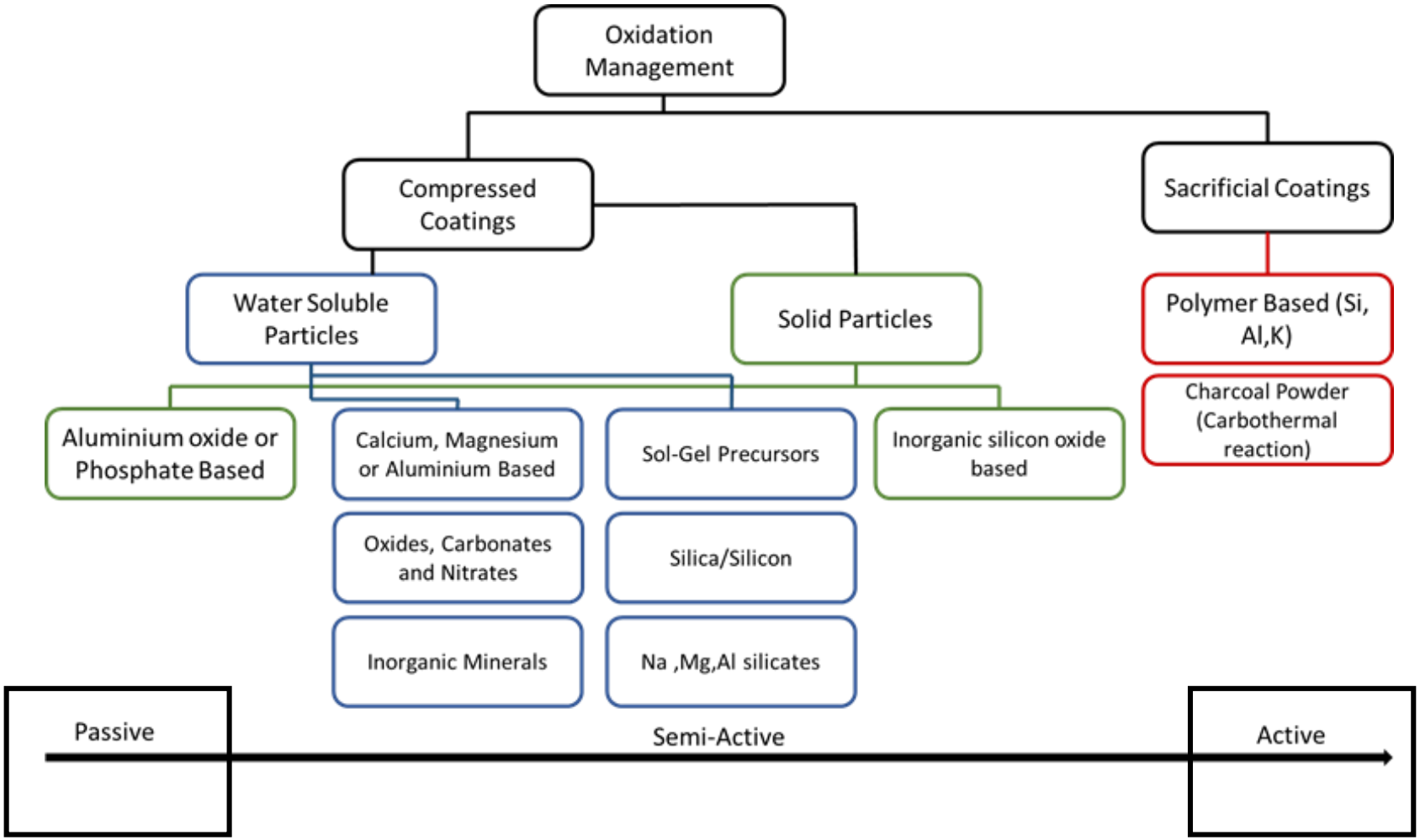
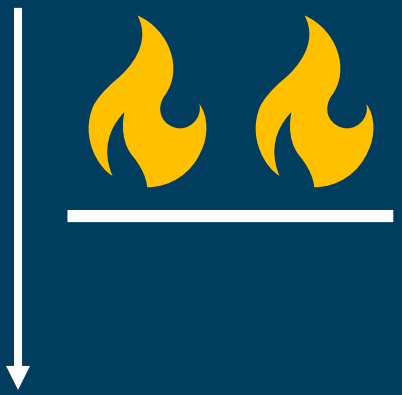

Oxidation Management
Compressed Coatings
Sacrificial Coatings
Water Soluble Particles
Solid Particles
Polymer Based (Si, Al,K)
Charcoal Powder (Carbothermal reaction)
Aluminium oxide or Phosphate Based
Calcium, Magnesium or Aluminium Based
Sol-Gel Precursors
Inorganic silicon oxide based
Oxides, Carbonates and Nitrates
Silica/Silicon
Inorganic Minerals
Na ,Mg,Al silicates
Passive
Semi-Active
Active

Active Coating



Oxygen scavenging reaction
Under high temperatures

Passive Coating

Blocks the O_2 diffusing to the steel surface by forming an impermeable barrier

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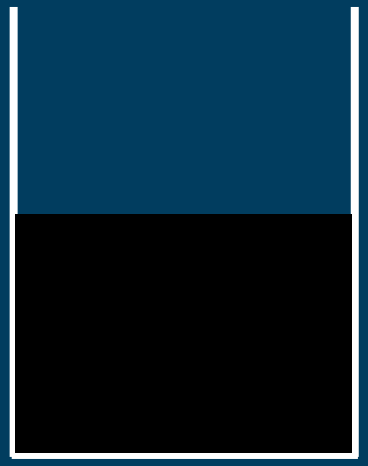
Charcoal Solution



Coal Powder + Polyethylene Glycol

Using up Oxygen → Which would have been used to form scale

Artificially creating a reducing atmosphere

$$C + O_2 = CO_2$$

However...

$$2C + O_2 = 2CO$$ Insufficient O2 = Carbon Monoxide

$$S + O_2 = SO_2$$ Sulphur Present = Sulphur Dioxide

Passive Coating

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Silicates

The Mullite Solution

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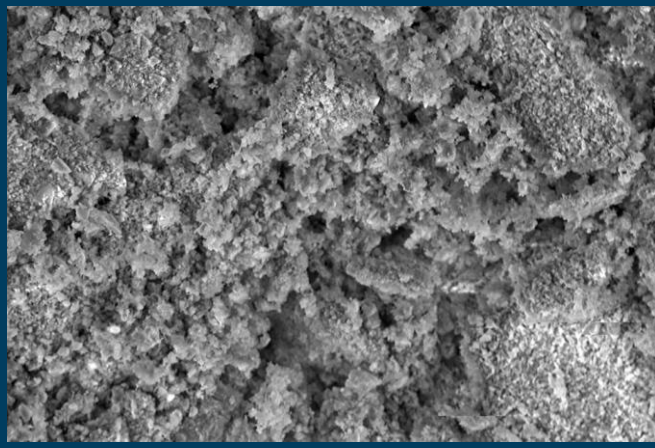
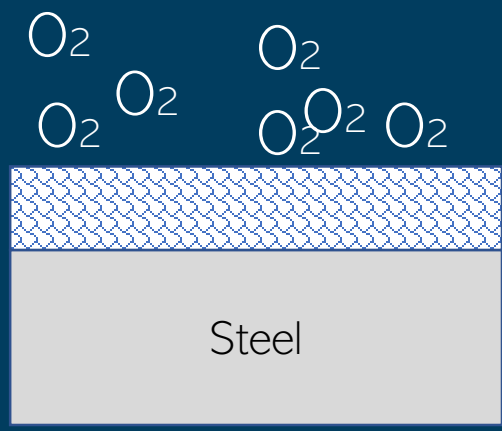
Aluminium Silicate

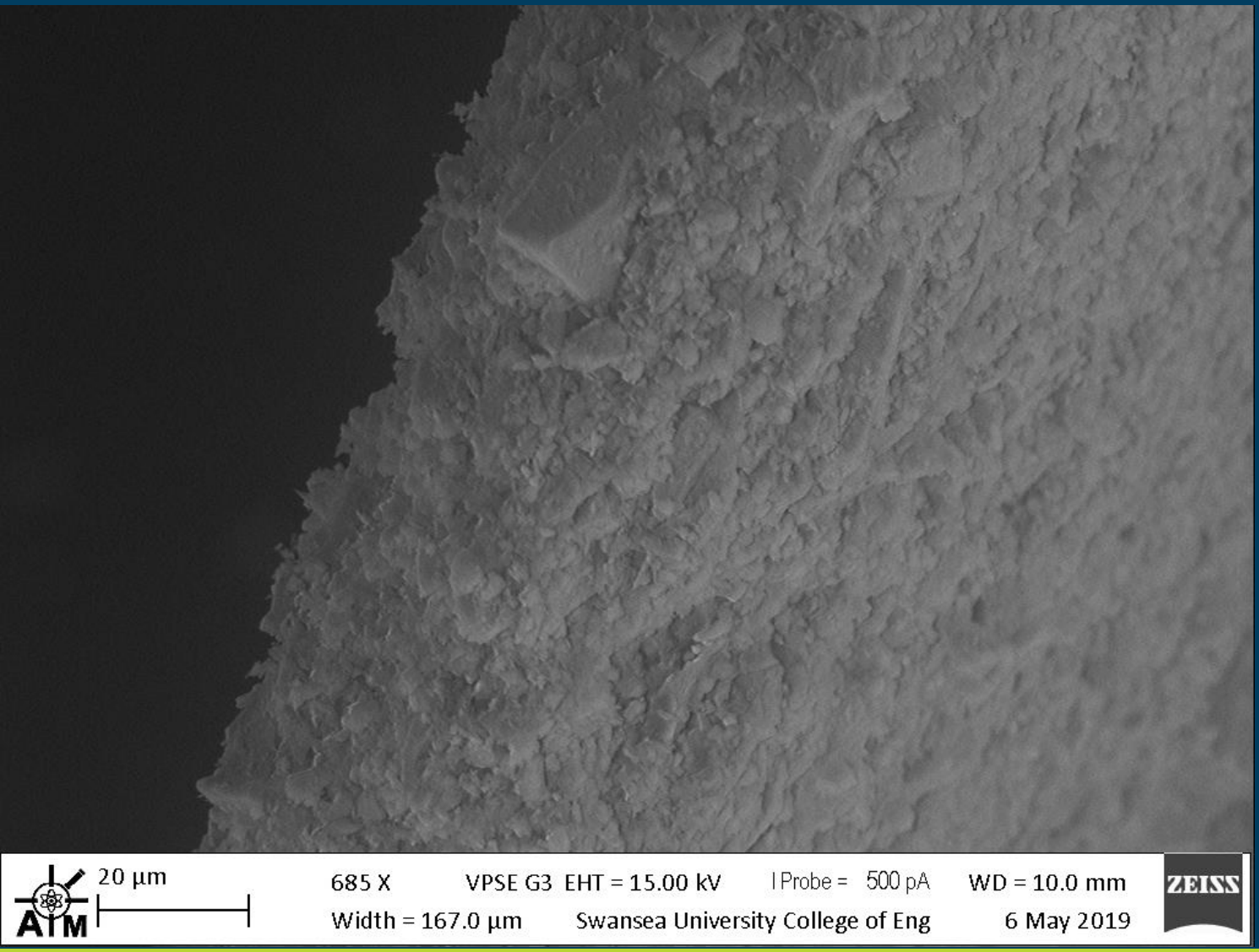
Al Powder
Silica
Frit
Solvents
Binding Agents



Al powder → Al transforms to Liquid Al at 700°C → Al liquid begins to oxidise forming Al_2O_3

Combines with silica to form **Mullite (barrier)** → $2SiO_2 \bullet 3Al_2O_3$ →





Mullite

Orthorhombic Crystal Structure

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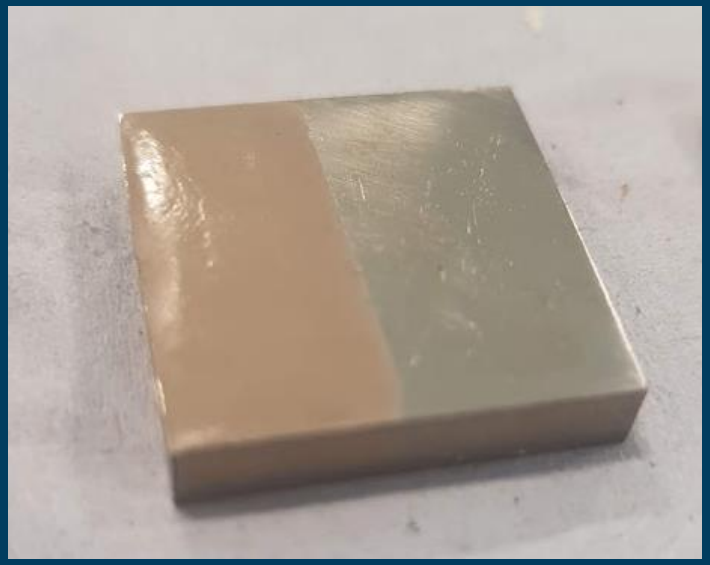
Initial mullite (25μm) Trail against oxide kinetics

Coated/Uncoated E1



Heat Treated Coated/Uncoated

Mullite

What's the problem?

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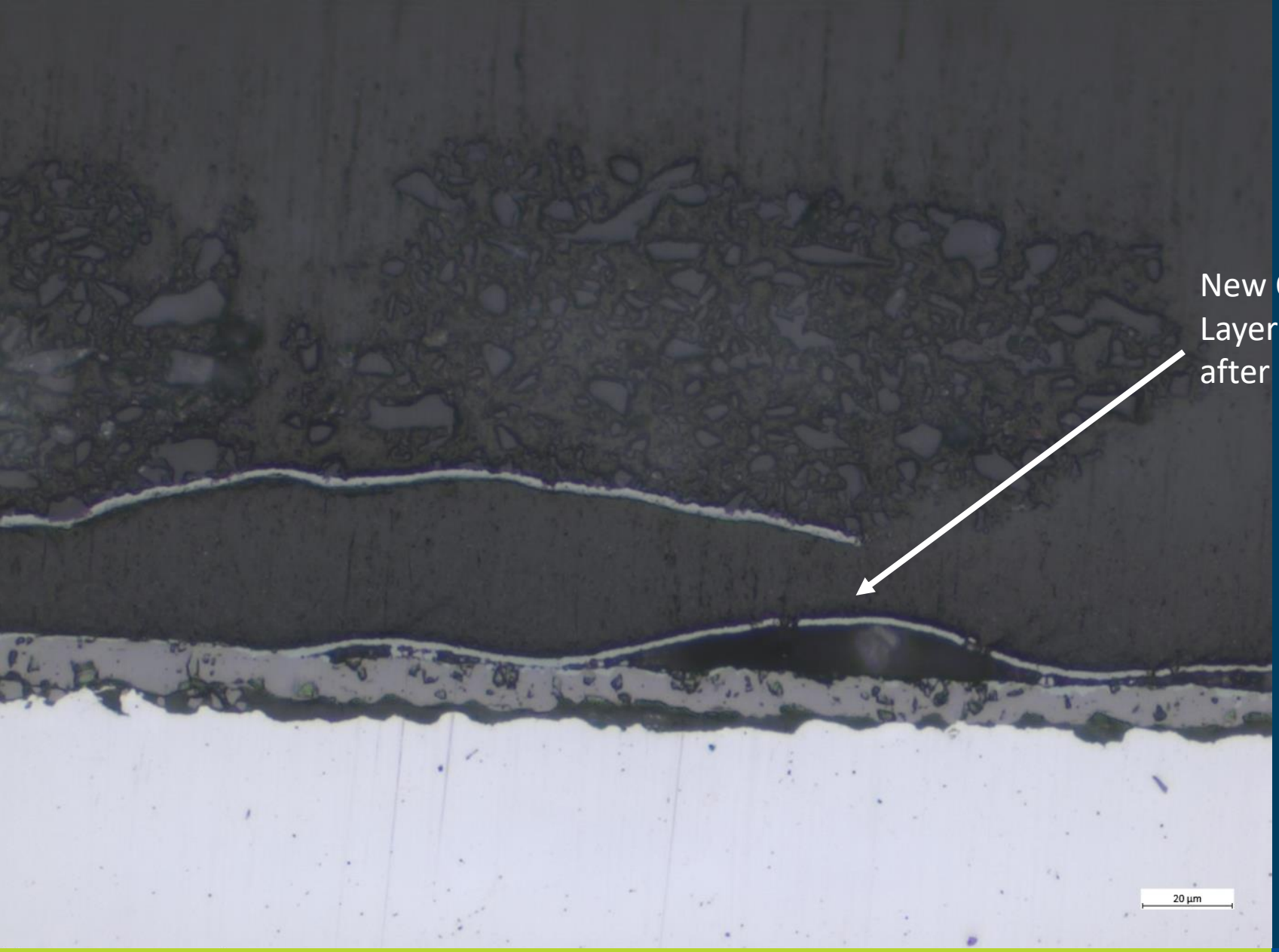

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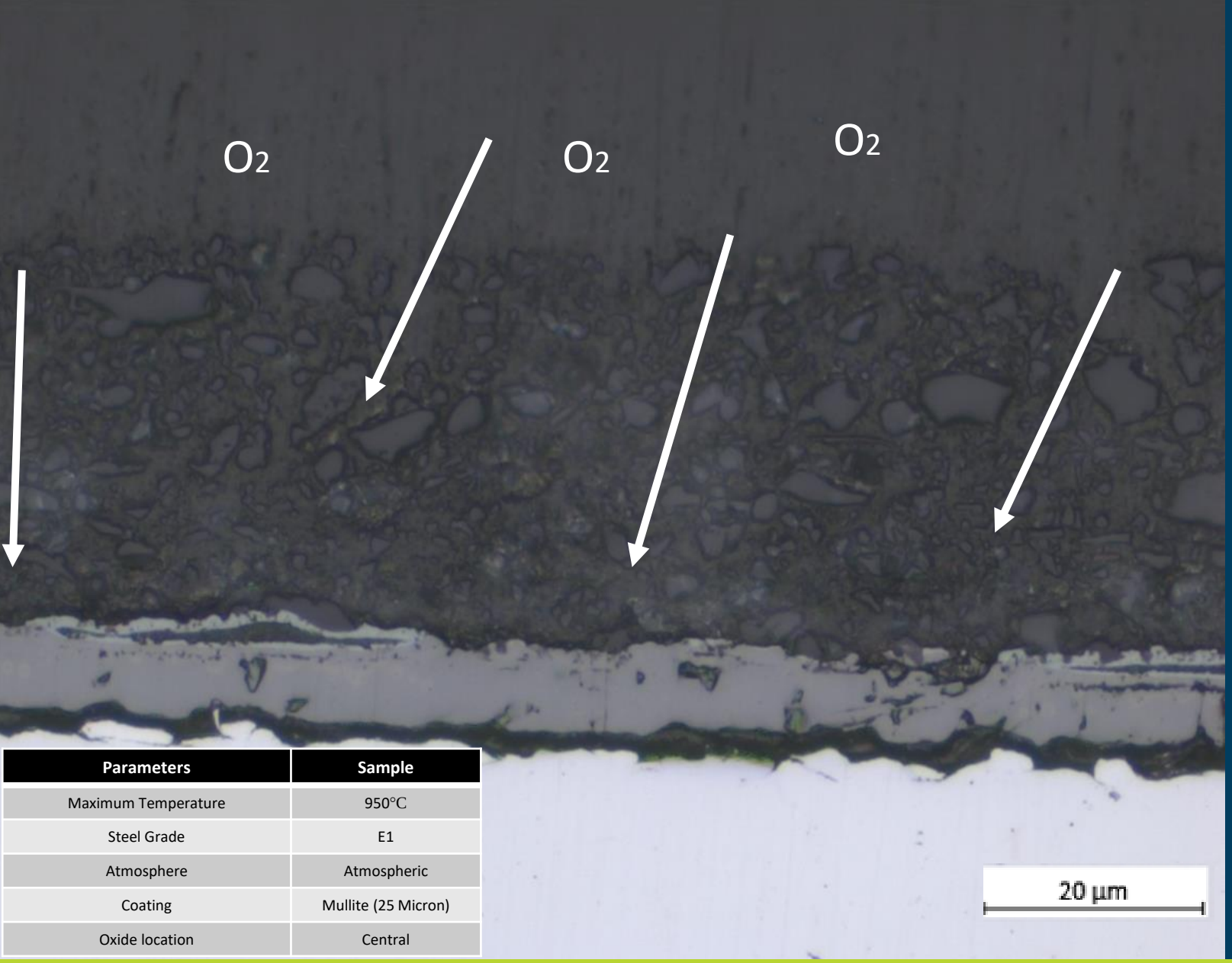

Parameters	Sample
Maximum Temperature	950°C
Steel Grade	E1
Atmosphere	Atmospheric
Coating	Mullite (25 Micron)
Oxide location	Central

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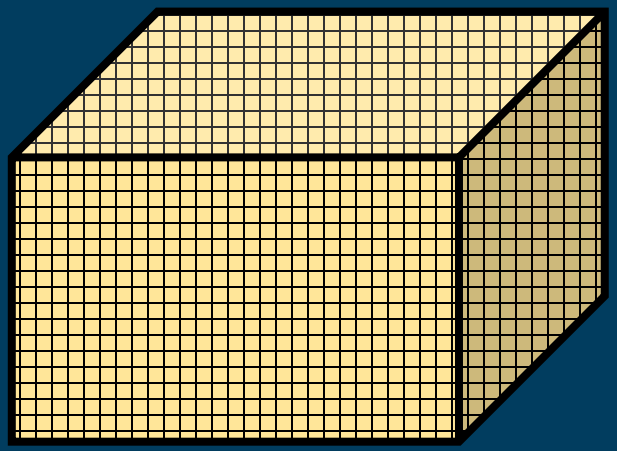
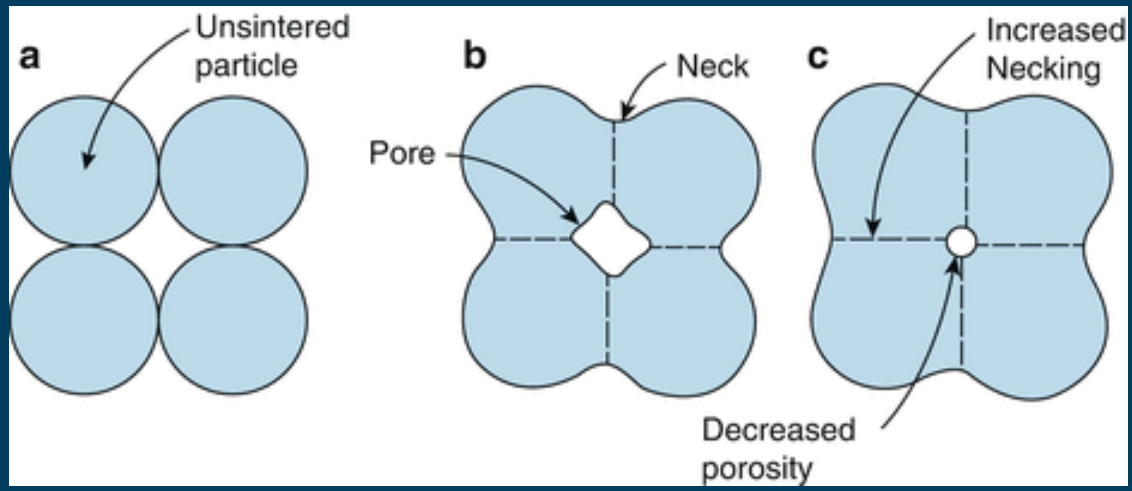
Porosity Issues in pure mullite solutions

Large areas of continuous non – uniform unsintered mullite particles.



Sintered $2SiO_2 \bullet 3Al_2O_3$ is causing an unwanted 'necking' effect



Reduced necking allows a highly porous structure from which oxygen can permeate to the steel surface

Solution : Form a composite with another (high temperature stable) material to fill/trap the pores

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Zirconia Oxide

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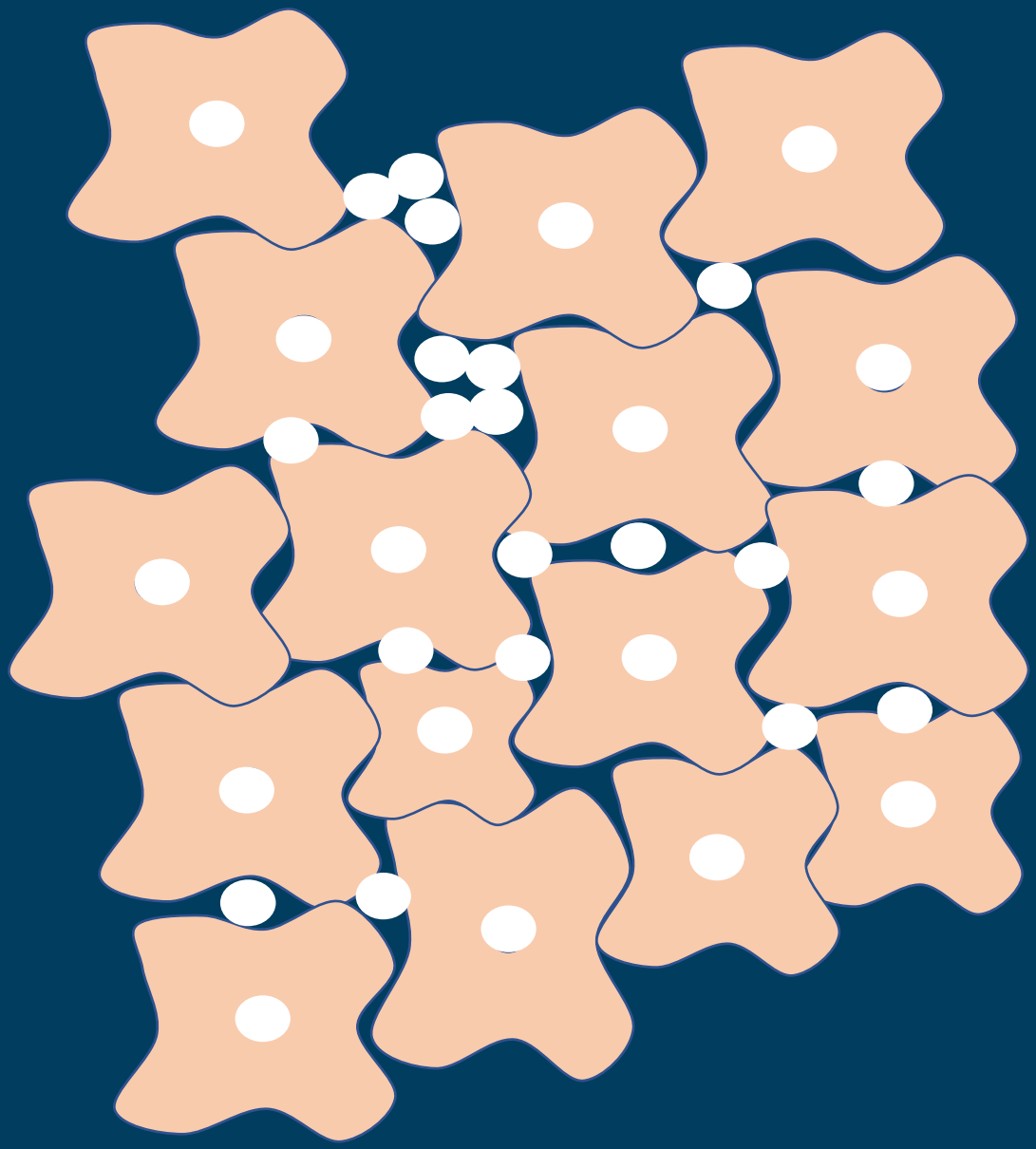
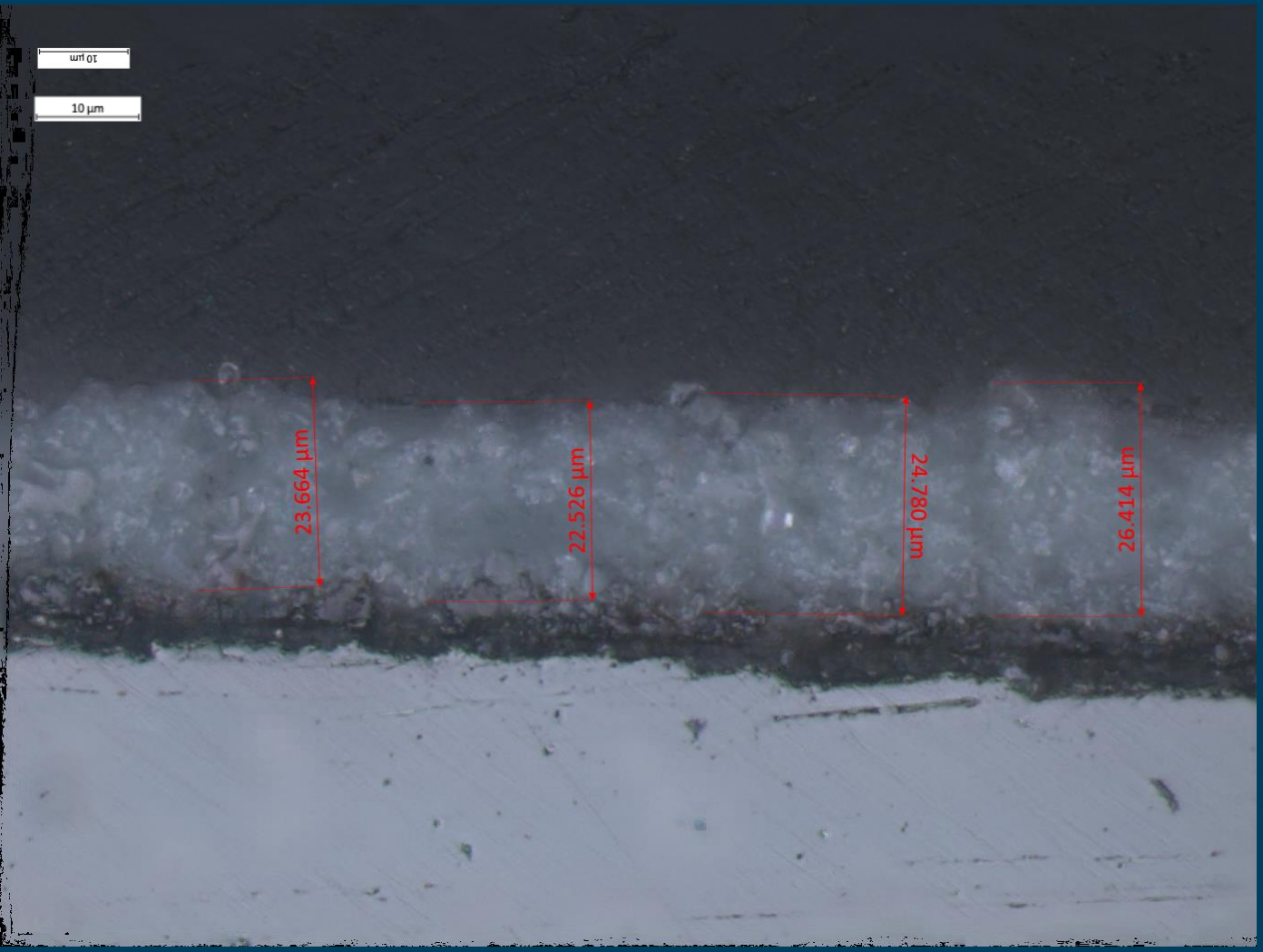
Mullite Layer



- Zirconia particles are trapped by the porous regions of the mullite during sintering
- Forming a more compact and impermeable layer for O_2 prevention

ZrO_2



- ZrO_2 won't sinter until over 1400°C
- Stable and unreactive
- Not as brittle as Al particle and can be made to a smaller particle size to fill the mullite pores

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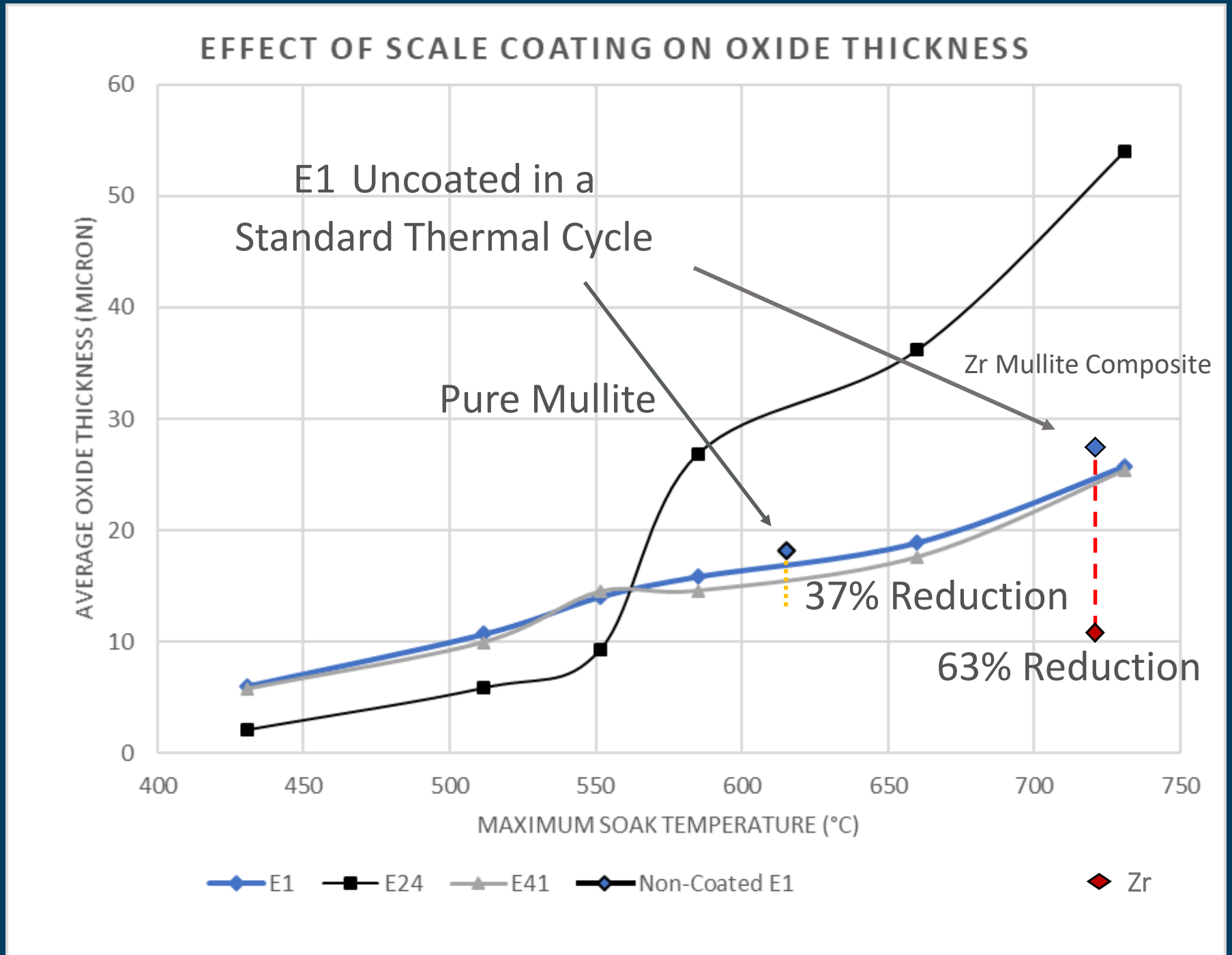
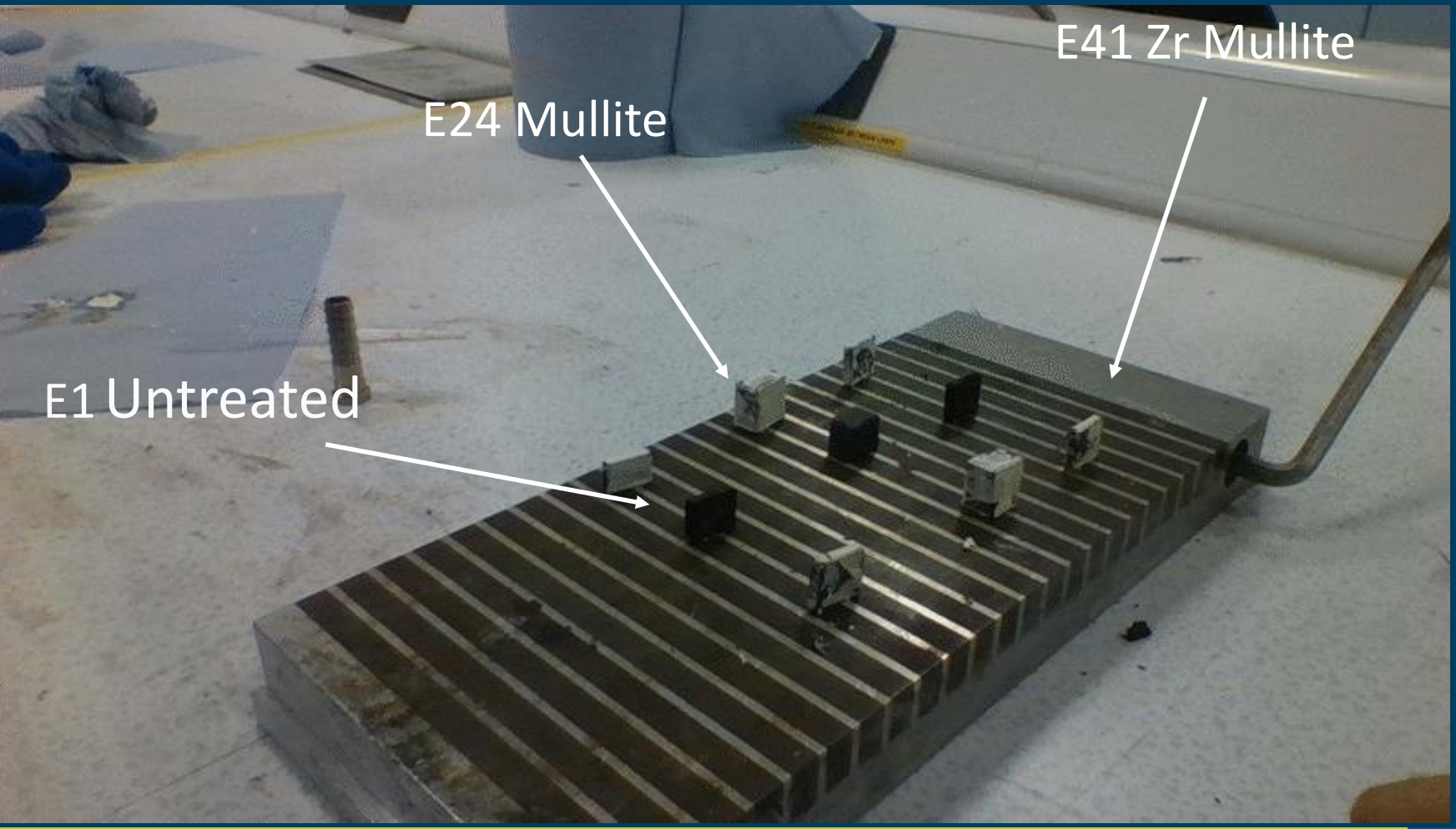

EFFECT OF SCALE COATING ON OXIDE THICKNESS
AVERAGE OXIDE THICKNESS (MICRON)
60
50
40
30
20
10
0
400
450
500
550
600
650
700
750
MAXIMUM SOAK TEMPERATURE (°C)
E1 Uncoated in a Standard Thermal Cycle
Pure Mullite
Zr Mullite Composite
37% Reduction
63% Reduction
E1
E24
E41
Non-Coated E1
Zr

Future Work – Descalability of the coatings and their adhesion to scale traits



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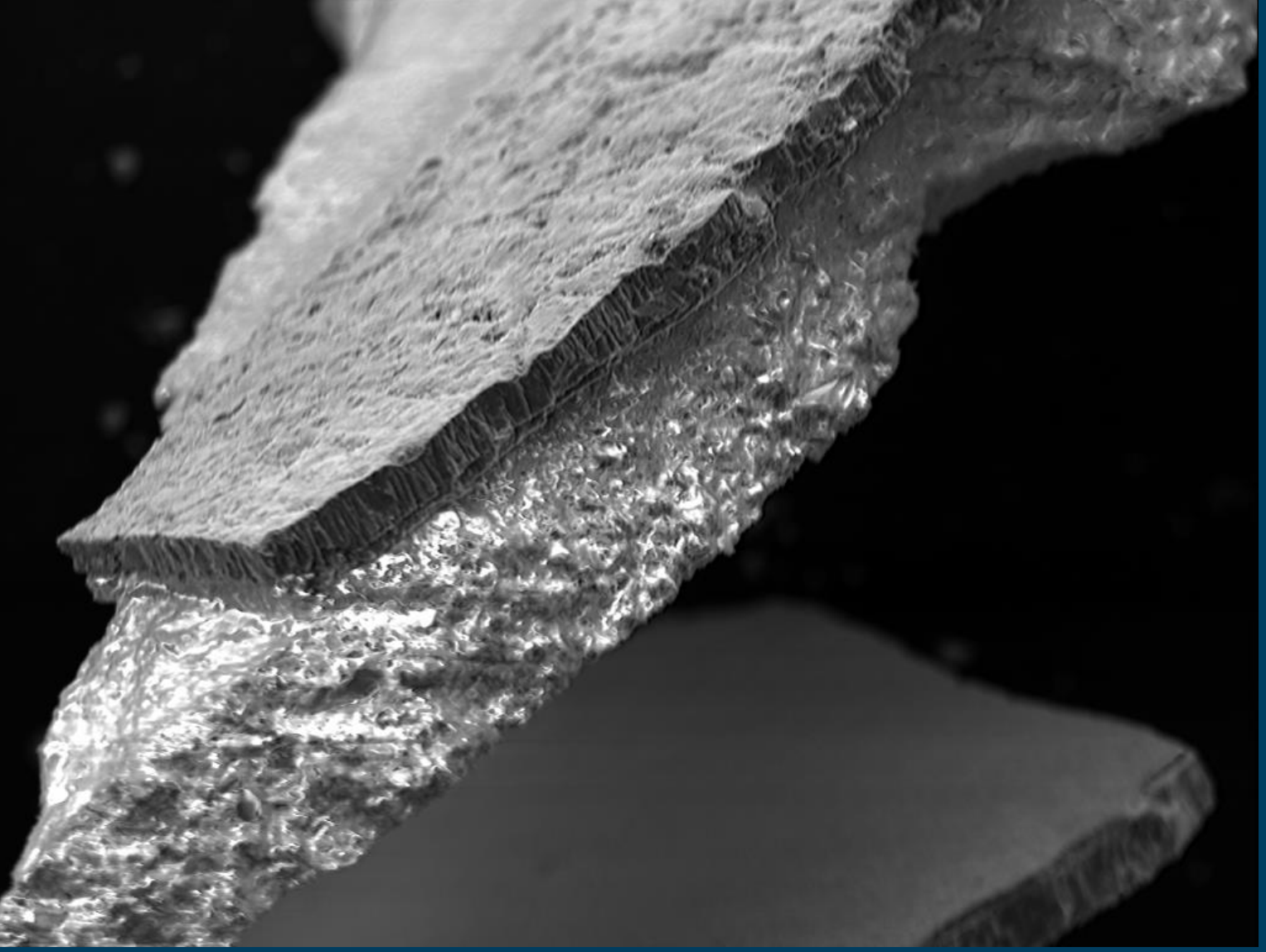

Reducing the oxide and ripping it away from the steel surface

Improving Surface quality by coating spallation

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Conclusions

- Characterisation of the scale generated by the mill using SEM, EDS and Raman Spectroscopy
- Kinetics of the steel grade oxidation performed using STA and further experimentation developed to quantify the reduction in oxide growth
- Initial trialling of mullite solutions aiming to produce a passive barrier layer and reductions of up to 63% observed.

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James Grant
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