# Multilayer Coatings & Compsite Coatings

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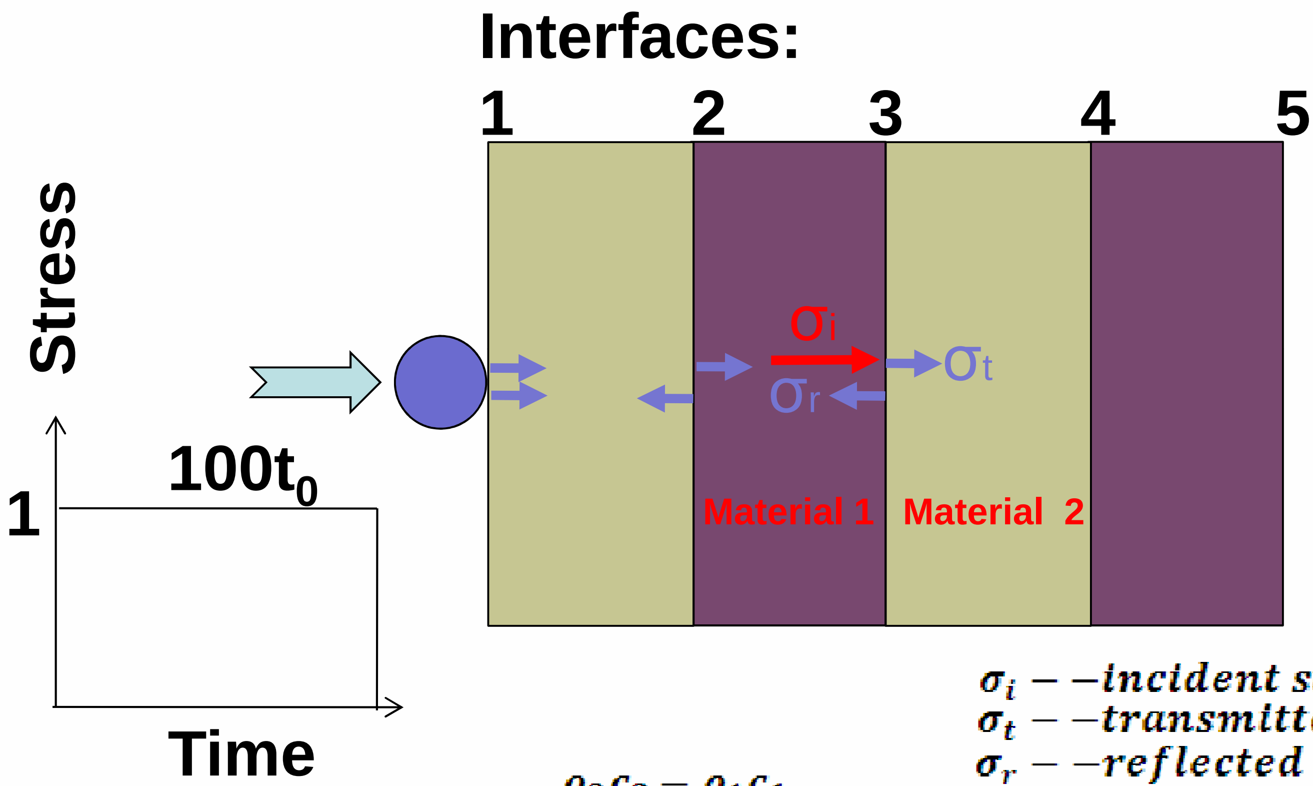
# OUTLINES

- Part I - Stress wave analysis & multilayer coatings
- Part II - Boronizing coatings
- Part III - $TiC$-$TiB_2$-$TiAl_3$ coatings
- Part IV - Laser cladding coatings

# Part I

# Stress Wave Analysis & Multilayer Coatings

# Background: Simplifications




$$\sigma_r = \frac{\rho_2 c_2 - \rho_1 c_1}{\rho_2 c_2 + \rho_1 c_1} \cdot \sigma_i$$

$$\sigma_t = \frac{2\rho_2 c_2}{\rho_2 c_2 + \rho_1 c_1} \cdot \sigma_i$$

$\sigma_i$ – –*incident stress*
$\sigma_t$ – –*transmitted stress*
$\sigma_r$ – –*reflected stress*
$\rho$ – –*medium density*
$c$ – –*wave speed in medium*

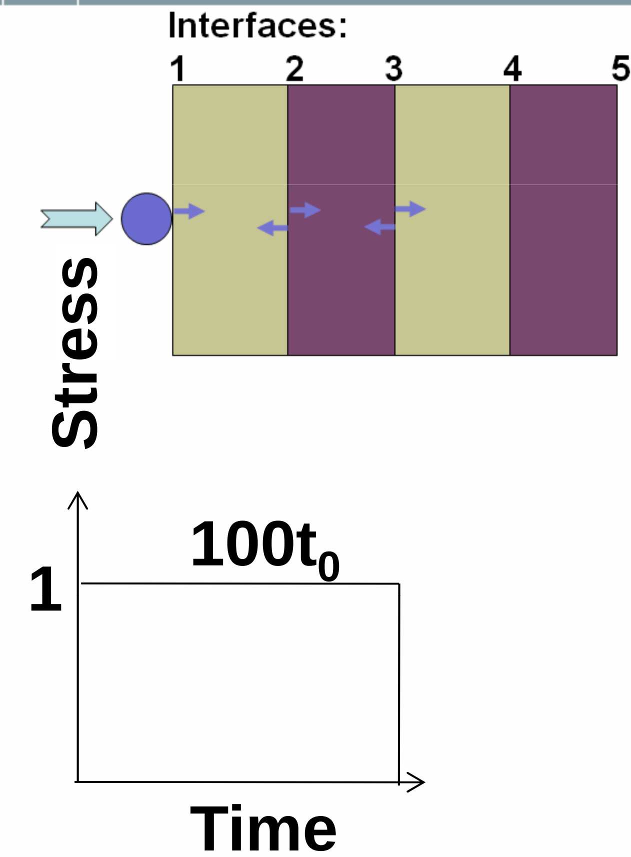
$$c = \sqrt{\frac{E}{\rho}}$$

$E$— *Young's modulus*



*Ref 3: Li, 2003.*

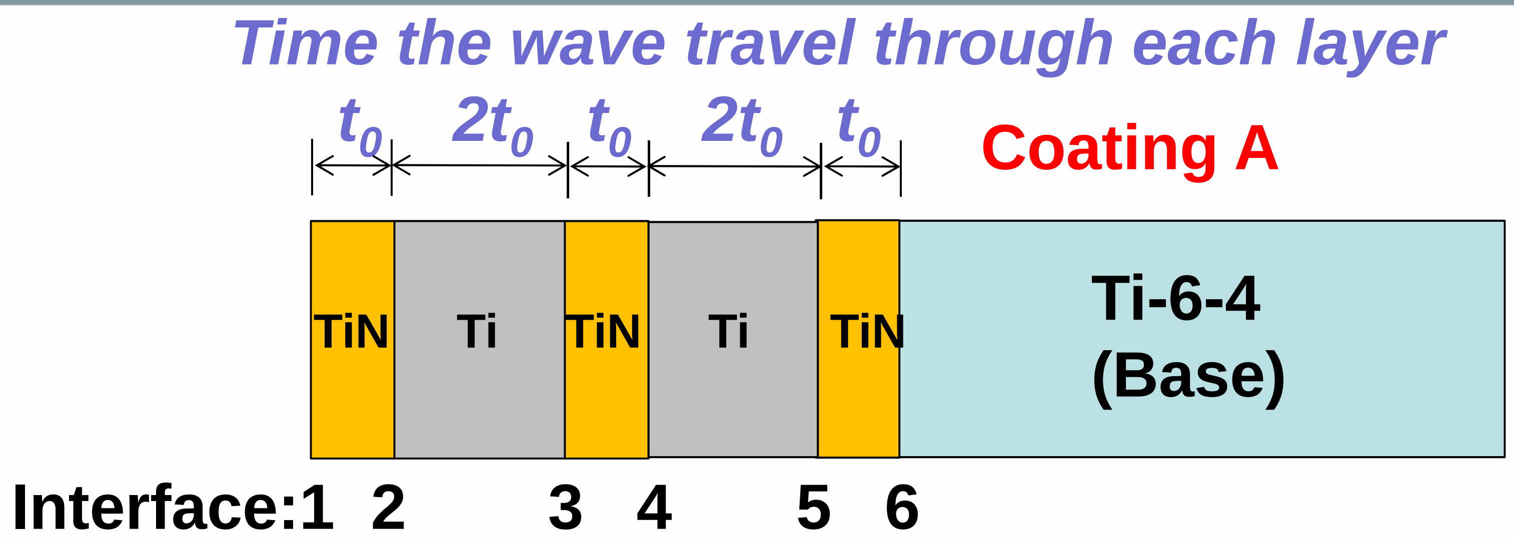
# Background: Simplifications




## Simplifications:

1. **Single droplet**
   (Φ16.65 μm, 500m/s)
2. **Linear and rectangle wave**
3. **Incident stress = 1 unit**
4. **Incident time range = 100$t_0$**
   ($t_0$=3.3E-10 s)
5. **100 pulses**
6. **No energy loss**
7. **Thickness design** ($t_0$ by integer)
   1$t_0$------3.5 μm TiN / 1.6 μm Ti layer

## Coating A



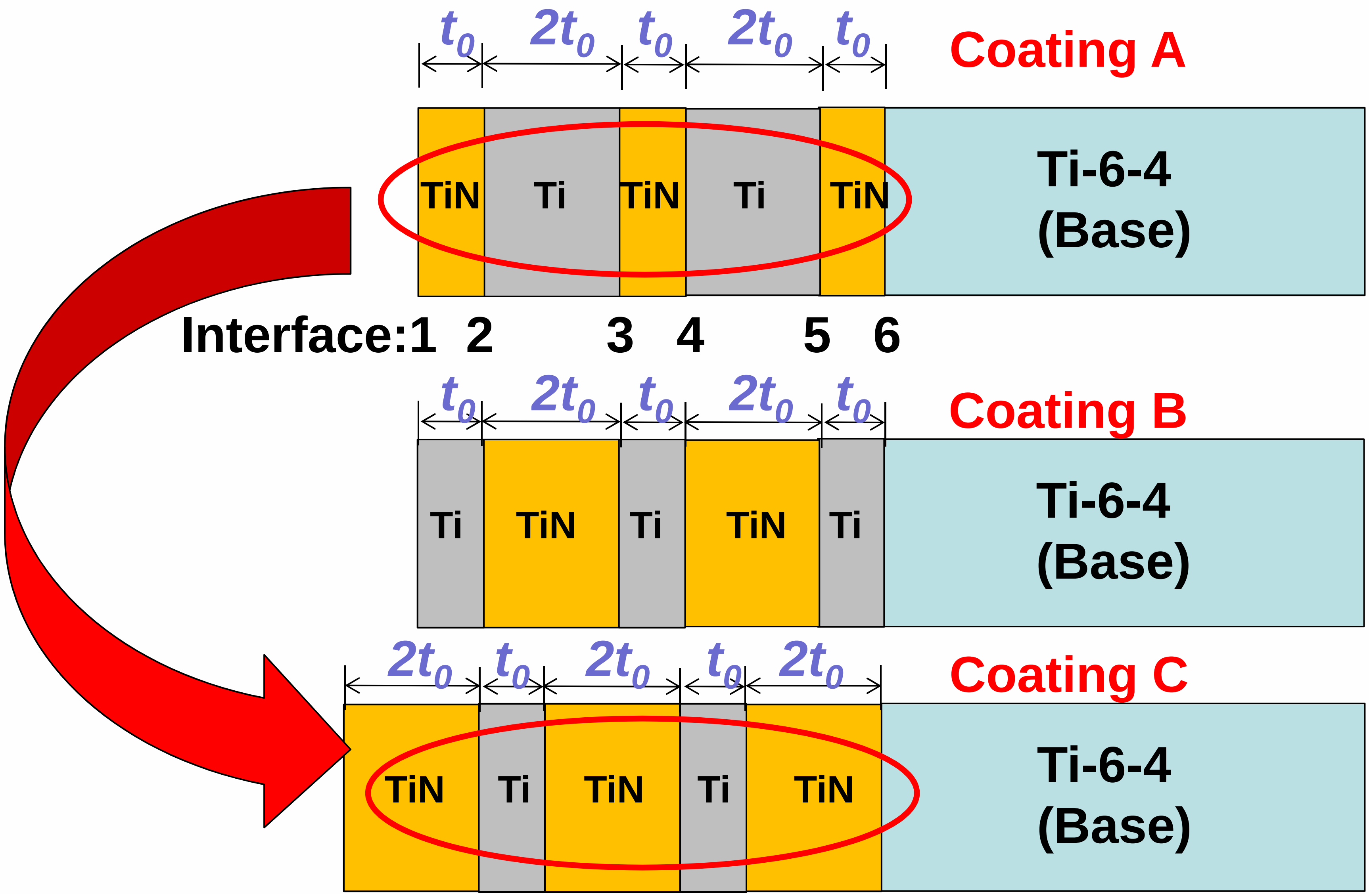

# Simplifications:

8. Base metal thickness: infinite

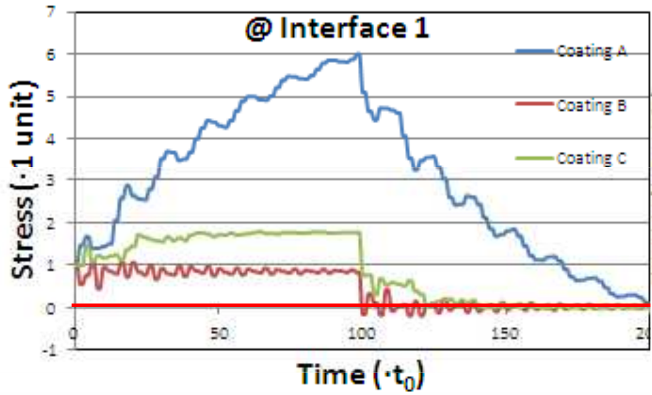
## 2 Calculated Results

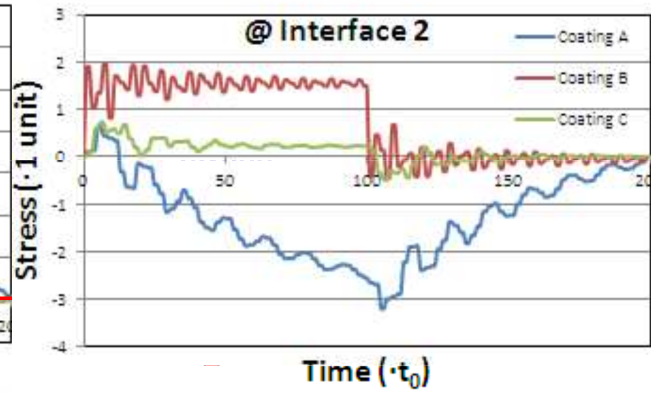
# Coating A Vs. Coating C

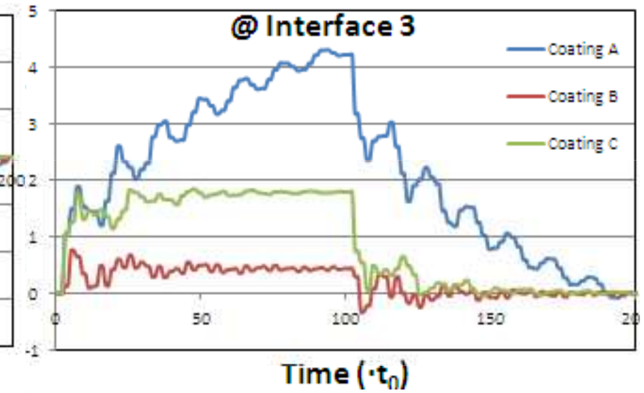
*Time the wave travel through each layer*



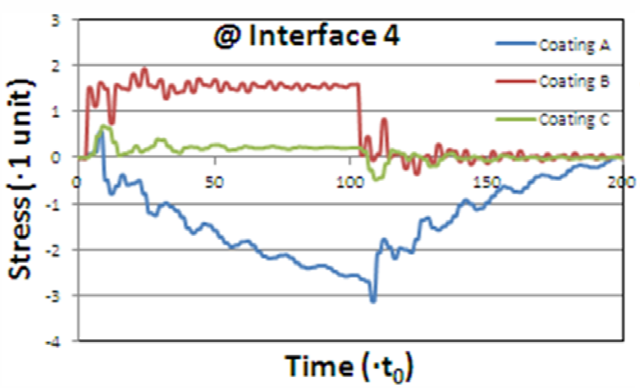
## 2 Calculated Results

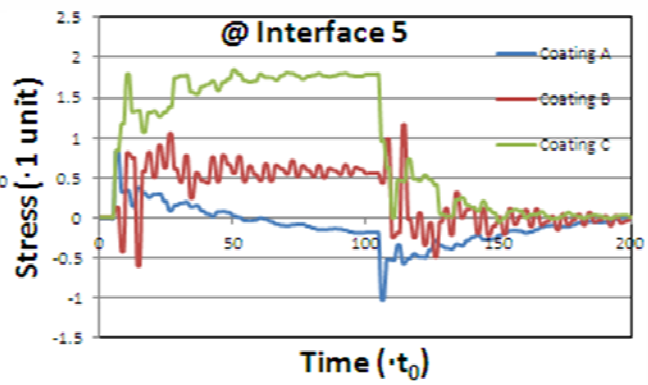


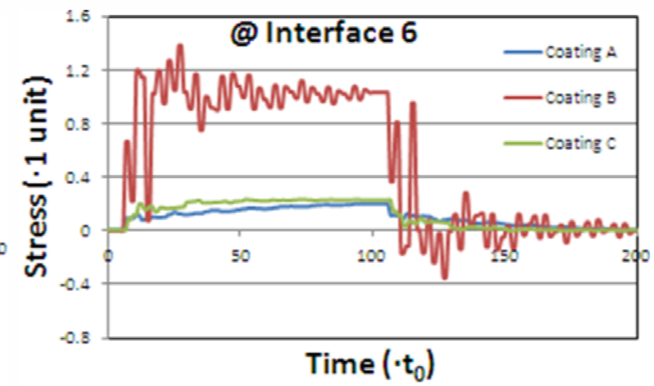




Tensile stress



## 3 Coating Design

## 3 Coating Design

| Coating # | Configuration (From inner to outside) | Layers Amounts | Thickness of Layer 1 (μm) | Thickness of Layer 2 (μm) | Thickness of Layer 3 (μm) | Thickness of Layer 4 (μm) | Thickness of Layer 5 (μm) | Thickness of Layer 6(μm) | Thickness of Layer 7 (μm) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | TiN (1T) | 1 | 1.10 | | | | | | |
| 2 | TiN(5T) | 1 | 5.52 | | | | | | |
| 3 | TiN(9T) | 1 | 9.93 | | | | | | |
| 4 | TiN-Ti-TiN (2T-T-2T) | 3 | 2.21 | 0.50 | 2.21 | | | | |
| 5 | TiN-Ti-TiN-Ti-TiN (2T-T-2T-T-2T) | 5 | 2.21 | 0.50 | 2.21 | 0.50 | 2.21 | | |
| 6 | TiN-Ti-TiN-Ti-TiN (T-T-T-T-T) | 5 | 2.21 | 0.50 | 2.21 | 0.50 | 2.21 | | |
| 7 | TiN-Ti-TiN-Ti-TiN (3T-T-3T-T-3T) | 5 | 3.31 | 0.50 | 3.31 | 0.50 | 3.31 | | |
| 8 | TiN-Ti-TiN-Ti-TiN (T-2T-T-2T-T) | 5 | 1.10 | 0.50 | 1.10 | 0.50 | 1.10 | | |
| 9 | TiN-Ti-TiN-Ti-TiN-Ti-TiN-Ti-TiN (2T-T-2T-T-2T-T-2T-T-2T) | 9 | 2.21 | 0.50 | 2.21 | 0.50 | 2.21 | 0.50 | 2.21 |
| 10 | TiN-TiAlN-TiN-TiAlN-TiN (2T-T-2T-T-2T) | 5 | 2.21 | 1.07 | 2.21 | 1.07 | 2.21 | | |
| 11 | TiN-TiAlN-TiN-TiAlN-TiN-TiAlN (2T-T-2T-T-2T-T) | 6 | 2.21 | 1.07 | 2.21 | 1.07 | 2.21 | 1.07 | |
| 12 | TiN-CrN-TiN-CrN-TiN (2T-T-2T-T-2T) | 5 | 2.21 | 0.78 | 2.21 | 0.78 | 2.21 | | |
| 13 | TiN-CrN-TiN-CrN-TiN-CrN (2T-T-2T-T-2T-T) | 6 | 2.21 | 0.78 | 2.21 | 0.78 | 2.21 | 0.78 | |

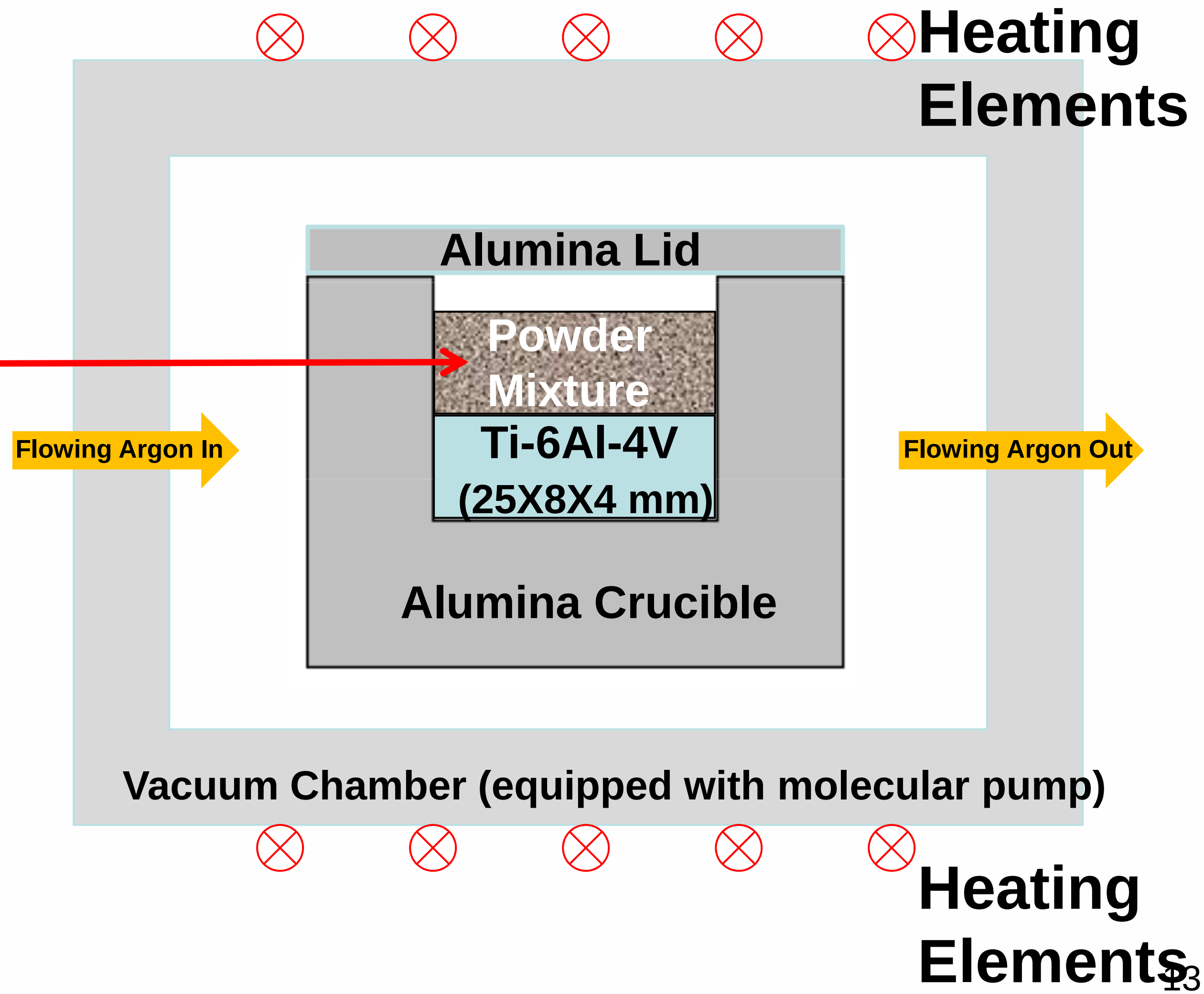
Part II

# Boronizing Coating Results

# Boronizing Diffusion Coatings

**Powder Mixture:**
*[US Patent 7264682, 2007]*
* Boron, amorphous (reactant)
* $Na_2CO_3$ (activator)
* Carbon, activated (filler material)

**Setup Illustration**

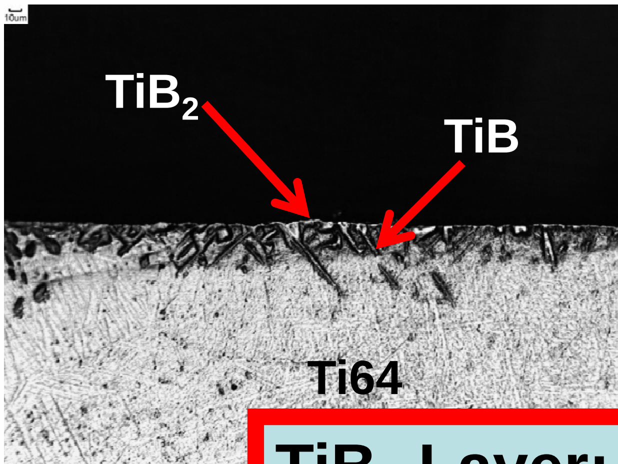


# Experiment Design

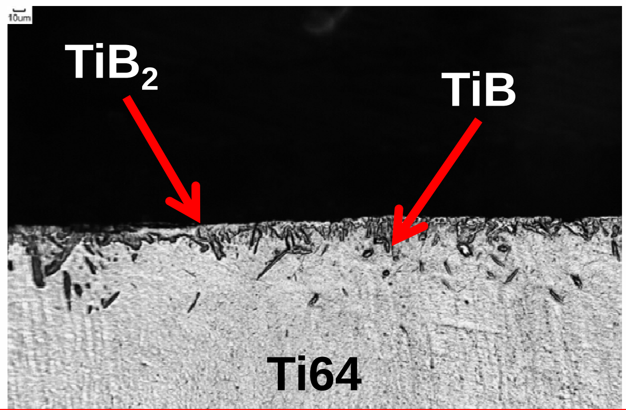
| Composition # | B (wt%) | $Na_2CO_3$(wt%) | C(wt%) | Holding Time@1050C | Holding Time@1200C |
|---|---|---|---|---|---|
| B0 | 100% | - | - | 24hrs | |
| B1 | 50% | 45% | 5% | 24hrs | |
| B2 | 50% | 35% | 15% | 12, 24, 48hrs | 12 hrs |
| B3 | 50% | 25% | 25% | 12, 24, 48hrs | 12 hrs, 72hrs |
| B4 | 50% | 15% | 35% | | 72hrs |
| B5 | 50% | 5% | 45% | 24, 72hrs | 72hrs |

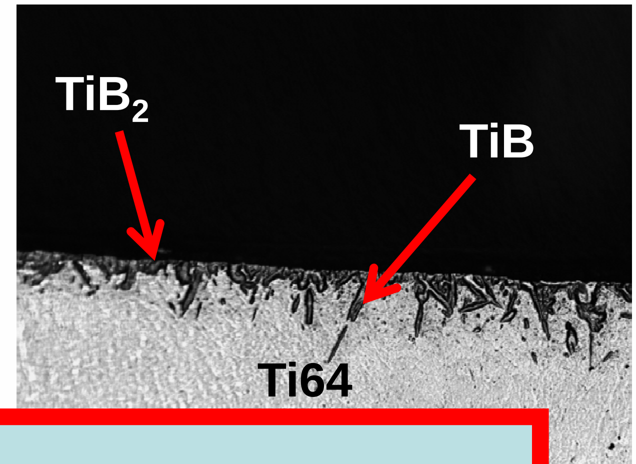
# Effect of Powder Composition I

## Same Heat Treatment: 1050ºC 24 hrs



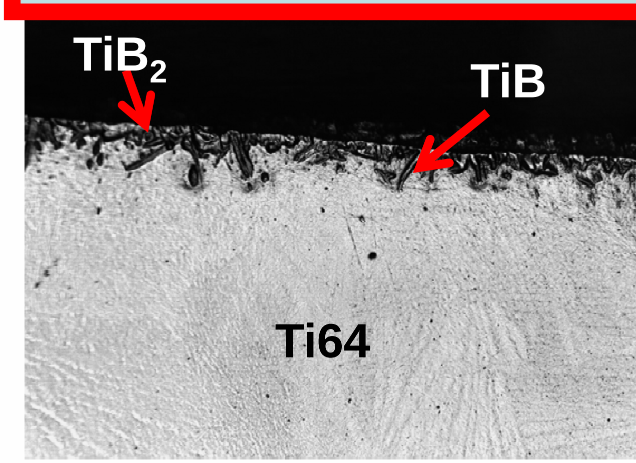

B0



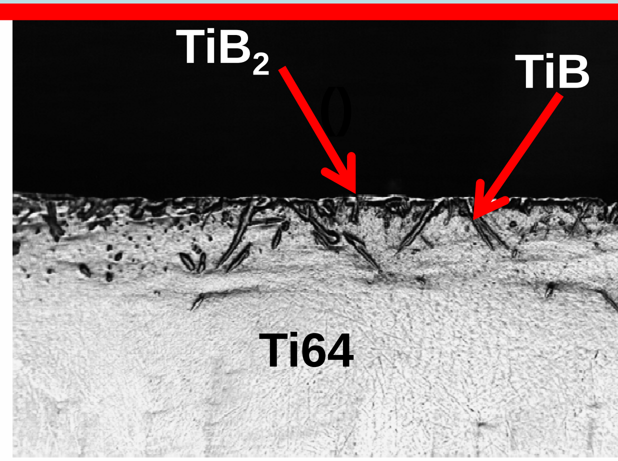




5%C

**$TiB_2$ Layer: Discontinuous**
**Overall coating structure: No significant change**



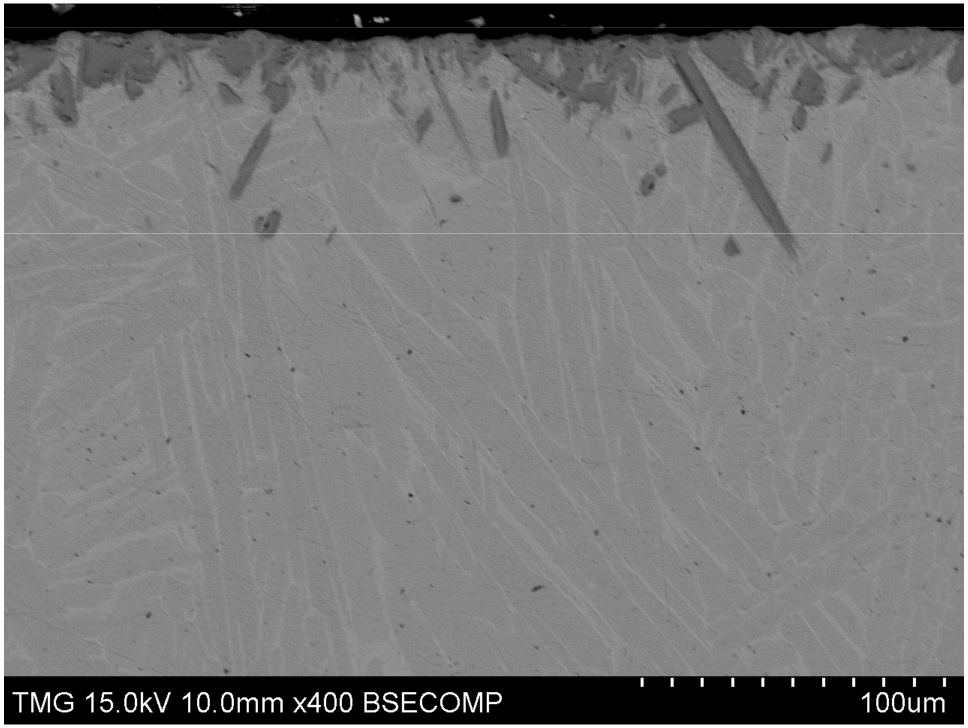

**B3: 50%B, 25%$Na_2CO_3$, 25%C**

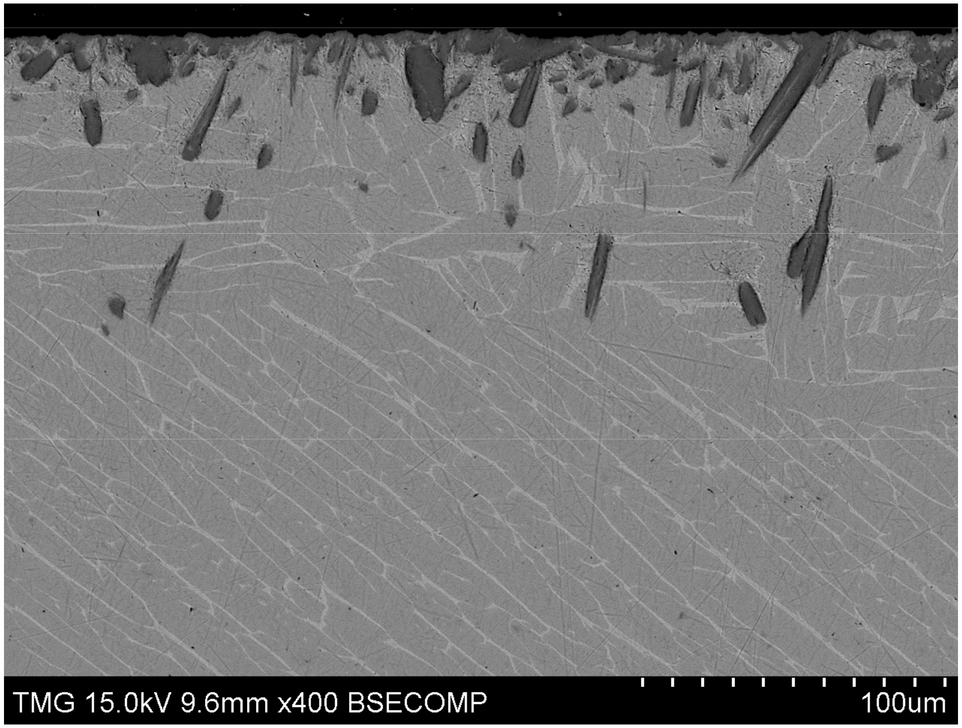



**B5: 50%B, 5%$Na_2CO_3$, 45%C**

# Effect of Powder Composition II

## Same Heat Treatment:  1050ºC 72 hrs



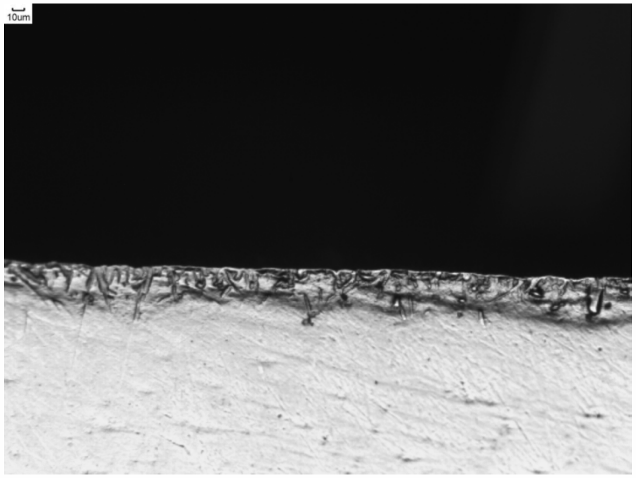




**B1, 1050ºC 72 hrs**

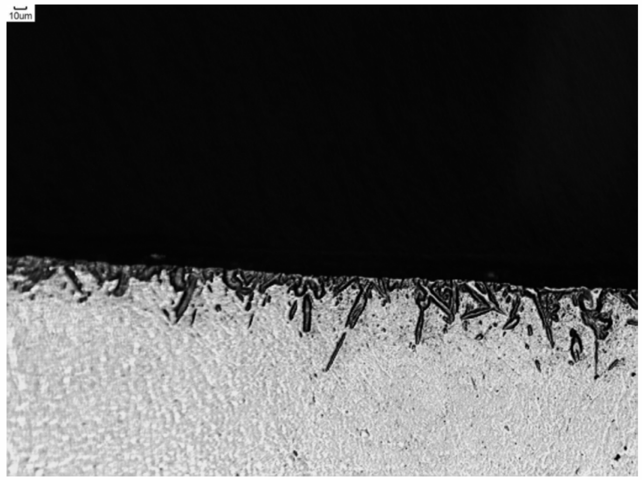
**B5, 1050ºC 72 hrs**

# Effect of Holding Time I

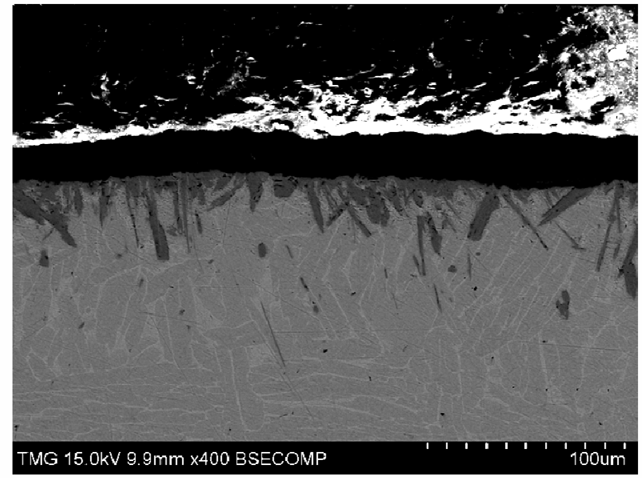
## Same Composition B3, Same heating temperature



**1050ºC 12 hrs**
**B3: 50%B, 25%$Na_2CO_3$, 25%C**
*Optical*



**1050ºC 24 hrs**
**B3: 50%B, 25%$Na_2CO_3$, 25%C**
*Optical*

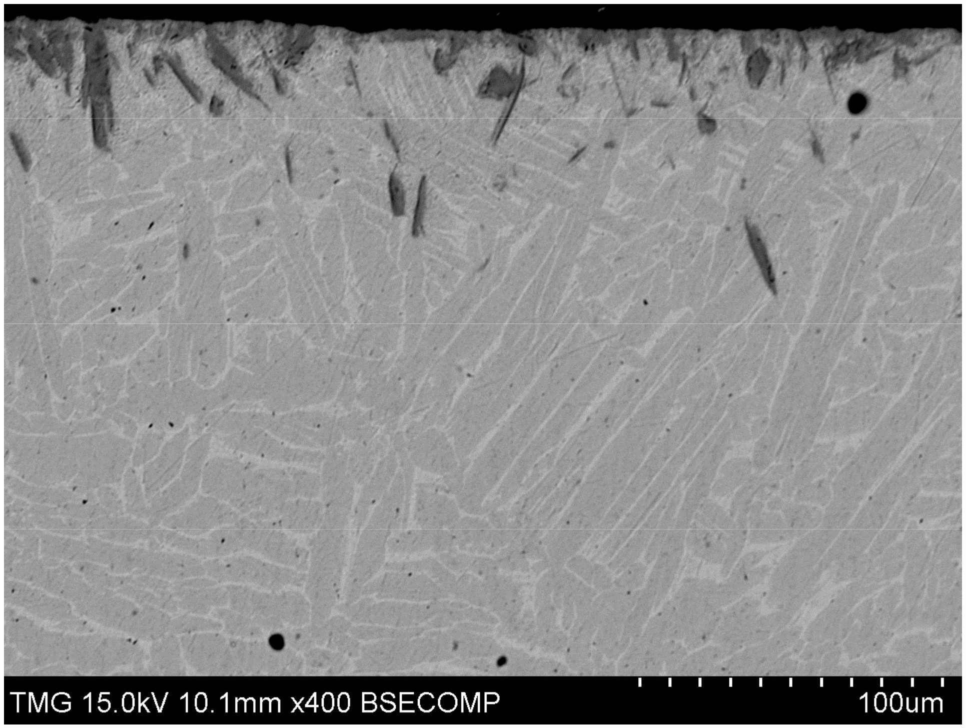



**1050ºC 48 hrs**
**B3: 50%B, 25%$Na_2CO_3$, 25%C**
*SEM (BSE)*

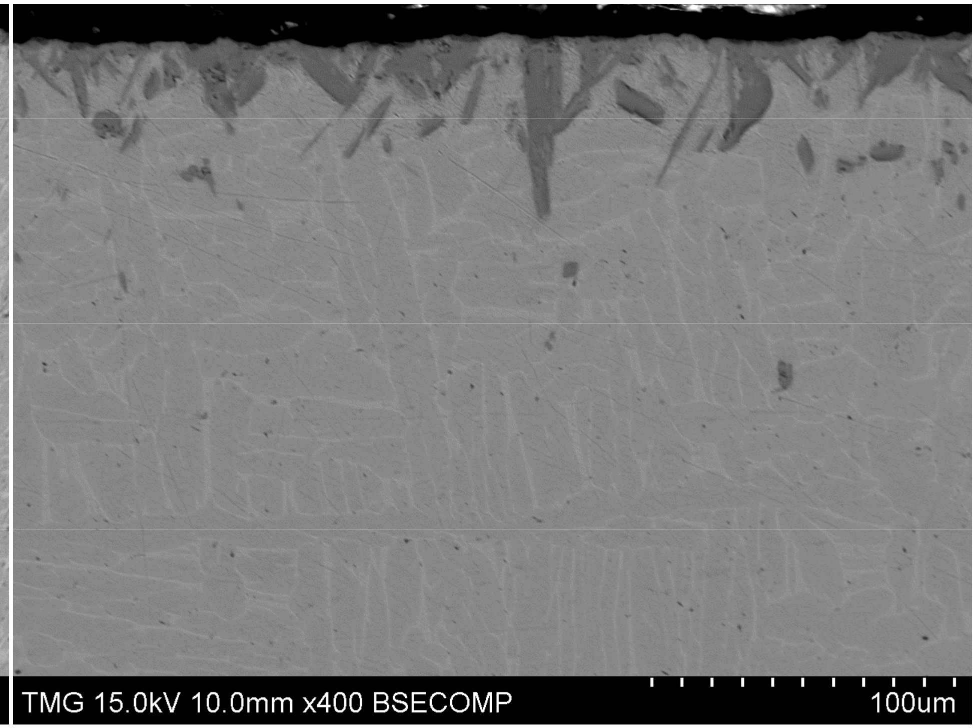
**$TiB_2$ Layer: Discontinuous**
**Overall coating structure: No significant change**

# Effect of Holding Time II

## Same Composition B1, Same heating temperature



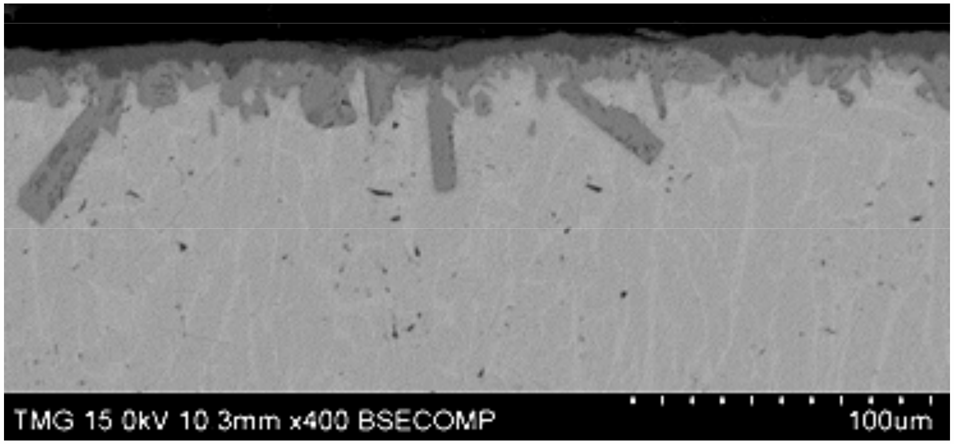

B1, 1050ºC 24 hrs



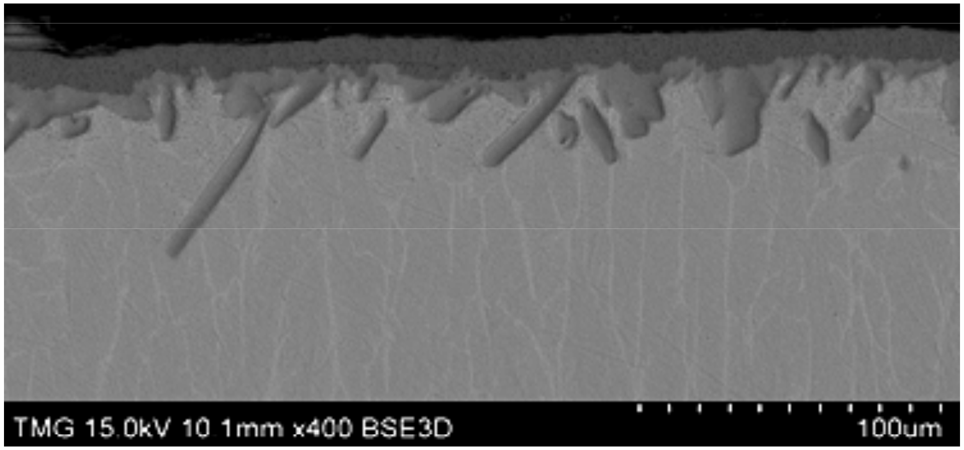

B1, 1050ºC 72 hrs

**$TiB_2$ Layer: Discontinuous**
**Overall coating structure: some change in TiB size**

# Further Exploration for Better Coatings




B3, 1200ºC, 72hrs

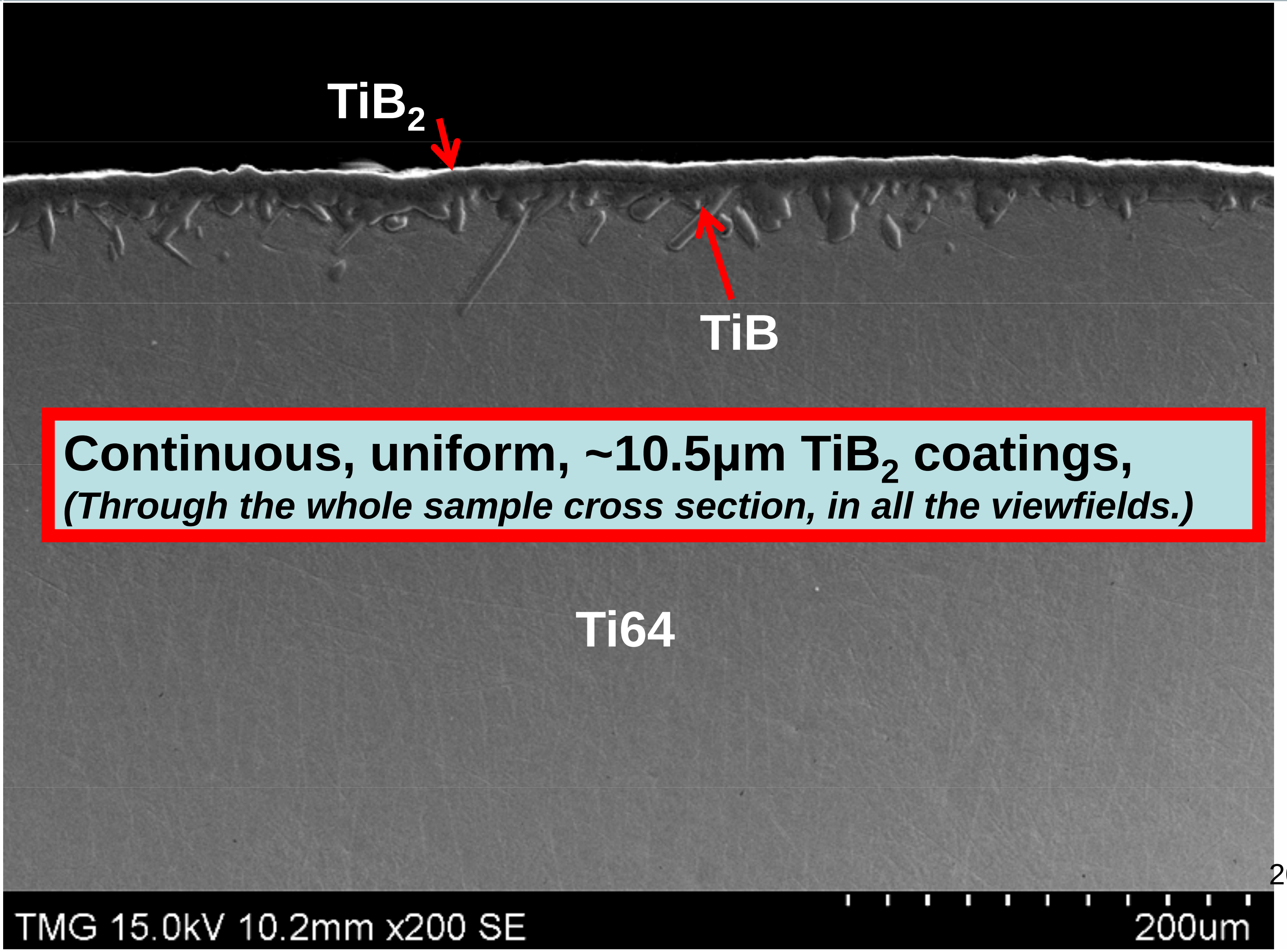



B5, 1200ºC, 72hrs

# Best Coatings So Far

**SEM (SE)**

## Composition B5, 1200ºC X 72 hrs

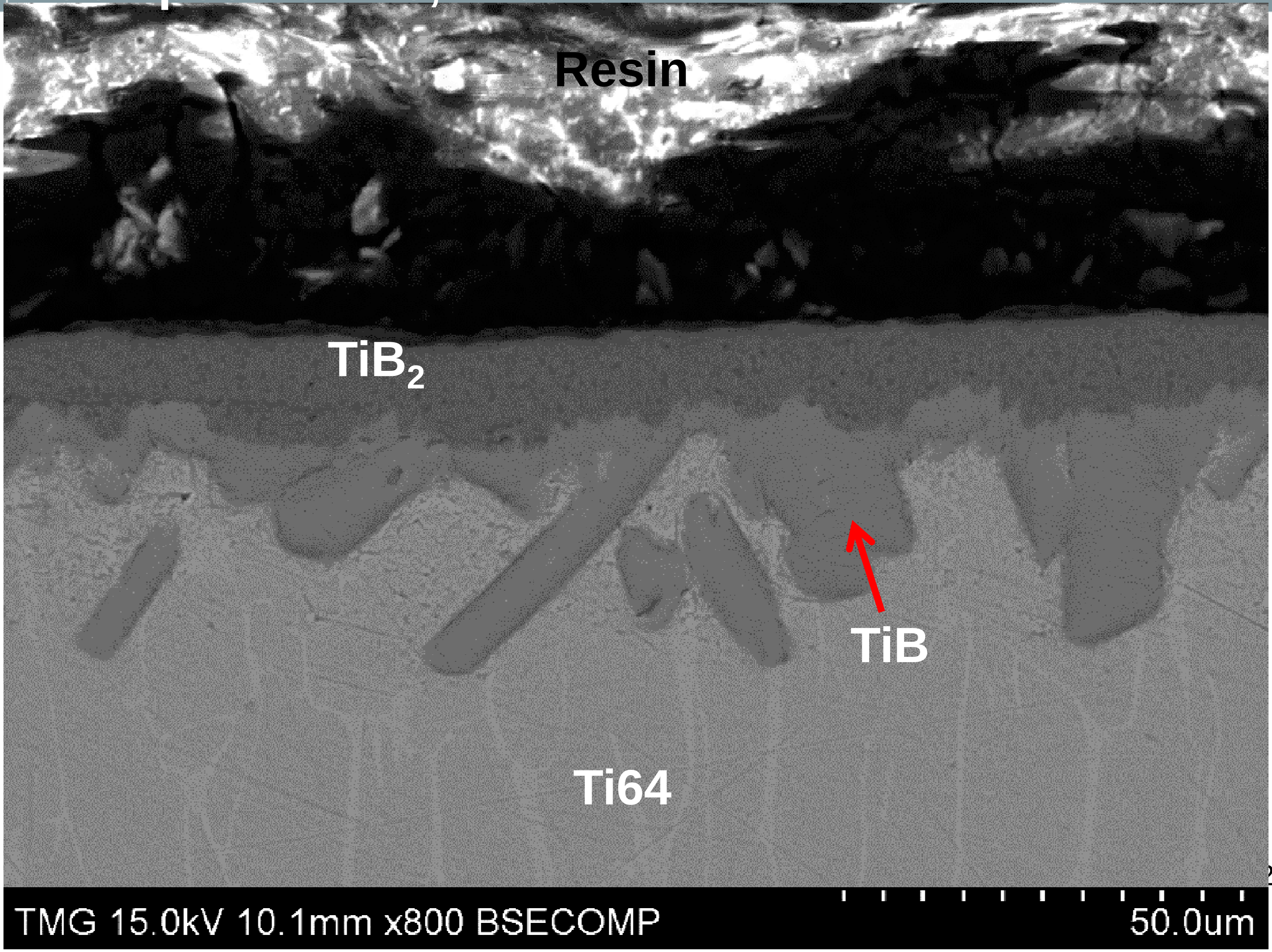



**Continuous, uniform, ~10.5µm $TiB_2$ coatings,**
***(Through the whole sample cross section, in all the viewfields.)***

# Best Coatings So Far

**SEM (BSE)**

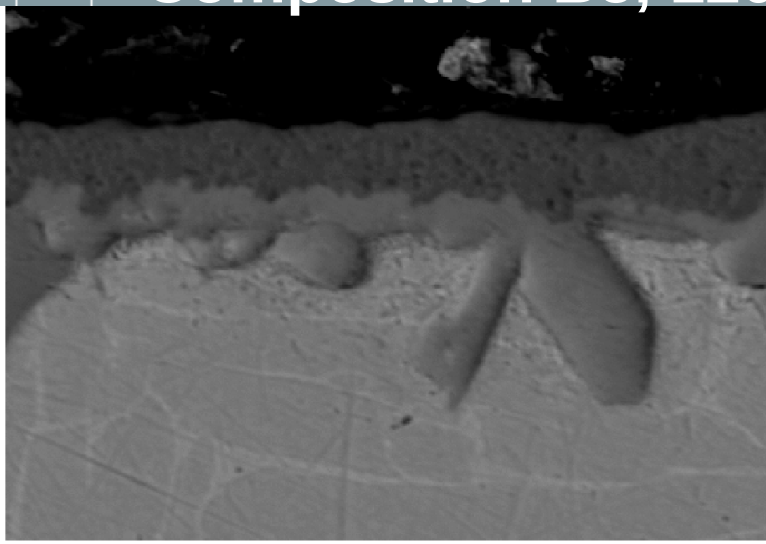
## Composition B5, 1200ºC X 72 hrs



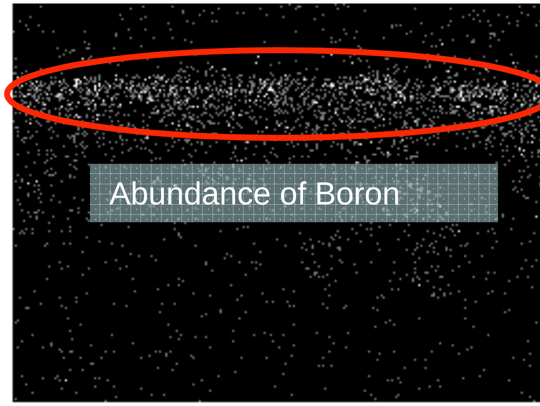
# Best Coatings So Far

## EDS (Mapping)

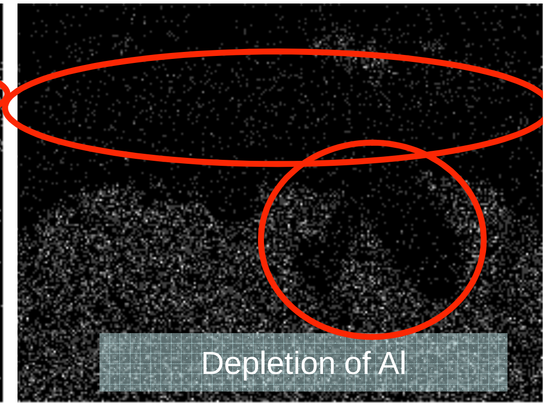
## Composition B5, 1200ºC X 72 hrs



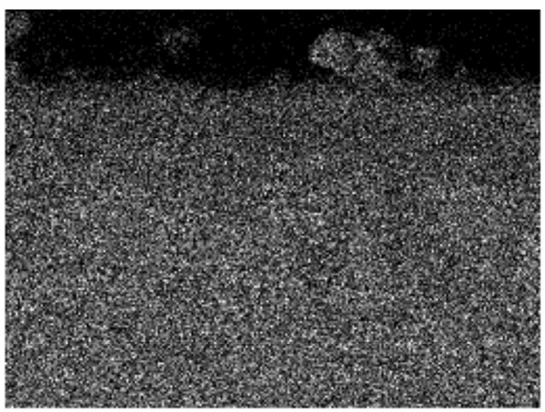
Electron Image 1



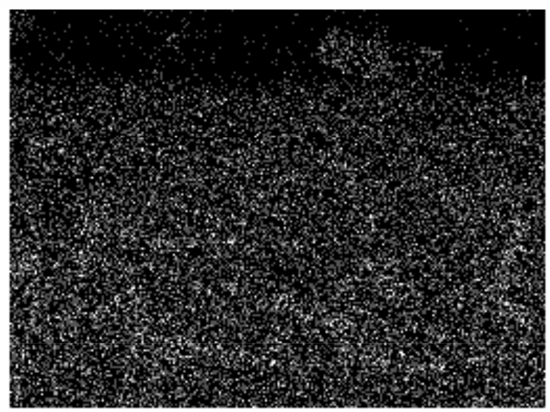

B Ka1_2




Al Ka1



Ti Ka1

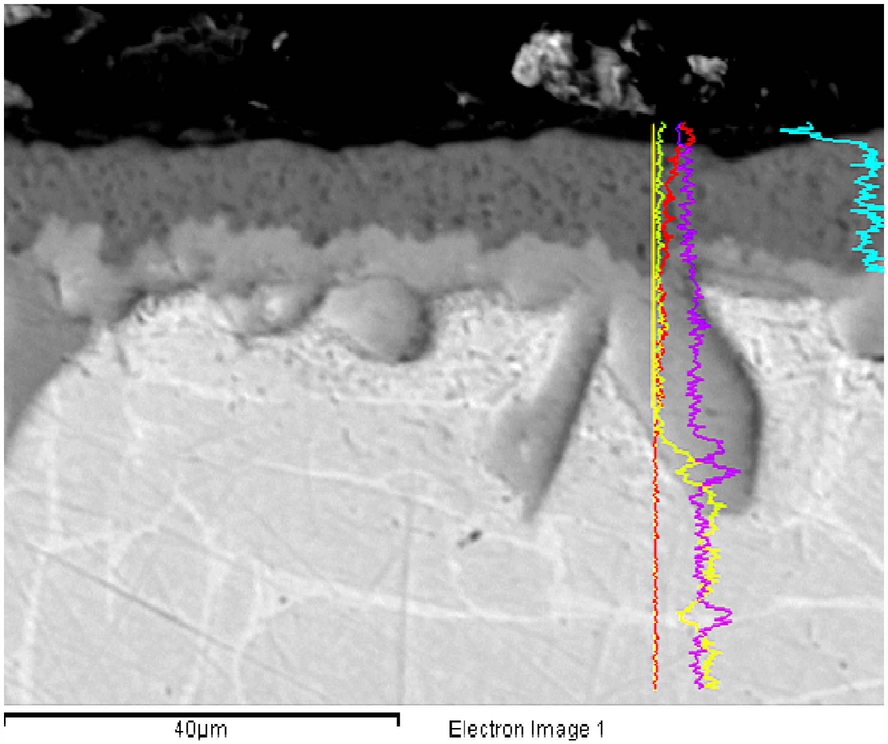


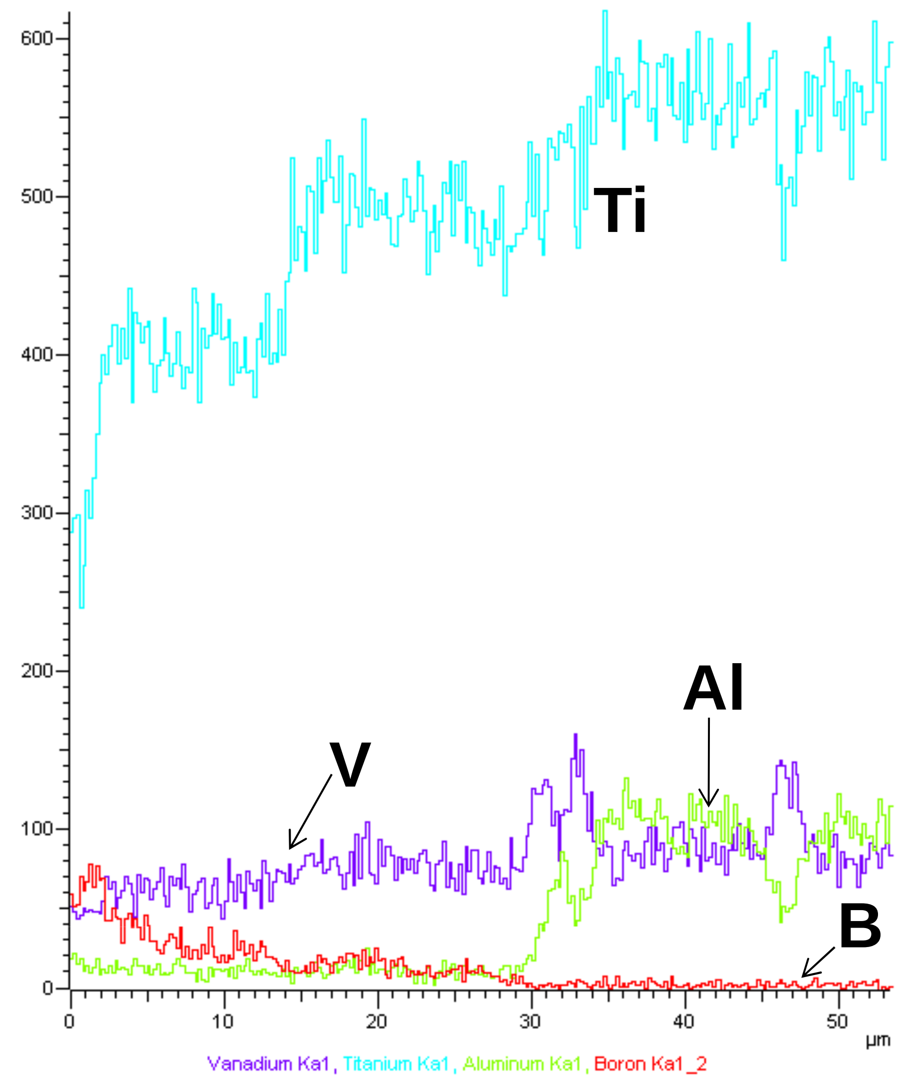
V Ka1

# Best Coatings So Far

## EDS (Line Scan)

## Composition B5, 1200ºC X 72 hrs

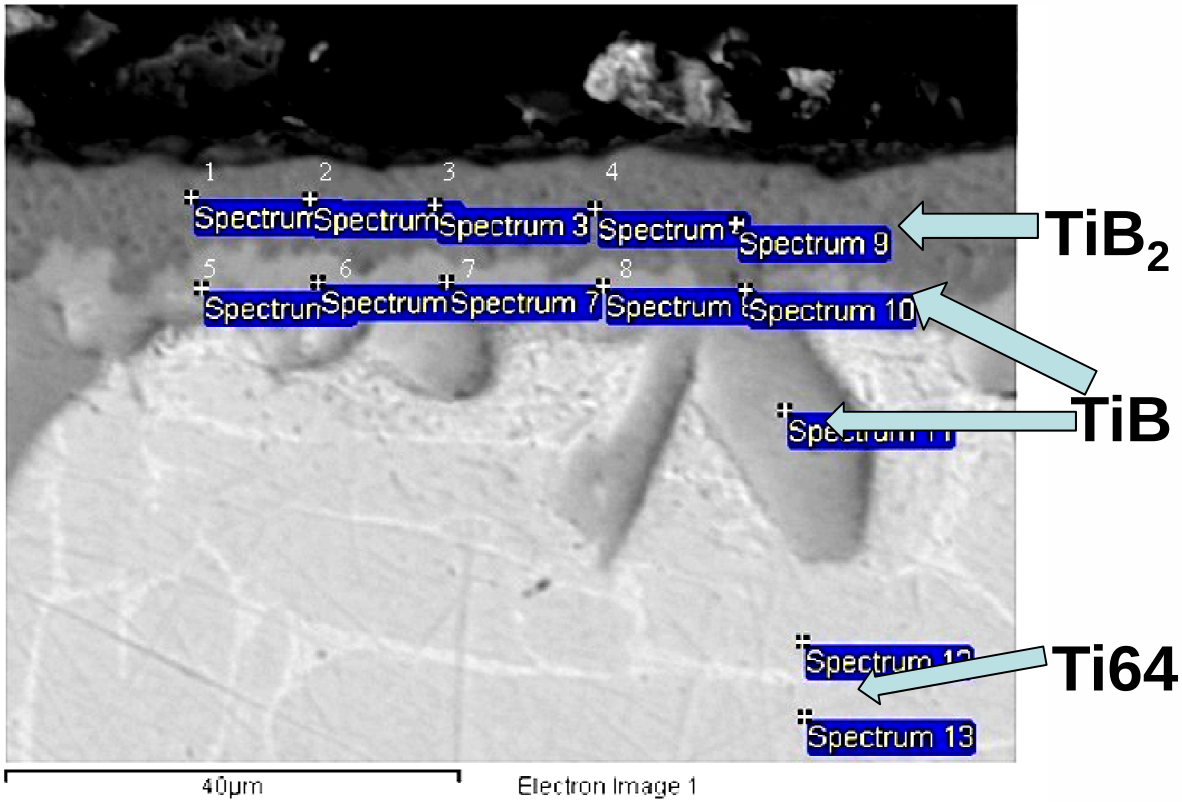


# Best Coatings So Far

## EDS (Point Identification)

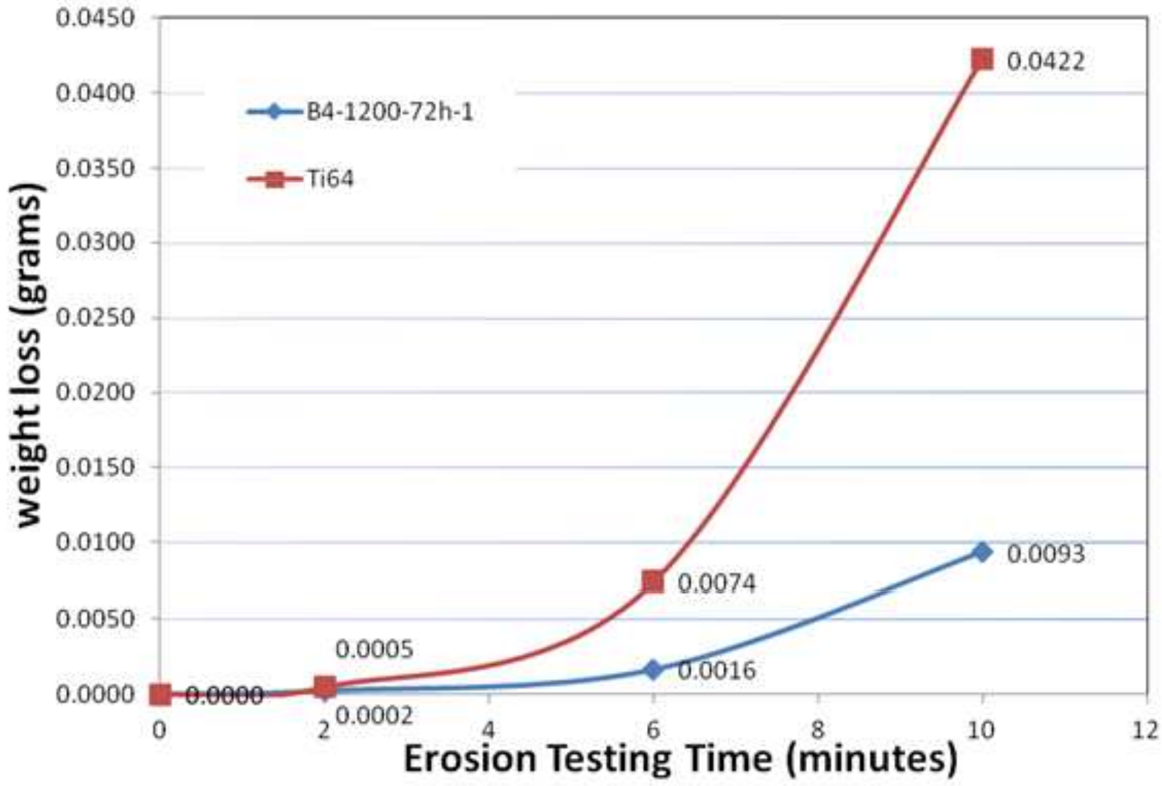
## Composition B5, 1200ºC X 72 hrs



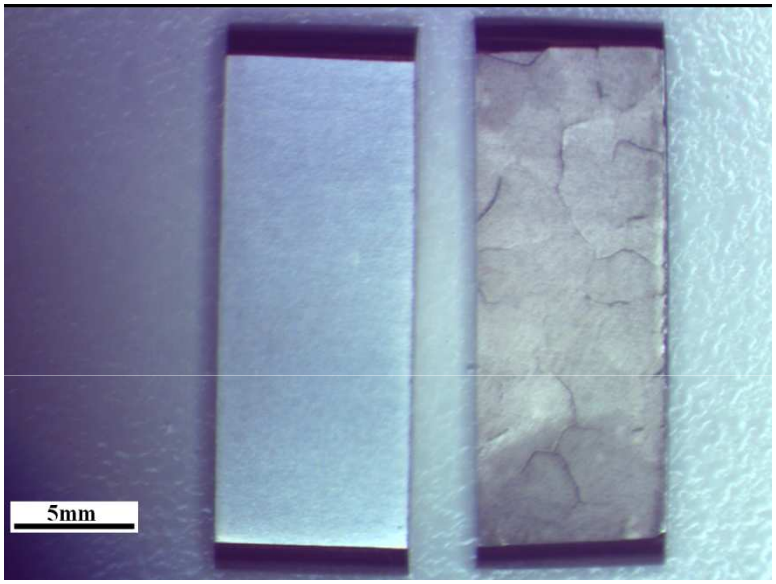

| Spectrum | B, at% | Al, at% | Ti, at% | V, at% |
|---|---|---|---|---|
| Spectrum 1 | 74.08 | 0.02 | 24.86 | 1.05 |
| Spectrum 2 | 72.98 | 0.01 | 26.15 | 0.86 |
| Spectrum 3 | 73.77 | 0.03 | 25.48 | 0.71 |
| Spectrum 4 | 72.18 | 0.02 | 27.04 | 0.75 |
| Spectrum 9 | 70.69 | 0.05 | 28.24 | 1.02 |
| Spectrum 5 | 25.02 | 2.35 | 70.20 | 2.43 |
| Spectrum 6 | 41.13 | 0.07 | 56.85 | 1.95 |
| Spectrum 7 | 41.72 | 0.56 | 55.74 | 1.98 |
| Spectrum 8 | 44.72 | -0.17 | 53.37 | 2.08 |
| Spectrum 10 | 61.82 | 0.07 | 37.14 | 0.96 |
| Spectrum 11 | 50.09 | 0.16 | 48.39 | 1.35 |
| Spectrum 12 | | 12.06 | 85.44 | 2.50 |
| Spectrum 13 | | 11.74 | 86.20 | 2.06 |
| Ti64 Nomination | | 10.20 | 86.20 | 3.60 |

# Water Erosion Testing

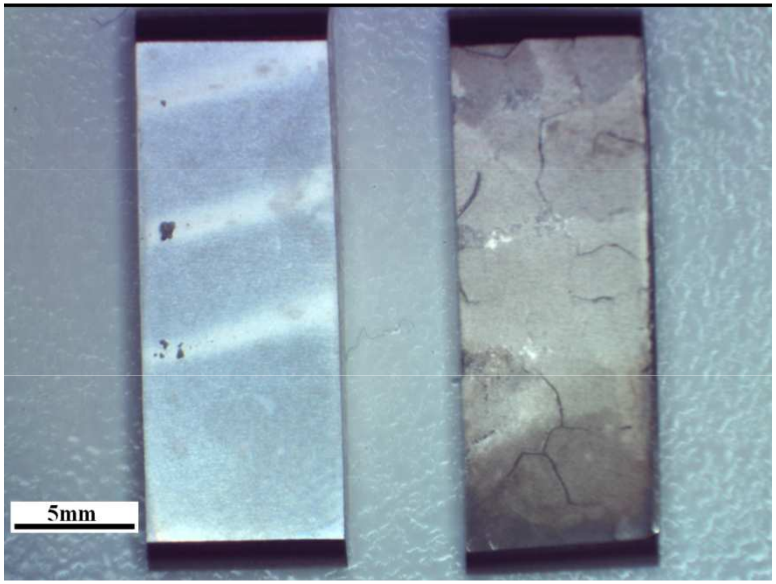
- Samples preparation: Boronizing with powder compostion B4, @1200ºCX72hous
- RIG testing parameter:
- **Speed: 14000 RPM**
- **Time: 0-10 minutes**
- **Nozzle size: 400 µm**
- **Flow Rate: 0.050-0.070 L/min**



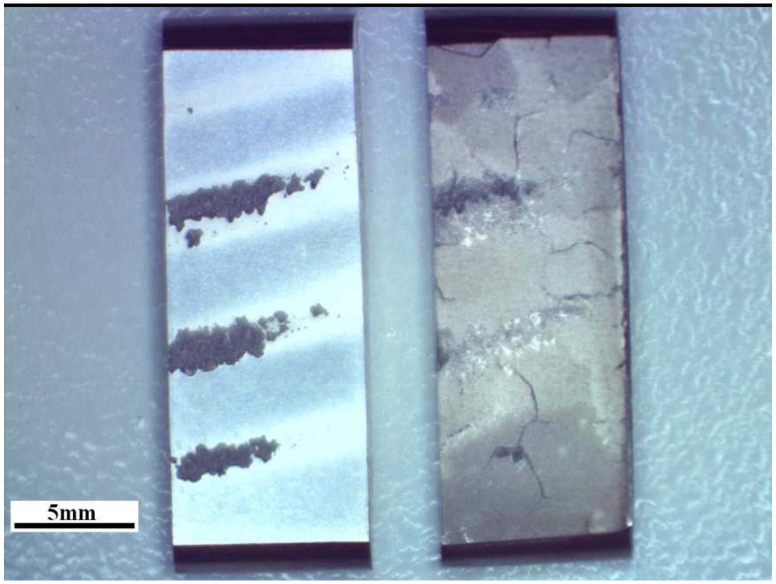
# Water Erosion Testing, B4- 1200ºC X 72 hrs  Vs Ti64



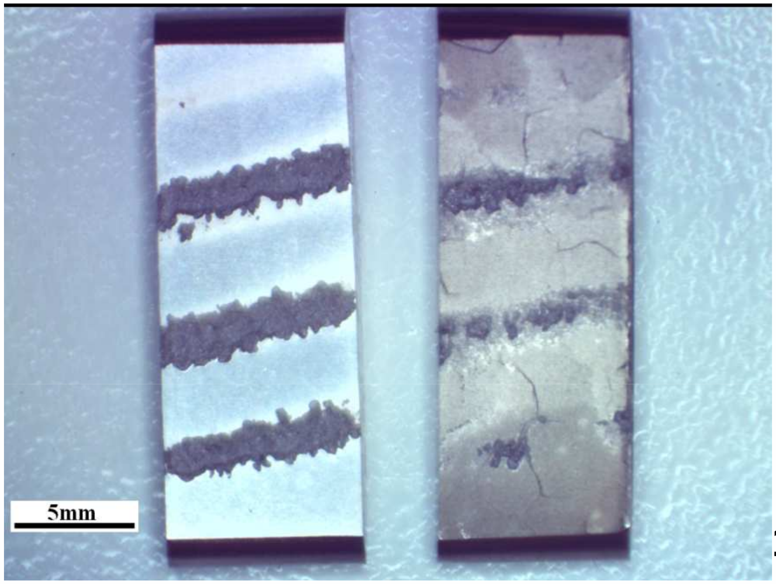

0 min




2 min



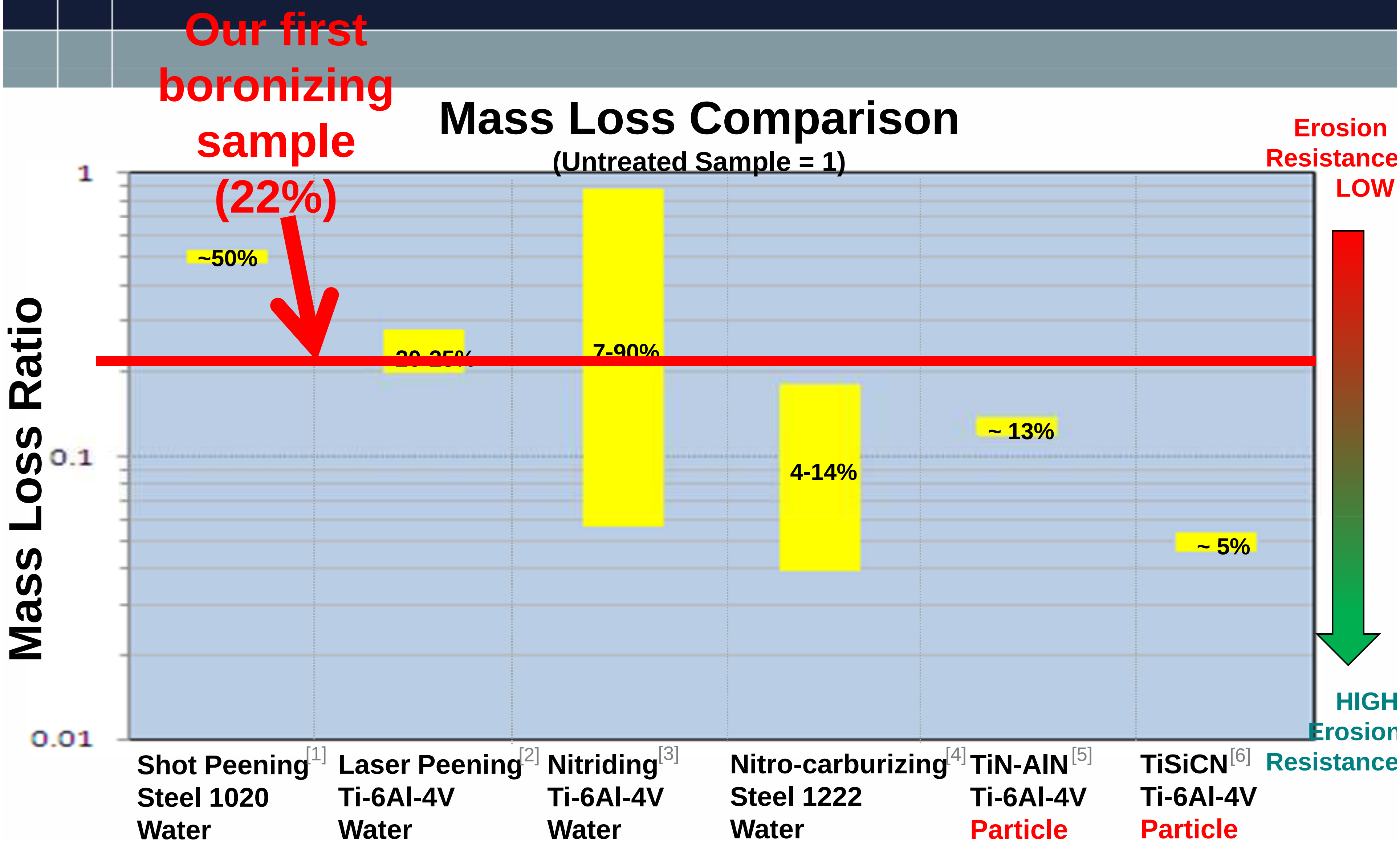

6 min




10 min

# Mass Loss Comparison

(Untreated Sample = 1)



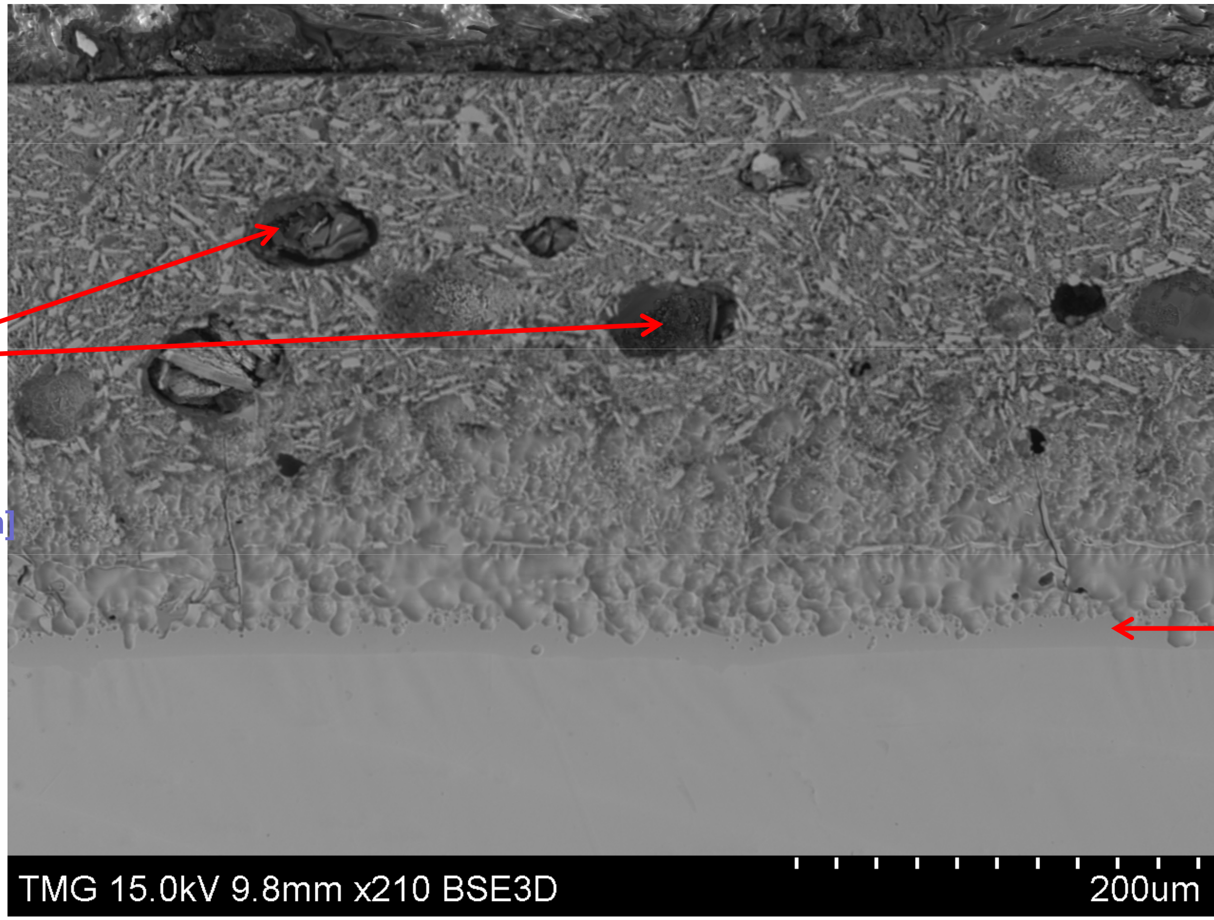


*Ref: [1] MIC 1988, [2] Robbinson 1995, [3] Ahmad 2009,*
*[4] Pant 2010, [5] Rogers 2008, [6] Swaminathan 2008*

Part III

# $TiC$-$TiB_2$-$TiAl_3$ Coatings

# $TiC-TiB_2/TiAl_3$ In-situ Coatings




Porosities

[expected to be reduced by new experiment design]

Resin

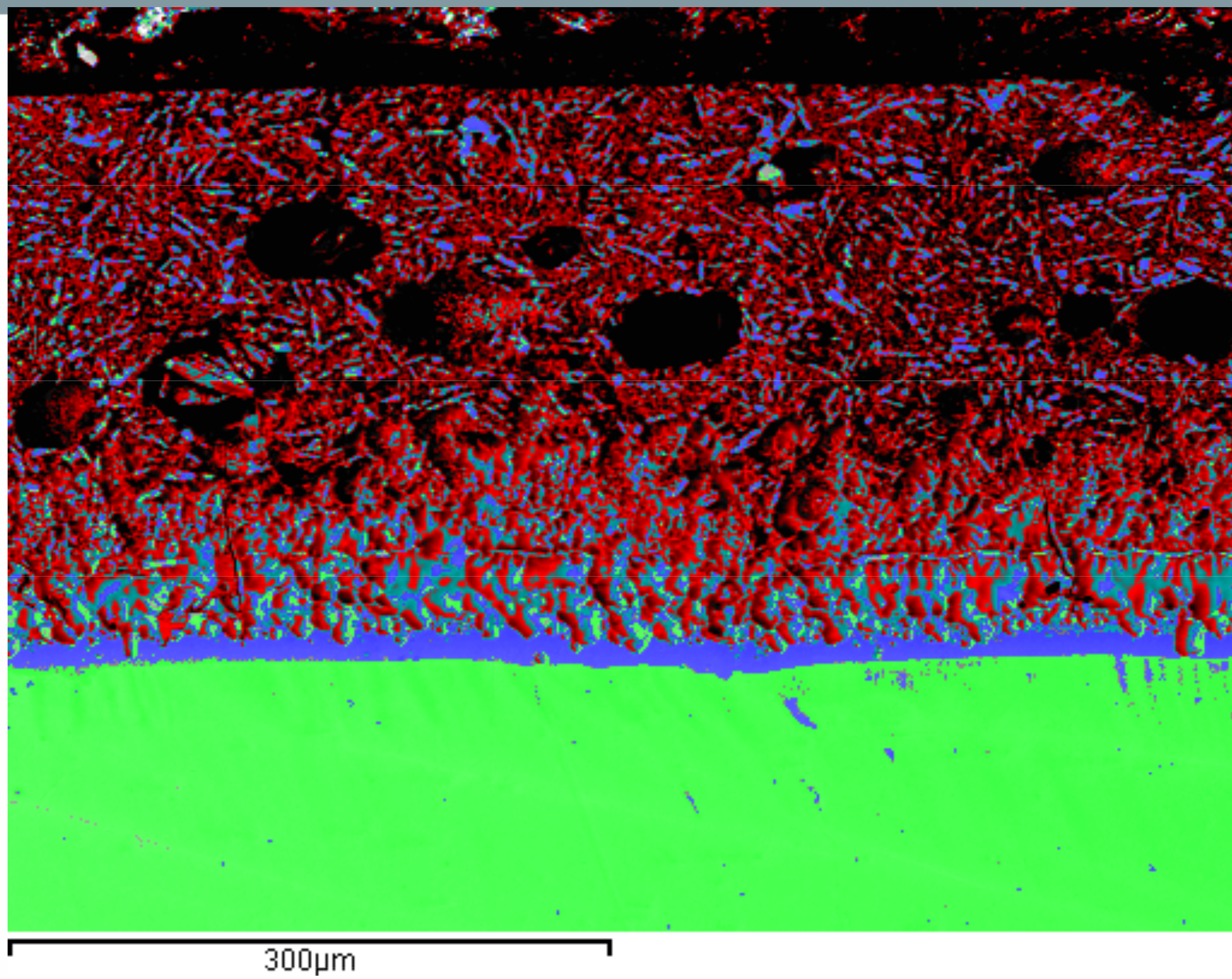
Coating layer

TiAl3 layer

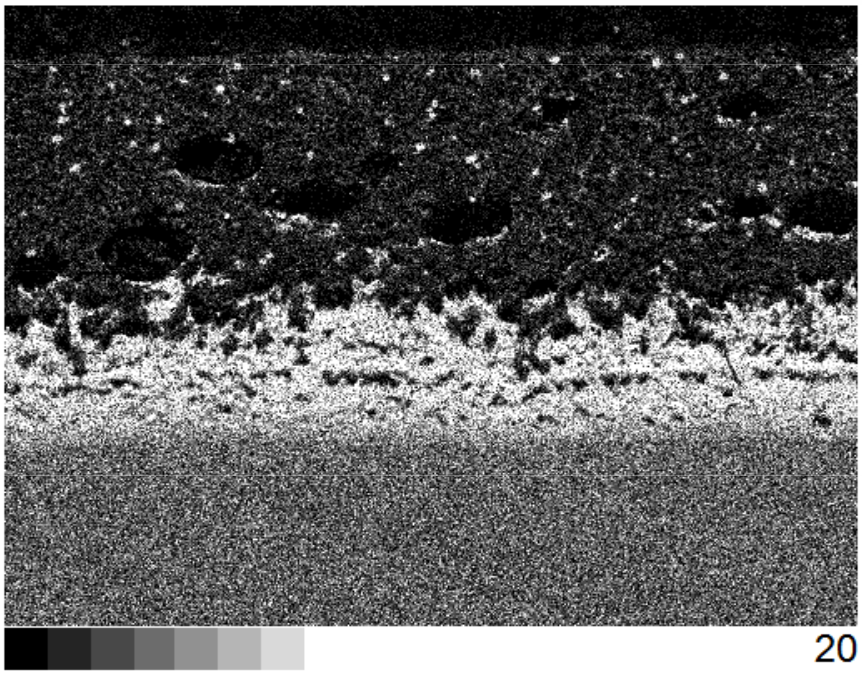
New-found diffusion layer

Ti64

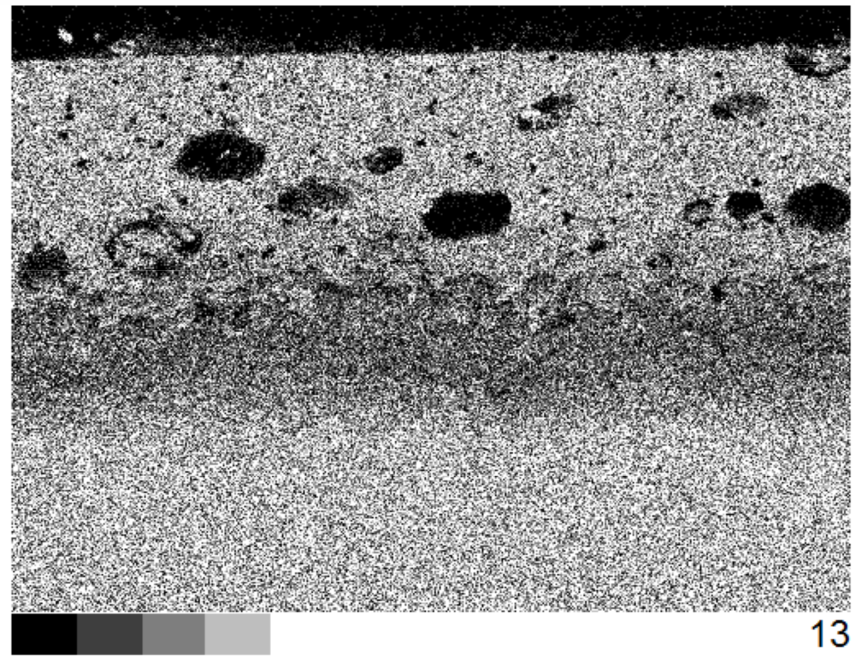
# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings



# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings



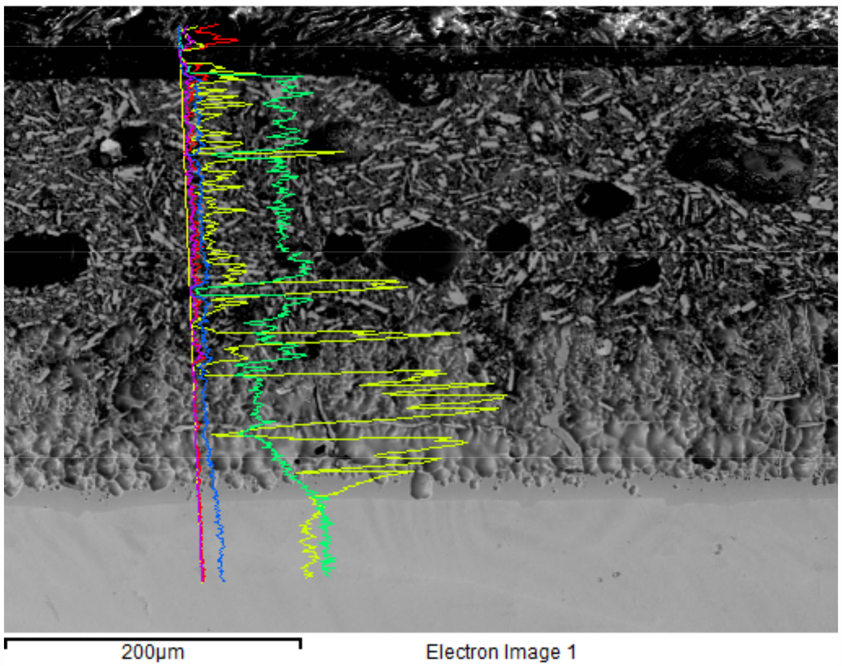

Al Ka1

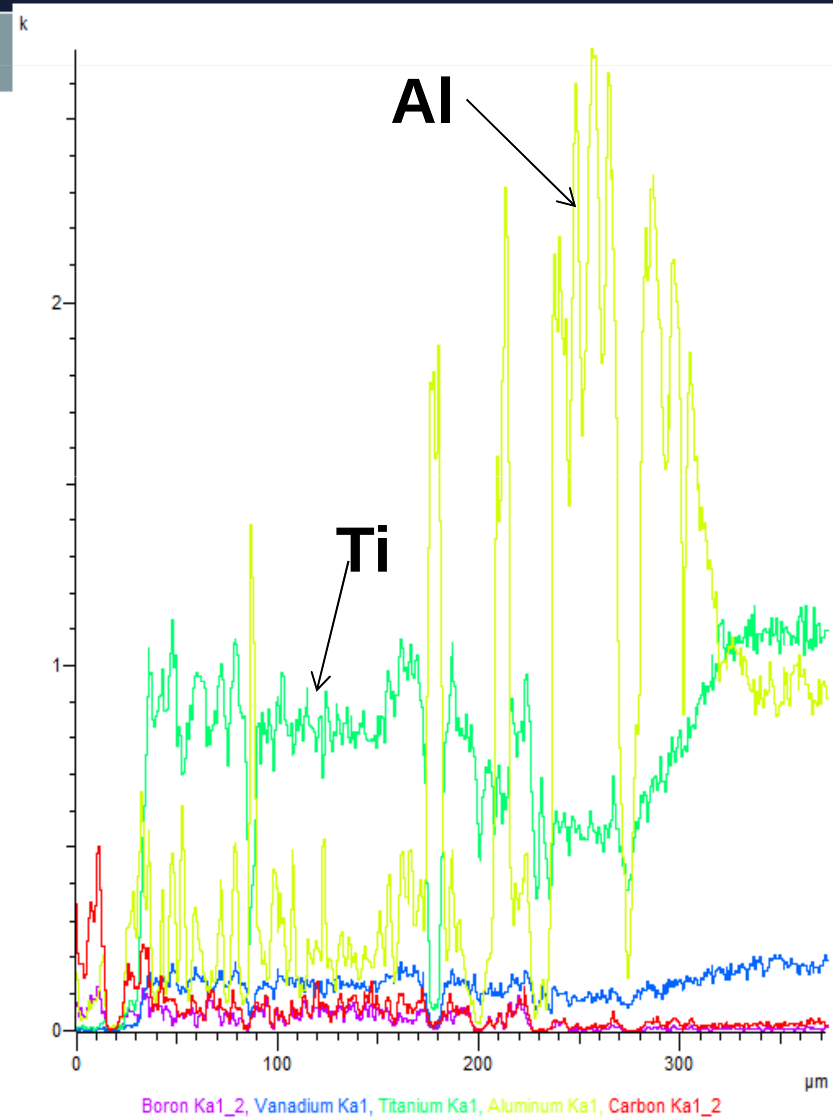



Ti Ka1

**Al concentrated in the bonding field, indicating possibility of *good bonding* & *higher percentage of strengthening phases* in the coating**

# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings



# Water Erosin Testing

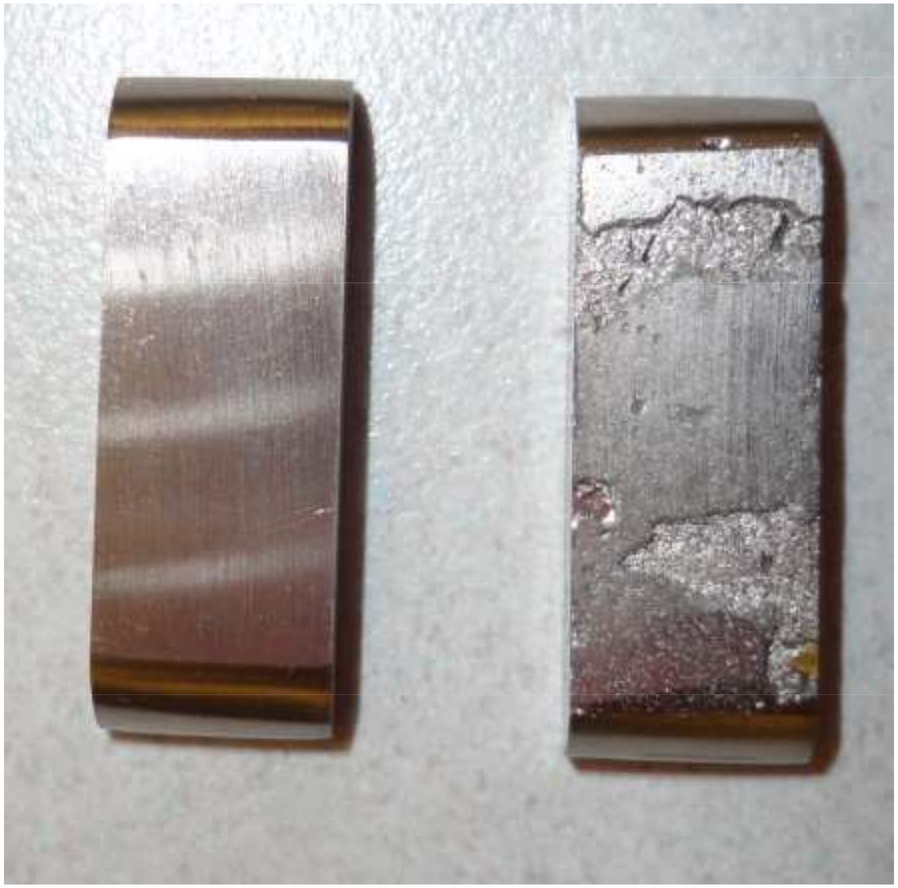
**Ti64**
**Uncoated**

**Vacuum: 44-50 mBar**
**Speed: 8000 RPM**
**Time: 40 minutes**
**Nozzle distance: 30mm**
**Flow Rate: 0.065-0.070 L/min**

**$TiC$-$TiB_2$-$TiAl_3$**
**coating**

**Weight Loss:**

**0.0005 g**



**Weight Loss:**

**0.0358 g**

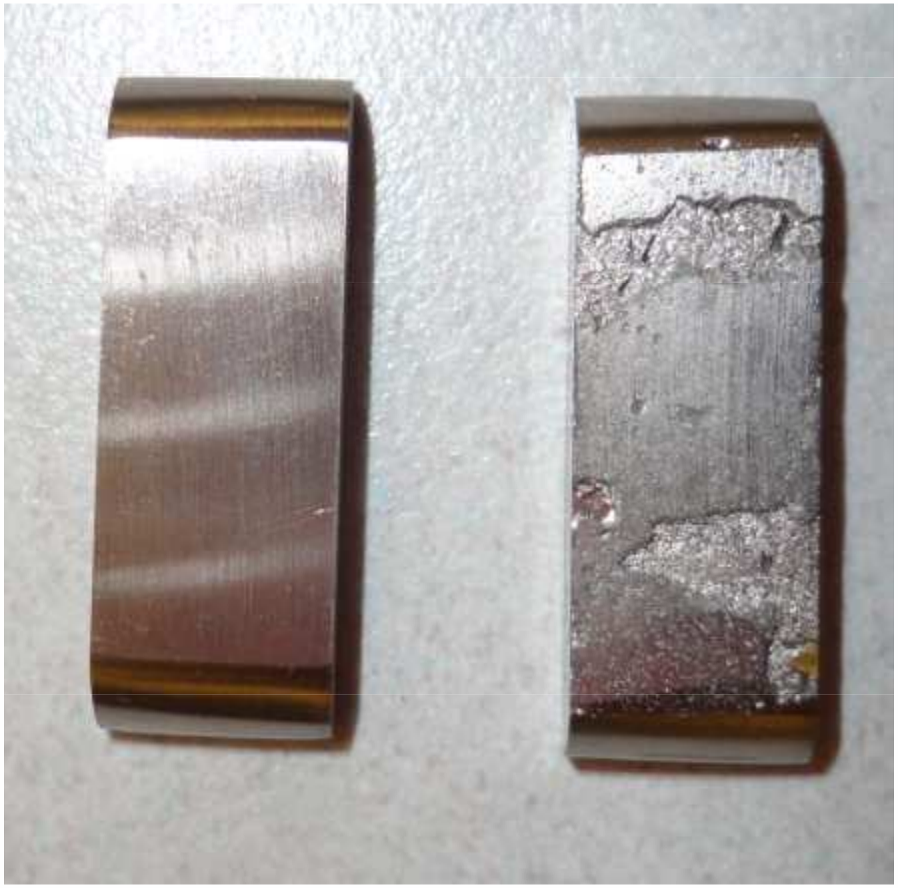
# $TiC-TiB_2/TiAl_3$ In-situ Coatings

Vacuum: 44-50 mBar
Speed: 8000 RPM
Time: 40 minutes
Nozzle size: 600 µm
Nozzle distance: 30mm
Flow Rate: 0.065-0.07 L/min

**Ti64 Uncoated**

**Weight Loss:**

**0.0005 g**

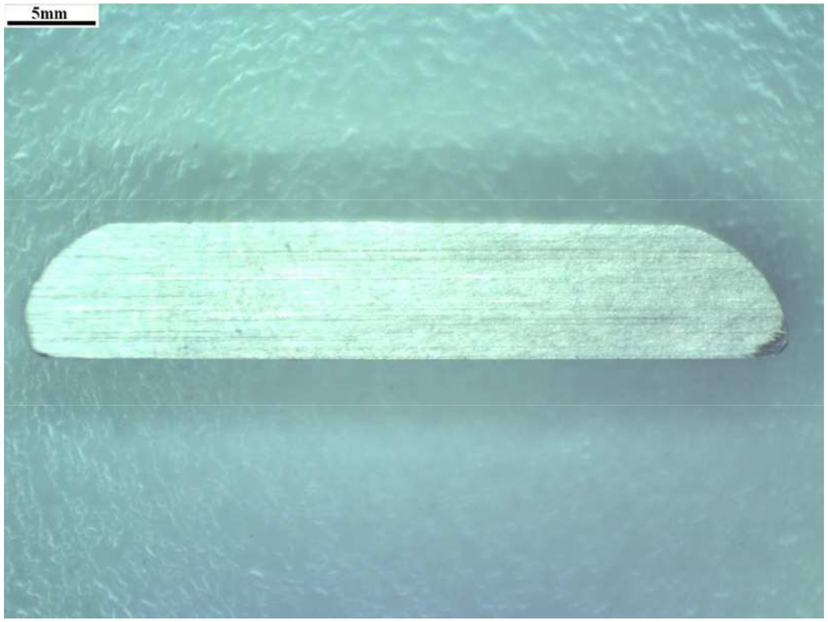


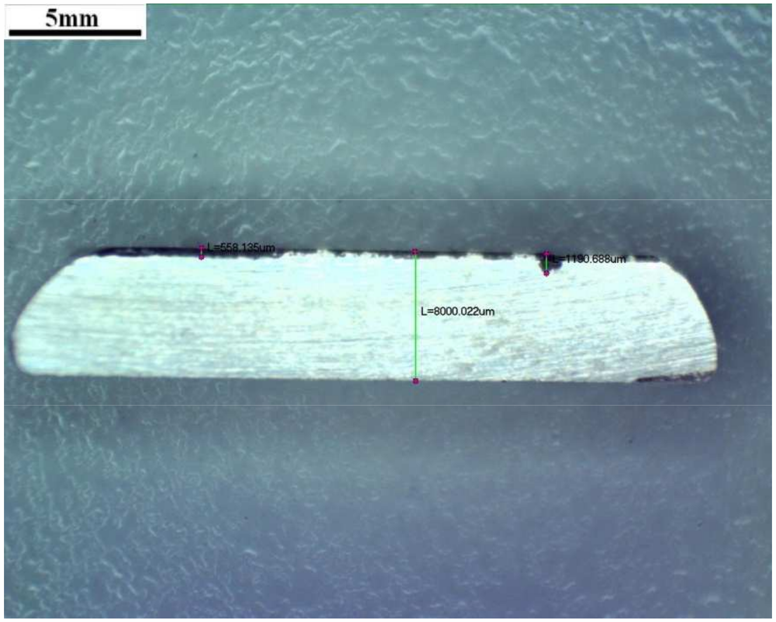
**$TiC-TiB_2-TiAl_3$ coating**

**Weight Loss:**

**0.0358 g**

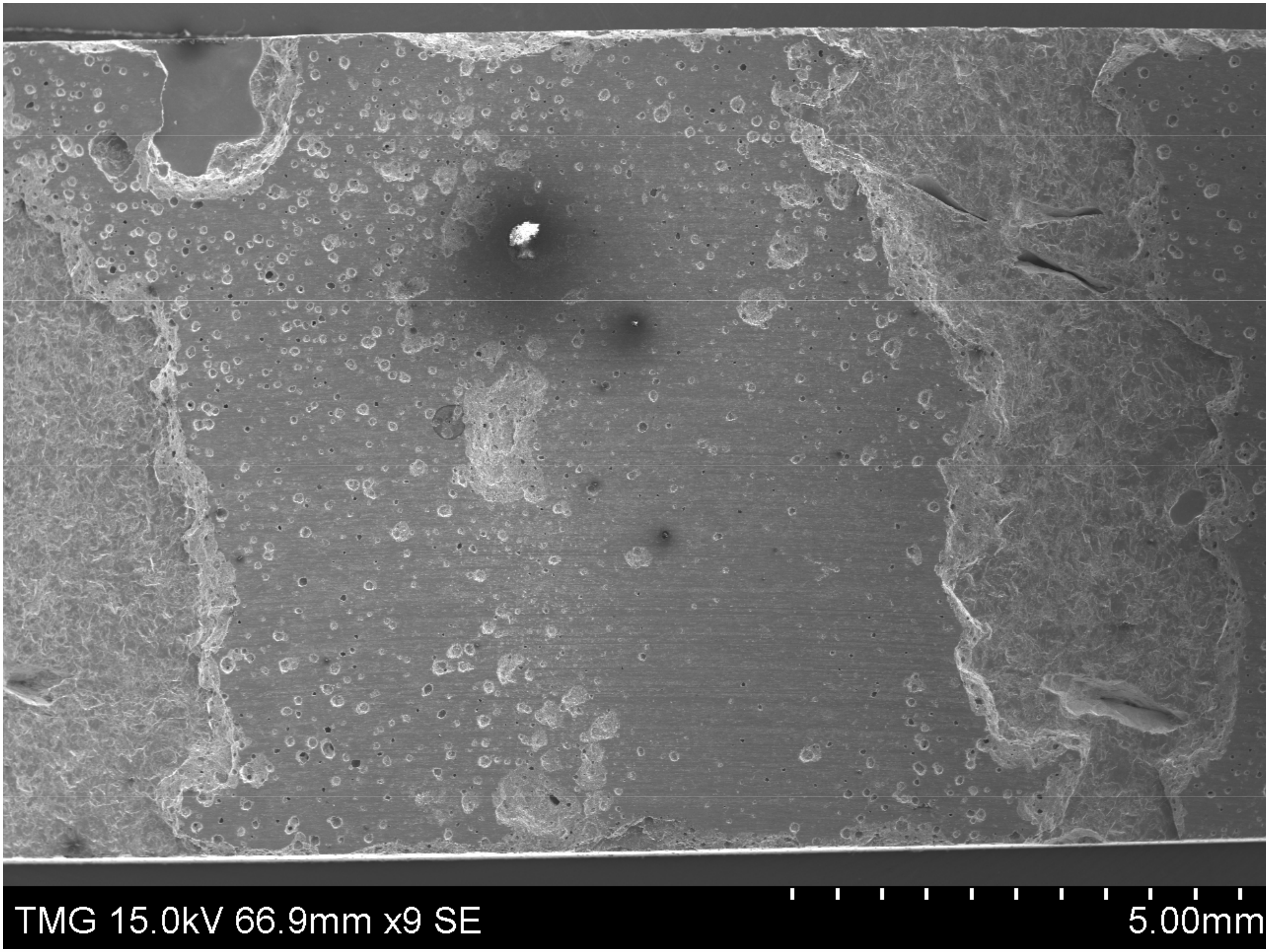
# Profile of the edge of eroded coupons



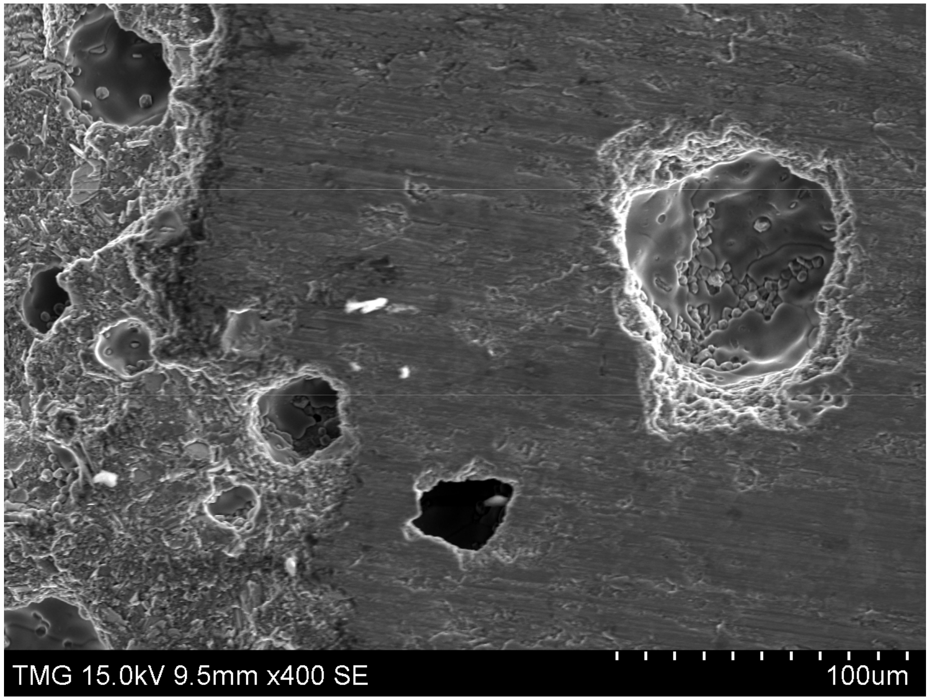
# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings

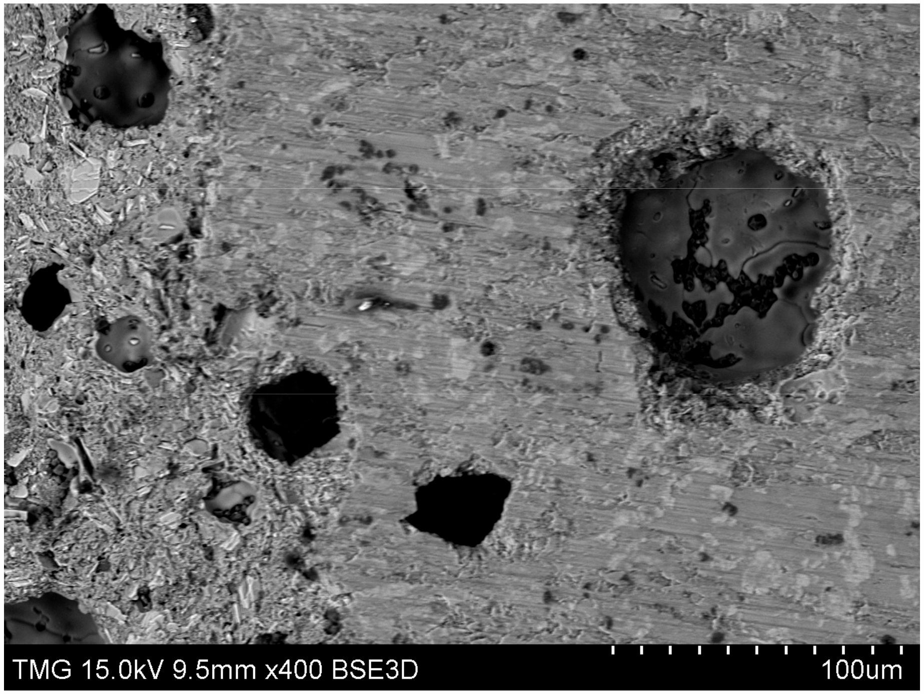
## Eroded Surface SEM – Low magnification



# TiC-$TiB_2$/$TiAl_3$ In-situ Coatings

## Edge of the stream – Original pores on the coating



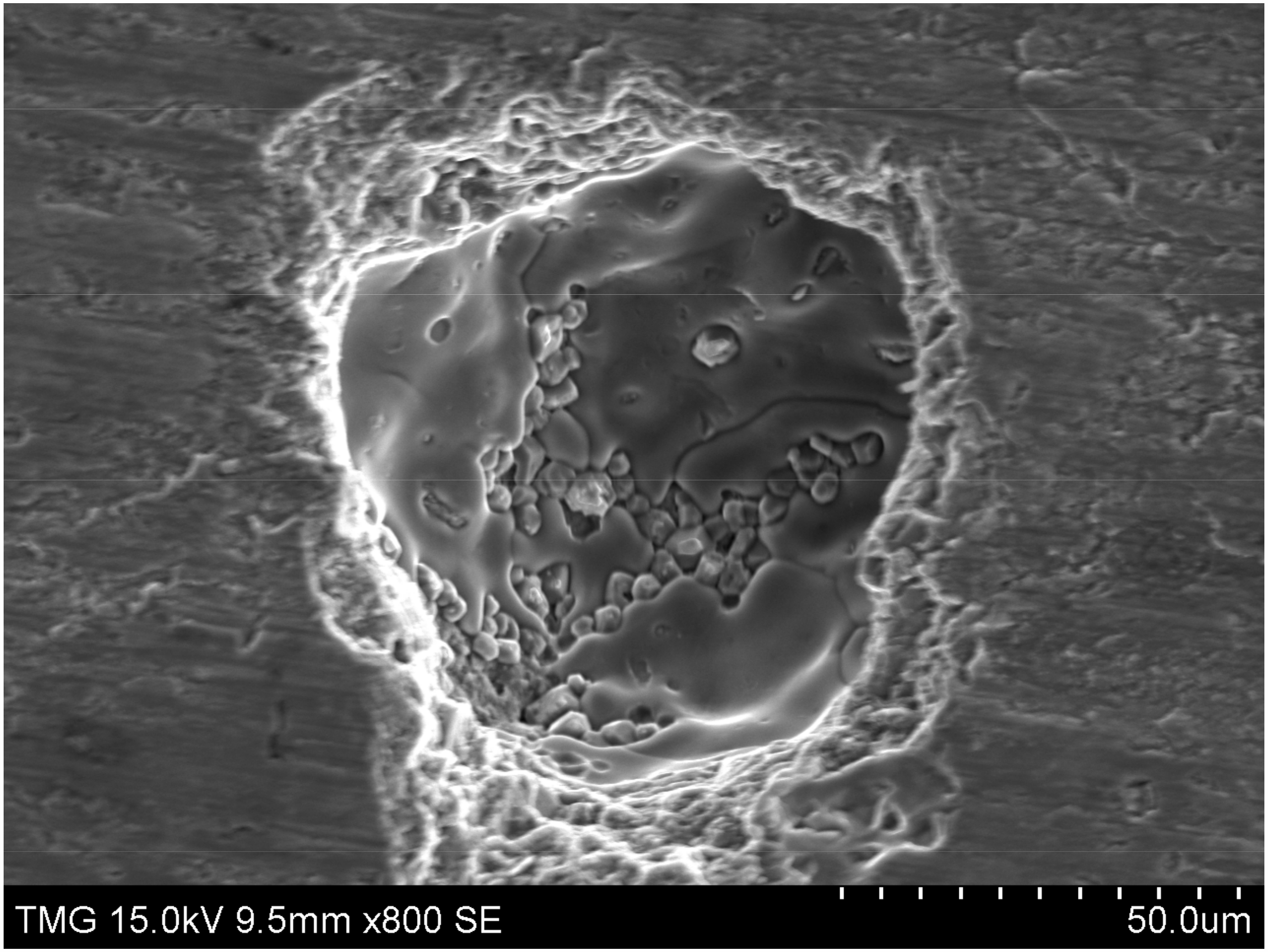




**stream** ←

**Uneroded** →

# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings

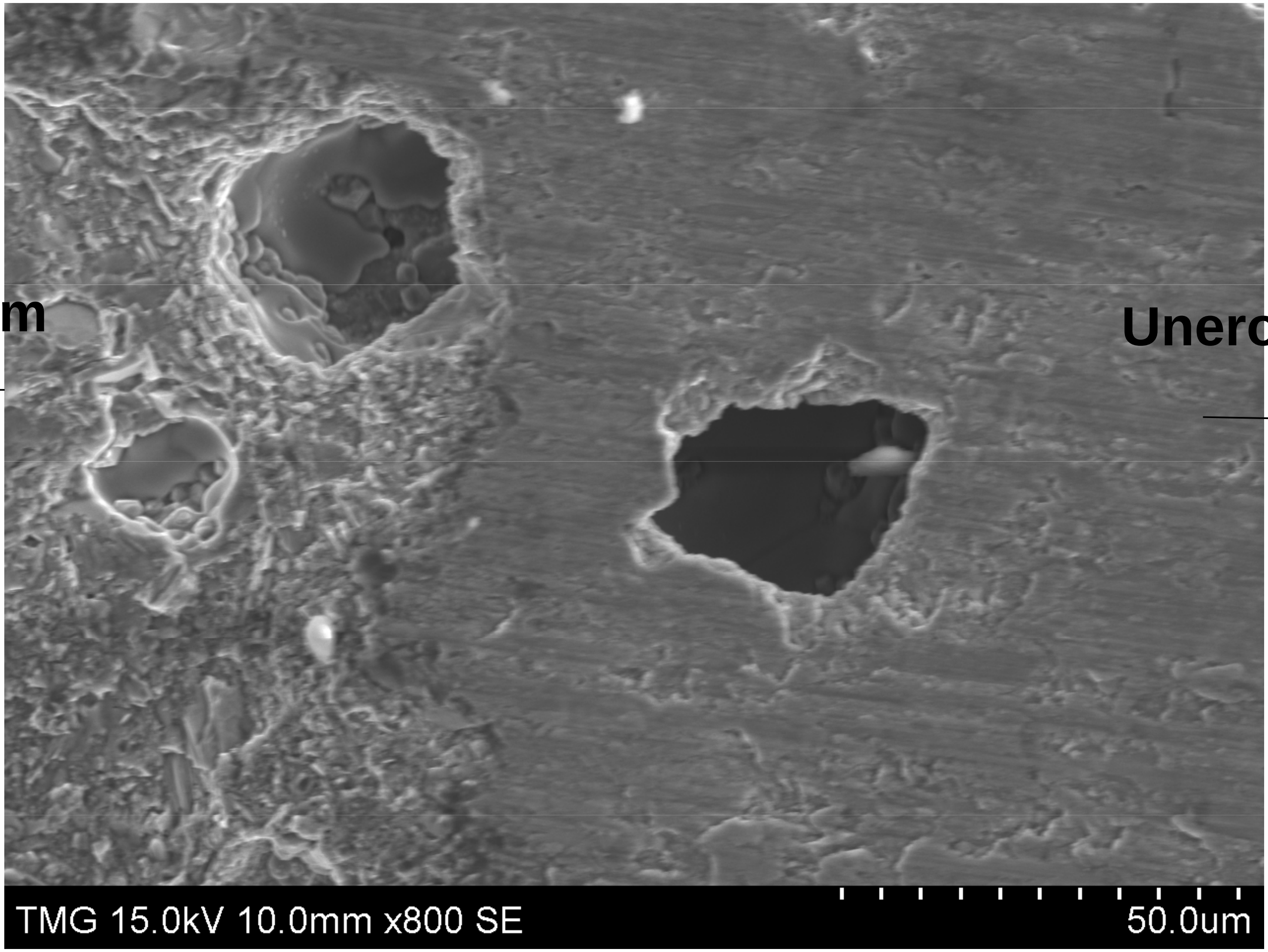
## Edge of the stream – Original pores on the coating



# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings

## Edge of the stream – Erosion of the coating

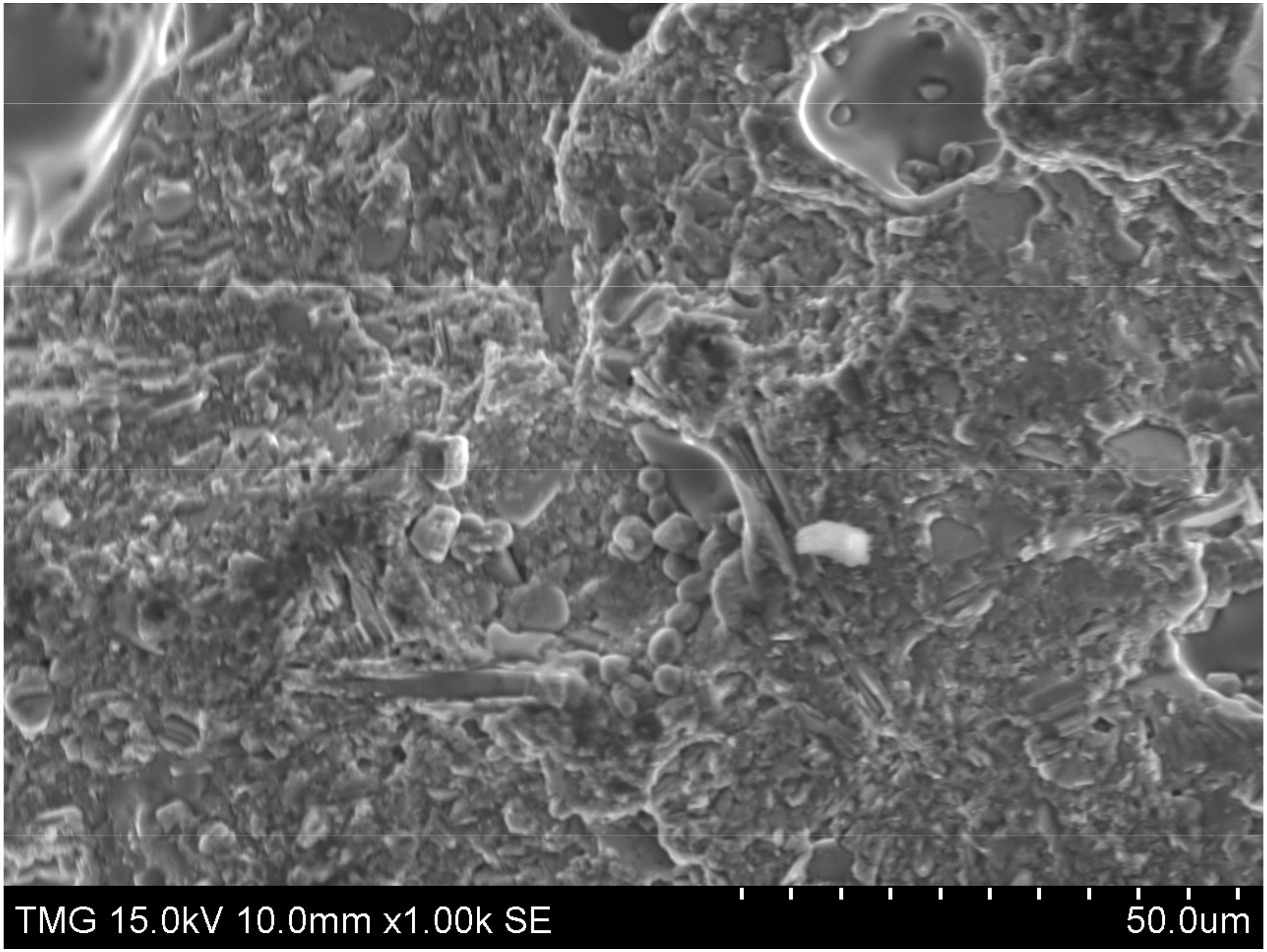
stream ←




Uneroded →

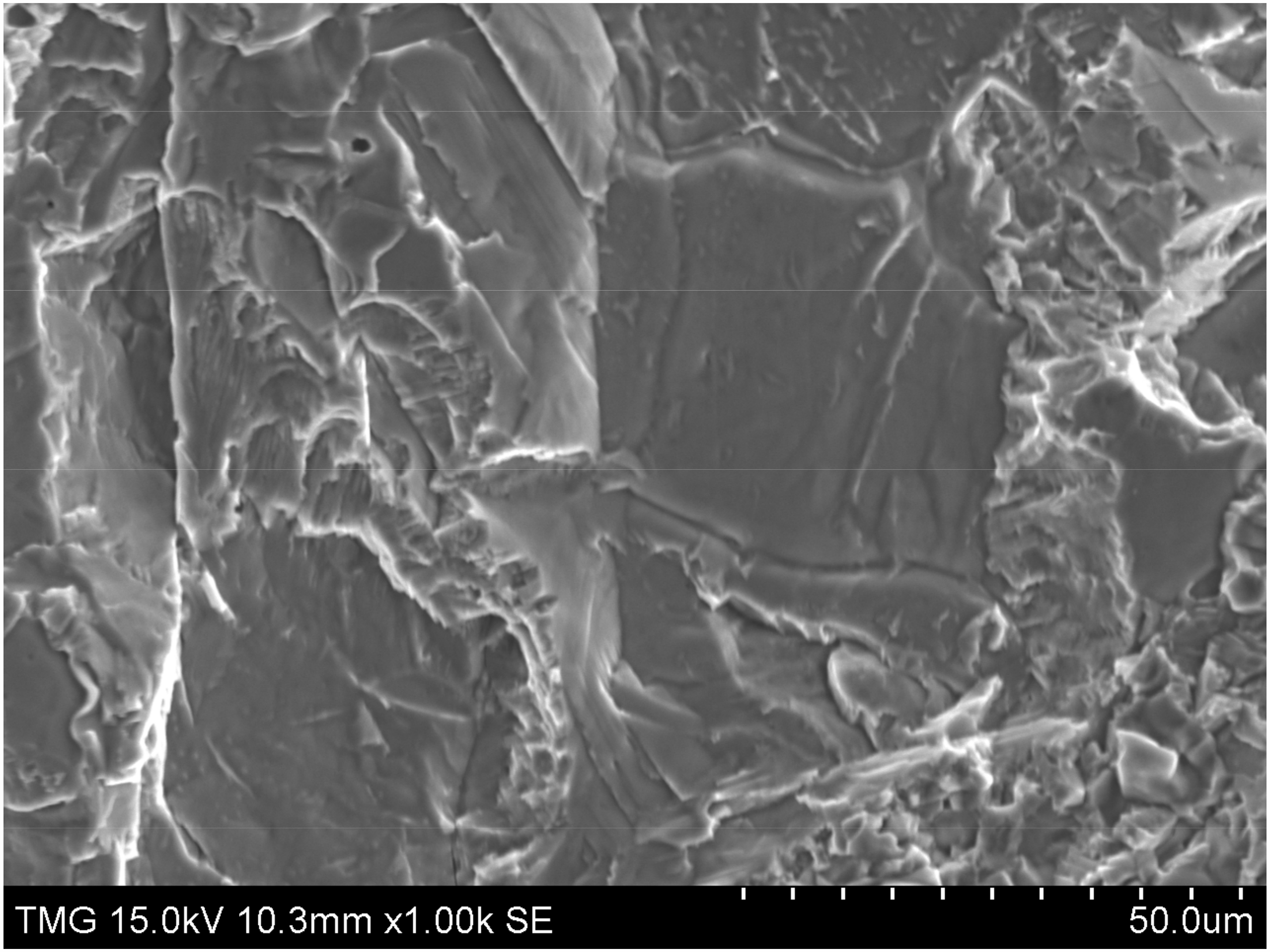
# $TiC-TiB_2/TiAl_3$ In-situ Coatings

## Edge of the stream – Erosion of the coating



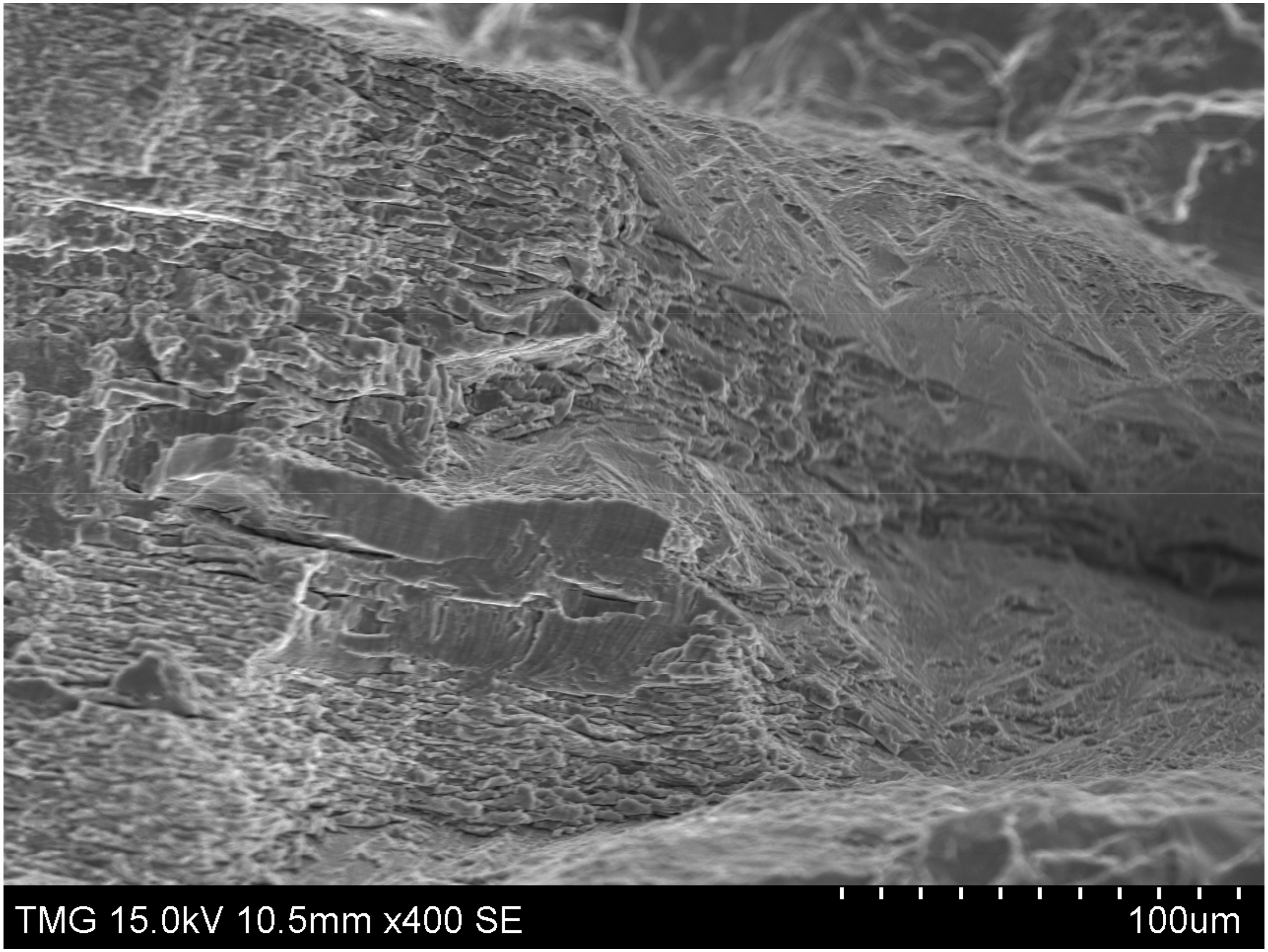
# TiC-TiB$_2$/TiAl$_3$ In-situ Coatings

## Center of the stream – Erosion of the substrate



# $TiC\text{-}TiB_2/TiAl_3$ In-situ Coatings

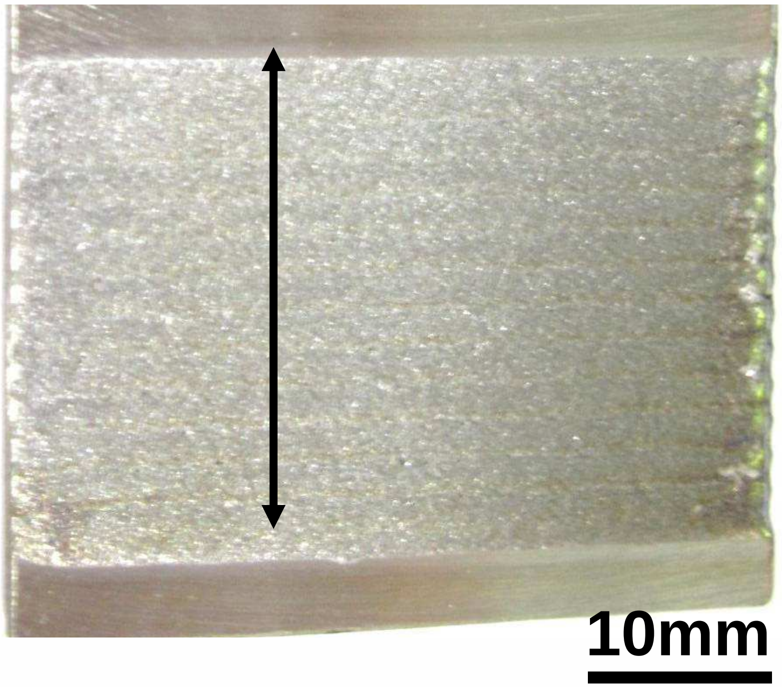
## Center of the stream – Erosion of the substrate

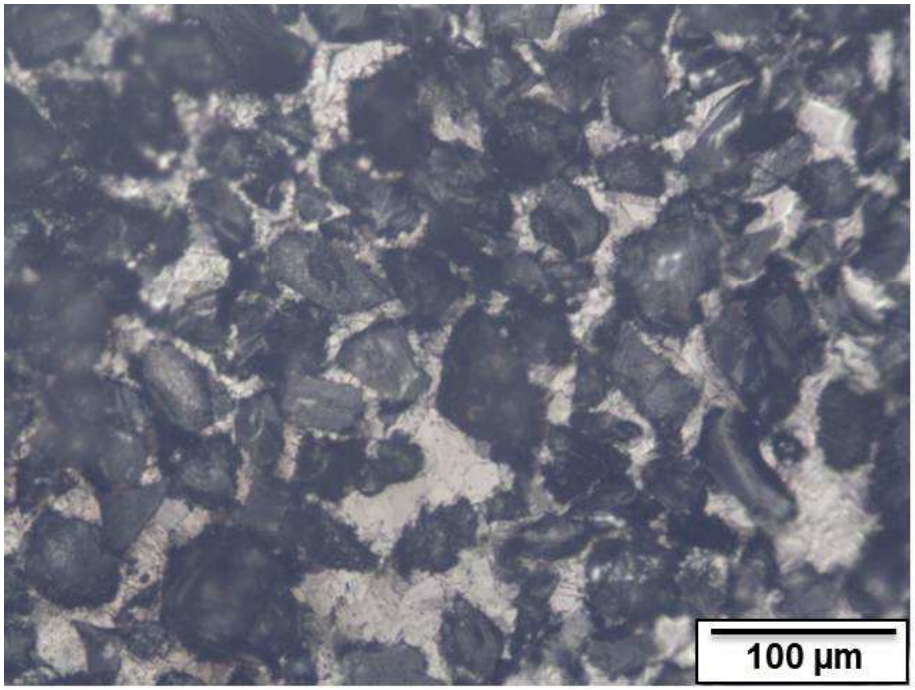


# Part IV

## Erosion Testing of $Al_2O_3$ Laser Cladding Coatings

# $Al_2O_3$ Laser Cladding Coating




**cladding surface**

***by Thuan***

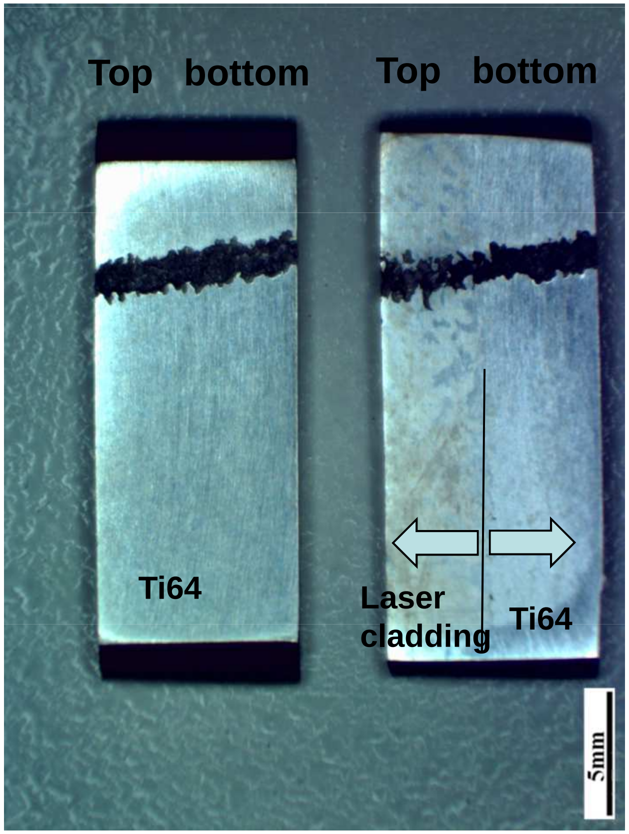
# $Al_2O_3$ Laser Cladding Coating

**Vacuum: 18-40 mBar**
**Speed: 14000 RPM**
**Time: 6 minutes**
**Nozzle size: 400 µm**
**Flow Rate: 0.03 L/min**

**Ti64 Uncoated**

**Weight Loss:**

**0.0135 g**




**$Al_2O_3$ Laser cladding coating**

**Weight Loss:**

**0.0095 g**

**70% weight loss for half cladding**

**~41% weight loss for full cladding**

# Thank you!

# Questions?