

Microstructural Characterization of Semi Conductive Coatings on Ceramic Insulators for Improved High Voltage Hold off Performance

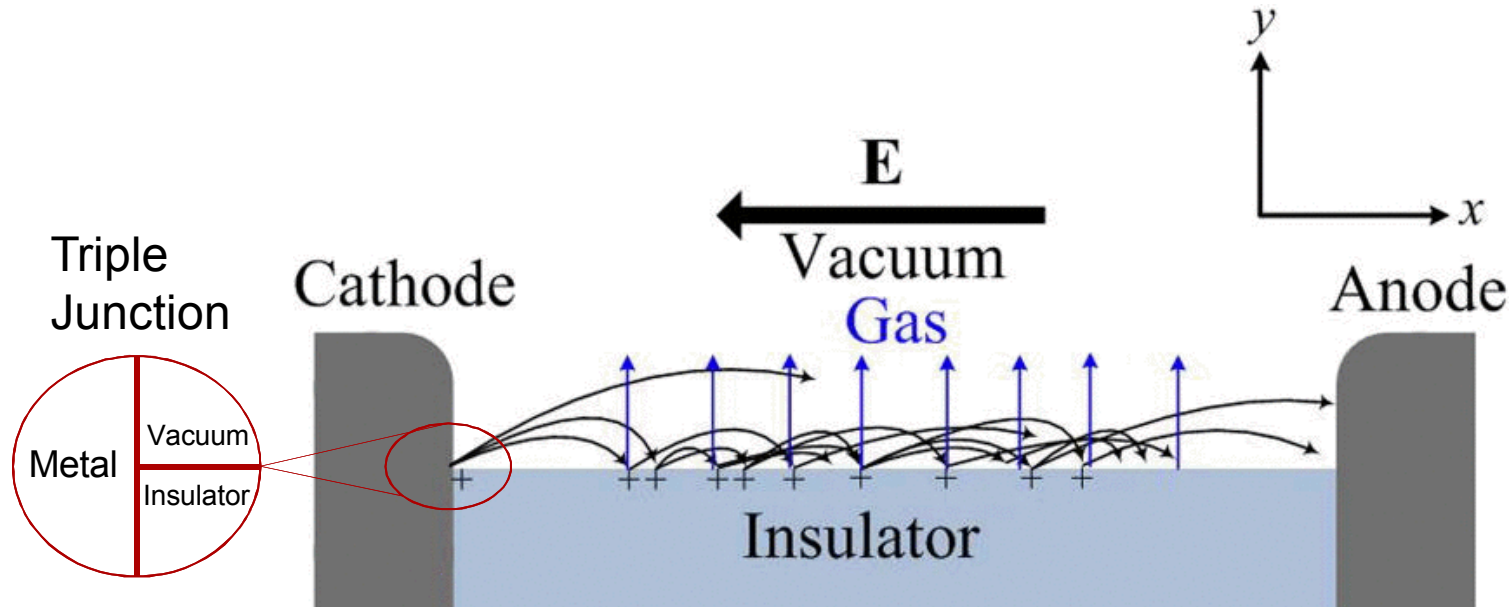
Sandra Stangebye

Brittany Muntifering, Elizabeth Paisley, Rajan Tandon

Outline

- Background
 - Characteristics of solid insulators in vacuum
- Characteristics of bulk ceramic
- Procedure
- Ti/Mn coating microstructure analysis
 - Optical gradation
 - SEM, EDS, and TEM data
- Ti/Cr coating microstructure analysis
- Compare Ti/Mn and Ti/Cr coatings
- Preliminary electrical property results and future work

High Voltage Performance of Solid Insulators

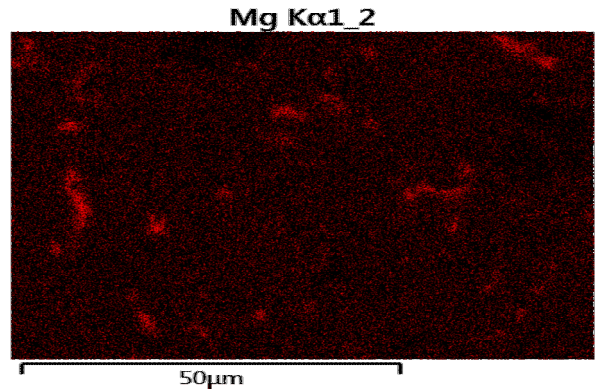
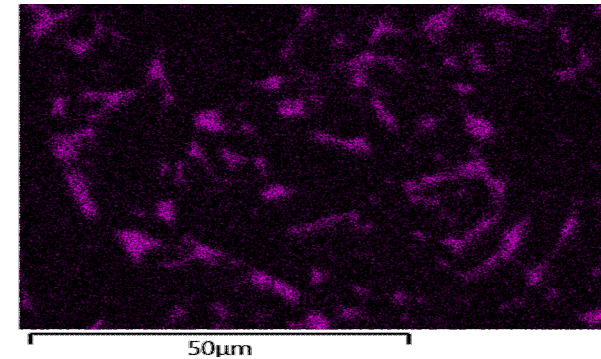
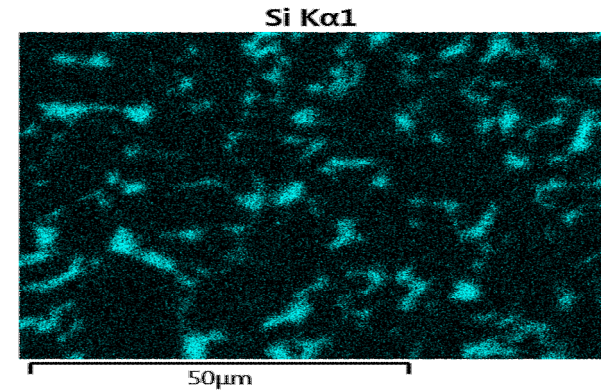
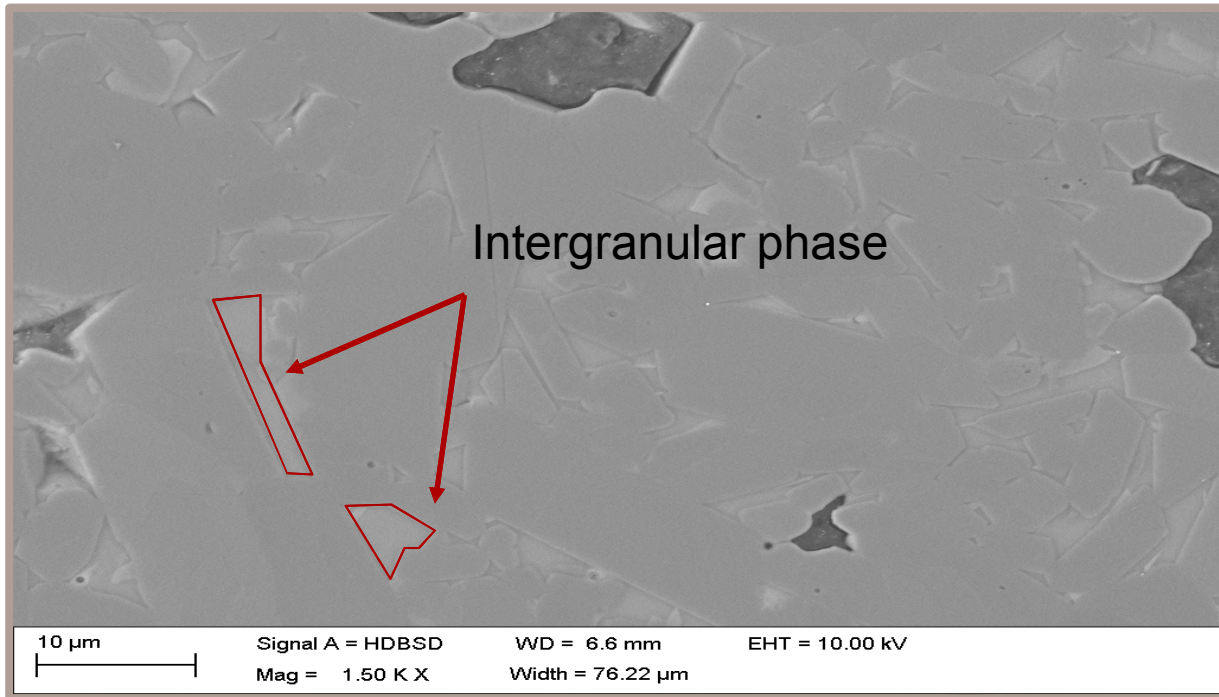


- Secondary electron emission (SEE) leads to voltage breakdown
- Positive feedback loop can cause continuation of SEE

Goals

- Decrease secondary electron emission
- Increase surface electrical uniformity
- Decrease surface resistivity

Bulk Ceramic Insulator



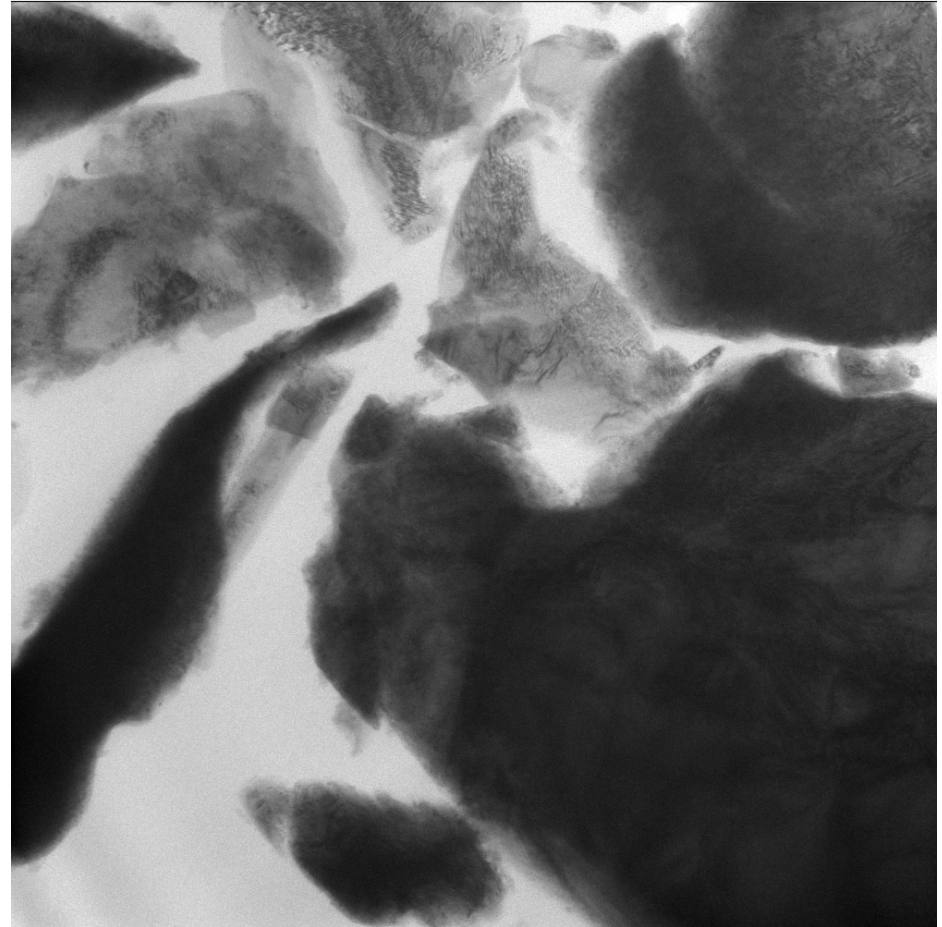
Bulk: 94% Alumina (Al_2O_3)

Intergranular Phases:

Primarily SiO_2 and CaO (some MgO and Cr_2O_3)

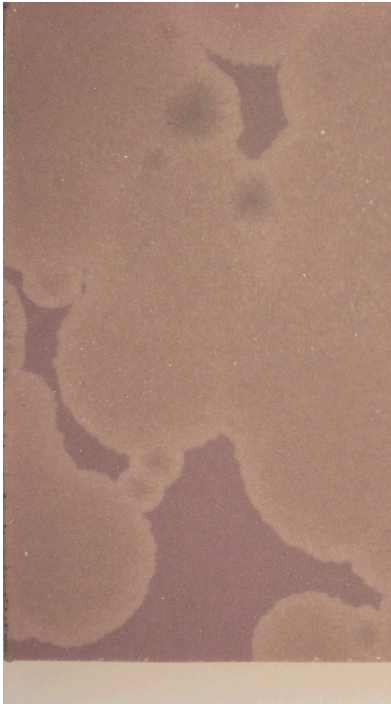
Procedure

- Ti/Mn and Mn/Cr powders
- Powders in solvent painted onto ceramic and fired at temperatures above 1300 C
- Compared Ti/Cr and Ti/Mn coatings

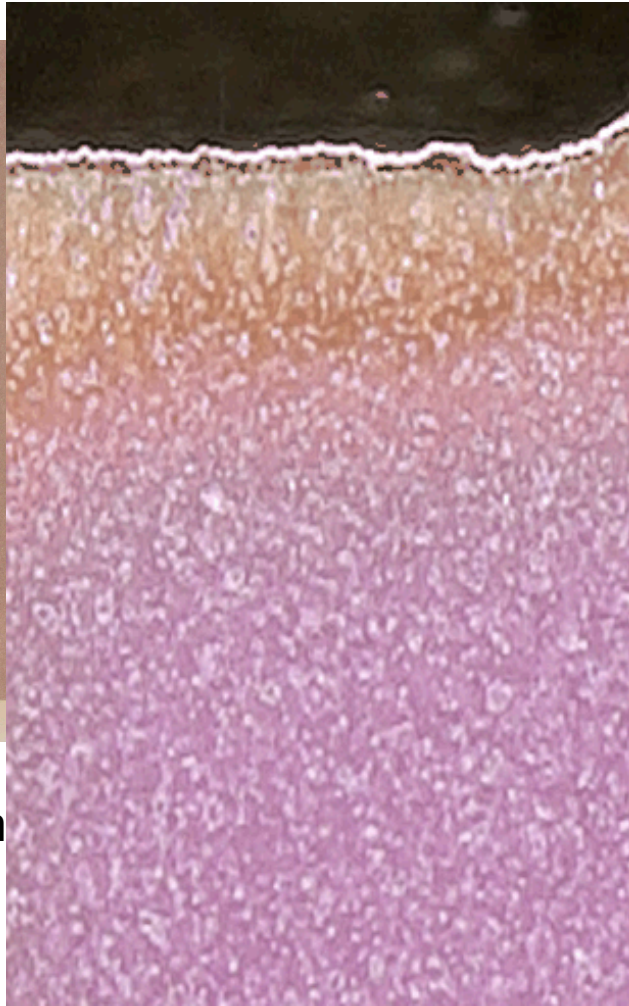


TEM Images of semi-conductive powder

Ti/Mn Coating



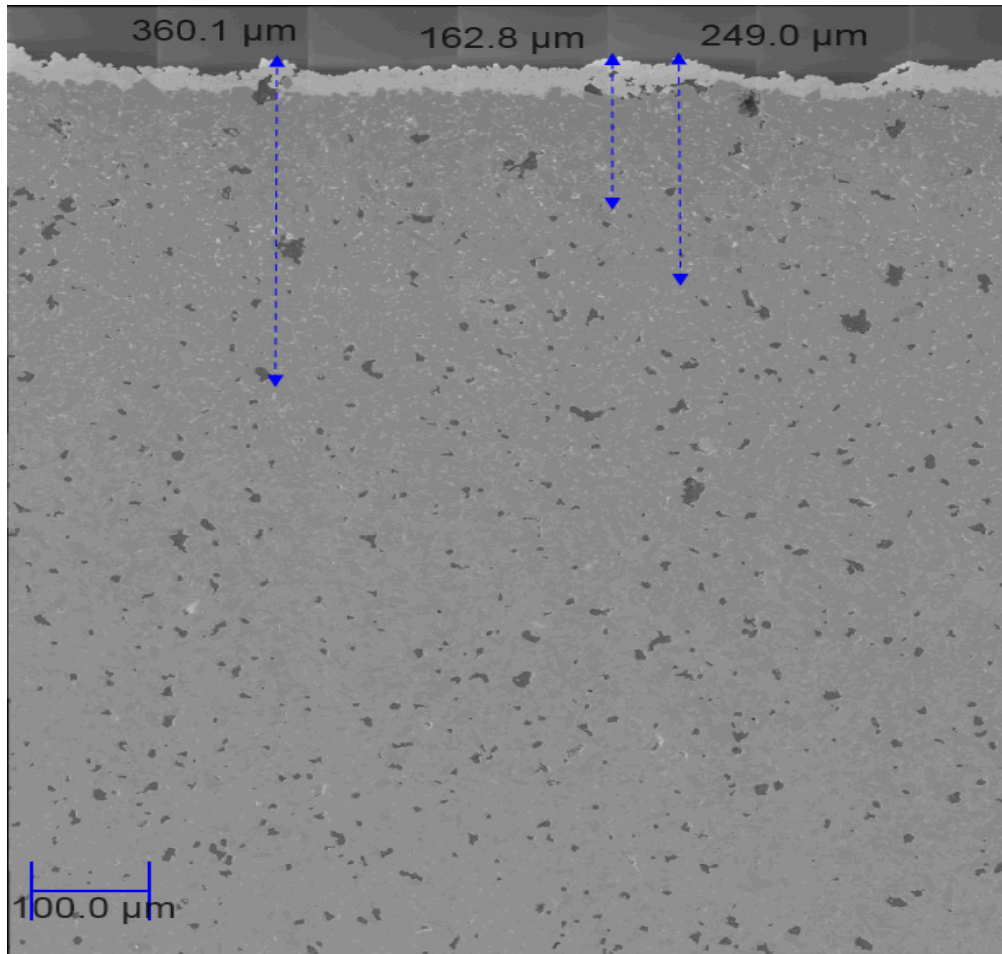
Top view of Alum



5 colors observed



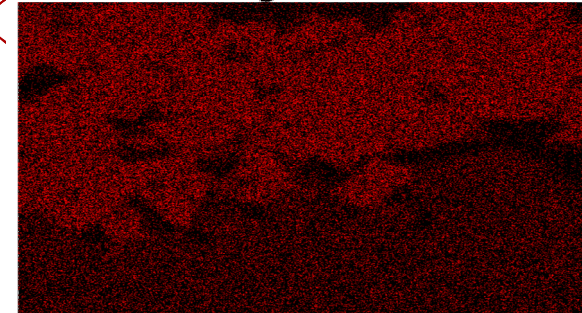
SEM Cross Section



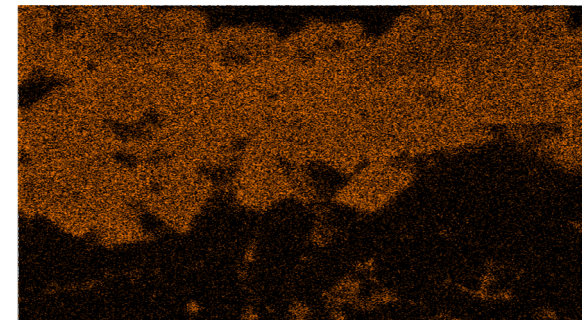
Top Layer

- Mg has diffused to surface where it interacts with MnAl_2O_4 leaving denuded zone
- Al has also diffused to surface to form a $(\text{Mn},\text{Mg})\text{Al}_2\text{O}_4$ spinel structure.
- Ti is align with Ca and Si phases

Mg K α 1_2

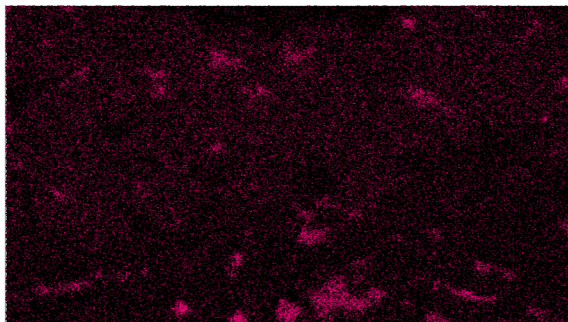


Mn K α 1



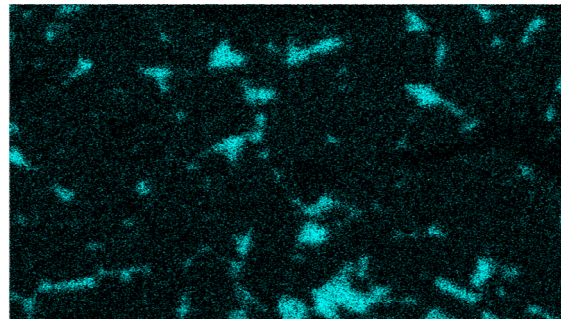
50 μm

Ti K α 1



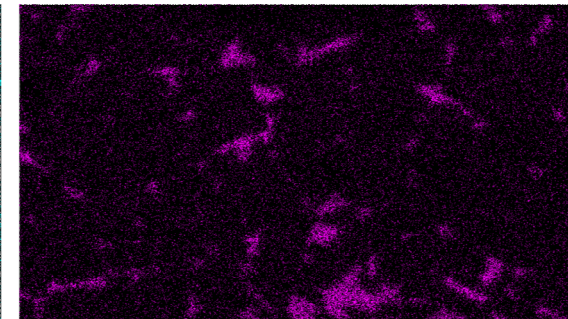
50 μm

Si K α 1



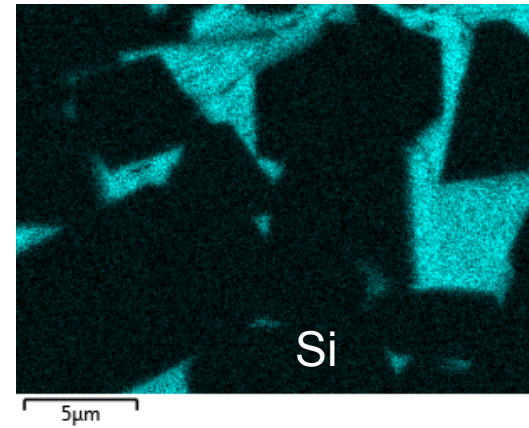
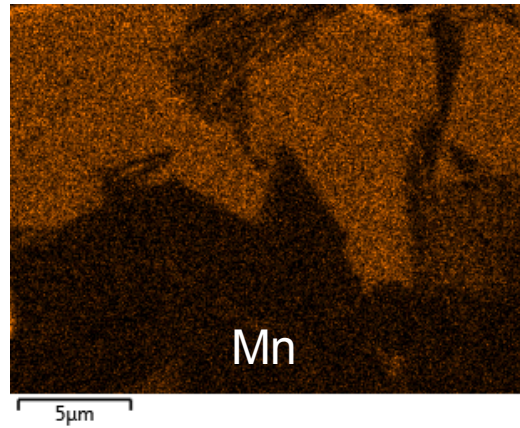
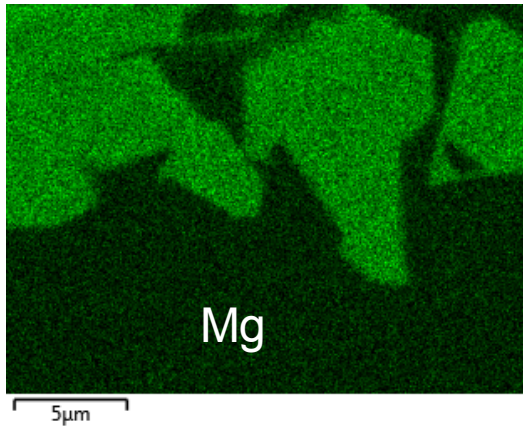
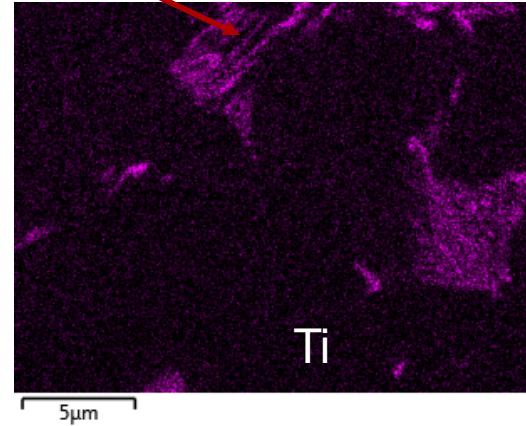
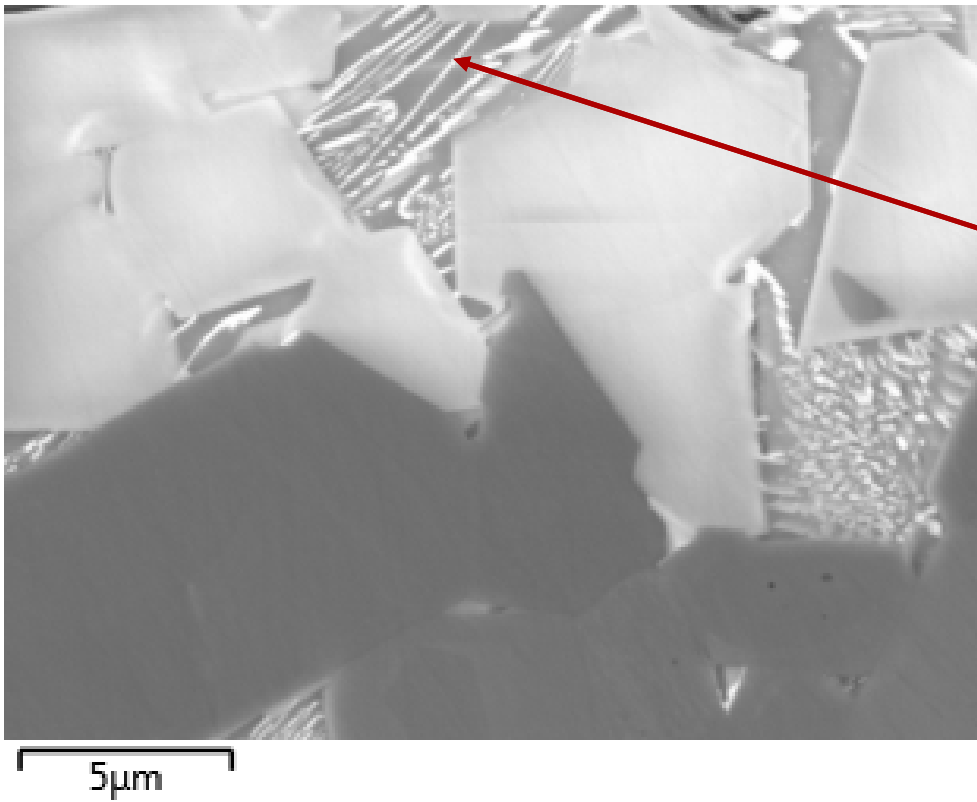
50 μm

Ca K α 1

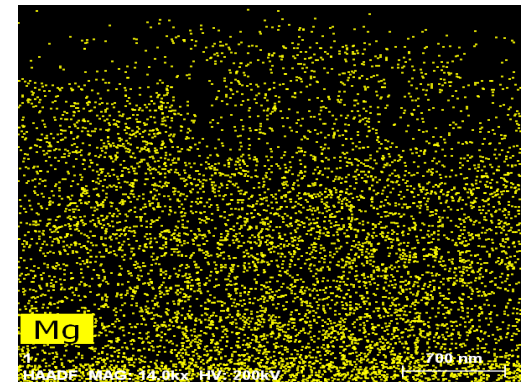
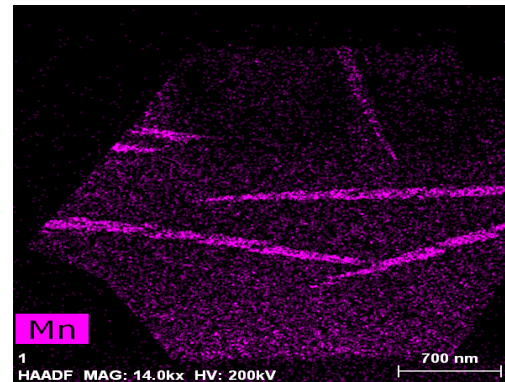
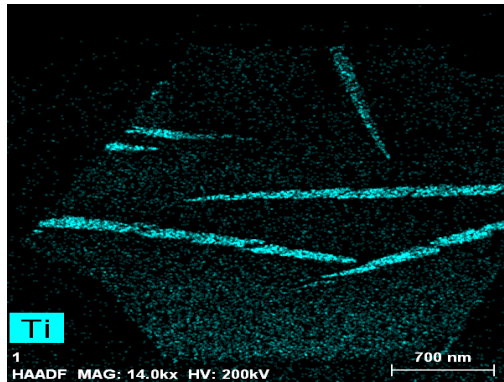
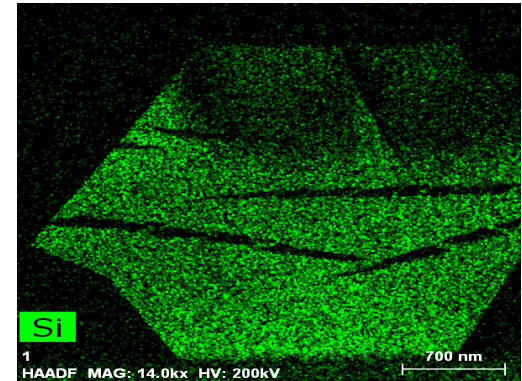
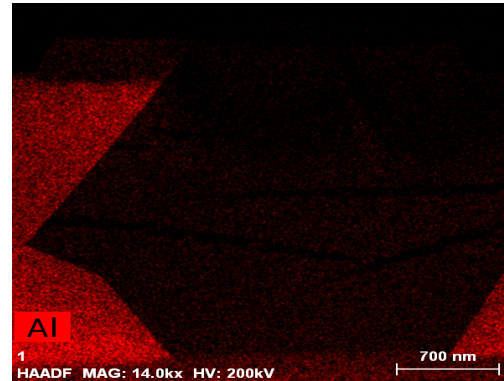
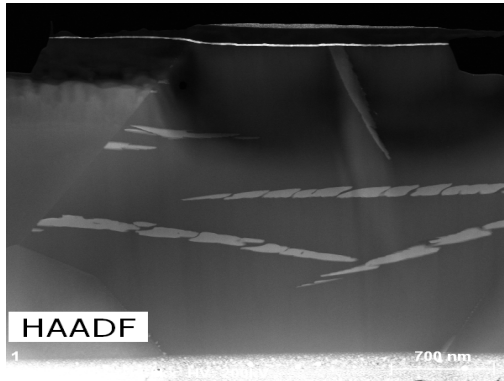


50 μm

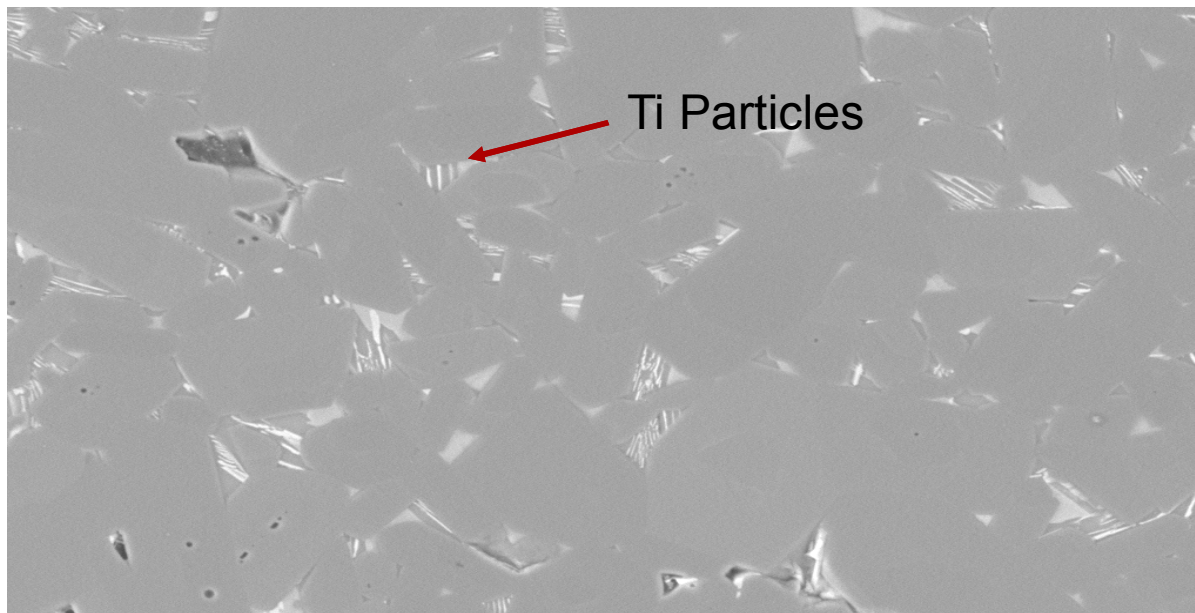
Higher mag shows
Ti/Mn rich precipitates
in intergranular phase



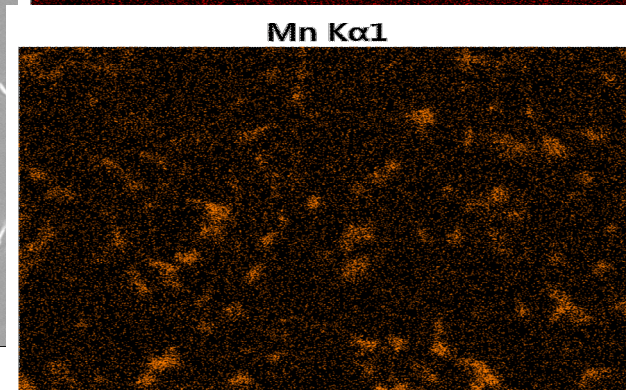
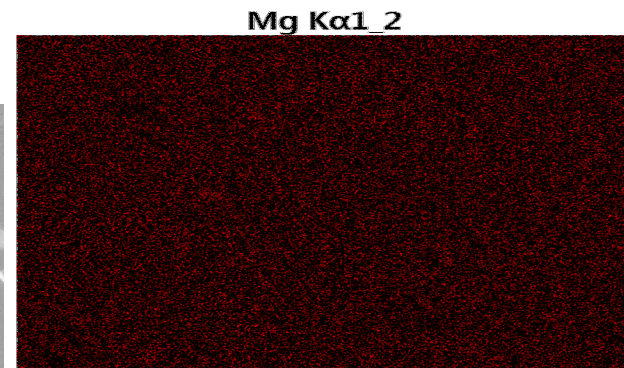
TEM Analysis of Ti rich precipitates



“Light-Orange” Layer



10 μ m Signal A = HDBSD WD = 6.6 mm EHT = 10.00 kV
Mag = 1.50 K X Width = 76.22 μ m

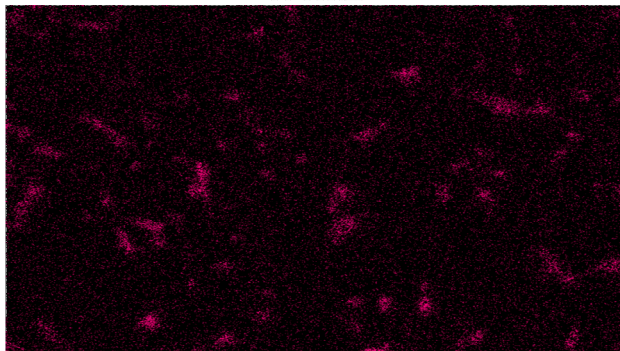


50 μ m

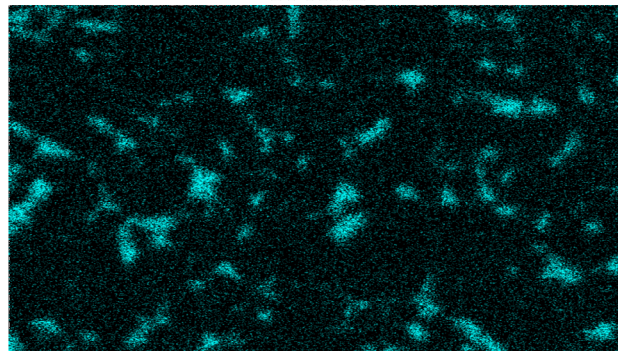
Ti K α 1

Si K α 1

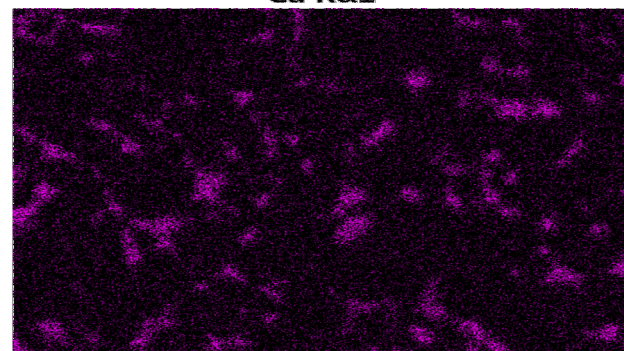
Sr K α 1



50 μ m

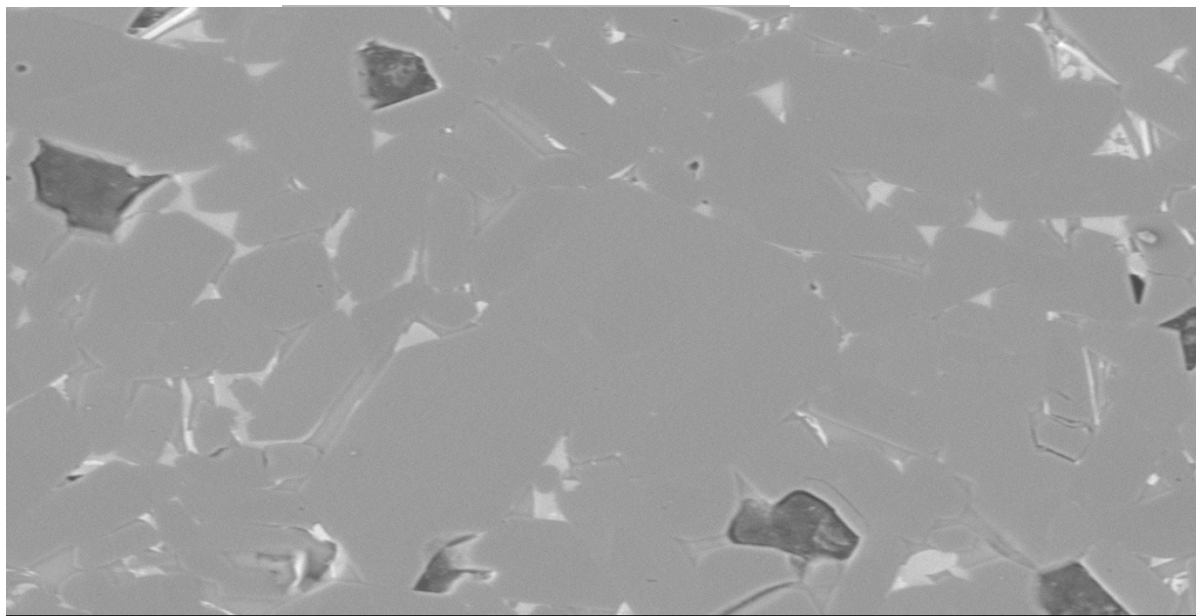


50 μ m



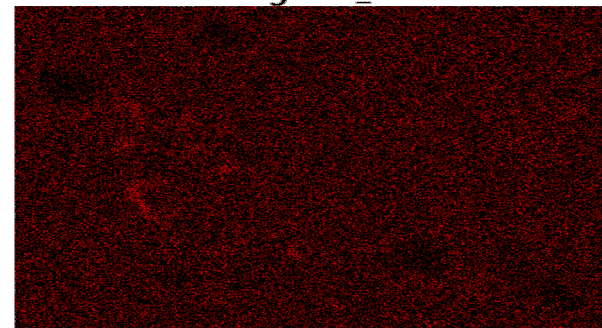
50 μ m

“Brown” Layer

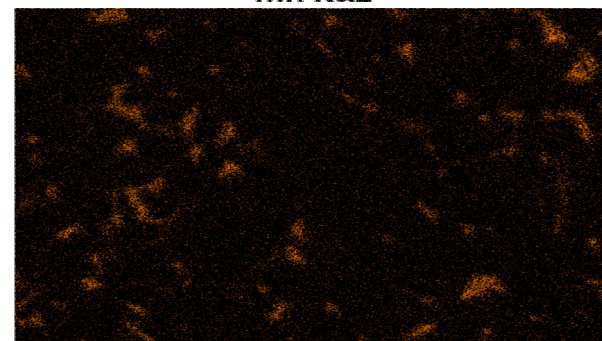


10 μ m Signal A = HDBSD WD = 6.5 mm EHT = 10.00 kV
Mag = 1.50 K X Width = 76.22 μ m

Mg K α 1_2



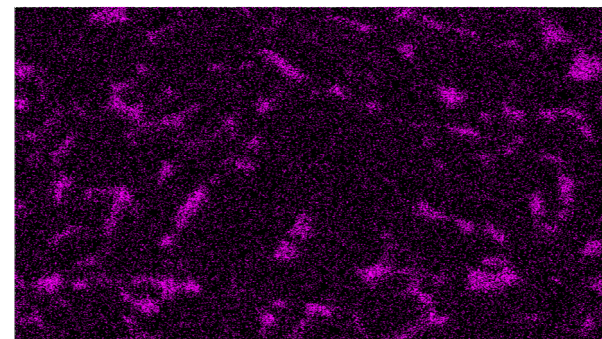
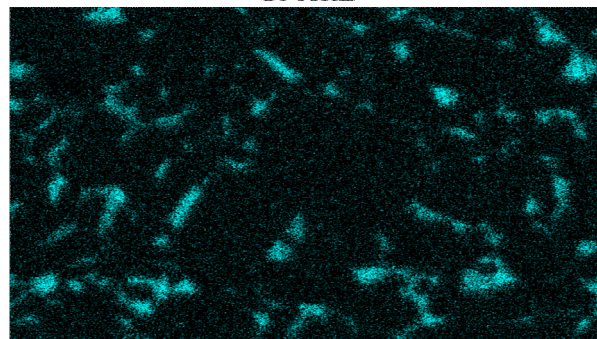
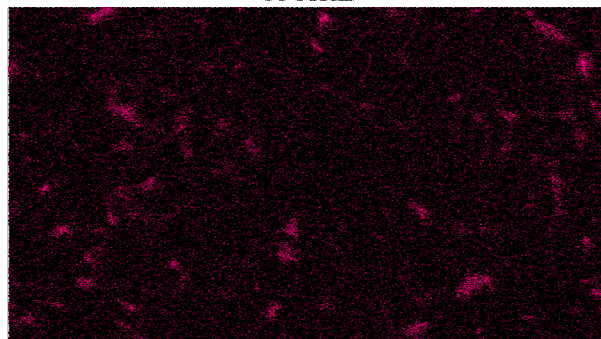
Mn K α 1



50 μ m

Ti K α 1

Si K α 1



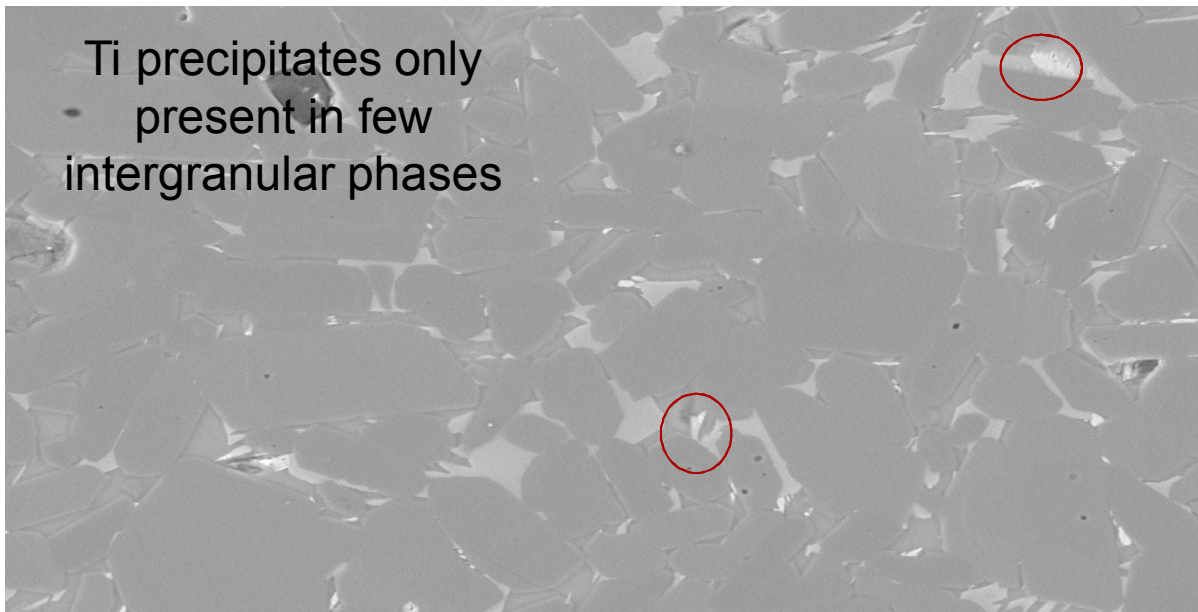
50 μ m

50 μ m

50 μ m

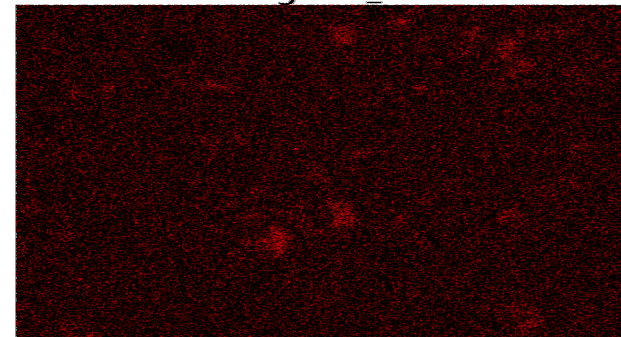
“Light Pink” Layer

Ti precipitates only present in few intergranular phases

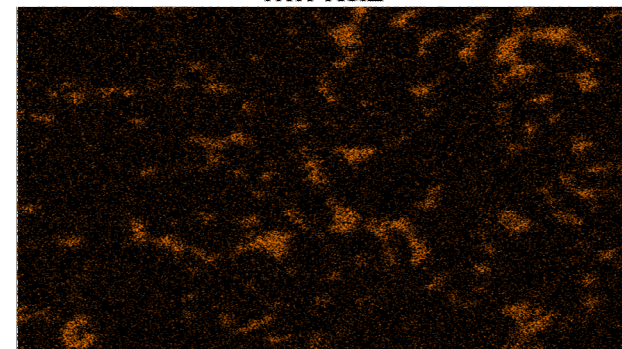


10 μm | Signal A = HDBSD | WD = 6.6 mm | EHT = 10.00 kV
Mag = 1.50 K X | Width = 76.22 μm

Mg K α 1_2



Mn K α 1

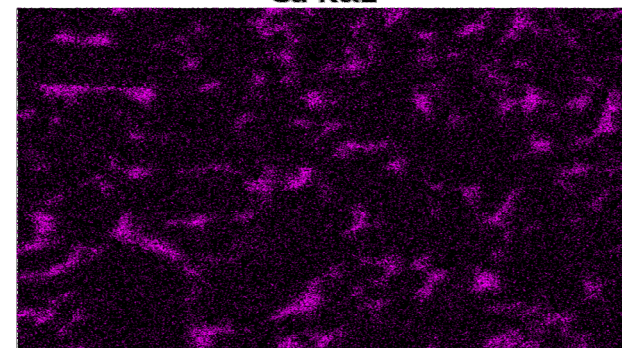
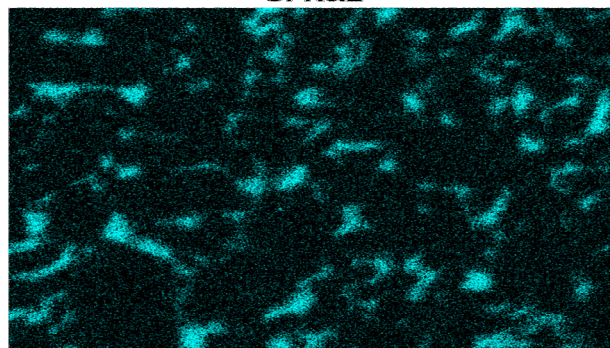
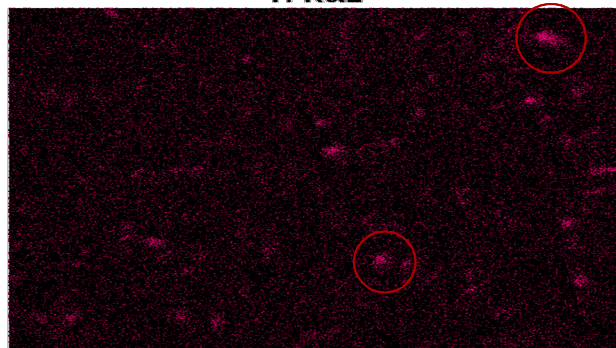


50 μm

Ti K α 1

Si K α 1

Sr K α 1

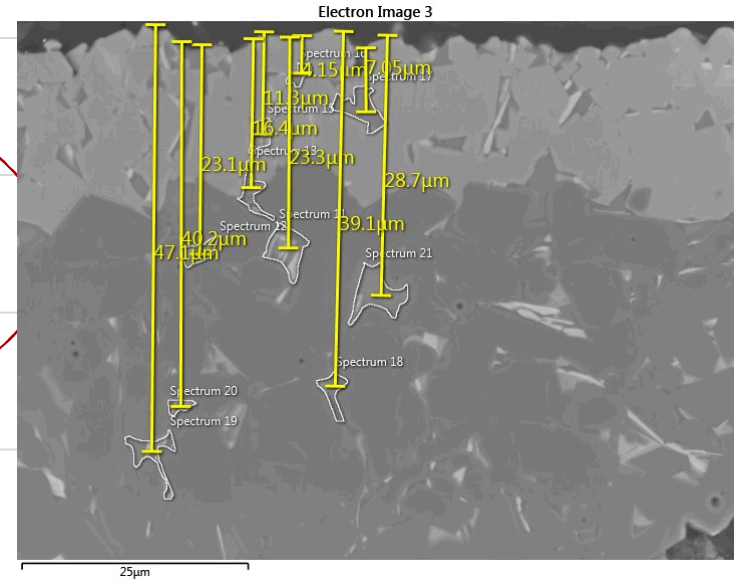
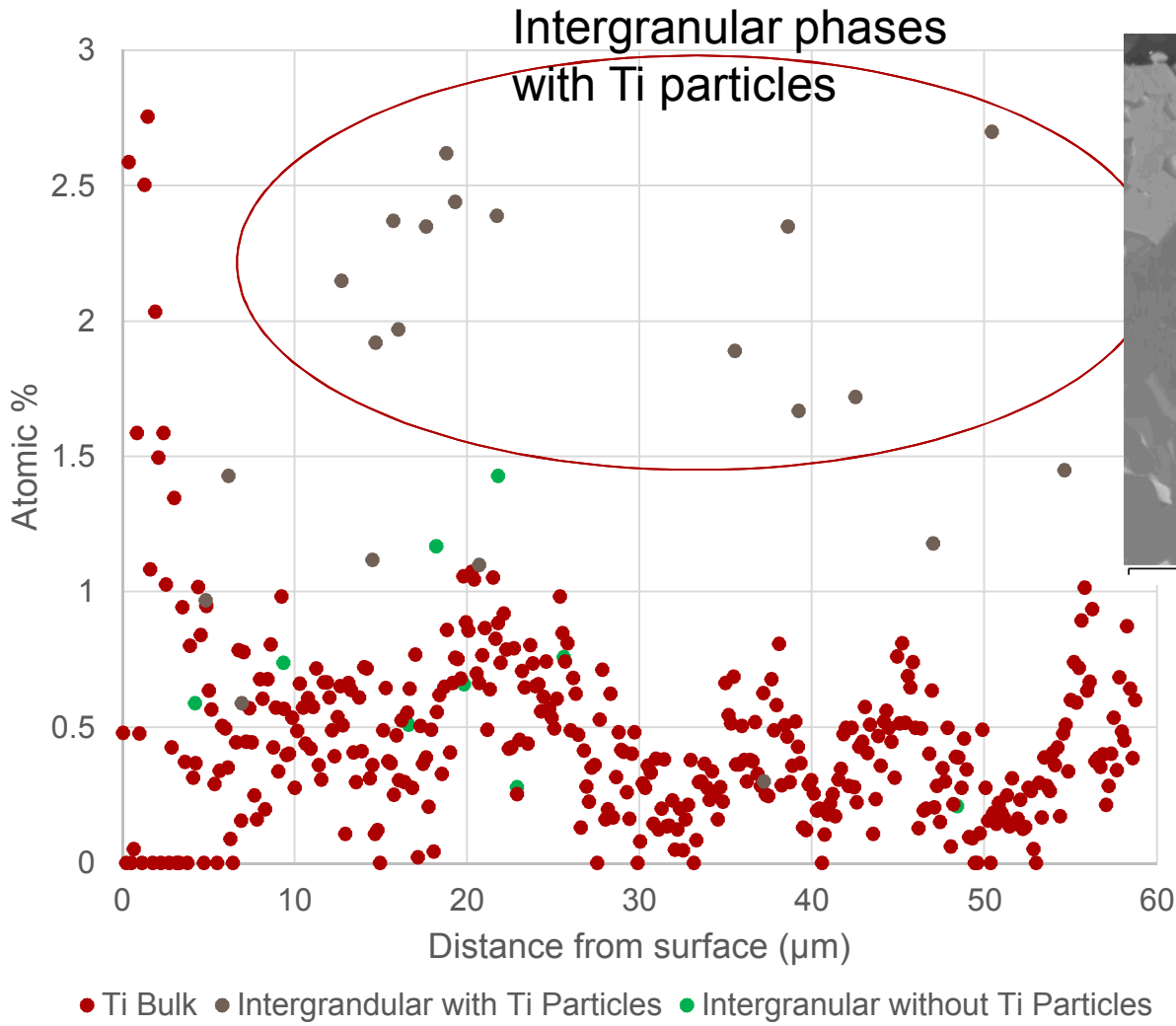


50 μm

50 μm

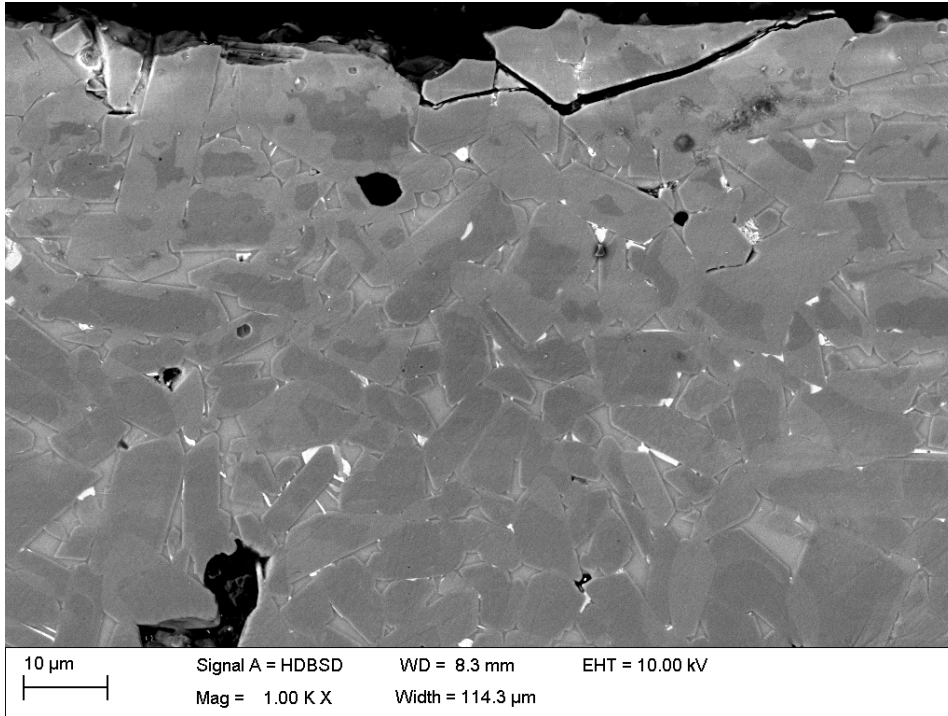
50 μm

Ti Atomic %

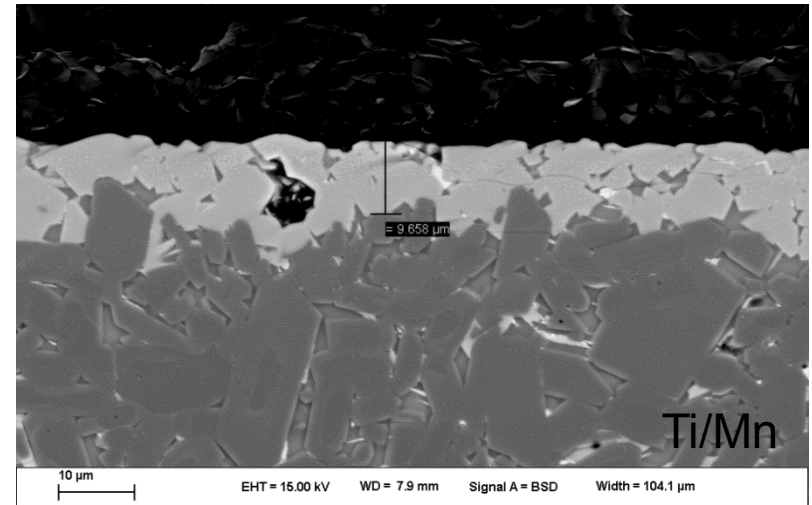


	Atomic %
Bulk	0.46% \pm 0.34
Intergranular without Ti particles	0.71% \pm 0.36
Intergranular with Ti particles	1.75% \pm 0.66

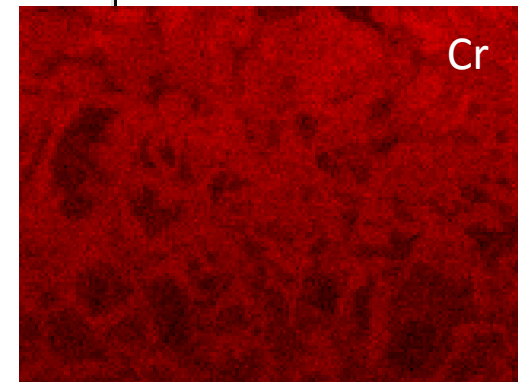
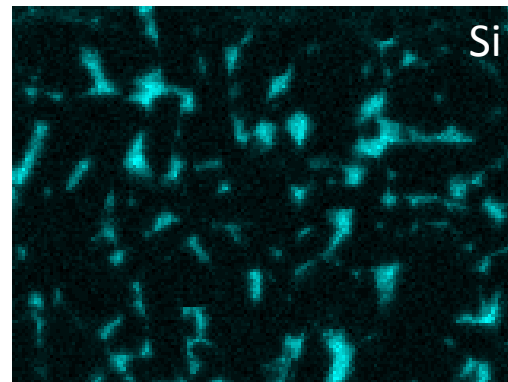
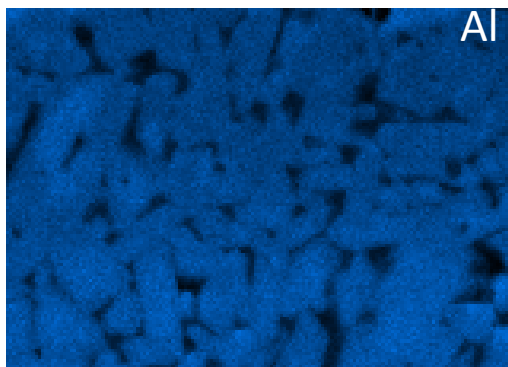
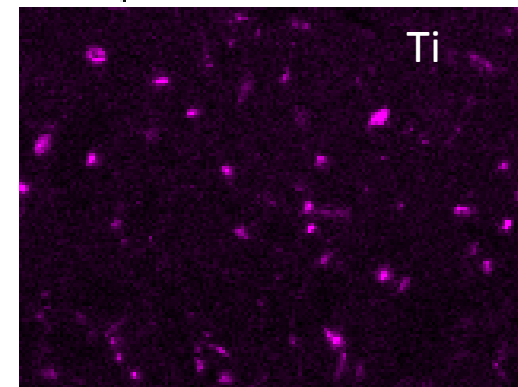
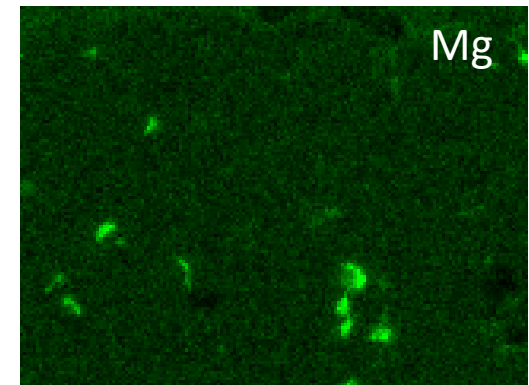
Ti/Cr Coating



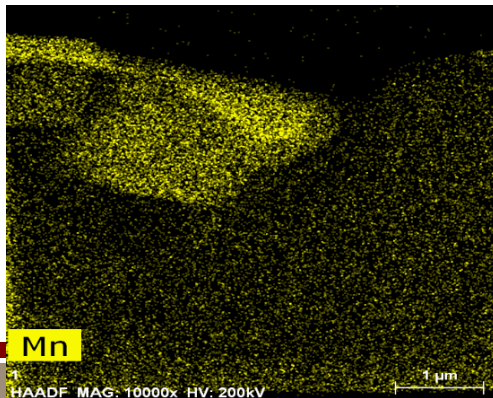
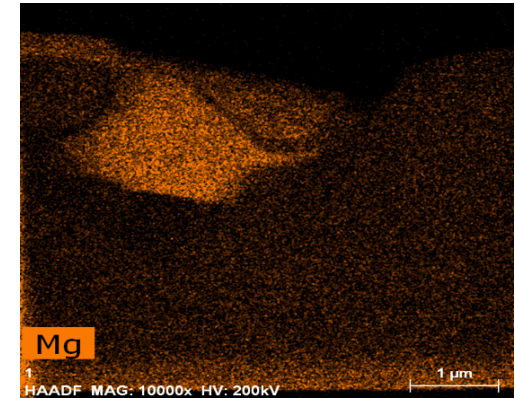
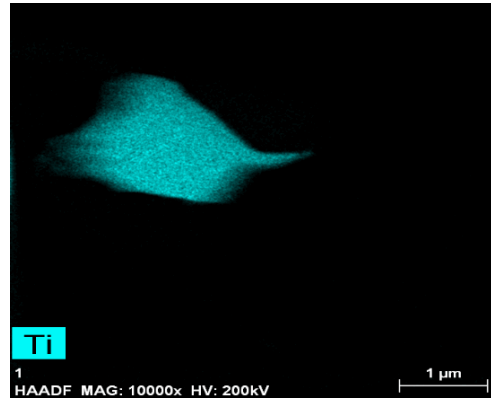
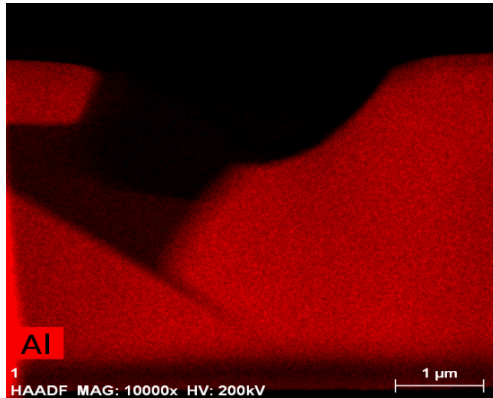
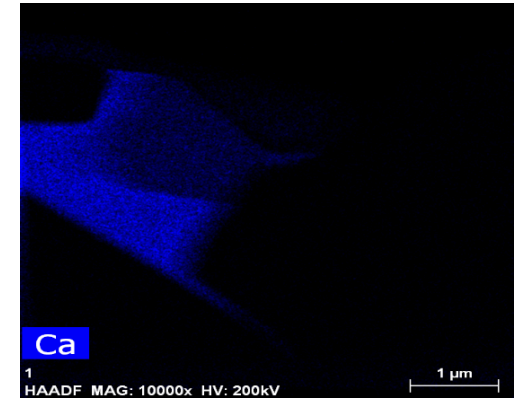
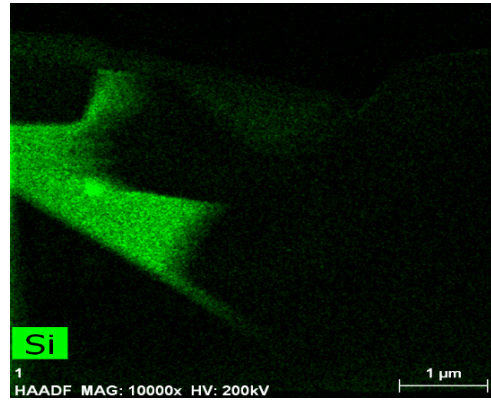
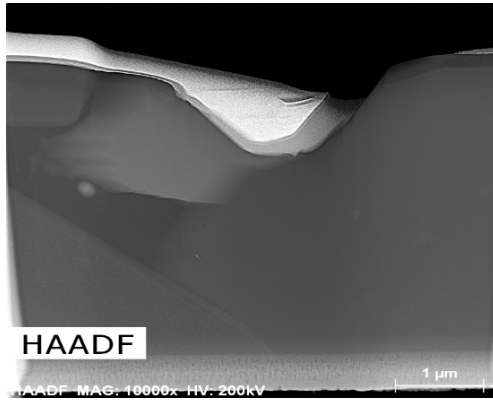
} No visible reaction layer



- Mg does not segregate to surface but instead into intergranular phases
- Ti segregation in intergranular phases is not the same as Mg
- Cr substitutes for Al
- Core-shell type grains

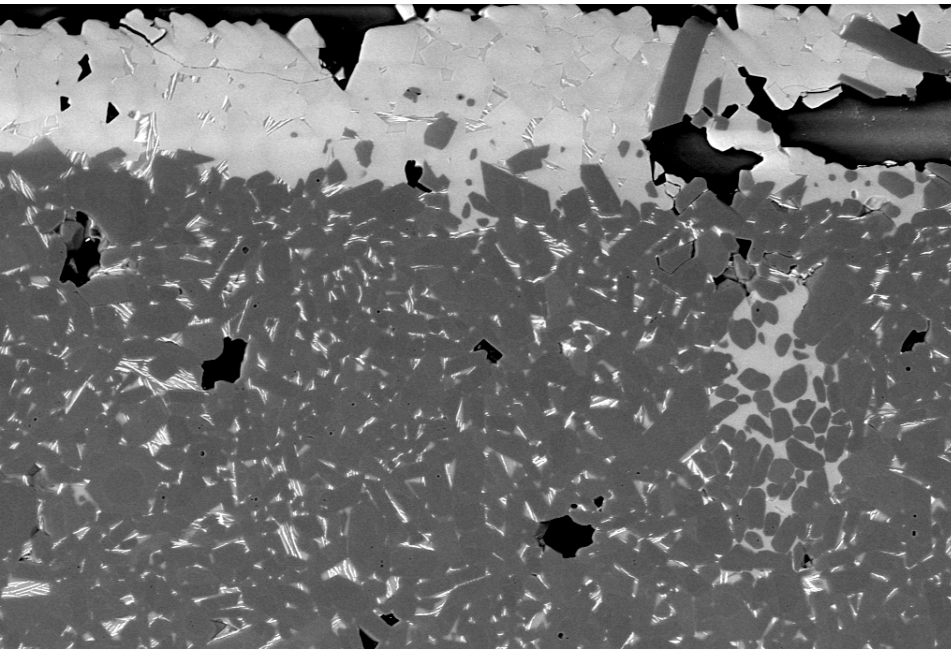


TEM of Ti rich phase



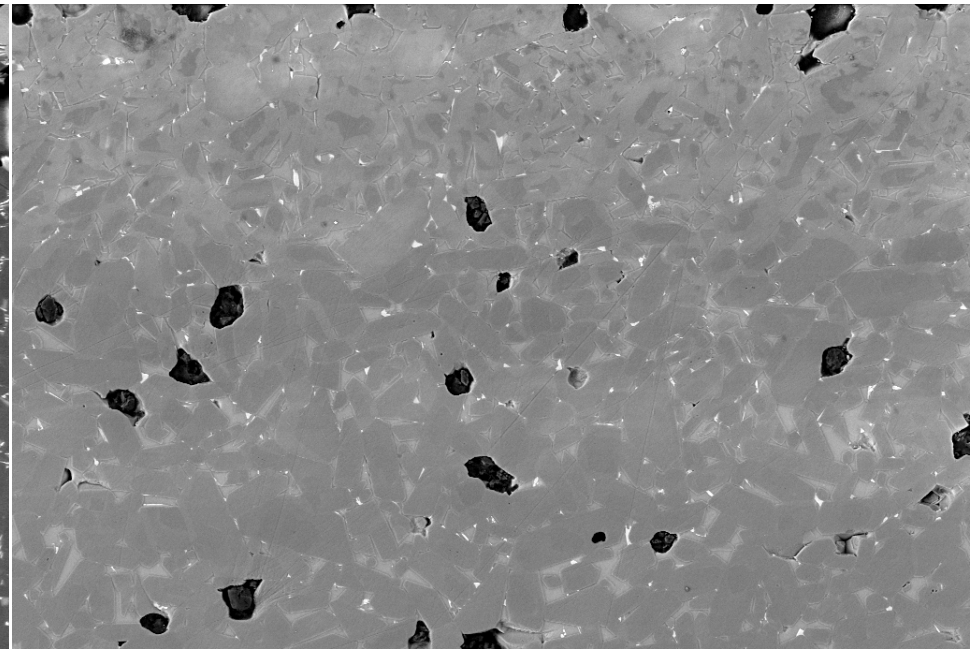
Comparison of Microstructure

Ti/Mn



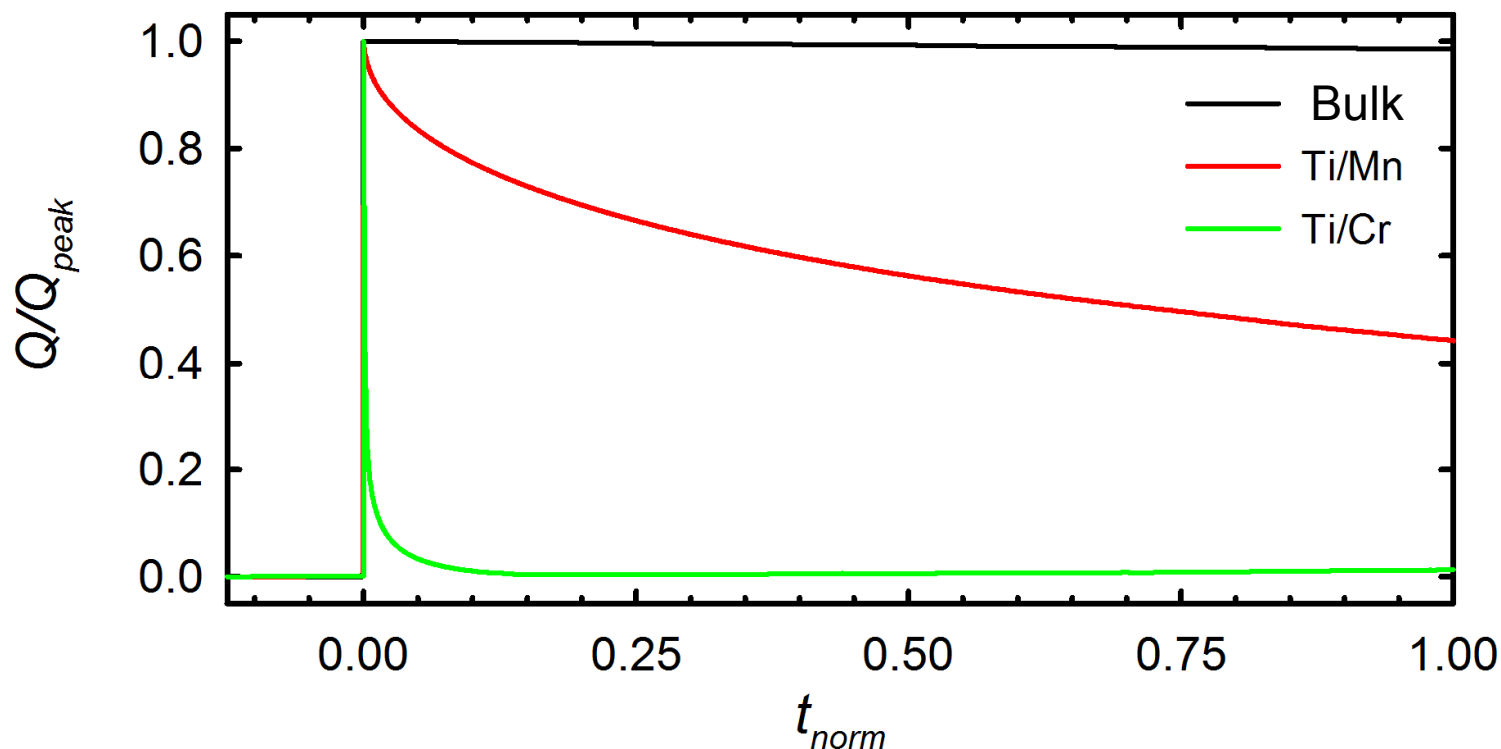
20 μm | Signal A = HDBSD | WD = 8.5 mm | EHT = 10.00 kV
Mag = 500 X | Width = 228.7 μm

Ti/Cr



20 μm | Signal A = HDBSD | WD = 8.3 mm | EHT = 10.00 kV
Mag = 500 X | Width = 228.7 μm

Charge bleed off comparison



- SEE measurements in progress with a collaboration with Princeton Plasma Physics Labs
- Goal to correlate resistivity, charge bleed off, and SEE with microstructure to optimize high voltage insulating performance