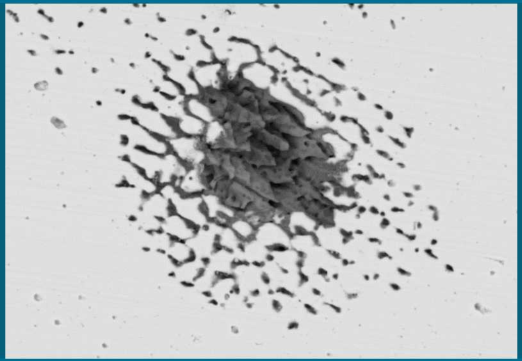
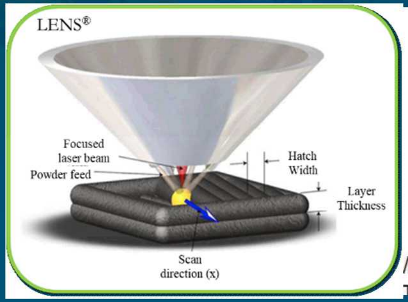
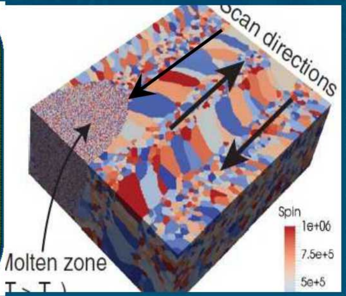
C05-1298

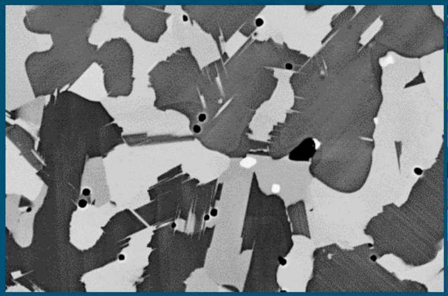
SAND2020-9897C

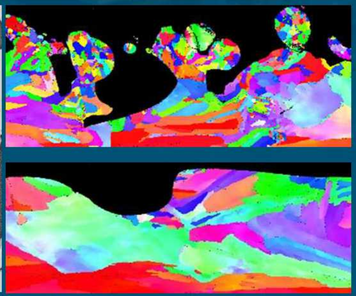
Additive Manufacturing to Compositionally Grade Metals for High Throughput Alloy Screening











Michael Melia (SNL)

PRESENTED BY

Michael Melia (SNL)

Co-authors: Jonathan Pegues, Morgan Jones, Brendan Nation, Nicolas Argibay, Andrew Kustas (SNL)

Kodi Summers and Dev Chidambaram (UN-Reno)

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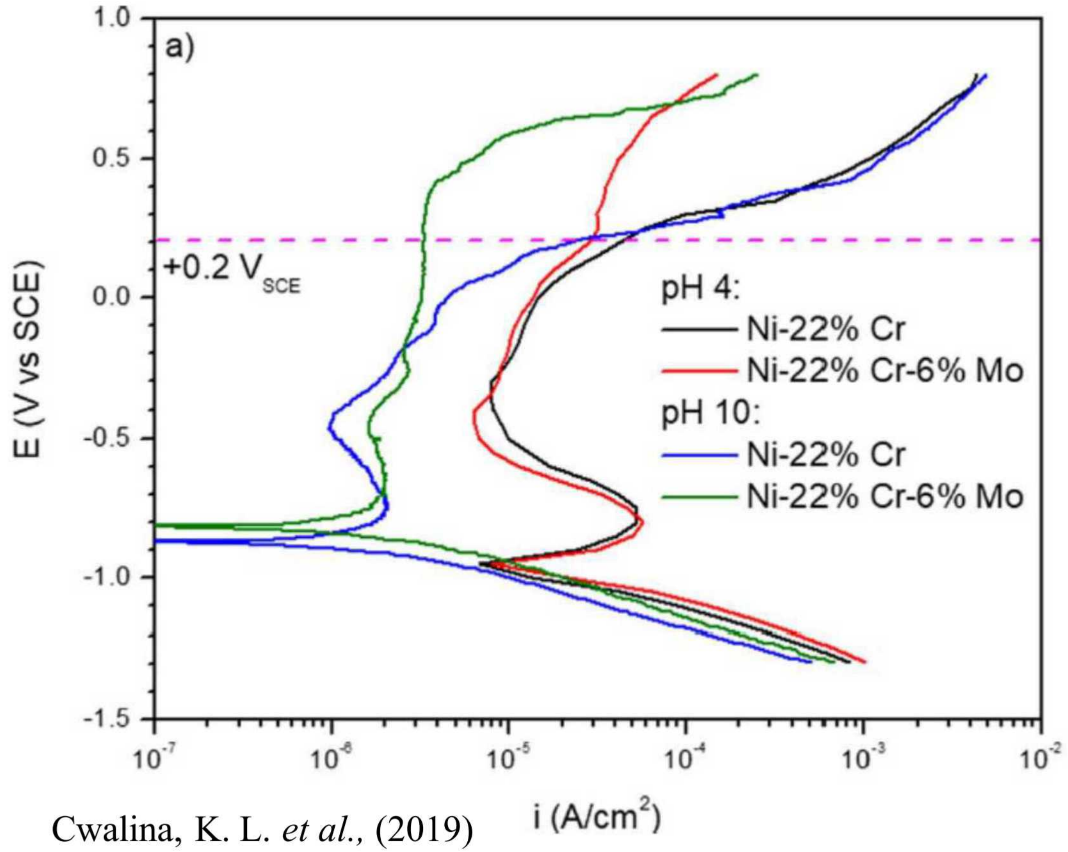
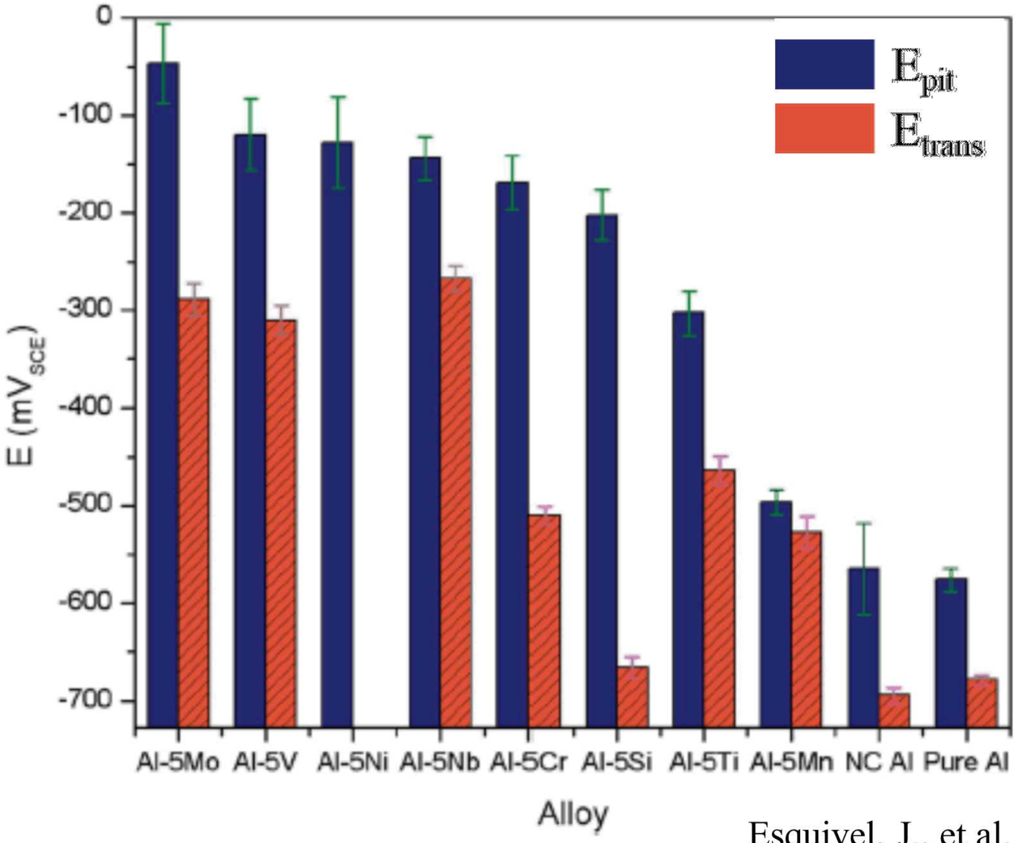
SAND2020-XXXXX

Alloying impact on passivity



Cwalina, K. L. *et al.,* (2019)



Esquivel, J., et al. (2018)

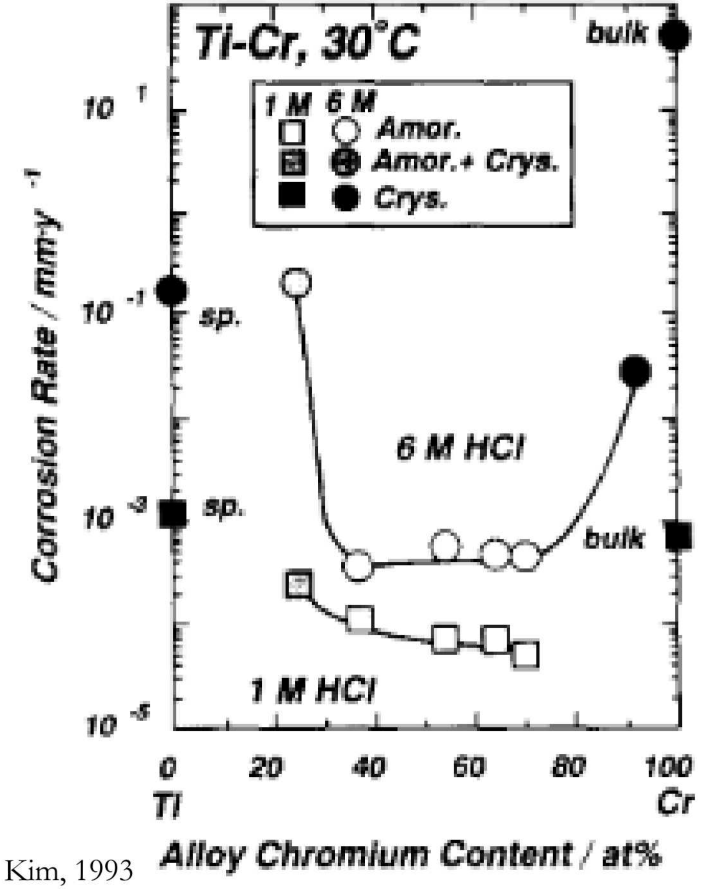
Passivity is heavily influenced by alloying elements, i.e. Cr/Mo additions to steel or Ni alloys.

Other passivity promoting elements (Ti, Mo, Nb, etc.) show beneficial effects when added to pure Al, assuming the elements are kept in solid solution.

Alloying impact on passivity – Cr-Ti system

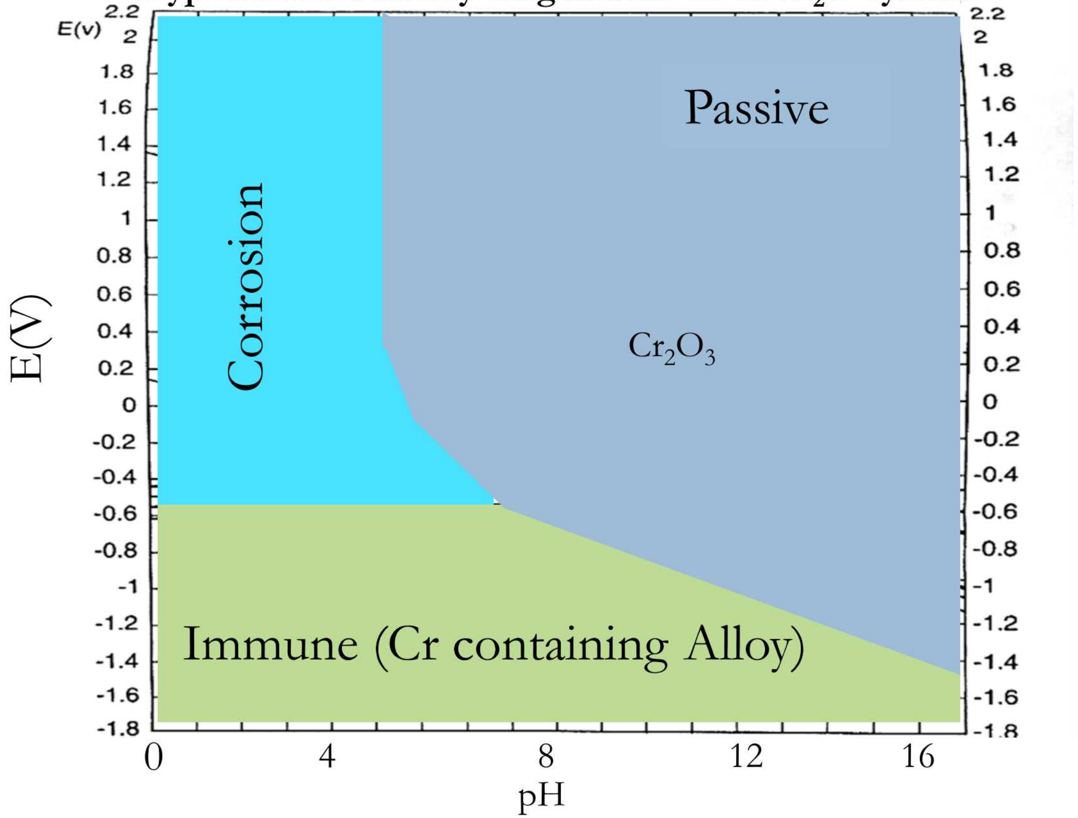
The combination of Ti and Cr has been shown to have synergistic effects for corrosion resistance/passivity across a range of aggressive environments (concentrated acids).

Concentrated HCl



J.H. Kim, 1993

Hypothetical stability diagram for Cr-Ti-H_2O system

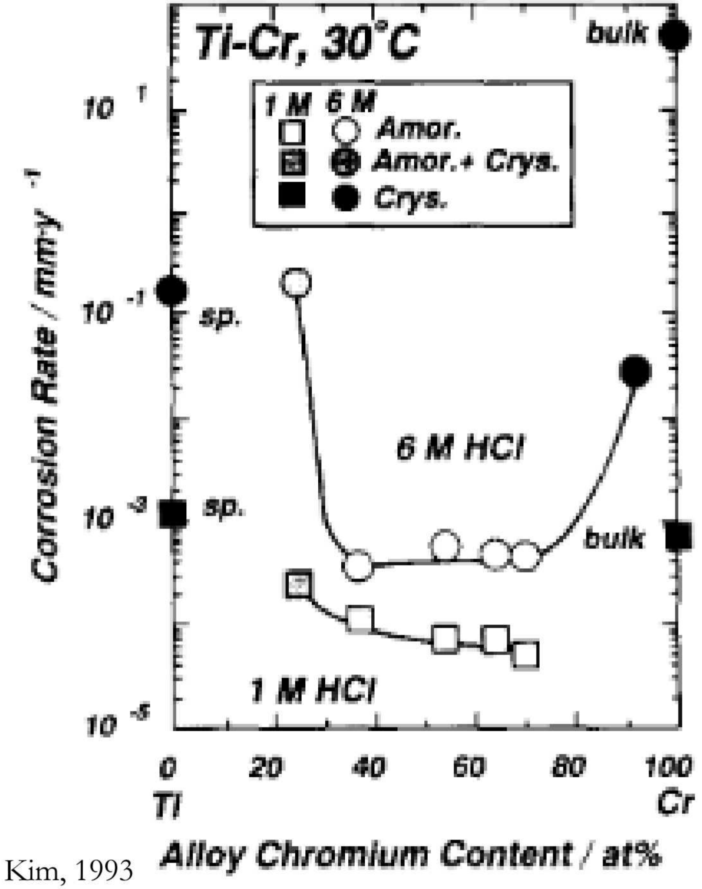


The underlying passivating mechanism is expected, for these Ti-Cr systems, to be caused by the formation of a homogeneous double oxy-hydroxide of Cr^{3+} and Ti^{4+}, stable across a larger pH and potential range than typically experienced by pure Ti or Cr.

M. Mehmood, 1999

Alloying impact on passivity – Cr-Ti system

Concentrated HCl



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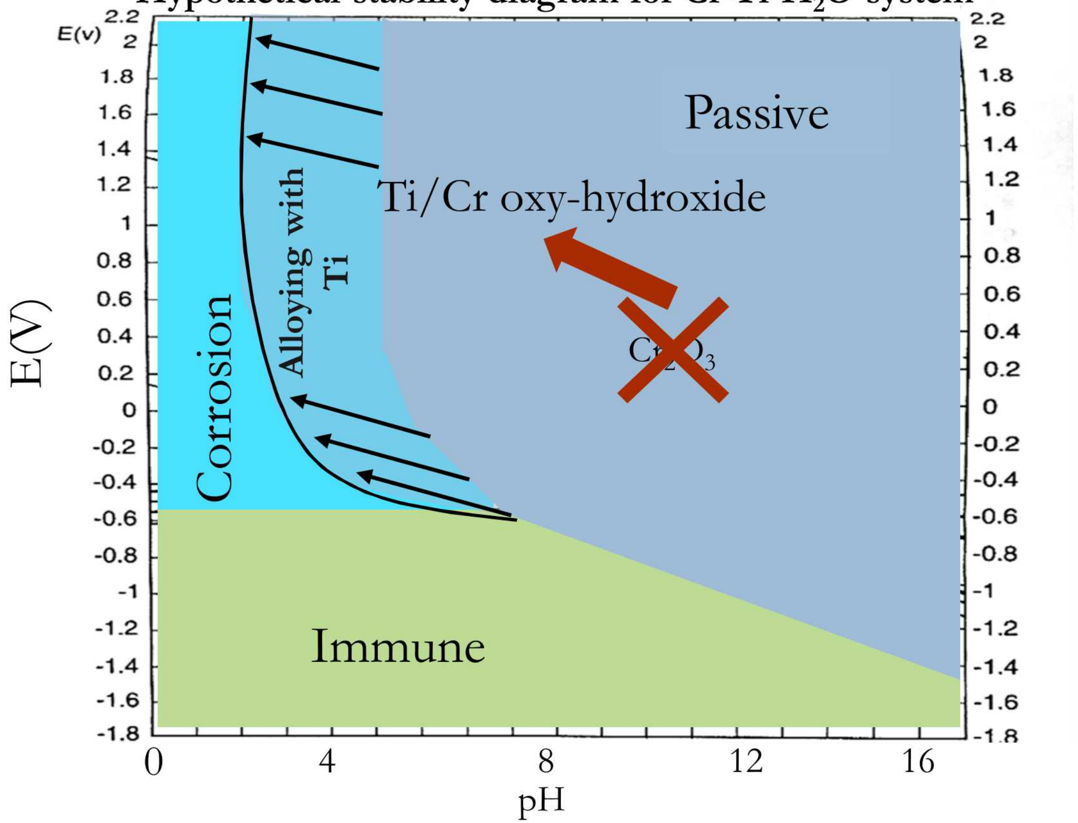

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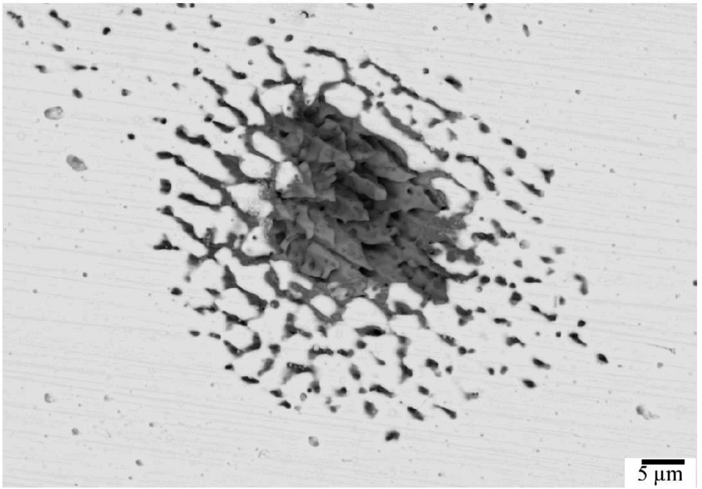
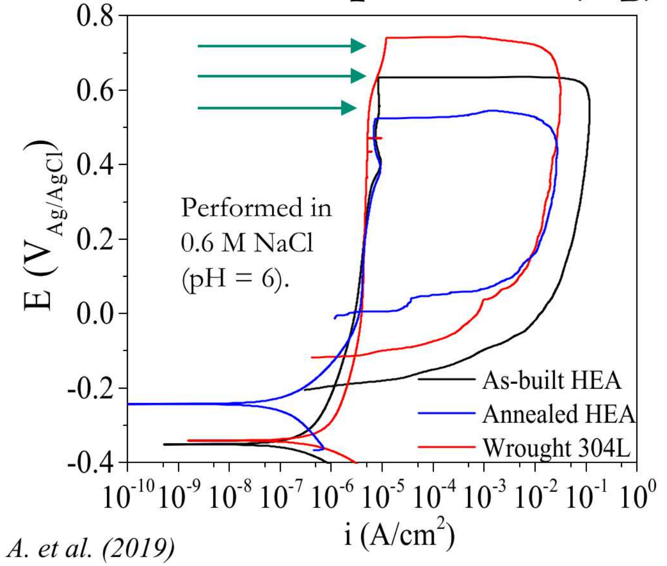
M. Mehmood, 1999

Corrosion of high entropy alloys (HEAs)

Pit morphology of CoCrFeMnNi HEA



Breakdown potential (E_B)



Melia, M. A. et al. (2019)

Corrosion of high entropy alloys (HEAs)

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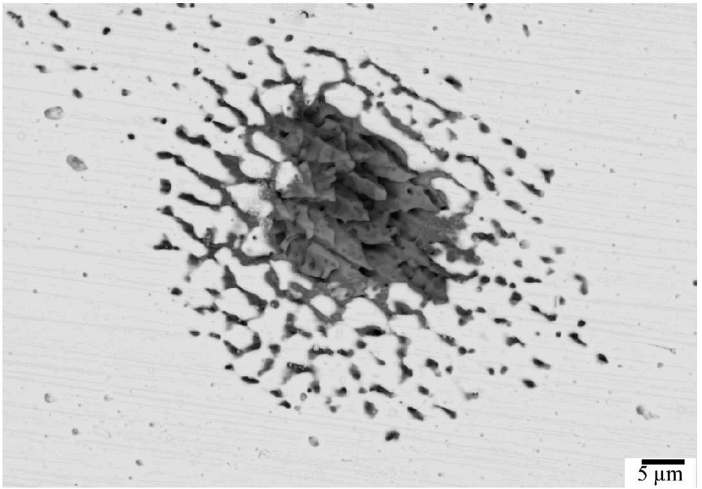
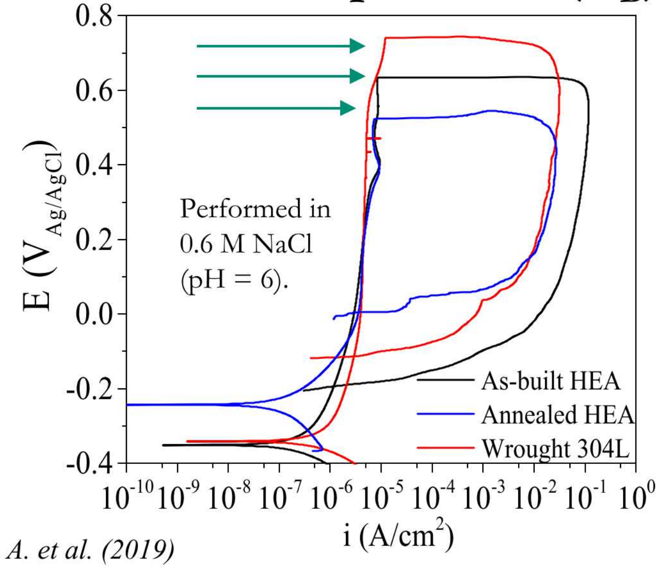
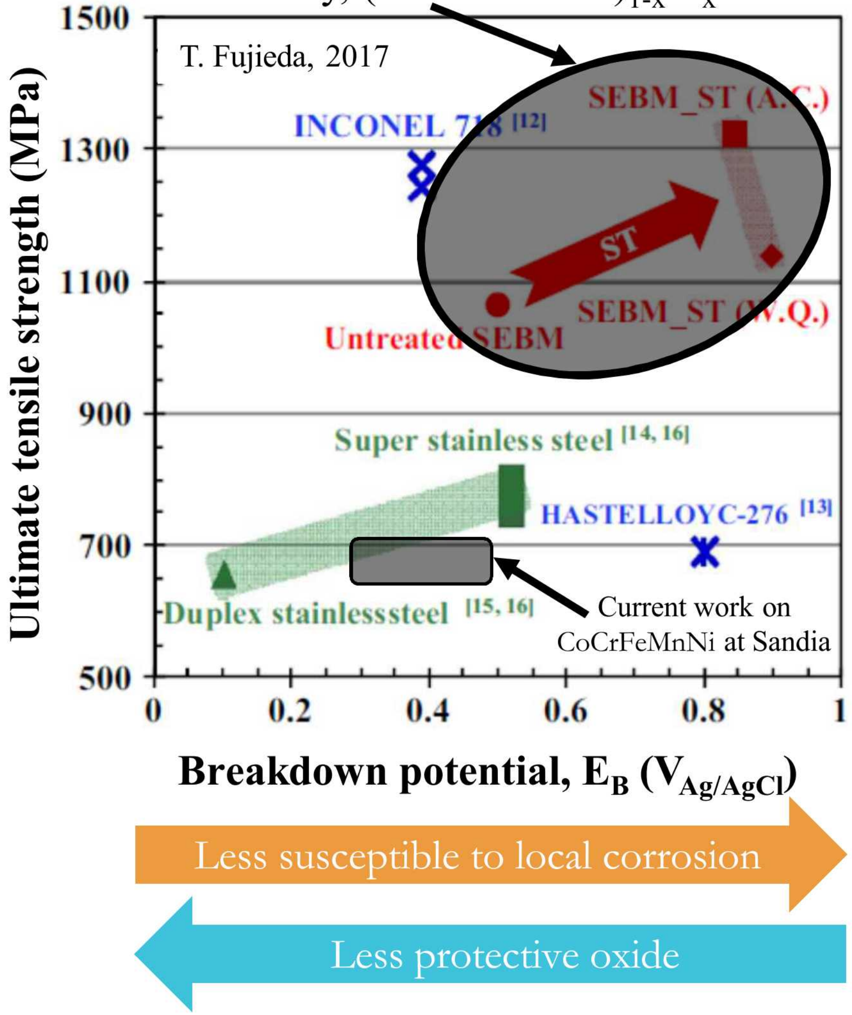


Breakdown potential (E_B)



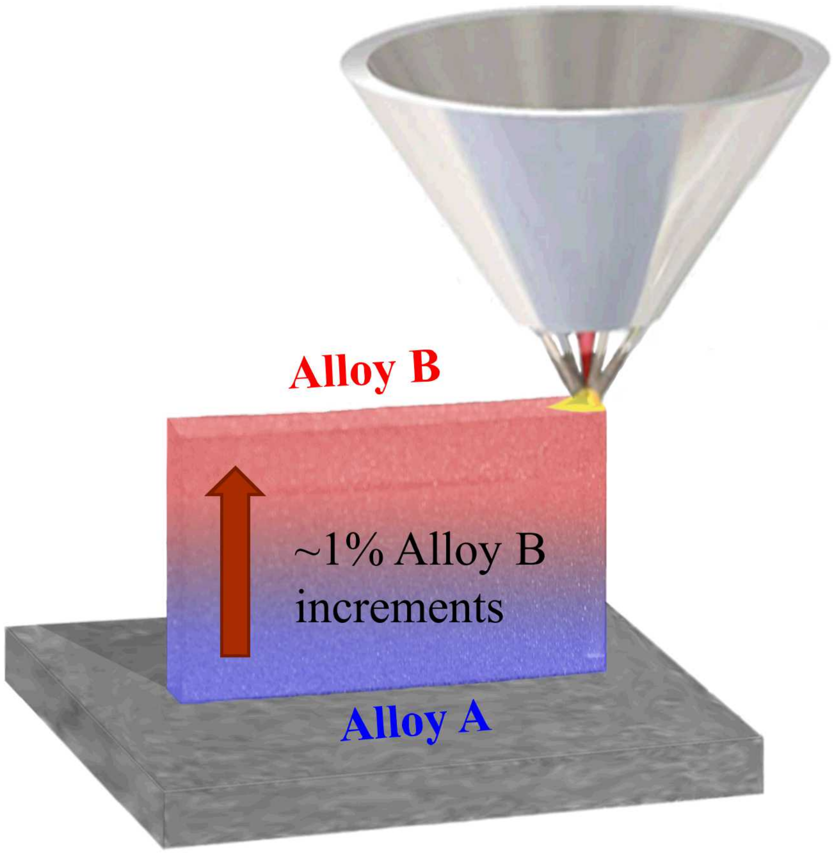
Melia, M. A. et al. (2019)

Potential property space for proposed alloy, $(CoCrFeMnNi)_{1-x}Ti_x$



High-throughput Alloy Screening by AM

Laser Beam – Directed Energy Deposition (LB-DED)



Samples were printed with an in-house LB-DED system with potential for mixing 5 powders.

A pre-mixed equiatomic CoCrFeMnNi alloy powder and commercial purity (CP) Ti powder were used.

Material characterization:

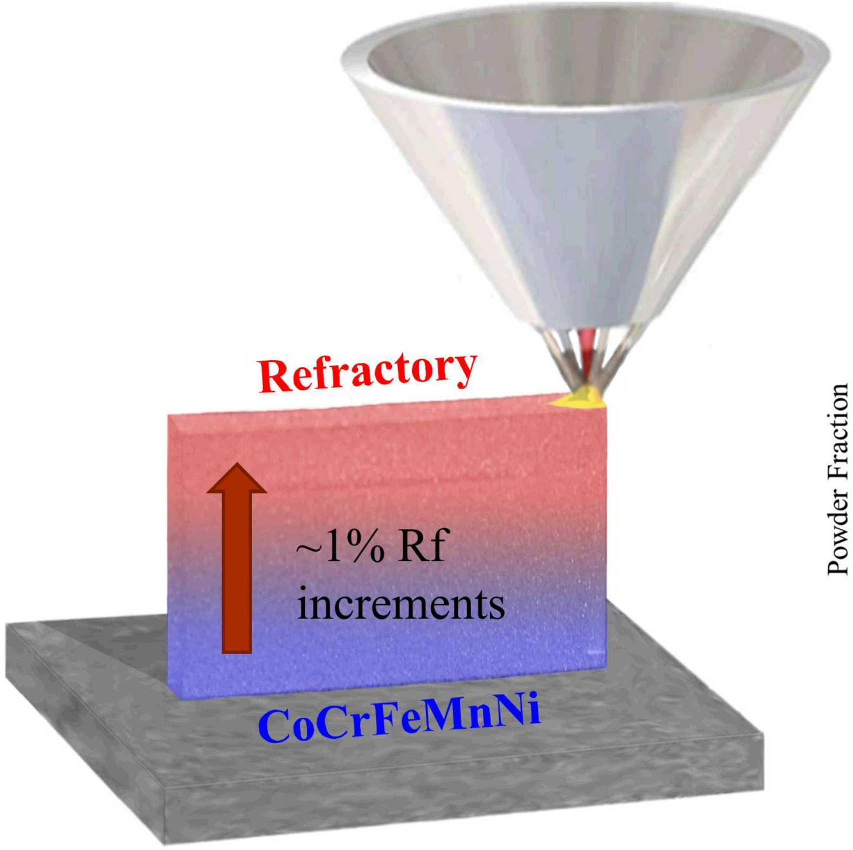
- Site specific x-ray diffraction (XRD).
- Energy dispersive spectroscopy (EDS) and Electron backscattered diffraction (EBSD).

Electrochemical measurements:

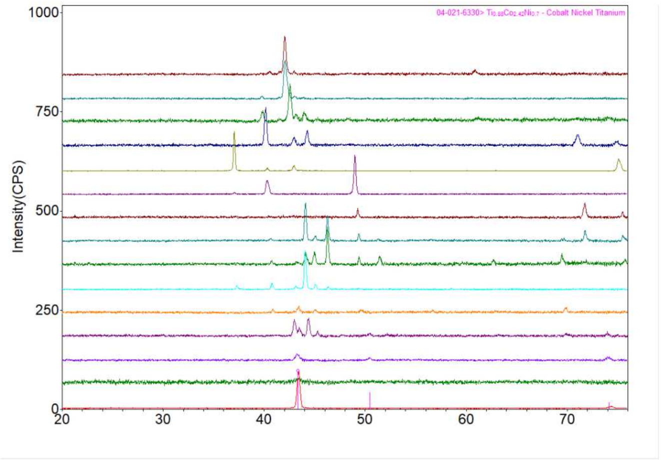
- Capillary cell polarization measurements.
- Repassivation kinetics by scratch testing.

High-throughput Alloy Screening

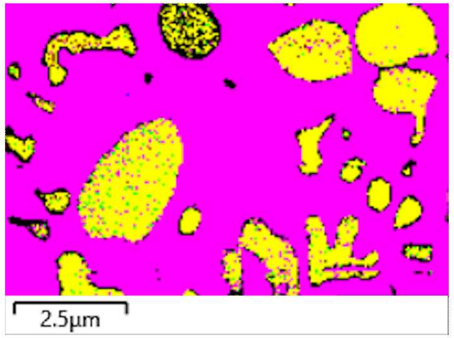
Laser Beam – Directed Energy Deposition (LB-DED)

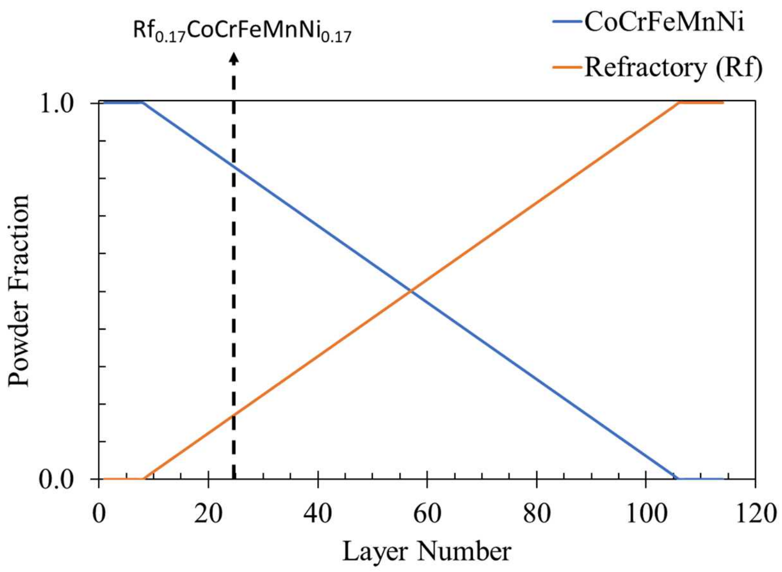


Site specific XRD



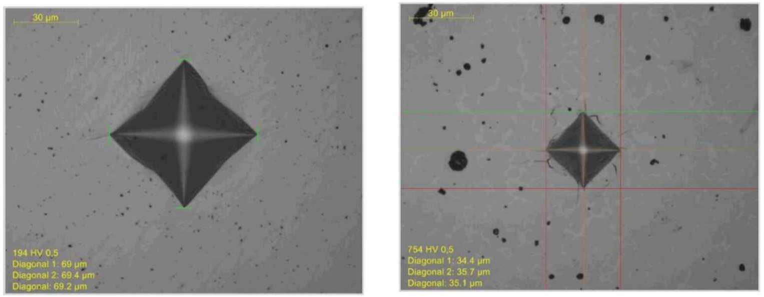
SEM – EDS/EBSD

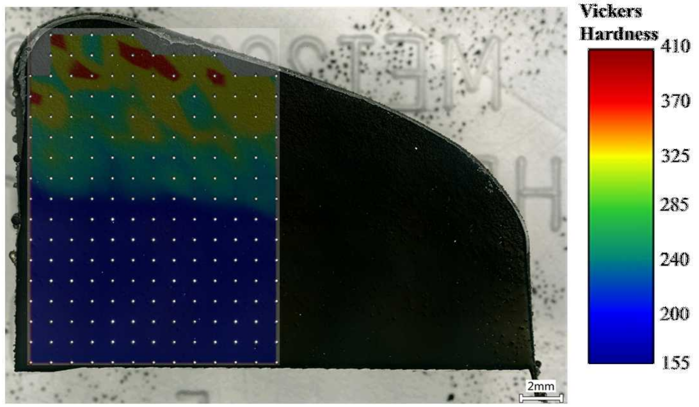




$$Rf_x[CoCrFeMnNi]_{(1-x)/5}$$

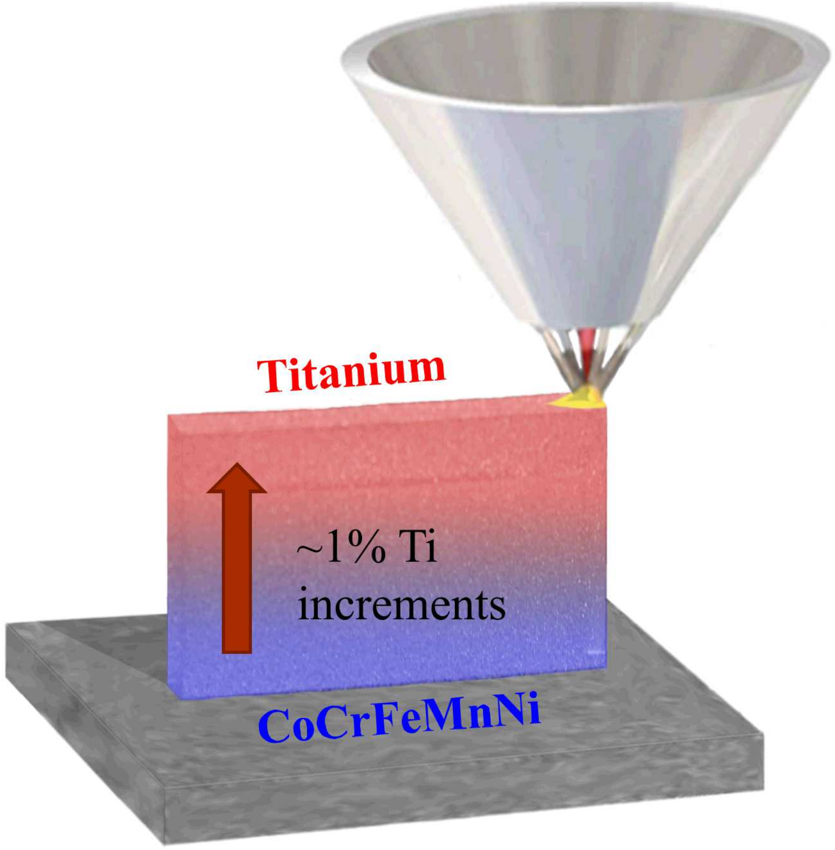
Vickers Hardness mapping



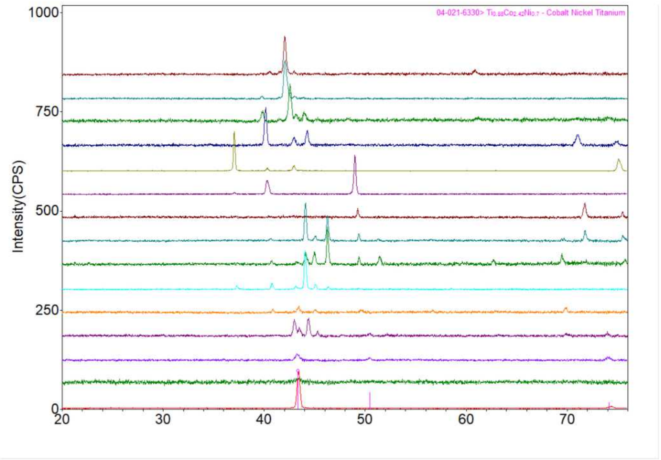


High-throughput Alloy Screening – $Ti_x[CoCrFeMnNi]_{(1-x)/5}$

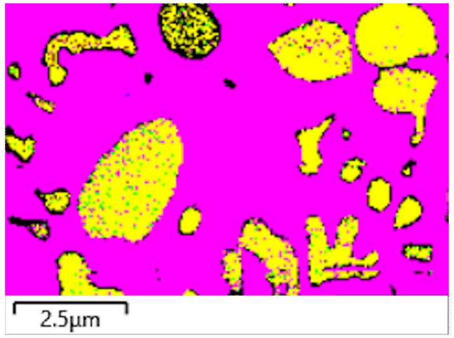
Laser Beam – Directed Energy Deposition (LB-DED)



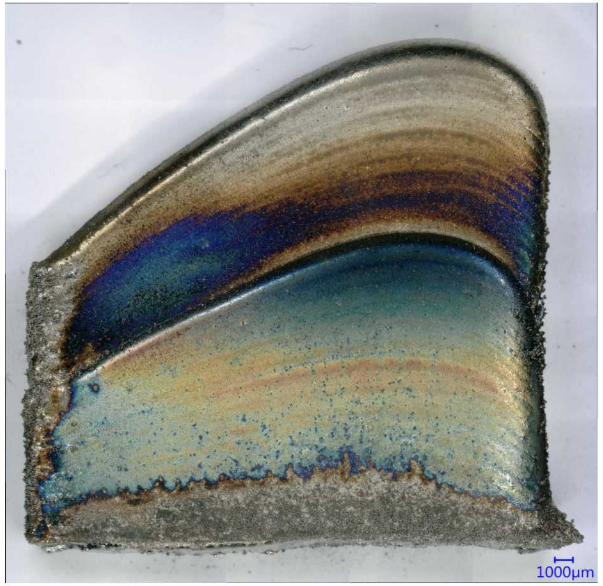
Site specific XRD



SEM – EDS/EBSD

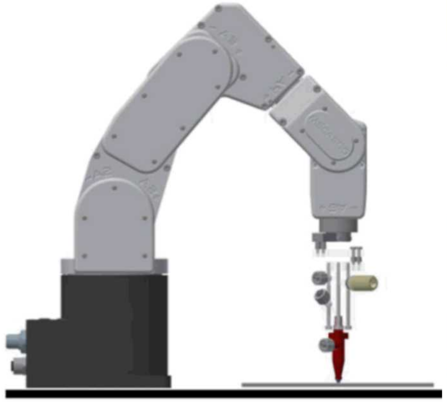


Actual printed HEA/Ti sample



Repassivation kinetics (scratch testing)

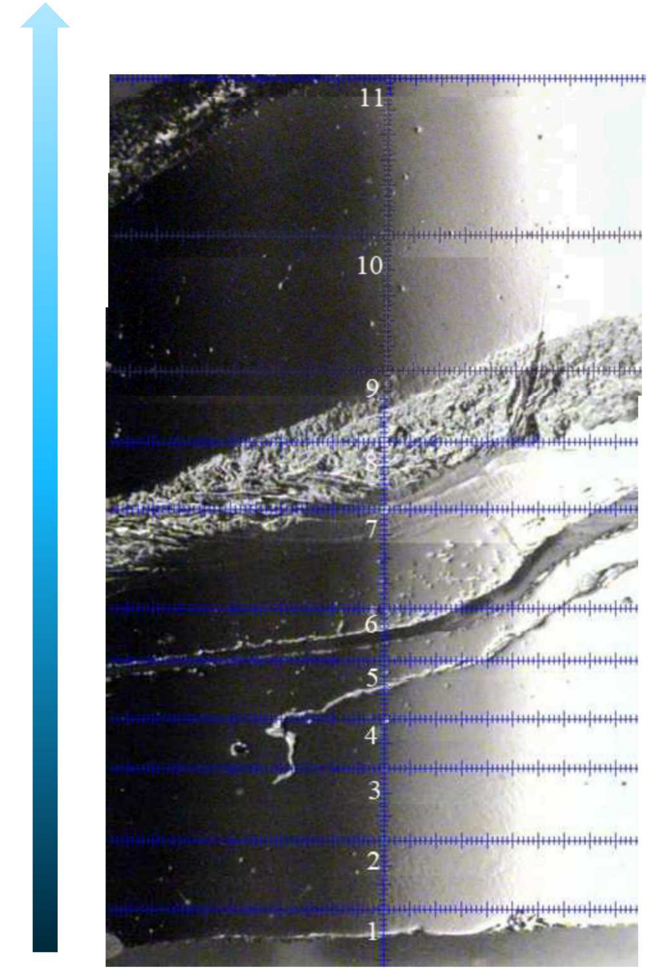
Capillary cell electrochemistry



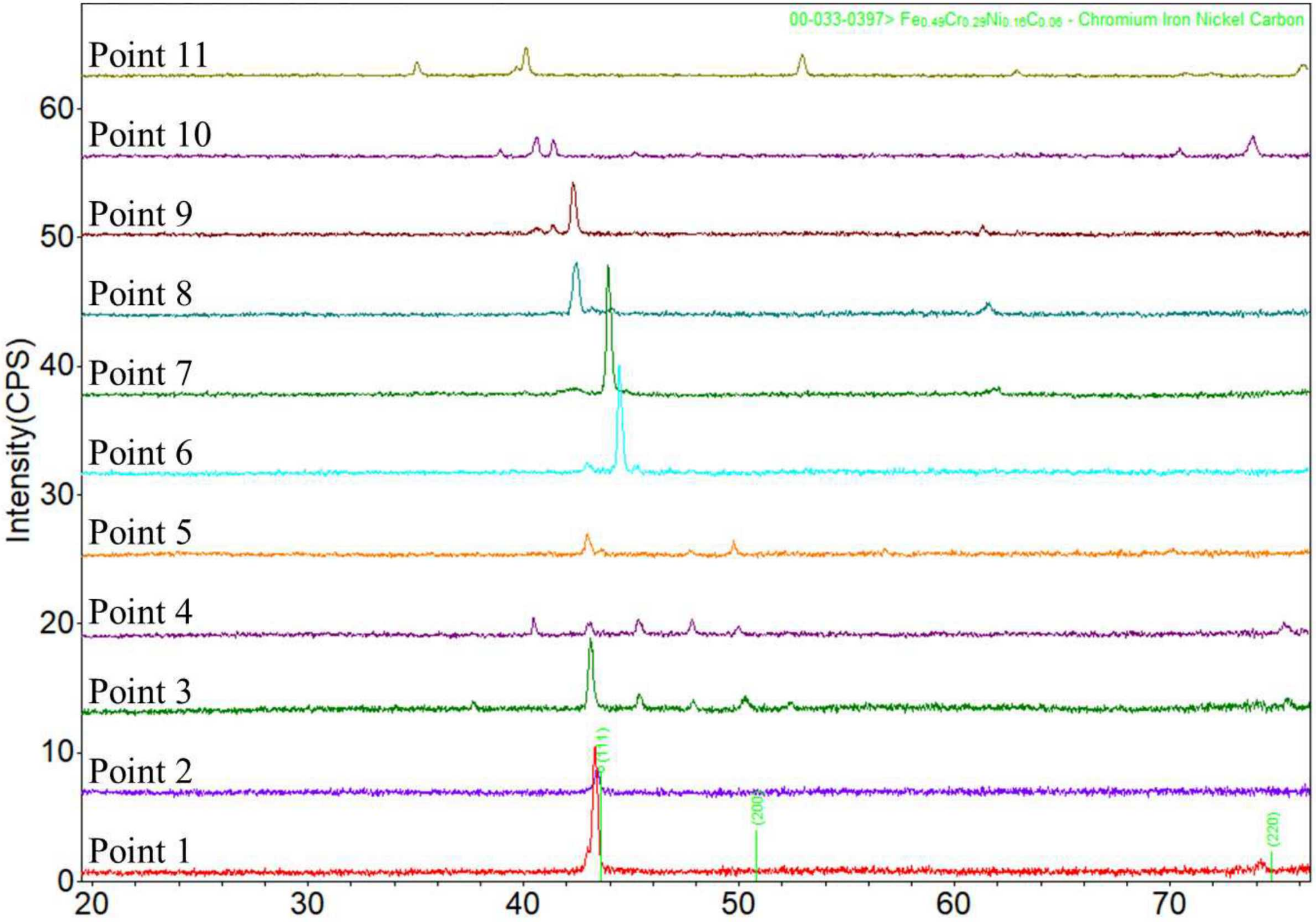
Summers, K. & Chidambaram, D. (2020)

$Ti_x[CoCrFeMnNi]_{(1-x)/5}$ – graded composition phase analysis

Pure Ti



Pure CoCrFeMnNi alloy



Start to see IMCs

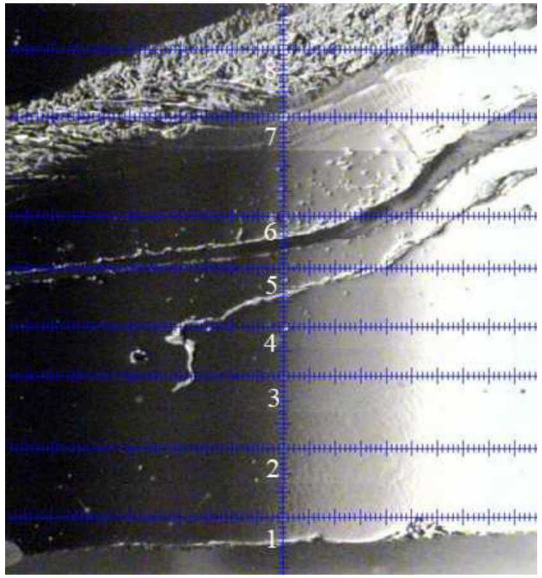
$Ti_x[CoCrFeMnNi]_{(1-x)/5}$ – graded composition phase analysis

Pure Ti

Hexagonal Ti + Ti rich BCC intermetallic

BCC Ti + cubic Ti_2Ni – Ni component is like a combination of other transition metals.

BCC NiTi + BCC Ti + cubic Ti_2Ni



BCC NiTi + hexagonal $TiFe_2$

BCC $Ti_{0.16}Cr_{0.26}Fe_{.58}$ + hexagonal TiCrMn (similar to $TiFe_2$)

BCC $Ti_{0.16}Cr_{0.26}Fe_{.58}$ + hexagonal $TiFe_2$

FCC steel + BCC $Ti_{0.16}Cr_{0.26}Fe_{.58}$ + hexagonal $TiFe_2$

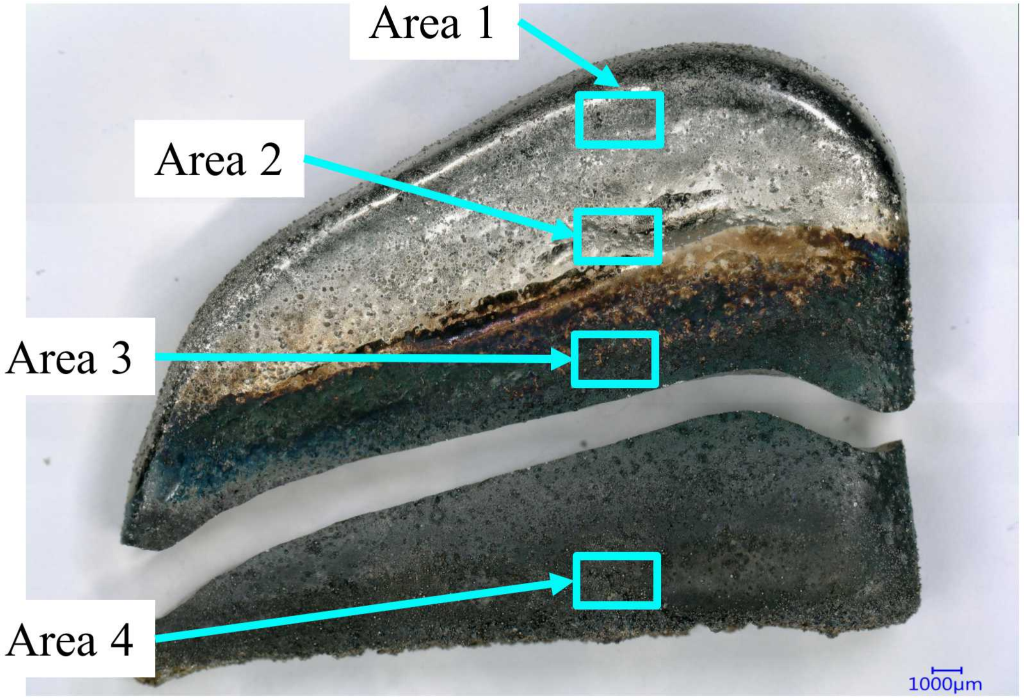
FCC steel

Pure CoCrFeMnNi alloy

$Ti_x[CoCrFeMnNi]_{(1-x)/5}$ – graded composition phase analysis

Ti BCC | Ti Hex | Ni_3Ti

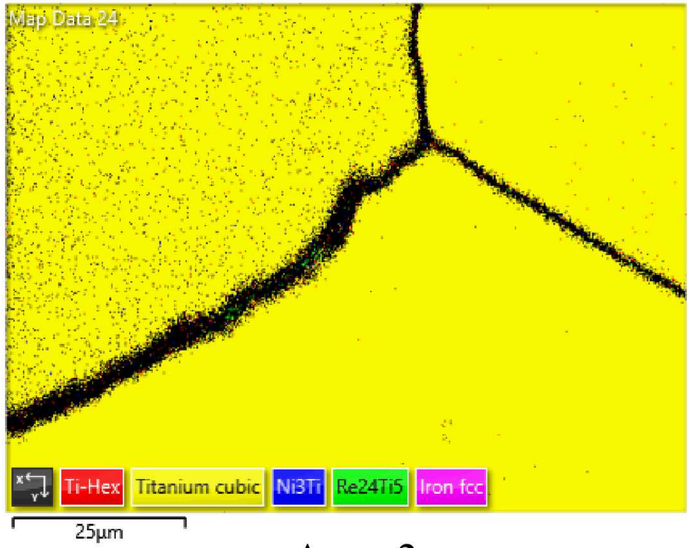
$Tm_{24}Ti_5$ | Fe FCC



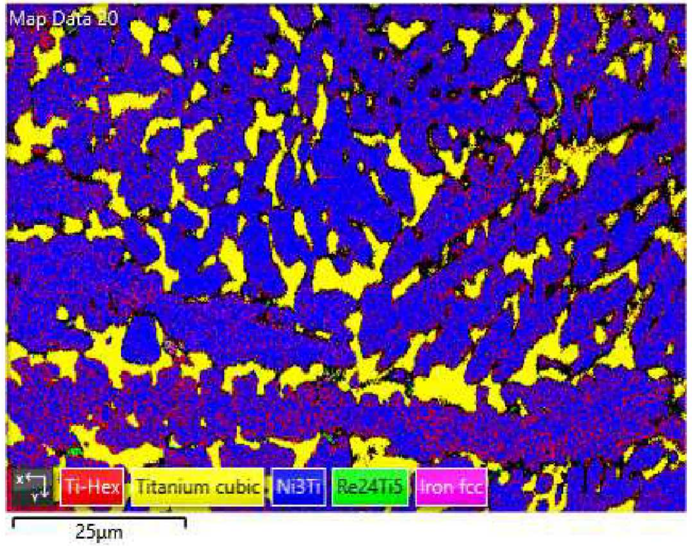
Using EBSD alone does not allow for accurate phase identification, analysis of material with XRD informed phase ID appears to be more accurate.

XRD helps distinguish between Ti right and transition metal rich intermetallic phases.

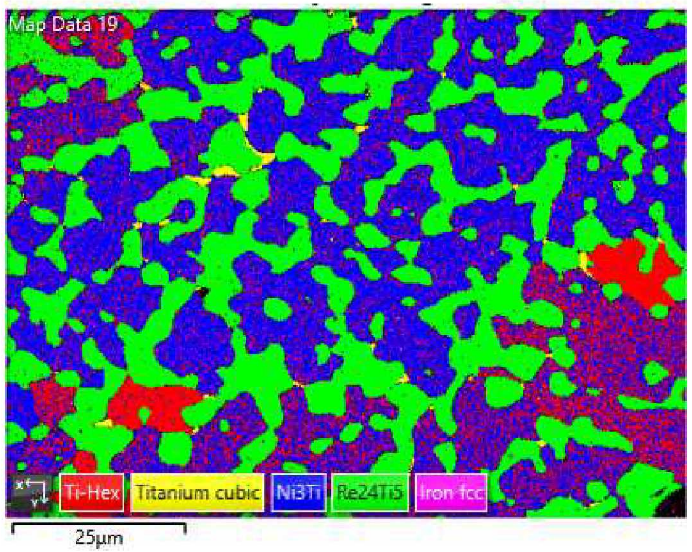
Area 1



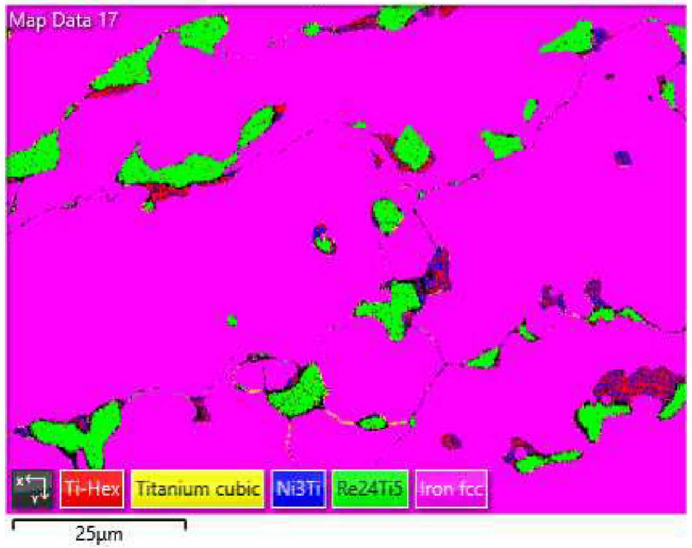
Area 2



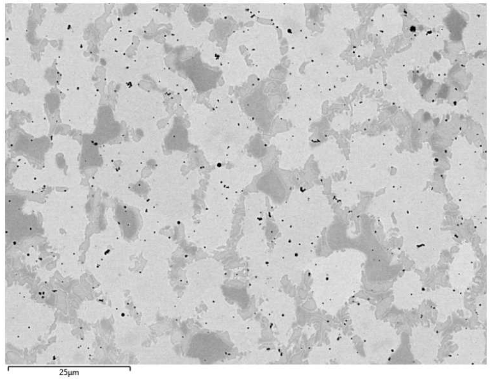
Area 3



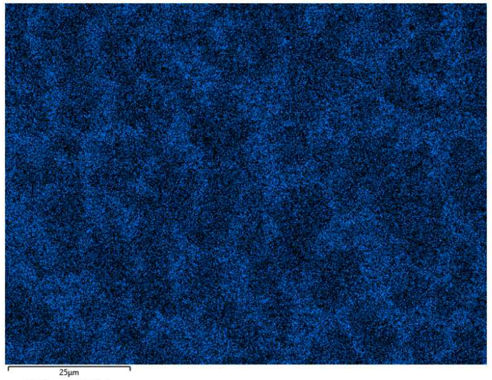
Area 4



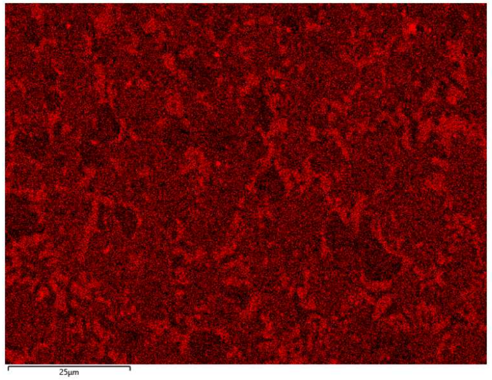
$Ti_x[CoCrFeMnNi]_{(1-x)/5}$ – Equi-atomic composition



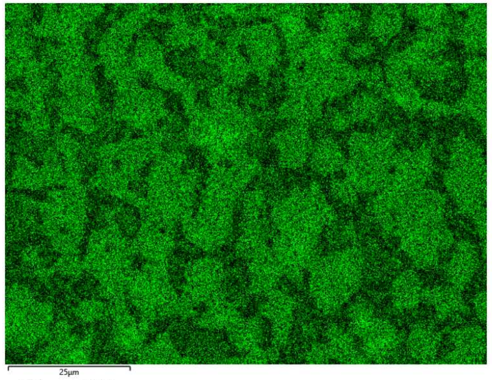
Ni K-α



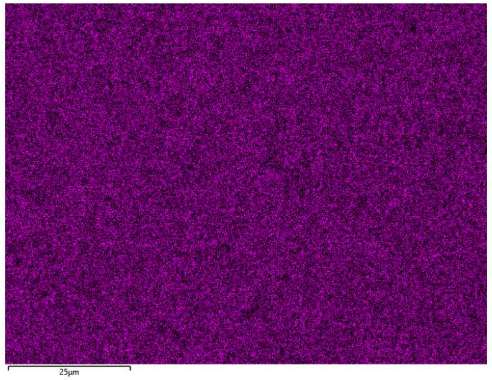
Ti K-α



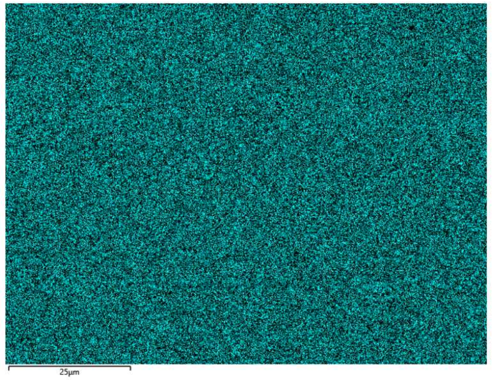
Cr K-α

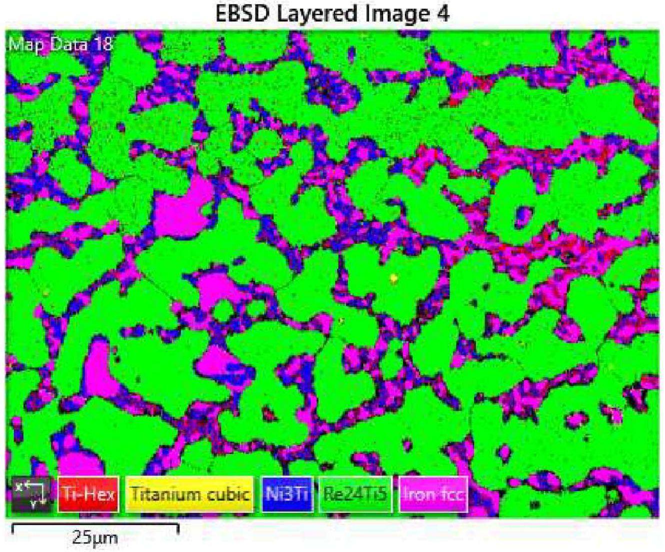


Mn K-α



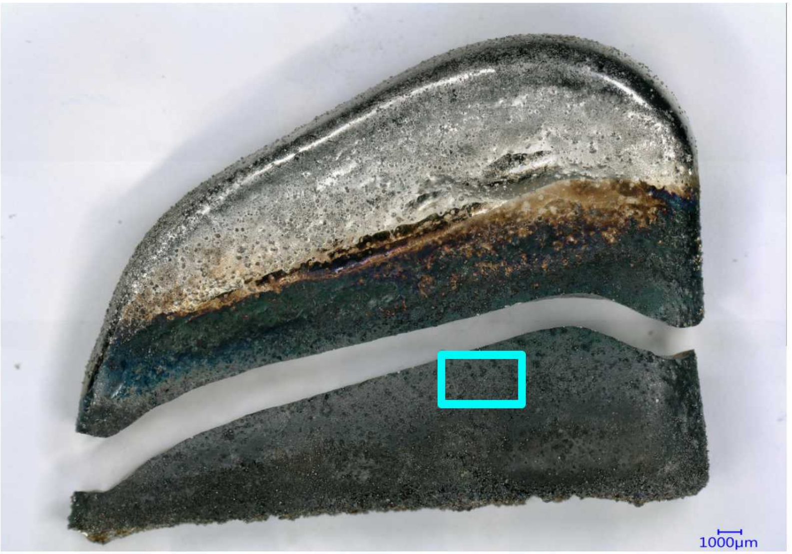
Co K-α





Expected phases from XRD/EBSD/EDS

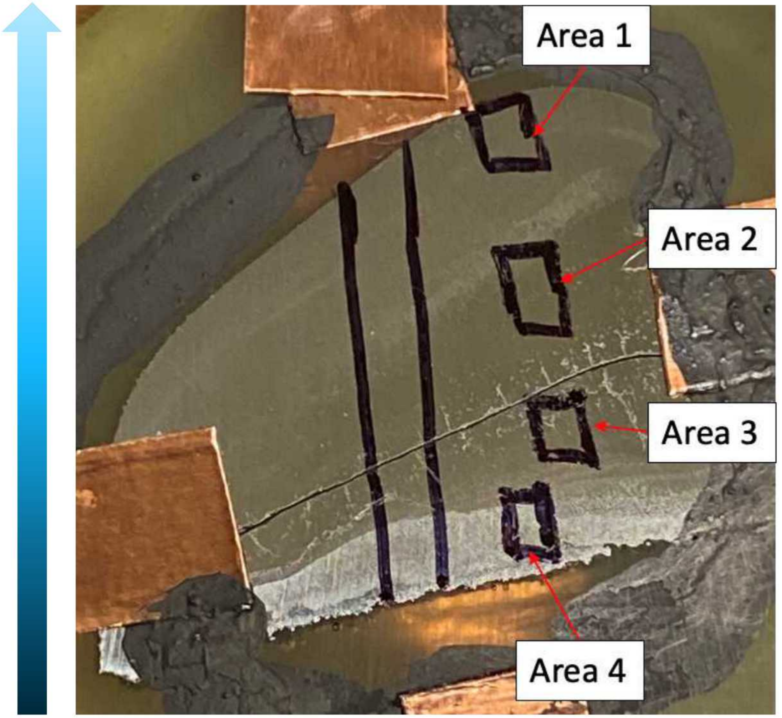
- BCC $Ti_{0.16}Cr_{0.26}Fe_{.58}$
- Hexagonal $TiFe_2$
- FCC - steel



SNL Materials Characterization – Luis Jauregui and Sara Dickens

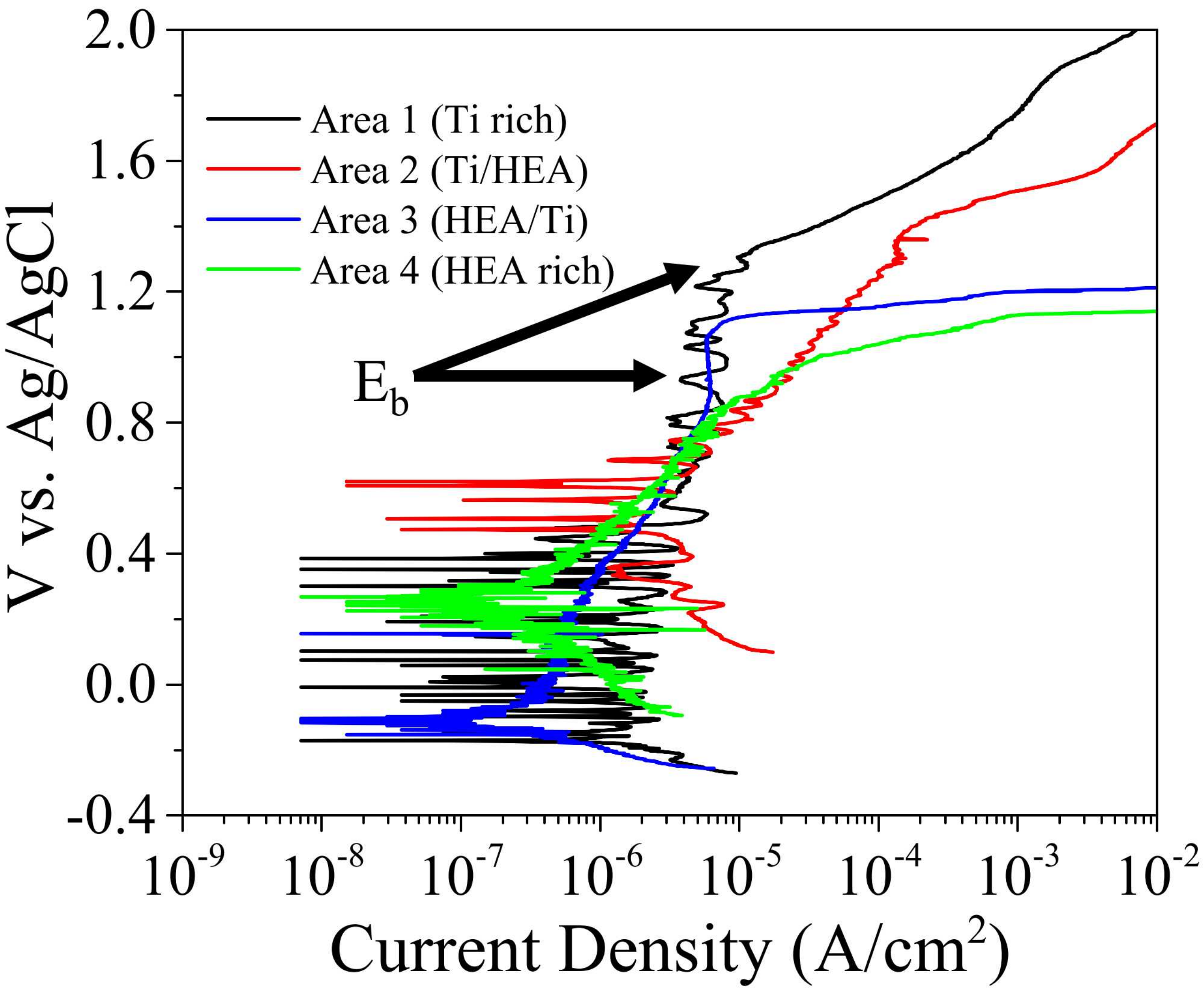
Electrochemical behavior – Capillary cell measurements

Pure Ti



Pure CoCrFeMnNi alloy

Parameters	
WE	Ti-graded Cantor
CE	Pt wire
RE	3M Ag|AgCl
Electrolyte	0.599 M NaCl
Scan Range	-0.05V vs. Eoc to 2.0V vs. E_{ref}
Scan rate	1 mV/s
Flow rate	0.075 mL/min

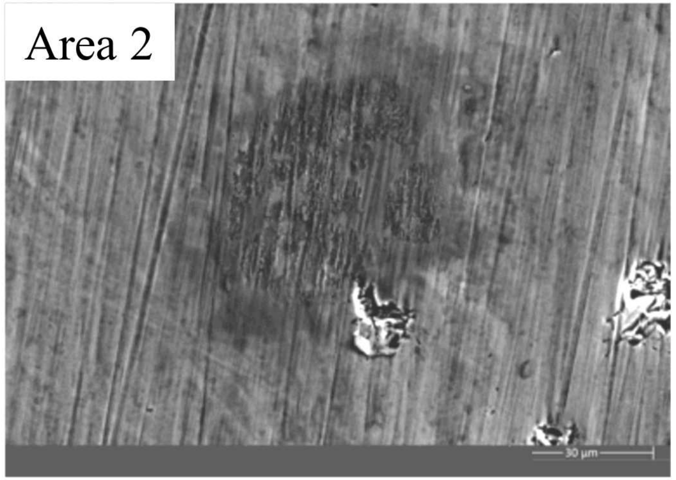


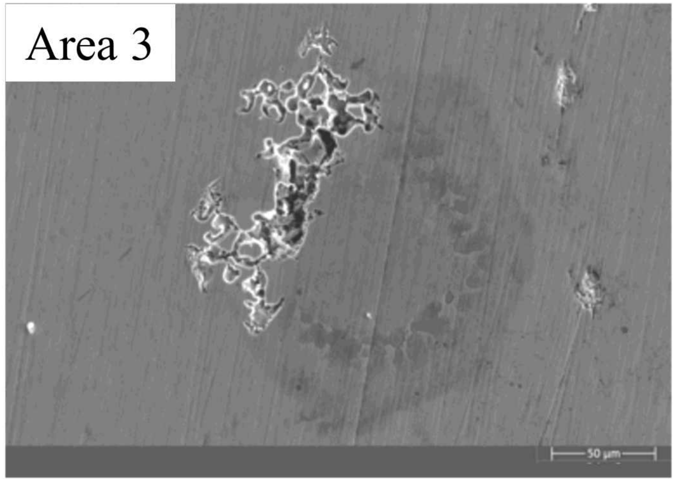
UN-Reno collaboration with Dr. Chidambaram and Mr. Summers

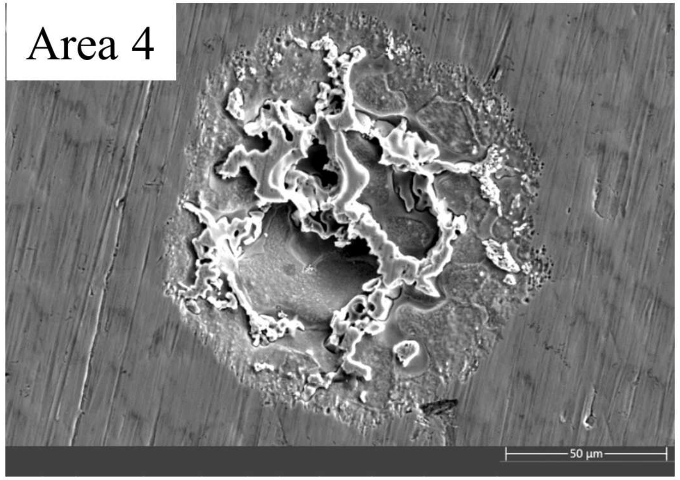
Electrochemical behavior – Capillary cell measurements

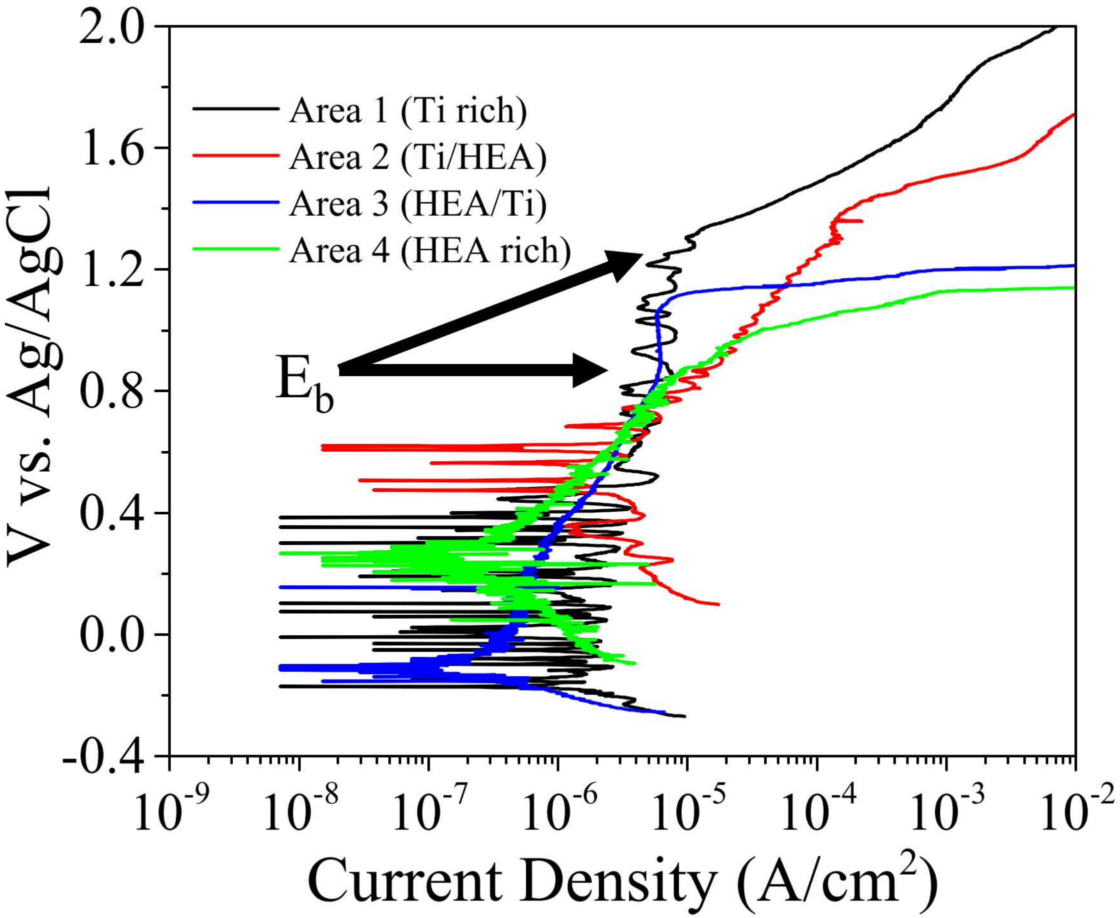
Area 1

No image





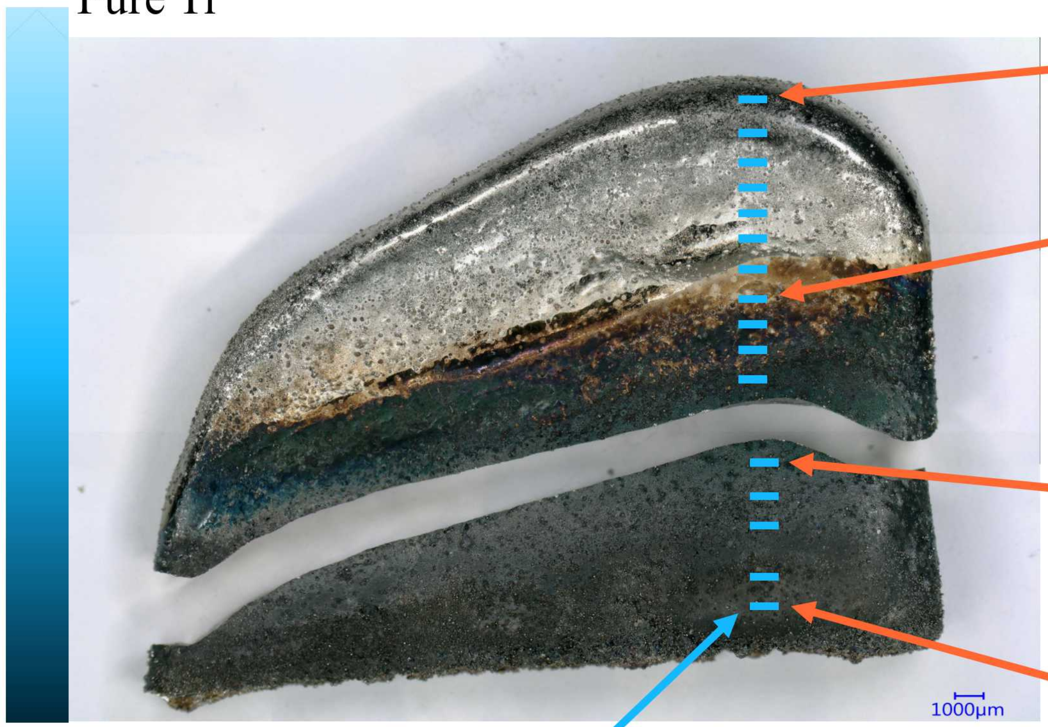


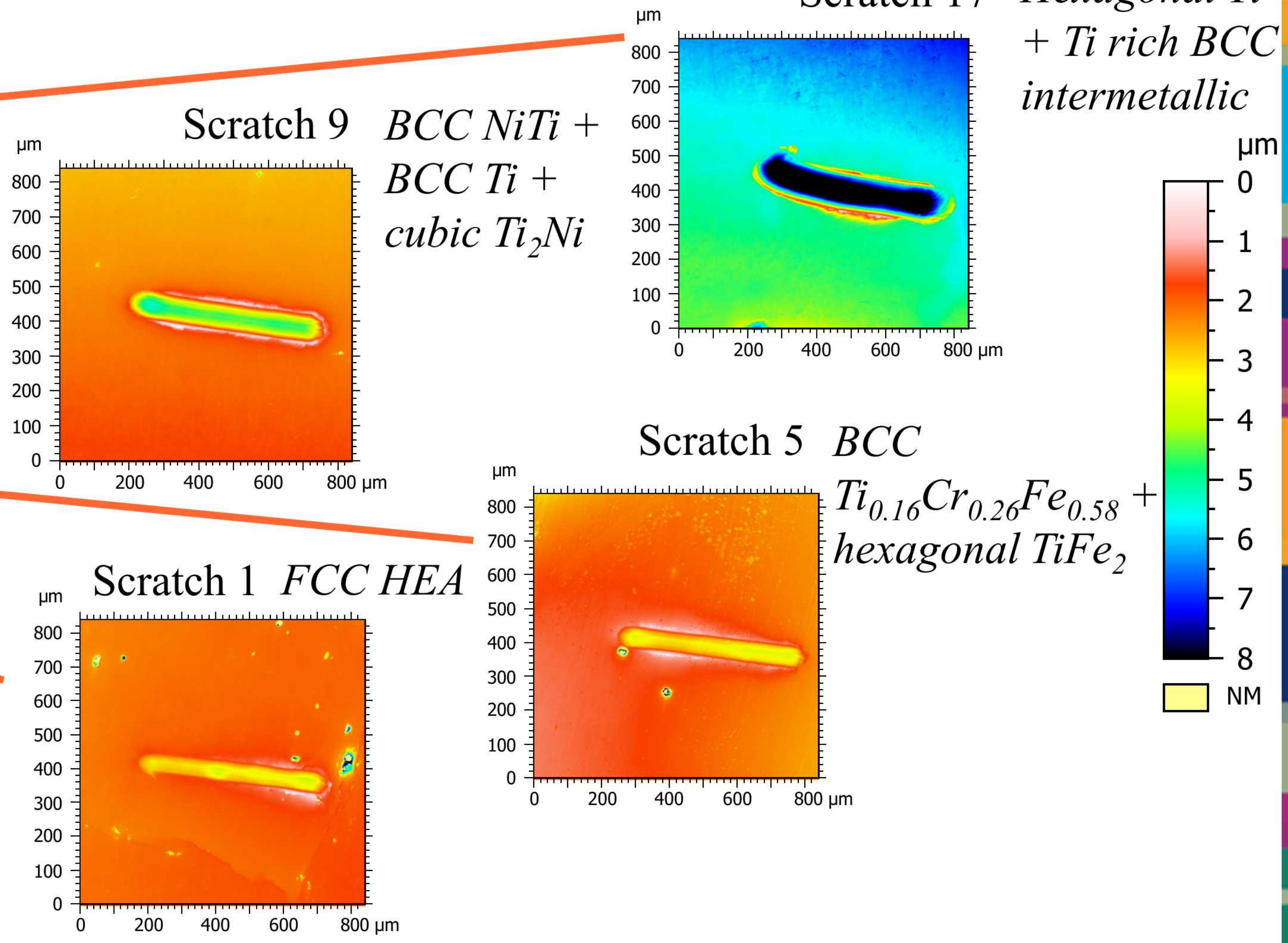


Area 1 E_b ~ 1.3 V vs. Ag/AgCl
Area 2 E_b ~ 1.3
Area 3 E_b ~ 1.1
Area 4 E_b ~ 0.9

UN-Reno collaboration with Dr. Chidambaram and Mr. Summers

Electrochemical behavior – Scratch testing for repassivation kinetics





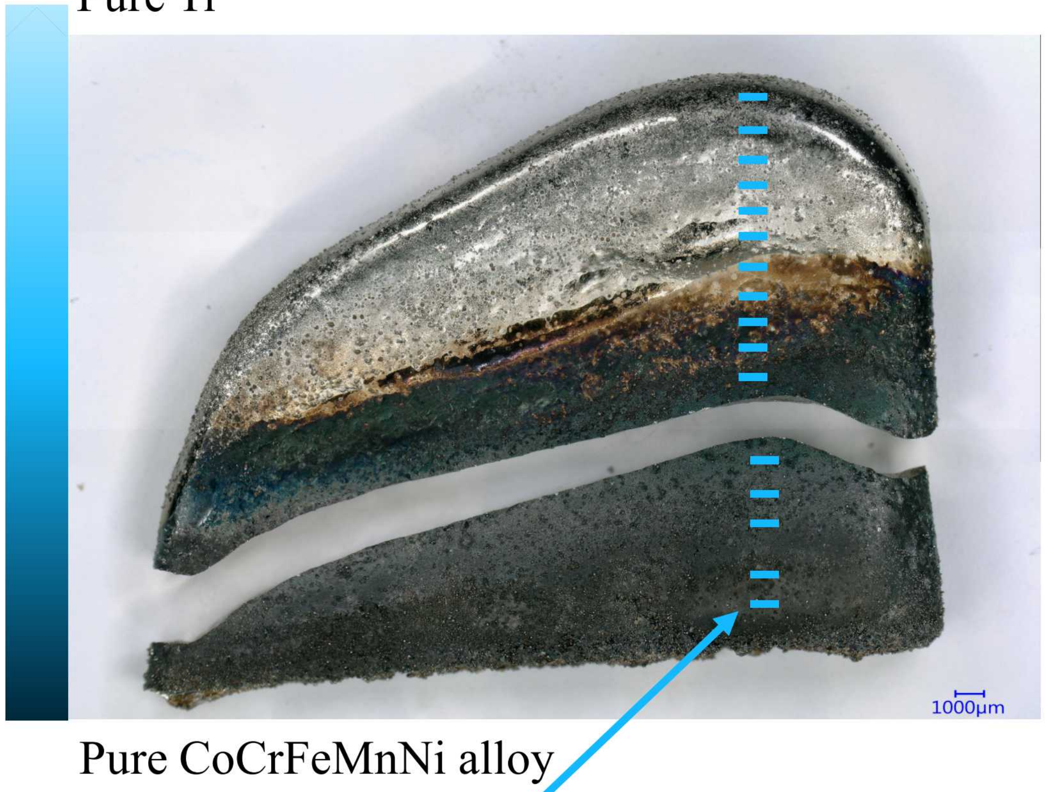
First scratch after 5500 second anodic polarization (+ 200 mV vs. Ag/AgCl) in 1 M Na_2SO_4 solution.

All performed with scratches at 20 N, 60 mm/min, 0.5 mm long.

17 scratches by a spheroconical diamond scribe (r = 100 µm) along the composition gradient.

SNL tribology lab – Brendan Nation, Morgan Jones, and Nic Argibay

Electrochemical behavior – Scratch testing for hardness measurement



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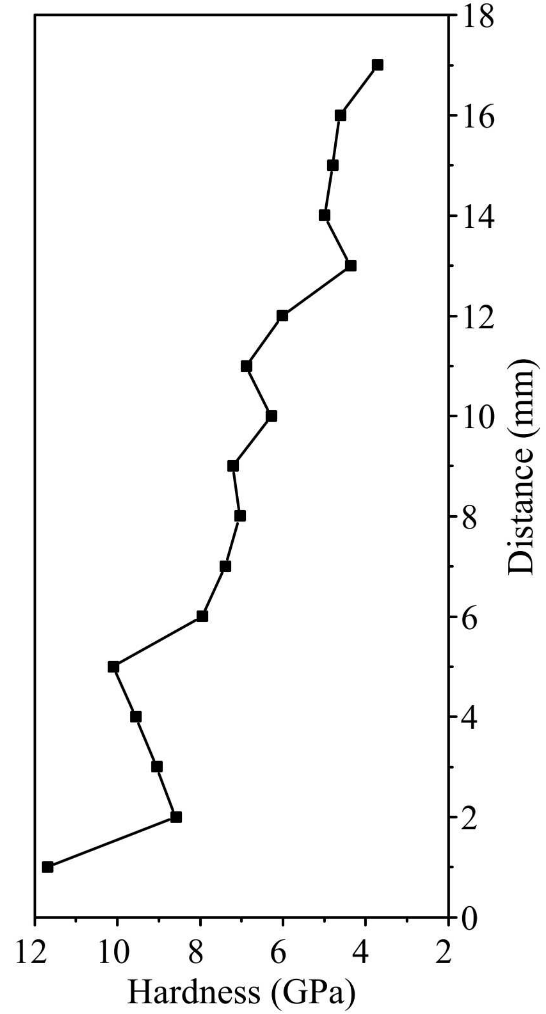

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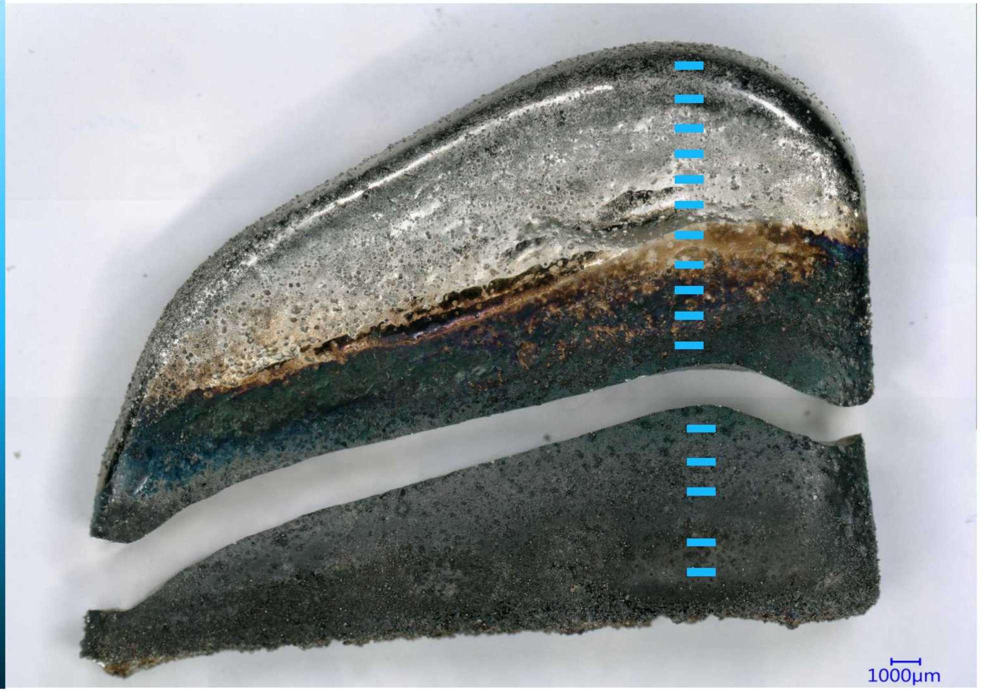
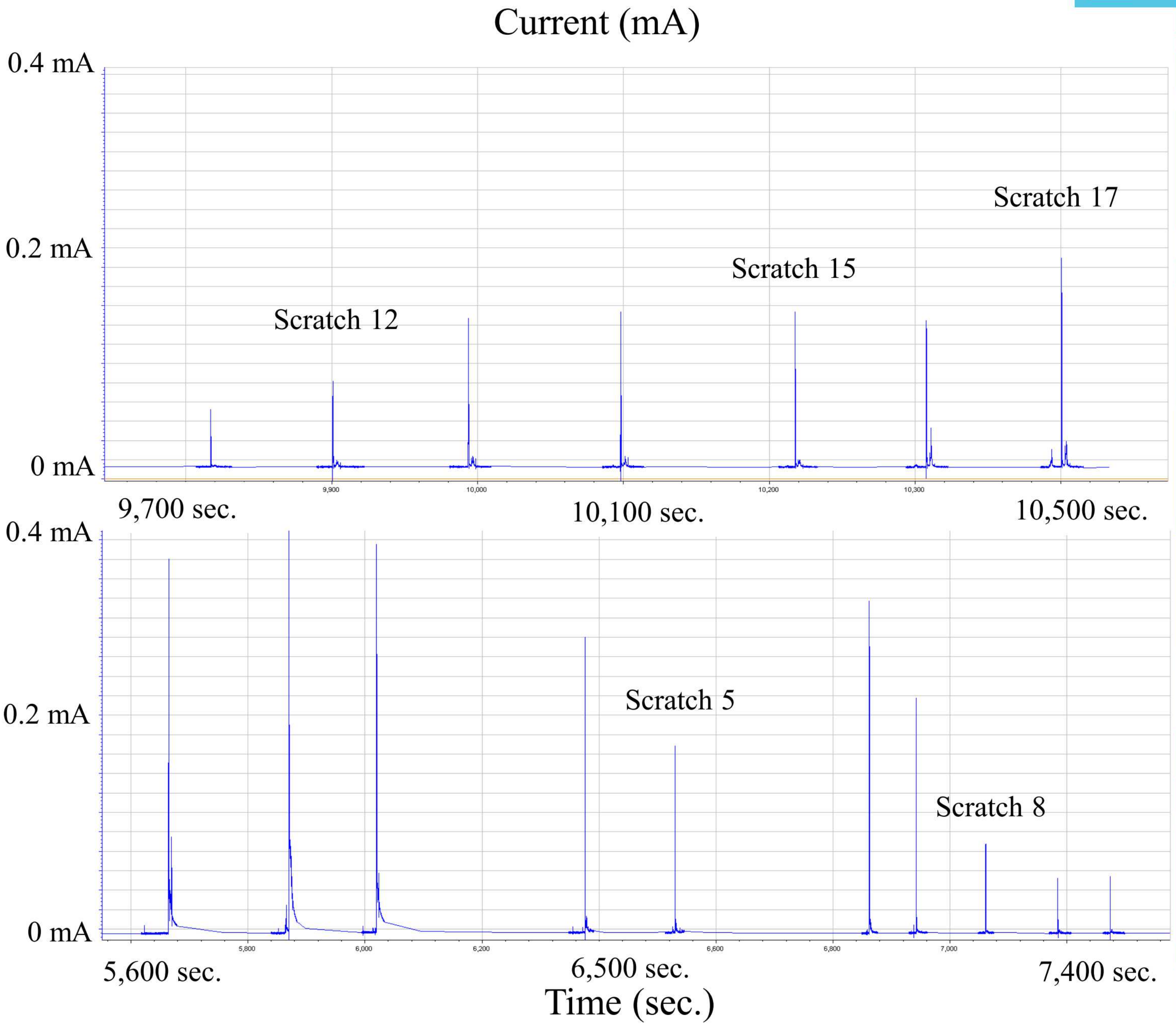
Electrochemical behavior – Scratch testing for hardness measurement

Pure Ti



Pure CoCrFeMnNi alloy



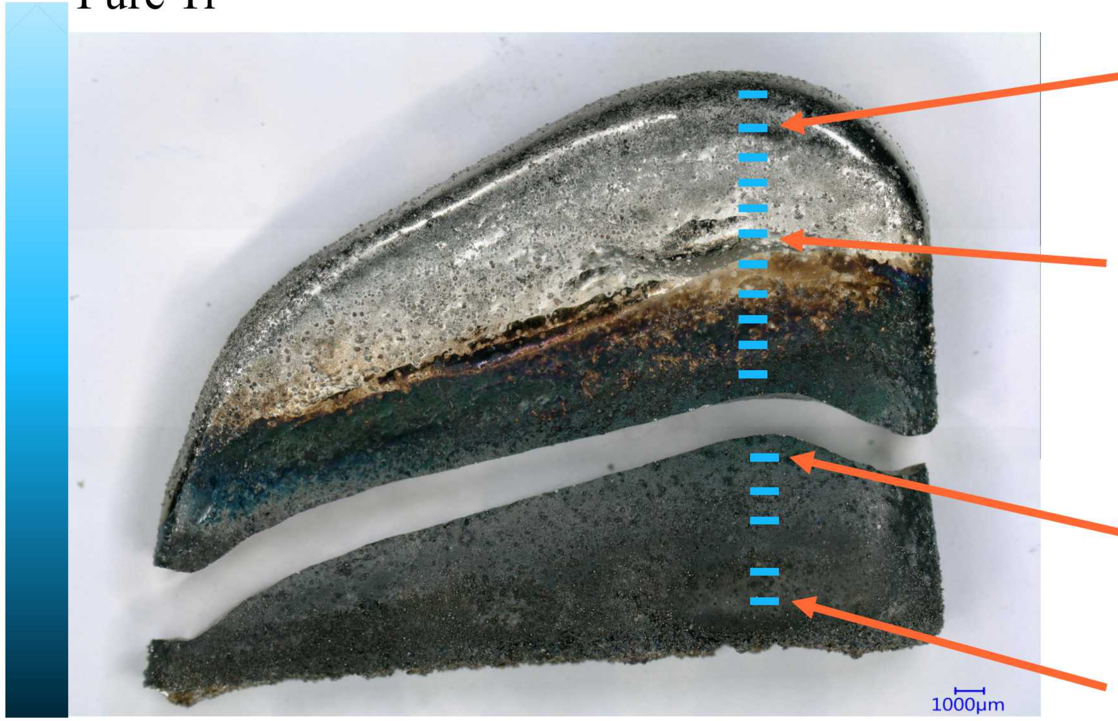
Prior to the scribe touching the sample, the data acquisition rate was switched to 10,000 pt./sec.

Peak current is maximum for the CoCrFeMnNi rich side of the sample, minimum at the Ti rich/IMC rich region, and increased when moving closer to pure Ti.

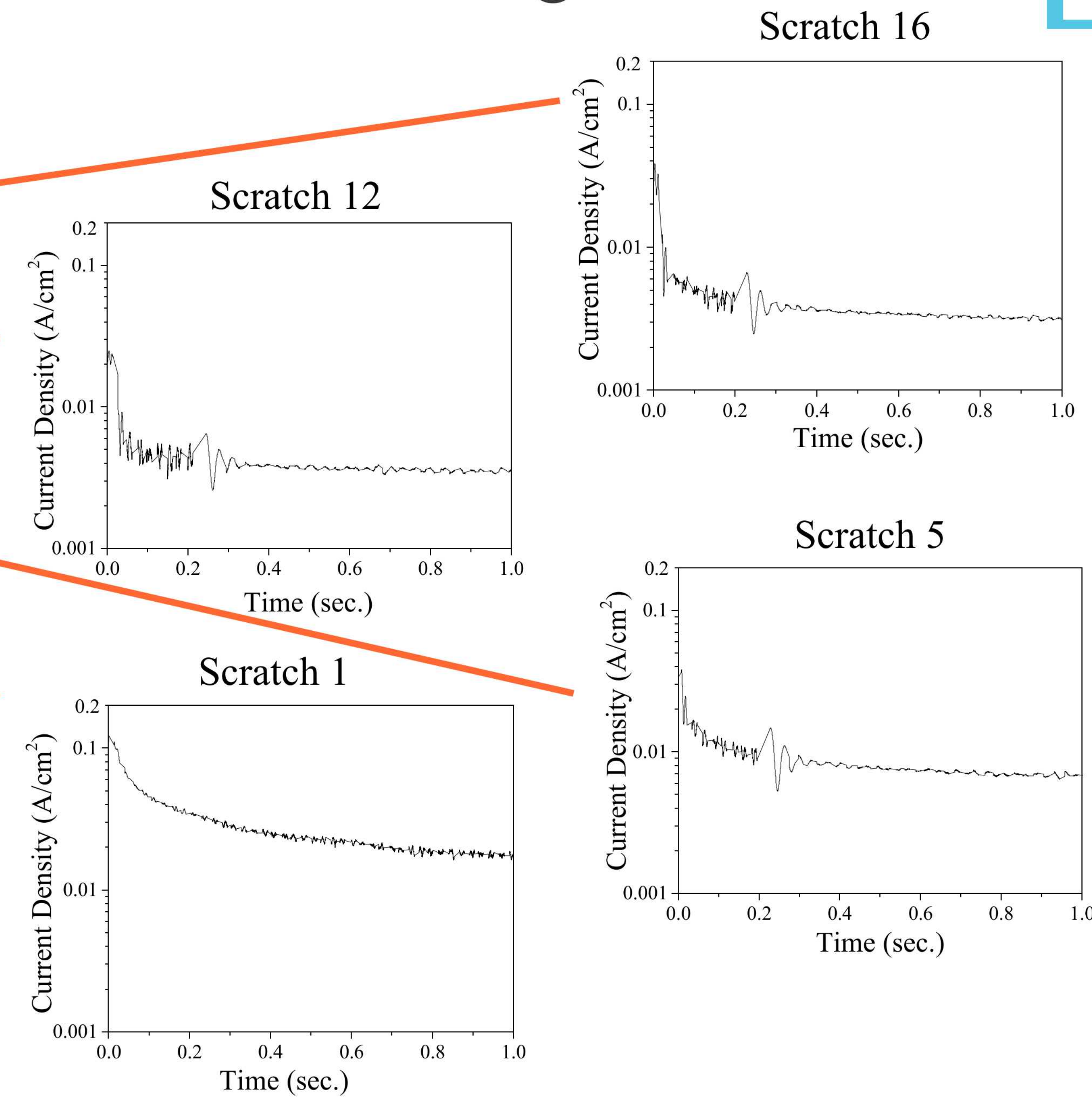
SNL tribology lab – Brendan Nation, Morgan Jones, and Nic Argibay

Electrochemical behavior – Scratch testing for repassivation kinetics

Pure Ti



Pure CoCrFeMnNi alloy



There is a lot of potential for this rapid experimental approach, but fine tuning will be required in the acquisition and analysis.

SNL tribology lab – Brendan Nation, Morgan Jones, and Nic Argibay

Conclusions

- The phase analysis portion of the high throughput alloy screening approach is able to accurately determine phase constitution across these compositionally graded alloys.
  - Doing site specific XRD first, prior to EDS/EBSD, helps with more site specific phase analysis.

- Electrochemical measurements using a capillary cell showed the breakdown potential of the Ti rich side of the sample to be larger than the CoCrFeMnNi rich side of the sample.
  - Mitigating noise in measurements and limiting crevice corrosion will be critical to generating consistent results.

- Scratch testing shows promise as an approach to determine mechanical and corrosion properties of the compositionally graded alloys.
  - The quicker repassivation (return to stable current) for the Ti rich region of the sample correlates well with the larger breakdown potential seen for capillary cell measurements.

Acknowledgements

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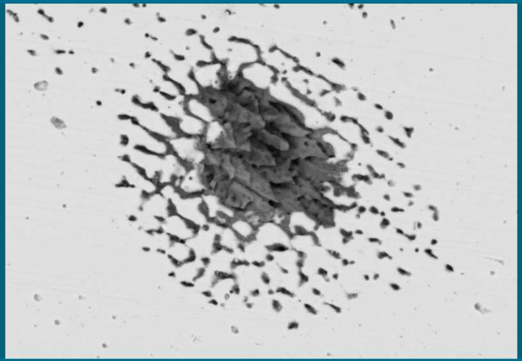
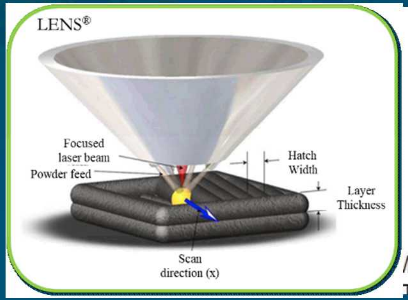
Slide 21

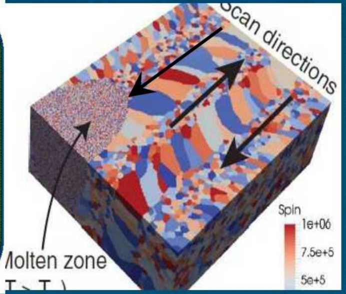
SR13 Probably don't need extra slides as you won't be able to present them...also ecs suggest putting your title slide again as your last slide

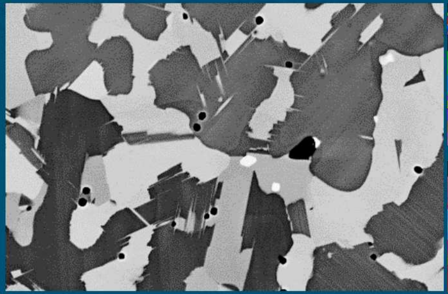
Schaller, Rebecca, 9/16/2020

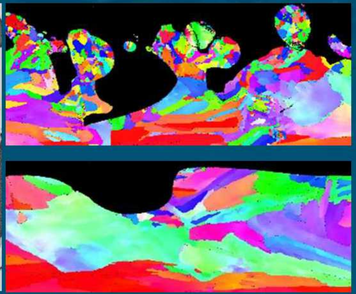
Additive Manufacturing to Compositionally Grade Metals for High Throughput Alloy Screening











Michael Melia (SNL)

PRESENTED BY

Michael Melia (SNL)

Co-authors: Jonathan Pegues, Morgan Jones, Brendan Nation, Nicolas Argibay, Andrew Kustas (SNL)

Kodi Summers and Dev Chidambaram (UN-Reno)

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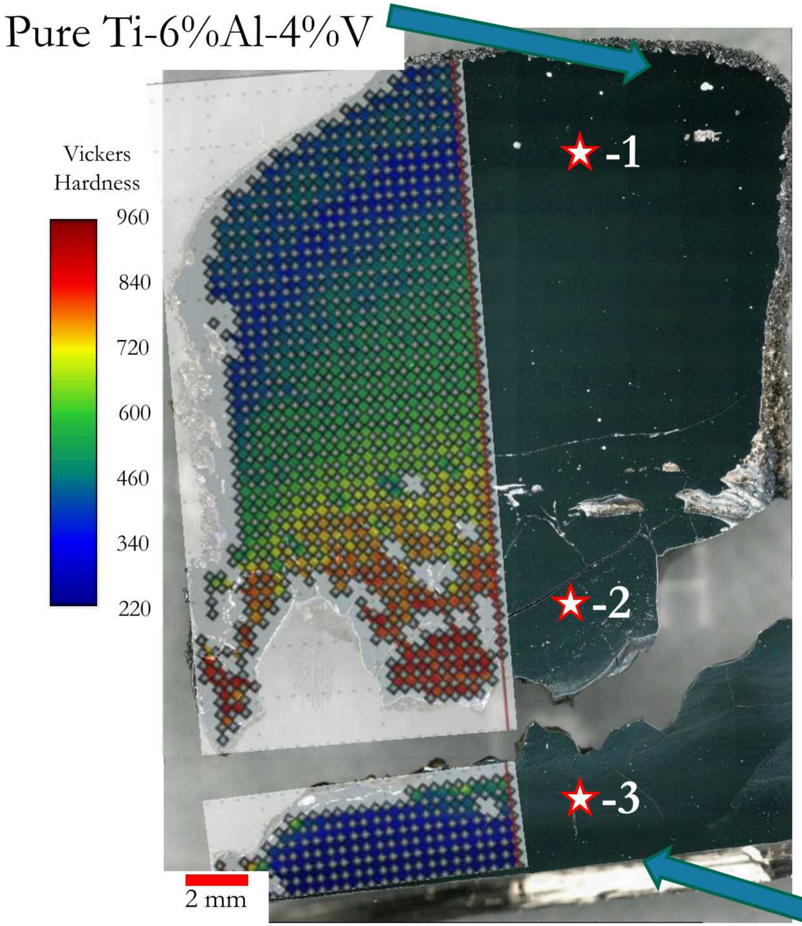
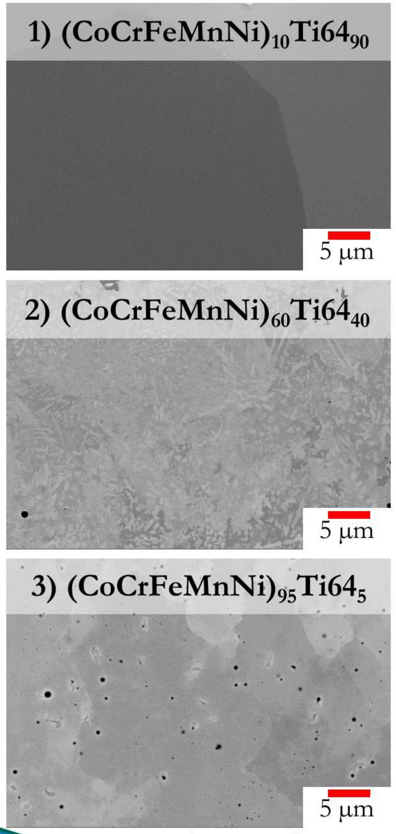
Extra slides

Background: Current HEA studies at Sandia

The most recent studies at Sandia used the LENS process to create a compositionally graded CoCrFeMnNi HEA with the Ti-6%Al-4%V alloy. These types of samples allow for rapid screening of properties such as microhardness (map in left of image).

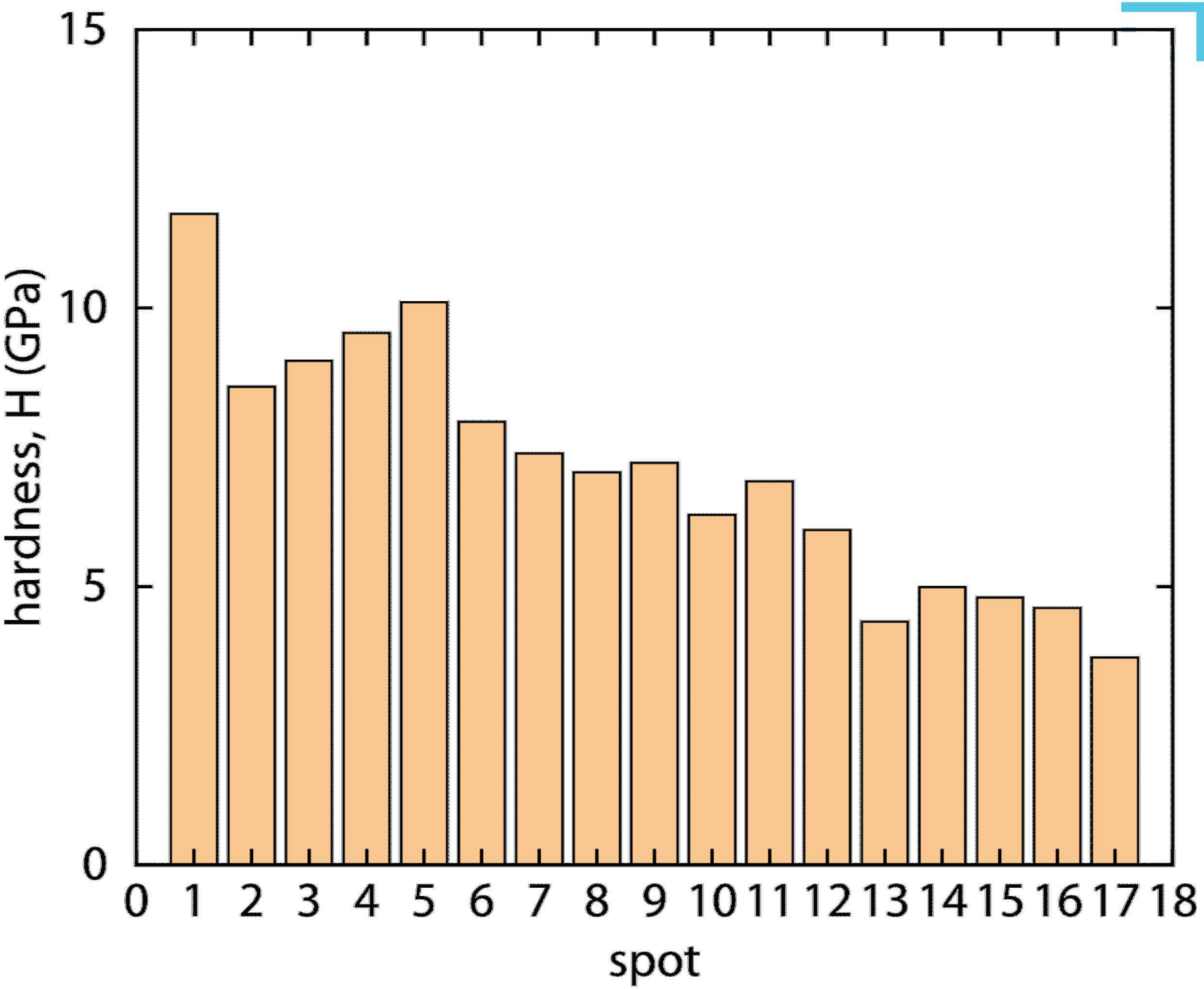


Example electron micrographs



Microstructural characterization along this sample helped refine composition selections to ones that exhibit chemical homogeneity, expected to be a requirement for the Cr and Ti to behave synergistically in corrosive environments.

spot	hardenss (GPa)
1	11.7
2	8.6
3	9.1
4	9.6
5	10.1
6	8.0
7	7.4
8	7.0
9	7.2
10	6.3
11	6.9
12	6.0
13	4.4
14	5.0
15	4.8
16	4.6
17	3.7



hardness (GPa)

$$H_s = \frac{8F_n}{\pi w^2}$$

$$K_c = \frac{F_t}{(2pA)^{1/2}}$$

$$A = \frac{1}{2}Rl_p - (R - d_p)\sqrt{R^2 - (R - d_p)^2}$$

$$l_p = 2R\arccos\frac{R - p_d}{R}$$

K_c is fracture toughness

F_t is friction force, measured and recorded during scratch

A is projected frontal area

p is maximum penetration depth, SWLI

l_p is perimeter length

R is radius of indenter