INL/MIS-22-66184-Revision-0

In-Situ Powder-Directed Energy Process Control for Additively ManufacturedMulti-Layer, Functionally Graded Components

February 2022

Changing the World's Energy Future

Calvin Myer Downey, Isabella van Rooyen, Luis Nunez, Indrajit Charit, Michael Maughan, Edward Herderick

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In-Situ Powder-Directed Energy Process Control for Additively Manufactured Multi-Layer, Functionally Graded Components

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About the Presenter

- Calvin M. Downey (calvin.downey@inl.gov);
  - BS Mechanical Engineering, Worcester Polytechnic Institute, 2019
  - MS Thesis Student at University of Idaho in Materials Science and Engineering focused on Additive Manufacturing
  - Experiment Design Engineer at Idaho National Laboratory (INL)
  - Research interests include; nuclear reactor experiment design, additive manufactured (AM) materials for nuclear appplications, coating technologies

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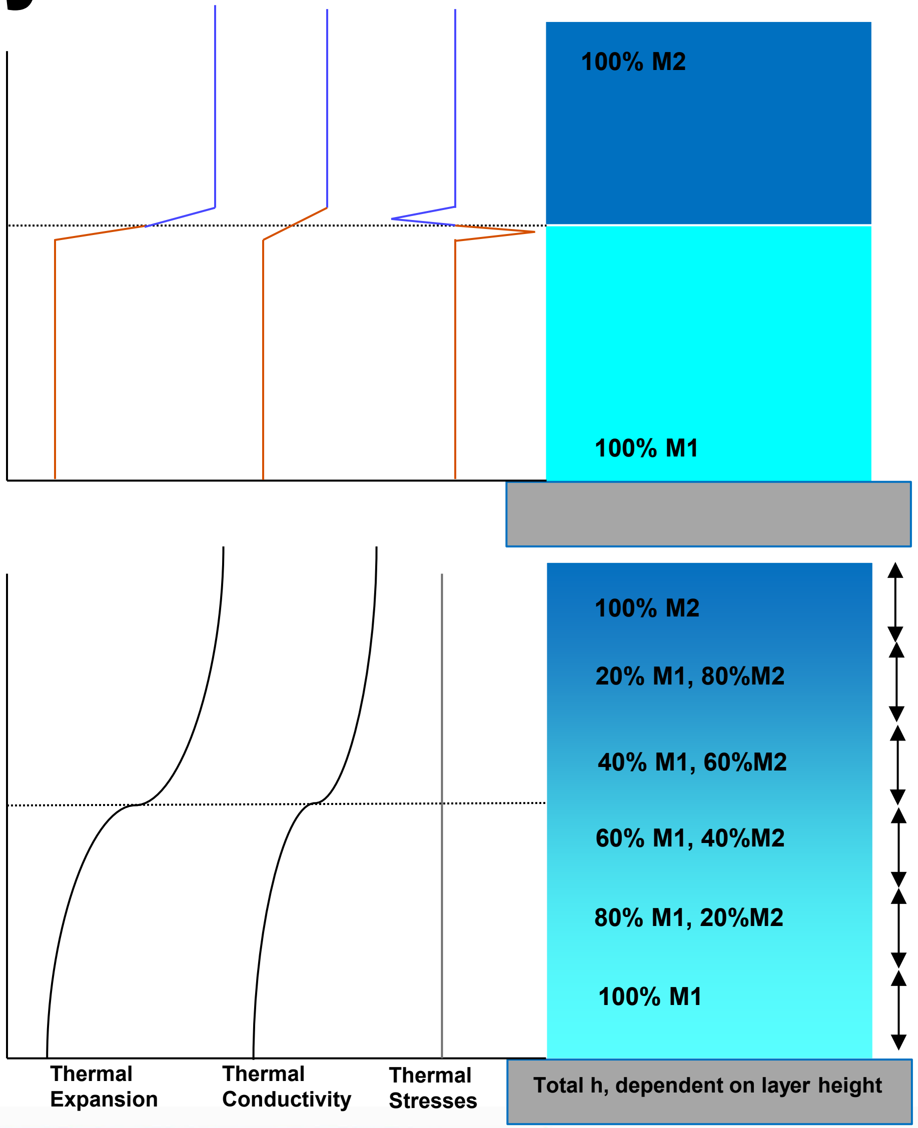
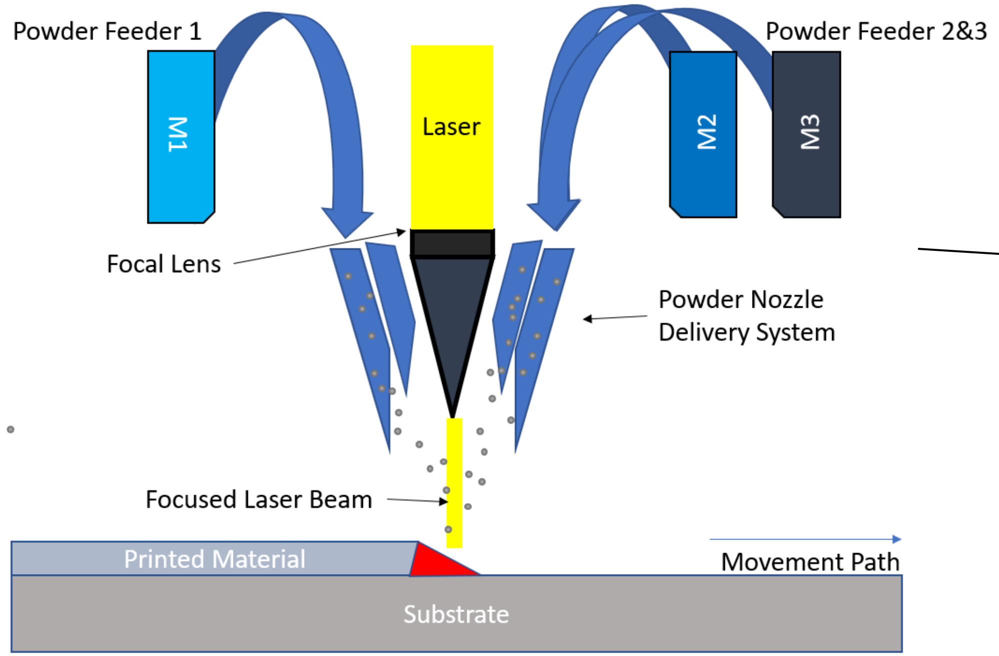
Introduction to AM Functionally Graded Materials

- **Challenges of coatings and claddings in harsh environments due to:**
  - **Erosion, Oxidation**
  - **Delamination (thermal stress)**
  - **External mechanical, radiation damage**
- **Functionally Graded Materials (FGMs) show many benefits including:**
  1. **Reduce thermal stress in material (thermal barrier coatings)**
  2. **Joining of dissimilar materials**
  3. **Application specific corrosion resistance and surface resistance to mechanical wear**
  4. **Diffusion barriers**

Powder-fed DED with multiple independent feeders allows for AM FGMs, and provides robust control over process parameters to optimize microstructure and performance [1]. With the mentioned benefits, FGMs have a great opportunity for applications in energy applications as a strategy to increase plant efficiency and lifetime [2].



AM Powder-DED System Used in This Study

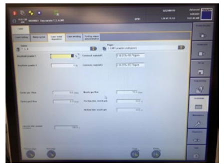


Model	Trumpf TruLaser Cell 3000
Atmosphere	Argon
Chamber Pressure	0.500 kPa
Carrier Gas Flow Rate	4 lpm
Center Purge Pressure	25 lpm
Laser Type	2 kW Nd:Yag
Wavelength	1064 nm
Laser spot size	~1 mm
Controller	Siemens 828D

AM Process Flow Parameters and In-situ Control

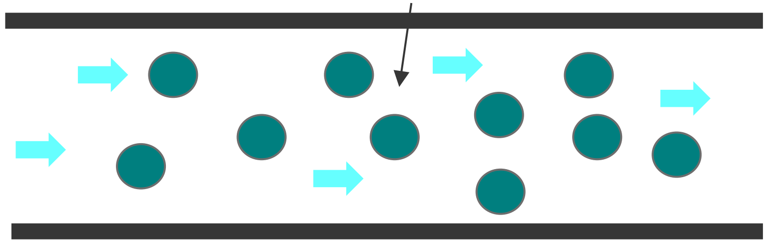
Powder-DED controls: laser power, scan speed, powder flow rate into melt pool which controls: Energy Density, Linear Mass Density.

Optimization of suitable parameters studied for powder-DED. Bulk builds parameters held constant, however carrier gas flow had to be adjusted in-situ for the different materials:

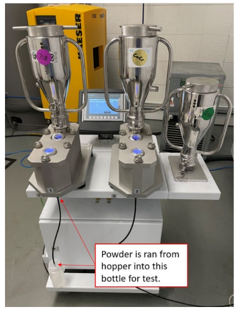


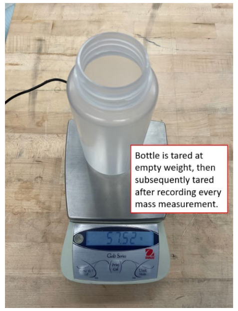
Material Properties (Density, Powder Mesh, Humidity, Temperature) Affect Flowability

Carrier Gas Flow Rate Amplitude



Collected Grams/Unit Time





Multiple Trials of Various Amplitude; Regression

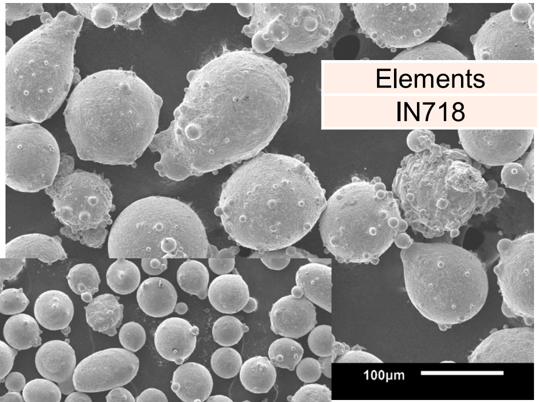
Independent Flow Rates for Each Powder Material

Material:	Carrier Gas Flow:	Nozzle Flow:	Amplitude:
IN718:	718: 9L/min	718: 15L/min	718: 16%
SS316L:	316L: 9L/min	316L: 15L/min	316L: 20%
CoCr:	CoCr: 9L/min	CoCr: 15L/min	CoCr: 20%

Target: 6.79g/min

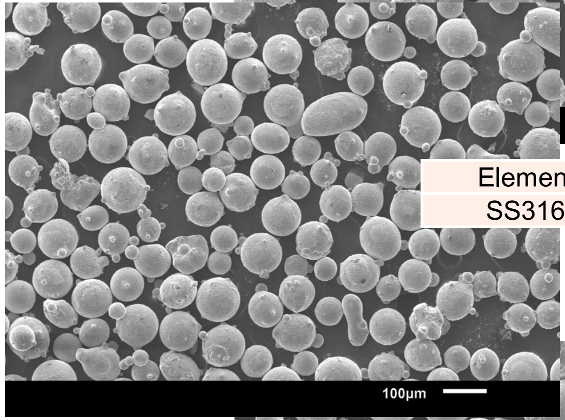
Materials and Methods

AM DED Powders



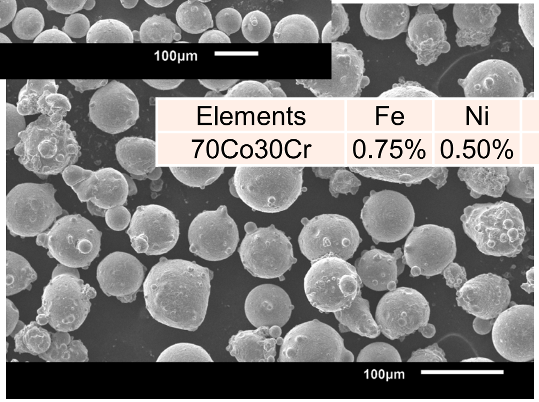
Elements	Fe	Ni	Cr	Mo	Nb	Ti
IN718	14%	55%	21%	3.30%	5.50%	1.20%

-45+105 Mesh



Elements	Fe	Ni	Cr	Mo	Mn	Si	C
SS316L	62.25%	14%	18%	3%	2%	0.75%	0.03%

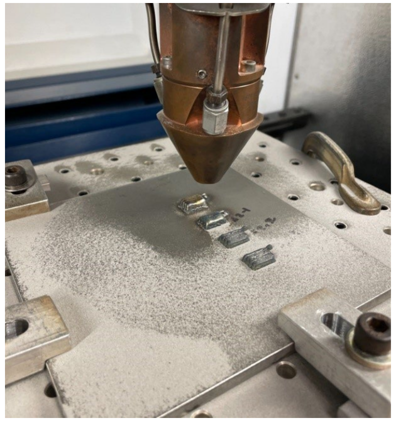
-45+105 Mesh



Elements	Fe	Ni	Cr	Mo	Co	Mn	Si	Ti	C
70Co30Cr	0.75%	0.50%	30%	5%	62%	1%	1%	0.10%	0.16%

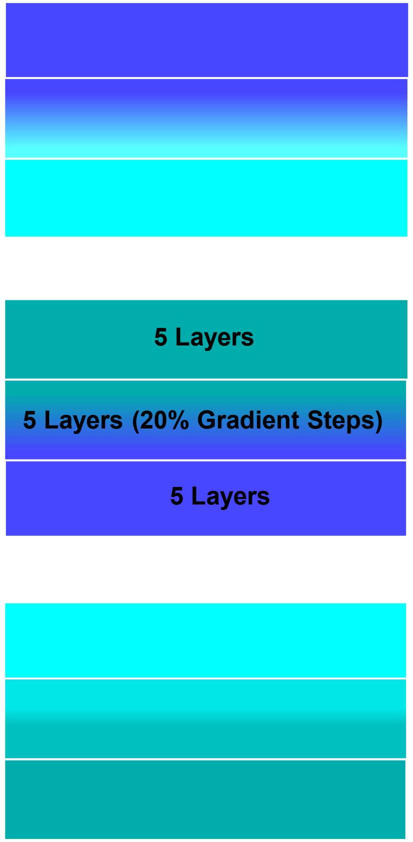
-45+106 Mesh

AM Bulk Builds



500W, 20ipm

Each configuration of the three materials graded to each other in ~20% grades

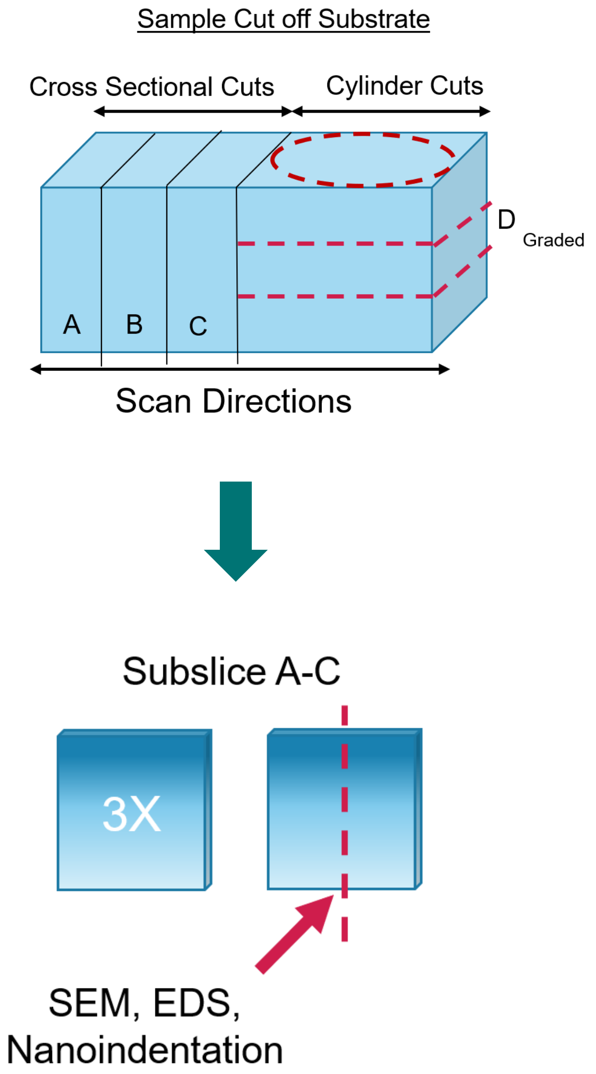


AM Sample Plan

Materials and Methods

Sample Preparation:

1. **Electrical Discharge Machining (EDM) to cut sample cross-sections**
2. **Puck mount with crystal bond**
3. **Sanded to 1200 grit**
4. **Polished to 0.05μm with alumina suspension**
5. **Electropolishing with oxylic acid at 4V potential**

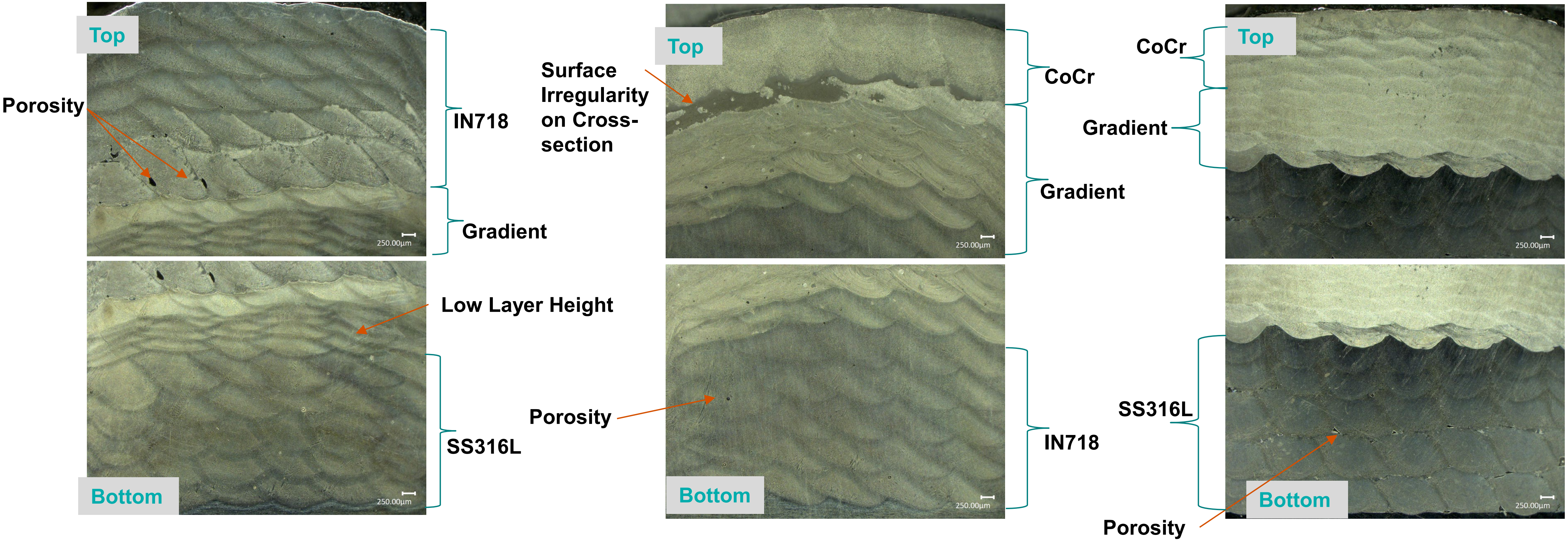


Resulting Bulk Builds

B1 Build; IN718 → SS316L

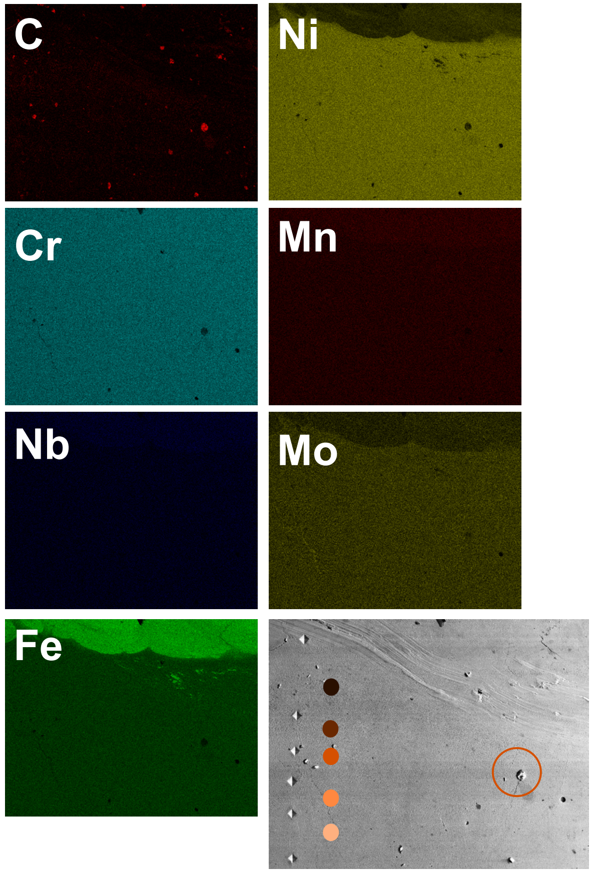
B2 Build; IN718 → 70Co30Cr

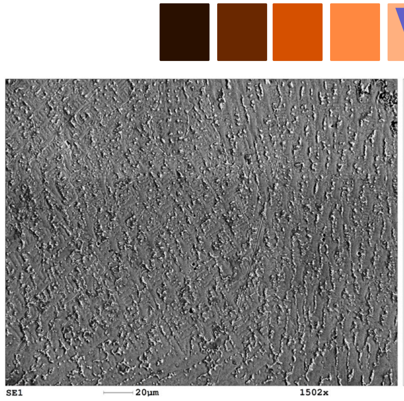
B3 Build; SS316L → 70Co30Cr



Resulting Compositions B1

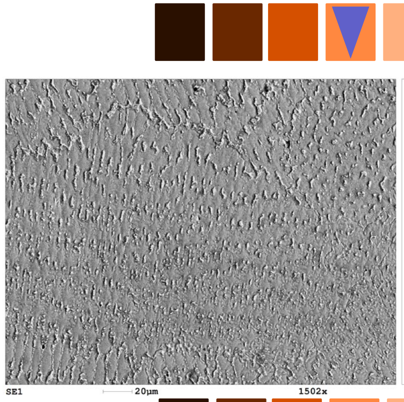
B1 Build; IN718 → SS316L
EDS 5 Gradient Layers





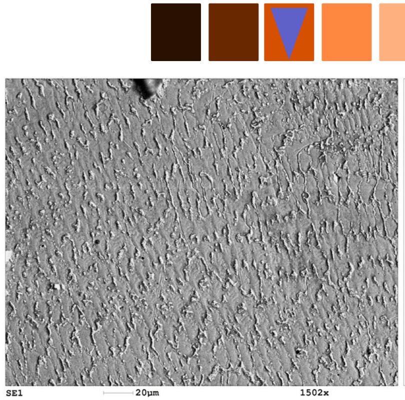
EDS Quantitative Results

Element	Wt%	At%
CK	4.69	19.35
SiK	0.10	0.17
NbL	4.65	2.48
MoL	3.81	1.97
TiK	0.90	0.94
CrK	17.59	16.76
MnK	0.00	0.00
FeK	17.52	15.54
CoK	0.11	0.09
NiK	50.62	42.71



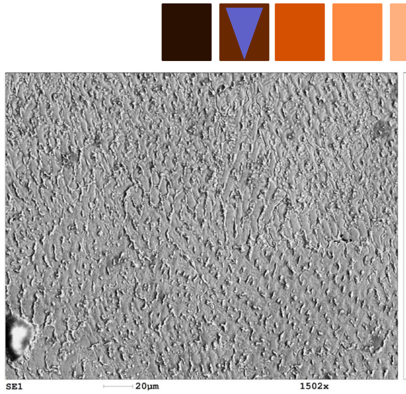
EDS Quantitative Results

Element	Wt%	At%
CK	4.25	17.77
SiK	0.12	0.21
NbL	4.52	2.45
MoL	3.90	2.04
TiK	0.89	0.94
CrK	17.56	16.97
MnK	0.00	0.00
FeK	17.57	15.81
CoK	0.19	0.16
NiK	50.99	43.64



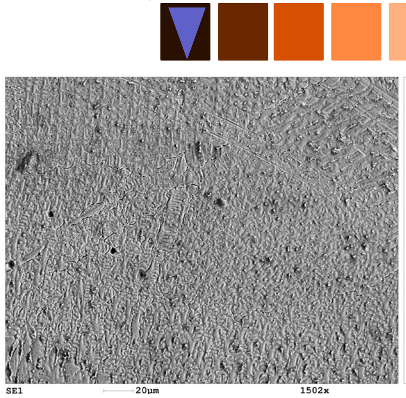
EDS Quantitative Results

Element	Wt%	At%
CK	5.24	21.25
SiK	0.09	0.15
NbL	4.73	2.48
MoL	3.95	2.01
TiK	0.75	0.76
CrK	17.41	16.32
MnK	0.00	0.00
FeK	17.14	14.95
CoK	0.08	0.07
NiK	50.61	42.01



EDS Quantitative Results

Element	Wt%	At%
CK	6.89	26.50
SiK	0.10	0.16
NbL	4.37	2.17
MoL	3.78	1.82
TiK	0.63	0.60
CrK	17.43	15.48
MnK	0.00	0.00
FeK	17.05	14.11
CoK	0.00	0.00
NiK	49.76	39.15



EDS Quantitative Results

Element	Wt%	At%
CK	4.24	17.73
SiK	0.13	0.24
NbL	4.63	2.50
MoL	3.78	1.98
TiK	0.59	0.62
CrK	17.74	17.16
MnK	0.00	0.00
FeK	17.51	15.76
CoK	0.07	0.06
NiK	51.31	43.94

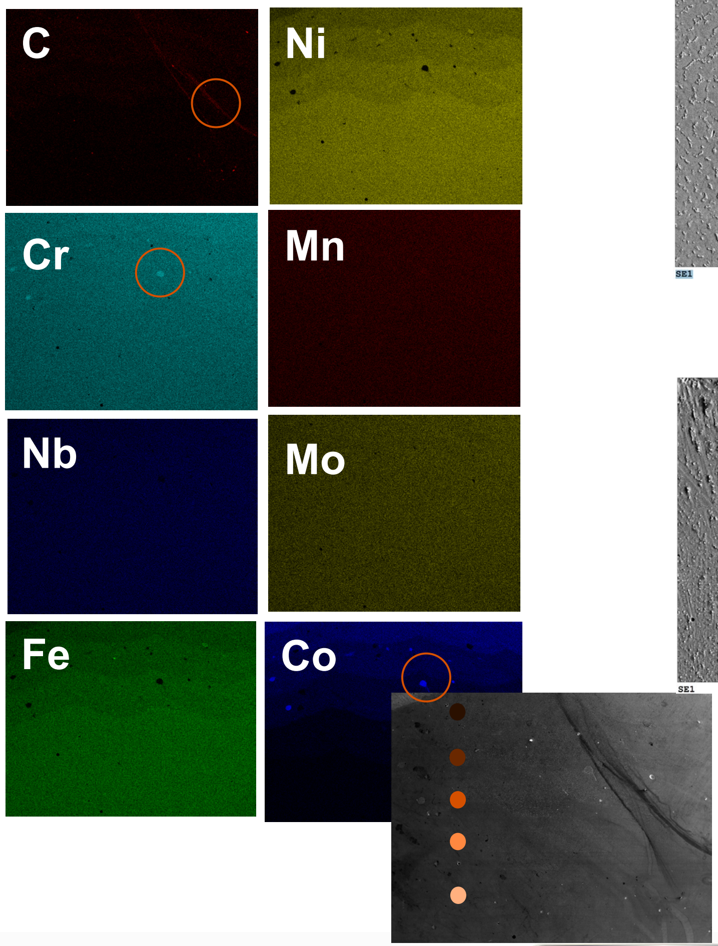
There is no gradient in the material, the 5 middle layers are showing small layer height and only Inconel 718 composition

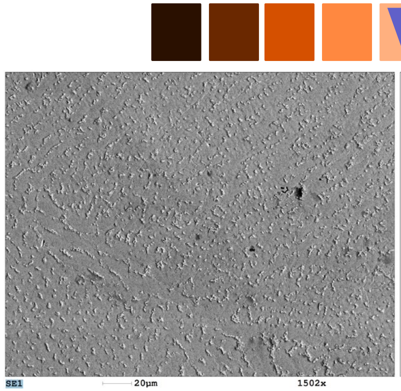
Carbon may be incorrectly identified in EDS as a low Z element

Powder flowability in SS316L (humidity in the powder flow lines). → Low layer height (lower linear mass density)

Resulting Compositions B2

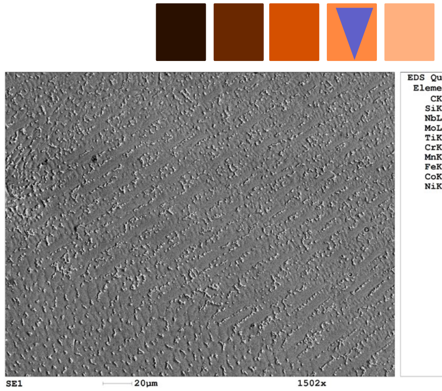
B2 Build; IN718 → 70Co30Cr
EDS 5 Gradient Layers





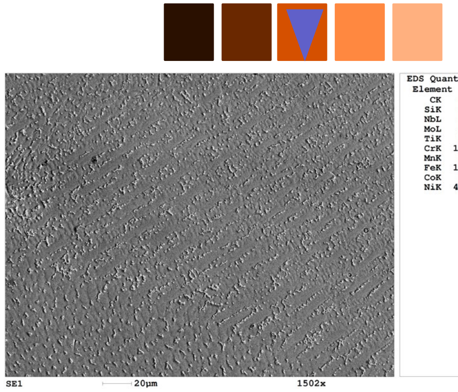
EDS Quantitative Results

Element	Wt%	At%
CK	1.98	8.97
SiK	0.13	0.25
NbL	4.77	2.79
MoL	4.08	2.31
TiK	0.83	0.95
CrK	17.97	18.80
MnK	0.00	0.00
FeK	17.79	17.33
CoK	0.20	0.18
NiK	52.25	48.42



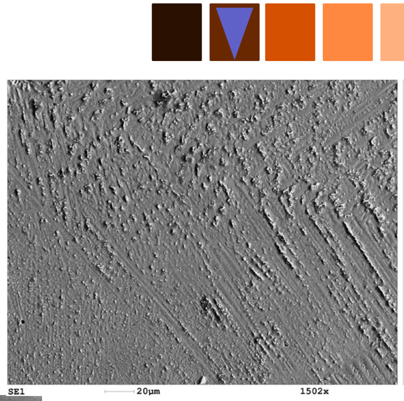
EDS Quantitative Results

Element	Wt%	At%
CK	2.29	10.25
SiK	0.16	0.32
NbL	4.58	2.65
MoL	3.98	2.23
TiK	0.85	0.95
CrK	18.49	19.09
MnK	0.00	0.00
FeK	17.27	16.61
CoK	2.21	2.01
NiK	50.17	45.89



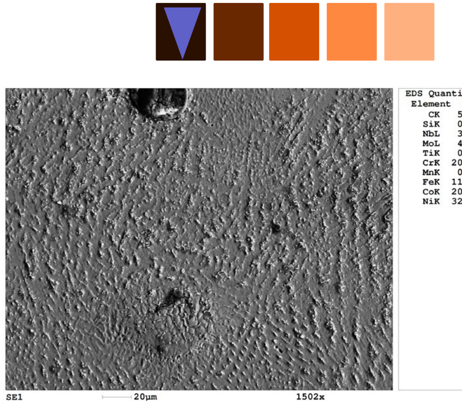
EDS Quantitative Results

Element	Wt%	At%
CK	4.02	16.94
SiK	0.16	0.28
NbL	4.29	2.34
MoL	4.12	2.17
TiK	0.73	0.77
CrK	18.91	18.41
MnK	0.00	0.00
FeK	15.12	13.71
CoK	8.36	7.18
NiK	44.29	38.19



EDS Quantitative Results

Element	Wt%	At%
CK	4.23	17.68
SiK	0.27	0.49
NbL	3.80	2.05
MoL	4.29	2.24
TiK	0.54	0.56
CrK	20.16	19.45
MnK	0.00	0.00
FeK	13.23	11.88
CoK	15.05	12.81
NiK	38.42	32.83

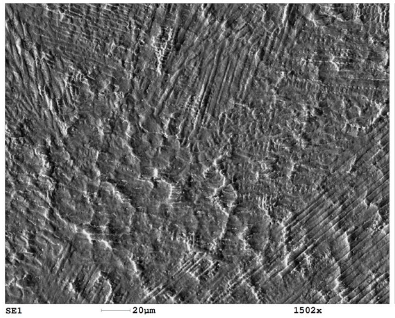


EDS Quantitative Results

Element	Wt%	At%
CK	5.02	20.35
SiK	0.76	1.33
NbL	3.69	1.94
MoL	4.65	2.36
TiK	0.54	0.55
CrK	20.79	19.49
MnK	0.00	0.00
FeK	11.16	9.74
CoK	20.80	17.20
NiK	32.58	27.05

Layer 5 (layer 10) of gradient showing Ni content decrease to 30%, but predicted should be 12%.

70Co30Cr



EDS Quantitative Results

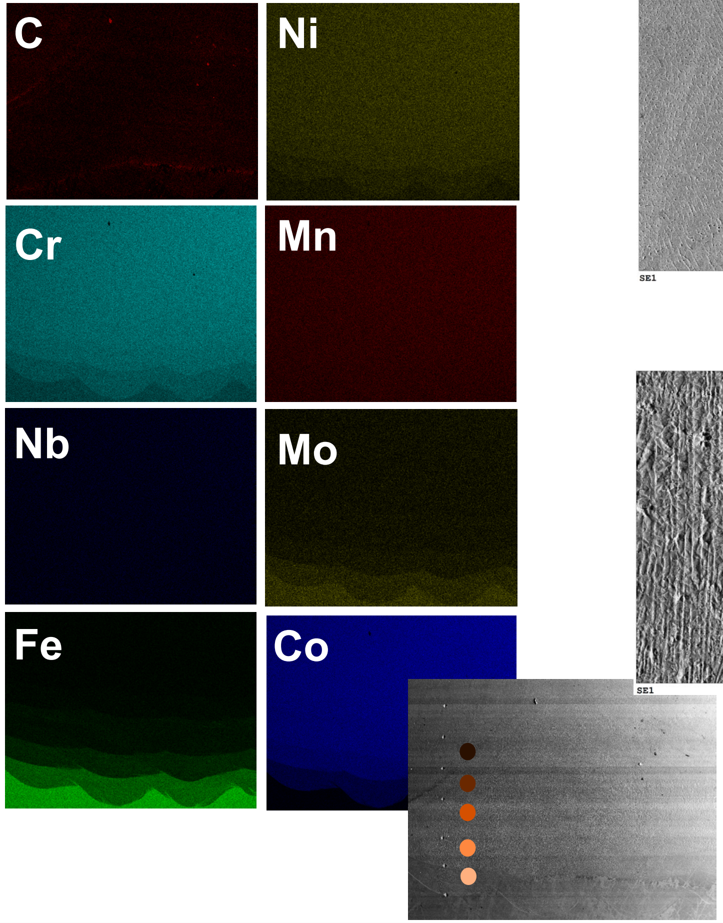
Element	Wt%	At%
CK	3.95	16.46
SiK	0.68	1.20
NbL	0.39	0.21
MoL	5.40	2.82
TiK	0.08	0.09
CrK	27.22	26.21
MnK	0.17	0.15
FeK	1.17	1.05
CoK	57.61	48.96
NiK	3.33	2.84

Layer 13 full CoCr composition

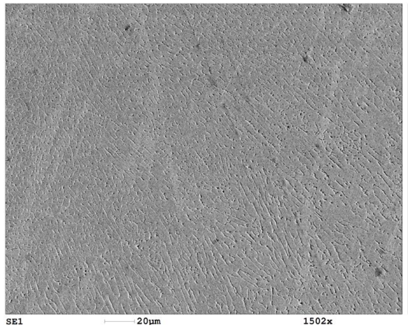
→ **Higher concentrations of carbon at bead boundaries. Chromium rich sigma phases or unmelted CoCr Powder.**

Resulting Compositions B3

B3 Build; SS316L → 70Co30Cr
EDS 5 Gradient Layers



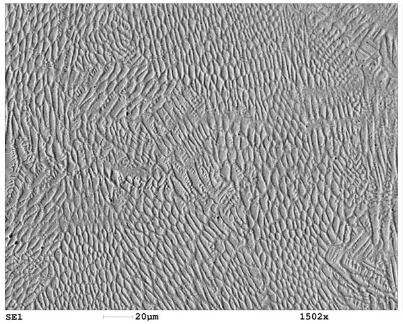
SS316L



EDS Quantitative Results

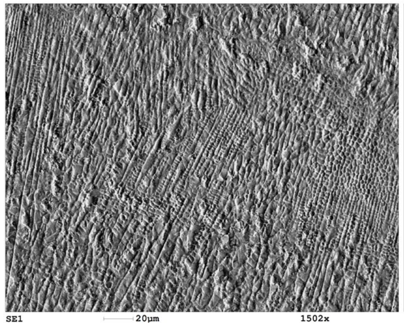
Element	Wt%	At%
CK	4.14	16.70
SiK	0.36	0.63
NbL	0.14	0.07
MoL	2.03	1.02
TiK	0.05	0.05
CrK	16.46	15.33
MnK	0.97	0.86
FeK	66.05	57.27
CoK	0.00	0.00
NiK	9.79	8.07

Layer 1 (layer 6) of gradient showing Fe content to be 35%, but predicted should be 50%.



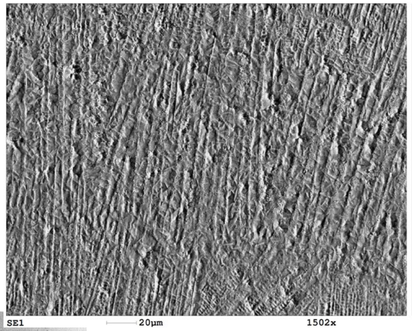
EDS Quantitative Results

Element	Wt%	At%
CK	4.40	17.81
SiK	0.50	0.86
NbL	0.13	0.07
MoL	3.42	1.73
TiK	0.05	0.05
CrK	21.89	20.45
MnK	0.58	0.52
FeK	34.94	30.39
CoK	28.93	23.84
NiK	5.16	4.27



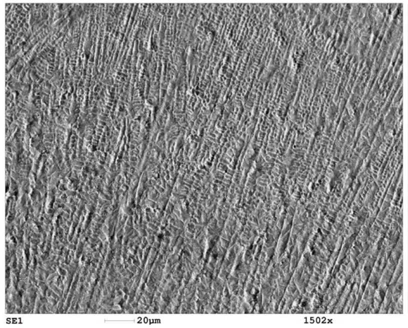
EDS Quantitative Results

Element	Wt%	At%
CK	5.69	22.32
SiK	0.76	1.27
NbL	0.28	0.14
MoL	4.96	2.43
TiK	0.03	0.03
CrK	25.47	23.07
MnK	0.22	0.19
FeK	11.97	10.10
CoK	48.83	39.02
NiK	1.78	1.43



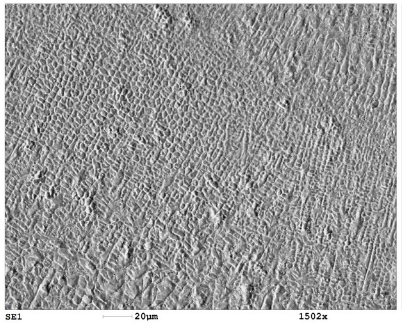
EDS Quantitative Results

Element	Wt%	At%
CK	5.79	22.69
SiK	0.68	1.14
NbL	0.03	0.02
MoL	5.32	2.61
TiK	0.05	0.05
CrK	27.01	24.47
MnK	0.09	0.07
FeK	3.72	3.14
CoK	56.77	45.37
NiK	0.54	0.43



EDS Quantitative Results

Element	Wt%	At%
CK	7.97	29.30
SiK	0.70	1.10
NbL	0.10	0.05
MoL	5.46	2.52
TiK	0.06	0.06
CrK	26.51	22.53
MnK	0.14	0.11
FeK	1.57	1.24
CoK	57.14	42.84
NiK	0.34	0.26



EDS Quantitative Results

Element	Wt%	At%
CK	8.34	30.34
SiK	0.73	1.13
NbL	0.13	0.06
MoL	5.38	2.45
TiK	0.03	0.03
CrK	26.87	22.57
MnK	0.07	0.06
FeK	0.57	0.44
CoK	57.78	42.83
NiK	0.11	0.08

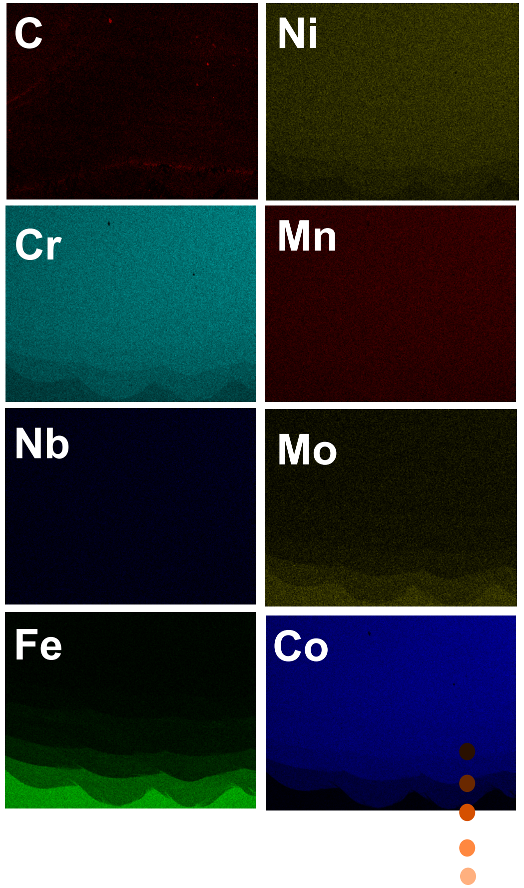
Layer 5 (layer 10) of gradient showing Fe content decrease to 0.5%, but predicted should be 13%.

Carbon is incorrectly being identified in the higher layers due to low Z, likely reducing the reported Co content.

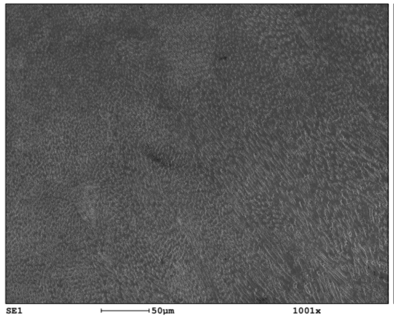
Resulting Compositions C2

B3 Build; SS316L → 70Co30Cr

EDS 5 Gradient Layers

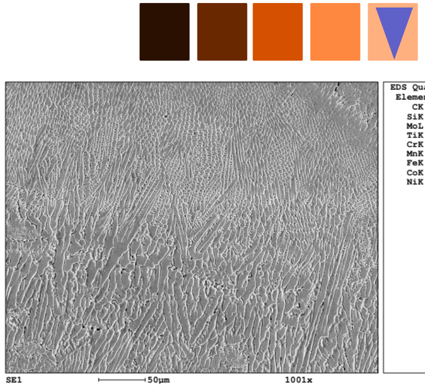


SS316L



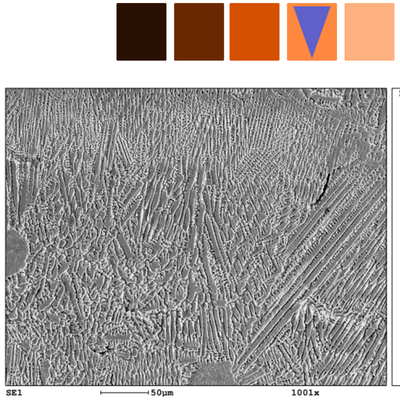
EDS Quantitative Results

Element	Wt%	At%
CK	0.71	3.20
SiK	0.40	0.76
MoL	2.04	1.15
TiK	0.00	0.00
CrK	16.94	17.69
MnK	0.83	0.82
FeK	68.81	66.88
CoK	0.00	0.00
NiK	10.27	9.50



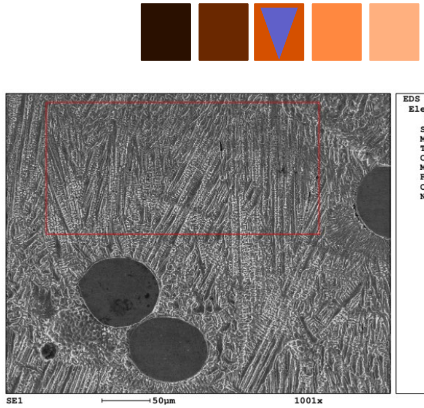
EDS Quantitative Results

Element	Wt%	At%
CK	0.87	3.93
SiK	0.33	0.64
MoL	2.05	1.16
TiK	0.01	0.02
CrK	17.71	18.41
MnK	0.55	0.54
FeK	65.44	63.33
CoK	1.63	1.50
NiK	11.39	10.48



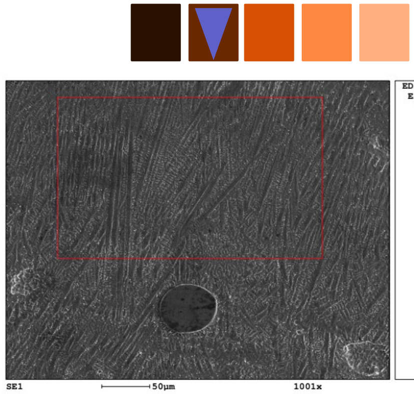
EDS Quantitative Results

Element	Wt%	At%
CK	0.89	4.05
SiK	0.29	0.55
MoL	2.36	1.34
TiK	0.11	0.12
CrK	19.15	20.00
MnK	0.41	0.41
FeK	53.90	52.40
CoK	8.40	7.73
NiK	14.49	13.40



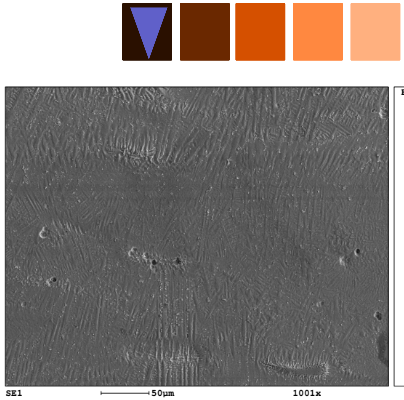
EDS Quantitative Results

Element	Wt%	At%
CK	1.83	8.12
SiK	0.28	0.53
MoL	4.15	2.30
TiK	0.20	0.22
CrK	21.37	21.90
MnK	0.20	0.19
FeK	31.66	30.21
CoK	19.99	18.07
NiK	20.33	18.45



EDS Quantitative Results

Element	Wt%	At%
CK	1.92	8.54
SiK	0.41	0.77
MoL	4.96	2.76
TiK	0.21	0.24
CrK	21.60	22.16
MnK	0.03	0.03
FeK	25.05	23.93
CoK	22.51	20.38
NiK	23.32	21.19

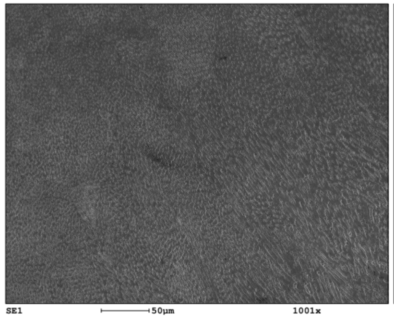


EDS Quantitative Results

Element	Wt%	At%
CK	1.81	8.12
SiK	0.44	0.85
NbL	2.34	1.36
MoL	4.49	2.53
TiK	0.27	0.31
CrK	21.91	22.76
MnK	0.00	0.00
FeK	19.52	18.88
CoK	25.13	23.03
NiK	24.09	22.16
TaL	0.00	0.00

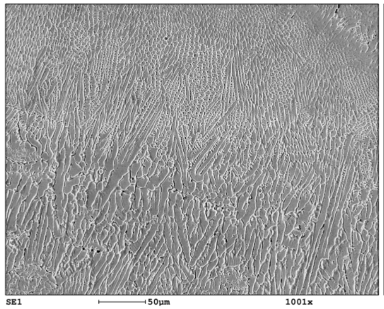
Carbon is incorrectly being identified in the higher layers due to low Z, likely reducing the reported Co content.

SS316L



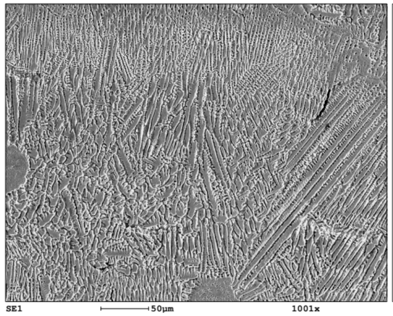
EDS Quantitative Results

Element	Wt%	At%
CK	0.71	3.20
SiK	0.40	0.76
MoL	2.04	1.15
TiK	0.00	0.00
CrK	16.94	17.69
MnK	0.83	0.82
FeK	68.81	66.88
CoK	0.00	0.00
NiK	10.27	9.50



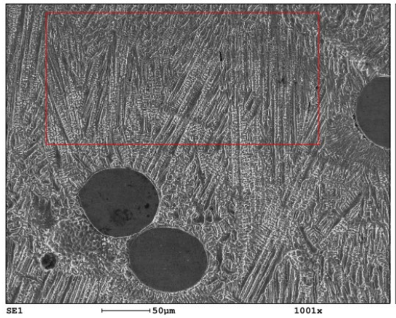
EDS Quantitative Results

Element	Wt%	At%
CK	0.87	3.93
SiK	0.33	0.64
MoL	2.05	1.16
TiK	0.01	0.02
CrK	17.71	18.41
MnK	0.55	0.54
FeK	65.44	63.33
CoK	1.63	1.50
NiK	11.39	10.48



EDS Quantitative Results

Element	Wt%	At%
CK	0.89	4.05
SiK	0.29	0.55
MoL	2.36	1.34
TiK	0.11	0.12
CrK	19.15	20.00
MnK	0.41	0.41
FeK	53.90	52.40
CoK	8.40	7.73
NiK	14.49	13.40



EDS Quantitative Results

Element	Wt%	At%
CK	1.83	8.12
SiK	0.28	0.53
MoL	4.15	2.30
TiK	0.20	0.22
CrK	21.37	21.90
MnK	0.20	0.19
FeK	31.66	30.21
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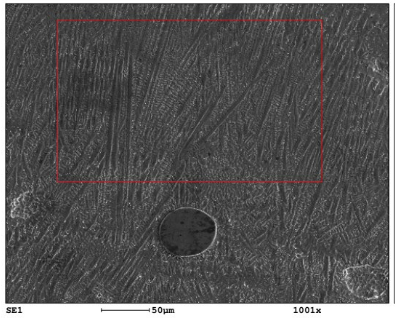

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Element	Wt%	At%
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SiK	0.41	0.77
MoL	4.96	2.76
TiK	0.21	0.24
CrK	21.60	22.16
MnK	0.03	0.03
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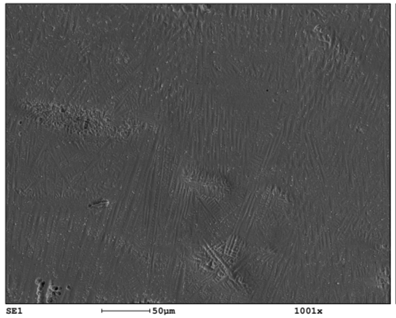
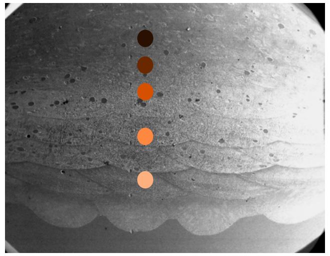
CoK	22.51	20.38
NiK	23.32	21.19



EDS Quantitative Results

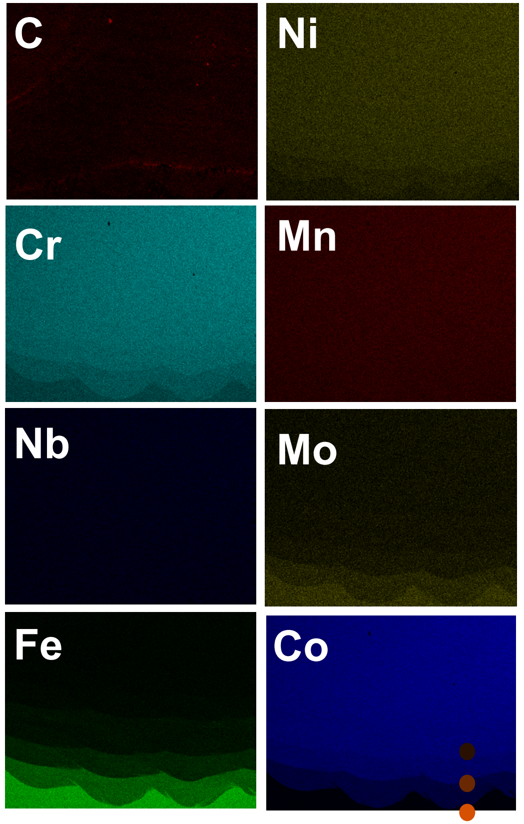
Element	Wt%	At%
CK	1.84	8.24
SiK	0.52	1.01
NbL	2.41	1.40
MoL	4.43	2.49
TiK	0.25	0.29
CrK	22.05	22.87
MnK	0.00	0.00
FeK	18.65	18.01
CoK	25.70	23.52
NiK	24.14	22.18
TaL	0.00	0.00



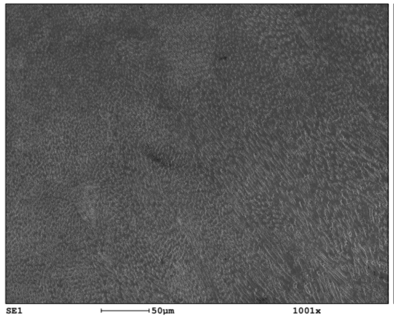
Resulting Compositions C2

B3 Build; SS316L → 70Co30Cr

EDS 5 Gradient Layers

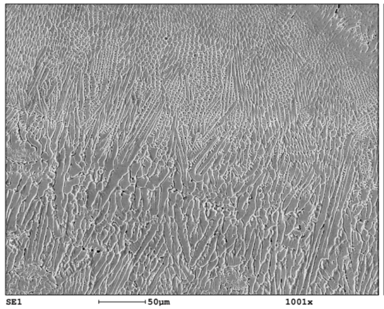


SS316L



EDS Quantitative Results

Element	Wt%	At%
CK	0.71	3.20
SiK	0.40	0.76
MoL	2.04	1.15
TiK	0.00	0.00
CrK	16.94	17.69
MnK	0.83	0.82
FeK	68.81	66.88
CoK	0.00	0.00
NiK	10.27	9.50



EDS Quantitative Results

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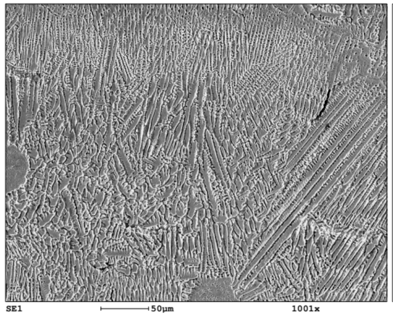
MnK	0.55	0.54
FeK	65.44	63.33
CoK	1.63	1.50
NiK	11.39	10.48



EDS Quantitative Results

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MnK	0.41	0.41
FeK	53.90	52.40
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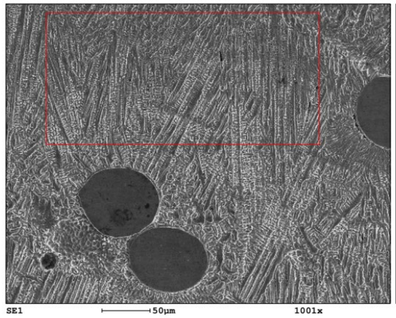

EDS Quantitative Results

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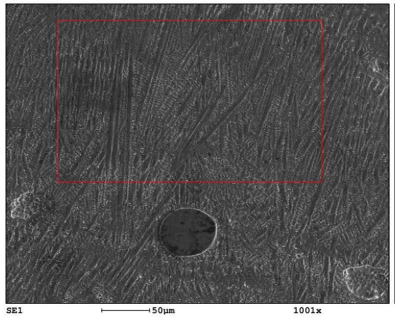
FeK	31.66	30.21
CoK	19.99	18.07
NiK	20.33	18.45



EDS Quantitative Results

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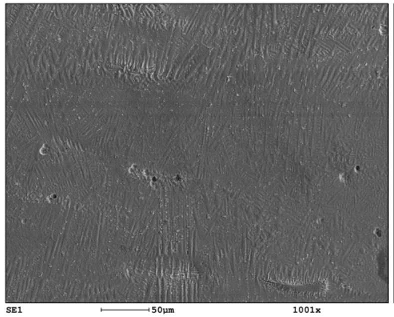
FeK	25.05	23.93
CoK	22.51	20.38
NiK	23.32	21.19



EDS Quantitative Results

Element	Wt%	At%
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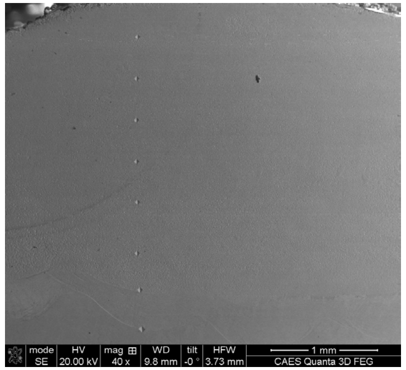
SiK	0.44	0.85
NbL	2.34	1.36
MoL	4.49	2.53
TiK	0.27	0.31
CrK	21.91	22.76
MnK	0.00	0.00
FeK	19.52	18.88
CoK	25.13	23.03
NiK	24.09	22.16
TaL	0.00	0.00

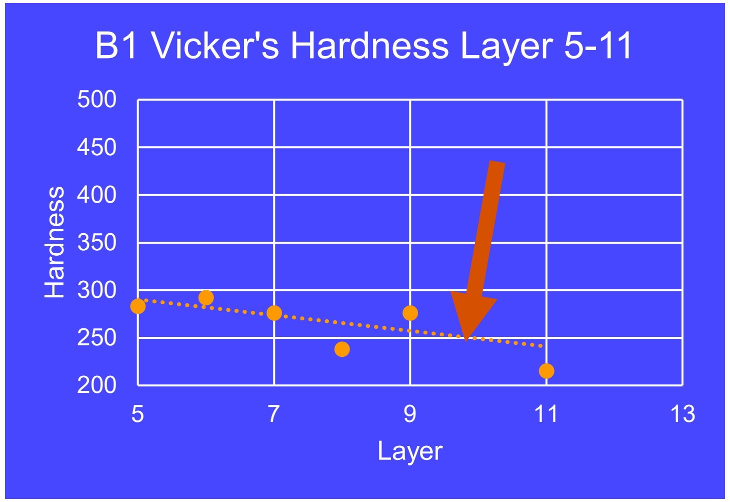
Carbon is incorrectly being identified in the higher layers due to low Z, likely reducing the reported Co content.

Initial Mechanical Properties

- **Vicker's hardness data taken at the bead boundary of every layer in order to both mark the layer boundary and get preliminary mechanical results. Trends match current literature [3,4]**

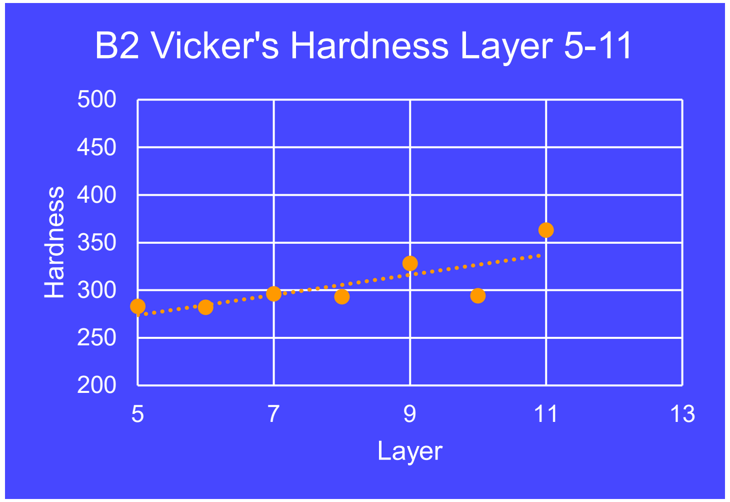


B1 Build; IN718 → SS316L



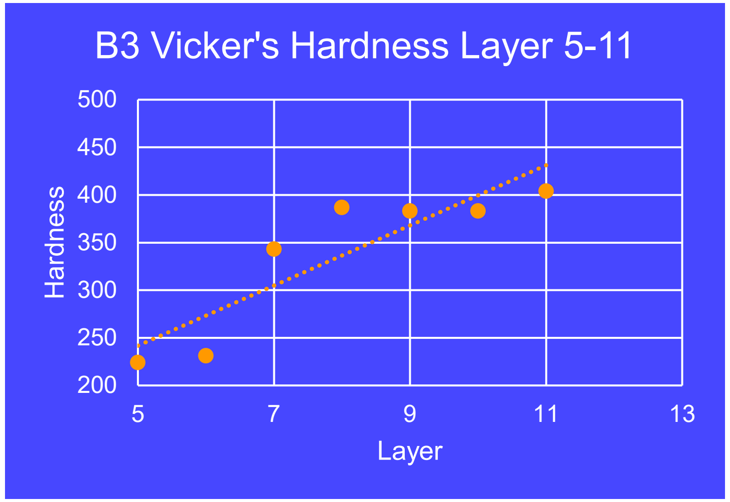
Decrease in Vickers by ~68 at layer

B2 Build; IN718 → 70Co30Cr



Increase by ~80 Vickers

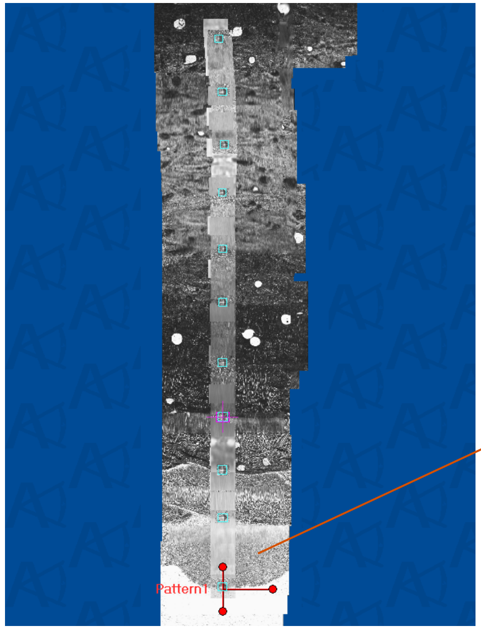
B3 Build; SS316L → 70Co30Cr

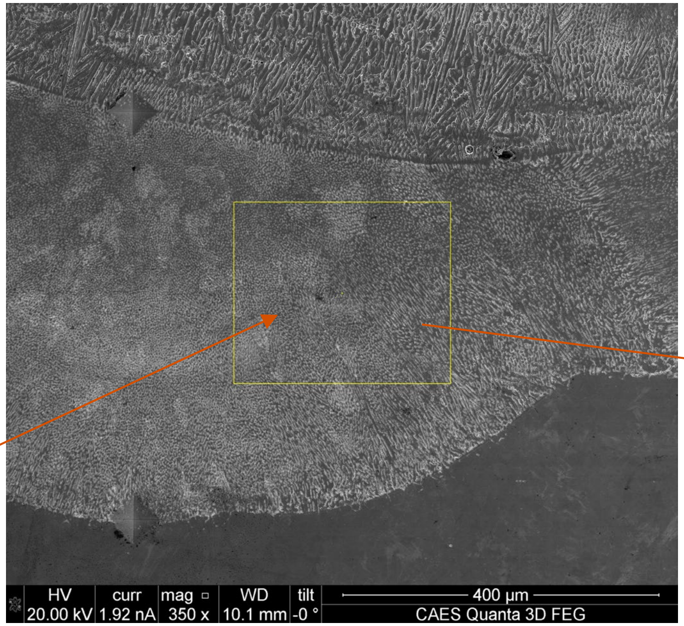


Increase by ~180

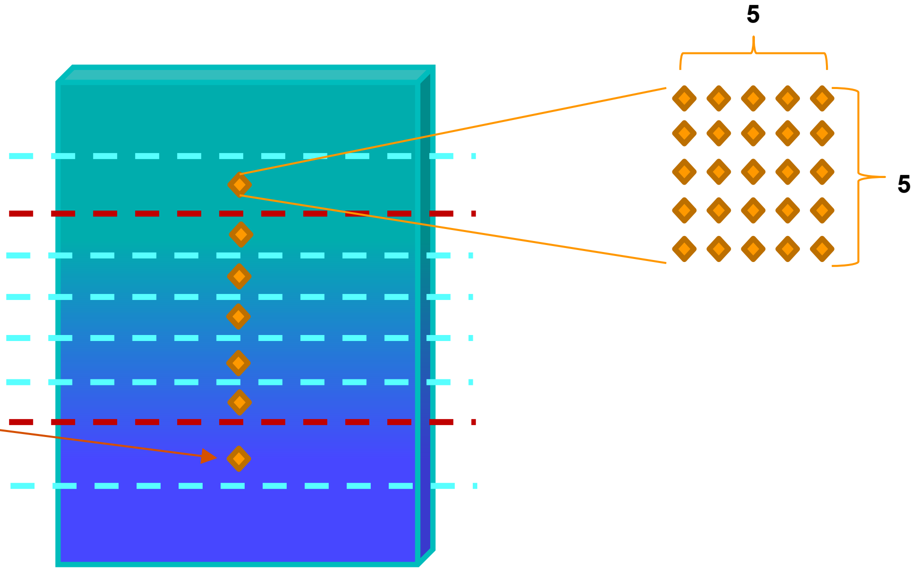
Nanoindentation and Bulk Modulus

Vicker's Hardness Indent Locations





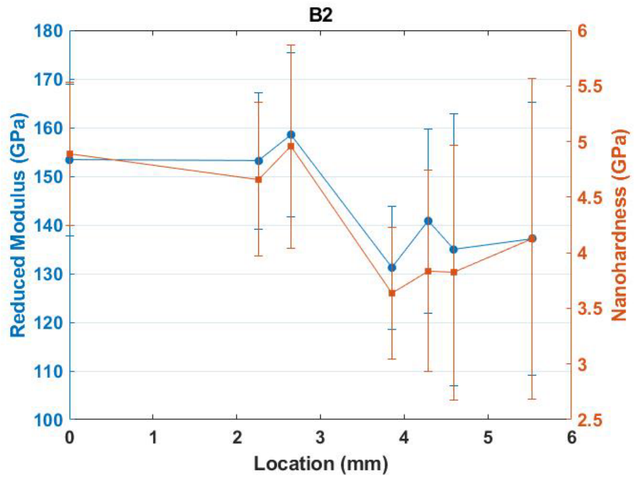
Planned Nanoindentation Area



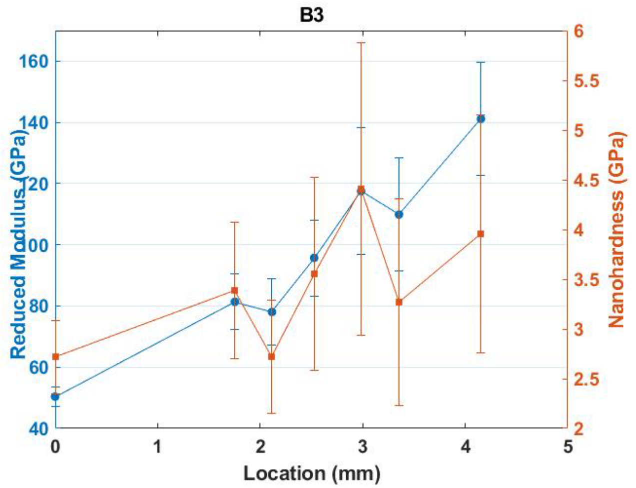
- 5x5 indents per area of interest, approximately in the middle of each graded layer
- 10 **µm** grid spacing for each indent
- 9mN force, 10-20-10 time program

Nanoindentation and Bulk Modulus

B2 Build; IN718 → 70Co30Cr



B3 Build; SS316L → 70Co30Cr



Nanohardness and Microhardness should theoretically show similar trends in a homogenous region

- **B2 Build shows a contrary trend to expected values based on preliminary microhardness. Reasons could include:**
  - **Sample prep issues, programming issues, areas probed have nanograins of varying phase not taken into account in microhardness**

Conclusion

- Multiple configurations of bulk build FGMs were fabricated using powder-DED of three common alloys and in-situ powder feed adjustment.
- Bulk cross-sections were assessed for successful fusion and lack of macroscopic defects (unmelted particles, separation of layers, etc.)
- Graded sections of materials were characterized using EDS and SEM for composition. B1 was a bi-metal build due to issues with material feed, B2 & B3 showed a successful FGM distribution.
- Layers were marked using Vicker's Hardness, preliminary mechanical results align with compositional data and what is expected based on literature.
- Nanoindentation and bulk hardness was performed. Some results inconsistent. Little nanoindentation data has been reported on FGMs and techniques may need improvement for bulk gradient samples to identify trends along complex sample compositions .

- Two configurations of FGMs (IN718→CoCr & SS316:→ CoCr) were fabricated using powder-DED
- FGMs manifested good mechanical results
- Nanoindentation was a valuable tool in acquiring useful preliminary data for FGM performance

Future Work

- **Improve nanoindentation technique. Further characterization of phases present in the material using EBSD and TEM for further composition and microstructural evaluation.**
- **Optimization of printing parameters (adjust hatch spacing, energy density, potentially dynamic process parameter adjustment) to increase homogeneity and performance**
  - **Feed into models for ML and digital twin models for Powder-DED.**
- **Expand work to other alloy systems.**

Special Thanks to:
Edward Hederick, PhD for coordination from the Center for Design and Manufacturing Excellence and Ben Dimarco for his assistance with fabricating bulk samples for this work and providing ample collaboration on the fabrication process. Also to the Center for Advanced Energy Studies for the use of the Materials and Characterization Suite.
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CDME

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Center for Advanced Energy Studies

Questions?

Sources

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3. Mei, Xinliang, et al. “Interfacial Characterization and Mechanical Properties of 316L Stainless Steel/Inconel 718 Manufactured by Selective Laser Melting.” *Materials Science and Engineering: A*, vol. 758, 2019, pp. 185–191., https://doi.org/10.1016/j.msea.2019.05.011.
4. Smoqi, Ziyad, et al. “Process-Structure Relationship in the Directed Energy Deposition of Cobalt-Chromium Alloy (Stellite 21) Coatings.” *Materials & Design*, vol. 197, 2021, p. 109229., https://doi.org/10.1016/j.matdes.2020.109229.