

High-Throughput Rapid Experimental Alloy Development (HT-READ)

Kenneth S. Vecchio

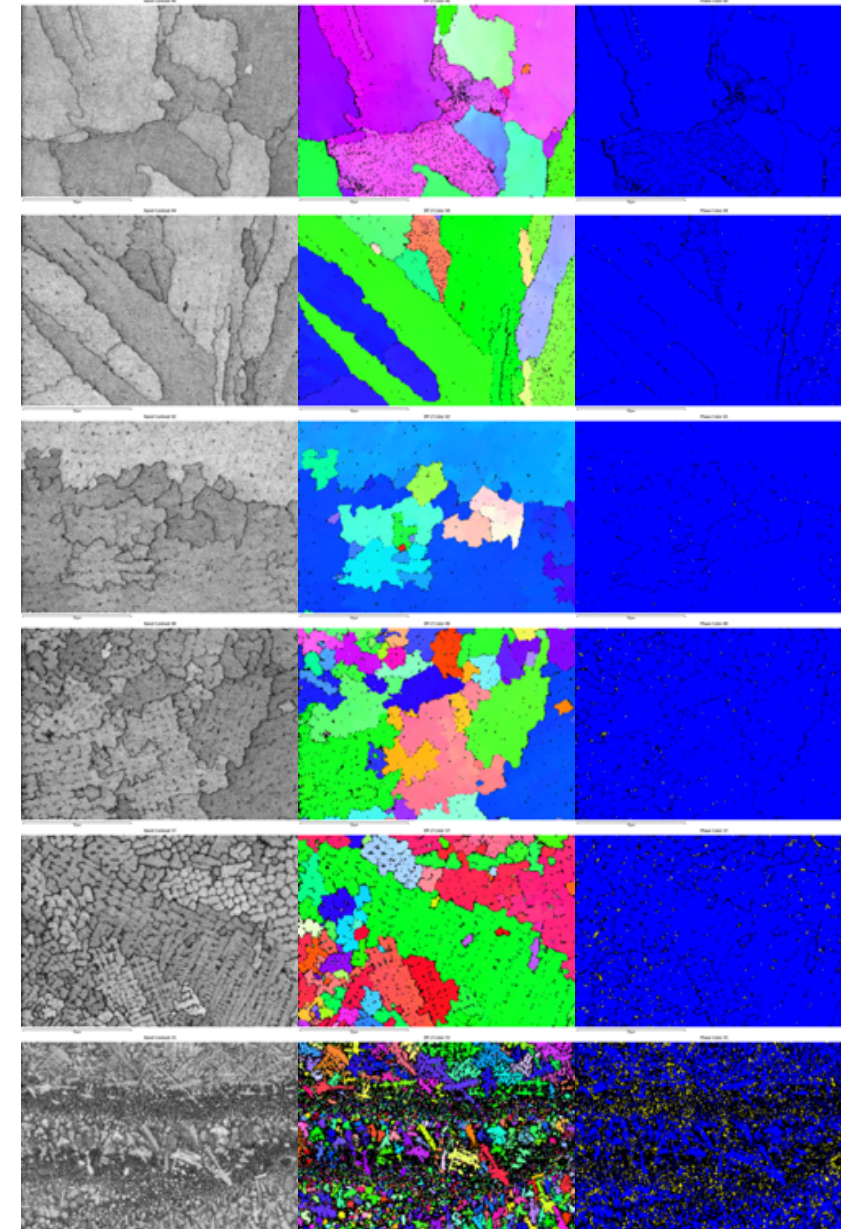
Professor of Materials Engineering, Department of NanoEngineering, UC San Diego

Founding Member of the Materials Science and Engineering Program, UC San Diego

Founding Chair of the NanoEngineering Department, UC San Diego, 2007-2013

Director, NanoEngineering Materials Research Center

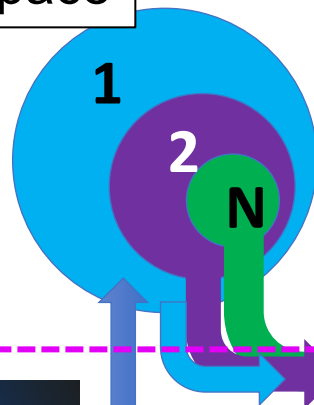
Founder, Scoperta Inc. a computational materials science startup founded in 2007,
acquired by Oerlikon-Metco in 2017



Featurize Compositions

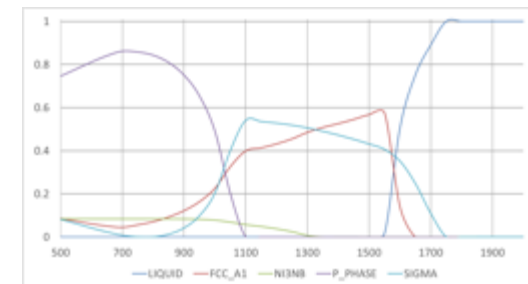
Computation/Modeling

Composition Space

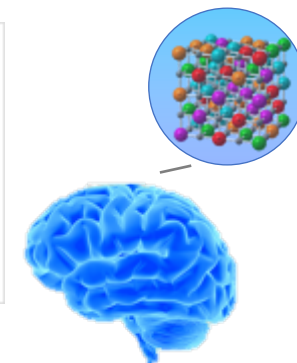


Material	Feature Vector				
Material 1	A_{11}	A_{12}	A_{13}	A_{14}	A_{15}
Material N	A_{N1}	A_{N2}	A_{N3}	A_{N4}	A_{N5}

CALPHAD



ML



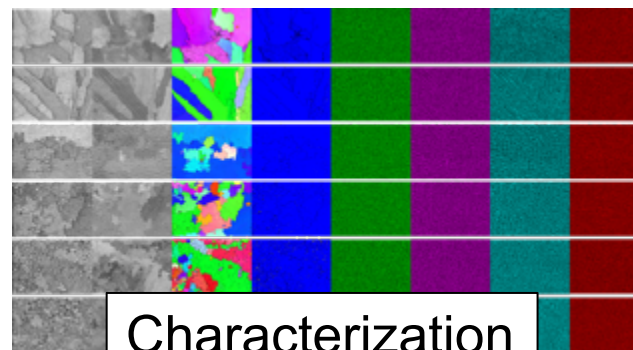
High-Throughput Materials Research Cycle



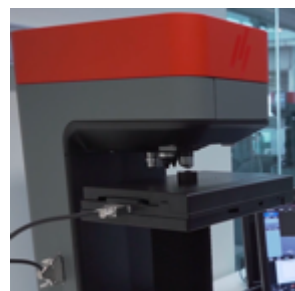
Synthesis



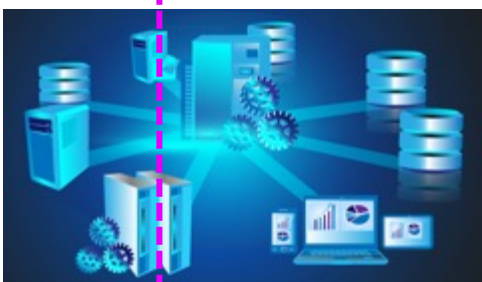
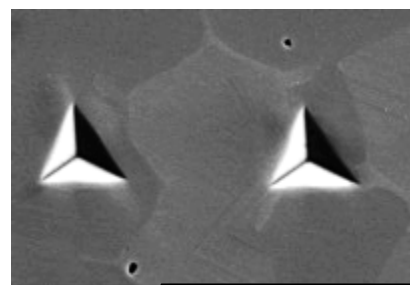
Processing



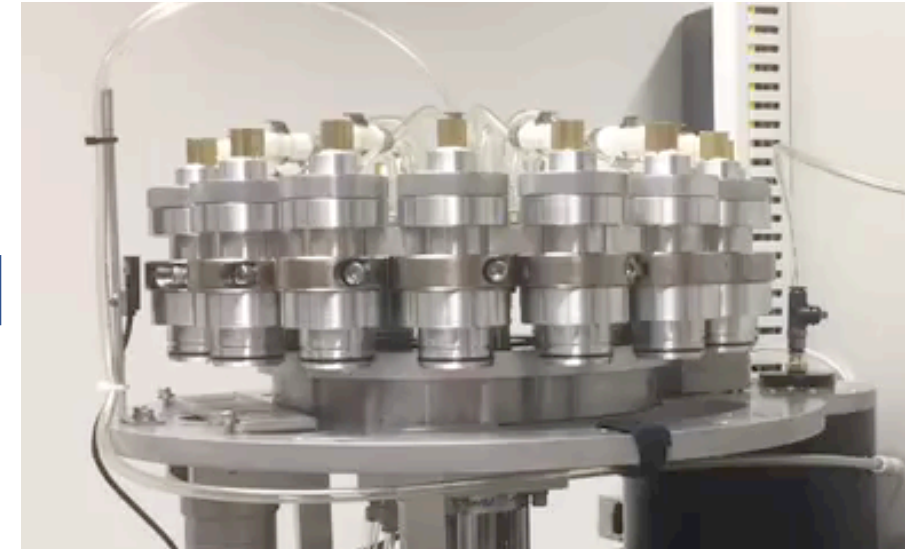
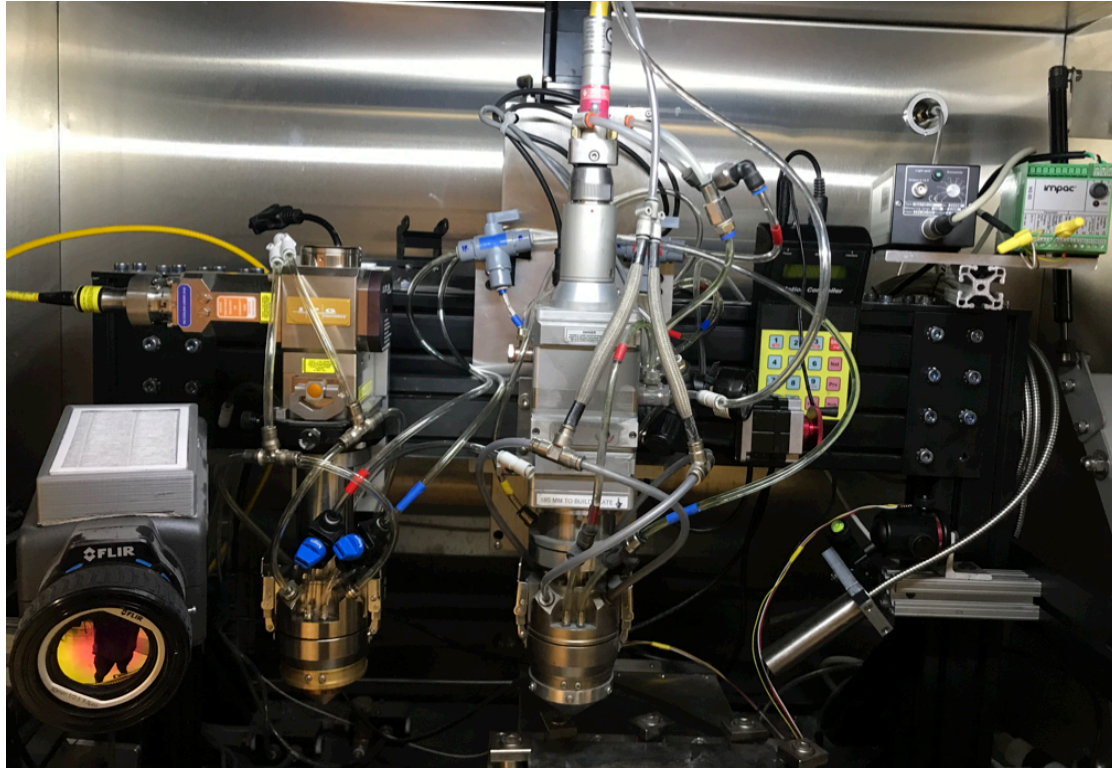
Characterization



Property Measurements

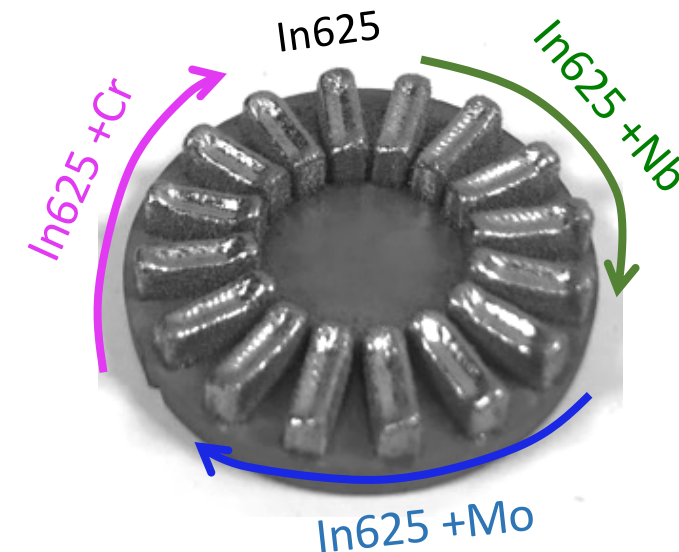


Data Management



16-powder hopper allows for manufacturing of complex sample libraries

Aspirational Goal: Fabricate and Fully Characterize ~100 different bulk alloy compositions in a 24-hour period. 6 Samples with 16 different compositions = 96 alloys. Sample Geometry is critical to high-throughput. Rotation better than translation for fast characterization, particularly SEM-EBSD.

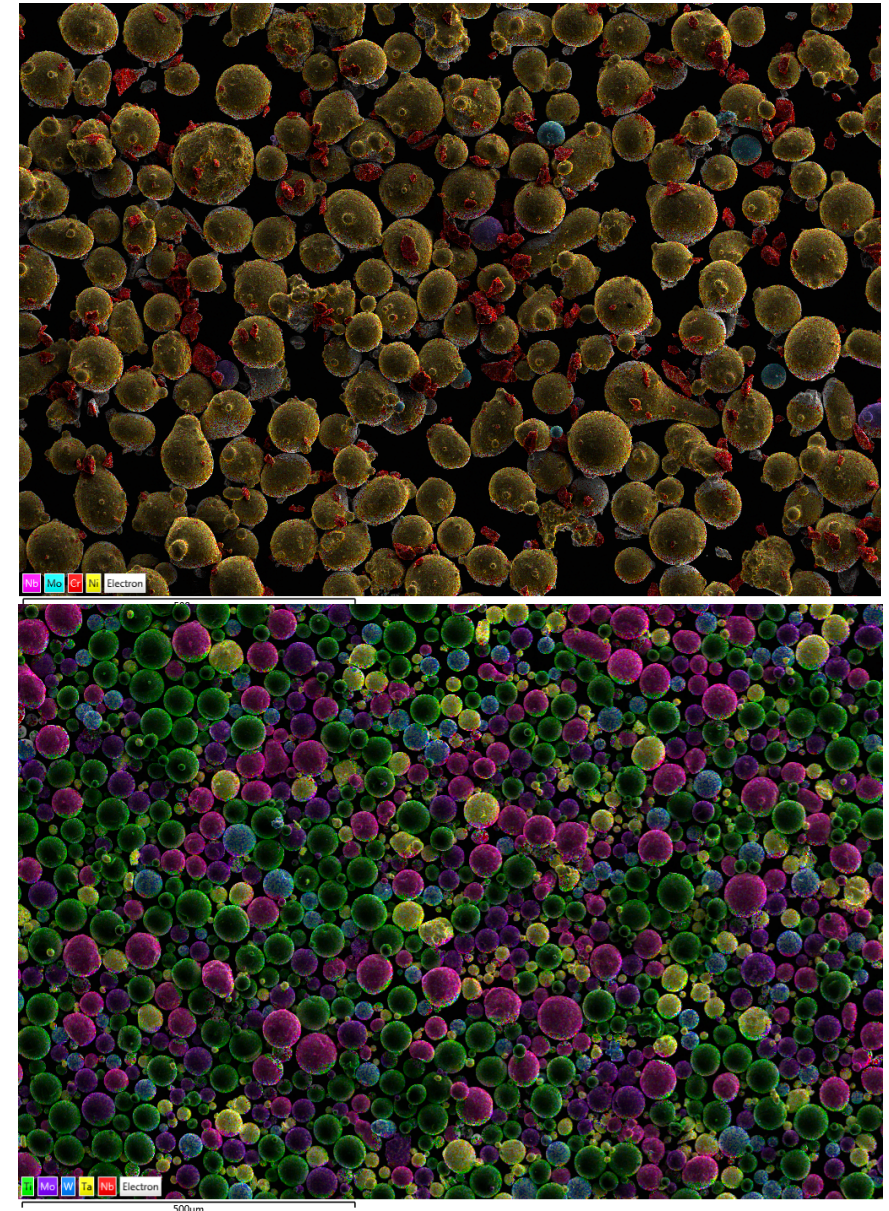


Unlikely high-throughput alloy development can be achieved using powder-bed platforms due to powder handling limitations.

Employing a powder-fed system is much better, but the use of multiple individual powder feeders to 'on-the-fly' blend powders has enormous challenges associated with consistent powder flow, complexity of powder compositions, need for large number of feeders to address all required elements, and perhaps the biggest hurdle is adjusting the feeder flow rates to achieve desired compositions, which again needs to be modified for each new powder used.

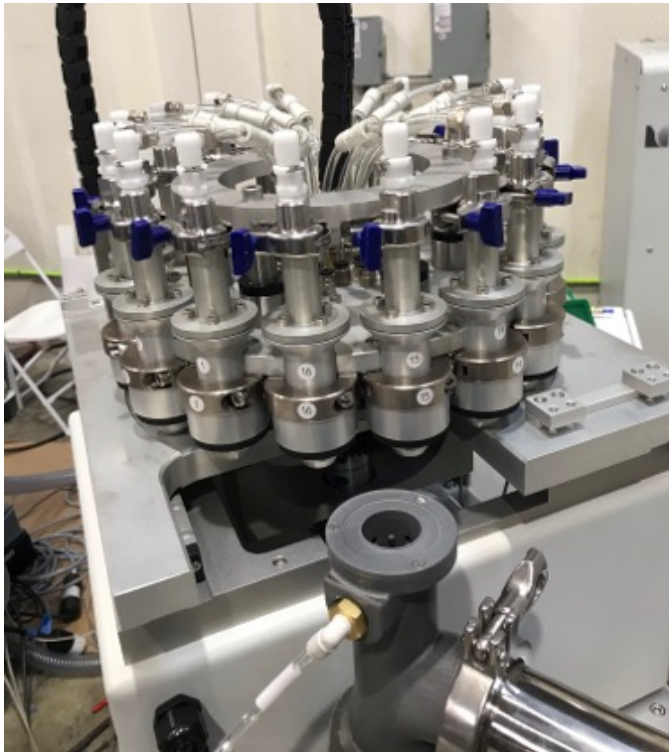
Huge advantages of blended powders in separate vials:

- Powder samples blends can be accurately achieved by weighting powders while dispensing into blending bottles.
- Powders can be easily blended using bottle rollers.
- Entire powder weighting, blending, dispensing into vials can be easily automated using simple robotics.
- Much easier path than trying to solve the 'on-the-fly' feeder system.

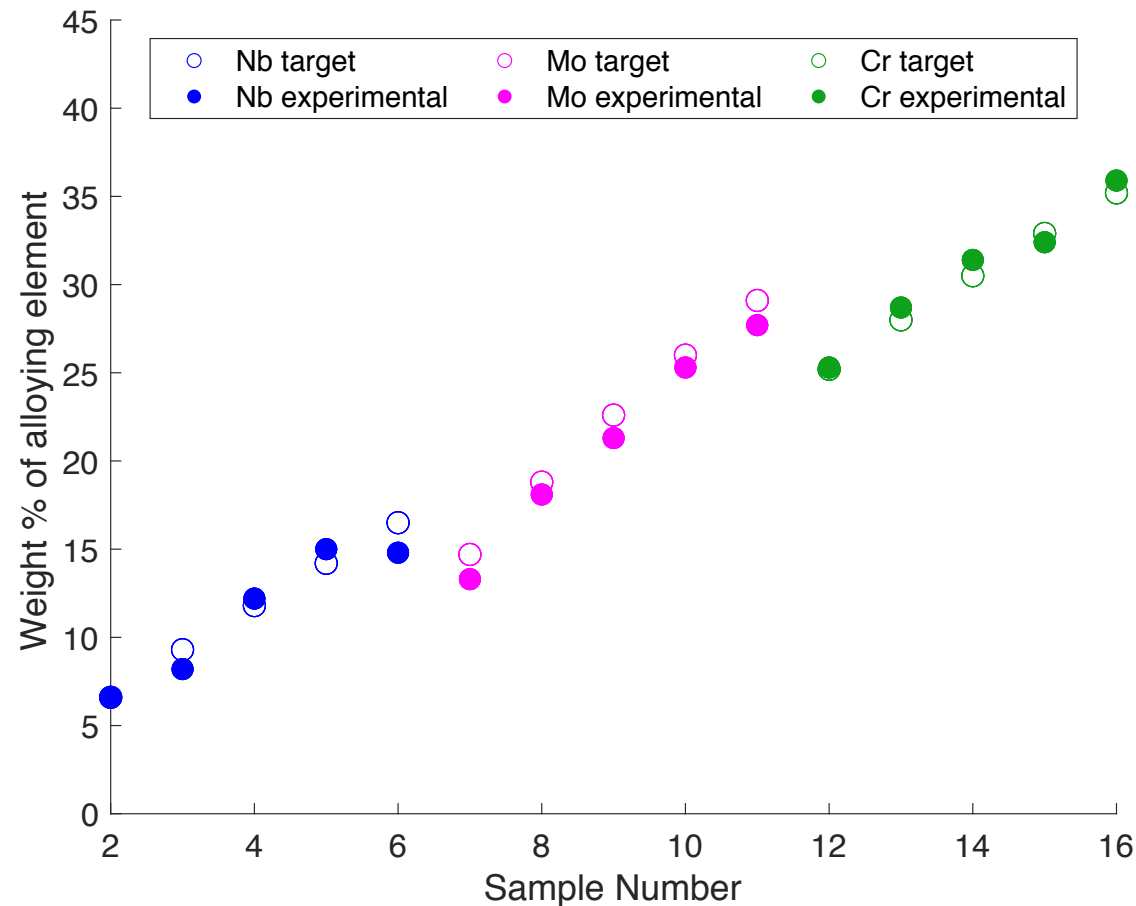


FORMALLOY ADF Alloy Development Feeder

FORMALLOY's ADF Alloy Development Feeder enables the rapid deposition of up to 16 different alloys or alloy blends. Utilizing a revolver-style motion, each powder vial can be accessed and deposited quickly and efficiently to reduce alloy development time by orders of magnitude. The FormAlloy ADF is compatible with the standard FormAlloy PF feeder and can be used in parallel with multiple ADF's or PF feeders to enable interchangeability and seamless integration with the X-Series or L-Series systems. The FormAlloy ADF provides an unprecedented capability for material development and research.



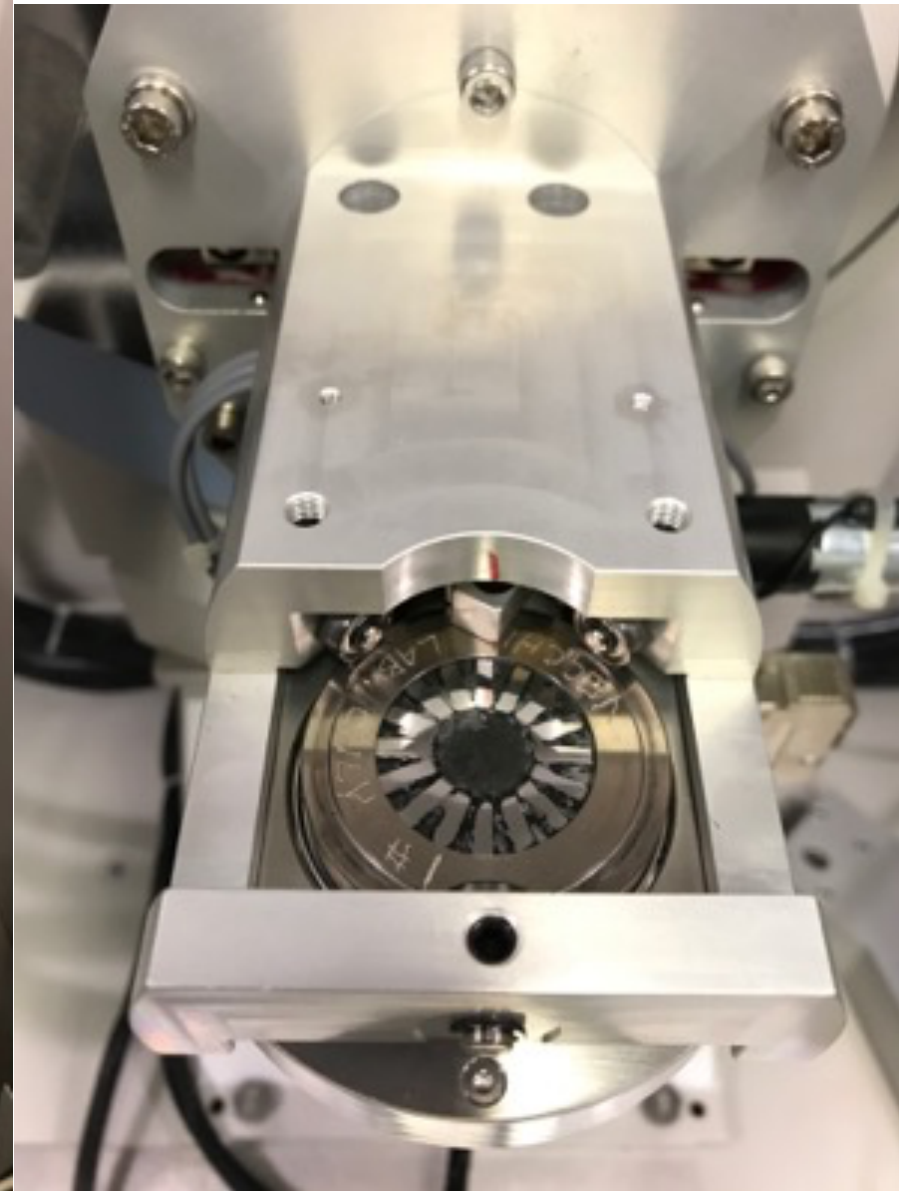
Compositional accuracy depends on advancement of powder handling system.

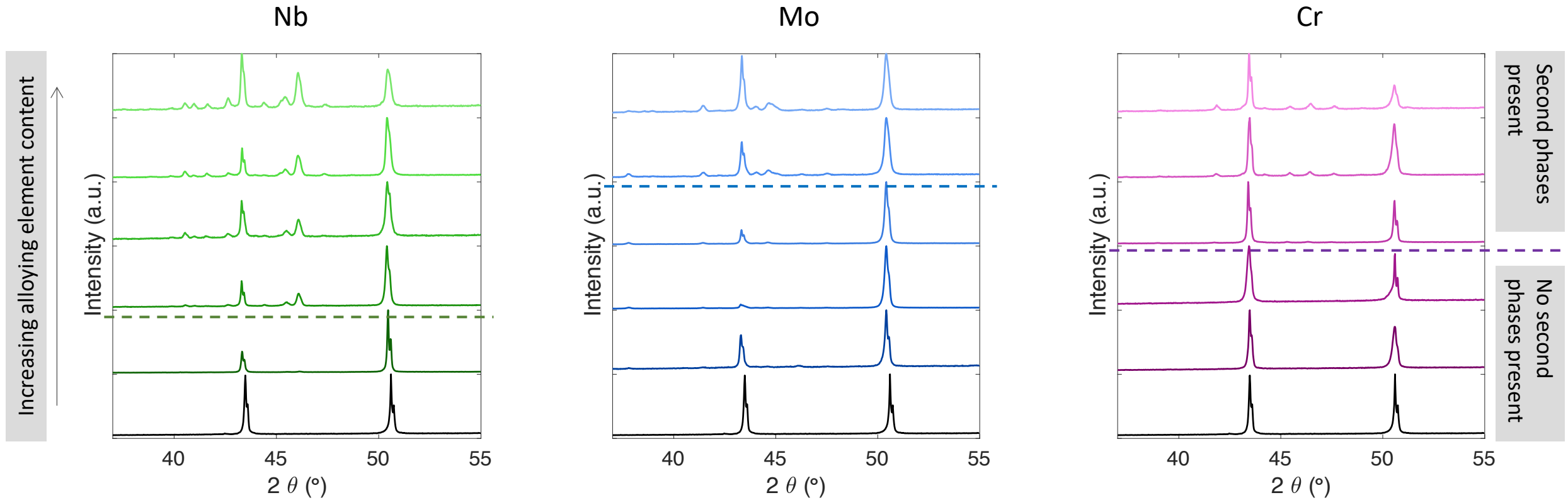


#	Target	Experimental	Difference
1	3.7	3.7	0.0
2	6.6	6.6	0.0
3	9.3	8.2	1.1
4	11.8	12.2	0.4
5	14.2	15.0	0.8
6	16.5	14.8	1.7
7	14.7	13.3	1.4
8	18.8	18.1	0.7
9	22.6	21.3	1.3
10	26.0	25.3	0.7
11	29.1	27.7	1.4
12	25.2	25.3	0.1
13	28.0	28.7	0.7
14	30.5	31.4	0.9
15	32.9	32.4	0.5
16	35.2	35.9	0.7

Compositions of additive samples from EDS analysis are now **consistent** with composition targets.

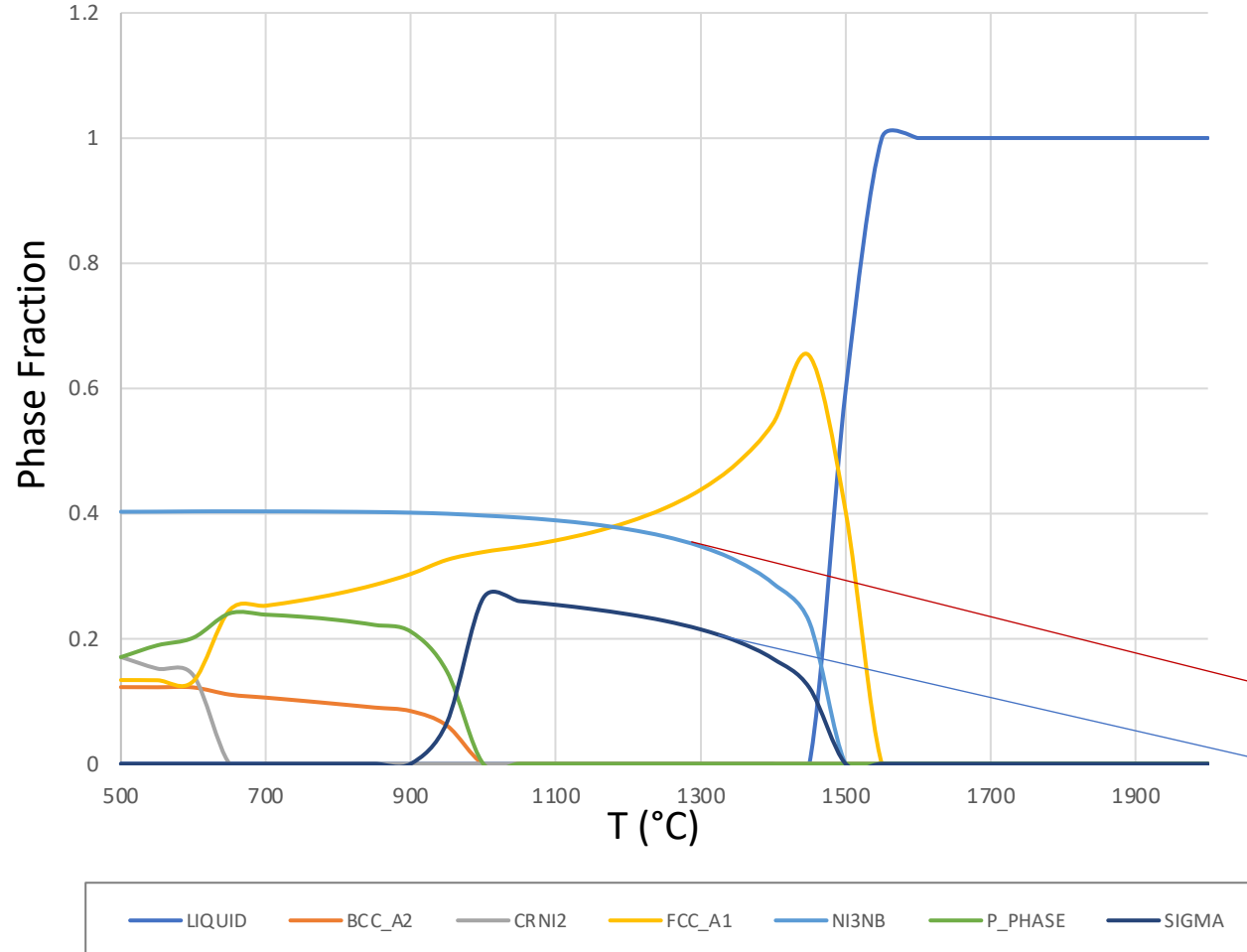
- Panalytical X'Pert Pro XRD has an automated rotational axis
- “wobble” feature allows for negation of texture effects.



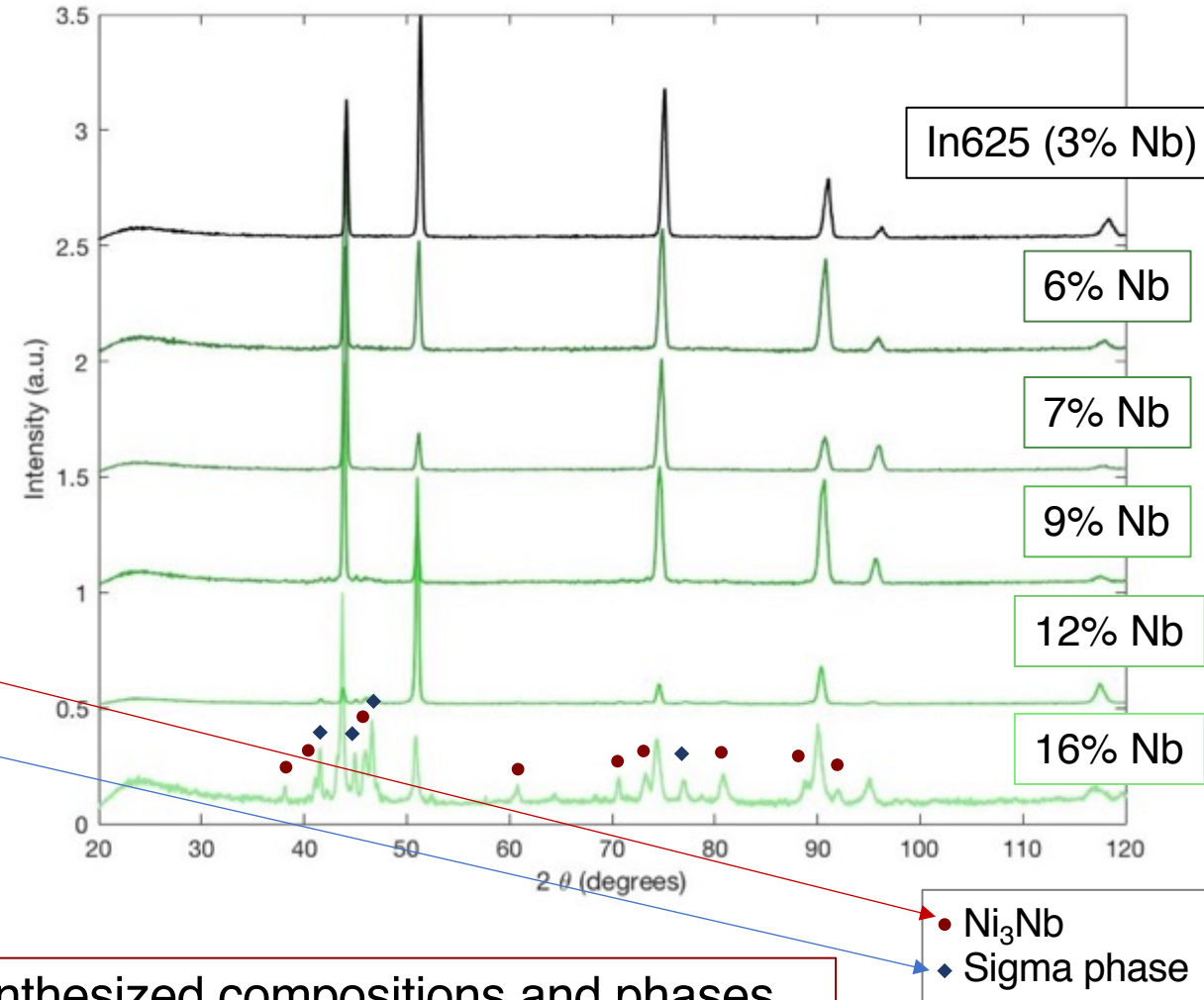


Presence of second phase particles in XRD is used to verify CALPHAD

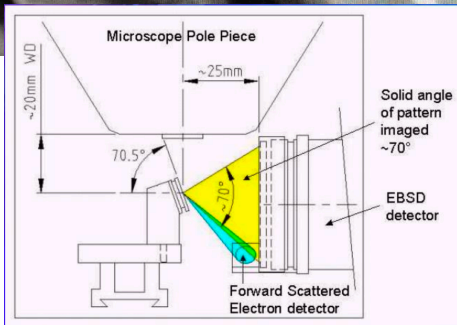
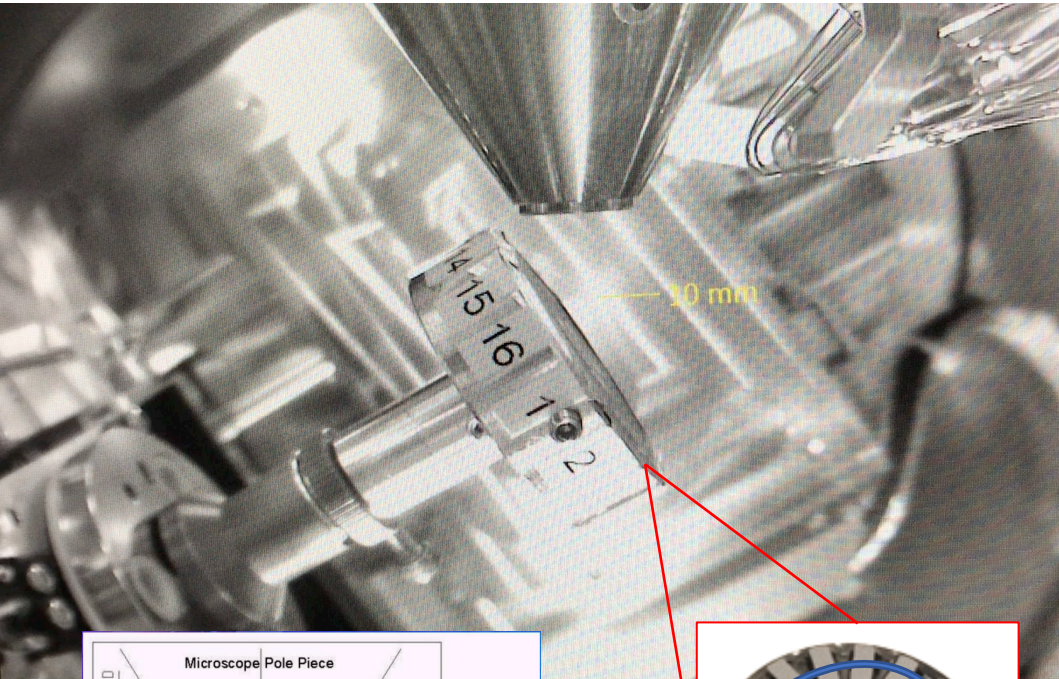
CALPHAD Predictions for 16% Nb:



XRD for In625 + Nb progression:



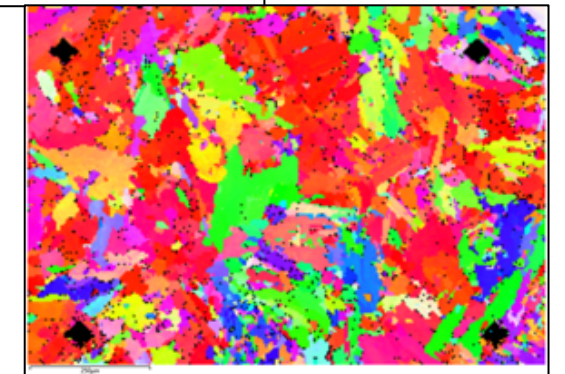
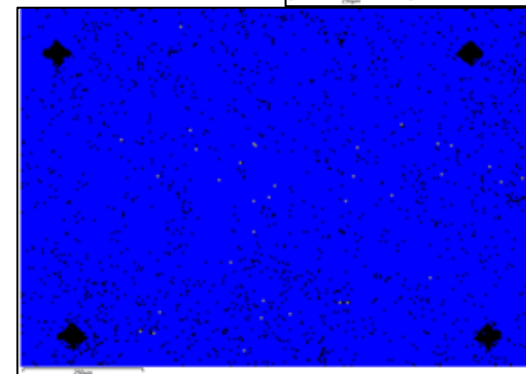
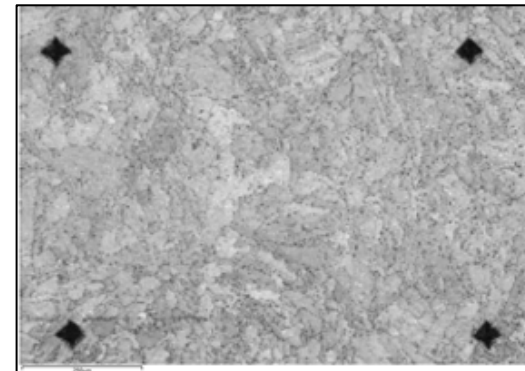
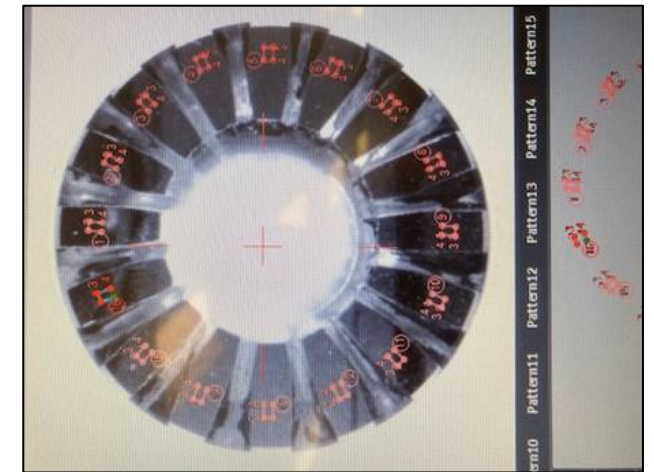
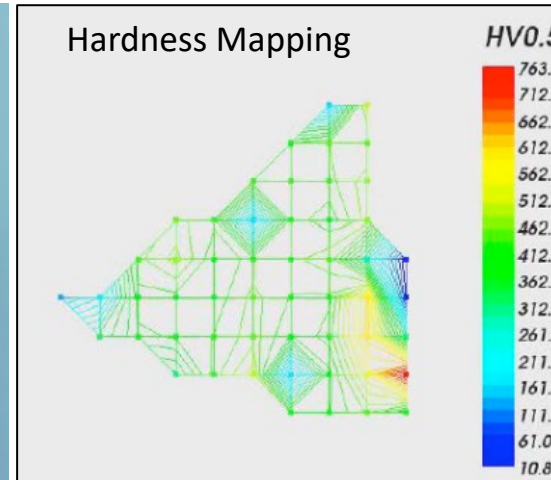
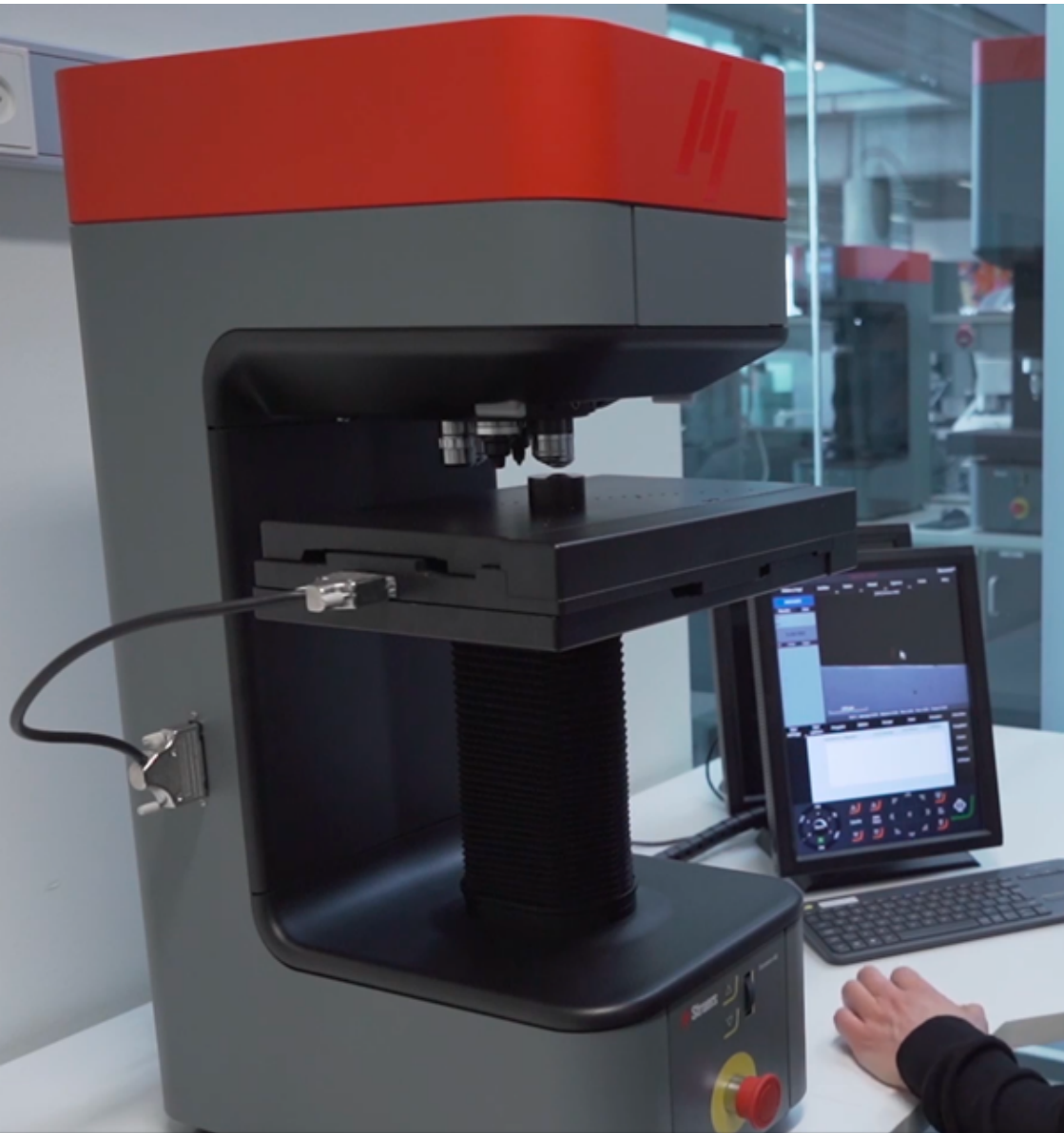
CALPHAD predictions match with synthesized compositions and phases.

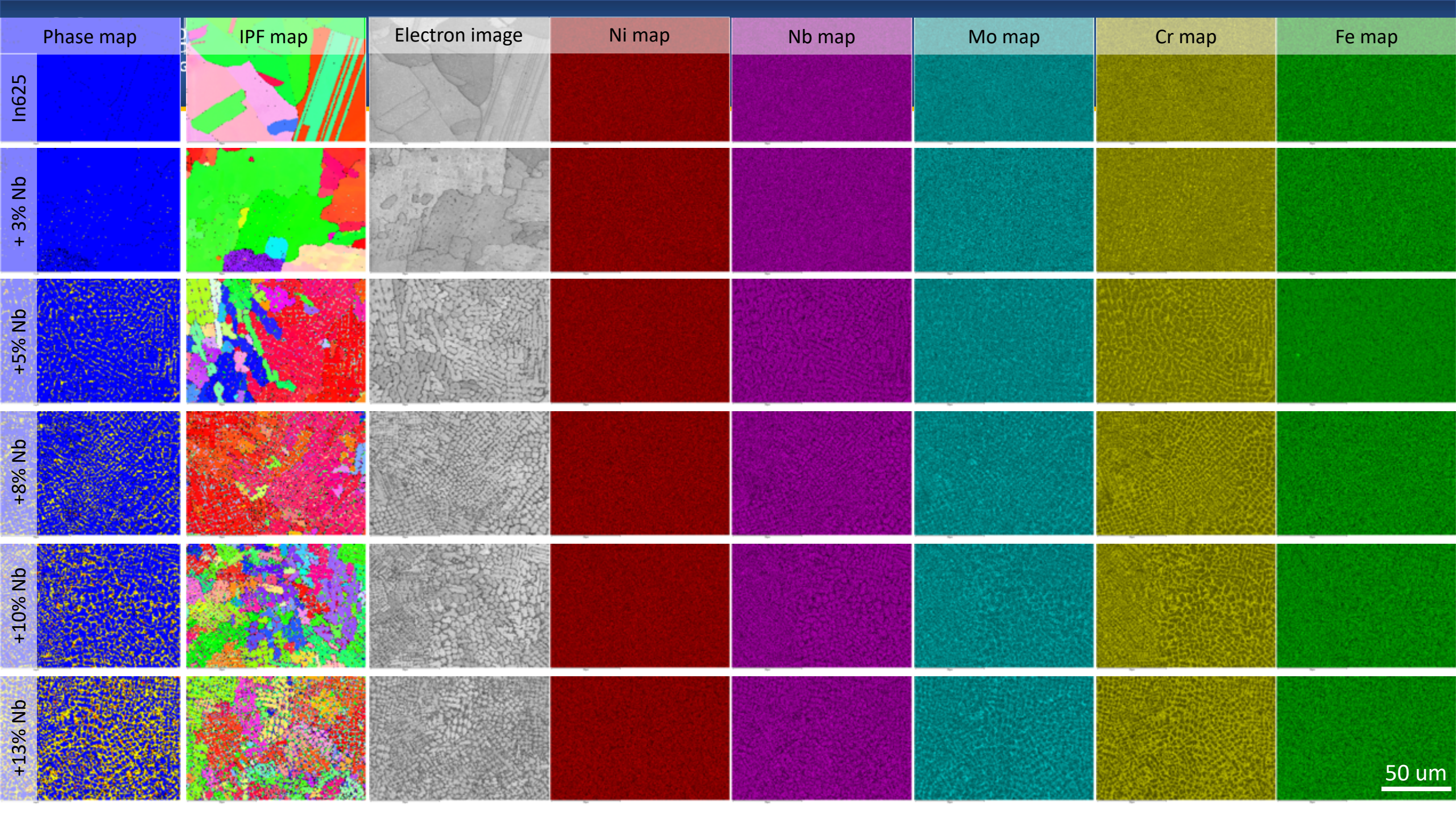


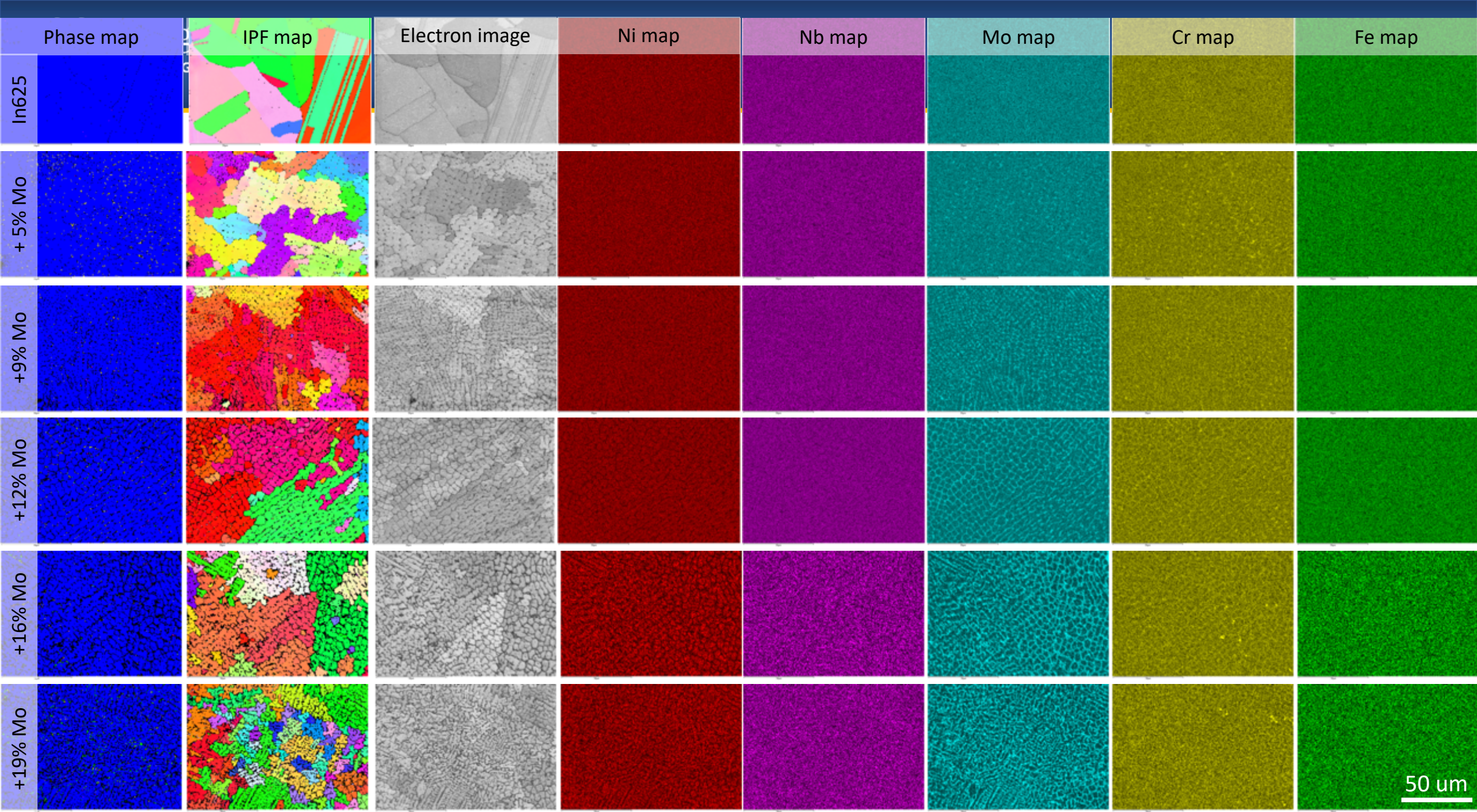
New CMOS-technology for EBSD allows higher pattern resolution, high acquisition rates that are comparable now to EDS

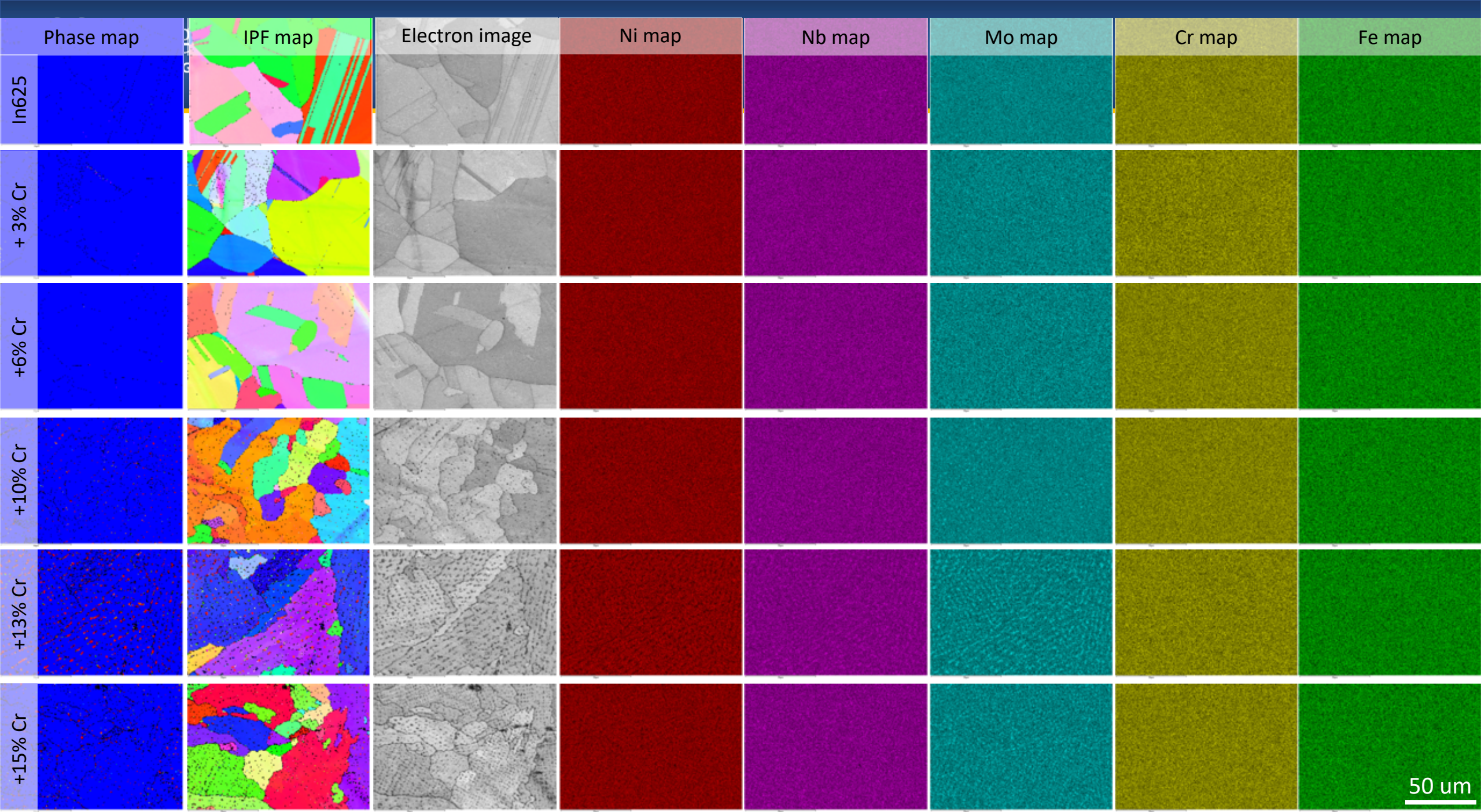


By combining simultaneous EDS and EBSD (chemistry and structure) information, complete phase identification is possible: number of phases, phase types, volume fraction of phases, phase and grain scale, phase location (in grains or at grain boundaries).



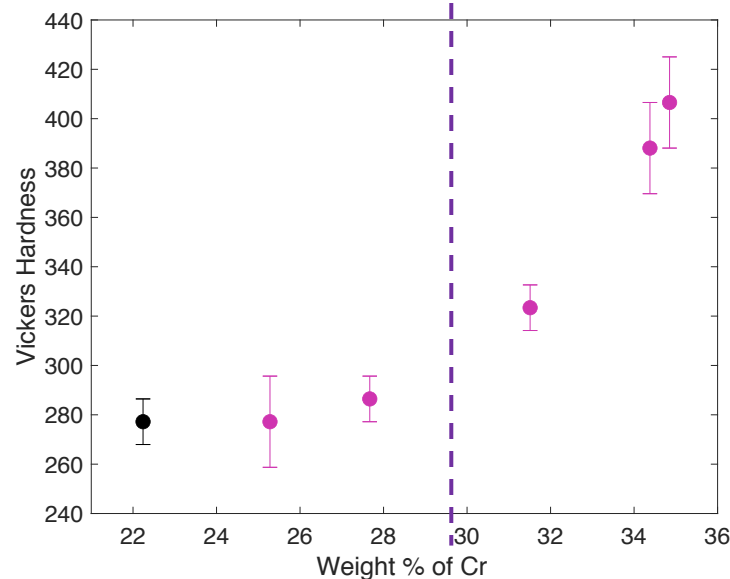
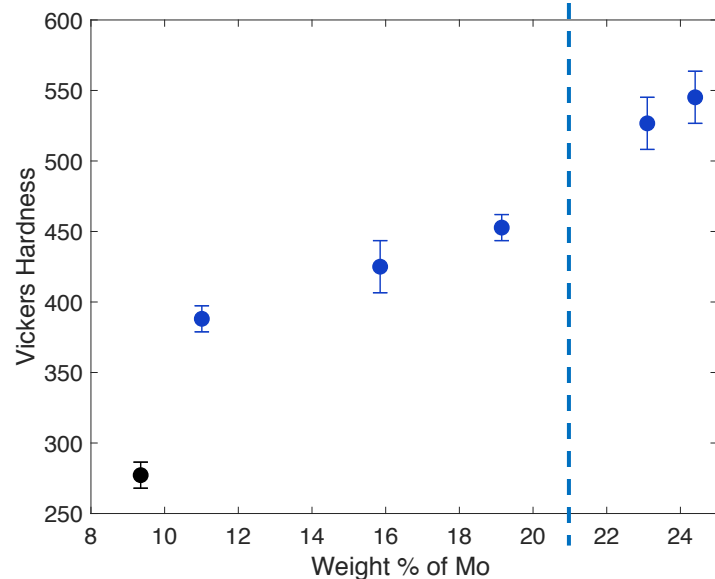
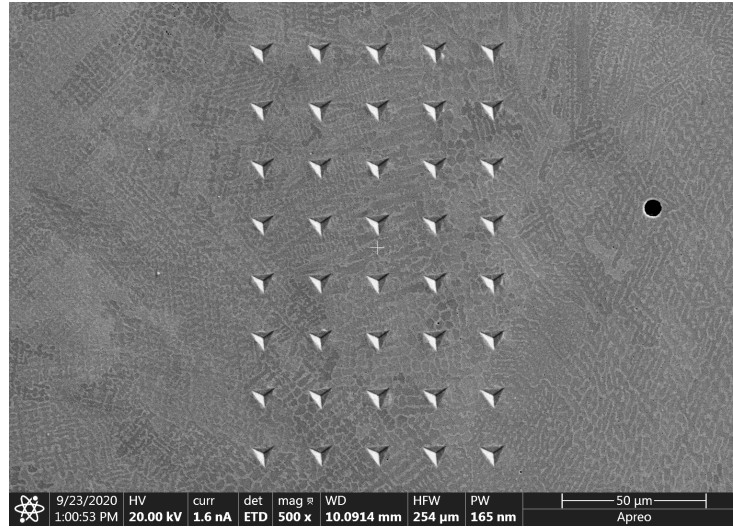
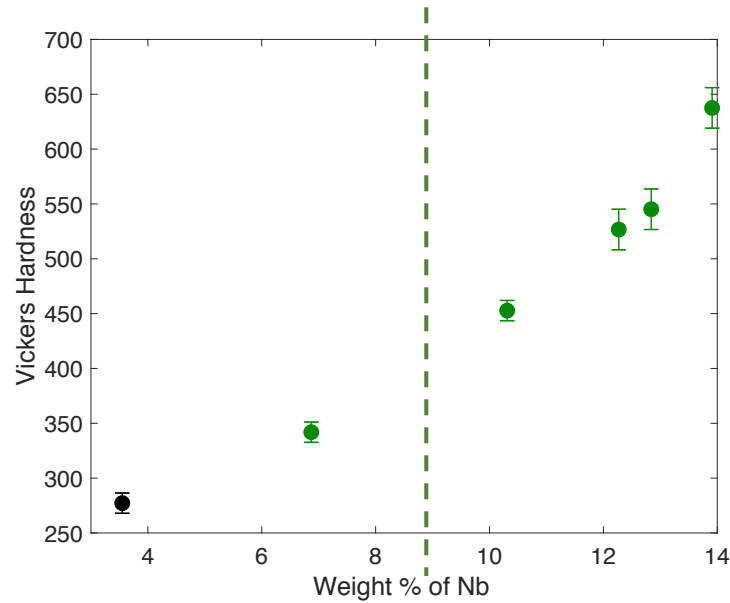






50 μ m

Automated Nanoindentation can extend characterization to 'Phase-specific' data



- Instrumented indentation techniques, such as nanoindentation (and some microhardness) automate the mechanical testing process
- An 8x5 grid was indented on each sample
- Vickers Hardness calculated from nanoindentation hardness results
- Increasing hardness with increased alloying elements – but where is solubility limit?
- Finding the solubility limit via XRD (and EBSD/EDS)
- Alloys for increased hardness, without brittle precipitates – increase in strength

The most challenging hurdles to High Throughout Rapid Experimental Alloy Development (HT-READ) are associated with accurate and fast data analysis, materials characterization setups, and the heavy reliance on an engineer's experiential knowledge. We are trying to minimize the reliance on user decision making in all aspects of the HT-READ pipeline.

One example is the use of EBSD technology as a characterization step. Current EBSD technology relies on 'User selected phases' due to the implementation of Hough-based pattern transformation. Hough-based EBSD is not phase identification. The user must know which phases to select for each sample, then the Hough approach determines the orientation of those phases based on the transformed patterns. The user must know the answer of what phases are present, and Hough-based EBSD can fit a very wide range of incorrect structures to the phases.

We have developed a machine learning approach to replace Hough-based EBSD that can be performed autonomously.
No user phase selection required.

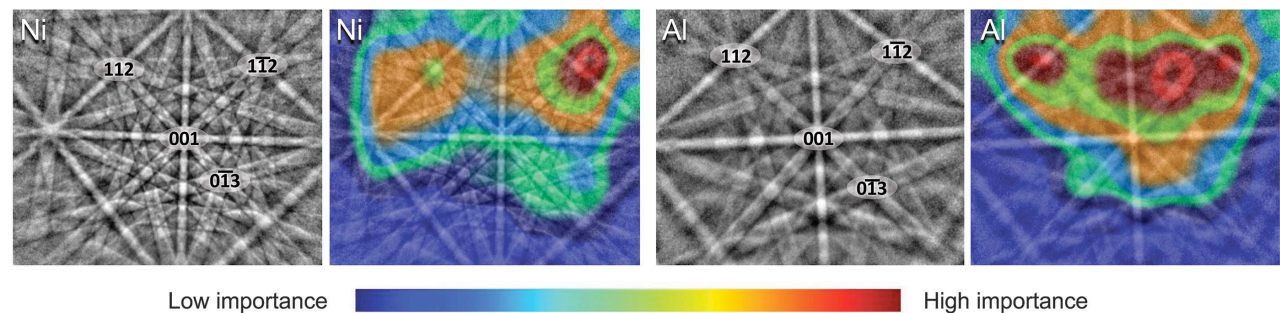
CRYSTALLOGRAPHY

Crystal symmetry determination in electron diffraction using machine learning

Kevin Kaufmann¹, Chaoyi Zhu^{2*}, Alexander S. Rosengarten^{1*}, Daniel Maryanovsky³, Tyler J. Harrington², Eduardo Marin¹, Kenneth S. Vecchio^{1,2†}

Electron backscatter diffraction (EBSD) is one of the primary tools for crystal structure determination. However, this method requires human input to select potential phases for Hough-based or dictionary pattern matching and is not well suited for phase identification. Automated phase identification is the first step in making EBSD into a high-throughput technique. We used a machine learning-based approach and developed a general methodology for rapid and autonomous identification of the crystal symmetry from EBSD patterns. We evaluated our algorithm with diffraction patterns from materials outside the training set. The neural network assigned importance to the same symmetry features that a crystallographer would use for structure identification.

Science
AAAS



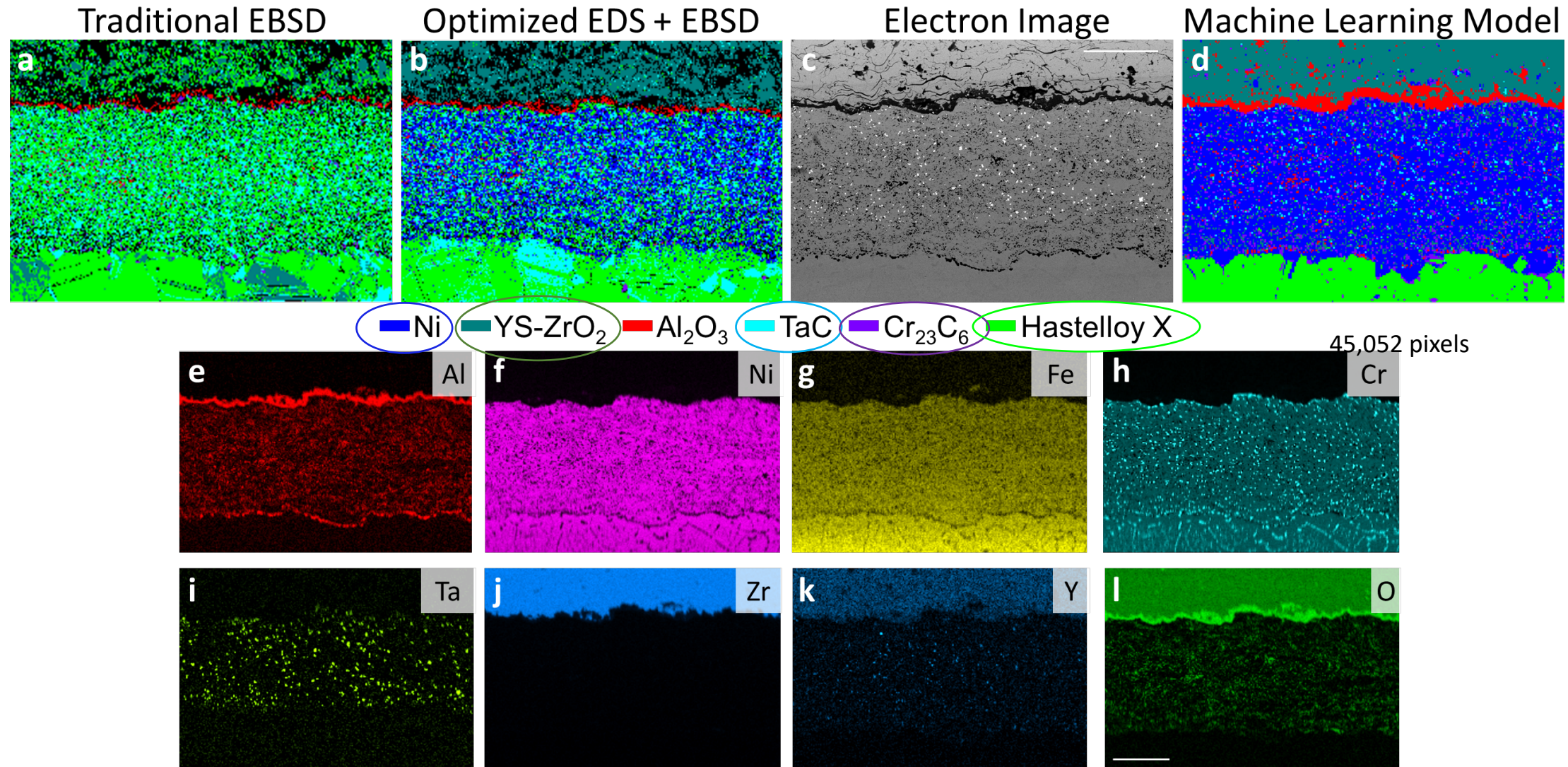
KAUFMANN, K., ZHU, C., ROSENGARTEN, A. S., MARYANOVSKY, D., HARRINGTON, T. J., MARIN, E. & VECCHIO, K. S., *SCIENCE* (2020).

Crystal symmetry determination in electron diffraction using machine learning, **367**, 564-568.

Material Set 2

Correct
classification
along diagonal

True class	Primitive Cubic	7368	4	2	6	22	10	0	3	45	2	424	14	2	12	93.1%	6.9%	Mo ₃ Si
	Face-Centered Cubic	2	8001	7	0	11	1	0	0	60	0	0	10	59	1	98.1%	1.9%	Al
	Body-Centered Cubic	20	50	14463	196	71	75	83	2	1	9	86	14	38	0	95.2%	4.3%	Ta
	Hexagonal	0	3	2	5896	10	7	0	0	3	4	6	9	0	2	99.2%	0.8%	Ti
	Rhombohedral	68	9	11	12	4548	189	4	5	75	5	28	52	13	53	89.7%	10.3%	Ilmenite
	Primitive Tetragonal	2	5	8	3	6	8542	0	0	0	0	0	28	12	29	98.9%	1.1%	Sn
	Body-Centered Tetragonal	0	2	2	1	16	0	12622	0	0	0	0	0	4	0	99.8%	0.2%	Anatase
	Primitive Orthorhombic	0	0	0	3	3	1	0	3034	0	1	6	0	0	1	99.5%	0.5%	Enstatite
	Face-Centered Orthorhombic	22	0	1	3	11	7	1	1	8706	0	226	21	1	3	96.7%	3.3%	Al ₃ Zr ₂
	Body-Centered Orthorhombic	0	2	11	9	94	1	0	1	1	24637	2	0	1	0	99.5%	0.5%	MoPt ₂
	Base-Centered Orthorhombic	0	0	0	1	0	19	0	53	0	0	6628	86	0	0	97.7%	2.3%	AuSn ₄
	Primitive Monoclinic	1	0	0	0	1	1	2	1	0	0	1	5481	34	29	98.7%	1.3%	Malachite
	Base-Centered Monoclinic	1	41	7	0	9	5	0	0	0	0	2	58	9043	34	98.3%	1.7%	Fe ₄ Al ₁₃
	Triclinic	2	0	0	0	7	4	0	0	2	0	0	14	10	3544	98.9%	1.1%	Labradorite
		Primitive Cubic	Face-Centered Cubic	Body-Centered Cubic	Hexagonal	Rhombohedral	Primitive Tetragonal	Body-Centered Tetragonal	Primitive Orthorhombic	Face-Centered Orthorhombic	Body-Centered Orthorhombic	Base-Centered Orthorhombic	Primitive Monoclinic	Base-Centered Monoclinic	Triclinic			
	Predicted class																	



Five of the six phases present belong to space group 225 (FCC), but have different lattice parameters and chemistries.

- High-Throughput Rapid Alloy Experimental Development (HT-READ)
 - Enables rapid experimental assessment of complex materials space
 - Instrumentation Pipeline has been established and Critical Components are Being Optimized.
 - Aspirational Goal of Fabrication and Characterization of ~100 bulk alloy compositions in a 24-hour period is within reach.
- Machine Learning being used to augment data analysis
 - ML being applied to Front-end alloy selection
 - ML being used to automate phase identification and phase fractions via EBSD
 - ML being used to automate phase identification and phase fractions via XRD
- Major Challenge: Data Management!