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

TEXAS A&M UNIVERSITY Engineering

Kenneth S. Vecchio

Professor of Materials Engineering, Department of NanoEngineering, 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, now called Oerlikon-Scoperta.

(Scoperta is the Italian word for 'Discovery').

Raymundo Arróyave

Ibrahim Karaman

DEVCOM ARMY RESEARCH LABORATORY

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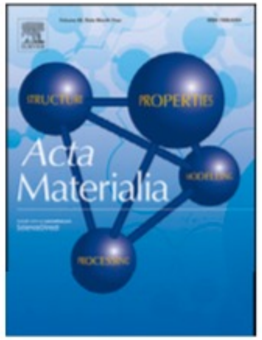


Full length article

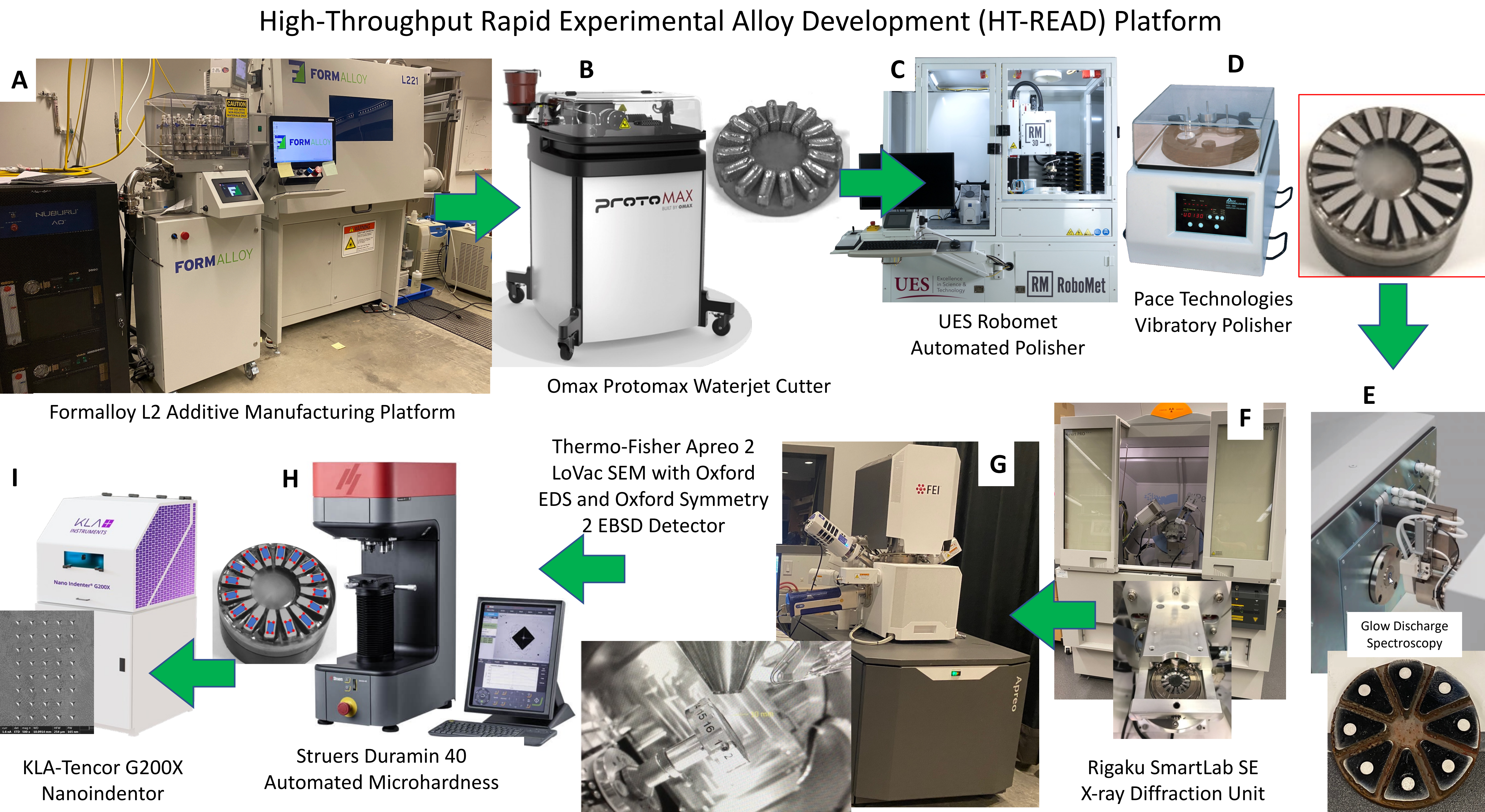
High-throughput rapid experimental alloy development (HT-READ)

Kenneth S. Vecchio[a,b,1,*], Olivia F. Dippo[a,1], Kevin R. Kaufmann[b], Xiao Liu[a]

[a] Materials Science and Engineering Program, UC San Diego, La Jolla CA 92093, USA
[b] Department of NanoEngineering, UC San Diego, La Jolla CA 92093, USA

Check for updates


High-Throughput Rapid Experimental Alloy Development (HT-READ) Platform
A
Formalloy L2 Additive Manufacturing Platform
B
Omax Protomax Waterjet Cutter
C
Struers Tegramin Automated Polisher
D
Pace Technologies Vibratory Polisher
E
Glow Discharge Spectroscopy
F
Rigaku SmartLab SE X-ray Diffraction Unit
G
Thermo-Fisher Apreo 2 LoVac SEM with Oxford EDS and Oxford Symmetry 2 EBSD Detector
H
Struers Duramin 40 Automated Microhardness
I
KLA-Tencor G200X Nanoindentor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Additive Manufacturing of Sample Libraries

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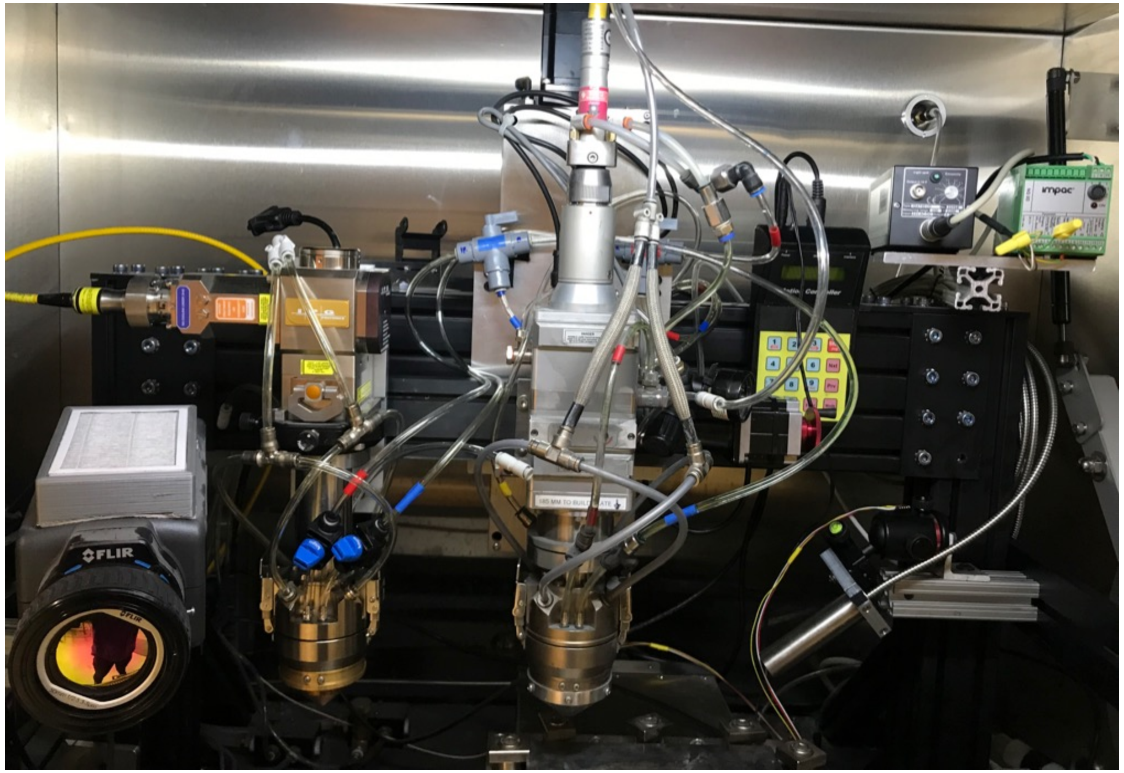
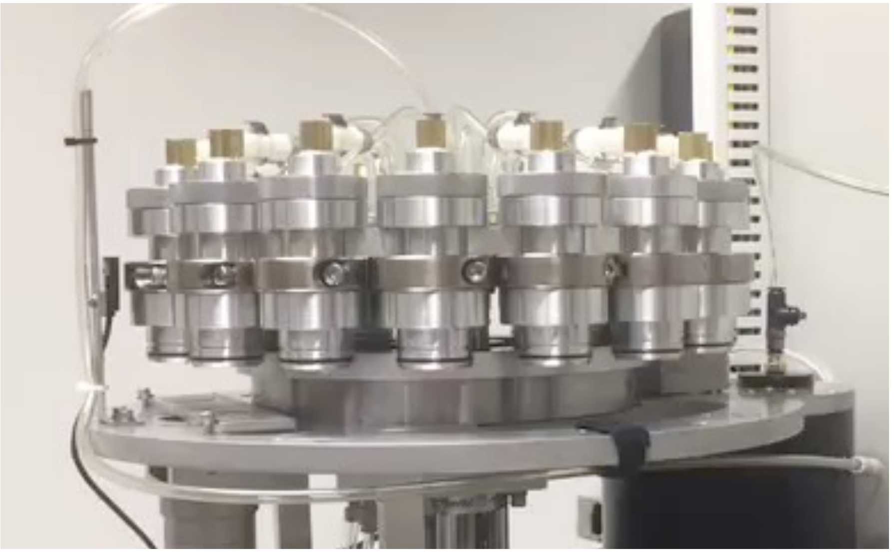
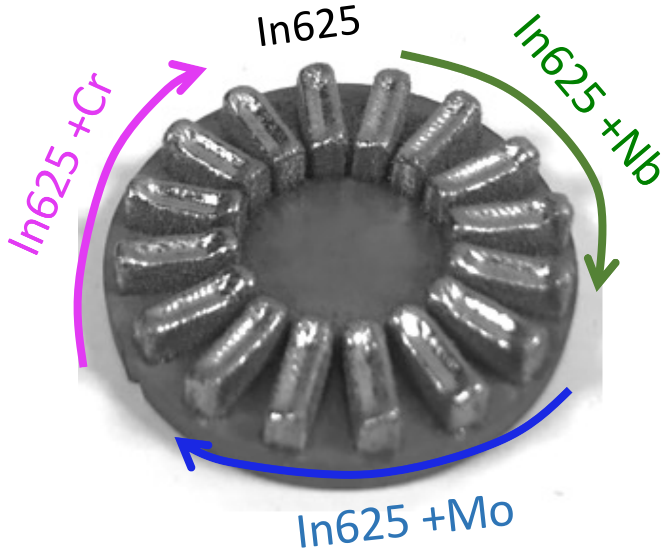




16-powder hopper allows for manufacturing of complex sample libraries



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 XRD and SEM-EBSD.

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Commercialized Alloy Development Feeder (ADF) Jointly developed between UC San Diego and Formalloy

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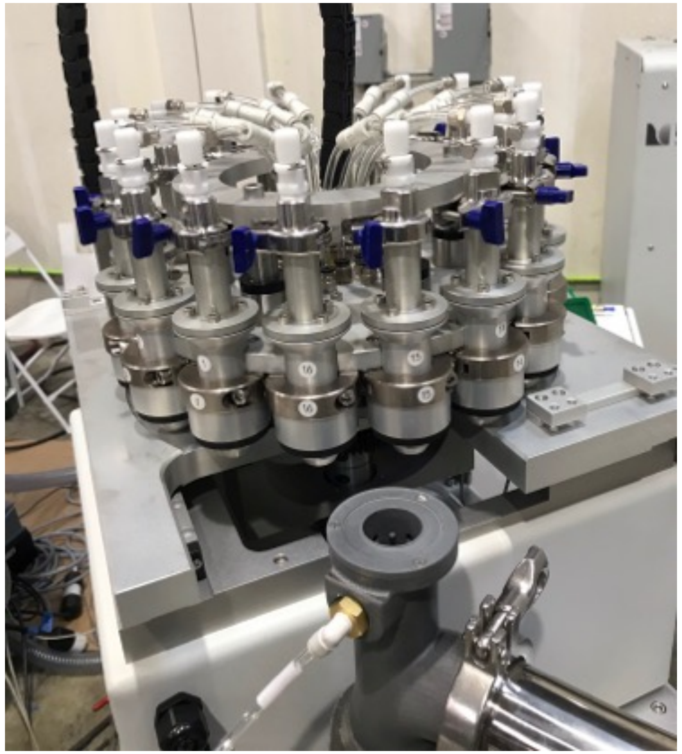
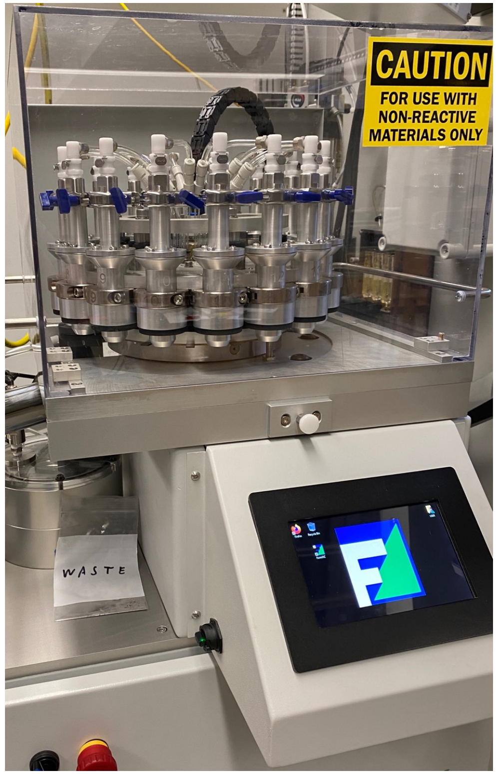
FORMALLOY ADF Alloy Development Feeder

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.

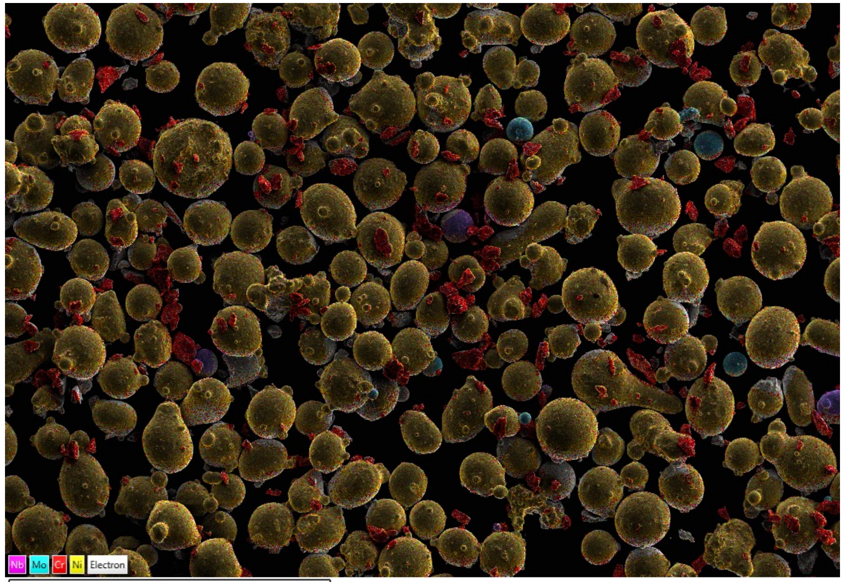
Powder Feeder Vials have Exact Mix of Powders to Achieve Desired Alloy Composition

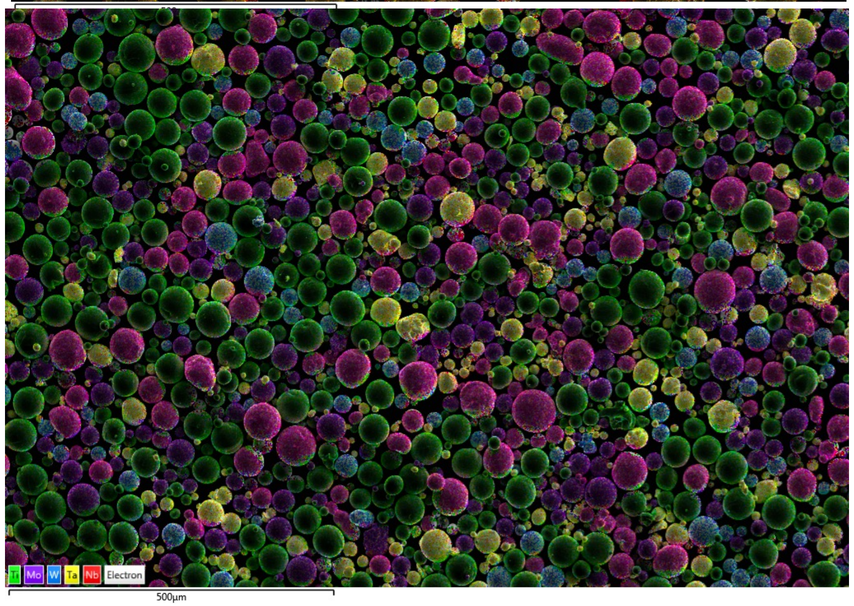
Unlikely high-throughput alloy development can be achieved using powder-bed platforms due to powder handling limitations.

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.

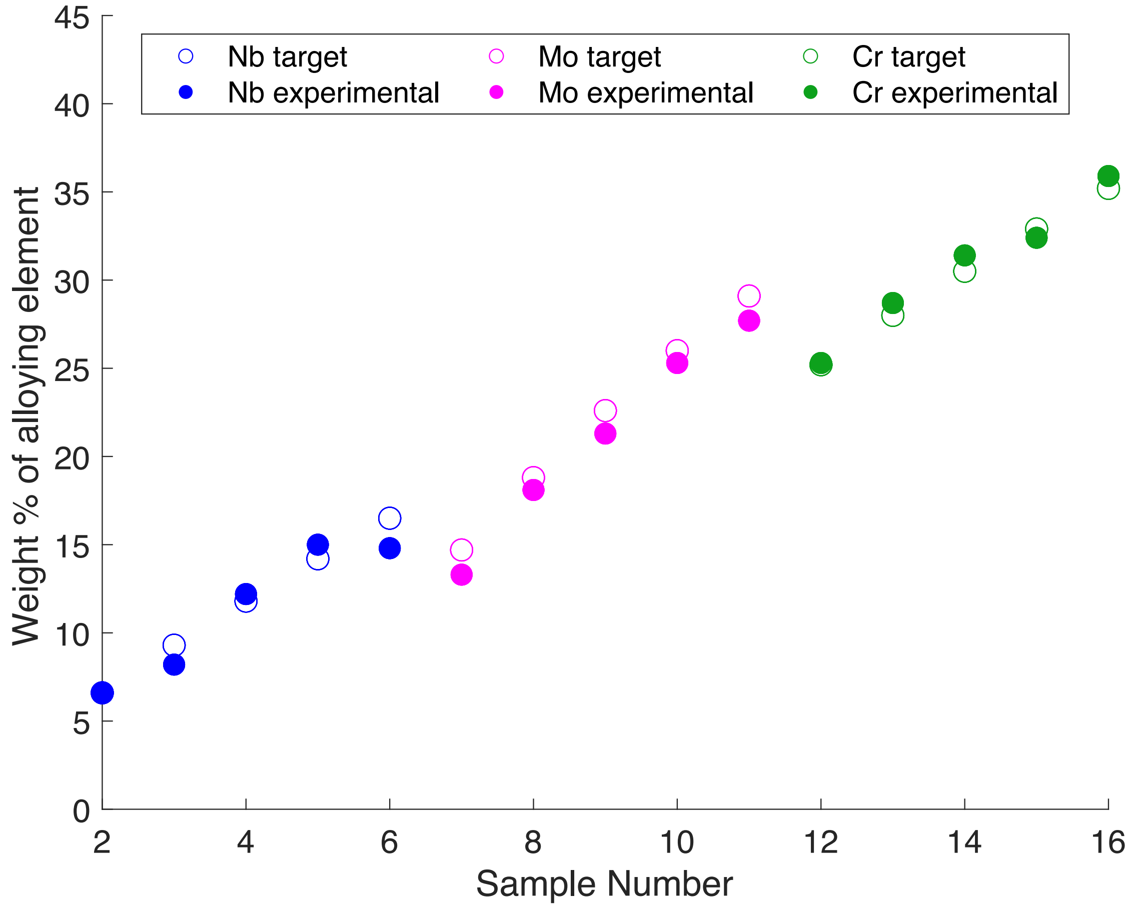
Huge advantages of blended powders in separate vials:

- 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.





16-Build Sample
EDS: Elemental Composition



#	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.

New Alloys to be Fabricated in this One-Year Program

Case Study #1:

Here, our goal is to demonstrate that the HT-READ platform can fabricate alloy samples of 5 elements (or greater) in the CCA space, Fe-Ni-Co-Cr-X (X = V, Ta, or Nb), with better than 1 wt.% accuracy for each element. The compositions will be systematically varied from equiatomic Fe-Ni-Co-Cr-X, to far from equiatomic compositions, with the results of both compositions fabricated and microstructures produced (in terms of phases, phase fractions, and phase compositions) compared with their CALPHAD predicted phase constitution. Our plan is to complete at least **four iterations** over one year of the present effort. (32 to 48 separate composition in Year 1).

Case Study #2:

For many materials, particularly steels, their microstructure and properties of the materials are strongly influenced by light elements, principally carbon, boron, and nitrogen. Both fabricating samples with specific concentrations of C, for example, and being able to quantify the C content in a rapid, HTP manner are significant challenges. This is an important limitation given the Army-relevant ballistic applications are steels. Here, we will investigate the ability to achieve C content accuracy of 0.1 wt.% across a range of compositions from 0 to 1 wt.% C added to 316L Stainless Steel. Since accurate quantification of C, particularly at low concentrations, is problematic for SEM-EDS, a different approach will be developed here. Glow Discharge Optical Emission Spectroscopy (GDS) will be employed to determine the composition of the samples built for this C concentration study. (16-32 separate compositions in Year 1) .

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Glow Discharge Spectroscopy (GDS) Elemental Composition

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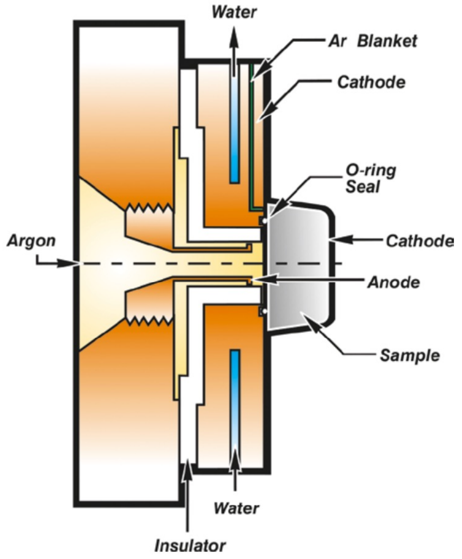
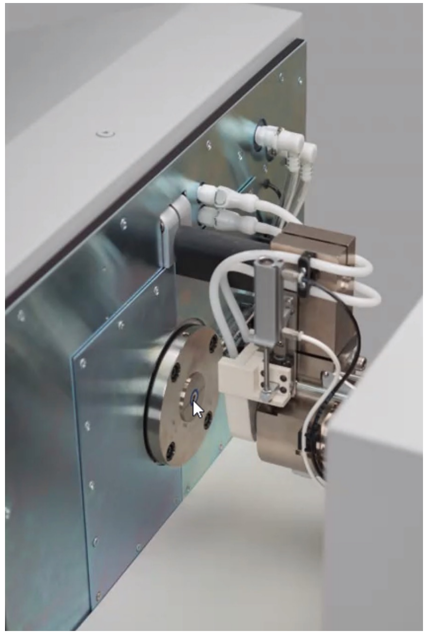


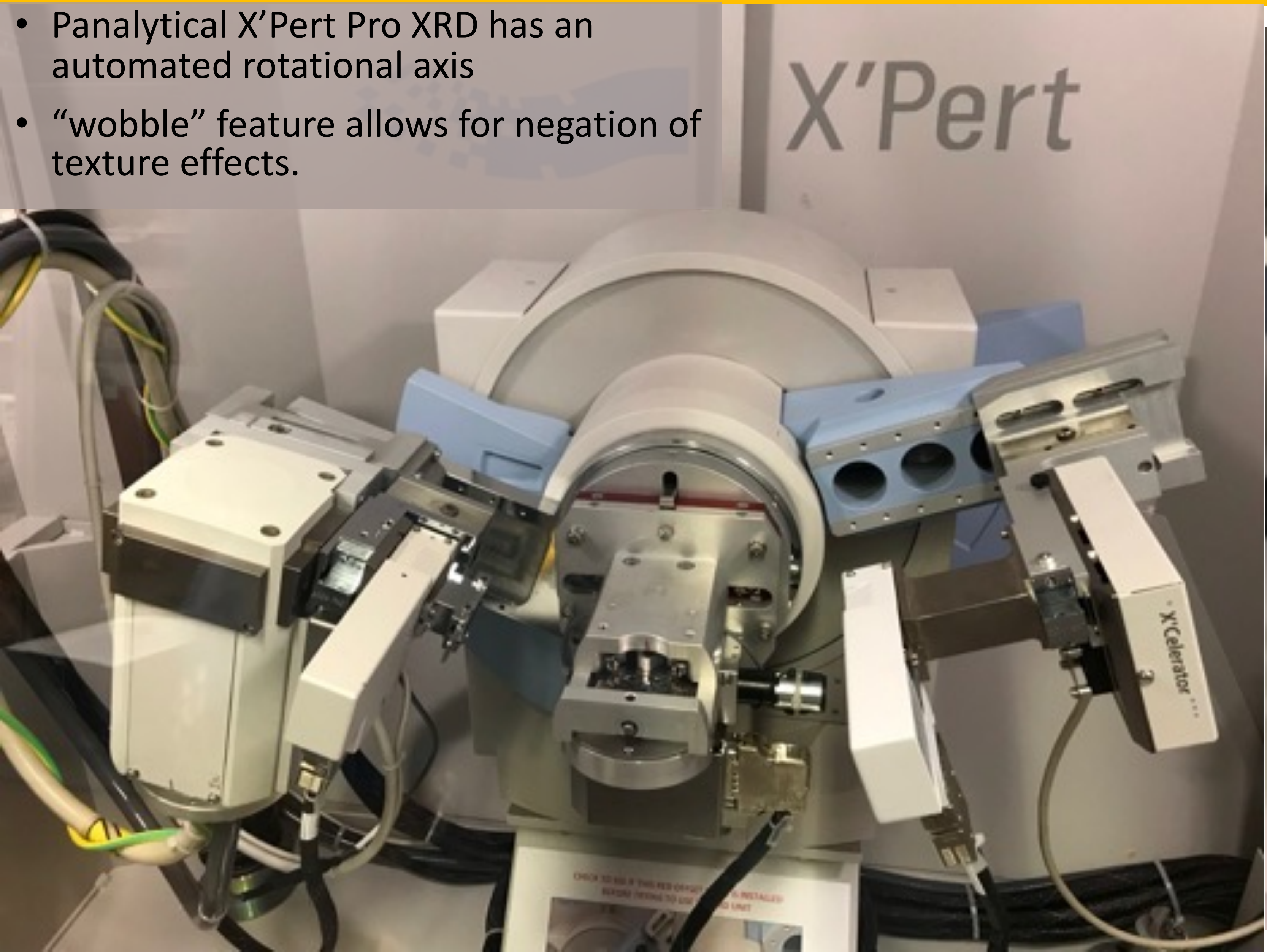


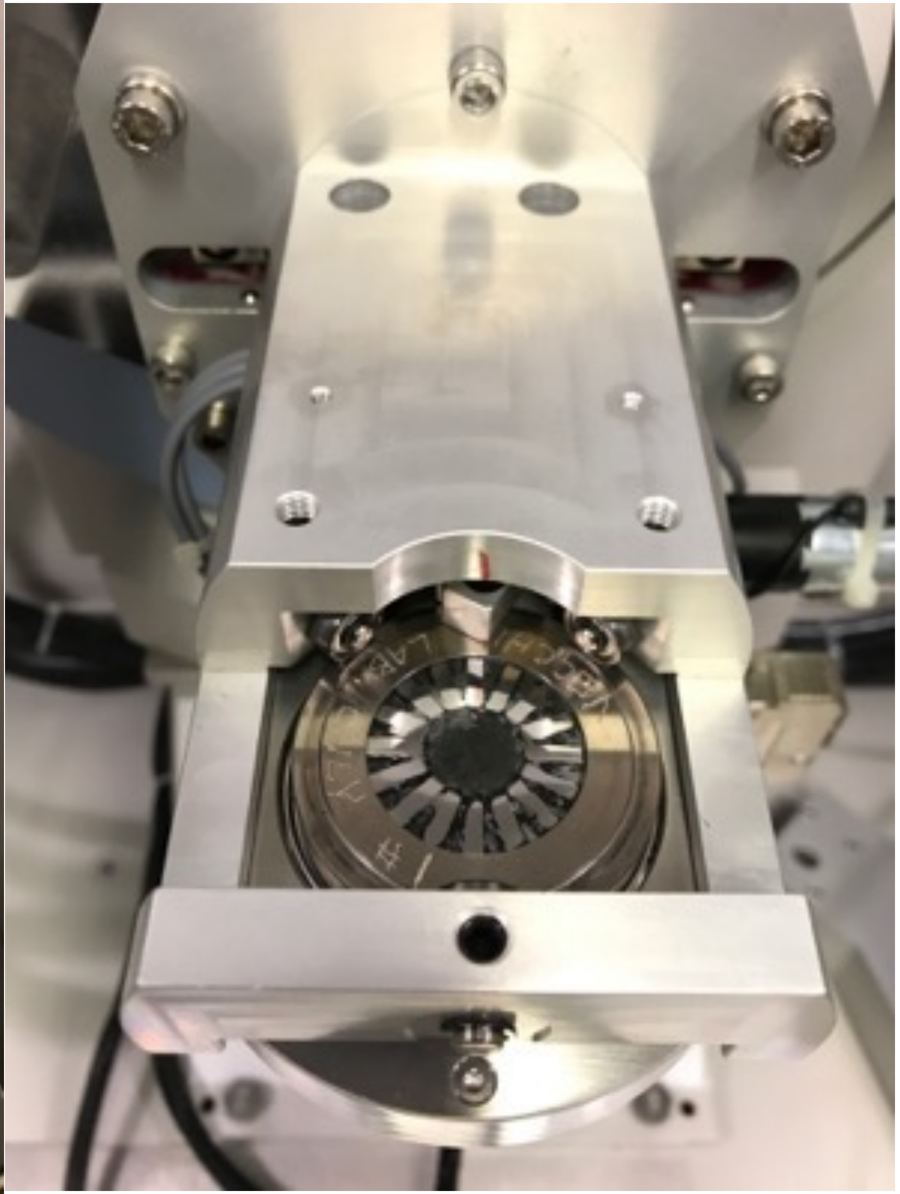
Analysis	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	V	Al	Ti	Nb	Co	W	Sn	Ta	B	Zr	N	Fe
Designed Composition	**0.125**	**0.000**	**0.000**	**0.000**	**0.000**	**10.300**	**0.700**	**1.100**	**0.000**	**0.200**	**0.000**	**0.000**	**0.055**	**0.000**	**1.025**	**0.000**	**0.000**	**0.000**	**0.000**	**0.055**	**86.440**
5/3/22	0.141	0.435	0.282	0.016	0.011	10	1.23	1.08	0.037	0.207	0.004	0.008	0.114	0.032	1.12	0.009	0.027	0.019	0.024	0.000	85.1
5/3/22	0.135	0.46	0.281	0.018	0.012	10.3	1.11	1.05	0.039	0.211	0.005	0.012	0.104	0.04	1.09	0	0.036	0.018	0.056	0.000	84.9
	0.138	**0.448**	**0.282**	**0.017**	**0.012**	**10.150**	**1.170**	**1.065**	**0.038**	**0.209**	**0.005**	**0.010**	**0.109**	**0.036**	**1.105**	**0.005**	**0.032**	**0.019**	**0.040**	**0.000**	**85.000**
	0.013	0.4475	0.2815	0.017	0.0115	-0.15	0.47	-0.035	0.038	0.009	0.0045	0.01	0.054	0.036	0.08	0.0045	0.0315	0.0185	0.04	-0.055	-1.44

Analysis	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	V	Al	Ti	Nb	Co	W	Sn	Ta	B	Zr	N	Fe
Designed Composition	**0.155**	**0.500**	**0.000**	**0.000**	**0.000**	**12.250**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**	**0.200**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**	**86.895**
5/3/22	0.176	0.512	0.433	0.017	0.011	12.4	0.3	0.03	0.044	0.022	0.006	0.009	0.241	0.044	0.023	0.026	0	0.008	0.045	0.000	85.3
5/3/22	0.225	0.542	0.473	0.02	0.014	12.8	0.399	0.043	0.054	0.029	0.004	0.019	0.266	0.08	0.045	0.055	0	0.008	0.1	0.000	84.4
	0.201	**0.527**	**0.453**	**0.019**	**0.013**	**12.600**	**0.350**	**0.037**	**0.049**	**0.026**	**0.005**	**0.014**	**0.254**	**0.062**	**0.034**	**0.041**	**0.000**	**0.008**	**0.073**	**0.000**	**84.850**
	0.0455	0.027	0.453	0.0185	0.0125	0.35	0.3495	0.0365	0.049	0.0255	0.005	0.014	0.0535	0.062	0.034	0.0405	0	0.008	0.0725	0	-2.045

Automated XRD of 16-Build Sample

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



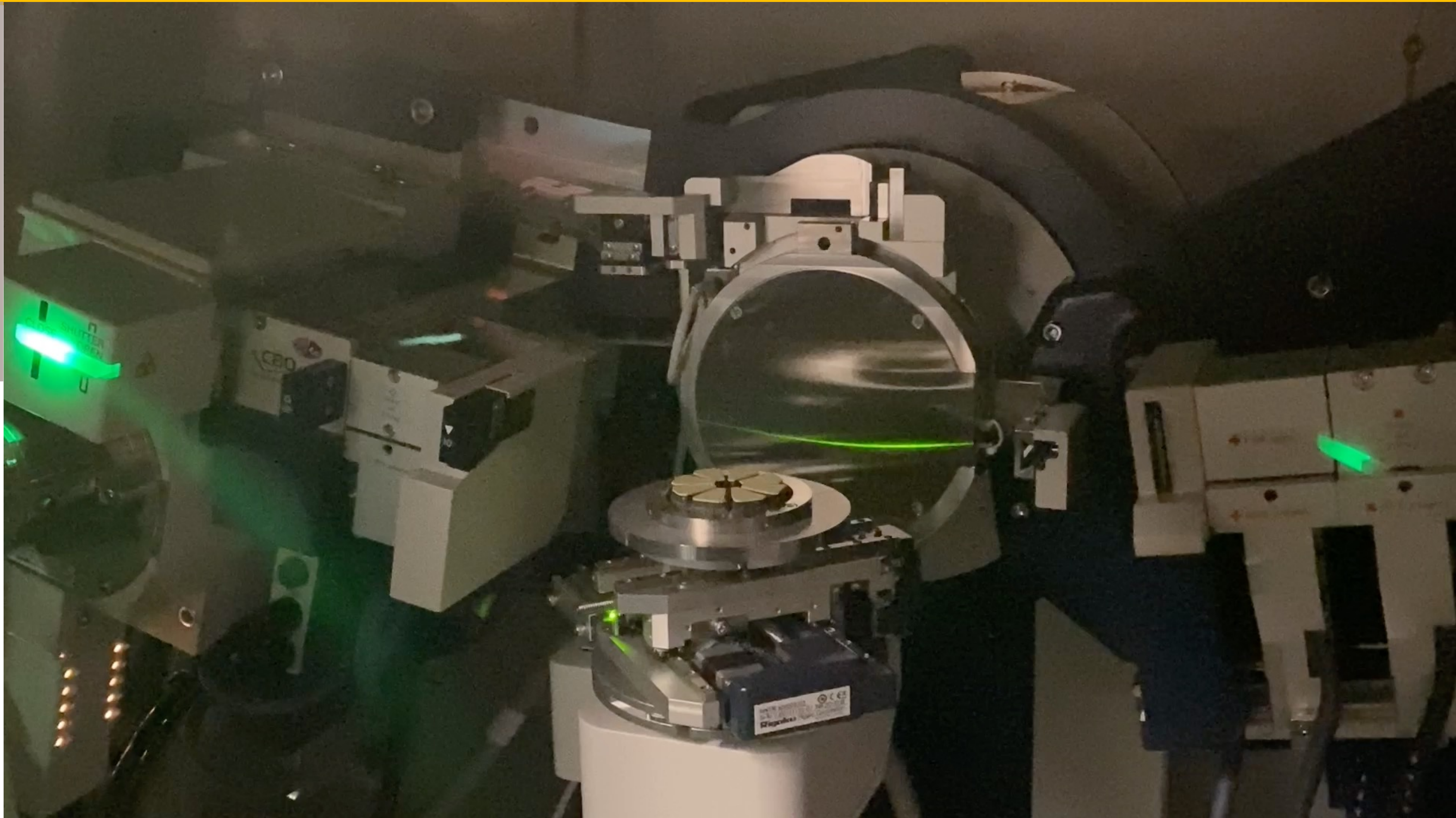


New Rigaku SmartLab XRD Platform

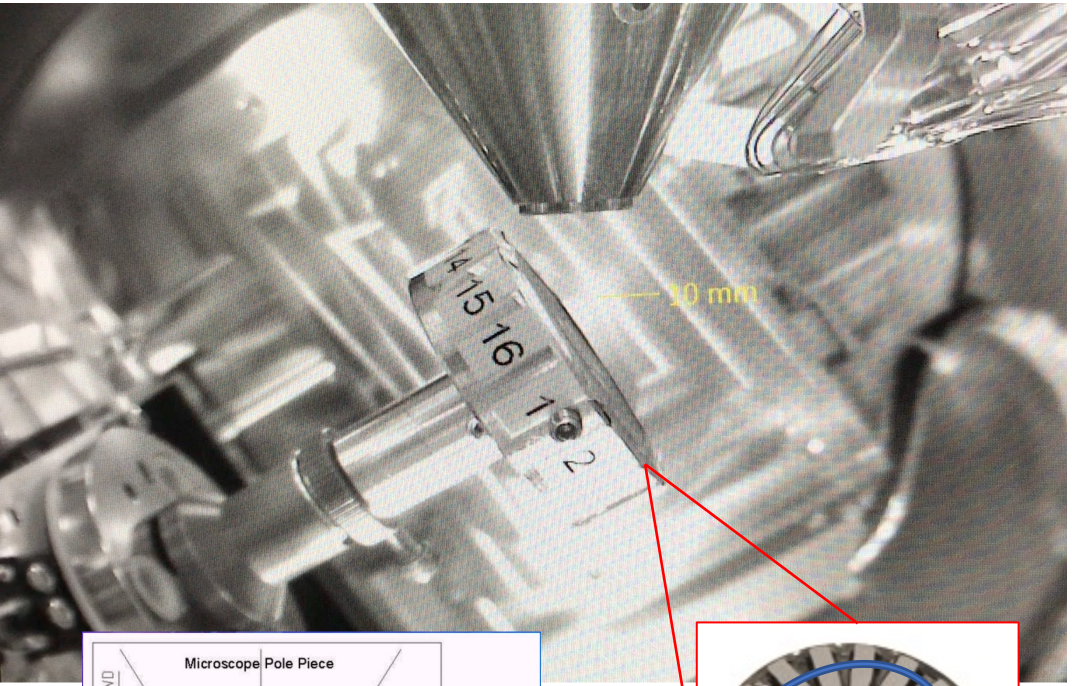
Automated XRD of 8-Build Sample

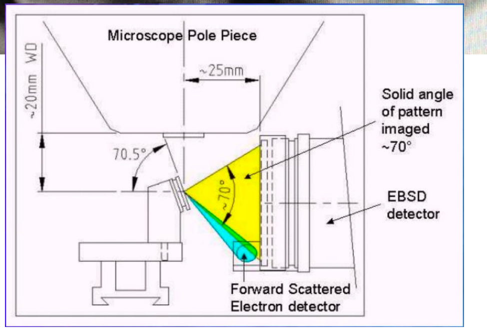
New Rigaku SmartLab 9-kW XRD with fully-programmable stage

"wobble" feature allows for negation of texture effects.



Characterization: Automated EBSD and EDS





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).

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Automated Microhardness Measurements and Automated Nano-Indentation Hardness

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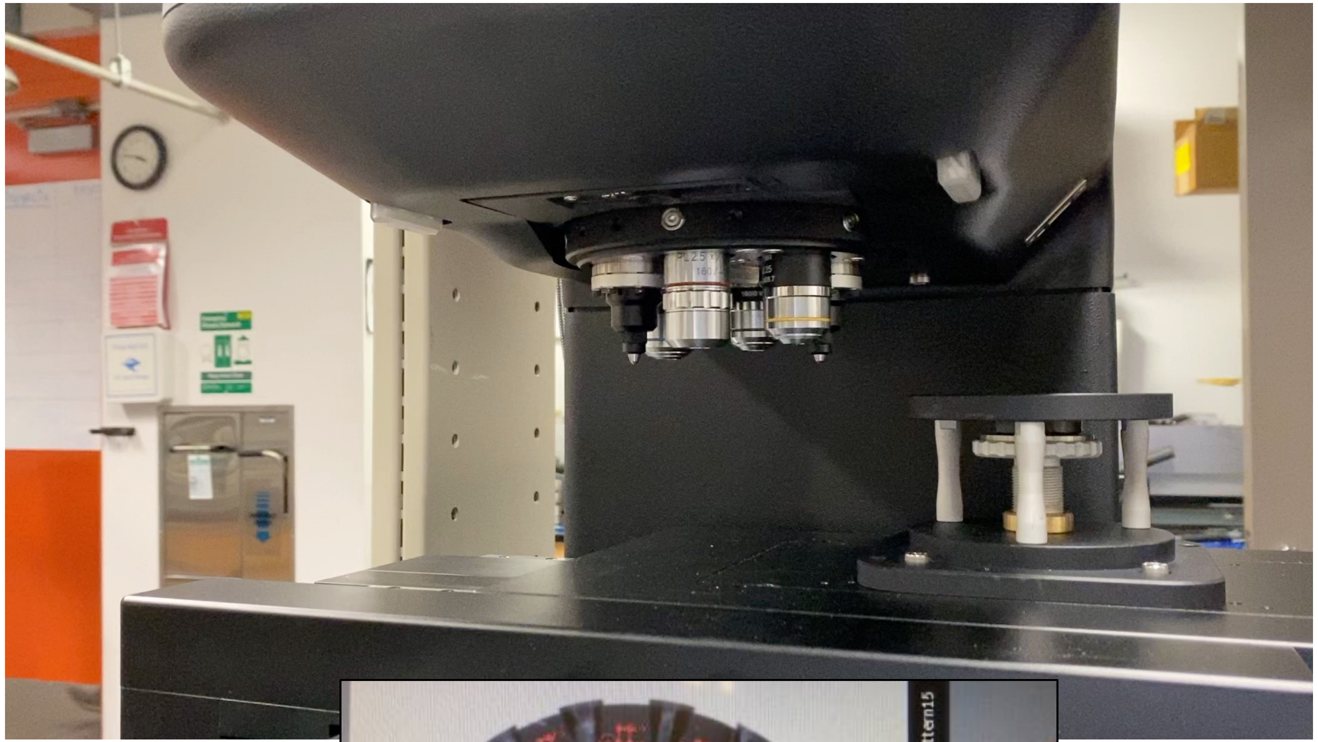
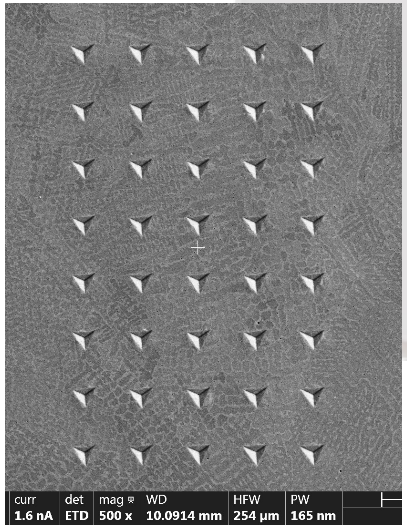
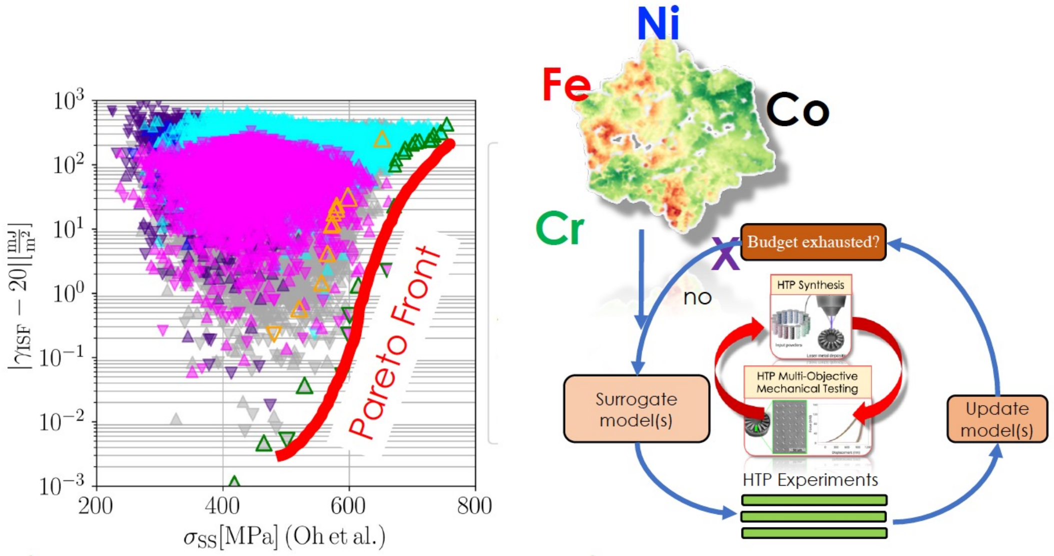


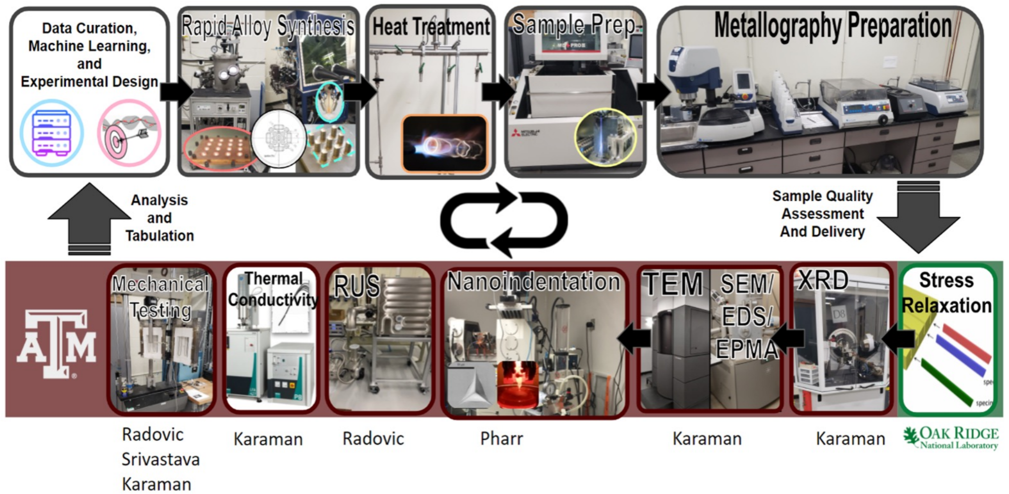


Texas A&M University (TAMU) Year 1 Activities

Efficient ML-based Alloy Discovery

We propose to use advanced Bayesian Materials Discovery (BMD) frameworks to efficiently detect property Pareto fronts.





On-demand True Property Gradients

We will leverage advanced path planning algorithms for functionally graded materials (FGM) to design and fabricate property-graded samples.