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Award No. DE-FEOO08719

Synergistic Computational and Microstructural Design of Next-Generation High-Temperature Austenitic Stainless Steels

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❖ PROJECT GOAL

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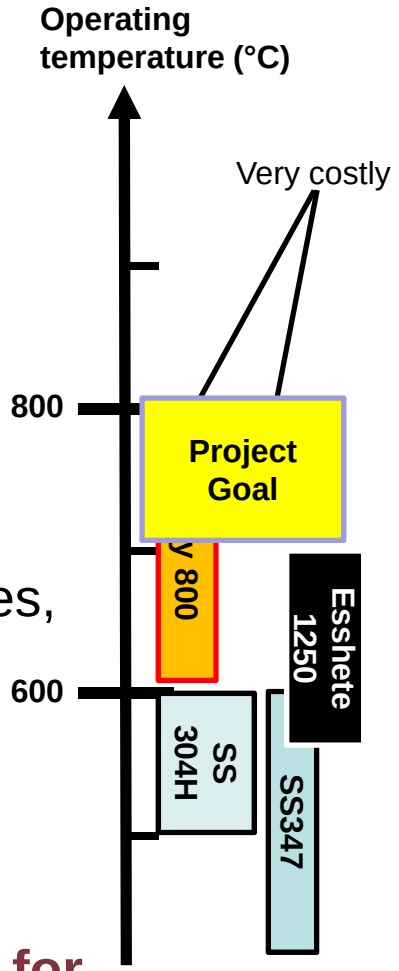
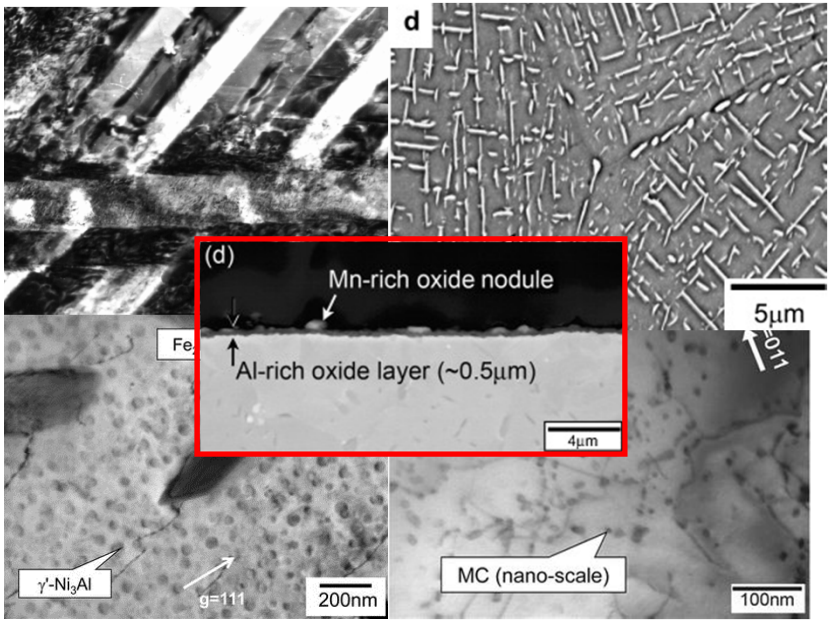
- Design new austenitic stainless steels (ASS) for advanced ultra supercritical combustion coal-fired power systems
  - ✓ High temperature strength
  - ✓ High ductility
  - ✓ Good creep resistance
  - ✓ Good high temperature oxidation/corrosion resistance
- Design of micro-alloying additions, heat treatment schedules, and microstructure
  - Cost-effective alternatives to Ni-base superalloys
  - Higher-temperature alternatives to ferritic steels
- **Develop a robust ICME design/optimization framework for high temperature ASS.**

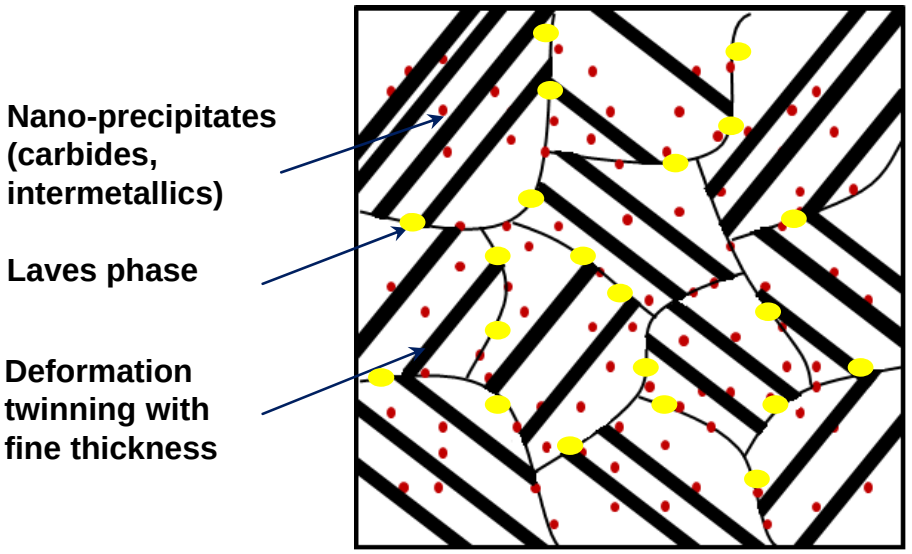


❖ Approach

Alloy + Microstructure Design

- Austenitic structure
- High density of low energy nano-twin boundaries
- Nano-scale precipitates, intermetallics, laves phases stable at high temperature
- Formation of alumina surface oxide





❖ OUTLINE

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❶ THE STUDY OF DEFORMATION TWINNING

- Evolution with applied strain
- Thermal stability
- Interactions of twins
- Effect of deformation twins on mechanical response

❷ NOVEL AFASS ALLOY DESIGN

- Initial characterization and evaluation of first-generation alloys
- Processing of candidates from first-generation alloys
- Introducing the second-generation alloys
- Characterization, evaluation, and processing of second generation alloys

❖ Overview

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		Fe	Ni	Cr	Mn	Nb	Si	Al	Mo	C	N	B	Deformation twin	FCC stability at high temperatures
Single crystals	Fe-Mn-C	Ba.			13					1.1			✓	×
	316L	Ba.	12	17.8	1.8		0.5		2.4	0.03			✓	?
	316 L+N	Ba.	11.8	17.7	1.1		0.44		2.3	0.08	0.2		×	?
	316+N		10	17	1.5		0.6		1.8	0.05	~0.1		×	?
	316	Ba.	9.5	17.5	1.6		0.72		2.51	0.03			✓	✓
	Alloy 1	Ba.	20	14	2	0.86	0.15	2.5	2.5	0.08		0.01	×	✓

First-Generation Alloys

all in wt%	Fe	Ni	Cr	Mn	Nb	Si	Al	Ti	Mo	V	C	N	B	Twinning	FCC stability	Precipitates	Alumina
Alloy 1	Ba.	20	14	2	0.86	0.15	2.5	0	2.5	0	0.08	0	0.01	×	✓	×	✓
Alloy 2	Ba.	12	14	10	1	0.2	2.5	0.3	2.5	0.5	0.08	0.01	0	?	×	×	?
Alloy 3	Ba.	17	14	10	1	0.2	2.5	0.3	2.5	0.5	0.08	0.01	0	?	×	×	?
Alloy 4	Ba.	10	16	10	0	0.5	3	0	2.5	0	0.05	0	0	?	×	×	?

- Alloy 1: Need to validate the predictive power of thermodynamic databases and models developed (oxidation, twinning ability). Selected based on the literature material developed by Yamamoto et al., at ORNL
- Alloy 2: lower expensive Ni, V to form precipitate at high temp., N to improve twin-ability. Nb is for carbides and Laves phases
- Alloy 3: higher Ni than alloy 2 for FCC stability
- Alloy 4: between Alloy 1 (AFA) and 316 SS (twin)

Second-Generation Alloys

wt.%	C	Mn	Ni	Mo	Al	Cr	Si	Fe	Twinning	FCC stability	Precipitates	Alumina
PGAA 2	0.088	9	17.15	2.24	3.11	15.3	0.19	bal.	?	✓	×	?

❖ OUTLINE

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❶ THE STUDY OF DEFORMATION TWINNING

- ➢Evolution with applied strain
- ➢Thermal stability
- ➢Interactions of twins
- ➢Effect of deformation twins on mechanical response

QUESTIONS AND CHALLENGES

- Fundamental study of recovery and recrystallization (ReX) of deformation twins in low SFE steels in the presence of various densities of dislocations
- The optimum thermo-mechanical processing path for high volume fraction of deformation twins
- Role of in-situ carbides and nitrides during recovery and ReX in the presence of deformation twins?
- Role of deformation twins and nano-particles on creep and stress rupture behavior of designed steels.

❖ TWINNING STUDIES

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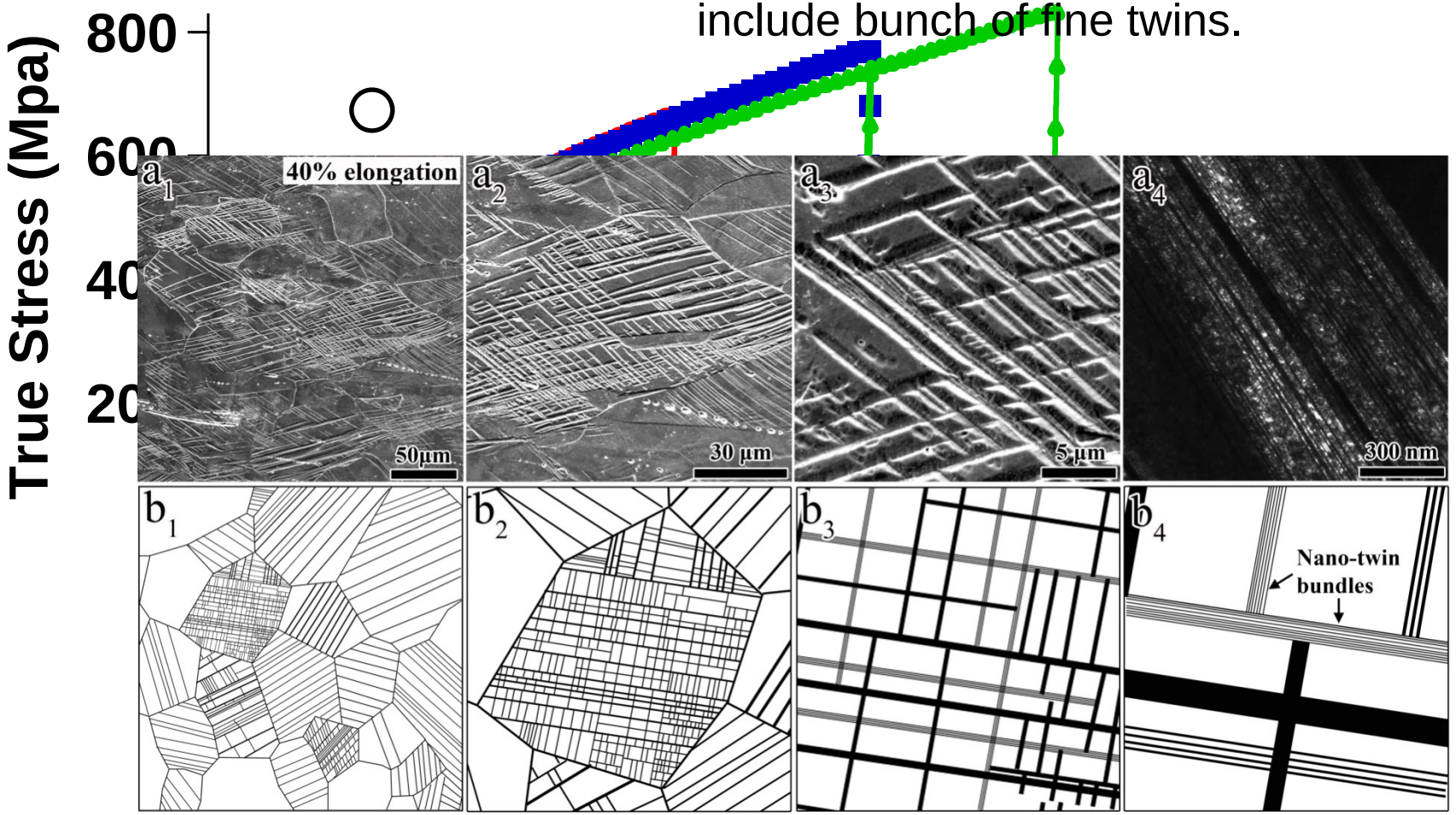
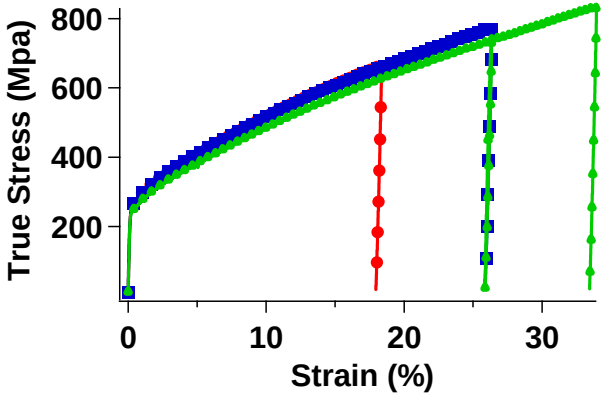
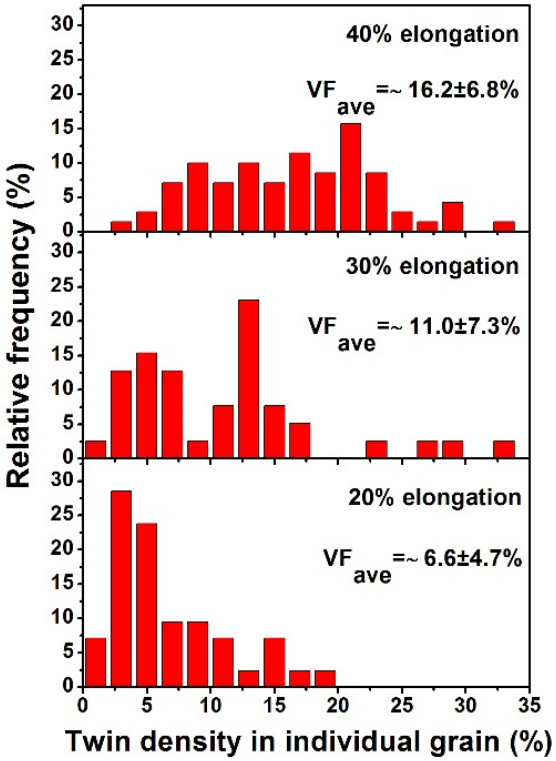
316 Stainless Steel, 40% Tension at RT

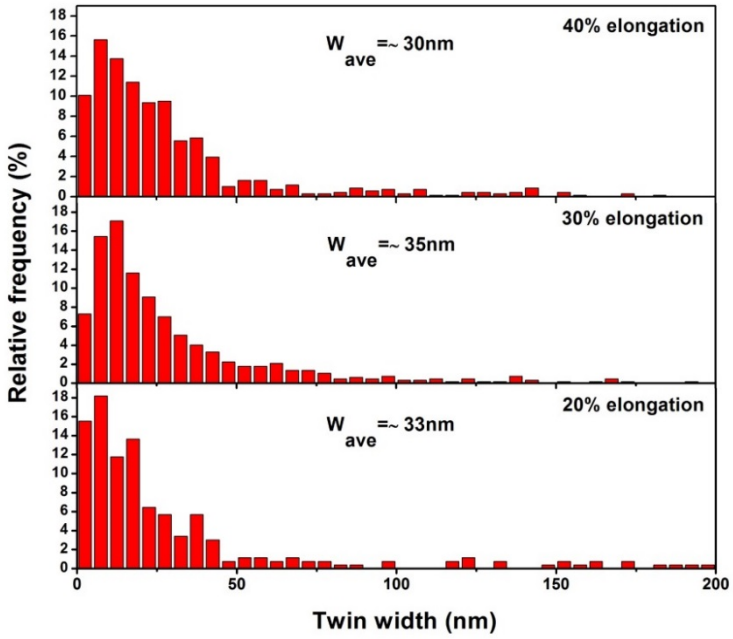
High density twins. Twins intersect with each other. Some twin bands include bunch of fine twins.



316 Stainless Steel, Tension at RT



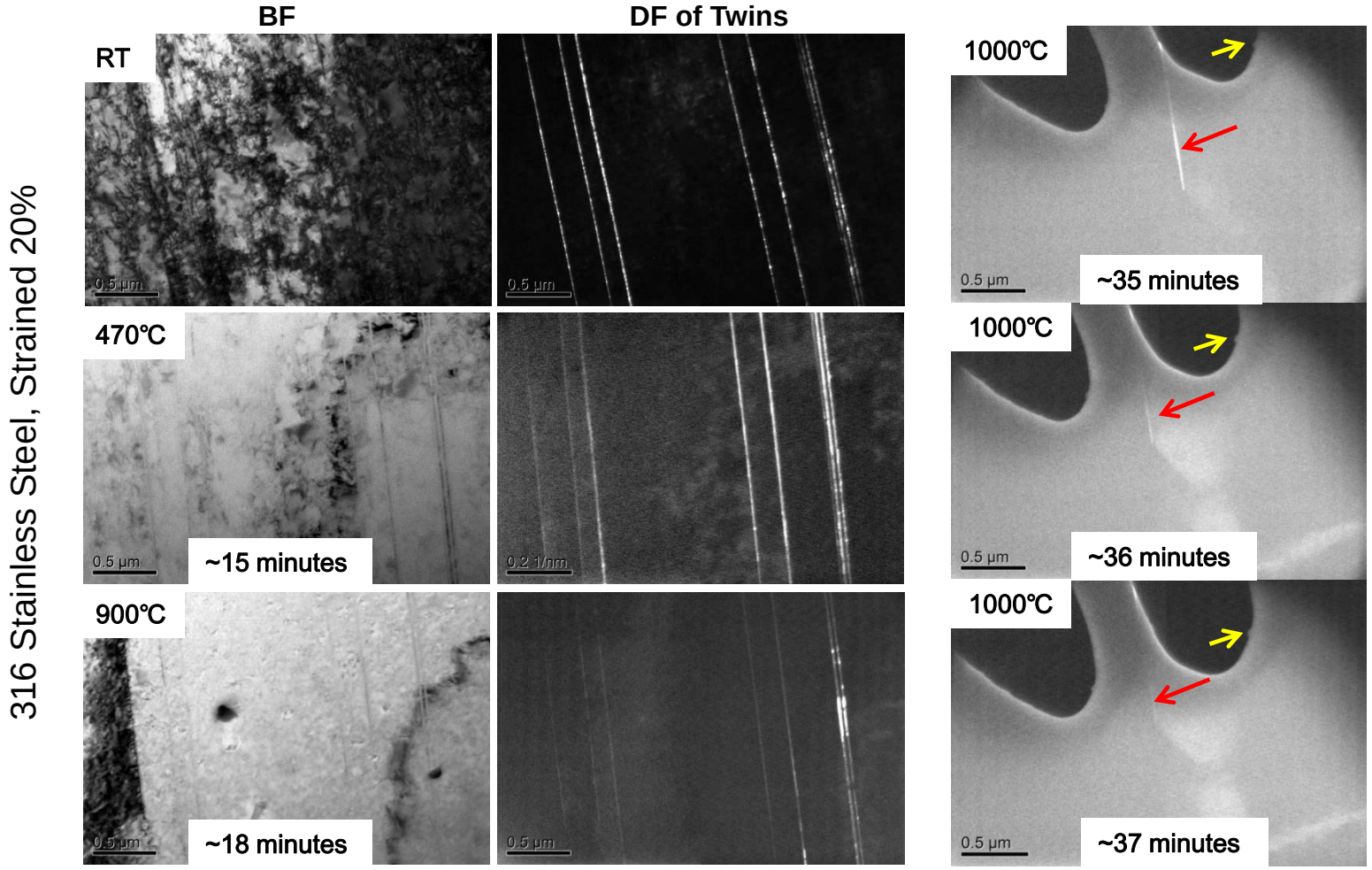




	Twinned Grains (%, from ~500 grains)	Twin VF in Twinned Grains (%, from ~70 Grains)	Twins in Whole Sample (%, from ~500 grains)
20%	62.6 ± 4.6	6.6 ± 4.7	5.8 ±1.5
30%	77.3 ± 3.1	11.0 ± 7.3	9.4 ± 1.6

- The twin density increases with increasing strain.
- Twin width stays similar.

Thermal Stability of Deformation Twins During In-situ TEM Heating



The nano twins are stable under 900 ºC, no coarsening and detwinning were discerned. Dislocations were recovered by annealing.

❷ NOVEL AFASS ALLOY DESIGN

- ➢Initial characterization and evaluation of first-generation alloys
- ➢Processing of candidates from first-generation alloys
- ➢Introducing the second-generation alloys
- ➢Characterization, evaluation, and processing of second generation alloys

First-Generation Alloys

all in wt%	Fe	Ni	Cr	Mn	Nb	Si	Al	Ti	Mo	V	C	N	B
Alloy 1	Ba.	20	14	2	0.86	0.15	2.5	0	2.5	0	0.08	0	0.01
Alloy 2	Ba.	12	14	10	1	0.2	2.5	0.3	2.5	0.5	0.08	0.01	0
Alloy 3	Ba.	17	14	10	1	0.2	2.5	0.3	2.5	0.5	0.08	0.01	0
Alloy 4	Ba.	10	16	10	0	0.5	3	0	2.5	0	0.05	0	0

- Alloy 1: Need to validate the predictive power of thermodynamic databases and models developed (oxidation, twinning ability). Selected based on the literature material developed by Yamamoto et al., at ORNL
- Alloy 2: lower expensive Ni, V to form precipitate at high temp., N to improve twin-ability. Nb is for carbides and Laves phases
- Alloy 3: higher Ni than alloy 2 for FCC stability
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❖ ALLOY DESIGN

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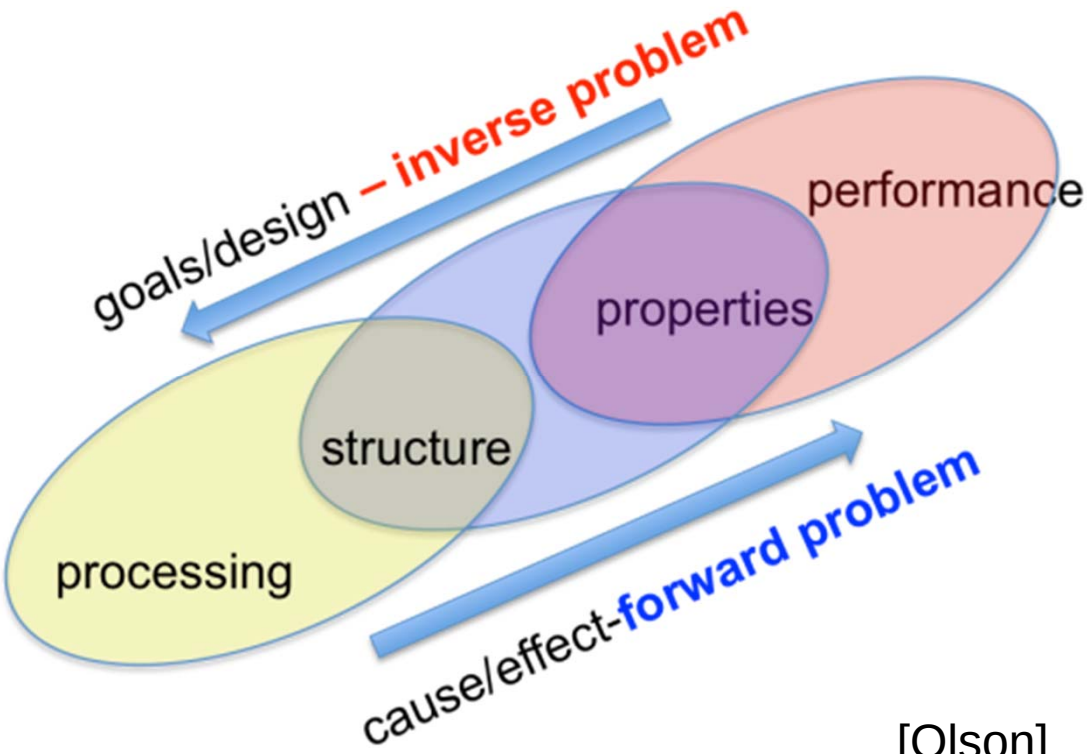
First-Generation Alloys

Alloy 1	✓ Fully austenite
	× Uncontrollable NbC precipitation
	× No Twinning
	✓ Alumina scale formation
Alloy 2	× Second phase formation
	× Uncontrollable Ti-rich NbC
Alloy 3	✓ Austenite, intra-granular second phase
	× Uncontrollable Ti-Nb carbo-nitrides and AlN precipitation
Alloy 4	× Second phase formation

all in wt%	Fe	Ni	Cr	Mn	Nb	Si	Al	Ti	Mo	V	C	N	B
Alloy 1	Ba.	20	14	2	0.86	0.15	2.5	0	2.5	0	0.08	0	0.01
Alloy 2	Ba.	12	14	10	1	0.2	2.5	0.3	2.5	0.5	0.08	0.01	0
Alloy 3	Ba.	17	14	10	1	0.2	2.5	0.3	2.5	0.5	0.08	0.01	0
Alloy 4	Ba.	10	16	10	0	0.5	3	0	2.5	0	0.05	0	0

What is Materials Design?



[Olson]

Materials Design is ultimately the solution to an inverse problem
Ideally, MD should be materials-agnostic (not realistic at this tim

Design Criteria

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- Alumina Formation
- Low SFE – Twinnability
- Large stability region for FCC
- Low Ms
- Competing factors:
  - Al necessary for alumina formation
  - Al increases SFE, decreases twinnability
  - Al stabilizes BCC against FCC
  - Optimization is necessary

Today: Formation of Stable Alumina Layer

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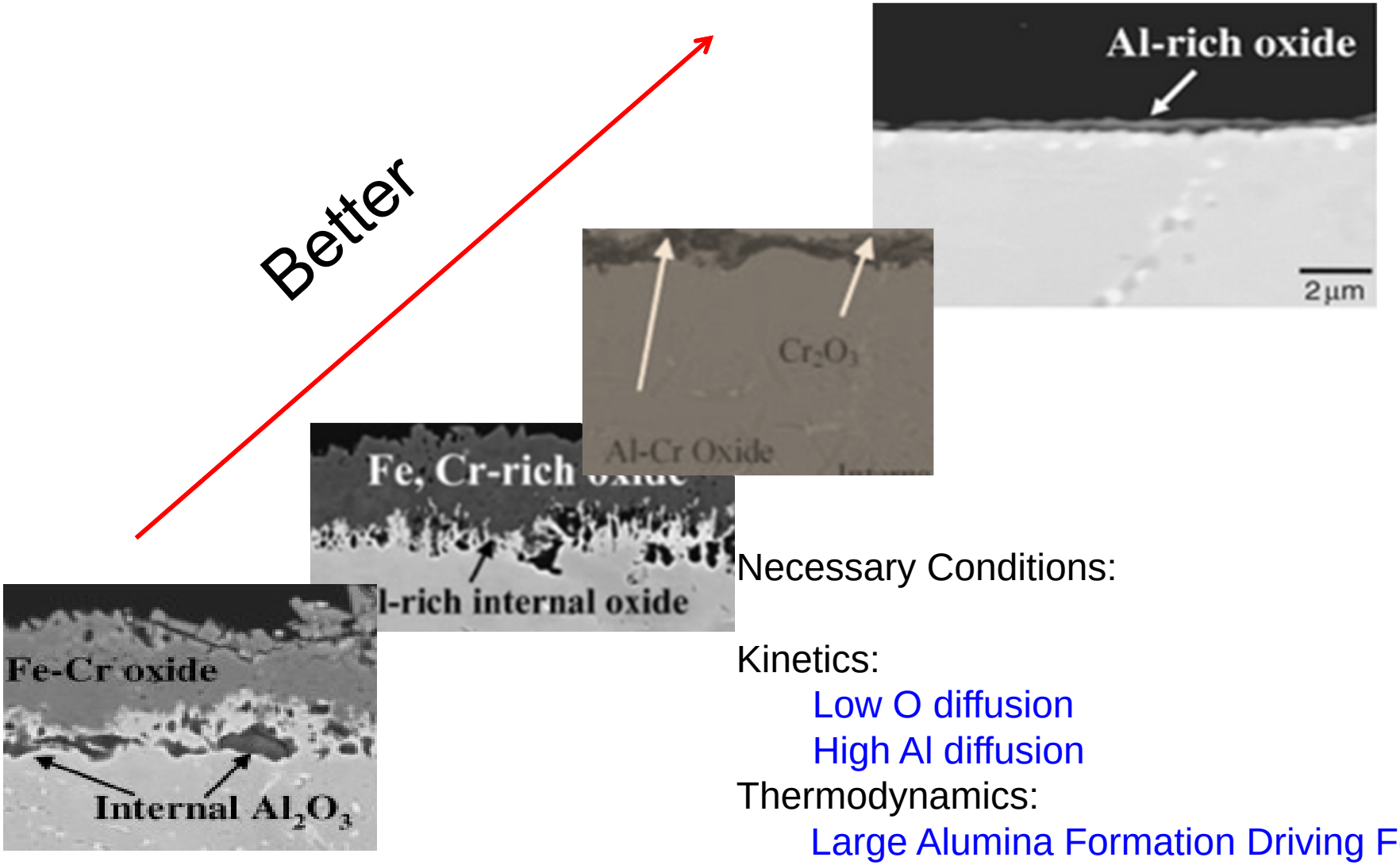

Necessary Conditions:

Kinetics:

- Low O diffusion
- High Al diffusion

Thermodynamics:

- Large Alumina Formation Driving Forc

❖ Testing the 'Effective Growth Constant Criterion'

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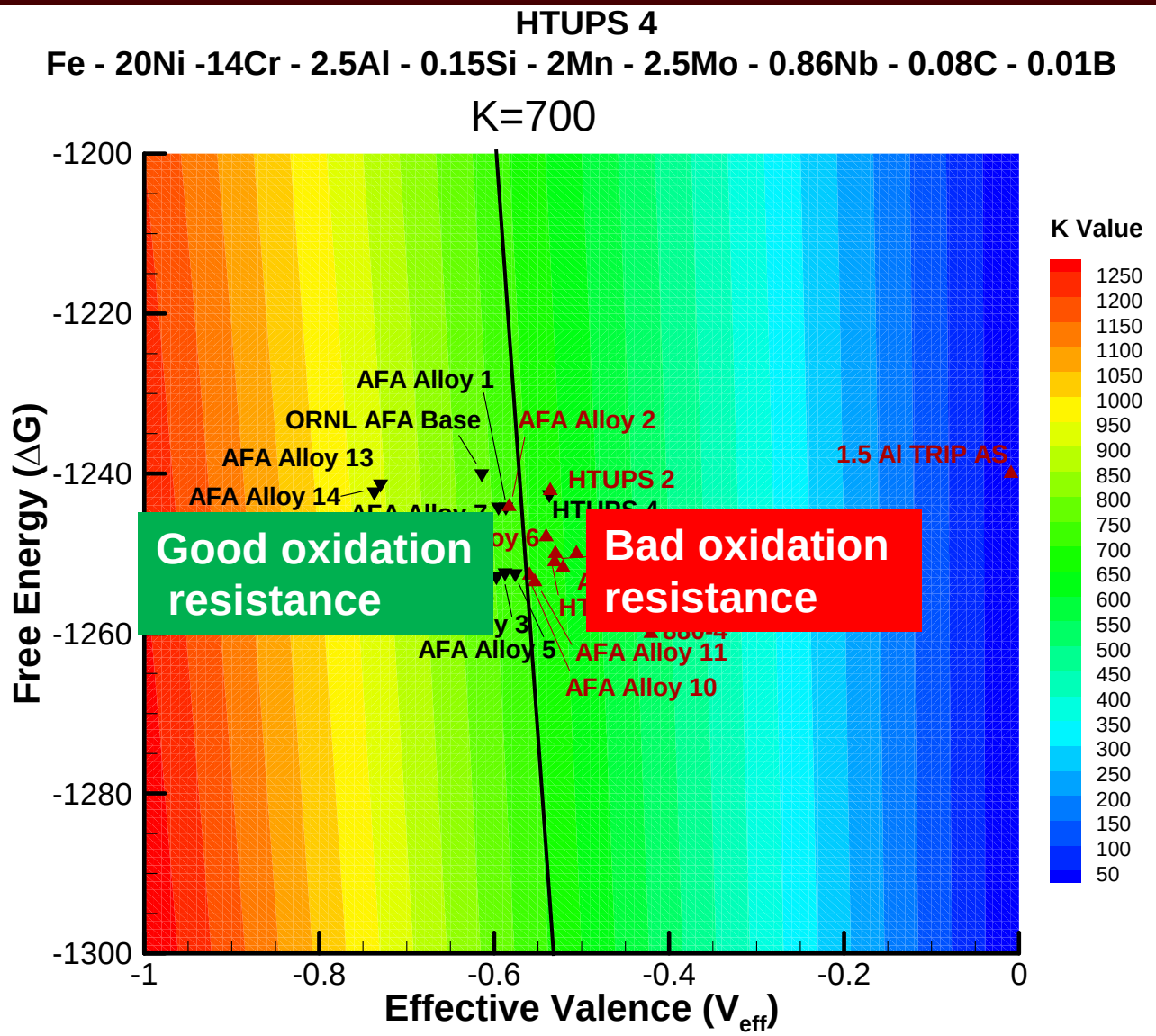
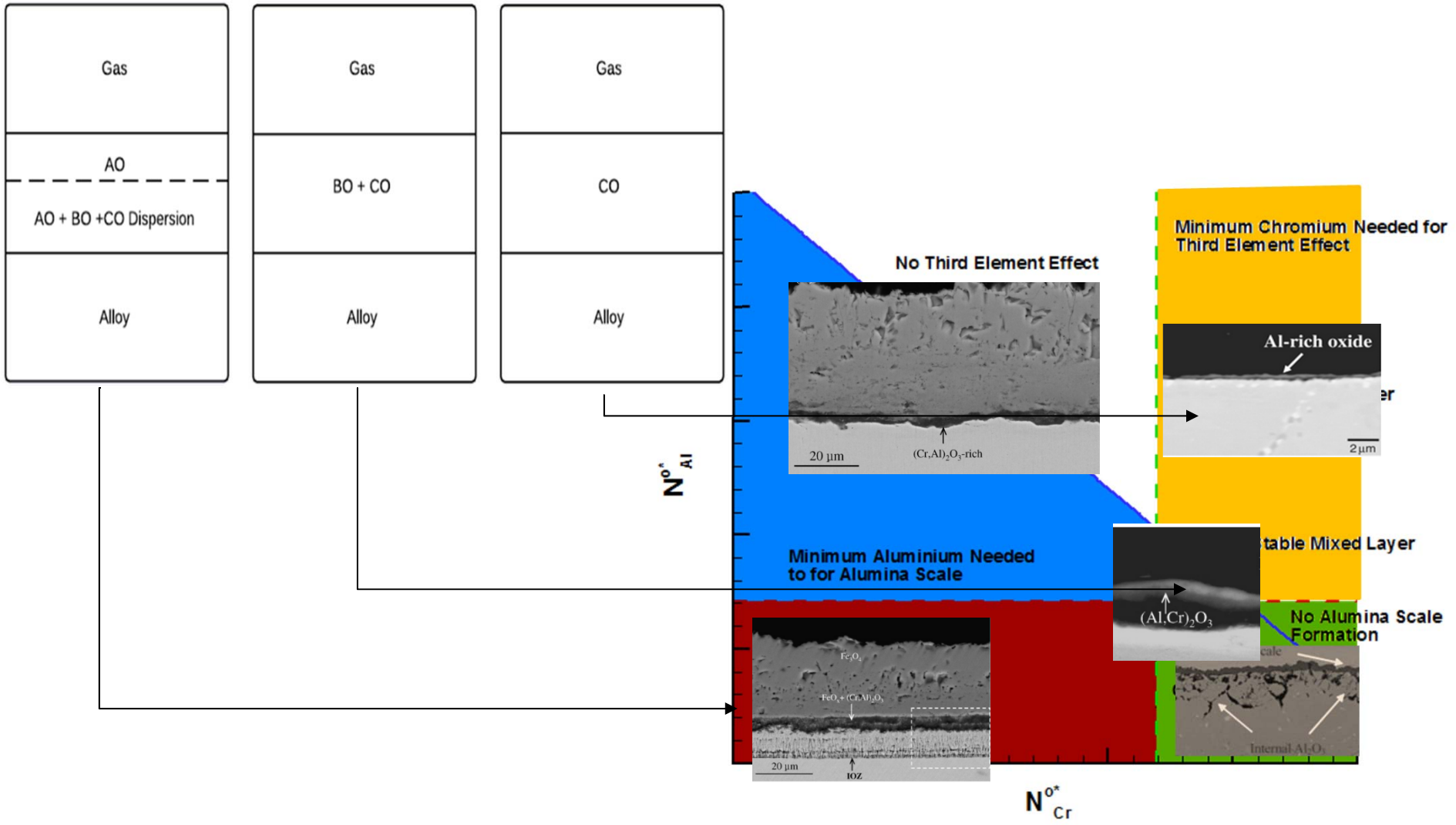
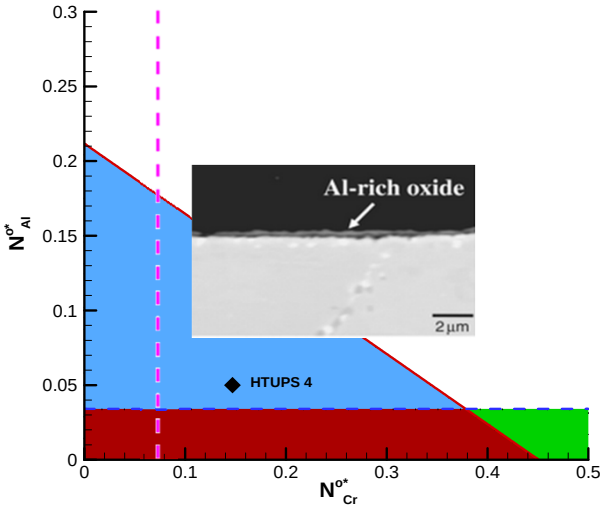


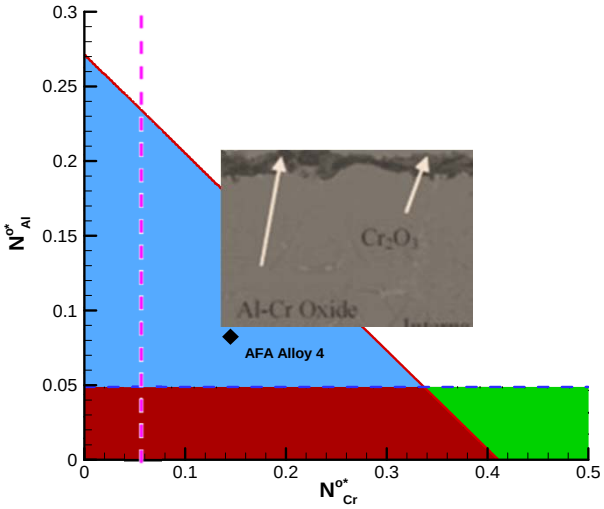
❖ Third Element Effect Predominance Maps

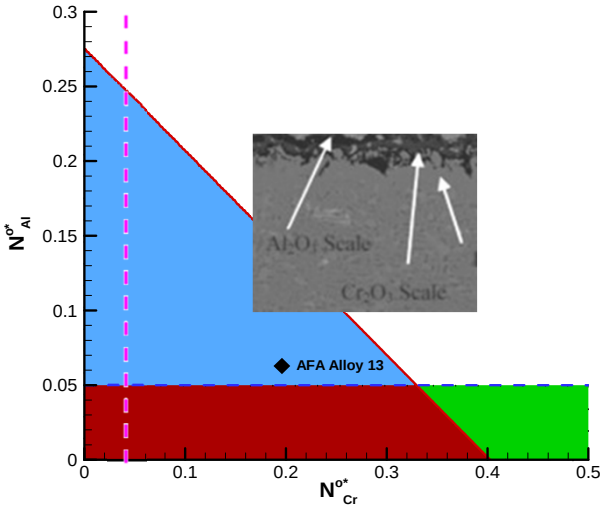
Unstable Oxide Layer → Mixed Stable Layer **or** Stable Layer



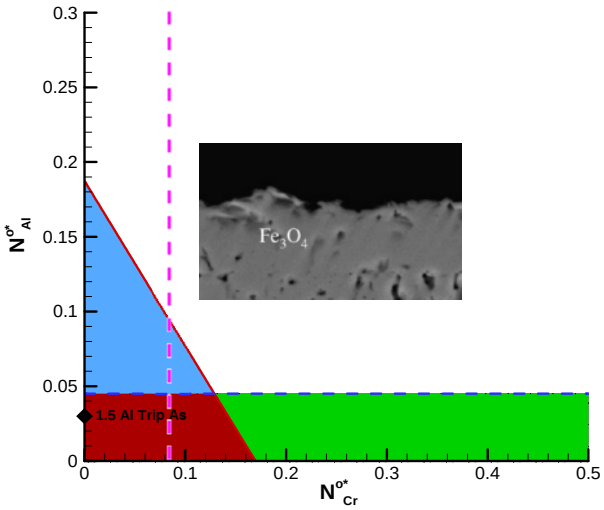
Testing Third Element Effect Predominance Maps

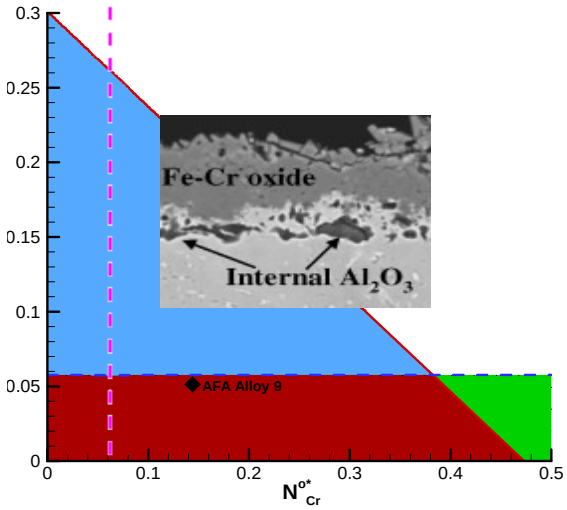


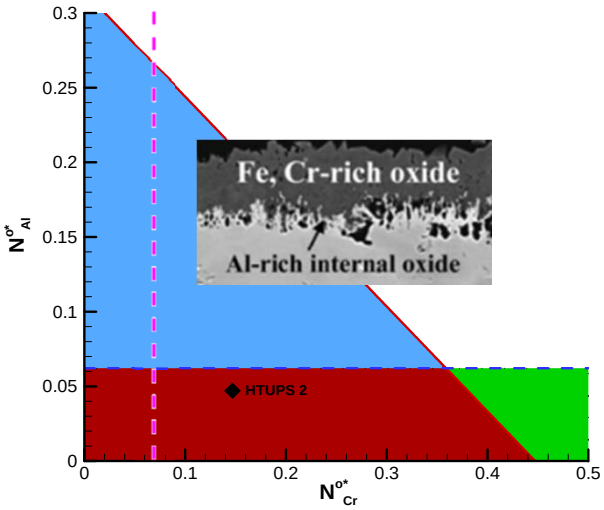




Fe	C	Mn	Si	Cr	Ni	Mo	Cu	W	V	Ti	Al	Nb
Bal.	0.076	1.95	0.15	14.20	20.00	2.46	0.00	0.00	0.50	0.31	2.40	0.14







Comparison of two Criteria

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	Fe	Ni	Cr	Al	Si	Mn	Mo	Nb	Ti	V	C	B	Cu	W	Effective Valence Model	Third Element Model	Experimental Result	Ref
AFA Alloy 1	Bal.	25.02	14.06	3.06	0.14	2.00	2.00	1.02	0.05	0.05	0.0470	0.0096	0.52	0.96	Pass	Pass	Pass	[64]
AFA Alloy 2	Bal.	25.05	13.99	3.03	0.15	2.00	2.00	1.00	0.05	0.05	0.2040	0.0104	0.51	0.96	Fail	Pass	Fail	[64]
AFA Alloy 3	Bal.	25.05	14.03	4.13	0.14	2.00	2.00	0.99	0.05	0.05	0.0490	0.0100	0.52	0.96	Pass	Pass	Pass	[64]
AFA Alloy 4	Bal.	25.03	13.97	4.11	0.14	2.00	1.99	1.01	0.05	0.05	0.2090	0.0104	0.52	0.96	Pass	Pass	Pass	[64]
AFA Alloy 5	Bal.	25.02	13.84	3.06	0.13	1.99	2.00	1.02	0.05	0.05	0.1060	0.0078	0.51	0.96	Pass	Pass	Pass	[64]
AFA Alloy 6	Bal.	20.05	13.84	3.07	0.13	2.00	1.99	1.01	0.05	0.05	0.2000	0.0080	0.52	0.97	Fail	Pass	Fail	[64]
AFA Alloy 7	Bal.	25.05	13.98	4.17	0.14	1.99	1.98	2.53	0.05	0.05	0.2010	0.0092	0.52	0.97	Pass	Pass	Pass	[64]
AFA Alloy 8	Bal.	12.08	13.84	2.52	0.13	4.99	0.15	1.03	0.05	0.05	0.1710	0.0110	3.04	0.15	Fail	Fail	Fail	[64]
AFA Alloy 9	Bal.	12.05	13.84	2.52	0.13	6.79	0.15	1.01	0.05	0.05	0.2000	0.0090	3.06	0.15	Fail	Fail	Fail	[64]
AFA Alloy 10	Bal.	12.04	13.92	2.52	0.14	9.93	0.15	1.01	0.05	0.05	0.1000	0.0090	3.06	0.14	Pass	Fail	Fail	[64]
AFA Alloy 11	Bal.	12.09	13.89	2.54	0.14	9.96	0.15	1.01	0.05	0.05	0.2000	0.0090	3.06	0.15	Fail	Fail	Fail	[64]
AFA Alloy 12	Bal.	32.06	18.69	3.10	0.13	6.96	0.15	3.32	0.05	0.05	0.1140	0.0017	0.15	0.14	Fail	Fail	Fail	[64]
AFA Alloy 13	Bal.	32.06	18.69	3.10	0.13	0.15	0.15	3.32	0.05	0.05	0.1110	0.0017	0.15	0.14	Pass	Pass	Pass	[64]
AFA Alloy 14	Bal.	32.08	18.72	3.08	0.13	0.15	0.15	3.27	0.05	0.05	0.0160	0.0018	0.15	0.14	Pass	Pass	Pass	[64]
AFA Alloy Base	Bal.	25.20	14.90	3.00	0.15	1.90	2.00	2.50	0.00	0.00	0.0900	0.0100	0.00	0.00	Pass	Pass	Pass	[8]
880-4	Bal.	24.40	9.70	4.80	0.40	0.15	0.00	0.00	0.00	0.00	0.0300	0.0005	0.00	0.00	Fail	Pass	Fail	[8]
1.5 Al Trip As	Bal.	0.00	0.00	1.50	0.06	1.55	0.00	0.00	0.00	0.00	0.1100	0.0000	0.00	0.00	Fail	Fail	Fail	[8]
HTUPS 2	Bal.	20.00	14.20	2.40	0.15	1.95	2.46	0.14	0.31	0.50	0.0760	0.0110	0.00	0.00	Fail	Fail	Fail	[6, 15]
HTUPS 3	Bal.	19.98	14.21	3.67	0.10	1.92	2.46	0.14	0.31	0.49	0.0790	0.0110	0.00	0.00	Fail	Pass	Fail	[6, 15]
HTUPS 4	Bal.	19.95	14.19	2.48	0.15	1.95	2.46	0.86	0.00	0.00	0.0750	0.0100	0.00	0.00	Fail	Pass	Pass	[6, 15]

❖ Prediction of Stacking Fault Energy as a Function of Alloying Additions

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Models:

Experimental Measurements
- (A. Dumay 2006)
- (Schramm 1975)
- (Xing Tian 2008)
- Many more

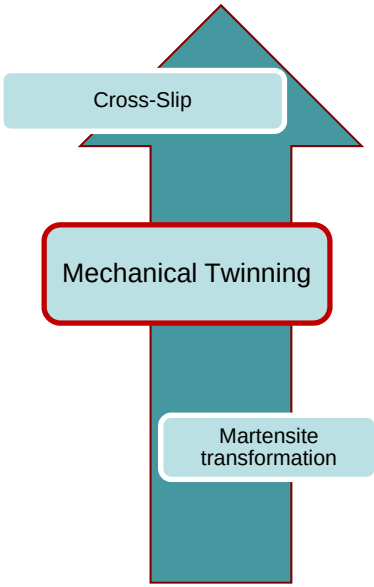
Theoretical Predictions
- (Cohen 1976)
- (Mullner 1998)
- (Jacques 2010)
- (Vitos 2011)
- (Q. Lu 2013)
- (K. Ishida 1976)
- Many more

Effects on SFE:

1. Alloying elements
2. Temperature
3. Interstitials

Prediction:

SFE: High / Low



❖ Relevant to creep, strain deformation, annealing twins, formation of dislocations, stress corrosion cracking, phase transformation stability, and electron/vacancy density, but we want to optimize SFE to ensure formation of deformation twins

The problem : Unpredictability !

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- Significant inconsistencies

(computational and experimental)

"..same alloying element cause totally opposite changes....no universal composition equations for SFE can be established...."

Vitos et al., 2006

Yet many linear regression equations relating SFE to composition in the literature.

AISI 316

44 mJ/m^2
Yonezawa et al., 2013

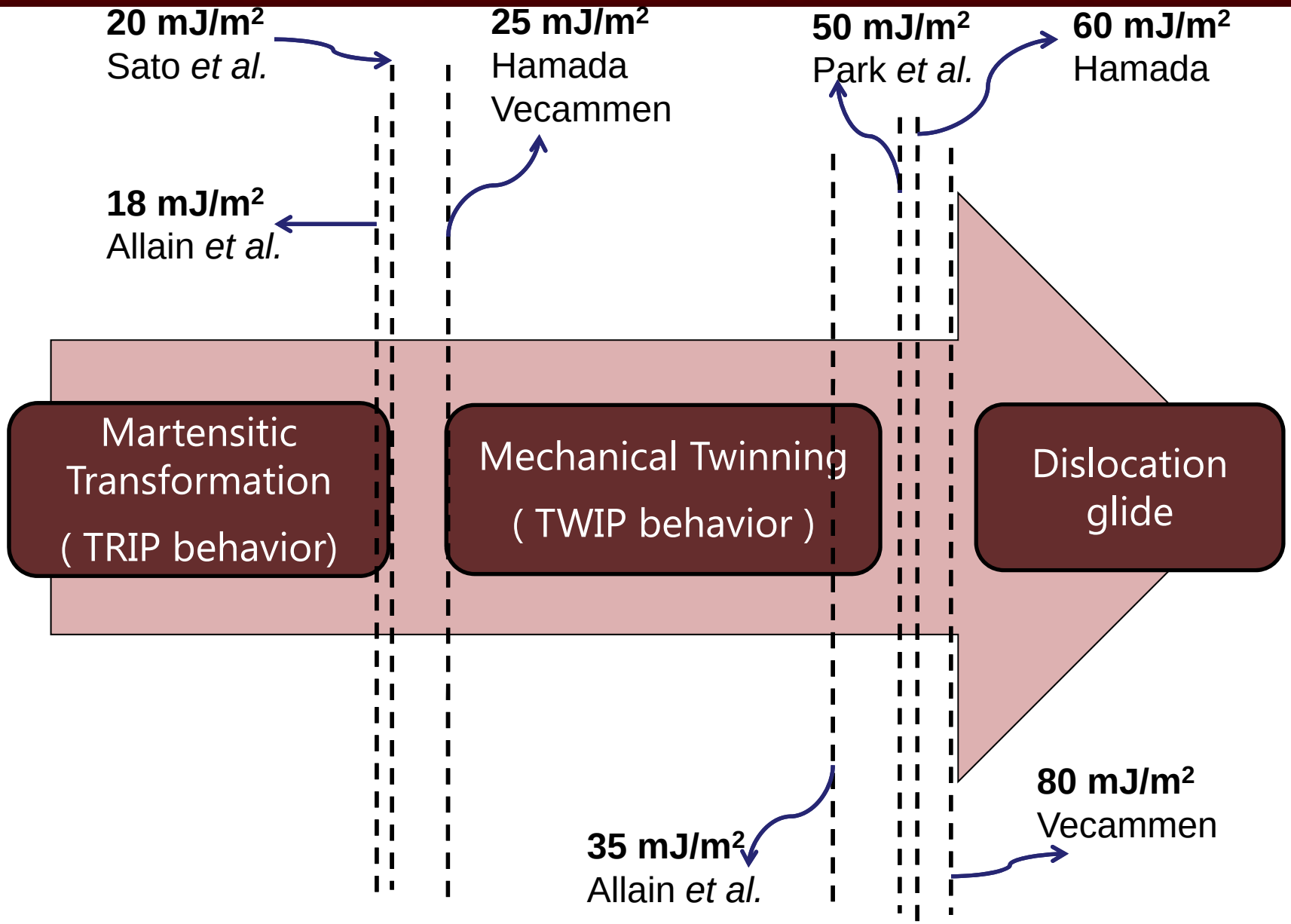
78 +/- 6 mJ/m^2
(Xray profile analysis)
Schramm and Reed , 1975

AISI 310

94 +/- 14 mJ/m^2
(Xray profile analysis)
Schramm and Reed , 1975

40 +/- 5 mJ/m^2
(TEM extended nodes)
Rhodes and Thompson, 1977

From Prediction to Classification



Neural Network-based Classifiers

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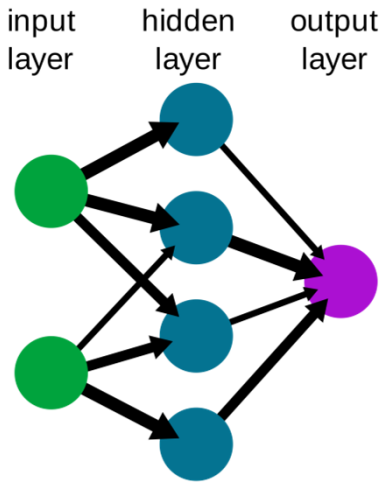
Machine Learning - **Artificial Neural Networks** for Classification



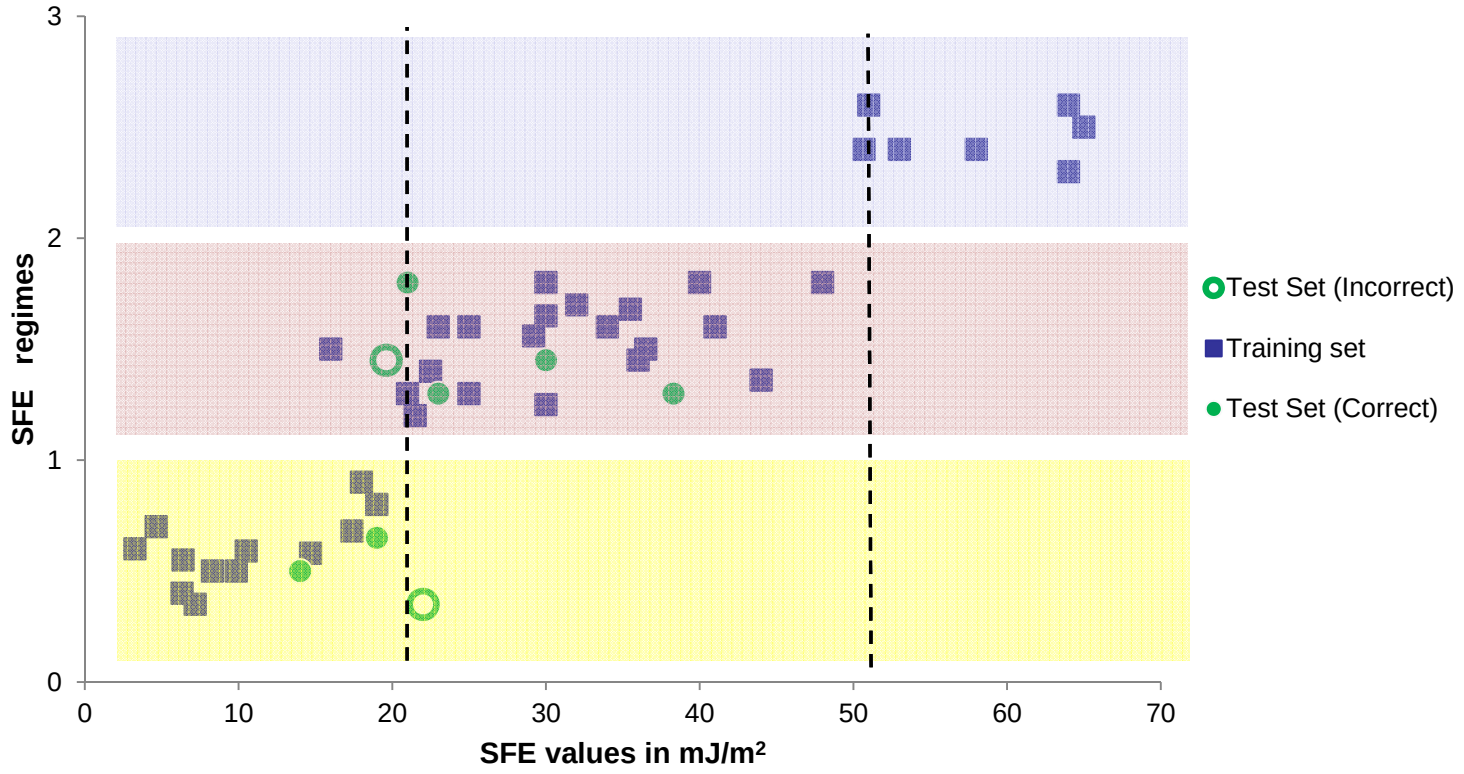
A basic neural network representation

Our Model

Input Layer	Hidden Layer	Output Layer
9 units representing wt.% of different elements	40 units to capture complex relationships between elements	3 units representing 3 different SFE regimes

Training Set: 60 alloy compositions
Accuracy : 97%

Model training and testing



1	Low SFE (<20 mJ/m^2)
2	Medium SFE (20-50 mJ/m^2)
3	High SFE (>50 mJ/m^2)

- The data collected was broken into training and testing sets.
- The ANN has trained well as evident (~97% accuracy)
- The ANN has generalized well which is shown from good predictions on Test set.

❖ Genetic Algorithm-based Optimization

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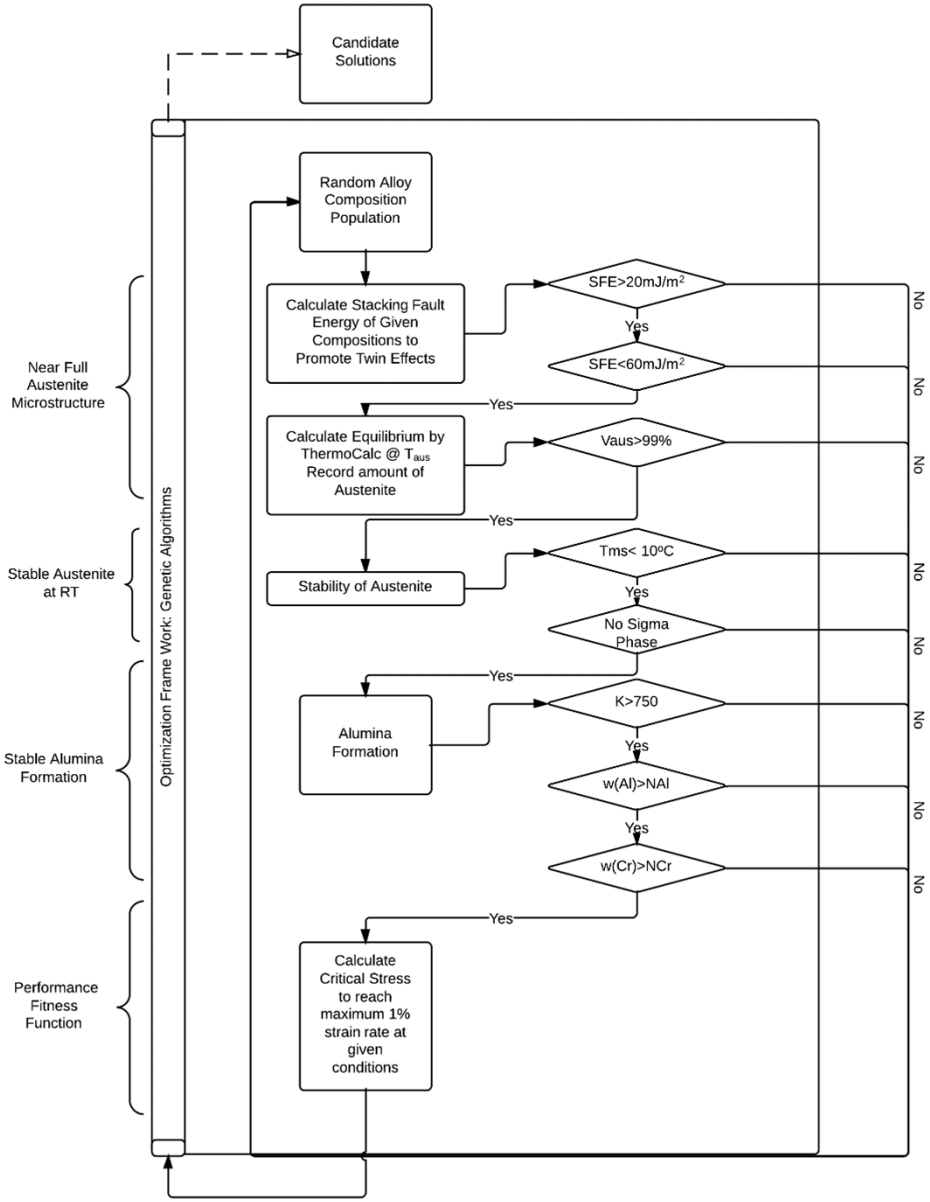
- Computational Genetic Algorithms are a necessity to
  - Streamline Alloy Design Process
  - Decrease Time and Cost of Alloy Discovery
  - Decrease Time and Cost of Alloy Refinement
- GA will be used to find a heat treatment process for
  - Maximizing FCC Phase
  - Minimize BCC and Unwanted Phases
  - Ensure Twinnability through control of Stacking Fault Energy
  - Austenite Stability
  - Alumina Formation
  - Critical Stress for Creep



❖ Proposed Genetic Algorithm Alloys

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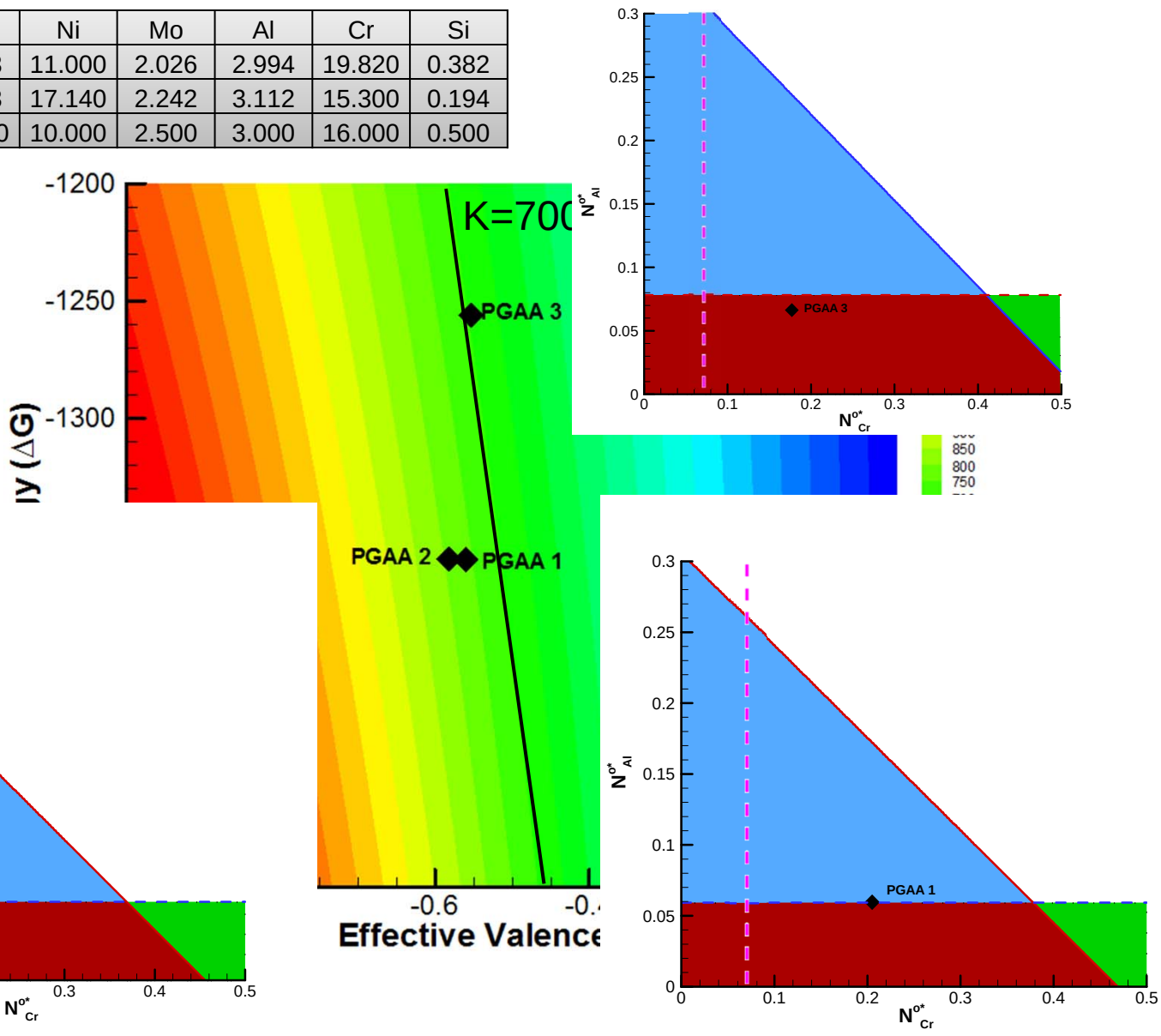
PGAA	Fe	C	Mn	Ni	Mo	Al	Cr	Si
1	Bal.	0.073	3.893	11.000	2.026	2.994	19.820	0.382
2	Bal.	0.088	8.993	17.140	2.242	3.112	15.300	0.194
3	Bal.	0.050	10.000	10.000	2.500	3.000	16.000	0.500

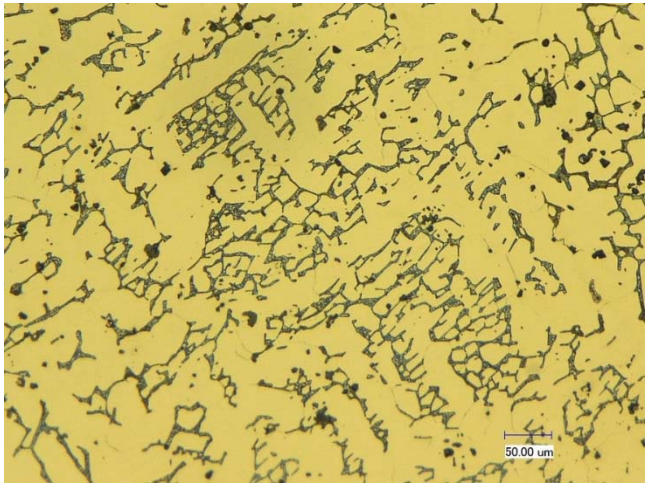


Computational Alloy Design

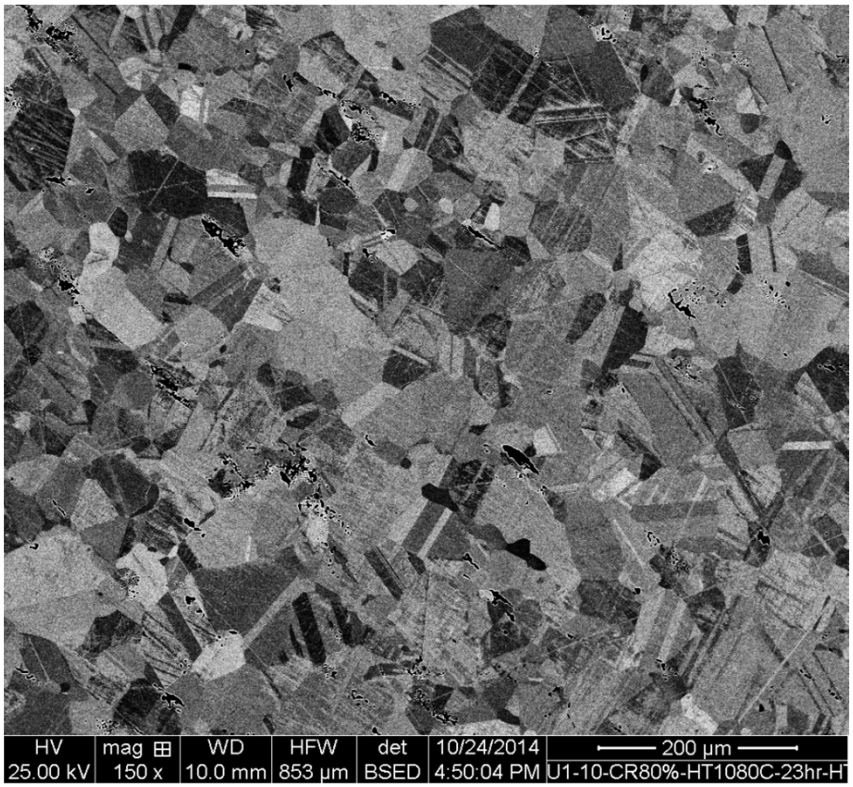
Second-Generation Alloys

wt.%	C	Mn	Ni	Mo	Al	Cr	Si	Fe
PGAA2	0.088	9	17.15	2.24	3.11	15.3	0.19	bal.

As-received, etched



Cold rolled 80%
HT1080°C-23ht
HT1150°C-1hr



Can the second generation alloy

- Form alumina?
- Undergo deformation twinning?

❖ ALLOY DESIGN

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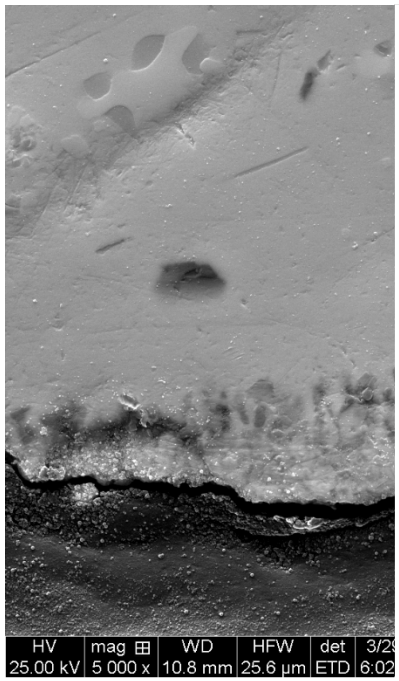
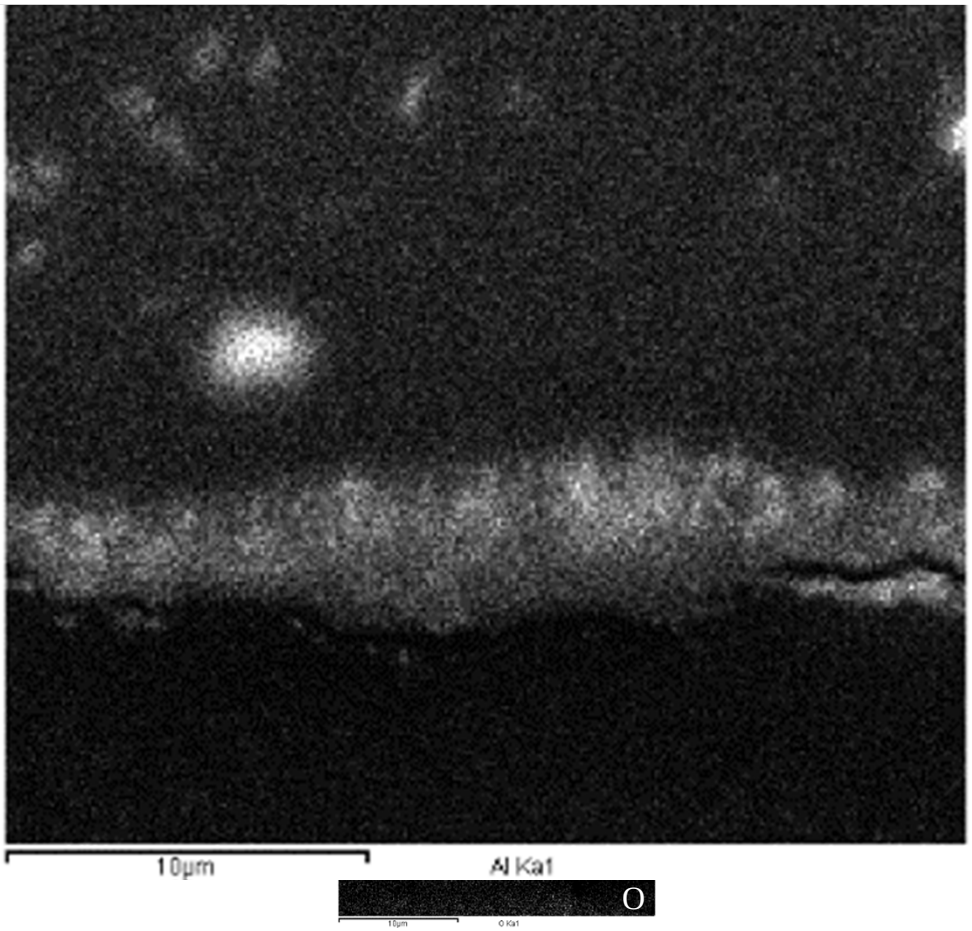
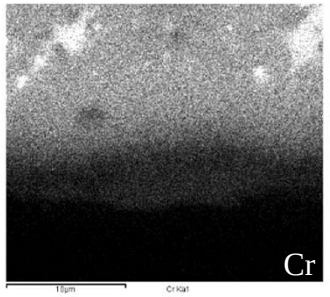
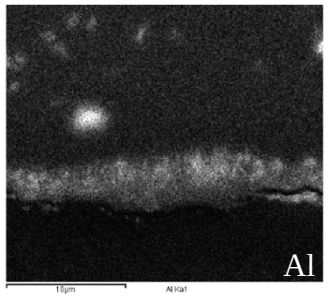
PGAA2: Oxidation at 850°C



156hrs



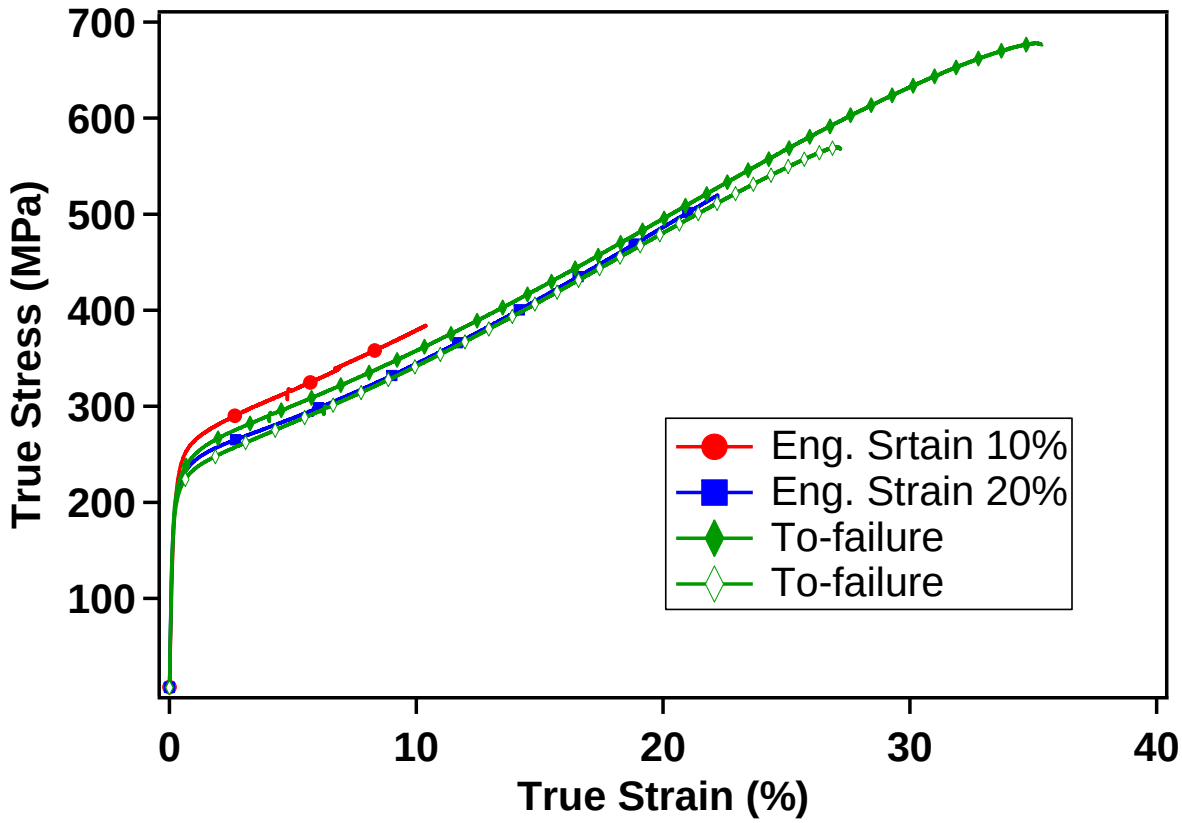




Aluminum diffusion zone gets thicker with time

❖ Does PGAA2 Show Deformation Twinning?

- Stress-strain curve suggests deformation by twinning. TEM in progress…



❖ SUMMARY

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1. Work on twinning:
   - Single crystals of three austenitic steels have been grown.
   - High volume fractions and hierarchical structure of deformation nano-twins were confirmed in single and polycrystals.
   - Nano-twins are thermally stable up to 900˚C. Deformation – annealing – deformation route can increase the twin density and the strength levels
2. A new method has been developed to successfully predict the alumina formation in multicomponent alloys.
3. We have developed an extensive datasets for SFE of austenitic stainless steels
4. We have developed a classifier to predict in a robust manner whether any alloy would have low, medium or high SFE
5. A preliminary alloy design framework has been developed through the use of Genetic Algorithms.
6. Two generations of new alloys have been designed and characterized.
7. First generation of designed alloys suffers from lack of twinning, two phase formation, and AlN formation
8. Second generation of designed alloys look more promising for alumina formation and twinning, more work is needed.

Computational Alloy Design

- ICME---Integrated Computational Materials Engineering
- Multi-objective optimization through Generic Algorithms