WHEN TRUST MATTERS

This work is supported by the Department of Energy, National Energy Technology Laboratory under award #DE-FE0031631, with NETL program manager Dr. Vito Cedro

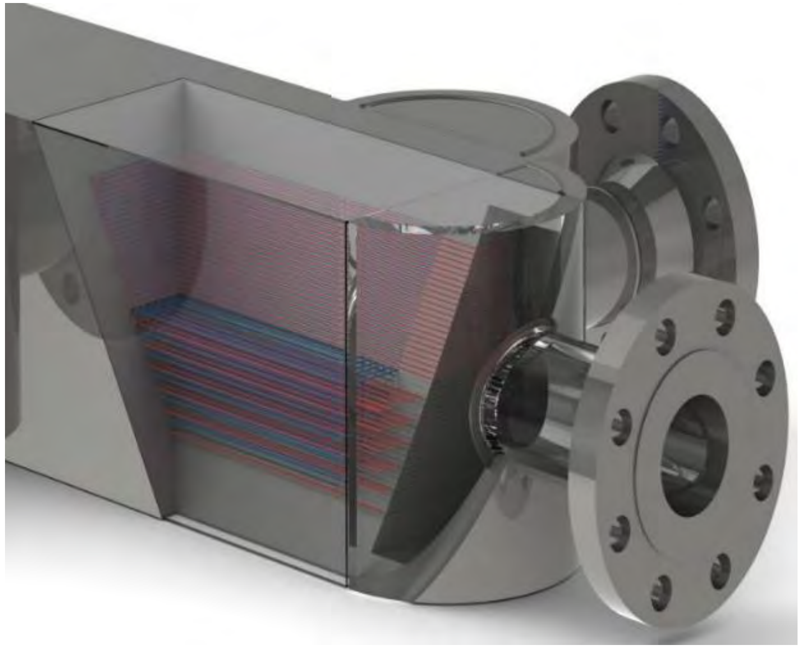
ICME for Advanced Manufacturing of Ni Superalloy Heat Exchangers with High Temperature Creep + Oxidation Resistance for Supercritical CO_2

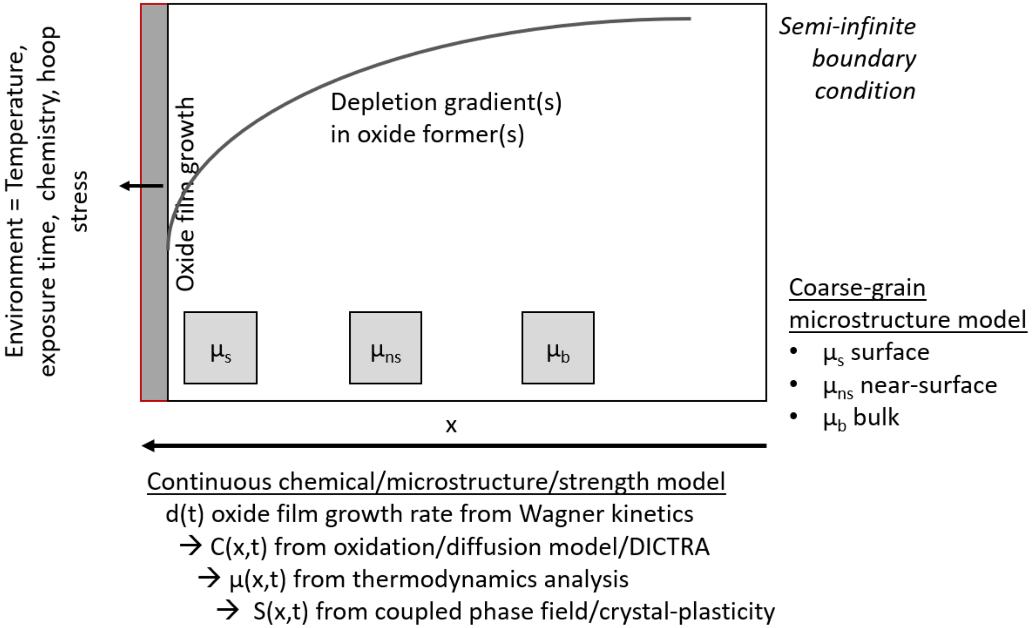
Christopher Taylor, Taiwu Yu, Pengyang Zhao, Supriyo Chakraborty, Steve Niezgoda, Yunzhi Wang, Brett Tossey
DNV GL, The Ohio State University, Quintus Technologies

08 June 2021

Objective

- Microchannel heat-exchangers with optimal durability
- Targeting additive manufacturing design for supercritical CO_2 power
  - 700-1000 C, 50+ years
- Optimized material combinations:
  - Surface skin: alumina former, such as Haynes alloy 224 (high temperature oxidation resistance)
  - Internal layer: chromia former having high creep resistance
- Avoid internal oxidation and dissolution of γ’ in near-surface
- Develop and Validate an Integrated Computational Materials Engineering approach to materials design

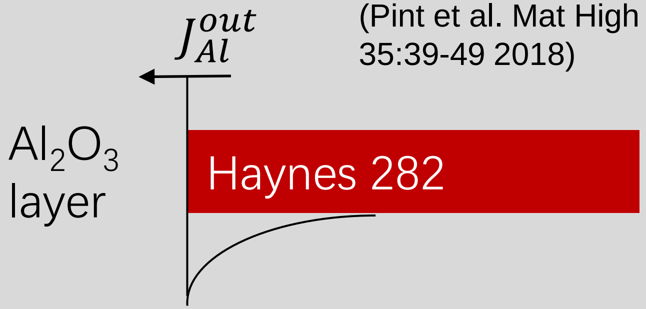




DICTRA Simulation Setup
flux boundary condition with k_p

$$J_{Al}^{out} = \frac{1}{2V_m^{Al_2O_3}}\sqrt{\frac{k_c}{t+t_0}}$$

At 900℃, k_c=5.49× 10^{-18} m²/s

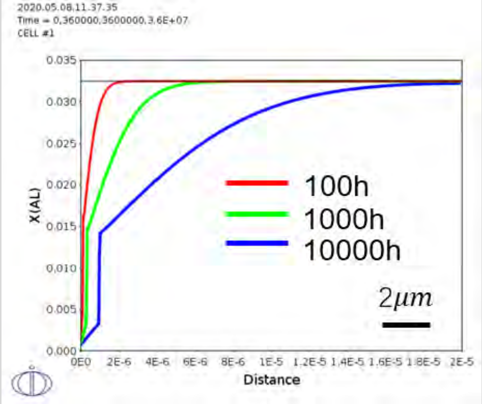


(Pint et al. Mat High Temp 35:39-49 2018)

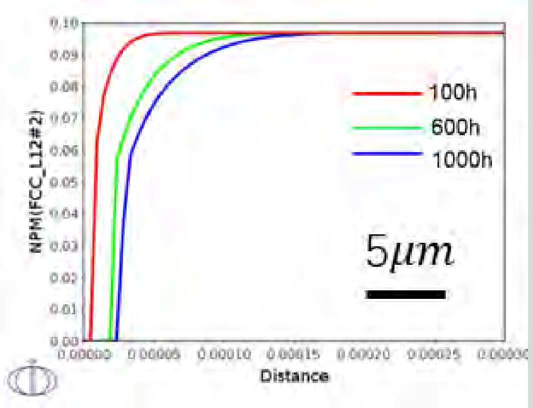
DICTRA calculation

DICTRA Simulation result

Concentration profile



Volume fraction of phases



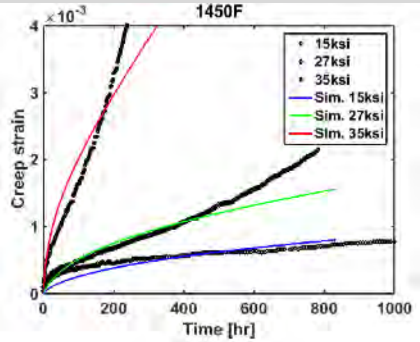
- Precipitates-free zone forms due to the loss of Al.
- Precipitates dissolves up to 3.4% of total volume in the two-phase region

ICME approach

Simulation of particle dissolution and prediction of heterogeneous microstructure: phase field method

Crystal plasticity model

Homogenized model

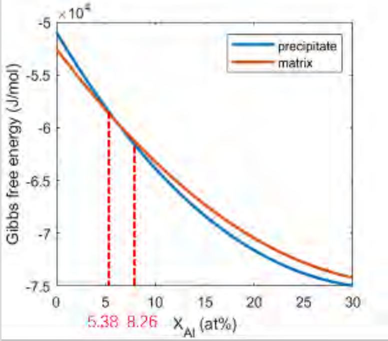


Predict the creep behavior with microstructure obtained through PFM

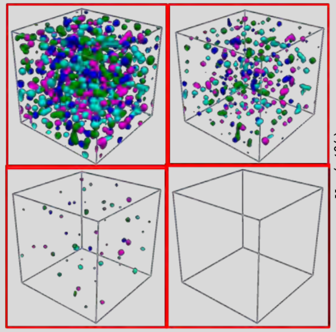
Simulation of creep

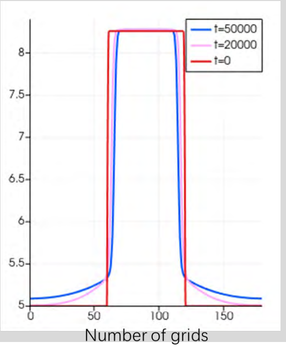
Crystal plasticity method

Phase field model (PFM) & results



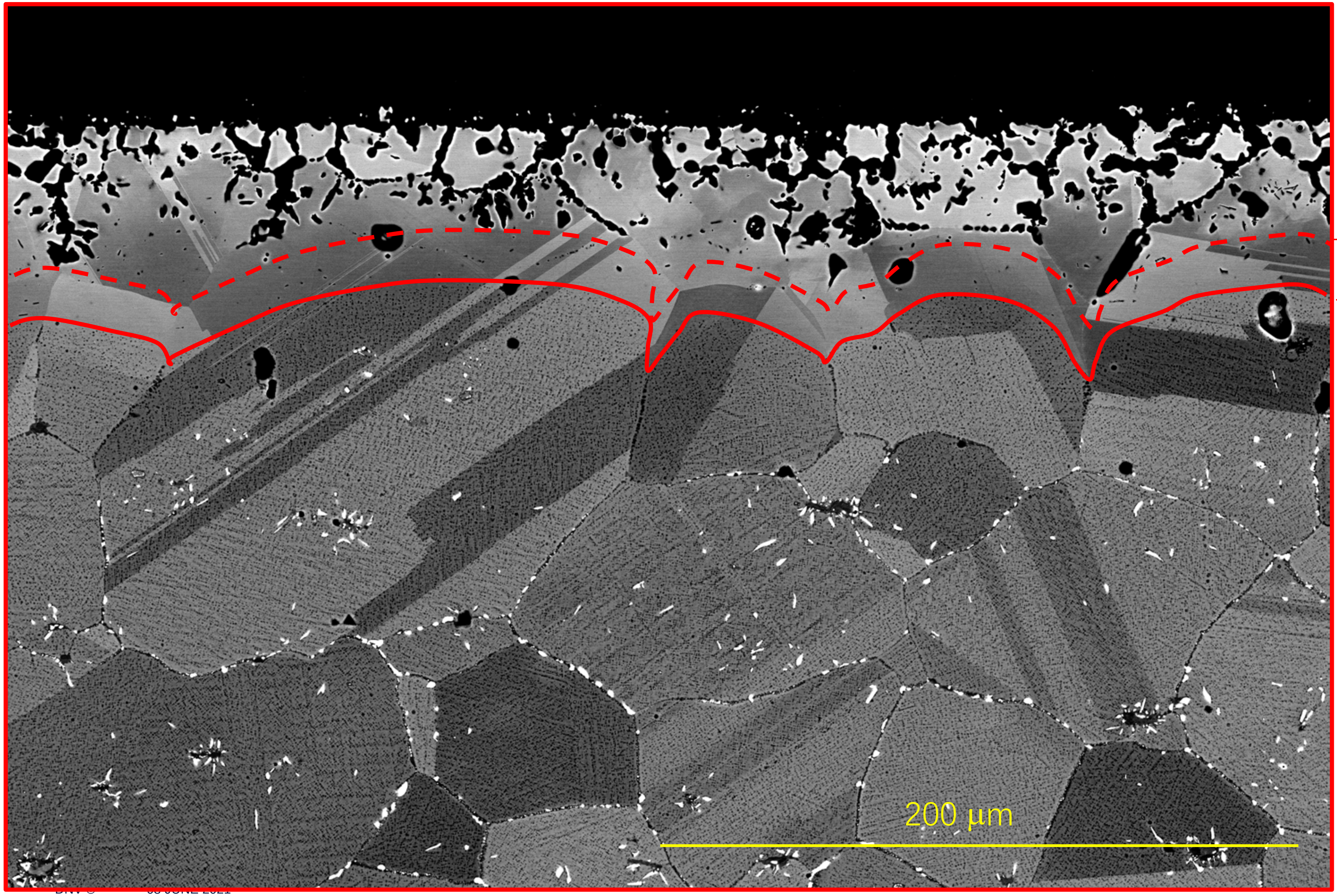
Particle dissolution





Microstructure

Alloy 282 - Exposed at 857°C , 4380 hrs



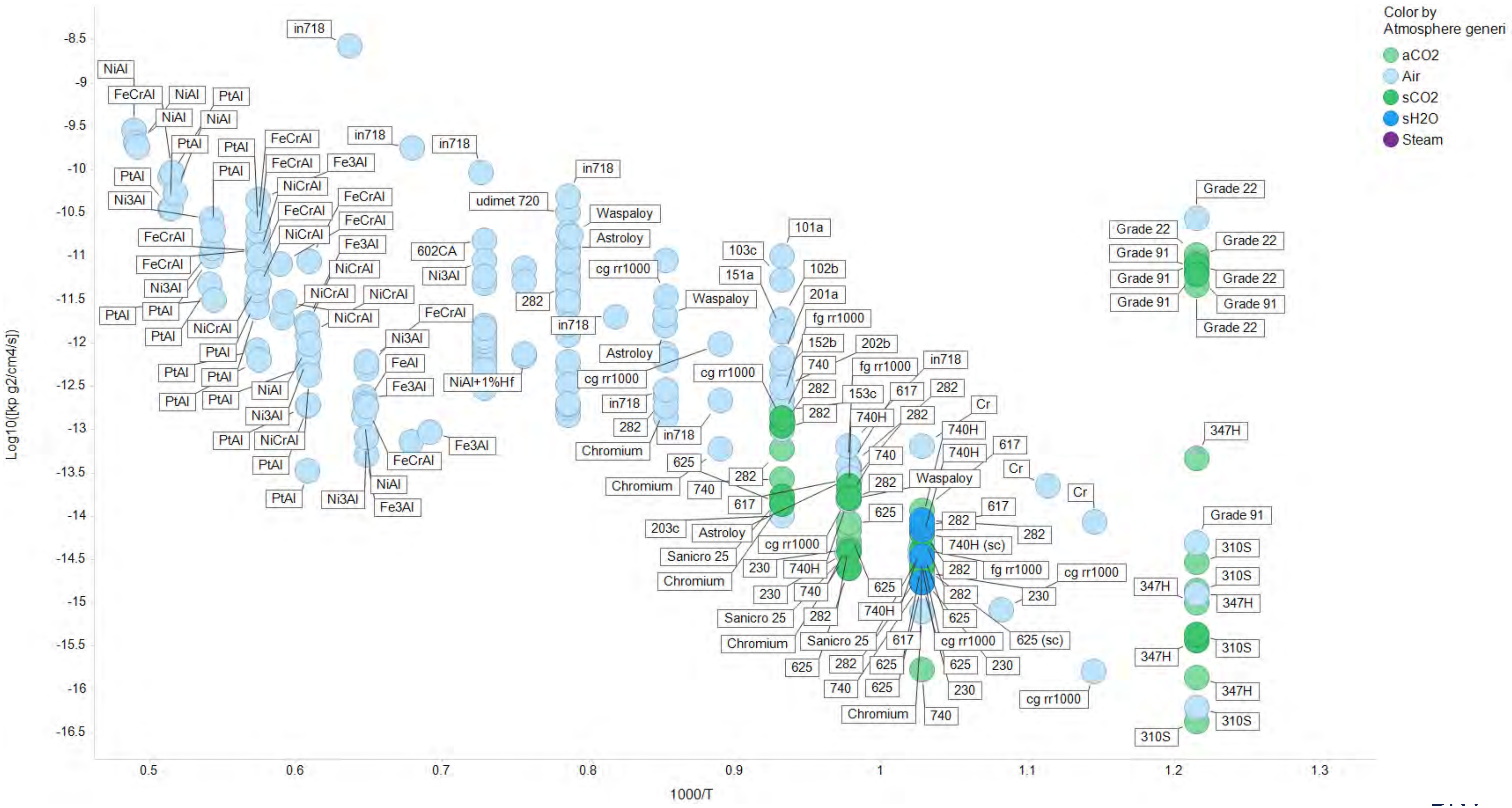
Al_2O_3 oxide network

γ' depletion zone 18.23μm

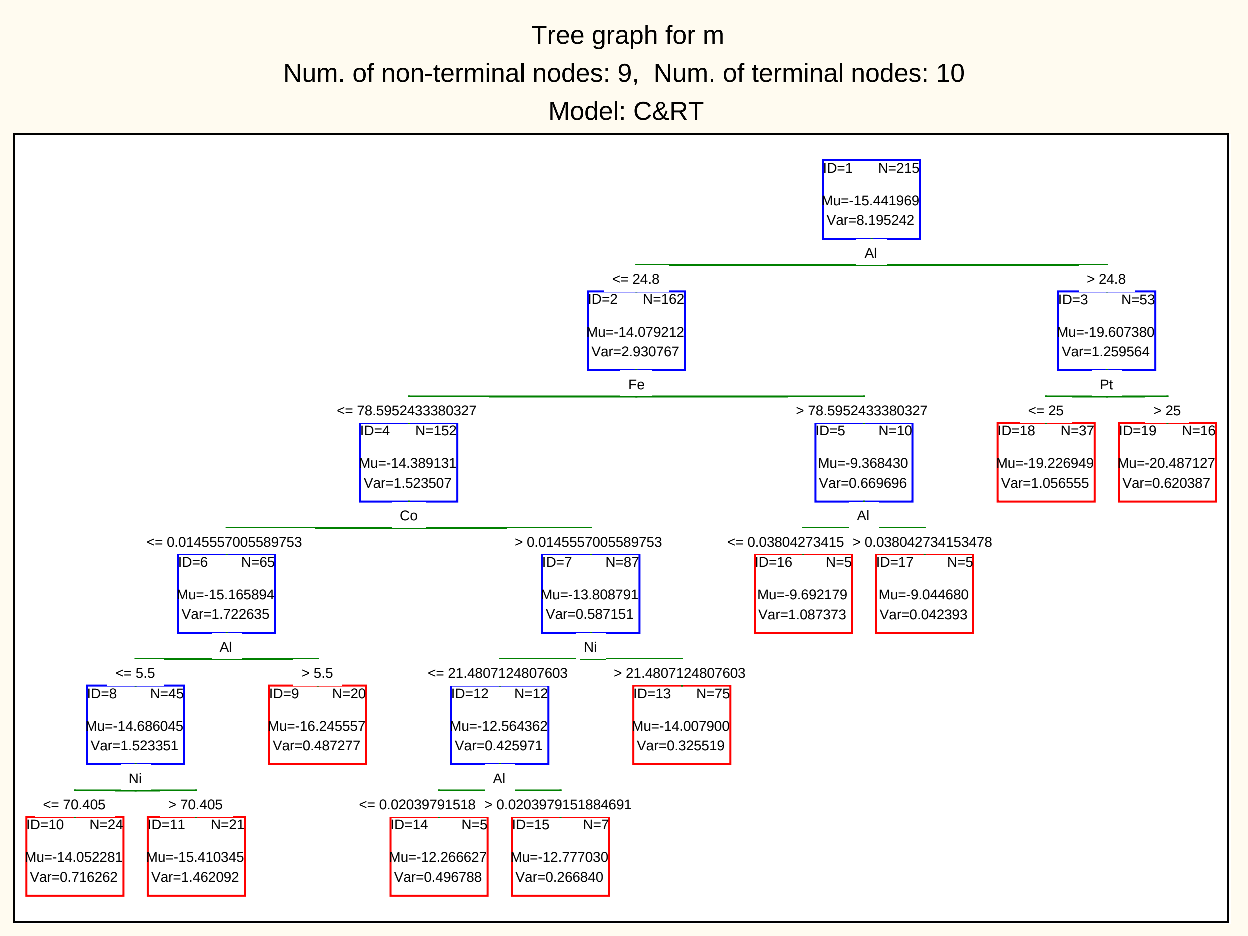
Matrix

(Credit to Gopal Babu Viswanathan)

Oxidation Rate Constants

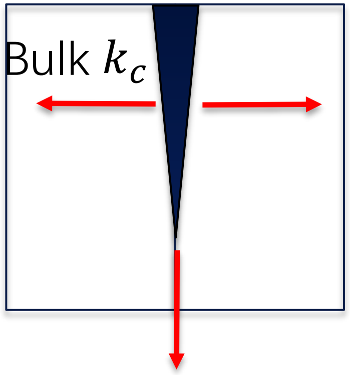


Pruned Decision Tree on Composition



- Decision tree mimics ‘family tree’ of alloys and superalloys
- Top split:
  - Aluminides vs other alloys
  - Then Pt vs other aluminides
- Second split:
  - Ferritic, austenitic low alloy steels with low activation energy
  - Stainless steels and nickel alloys
- Third split:
  - Low Ni vs High Ni
  - Alumina formers vs chromia formers
- Multiple trees possible (=> random forest)

Diffusion along grain boundary



Grain boundary k_c

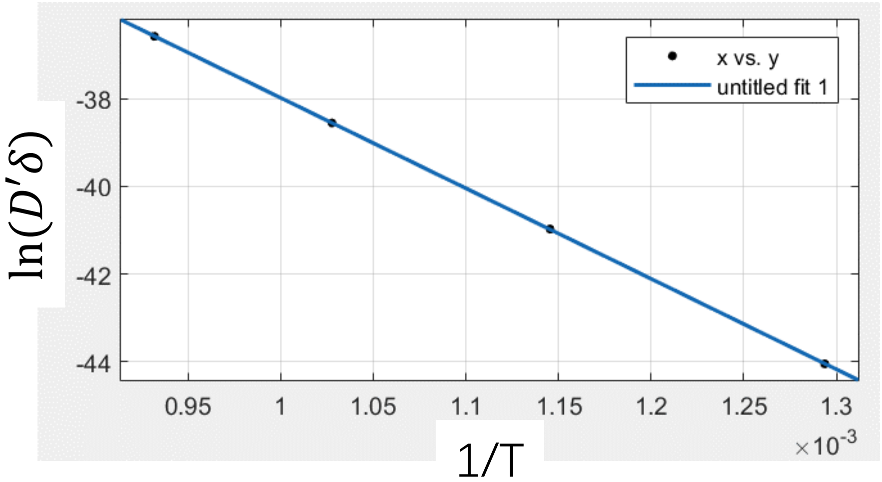
$$k_c \cong 6.4\left\{D_L^* + \frac{2(D'\delta)^*}{G_t}\right\}$$

δ=1nm

Table 1. Experimental results for oxide thickness and grain size, and values of diffusion coefficients.

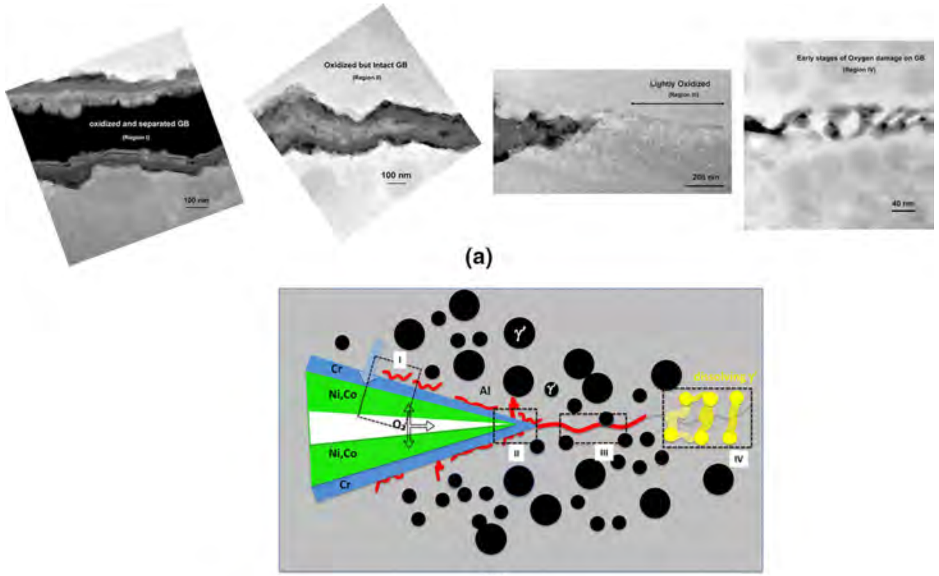
T (°C)	t (hour)	x (μm)	g† (μm)	$(D'\delta)^*$ (cm³ s⁻¹)	D^* (cm² s⁻¹)
800	1	4·2	0·54	$1{\cdot}3\times10^{-16}$	$2{\cdot}1\times10^{-14}$
	5	7·9	0·84		
	29	16·1	1·24		
	109	27·2	1·39		
700	1	1·9	0·41	$1{\cdot}8\times10^{-17}$	$1{\cdot}2\times10^{-15}$
	5	3·8	0·50		
	35	8·9	0·51		
	75	12·4	0·54		
	115	14·7	0·55		
600	1	0·79	0·23	$1{\cdot}6\times10^{-18}$	$3{\cdot}7\times10^{-17}$
	6	1·7	0·35		
	29	3·3	0·37		
	109	6·0	—		
500	5	0·28	0·15	$7{\cdot}4\times10^{-20}$	$4{\cdot}5\times10^{-19}$
	88	0·82	0·16		
	185	1·23	0·16		

A. Atkinson , R. I. Taylor & A. E. Hughes (1982) A quantitative demonstration of the grain boundary diffusion mechanism for the oxidation of metals, Philosophical Magazine A, 45:5, 823-833, DOI: 10.1080/01418618208239905



Time (h)	Estimated grain size G_t (um)	k_c of deepening along GB (m²/s)	Oxide intrusion depth (um)
1140 (T=925℃)	50	4.28× 10^{-16}	41.9
4380 (T=899℃)	80	3.17× 10^{-16}	70.6

Analysis

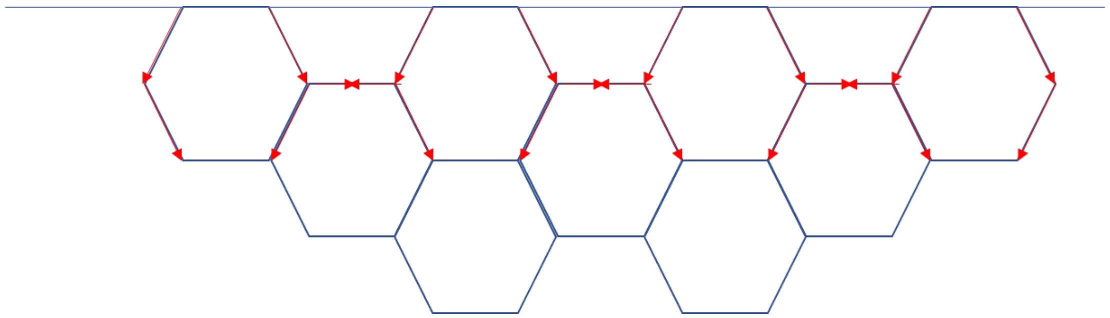


(a)

(b)

Viswanathan, G. B., David E. Mills, and Michael J. Mills. "Oxidation-Related Microstructural Changes at a Crack Tip in Waspaloy After Elevated-Temperature Dwell-Fatigue Testing." *Metallurgical and Materials Transactions A* 50.12 (2019): 5574-5580.

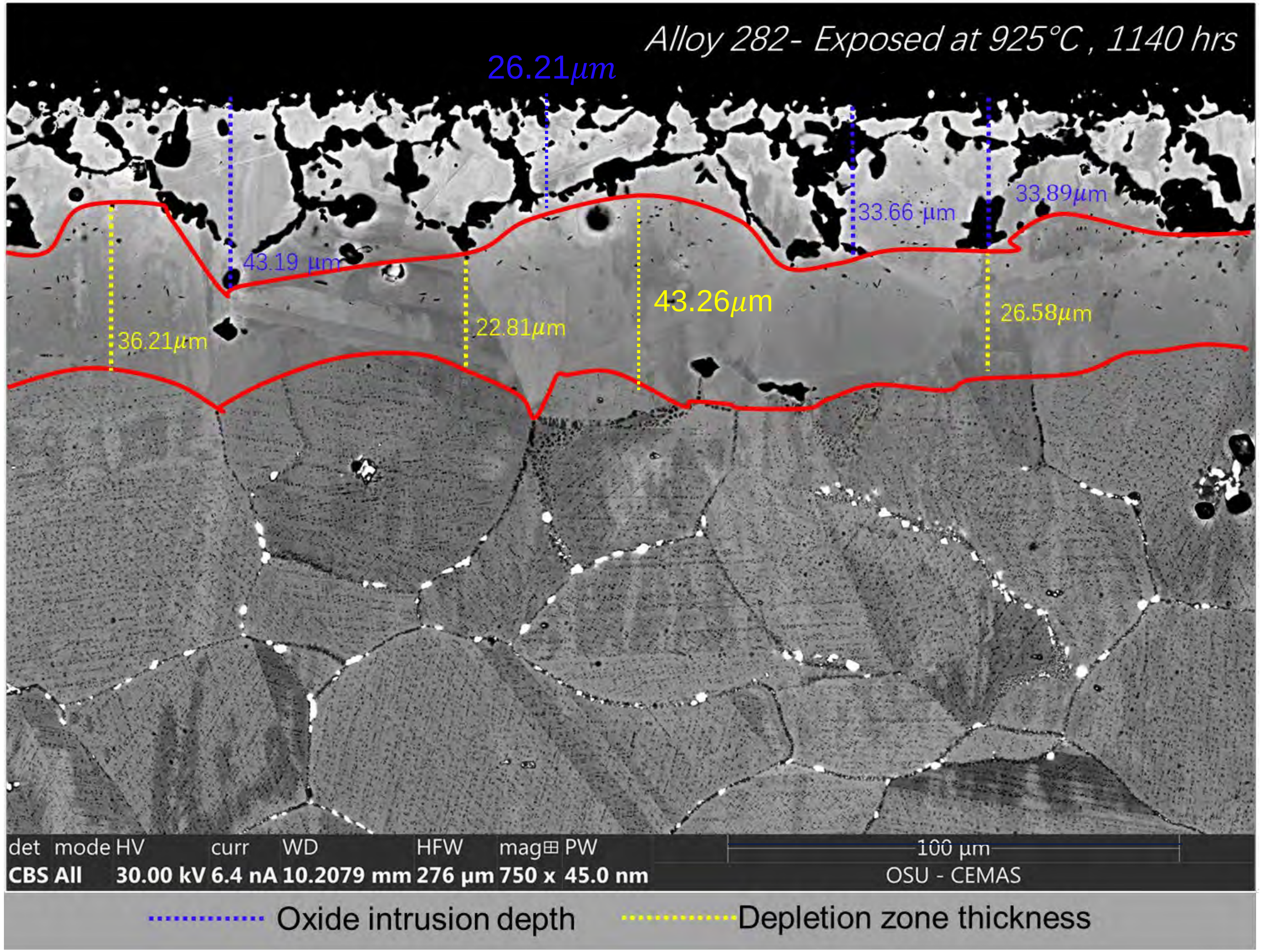
Al_2O_3 network



Oxide propagation path

Time (h)	Oxide intrusion depth (um)	Actual depth ($\times \sin 60°$) (um)	Equivalent bulk k_c (m^2/s)
1140 (T=925℃)	41.9	36.3	6.94×10^{-18}
4380 (T=899℃)	70.6	61.2	5.49×10^{-18}

Compare with the experiment



(Credit to Gopal Babu Viswanathan)

Oxide intrusion depth

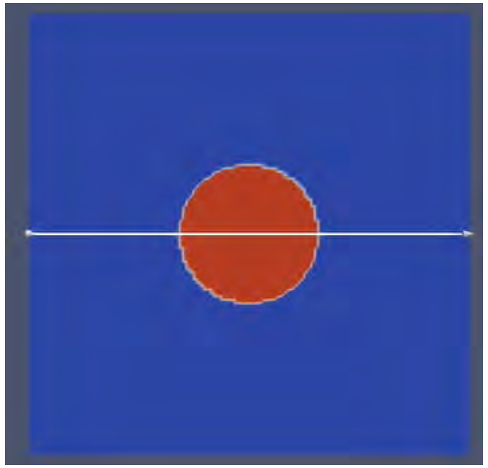
Time (h)	Oxide intrusion depth (um)	Exp results (um)
1140 (T=925℃)	36.3	33.0±6.6

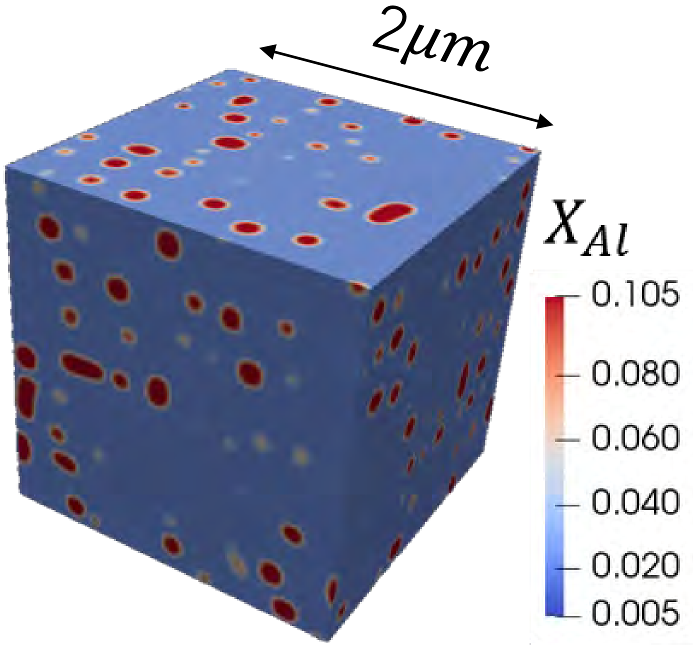
Depletion zone thickness

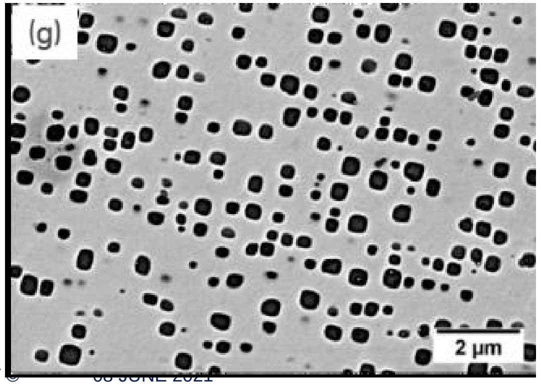
Time (h)	Depletion zone thickness (DICTRA) (um)	Exp results (um)
1140 (T=925℃)	23.9	32.2±9.3

Phase Field Simulation in Depleted Zone

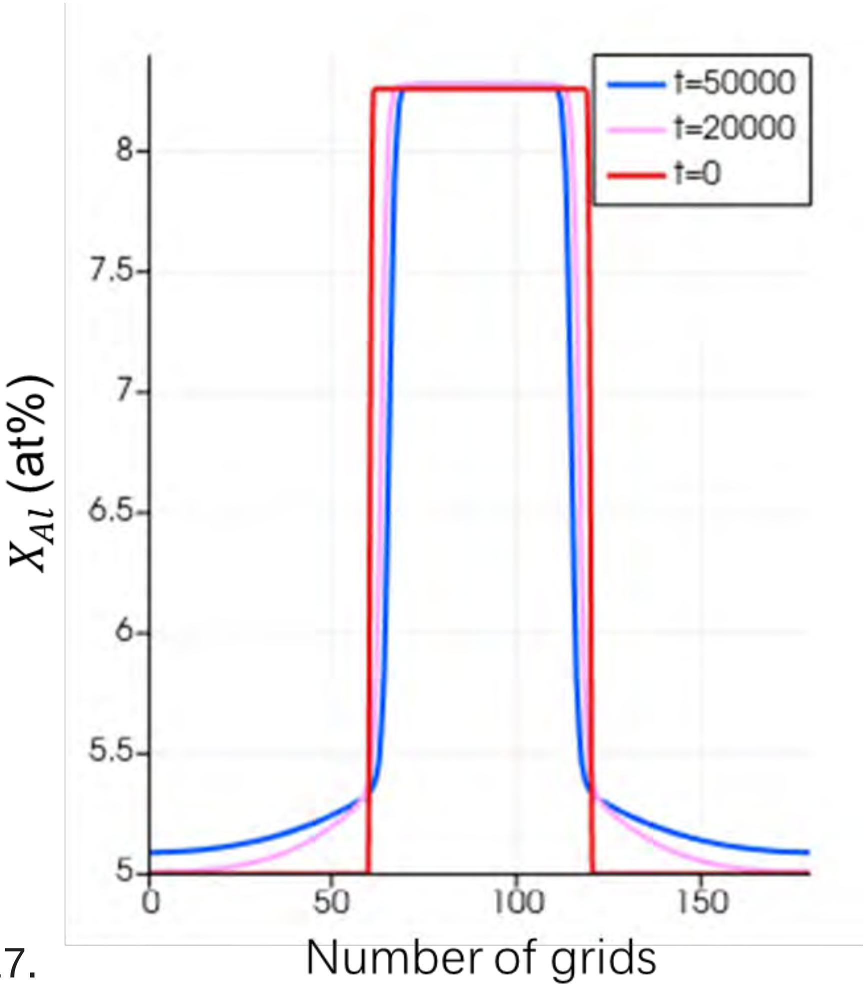
Particle growth





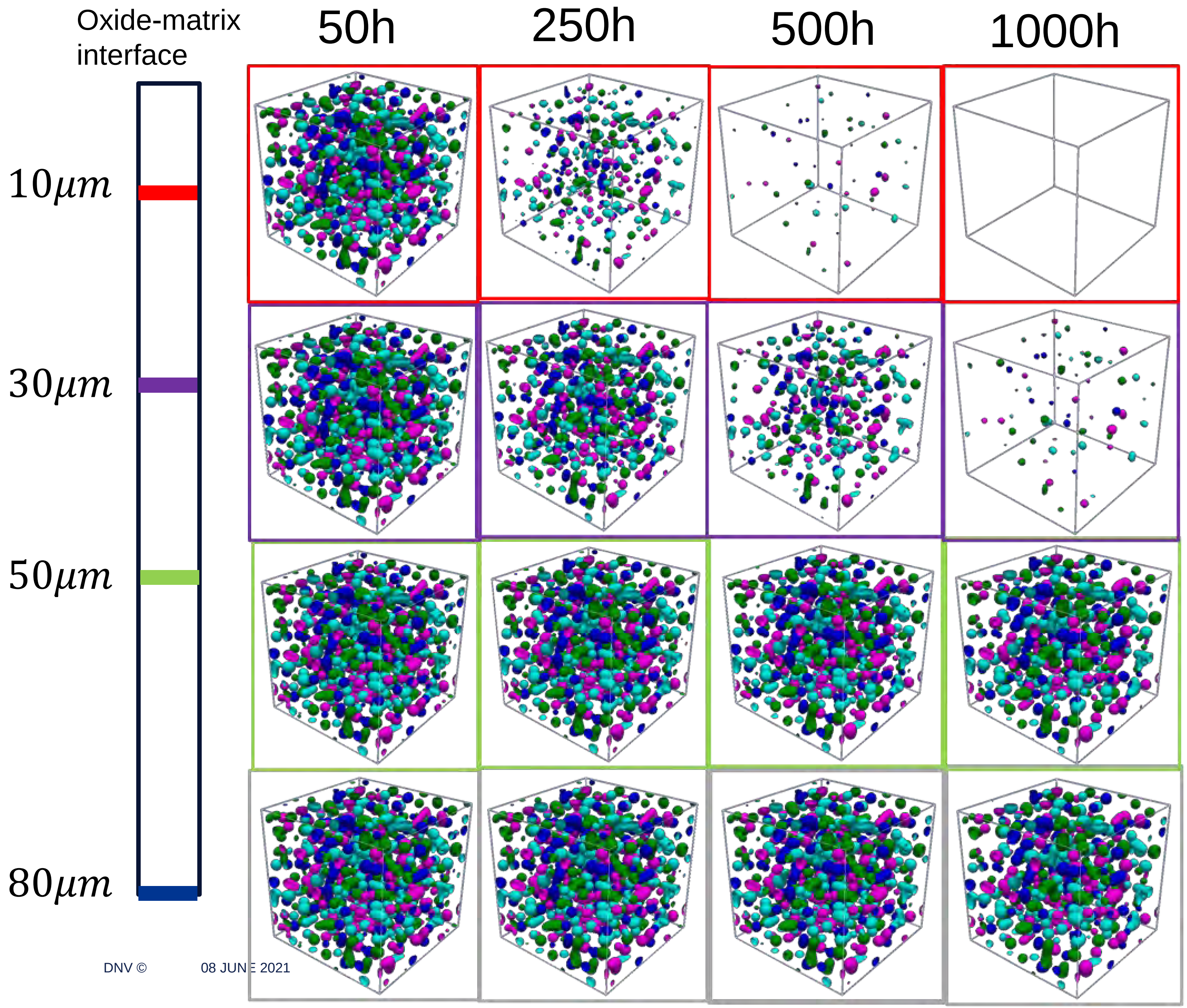


Particle dissolution



Shen, Chen, et al. No. DOE-GE-FE24027. GE Global Research, NIskayuna, NY (United States), 2017.

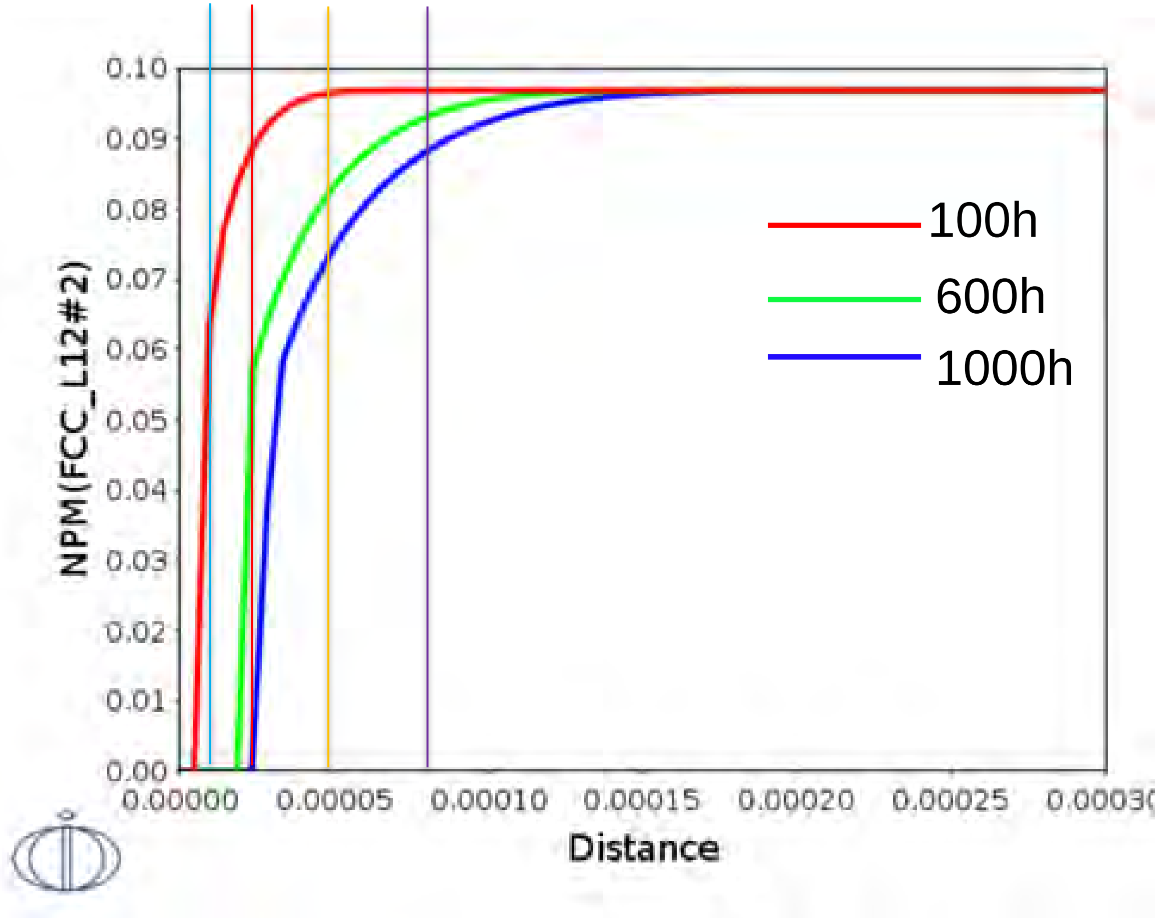
Simulation results

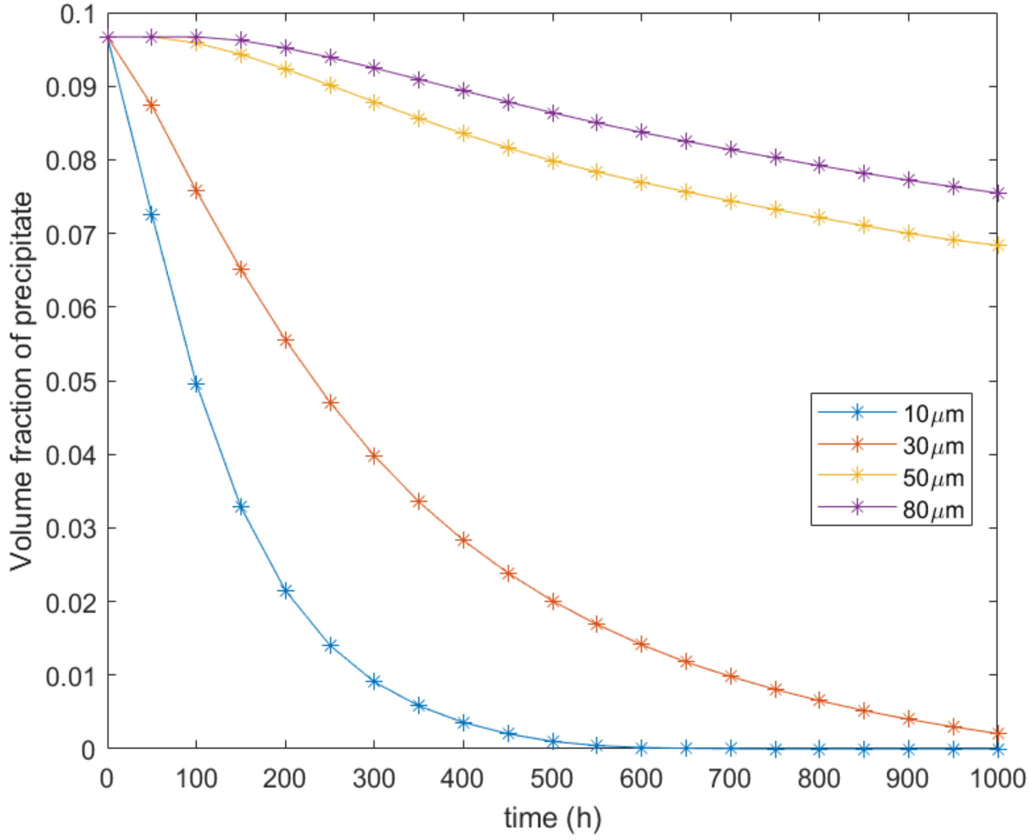


- Antiphase Domain 1
- Antiphase Domain 2
- Antiphase Domain 3
- Antiphase Domain 4

Simulation results

Oxidation time vs volume fraction of precipitates (phase field simulation)

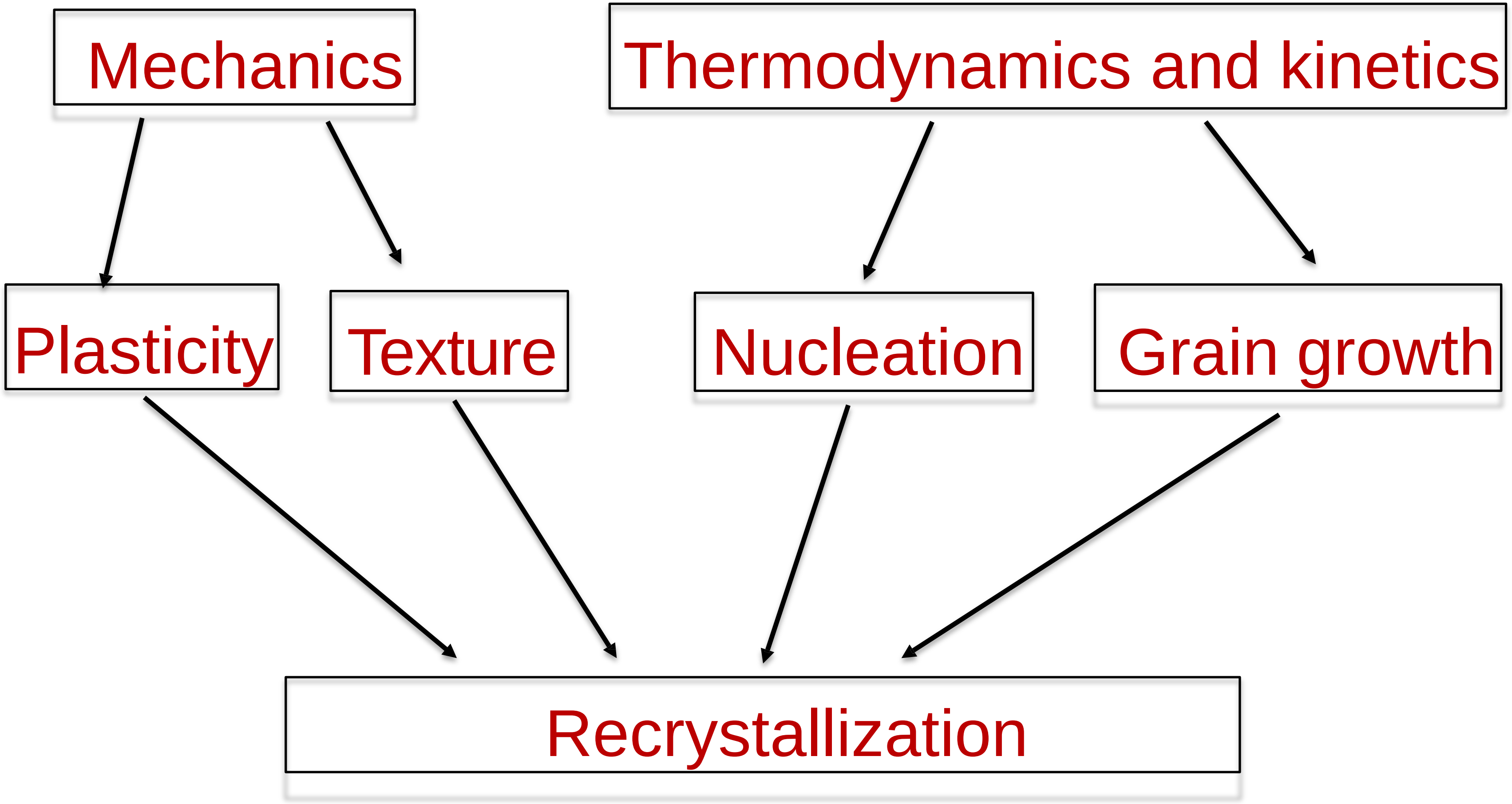




THE OHIO STATE UNIVERSITY

Mesoscale modeling of Creep and Recrystallization

Stephen Niezgoda

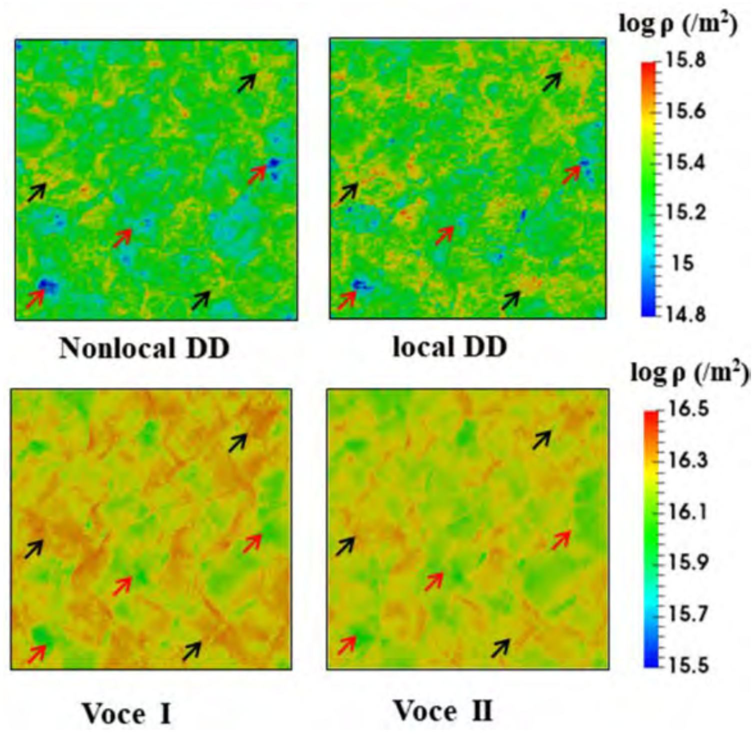

Mechanics
Thermodynamics and kinetics
Plasticity
Texture
Nucleation
Grain growth
Recrystallization

Dislocation density based model

- Phenomenological models are well suited for overall mechanical field predictions and texture evolution, however, it does not perform well in case of localized fields (e.g. dislocation density).

- In the phenomenological model, it is difficult to include different physical mechanisms of strain hardening (e.g. forest dislocation hardening) and recovery (e.g. climb and cross-slip).

- Finally, temperature dependance of the flow curve can not be predicted naturally from phenomenological model.



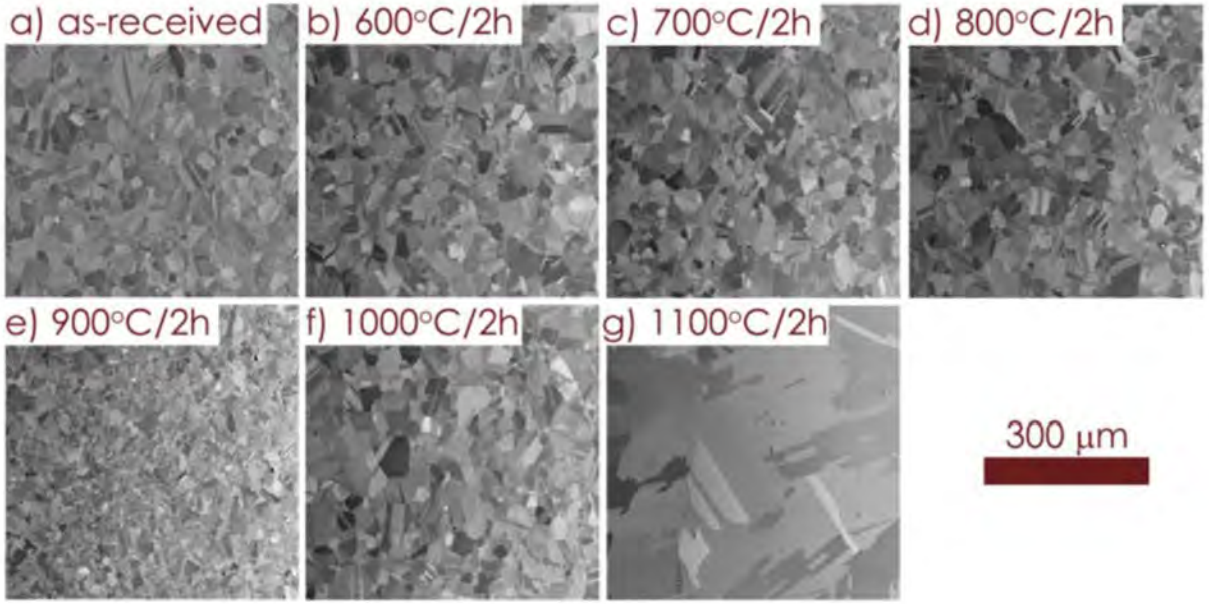
Materials Characterization
Volume 171, January 2021, 110737

Microstructure and mechanical properties of Ni-Fe-Cr-Al wrought alumina forming superalloy heat-treated at 600–1100 °C

Adelajda Polkowska, Sebastian Lech, Piotr Bała, Wojciech Polk

Show more

- 224 is Solution heat treated by Haynes and original data from Haynes suggested that under in-service condition we would expect the material to be in the solid solution state
- Not a problem use Dislocation Creep Model

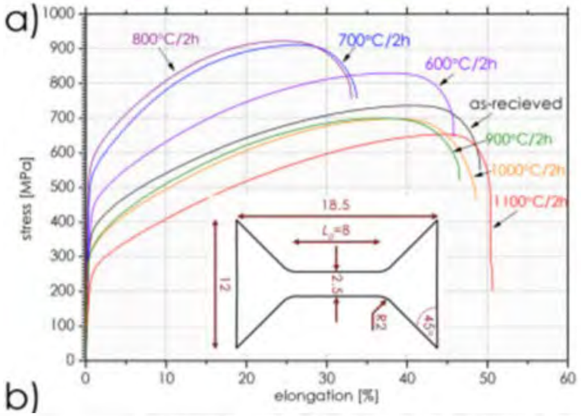


Download : Download high-res image (643KB)   Download : Download full-size image

Fig. 3. SEM images showing a grain structure evolution of Haynes® HR-224® alloy heat-treated at 600–1100 °C/2 h (polished on silica suspensions, non-etched).

- Vinay Deodeshmukh from Haynes was able to share all of the creep data on 224 with the team
  - 25 tests
  - Wide range of tempertatures
  - Wide range of stress
- RT stress strain tests from lit

a)



b)

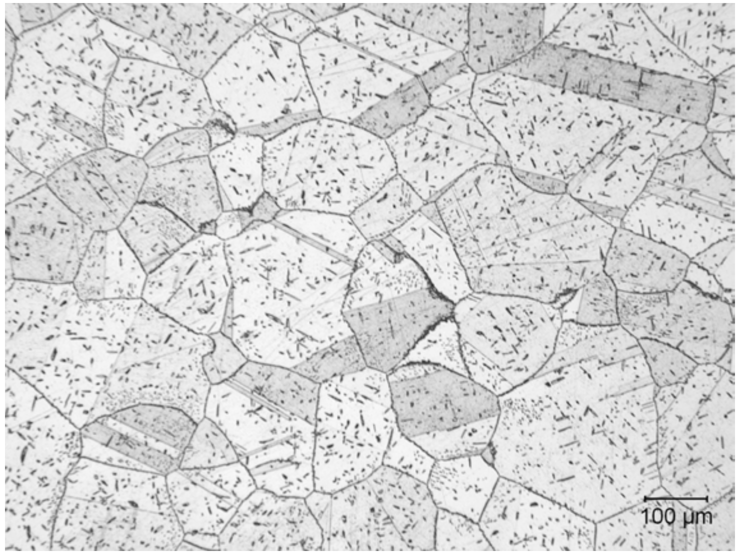
HAYNES® HR-224® Tensile Properties - Bar

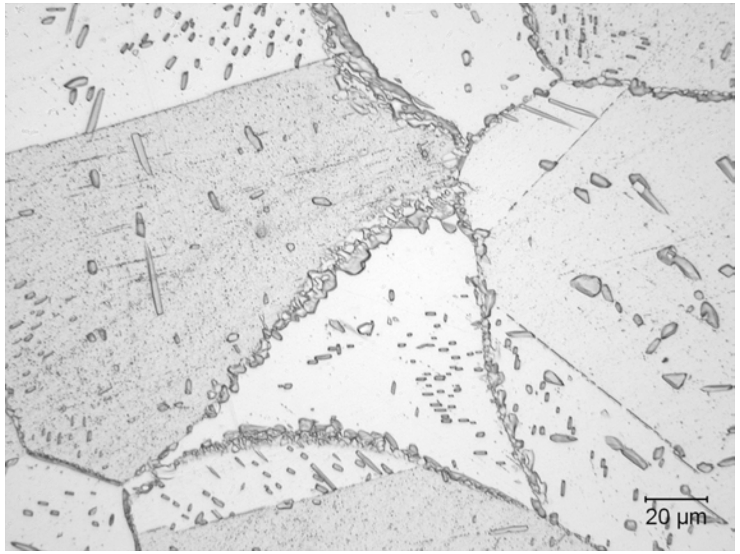
Test Temperature		Yield Strength 0.2% Offset		Ultimate Tensile Strength		Elongation	Reduction of Area
°F	°C	ksi	MPa	ksi	MPa	%	%
RT	RT	45.8	316	106.5	734	48	72
1000	538	43.0	296	93.4	644	53	61
1200	649	54.8	378	74.5	514	13	22
1400	760	57.5	396	69.6	480	11	12
1600	871	12.9	89	19.5	135	106	93
1800	982	6.3	43	9.8	67	101	95

Creep-Rupture Strength

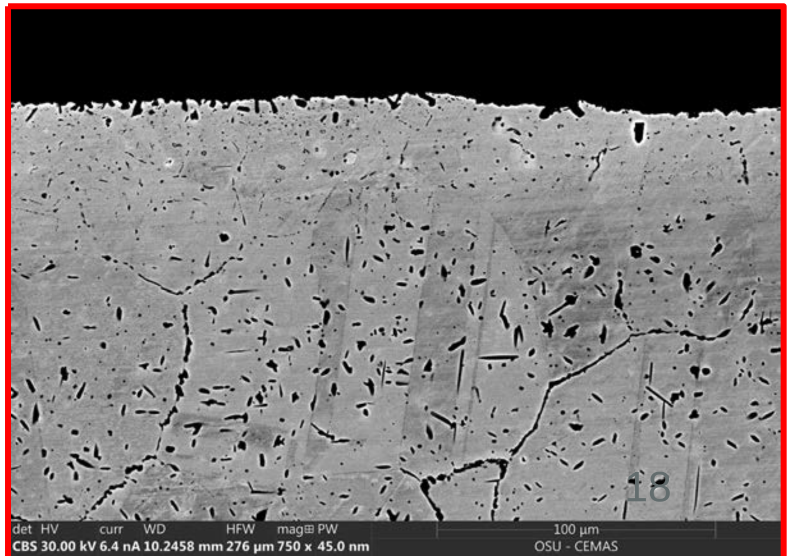
Temperature		Creep	Approximate Initial Stress to Produce Specified Creep in 10 hours		100 hours		1000 hours		10,000 hours	
°F	°C	%	ksi	MPa	ksi	MPa	ksi	MPa	ksi	MPa
1200	649	0.1	36.9	254	26.8	185	19.6	135	14.5	100
		1.0	59.7	412	39.1	270	26.1	180	17.7	122
		R	82.8	571	18.3	126	29.1	201	18.0	124
1300	704	0.1	19.1	132	13.9	96	10.2	70	7.6	52
		1.0	29.0	200	19.2	132	12.9	89	8.9	61
		R	43.4	299	25.5	176	15.5	107	9.7	67
1400	760	0.1	10.3	71	7.5	52	5.6	39	4.2	29
		1.0	14.9	103	9.9	68	6.8	47	4.8	33
		R	23.7	163	14.1	97	8.7	60	5.5	38
1500	816	0.1	5.8	40	4.3	30	3.2	22	2.4	17
		1.0	8.1	56	5.5	38	3.8	26	2.7	19
		R	13.5	93	8.1	56	5.1	35	3.3	23
1600	871	0.1	3.4	23	2.5	17	1.9	13	1.5	10
		1.0	4.6	32	3.2	22	2.3	16	1.7	12
		R	8.0	55	4.9	34	3.2	22	2.1	14
1700	927	0.1	2.1	14	1.6	11	1.2	8	0.9	6
		1.0	2.8	19	2.0	14	1.4	10	1.1	8
		R	5.0	34	3.1	21	2.1	14	1.4	10
1800	982	0.1	1.3	9	1.0	7	0.8	6	0.6	4
		1.0	1.8	12	1.3	9	1.0	7	0.8	6
		R	3.2	22	2.1	14	1.4	10	1.0	7
1900	1038	0.1	0.9	6	0.7	5	0.5	3	0.4	3
		1.0	1.2	8	0.9	6	0.7	5	0.6	4
		R	2.2	15	1.4	10	1.0	7	0.8	6
2000	1093	0.1	0.6	4	0.5	3	0.4	3	0.3	2
		1.0	0.8	6	0.7	5	0.5	3	0.4	3
		R	1.5	10	1.1	8	0.8	6	0.6	4

R = Rupture



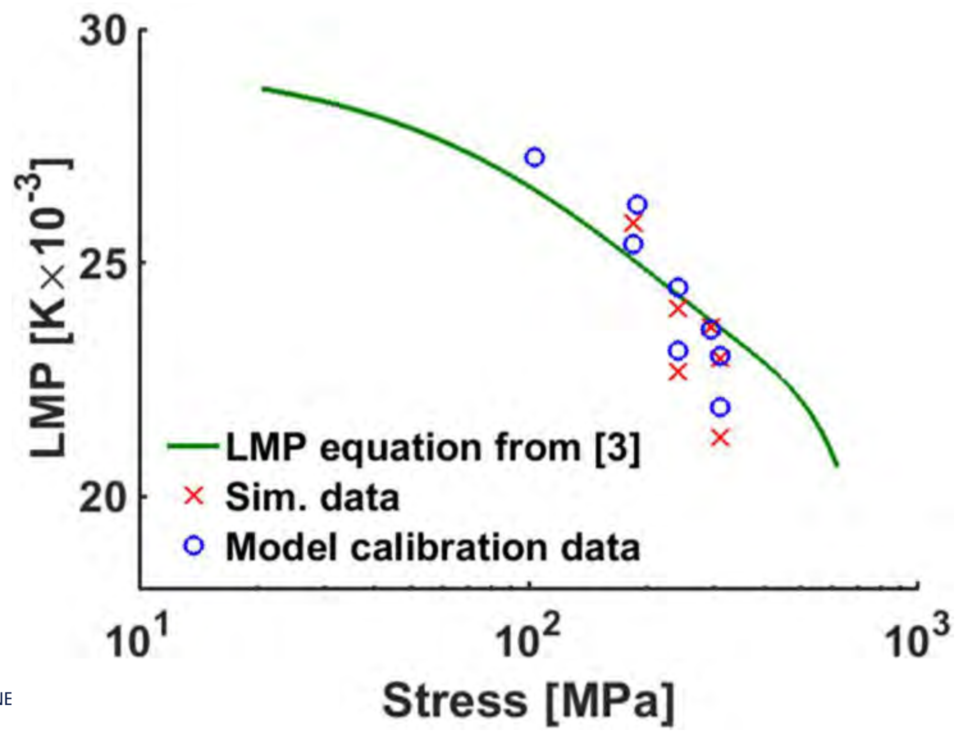


Top Haynes, Bottom Babu (OSU) 224 @ 4K 850C



Creep Modeling 282

- Capture primary and secondary creep
- No explicit tertiary creep effects - damage etc
- Model still able to accurately capture LMP for lifing purposes
  - I.G. Wright, P.J. Maziasz, F.V. Ellis, T.B. Gibbons, D.A. Woodford, Materials issues for turbines fo operation in ultra-supercritical steam (2004).



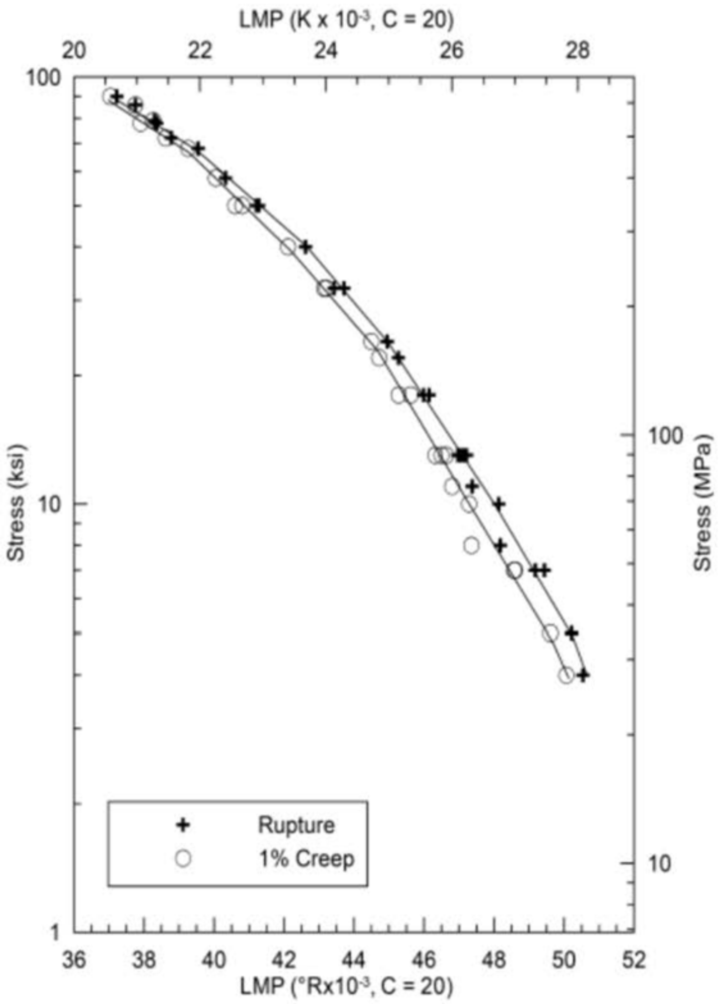


Fig. 14: Larson-Miller plot of rupture and 1% creep for Haynes 282 alloy.

What needs to be done?

- As a team we need to decide what is the best way to handle precipitate growth in 224 and its effect on creep
- Precipitate growth happens at the same time scale as creep meaning that we can’t assume a static microstructure or properties

- Next step simulation of effect of oxide/depletion zone in 282 using heterogenous microstructure creep model

ELSEVIER

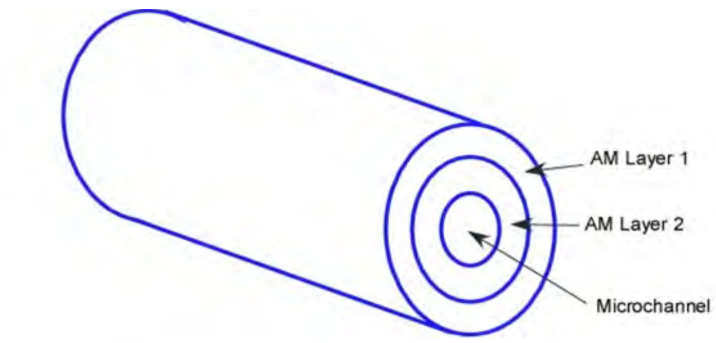
International Journal of Plasticity
Volume 109, October 2018, Pages 153-168

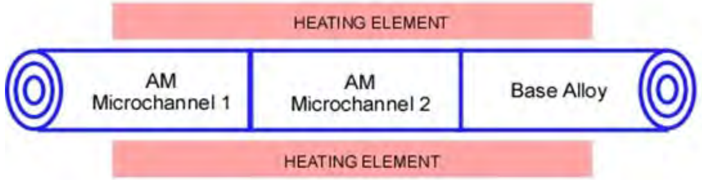
Heterogeneous γ' microstructures in nickel-base superalloys and their influence on tensile and creep performance

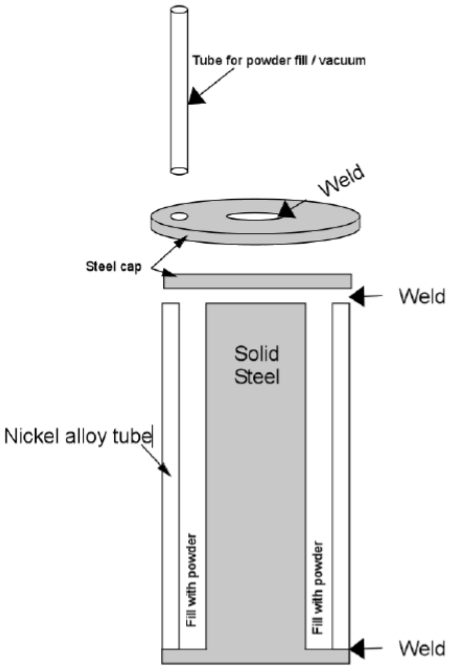
Pengyang Zhao [a], Chen Shen [b], Stephen R. Niezgoda [a, c], Yunzhi Wang [a]

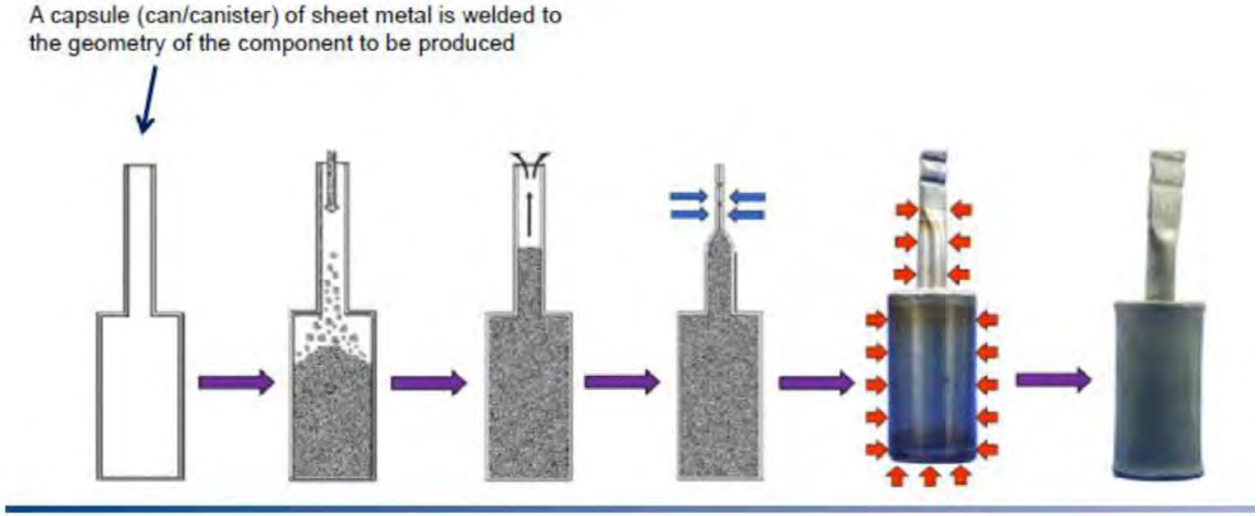
High Temperature Testing

- Wrought flat and tubular specimens exposed to sCO2 for oxidation
  - ~ 1khr , 5khr , 10 khr
- Oxidation CO_2, 725ºC, 850ºC, and 925ºC
- Creep testing tubular pressurized tubular sample
  - Test duration 100 to 2.5 khrs

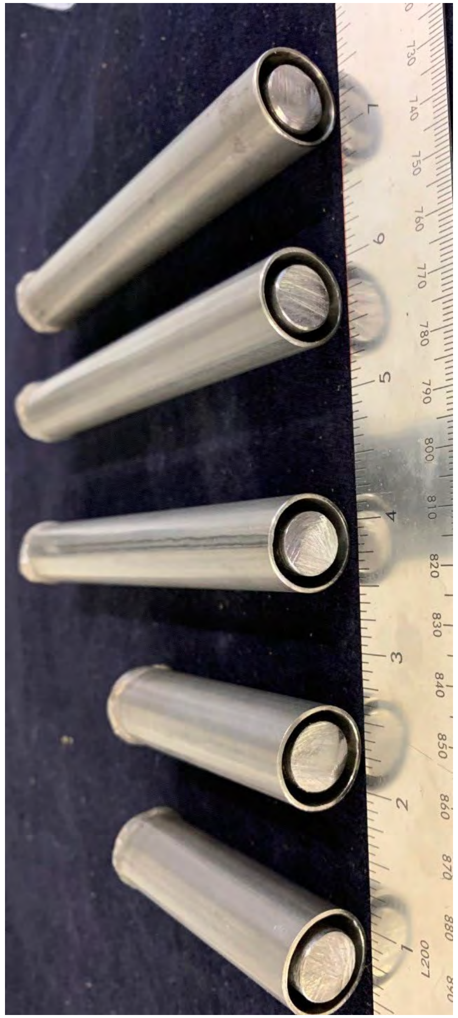








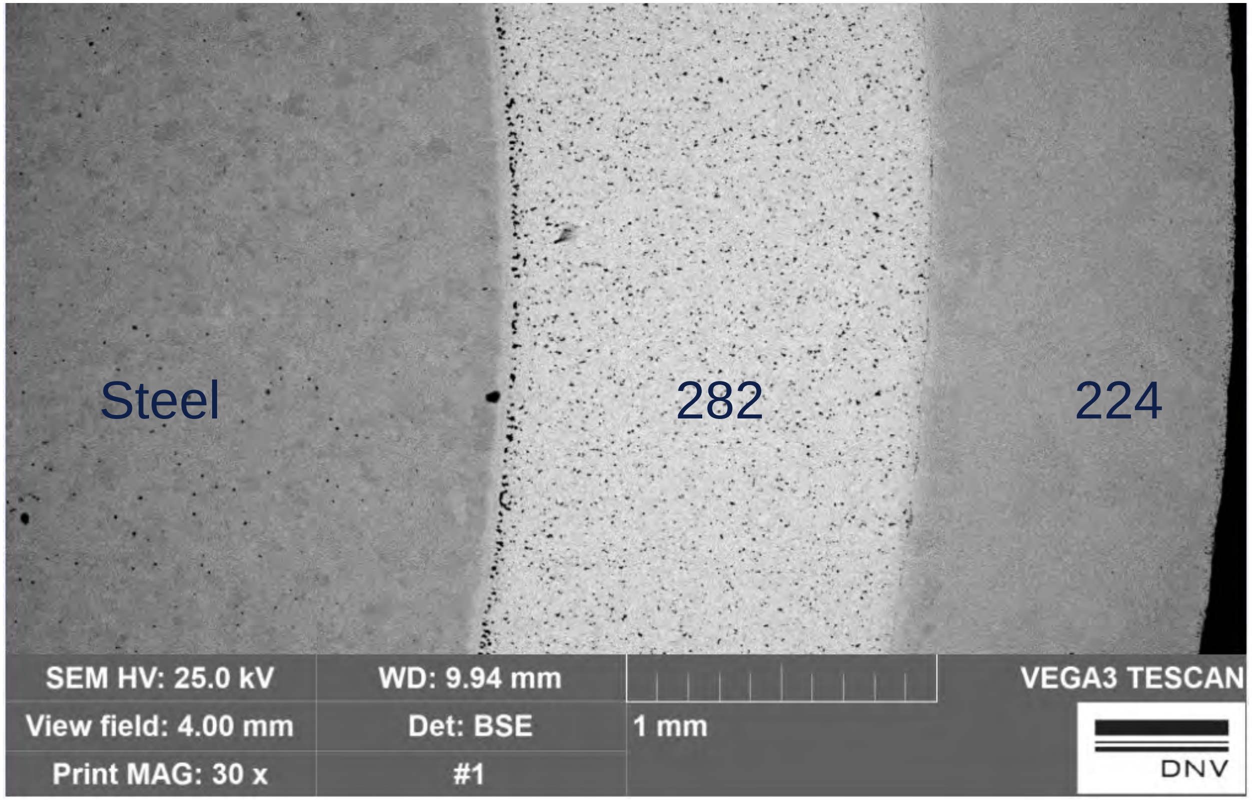
HIP of Graded Alloy Microchannel (actual)



Note collapsed fill tube
= HIP success

HIP of Graded Alloy Microchannel

- Create creep test sample
  - Remove center steel rod
  - Measure wall thickness and diameter at multiple locations
  - Weld Ni-alloy tubing to 282/224 HIP sample
  - Pressurize with CO_2
  - Heat to 850^{o}C, 10-40 hours should show appreciable/measurable creep
  - Expose, measure diameter at end of test
    - Estimate creep strain
  - Characterize
  - Verify / update models



Characterization Efforts: Targeted for Obtaining and Providing the Necessary Parameters for Modeling Efforts

- Optimizing experimental parameters for imaging in the Scanning Electron Microscope (SEM)
  (i) oxide scale thickness (ii) grain boundary oxide intrusion and (iii) imaging γ' precipitate
- Imaging the extent of γ' precipitate dissolution zones for various exposure temperatures and times
- Energy Dispersive Spectroscopy (EDS) to discern the oxidation products and elemental segregation.

Target Alloys:

Haynes Alloy® HR-282 : Characterization is Complete.

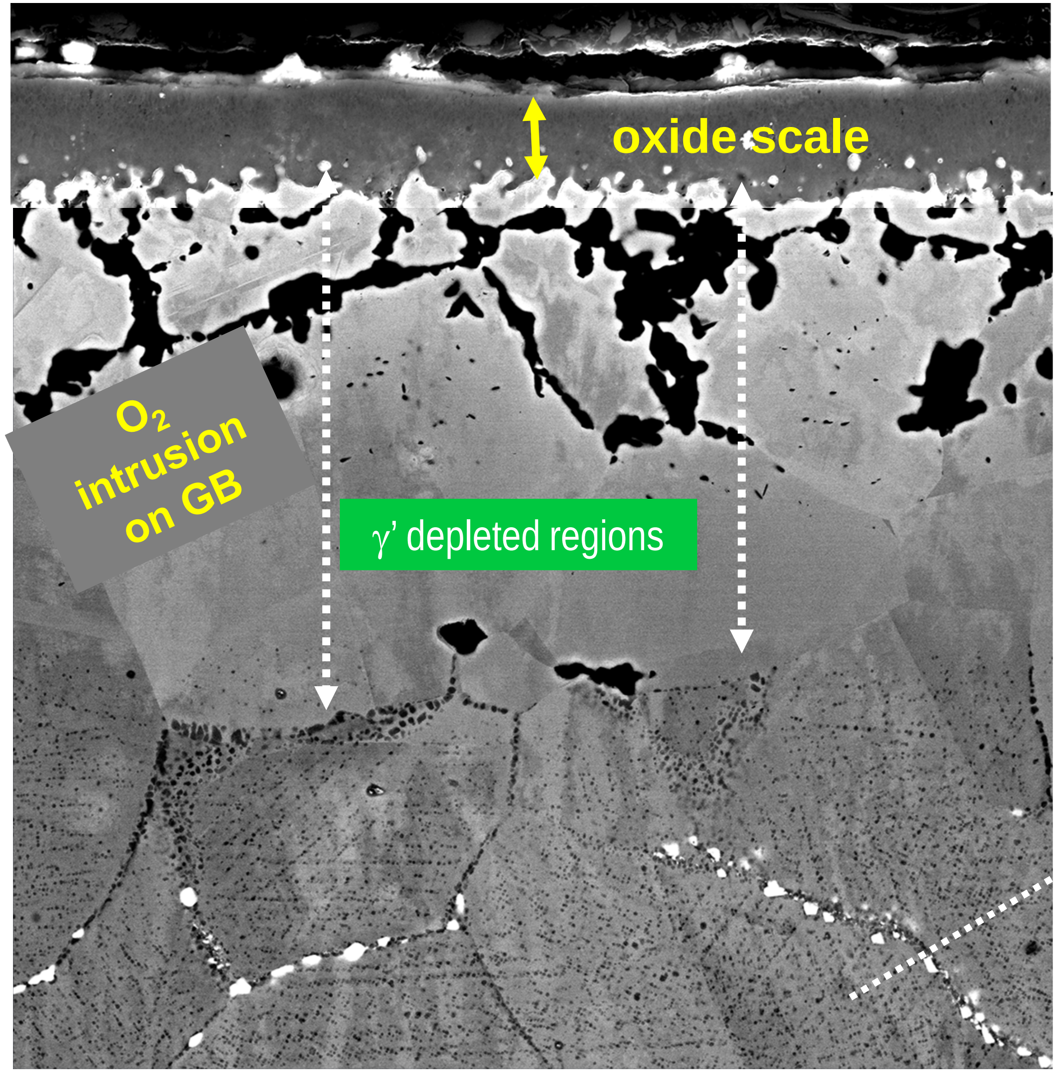
Haynes Alloy® HR-224: Characterization is underway. Expected to be completed in June 2021

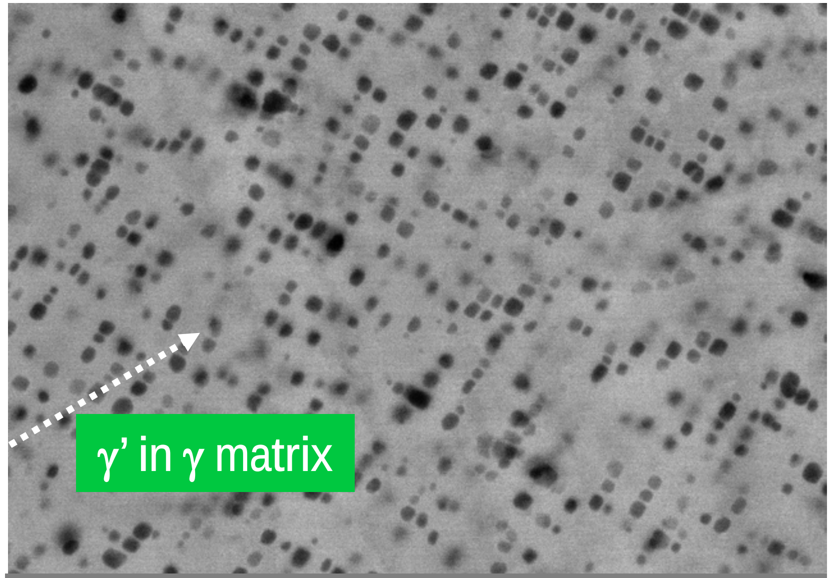
Typical Examples of Characterization Efforts are shown in the following Slides

Typical Example of High-Resolution Imaging in the SEM to characterize the extent of γ’ precipitate dissolution

Alloy 282- Exposed at 925°C,1140 hrs

The depth of γ’ depleted region varies between 55 – 67 μm.



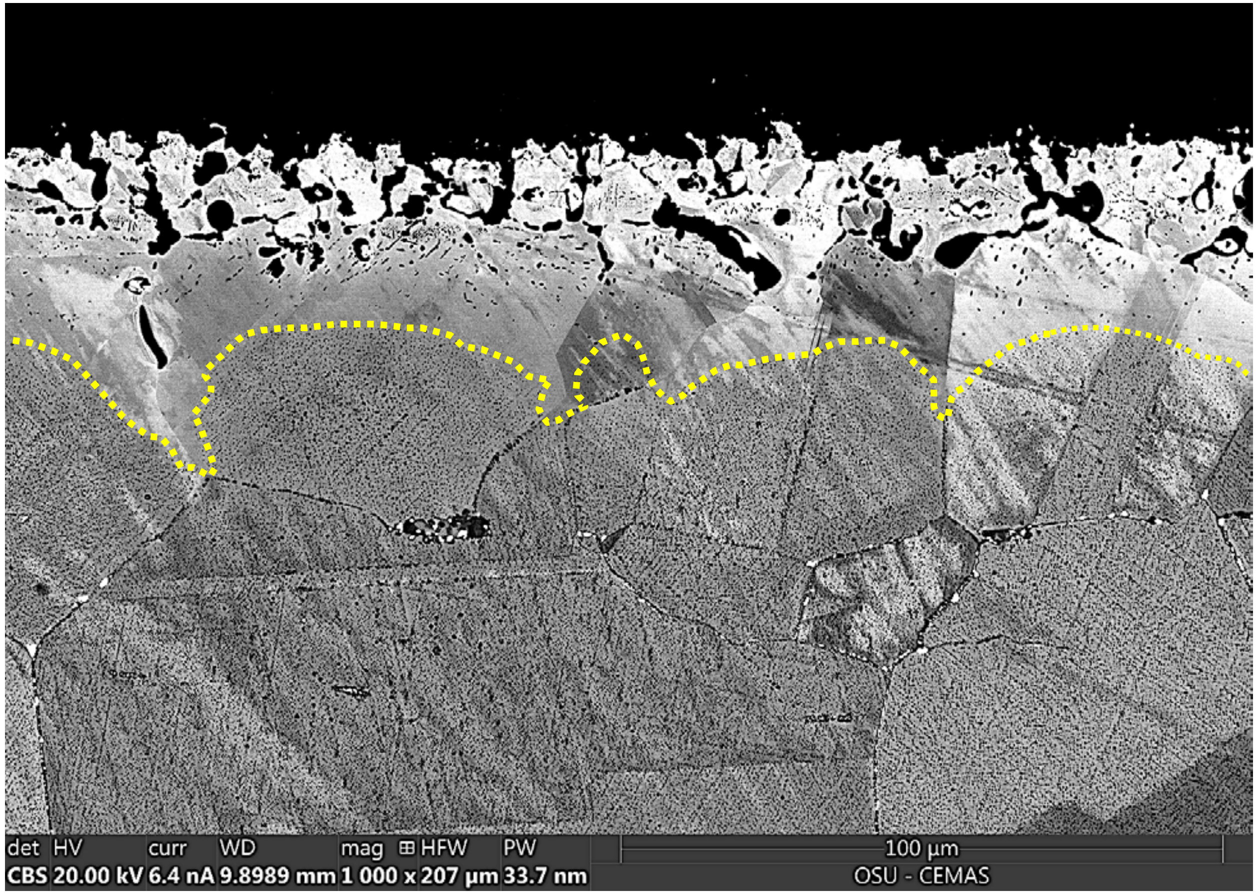


Imaged with Thermo Scientific Apreo FEG-SEM

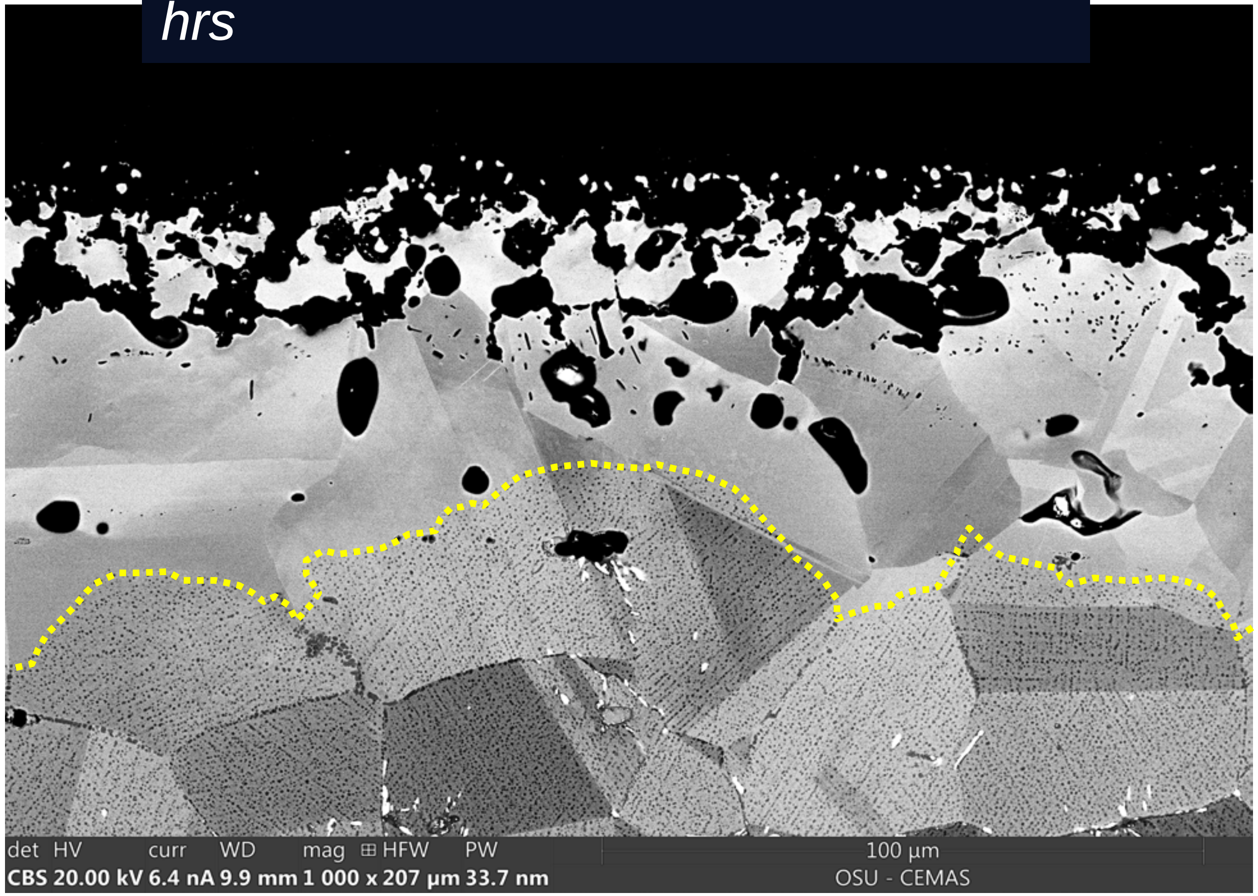
Microstructure: Typically 15-20 μm thick oxide scale is observed on the surface with additional oxygen intrusion of 1-1.5 grain diameter depth along the grain boundary.

Typical Example: Extent of γ' precipitate dissolution: Comparison Short and Long Exposure Times

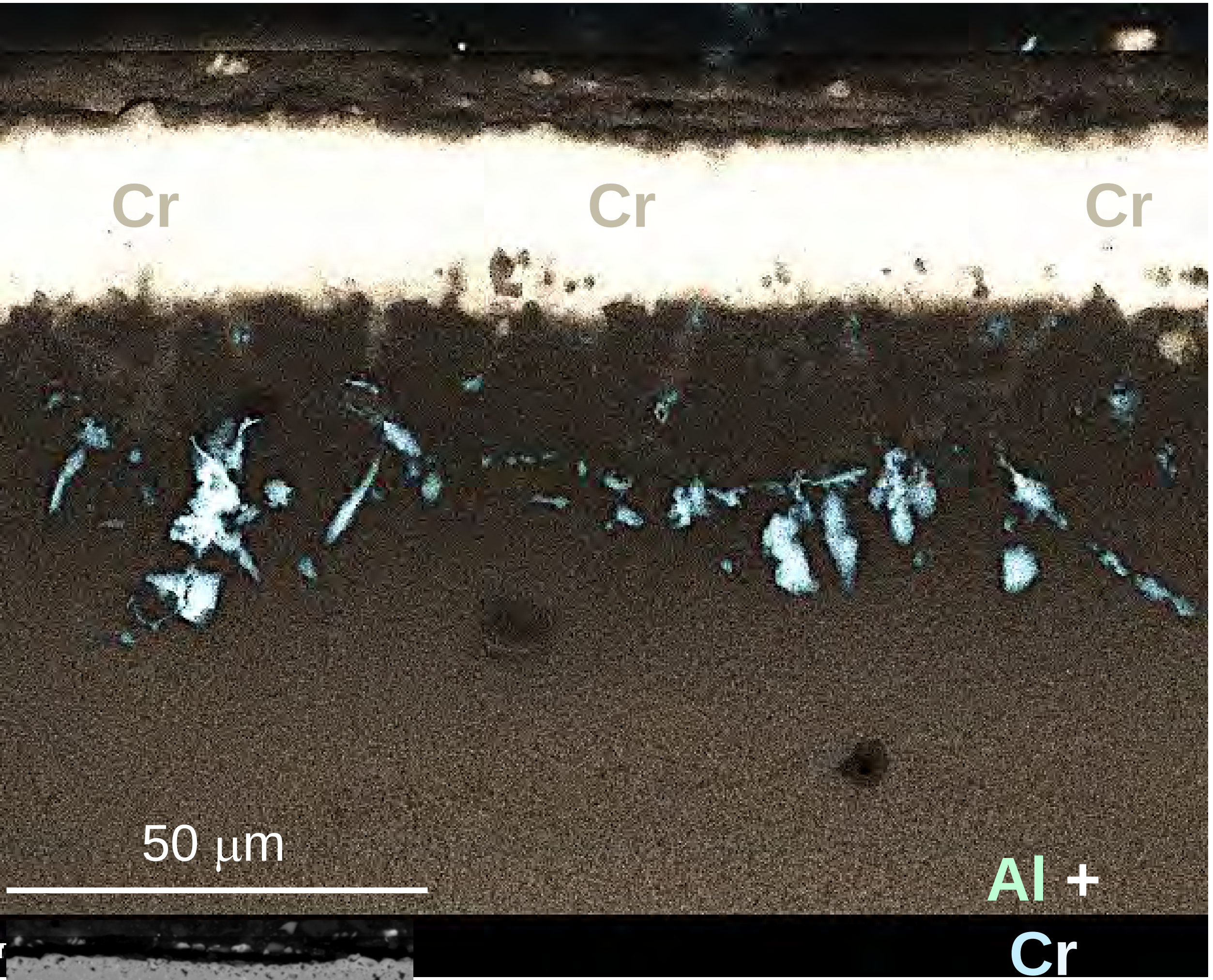
Alloy 282- Exposed at 845°C , 1140 hrs

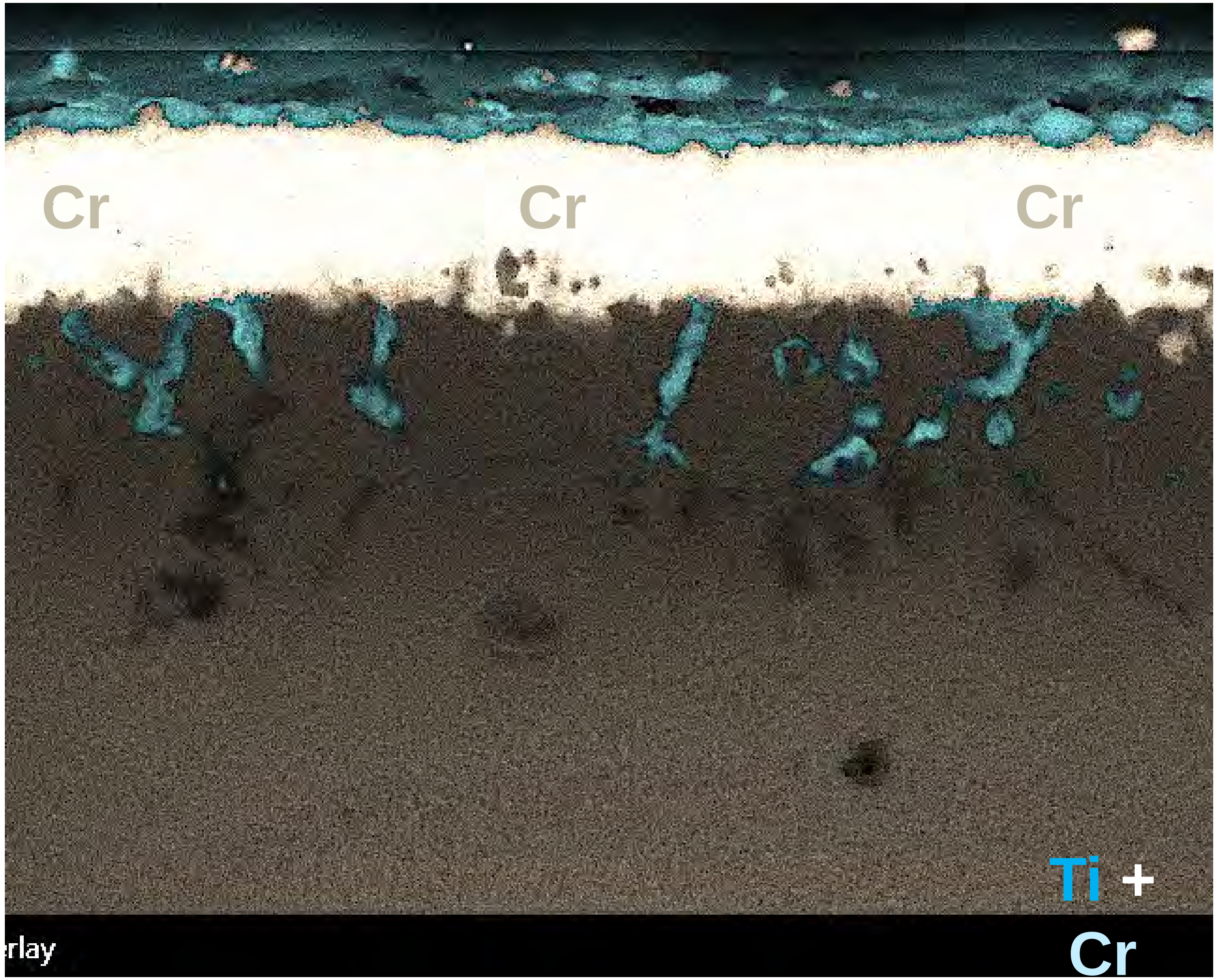


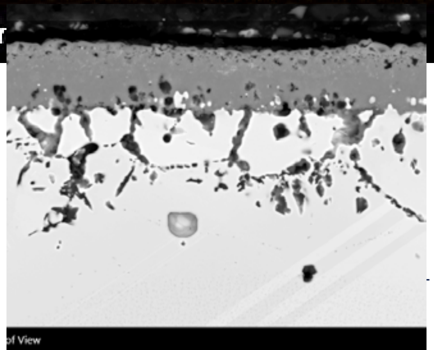
Alloy 282- Exposed at 857°C , 10000 hrs



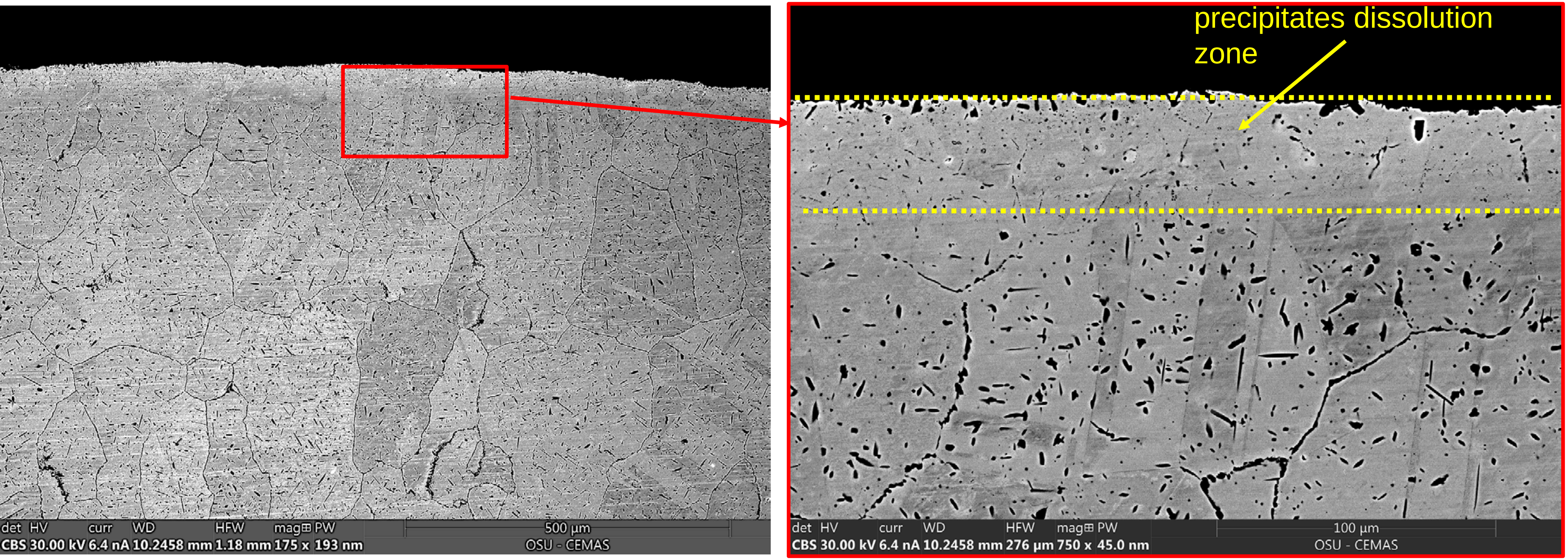
Typical Example: Comparison Between Al and Ti Segregation Behavior Exposed at 855°C



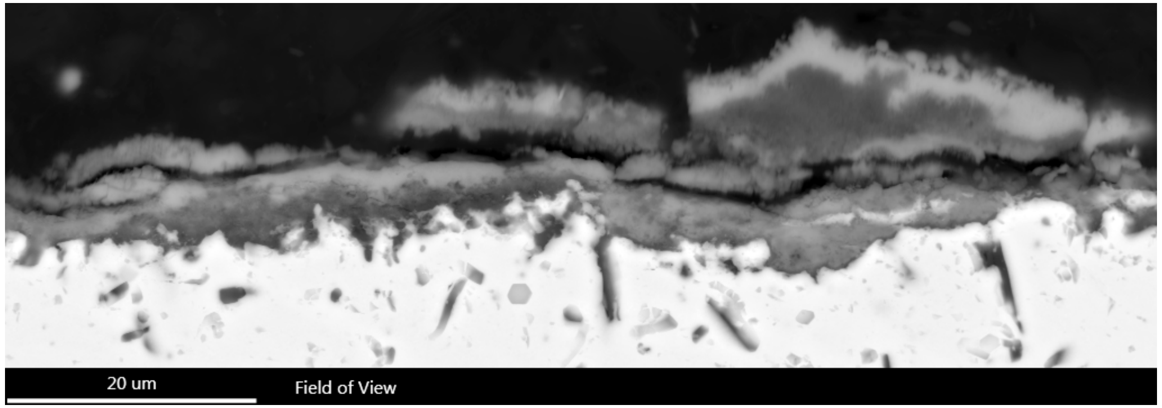


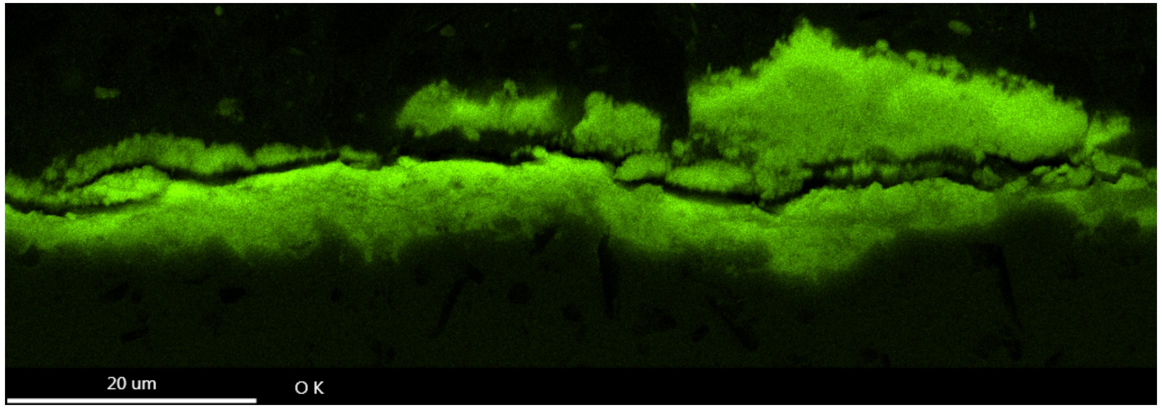


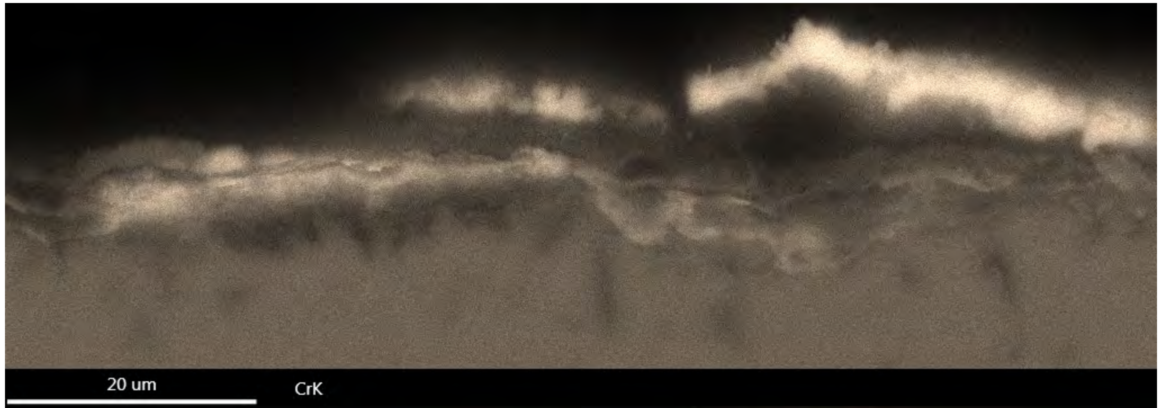
Alloy 224 Exposed at 857°C, 1140 hrs- Typical Example of Precipitates Dissolution

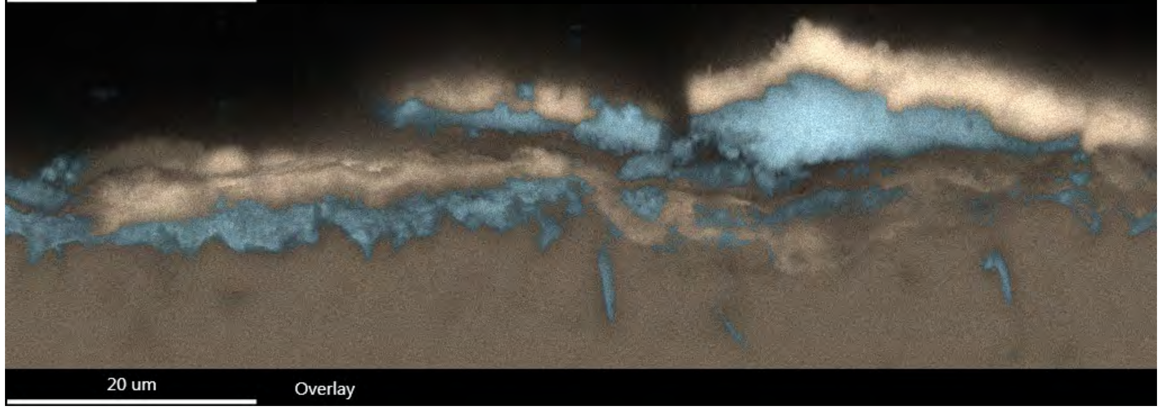


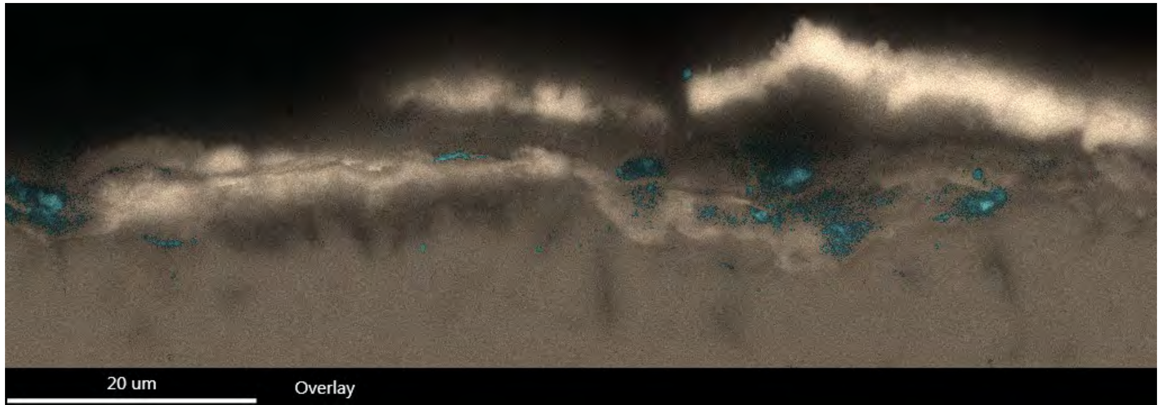
Typical Example: Elemental mapping in Alloy 224 Exposed at 857°C, 1140 hrs











Milestones

Date	Milestone	Status	Updated Target
2-1-2019	MS 1. ICME Integration Plan	Complete	
2-1-2019	MS 2. Sample Fabrication: High Temperature Oxidation Coupons	Complete	
9-1-2019	MS 3. Fabrication of microchannel-like prototype component	Complete	
11-1-2019	MS 4. High temperature oxidation testing	Complete	
6-1-2020	MS 5. High temperature creep testing of prototype component	Delayed	8-1-2021
9-30-2020	MS 6. Demonstration, verification and validation of model	Delayed	9-30-2021

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