An Abrupt Transition to an Intergranular Failure Mode in the Near-Threshold Fatigue Crack Growth Regime in Ni-Based Superalloys

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Motivation

- Most of cyclic fatigue crack growth (FCG) life occurs in the near threshold regime; limited research published in superalloys.
- Recent discovery of sudden transition to an intergranular failure mode in the near-threshold cyclic FCG regime – need to understand the underlying science.
- Surprising, since intergranular failure is typically associated with increase in FCG and not slow FCG rates of the threshold regime.
- What causes the transition in the failure mode? Detailed look at crack tip behavior.
- What are the crack driving forces for intergranular failure?
- Mixed mode failure typically observed in the Paris FCG regime – what does this actually mean and how does it effect FCG propagation rates?

Experimental

Material: Low Solvus High Refractory (LSHR) and ME3 P/M nickel-base disk alloys

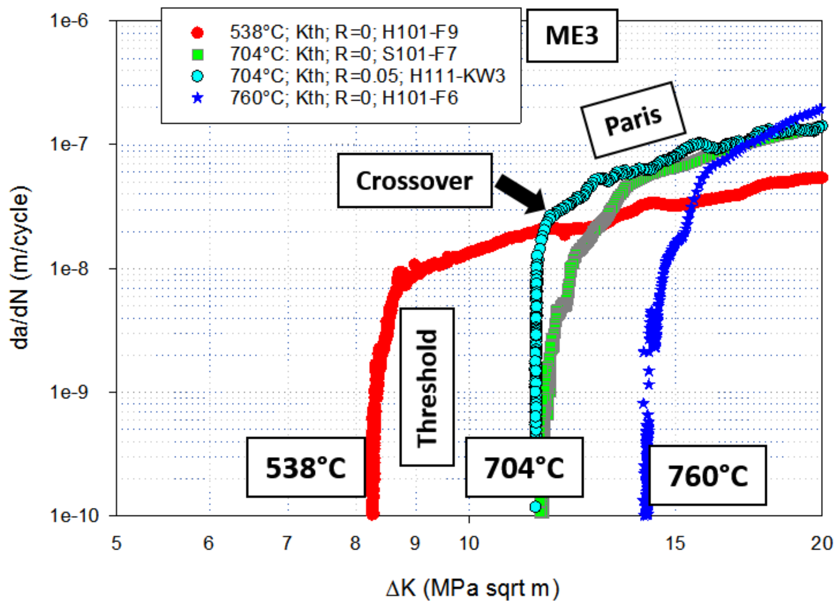
Alloy	Ni	Cr	Co	Mo	W	Nb	Ta	Al	Ti	Zr	B	C
ME3	Bal.	13	21	3.7	2.1	0.8	2.4	3.4	3.8	0.05	0.02	0.05
LSHR	Bal.	12.5	20.4	2.7	4.3	1.5	1.5	3.5	3.5	0.05	0.03	0.045

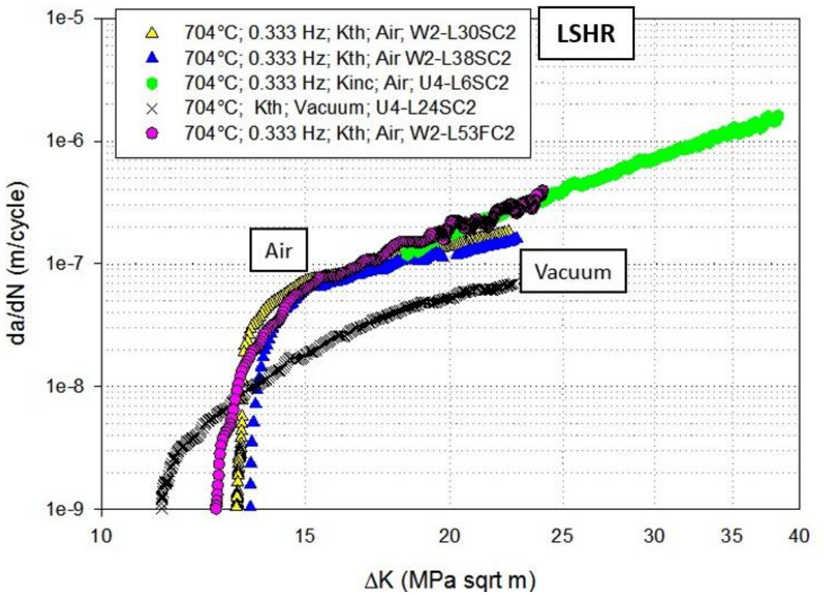
- Supersolvus Grain Size: LSHR – ASTM 8; ME3 – ASTM 7

FCG Testing:

- Mostly at 704°C; selected specimens at 538°C and 760°C; R ratio of 0.05; few at 0.5
- Test frequency range: from 0.333 Hz to 30 Hz (majority at 0.333 Hz)
- Environment: air with additional selected vacuum tests
- Threshold testing: K-decreasing (load shed); threshold = $1x10^{-10}$ m/cyc
- Near threshold testing: constant load tests started at near-threshold ΔK values
- Paris regime: Constant ΔK=25 MPa√m at 0.333 Hz, 2 Hz and 10 Hz
- Specimen geometry: Surface flaw (K_B bar)

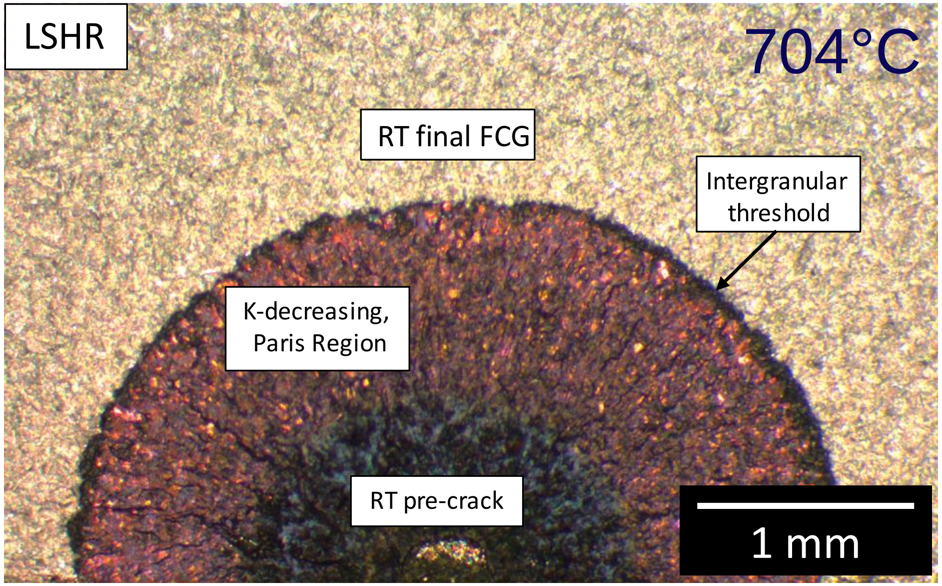
Near Threshold Crossover Effect

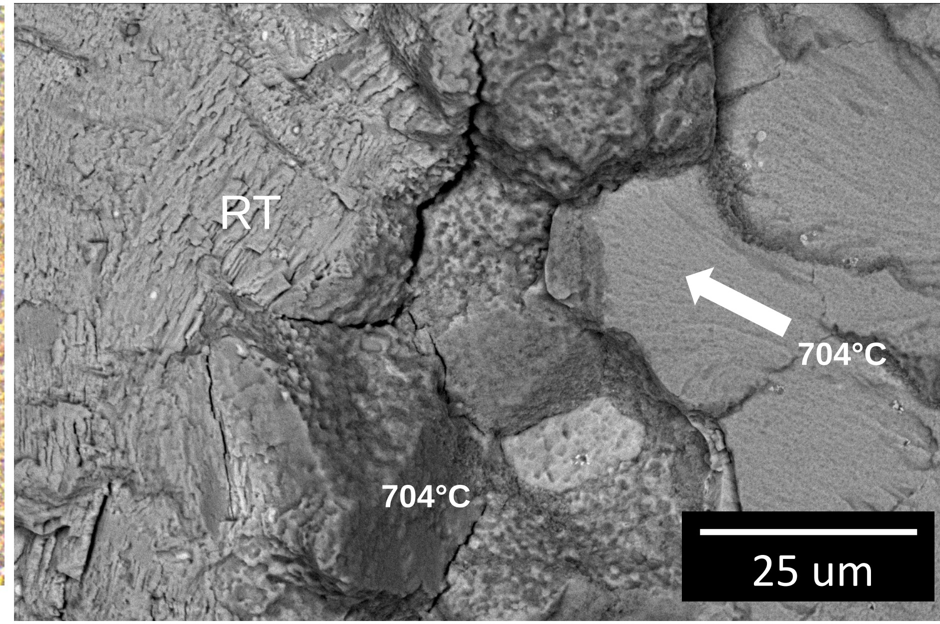


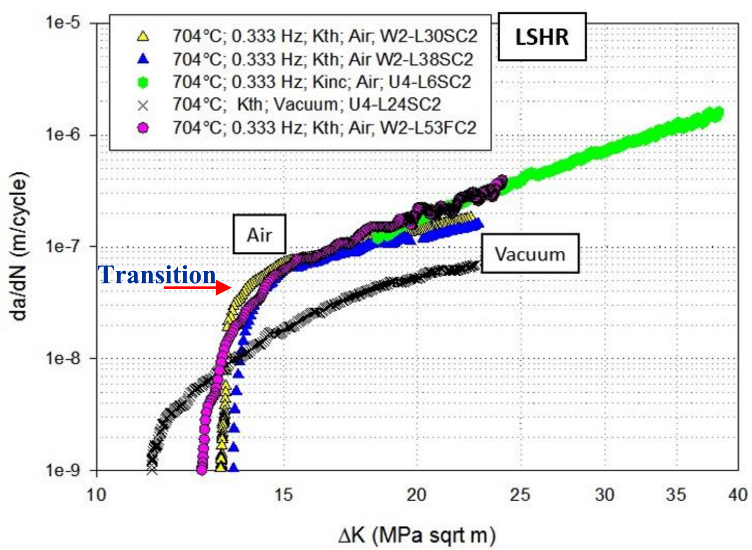


- Higher FCG rates in the Paris regime at higher temperatures, air vs vacuum.
- Higher resistance to FCG in the near-threshold region.
- Limited literature on the subject, crossover attributed to increase in oxide induced crack closure

Abrupt Change in Failure Mode at the onset of Near-Threshold Regime; LSHR



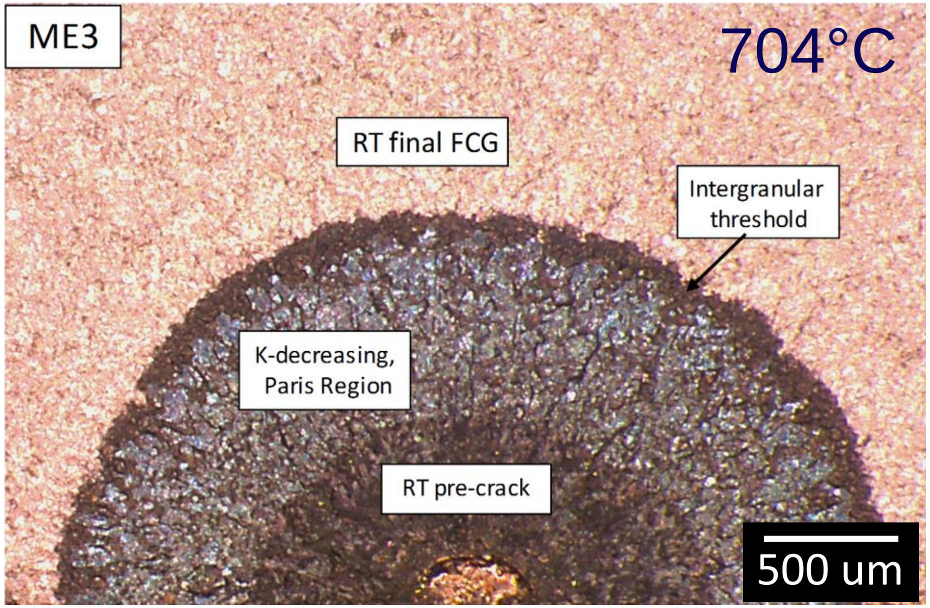


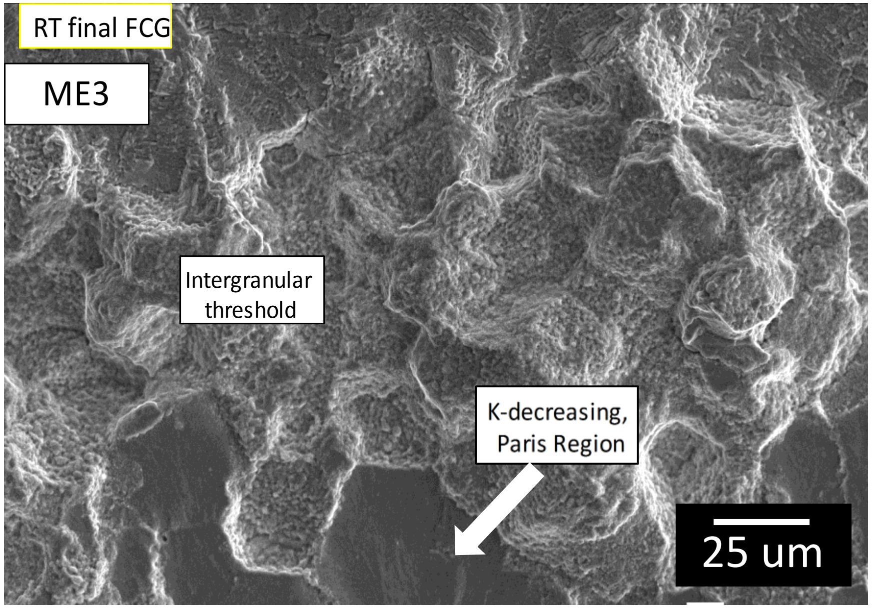


- Intergranular zone develops suddenly at the transition from the Paris regime to the near threshold regime.
- Intergranular zone is 2-5 grains in depth and covers the entire crack front perimeter.
- Unexpected change to intergranular failure mode, previously not reported.

ME3 - Near Threshold FCG Abrupt Failure Mode Transition

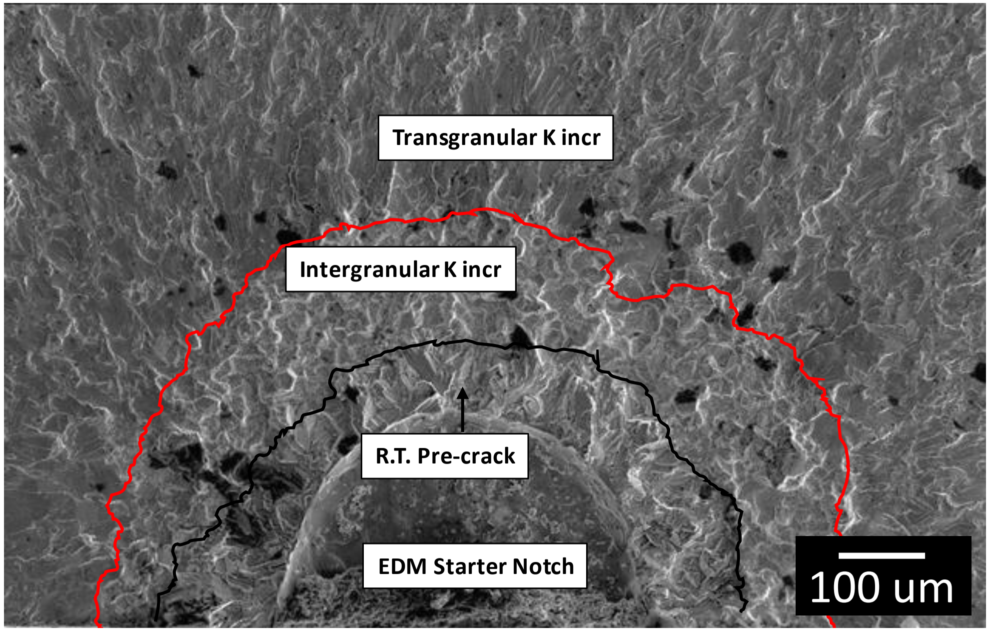
704°C; 0.333 Hz; K-decreasing threshold test

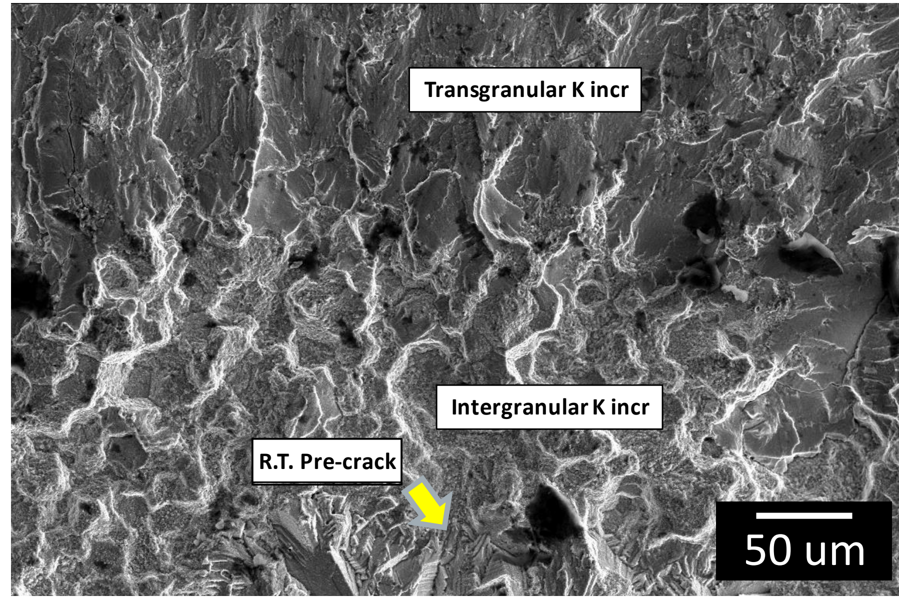


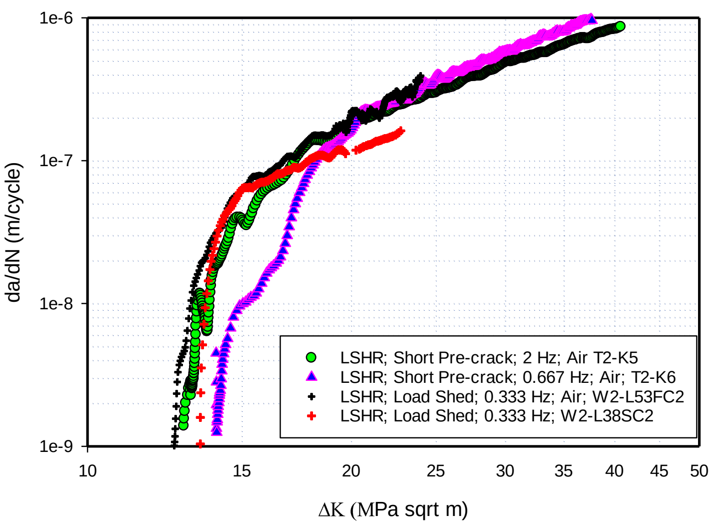


- Abrupt failure mode transition also occurs during threshold tests in ME3

LSHR K-Increasing FCG Tests: Abrupt Failure Mode Transition

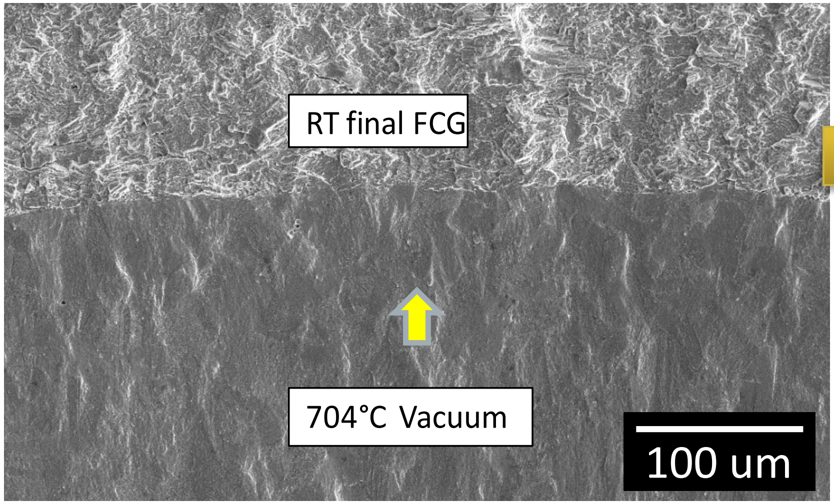




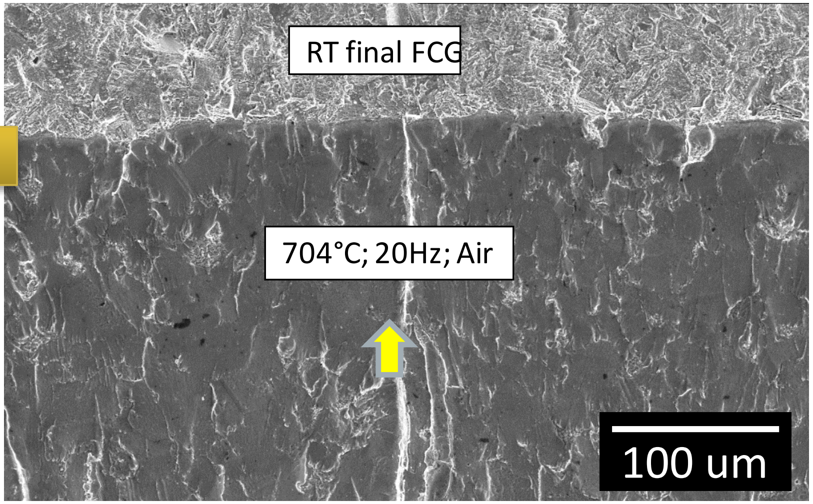


- Abrupt failure mode transition also occurs during constant load, K increasing testing.
- The width of the intergranular zone somewhat greater than in K-decreasing tests
- FCG results for K increasing tests are in general agreement with K decreasing tests

Existence of Failure Mode Transition Depends on Test Conditions



Near Threshold

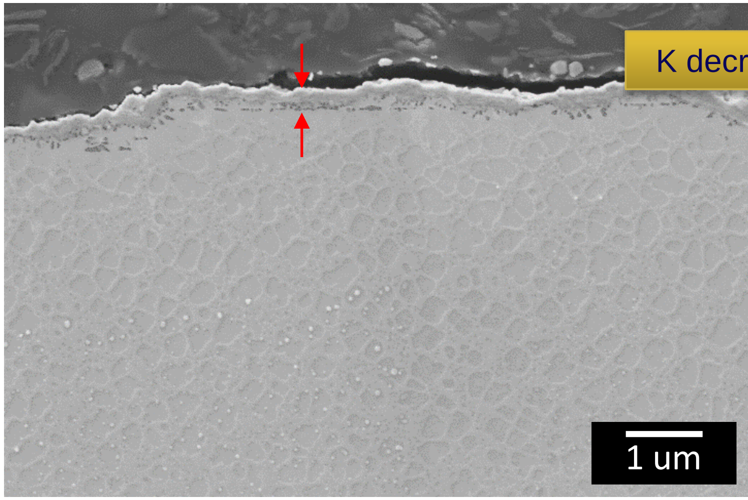


- No failure mode transition for tests performed in vacuum, at high frequencies or lower temperatures.
- Failure mode transition phenomenon dependent on environmental interactions.
- Kinetic requirements need to be satisfied for appropriate reactions to take place to cause failure mode transition.

Variation in Oxide Thickness for the Two Failure Modes

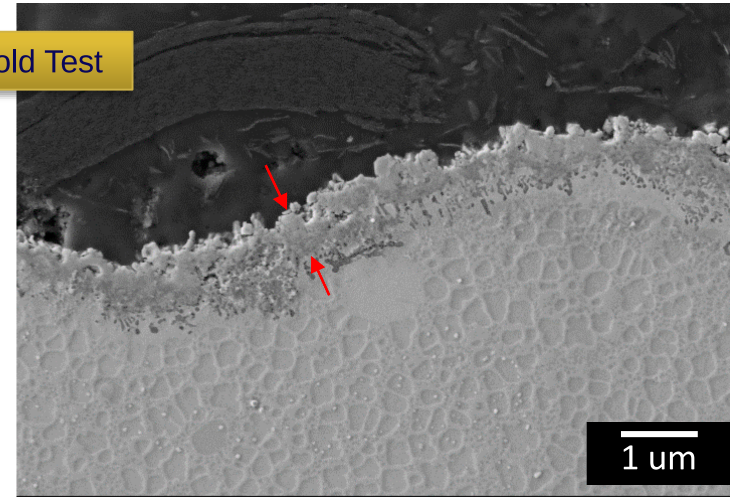
K decreasing Threshold Test

Paris Regime, Transgranular



Average: 260 nm
95% Confidence interval: 34 nm

Near Threshold, Intergranular

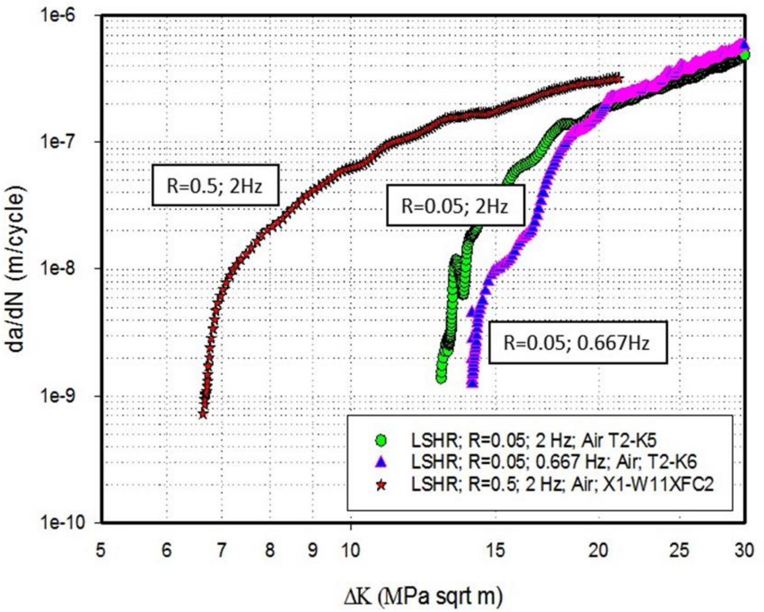


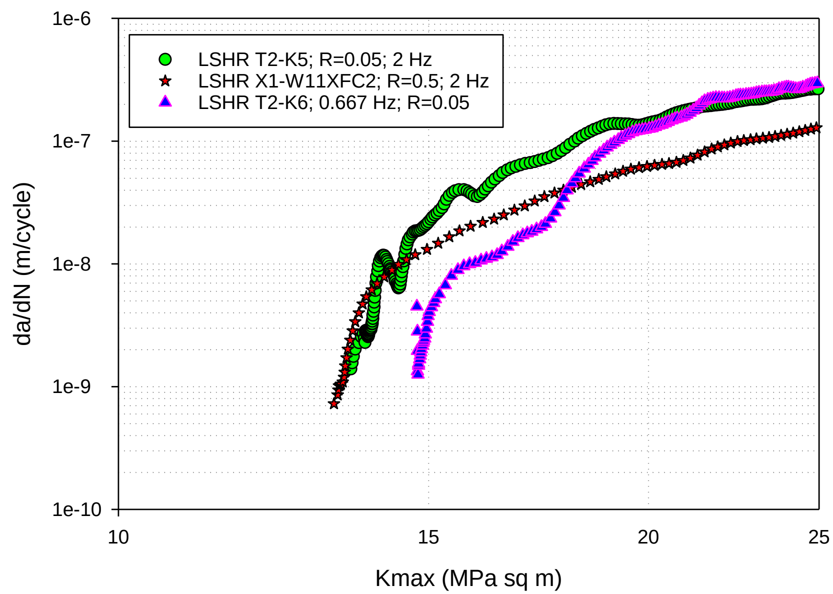
Average: 636 nm
95% Confidence interval: 124 nm

- Oxide formation accelerated by presence of grain boundaries.
- Intergranular oxide thickness > 2X transgranular layer.
- Change in oxide thickness increases crack closure, partly responsible for the crossover effect (does not explain why failure mode transition takes place).

What Crack Driving Force is Most Suitable for Correlating Intergranular Near Threshold Failure Mode?

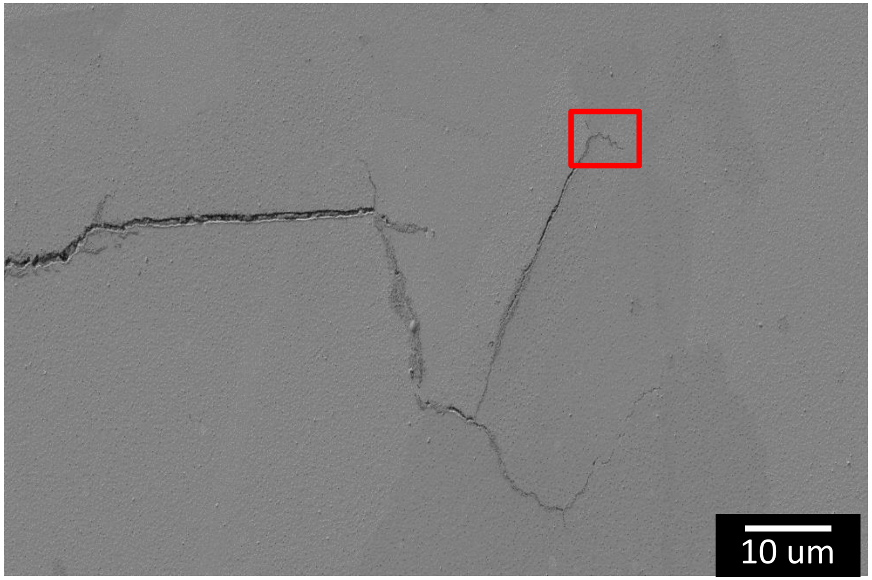
- Transgranular FCG process governed by cyclic plasticity → ΔK parameter
- Intergranular crack growth during hold time tests → K_{max} parameter
- Cyclic crack advancement through oxidized grain boundaries → ?

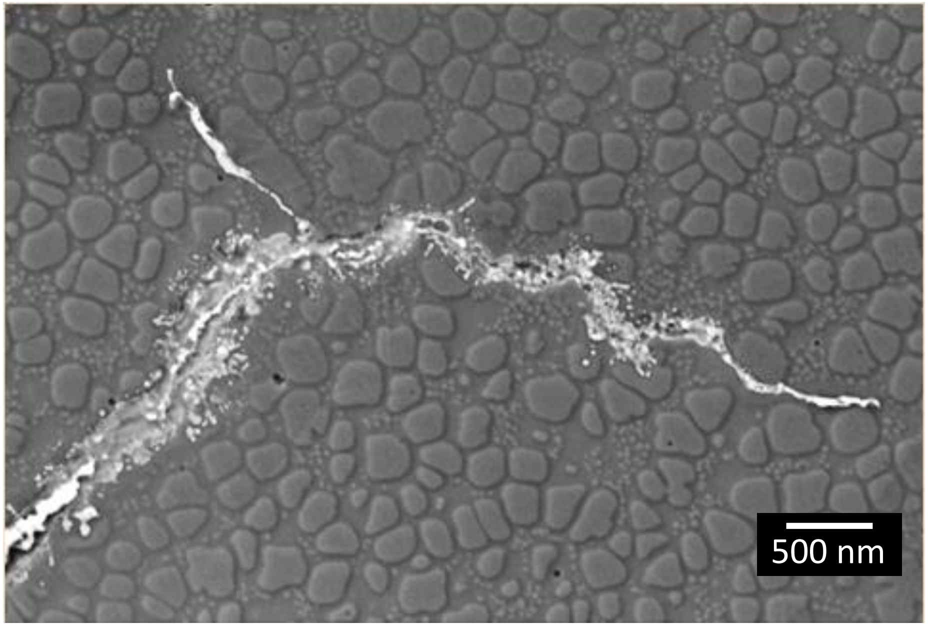


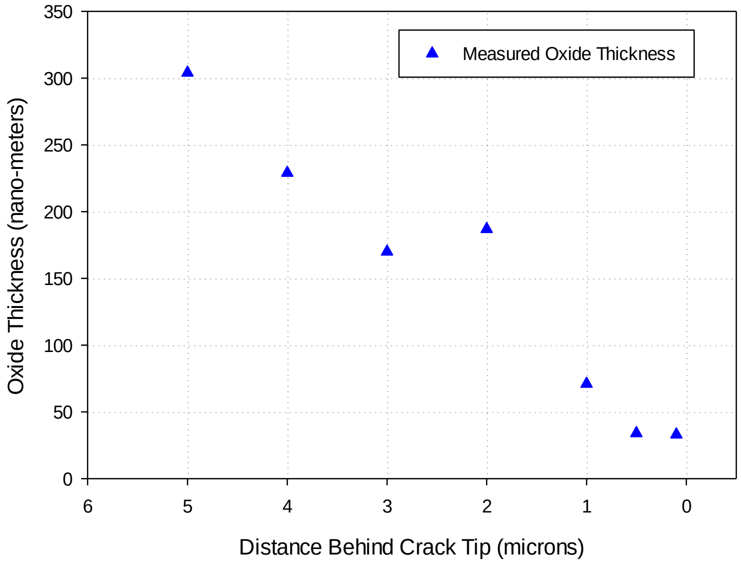


- Difficult to visualize cyclic plasticity governing FCG of oxidized grain boundaries.
- Crack growth in embrittled materials is governed by Kmax.
- Kmax parameter does a better job in correlating near threshold regime.
- Crack advancement through oxide micro-cracking process.

Interrupted K-decreasing Threshold Test

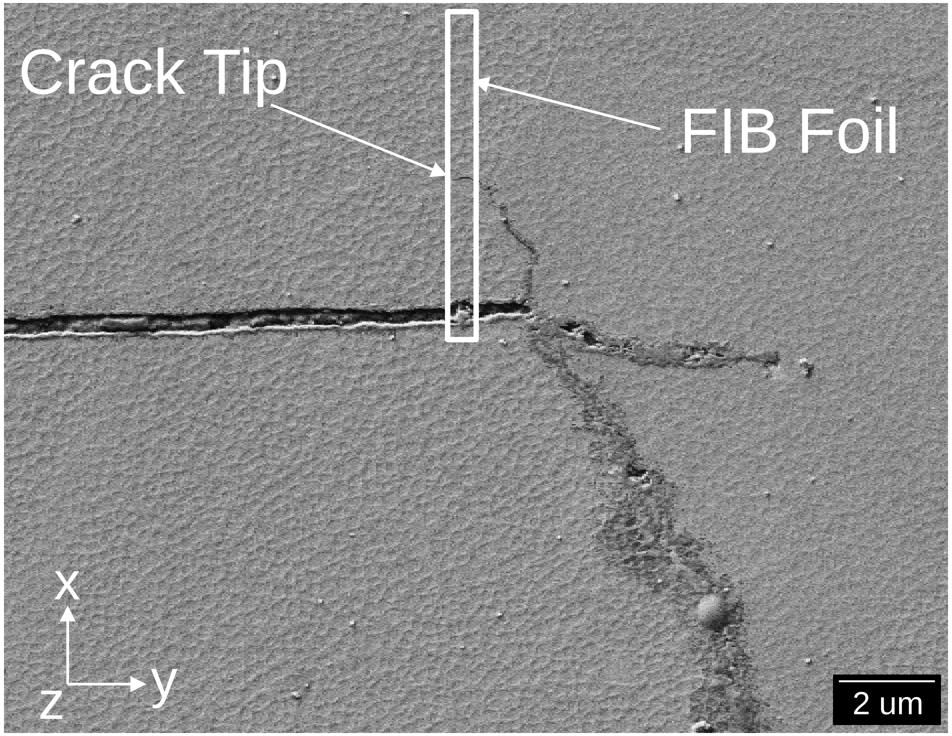


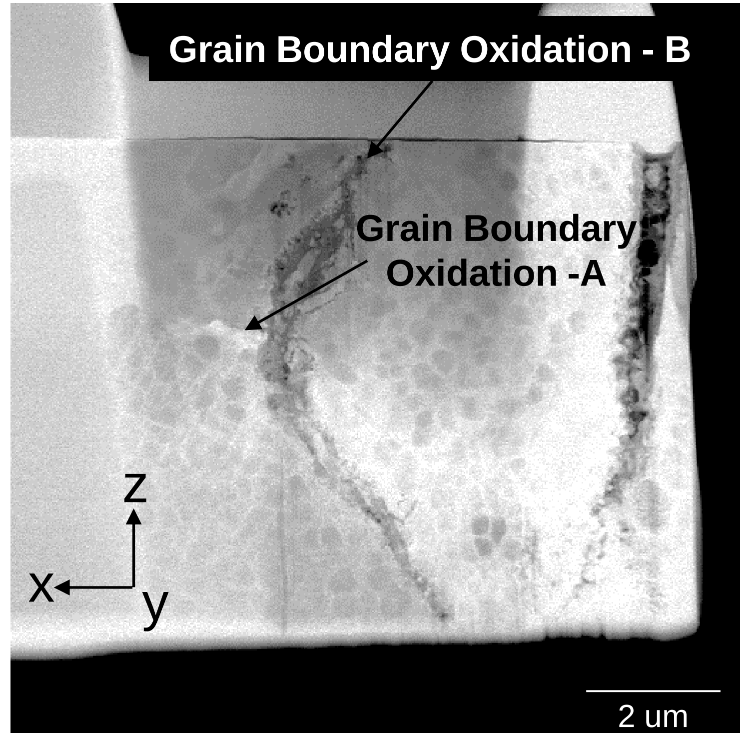




- Heating system automatically turned off.
- Transition to intergranular failure within 30 µm of crack tip.
- Intergranular crack appears to avoids γ′ precipitates.
- Oxide layer continues to widen by growth of “oxide fingers” behind crack tip.
- “Oxide fingers” grow into γ′.

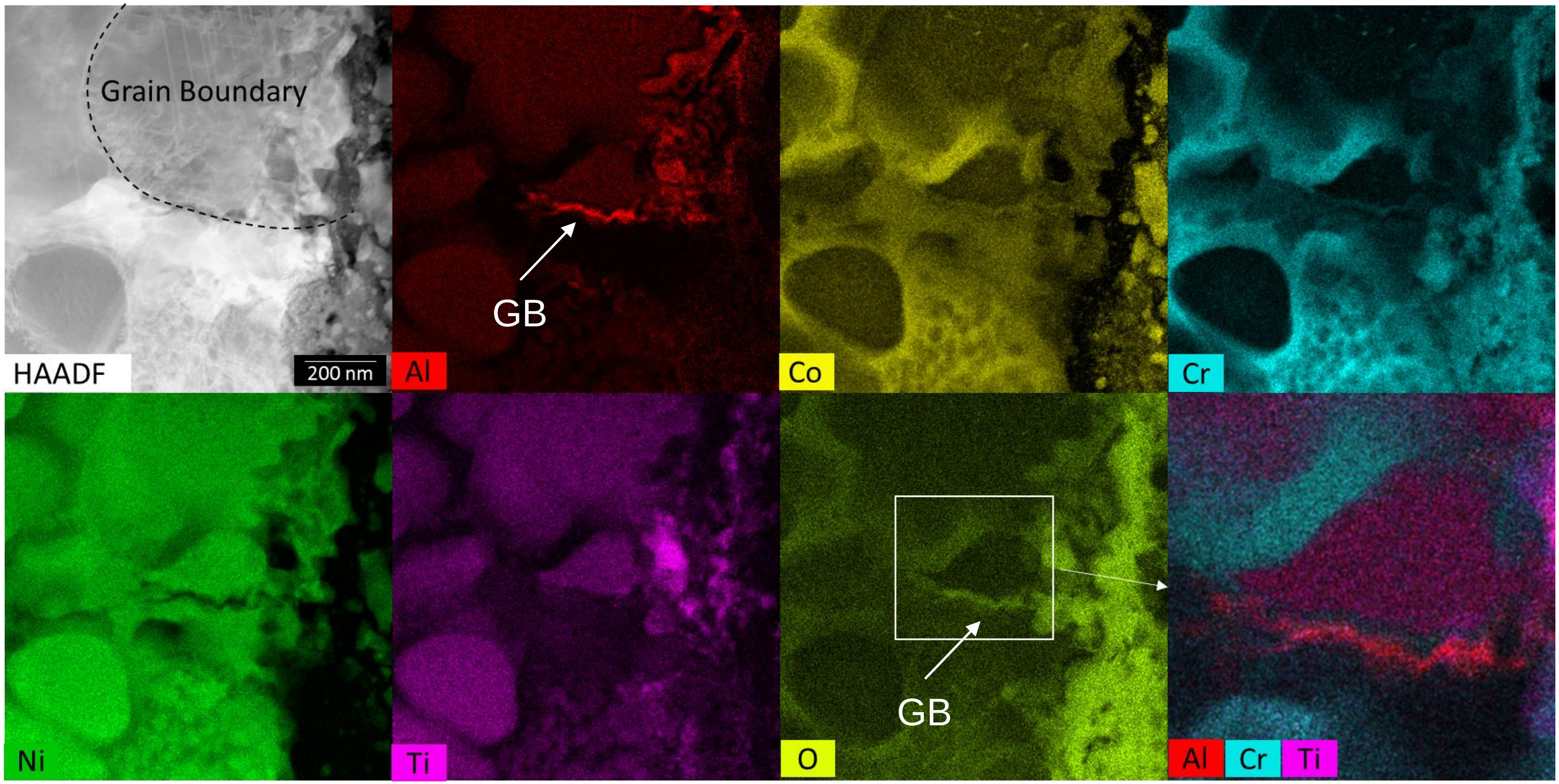
Scanning Transmission Electron Microscopy (STEM) Analysis of Crack Oxidation





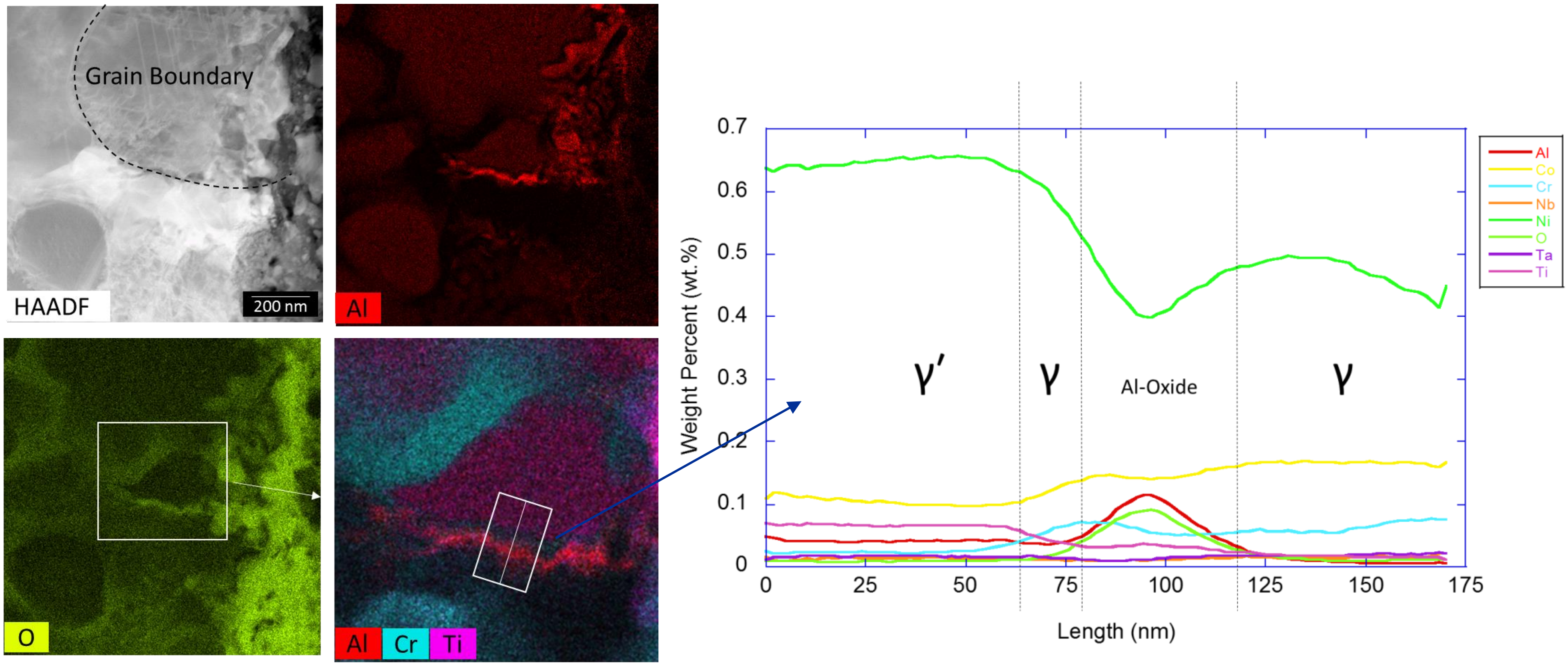
STEM FIB foil was extracted at a crack tip in order to better understand what causes the transition in the failure mode.

STEM EDS of Crack Tip Oxidation – Region A



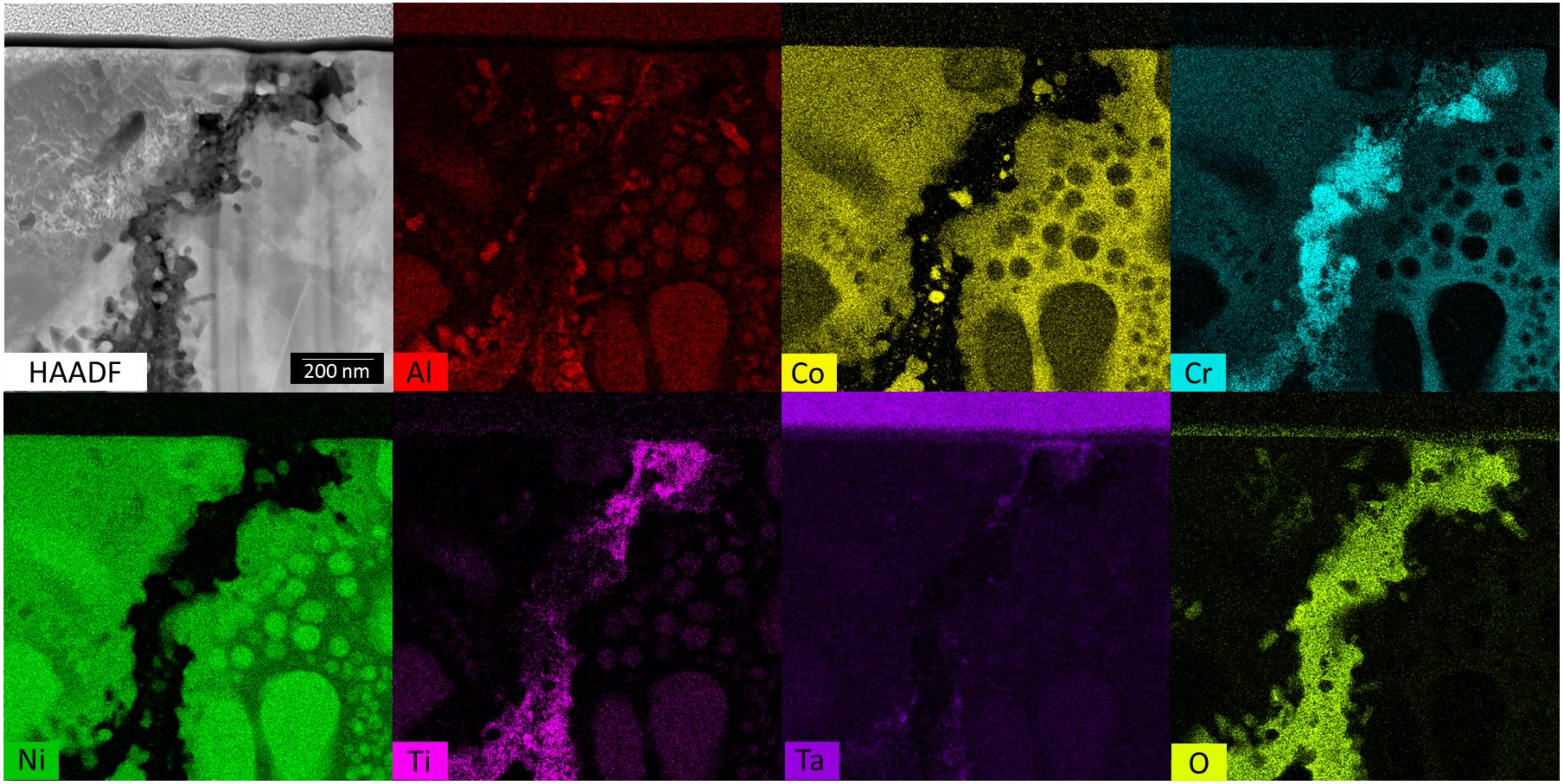
- High resolution STEM-EDS maps reveal Al-oxidation along a grain boundary.
- The composite map reveals a layer of γ between the Al-oxide and the γ' precipitate.

STEM EDS of Crack Tip Oxidation – Region A



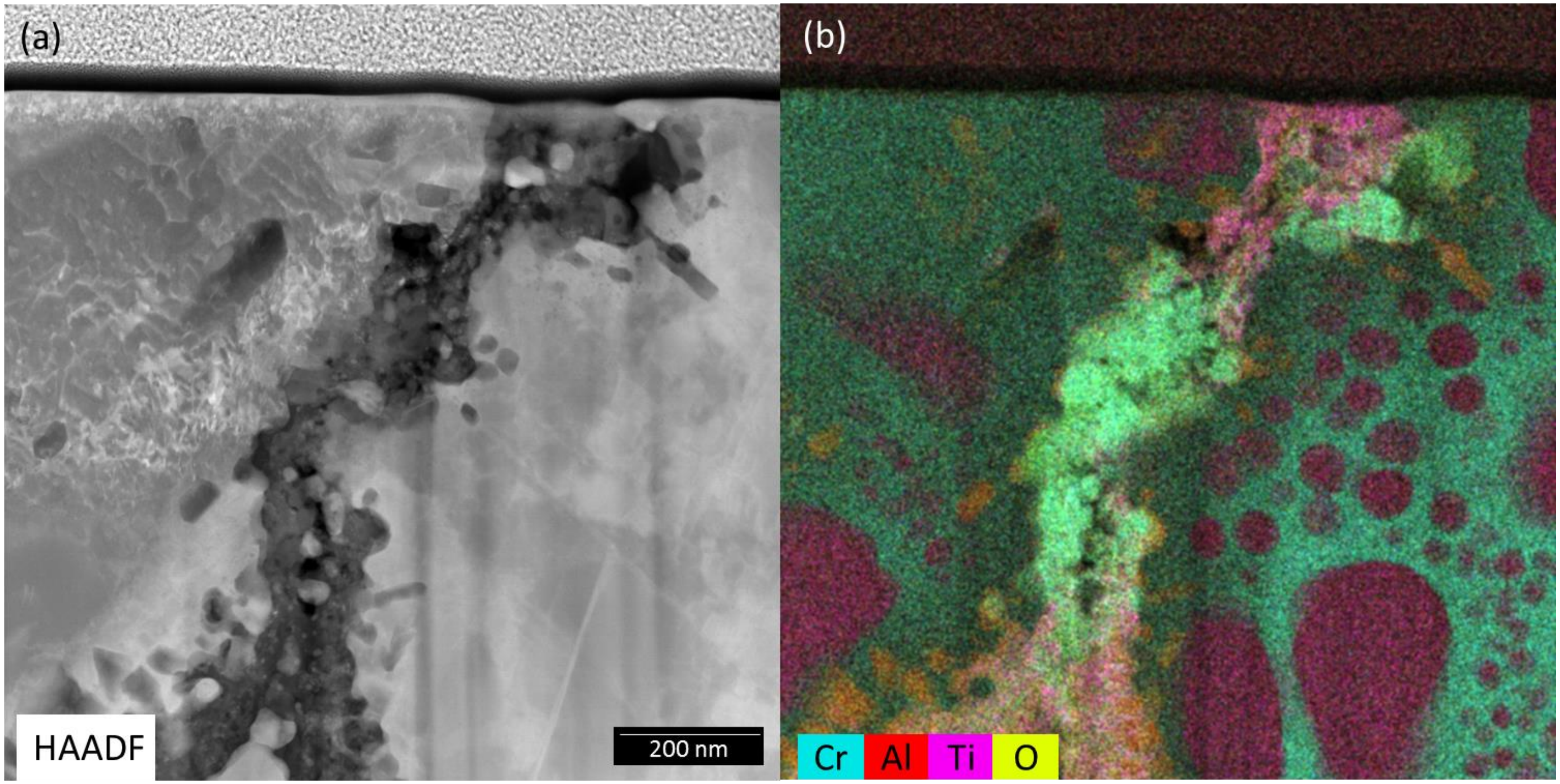
- High resolution STEM-EDS maps reveal Al-oxidation along a grain boundary.
- The composite map reveals a layer of γ between the Al-oxide and the γ' precipitate.

STEM EDS of Crack Tip Oxidation – Region B



- High resolution STEM-EDS maps reveal Ti and Cr oxidation forming along a grain boundary after the initial Al-oxide formation.
- The composite map reveals the presence of Ti or Cr oxidation is dictated by the surrounding microstructure

STEM EDS of Crack Tip Oxidation – Region B



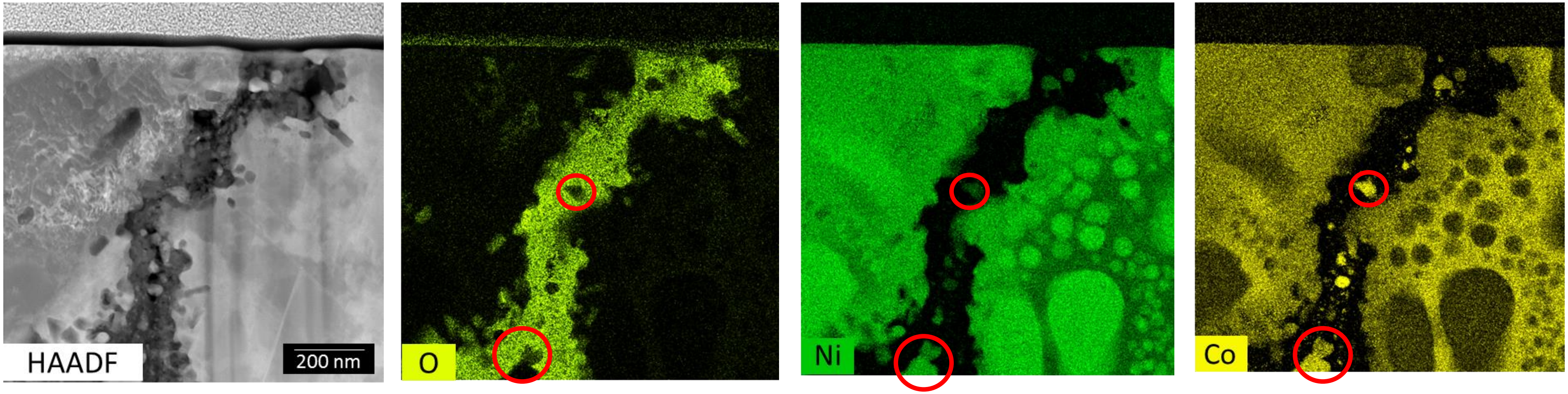
- High resolution STEM-EDS maps reveal Ti and Cr oxidation forming along a grain boundary after the initial Al-oxide formation.
- The composite map reveals the presence of Ti or Cr oxidation is dictated by the surrounding microstructure

Crack Tip Protection by Stable Oxide Layer and Failure Mode Transition

- Layered crack tip oxides order of formation in the intergranular threshold region:

  Al oxide→ Cr oxide → Ti oxide

- Delays formation of deleterious Ni and Co oxides

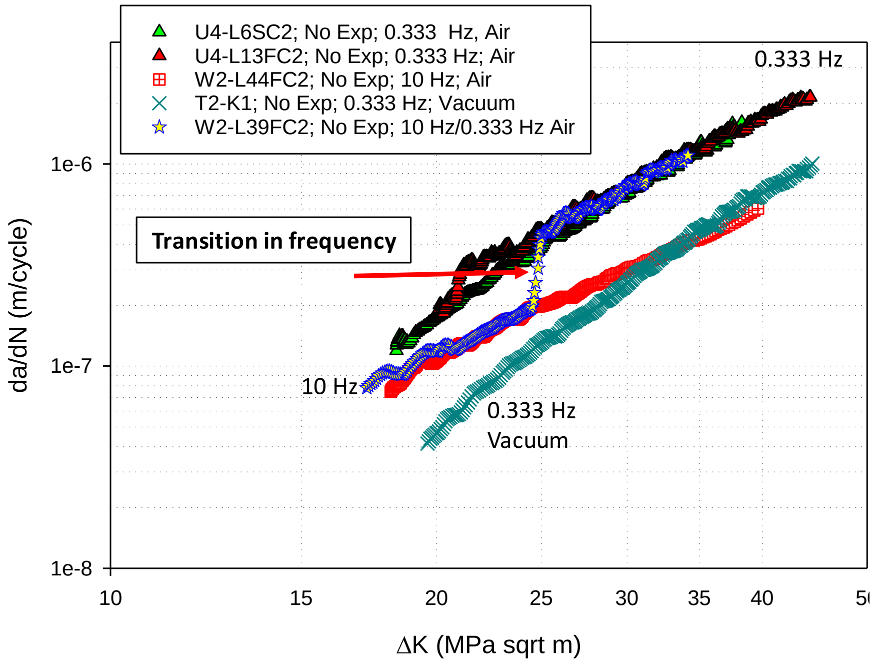


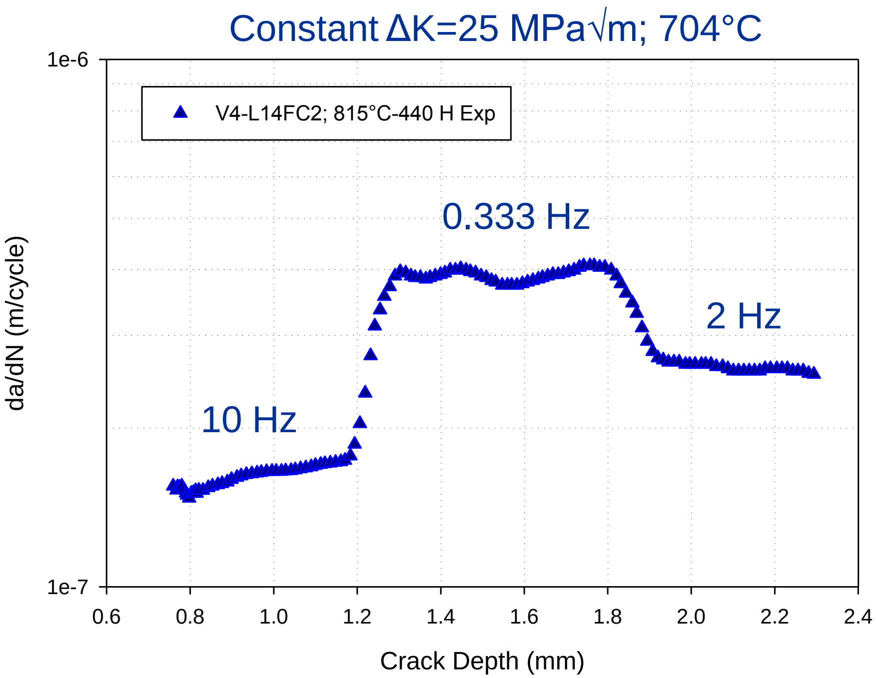
- Once near-threshold FCG rates become higher than the rate of formation of stable oxides, cyclic plasticity driven transgranular failure mode becomes dominant → Paris regime

- High freq., lower T or low PO_2 prevent stable oxide formation; no failure mode transition

Role of Intergranular Failure on Cyclic FCG in the Paris Regime

- Mixed mode failure mode commonly encountered in the Paris regime at T>600°C
- What is the impact of intergranular failure component on FCG in Paris regime?

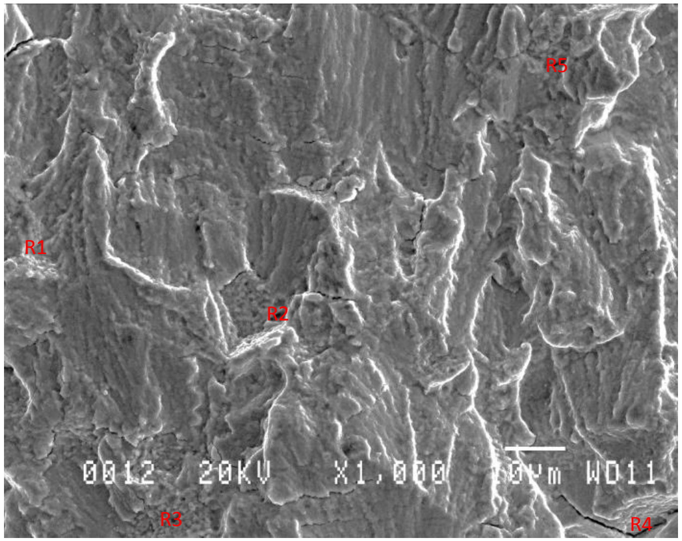


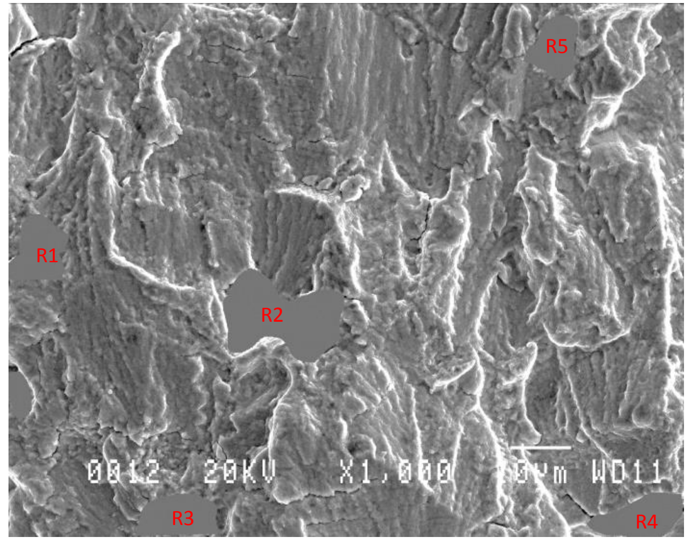


- Decreasing frequency increases FCG rates and intergranular failure content

Percent of Intergranular Content Quantified for Varied Heat Treatments at Different Test Frequencies

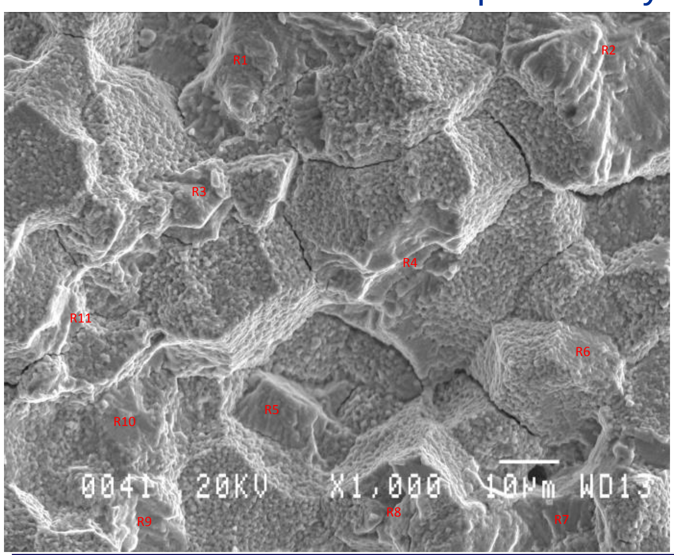
Heat treatment resistant to cyclic FCG integranular failure

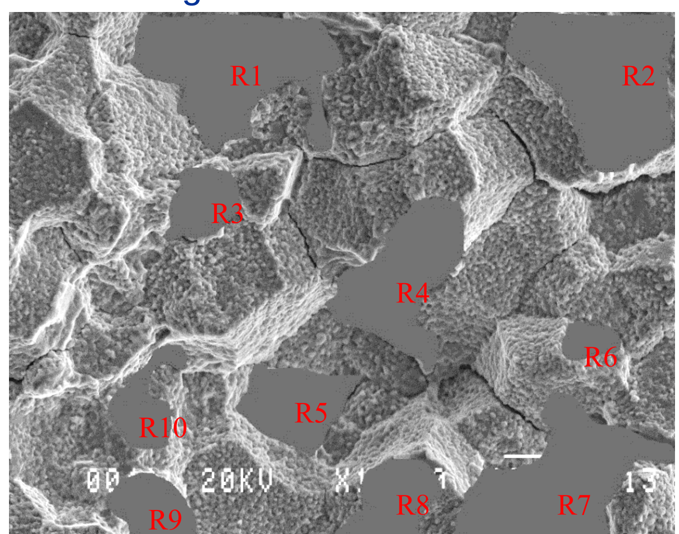




LSHR: 704°C;
ΔK=25 MPa√m
≈ 5% IG;
0.333 Hz

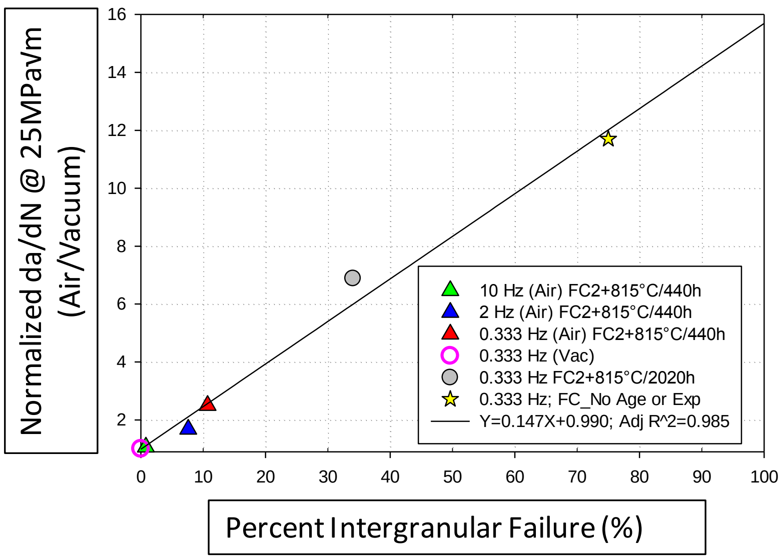
Heat treatment prone to cyclic FCG integranular failure

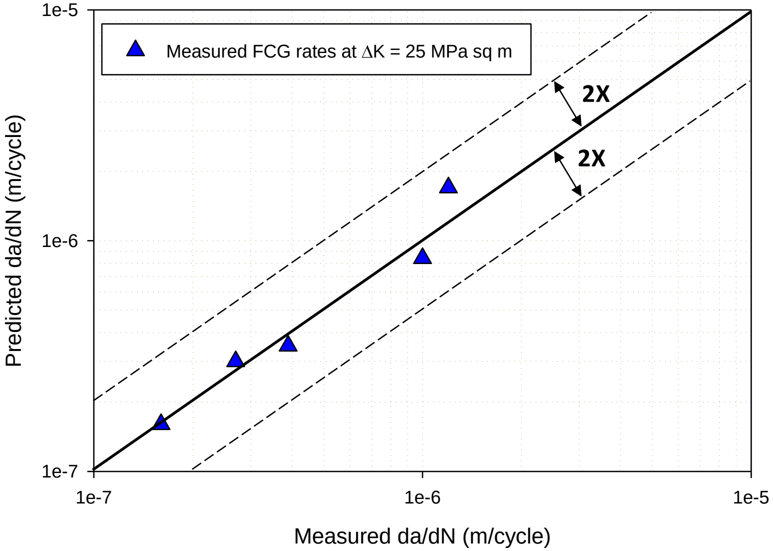




LSHR: 704°C;
ΔK=25 MPa√m
≈ 72% IG;
0.333 Hz

Relationship Between Percentage of Intergranular Failure and Cyclic FCG Rates – Paris Regime



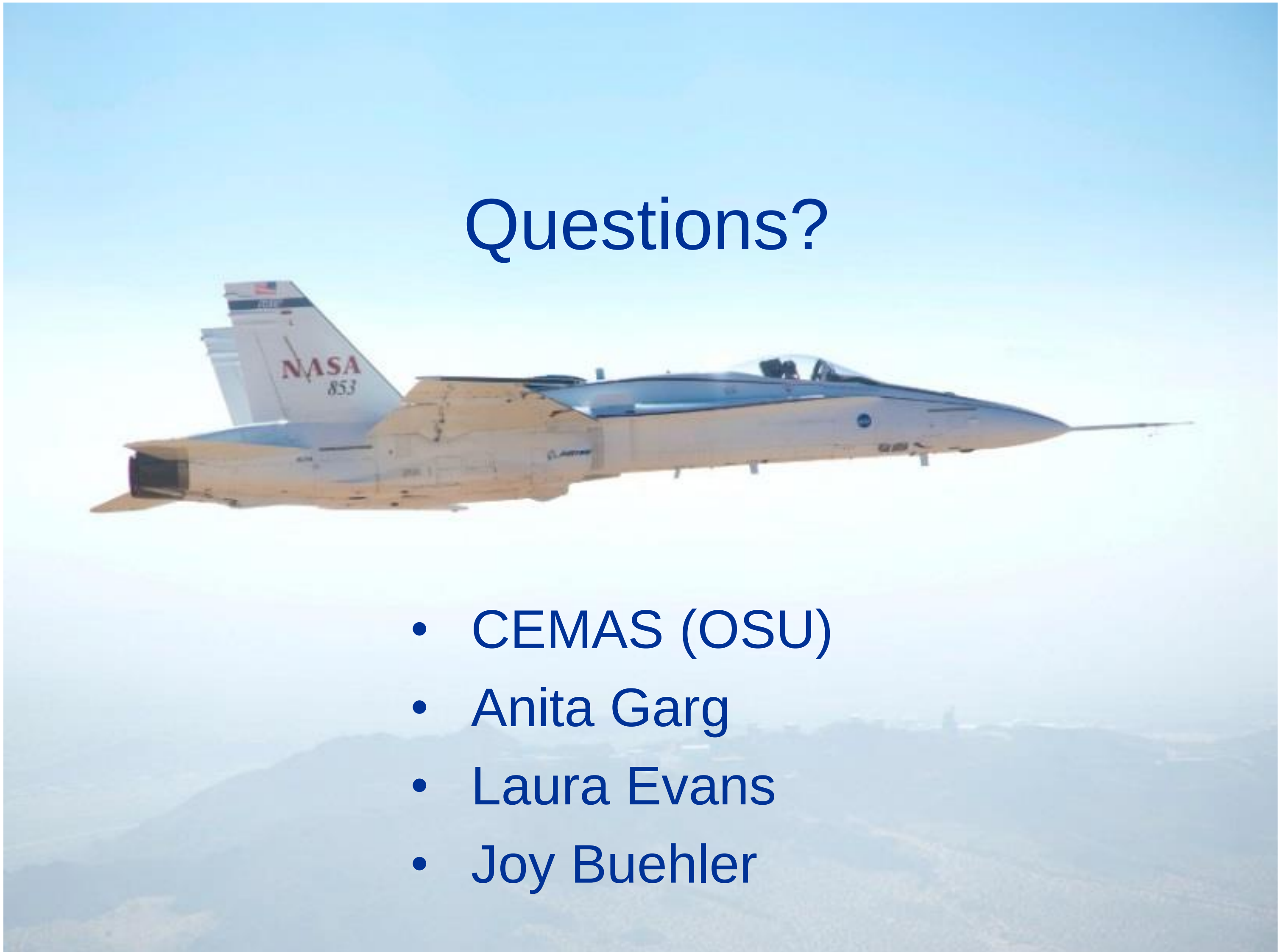


- Intergranular failure content dominant factor in Paris regime cyclic FCG resistance.
- 10% integranular failure content increases FCG rates by ≈ 2x
- Linear relationship between intergranular failure content and FCG rates.
- The linearity of the relationship enables use of Rule of Mixtures to predict FCG
- Once GB's transition to intergranular failure, their contribution to increasing FCG is the same without regard to their previous ability to resist intergranular failure.

Conclusions

- Unexpected failure mode transition to intergranular failure in the near-threshold FCG regime documented and characterized for LSHR and ME3 superalloys
- Formation of stable Al and Cr oxides protects the crack tip region from early formation of deleterious Ni oxides and is thought to slow down crack growth
- Failure mode transition proposed to occur when the rate of FCG exceeds the rate at which protective stable oxides can form.
- In the near-threshold regime crack advances through an oxide micro-cracking process which is mostly governed by Kmax parameter.
- In contrast, for the Paris regime the percentage of intergranular cracking in the mixed mode failure dominates FCG rates in comparison to transgranular failure.

Acknowledgments



Questions?

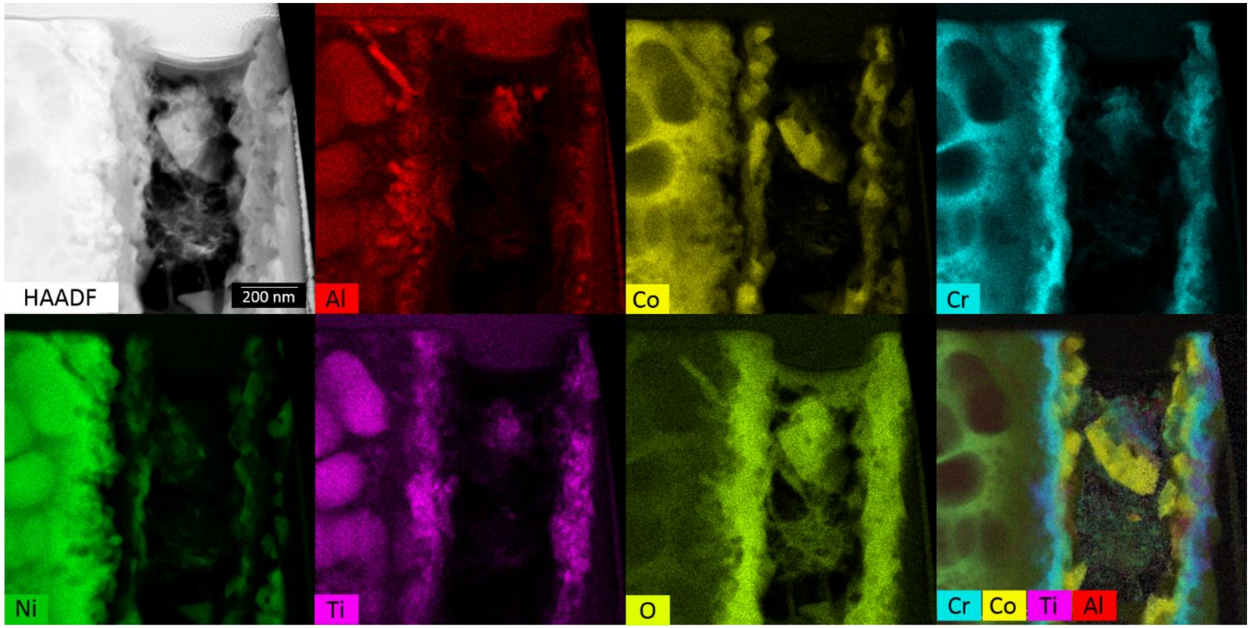
- CEMAS (OSU)
- Anita Garg
- Laura Evans
- Joy Buehler

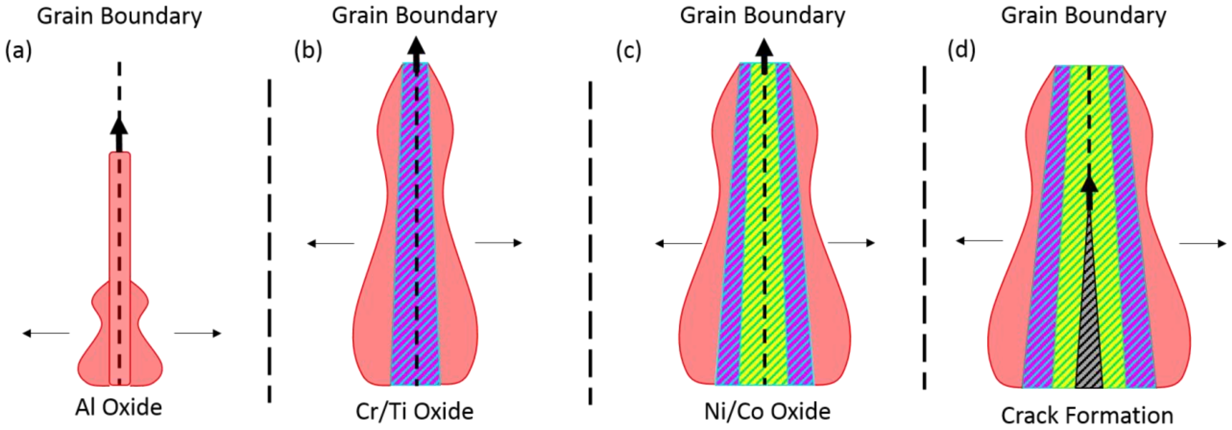
Backup

Table II. Comparison of Intergranular Band Width with Near-Threshold Region Crack Growth Distance at 704 °C, Tested at $R = 0.05$

Alloy/Specimen	Test Type	Frequency (Hz)	Crack Length Distance to/from 4×10^{-8} m/Cycle to/from Threshold (μm)	Average Intergranular Band Width (Five Measurements, μm)	K_{max} at Failure Mode Transition (MPa m$^{1/2}$)
LSHR/W2-L30SC2	load shed	0.333	75	72	14.5
LSHR/W2-L38SC2	load shed	0.333	75	82	14.5
LSHR/W2-L53FC2	load shed	0.333	187	170	15.5
ME3/H111-KW3	load shed	0.333	110	105	13
ME3/H111-KR3	load shed	0.333	120	110	13.8
LSHR/T2-K5	constant load/ short precrack	2	137	131	15.8
LSHR/T2-K6	constant load/ short precrack	0.667	205	222	18

Transgranular Crack Oxidation





Presence of Stacking Fault Segregation and Cottrell Atmospheres