

***PNNL Presentations on SCC Initiation
at the 2017 EPRI Alloy 690/152/52
Research Collaboration Meeting
Milestone Report: M3LW-18OR0402032***



Light Water Reactor Sustainability R&D Program

***Steve Bruemmer, Ziqing Zhai
and Mychailo Toloczko
Pacific Northwest National Laboratory***

*Alloy 690/ 152/ 52 Research
Collaboration Meeting*

November 28-30, 2017



PNNL Presentations at the 2017 EPRI Alloy 690/152/52 Collaboration Meeting:

- **ICG-EAC Alloy 600 PWSCC Initiation Round Robin Testing at PNNL** – review of LWRS-supported SCC initiation tests on all three alloy 600 materials and correlation with reported results from six other international laboratories.
- **Update on Alloy 690 SCC Initiation Research at PNNL** – summary of ongoing LWRS-supported SCC initiation research on cold-worked alloy 690 CRDM and plate materials. First ever detection of crack initiation during constant load testing
- **Additional PNNL Presentations (available by request):**
 - **EPRI-NRC Cooperative Project: PWSCC Crack Initiation Characterization of Alloy 182** – SCC initiation results were reviewed for alloy 182 welds including collaborative tests with our LWRS project plus ongoing tests on alloy 690/152/52.
 - **NRC Project: Update on Alloy 152/52 SCC Crack Growth Research at PNNL** - work on high-Cr welds was reviewed including test results on alloy 152, 152M, 52, 52M and 52MSS weld metals along with dissimilar metal welds, overlay welds and alloy 52M containing ductility dip cracks.

ICG-EAC Alloy 600 PWSCC Initiation Round Robin Testing at PNNL



***Steve Bruemmer, Ziqing Zhai
and Mychailo Toloczko***

Pacific Northwest National Laboratory

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Tampa, Florida November 28, 2017

Light Water Reactor Sustainability R&D Program



Presentation Overview

- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- *15%CF PNNL NX6106XK-11 MA600 Test Results*
- *15%CF EPRI/GE 31907 SA600 Test Results*
- *15%CF Rolls Royce 11415 SA600 Test Results*
- *Summary*
- *Additional SCC Initiation Test Results at 325°C, Estimated Activation Energy*

ICG-EAC Round Robin

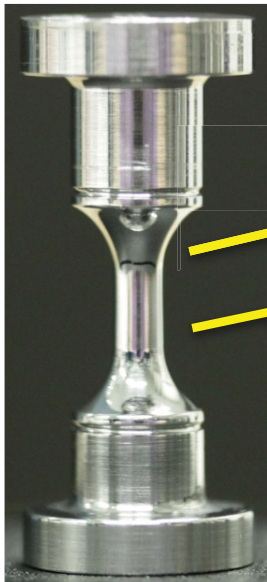
- *Recent growth in interest for quantitative measurement of SCC initiation times for LWR pressure boundary materials.*
- *Round Robin was proposed to analyze lab-to-lab variability.*
- *Emphasis on constant load tensile specimens with in-situ measurement of initiation by DCPD, but other test methods are welcome.*
- *Cold-worked alloy 600 in PWR primary water selected.*
 - *Substantial prior testing experience.*
 - *Relatively low initiation times for cold-worked material.*
 - *Primary, secondary and tertiary materials identified.*
 - *Primary and secondary materials distributed in late 2015.*
- *Additional details in Stuart Medway's presentation.*
- *PNNL has completed tests on all three round robin materials at 360°C along with tests on two materials at 325°C.*

SCC Initiation Testing at PNNL

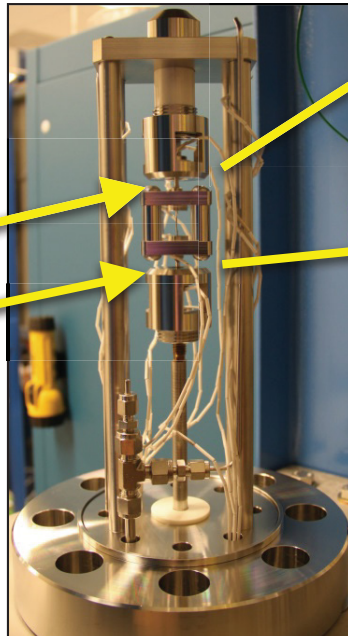
SCC initiation test systems with active loading and in-situ DCPD crack-detection:

- ▶ **LWRS:** two smaller systems recently converted to test 6 fully instrumented specimens + one 36-specimen system with up to 12 specimens instrumented.
- ▶ **NRC/EPRI:** two 36-specimen systems with 24 instrumented.
- ▶ **Other DOE:** three smaller systems to test 3 fully instrumented specimens.

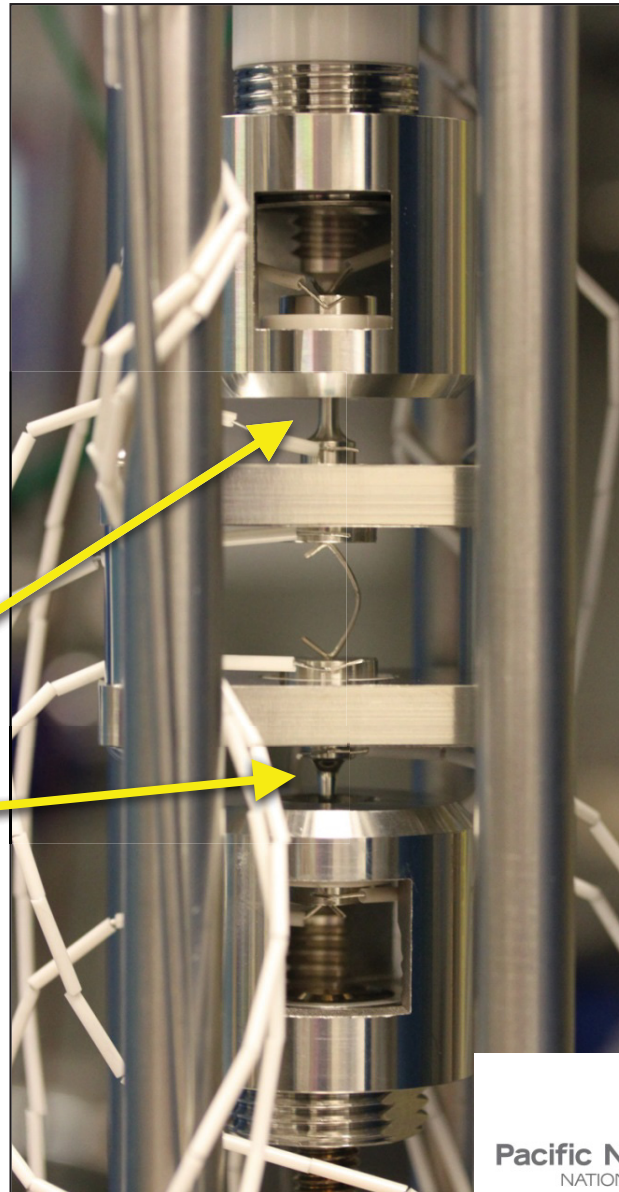
1.2" Tall SCC Initiation Specimen



Small SCC Initiation System



Small SCC Initiation System



36-Specimen SCC Initiation System

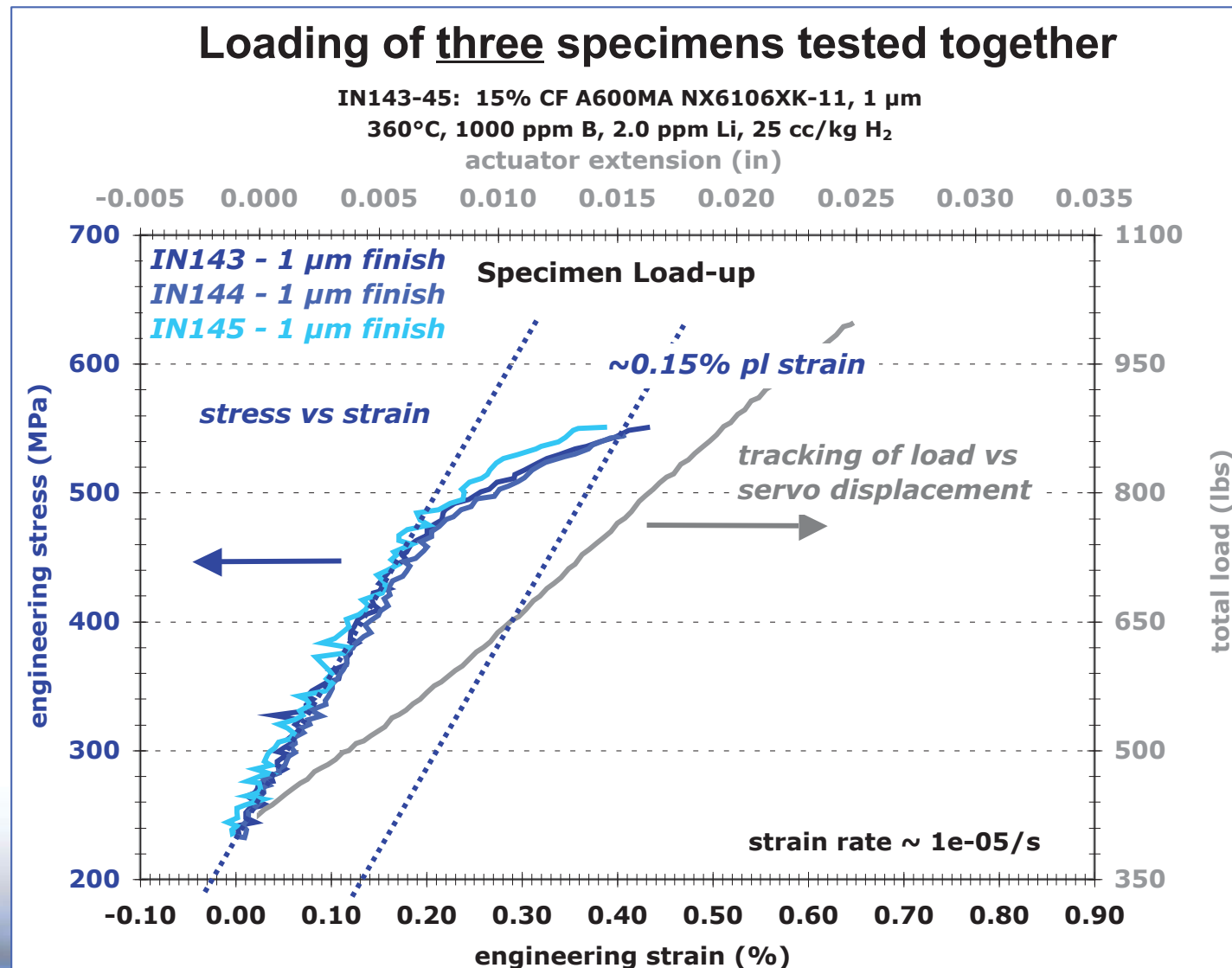


Test Conditions

- Gauge section of all specimens polished to a 1 μm finish.
- 360°C selected to match prior experience at PNNL. May perform another set of tests at 325°C.
- Water conditions (as defined by Round Robin)
 - PWR primary water
 - 1000 ppm B, 2 ppm Li
 - Ni/NiO stability line (25 cc/kg H_2)
- All specimens loaded to small scale plastic yielding (0.1-0.2% plastic strain).
- Testing started within 12 hours of reaching full temperature.
- For the 3-specimen test systems that were used, when a specimen initiates, the test is stopped to remove it. Remaining specimens are then reloaded to original load.

Test Methodology

- Gauge section is typically polished to a $1\ \mu\text{m}$ finish to facilitate detection of cracking via surface and cross section examinations.
- Testing typically conducted at the yield strength of the material.
- Confirmation of yield determined by monitoring stress versus strain (from DCPD) at a displacement rate of $\sim 1 \times 10^{-5}\ \text{s}^{-1}$ (~ 1 hour to load).
- Loading typically stopped at ~ 0.1 - 0.2% plastic strain.

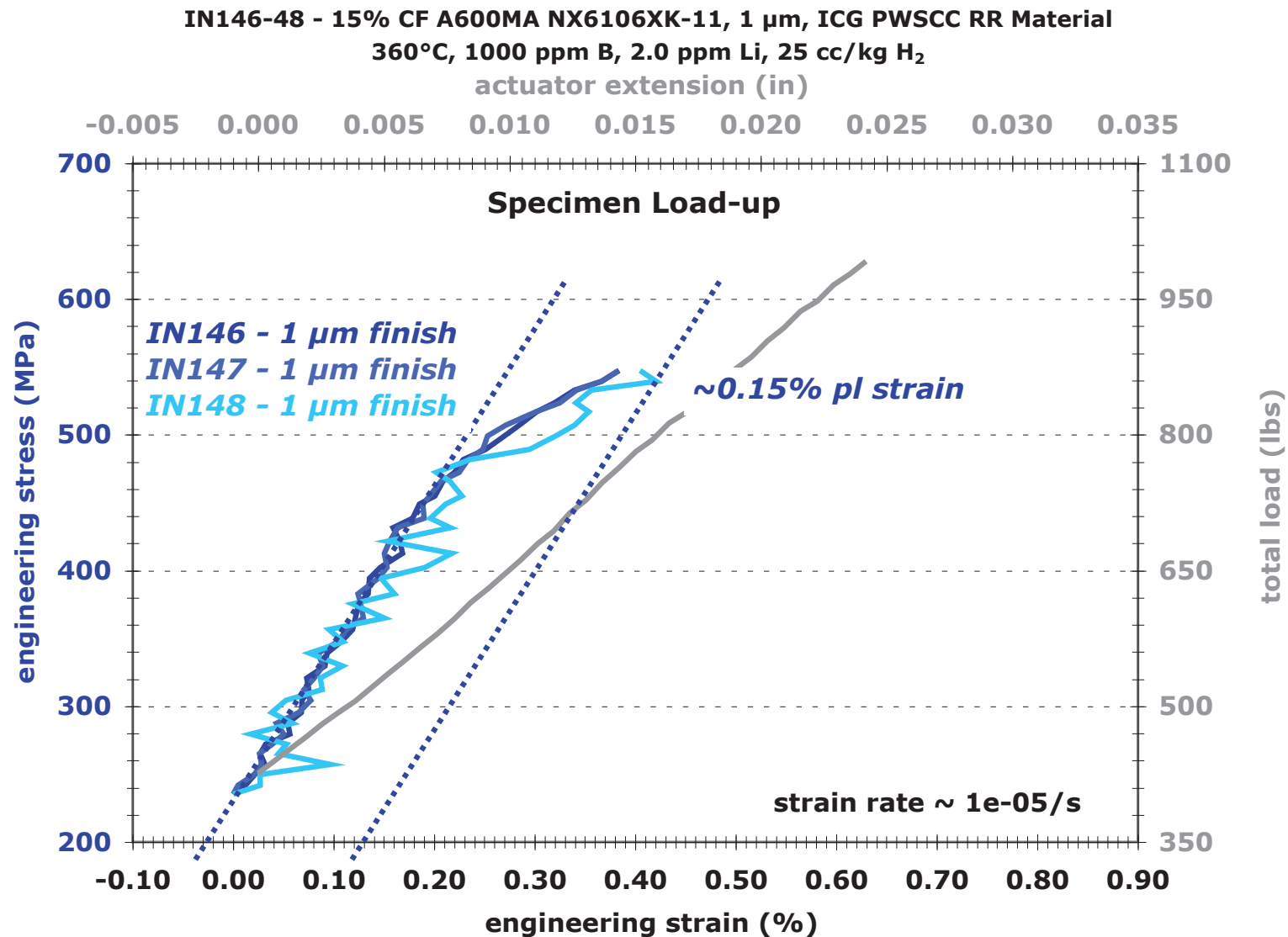


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Load-up of NX6106XK-11 Round Robin Specimens

- All three specimens behaved identically during tensile loading to reach the yield strength.
- Loading stopped at 0.12-0.15% plastic strain = **550 MPa**.
- 1 hour to reach full load.

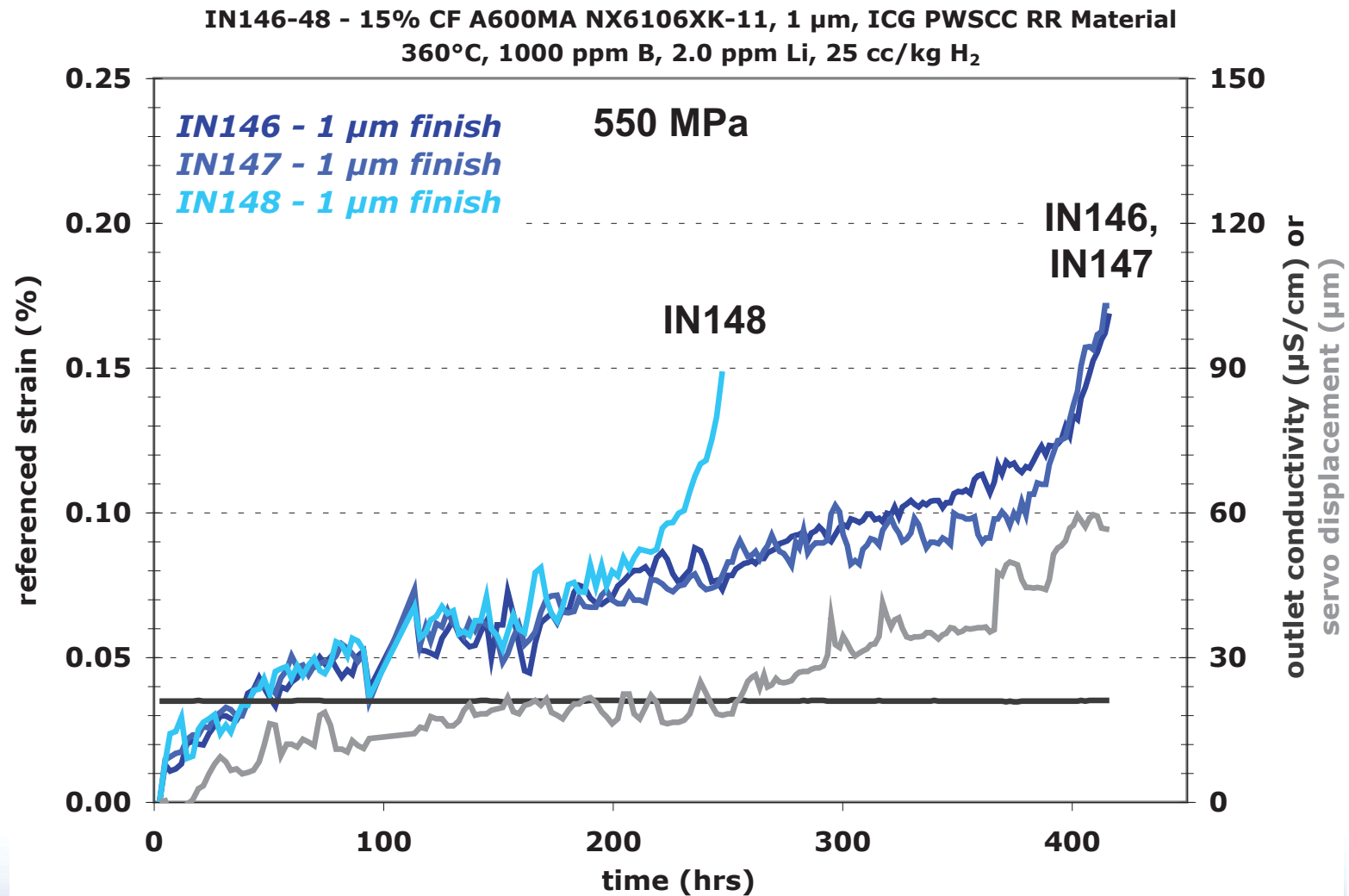


Initiation of NX6106XK-11 Round Robin Specimens

- Well behaved response, but unexpectedly low SCC initiation time for all three specimens (compared to prior results).

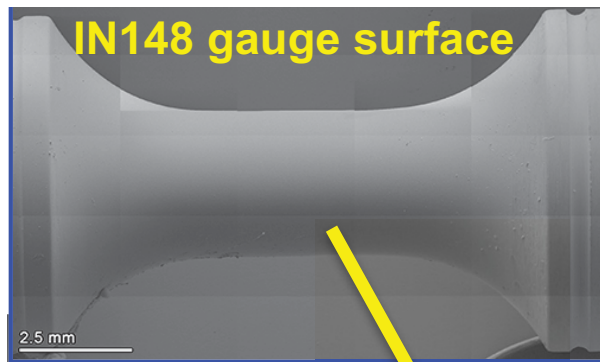
- SCC initiation times of 219, 385 and 396 hours.

- Well below prior tests on this plate heat in the cold-worked condition.

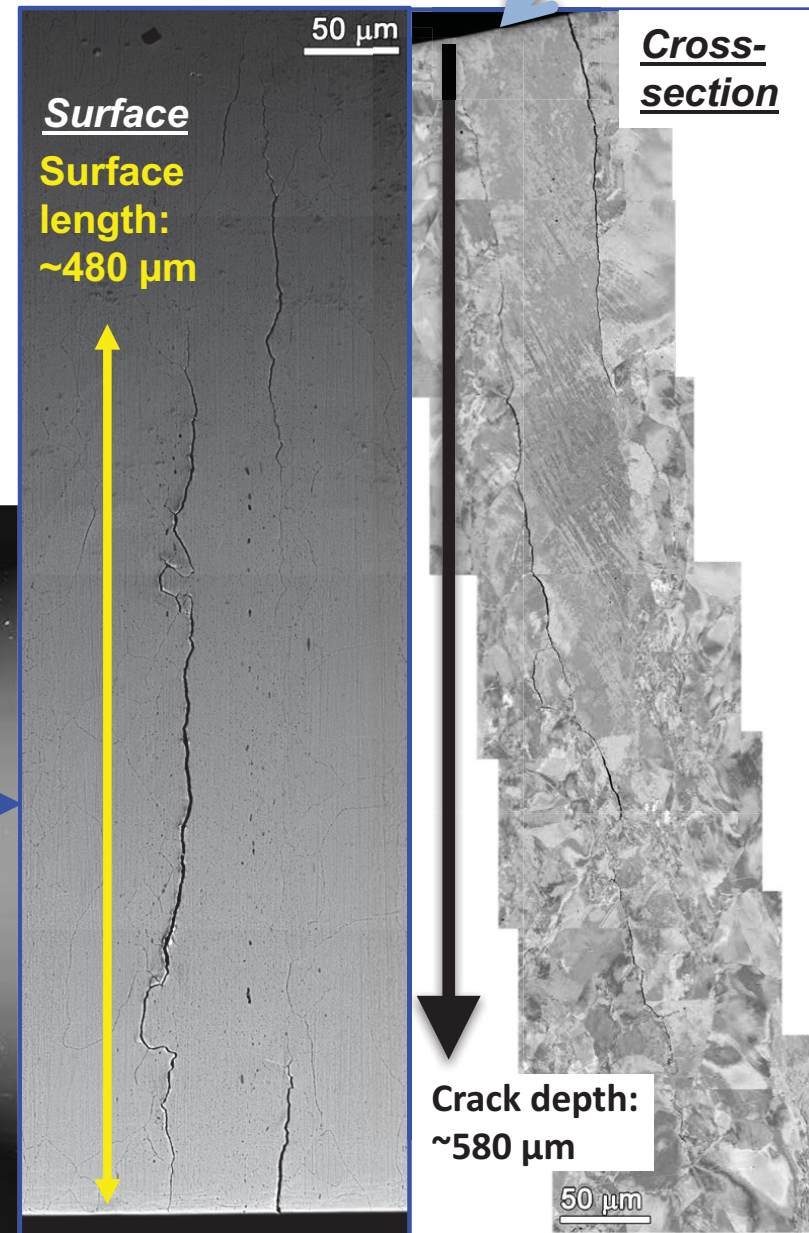
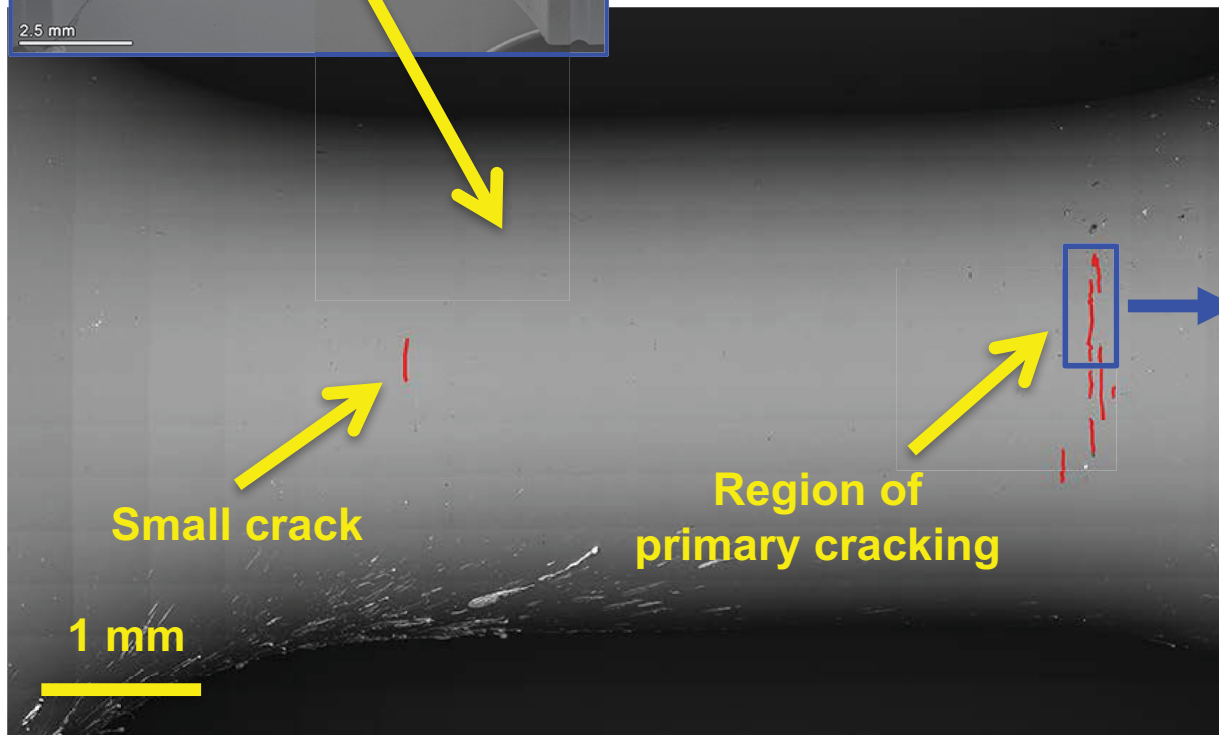


Observations of IN148 (219 h Initiation)

- Initiation at 219 hours. Test stopped at 250 hours for examinations.
- Region of primary cracking was easily found.
- Primary crack had surface length of $\sim 480\ \mu\text{m}$ and depth of $\sim 580\ \mu\text{m}$.

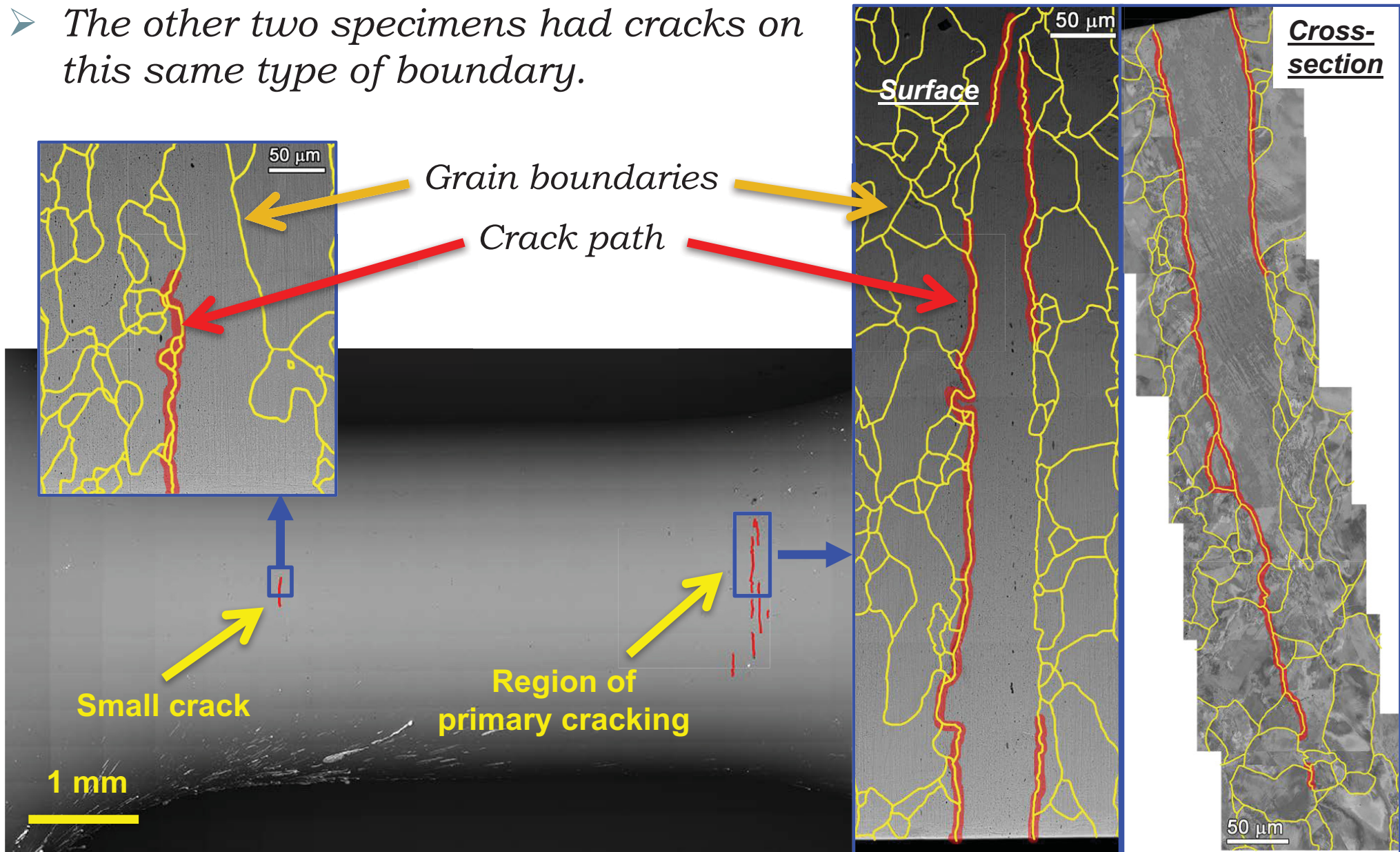


SEM surface and cross-section examinations



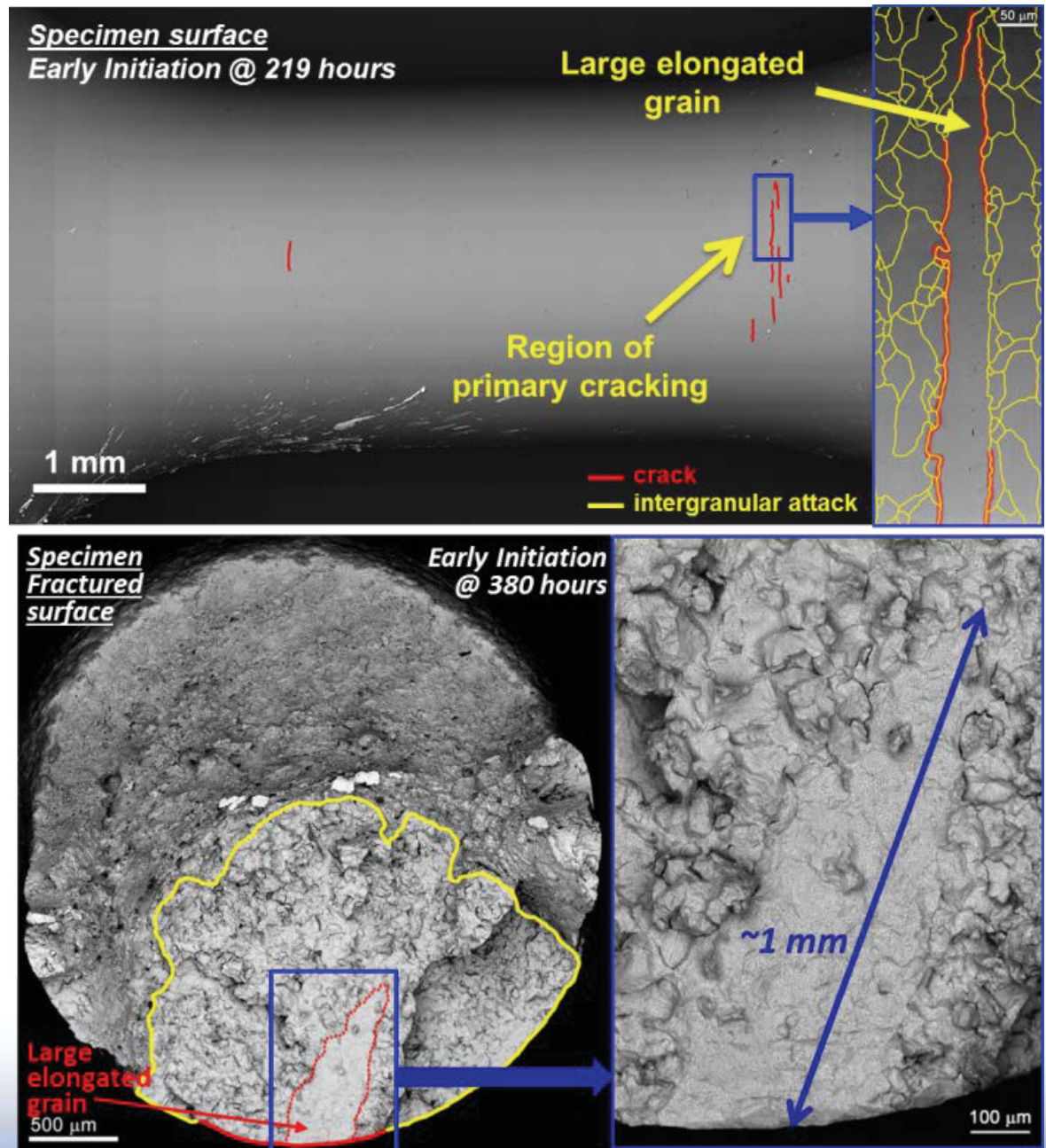
Observations of IN148 (219 h initiation)

- Cracking occurred on the grain boundaries of the sporadically distributed, large elongated grains.
- The other two specimens had cracks on this same type of boundary.



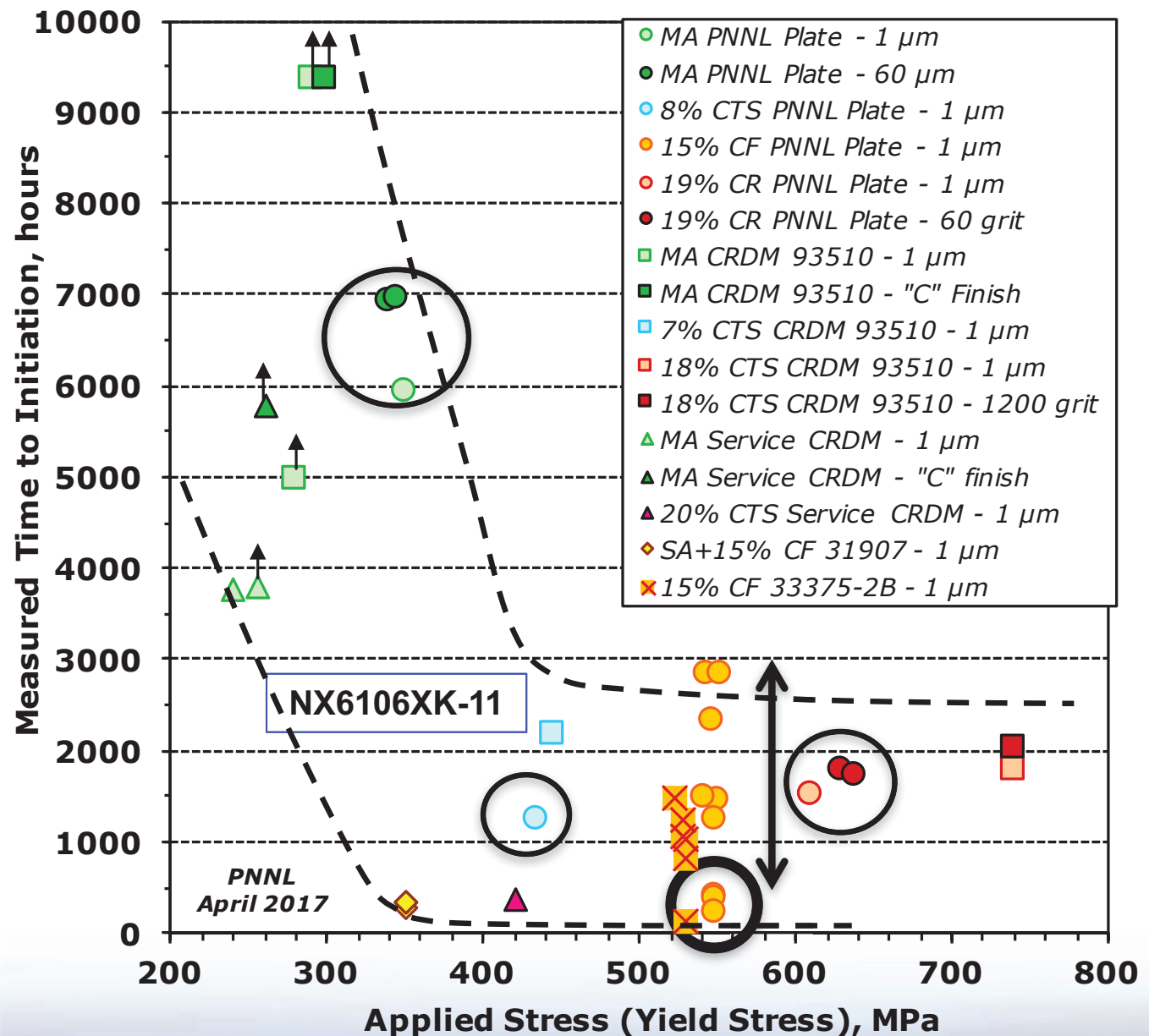
SCC Initiation Measurements on PNNL Plate

- Lower SCC initiation times for 15%CF PNNL plate in round robin tests are believed to result from cracking along sporadic large elongated grains in the banded microstructure that intersect the gauge surface.



SCC Initiation Measurements on PNNL Plate

- *NX6106XK-11: lower RR initiation times than previous tests may result from differences in microstructure, cracking observed along large elongated grains in RR tests.*
- Additional tests on 15%CF PNNL material from different plate location revealed longer (>5X) crack initiation times.***

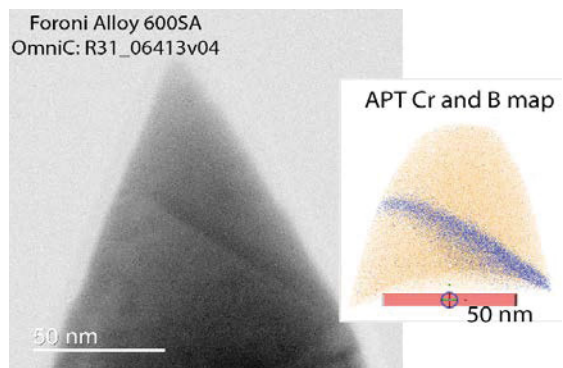
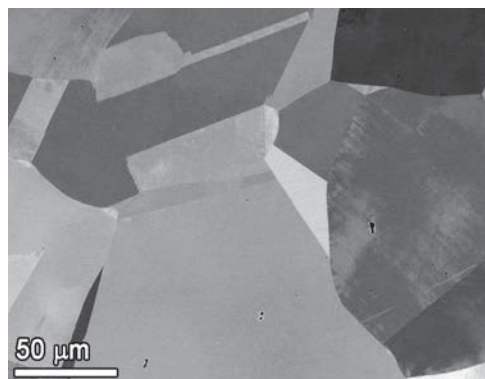
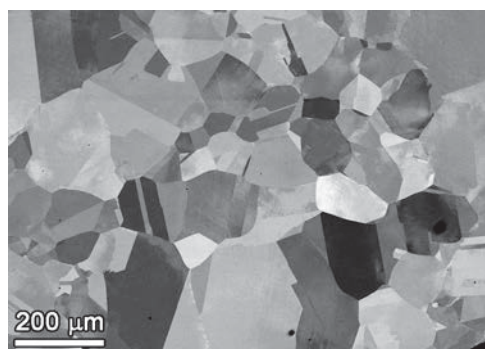


Presentation Overview

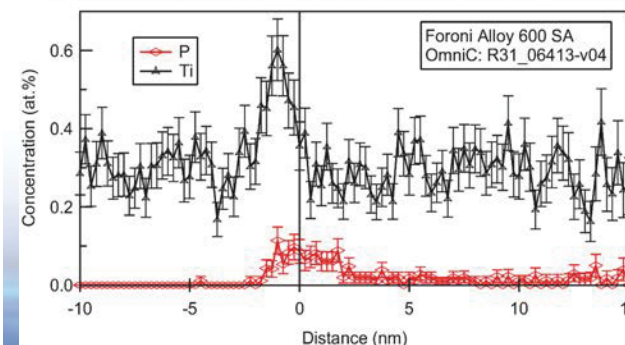
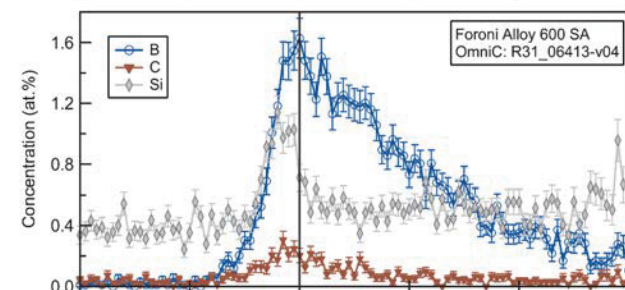
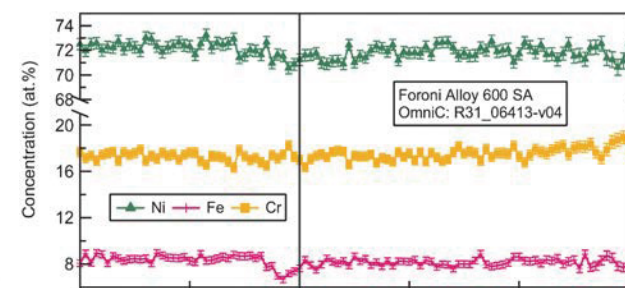
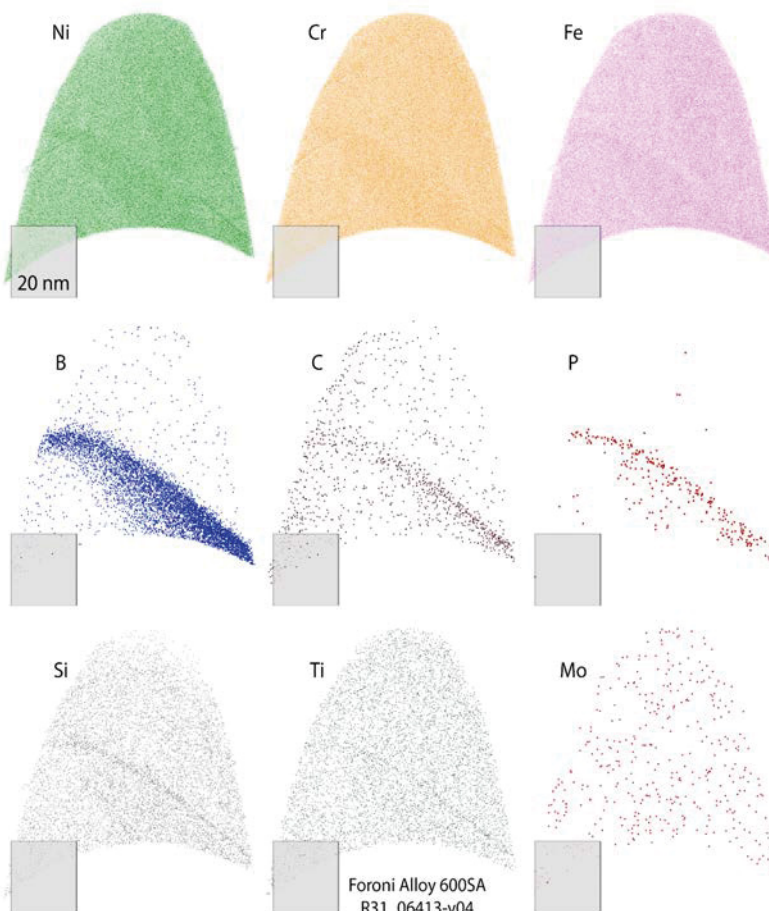
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Microstructure and Grain Boundary Composition in Solution-Annealed Alloy 600 Heat 31907

- Large grain size ranging from ~ 50 - $300\ \mu\text{m}$
- Clean grain boundaries

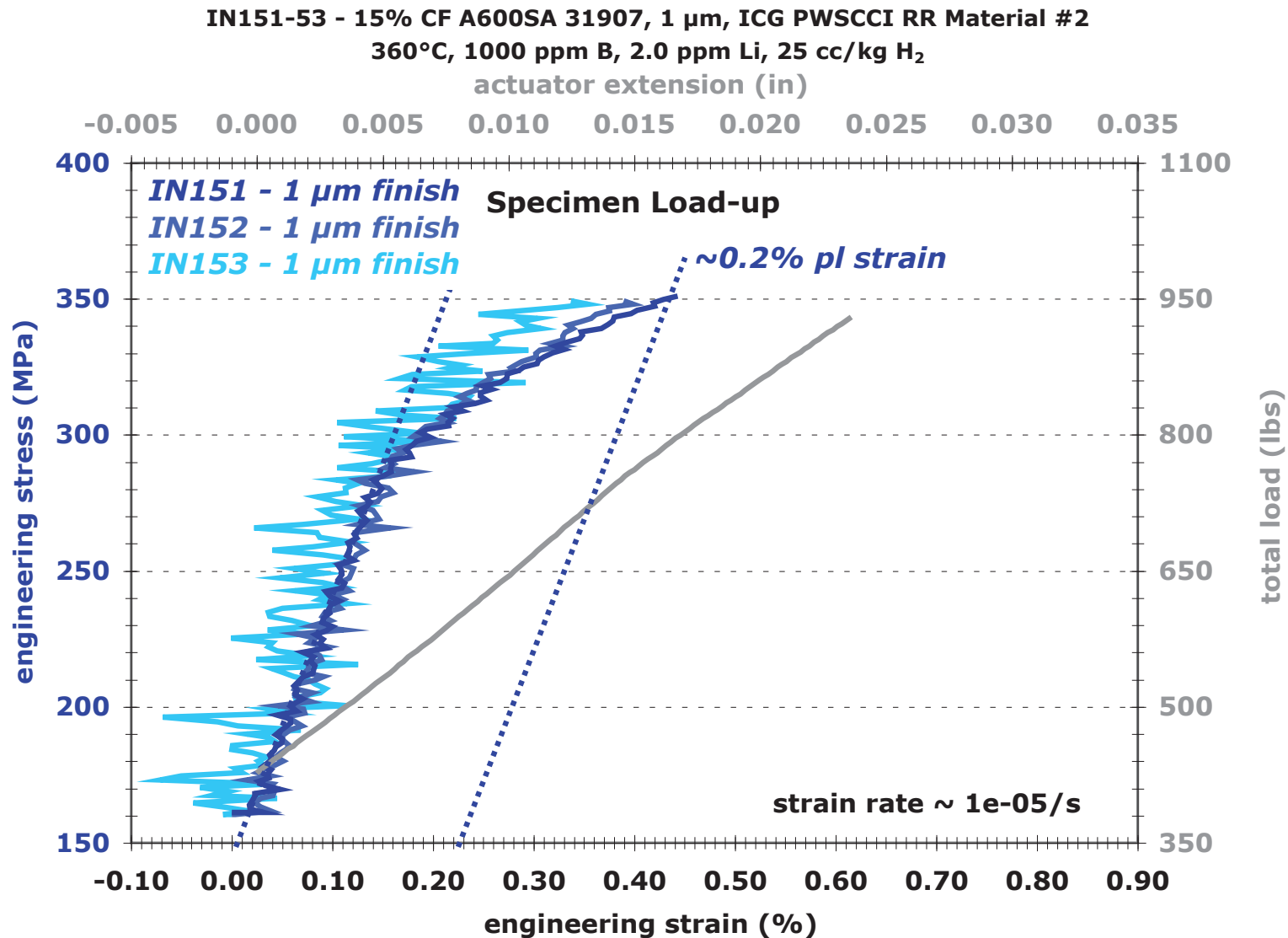


APT revealed boron grain boundary segregation to 1.8 at%, minor Si, Ti and P.



Load-up of 31907 Round Robin Specimens

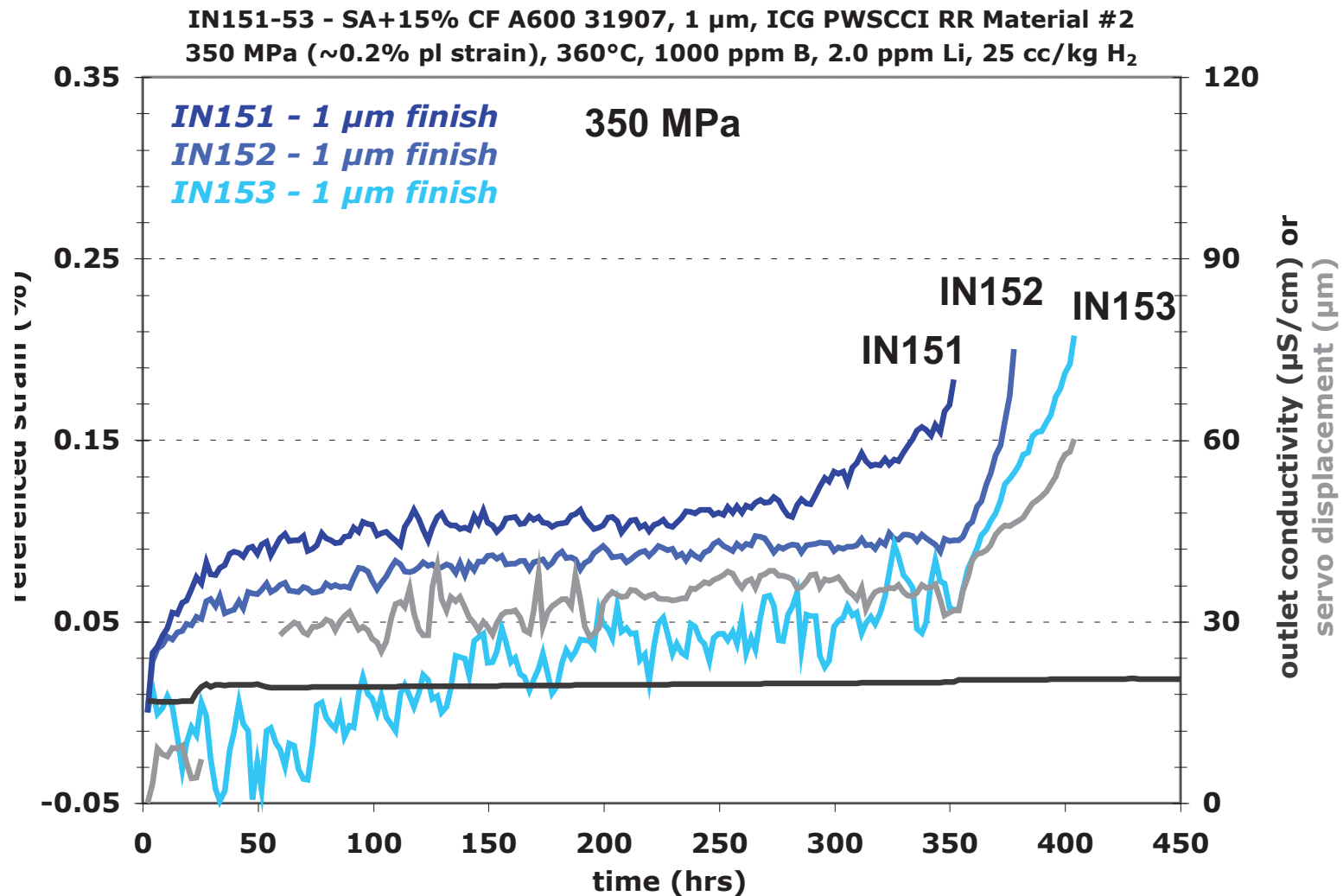
- All three specimens behaved nearly identically during tensile loading to reach the yield strength of the specimens.
- Loading was stopped at 0.1-0.2% plastic strain = **350 MPa**. Note lower YS than for 15%CF plate
- 1 hour to reach full load.



Initiation of 31907 Round Robin Specimens

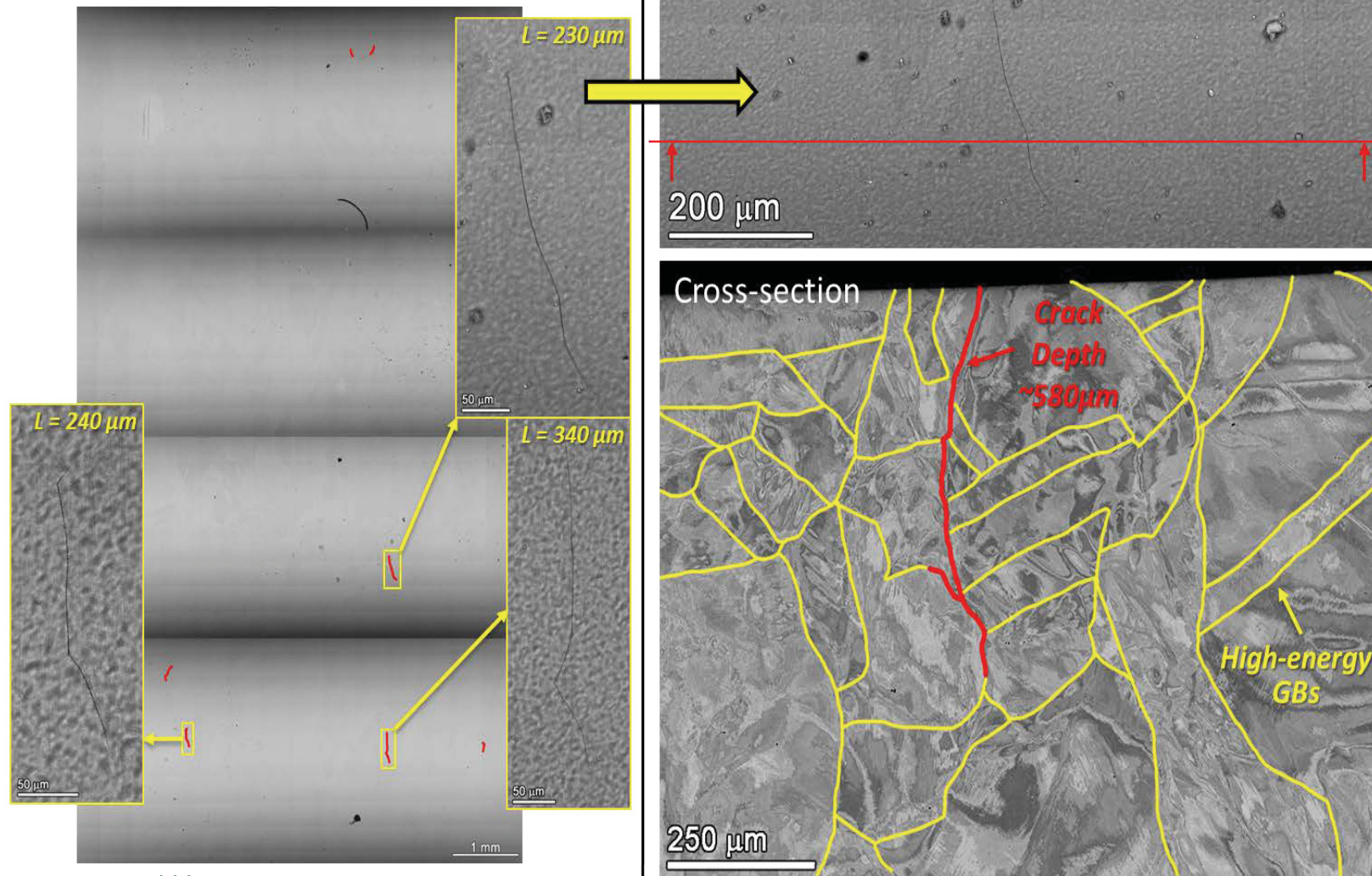
- First SCC initiation at 295 hours followed by other two specimens showing initiation at 351 and 368 hours.

- GE indicated similar SCC initiation times for SA+15%CF.
- Boron grain boundary segregation observed in the solution annealed material.



Examination of 31907 Round Robin Specimens

IN151 (SA+15%CF Alloy 600 heat 31907, 360°C, initiation at 295 h, total exposure of 352 h)

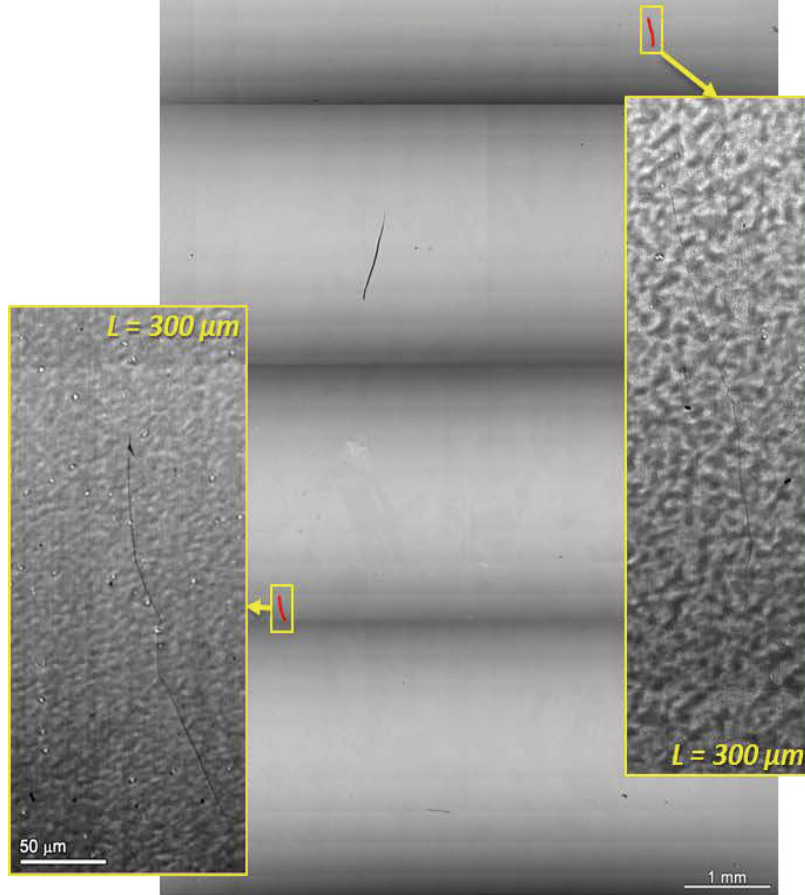


- Test stopped 57 h after DCPD initiation, main crack found to be >0.5 mm deep.

Examination of 31907 Round Robin Specimens

IN152 (SA+15%CF Alloy 600 heat 31907, 360°C, initiation at 354 h)

Gauge surface examination at 352 h



Crack surface examination at 514 h



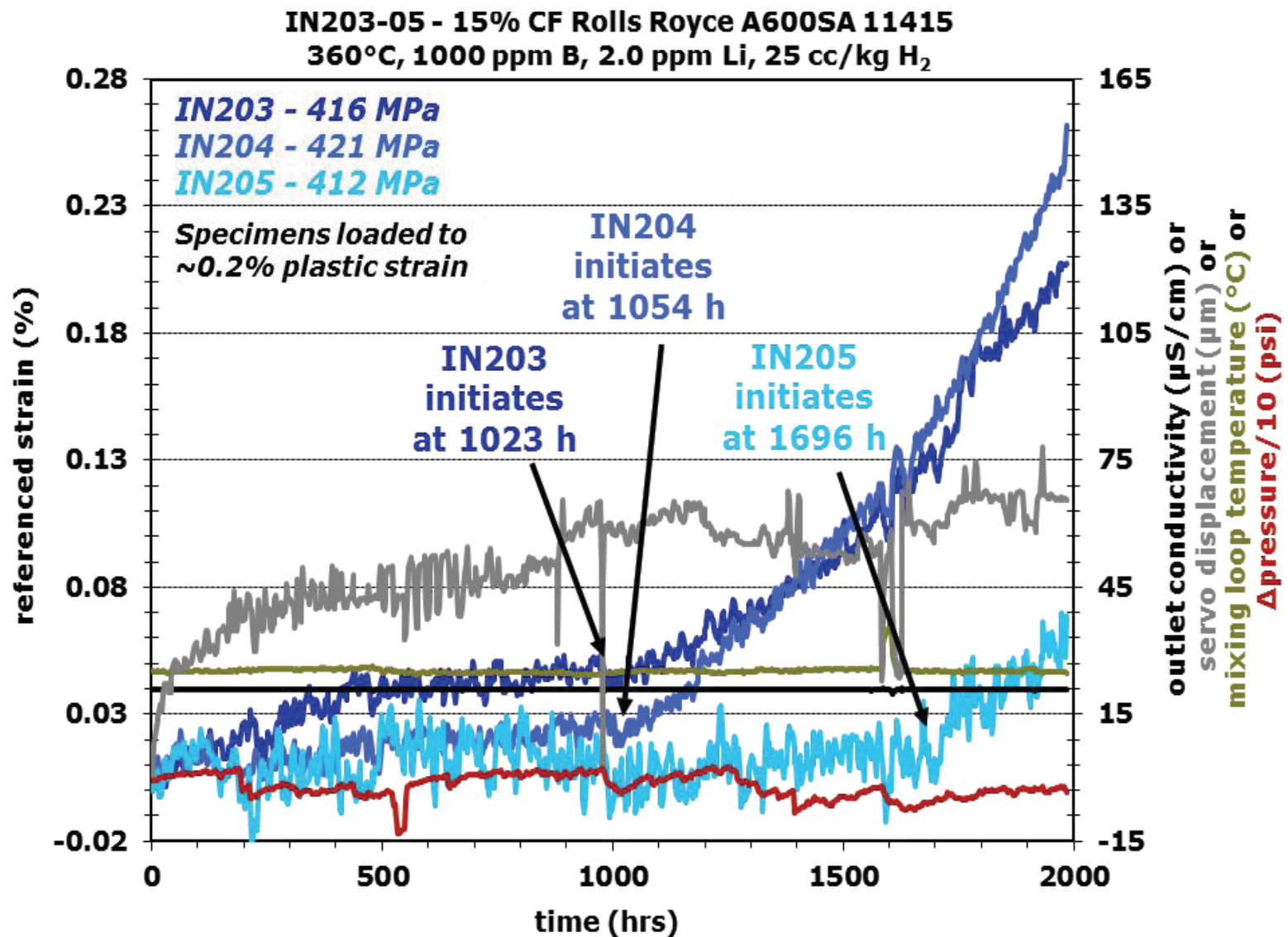
- *Full thickness IGSCC failure occurred in ~160 hours after DCPD-detected initiation.*

Presentation Overview

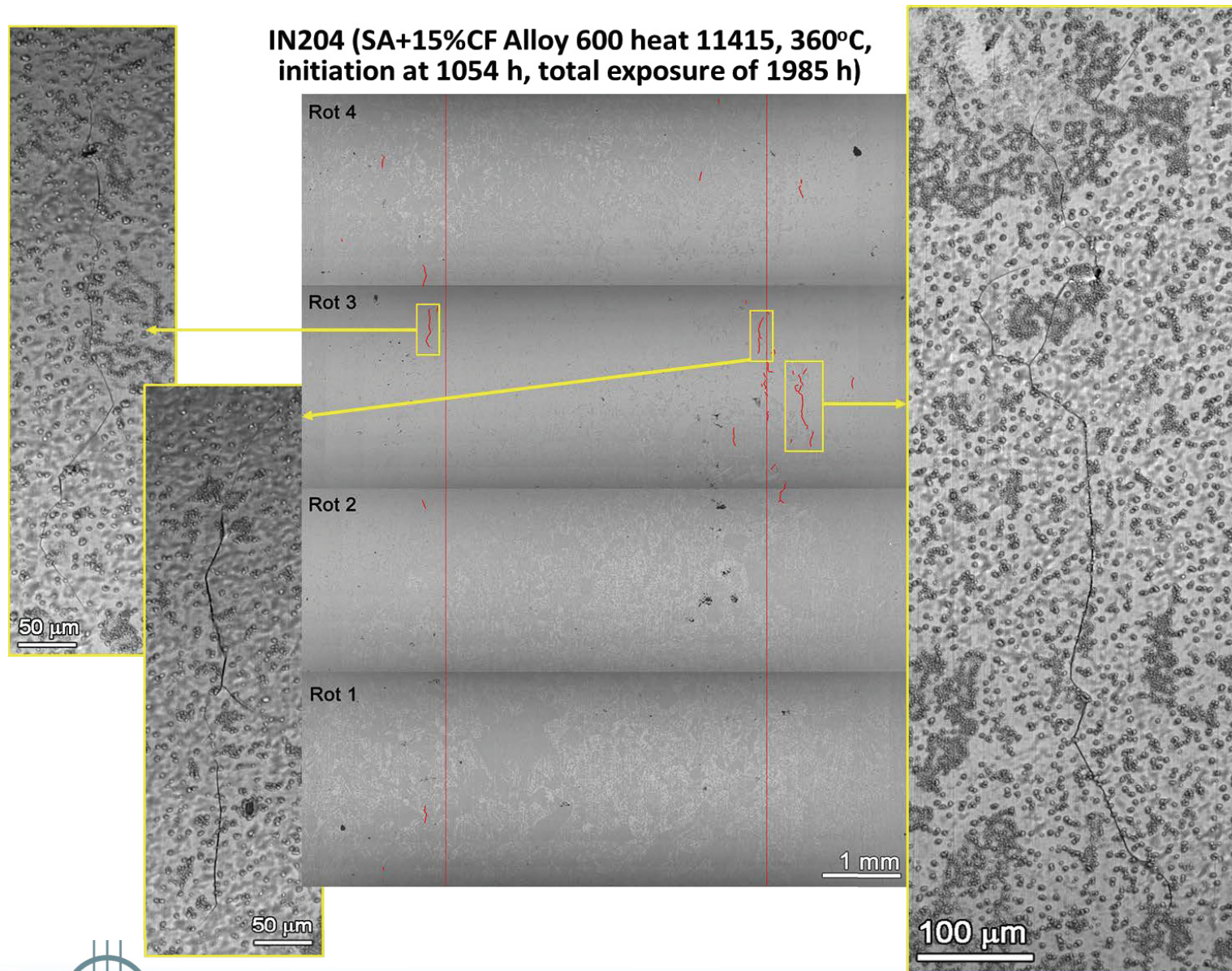
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Initiation of 11415 Round Robin Specimens

- First SCC initiation at 1023 hours followed by other two specimens showing initiation at 1054 and 1696 hours.
- Longer initiation times for this 15%CF SA alloy 600 heat.
- Distribution of grain size, no apparent grain boundary segregation,



Examination of 11415 Round Robin Specimens



- Test stopped 930 h after DCPD initiation, very tight surface cracks reaching $\sim 400 \mu\text{m}$ in length.

Presentation Overview

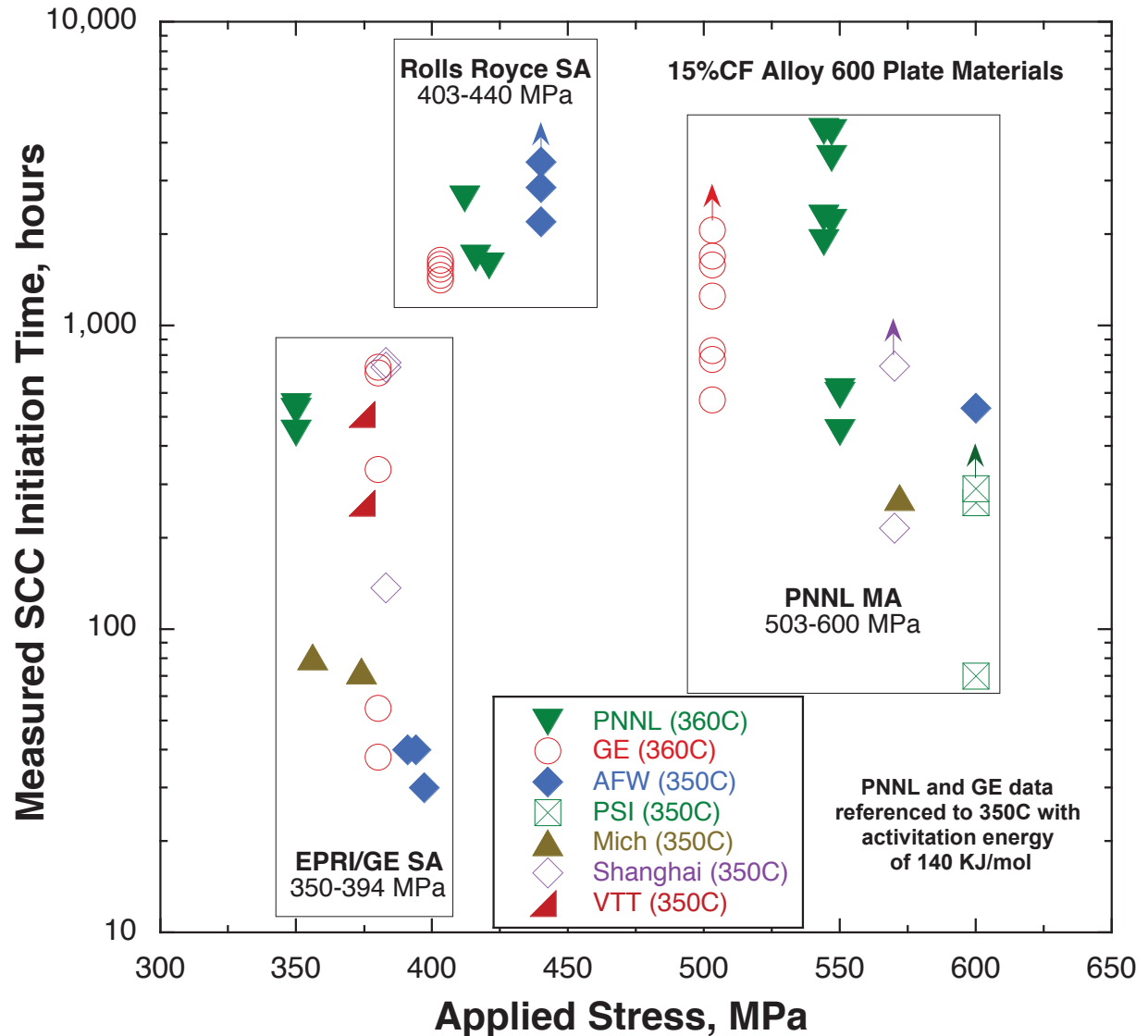
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Summary

- *Testing conditions*
 - *360°C simulated PWR primary water to match prior PNNL experience, other conditions aligned with round robin specification.*
 - *Stress and strain tracked during specimen load-up.*
- *Primary (NX6106XK-11) PNNL MA600 exhibited consistent SCC initiation response among the 3 specimens tested, however SCC initiation times were shorter than anticipated from prior PNNL tests.*
 - *Crack initiation found to occur at sporadic large grains in RR specimens, earlier plate specimens had a more equiaxed grain structure. The inhomogeneous microstructure in this RR plate promotes differences in SCC initiation response.*
- *Secondary (31907) EPRI/GE SA600 exhibited consistent SCC initiation response among the 3 specimens tested with low initiation times compared to PNNL experience. More uniform microstructure albeit with large grain size and grain boundary boron segregation.*
- *Tertiary (11415) Rolls Royce SA600 exhibited consistent SCC initiation response among the 3 specimens tested, longer initiation times than for other 15%CF RR materials.*

Preliminary Comparison to Other Labs

- *NX6106XK-11: range in SCC initiation times from 200-4000 hours may result from inhomogeneous microstructure.*
- *31907: range in SCC initiation times from 30-700 hours, lower initiation times at lower applied stress. Higher SCC susceptibility in this low C heat may result from higher creep rate.*
- *Heat 11415: longer SCC initiation times for this higher C heat, limited data but more consistent.*



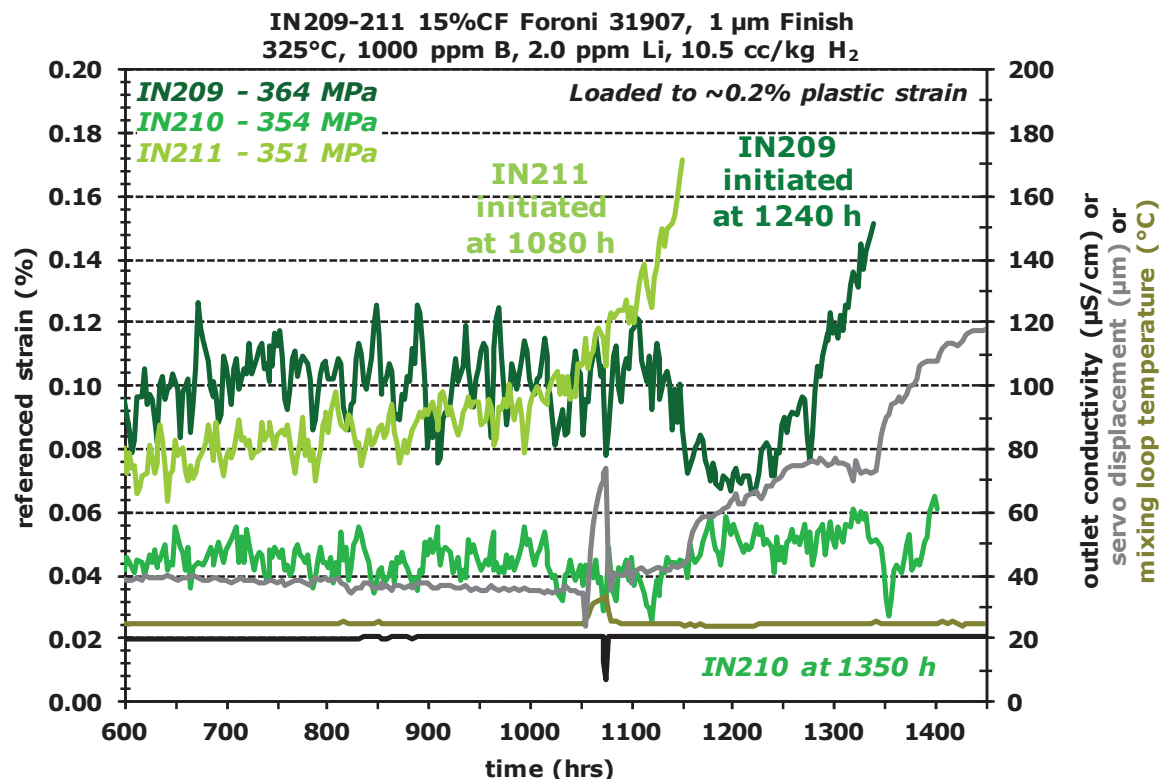
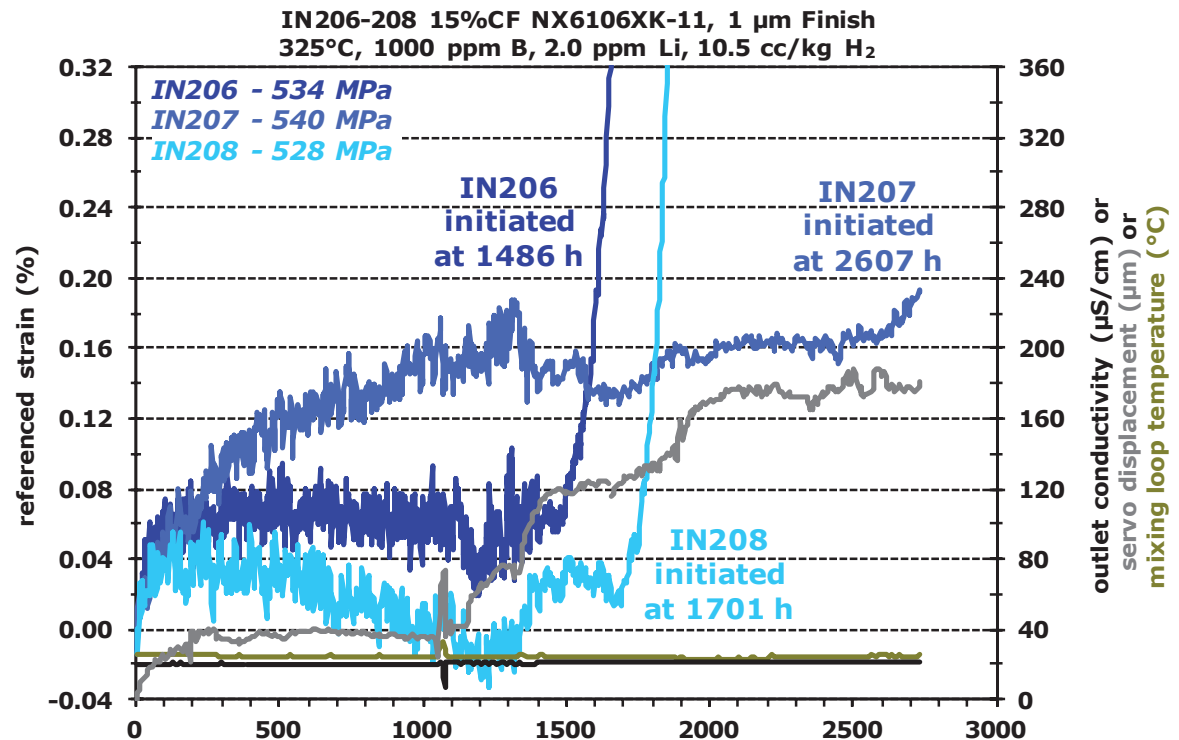
Data as of June 2017

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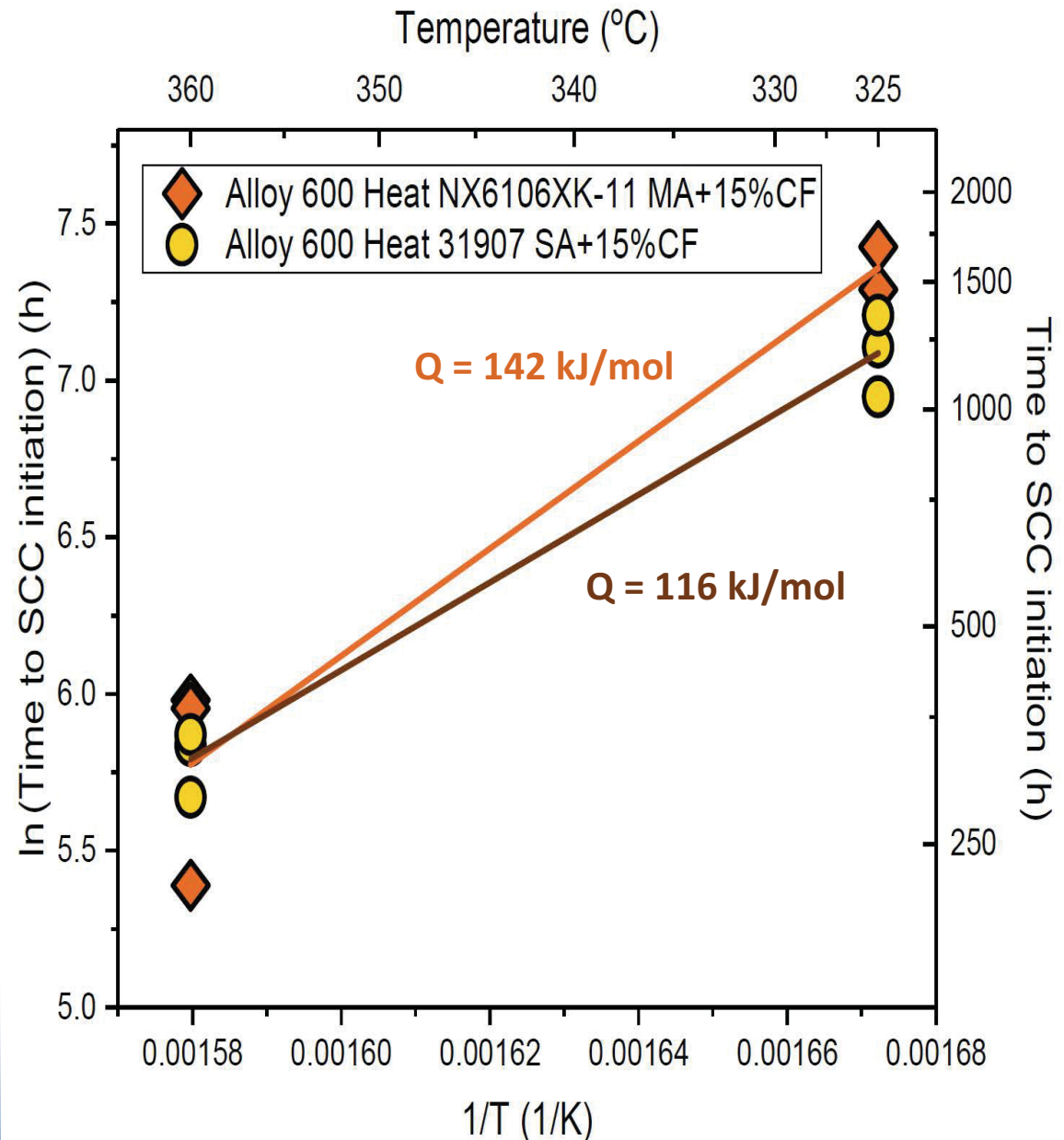
SCC Initiation Response at 325°C

- 15%CF PNNL MA plate initiated at 1486, 1701 and 2607 hours.
- 15%CF EPRI/GE SA plate initiated at 1080, 1240 and 1350 hours.
- General response is consistent with the 360°C behavior with EPRI/GE heat more susceptible.
- SCC initiation times at 325°C are ~4X longer than measured at 360°C.



Estimated Thermal Activation Energy of SCC Initiation Determined for Cold-Worked Alloy 600

- SCC initiation tests performed in 325°C and 360°C simulated PWR primary water at the Ni/NiO stability ECP.
- Measured SCC initiation times reveal estimated activation energies of ~116 kJ/mol for the 15%CF EPRI/GE SA600 plate and 142 kJ/mol for the 15%CF PNNL MA600 plate materials. Reasonably consistent with prior reported values by KAPL.



Update on Alloy 690 SCC Initiation Research at PNNL



***Ziqing Zhai, Mychailo Toloczko,
Karen Kruska and Steve Bruemmer
Pacific Northwest National Laboratory***

*Alloy 690/52/152 Research
Collaboration Meeting*

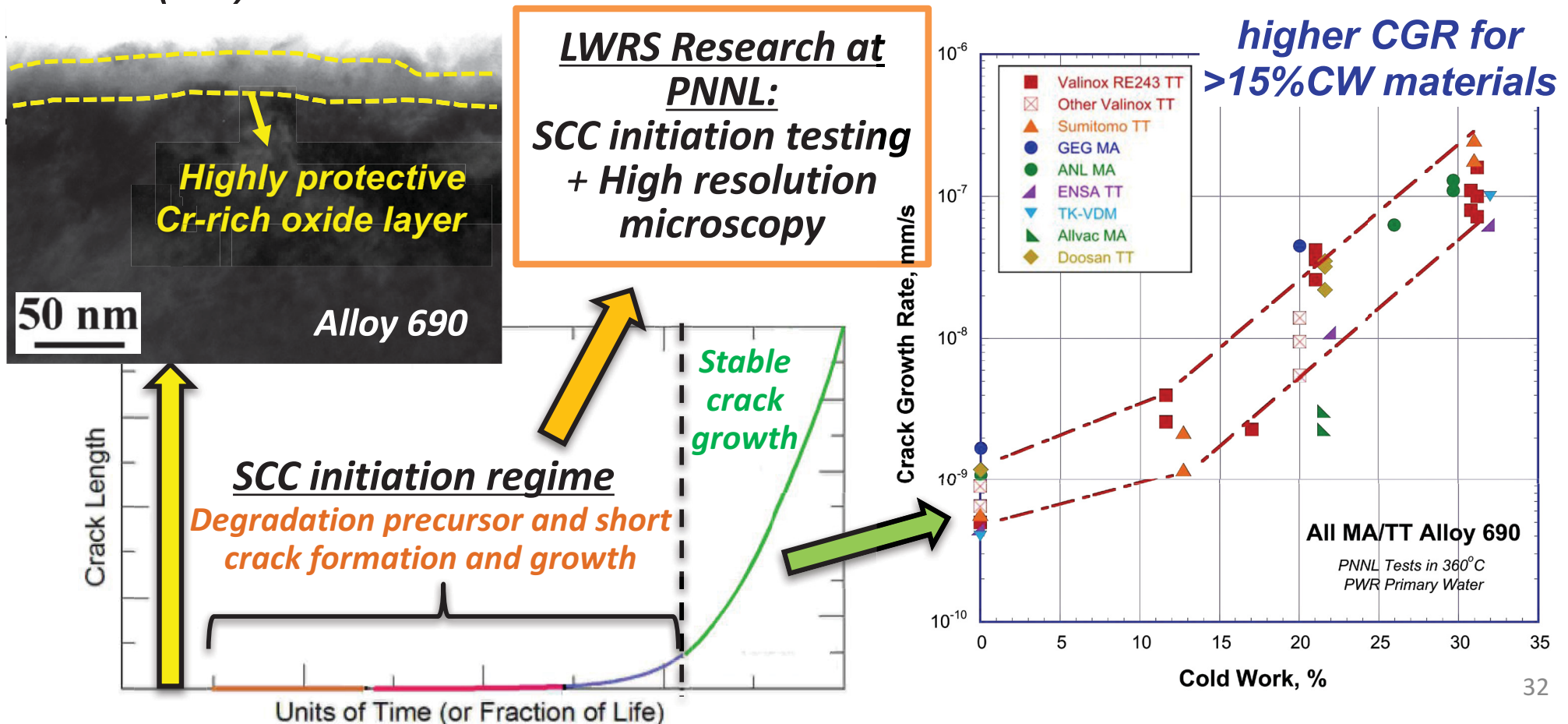
*Tampa, FL, USA
November 28-30, 2017*

Light Water Reactor Sustainability R&D Program



Background

- **Ni-base alloy 690 (Ni-30Cr-10Fe):**
 - Important structural material for pressurized water reactor (PWR) pressure boundary components.
 - Considered highly resistant to stress corrosion cracking (SCC), but high SCC crack growth rate observed at moderate-to-high cold worked (CW) conditions.



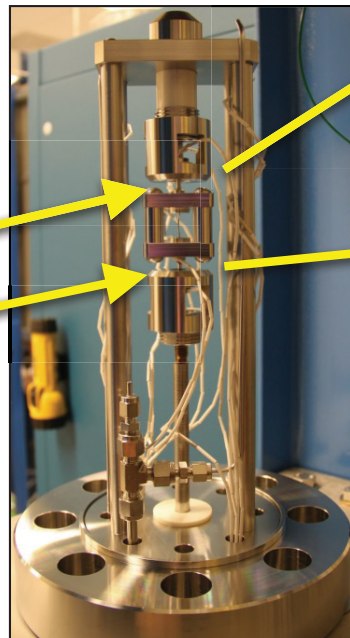
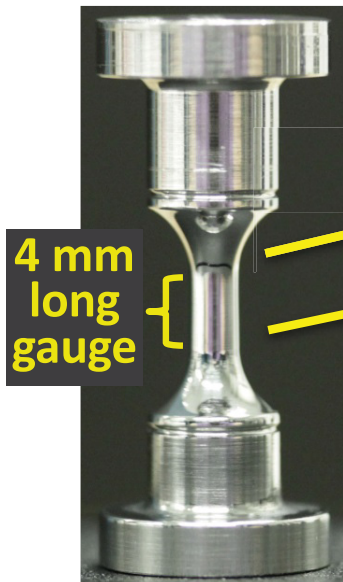
SCC Initiation Testing at PNNL

SCC initiation test systems with active loading and in-situ DCPD crack-detection:

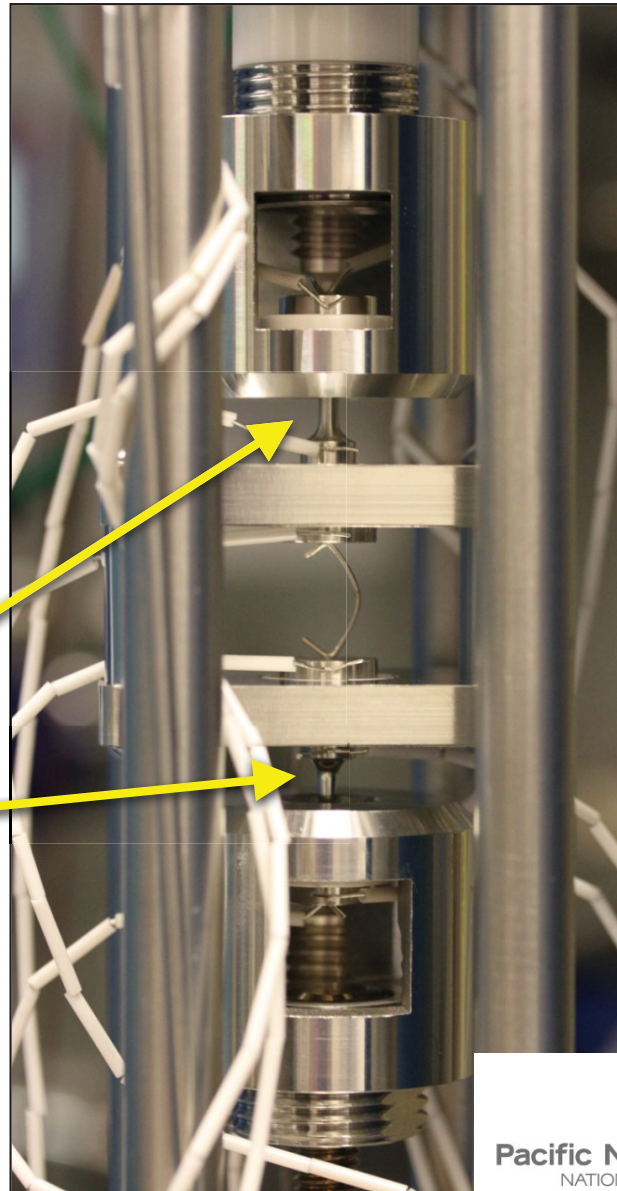
- ▶ **LWRS:** two smaller systems recently converted to test 6 fully instrumented specimens + one 36-specimen system with up to 12 specimens instrumented.
- ▶ **NRC/EPRI:** two 36-specimen systems with 24 instrumented.
- ▶ **Other DOE:** three smaller systems to test 3 fully instrumented specimens.

1.2" Tall SCC
Initiation Specimen

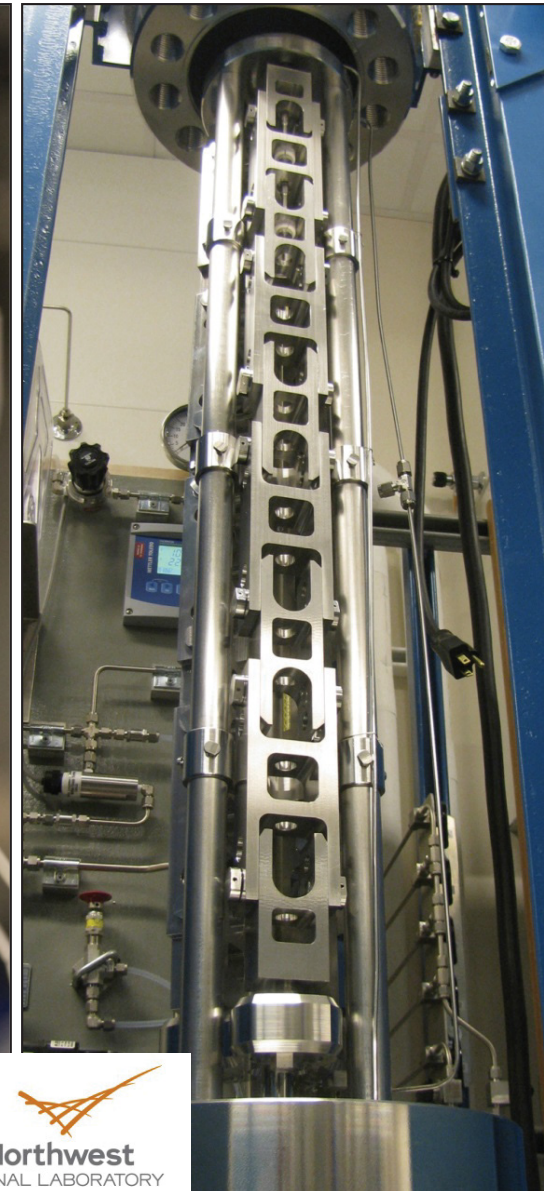
Small SCC
Initiation System



Small SCC
Initiation System

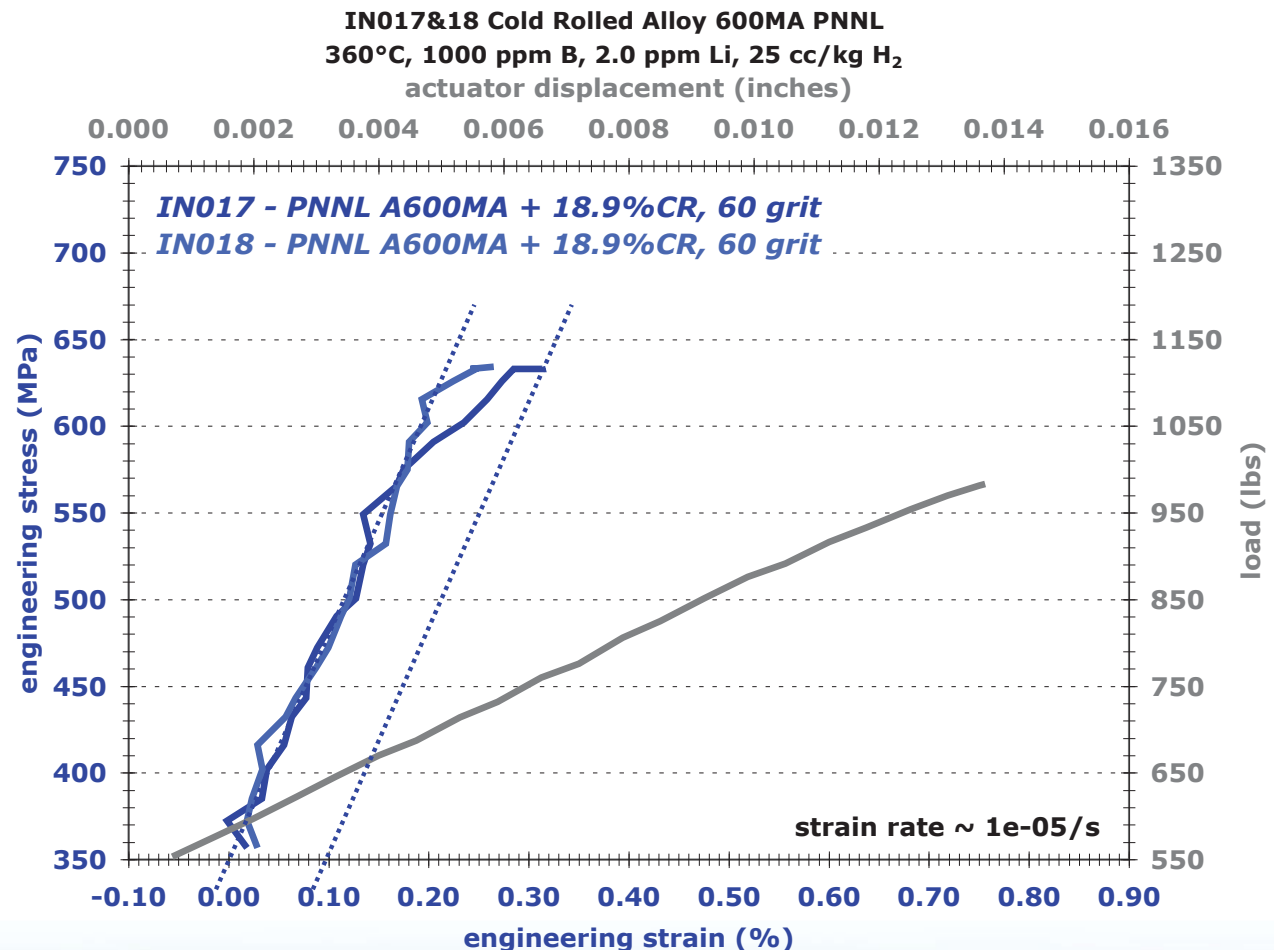


36-Specimen SCC
Initiation System



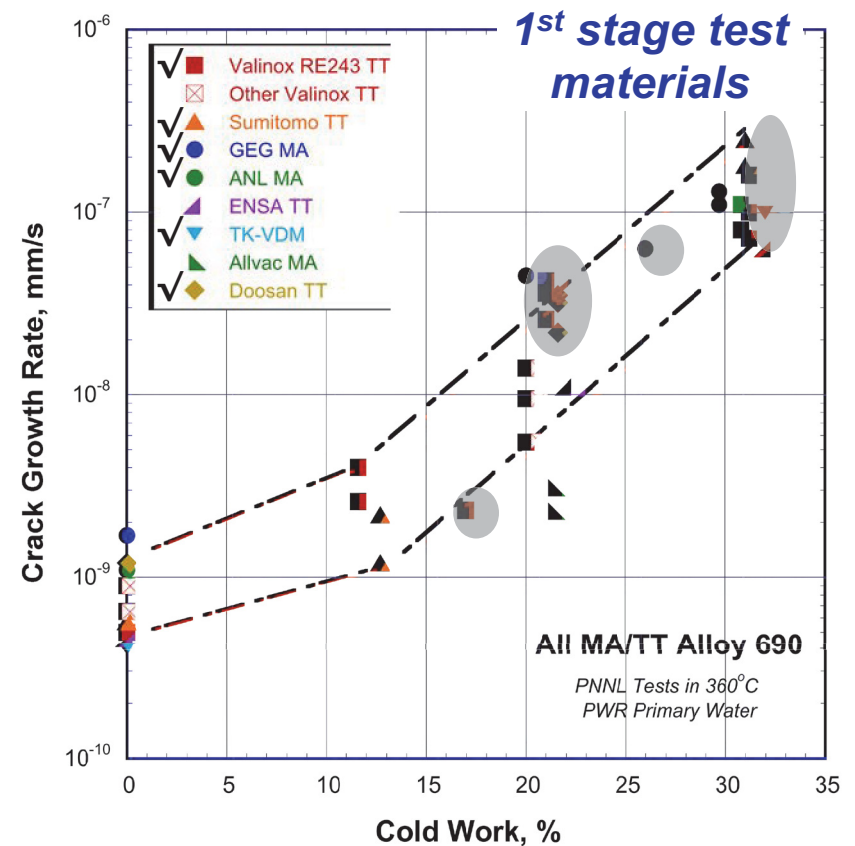
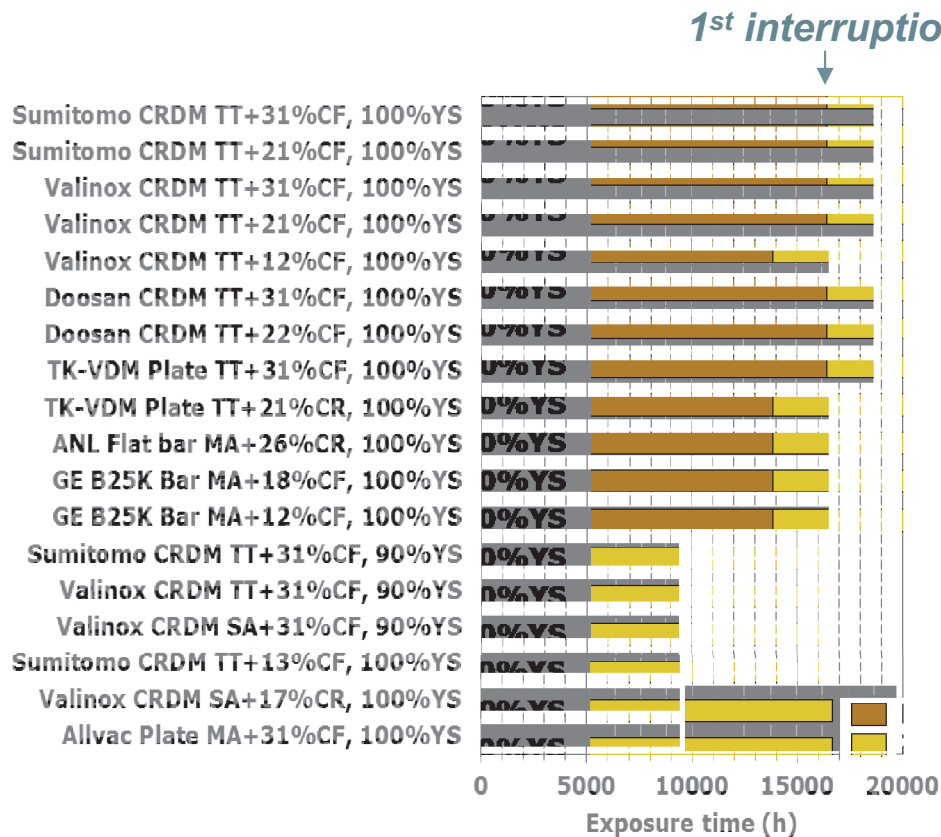
Typical SCC Initiation Test Conditions

- *Simulated PWR primary water – 1000 ppm B, 2 ppm Li, 360°C, 25 cc/kg H₂ (Ni/NiO stability line).*
- *All specimens loaded to their yield strength.*
- Yield observed by monitoring stress (from load) versus strain (from DCPD) at a displacement rate of $\sim 1 \times 10^{-5} \text{ s}^{-1}$ ($\sim 1 \text{ h}$ to load).
- For this multi-specimen test, only one specimen underwent $\sim 2\%$ plastic strain while the other specimens exhibit no obvious plastic strain.



SCC Initiation Test Status

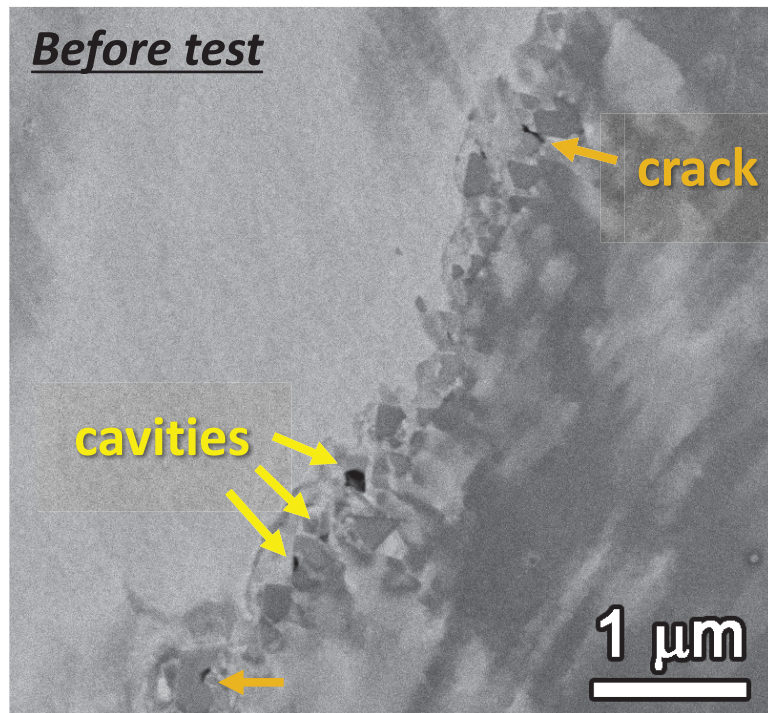
- 1st stage test: six alloy 690 CRDM TT and plate MA/TT heats with cold work levels of ~12%, 21% and ~31% reduction at yield stress.
- 2nd stage test: above conditions & a few more combinations of carbide microstructure (TT vs. SA), applied stress (100% vs. 90%YS), and heat IGSCC susceptibility (high vs. medium).
- SCC CGR data available for every material + cold work combination.



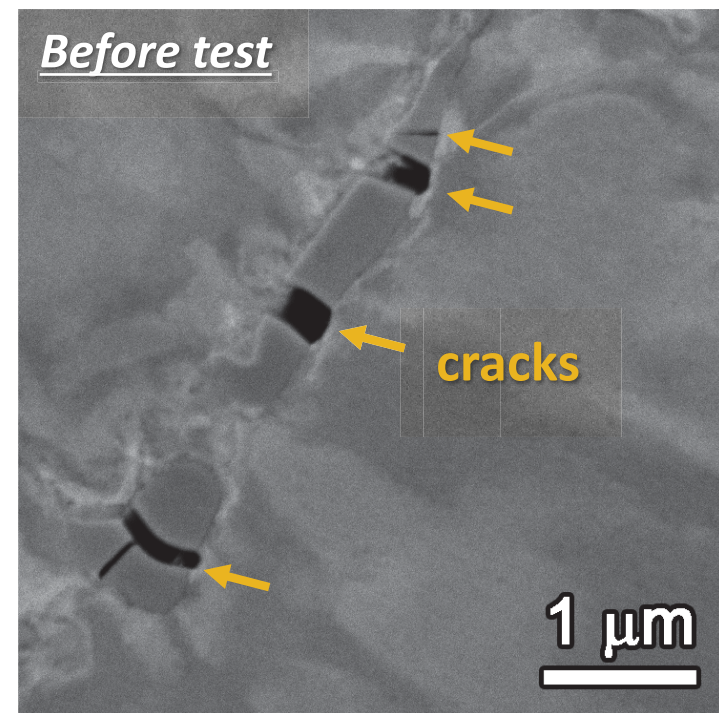
Cold-Worked Alloy 690 Microstructures

- Six alloy 690 CRDM TT and plate MA/TT heats with cold work levels of ~12%, 21% and ~31% reduction in 1st stage test materials.
- SCC CGR data available for every material + cold work combination. All highly cold-worked materials exhibited IGSCC susceptibility.

Valinox CRDM TT + 31%CF



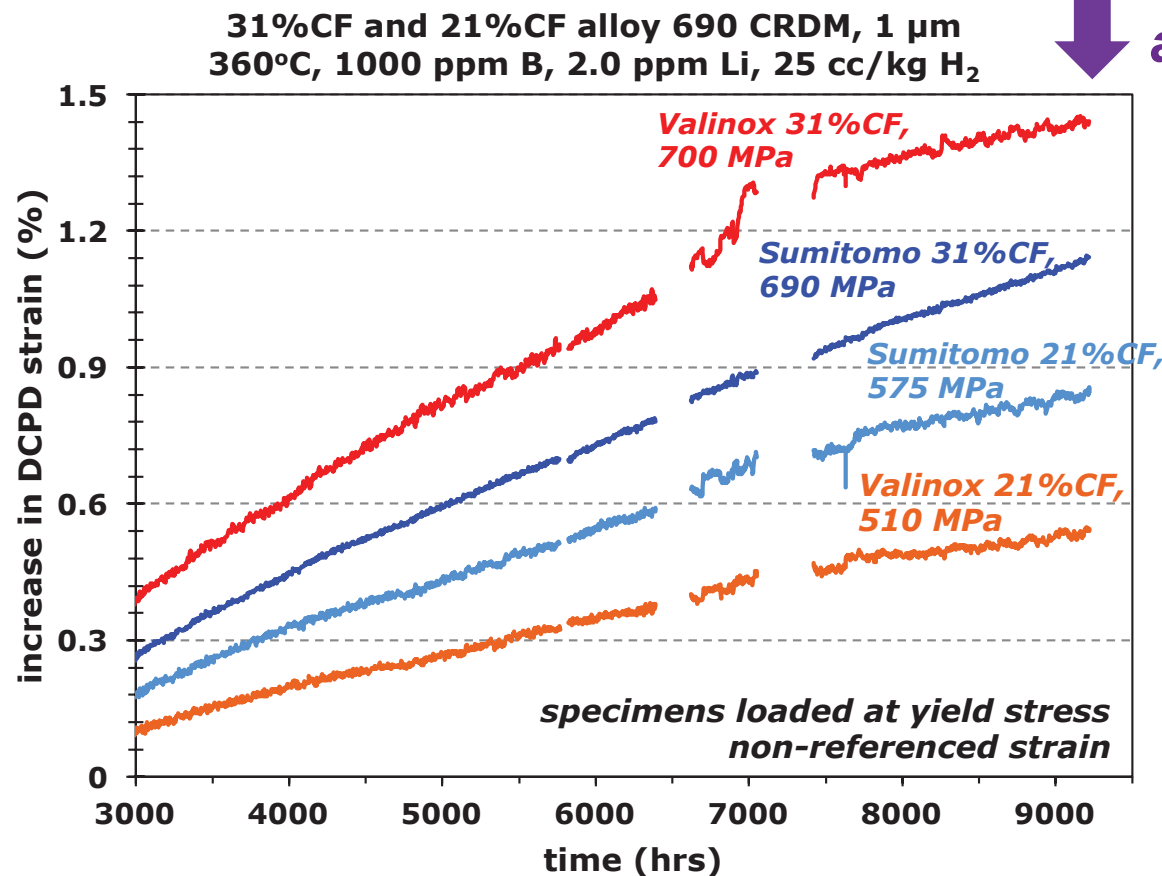
Doosan CRDM TT + 31%CF



Size and density of damage produced by cold work strongly depends on GB carbide distribution.

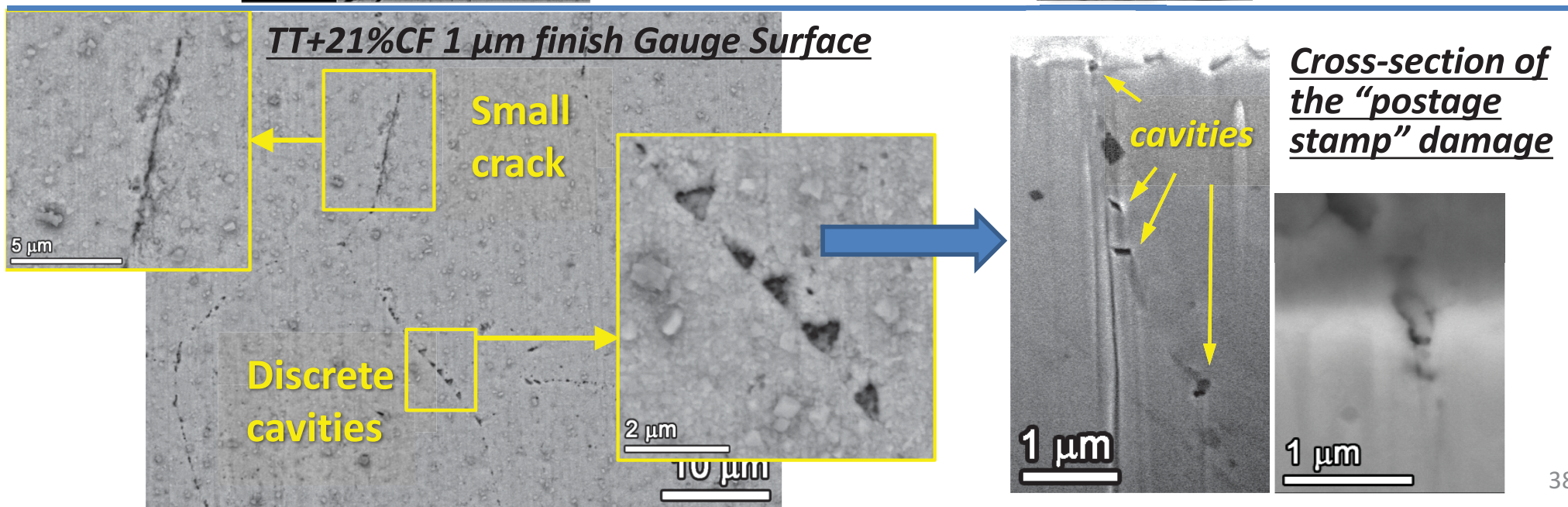
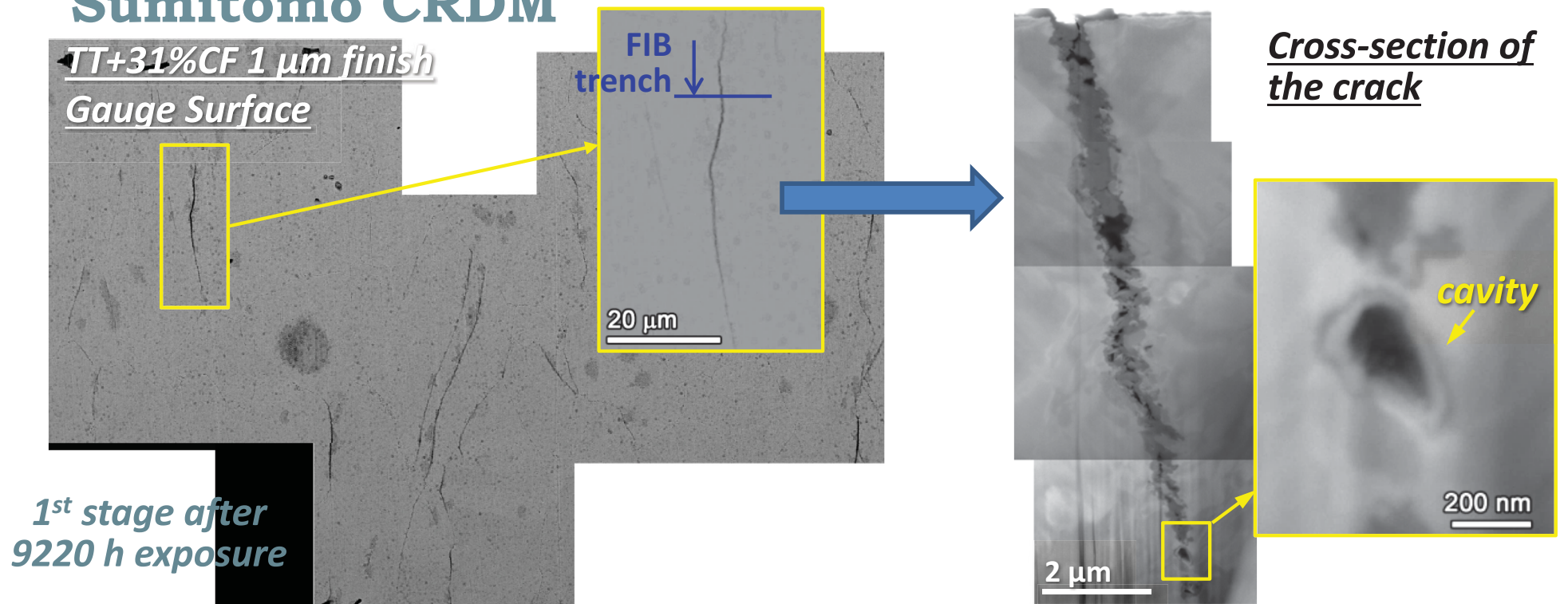
1st Stage Test: DCPD Responses

- ▶ Decreasing strain rate measured by DCPD with test time indicating no crack initiation.
- ▶ Test was stopped at ~9220 h of exposure to examine the gauge sections for precursor damage.



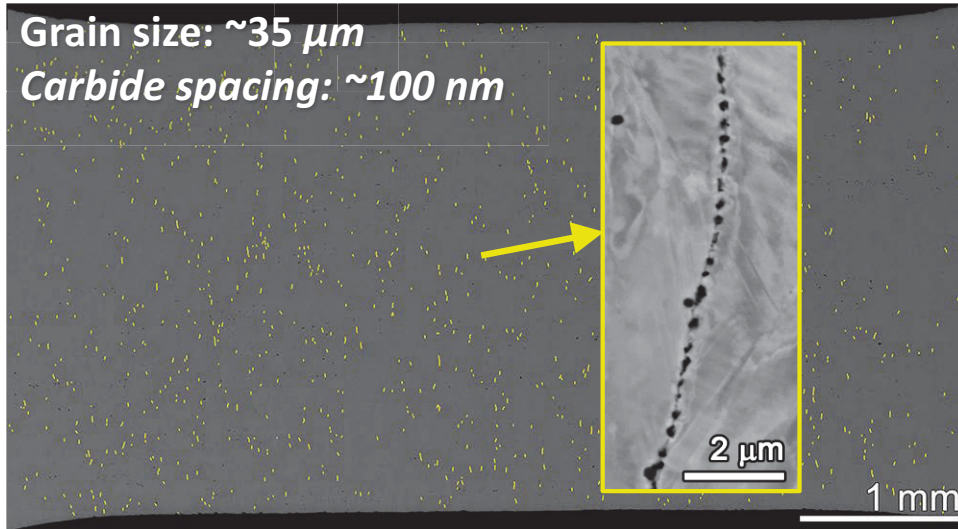
Test interruption
at 9220 h

1st Stage Test: Surface Damage Morphology – Sumitomo CRDM

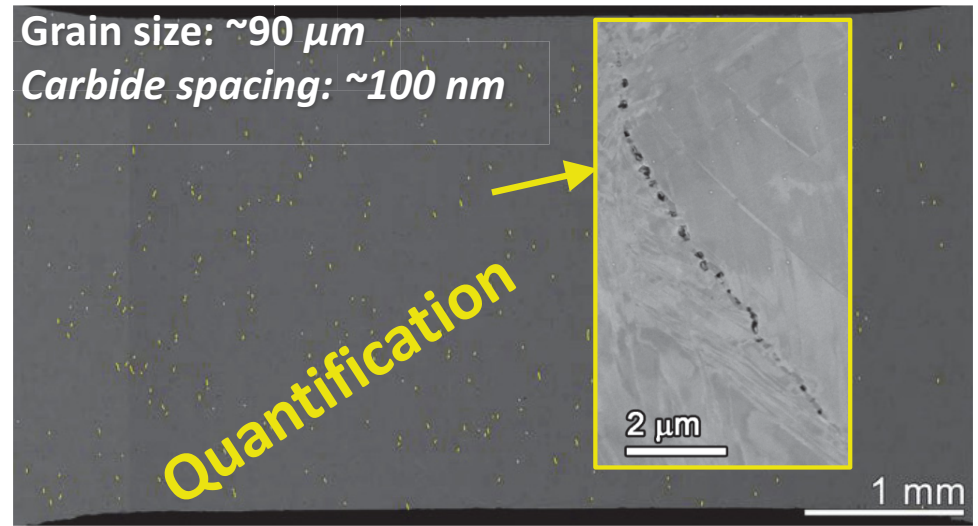


1st Stage Test: Gauge Cross-Section Morphology - Different GB Cavity Distribution in Bulk

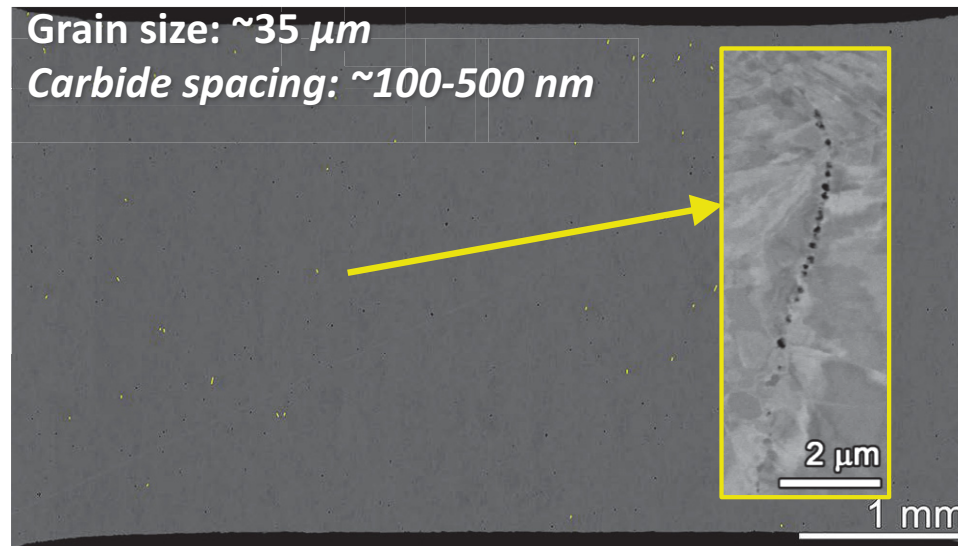
TT + 31%CF Sumitomo CRDM, 675 MPa (IN034)



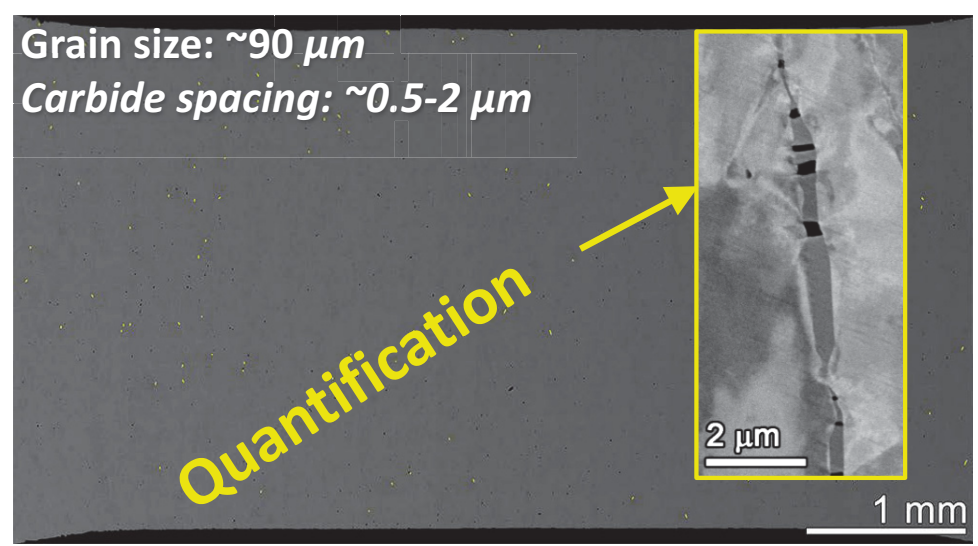
TT + 31%CF Valiniox CRDM, 690 MPa (IN038)



TT + 31.9%CF TK-VDM Plate, 675 MPa (IN044)

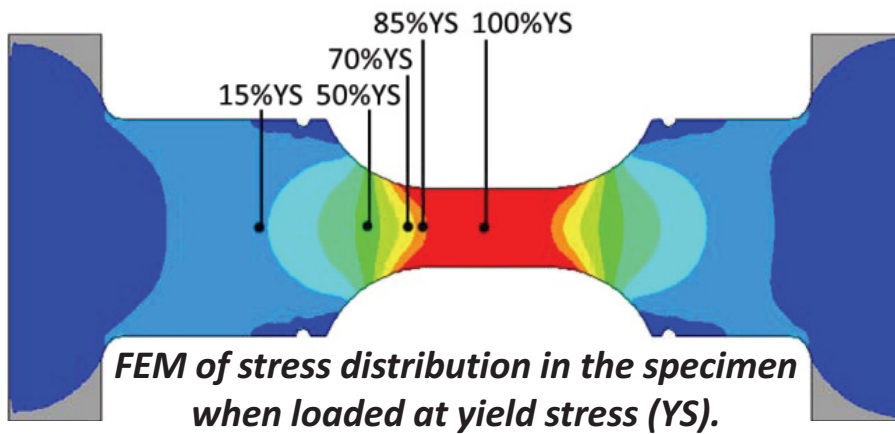


TT + 31%CF Doosan CRDM, 655 MPa (IN041)



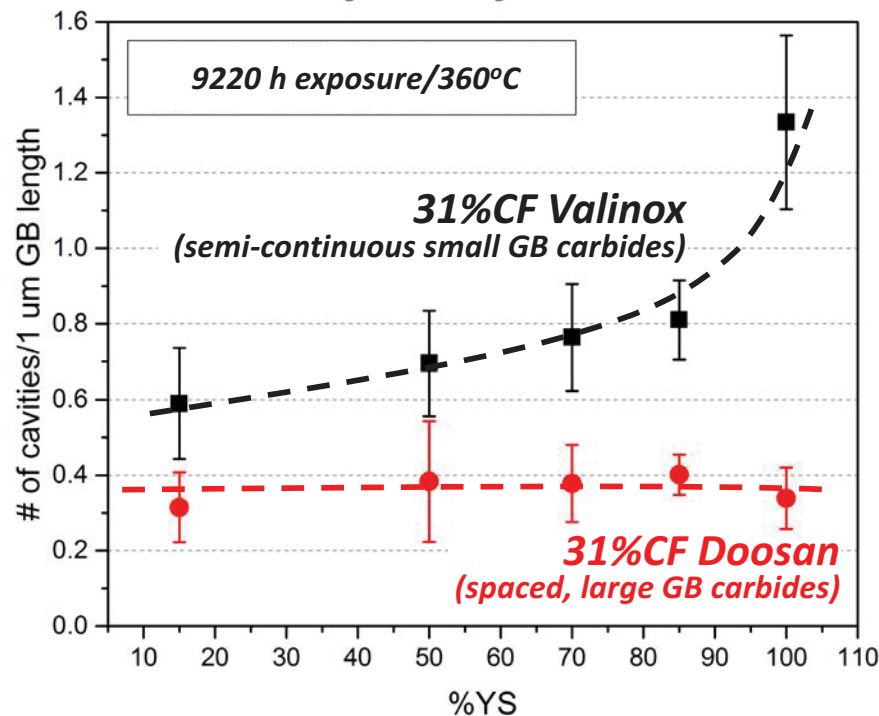
All four specimens tested in 360°C water at YS for 9220 hours, and all with ground “C” finish

Quantification of GB Cavity Distribution in 31%CF Valinox & Doosan CRDM Specimens

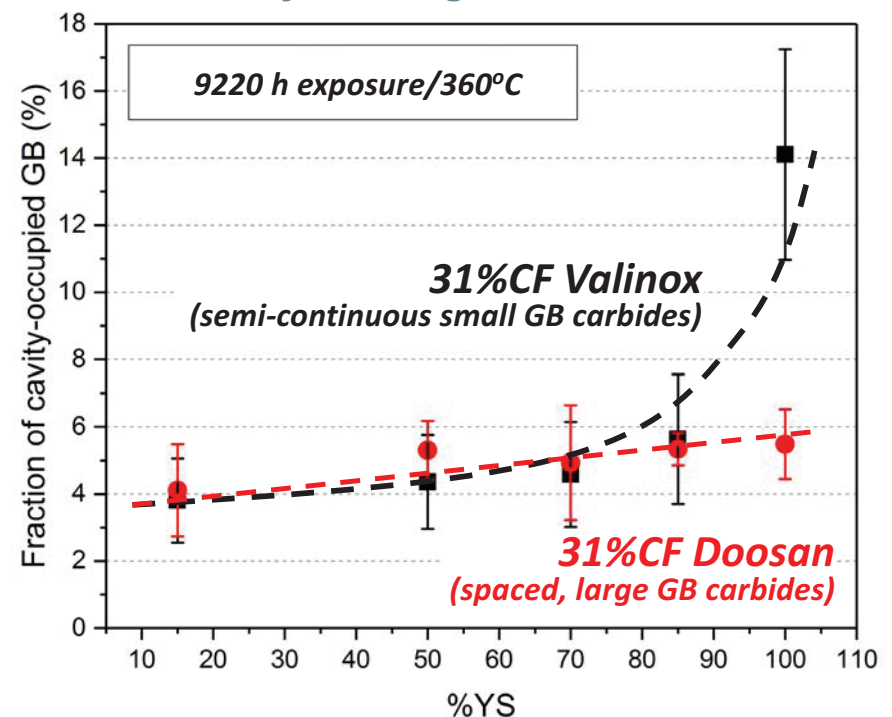


- **Approach:**
 - Analyses done at specimen regions where the applied stress equals 15% to 100% of the yield stress.
 - Five to six 150 μm -long GBs analyzed at each region.
 - ImageJ used for image processing and measurements.
- **Two parameters quantified for each GB:**
 - Cavity density = $\frac{\sum \# \text{ of IG cavities}}{\text{GB length}}$
 - Cavity coverage at GB = $\frac{\sum h \text{ of IG cavities}}{\text{GB length}} \%$

Cavity density vs. σ

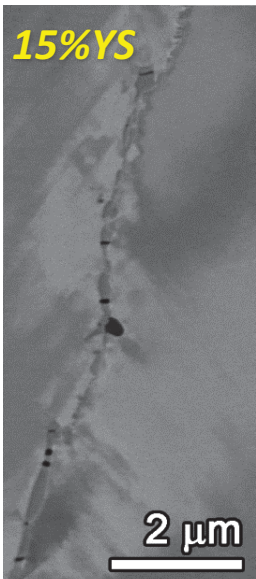
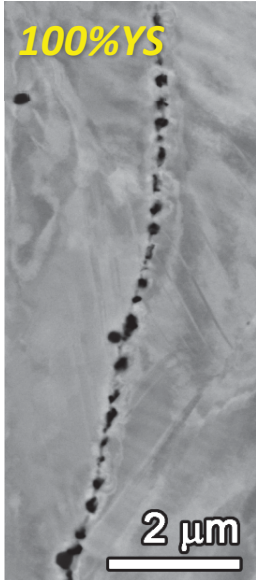


Cavity coverage at GB vs. σ

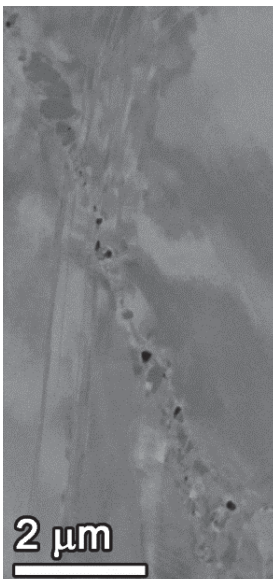
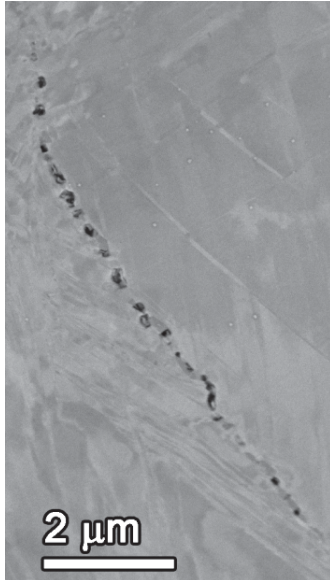


Cross-Section Cavity Morphology Comparison – Gauge vs. Shoulder in Highly CW Specimens

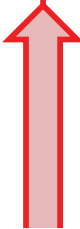
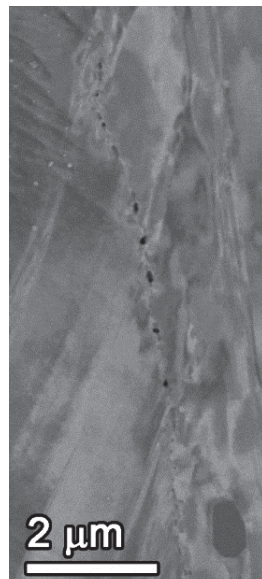
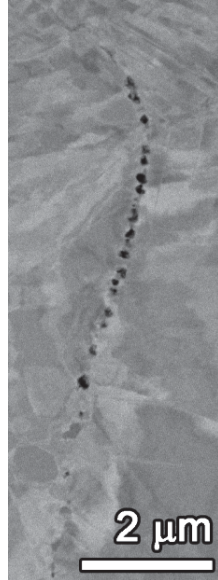
IN034 (Sumitomo
31%CF, C finish)



IN038 (Valinox
31%CF, C finish)



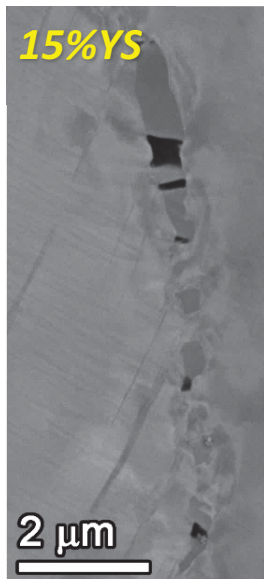
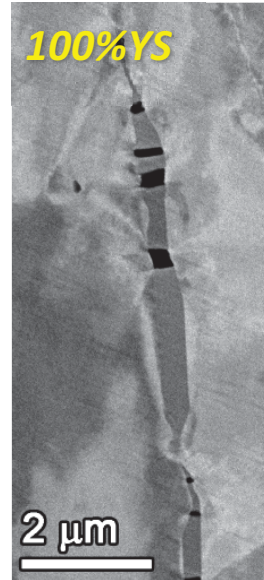
IN044 (TK-VDM
32%CF, C finish)



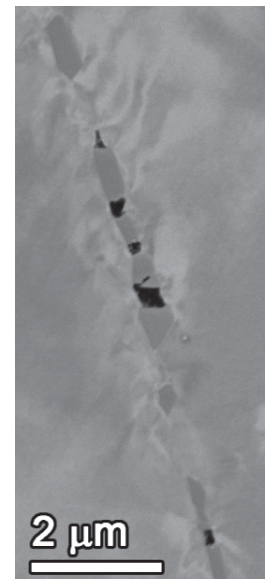
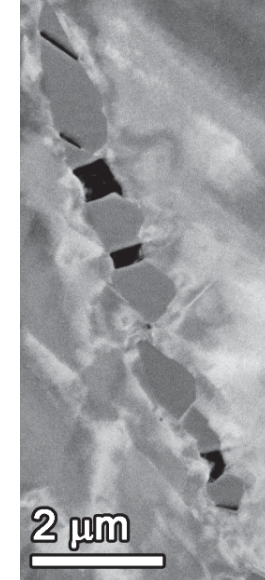
*Obvious
increase
in cavity
size and
density
from
shoulder
to gauge*



IN041 (Doosan
31%CF, C finish)



IN055 (ANL
26%CR, C finish)



High-
Stress
Gauge

*No
apparent
increase
in cavity
density*

Low-
Stress
Shoulder

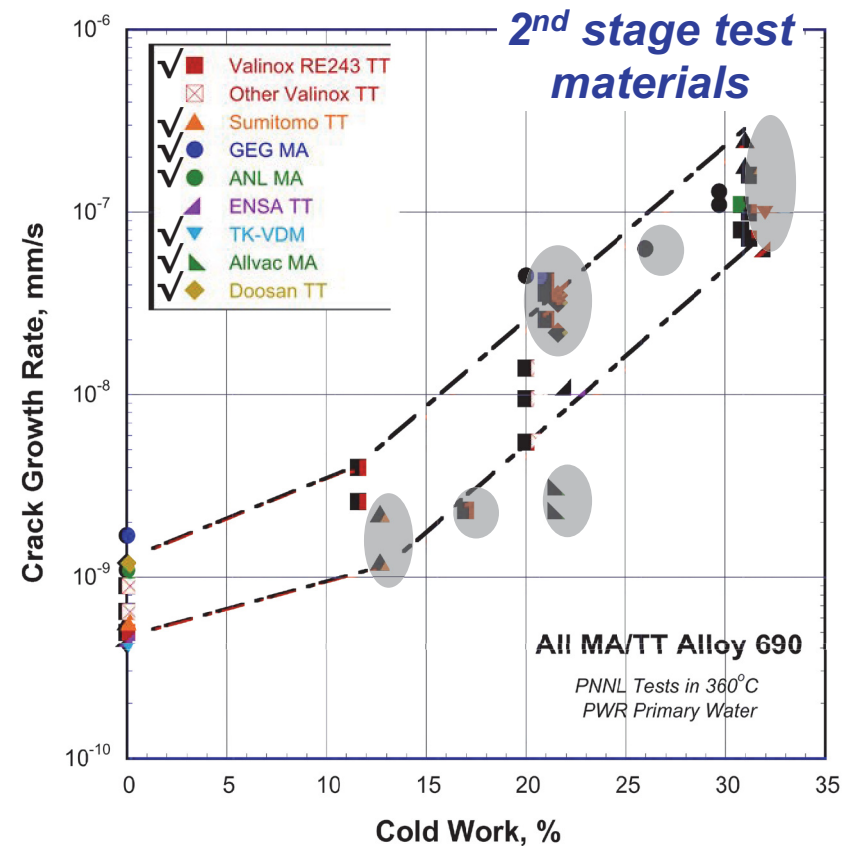
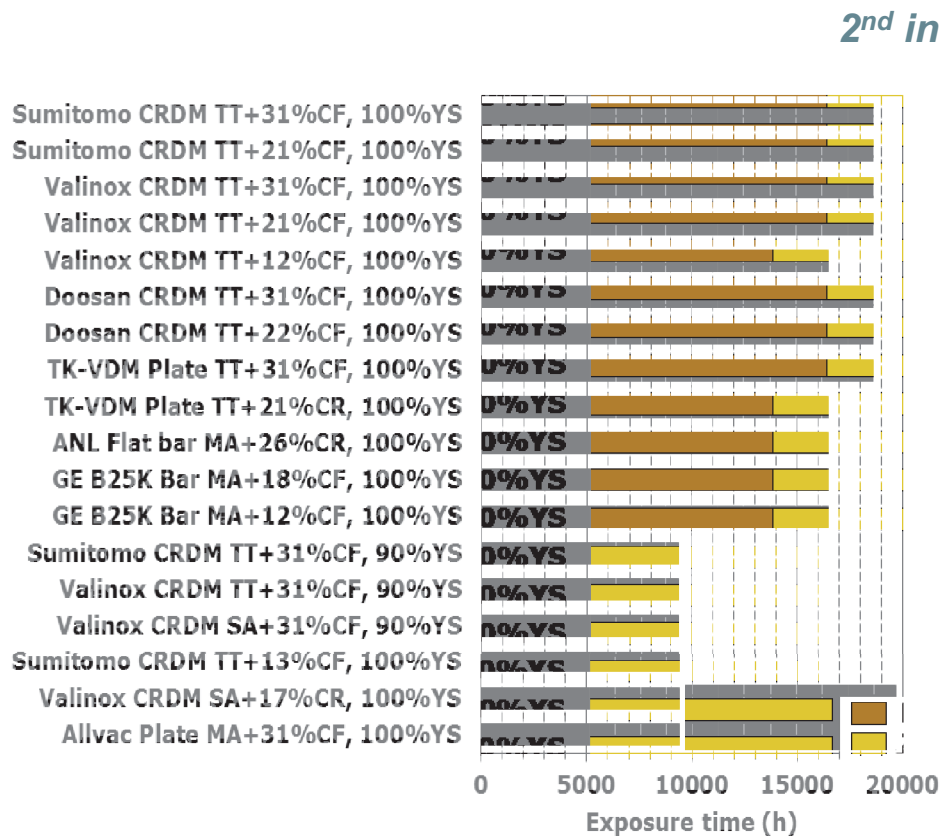
1st Stage Test (to 7110 or 9220 h): Summary of Alloy 690 Constant Load Test Results

Material, Condition	σ (MPa)	GB carbide distribution		SEM Observation on <i>Ground</i> Specimens		SEM Observation on <i>Polished</i> Specimens
		Spacing	Size	IG cavities aggregations <i>in bulk</i>	New GB cavities <i>in bulk</i>	IG cracks <i>on surface</i>
Sumitomo CRDM, TT+31%CF	675	~100 nm	Small	Many	Many	Many
Valinox CRDM, TT+31%CF	690	~100 nm	Small	Some	Some	Some
TK-VDM Plate, TT+32%CF	675	~100–500 nm	Medium	Few	Some	Very Few
Doosan CRDM, TT+31%CF	655	~0.5–2 μm	Large	Very Few	Very Few	Very Few
ANL Flat Bar, MA+26%CR	775	~0.2–2 μm	Large	None	Very Few	Few TG cracks
Sumitomo CRDM, TT+21%CF	590	~100 nm	Small	Few	Very Few	Very Few
Valinox CRDM, TT+21%CF	525	~100 nm	Small	None	None	None
Doosan CRDM, TT+21%CF	555	~0.5–2 μm	Large	None	None	None
TK-VDM Plate, TT+21%CR	655	~100–500 nm	Medium	None	None	None
GE B25K Bar, MA+18%CF	550	Few IG carbides		None	None	None
Valinox CRDM, TT+12%CF	365	~100 nm	Small	None	None	None
GE B25K Bar, MA+12%CF	510	Few IG carbides		None	None	None

Three highly CW alloy 690TT heats (Sumitomo, Valinox and TK-VDM) with nearly continuous distribution of small GB carbides exhibit formation of many new, closely spaced IG cavities that can lead to internal creep cracks. New IG cavities do not form in the Doosan and ANL heats with larger, well-spaced GB carbides.

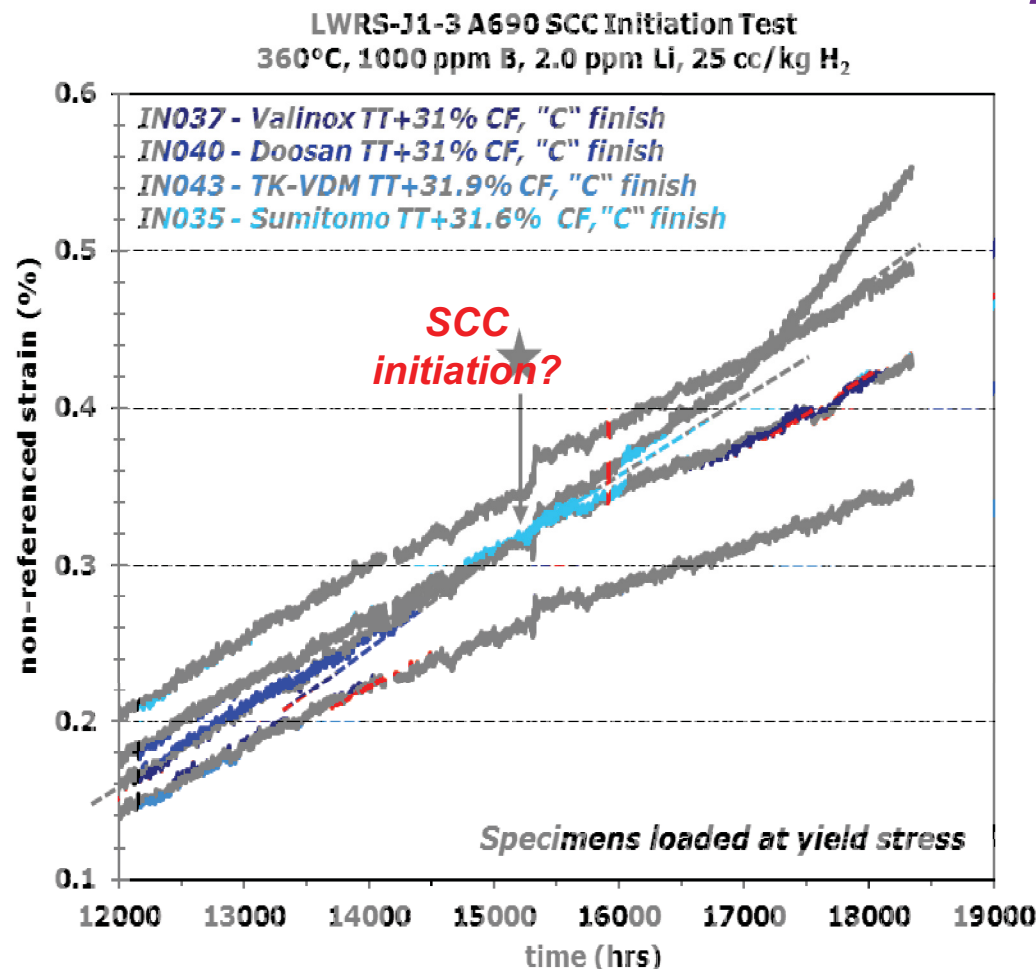
SCC Initiation Test Status

- 1st stage test: six alloy 690 CRDM TT and plate MA/TT heats with cold work levels of ~12%, 21% and ~31% reduction at yield stress.
- 2nd stage test: above materials & a few more combinations of carbide microstructure (TT vs. SA), applied stress (100% vs. 90%YS), and heat IGSCC susceptibility (high vs. medium).
- SCC CGR data available for every material + cold work combination.



2nd stage test: DCPD Responses

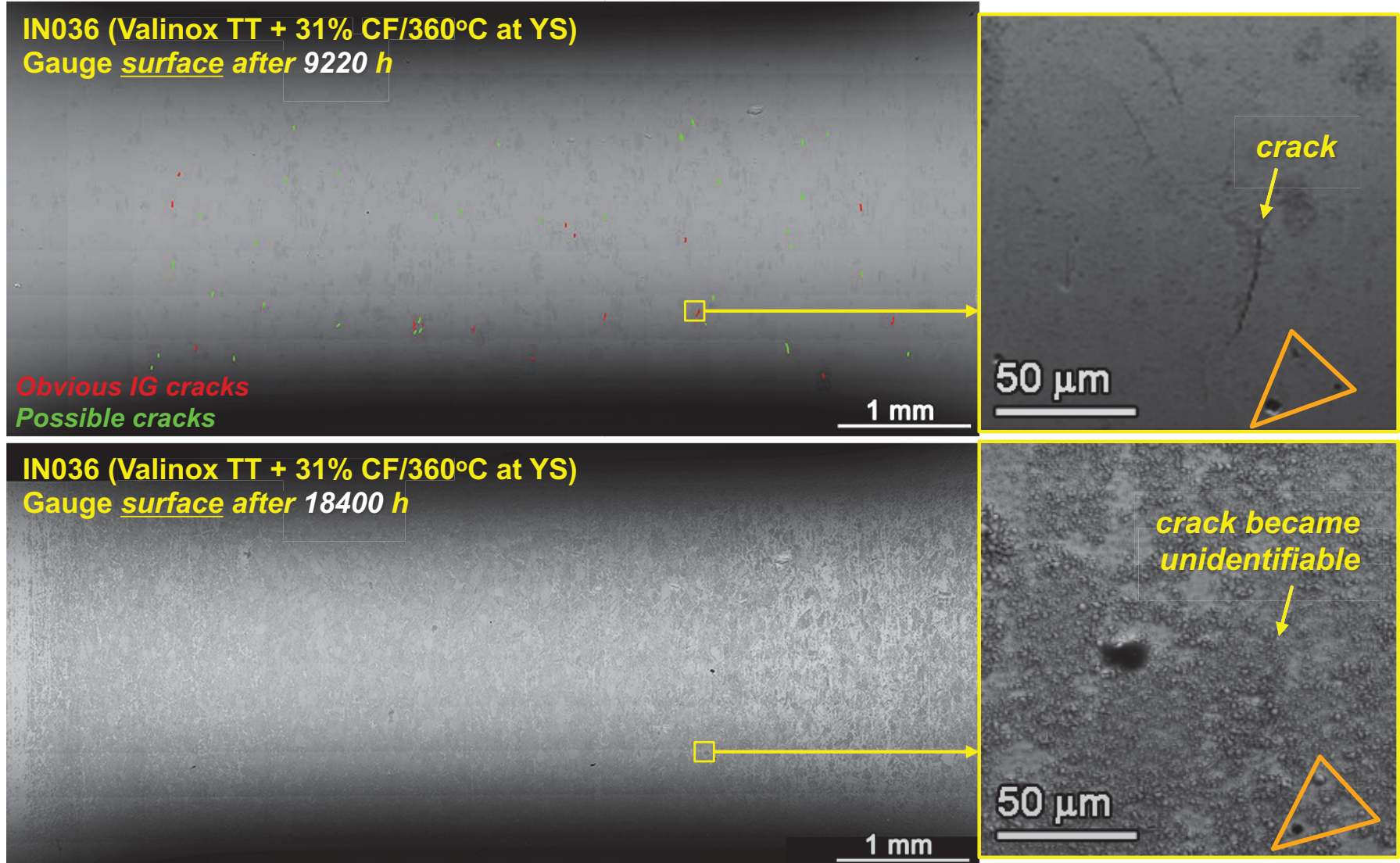
- ▶ Decreasing strain rate measured by DCPD with test time for most specimens except IN037 (Valinox CRDM TT+31%CF, “C” finish at YS).
- ▶ Test was stopped at ~18400 h of exposure to examine the gauge sections for precursor damage.



2nd test interruption
at 18400 h

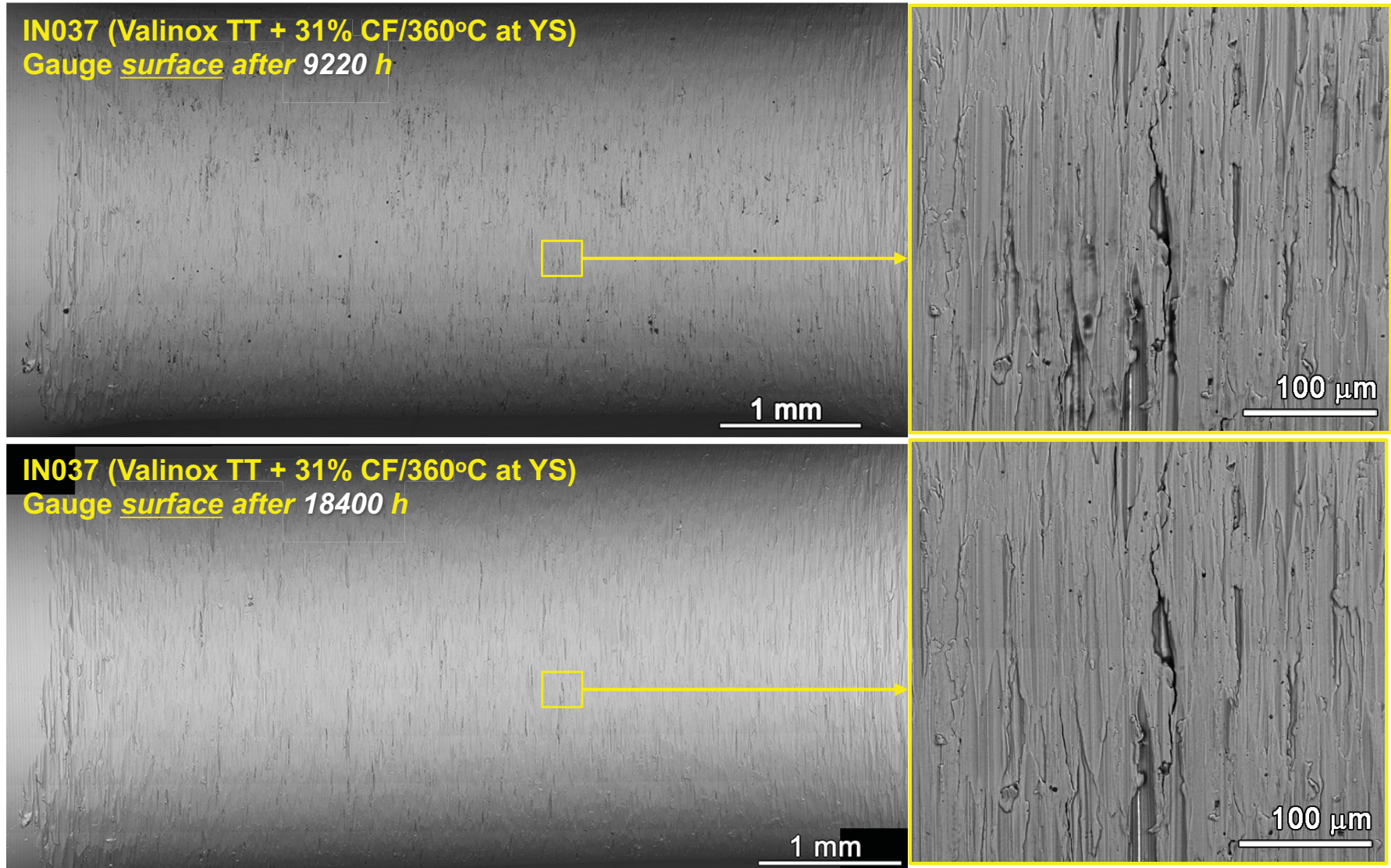
Highly CW Materials: Gauge Surface Morphology

Specimens with $1\text{ }\mu\text{m}$ finish: spinel-type oxides formed extensively on the surface, obscuring the identification of cracks even including previously obvious cracks.



Highly CW Materials: Gauge Surface Morphology

Specimens with **ground “C” finish**: rough surface resulted by grinding prevented a clear view of cracks on the surface. No obvious cracks were found.

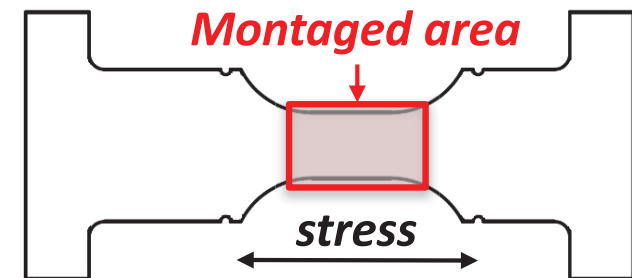


Specimens Selected for Cross-Section Examination (11/2017)

<i>Spec ID</i>	<i>Material & Condition</i>	<i>Surface</i>	<i>σ (MPa)</i>	<i>Exposure time (h)</i>
IN035	Sumitomo CRDM, TT+31% CF	Ground C	690 (100%YS)	18400
IN036	Valinox CRDM, TT+31% CF	Ground C	700 (100%YS)	18400
IN040	Doosan CRDM, TT+31% CF	Ground C	655 (100%YS)	18400
IN043	TK-VDM Plate, TT+31.9% CF	Ground C	675 (100%YS)	18400
IN025	Sumitomo CRDM, TT+21% CF	Ground C	590 (100%YS)	18400
IN028	Valinox CRDM, TT+21% CF	Ground C	525 (100%YS)	18400
IN031	Doosan CRDM, TT+21.6% CF	Ground C	555 (100%YS)	18400

- Preliminary SEM examination completed on selected specimens with ground surface finish.*
- The highly polished specimens from the material + CW conditions listed above are kept for further analysis/testing.*

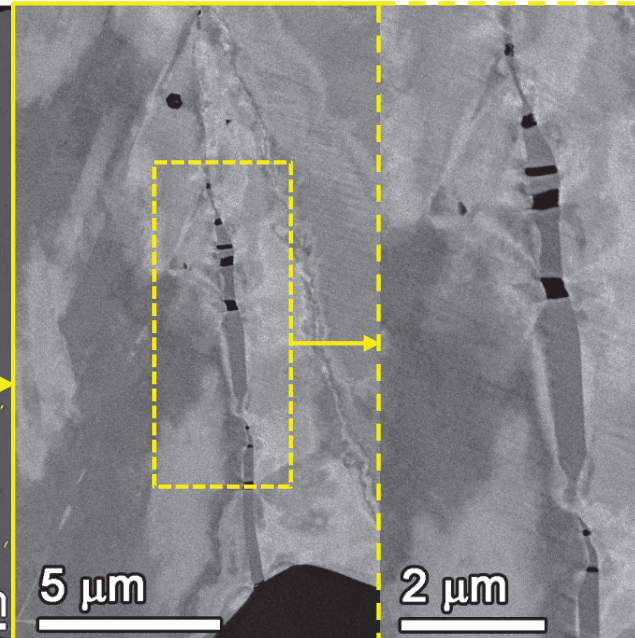
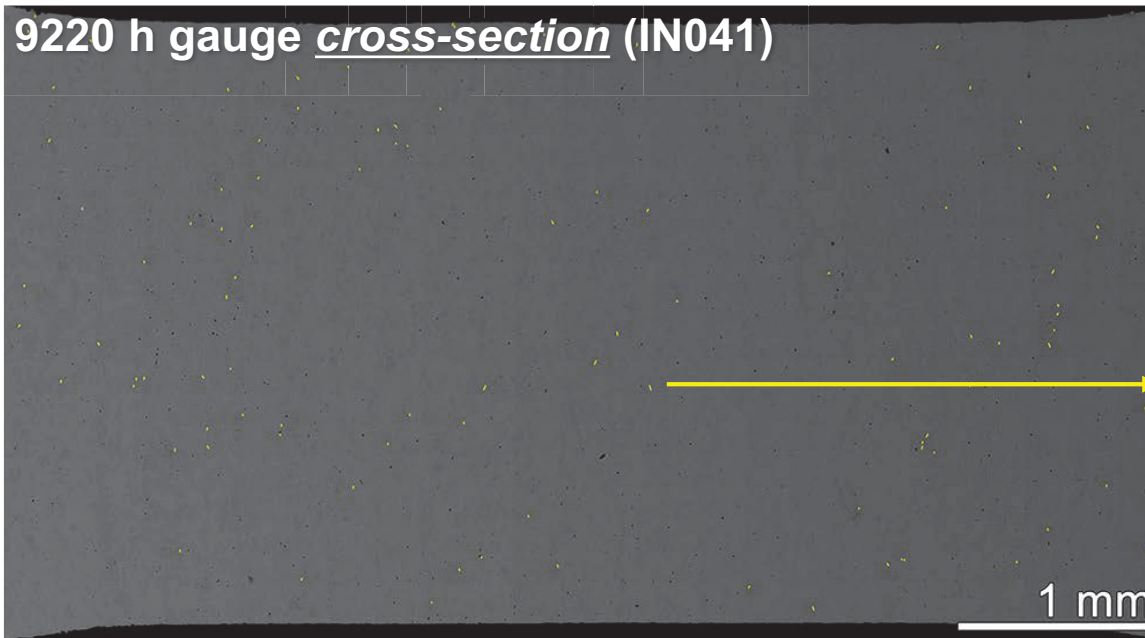
Cross-section of CLT specimen



Highly CW Materials: Cross-Section Morphology

Doosan CRDM TT+31%CF, 360°C at YS (~655 MPa), “C” finish

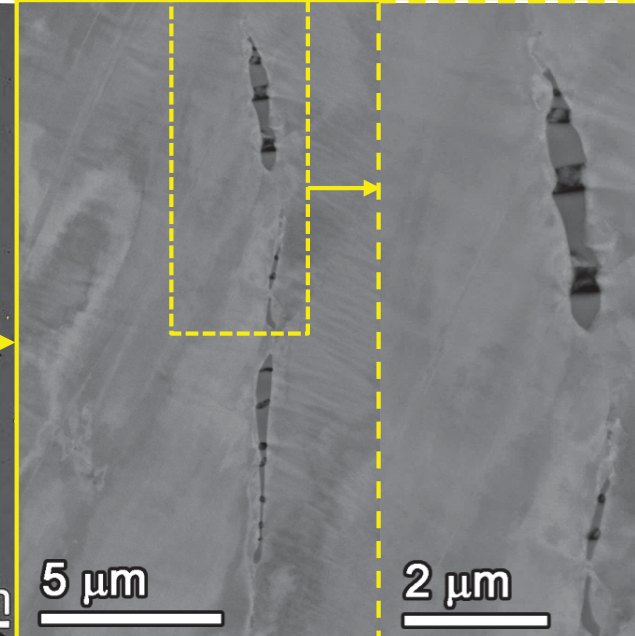
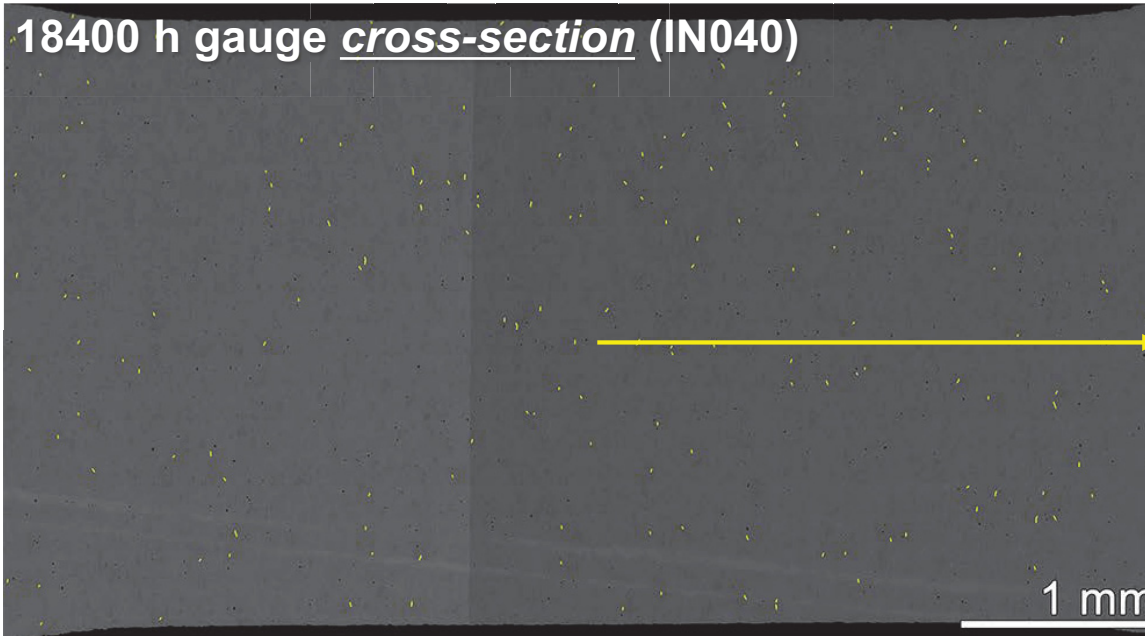
9220 h gauge cross-section (IN041)



stress

Slight increase in cavity size leading to higher visibility in the montage map, but no obvious change in cavity density and spacing

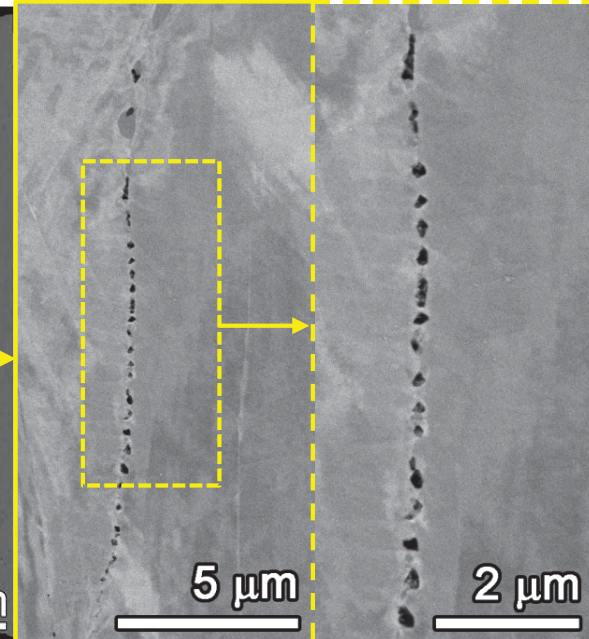
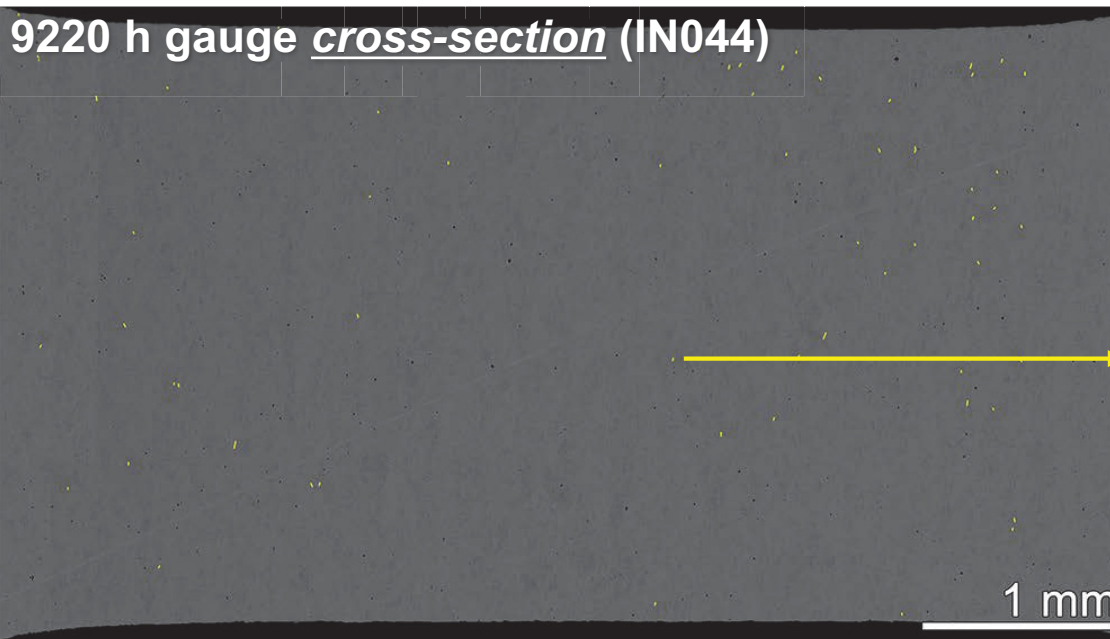
18400 h gauge cross-section (IN040)



Highly CW Materials: Cross-Section Morphology

TK-VDM Plate TT+31%CF, 360°C at YS (~675 MPa), “C” finish

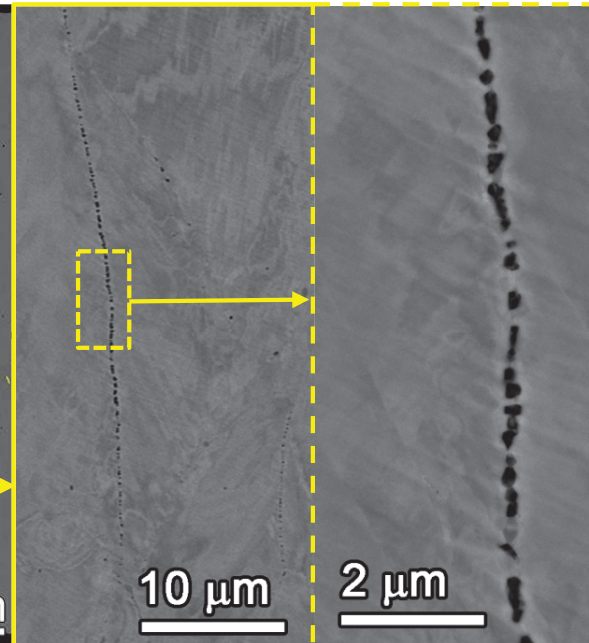
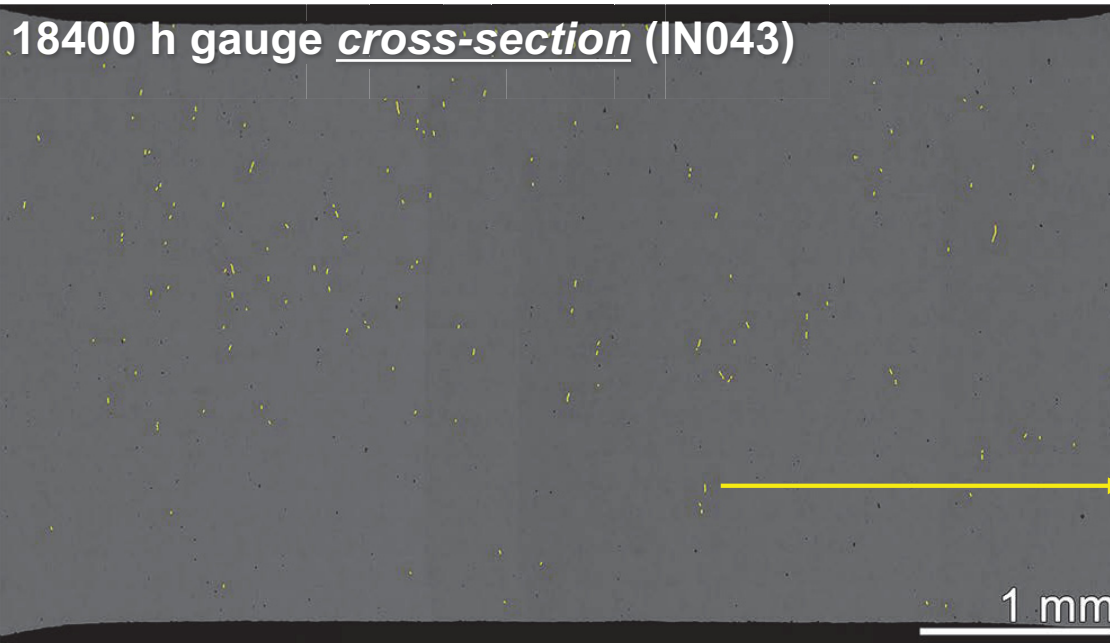
9220 h gauge cross-section (IN044)



stress
↔

*Relatively
short
aggregations
of discrete
GB cavities*

18400 h gauge cross-section (IN043)

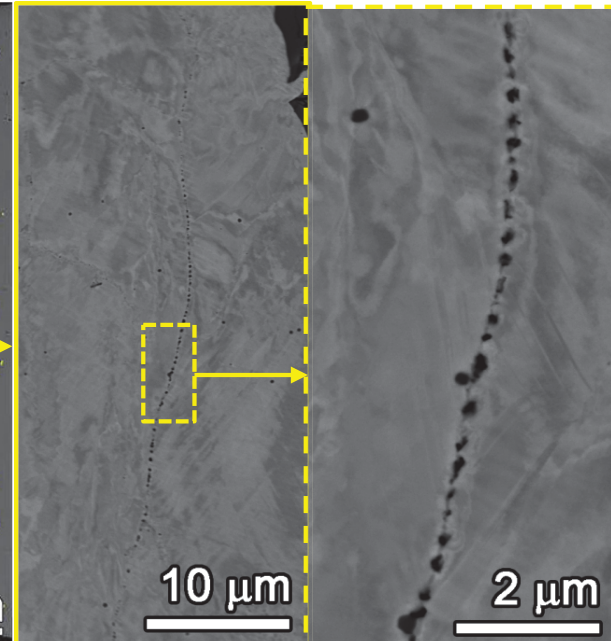
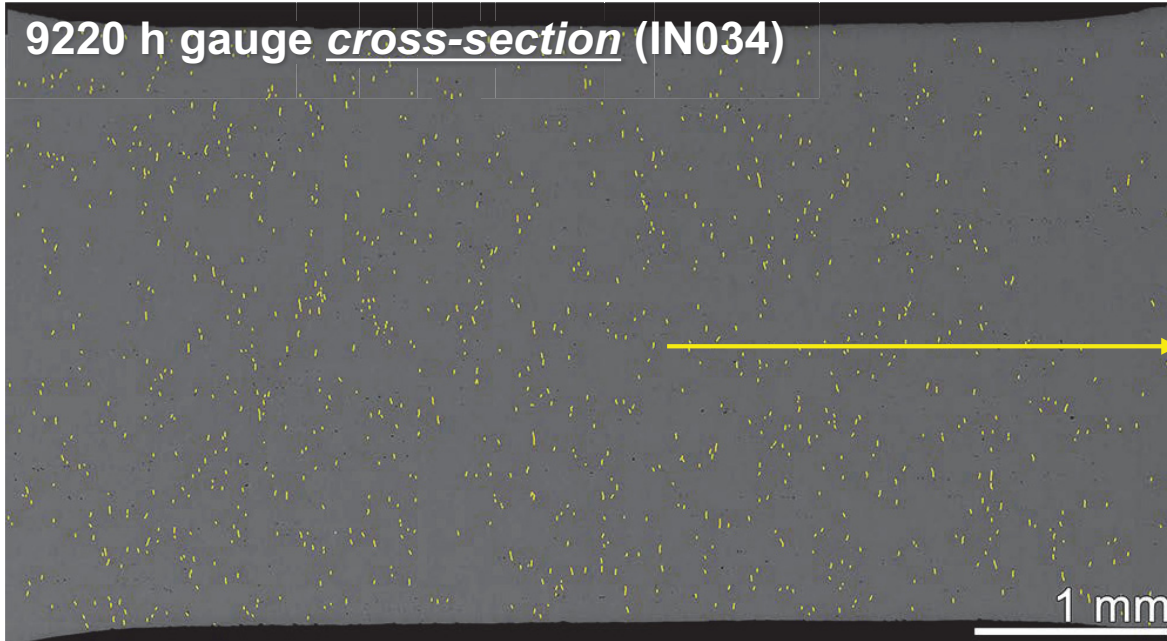


*Obviously
longer
aggregations
of denser GB
cavities*

Highly CW Materials: Cross-Section Morphology

Sumitomo CRDM TT+31%CF, 360°C at YS (~675 MPa), “C” finish

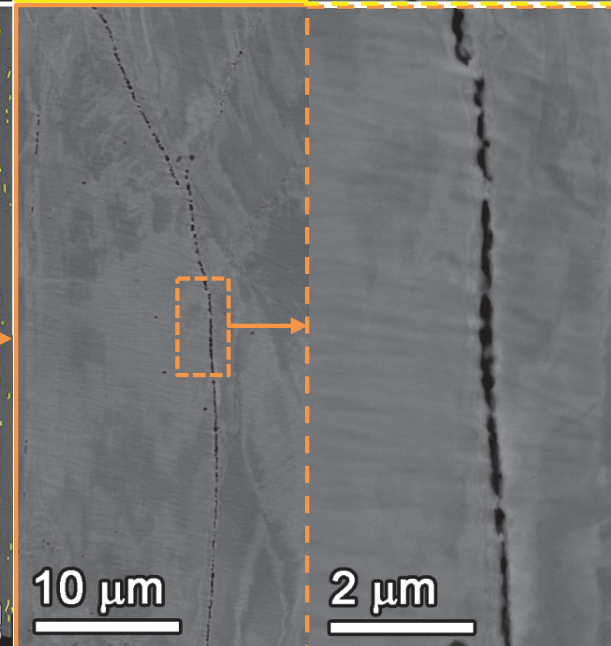
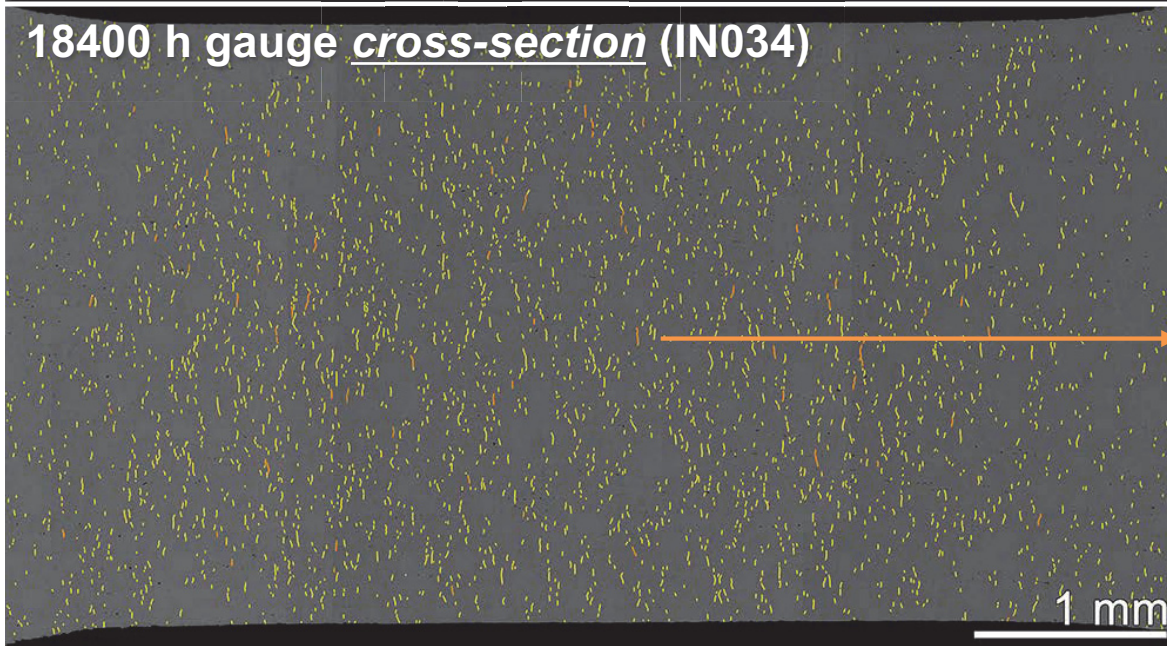
9220 h gauge cross-section (IN034)



stress

Closely spaced but discrete GB cavities

18400 h gauge cross-section (IN034)

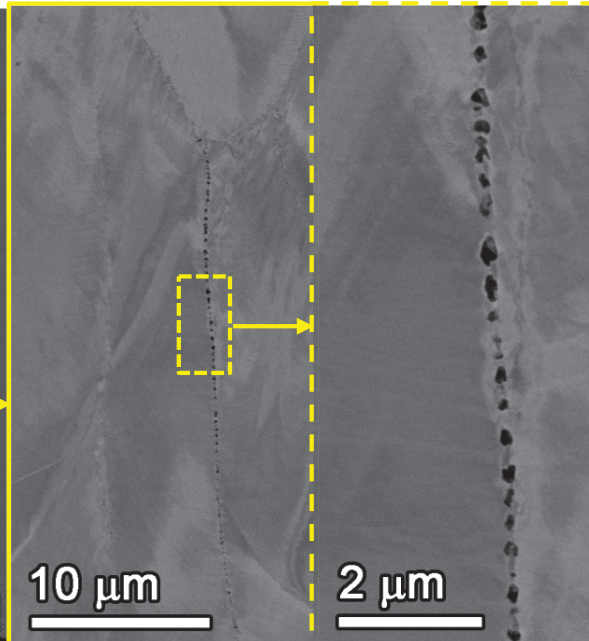
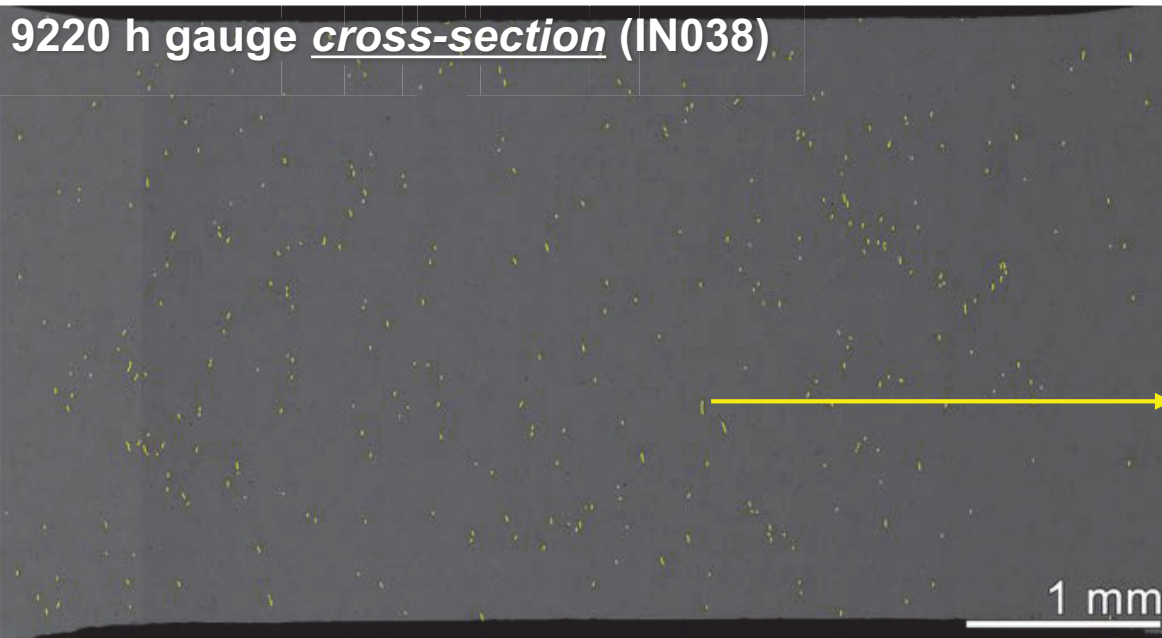


Semi-continuous and partially linked up GB cavities and cracks

Highly CW Materials: Cross-Section Morphology

Valinox CRDM TT+31%CF, 360°C at YS (~690 MPa), “C” finish

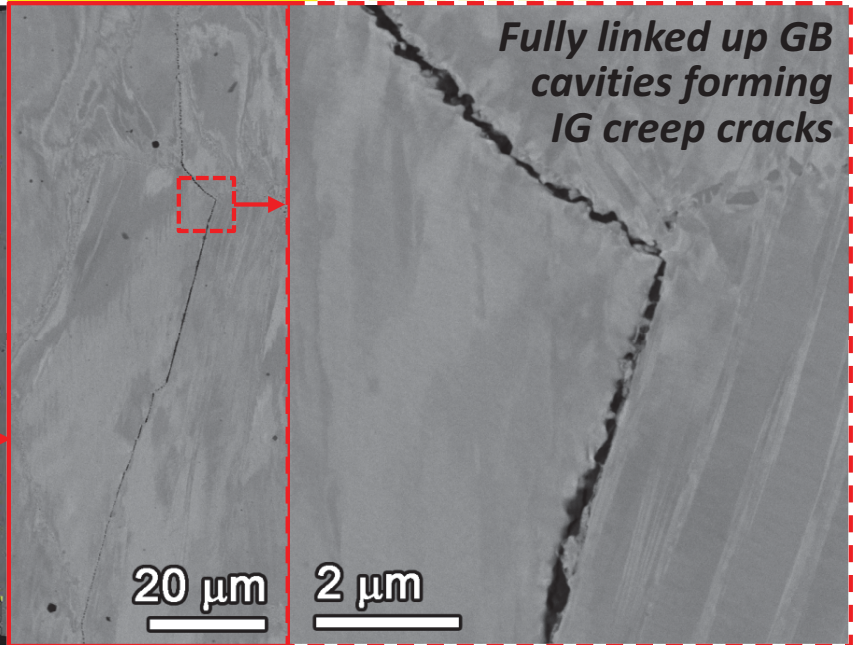
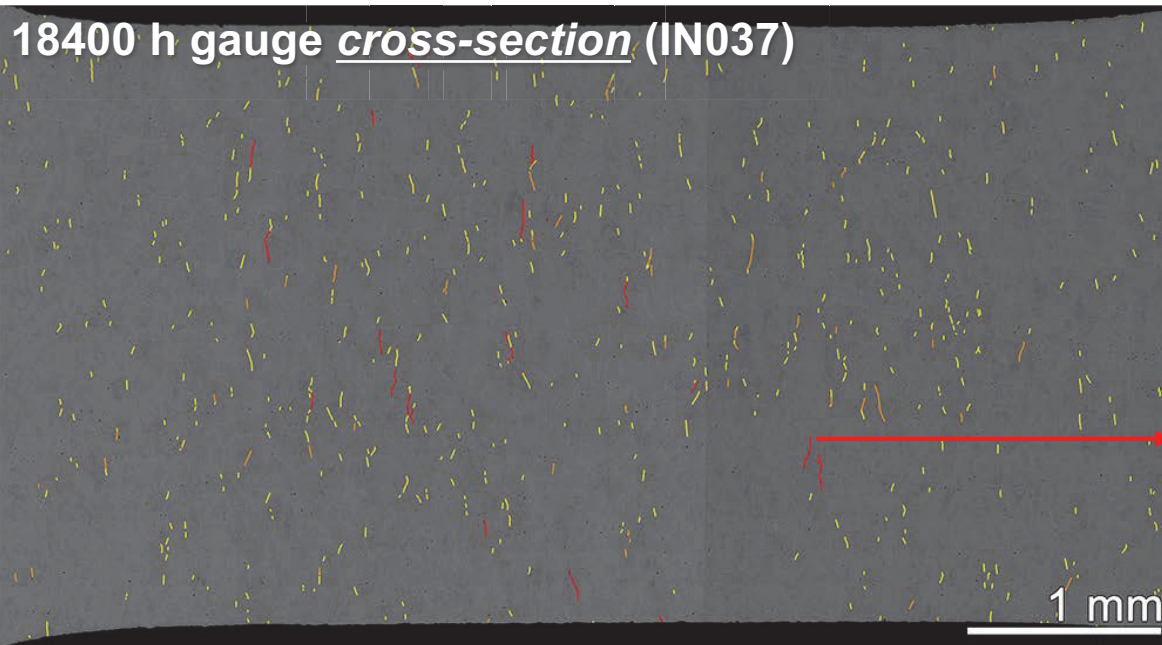
9220 h gauge cross-section (IN038)



stress
↔

Closely spaced but discrete GB cavities

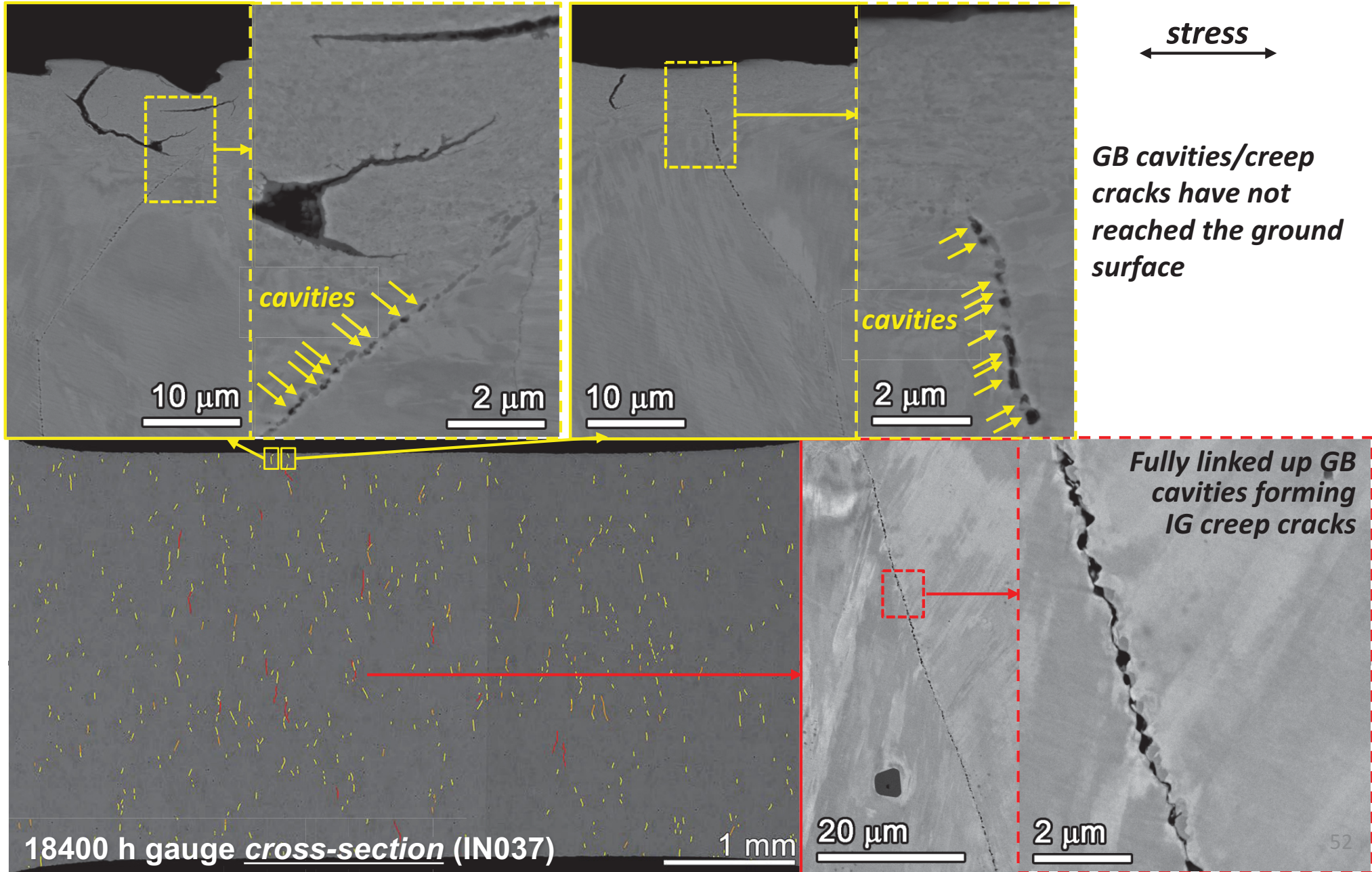
18400 h gauge cross-section (IN037)



Fully linked up GB cavities forming IG creep cracks

Highly CW Materials: Cross-Section Morphology

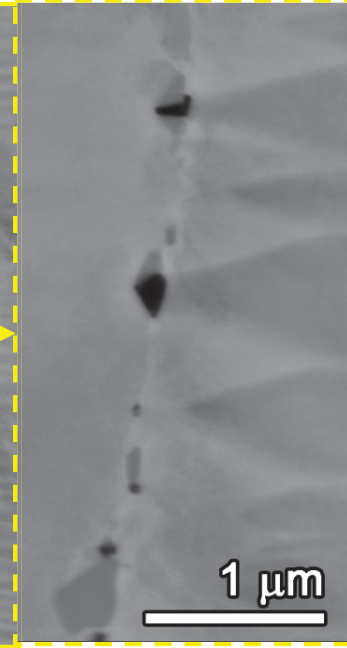
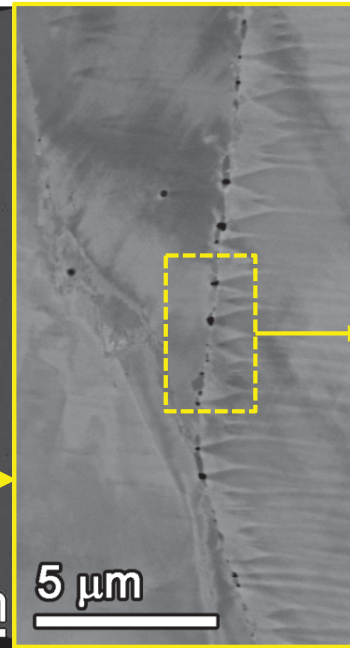
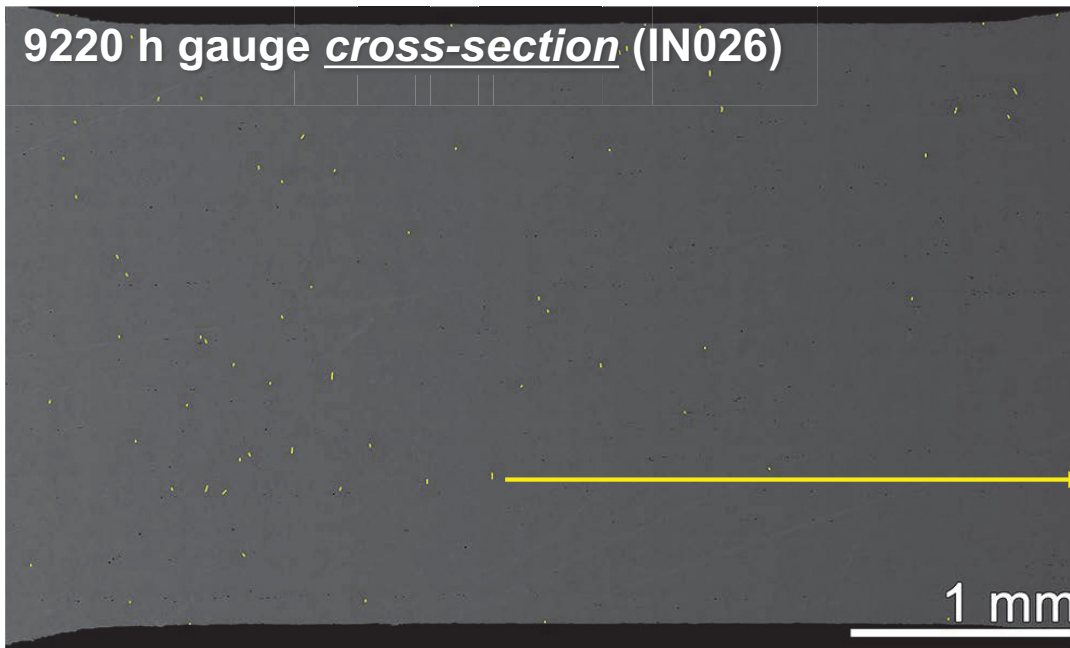
Valinox CRDM TT+31%CF, 360°C at YS (~690 MPa), "C" finish



Moderately CW Materials: Cross-Section Morphology

Sumitomo CRDM TT+21%CF, 360°C at YS (~590 MPa), “C” finish

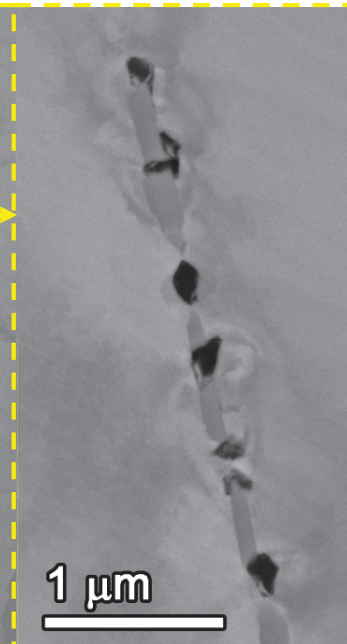
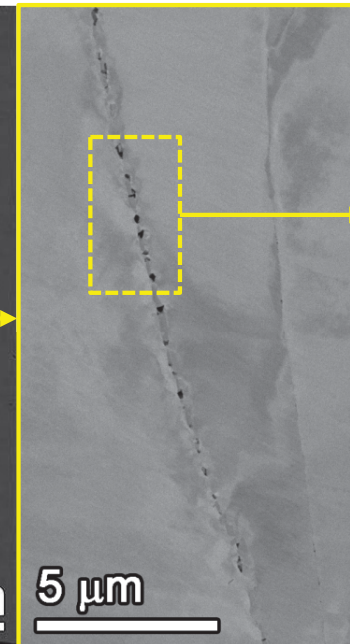
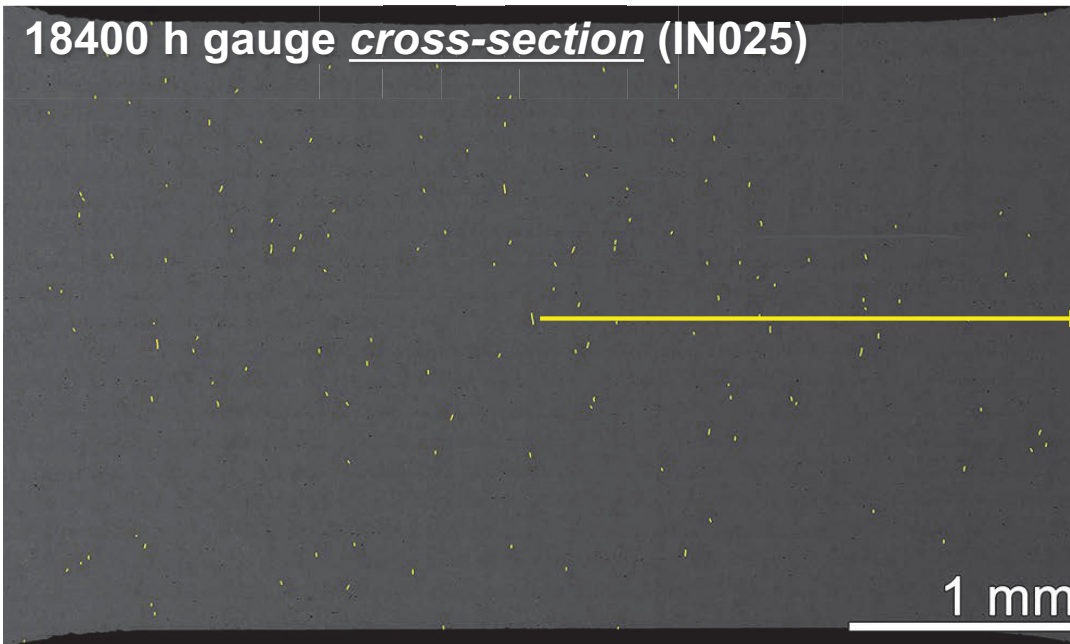
9220 h gauge cross-section (IN026)



stress

Slight increase in cavity size leading to higher visibility in the montage map, but no obvious change in cavity density and spacing

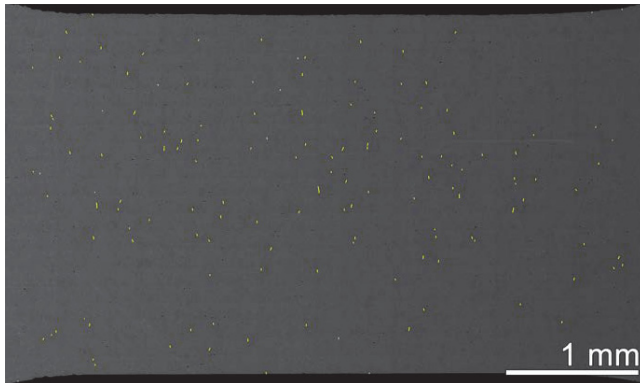
18400 h gauge cross-section (IN025)



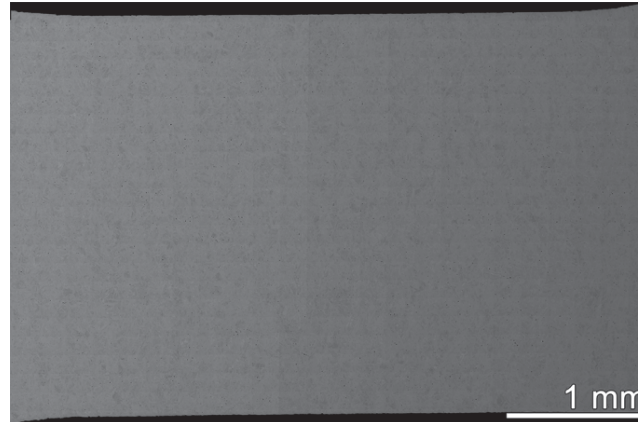
Moderately CW Materials: Cross-Section Morphology

Sporadically distributed GB cavities were observed in the TT+21%CF materials, but the size and density are not sufficient to allow them be fully identified in the montage maps.

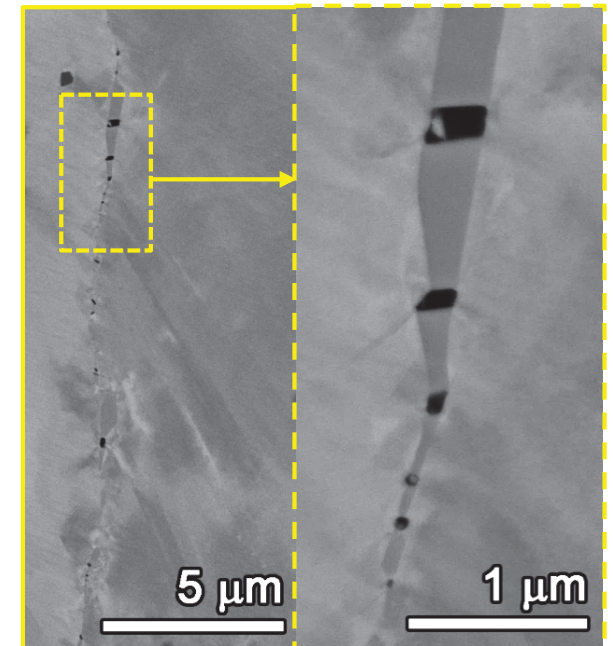
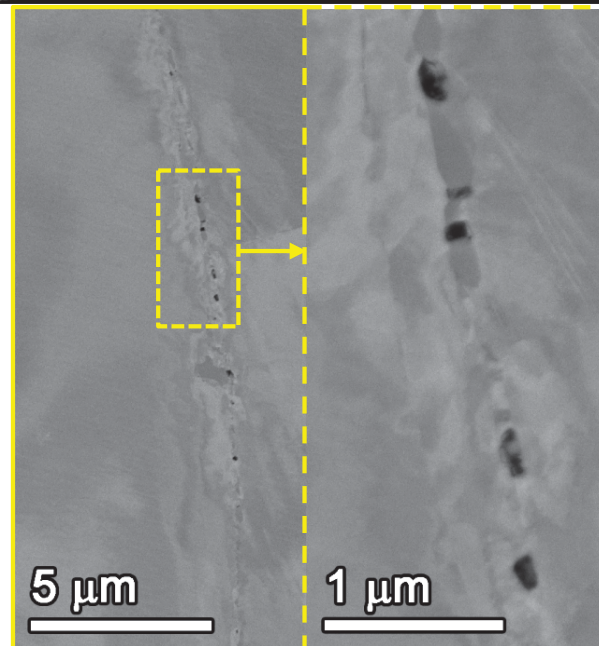
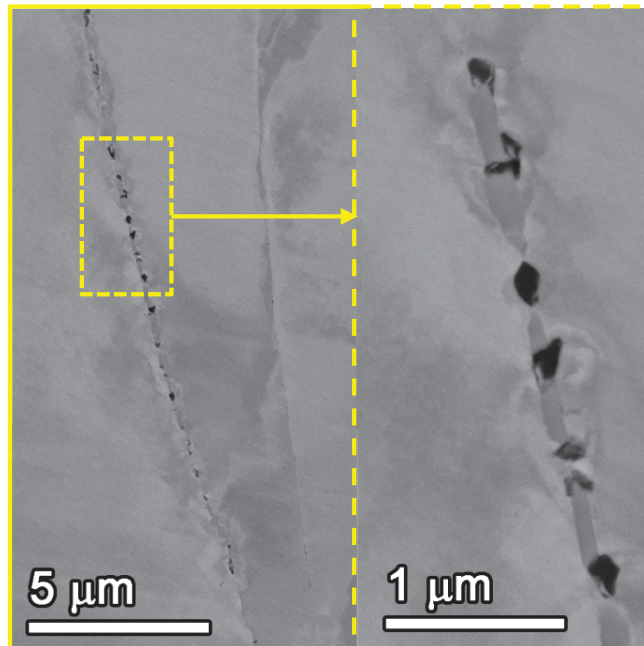
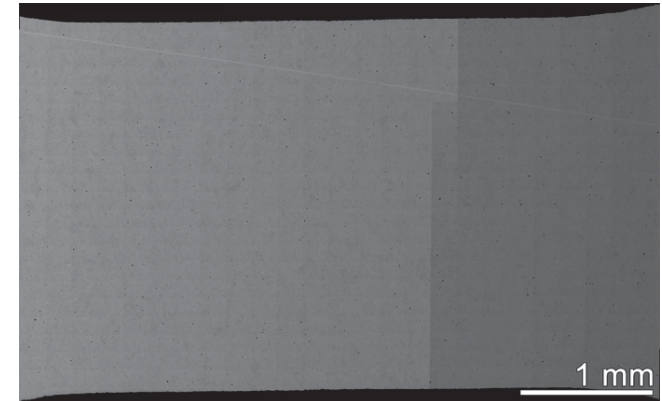
Sumitomo CRDM TT+21%CF



Valinox CRDM TT+21%CF



Doosan CRDM TT+21%CF

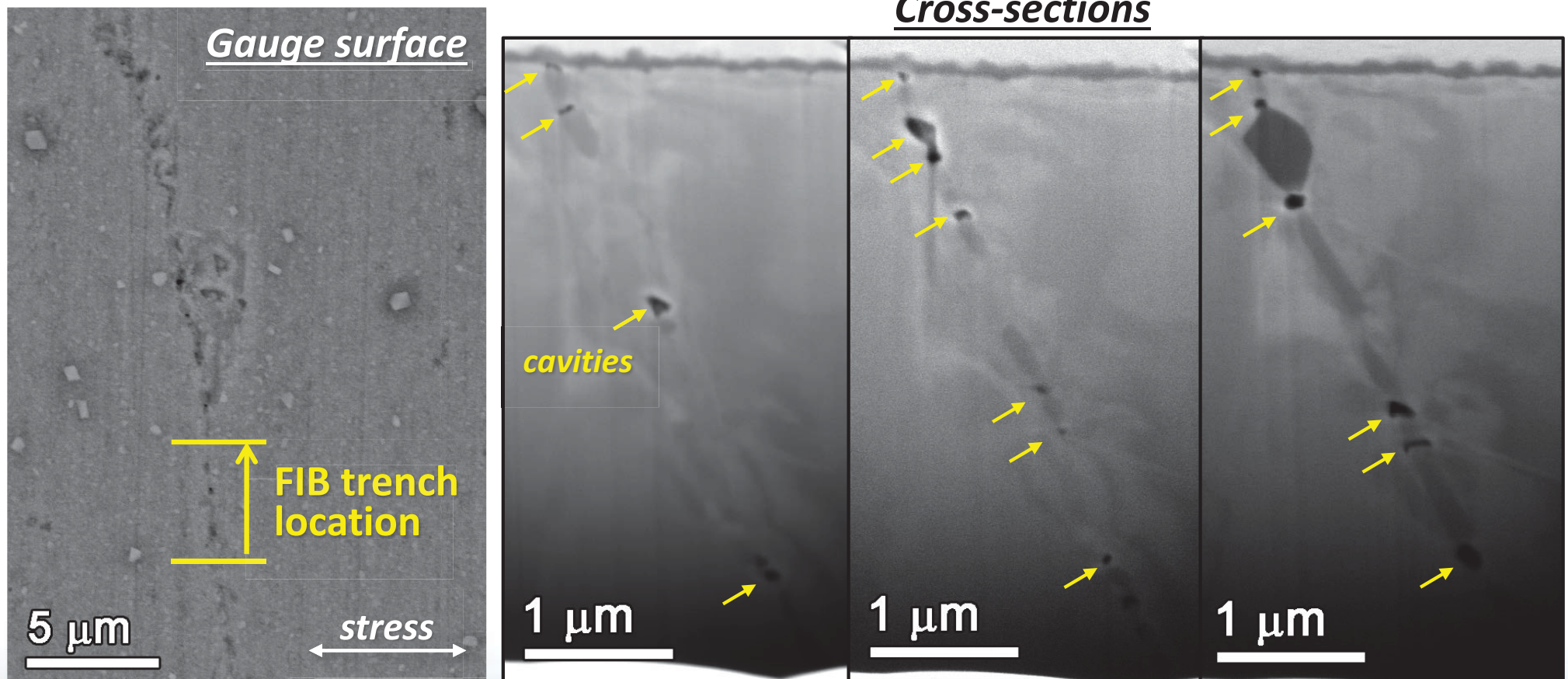


All three specimens tested in 360°C water at YS for 18400 hours

Replacement Specimens: FIB Trenching

Sumitomo CRDM TT+31%CF, 360°C at 90% YS (~621 MPa), 1 μm finish, 9180 h

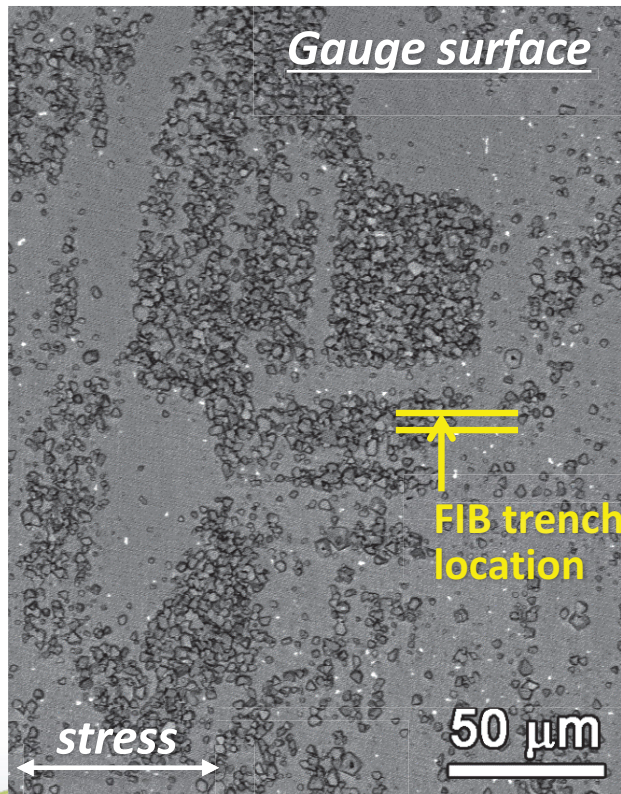
- High-energy GBs are extensively delineated with “postage stamp” damage.
- Serial FIB trenching of one GB revealed moderate density of IG cavities below the surface.



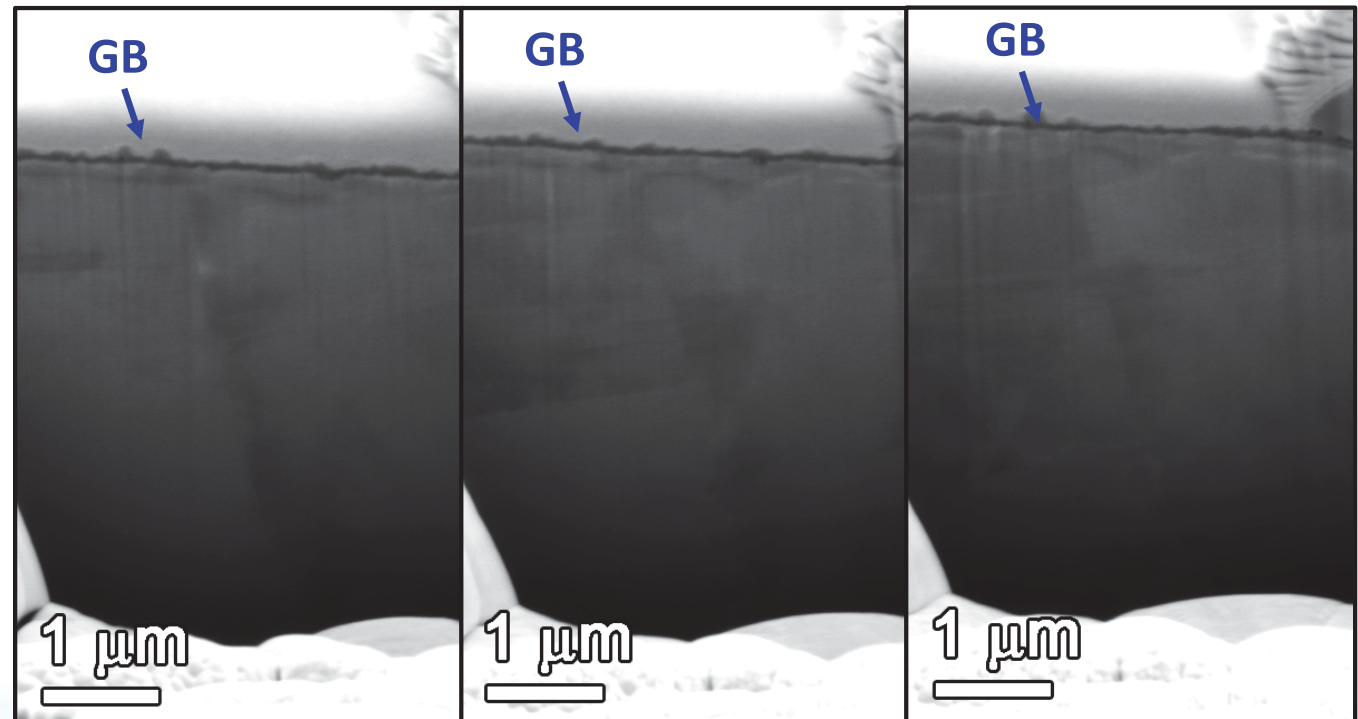
Replacement Specimens: FIB Trenching

Valinox CRDM SA+31%CR, 360°C at 100% YS (~650 MPa), 1 μm finish, 9180 h

- High-energy GBs are not identifiable but the shape of some grains are revealed by non-uniform distribution of spinel oxides on the surface.
- Serial FIB trenching of one high-energy GB revealed no IG cavities below the surface.



Cross-sections



2nd Stage Test: Summary of Preliminary Characterization Results

Specimens tested during both the 1st and 2nd stages:

Material, Condition	σ (MPa)	GB carbide distribution		SEM Cross-Section Observation on <i>new GB cavities</i>		SEM Cross-Section Observation on <i>IG cracks</i>	
		Spacing	Size	0-9220 h	9220-18400 h	0-9220 h	9220-18400 h
Sumitomo CRDM, TT+31%CF	675 (YS)	~100 nm	Small	Many	Many	None	Very few
Valinox CRDM, TT+31%CF	690 (YS)	~100 nm	Small	Some	Some	None	Some
TK-VDM Plate, TT+32%CF	675 (YS)	~0.1–0.5 μm	Medium	Some	Some	None	None
Doosan CRDM, TT+31%CF	655 (YS)	~0.5–2 μm	Large	Very few	Very few	None	None
Sumitomo CRDM, TT+21%CF	590 (YS)	~100 nm	Small	Very few	Very few	None	None
Valinox CRDM, TT+21%CF	525 (YS)	~100 nm	Small	Very few	Very few	None	None
Doosan CRDM, TT+21%CF	555 (YS)	~0.5–2 μm	Large	Very few	Very few	None	None

Replacement specimens tested only during the 2nd stage:

Material, Condition	σ (MPa)	GB carbide distribution		FIB Trenching Observation on <i>new GB cavities</i> (0-9180 h)
		Spacing	Size	
Sumitomo CRDM, TT+31%CF	621 (90% YS)	~100 nm	Small	Few?
Valinox CRDM, SA+31%CR	650 (YS)	N/A	N/A	No GB cavities observed

IG cavity size and density may be evolving more slowly during the 2nd stage exposure, but does continue for selected CW CRDM materials. Detailed characterizations underway and 3rd stage testing will begin.

LWRS

Light Water Reactor Sustainability

