SCC Initiation in Alloy 600 and Alloy 690

Presentations at the EPRI Alloy 690/152/52 Collaborative Research Meeting

Milestone: M3LW-17OR0402032

Steve Bruemmer, Ziqing Zhai and Mychailo Toloczko

Pacific Northwest National Laboratory

Milestone Submission

December 19, 2016

Light Water Reactor Sustainability

Light Water Reactor Sustainability R&D Program

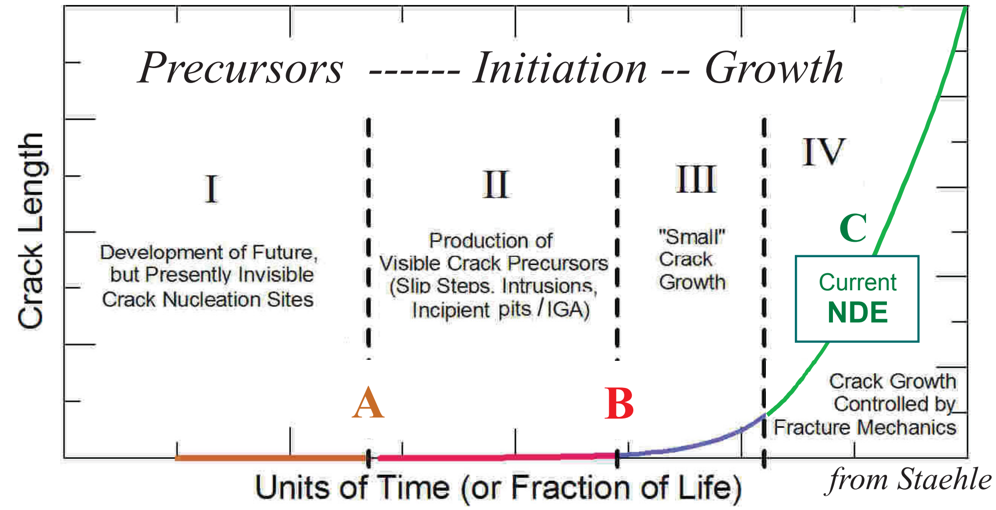
Overall Project Objectives and Approach

- *Focus is to identify mechanisms controlling SCC initiation of corrosion-resistant nickel-base alloys under realistic LWR service conditions.*
- *Research is investigating important material (composition, microstructure and surface condition) and environmental (water chemistry, temperature and electrochemical potential) effects on the susceptibility to localized corrosion/oxidation and SCC initiation.*
- *High-temperature autoclave test systems have been designed and constructed to enable evaluation of corrosion precursors and in-situ measurement of crack initiation in simulated LWR environments.*
- *Experiments are evaluating surface corrosion-oxidation, grain boundary damage and crack initiation* in PWR primary water for *alloy 600, 690 and their weld metals.*

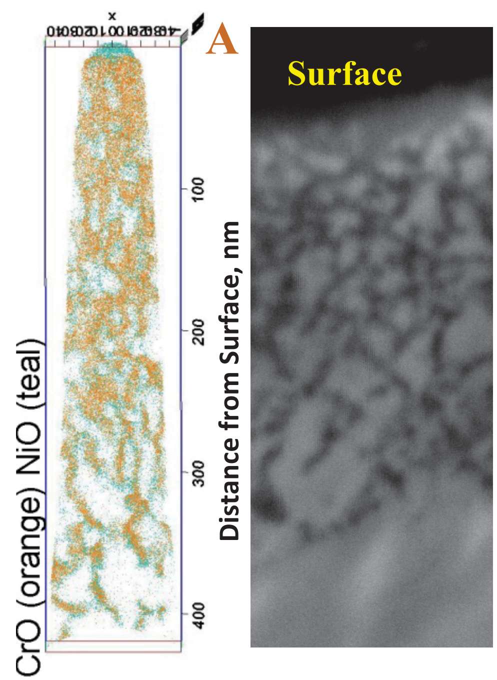
LWRS Light Water Reactor Sustainability

Background: Nano-to-Microscale SCC Initiation Precursors in LWR Service Environments

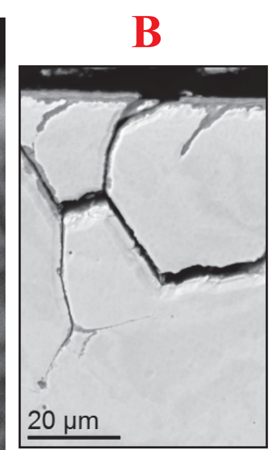
- *The evolution of stress-corrosion crack initiation may take decades in service to form, but is often followed by rapid crack growth and component failure.*
- *It is essential to detect early near-surface degradation precursors (**A**) and monitor development to crack initiation (**B**) enabling proactive mitigation before the rapid growth stage (**C**) is reached.*
- ***A key aspect of this research is on the characterization of localized damage, oxidation and crack precursors at the nano-to-microscale in LWR component alloys.***
- *Characterizations include high-resolution scanning (SEM) and transmission electron microscopy (TEM) plus atom probe tomography (APT) combined with state-of-the-art SCC initiation testing.*



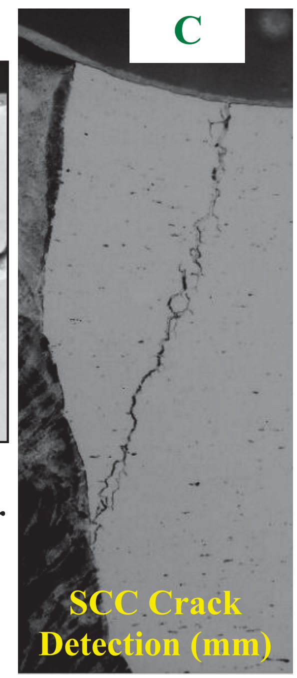
from Staehle



Precursor Damage Penetrative Oxidation (0.1-10 µm)



SCC Initiation from Precursor Damage (10-50 µm)



PNNL Presentations at the EPRI Research Collaboration Meeting: November 29, 2016

- **PNNL Status on ICG-EAC SCC Initiation Round Robin Testing**
  - *SCC initiation test results obtained at PNNL were summarized for the two 15%CW alloy 600 round robin materials in 360°C PWR primary water.*
  - *Consistent SCC initiation times were measured for the 15%CW PNNL alloy 600 plate, however new observations suggest that microstructure variations due to compositional banding influence cracking behavior.*
  - *Strong grain boundary (GB) segregation is present in the EPRI/GE alloy 600 plate and may promote its high susceptibility to IGSCC initiation.*
- **Mechanisms of *Crack Initiation in Cold-Worked Alloy 690***
  - *Results of long-term, constant-load SCC initiation tests on alloy 690 materials in 360°C simulated PWR primary water. No detection of SCC initiation, however surface nucleation of IG cracks occurred on certain highly CW alloy 690TT heats after exposure time of ~1 year.*
  - *Detailed high-resolution characterizations of surface cracks and sub-surface microstructures revealed nano-cavity formation at GB carbides throughout several high-CW specimens leading to crack nucleation.*
  - *The susceptibility to GB cavity formation and IG crack nucleation appears to be controlled by the carbide distribution and size in highly CW alloy 690 exposed at high stress in PWR primary water. Key issue is whether these damage processes will impact long-term performance in service.*

LWRS

Light Water Reactor Sustainability

PNNL Status on ICG-EAC PWSCC Initiation Round Robin Testing

Mychailo Toloczko, Ziqing Zhai and Steve Bruemmer

Pacific Northwest National Laboratory

Alloy 690/152/52 Research Collaboration Meeting

Tampa, Florida November 29, 2016

Light Water Reactor Sustainability

Light Water Reactor Sustainability R&D Program

Presentation Overview

- ***ICG-EAC Round Robin Overview***
- *PNNL SCC Initiation Testing Capability*
- *NX6106XK-11 Material Characterization*
- *Material Preparation and Specimen Fabrication*
- *Prior NX6016XK-11 SCC Initiation Test Results*
- *Round Robin NX6106XK-11 Test Results*
- *Round Robin 31907 Test Results*
- *Summary*

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 primary & secondary materials.

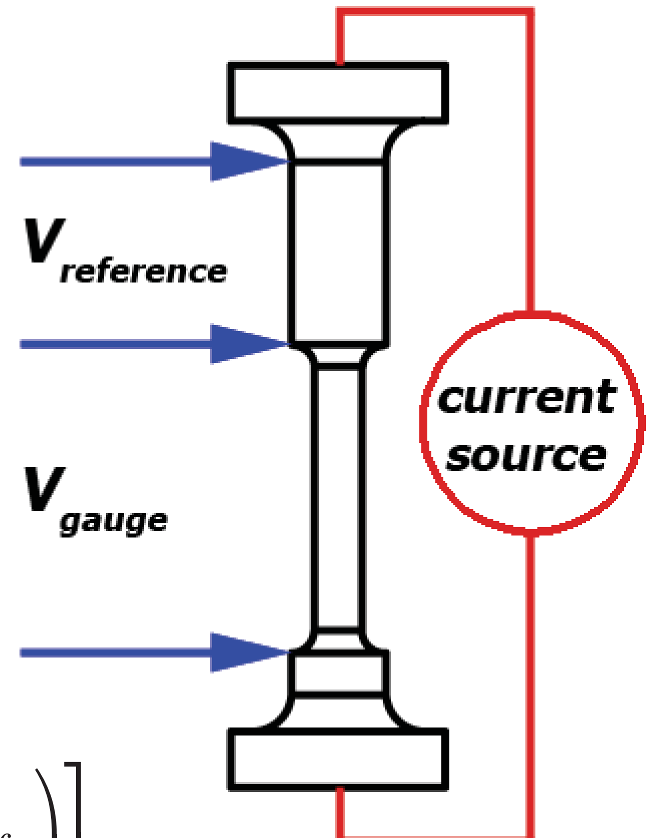
Presentation Overview

- *ICG-EAC Round Robin Overview*
- ***PNNL SCC Initiation Testing Capability***
- *NX6106XK-11 Material Characterization*
- *Material Preparation and Specimen Fabrication*
- *Prior NX6016XK-11 SCC Initiation Test Results*
- *Round Robin NX6106XK-11 Test Results*
- *Round Robin 31907 Test Results*
- *Summary*

DCPD to Measure Crack Initiation

- *DCPD voltage is sensitive to many factors:*
  - *Cracking (change in cross-section)*
  - *Creep and tensile straining (length and diameter change)*
  - *Material resistivity evolution (often due to aging)*
- *Tracking of a reference voltage on a large diameter region where no cracking occurs allows for removal of resistivity evolution.*
- *Strain is easily calculated from DCPD and is assumed to be the dominant effect on DCPD up to SCC initiation.*
- *True strain formulation used.*

Schematic of Idealized Voltage Measurement Points



$$\varepsilon_{referenced} = \frac{1}{2}\left[\ln\left(\frac{V_{gauge}}{V_{gauge_o}}\right) - \ln\left(\frac{V_{ref}}{V_{ref_o}}\right)\right]$$

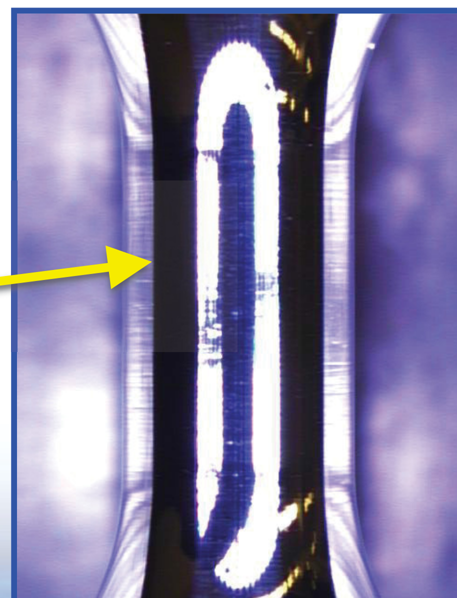
Specimen Selection and Preparation

- Tensile specimen: uniform, easily determined stress and strain.
- PNNL 1.2" length matches the height of a 0.5T CT specimen.
- Optimized geometry for DCPD-based detection of initiation
  - Short gauge length and small diameter accentuate DCPD initiation signal.
  - Small gauge dimensions allow complete documentation by SEM.
  - DCPD reference resistivity measured directly on the specimen.
- A range of surface finishes or notches can readily be applied.

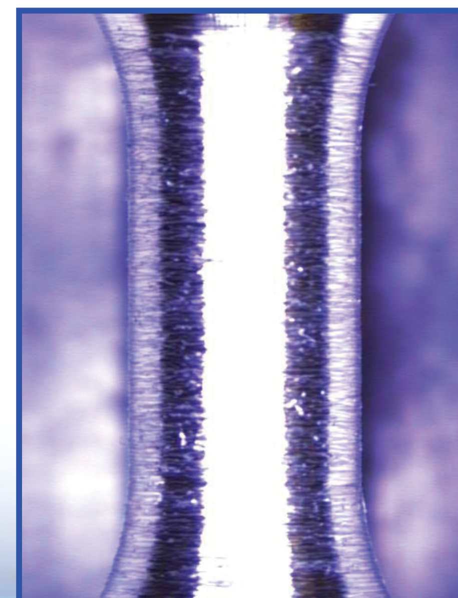
1.2" Tall SCC Initiation Specimen

Examples of Surface Finishes

1 µm finish



60 grit finish



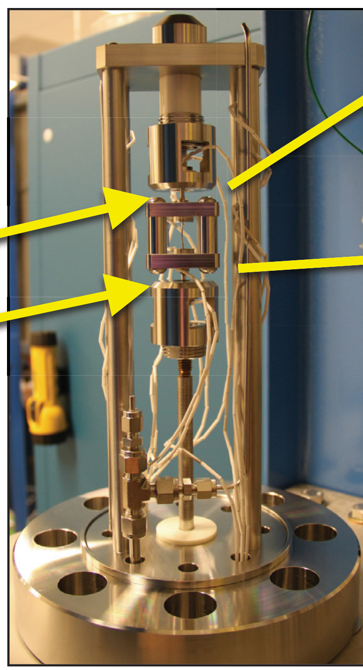
LWRS
Light Water Reactor Sustainability

PNNL LWRS Test Systems

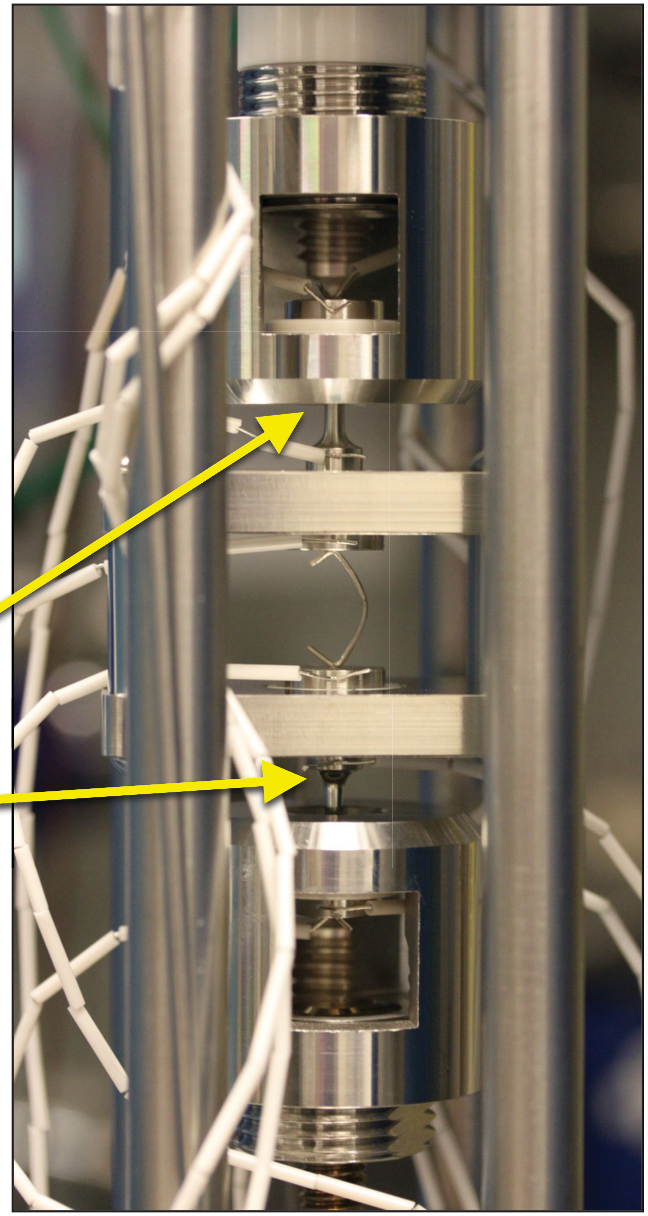
- *Based on PNNL SCC CGR test systems.*
- *Two systems that can test up to 6 specimens.*
- *One 36 specimen system with up to 12 specimens instrumented for initiation.*
- *All use DCPD for in-situ detection of crack initation.*

1.2" Tall SCC Initiation Specimen

2-6 Specimen SCC Initiation System



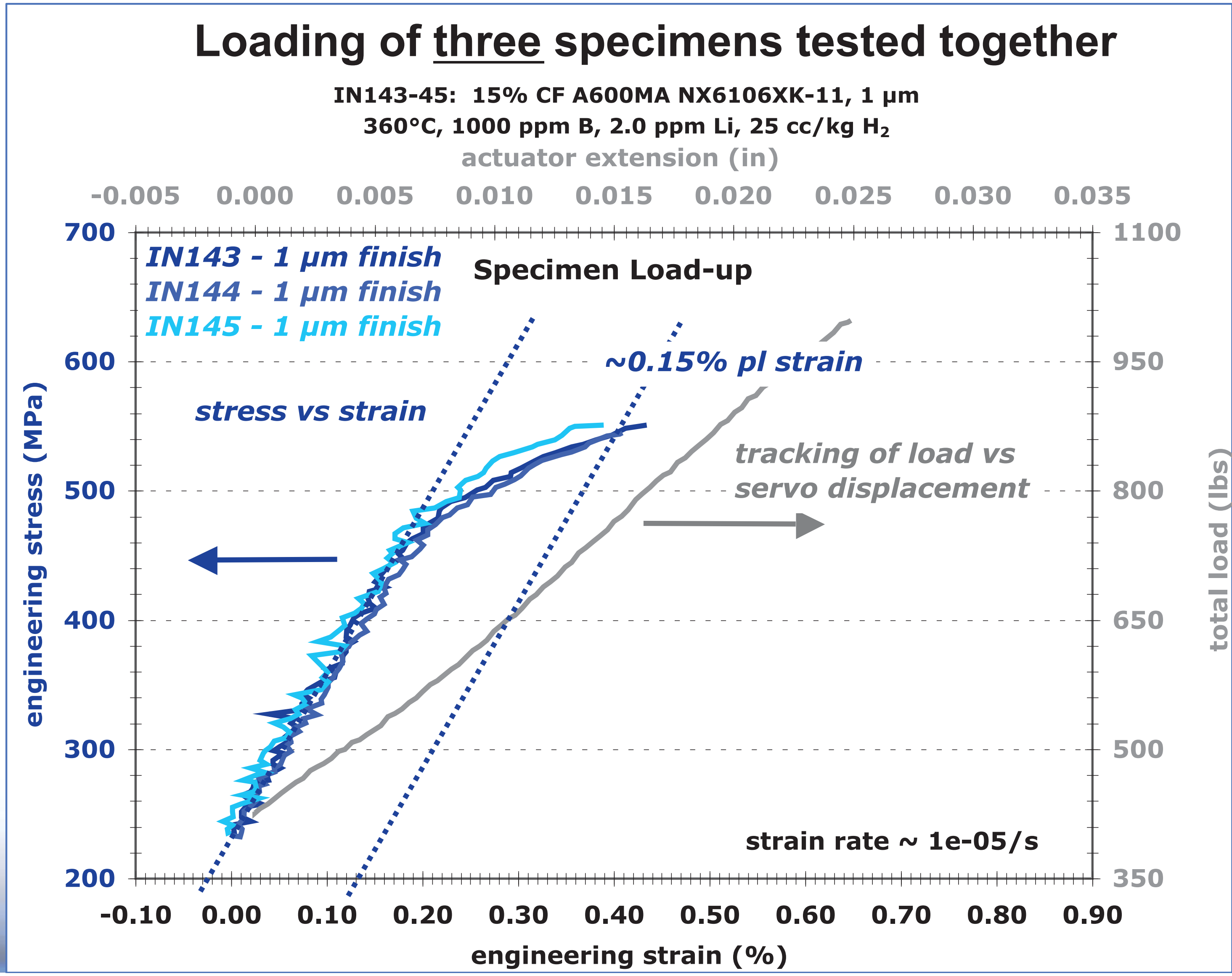
2-6 Specimen SCC Initiation System



36 Specimen SCC Initiation System

Test Methodology

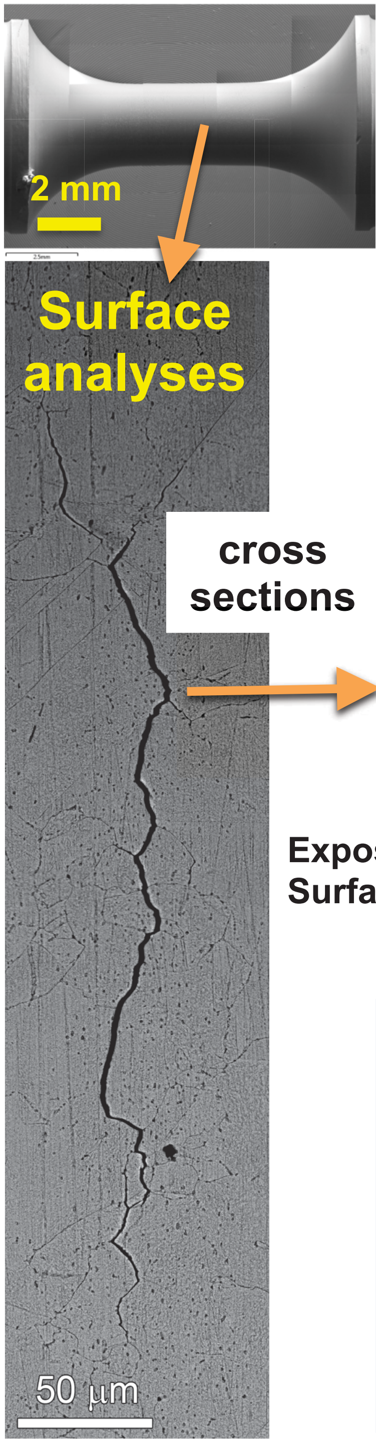
- *Gauge section is typically polished to a 1 µm finish to facilitate detection of cracking via surface and cross section examinations.*
- *Testing typically conducted at the yield strength of the material.*
- *Confirmation of yield determined by monitoring stress versus strain (from DCPD) at a displacement rate of ~1x10^{-5} s^{-1} (~1 hour to load).*
- *Loading typically stopped at ~0.1-0.2% plastic strain.*



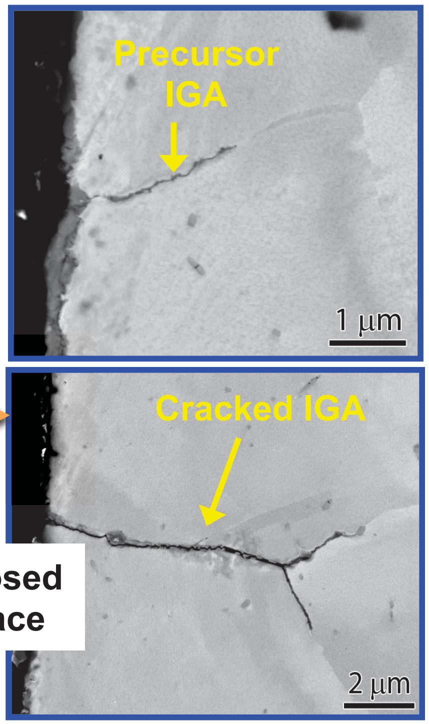
LWRS Light Water Reactor Sustainability

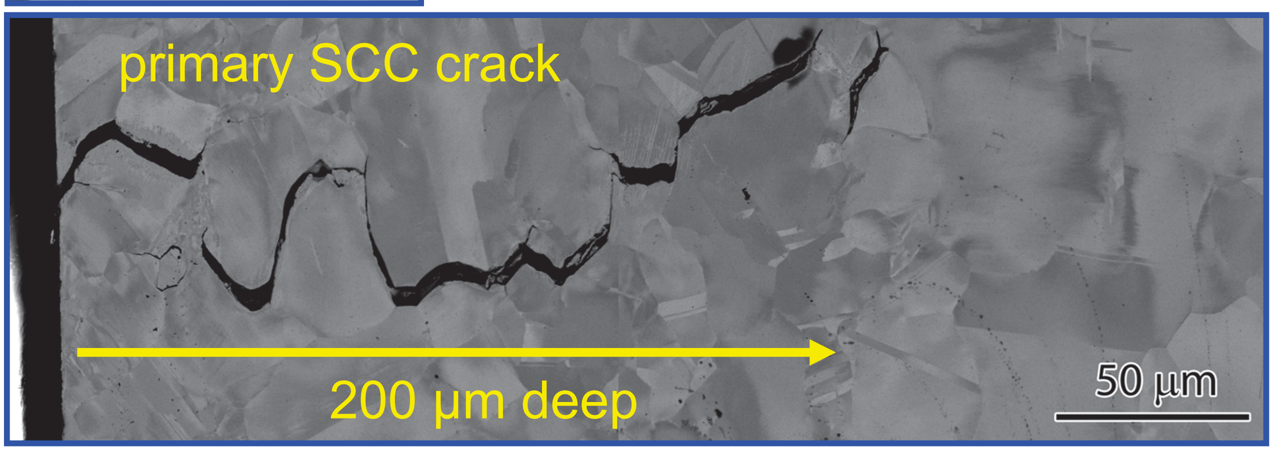
Detailed Analysis of Surface Length and Depth for Cracks

Gauge section

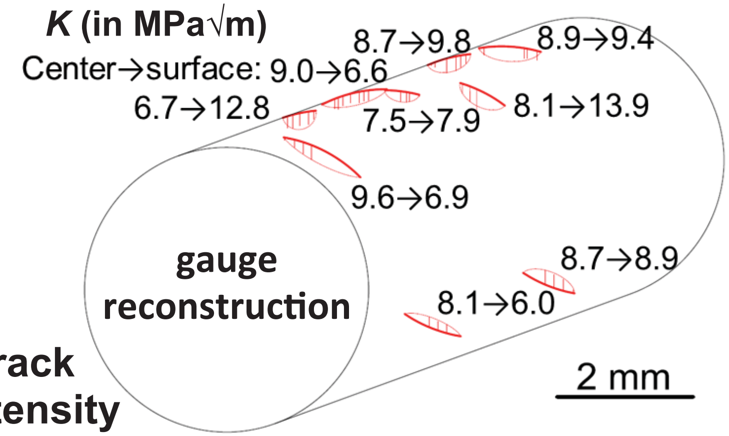


Cross section analyses

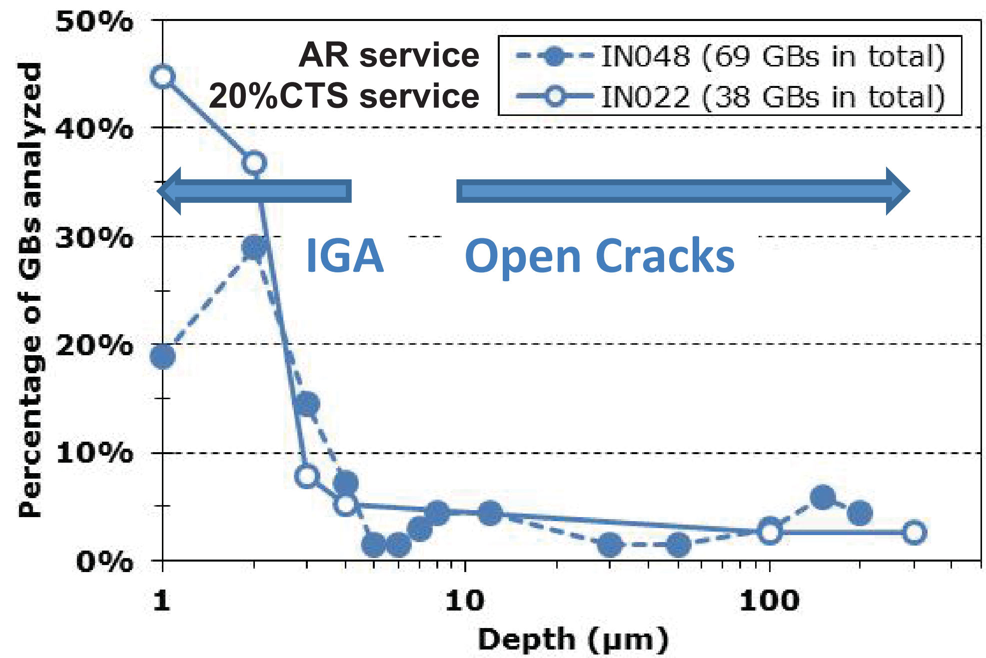




Reconstruction of crack shape and stress intensity



Crack type versus crack depth statistics



Presentation Overview

- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- ***NX6106XK-11 Material Characterization***
- Material Preparation and Specimen Fabrication
- Prior NX6106XK-11 SCC Initiation Test Results
- *Round Robin NX6106XK-11 Test Results*
- Round Robin 31907 Test Results
- *Summary*

Alloy 600 Heat NX6106XK-11 Information

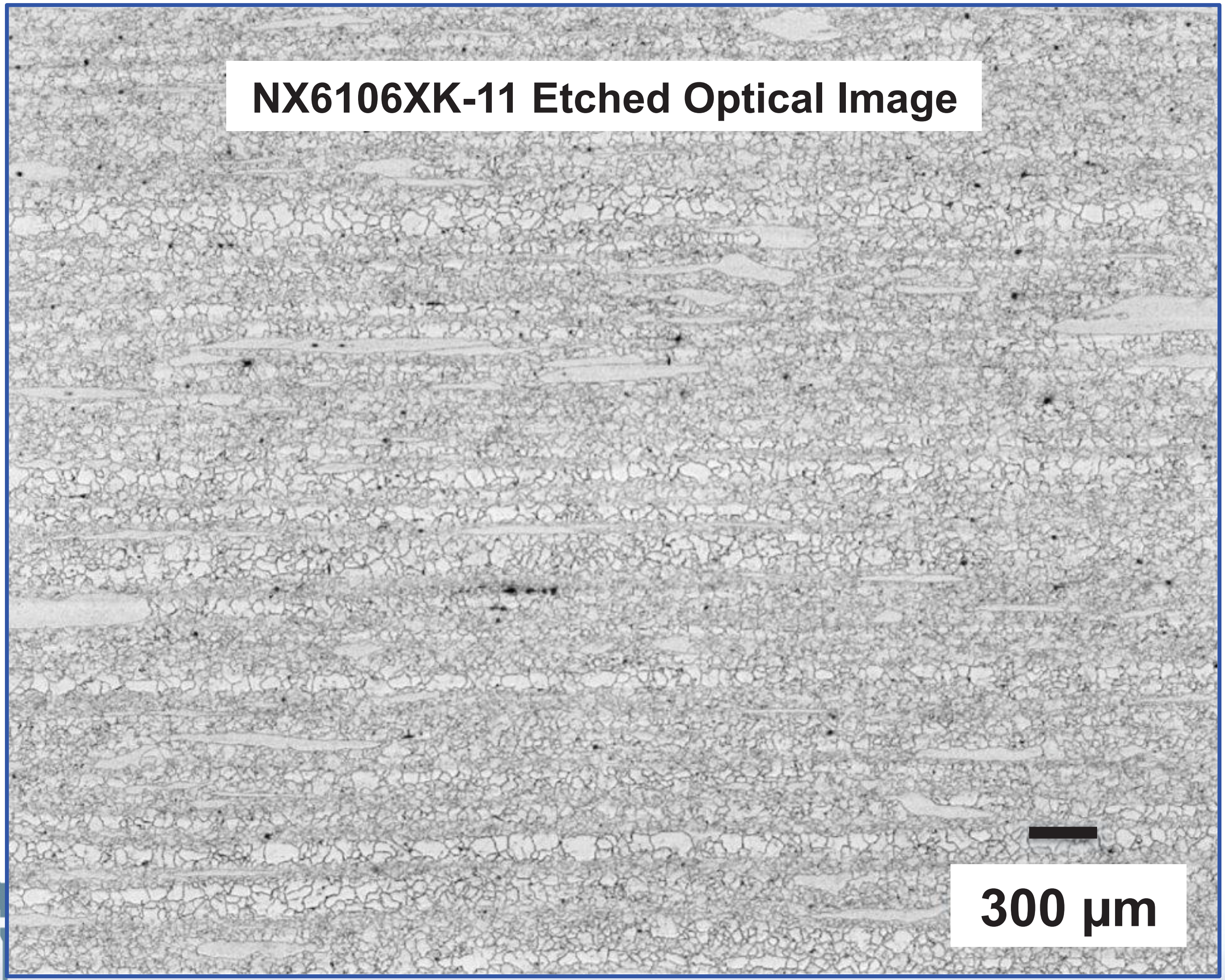
- *2 inch (50 mm) thick plate produced by Special Metals.*
- *Mill annealed at 1700°F (927°C) for 3.5 hours followed by water quench.*
- *Purchased by PNNL in 2009 for SCC initiation research.*
- *Has been initiation tested for PNNL LWRS program in 19%CR, 8% tensile strained and as-received condition.*
- *Have SCC crack growth rate data on 19%CR and as-received condition. SCC CGR testing of 15%CF in-progress.*

Product	Heat	Ni	Cr	Fe	C	Mn	Si	Cu	P	S	B
Plate	NX6106XK-11	74.0	16.4	8.5	0.06	0.23	0.22	0.01	.004	.001	NM
	A600 Spec	Bal	14-17	6-10	<.15	<1.0	<0.5	<0.5	---	<.015	---

NM = not measured

Alloy 600 Heat NX6106XK-11 Optical Imaging

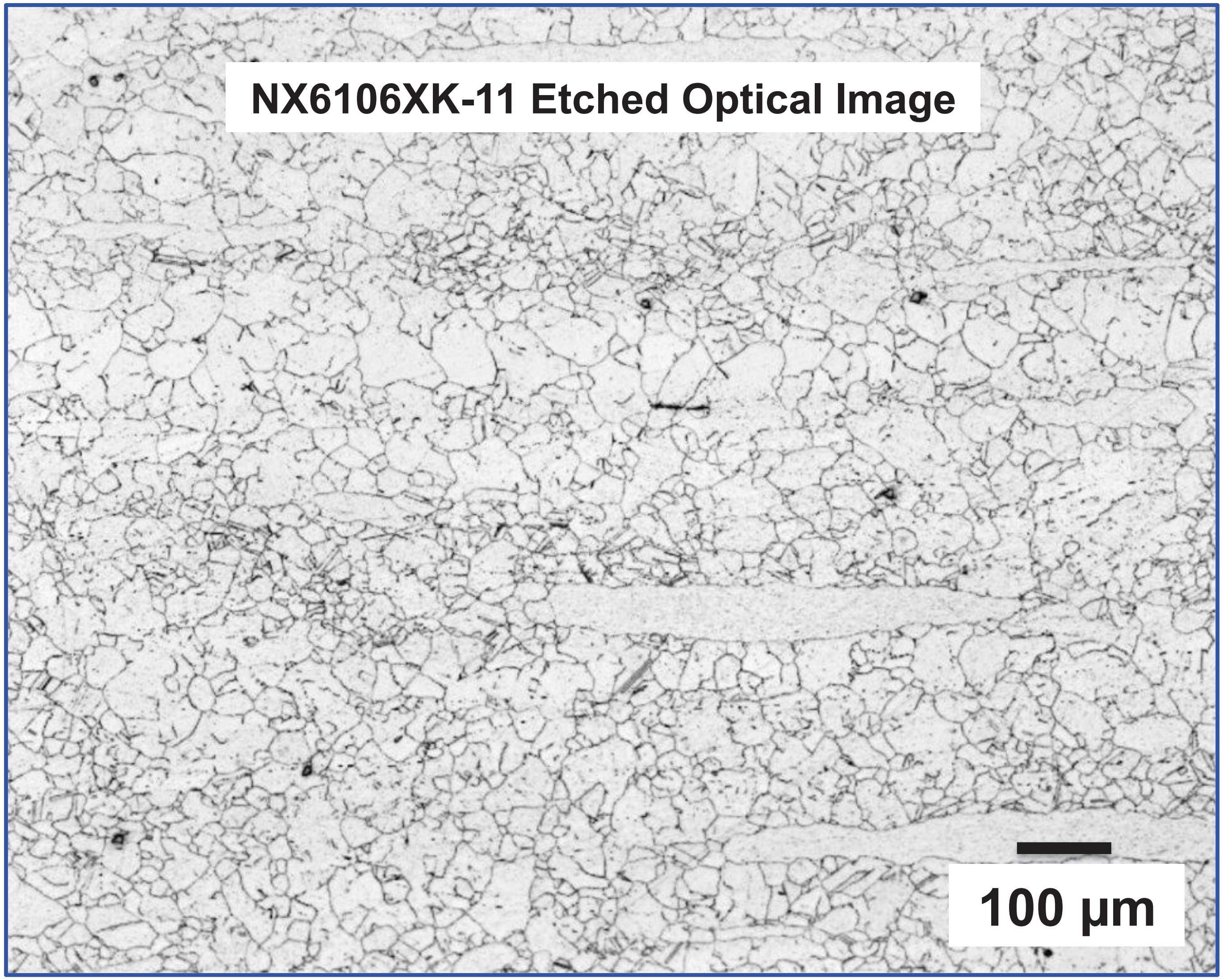
- *Inhomogeneous grain shape and size with banding in the plate processing direction.*



Light Water Reactor Sustainability

Alloy 600 Heat NX6106XK-11 Optical Imaging

- *Bands of larger and smaller grains. Sporadic distribution of highly elongated grains, some exceeding 500 μm.*
- *Concern that inhomogeneous microstructures may influence SCC initiation response.*



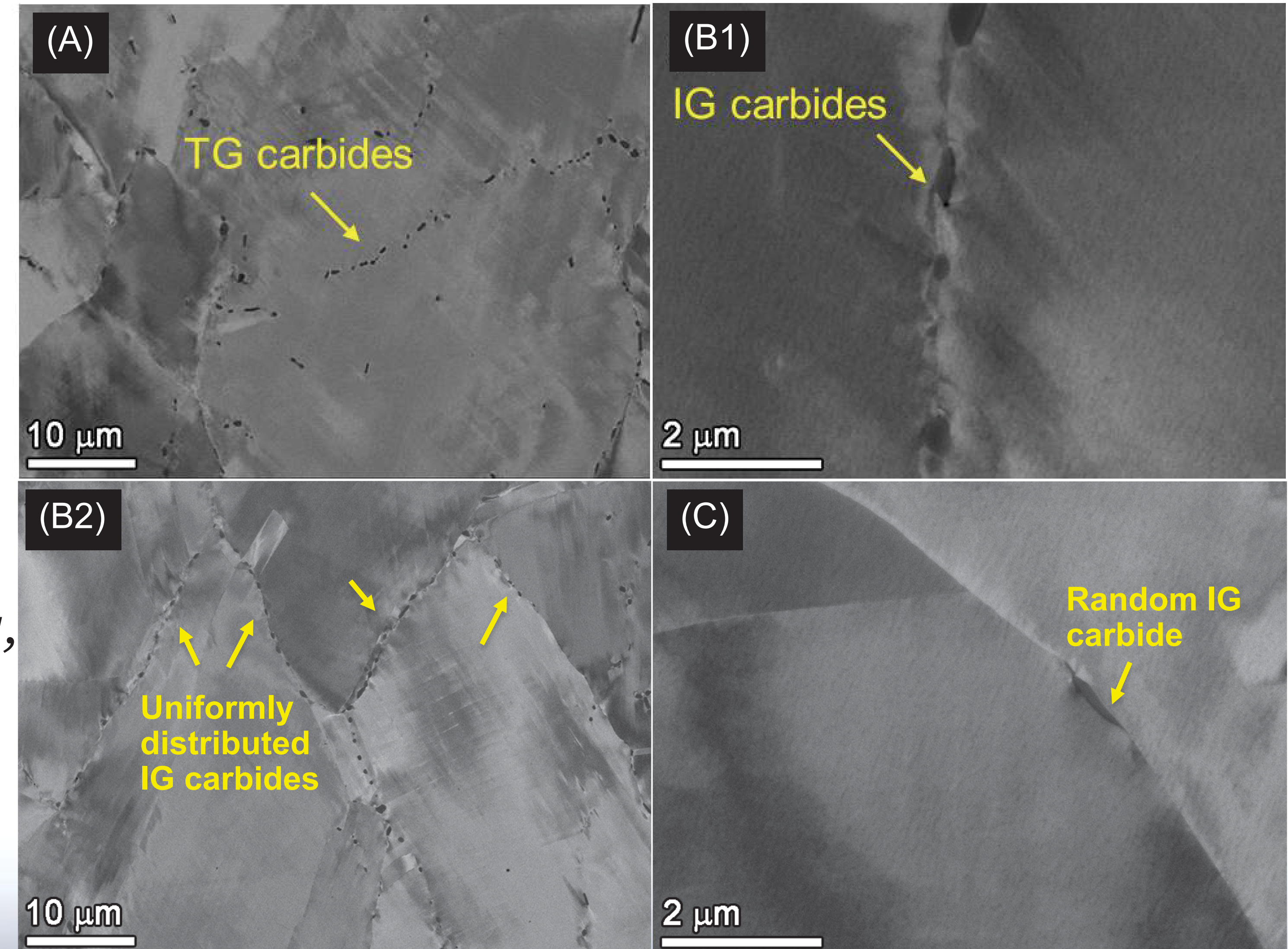
Alloy 600 Heat NX6106XK-11 SEM Imaging

- SEM imaging revealed both TG and IG carbides.
- Variable IG carbide density.

A. Most grains show primarily TG carbides.

B. Few grains show higher density of IG carbides.

C. More typical grain boundary, low density of IG carbides.



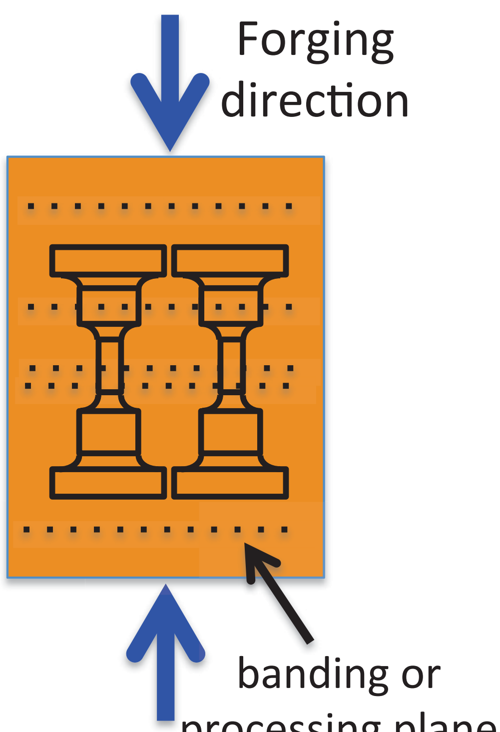
Presentation Overview

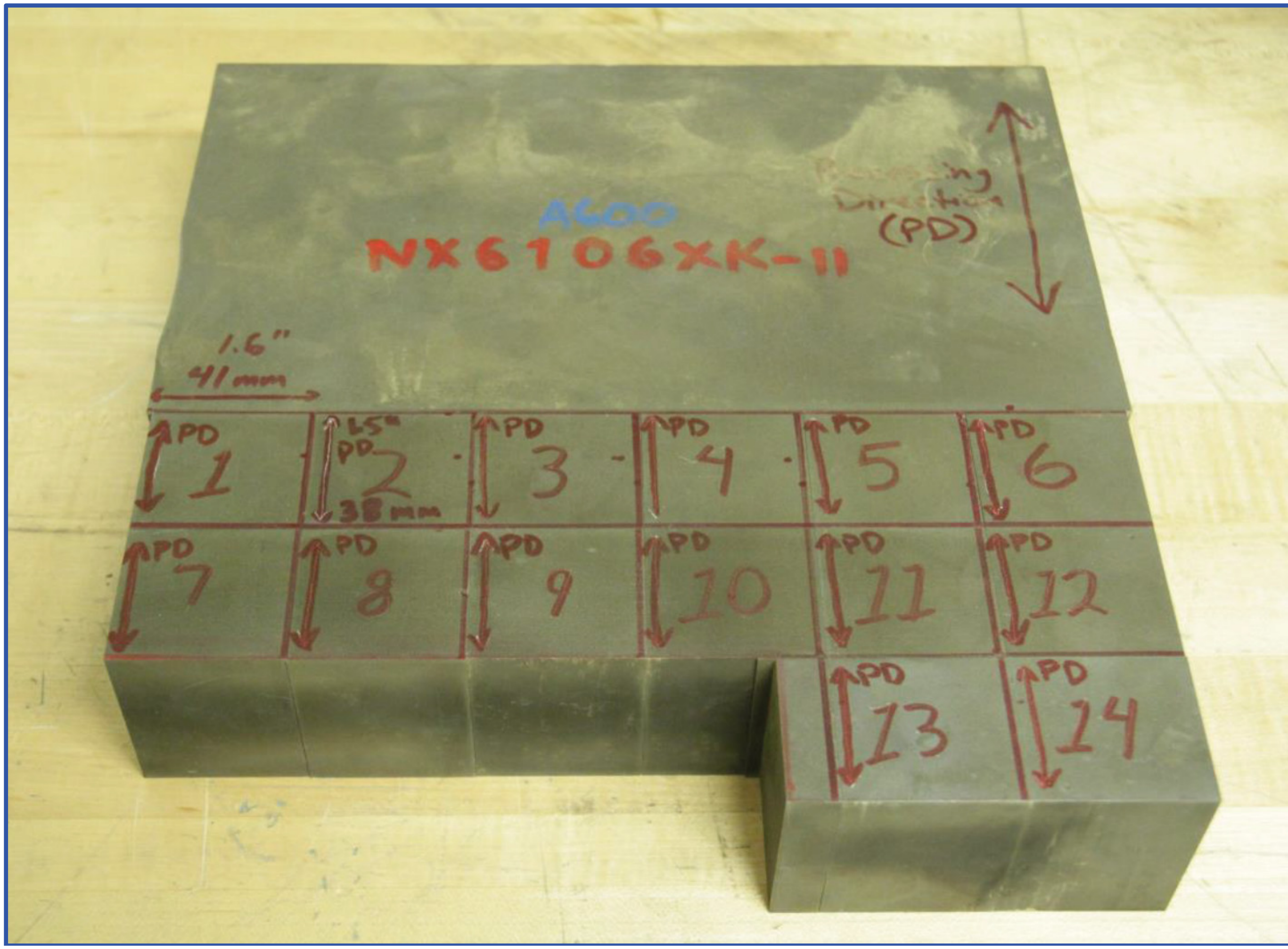
- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- *NX6106XK-11 Material Characterization*
- ***Material Preparation and Specimen Fabrication***
- *Prior NX6106XK-11 SCC Initiation Test Results*
- *Round Robin NX6106XK-11 Test Results*
- *Round Robin 31907 Test Results*
- *Summary*

Material Preparation and Specimen Fabrication

- *Plate material was cut into 1.5" long x 1.6" wide x 2" tall blocks.*
- *Blocks were numbered, and forging and mill processing direction (PD) were indicated on each block.*
- *Blocks were 15% cold-forged at GE Global by Peter Andresen.*
- *Forging and banding planes set to be coincident.*

Orientation of Specimens from Plate





Presentation Overview

- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- *NX6106XK-11 Material Characterization*
- *Material Preparation and Specimen Fabrication*
- ***Prior NX6106XK-11 SCC Initiation Test Results***
- *Round Robin NX6106XK-11 Test Results*
- *Round Robin 31907 Test Results*
- *Summary*

LWRS Light Water Reactor Sustainability

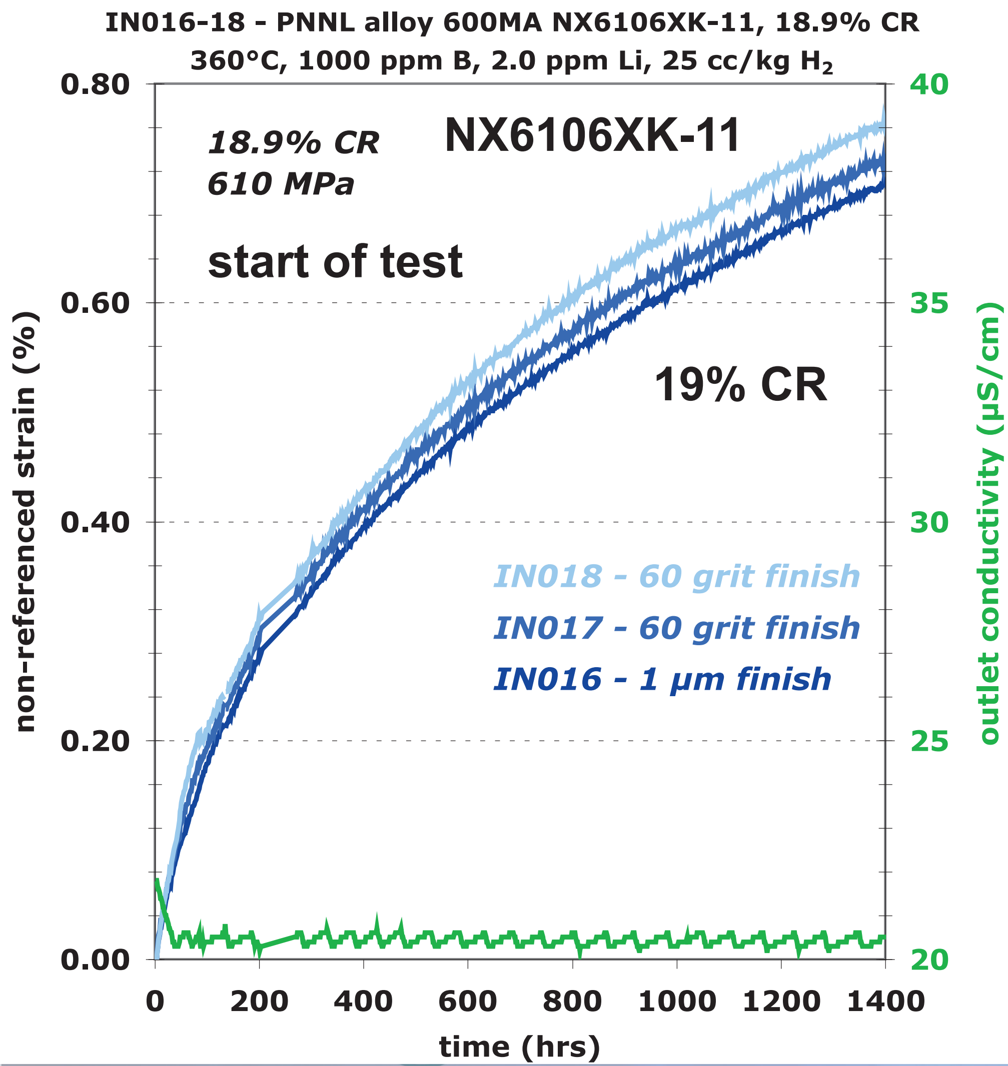
Test Conditions

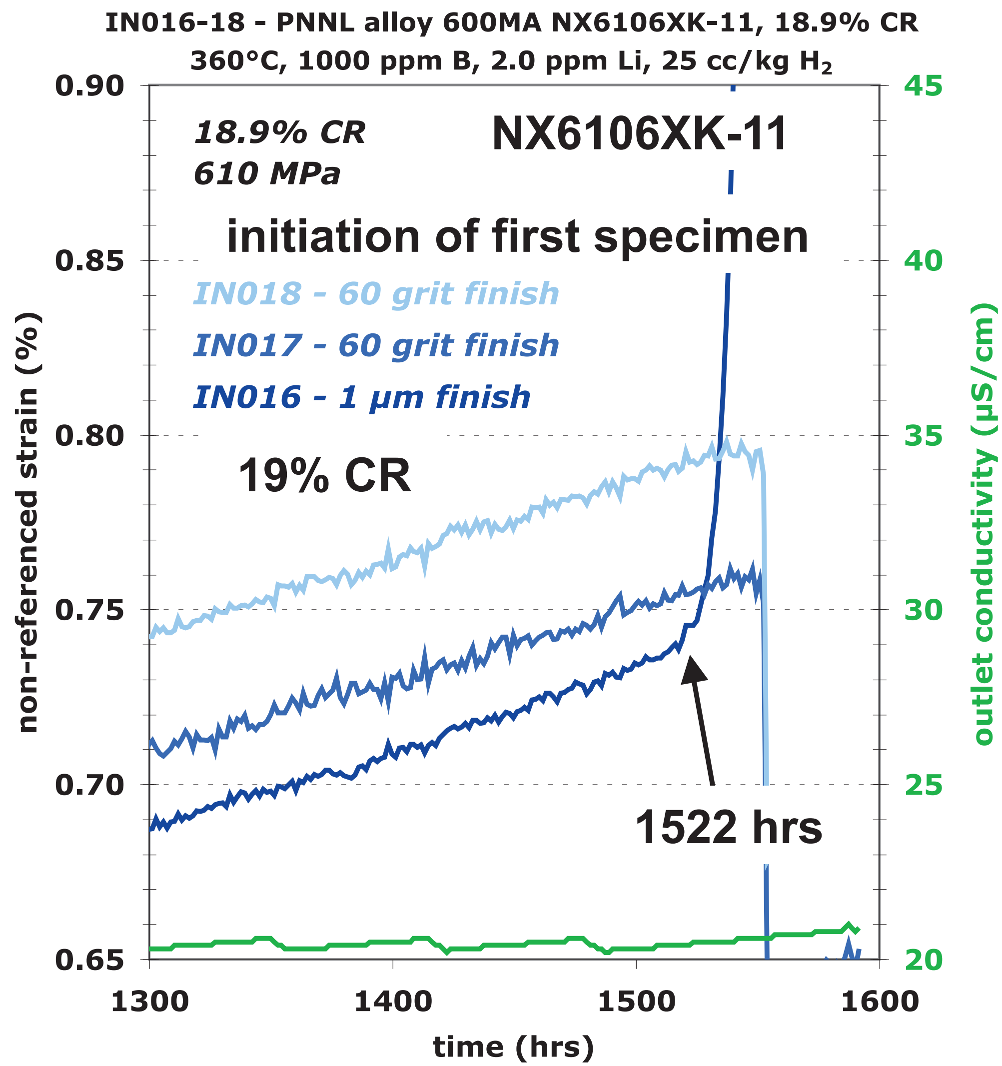
- *Gauge section of all specimens polished to a 1 μm finish.*
- *360°C selected to match prior experience at PNNL. May perform another set of tests at 325°C.*
- *Water conditions (as defined by Round Robin)*
  - *PWR primary water*
  - *1000 ppm B, 2 ppm Li*
  - *Ni/NiO stability line (25 cc/kg H_2)*
- *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.*

LWRS Light Water Reactor Sustainability

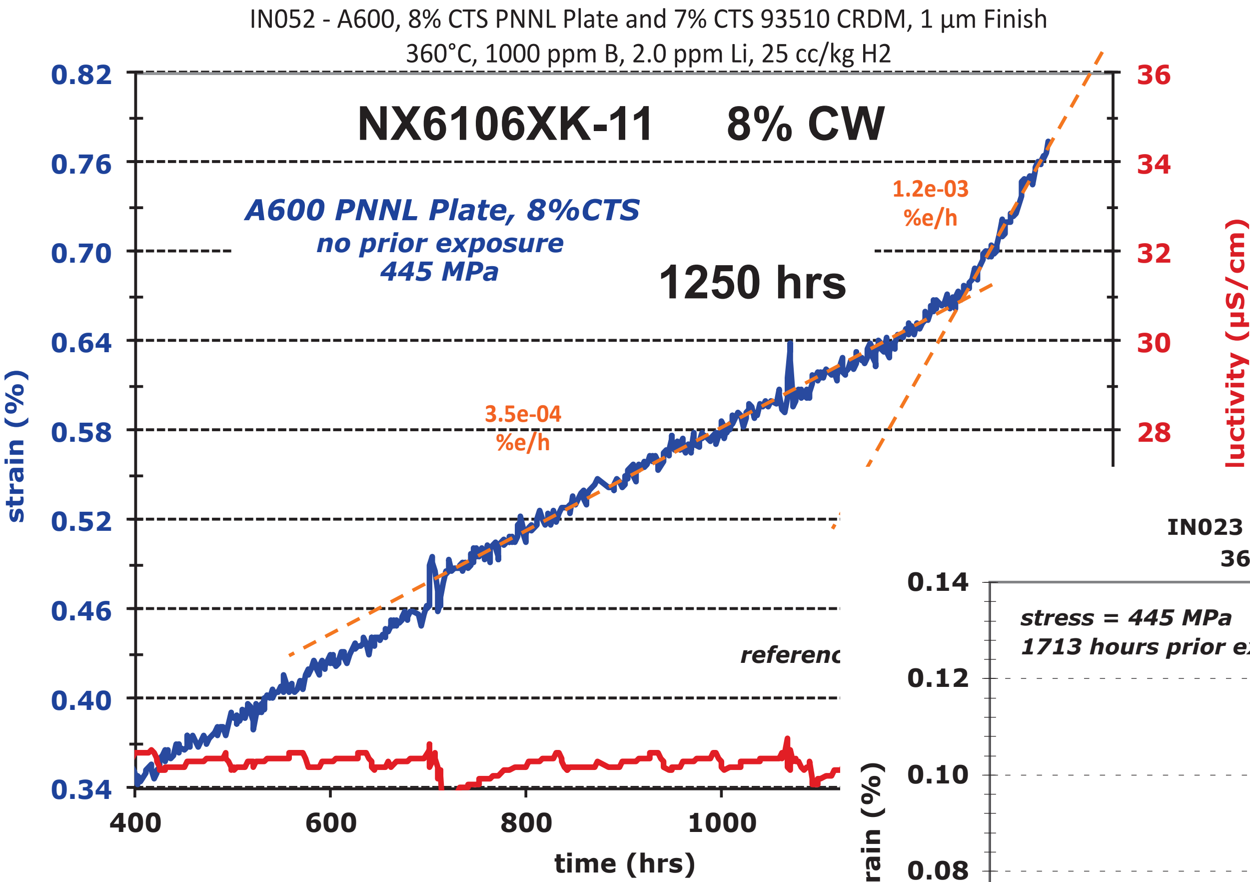
Response of Highly Cold-Worked Material

- *Highly cold-worked material shows a smooth but rapid transition to a higher indicated DCPD or strain slope over 2-4 days.*



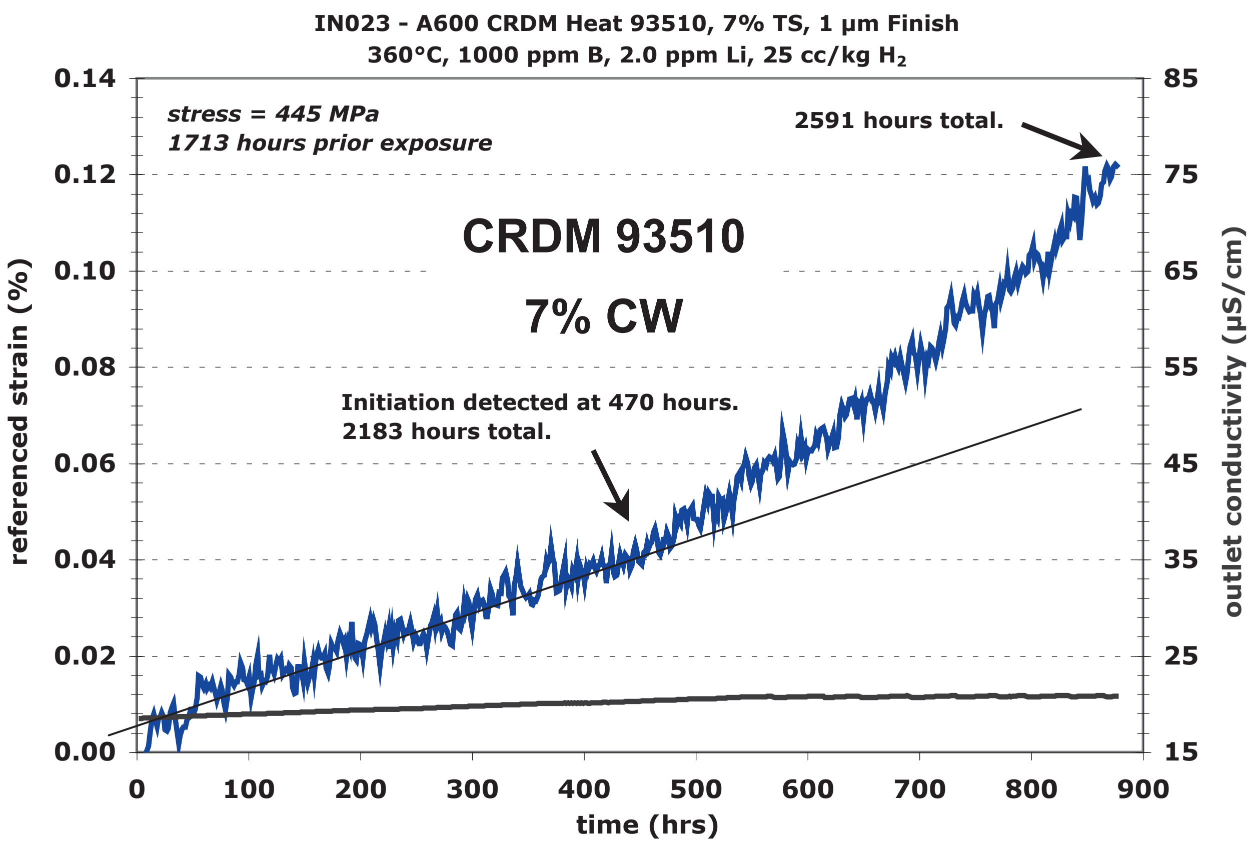


Response of Moderately Cold-Worked Material



Moderately cold-worked materials exhibit a transition to higher indicated strain rate over many days.

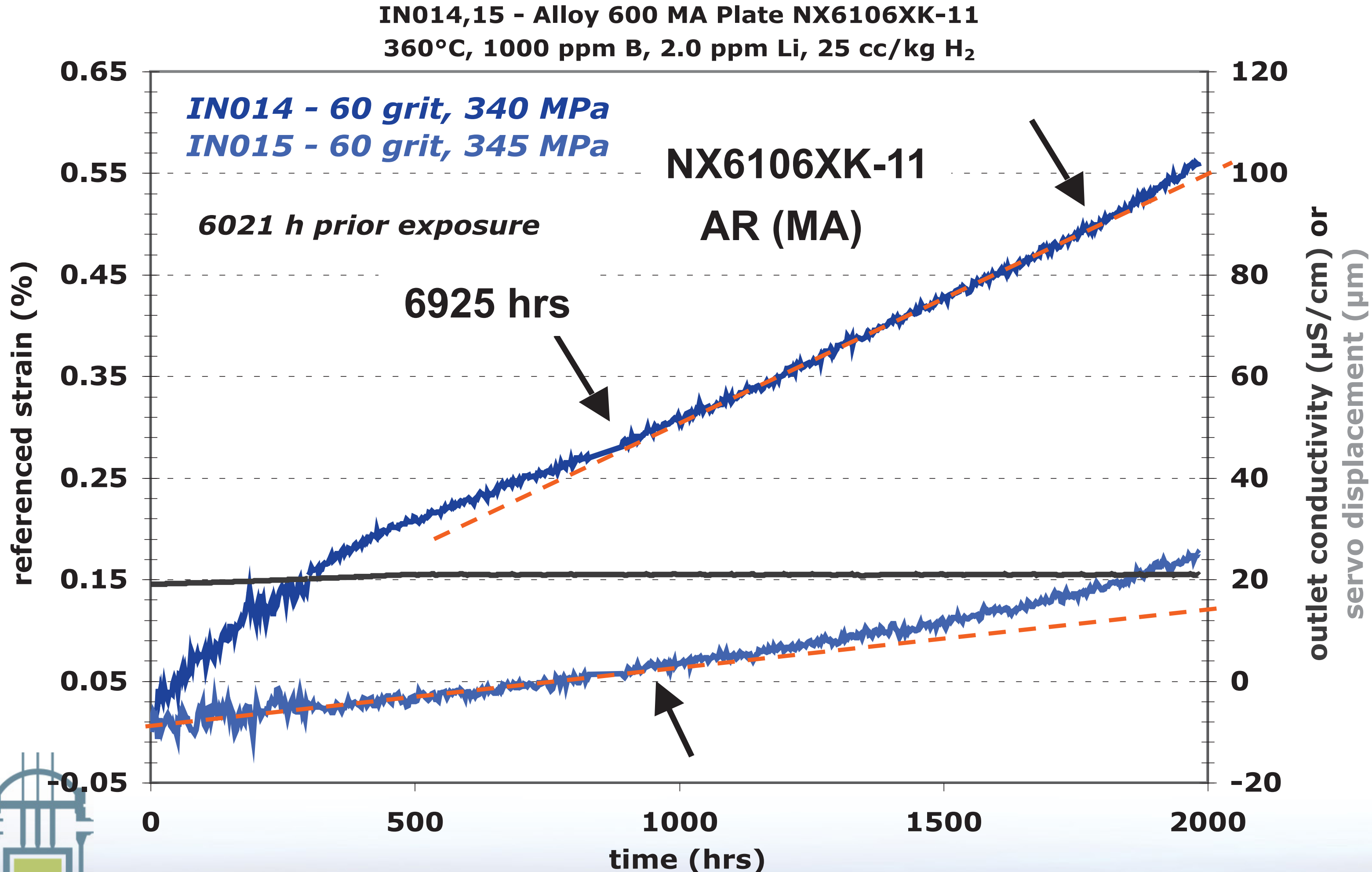
Slower evolution likely a combination of susceptibility and applied stress.



LWRS
Light Water Reactor Sustainability

As-Received, Non-Cold Worked Material

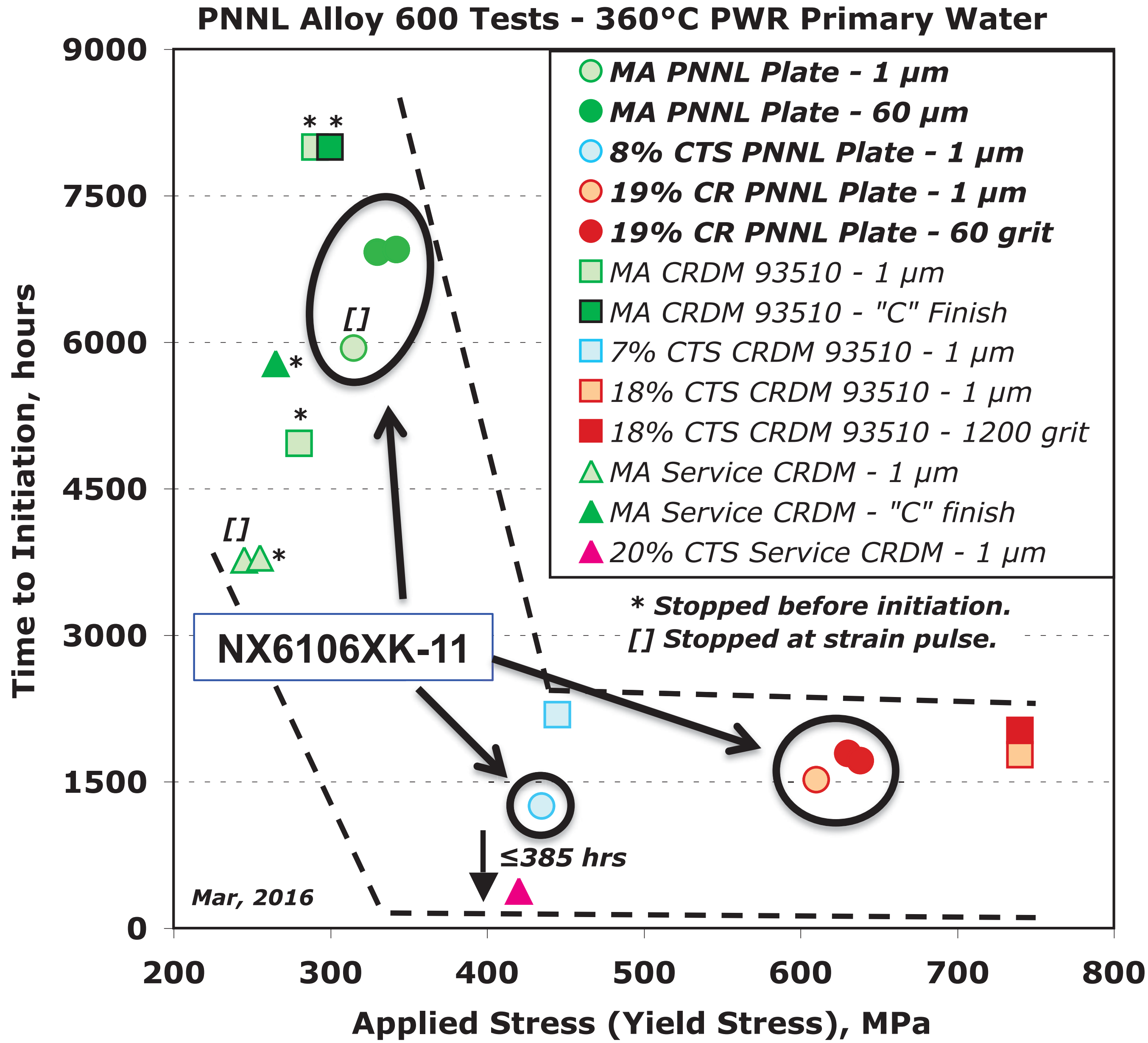
- *Transition to higher indicated slope is even more gradual in non-CW, low strength materials. Similar variability in SCC initiation response with CW/strength observed for other alloy 600 heats.*



Light Water Reactor Sustainability

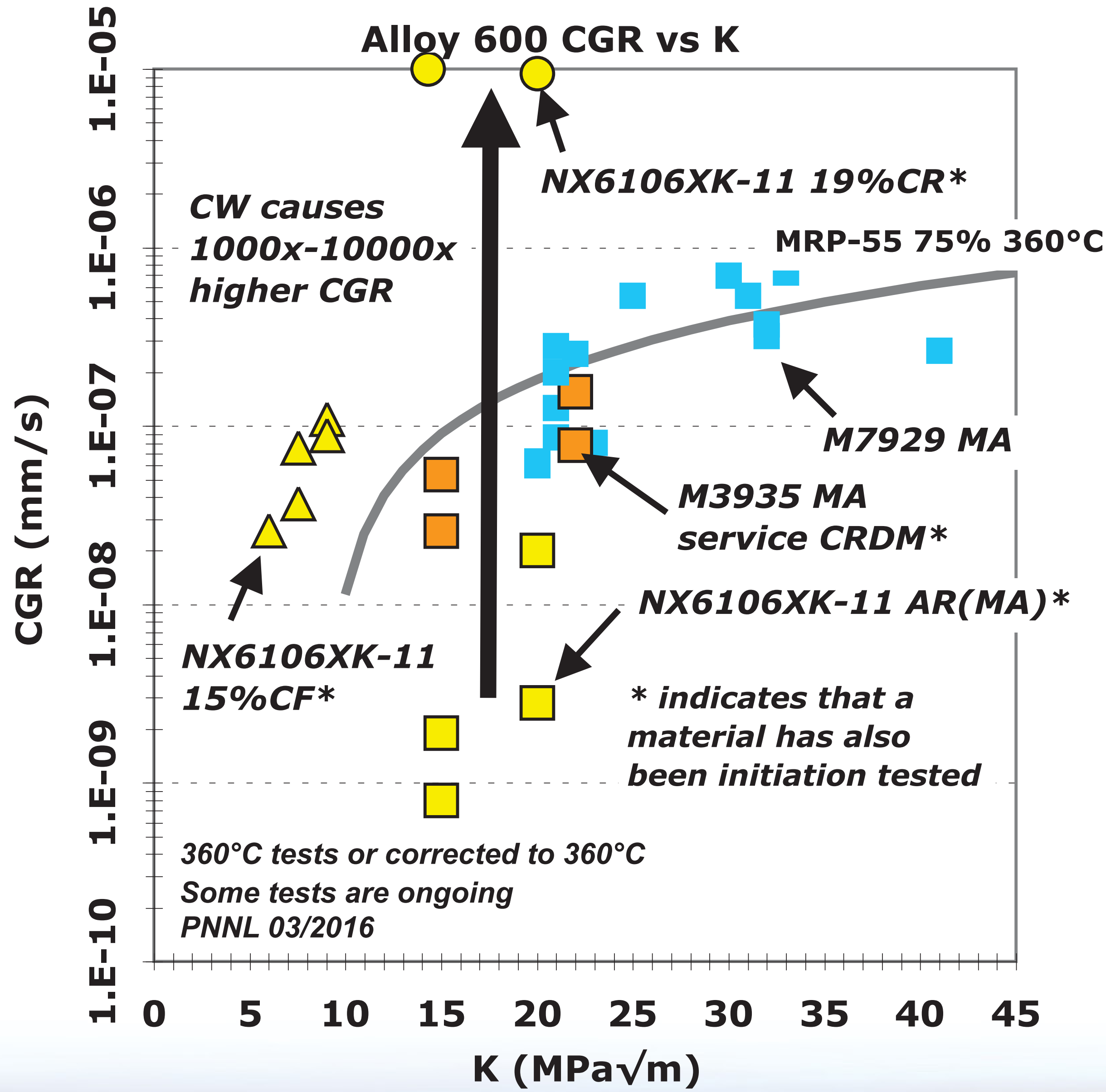
Summary of Prior Response vs Yield Stress

- *Small amounts of cold work cause large reductions in SCC initiation time.*
- *These prior results indicate initiation times of 1000-2000 hours for cold-worked NX6106XK-11.*
- *Microstructure exams of tested specimens revealed equiaxed grain structures for NX6106XK-11.*



Effect of Cold Work on Low K SCC Growth Rates

- *Effect of CW: Preliminary results indicate ≥1000x higher CGR for 19%CR material.*
- *Additional testing started on 15%CF to better establish low K response.*
- ***Difference between CW and non-CW CGR is consistent with strong difference in initiation time between CW and non-CW.***



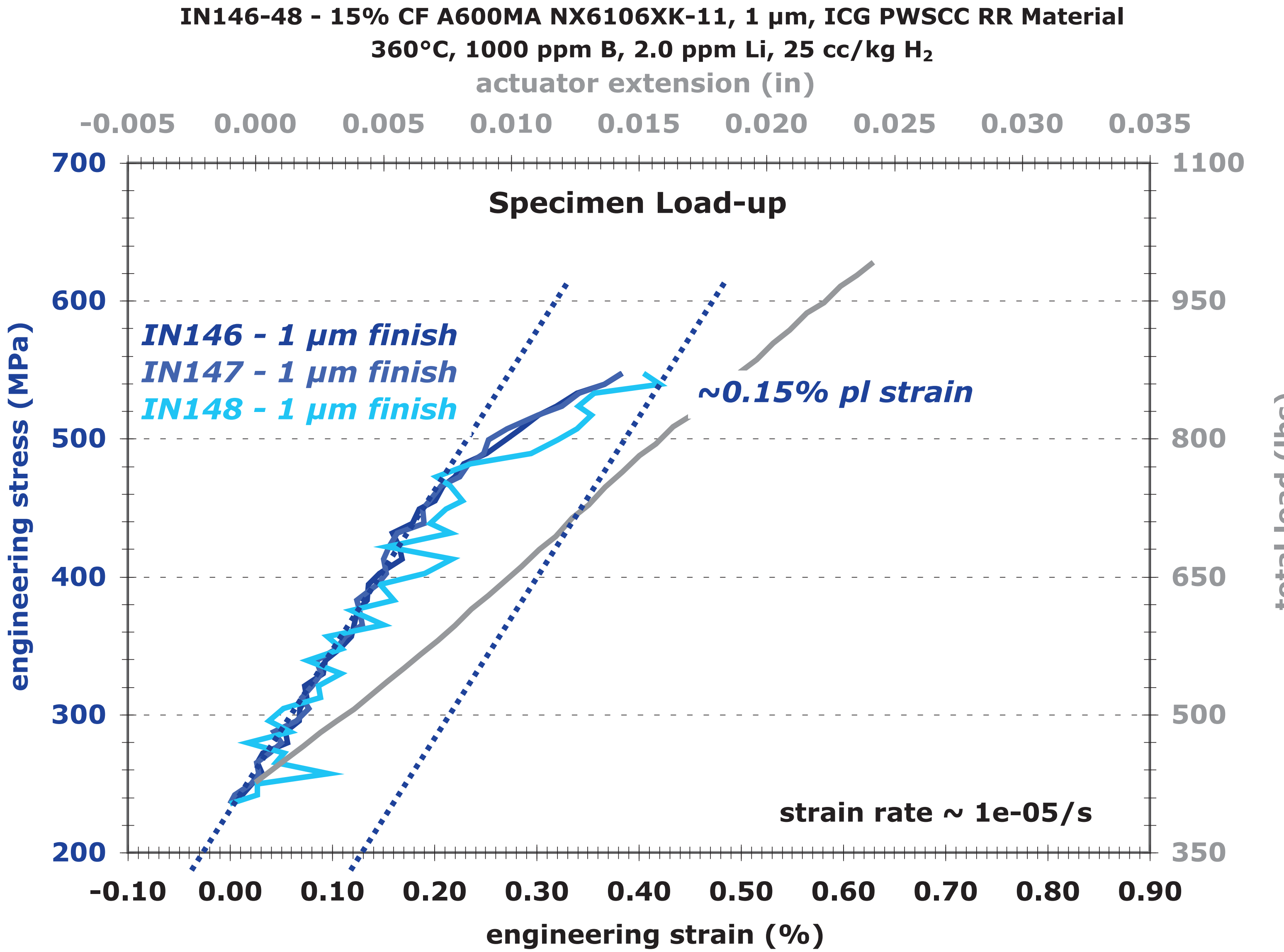
LWRS
Light Water Reactor Sustainability

Presentation Overview

- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- *NX6106XK-11 Material Characterization*
- *Material Preparation and Specimen Fabrication*
- *Prior NX6106XK-11 SCC Initiation Test Results*
- ***Round Robin NX6106XK-11 Test Results***
- *Round Robin 31907 Test Results*
- *Summary*

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.*

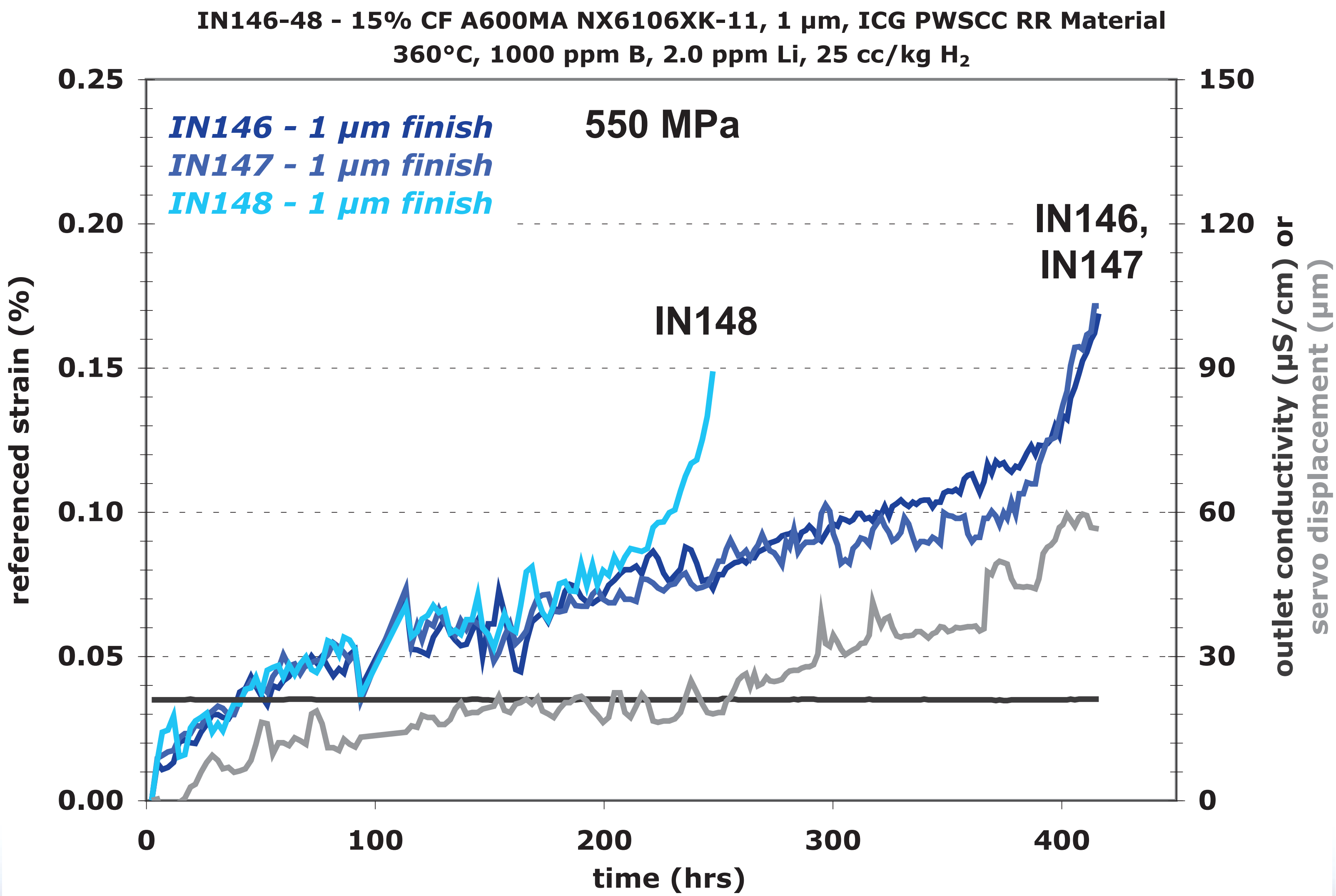


Initiation of NX6106XK-11 Round Robin Specimens

- *Well behaved response, but unexpectedly low SCC initiation time for all three specimens.*

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

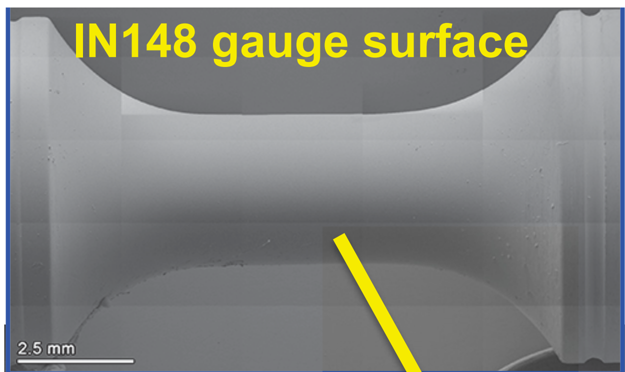
- *Well below prior tests on this plate heat in the cold-worked condition.*



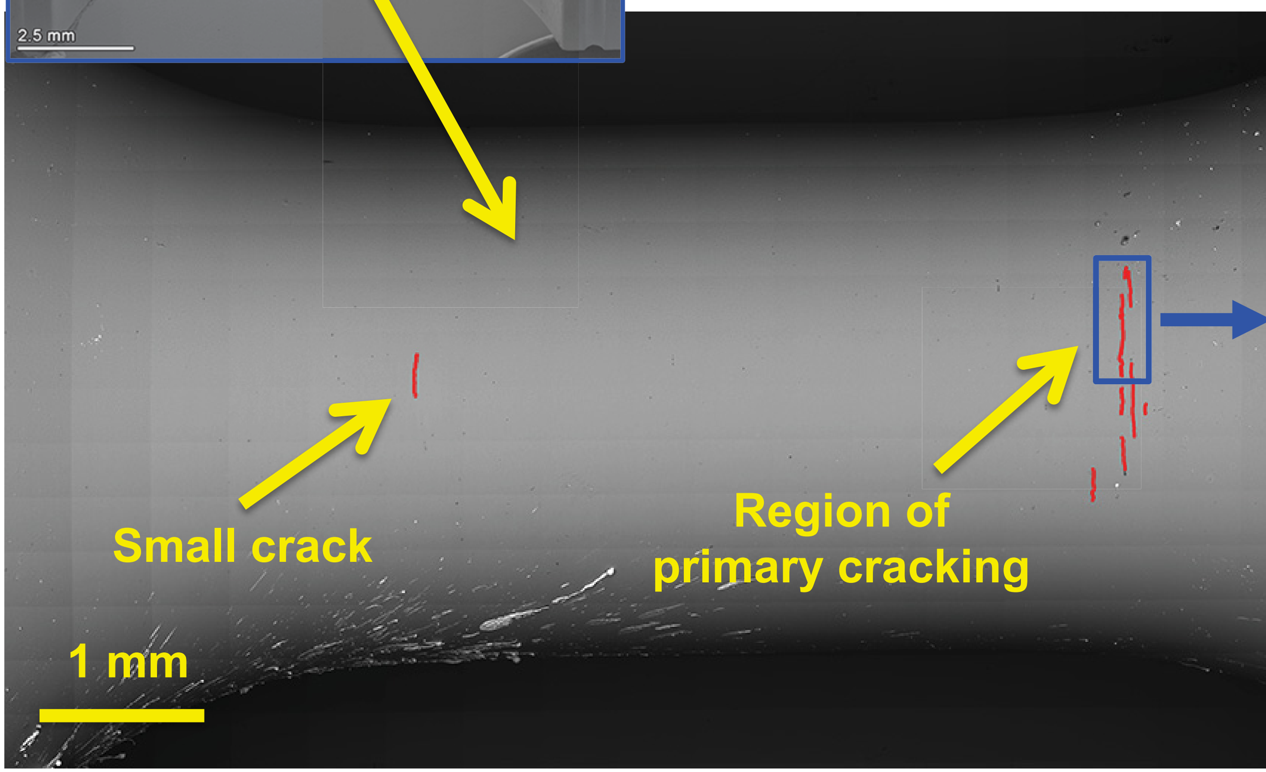
LWRS
Light Water Reactor Sustainability

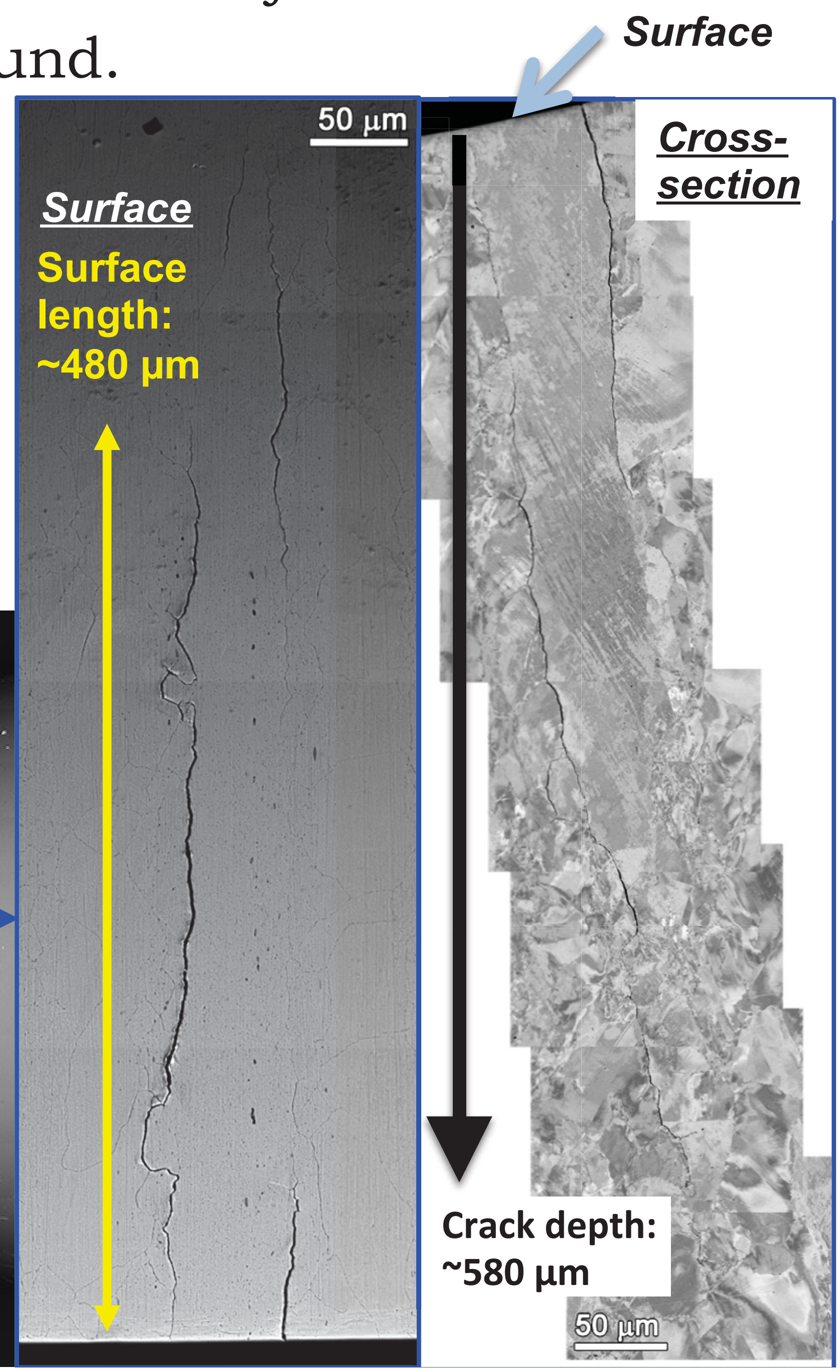
Observations of IN148 (219 h Initiation)

- *Initiation at 219 hours. Test stopped at 250 hours for examinations.*
- Region of primary cracking was easily found.
- *Primary crack had surface length of ~480 μm and depth of ~580 μm.*



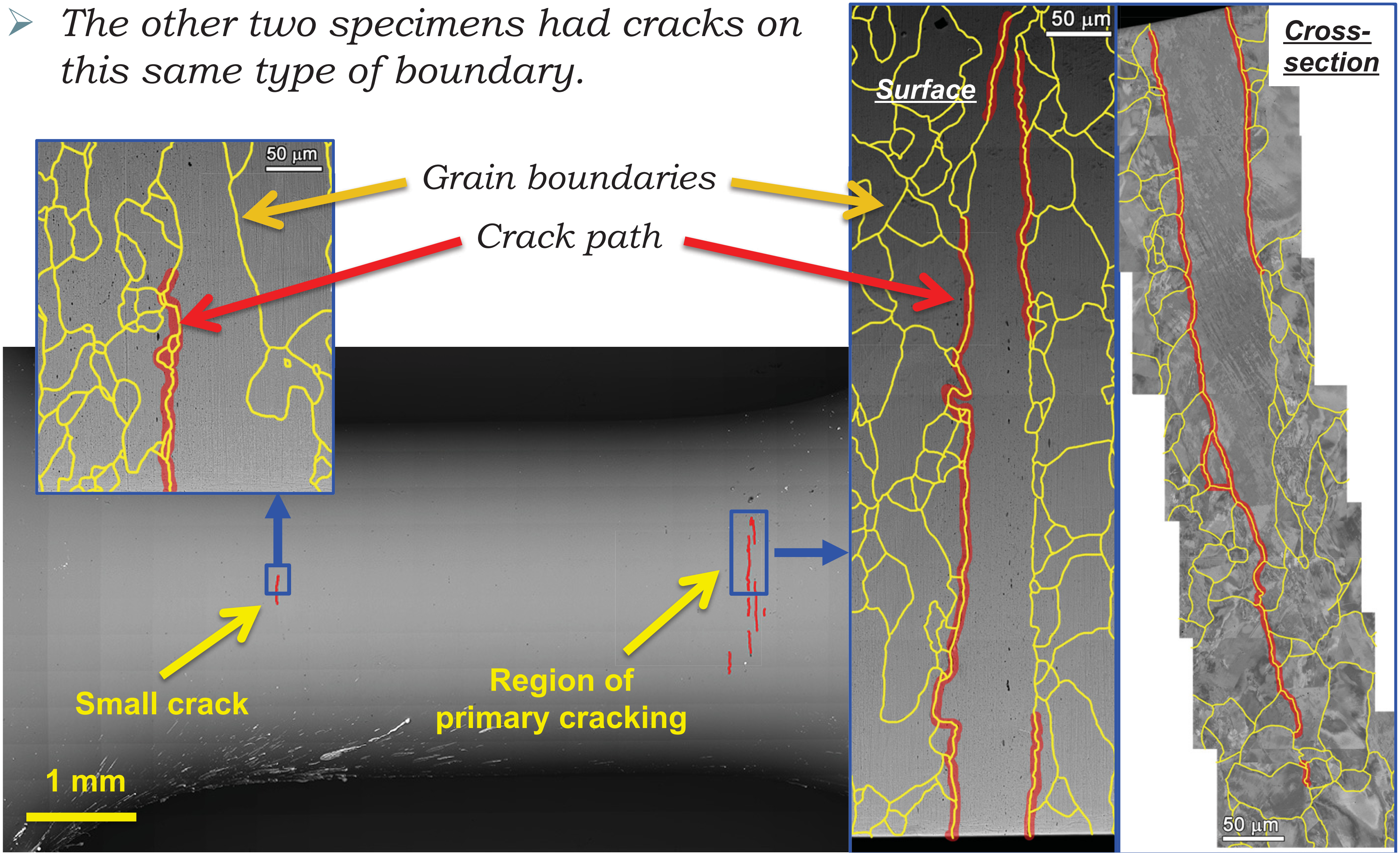
SEM surface and cross-section examinations





Observations of IN148 (219 h initiation)

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

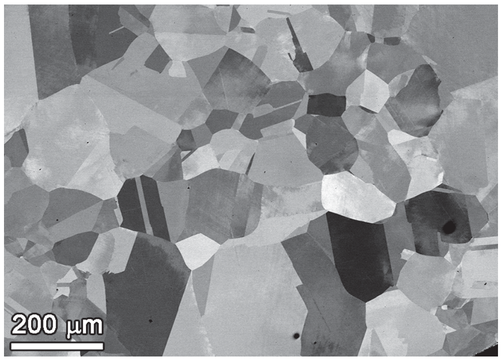


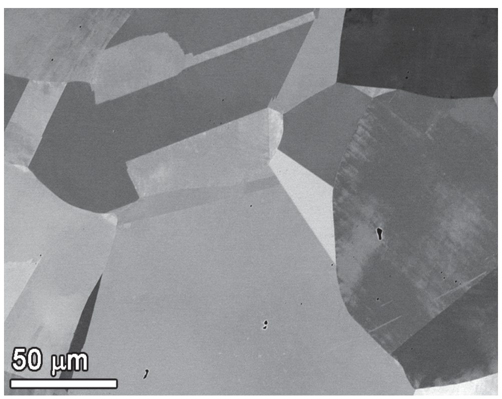
Presentation Overview

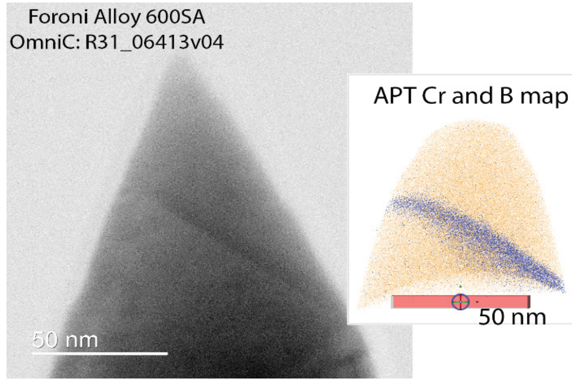
- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- *NX6106XK-11 Material Characterization*
- *Material Preparation and Specimen Fabrication*
- *Prior NX6106XK-11 SCC Initiation Test Results*
- *Round Robin NX6106XK-11 Test Results*
- ***Round Robin 31907 Test Results***
- *Summary*

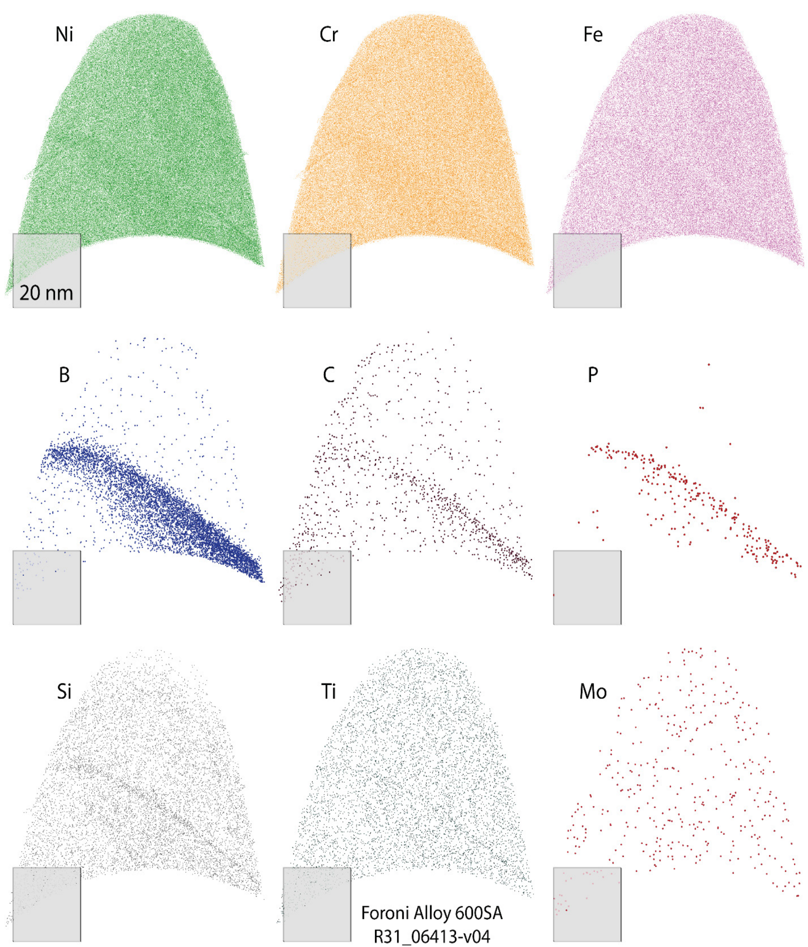
Microstructure and Grain Boundary Composition in Solution-Annealed Alloy 600 Heat 31907

- *Large grain size ranging from ~50-300 µm*
- *Clean grain boundaries*

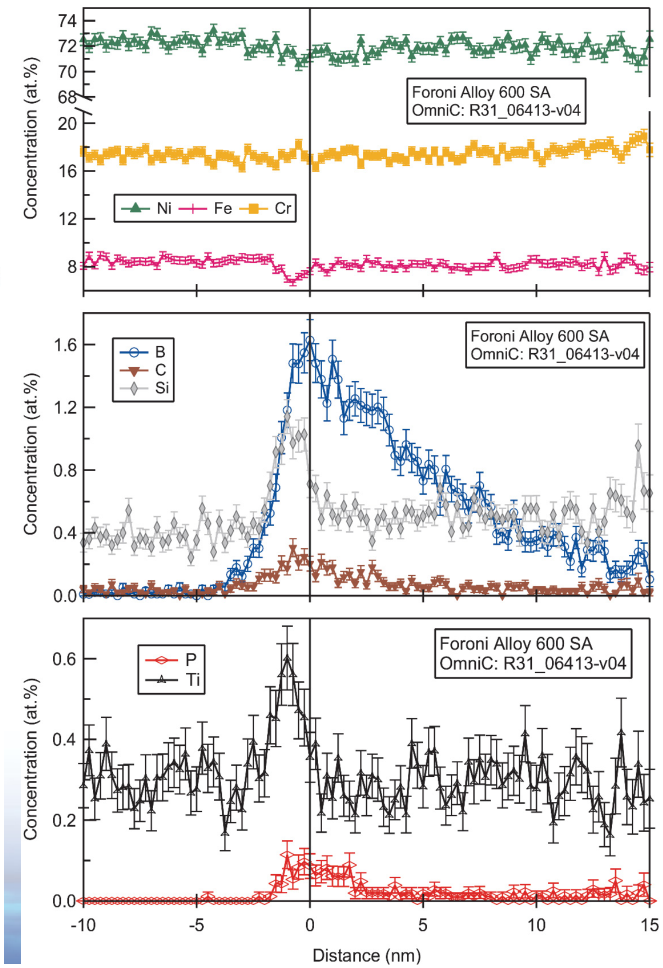








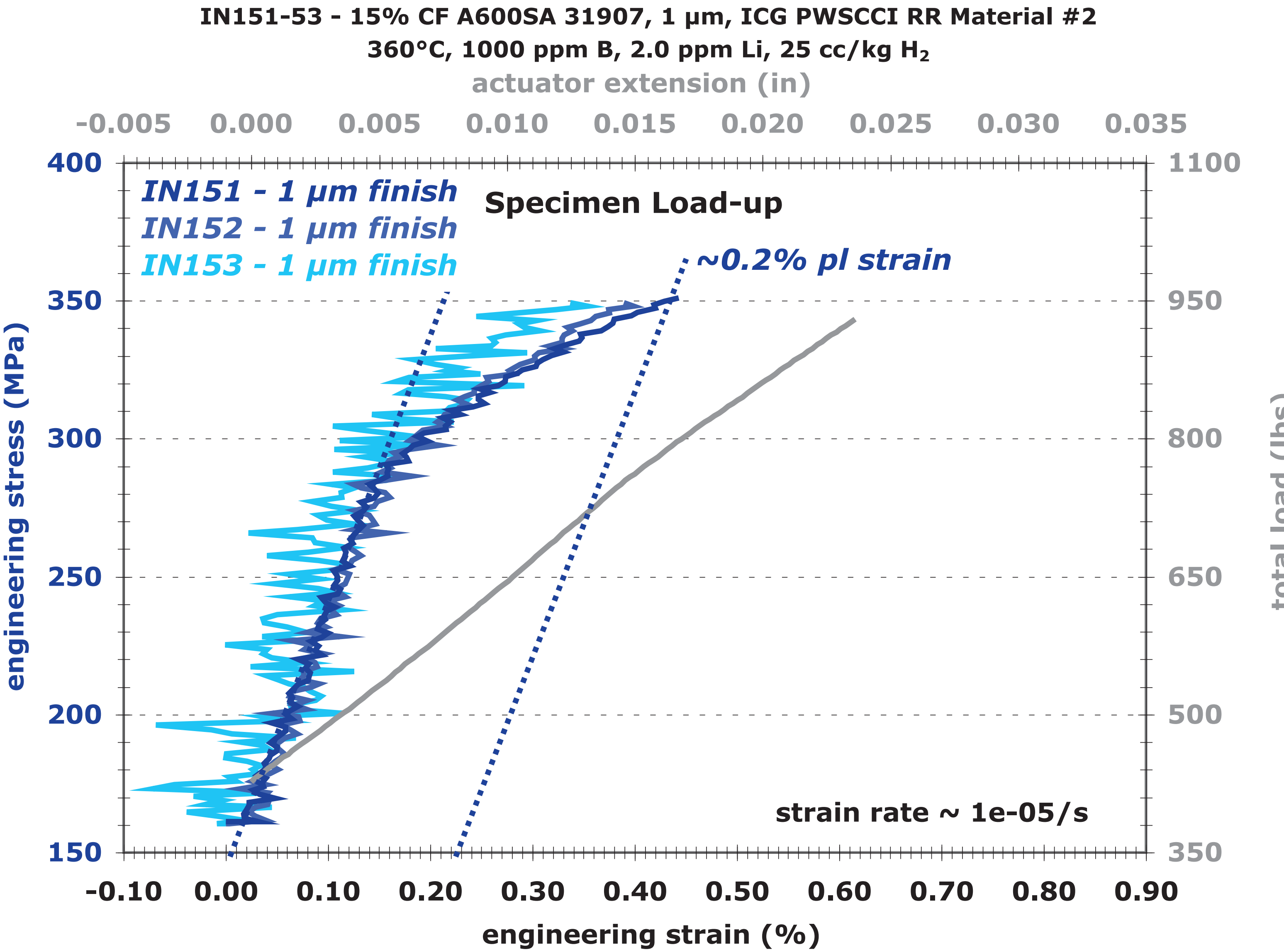
APT revealed boron grain boundary segregation to 1.8 at%, minor Si, Ti and P.



Light Water Reactor Sustainability

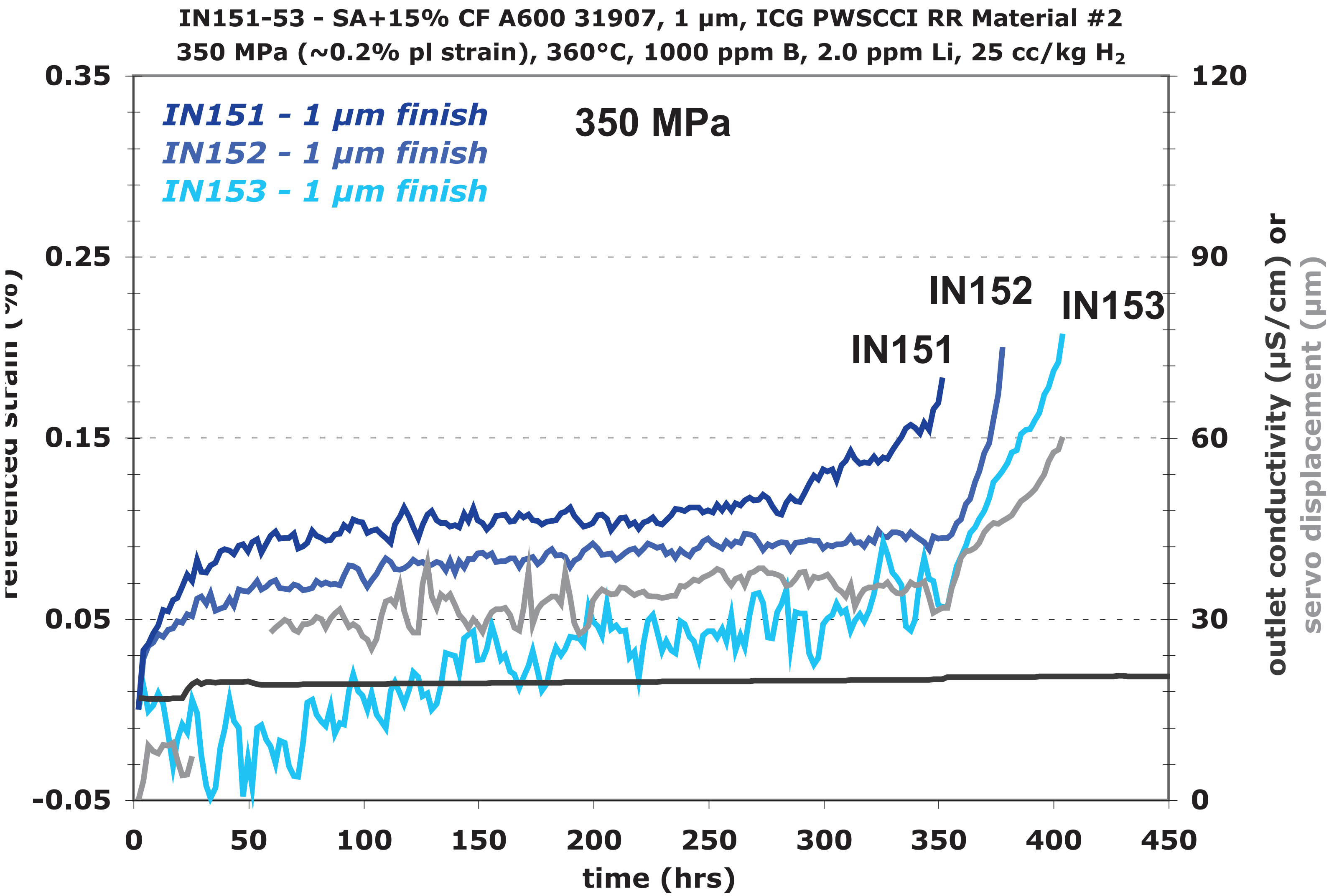
Load-up of 31907 Round Robin Specimens

- *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.*

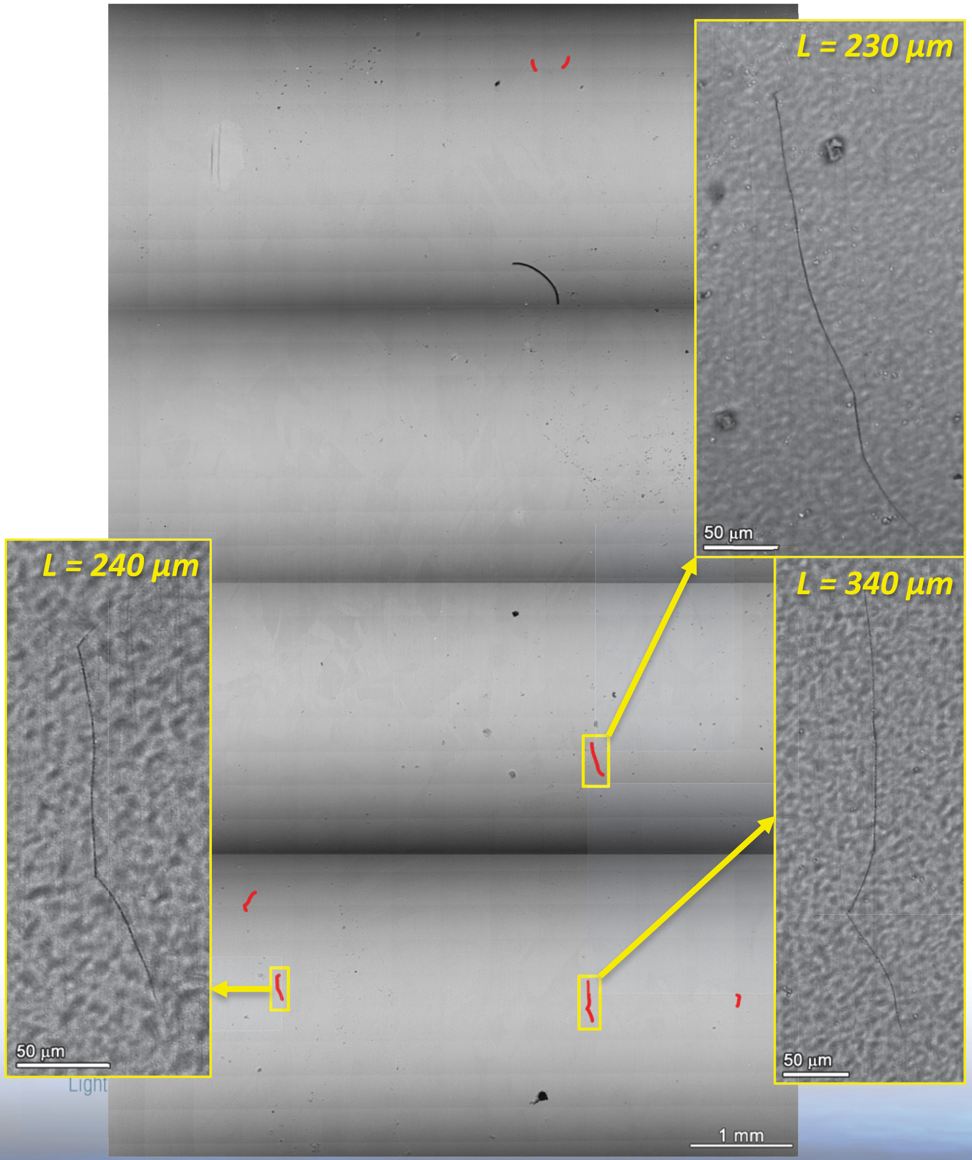


Light Water Reactor Sustainability

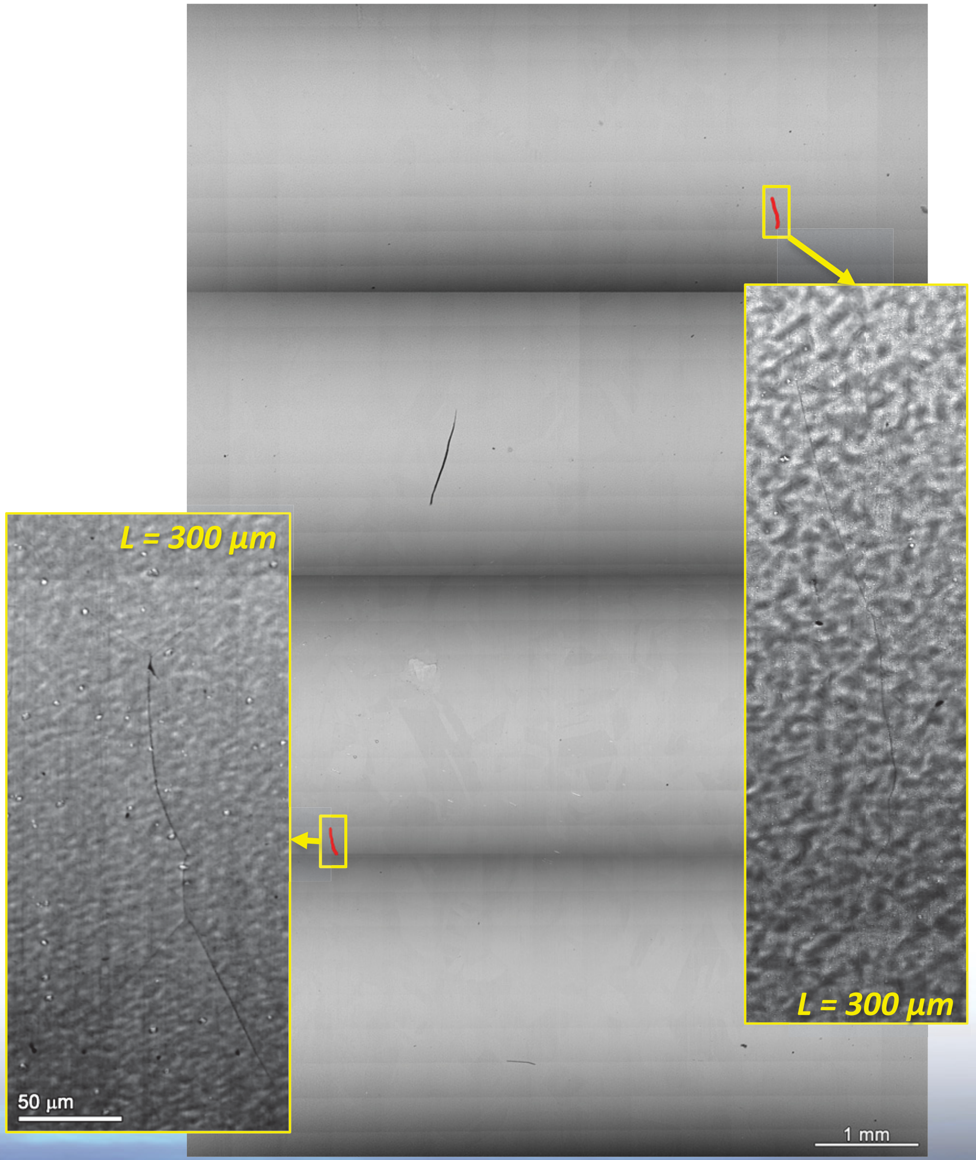
Examination of 31907 Round Robin Specimens

- *SEM examinations of entire gauge surface performed after SCC initiation of IN151, IN152 initiated soon after reloading.*

IN151 (initiated)



IN152 (just prior to initiation)



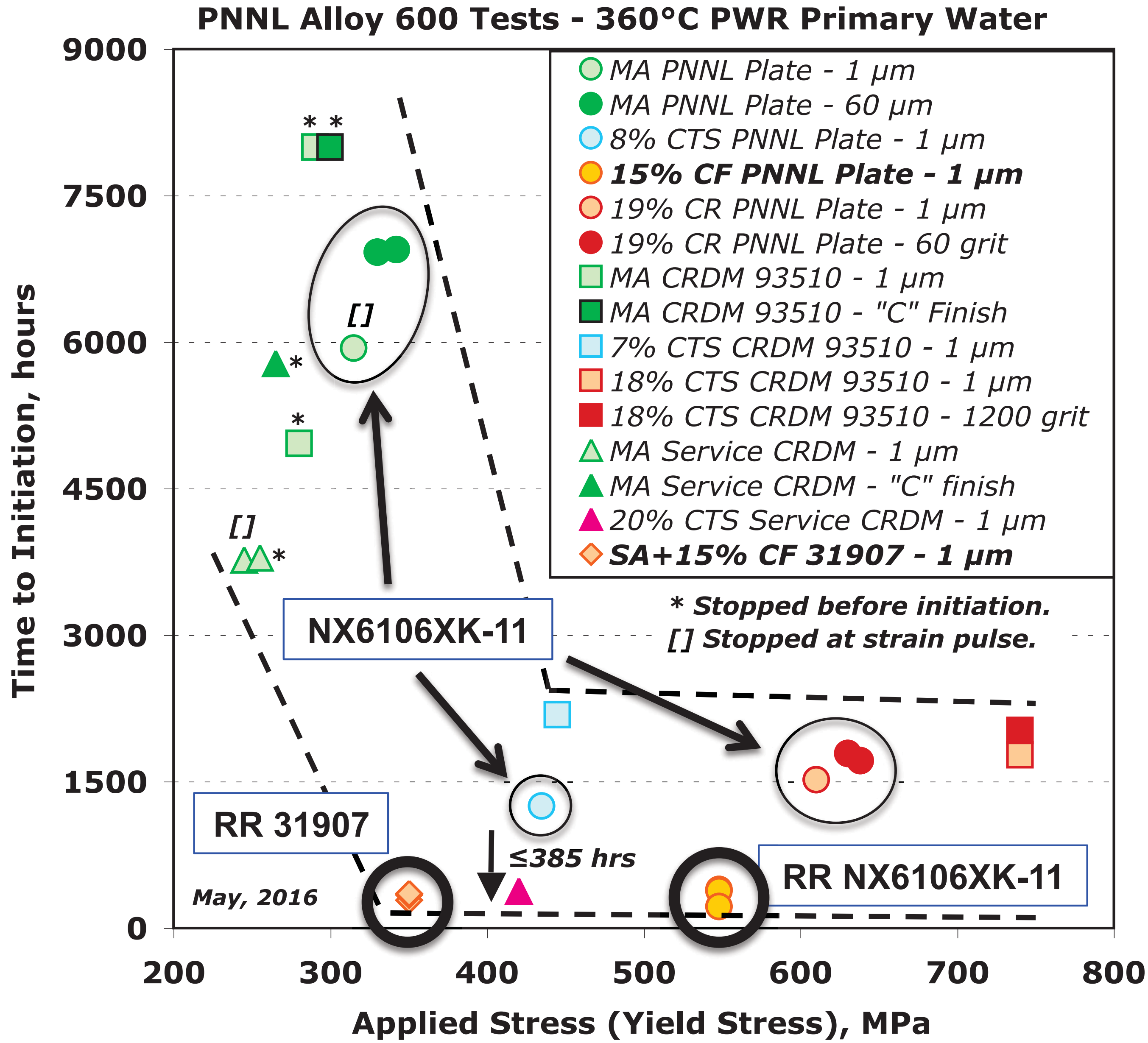
Presentation Overview

- *ICG-EAC Round Robin Overview*
- *PNNL SCC Initiation Testing Capability*
- *NX6106XK-11 Material Characterization*
- *Material Preparation and Specimen Fabrication*
- *Prior NX6106XK-11 SCC Initiation Test Results*
- *Round Robin NX6106XK-11 Test Results*
- *Round Robin 31907 Test Results*
- ***Summary***

LWRS Light Water Reactor Sustainability

Round Robin Response vs PNNL Experience

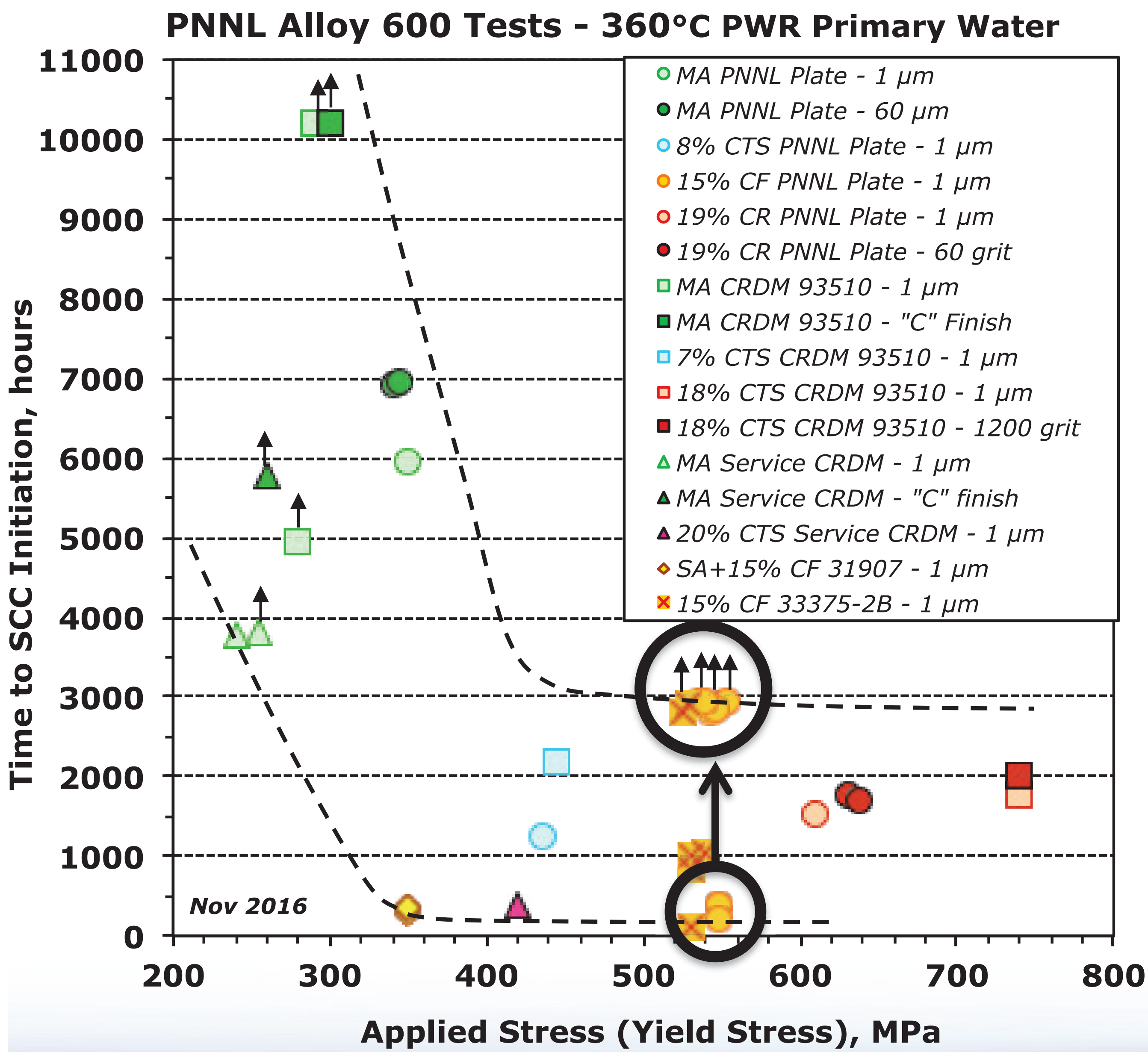
- *NX6106XK-11: lower RR initiation times than previous tests may result from differences in microstructure, cracking observed along large elongated grains in RR tests.*
- *31907: low initiation times at lower stress, comparable to PNNL results on alloy 600 service CRDM. Boron grain boundary segregation and large grain size observed for both materials.*



Round Robin Response vs PNNL Experience

- *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 revealing longer crack initiation times for 15%CF PNNL Plate.**

- *31907: low initiation times at lower stress, comparable to PNNL results on alloy 600 service CRDM. Boron grain boundary segregation and large grain size observed for both materials.*



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.*
    - *Confirmation of having reached yield stress.*
    - *Documentation of exact level of applied plastic strain.*
- *Primary (NX6106XK-11) material 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) RR material 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.*

LWRS

Light Water Reactor Sustainability

Grain Boundary Damage Evolution and SCC Initiation in Cold-Worked Alloy 690 Exposed to High-Temperature PWR Primary Water

Ziqing Zhai, Matt Olszta, Karen Kruska, Dan Schreiber, Mychailo Toloczko and Steve Bruemmer

Pacific Northwest National Laboratory

EPRI Alloy 690/52/152 Primary Water Stress Corrosion Cracking Research Collaboration Meeting

Light Water Reactor Sustainability R&D Program

November 29, 2016

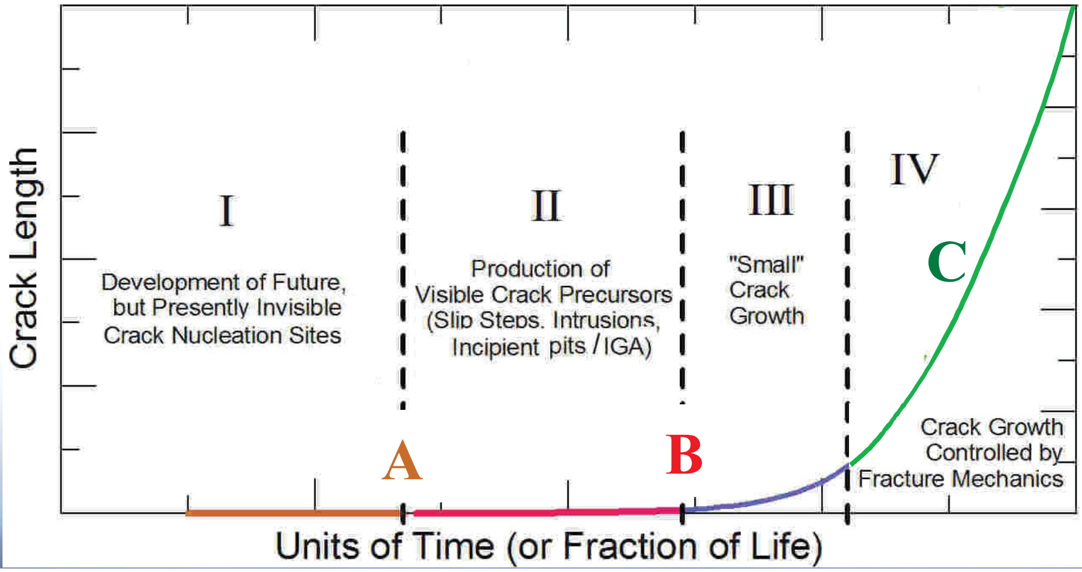
Research Objectives and Approach

- *To identify mechanisms controlling SCC initiation of corrosion-resistant nickel-base alloys under realistic LWR service conditions, with a focus on:*
  - *Early degradation precursors formation and growth (**A**)*
  - *Small crack nucleation and coalescence (**B**)*
  - *Transition to rapid growth stage (**C**)*
- *To investigate important influencing factors on the susceptibility to localized corrosion/oxidation and SCC initiation:*
  - *Material: composition, microstructure and surface condition*
  - *Environmental: water chemistry, temperature*
  - *Stress and strain: cold work, applied stress*

SCC initiation testing with in-situ monitoring

+

High resolution microscopy on localized damage and oxidation precursors at nano-to-micro scale



Stress Corrosion Crack Initiation Testing in LWR Environments at PNNL

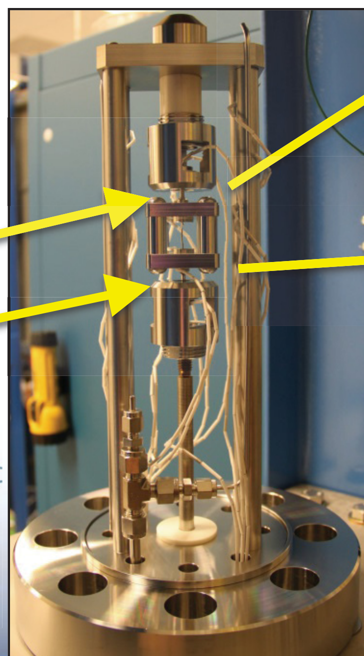
SCC initiation test systems assembled with in-situ DCPD crack detection.

-- Two LWRS high-temperature, high-pressure autoclave systems for 2-6 fully instrumented specimens and one LWRS system for 36 tensile samples with up to 20 instrumented.

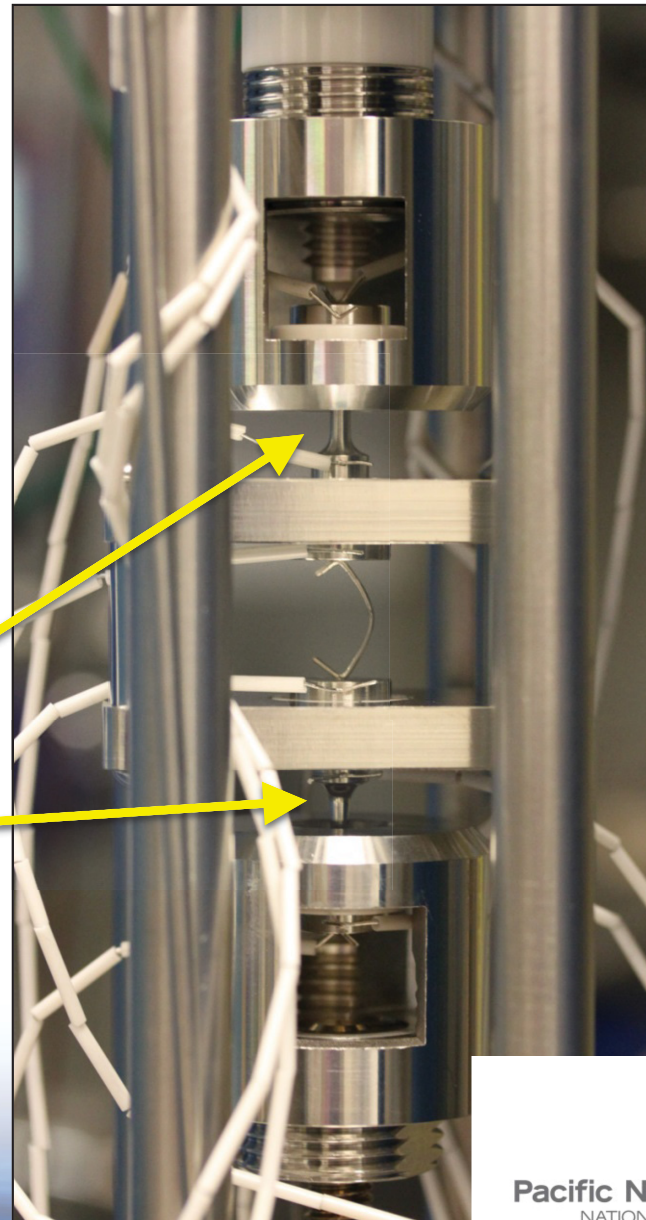
-- Two NRC/EPRI 36 tensile systems operating, three NRC systems being set up for 27 bent beam samples.

1.2" Tall SCC Initiation Specimen

2-6 Specimen SCC Initiation System



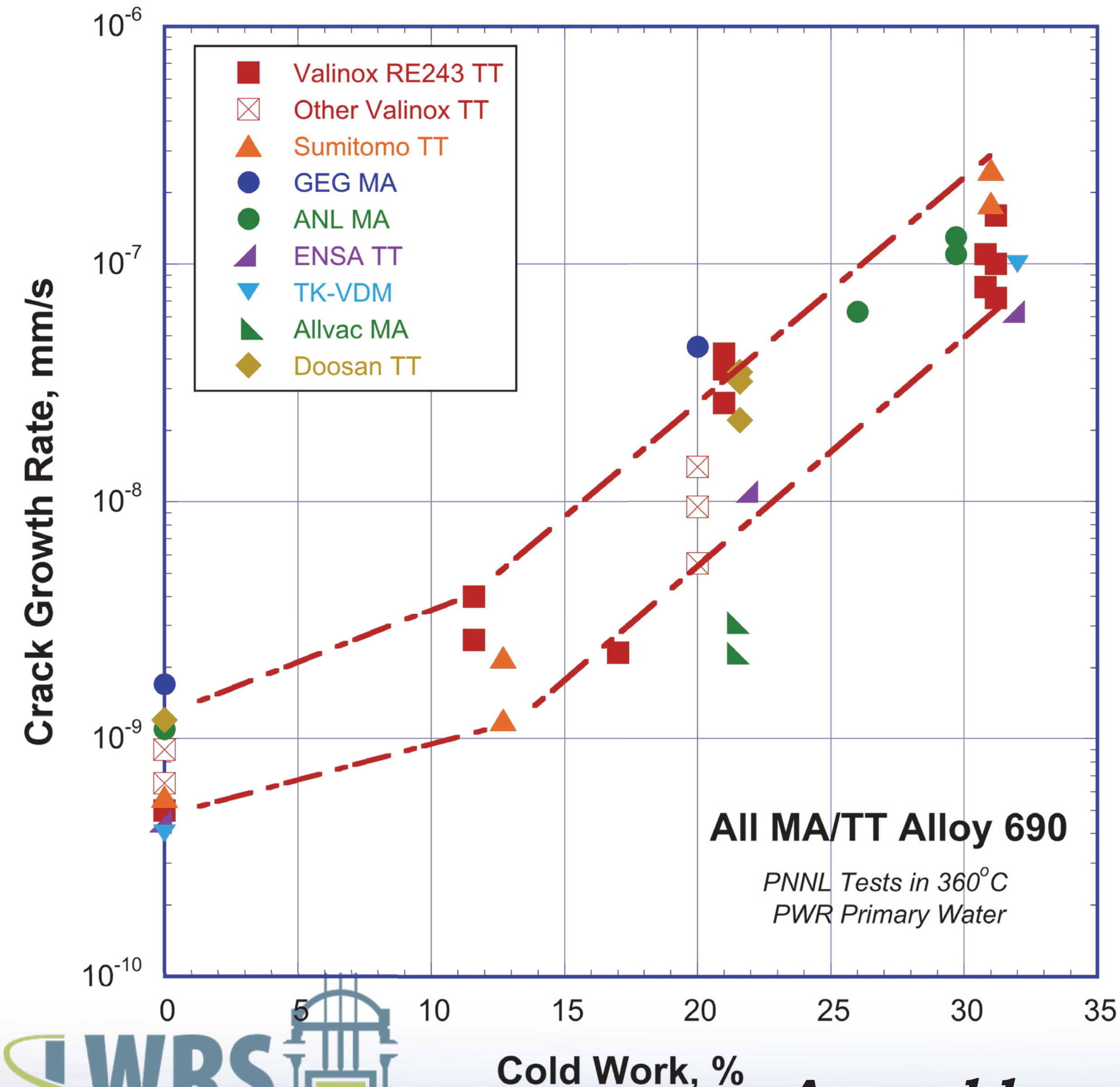
2-6 Specimen SCC Initiation System



36 Specimen SCC Initiation System

Pacific Northwest
NATIONAL LABORATORY

Summary of PNNL Alloy 690 Measurements of SCC Growth Rates



Consistent increase in measured SCC growth rates as a function of cold work for alloy 690 materials in the as-received MA or TT condition.

Data on these heats suggest a transition in SCC susceptibility for materials cold worked to >15% reduction. High SCC growth rates for many heats at ~30% cold work.

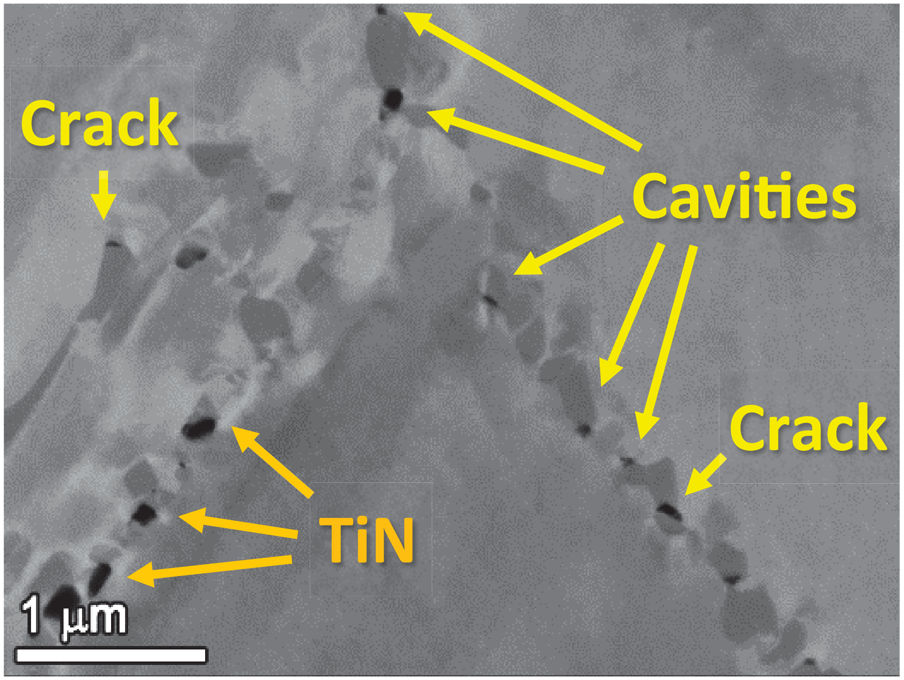
Are cold-worked alloy 690 materials susceptible to SCC initiation?

LWRS Light Water Reactor Sustainability

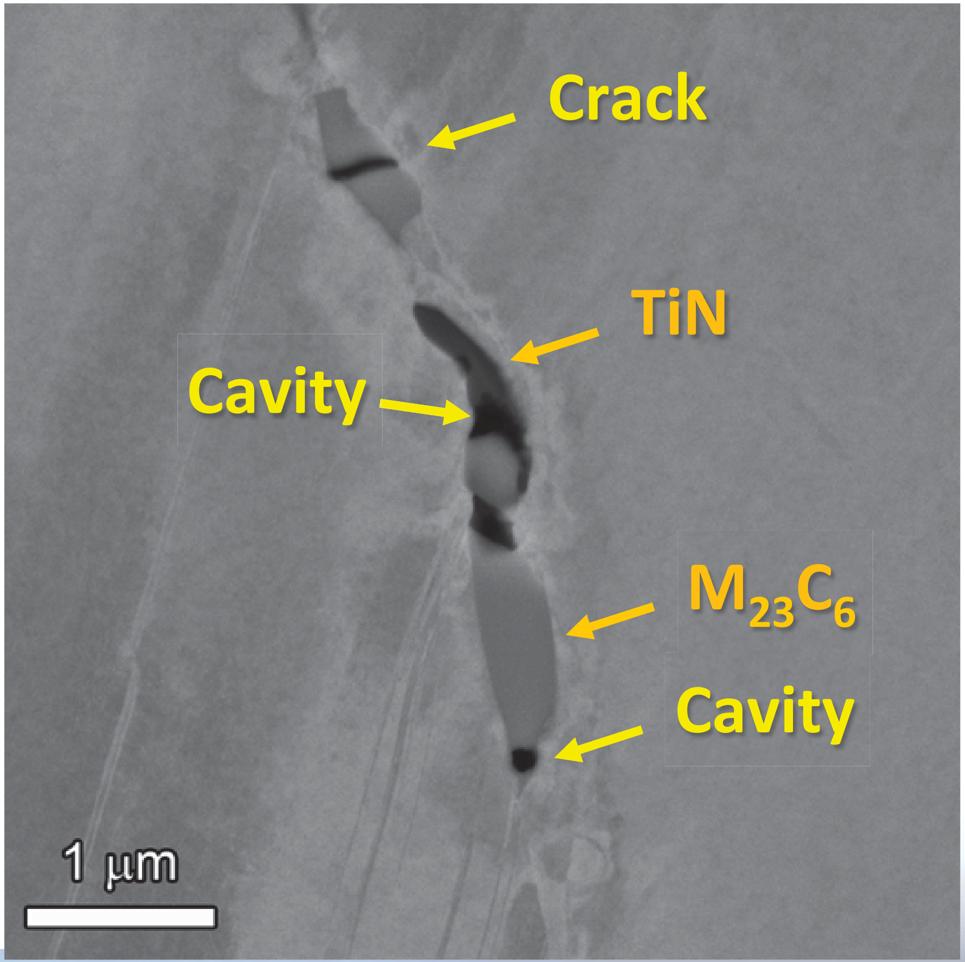
Cold-Worked Alloy 690 Materials

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

Valinox CRDM RE243 TT + 31%CF



ANL bar MA + 26%CR



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

Cold-Worked Alloy 690 Materials

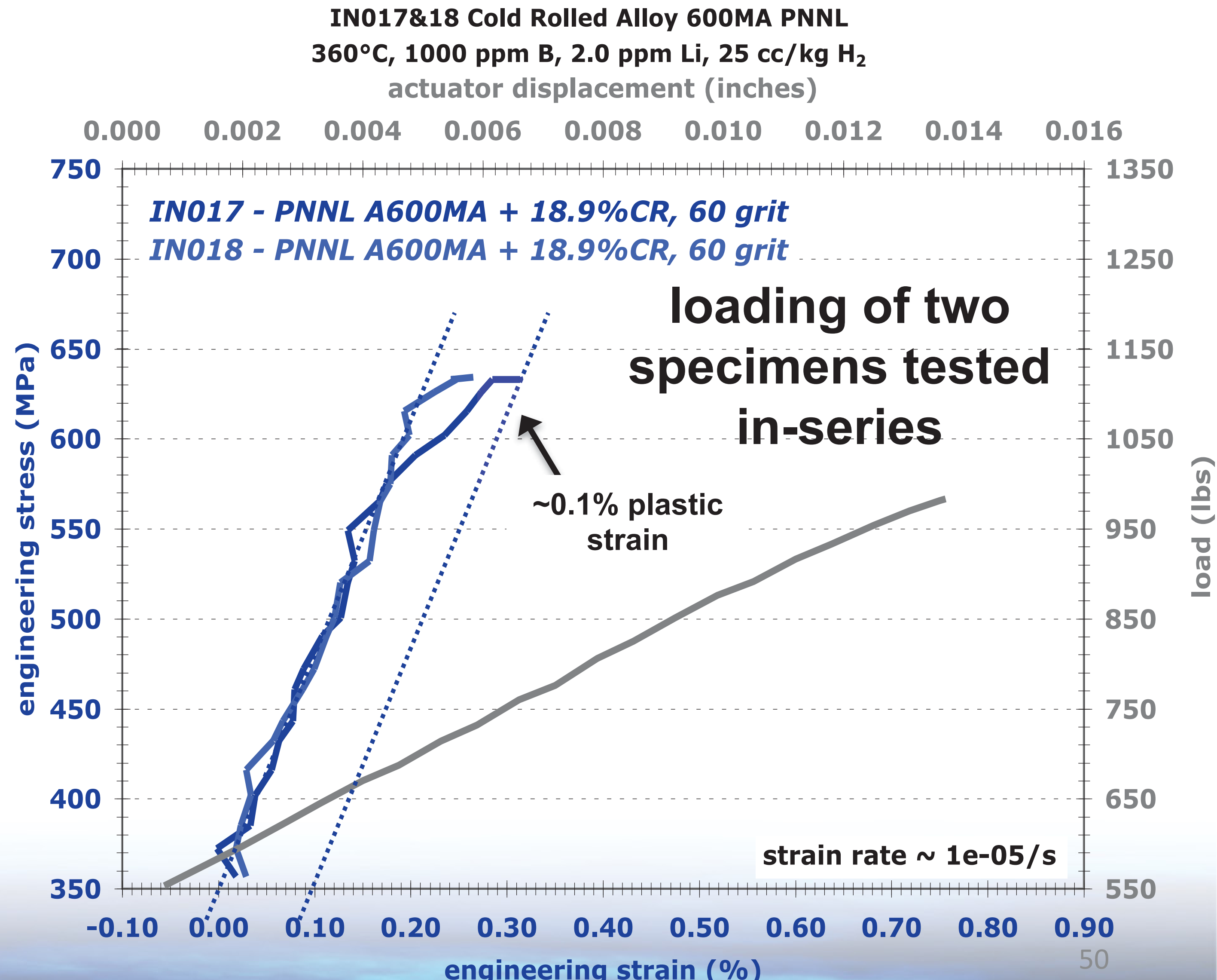
- *Six alloy 690 CRDM TT and plate MA/TT heats with cold work levels of ~12%, 21% and ~31%.*
- *SCC CGR data available for every material + cold work combination. All highly cold-worked materials exhibited IGSCC susceptibility.*

Material	AR GB Carbide Microstructure		Cold Work Induced GB Damage	
	Location, Size	Density, Spacing	IG Cavity Density	Density of Cracked GB Precipitates
Valinox CRDM RE243 31%CF	IG, 50–200 nm	Semi-continuous, spacing: ~100 nm	Moderate	Low
Sumitomo CRDM E67074C 31%CF	IG, 50–200 nm	Semi-continuous, Spacing: ~100 nm	Moderate	Low
Doosan CRDM 133454 31%CF	IG, 1–5 μm	Semi-continuous, Spacing: 0.5–2 μm	Low-Moderate	Moderate
TK-VDM Plate 114092 32%CF	IG, 50–200 nm	Semi-continuous, spacing: 0.2–0.5 μm	Moderate	Low
ANL Bar MA 26%CR	IG, 0.5–3 μm	Semi-continuous, spacing: 0.2–2 μm	Moderate-High	Moderate

High levels of cold work induced the formation of small IG cavities and cracked carbides in certain heats. The damage "density" was estimated as high when typical spacing was <1 μm, moderate from 1-10 μm and low >10 μm. This spacing is generally larger than the distance between IG carbides.

SCC Initiation Test Conditions

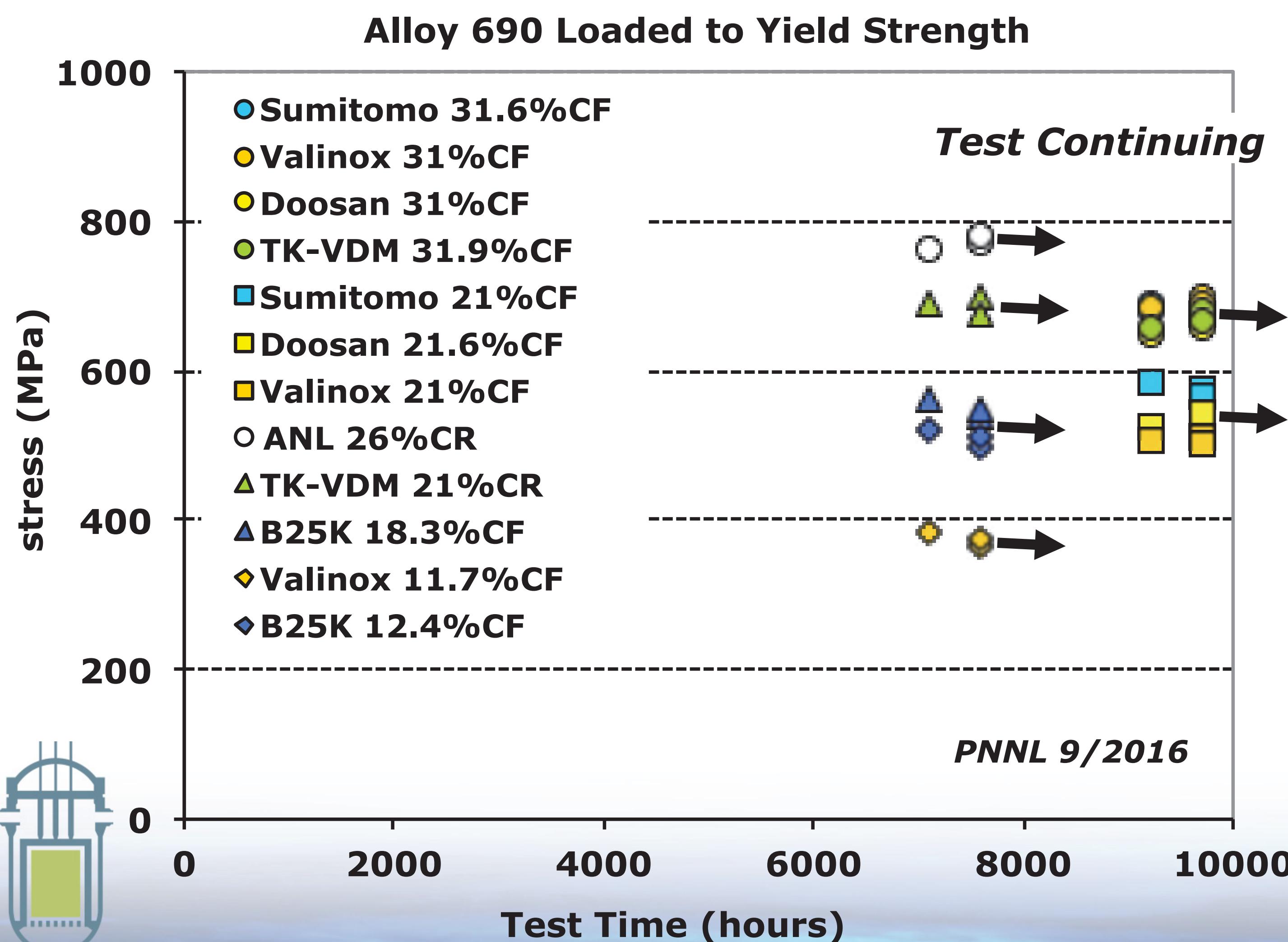
- *Simulated PWR primary water – 1000 ppm B, 2 ppm Li, 360°C, 25 cc/kg H_2 (Ni/NiO stability line).*
- *All specimens loaded to their yield strength.*
- *Yield observed by monitoring stress (from load) versus strain (from DCPD) at a displacement rate of ~$1x10^{-5}$ s^{-1} (~1 hour to load).*
- *For multi-specimen tests, goal is to limit plastic strain during loading to less than ~0.5%.*



Light Water Reactor Sustainability

Status of 36 Specimen SCC Initiation Test on Cold Worked Alloy 690

- *Constant load test at yield stress in 360°C simulated PWR primary water.*
- *No evidence for crack initiation from DCPD.*
- *Specimens were examined in SEM before restart of the test.*



LWRS Light Water Reactor Sustainability

Constant Load SCC Initiation Tests

Constant Load Alloy 690 SCC Initiation Tests in LWRS System 1: First Set of 21 Specimens after ~5750, 7050, 9220 hour Exposure Time

Specimen	Material	Heat Number	Material Condition	Surface Condition	Appl. Stress, MPa	DCPD Strain Rate, %ε/h
IN024 (CL)	**Sumitomo CRDM**	**EC7074C**	**TT + 21%CF**	**1 μm Polish**	**575**	**$1.1x10^{-4}$; $1.4x10^{-4}$; $7x10^{-5}$**
IN025 (CL)	Sumitomo CRDM	EC7074C	TT + 21%CF	Ground: C Finish	575	
IN026 (CL)	Sumitomo CRDM	EC7074C	TT + 21%CF	Ground: C Finish	575	
IN027 (CL)	**Valinox CRDM**	**RE243**	**TT + 21%CF**	**1 μm Polish**	**510**	**$1.0x10^{-4}$; $8x10^{-5}$; $4x10^{-5}$**
IN028 (CL)	Valinox CRDM	RE243	TT + 21%CF	Ground: C Finish	510	
IN029 (CL)	Valinox CRDM	RE243	TT + 21%CF	Ground: C Finish	510	
IN030 (CL)	**Doosan CRDM**	**133454**	**TT + 21.6%CF**	**1 μm Polish**	**540**	**$1.1x10^{-4}$; $6x10^{-5}$; $4x10^{-5}$**
IN031 (CL)	Doosan CRDM	133454	TT + 21.6%CF	Ground: C Finish	540	
IN032 (CL)	Doosan CRDM	133454	TT + 21.6%CF	Ground: C Finish	540	
IN033 (CL)	**Sumitomo CRDM**	**EC7074C**	**TT + 31%CF**	**1 μm Polish**	**690**	**$1.6x10^{-4}$; $1.2x10^{-4}$; $1.2x10^{-4}$**
IN034 (CL)	Sumitomo CRDM	EC7074C	TT + 31%CF	Ground: C Finish	690	
IN035 (CL)	**Sumitomo CRDM**	**EC7074C**	**TT + 31%CF**	**Ground: C Finish**	**690**	
IN036 (CL)	**Valinox CRDM**	**RE243**	**TT + 31%CF**	**1 μm Polish**	**700**	**$1.8x10^{-4}$; $2.8x10^{-4}$; $9x10^{-5}$**
IN037 (CL)	Valinox CRDM	RE243	TT + 31%CF	Ground: C Finish	700	
IN038 (CL)	Valinox CRDM	RE243	TT + 31%CF	Ground: C Finish	700	
IN039 (CL)	**Doosan CRDM**	**133454**	**TT + 31%CF**	**1 μm Polish**	**665**	**- - - ; $1.6x10^{-4}$; ~0**
IN040 (CL)	Doosan CRDM	133454	TT + 31%CF	Ground: C Finish	665	
IN041 (CL)	Doosan CRDM	133454	TT + 31%CF	Ground: C Finish	665	
IN042 (CL)	**TK-VDM Plate**	**114092**	**TT + 31.9%CF**	**1 μm Polish**	**680**	**$1.8x10^{-4}$; $1.7x10^{-4}$; $1.2x10^{-4}$**
IN043 (CL)	TK-VDM Plate	114092	TT + 31.9%CF	Ground: C Finish	680	
IN044 (CL)	TK-VDM Plate	114092	TT + 31.9%CF	Ground: C Finish	680	

Servo displacement of ~0.005 μm/h from 7000-9220 hours

Highlighted specimens: detailed surface exams by SEM and FIB.

Constant Load SCC Initiation Tests

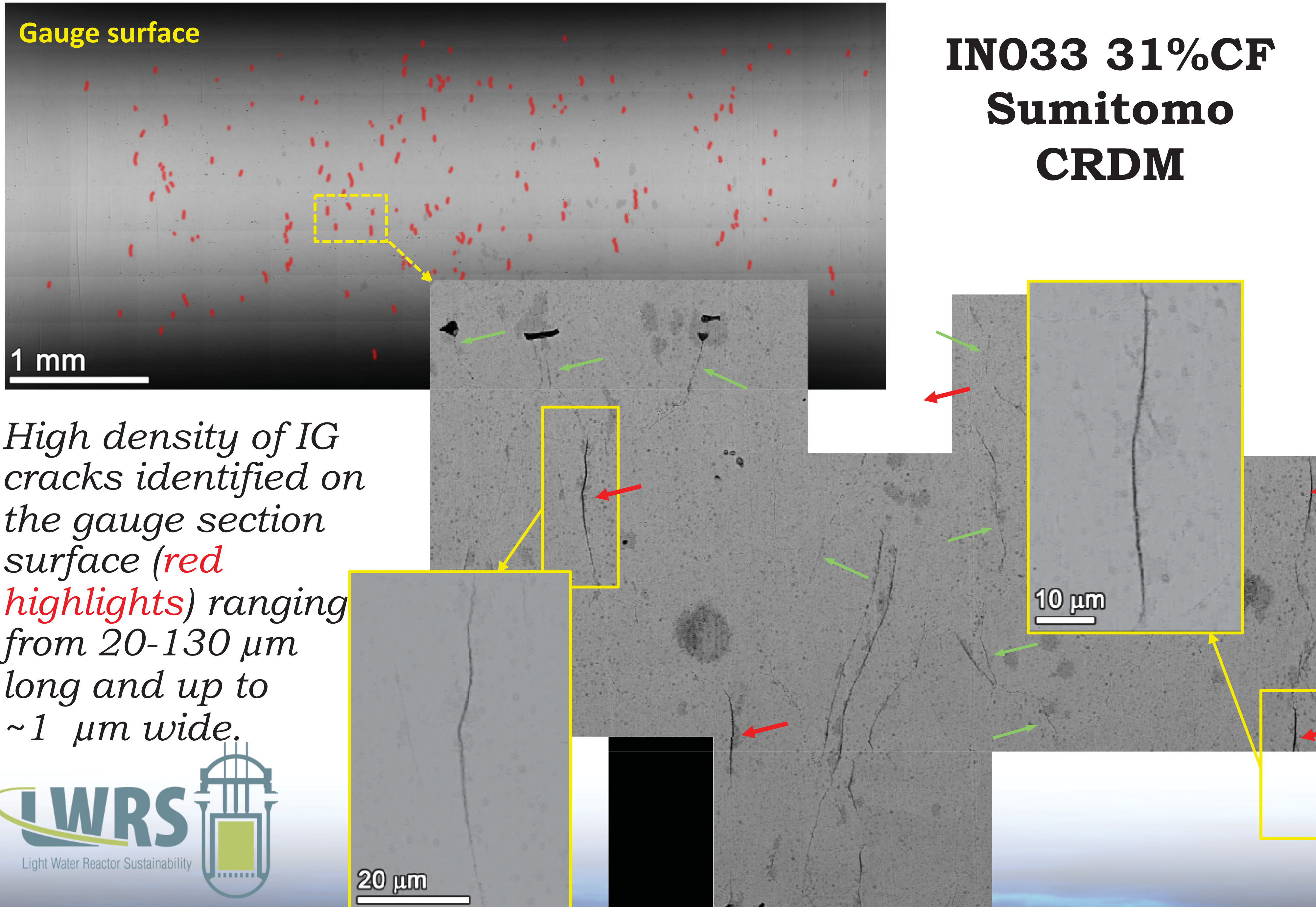
Constant Load Alloy 690 SCC Initiation Tests in LWRS System 1: Second Set of 15 Specimens after ~3610, 4910 and 7100 hour Exposure Time

Specimen	Material	Heat Number	Material Condition	Surface Condition	Applied Stress, MPa	Non-Ref DCPD, %ε/h
IN053 (CL)	**ANL Flat Bar**	**NX3297HK12**	**MA + 26%CR**	**1 μm Polish**	**775**	**$1.6x10^{-4}$; $1x10^{-4}$; $9x10^{-5}$**
IN054 (CL)	ANL Flat Bar	NX3297HK12	MA + 26%CR	1 μm Polish	775	
IN055 (CL)	ANL Flat Bar	NX3297HK12	MA + 26%CR	Ground: C Finish	775	
IN056(CL)	**GE B25K Bar**	**B25K**	**MA + 18.3%CF**	**1 μm Polish**	**550**	**$1.6x10^{-4}$; $1x10^{-4}$; $8x10^{-5}$**
IN057(CL)	GE B25K Bar	B25K	MA + 18.3%CF	1 μm Polish	550	
IN058 (CL)	GE B25K Bar	B25K	MA + 18.3%CF	Ground: C Finish	550	
IN059 (CL)	**TK-VDM Plate**	**114092**	**TT + 21%CR**	**1 μm Polish**	**660**	**$1.6x10^{-4}$; $1x10^{-4}$; $9x10^{-5}$**
IN060 (CL)	TK-VDM Plate	114092	TT + 21%CR	1 μm Polish	660	
IN061 (CL)	TK-VDM Plate	114092	TT + 21%CR	Ground: C Finish	660	
IN062 (CL)	**GE B25K Bar**	**B25K**	**MA + 12.4%CF**	**1 μm Polish**	**510**	**$1.3x10^{-4}$; $8x10^{-5}$; $7x10^{-5}$**
IN063 (CL)	GE B25K Bar	B25K	MA + 12.4%CF	1 μm Polish	510	
IN064 (CL)	GE B25K Bar	B25K	MA + 12.4%CF	Ground: C Finish	510	
IN065 (CL)	**Valinox CRDM**	**RE243**	**TT + 11.7%CF**	**1 μm Polish**	**365**	**$1.0x10^{-4}$; $7x10^{-5}$; $5x10^{-5}$**
IN066 (CL)	Valinox CRDM	RE243	TT + 11.7%CF	1 μm Polish	365	
IN067 (CL)	Valinox CRDM	RE243	TT + 11.7%CF	Ground: C Finish	365	

Servo displacement of ~0.005 μm/h from 5000-7100 hours

Highlighted specimens: detailed surface exams by SEM and FIB.

SEM Examinations of Surface Damage

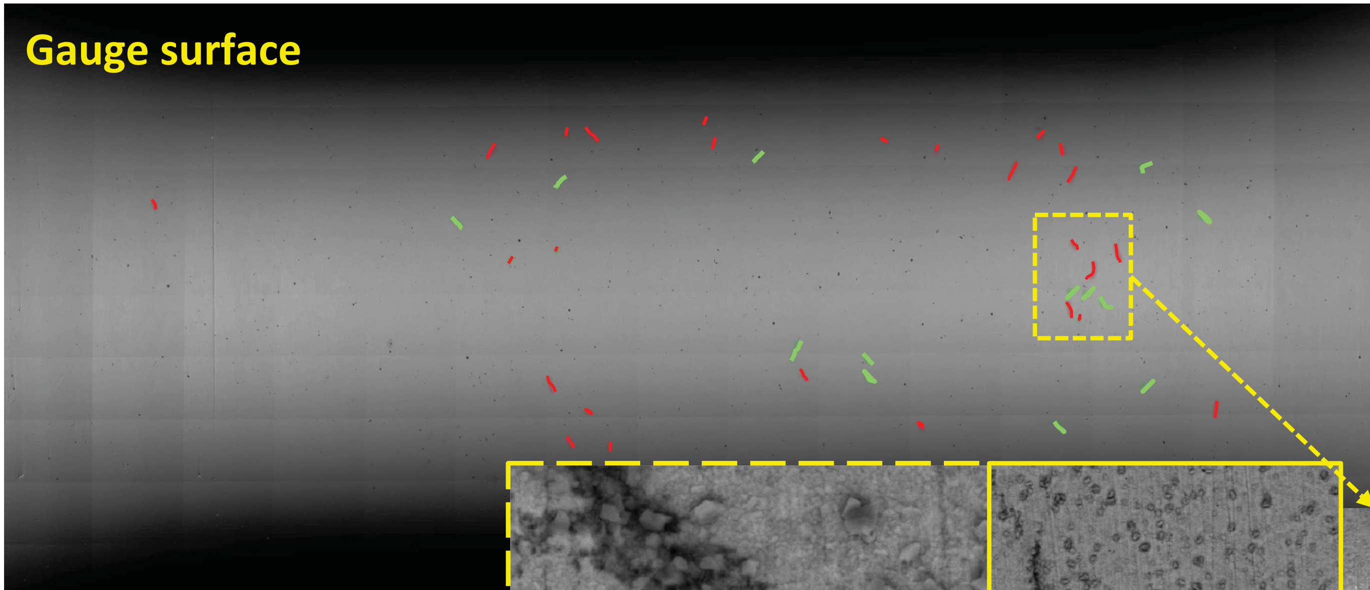


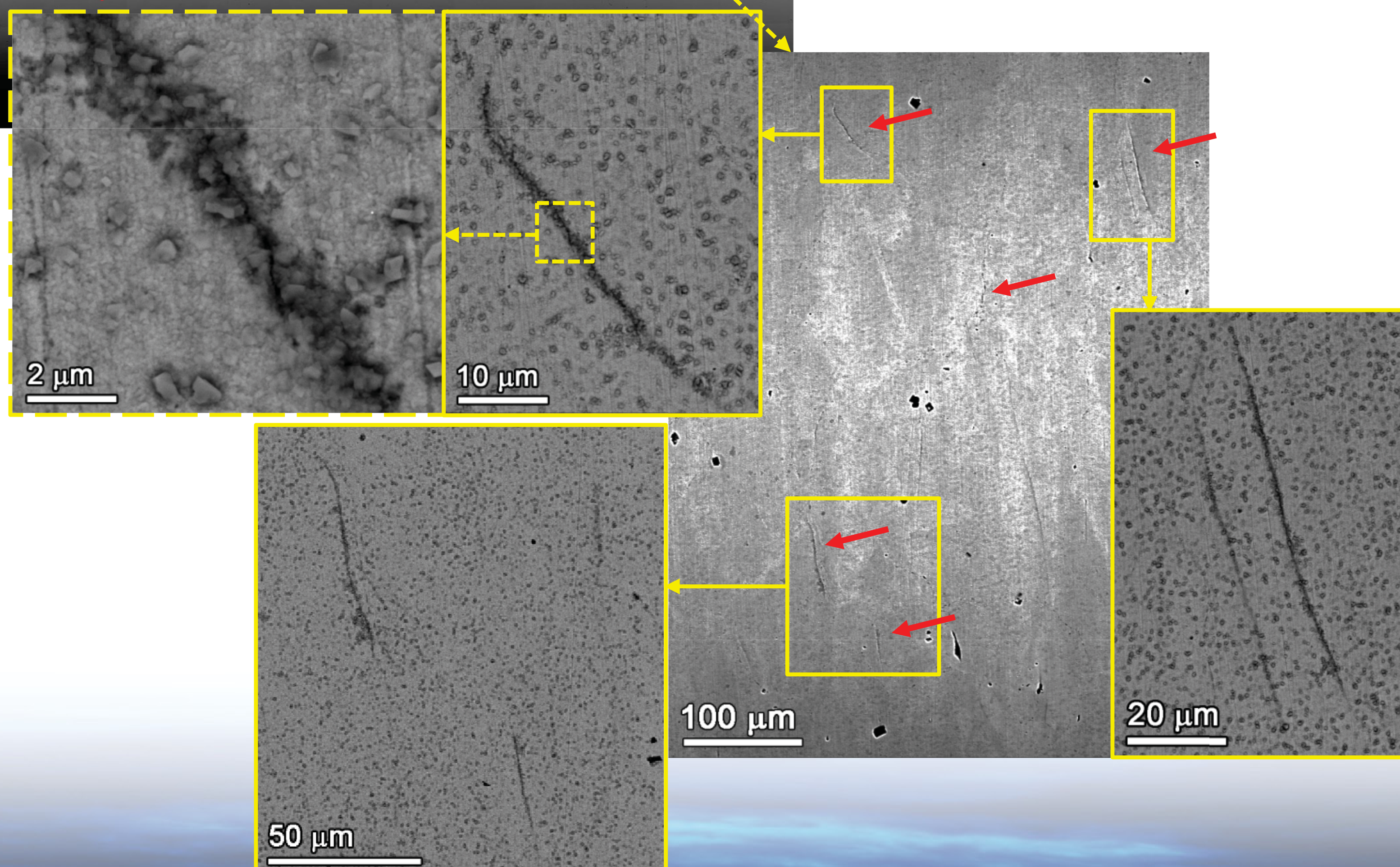
IN033 31%CF
Sumitomo
CRDM

High density of IG cracks identified on the gauge section surface (red highlights) ranging from 20-130 µm long and up to ~1 µm wide.

Light Water Reactor Sustainability

SEM Examinations of Surface Damage





IN036 31%CF
Doosan
CRDM

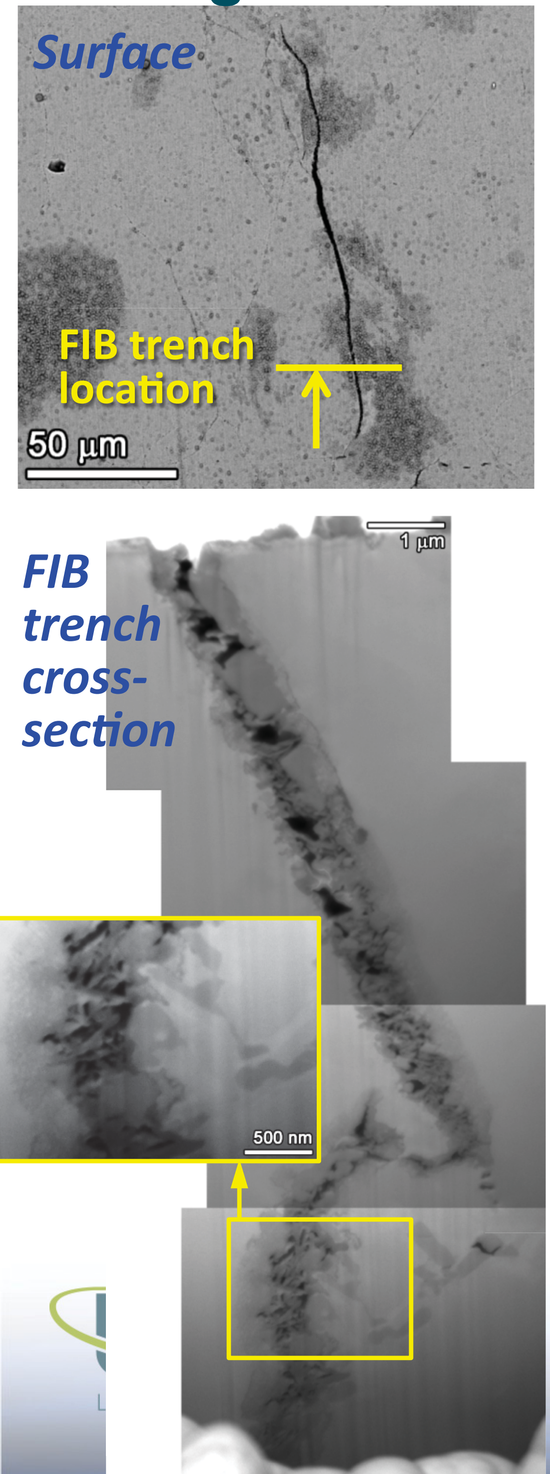
Moderate density of IG cracks identified on the gauge section surface (red/ green) ranging with length <50 µm.

Light Water Reactor Sustainability

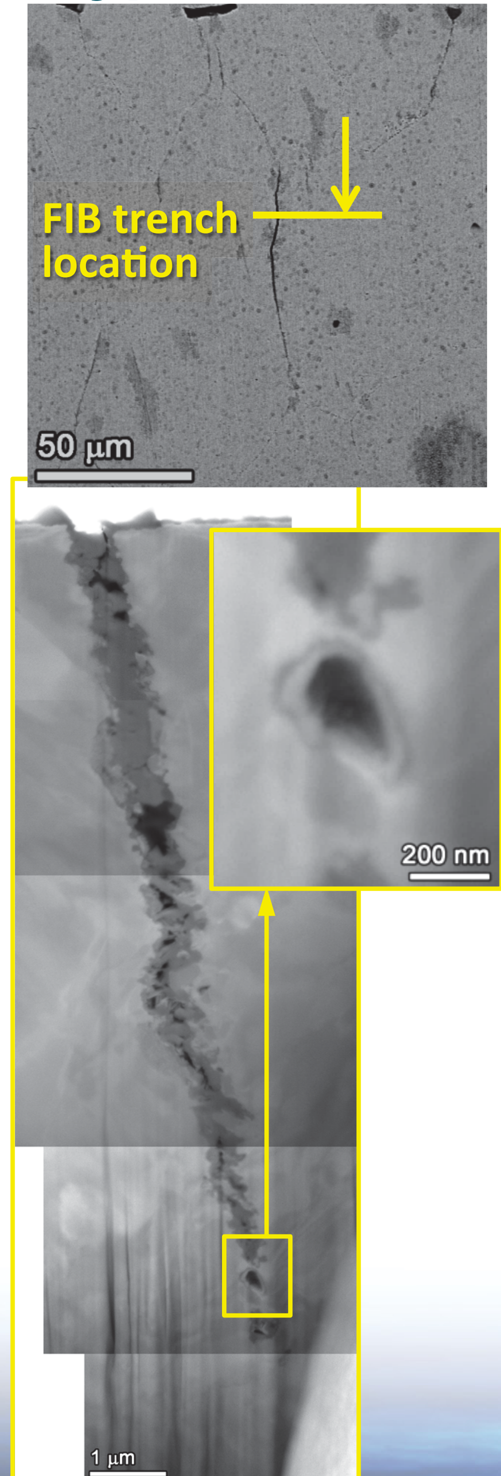
Gauge Surface Examination Results

Spec	Material & Condition	Surface	σ (MPa)	SEM Observations
IN033	**Sumitomo CRDM, TT+31%CF**	1 µm	690	**Many small IG cracks**
IN024	**Sumitomo CRDM, TT+21%CF**	1 µm	575	**Few very small IG cracks**
IN036	**Valinox CRDM, TT+31%CF**	1 µm	700	**Many small IG cracks**
IN027	Valinox CRDM, TT+21%CF	1 µm	510	No cracks
IN065	Valinox CRDM, TT+12%CF	1 µm	365	No cracks
IN039	**Doosan CRDM, TT+31%CF**	1 µm	665	**Few small IG cracks**
IN031	Doosan CRDM, TT+21%CF	1 µm	540	No cracks
IN042	**TK-VDM Plate, TT+32%CF**	1 µm	680	**Few small IG cracks**
IN059	TK-VDM Plate, TT+21%CR	1 µm	660	No cracks
IN053	**ANL Flat Bar, MA+26%CR**	1 µm	775	**Few TG cracks (TiN)**
IN065	GE B25K Bar, MA+18%CF	1 µm	550	No cracks
IN062	GE B25K Bar, MA+12%CF	1 µm	510	No cracks

Large crack



Typical crack



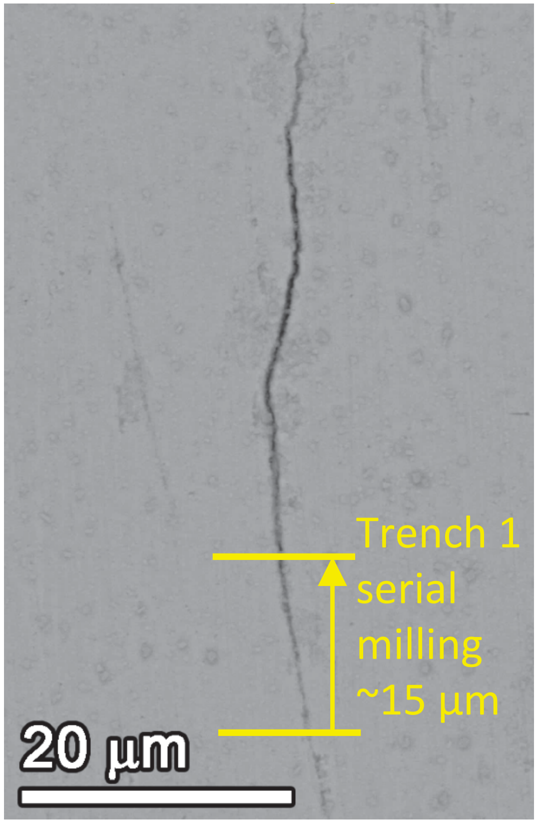
Tight crack

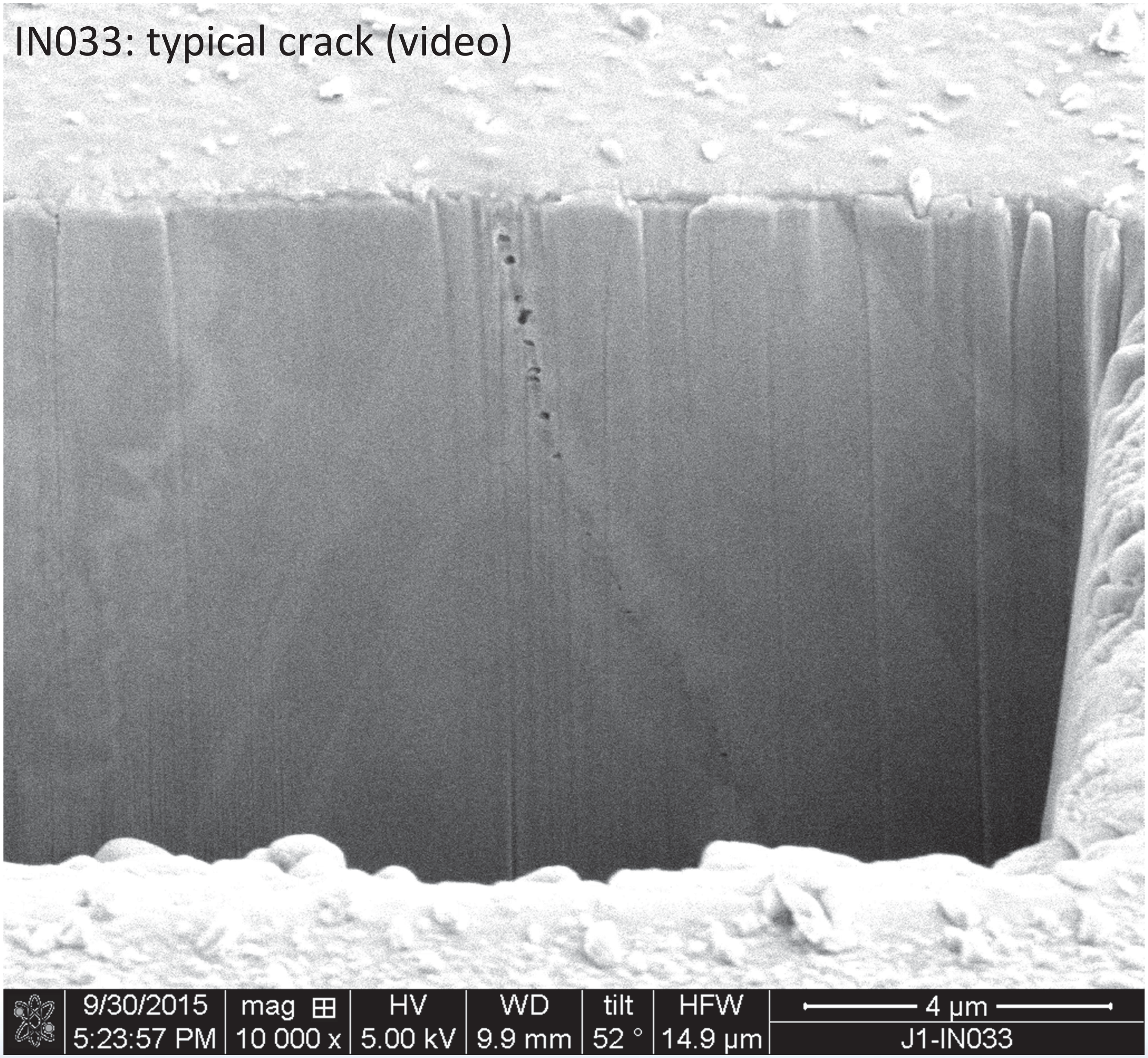
IN033 31%CF Sumitomo CRDM

FIB trenches, SEM of cracks in cross-section

Large surface cracks are >15 µm deep, short cracks end in GB cavities.

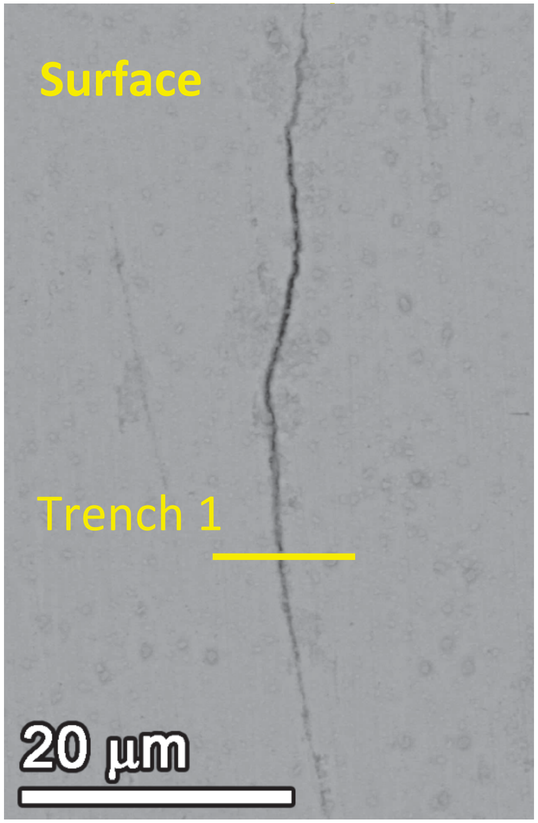
IN033 (Sumitomo 31%CF, 1 µm) FIB Serial Milling: Grain Boundary Cavities and IG Crack

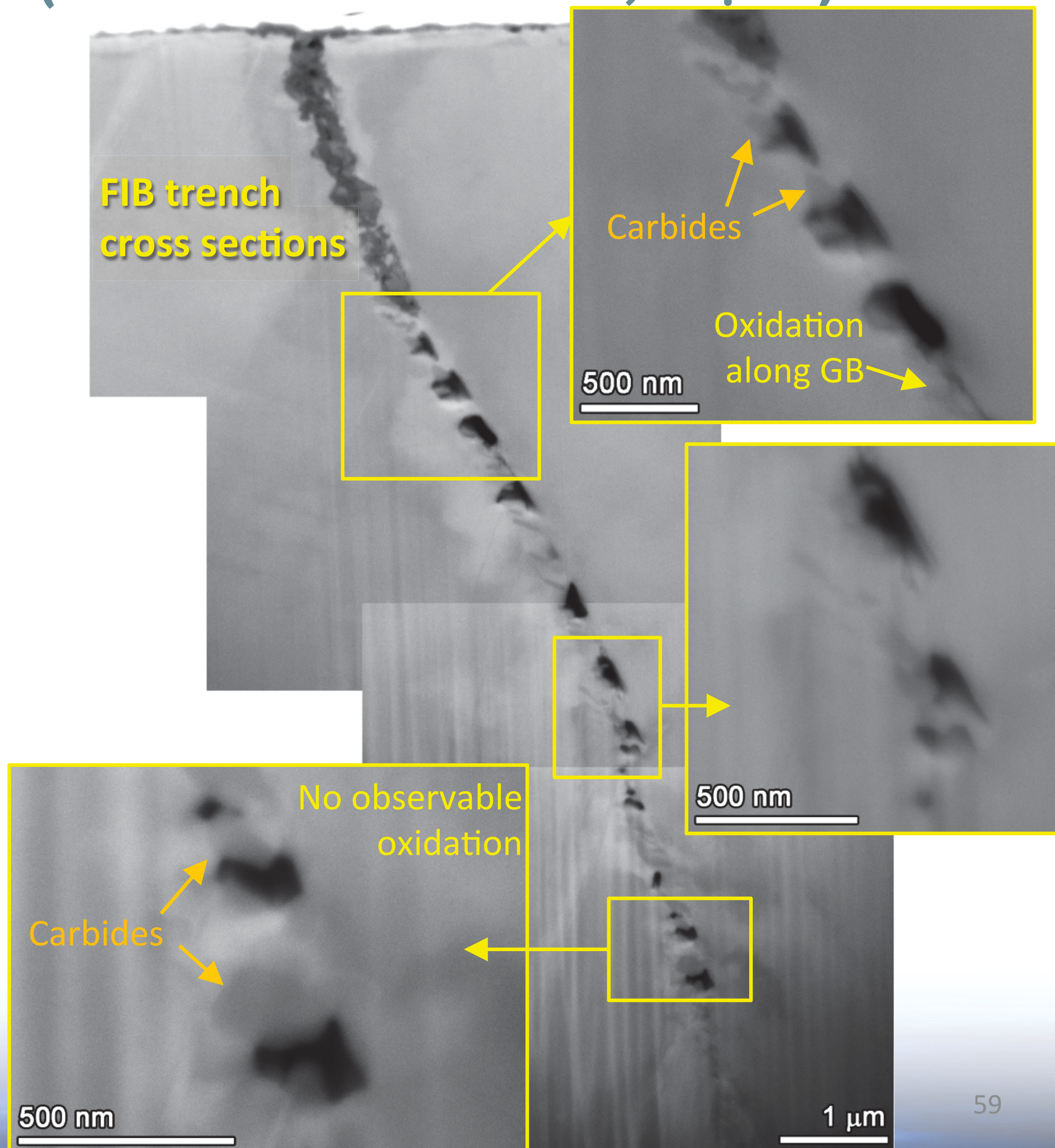




- *IG oxide filled crack reaching >15 µm deep.*
- *Grain boundary cavities found ahead of the crack/oxidation tip.*

IG Crack and Cavity Morphology in IN033 (Sumitomo 31%CF, 1 μm)

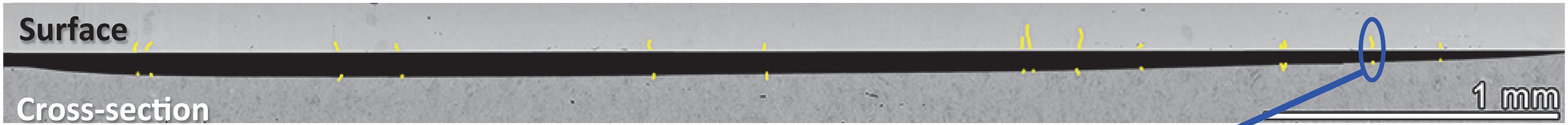




- *IG oxide filled crack reaching >15 μm deep.*
- *Grain boundary cavities found ahead of the crack/oxidation tip.*

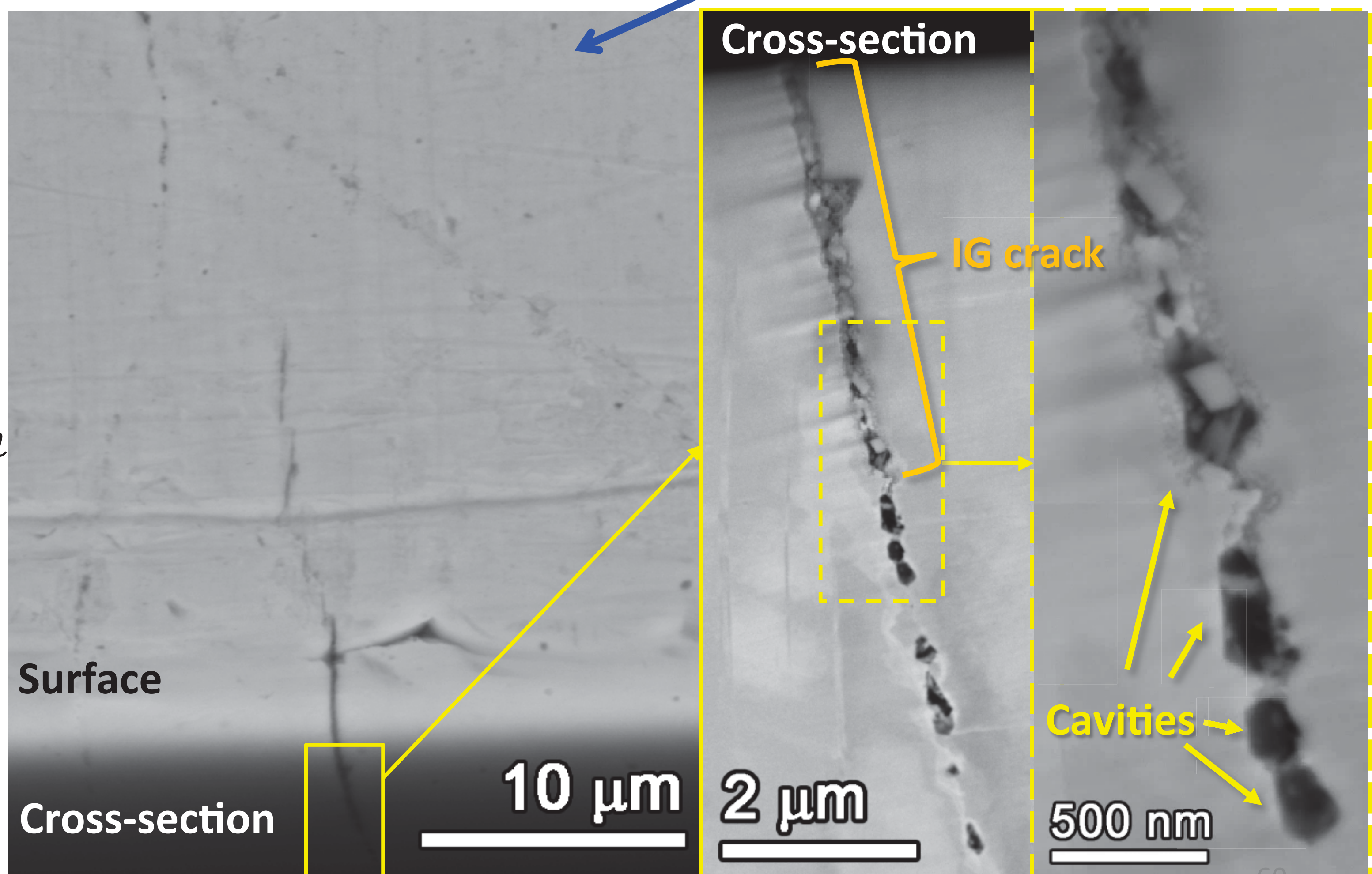
LWRS Light Water Reactor Sustainability

IN033 (Sumitomo 31%CF, 1 µm): Correlation of IG surface cracks and grain boundary cavities

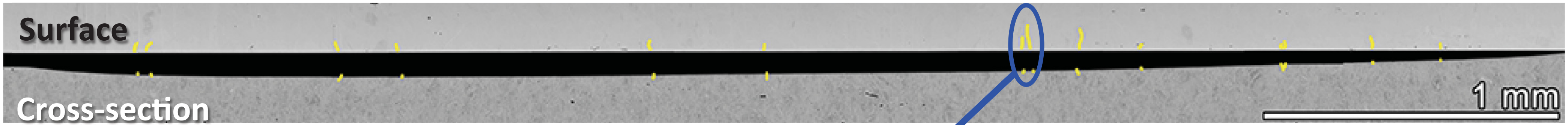


One-to-one match *between IG cracks on the surface and series of grain boundary cavities beneath that extend to the surface.*

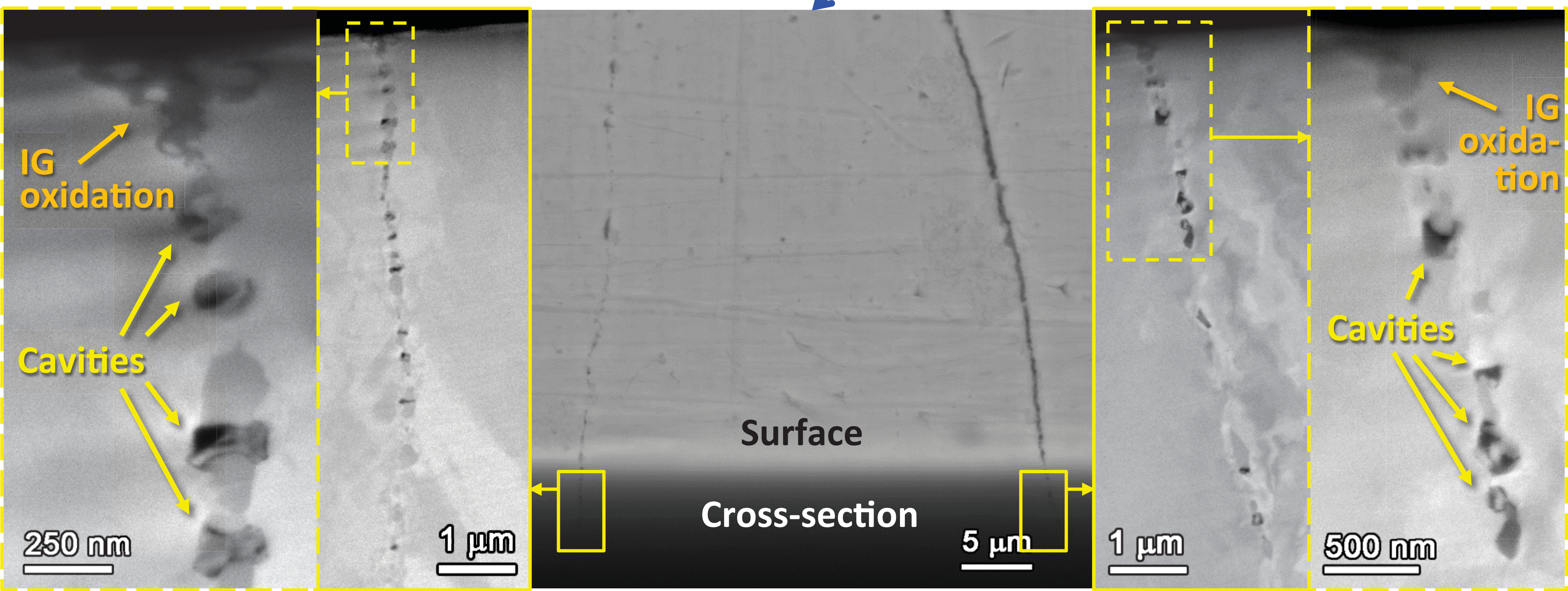
For obvious surface cracks: *oxidized IG crack depth reaches 5-20 µm with high density of grain boundary cavities ahead of crack oxidation front.*



IN033 (Sumitomo 31%CF, 1 μm): Correlation of IG surface cracks and grain boundary cavities

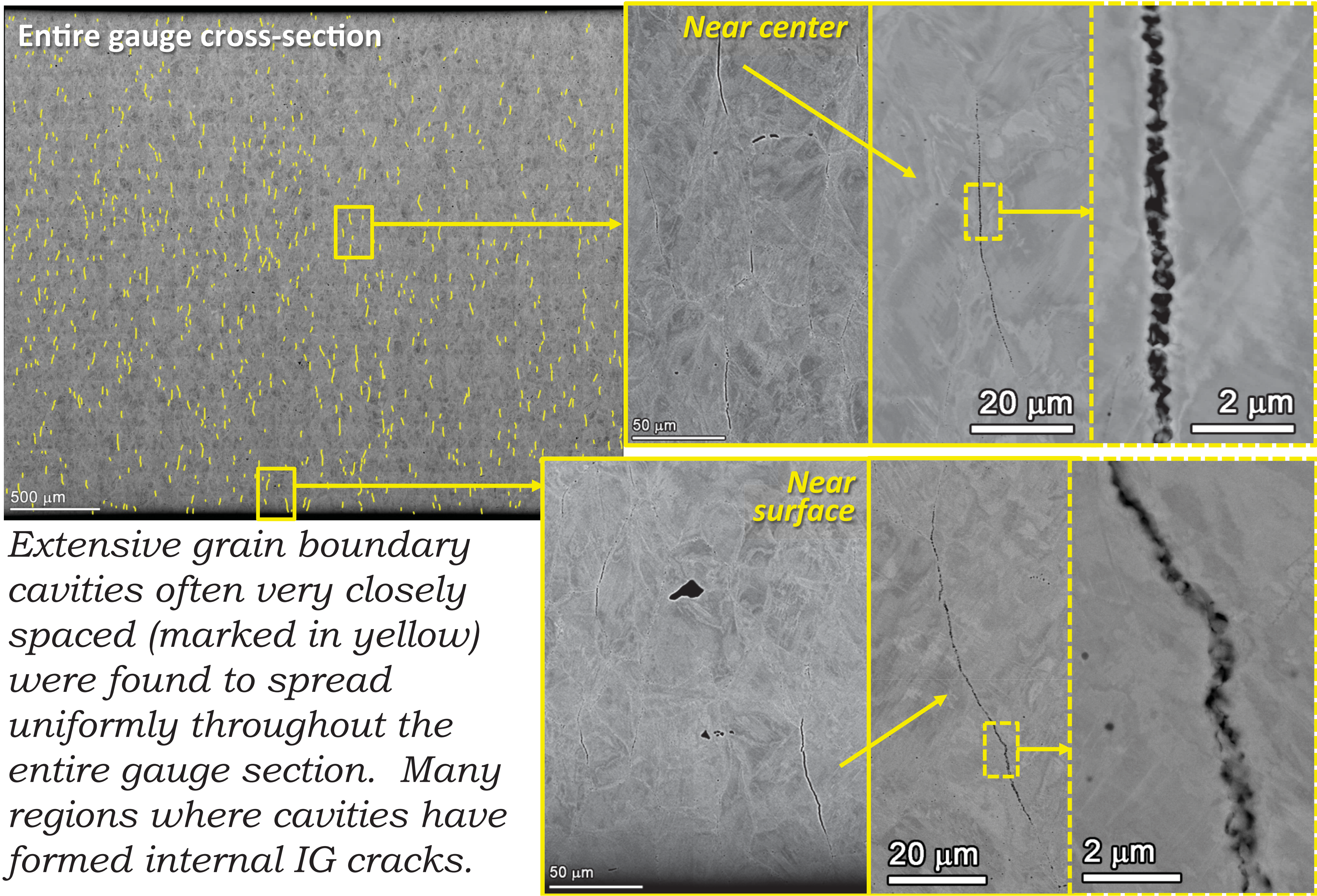


One-to-one match *between IG cracks on the surface and series of grain boundary cavities beneath that extend to the surface.*



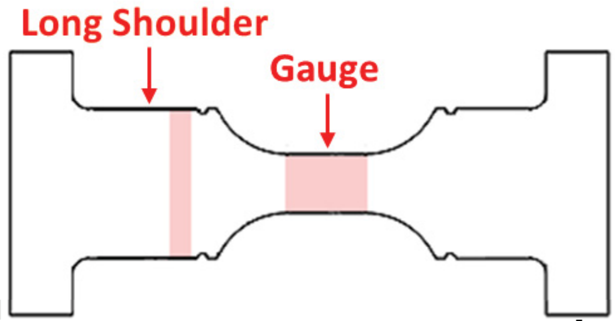
For shallow/discontinuous cracks observed on the surface: *IG crack depth is <1 μm and is followed by a series of grain boundary cavities.*

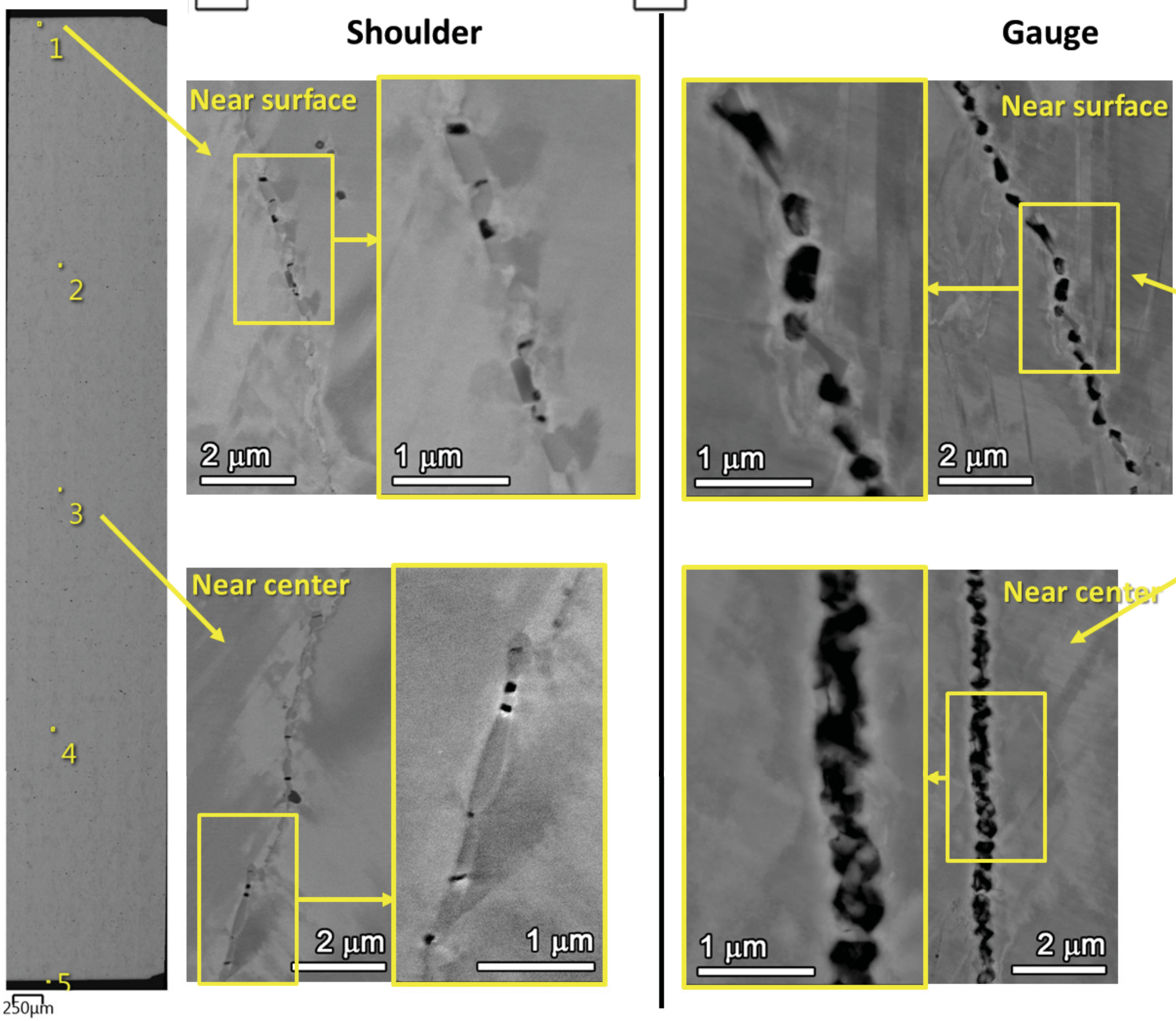
IN033 (Sumitomo 31%CF) Cross-Section: Grain Boundary Cavities Distribution in Bulk



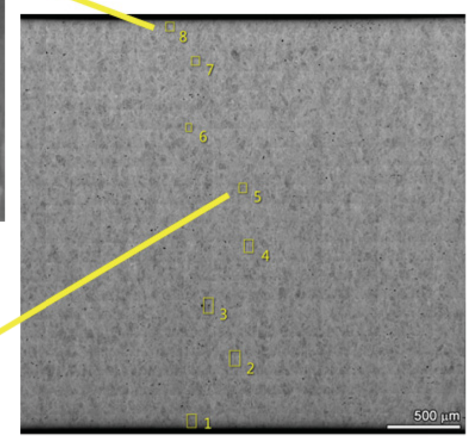
Extensive grain boundary cavities often very closely spaced (marked in yellow) were found to spread uniformly throughout the entire gauge section. Many regions where cavities have formed internal IG cracks.

IN033 (Sumitomo 31%CF) Cross-Section: Grain Boundary Cavities Distribution in Bulk





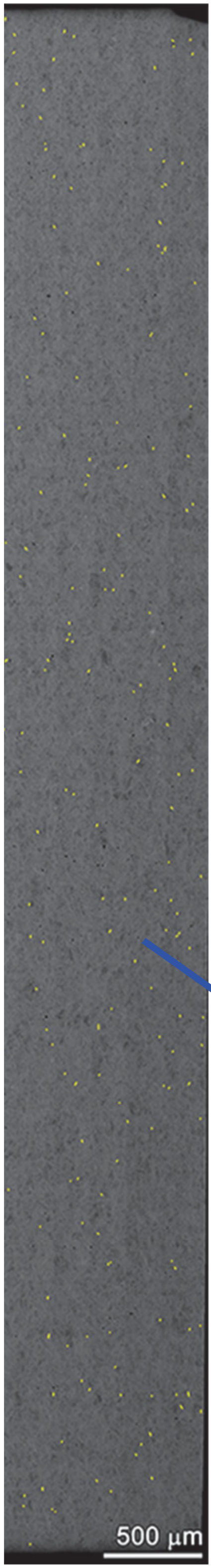
High density of cavities at GB carbide interfaces only in the high-stress gauge section.



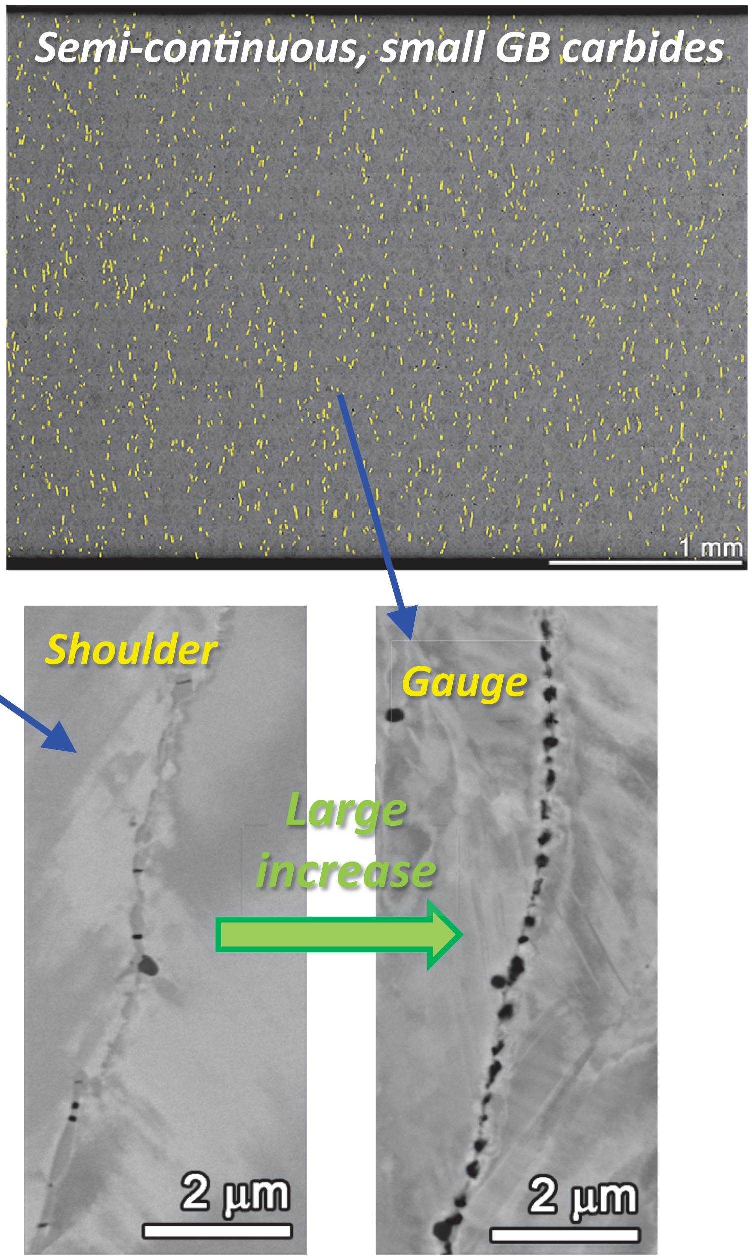
Limited GB damage found at low-stress shoulder similar to as-CW material.

Cross-Section Examination Results

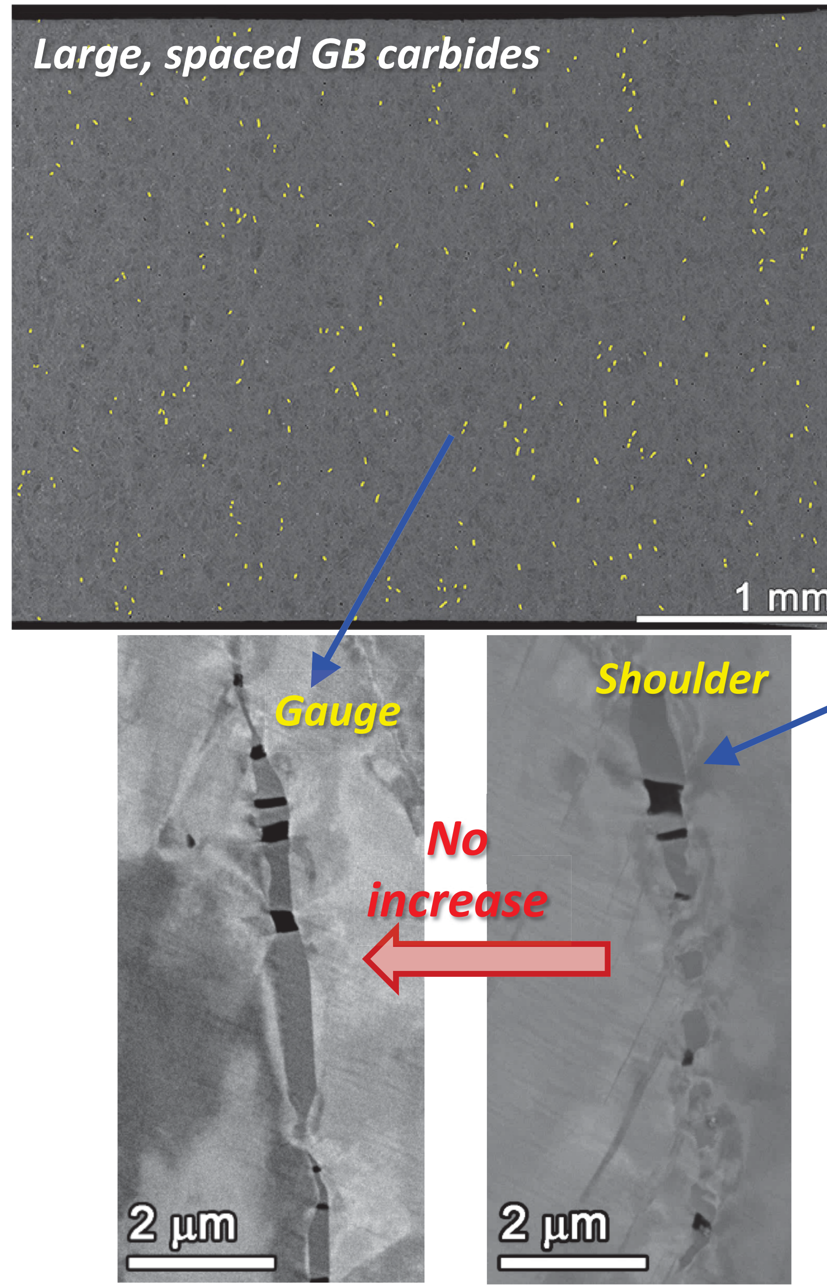
Cavity nucleation and growth depends strongly on GB carbide size and distribution.

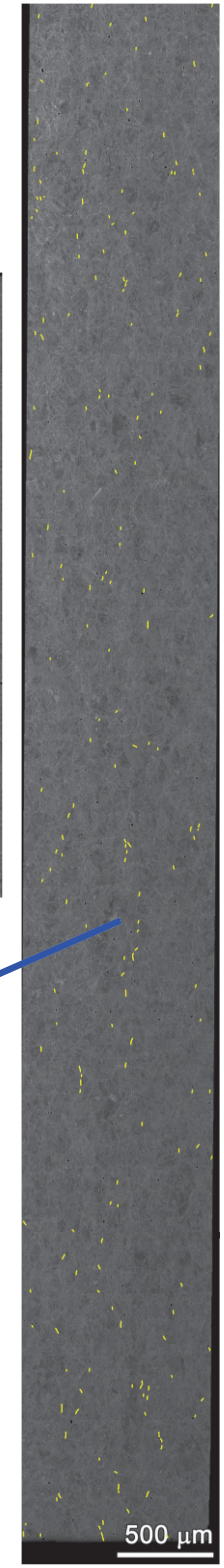


IN034 31%CF Sumitomo CRDM



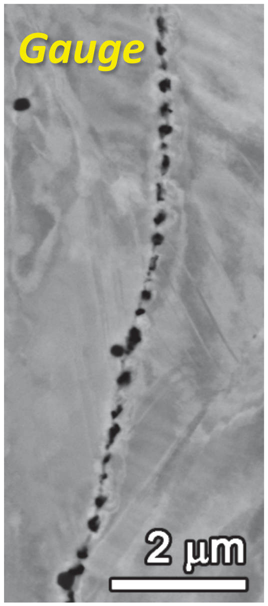
IN038 31%CF Doosan CRDM

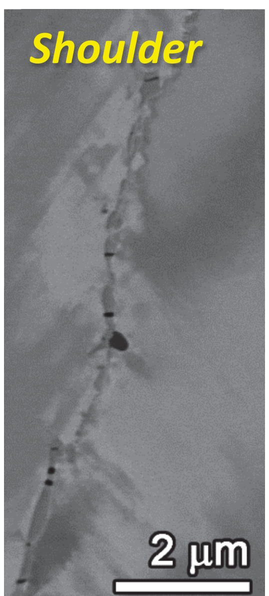




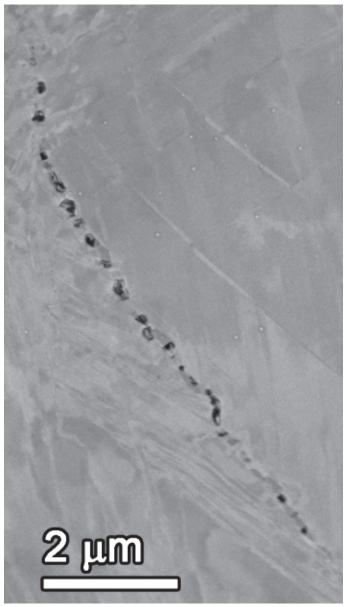
Cross-Section Cavity Morphology Comparison – Gauge vs. Shoulder in Highly CW Specimens

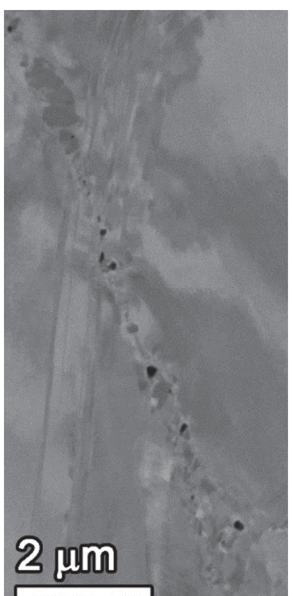
IN034 (Sumitomo 31%CF, C finish)



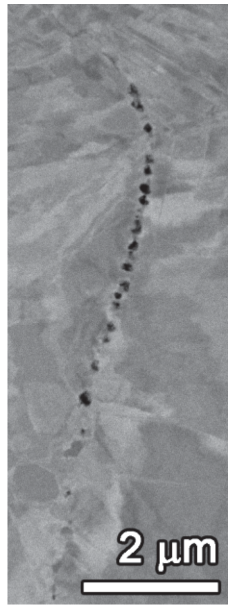


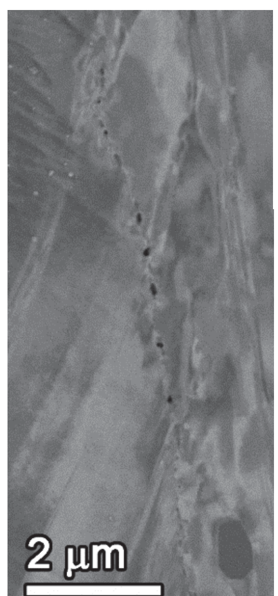
IN038 (Valinox 31%CF, C finish)





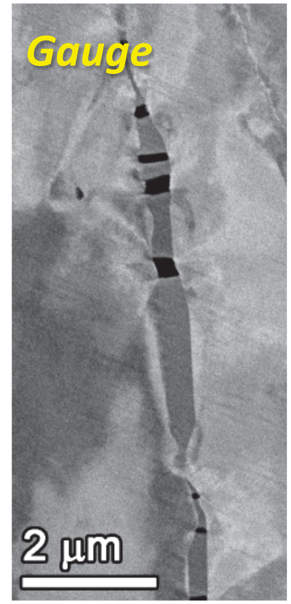
IN044 (TK-VDM 32%CF, C finish)

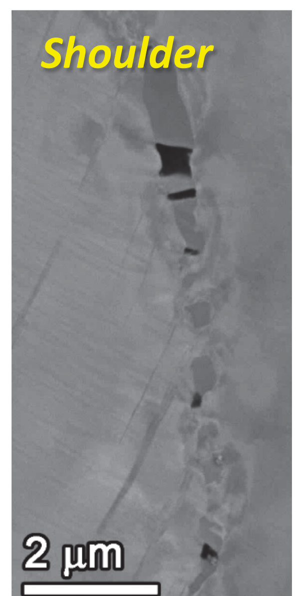




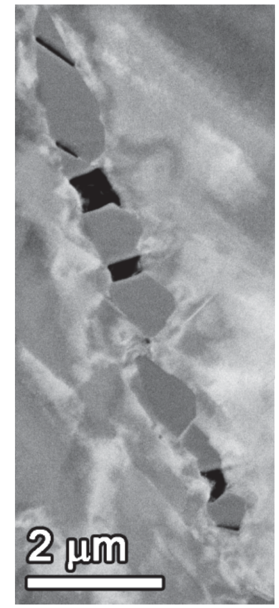
Large increase in cavity size and density from shoulder to gauge

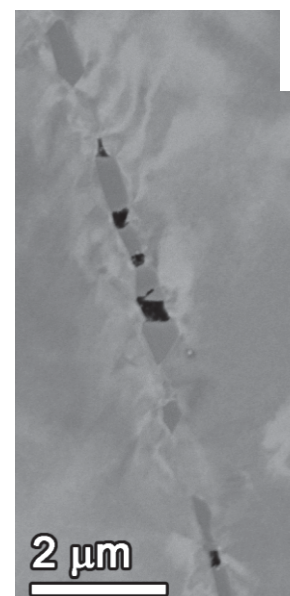
IN041 (Doosan 31%CF, C finish)





IN055 (ANL 26%CR, C finish)



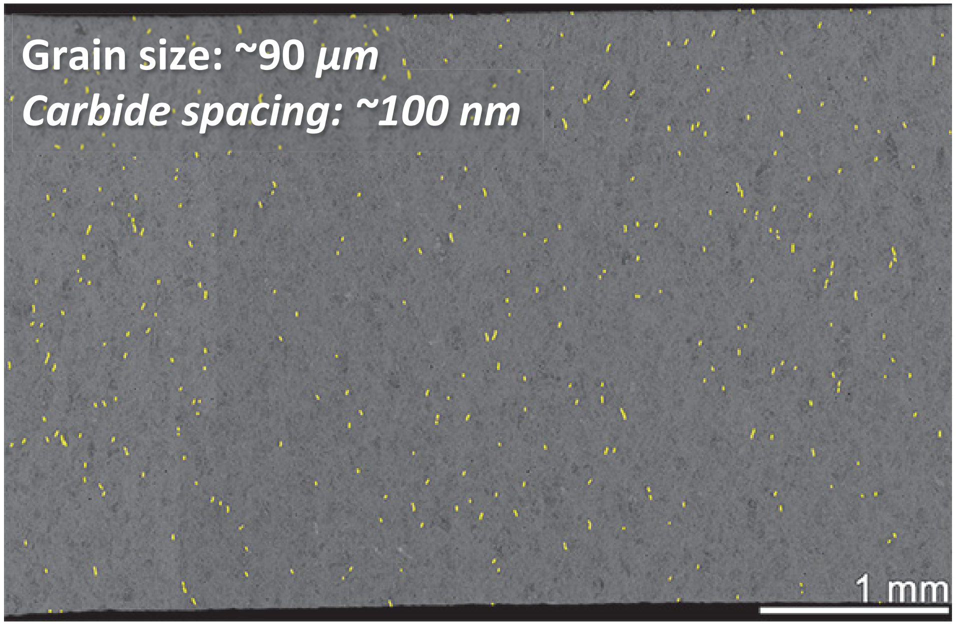


No apparent increase in cavity density

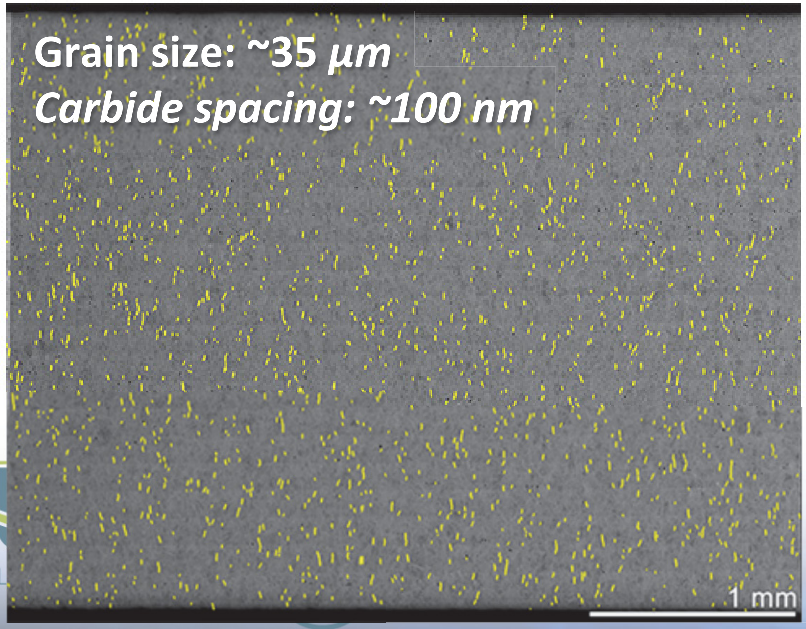
Cross-Section Examination Results

Higher density of GB cavities and IG creep cracks observed for the 31%CF Sumitomo than for the 31%CF Valinox and TK-VDM alloy 690TT specimens. No significant GB cavity formation during SCC testing for the other alloy 690 heats regardless of CW condition.

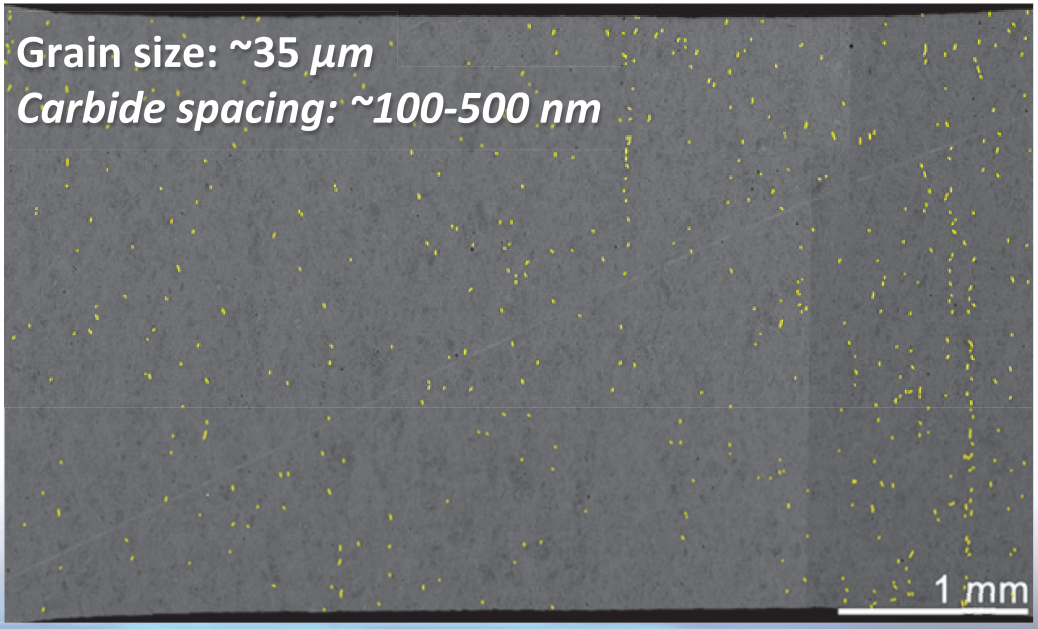
TT + 31%CF Valinox CRDM (IN038)



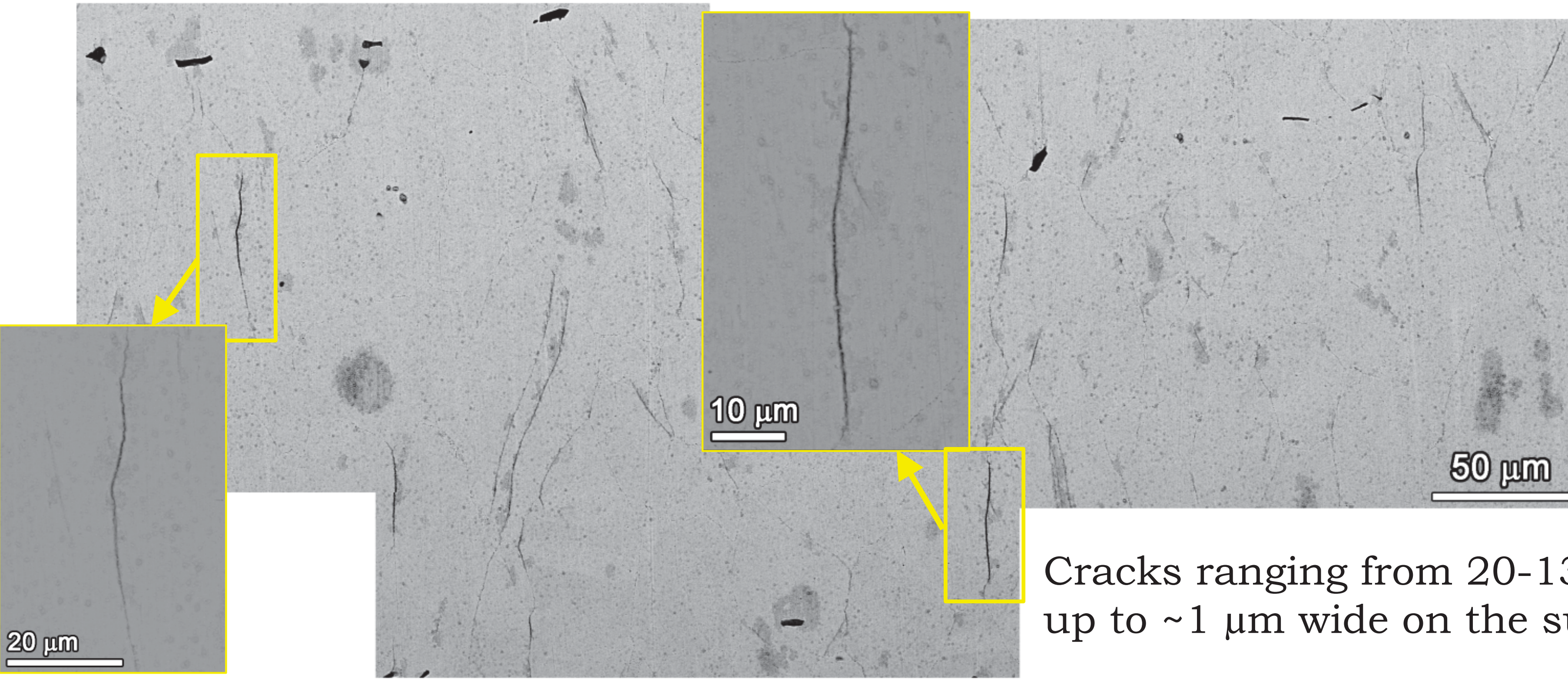
TT + 31%CF Sumitomo CRDM (IN034)



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

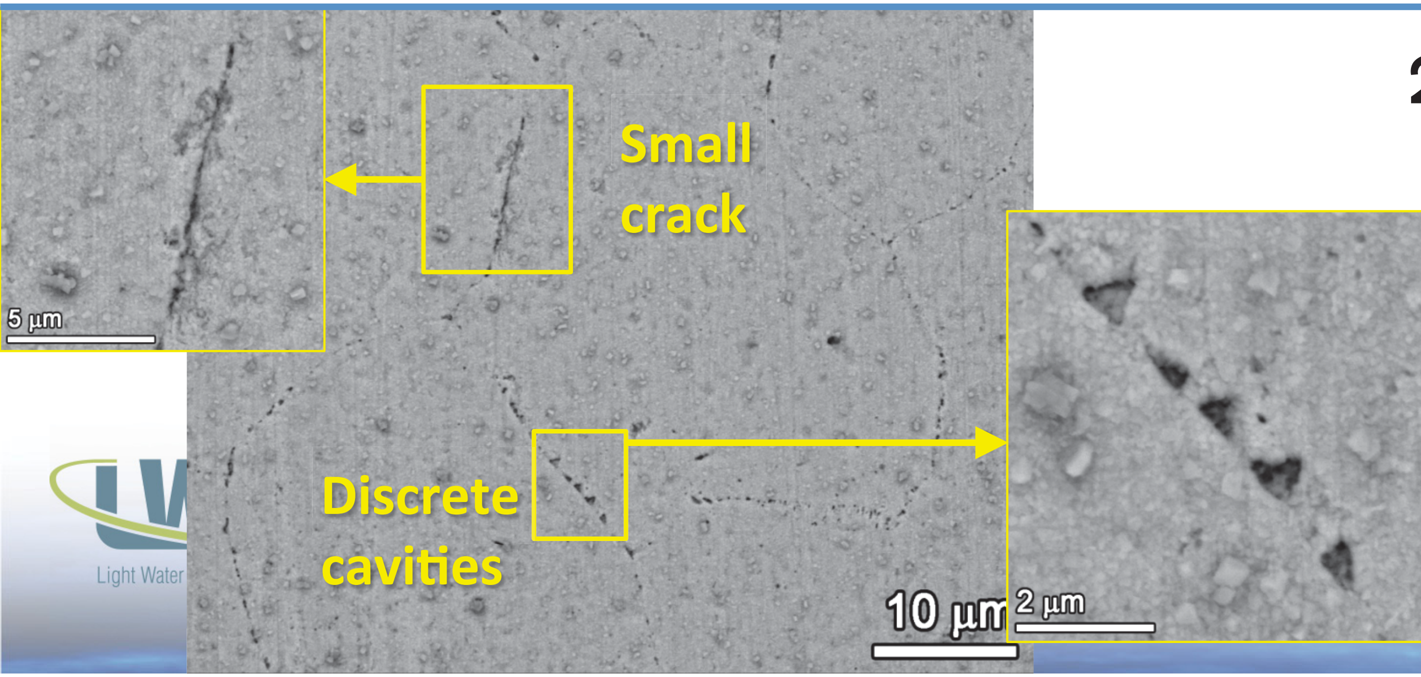


Surface Damage Morphology – Sumitomo CRDM



31%CF (IN033)

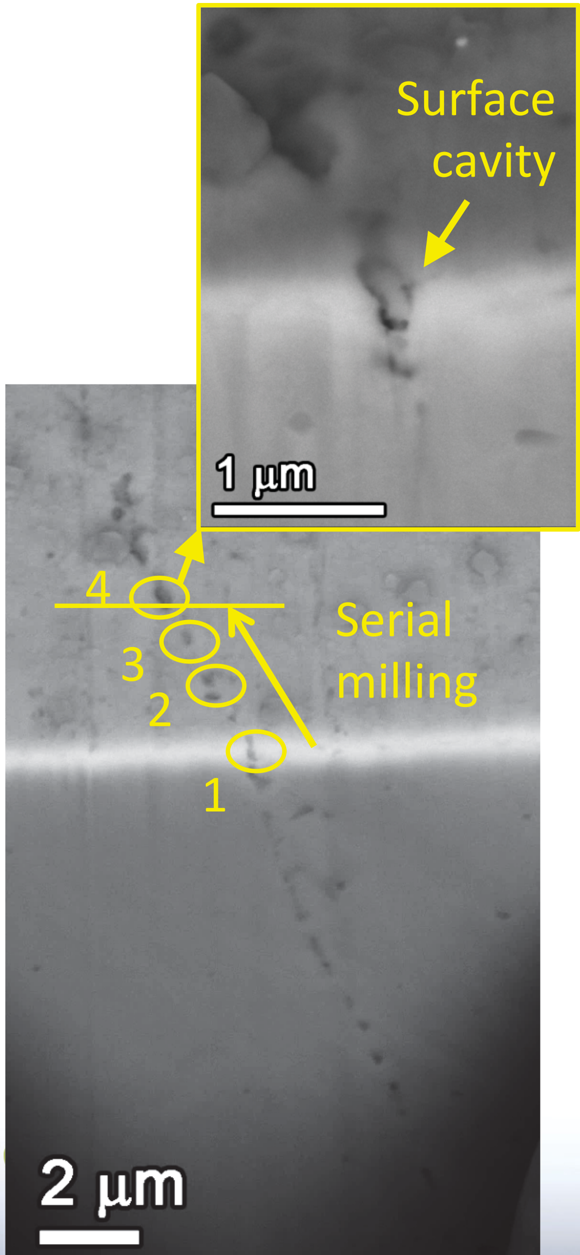
Cracks ranging from 20-130 µm long, up to ~1 µm wide on the surface

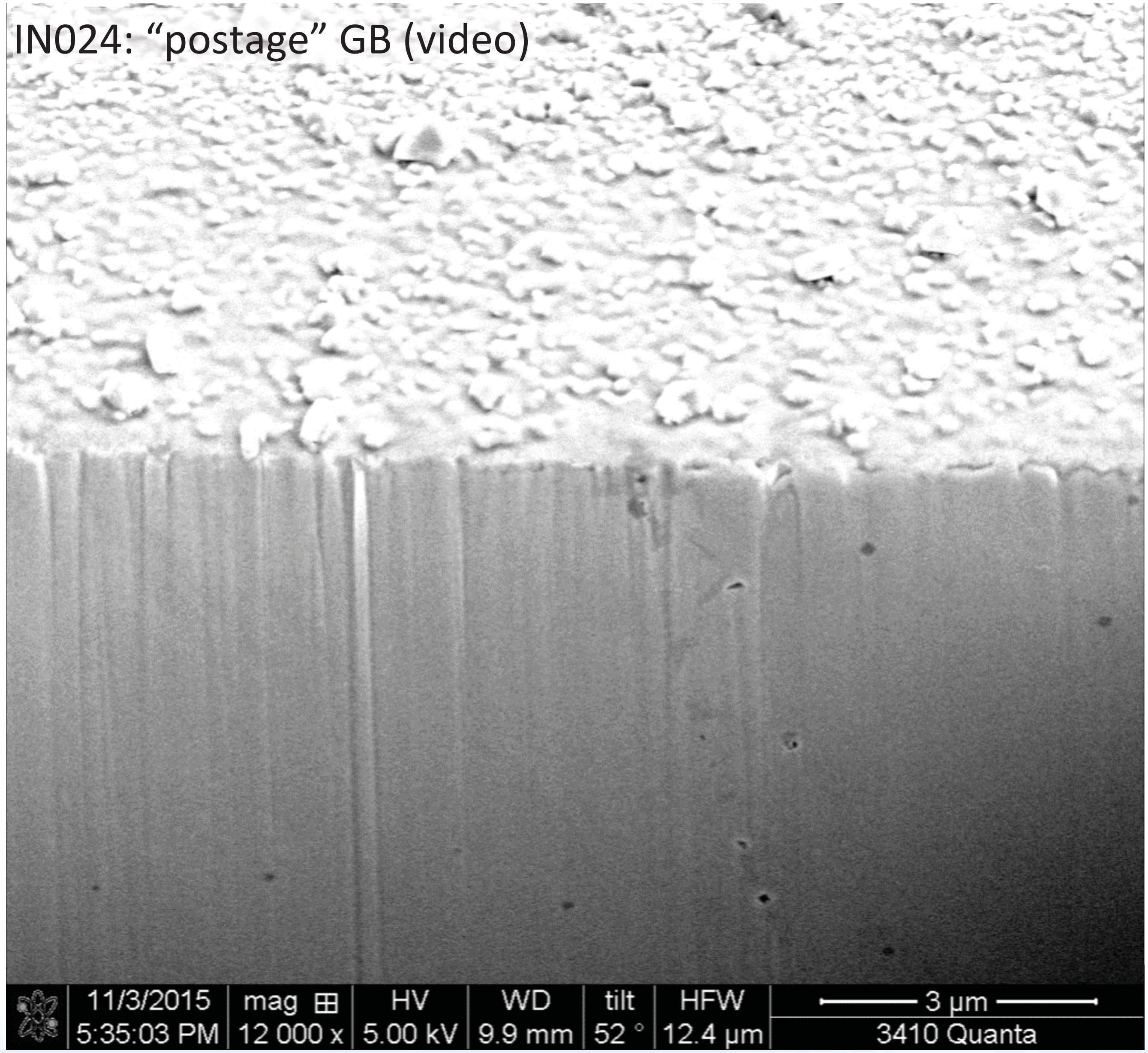


21%CF (IN024)

Only a few short, shallow cracks observed and “postage” damage (discrete cavities) along grain boundaries

IN024 (21%CF Sumitomo CRDM, 1 μm) FIB Serial Milling: Surface Cavity Distribution

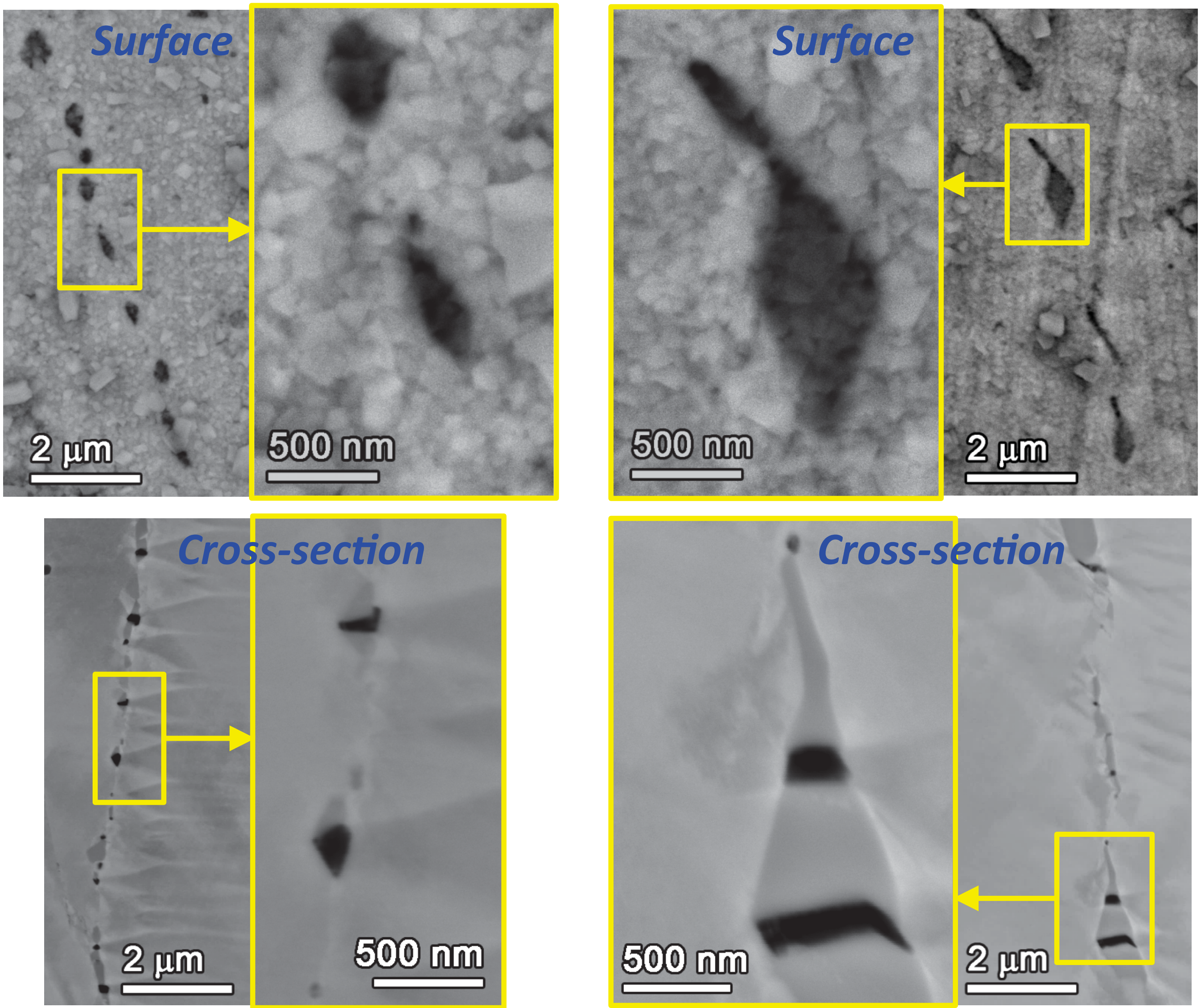




Damage Morphology: Surface vs Cross-Section

21%CF Sumitomo
Smaller cavities

21%CF Doosan
Larger cavities



The size and spacing of "postage stamp damage" on the surface is consistent with the size and spacing of GB cavities in the cross-section.

Strong correlation between damage distribution (cavities) and starting carbide microstructure.

Summary of Alloy 690 Constant Load Test Results

Material-Condition	GB Carbide Distribution	Density of New GB Cavities	Density of IG Cracks on Surface
Sumitomo TT+31%CF	Semi-continuous, small	High	Many small IG cracks
Sumitomo TT+21%CF	Semi-continuous, small	Low	Few small IG cracks
Valinox TT+31%CF	Semi-continuous, small	Moderate	Many small IG cracks
Valinox TT+21%CF	Semi-continuous, small	Very low	No cracks
Valinox TT+12%CF	Semi-continuous, small	No new cavities	No cracks
Doosan TT+31%CF	Spaced, large	No new cavities	Few small IG cracks
Doosan TT+21%CF	Spaced, large	No new cavities	No cracks
TK-VDM TT+32%CF	Semi-continuous, small	Moderate	Few small IG cracks
TK-VDM TT+21%CR	Semi-continuous, medium	No new cavities	No cracks
ANL MA+26%CR	Spaced, large	No new cavities	Few TG cracks (TiN)
GE MA+18%CF	Few IG carbides	No cavities	No cracks
GE MA+12%CF	Few IG carbides	No cavities	No cracks

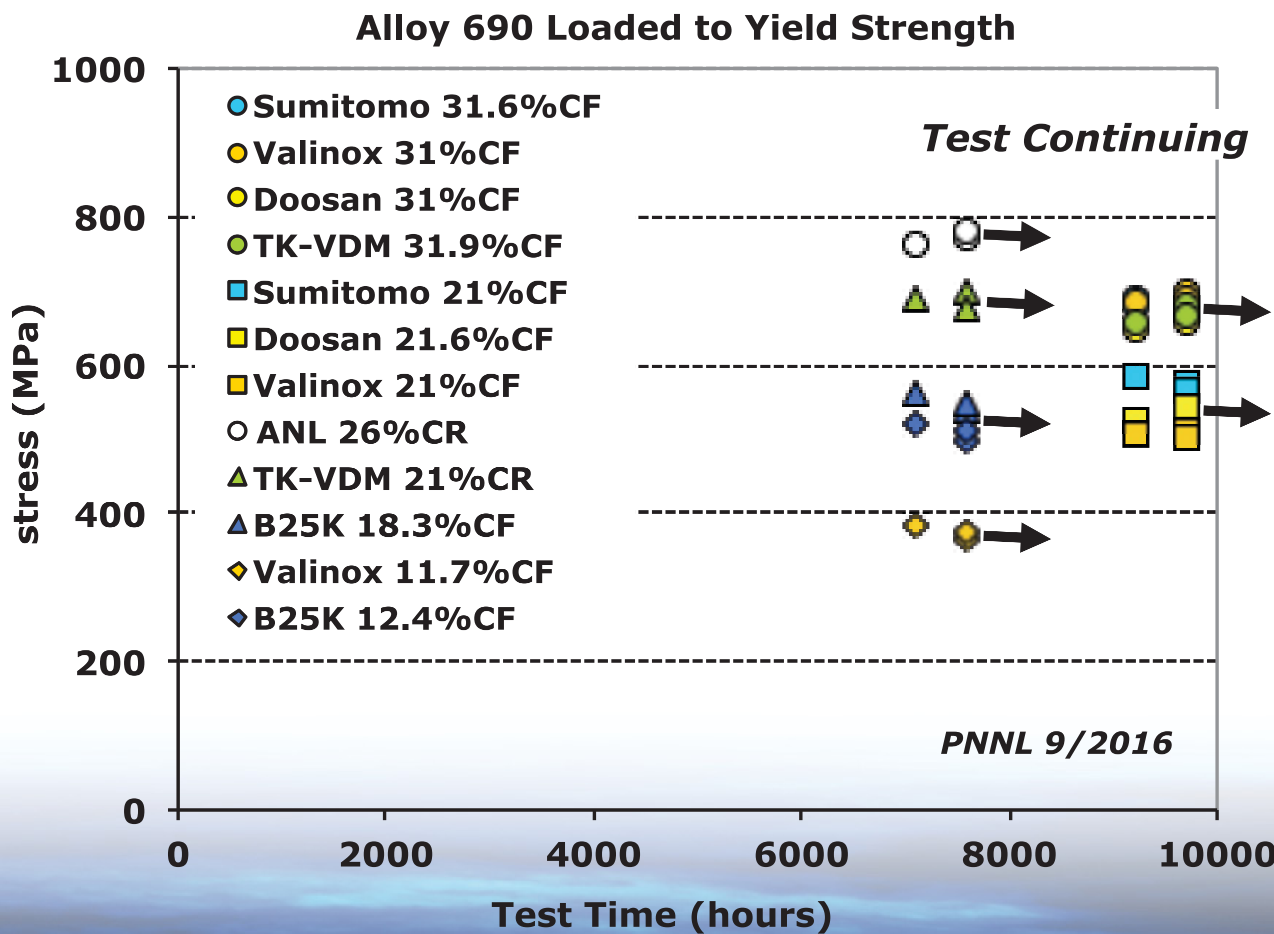
Three highly CW alloy 690TT heats with nearly continuous distribution of small GB carbides exhibit closely spaced IG cavity formation that can lead to internal creep cracks.

Summary of Alloy 690 Constant Load Test Results

- *Alloy 690 susceptibility to GB cavity formation and IG cracking directly depends on %CW, applied stress and GB carbide microstructure.*
- *It remains unclear whether this GB damage evolution will occur in alloy 690 materials with more realistic levels of cold/warm work (e.g., <15%) and at lower applied stresses.*
- *Constant load testing is continuing on 23 specimens and has expanded to include five new material/microstructural conditions.*

5 new conditions (13 new specimens) added to matrix:

- 31%CF Sumitomo TT at 90%YS
- 31%CF Valinox TT at 90%YS
- 31%CR Valinox SA at 100%YS
- 17%CR Valinox SA at 100%YS
- 31%CF Allvac MA at 100%YS



Light Water Reactor Sustainability

LWRS

Light Water Reactor Sustainability