STRESS CORROSION CRACK INITIATION MECHANISMS OF NICKEL-BASE ALLOYS IN SIMULATED PWR PRIMARY WATER

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Research Project:
Stress Corrosion Crack Initiation of
Nickel-Base Alloys in LWR Environments
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OVERVIEW

This report summarizes selected stress corrosion crack (SCC) initiation research results presented by Pacific Northwest National Laboratory (PNNL) staff in April 2018 at two international meetings: (1) CORROSION 2018 Conference & Expo organized by the NACE International held in Phoenix, AZ and (2) 2018 International Cooperative Group Meeting on Environment-Assisted Cracking (ICG-EAC) held in Knoxville, TN. It includes:

- A paper entitled "***Intergranular Stress Corrosion Crack Initiation and Temperature Dependence of Alloy 600 in Pressurized Water Reactor Primary Water,***" is given that was published in the CORROSION 2018 Conference proceeding. The paper describes SCC initiation results from our LWRS project for a cold-worked, solution-annealed alloy 600 heat that is part of an ongoing ICG-EAC round robin. Detailed characterizations of damage evolution are presented with a focus on the influence of temperature on SCC initiation.
- Three presentations are given in the Appendix:
  - "***Intergranular Stress Corrosion Crack Initiation and Temperature Dependence of Alloy 600 in Pressurized Water Reactor Primary Water***" presented in the Corrosion in Nuclear Systems Symposium as part of the CORROSION 2018 Conference. An overview of SCC initiation testing and analysis on alloy 600 materials at PNNL is provided highlighting damage evolution mechanisms leading to crack initiation as a function of material condition and test temperature.
  - "***Microstructural Comparison of Intergranular Attack in Alloy 600 in the SA versus TT Conditions Exposed to Simulated Primary Water***" presented in the Nickel-Base Alloys Session at the 2018 ICG-EAC meeting. High-resolution electron microscopy has been performed on intergranular attack evolution in selected alloy 600 materials after exposure in PWR primary water. Examinations are evaluating grain boundary corrosion/oxidation mechanisms and the influence of carbides on degradation. This research is primarily supported by EPRI and is in collaboration with our LWRS project where SCC initiation tests are being conducted on many of the same materials.
  - "***SCC Initiation of Alloy 182 in PWR Primary Water***" presented in the Weldments Session at the 2018 ICG-EAC meeting. An update of SCC initiation research on alloy 182 weld metals within a joint EPRI/NRC project is provided with a focus on the effects of cold work and applied stress. Many SCC initiation tests are being conducted in direct collaboration with our LWRS project employing experimental approaches and systems developed under LWRS support.

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Intergranular Stress Corrosion Crack Initiation and Temperature Dependence of Alloy 600 in Pressurized Water Reactor Primary Water

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ABSTRACT

Stress corrosion crack (SCC) initiation of a solution annealed, cold-worked (CW) UNS N06600 (Alloy 600) material was investigated in 360 and 325°C simulated PWR primary water using constant load tensile instrumented for in-situ detection by the direct current potential drop technique. This CW material with high boron and low carbon bulk concentrations was found to be highly susceptible to SCC. Consistent SCC initiation times were obtained at both temperatures and the thermal activation energy for SCC initiation is estimated at ~116 kJ/mol.

Key words: intergranular stress corrosion cracking, crack initiation, UNS N06600 (alloy 600), thermal activation energy

INTRODUCTION

In spite of the widespread use of UNS N06690 (Alloy 690) as a replacement material, many UNS N06600 (alloy 600) components remain in primary water reactor (PWR) service.[1] With the increasing demand for life extension of operating PWRs, it is essential to investigate the critical degradation modes that could impair the reliability of the thick section Alloy 600 components. In particular, detailed understanding of stress corrosion crack (SCC) initiation processes is still limited as is the ability to quantitatively estimate component SCC initiation times. In order to better investigate the SCC initiation behavior of Ni-base alloys in simulated PWR primary water, state-of-the-art SCC initiation testing facilities have been developed with active load control and in-situ direct current potential drop (DCPD) technique for crack detection. In addition, high-resolution microscopy including scanning electron microscopy (SEM), transmission electron microscopy and atom probe tomography (APT) has been utilized to examine precursor damage and short cracks in detail. This paper builds on recent publications[2-5] and presents new results on a solution annealed (SA) and cold-worked (CW) Alloy 600 plate heat describing crack initiation microstructures and the effect of test temperature on SCC initiation time.

EXPERIMENTAL PROCEDURE

Material

The bulk composition of the Alloy 600 plate material is listed in Table 1. This material was solution annealed at 1100°C for 30 min followed by a water quench, which produced a duplex grain size distribution consisting of 50–200 μm grains in addition to much larger grains ~400–600 μm in diameter. High resolution SEM imaging of grain boundaries revealed no evidence for intergranular (SA) precipitates in the SA condition (Figure 1). As reported in Table 1, this material has a low bulk carbon (C) and a high boron (B) content compared to typical alloy 600 compositions. In order to quantify grain boundary segregation, APT characterizations were performed on representative high energy grain boundaries (GBs). Two APT specimens successfully captured GB data with an example of atom maps and concentration profile shown in Figure 2. The two samples showed consistent levels of B segregation (1-1.6 at%) with minor segregation of Si, C, P and Ti at the grain boundary.[6]

Table 1
Bulk Composition of the UNS N06600SA (Alloy 600) Plate Material (wt%)

Product	Ni	Cr	Fe	Mn	C	Si	Cu	P	S	B, appm*
Plate	75.8	15.6	7.92	0.46	0.01	0.22	0.01	0.005	0.0002	46

* The B content is measured by glow discharge mass spectrometry.

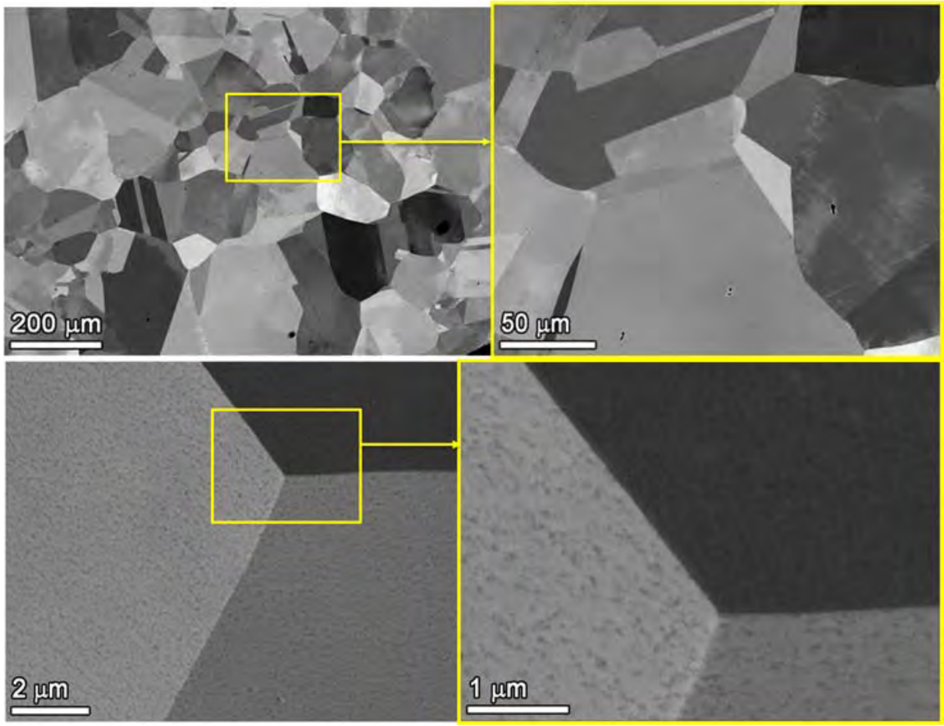

Figure 1: SEM-BSE images of grain size distribution (top) in the Alloy 600 plate material and higher-magnification images of a GB (bottom) revealing no IG precipitation.

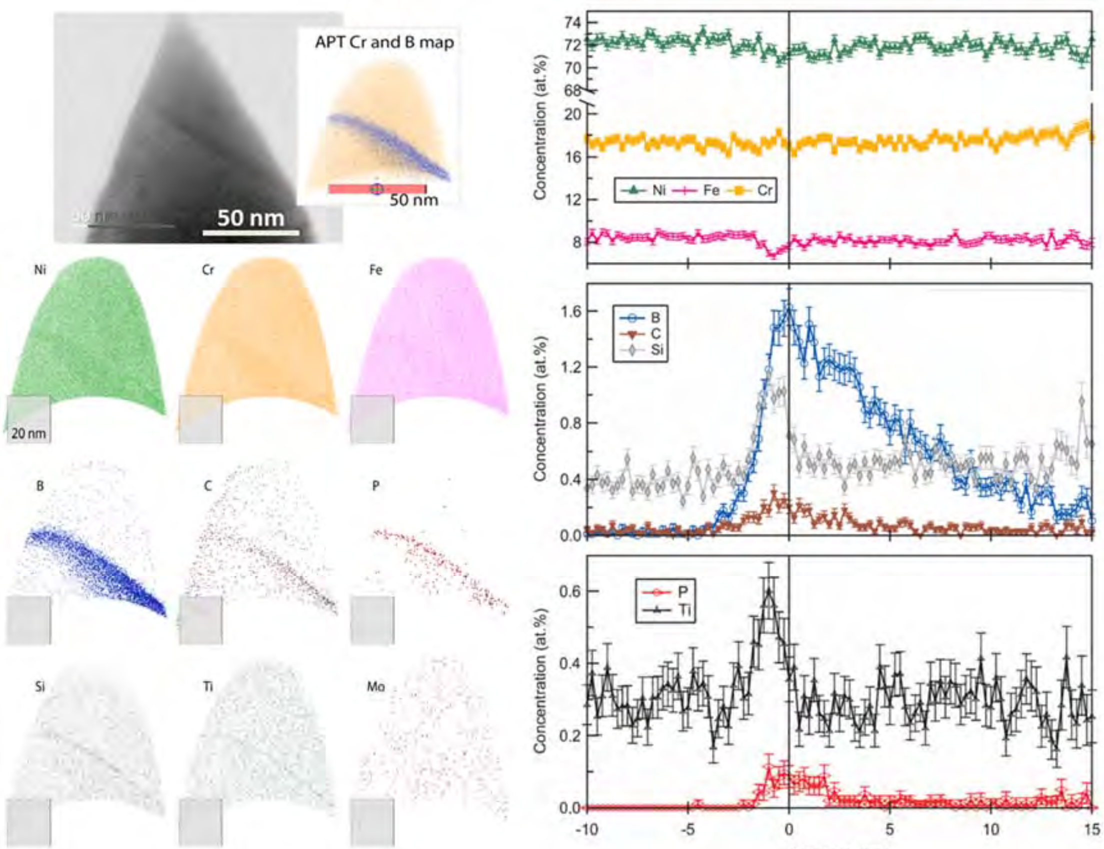

Figure 2: APT atom maps (left) and concentration maps (right) depicting segregation at a GB in the Alloy 600 plate material. Image depth of the atom maps is 20 nm.

SCC Initiation Testing

Constant Load Tensile (CLT) Test

A block that sectioned from the Alloy 600 plate was cold forged to 15% reduction in thickness. Uniaxial tensile specimens were machined with the gauge section along the thickness direction (short transverse) of the forged block. All specimens have a height of 30.4 mm (1.2 inches). This relatively small size was selected to enable multi-specimen serial loading in an autoclave and has the advantage of making DCPD more sensitive to changes in cross sectional area due to cracking. It also enables full characterization of the gauge surface by SEM in a reasonable period of time. The gauge diameter of the specimens is altered to control the stress level applied to each specimen in a loading string. This allows each specimen to be loaded to its yield strength (or any other target stress) for the applied load. As listed in Table 2, three specimens were prepared for each test temperature. In order to better characterize precursor damage and cracks, the gauge surface of every specimen was polished to 1 µm finish. The detailed surface preparation process is described elsewhere.[7]

Table 2
Summary of Alloy 600 plate specimens

Spec. ID	Material Type	CW level	Finish	Temp (^oC)	Applied Stress* (MPa)	Time to SCC initiation (h)
IN151	SA Plate	15%CF	1 µm	360	350	290
IN152	SA Plate	15%CF	1 µm	360	350	342
IN153	SA Plate	15%CF	1 µm	360	350	354
IN209	SA Plate	15%CF	1 µm	325	364	1220
IN210	SA Plate	15%CF	1 µm	325	354	1350
IN211	SA Plate	15%CF	1 µm	325	351	1040

* The applied stress is the yield stress of the specimens at the tested temperatures.

The SCC initiation tests were conducted in simulated PWR primary water with 1000 ppm of boron, 2 ppm of lithium and a dissolved hydrogen content corresponding to the Ni/NiO stability line at 360°C (25 cc/kg) and 325°C (10.5 cc/kg). As explained previously,[3, 4] a reversing DCPD technique was used to monitor the voltage across the gauge section of each specimen in-situ. The voltage is sensitive to multiple phenomena including cracking, elastic and plastic strain, and resistivity evolution of the material. At the start of the test, the specimens were loaded to their target load (equal to the yield stress) over a period of ~1 hour at a constant strain rate of $\sim 10^{-5}$ mm/s while the stress versus strain evolution was constantly monitored using DCPD, providing direct evidence that the specimens had reached their yield stress. During the test, the voltage evolution of each specimen was measured with the belief that any strong deviations in response are likely to be due to crack initiation.

Microstructural Characterization

Microstructural examinations were conducted using a JEOL 7600[†] SEM. The routine approach was to use the SEM Oxford Aztec software to acquire montages of the entire gauge surface of all the specimens with automated stage movement. In order to achieve this, four fiducial scribe marks (90° to one another) were made at the button ends of each specimen to keep track of the specimen orientation. Each of the four orientations was then mapped using high-keV backscatter electron (BSE) montage imaging so that features covered by thin surface oxides can be revealed. In this way, the morphology and location of SCC precursors and cracks could be quickly documented, enabling the evolution of these features to be tracked over time. In addition, one specimen (IN151) was cross-sectioned at the plane intersecting a large crack after the test, and the cross-section morphology of the crack was examined at high resolution under low-kV BSE imaging.

[†] Trade mark.

RESULTS AND DISCUSSIONS

The overall referenced DCPD strain responses for the SA+15%CF Alloy 600 plate specimens IN151-153 tested at 360°C and IN209-211 tested at 325°C are summarized in Figure 3. The measured SCC initiation times for these six specimens are listed in Table 2. In addition, the SCC initiation times of IN151-153 tested at 360°C were compared to the data obtained on five other Alloy 600 heats at the same temperature as shown in Figure 4. It is clear that compared to most of the other 15%CF specimens, IN151-153 exhibited significantly lower and very consistent SCC initiation times ranging from 295-356 hours (Figure 3a). It should also be noted that the SCC initiation times obtained at 325°C for this material are also very consistent at ~1000-1350 h (Figure 3b), which fall in the same range as the majority of specimens from other heats tested at 360°C, again indicating the high SCC initiation susceptibility of this material. As shown in Figure 2, this SA material has a high level of B segregation at GBs that is much greater than most of the other Alloy 600 materials included in Figure 4.[6] Previous experience with mill-annealed commercial heats has indicated enhanced SCC susceptibility with high B content, although the mechanism is still unclear.[8, 9] In addition, the current material has significantly lower C content than typical Alloy 600 materials and was tested in the SA condition with clean GBs, whereas most of the other heats are in the mill-annealed condition with a certain degree of carbide coverage at GBs. The low bulk C concentration and the lack of GB carbides may have promoted a lower creep resistance in this material, which also contributed to more rapid SCC initiation.

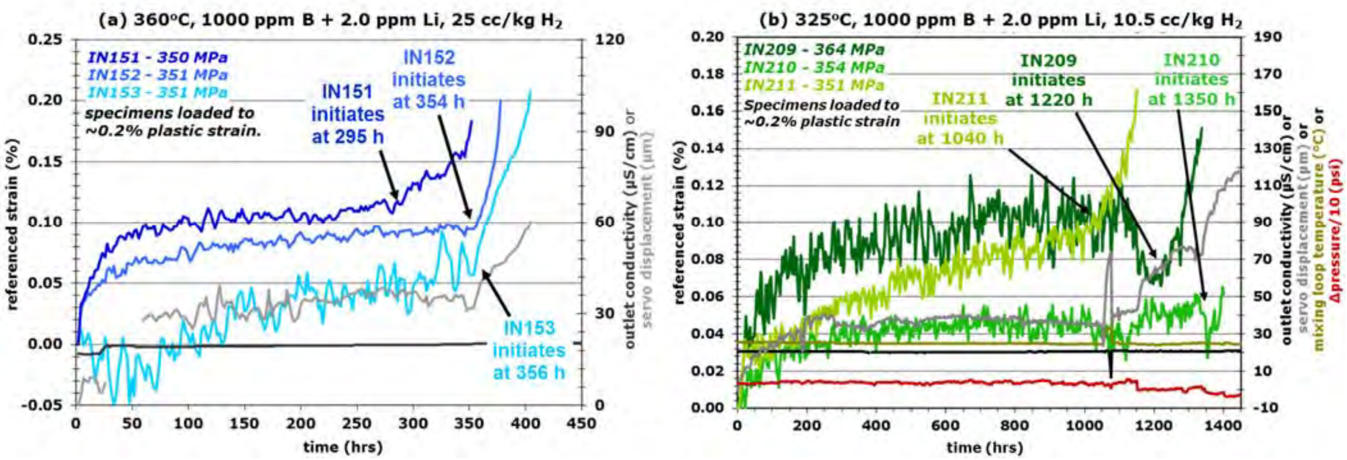

Figure 3: Referenced DCPD strain response showing SCC initiation for the SA+15%CF Alloy 600 plate specimens IN151-153 tested at 360°C (a) and IN209-211 at 325°C (b).

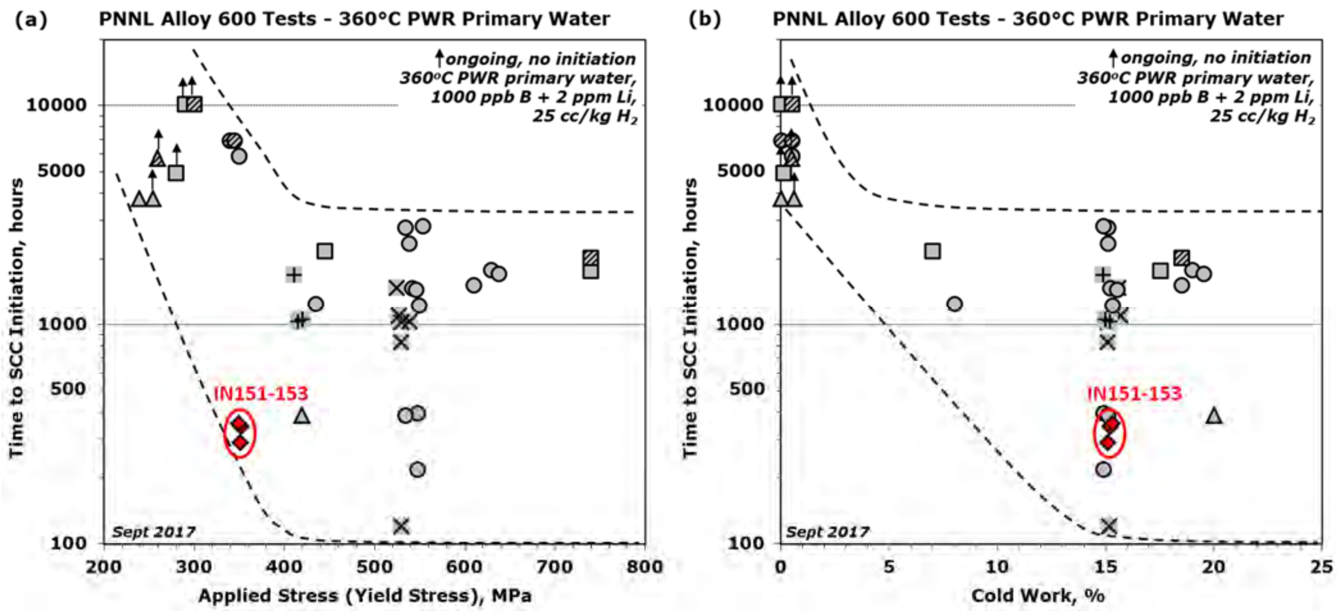

Figure 4: Measured SCC initiation time as a function of applied stress (a) and % cold work (b) of all Alloy 600 materials tested at 360°C at PNNL. The data for IN151-153 is highlighted in red. Dashed lines are meant to bound the data and aid in visualization of the initiation response.

<u>SCC Initiation Morphology of Specimens IN151-153 Tested at 360°C</u>

The earliest initiation was observed at 295 h for IN151, but with a slightly more gradual increasing slope in comparison to the other two specimens. Post-test examination at 352 h revealed a few large cracks with surface lengths ranging from ~150-350 µm. These cracks appear rather smooth on the surface indicating that they formed along only 1 or 2 grains. In order to have an idea on the depth of the cracks, this specimen was cross-sectioned along a plane intersecting a crack of medium size on the surface. As shown in Figure 5, this crack extended to a depth of ~580 µm, which is more than two times of its surface length. This confirmed that the specimen had fully initiated and it is expected that the cracks with longer surface length had grown even deeper inside the material.

IN152 and IN153 were also examined for indication of precursors and cracks when the test was interrupted at 352 h to remove the already initiated IN151. Somewhat surprisingly, two cracks (~300 µm long) were detected on the surface of IN152 (left image in Figure 6) while no obvious cracks were discovered on IN153 (left image in Figure 7). The two specimens were reloaded to yield stress and SCC initiation was detected almost immediately by DCPD at ~354 h for IN152 and ~356 hours for IN153. The test was then continued at a reduced load until IN152 failed in the system and was ended at 514 h. The fractured surface morphology is presented on the right side of Figure 6. Large grains of up to ~500 µm in diameter can be seen on the surface with sporadically distributed small grains of 100-200 µm in size. Interestingly, post-test gauge surface examination of IN153 only revealed three cracks with similar or shorter surface length ($\leq$200 µm) as those detected on IN151 and IN152 when exposed for much shorter time (Figures 5 and 6), suggesting little growth of the cracks after the load was reduced. It should also be noted that one crack in IN153 showed significantly wider opening in comparison to all cracks observed in the three specimens and had likely grown to a larger depth below the surface. The above findings suggest that instead of extending on the surface, the cracks in this CW alloy 600 plate material tend to favor growth in depth. This may indicate differences in SCC initiation susceptibility among GBs in this duplex microstructure. Additional investigations are planned to assess any local change in segregation and stress/strain distribution that may affect the SCC.

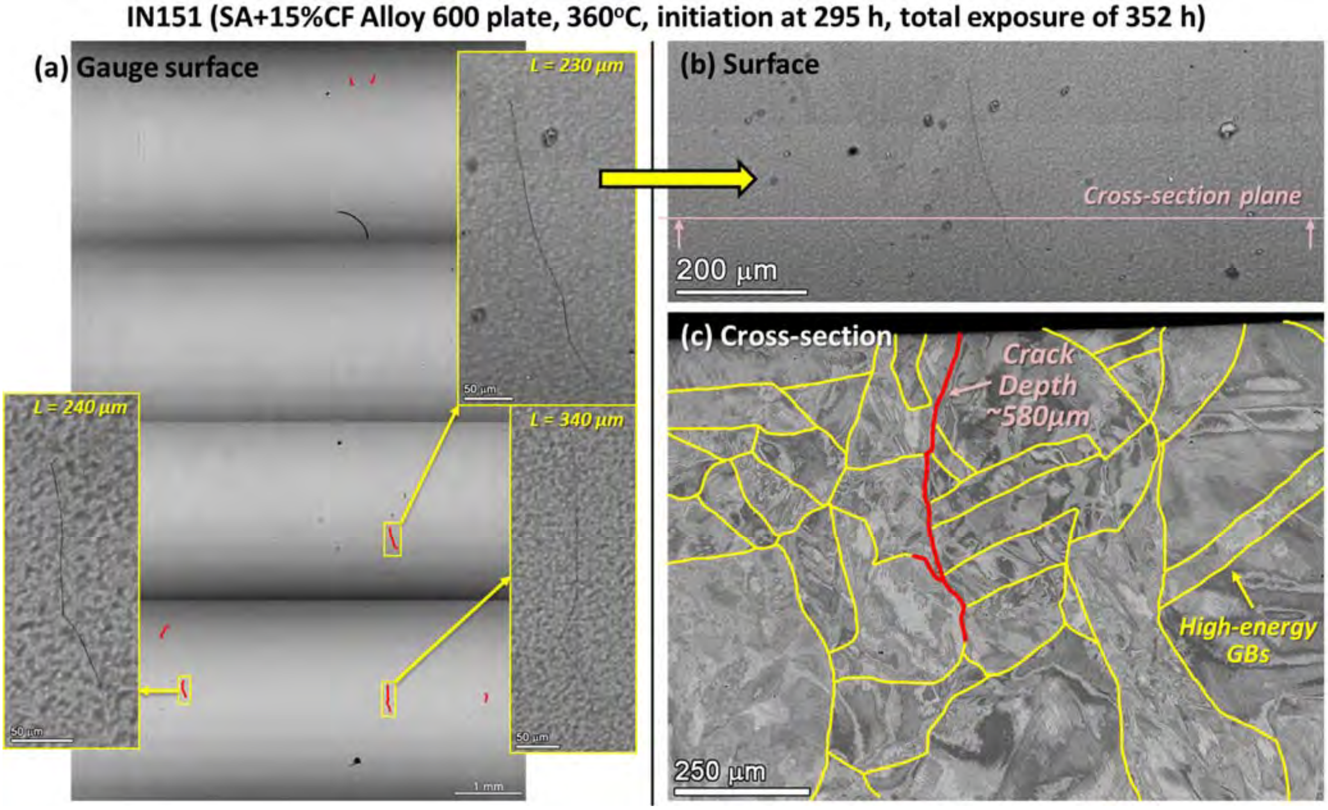

Figure 5: SEM-BSE montage image of the post-test gauge surface of SA+15%CF Alloy 600 plate specimen IN151 with obvious cracks are highlighted in red (a). The specimen was later cut into two halves (b) and the cross-section morphology of a medium size crack is shown with the crack highlighted in red and surrounding high-energy GBs in yellow (c).

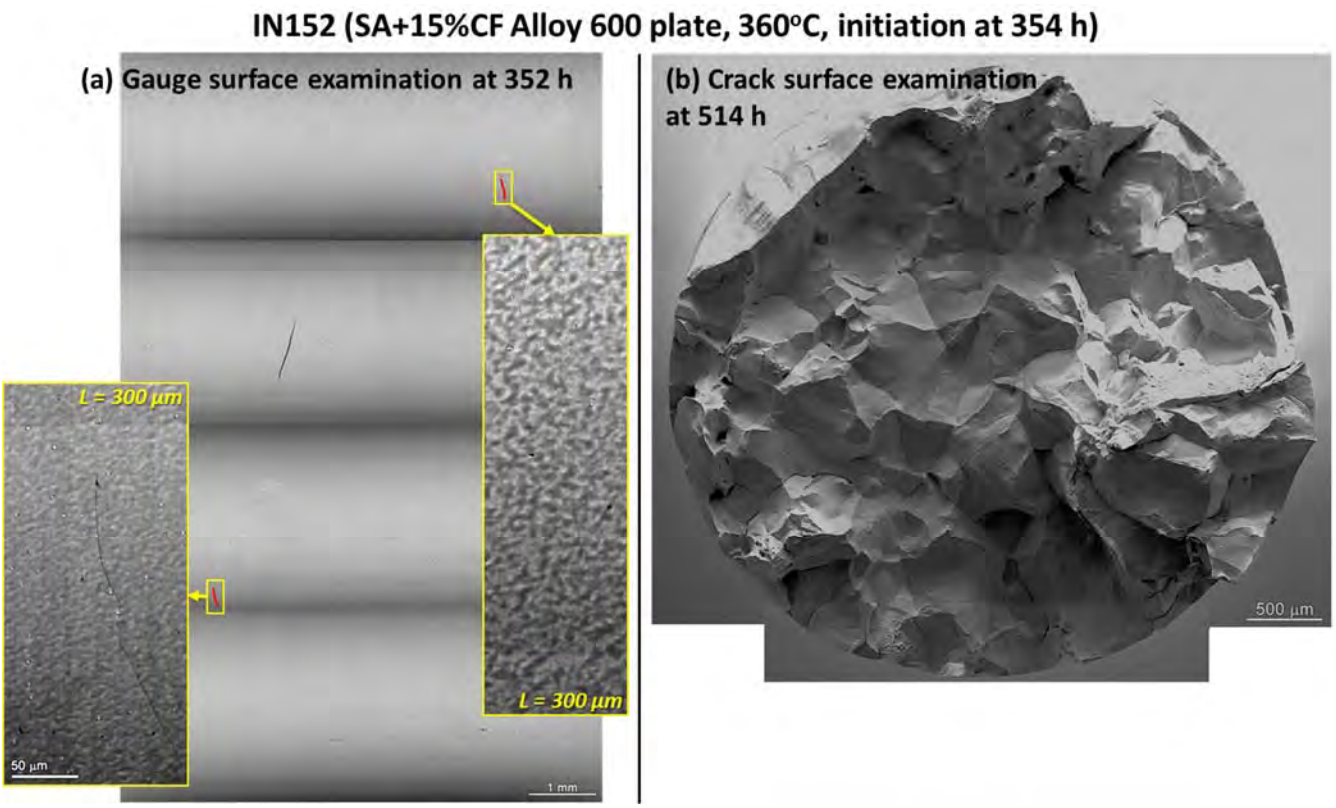

Figure 6: SEM-BSE montage image of the gauge surface of SA+15%CF Alloy 600 plate specimen IN152 right before SCC initiation detected ~2 h later, revealing two large cracks (a). The specimen was failed in the test and the fracture surface was examined after the test ended (b).

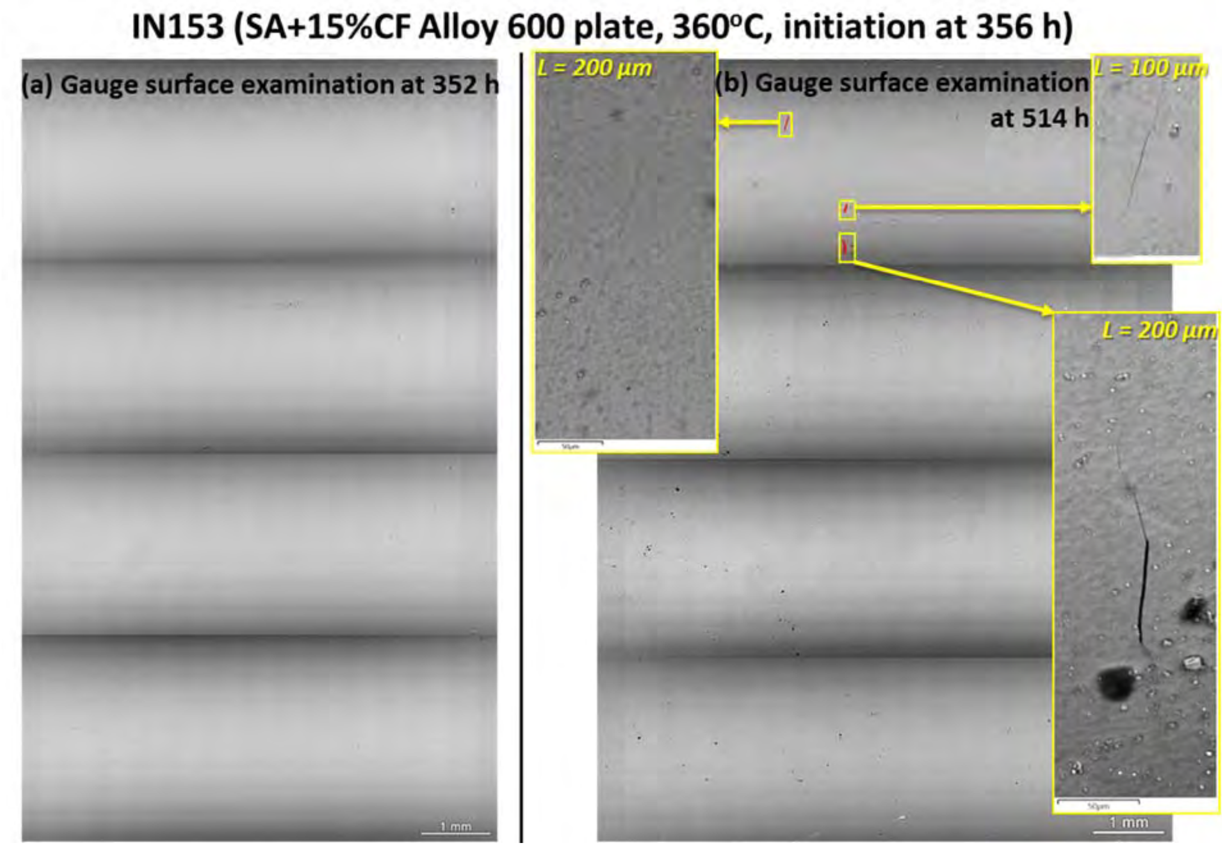

Figure 7: SEM-BSE montage image of the gauge surface of SA+15%CF Alloy 600 plate specimen IN153 at test interruption at 352 h right before SCC initiation detected ~4 h later (a) and after the conclusion of the test at 514 h (b).

<u>SCC Initiation Morphology of Specimens IN209-211 Tested at 325°C</u>

SCC initiation during 325°C testing first occurred in IN211 at 1040 hours and the test was interrupted at 1150 h to remove this specimen. As shown in Figure 8, only one large crack was found on the gauge surface. This crack extended to a surface length of ~800 µm and seems to have been created by coalescence of two smaller IG cracks as indicated in the image. SEM examinations were also performed on IN209 and IN210 after the test was interrupted at 1150 h and again shortly after SCC initiation was detected in each specimen, enabling evolution of surface morphology to be viewed over time. As shown in Figure 9, these two specimens didn't exhibit any obvious cracks at the test interruption after 1150 h of exposure, but both initiated within 100-200 h soon after the test was restarted. Closer examinations on surface morphology evolution were performed at selected sites in both specimens with examples shown in Figure 10. Relatively large cracks (surface length of 300-400 µm) were discovered after 1338-1400 h, while no evidence for obvious IGA or small cracks could be identified in these same locations during the 1150-h examinations. This indicates that the transition from IGA to short cracks then to macroscopic SCC initiation is very fast in this material. While high susceptibility of SCC initiation at certain large grains might have played an important role, further investigation is needed to better elucidate the mechanisms controlling the transition to SCC initiation.

Preliminary examinations of cracks suggest that the initiation time detected by DCPD represents a transition to rapid crack growth that occurs after the formation and coalescence of short cracks, which can be considered "practical" SCC initiation.[4, 7] Based on serial polishing and detailed examinations performed to record the crack shape on a large number of cracks in CW specimens in multiple alloy 600 heats, the onset of the macroscopic SCC initiation appears to correspond to the primary crack reaching a surface length of ~250 µm and a depth of ~100 µm. This was further correlated to a *K* level of ~10 MPa√m that is believed to trigger sustained crack growth at engineering relevant rates in these CW, highly SCC susceptible materials.[4] While serial polishing and detailed examinations were not performed on crack shapes for the newly tested UNS N06600SA plate specimens, the cracking morphology on gauge surfaces is in agreement with the previous findings. The primary cracks found after SCC initiation in these specimens normally have a surface length of 250-300 µm. As shown in Figure 5, these cracks tend to have a much greater depth than their surface length, confirming rapid crack propagation took place after SCC initiation. It should also be noted that based on surface morphology recorded at test interruptions and at the conclusion of the tests, the transition from shallow IGA to practical SCC initiation in the 15%CF specimens took place within a very short time period relative to the total exposure time before crack initiation was detected by DCPD. This is evidenced in Figures 7 and 9 where IG cracks were not observed during examinations immediately before SCC initiation was detected by DCPD in multiple specimens tested at different temperatures. While earlier results have shown a much more gradual and identifiable transition from shallow IGA to small cracks then to stable crack growth in the non-CW specimens,[4, 7] the new results suggest that this process was greatly shortened in this highly SCC susceptible CW alloy 600 heat.

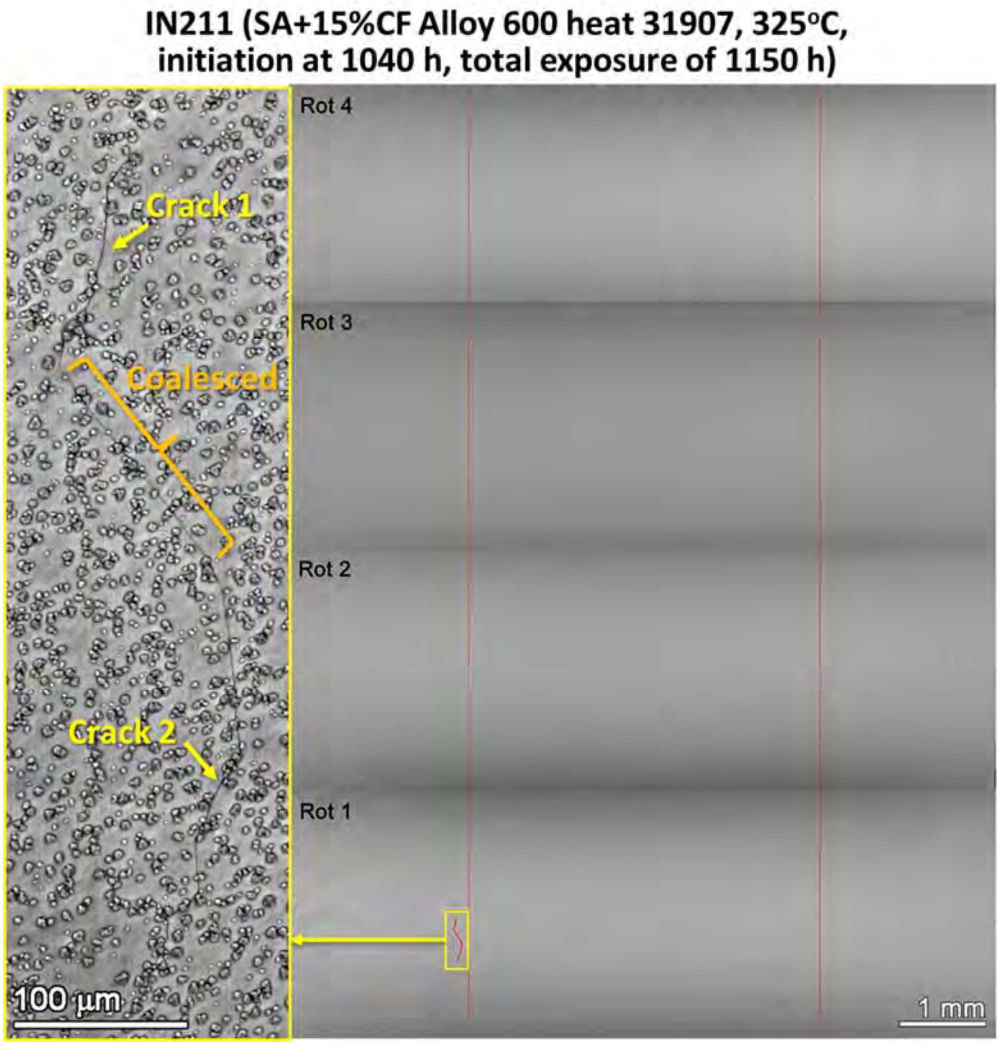

Figure 8. Post-test SEM-BSE montage image of the SA+15%CF SA+15%CF Alloy 600 plate specimen IN211.

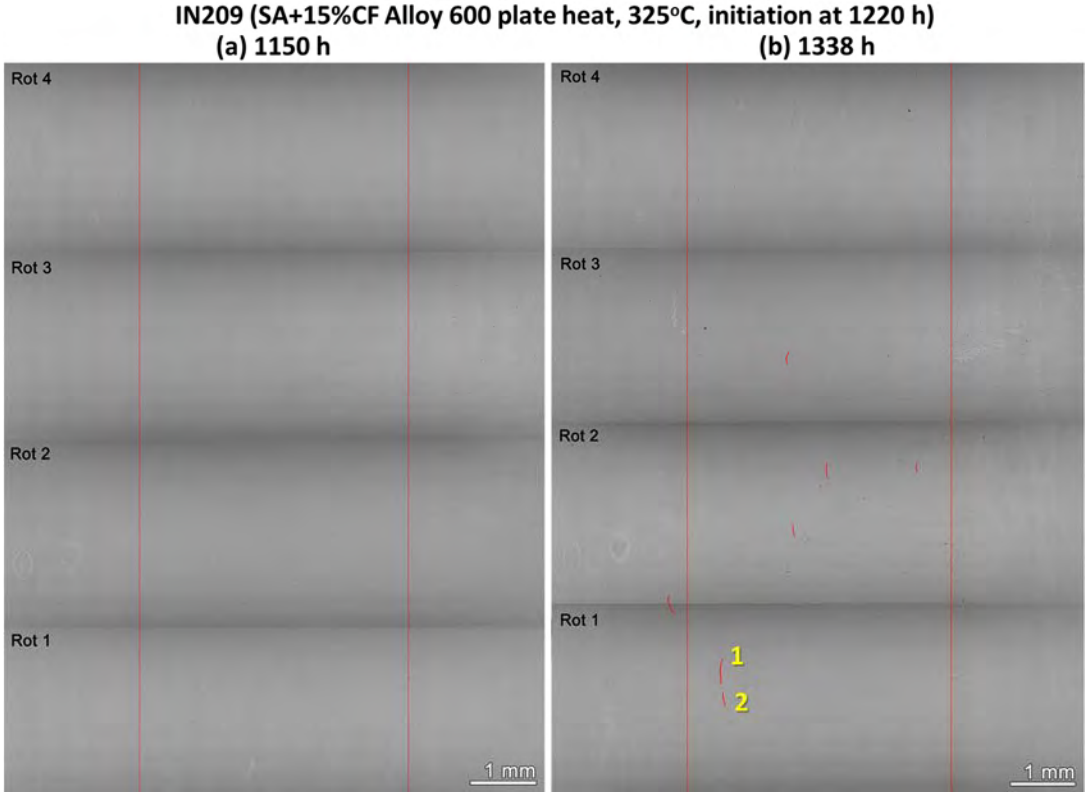

Figure 9: SEM-BSE montage image of the SA+15%CF Alloy 600 plate specimen IN209 after test interruption at 1150 h (a) and after being removed from the system at 1338 hours after detection of SCC initiation (b).

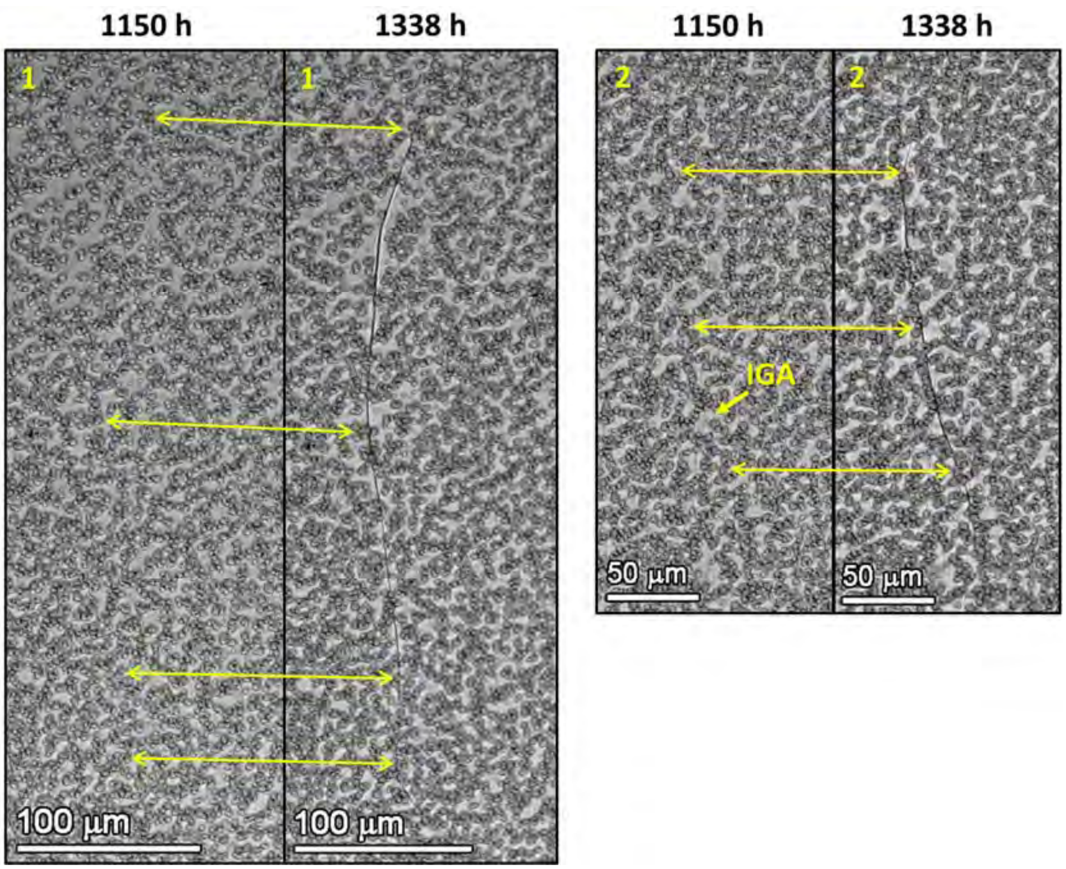

Figure 10. SEM-BSE montage image of surface morphology evolution at Sites 1 and 2 marked in Figure 9 in the SA+15%CF Alloy 600 plate specimen IN209 at test interruption and after SCC initiation was detected by DCPD.

Estimation of Thermal Activation Energy

Most of the SCC initiation testing at PNNL is performed in an accelerated manner at temperatures higher than those in normal PWR operation conditions. In order to better estimate SCC initiation behavior of materials used in service, the effect of test temperature on SCC initiation has been investigated using this SA+15%CF Alloy 600 material because its high susceptibility to SCC initiation. As a result, tests at lower temperatures could be completed within a reasonable amount of time. While data is still limited, the SCC initiation time data obtained on the specimens at 360 and 325ºC have enabled an estimation of thermal activation energy for SCC initiation. As shown in Figure 11, the SCC initiation results revealed a thermal activation energy of ~116 kJ/mol. It should also be noted that solution annealing is not a typical heat treatment for the Alloy 600 materials used in service. As this heat treatment and water quench essentially removes GB carbides, it may increase SCC susceptibility and reduce variability often observed for mill-annealed materials. However, the obtained activation energy is still somewhat lower than the values (134-140 kJ/mol) reported by other researchers[10] on multiple SA alloy 600 heats, while being slightly higher than that reported (103 kJ/mol) for a high-temperature annealed alloy 600 heat.[11] More study is needed to clarify the rate controlling process and better understand the heat-to-heat variability within Alloy 600 materials.

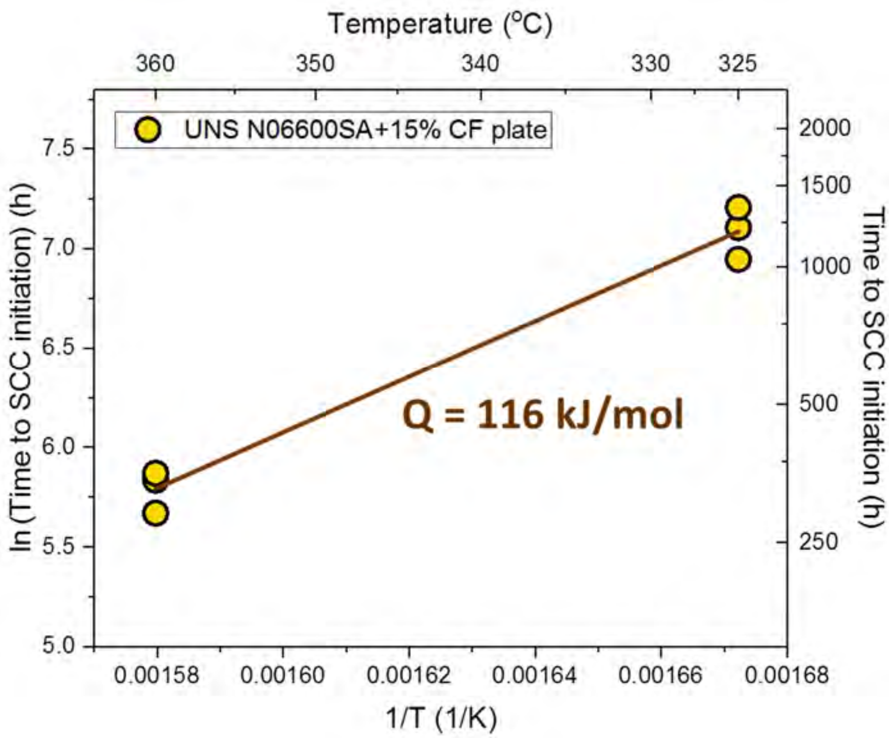

Figure 11. Extrapolated thermal activation energy for the SA+15%CF Alloy 600 plate tested at 360ºC and 325ºC with a dissolved H_2 concentration corresponding to the Ni/NiO stability line.

CONCLUSIONS

Constant load tensile tests with in-situ DCPD crack detection was performed on a SA+15%CF Alloy 600 material in simulated PWR primary water to investigate its SCC initiation behavior. Consistent SCC initiation times were obtained for this material at both 360°C (290-354 h) and 325°C (1040-1350 h) that were shorter than most other Alloy 600 heats in a similar CW condition. High level of B segregation at GBs and low C in the bulk is considered to have promoted the higher susceptibility, but further investigation is needed. SEM examinations at test interruptions and after the test revealed that the transition from IGA to large crack in the CW specimens took place over a very short time period relative to the total exposure time. A thermal activation energy of 116 kJ/mol is estimated for SCC initiation based on results obtained at 360 and 325°C.

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APPENDIX

1. "***Intergranular Stress Corrosion Crack Initiation and Temperature Dependence of Alloy 600 in Pressurized Water Reactor Primary Water***" presented in the Corrosion in Nuclear Systems Symposium as part of the CORROSION 2018 Conference.
2. "***Microstructural Comparison of Intergranular Attack in Alloy 600 in the SA versus TT Conditions Exposed to Simulated Primary Water***" presented in the Nickel-Base Alloys Session at the 2018 ICG-EAC meeting.
3. "***SCC Initiation of Alloy 182 in PWR Primary Water***" presented in the Weldments Session at the 2018 ICG-EAC meeting.

Pacific Northwest NATIONAL LABORATORY

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Intergranular Stress Corrosion Crack Initiation and Temperature Dependence of Alloy 600 in Pressurized Water Reactor Primary Water

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Pacific Northwest National Laboratory

CORROSION 2018, Phoenix, AZ, April 15-19, 2018

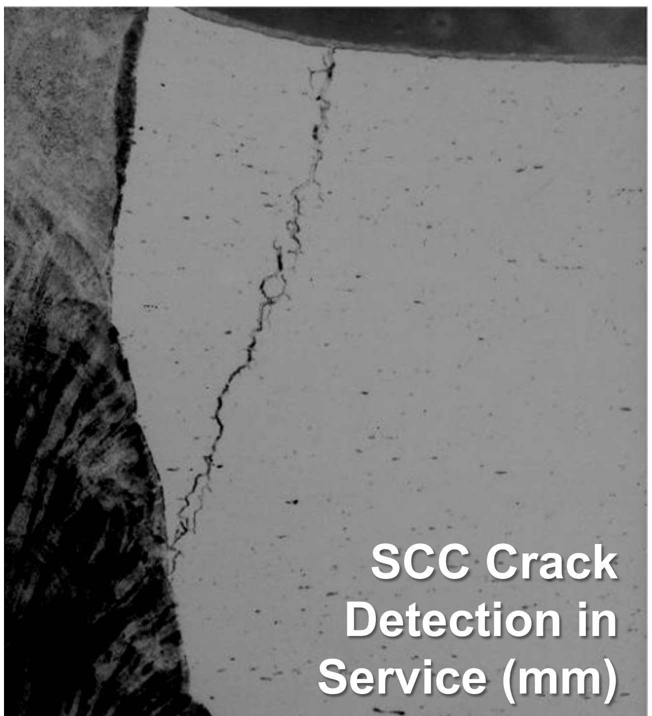
U.S. DEPARTMENT OF ENERGY

LWRS Light Water Reactor Sustainability

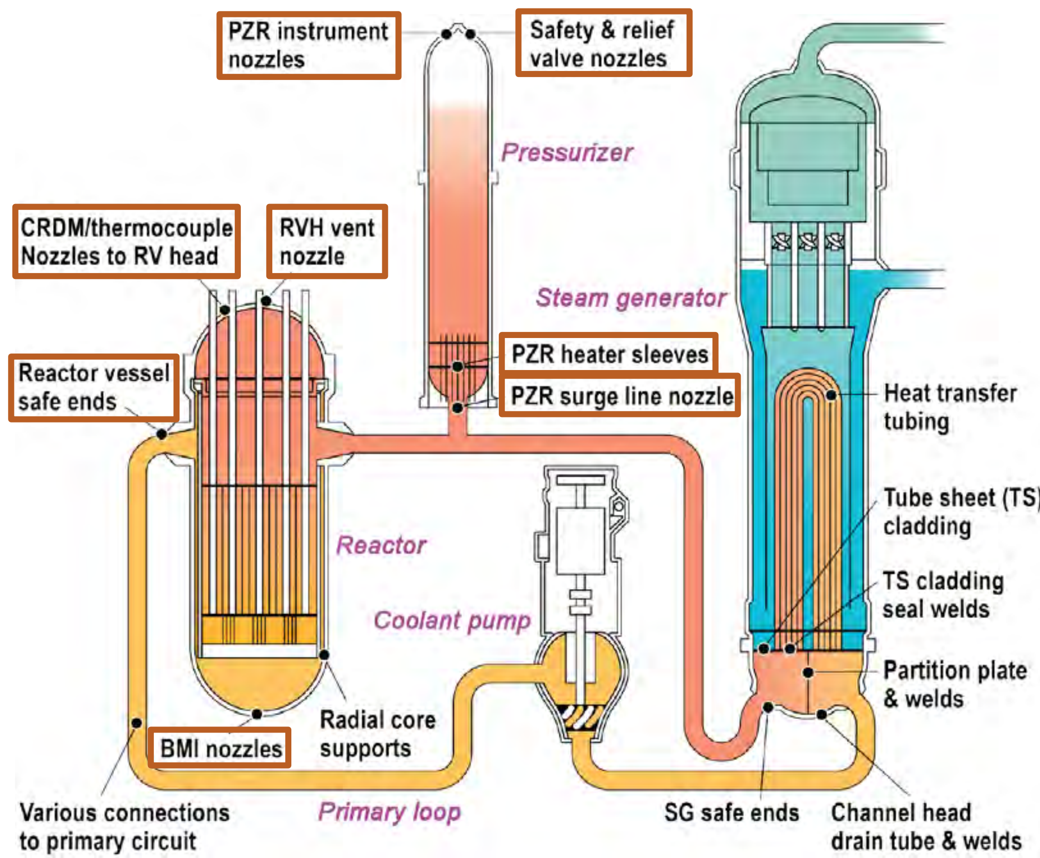
Background

- **Nickel-base alloy 600 (Ni-15Cr-8Fe)**
  - **Important structural materials for pressurized water reactor (PWR) pressure boundary components**
  - **Corrosion-resistant but susceptible to intergranular (IG) stress corrosion cracking (SCC) in high-temperature water**

IGSCC behavior needs to be evaluated for life extension of PWRs.

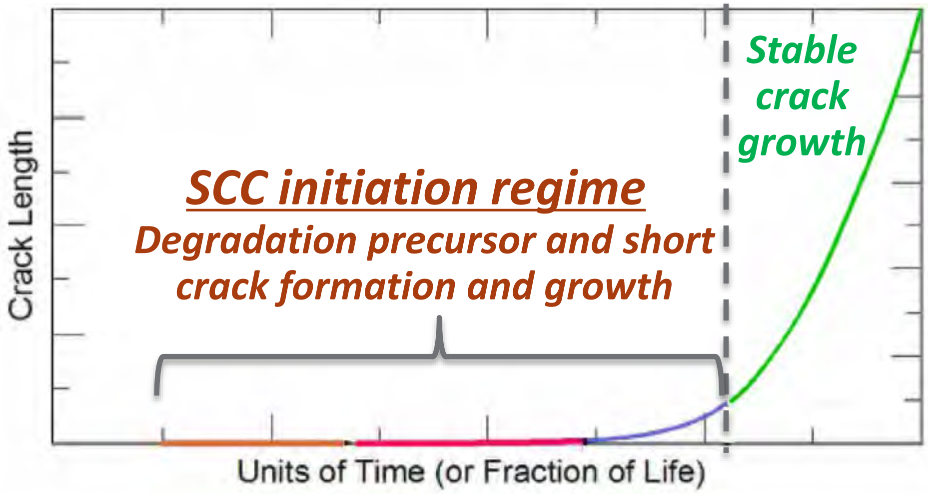


Locations in PWR primary circuits where Alloy 600 is still in use



Alloy 600 SCC Initiation Research at PNNL

- Focuses on mechanistic understanding and important influencing factors (material, environmental, stress and strain)
- Approach: advanced SCC initiation testing + high-resolution microscopy



PNNL Alloy 600 SCC Initiation Testing Status at PNNL

Heat	# of tested specimens		
	360°C	342°C	325°C
EPRI/GE SA Plate 31907	CW: 3	CW: 3 (ongoing)	CW: 3
PNNL MA Plate NX6106XK-11	Non-CW*: 3, CW: 13		CW: 3
Rolls Royce SA Plate 11415	CW: 3		
MA CRDM Plate 93510	Non-CW: 3, CW: 3		
Davis-Besse MA CRDM M3935	Non-CW: 3, CW: 1		
KAPL MA Plate 33375-2B	CW: 6		
MA Plate 522068	CW: 3		

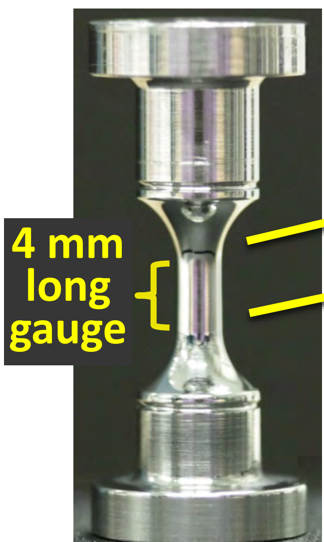
*CW = cold-worked

Alloy 600 SCC Initiation Testing at PNNL

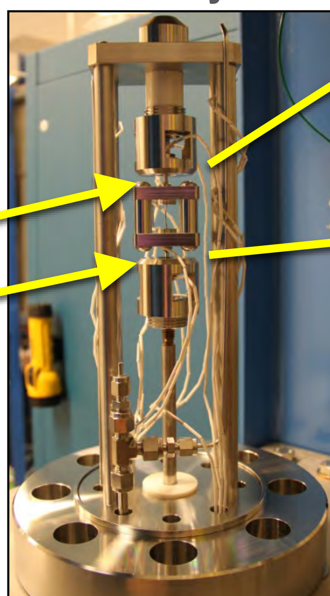
SCC initiation test systems with active loading and in-situ DCPD crack-detection:

- **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.

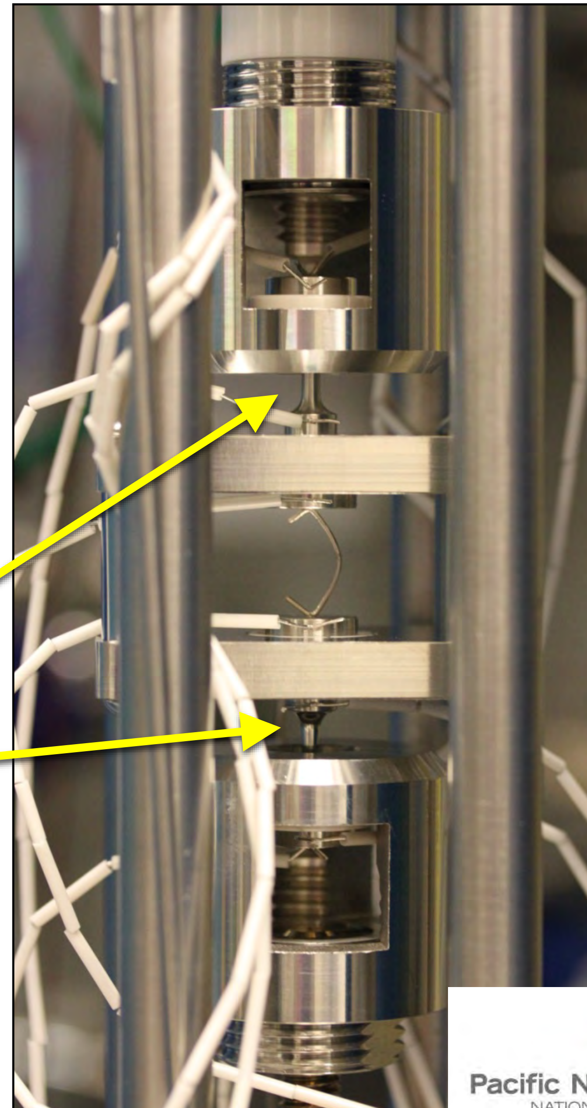
1.2" Tall SCC Initiation Specimen



Small SCC Initiation System



Small SCC Initiation System



36-Specimen SCC Initiation System

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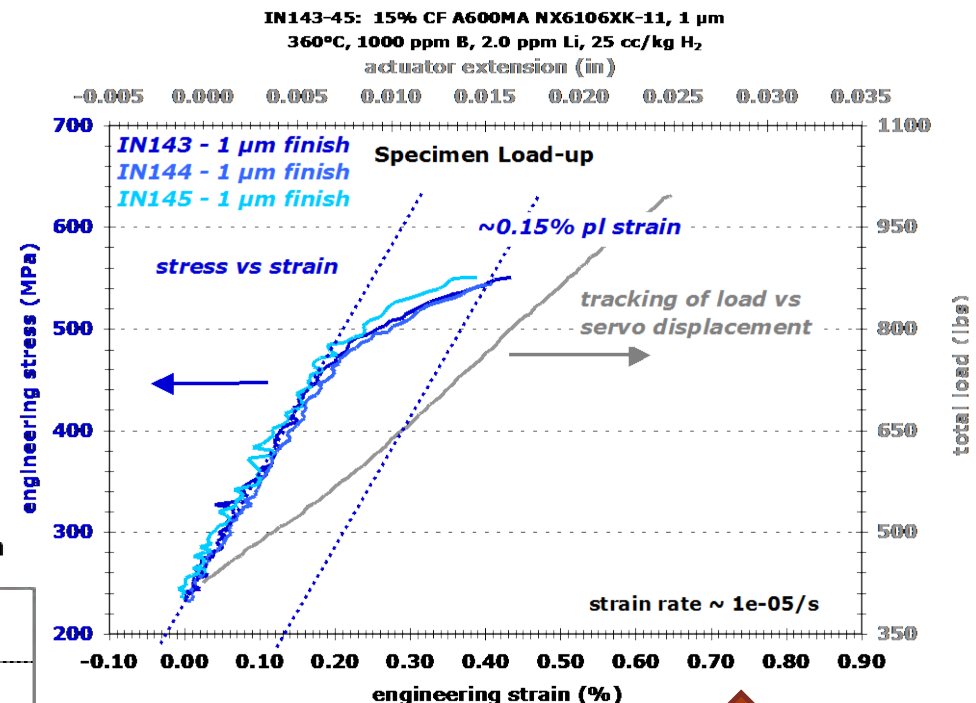
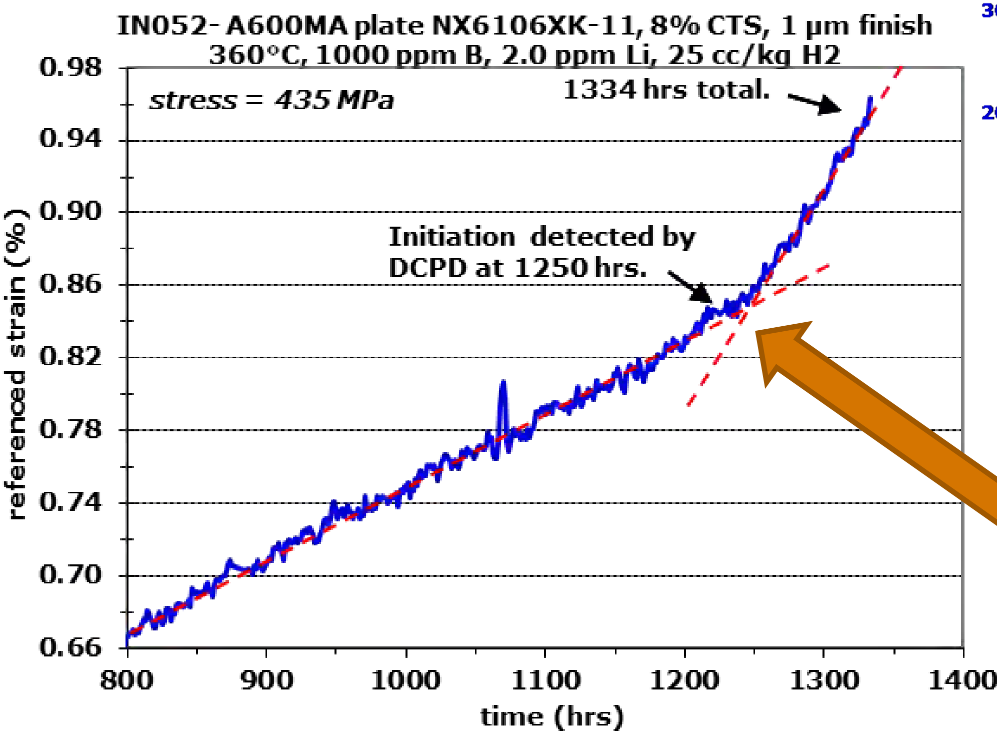
Alloy 600 SCC Initiation Testing at PNNL

- Tested in 360°C simulated PWR primary water at Ni/NiO stability line (1000 ppm B + 2 ppm Li, 25 cc/kg H_2).
- Constant load with applied stress at material yield strength.





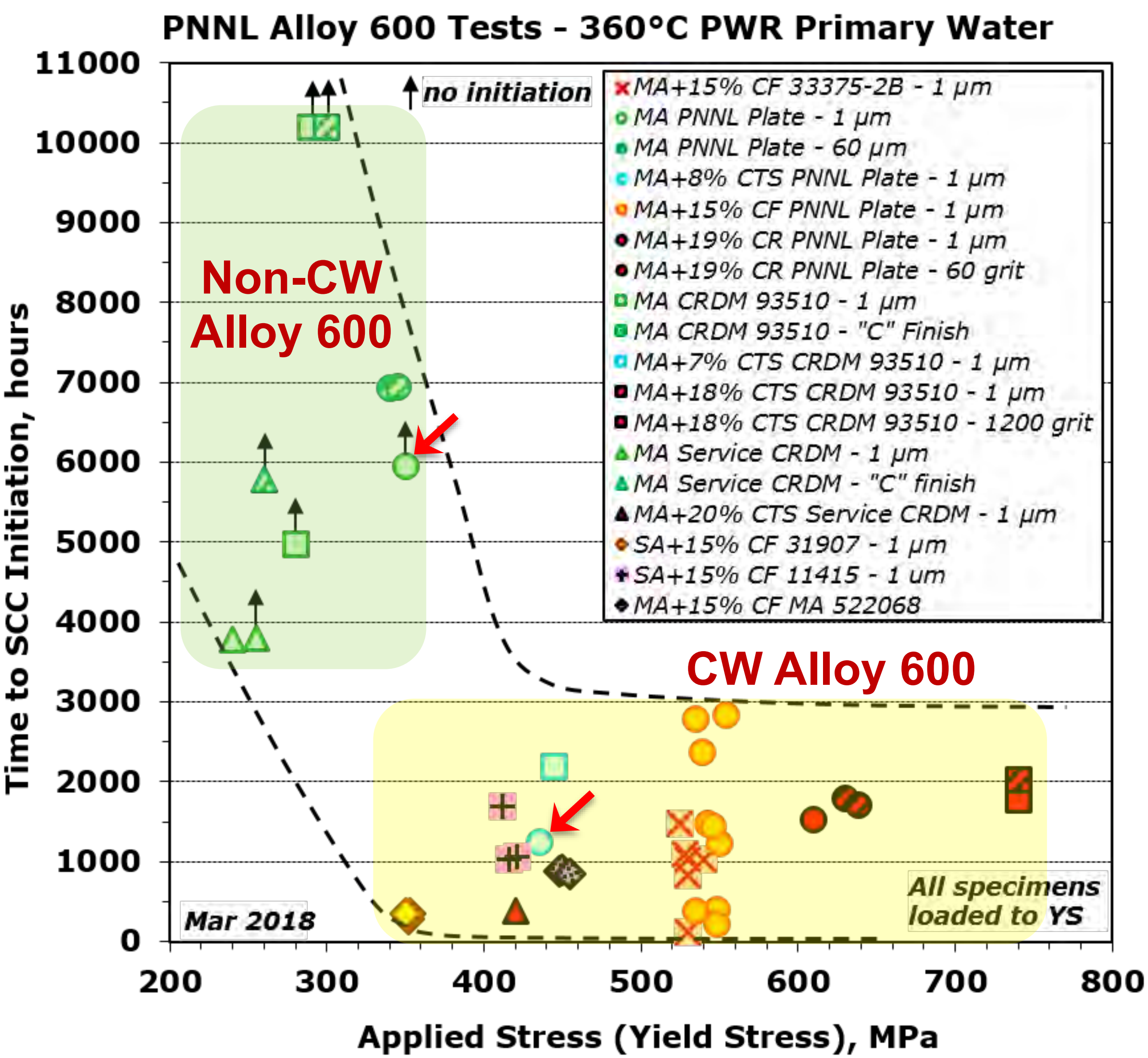
All specimens **loaded to yield stress at start of test** and **instrumented for crack initiation (also detects creep strain)**.

Alloy 600 SCC Initiation Testing at PNNL

Alloy 600 materials:

- 7 heats, mill-annealed (MA) or solution-annealed (SA)
- Non cold-worked (CW) and 7-20% CW
- 49 specimens in total

Small amount of cold work greatly reduces SCC initiation time to ≤3000 hours.

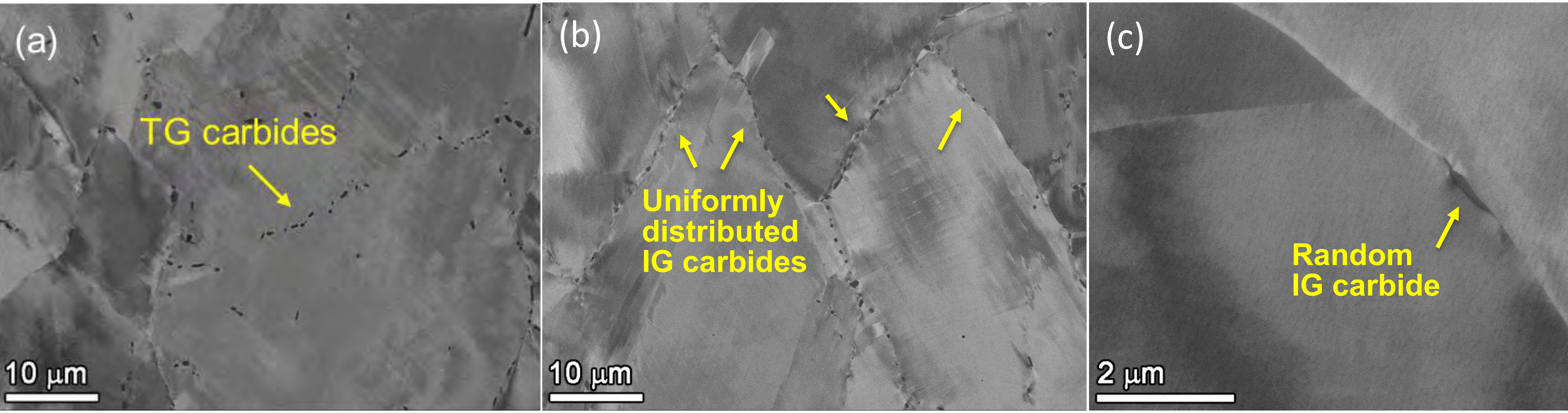


Alloy 600 Material (Example: Heat NX6106XK-11)

	Heat	Ni	Cr	Fe	Mn	C	Si	Cu	P	S	B, appm
Plate	NX6106XK-11	74.0	16.4	8.5	0.23	0.060	0.22	0.01	0.004	0.001	83

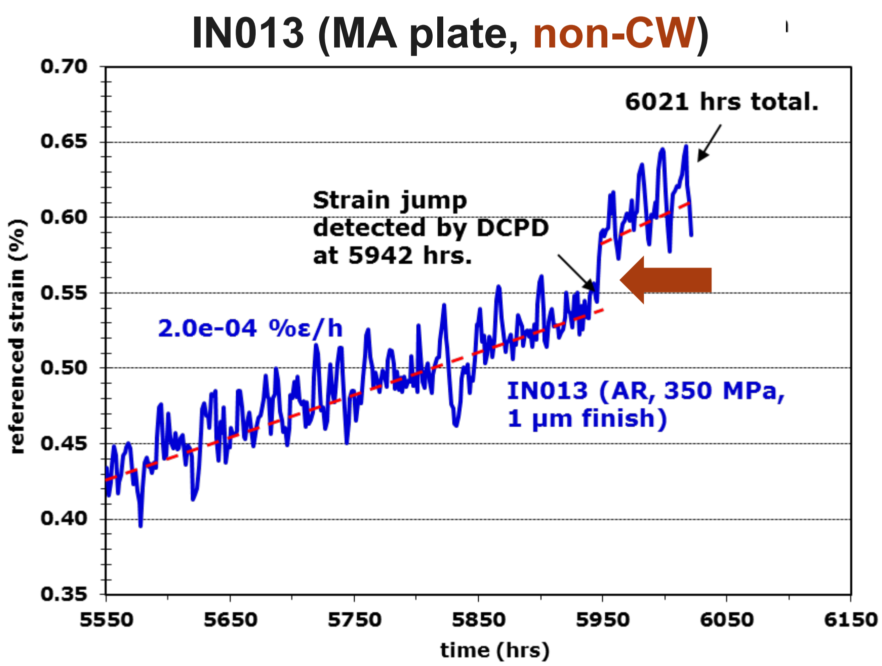
Mill-annealed at 927°C for 3.5 hours + water quench

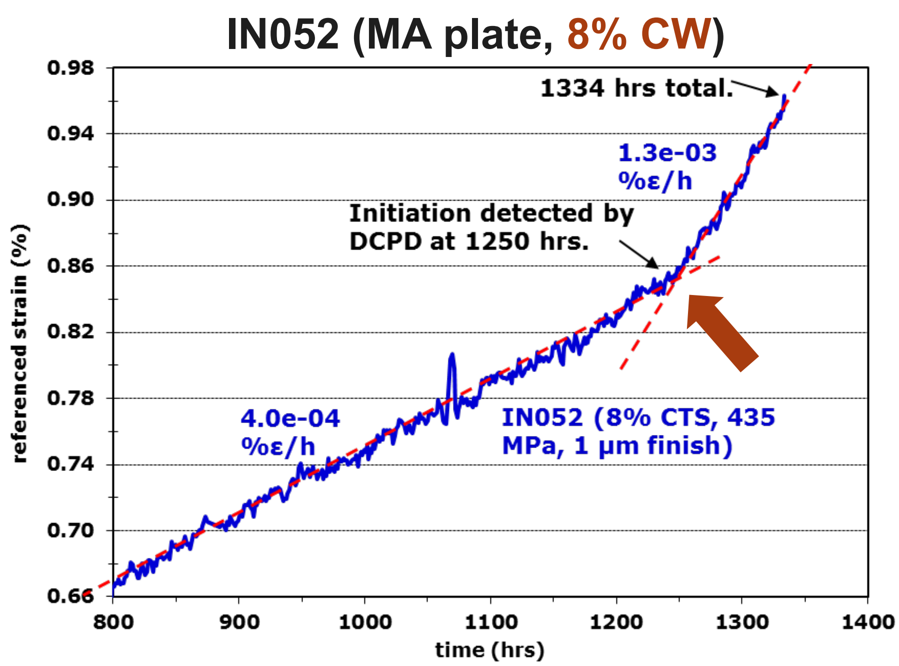
a) Most grains show primarily TG carbides.
b) Few grains show higher density of IG carbides.
c) More typical grain boundary, low density of IG carbides.



Alloy 600 SCC Initiation: Typical DCPD Response

Spec. ID	CW level	Finish	Applied stress (MPa)	Initiation Time (h)
IN013	Non-CW	1 µm	350	5942
IN052	8% CW	1 µm	435	1250



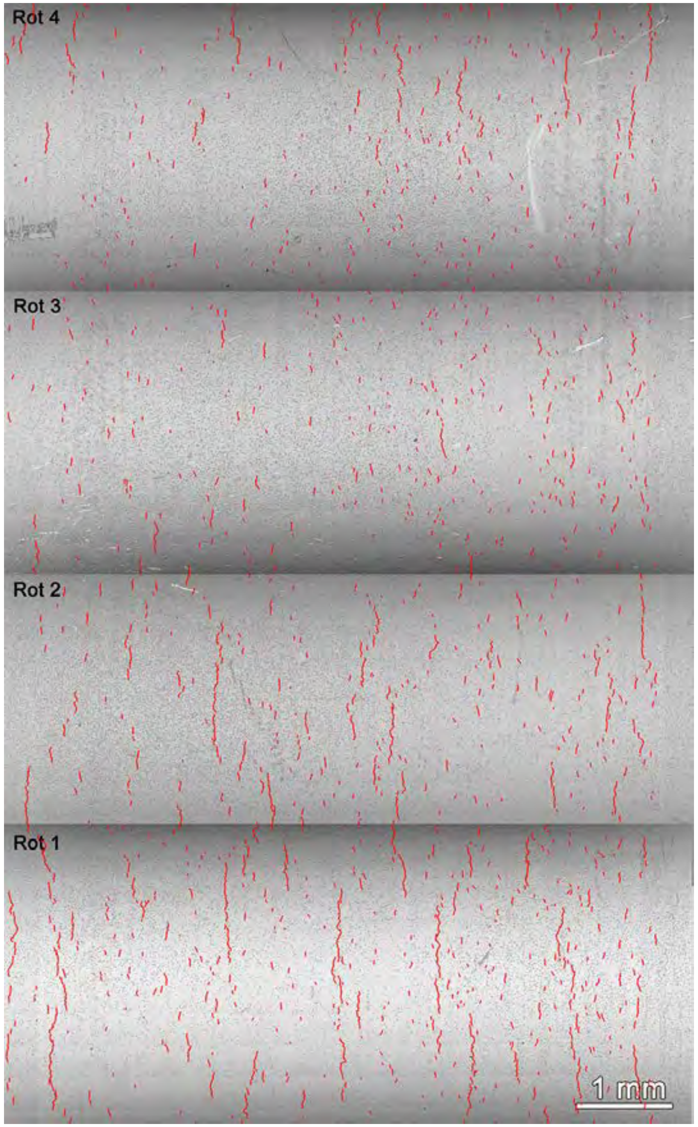


Gradual transition to a higher strain rate or strain jumps

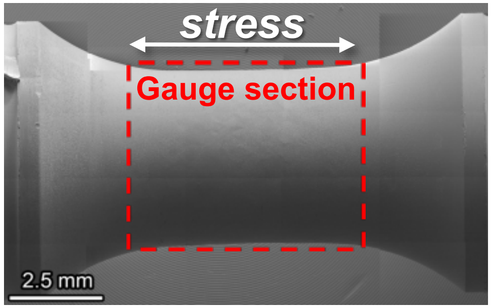
Distinct and rapid transition to a higher strain rate

Alloy 600 SCC Initiation: Morphology Overview

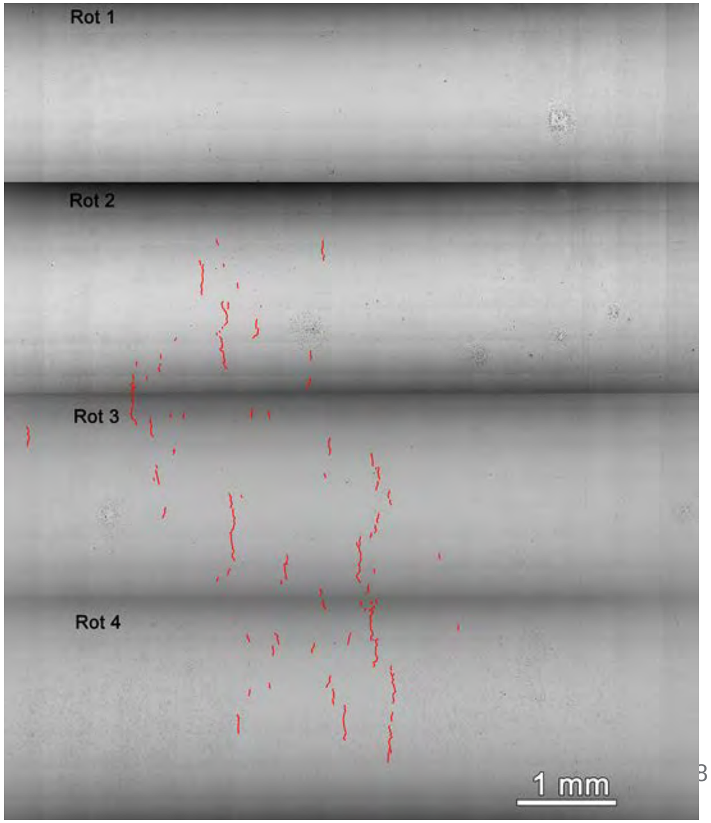
IN013 (MA plate, non-CW, 6021h)



Four rotations of the specimen was montaged using SEM to record the entire gauge surface



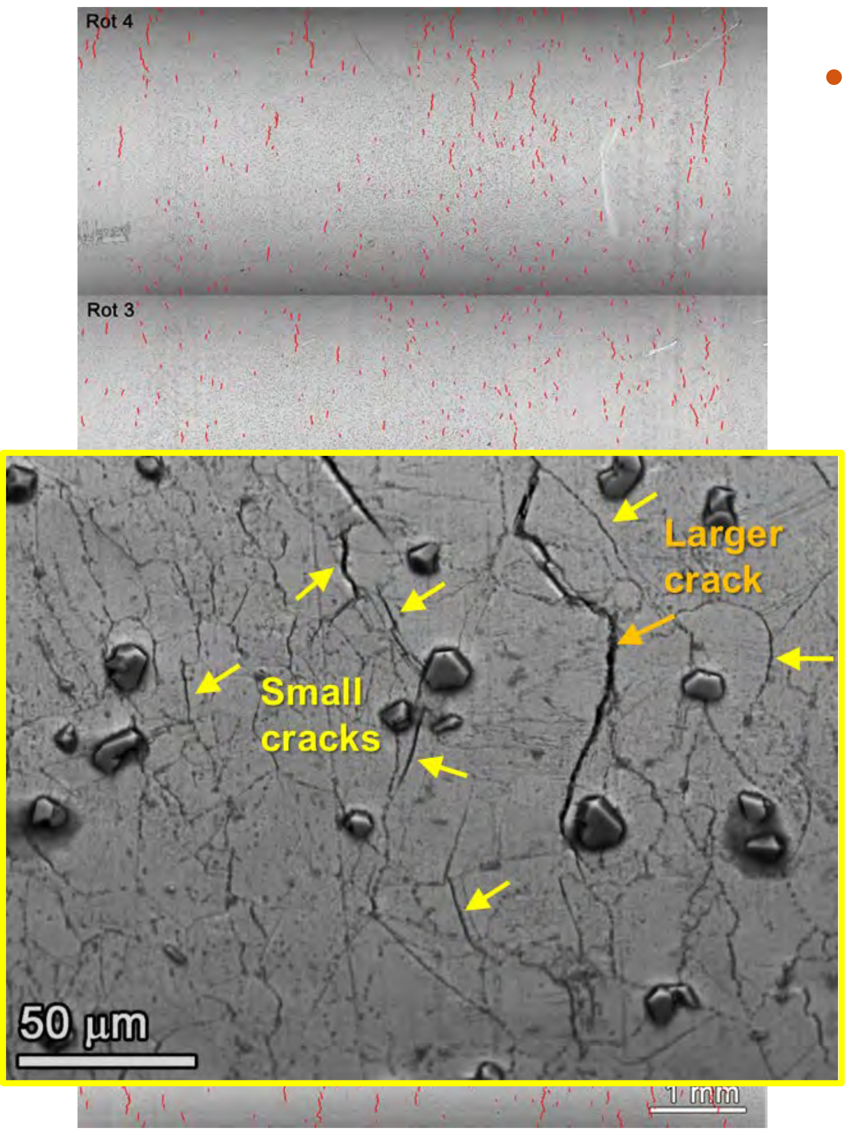
IN052 (MA plate, 8%CW, 1334h)



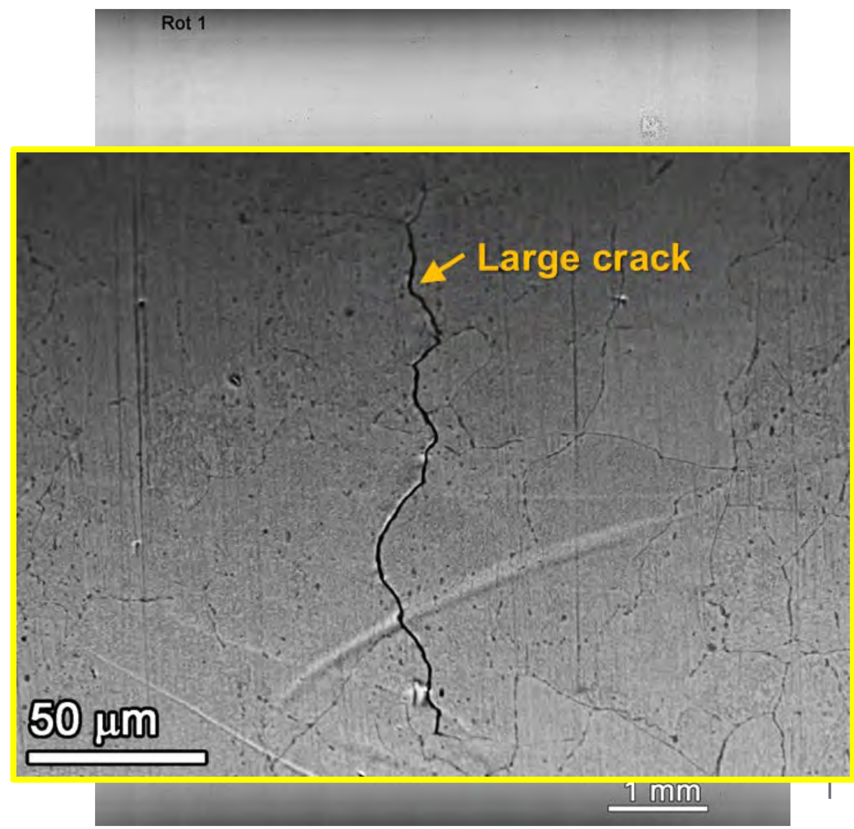
Alloy 600: Surface Morphology Overview

- Much higher crack density in as-received, non-CW specimens due to longer exposure.
- Extensive intergranular attack (IGA) on the surface transitions to small, shallow IG cracks early in alloy 600 specimen tests.

IN013 (MA plate, non-CW, 6021h)

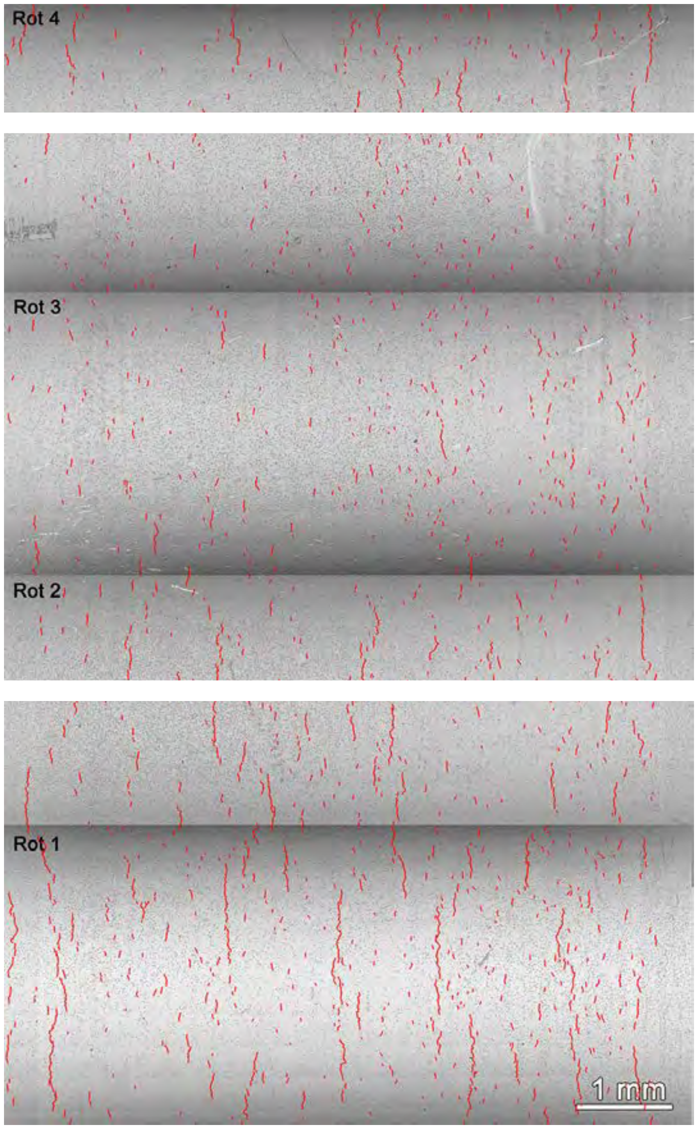


IN052 (MA plate, 8%CW, 1334h)



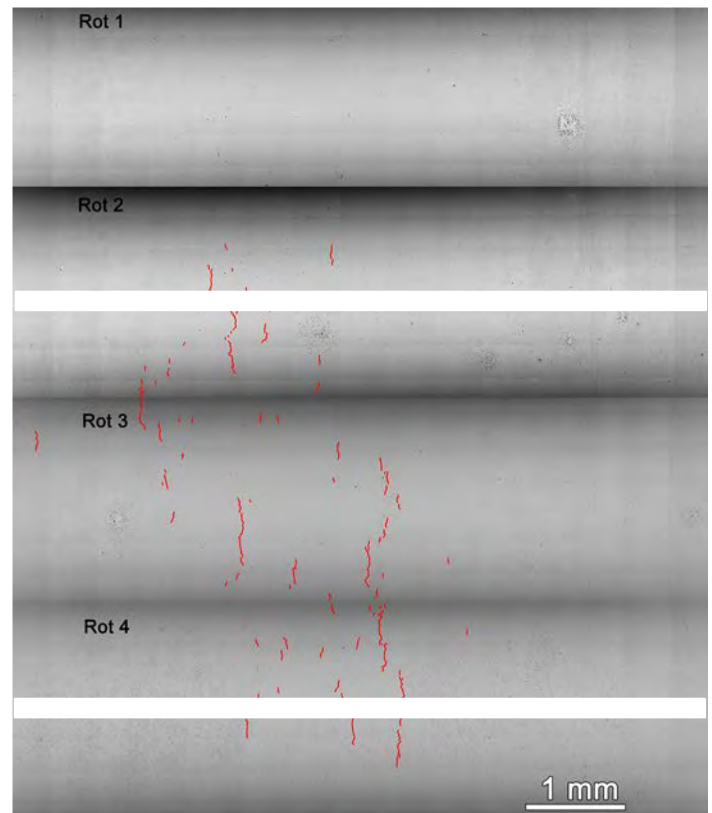
Alloy 600: Cross-Section Observation

IN013 (MA plate, non-CW, 6021h)



Cross-section plane

IN052 (MA plate, 8%CW, 1334h)



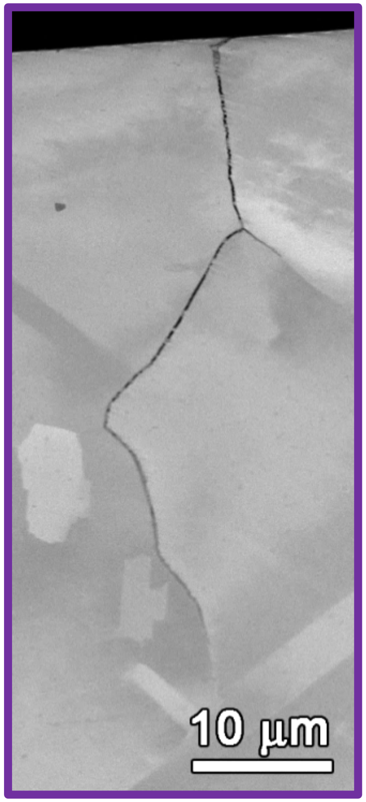
Alloy 600: Cross-Section Morphology Overview

SCC initiation in alloy 600 in PWR primary water evolves in 3 stages:

Stage A
Intergranular attack (IGA)

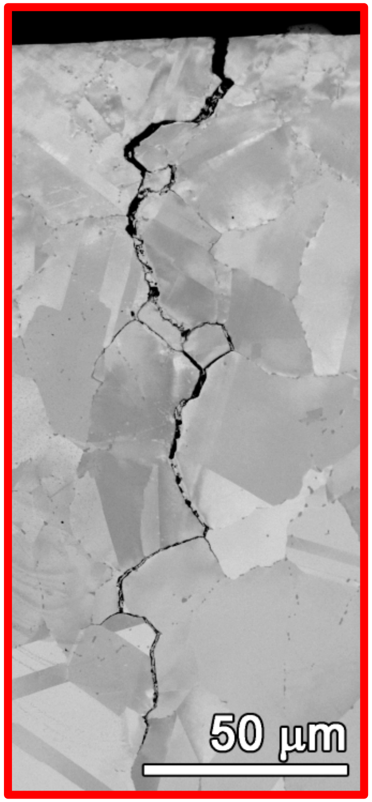
IGA begins immediately on high-energy GBs intersecting the surface.

Stage B
Short crack growth and coalescence



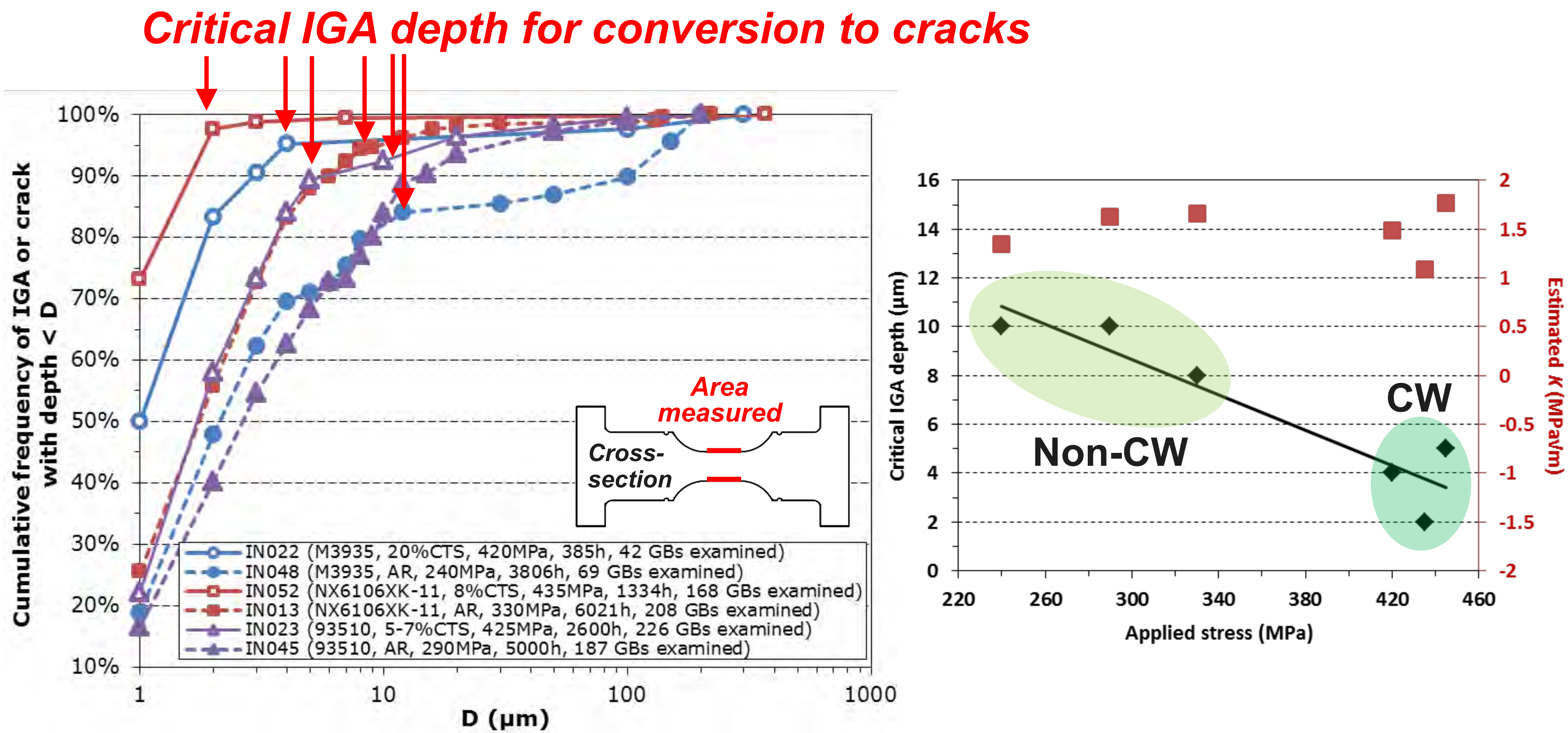
Crack nucleation from IGA, short crack growth and coalescence.

Stage C
Transition to stable crack growth



Cracks reach a sufficient size to produce a stress intensity for sustained growth.

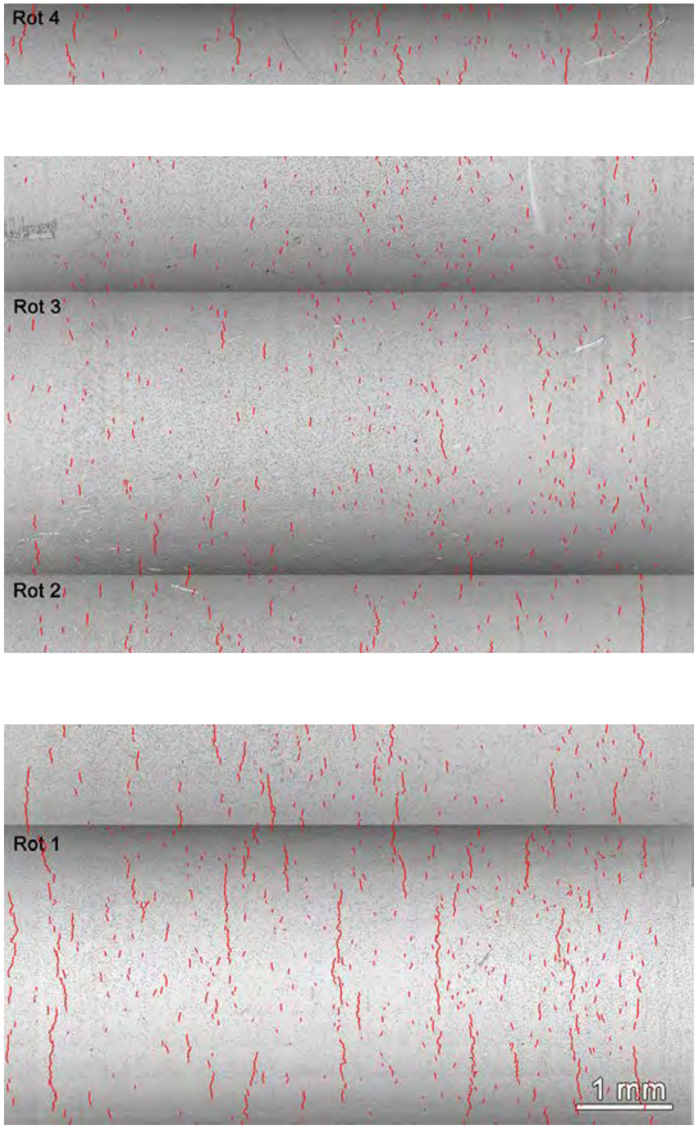
Alloy 600 SCC Initiation:
IGA and Short Crack Distribution



- More than 90% of the high energy grain boundaries are associated with shallow intergranular attacks (IGA) and cracks.
- The critical IGA depth exhibits an inverse linear dependence on applied stress, indicating the onset of a more important role played by stress.

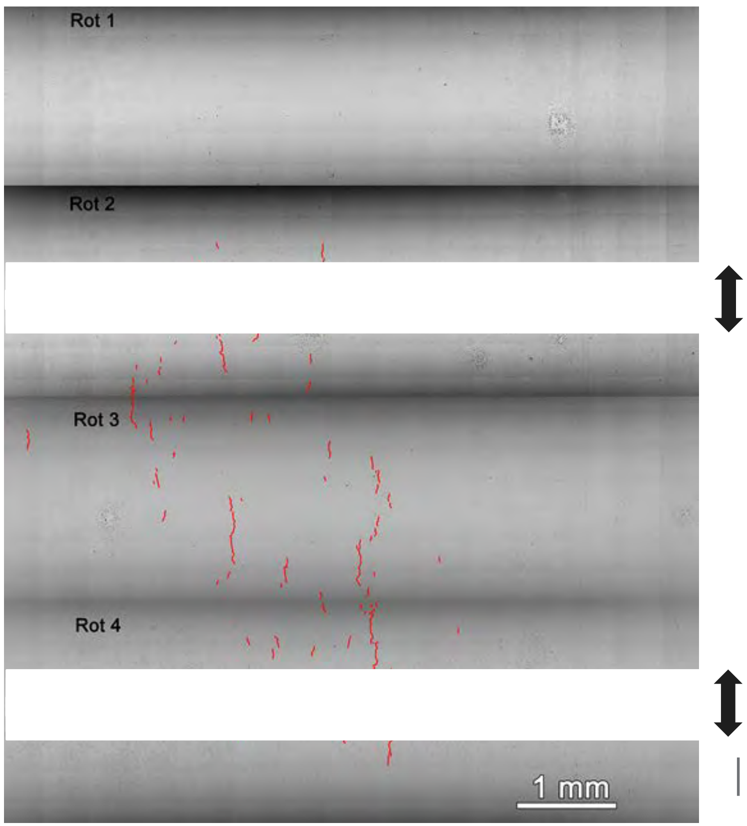
Alloy 600: Serial Polish and Examination

IN013 (MA plate, non-CW, 6021h)



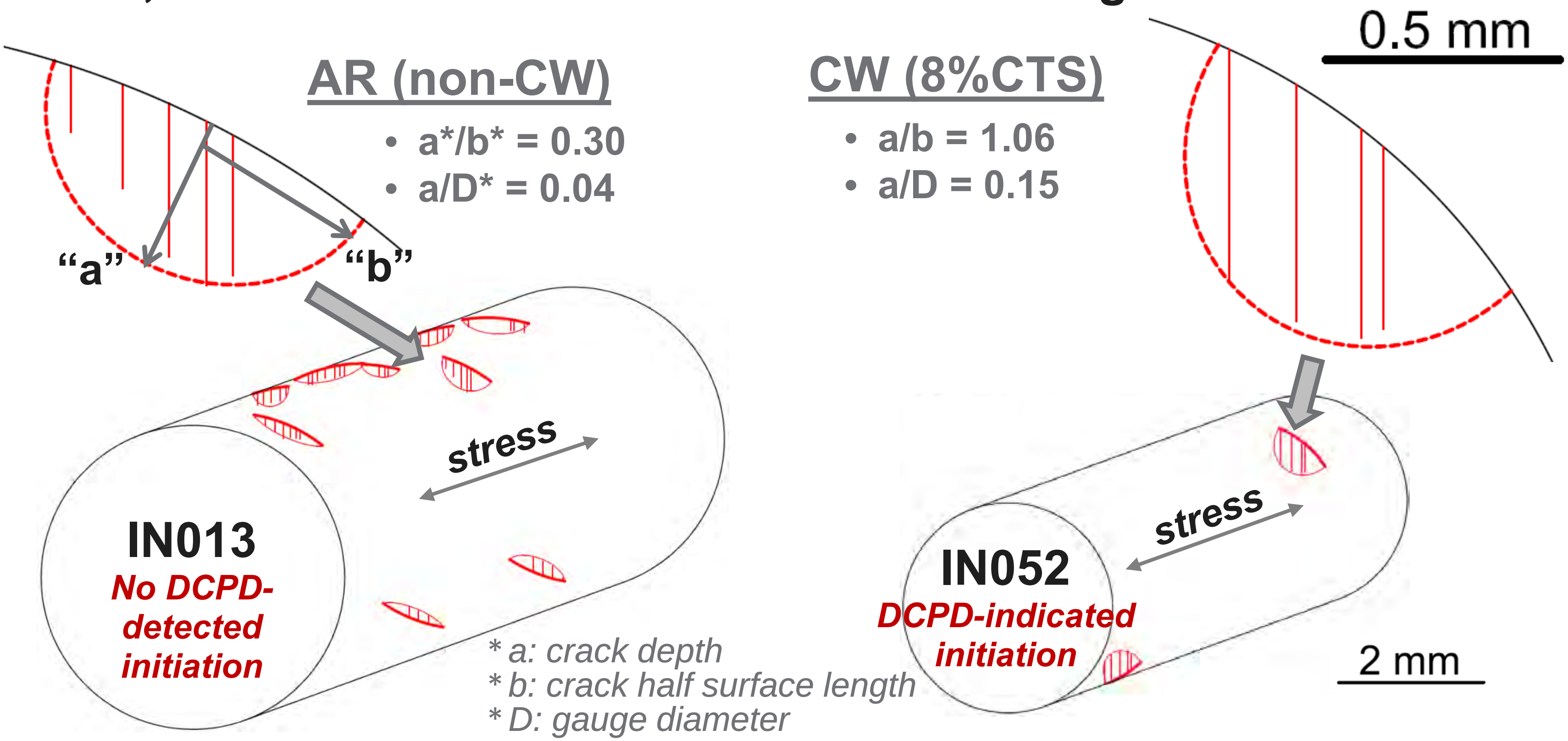
Serial polish (10-50 µm each time)

IN052 (MA plate, 8%CW, 1334h)



Alloy 600 SCC Initiation: Crack Depth Profile

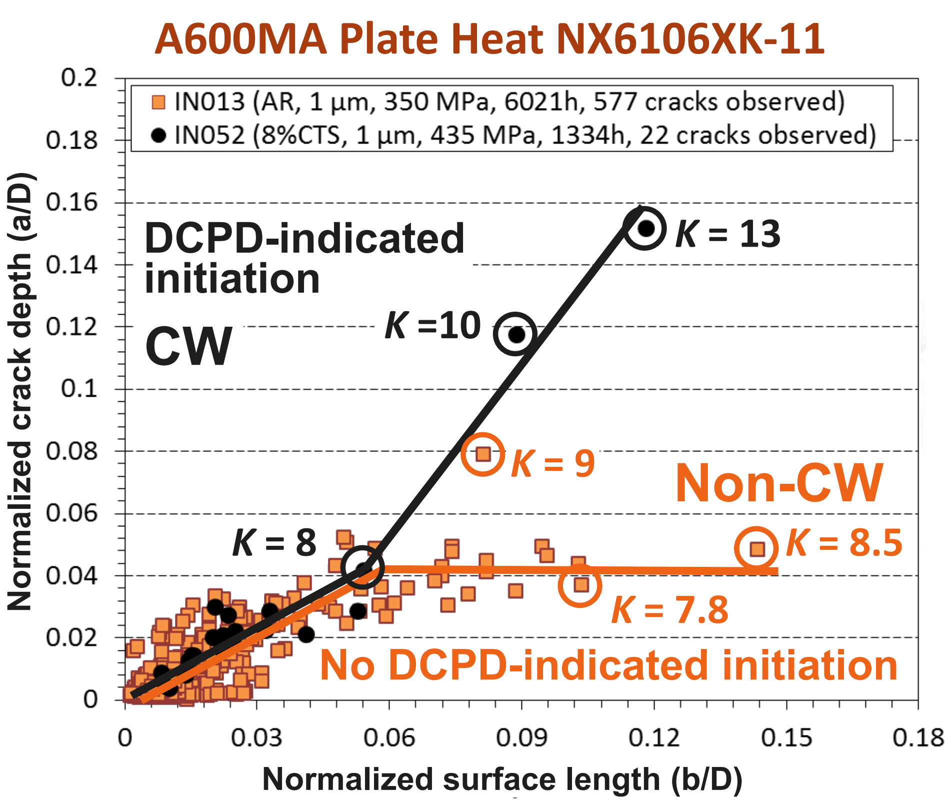
- Depth profiles have been collected for large cracks in selected specimens by serial polishing and sequential SEM examinations.
- Most cracks can be represented by a semi-elliptical shape.
- For large cracks, cold working promotes a fewer number of deeper cracks, but with the same or shorter surface length.

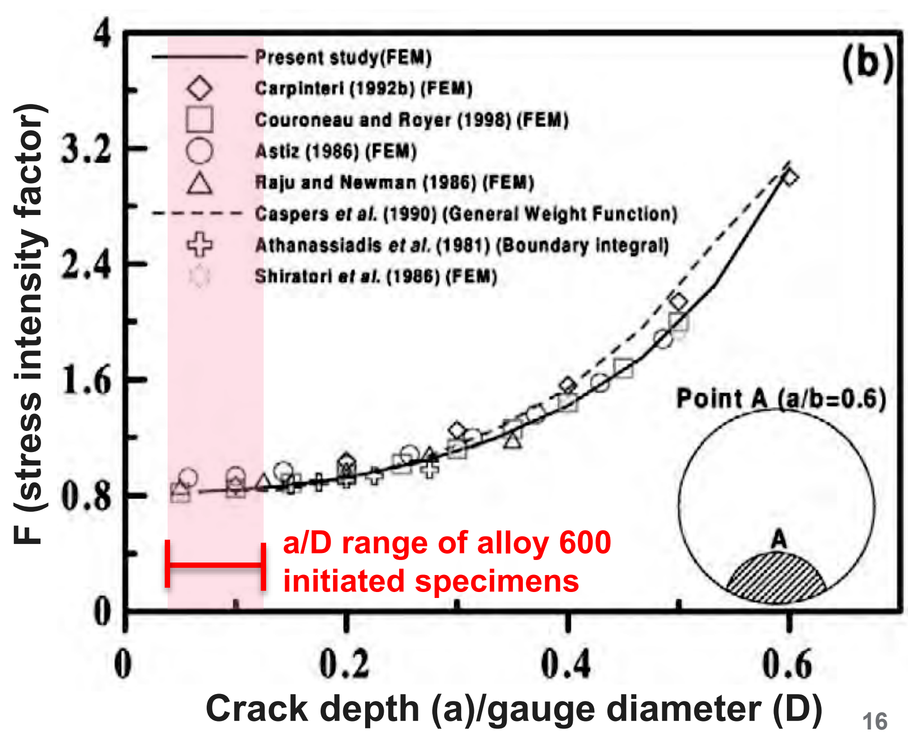


Crack depth profile of large cracks observed in gauge section of initiation specimens.

Alloy 600 SCC Initiation: Stress Intensity Estimation

- AR and CW follow a similar trend on crack depth versus surface length for short cracks until reaching a similar critical size relative to gauge diameter.
- Crack shape statistics and published FEM studies were used to estimate K for large cracks nucleated from initiation specimens.
- Results suggest a higher K is needed in non-CW materials compared to CW materials to sustain stable crack growth.

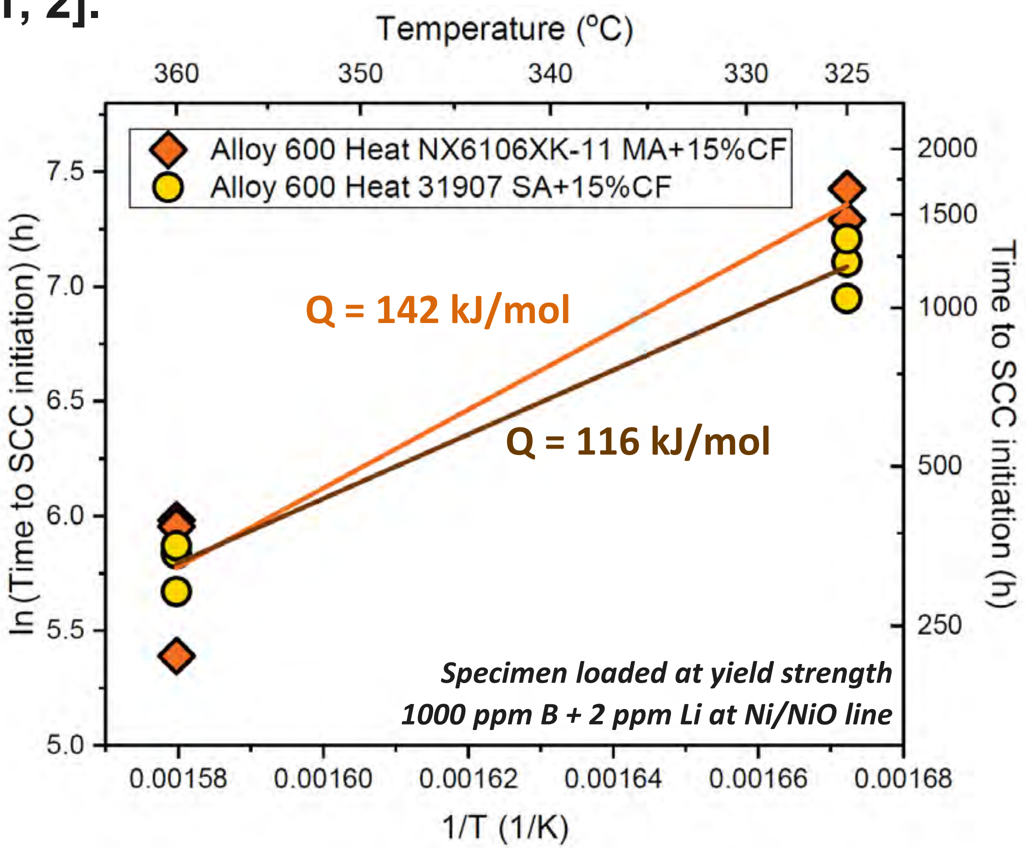




Shin et al (2004), Int. J. Fract. 129 (3) 239-264.

Alloy 600 SCC Initiation: Temperature Dependence

- **Objective**: To best estimate SCC initiation behavior of materials used in service.
- **Approach**: SCC initiation testing of selected heats at 360 and 325°C PWR primary water with dissolved H_2 corresponding to the Ni/NiO stability line.
- **Findings**: Thermal activation energy (Q) = 116 kJ/mol for an solution-annealed heat and Q = 142 kJ/mol for a mill-annealed heat in good agreement with published data [1, 2].

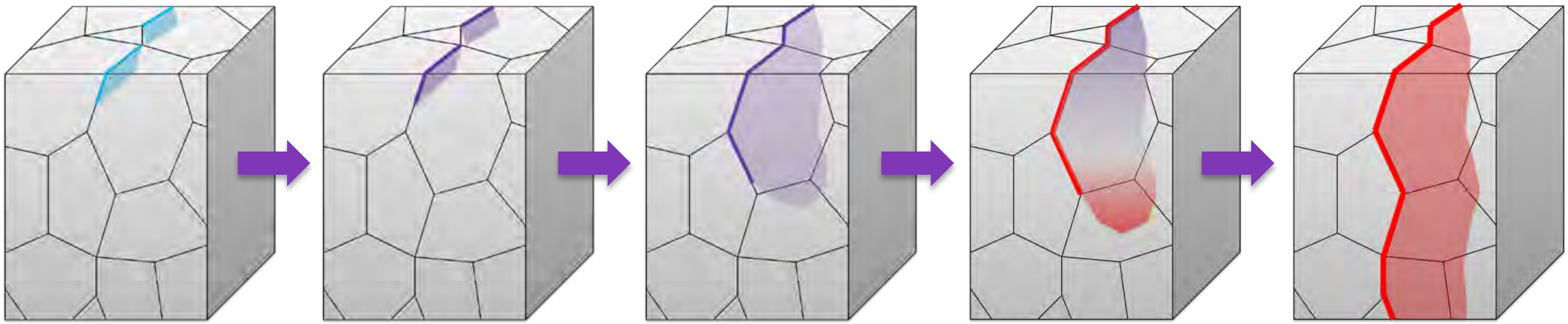


[1] Richey et al, 2007, 13th Env Deg
[2] Etien et al, 2011, 15th Env Deg

Alloy 600: Proposed SCC Initiation Mechanism

Non-CW & CW Alloy 600 loaded at yield strength (YS)

IGA development and growth → *Crack nucleation from IGA* → *Short crack growth and coalescence* → *Transition to stable crack at a critical depth* → *Stable crack grows at engineering relevant rate*



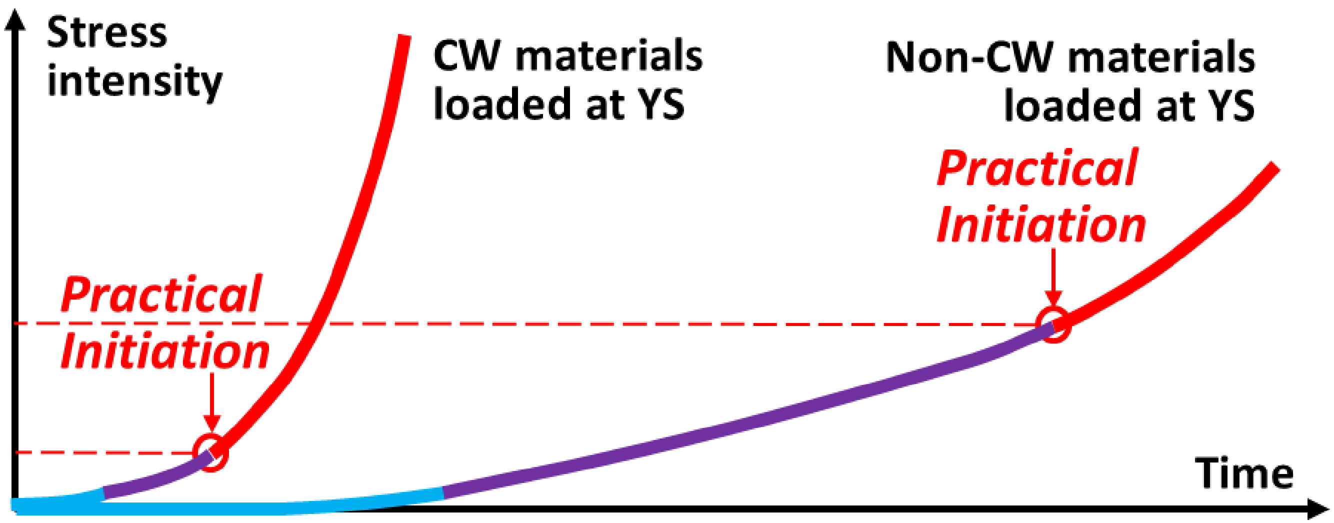
Reaching a critical IGA depth where the local K is sufficient to promote growth in depth.

Reaching a critical crack size where the local K is sufficient for sustained growth at engineering relevant rate.

DCPD-detected initiation considered to be "practical initiation"

Alloy 600 SCC Initiation: Summary

SCC initiation for alloy 600 in PWR primary water evolves in three stages:



Stage A
Intergranular attack

Stage B
Short crack growth and coalescence

Stage C
Transition to stable crack growth

Reaching a critical IGA depth where the local K is sufficient to promote growth in depth

Reaching a critical crack size where the local K is sufficient for sustained growth at engineering relevant rate.

Questions?

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Thanks for Your Attention!

Microstructural Comparison of Intergranular Attack in Alloy 600 in the SA versus TT Conditions Exposed to Simulated Primary Water

Matthew Olszta, Karen Kruska, Dan Schreiber, and Steve Bruemmer

Pacific Northwest National Laboratory

Research Supported by
EPRI and Rolls Royce

ICG-EAC Meeting

April 15-20th, 2018 Knoxville, TN USA

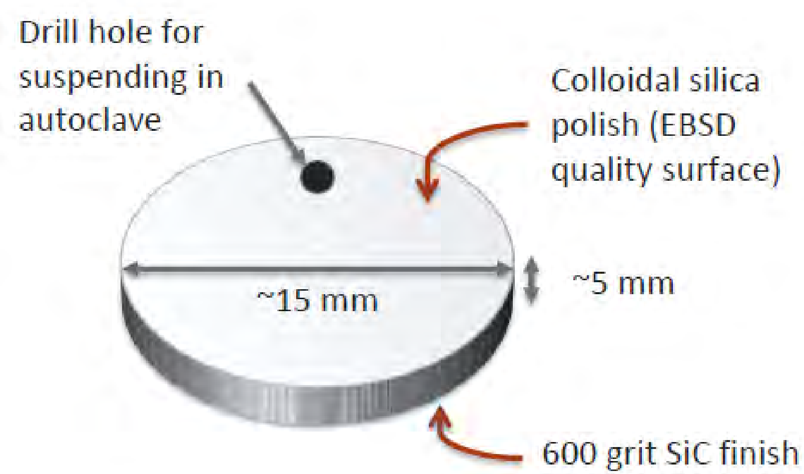
Pacific Northwest NATIONAL LABORATORY

Role of IG Carbides on Alloy 600 Initiation and SCC Cracking

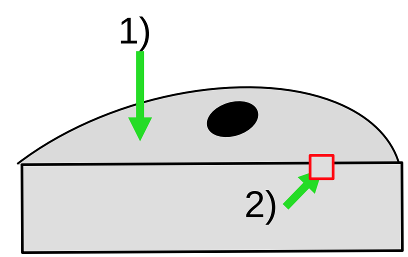
- *International Round Robin on Crack Initiation*
  - *Developed to understand variability in alloy 600 crack initiation*
  - *Three alloy 600 heats being investigated in SA or MA conditions*
    - *Clear differences observed for SCC initiation times among the round robin samples*
- *New interest in understanding material condition effects on SCC initiation particularly thermal treatment. Grain boundary Cr carbide precipitates believed to be beneficial to SCC response of alloy 600 via one of two proposed mechanisms*
  - *Carbides inhibit intergranular attack (IGA)*
  - *Carbides inhibit crack path mechanically*
- *Collaboration among EPRI, Rolls Royce and DOE LWRS projects at PNNL to understand IGA and SCC response to high temperature simulated PWR primary water.*
- *High resolution SEM, TEM and APT characterizations were performed to investigate grain boundary corrosion behavior in unstressed alloy 600 materials.*

Exposure Coupons

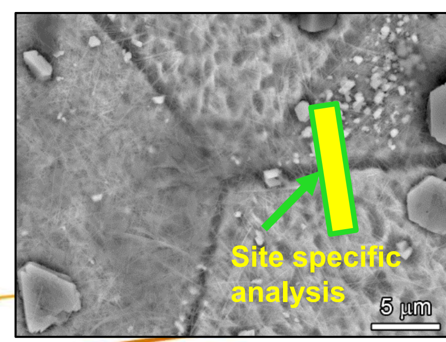
Material	Heat	Ni	Cr	Fe	Mn	C	Si	S	B (appm)
Rolls Royce A600	11415	75.6	15.6	8.36	0.19	0.037	0.20	0.001	1.7



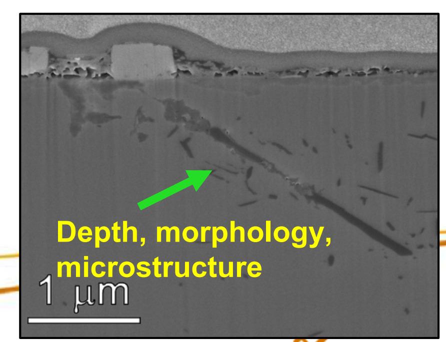
Unstressed Exposure Coupons



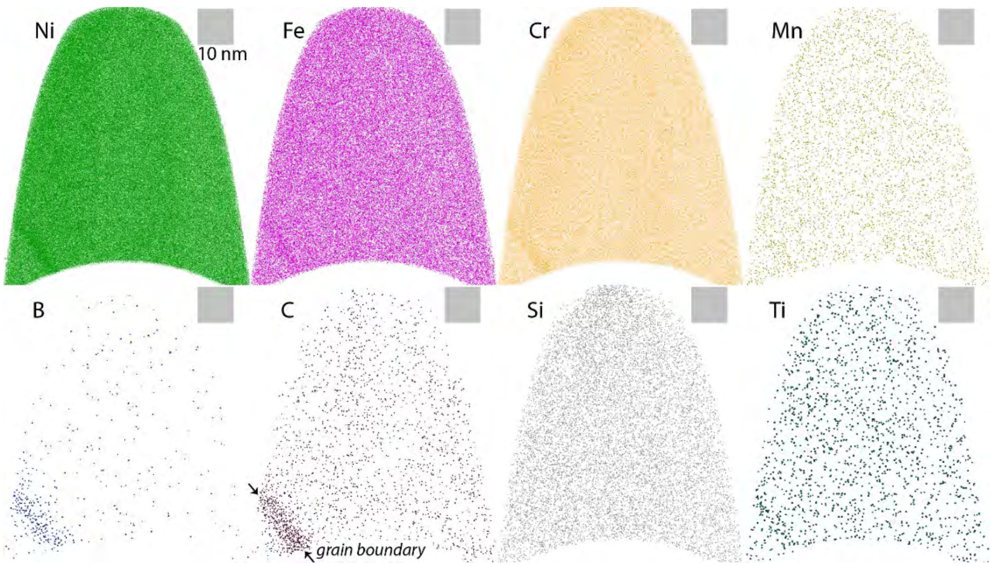
1) Corrosion surface

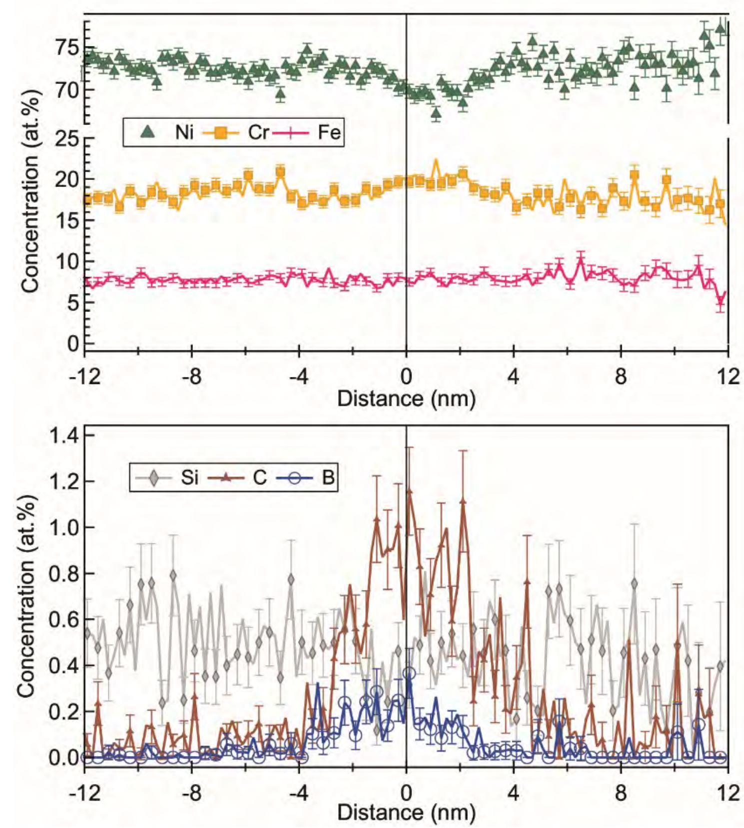


2) Cross-section



Rolls Royce A600 11415 SA





- *APT on solution annealed material*
  - *Slight Cr enrichment/Ni depletion*
  - *B and C enrichment*

Heat Treatments/Test Conditions

PNNL Exposure Matrix (3 coupons/sample)

Sample	Heat	Condition
EPRI/GE (Foroni)	31907	SA + 15% CF
		SA+TT + 15% CF
		SA (no CW)
		SA+TT(no CW)
PNNL Plate	NX6016XK-11	MA+15%CF
		SA
		SA+TT
		SA+15%CF
		SA+TT+15%CF
Rolls Royce	11415	***SA***
		SA+TT
		SA+15%CF

SA = 1100˚C for 0.5 h, WQ
TT= SA +704˚C for 12 h, AC

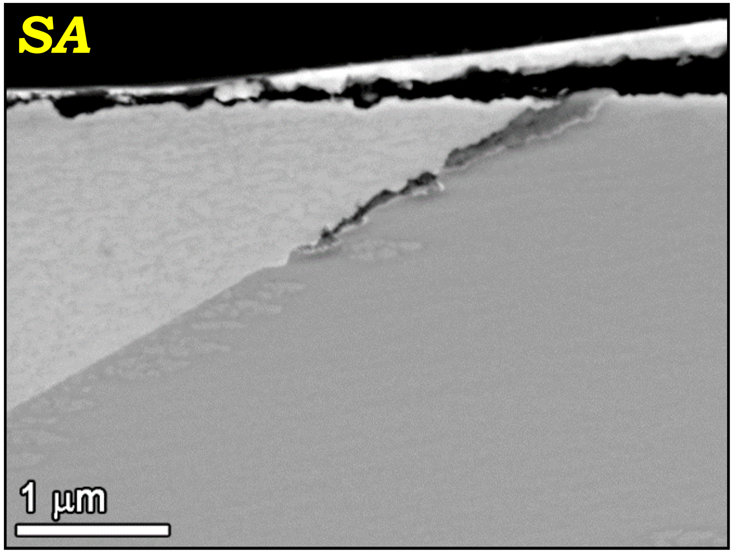
360°C, simulated PWR primary water @ Ni/NiO stability

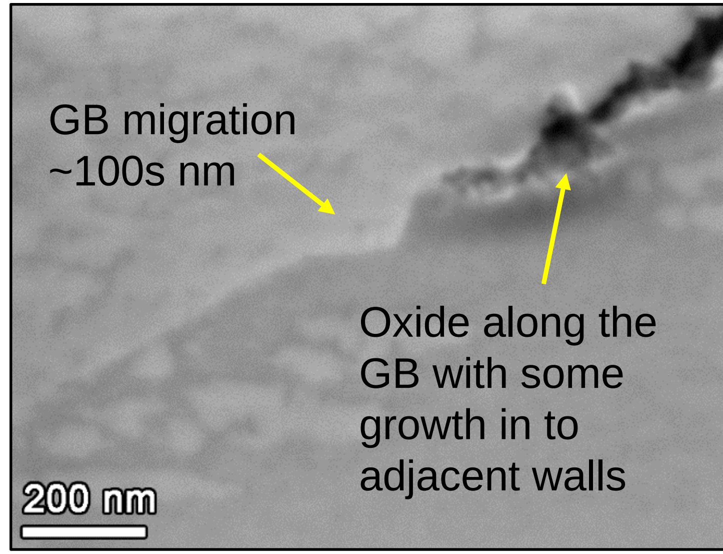
Examined the 1000 and 4400 h samples

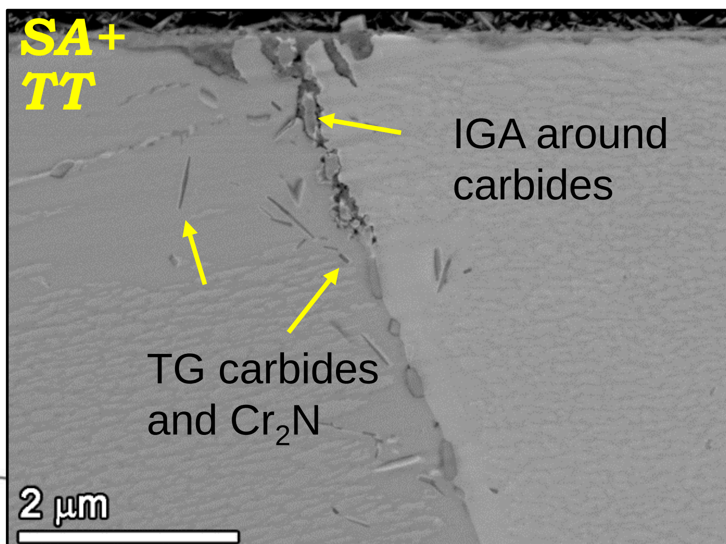
Schreiber, et al., Role of Grain Boundary Cr_5B_3 Precipitates on IGA in A600 Env. Deg. 2018

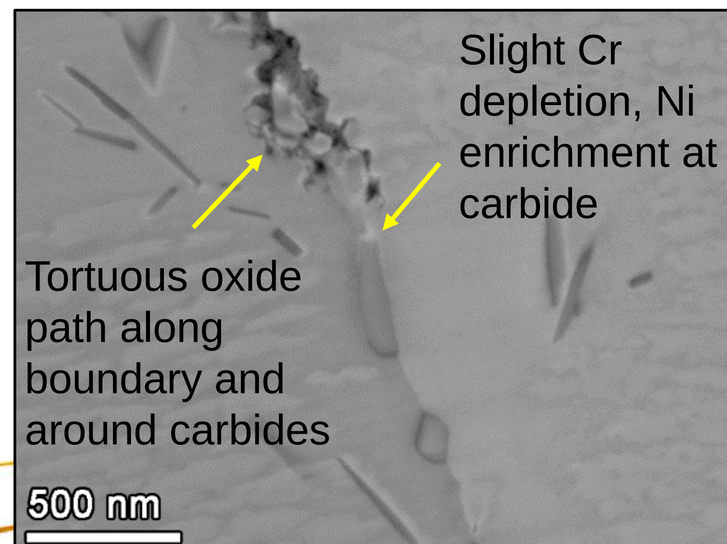
Cross-Section SEM 1000 h Exposure

Rolls Royce 11415



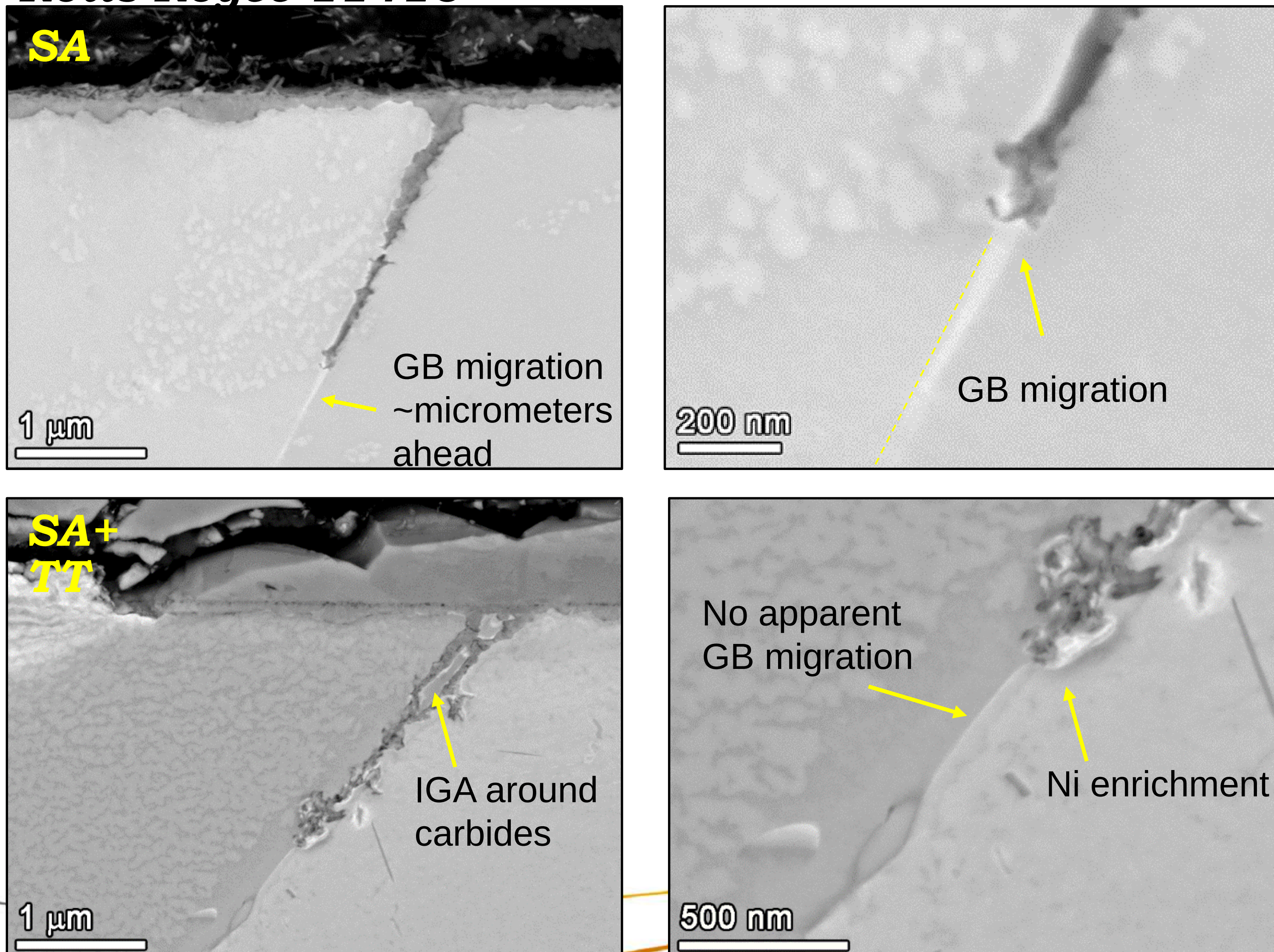






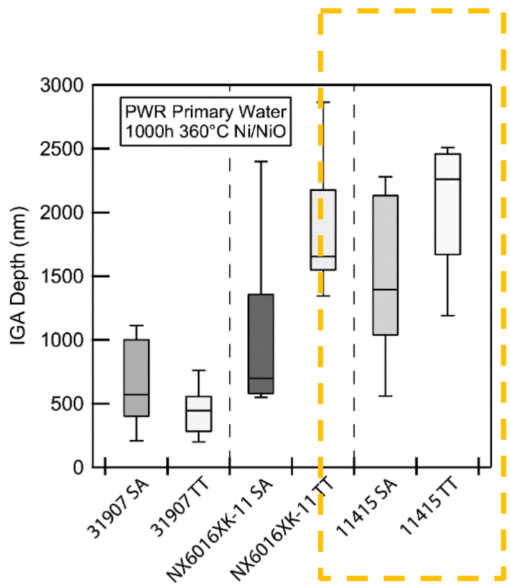
Cross-Section SEM 4400 h Exposure

Rolls Royce 11415

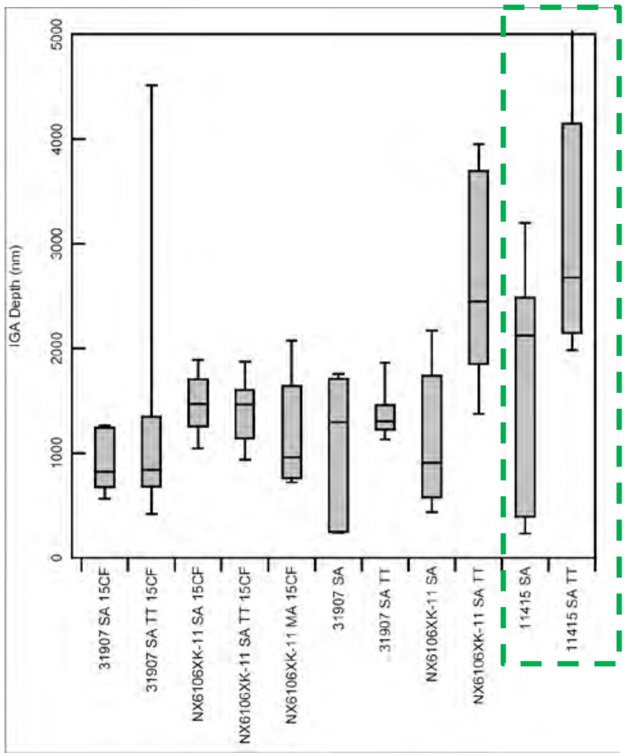


IGA: Depth Analysis

1000 h



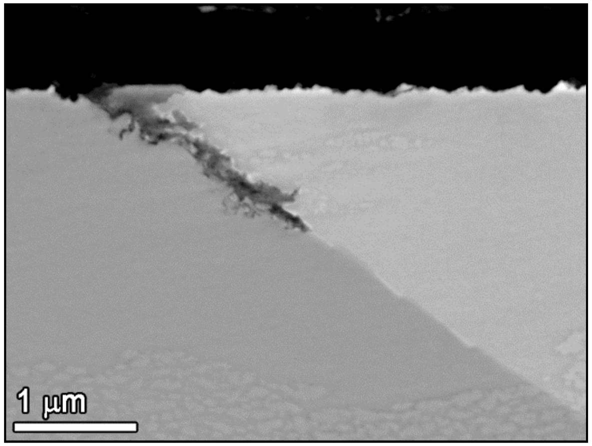
4400 h

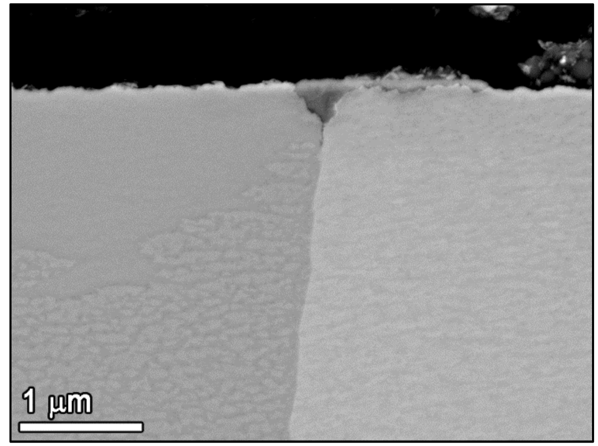


- *Measurement of ~8 boundaries shows no statistical difference between IGA depth of SA and SA+TT at neither 1000 nor 4400 h.*
- *Overall TT samples had the longest recorded IGA, but variability of random GB provided no statistical differences*

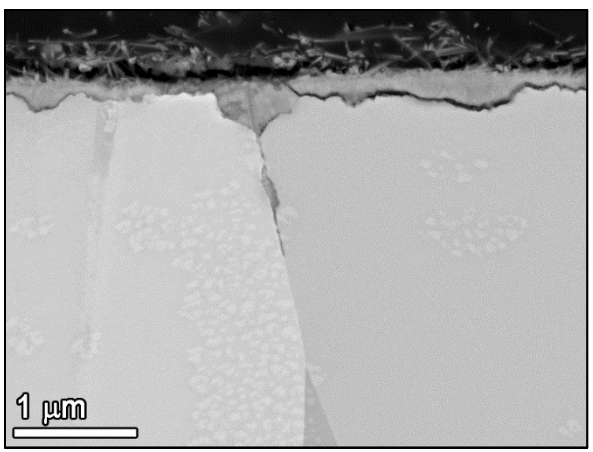
IGA Variability

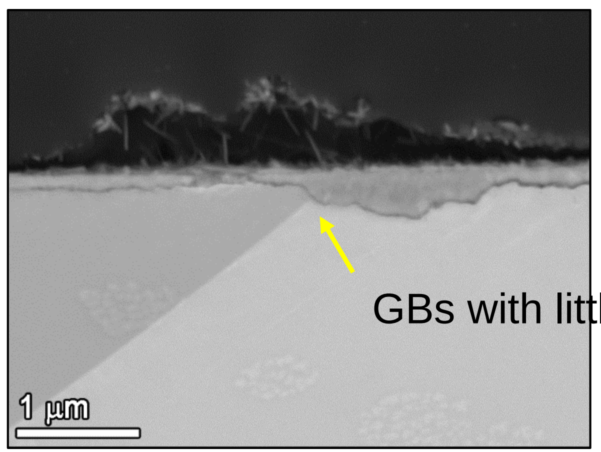
SA 1000 h

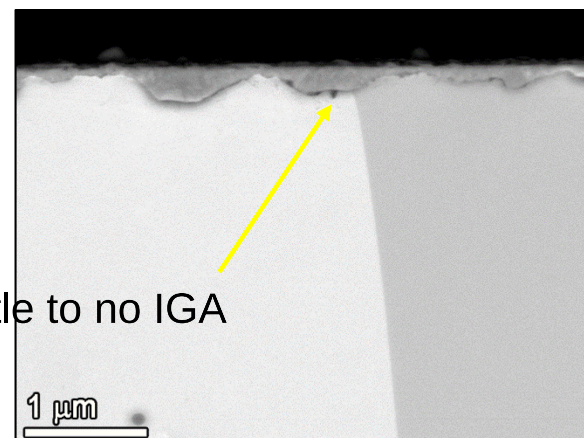




SA 4400 h



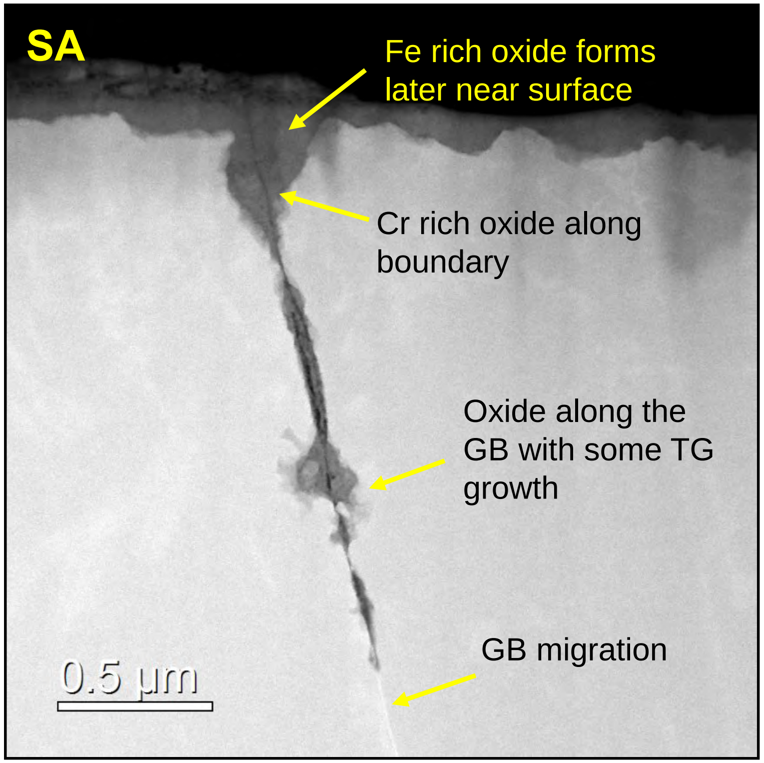




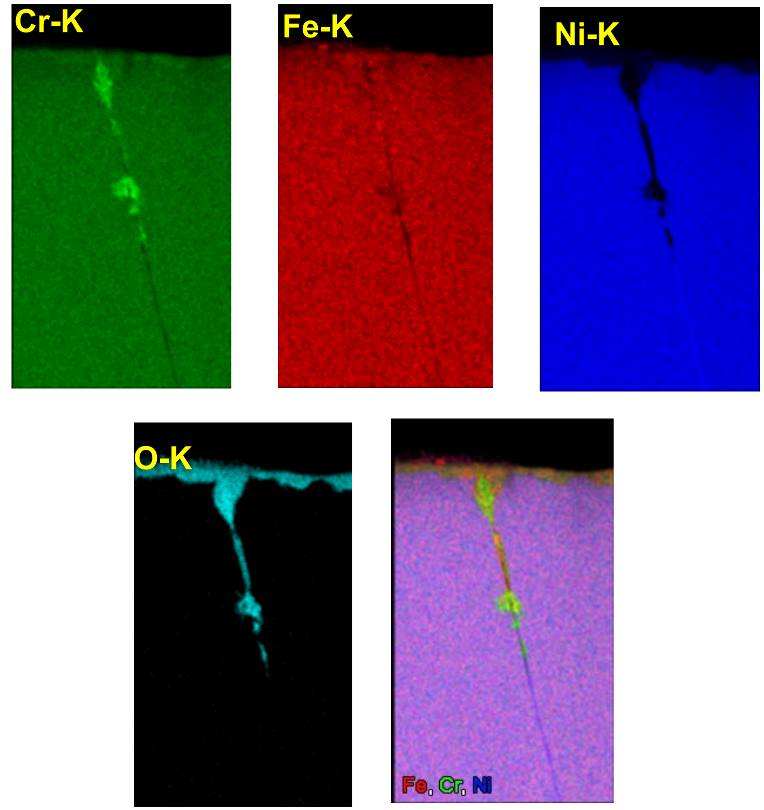
GBs with little to no IGA

TEM of IGA in SA Condition

Annular DF Imaging



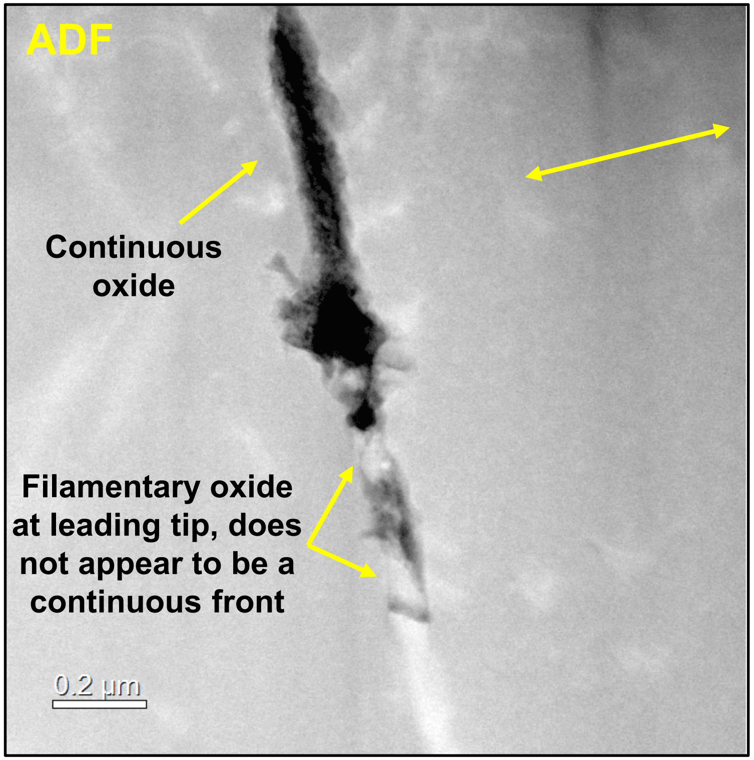
EDS Mapping

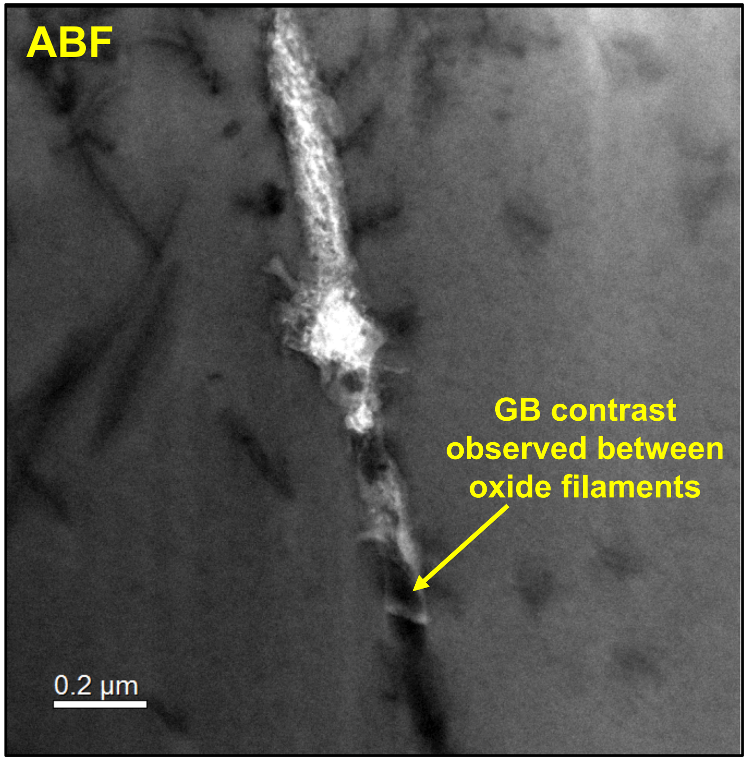


- *Oxide along the boundaries is Cr rich, with Fe rich oxides forming transgranularly away from the original boundary plane.*

SA Leading IGA: Tilt Series

Tilt Series taken at 5° steps





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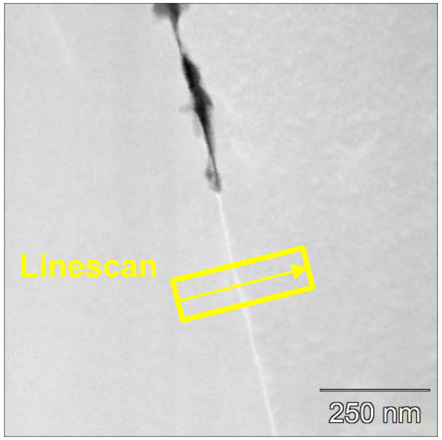
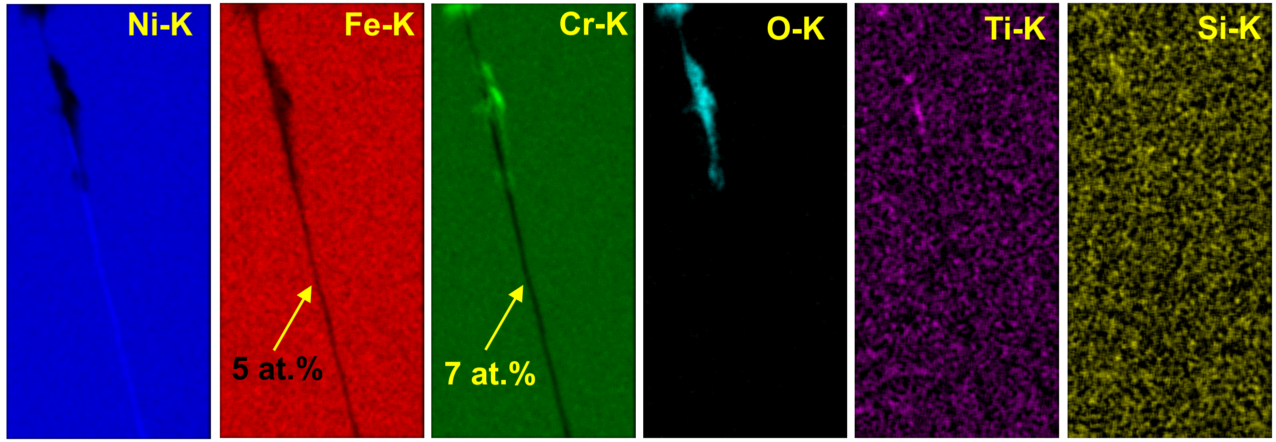
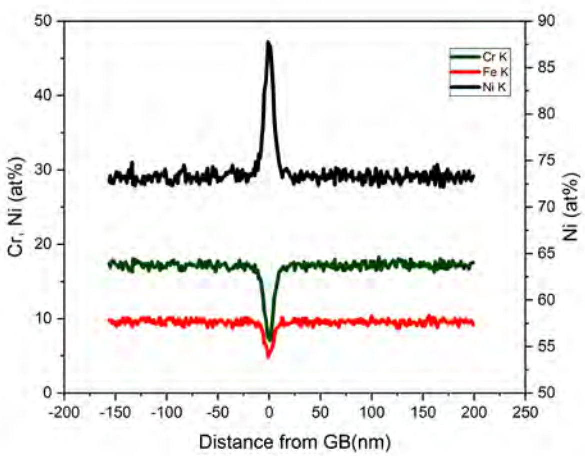
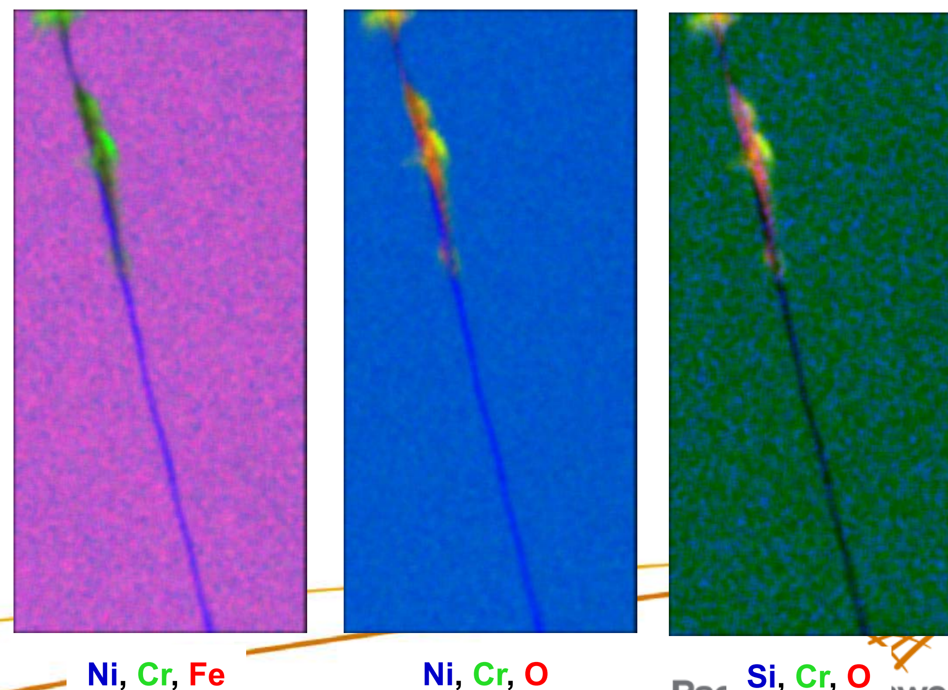
EDS Mapping of SA Leading IGA





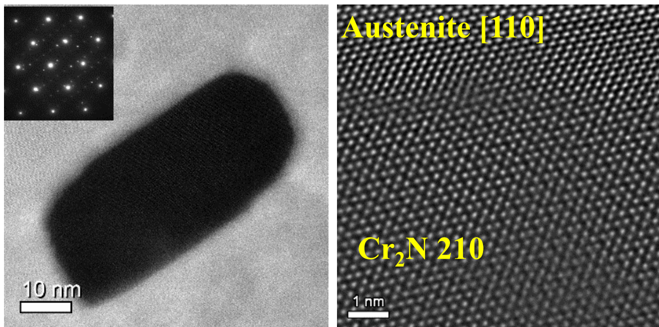


12 nm wide depletion profile ahead of leading IGA

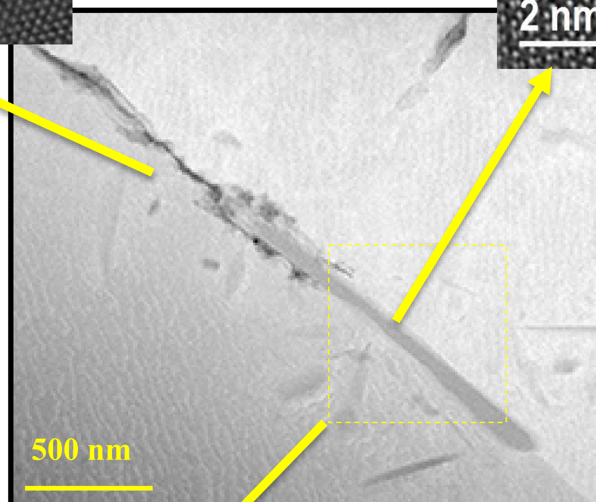


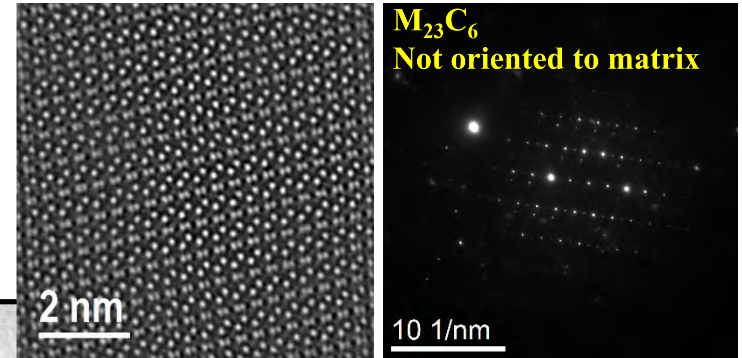
Microstructure of TT Material

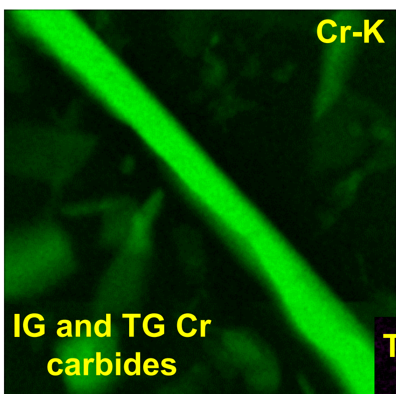
SA +704˚C for 12 h, AC

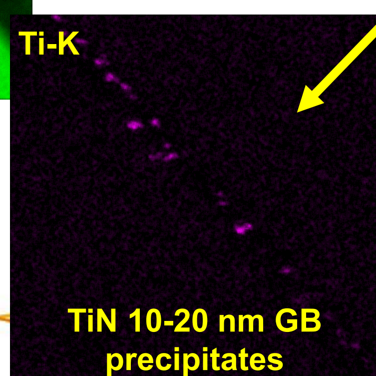


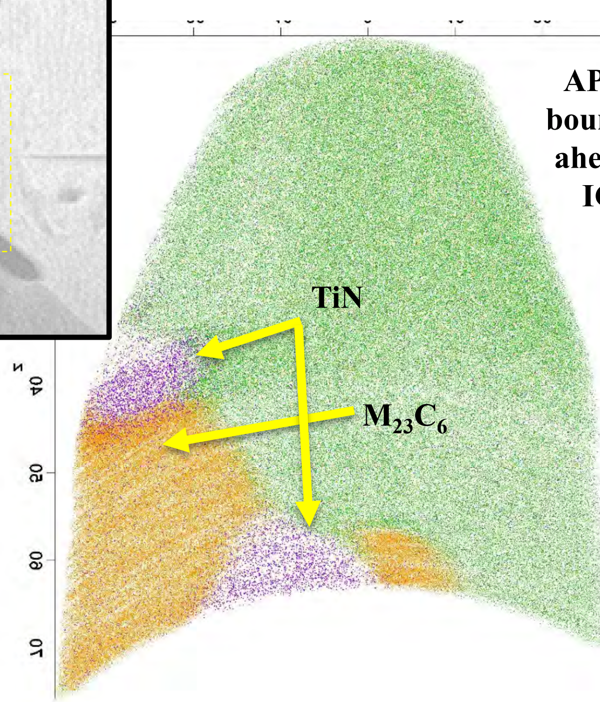
TG precipitate





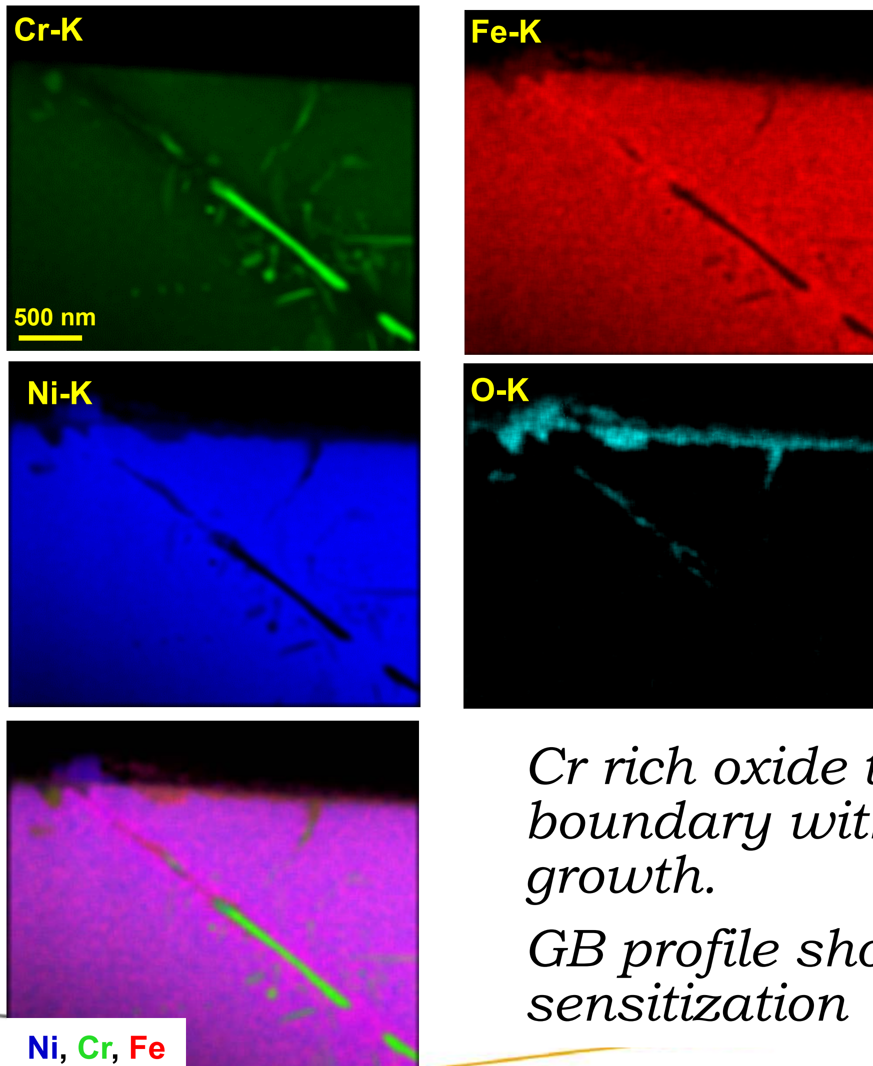


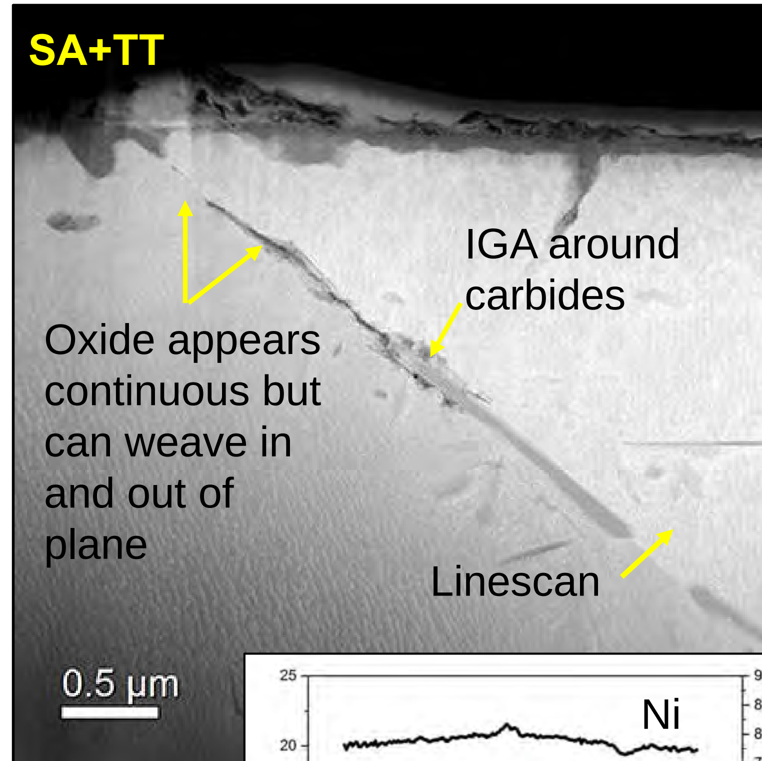


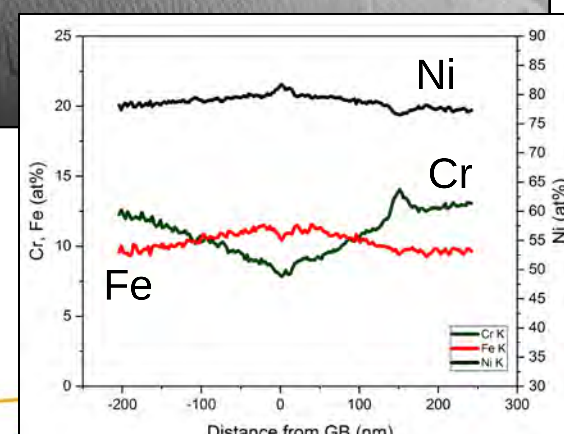


APT of boundary ahead of IGA

TEM of IGA in SA+TT Condition



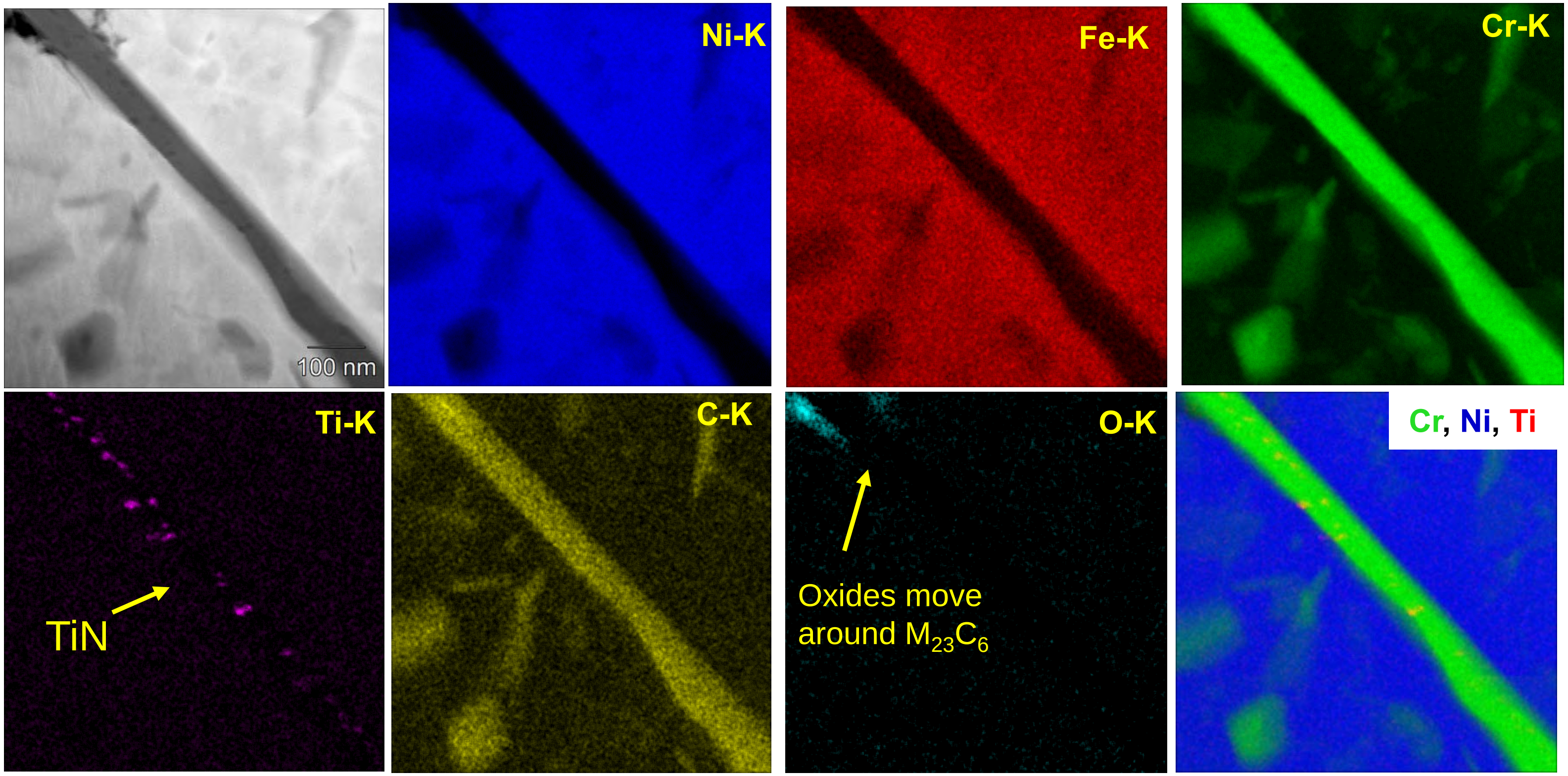




Cr rich oxide tight to the boundary with some TG growth.

GB profile shows slight sensitization

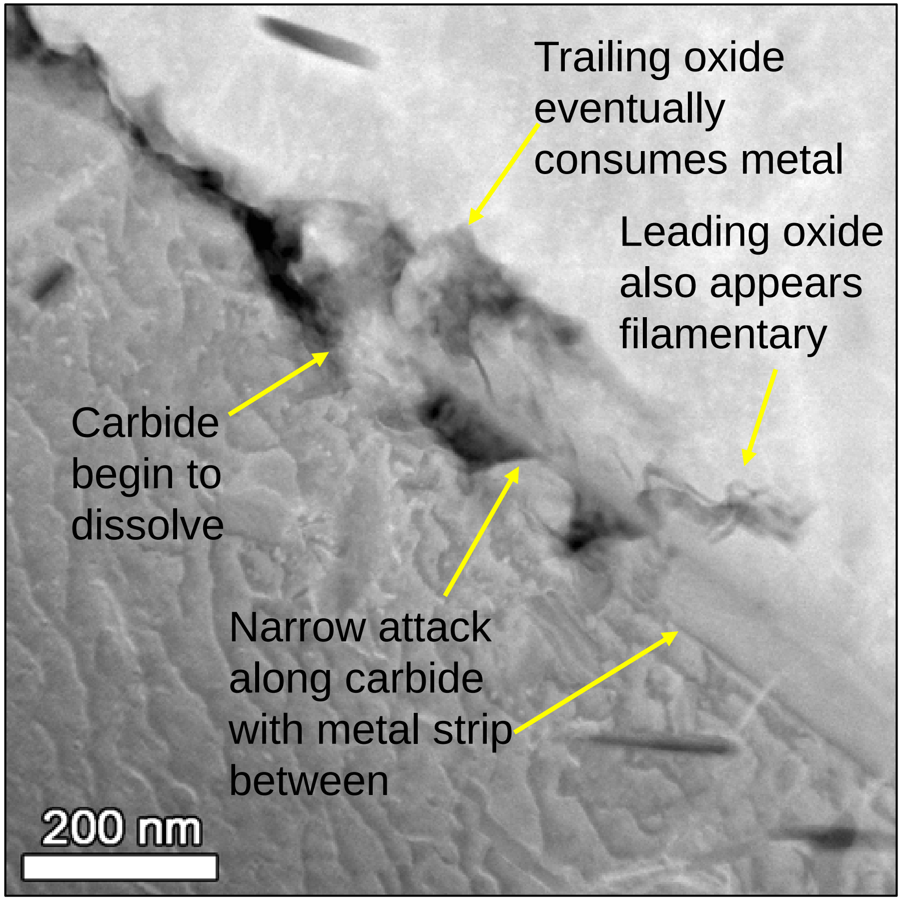
RR SA+TT Leading IGA



- *TiN observed inside the $M_{23}C_6$ carbide*
- *Leading IGA observed moving around the carbide*
- *Sample slightly too thick at leading IGA for good chemistry*

Leading IGA of SA+TT in Motion

- *Sample foil thickness precluded capturing good EDS, but thickness highly beneficial for seeing morphology at the leading tip.*
- *Tilt series provides better look at fine detail.*
  - *Oxide traveling adjacent to carbide with thin metal layer in between carbide/oxide.*
  - *Carbide eventually beings to dissolve*
  - *Finger like TG penetrations as well*



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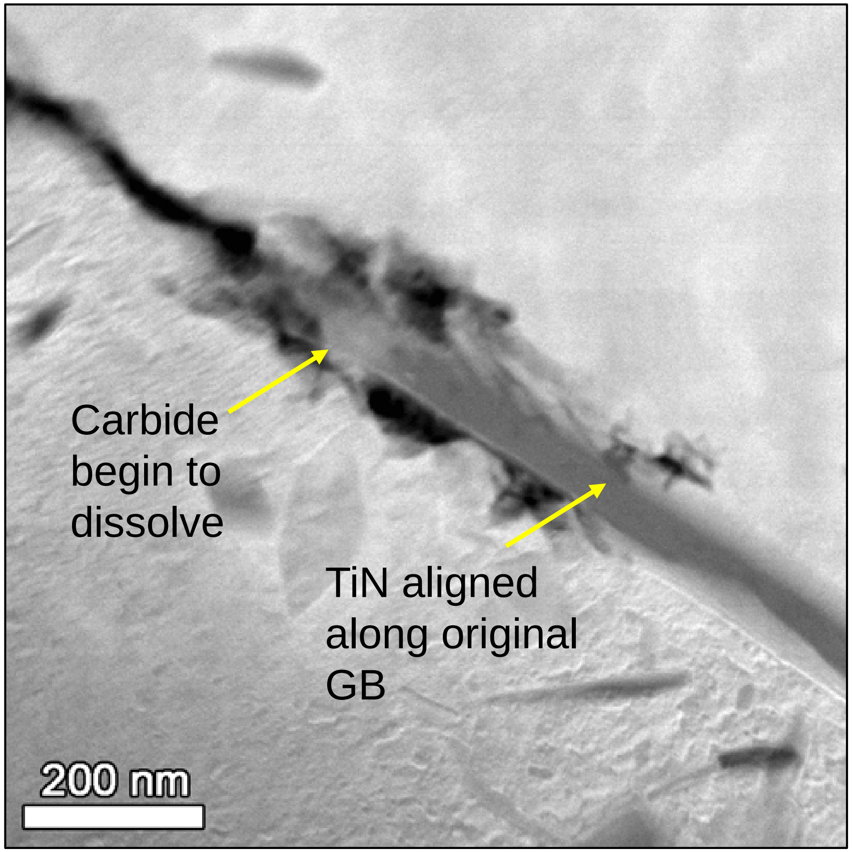
Second View of Leading IGA

- Tilt series performed along the IGA to get different opinion
  - Carbide dissolution more apparent
  - TiN along the original boundary before carbide formed is also evident



Summary/Conclusions

- *Rolls Royce Alloy 600 #11415 examined as part of Crack Initiation Round Robin*
  - *SA condition has shown longer initiation times*
- *PNNL Exposed Unstressed Coupons of #11415 in the SA and SA+TT condition for 1000 and 4400h.*
- *SA condition*
  - *migrated boundaries with Cr depletion/Ni enrichment ahead of IGA*
  - *Leading IGA appeared to be filamentary in regions*
- *SA+TT condition*
  - *showed small (10-20 nm) TiN particles along the boundary and inside IG carbides.*
  - *IGA appeared to attack around carbides leaving thin metal layer, with eventual carbide dissolution*

Acknowledgements

- Funding
  - EPRI (Peter Chou)
  - Rolls Royce (Tony Horner)
- Collaboration with DOE Light Water Reactor Sustainability project at PNNL where SCC initiation testing is being performed on these same materials.
- FIB and APT work were performed using EMSL, a U.S. DOE Office of Science, Biological and Environmental Research (DOE-BER) national user facility located at PNNL

- Questions?

SCC Initiation of Alloy 182 in PWR Primary Water

Mychailo Toloczko (PNNL), Ziqing Zhai (PNNL), Steve Bruemmer (PNNL), Eric Focht (NRC), Paul Crooker (EPRI)

ICG-EAC Meeting
Knoxville, TN
April 15-20, 2018

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The views expressed in this presentation are those of the authors, not necessarily those of the U.S. NRC

Outline

- **Objectives**
- **Material Selection and Fabrication**
- **Test Systems and Testing Approach ****
- 15% CF Alloy 182 SCC Initiation Behavior
- Alloy 182 Initiation Behavior Versus Cold Work
- Summary

** SCC initiation testing is being conducted in collaboration with a DOE Light Water Reactor Sustainability project at PNNL entitled Stress Corrosion Crack Initiation of Nickel-Base Alloys in LWR Environments.

Test Program Overview

SCC Initiation of Alloys 600/182 and Alloys 690/152/52

- This research arises from a cooperative effort between the USNRC and EPRI to evaluate SCC initiation behavior of Alloys 600/182 and Alloys 690/152/52.

- **Majority of the program is devoted to determining material and environmental dependencies of Alloy 182 PWSCC initiation to support calibration of xLPR models.**
  - xLPR aims to develop a fully probabilistic computational tool to evaluate the rupture probability of reactor coolant piping, starting first with Alloy 82/182.
  - Tool incorporates estimates of PWSCC initiation time of Alloy 82/182.
  - **Dependencies of interest for xLPR**

1. **Alloy 182 weld-to-weld variability → Four 15% CF welds at yield stress (YS)**
2. Stress (or stress ratio = stress/YS) → Two 15% CF welds each at 0.90YS and 0.80YS
3. **Yield strength → Two welds each at 15% CF, 7.5% CF, and as-welded; all at YS**
4. Temperature → Two 15% CF welds each at 345°C and 330°C
5. Hydrogen → Two 15% CF welds at Ni/NiO stability line and at Ni-metal stable
6. Surface condition → Two 15% CF welds each at polished and ground finish

Material Selection

Overview

- Four welds of Alloy 182 selected from different sources.
- PNNL selected 15% CW through forging as the baseline condition for assessing environmental effects. CW provides relevance to service components (i.e., cold-worked surface layer found in service) and serves as a test accelerant.
- YS, hardness, and other characterizations have been performed on the 15% CF welds.
- SCCGR rate measured in the non-cold worked (CW) condition.

Values are for 15% Cold Forged Material

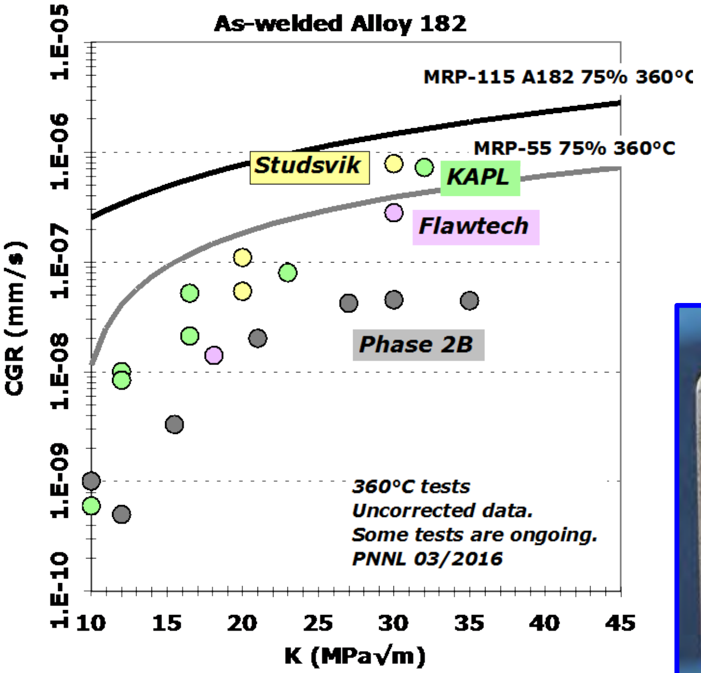
Material	ID	YS@360°C (MPa)	Hardness (kg/mm^2)
Alloy 182 Build-up	Studsvik 8001231	550	240-345
Alloy 182 DMW	Flawtech 844305	515, 525	225-345
Alloy 182 DMW	Phase 2B	460, 500	225-330
Alloy 182 U-Groove	KAPL 823030	580, 590	250-350

Alloy 182 Welds

Material Selection

Alloy 182 CGR Characterization

- Crack growth rate characterization of all four Alloy 182 welds has been completed in non-CW condition
- Response measured to stress intensity of as low as 10 MPa√m
- Studsvik and KAPL welds exhibited highest SCC susceptibility
- Flawtech was midrange
- Phase 2B lowest
- *High SCC CGRs in Studsvik and KAPL suggest possibility of very low SCC initiation times*



Alloy 182 Welds

Technical Approach

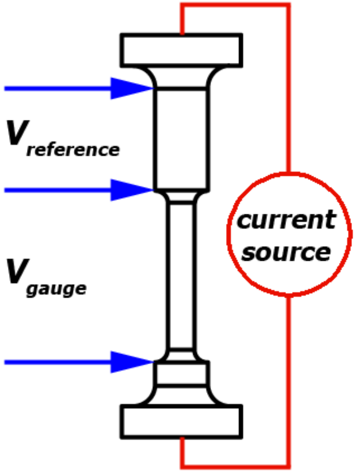
Constant Load Tensile Initiation Tests

- 1.2” (30 mm) tall – Matches height of a 0.5T CT
- 3 to 4.5 mm gauge diameter
- 4 mm long gauge length – Allows complete observation of entire gauge region by SEM in a reasonable period of time. Also improves DCPD sensitivity.
- Includes an on-specimen reference region – Used to subtract off resistivity shift that occurs in Alloy 690/152/52/600/182 when exposed to PWR primary water temperatures.

- DCPD sensitive to cracking, creep, resistivity evolution.
- PNNL assumes primary contribution to DCPD up to point of DCPD-initiation is creep. Plot data as strain.
- Basic approach developed in our DOE LWRS project.

1.2" Tall SCC Initiation Specimen

Schematic of Idealized Voltage Measurement Points



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

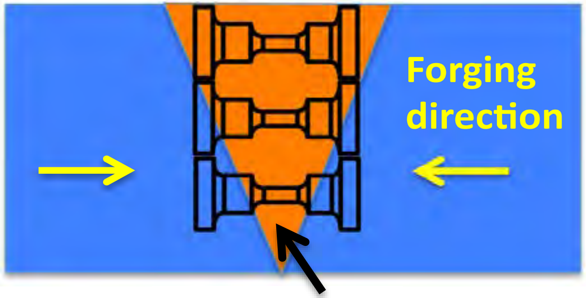
Specimens

Fabrication From Welds

- Gauge, fillet, and reference region of specimen are always made entirely of the weld metal.
- Forging plane is aligned to T-S orientation of weld.
- Specimen cracking plane is aligned to forging plane and to T-S orientation.

- The tensile specimen cracking plane matches the orientation that has been used for SCC CGR testing of these materials.

Orientation of Specimens from Welds (T-S relative to weld)



V-groove weld shown in this example sketch

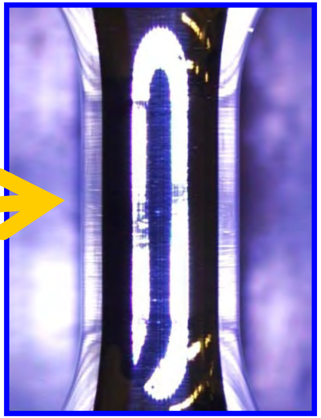
Specimens
Surface Finish

- Gauge section of all specimens are polished to a ***1 µm or colloidal silica finish.***
  - Allows ***characterization of surface features prior to testing***
  - Easier identification of cracks after initiation (compared to a ground or machined surface)
  - PNNL Alloy 600 experience gained through our LWRS project for this specimen type is that the initiation time for a ground surface is slightly longer than for a polished surface.

1.2" Tall SCC Initiation Specimen

Examples of Surface Finishes

1 µm finish



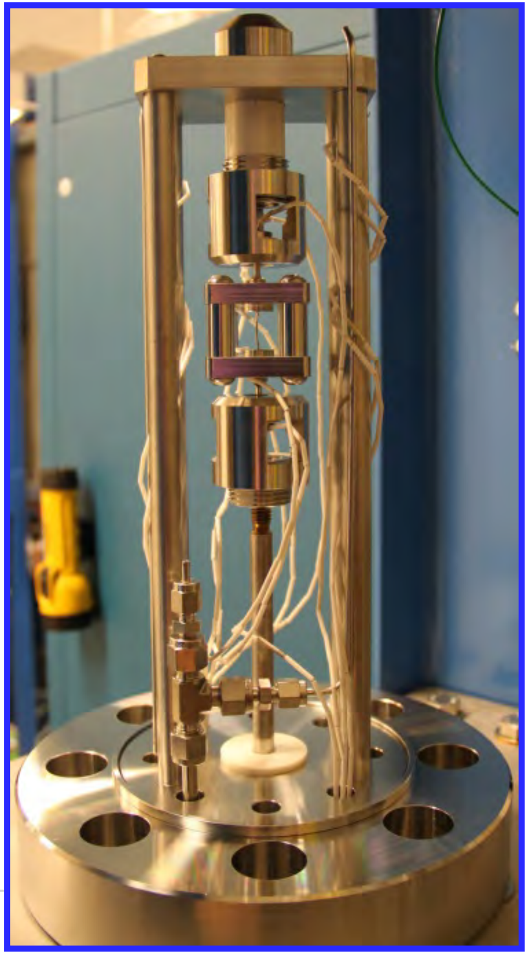
60 grit finish

Technical Approach

Multi-specimen Test Systems Developed in our LWRS Project

- Multiple specimens serially loaded.
  - All see the same load.
- Target baseline test load is 0.2% offset YS.
- Adjust gauge diameter to achieve this stress (or a fraction of the yield stress) at a set load of 1000 lbs.
- Requires pretest measurement of YS.
- Can test materials of different strength in the same string by varying gauge diameter.
- DCPD measurement of cracking behavior continually observed on each specimen sequentially.
- Measurement of servo load, tare load, servo position, autoclave temperature, and water conductivity are performed approximately once per minute.
  - Total applied load stable to within +/- 1.0% of target value
  - Temperature stable to within resolution limit (+/- 0.5 deg)

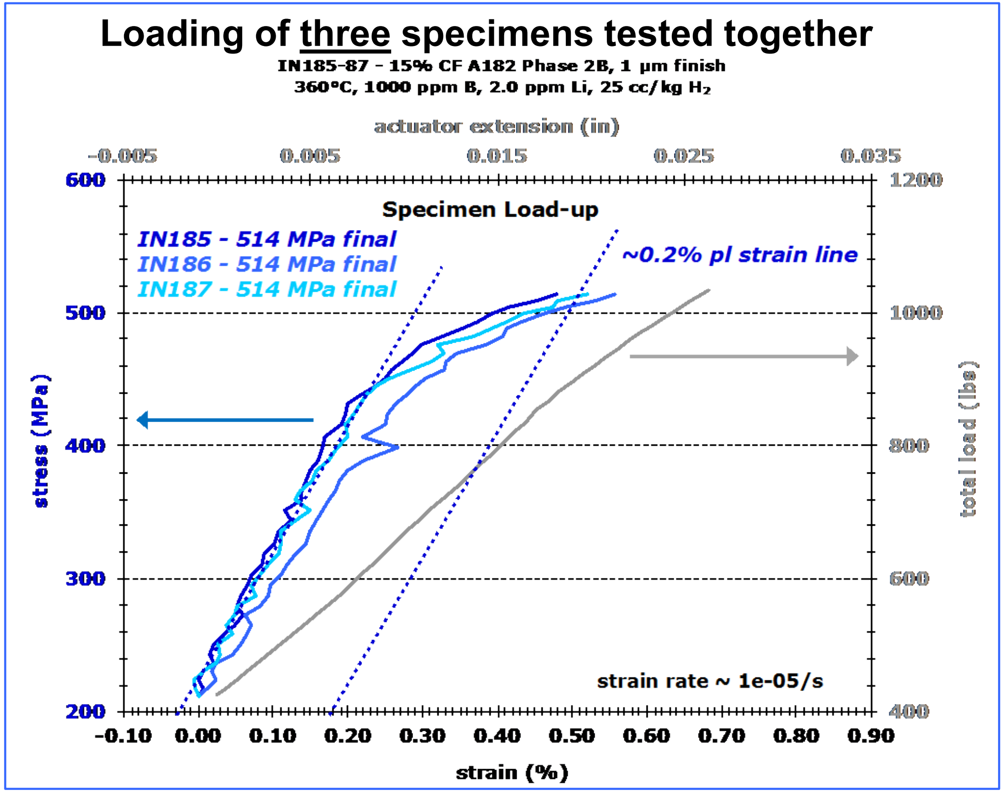
PNNL 2-3 Specimen SCC Initiation System



Testing Methodology

Environment and Loading Technique

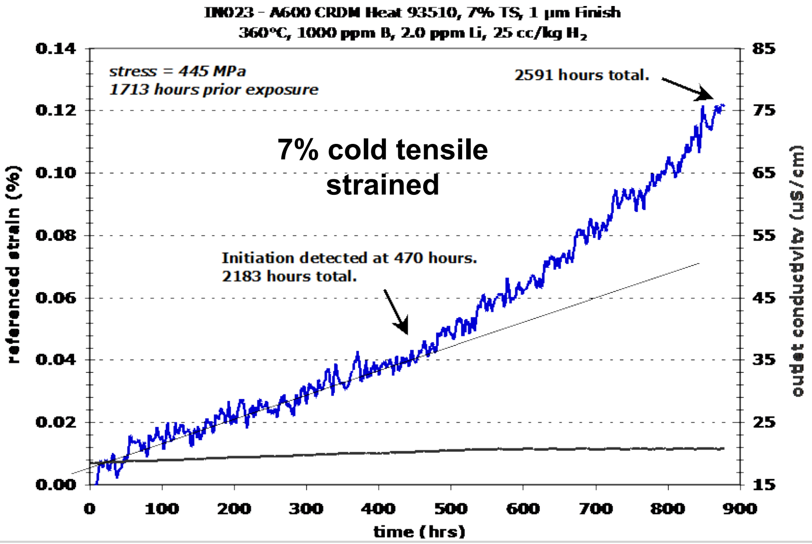
- Standard environment is 360°C, 25 cc/kg (Ni/NiO stability line).
- Within 24 hours after reaching full temperature, specimens are brought up to test load.
- During the 1-1.5 hour loading period, stress versus strain traces recorded in exactly the same manner as a tensile test.
  - Stress determined from applied load and gauge diameter.
  - Strain determined from DCPD. Sensitivity is sufficiently high to be able to track elastic and plastic strains.
- Loading is stopped when small plastic strains are observed in all specimens. Aim is for 0.1-0.3% plastic strain, but will allow up to 2% plastic strain if needed for all specimens to reach yield.

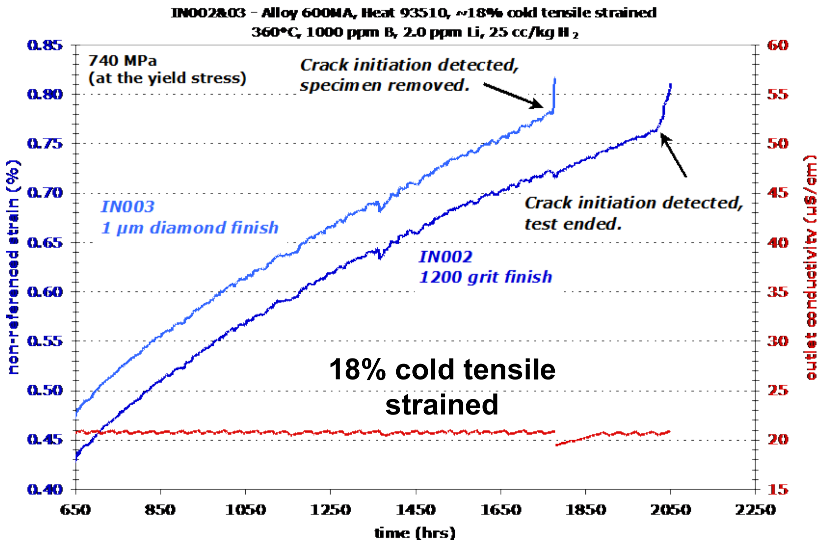


Technical Approach

Constant Load Observation and Detection of SCC Initiation

- Strain vs time plot shows a steady or decreasing slope up to the point of initiation.
- Initiation marked by a transition to an increasing slope.
- PNNL observations of Alloy 600 in our LWRS project indicate that the strength of this transition correlates with the SCC crack growth rate of the material.
  - Non-cold worked materials have a gentle transition while highly cold worked show a rapid transition.





Outline

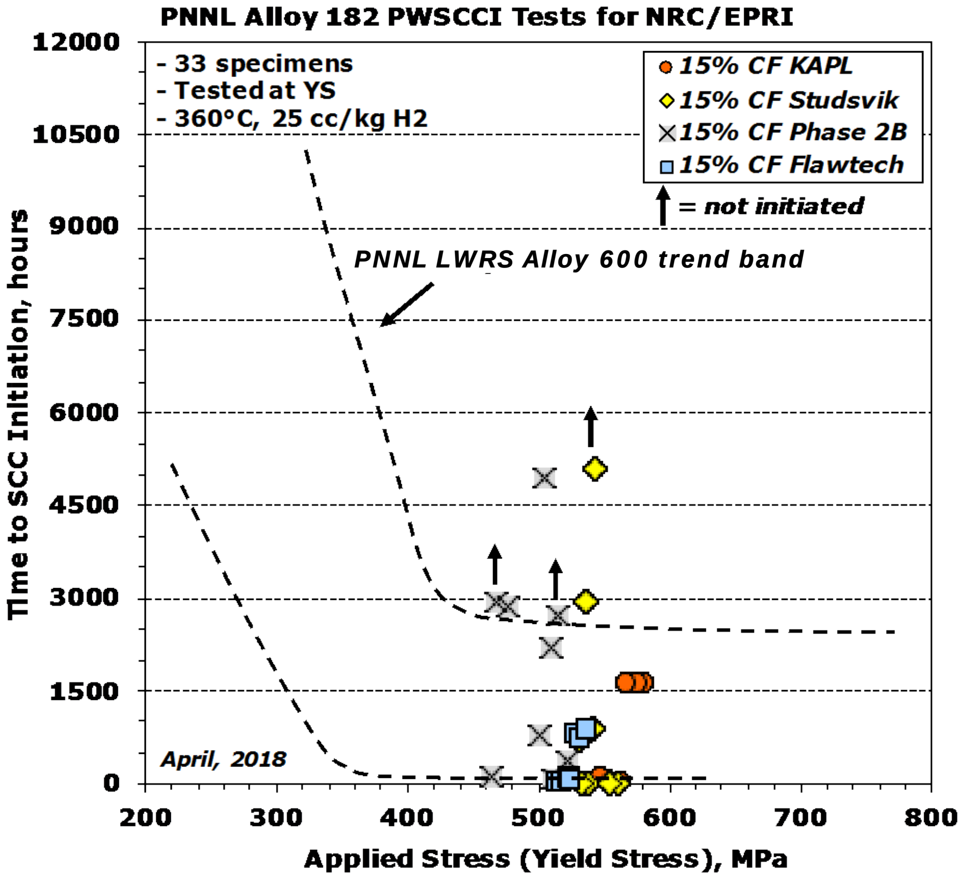
- Objectives
- Material Selection and Fabrication
- Test Systems and Testing Approach
- **15% CF Alloy 182 SCC Initiation Behavior**
- Alloy 182 Initiation Behavior Versus Cold Work
- Summary

15% CF Alloy 182 Initiation Testing

SCC Initiation Time vs Stress Plot

- 9 specimens each of the four different welds – most tests completed.
- Distinct grouping of data at <150 hours.
  - Many of these appear to be SCC initiations occurring upon reaching full load.
- Decreasing distribution all the way out to ~5000 hours.
- Most data are in the bottom half of the alloy 600 trend band.

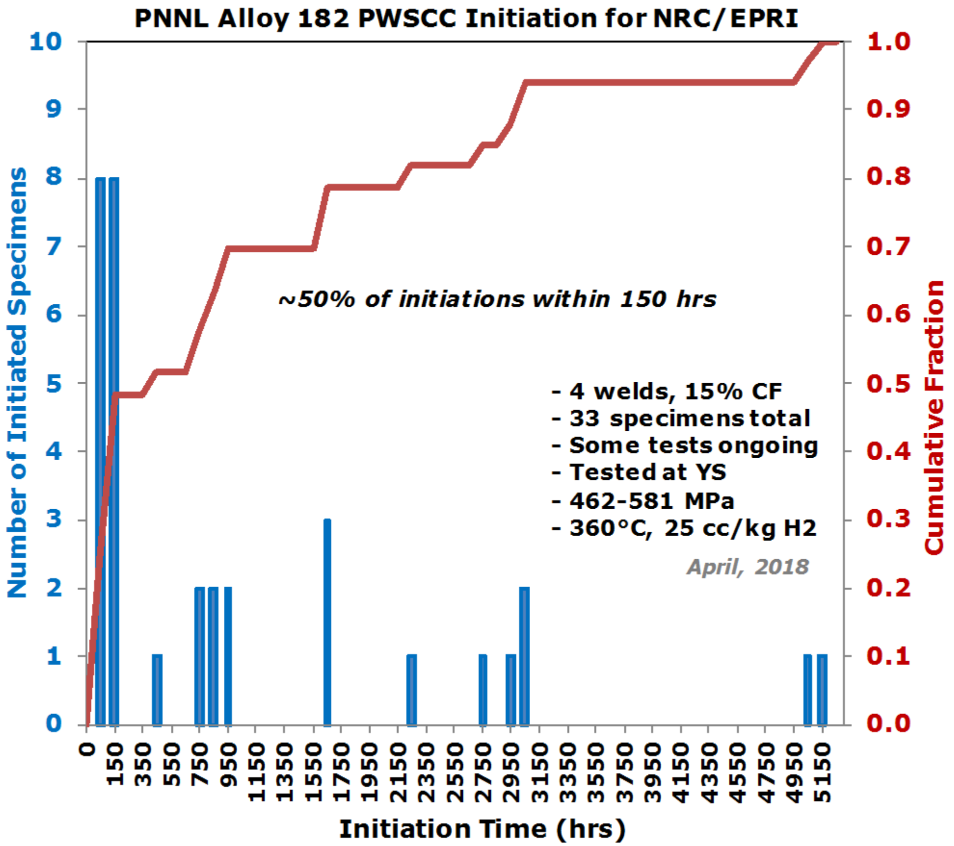
- 3 specimens with long exposures (≥2730 hours) not yet initiated.
- 3 KAPL Alloy 182 specimens not yet tested.



15% CF Alloy 182 Initiation Testing

SCC Initiation Time Histogram

- Approx 50% of initiations at <150 hours.
  - Many of these appear to be SCC initiations occurring upon reaching full load.
  - These are below the PNNL Alloy 600 trend band.
- Decreasing distribution all the way out to ~5000 hours.
- 3 specimens with long exposures (≥2730 hours) not yet initiated.
- 3 KAPL Alloy 182 specimens not yet tested.



15% CF Alloy 182 Initiation Testing

Current 15% Cold Forged Results

- Testing 9 specimens of each weld in 15% CF condition – most tests completed.
- Some tests not yet started or were stopped before SCC initiation.
- Wide range of response for each weld.
- Many very low SCC initiation times for each weld.

KAPL	Stress (MPa)	t_{init} (h)	Studsvik	Stress (MPa)	t_{init} (h)	Phase 2B	Stress (MPa)	t_{init} (h)	Flawtech	Stress (MPa)	t_{init} (h)
IN166	***563***	***≤30****	*IN169*	*541*	*>5126†*	***IN185***	***514***	***≤105***	***IN188***	***518***	***≤30***
IN167	***552***	***≤30***	***IN170***	***536***	***30***	*IN186*	*514*	*>2730†*	***IN189***	***518***	***≤30***
IN168	***547***	***113***	***IN171***	***534***	***2957***	***IN187***	***514***	***409***	***IN190***	***518***	***90***
IN194	***581***	***1635***	***IN191***	***553***	***83***	***IN197***	***500***	***806***	***IN200***	***528***	***825***
IN195	***575***	***1625***	***IN192***	***559***	***41***	***IN198***	***506***	***4964***	***IN201***	***528***	***746***
IN196	***567***	***1642***	***IN193***	***555***	***41***	***IN199***	***506***	***2238***	***IN202***	***528***	***900***
IN230	TDB‡	TBD	***IN233***	***532***	***≤30***	***IN216***	***462***	***132***	***IN221***	***525***	***106***
IN231	TBD	TBD	***IN234***	***529***	***725***	*IN217*	*467*	*>2971†*	***IN222***	***525***	***113***
IN232	TBD	TBD	***IN235***	***532***	***910***	***IN218***	***467***	***2908***	***IN223***	***525***	***79***

***Bold = Initiated** *† No initiation, testing stopped* *‡ Test not yet started*

15% CF Alloy 182 Initiation Testing

Correlation of SCC Initiation Time to Pre-existing Defects

- Surface breaking weld defects observed on many specimens. Relationship to SCC initiation time?
- Defects in the form of clusters of inclusions or sometimes apparent pre-existing cracks.
- No mechanical damage in the gauge region

15% CF Alloy 182 Initiation Testing

Correlation of SCC Initiation Time to Pre-existing Defects

- Initiation not associated with pre-existing defects in 24 out of 26 of the examined specimens.

KAPL	Stress (MPa)	t_{init} (h)	Studsvik	Stress (MPa)	t_{init} (h)	Phase 2B	Stress (MPa)	t_{init} (h)	Flawtech	Stress (MPa)	t_{init} (h)
IN166	**563**	**≤30***	IN169	541	>5126†	***IN185***	***514***	***≤105***	***IN188***	***518***	***≤30***
IN167	***552***	***≤30***	***IN170***	***536***	***30***	IN186	514	>2730†	***IN189***	***518***	***≤30***
IN168	***547***	***113***	***IN171***	***534***	***2957***	***IN187***	***514***	***409***	***IN190***	***518***	***90***
IN194	***581***	***1635***	***IN191***	***553***	***83***	***IN197***	***500***	***806***	***IN200***	***528***	***825***
IN195	***575***	***1625***	***IN192***	***559***	***41***	***IN198***	***506***	***4964***	***IN201***	***528***	***746***
IN196	***567***	***1642***	***IN193***	***555***	***41***	***IN199***	***506***	***2238***	***IN202***	***528***	***900***
IN230	TDB‡	TBD	***IN233***	***532***	***≤30***	***IN216***	***462***	***132***	***IN221***	***525***	***106***
IN231	TBD	TBD	***IN234***	***529***	***725***	IN217	467	>2971†	***IN222***	***525***	***113***
IN232	TBD	TBD	***IN235***	***532***	***910***	***IN218***	***467***	***2908***	***IN223***	***525***	***79***

**** Bold = Initiated*** *† No initiation, testing stopped*

Red = Initiation associated with SEM observable pre-existing defect

Yellow = Cracking from pre-existing defect, but not dominant crack

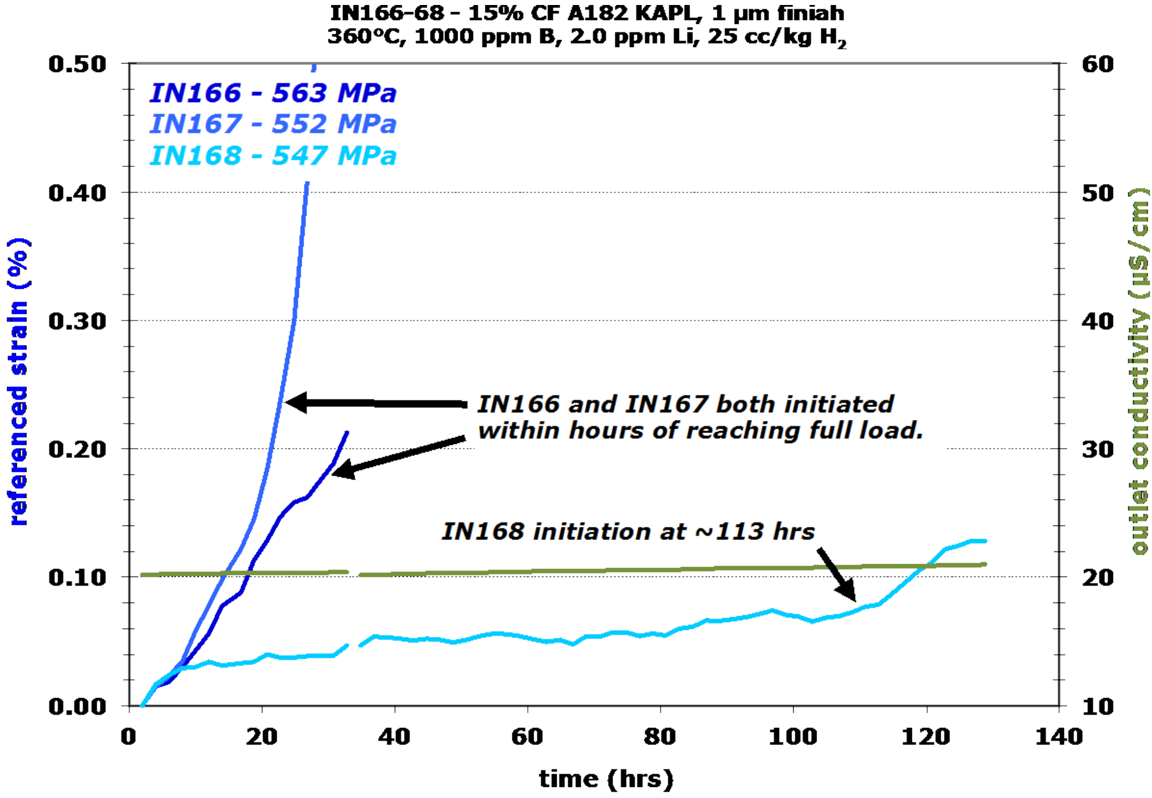
Blue = Initiation not associated with SEM observable pre-existing defect

Grey = No pre-test SEM examination, or post-test exam not yet performed

15% Alloy 182 Initiation Testing

First Three 15% CF KAPL SCC Initiation Tests

- ***Examples of very low SCC initiation times.***
- Specimens tested together.
- IN166, IN167 exhibited very high slope upon reaching full load.
- Upturn in DCPD response at ~15 hours evident for IN167.
- No upturn in IN166 but high slope.
- If high slope is sufficient indication for SCC initiation, then IN166 and IN167 both initiated at the onset of testing.
- Assume initiation time of <30 hours for IN166, IN167.
- IN168 initiated at 113 hours.



15% CF Alloy 182 Initiation Testing

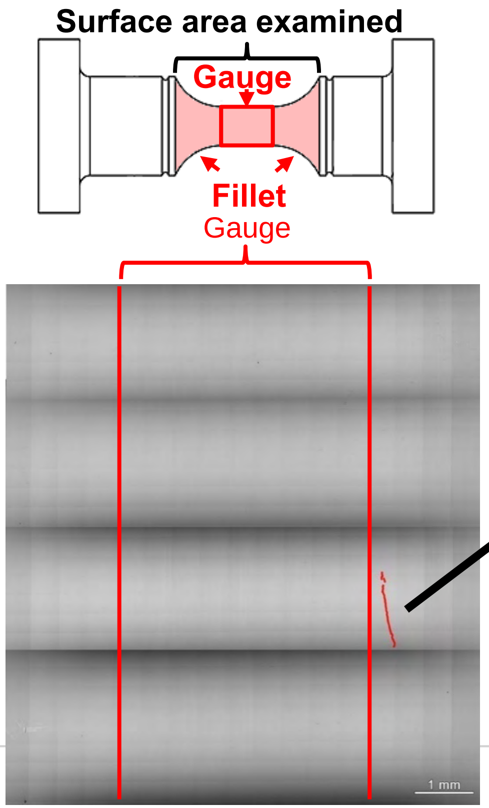
KAPL IN166 Gauge Surface Observations

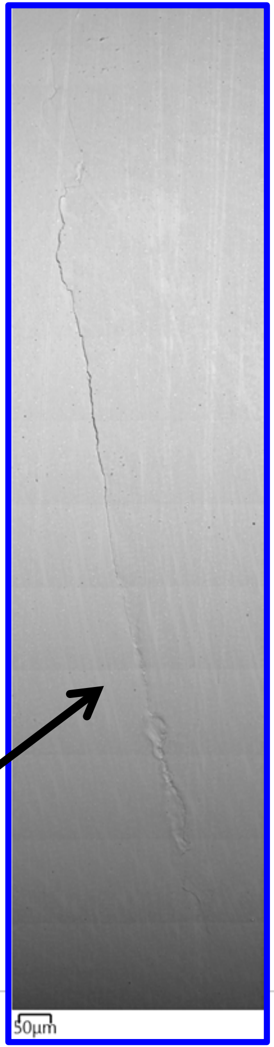
- Initiation in <30 hours.
- No unusual features during optical examinations prior to SCC initiation testing.
- Gauge surface observed by SEM after initiation.

- Entire gauge region and fillets are recorded.
- Four “rotations” required to image entire surface.

- One crack found with surface length >1 mm.

Post-test SEM observation of entire gauge surface

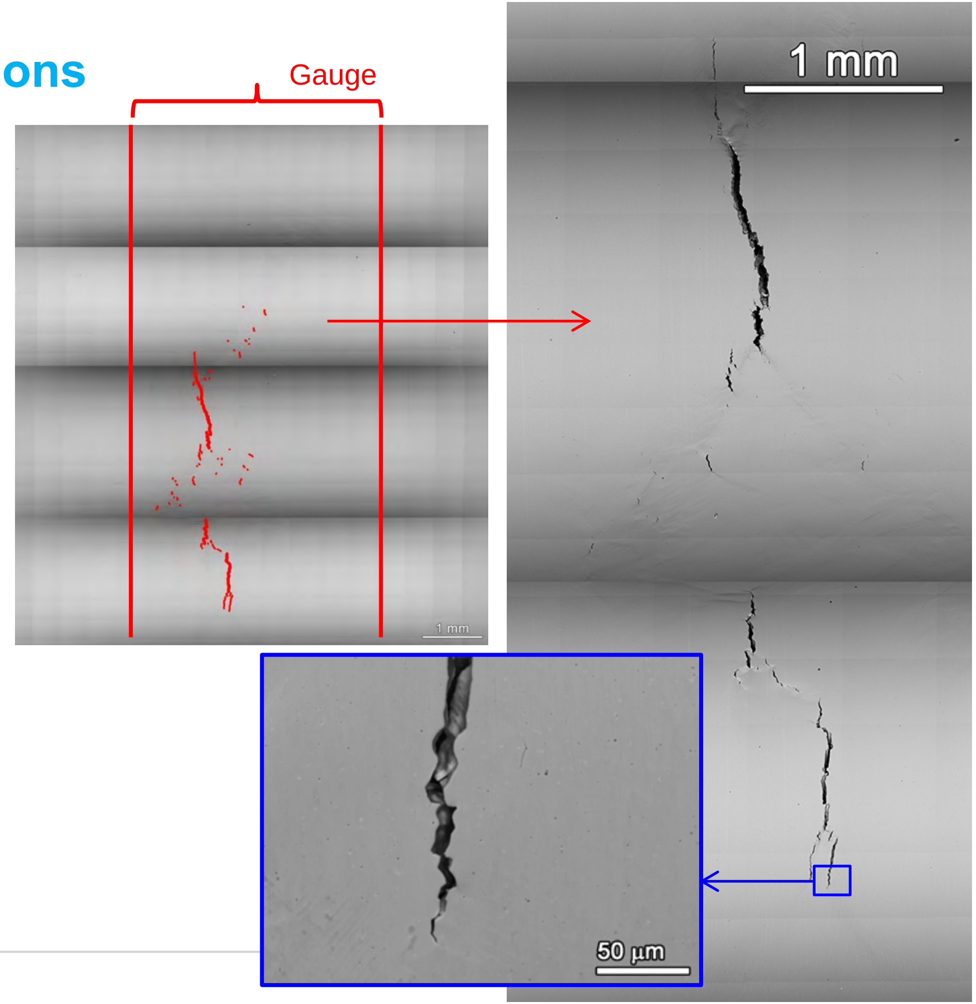




15% Alloy 182 Initiation Testing

KAPL IN167 Gauge Surface Observations

- Initiation in <30 hours.
- No unusual features during <u>optical</u> examinations prior to SCC initiation testing.
- Two long cracks in gauge region with large openings.
- Many smaller cracks in the vicinity of the main cracks.

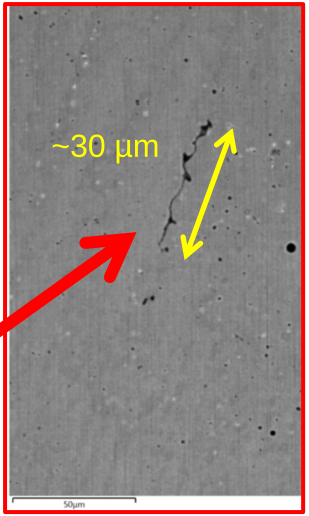


15% CF Alloy 182 Initiation Testing

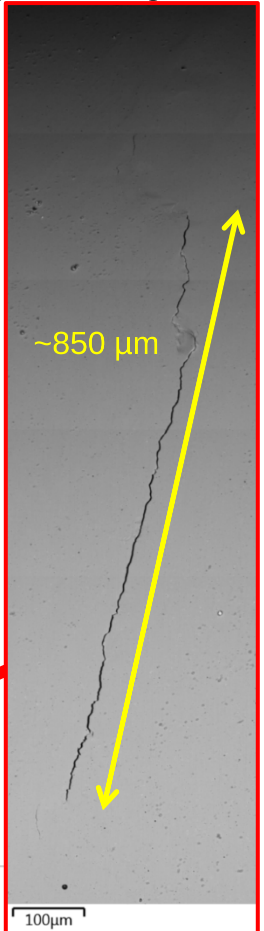
KAPL IN168 Gauge Surface Observations

- 113 hour initiation time.
- Optical examination prior to testing did not show any pre-existing defects.
- Midtest examination at 33 hours did reveal a small crack-like defect with smooth appearance suggesting a pre-existing defect.
- After initiation, this defect had clearly grown to become the primary crack.

33 hours: ***Smooth appearance suggests a pre-existing defect***



130 hours: ***Primary crack nucleated from what appears to be a pre-existing defect.***

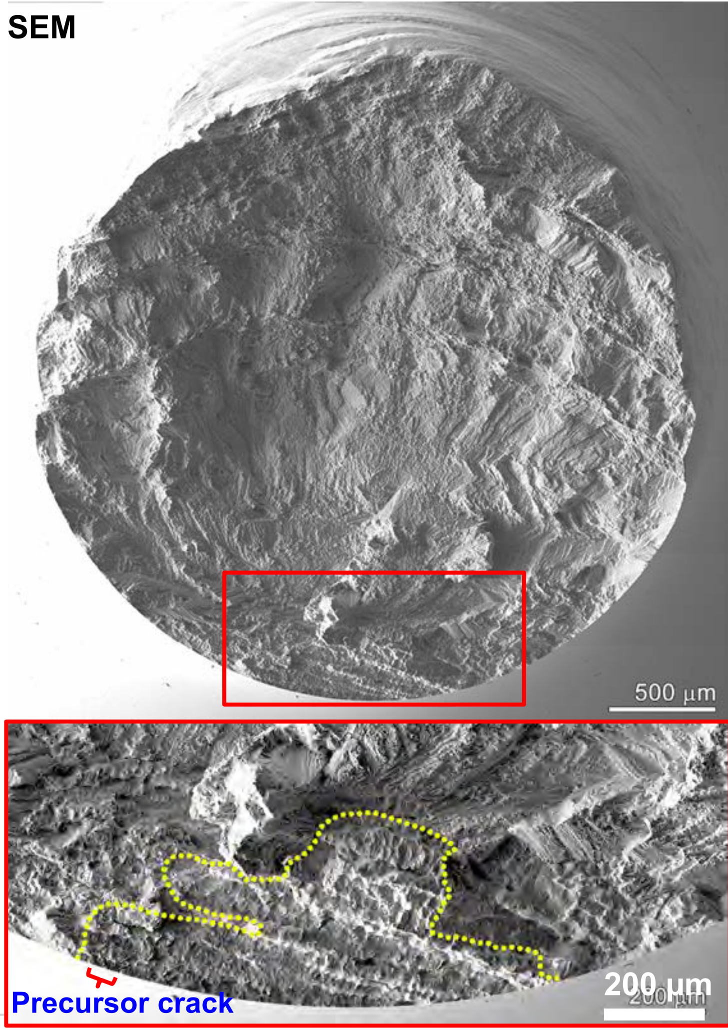


33 hours exposure (before DCPD initiation)

130 hours exposure (after DCPD initiation)

15% CF Alloy 182 Initiation Testing
KAPL IN168 Crack Surface Observations

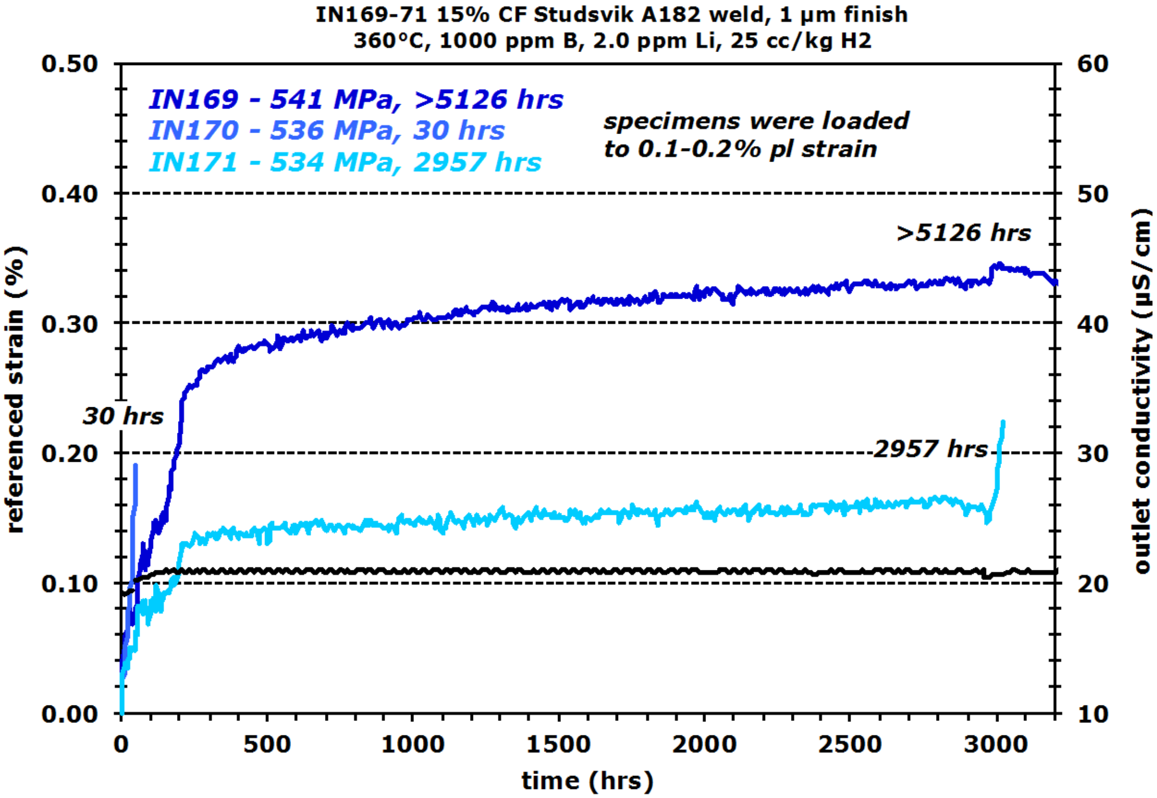
- 113 hour initiation time.
- After surface examinations are complete, specimens are air fatigue cracked to failure to expose crack surface.
- Allows characterization of crack shape and depth.
- This is the first attempt at performing this procedure.



15% Alloy 182 Initiation Testing

First Three 15% CF Studsvik SCC Initiation Tests

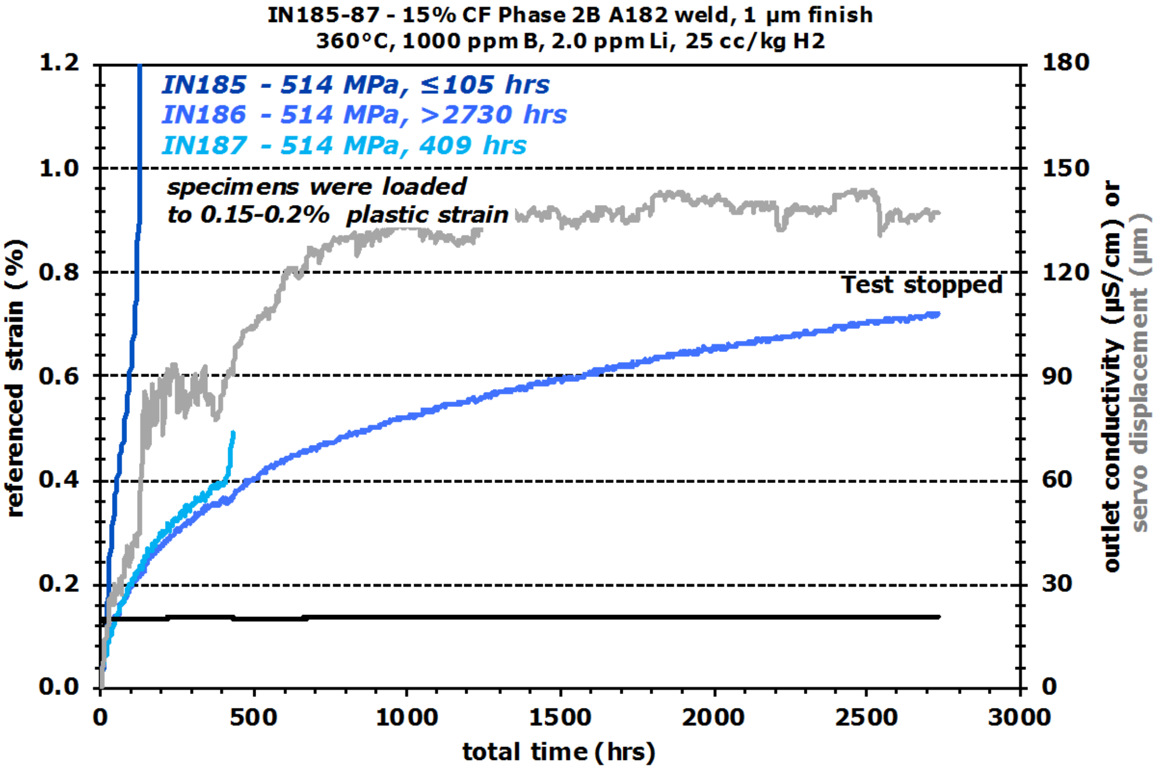
- Specimens tested together.
- IN170 exhibited very high slope upon reaching full load with initiation in 30 hours.
- The other two specimens exhibited much longer initiation times.



15% Alloy 182 Initiation Testing

First Three 15% CF Phase 2B SCC Initiation Tests

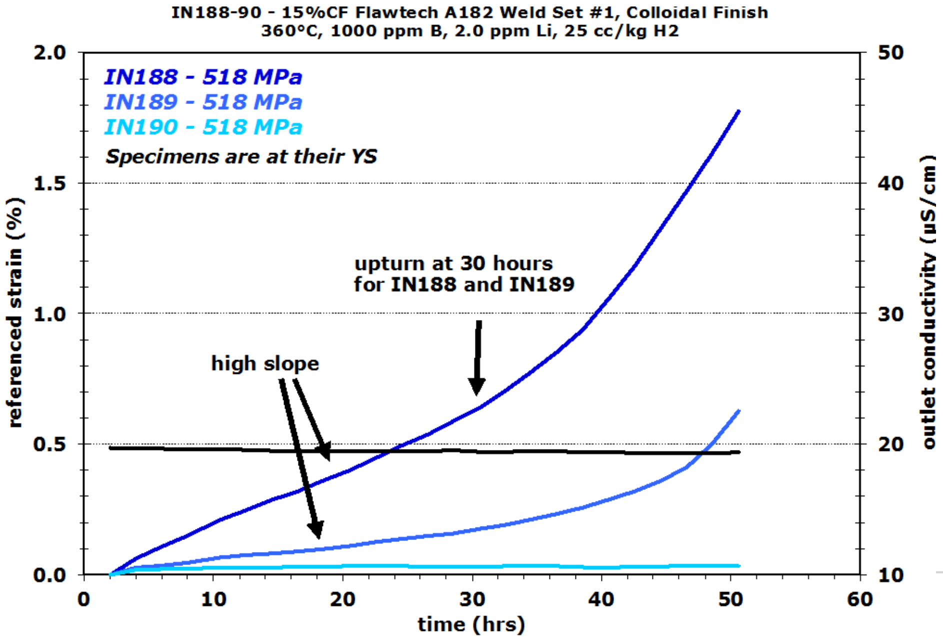
- Specimens tested together.
- IN185 exhibited very high slope upon reaching full load. Change to increasing slope at 105 hrs.
- IN187 initiated at 409 hrs.
- Testing of IN186 stopped at 2730 hrs to allow starting tests on other specimens.

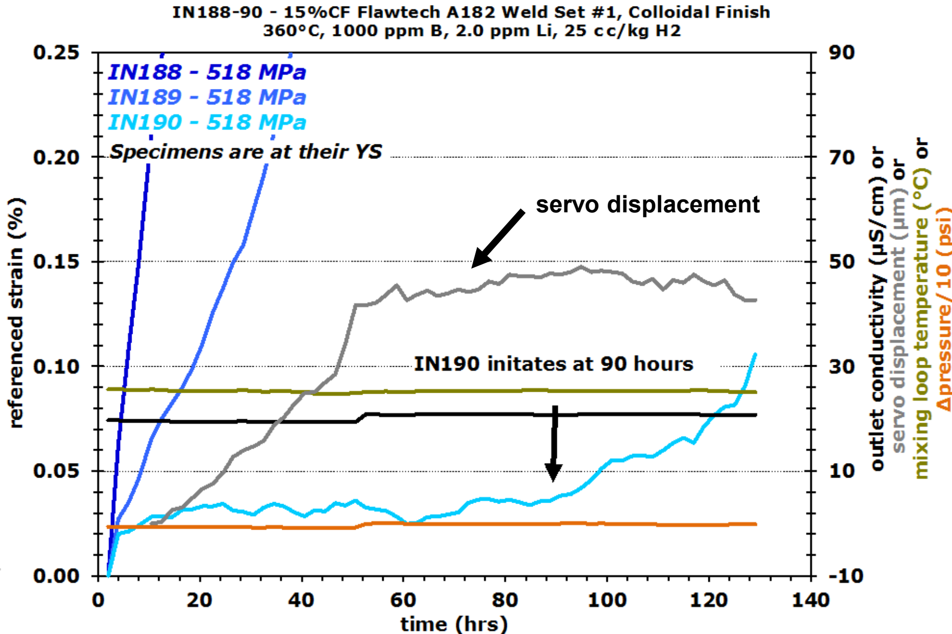


15% CF Alloy 182 Initiation Testing

First Round of 15% CF Flawtech SCC Initiation Tests

- IN188 and IN189 both exhibited high initial slope followed an increase in slope at 30 hours.
- IN190 initiated at 90 hours.
- Detailed pre-test SEM examinations were performed on these specimens.

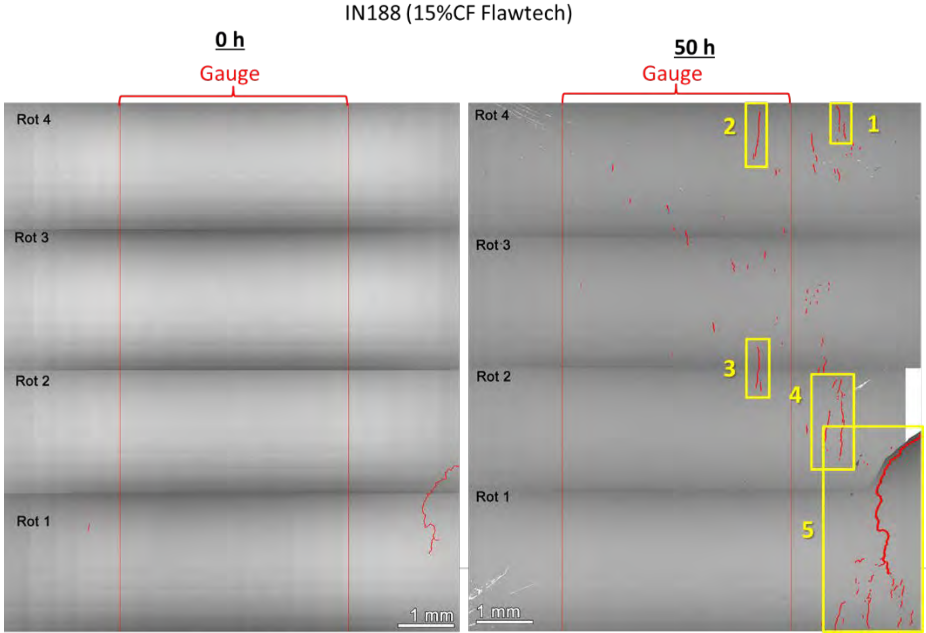




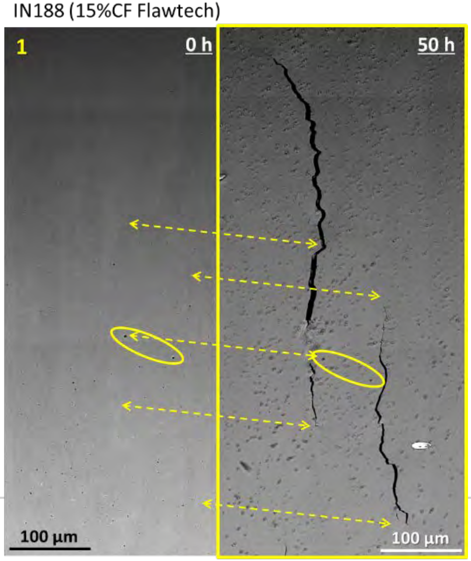
15% CF Alloy 182 Initiation Testing

Flawtech IN188 Gauge Surface Observations

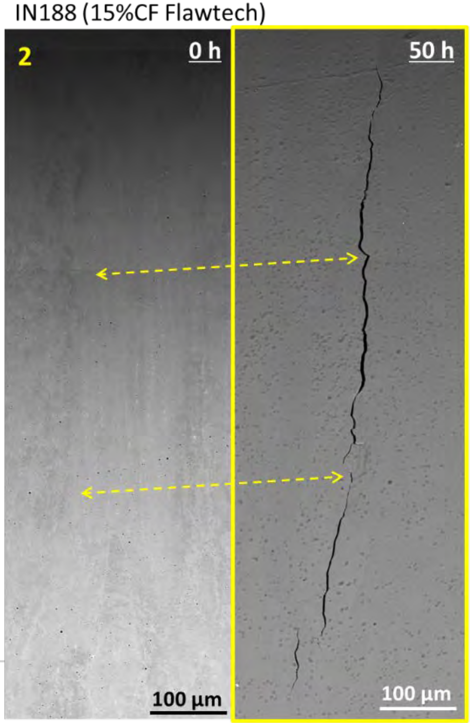
- SCC initiation in ≤30 hours.
- No detectable defects in pre-test examination.
- Extensive cracking after 50 hours at full load.
- Comparison of crack locations to pre-test images confirm no evidence of pre-existing defects.



Site #1 (crack develops from apparent defect-free region)



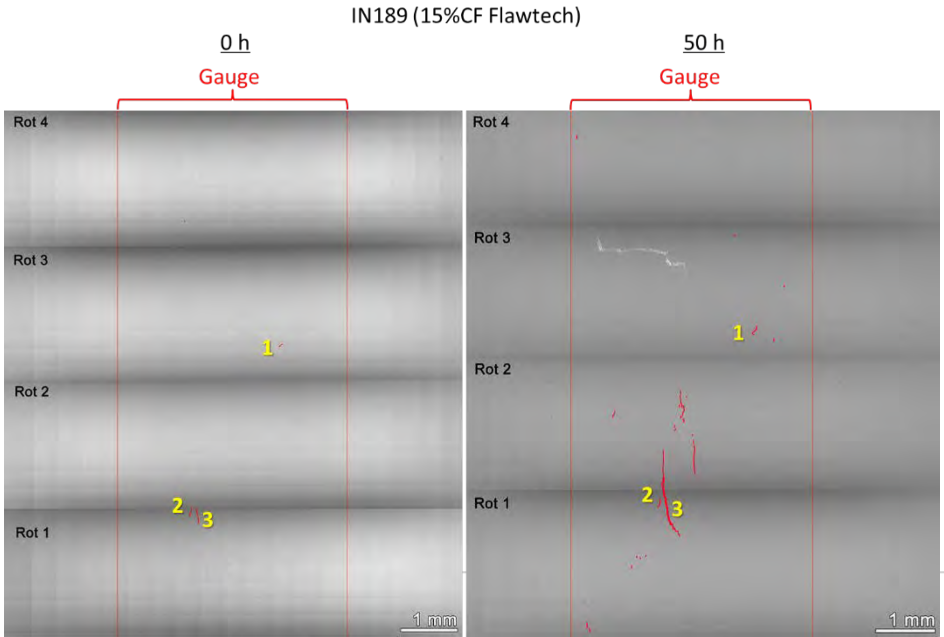
Site #2 (crack develops from apparent defect-free region)



15% CF Alloy 182 Initiation Testing

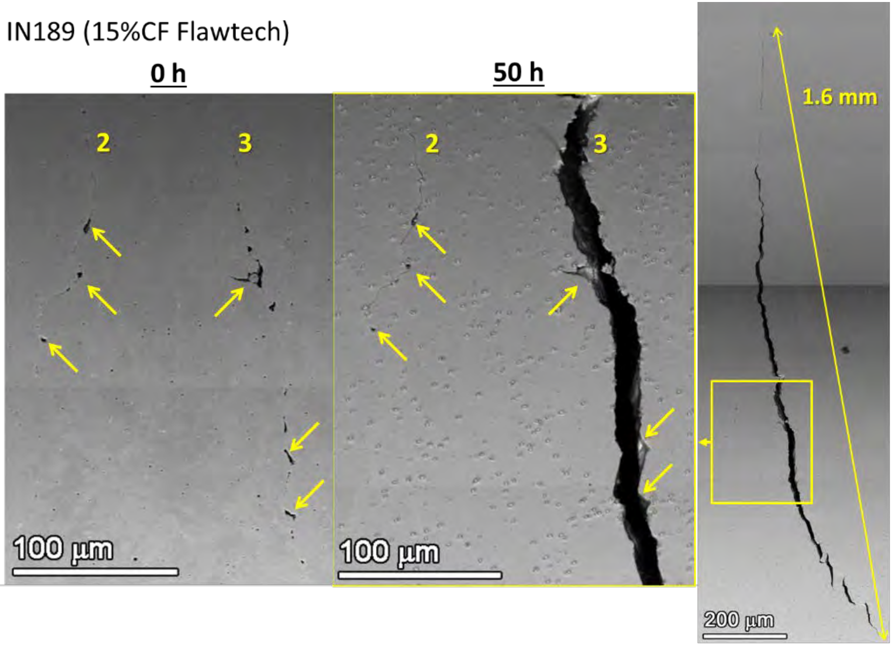
Flawtech IN189 Gauge Surface Observations

- SCC initiation in ≤30 hours.
- Crack-like defects observed in pre-test examination.
- Extensive cracking after 50 hours at full load.
- Largest crack corresponds to a pre-test defect, but some cracks again nucleated from apparent defect-free regions.



Site #2 (pre-test defect does not mature)

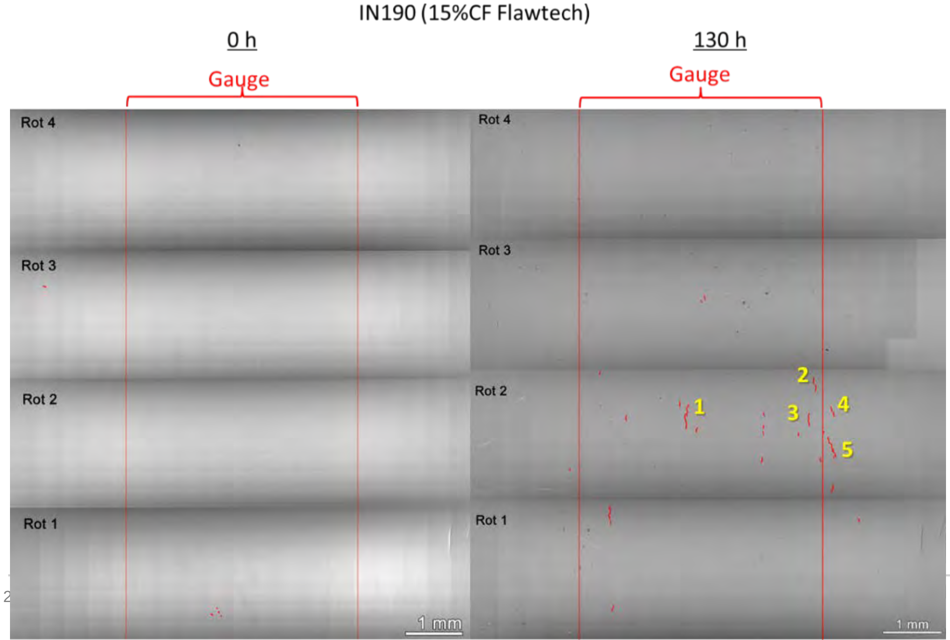
Site #3 (pre-test defect becomes dominant crack)



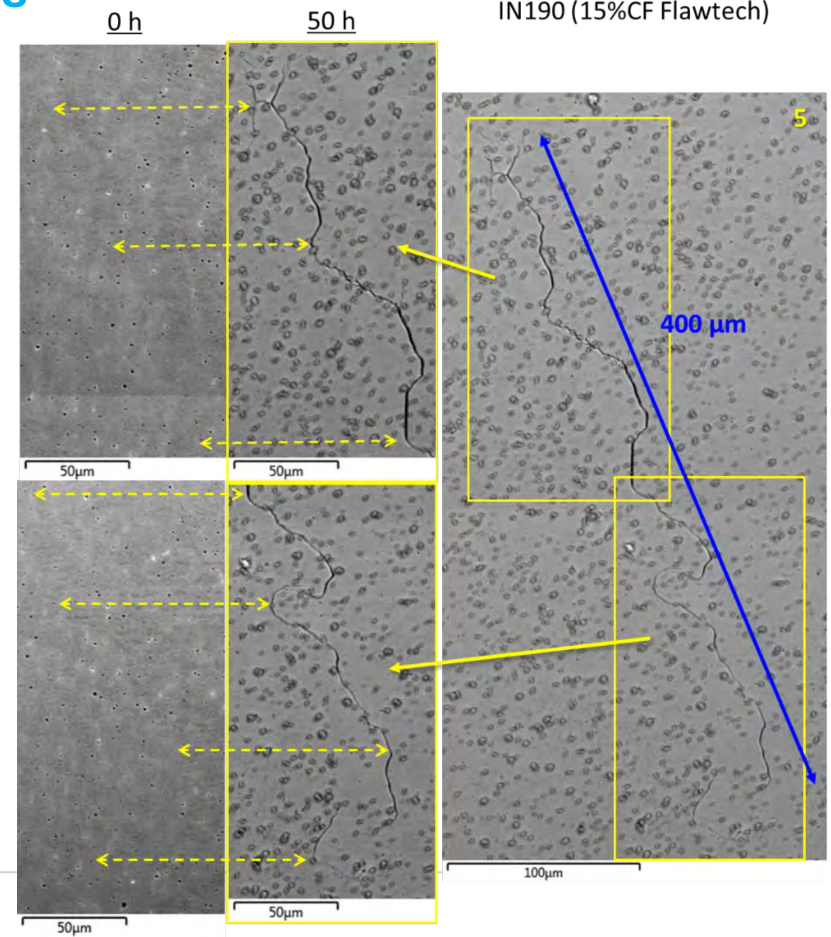
15% CF Alloy 182 Initiation Testing

Flawtech IN190 Gauge Surface Observations

- SCC initiation in 90 hours.
- No detectable defects in pre-test examination.
- Extensive cracking after 50 hours at full load.
- Comparison of crack locations to pre-test images confirms no evidence of pre-existing defects.



Site #5 (largest crack on specimen develops from apparent defect-free region)



15% CF Alloy 182 Initiation Testing

Correlation of SCC Initiation Time to Pre-existing Defects

- Initiation not associated with pre-existing defects in 24 out of 26 of the examined specimens.

KAPL	Stress (MPa)	t_{init} (h)	Studsvik	Stress (MPa)	t_{init} (h)	Phase 2B	Stress (MPa)	t_{init} (h)	Flawtech	Stress (MPa)	t_{init} (h)
IN166	**563**	**≤30***	IN169	541	>5126†	***IN185***	***514***	***≤105***	***IN188***	***518***	***≤30***
IN167	***552***	***≤30***	***IN170***	***536***	***30***	IN186	514	>2730†	***IN189***	***518***	***≤30***
IN168	***547***	***113***	***IN171***	***534***	***2957***	***IN187***	***514***	***409***	***IN190***	***518***	***90***
IN194	***581***	***1635***	***IN191***	***553***	***83***	***IN197***	***500***	***806***	***IN200***	***528***	***825***
IN195	***575***	***1625***	***IN192***	***559***	***41***	***IN198***	***506***	***4964***	***IN201***	***528***	***746***
IN196	***567***	***1642***	***IN193***	***555***	***41***	***IN199***	***506***	***2238***	***IN202***	***528***	***900***
IN230	TDB‡	TBD	***IN233***	***532***	***≤30***	***IN216***	***462***	***132***	***IN221***	***525***	***106***
IN231	TBD	TBD	***IN234***	***529***	***725***	IN217	467	>2971†	***IN222***	***525***	***113***
IN232	TBD	TBD	***IN235***	***532***	***910***	***IN218***	***467***	***2908***	***IN223***	***525***	***79***

*** Bold = Initiated*** *† No initiation, testing stopped*

Red = Initiation associated with SEM observable pre-existing defect

Yellow = Cracking from pre-existing defect, but not dominant crack

Blue = Initiation not associated with SEM observable pre-existing defect

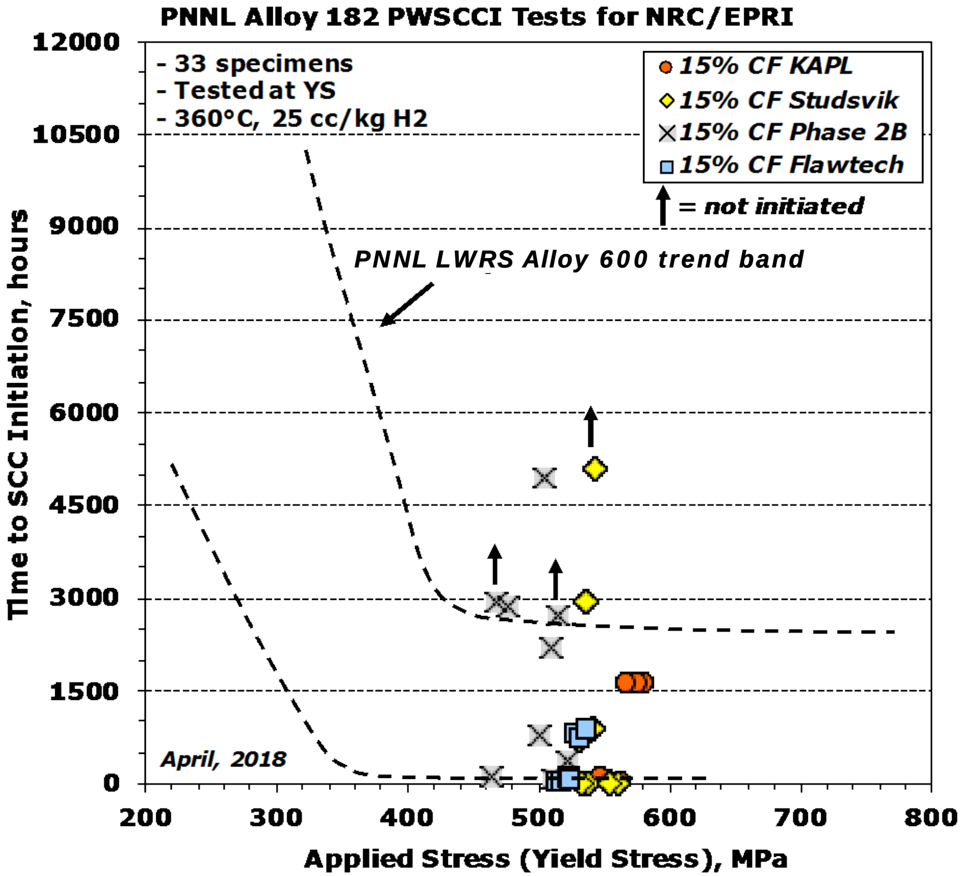
Grey = No pre-test SEM examination, or post-test exam not yet performed

15% CF Alloy 182 Initiation Testing

SCC Initiation Time vs Stress Plot

- Distinct grouping of data at <150 hours.
  - Many of these appear to be SCC initiations occurring upon reaching full load.
- Decreasing distribution all the way out to ~5000 hours.
- Most data are in the bottom half of the alloy 600 trend band.

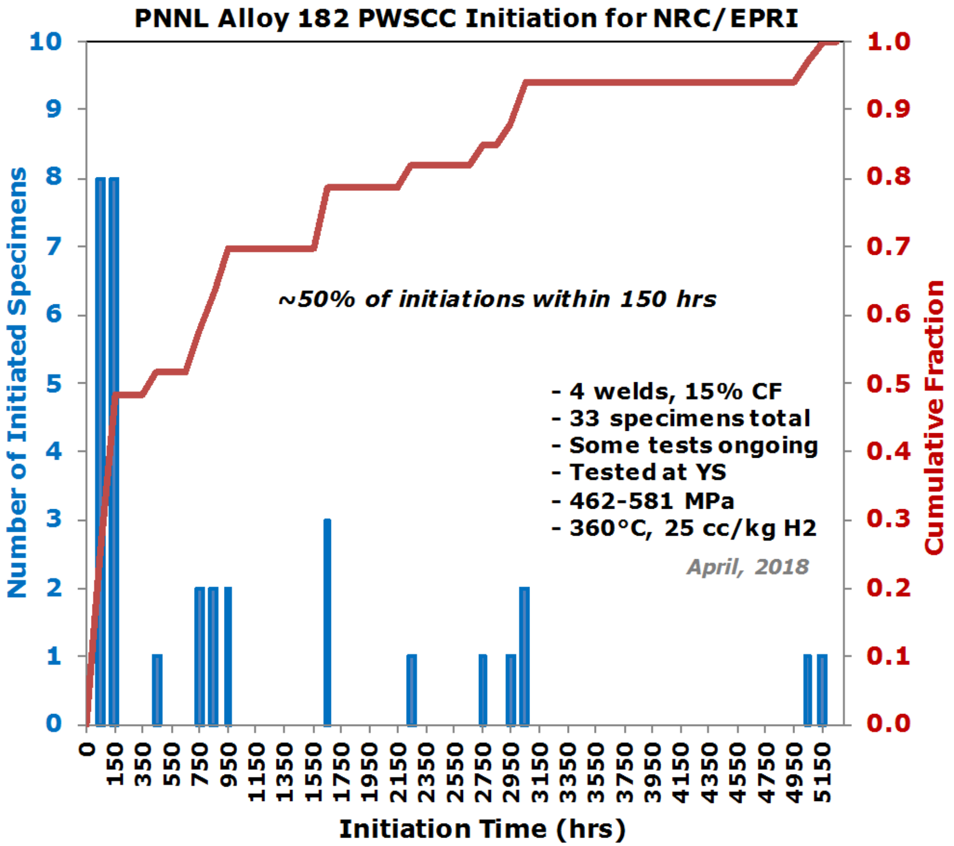
- 3 specimens with long exposures (≥2730 hours) not yet initiated.
- 3 KAPL Alloy 182 specimens not yet tested.



15% CF Alloy 182 Initiation Testing

SCC Initiation Time Histogram

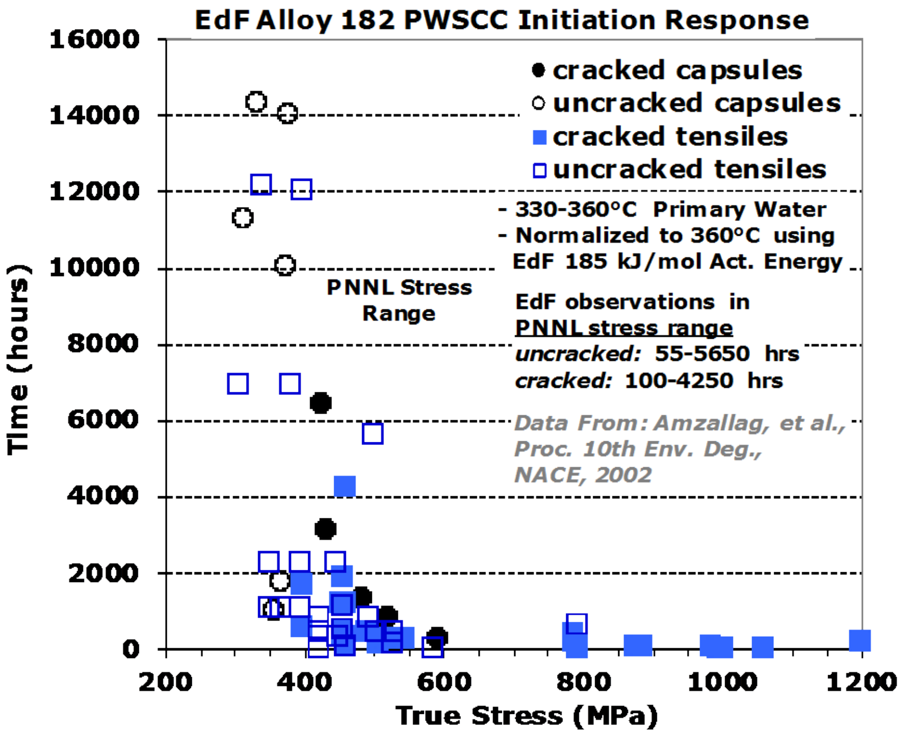
- Approx 50% of initiations at <150 hours.
  - Many of these appear to be SCC initiations occurring upon reaching full load.
  - These are below the PNNL Alloy 600 trend band.
- Decreasing distribution all the way out to ~5000 hours.
- 3 specimens with long exposures (≥2730 hours) not yet initiated.
- 3 KAPL Alloy 182 specimens not yet tested.



Alloy 182 Initiation Testing

Comparison to Other Labs – EdF

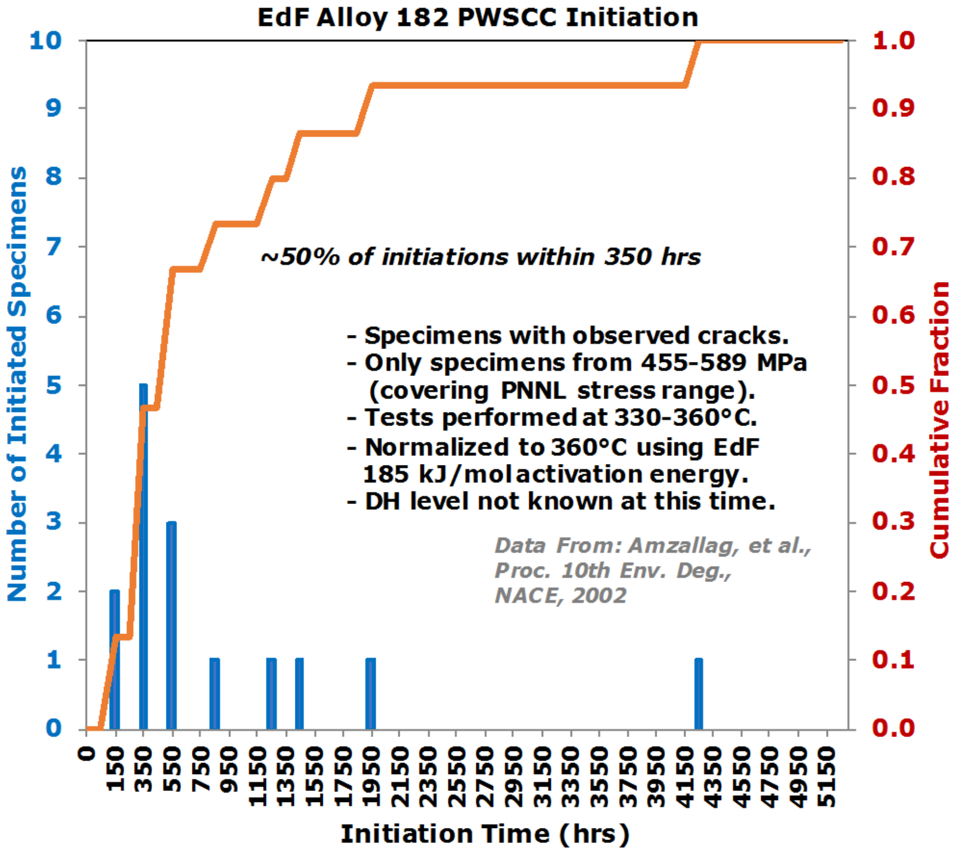
- Large amount of constant load data generated by EdF.
- For similar stresses and test temperature to PNNL testing, EdF Alloy 182 initiation times are ~100-5650 hours.
- Good overlap between PNNL and EdF times.
- Broad range of initiation times for PNNL and EdF suggest that Alloy 182 may have local highly susceptible regions. This is consistent with highly variable *SCCGR* response of Alloy 182.



Alloy 182 Initiation Testing

Comparison to Other Labs – EdF Histogram

- EdF data at similar stresses and test temperature to PNNL tests.
- Only specimens with observed cracks.
- Approx 50% of initiations at <350 hours, similar to PNNL observation of many low SCC initiation times.
- Decreasing distribution all the way out to ~4500 hours. Distribution tail goes out to >5650 hours when including exposure times for EdF specimens that have not cracked.
- Trend in distribution matches PNNL test results.

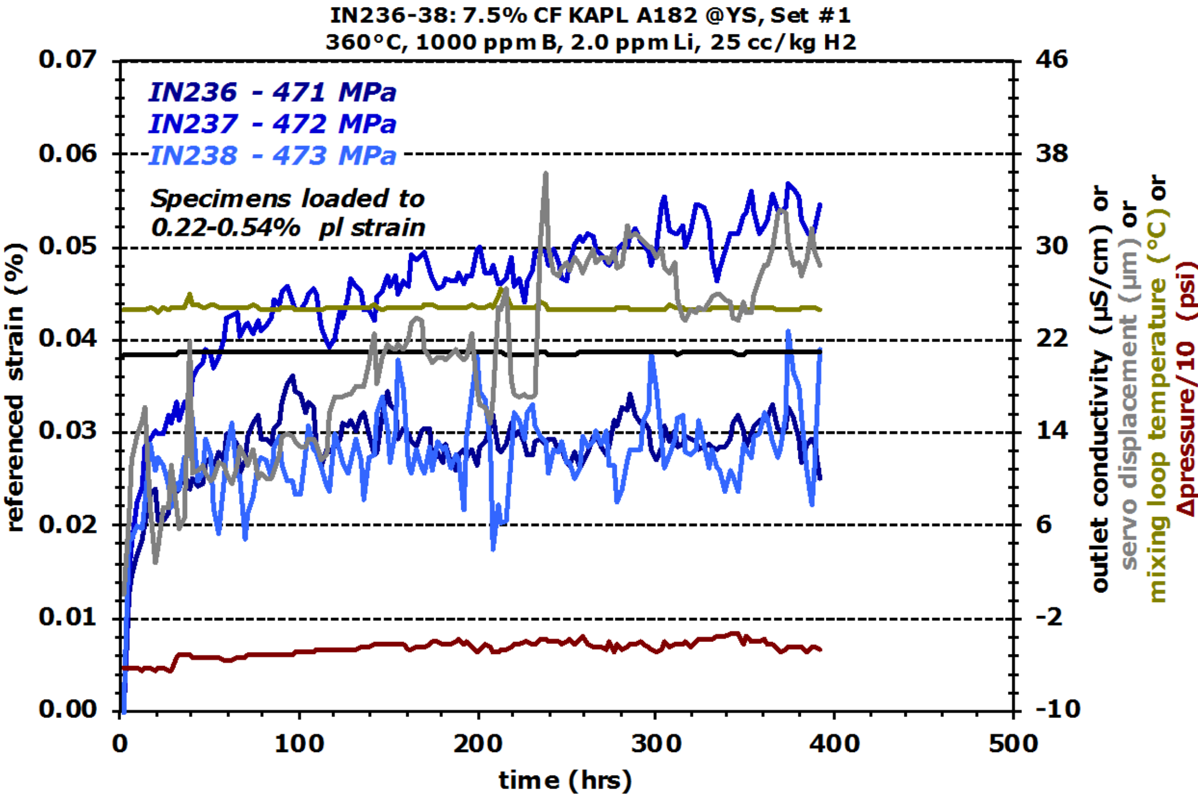


Outline

- Objectives
- Material Selection and Fabrication
- Test Systems and Testing Approach
- 15% CF Alloy 182 SCC Initiation Behavior
- **Alloy 182 Initiation Behavior Versus Cold Work**
- Summary

7.5% CF Alloy 182 Initiation Testing
KAPL Alloy 182

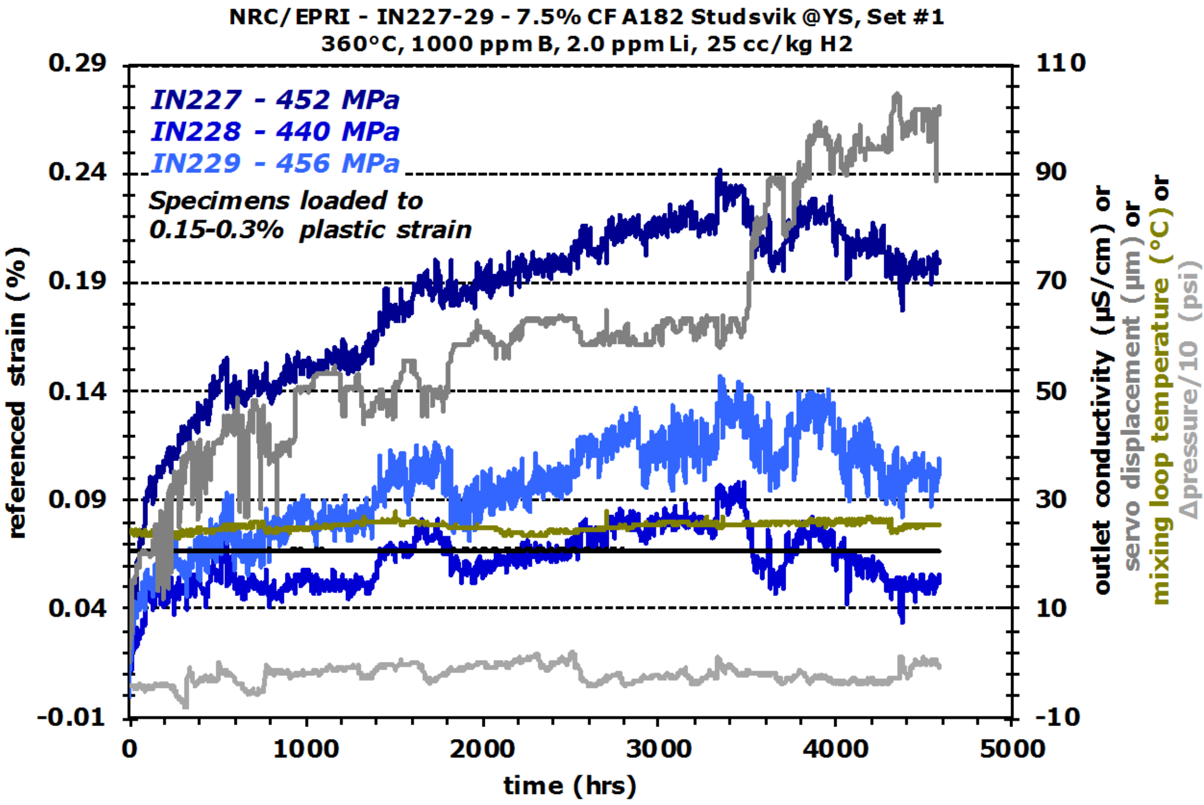
- First 3 out of 6 specimens to be tested.
- Test stress is ~470 MPa.
  - Test stress of 15% CF is 547-581 MPa.
- Only 391 hours accumulated.
- No indication of SCC initiation



7.5% CF Alloy 182 Initiation Testing

Studsvik Alloy 182

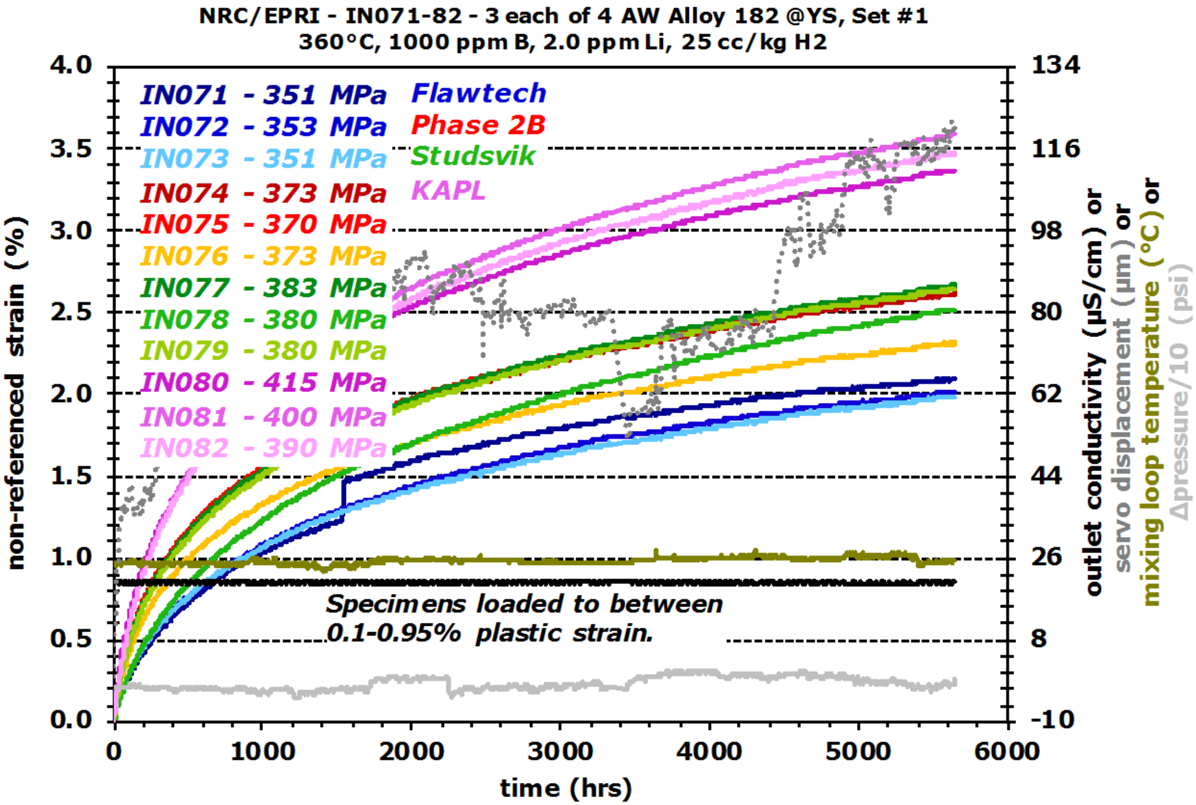
- First 3 out of 6 specimens to be tested.
- Test stress is ~450 MPa.
  - Test stress of 15% CF is 529-559 MPa.
- 4582 hours accumulated.
- Some apparent variability in the referenced strains due to operation of this particular test system (Valving in/out of a piggyback autoclave system).
- However, no indication of SCC initiation.



As-welded Alloy 182 Initiation Testing

All Four Welds

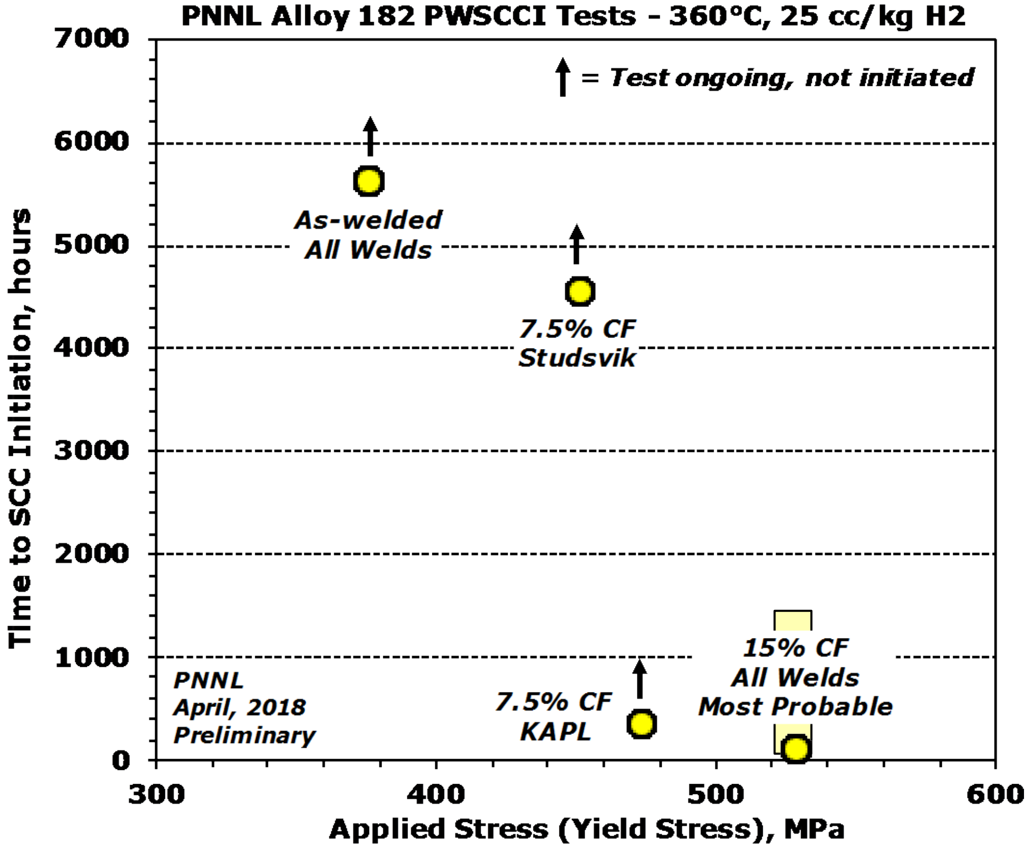
- Current plan is to test 3 of each weld, but will likely expand to 6 of each weld.
- 5650 hours accumulated.
- Monitoring non-referenced strain. Capability to monitor reference for 12 specimens not yet established.
- Indication of a strain jump in one KAPL weld specimen.
  - Often a precursor to initiated behavior, but no sign of SCCI.
  - May have been due to handling of the DCPD wiring.



As-welded Alloy 182 Initiation Testing

SCC Initiation Time Versus Yield Strength by Cold Work

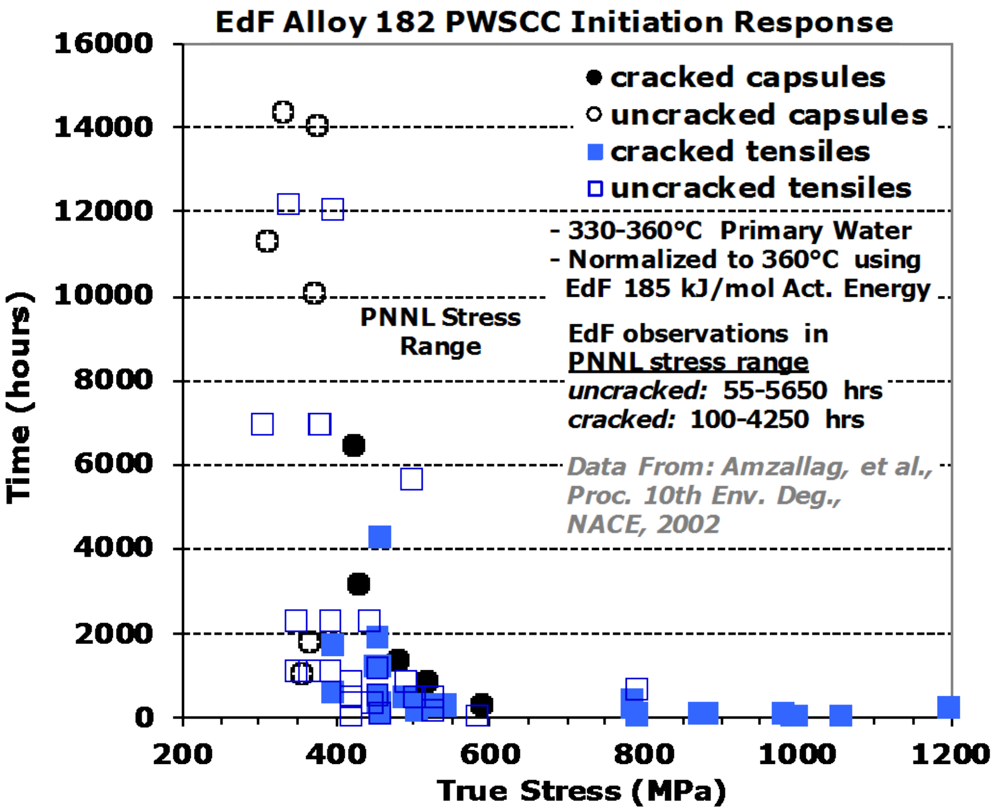
- Preliminary result.
- Nearly half of 15% CF weld initiations below 150 hours.
- Data suggesting a strong reduction in initiation time as applied stress increases from 450 and 550 MPa, same as with EdF data.



Alloy 182 Initiation Testing

Comparison to Other Labs – EdF Trend

- EdF SCC initiation times increase dramatically as stresses drop below 450 MPa.



15% CF Alloy 182 Summary

- 15% CF Alloy 182 is exhibiting a wide range of SCC initiation times.
- Initiation times are well below and above that of Alloy 600 with similar cold work levels.
- Many initiation events appear to have occurred almost from the moment a specimen reached full load.
- Gauge surface of specimens documented before and after SCC initiation.
  - In most cases, low initiation times associated with grain boundaries having no observable pre-existing microstructural or macroscopic defects.
  - In a few cases, low SCC initiation times correlated to pre-existing weld defects.
  - These pre-existing defects are not due to physical damage to the specimens.
- PNNL observations match up well with EdF constant load tests.
- Trend to-date suggests localized regions of very high susceptibility in Alloy 182.
- Ongoing tests and microstructural observations to better understand the distribution of response.