Investigation of deformation localization in irradiated austenitic steels

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Light Water Reactor Sustainability

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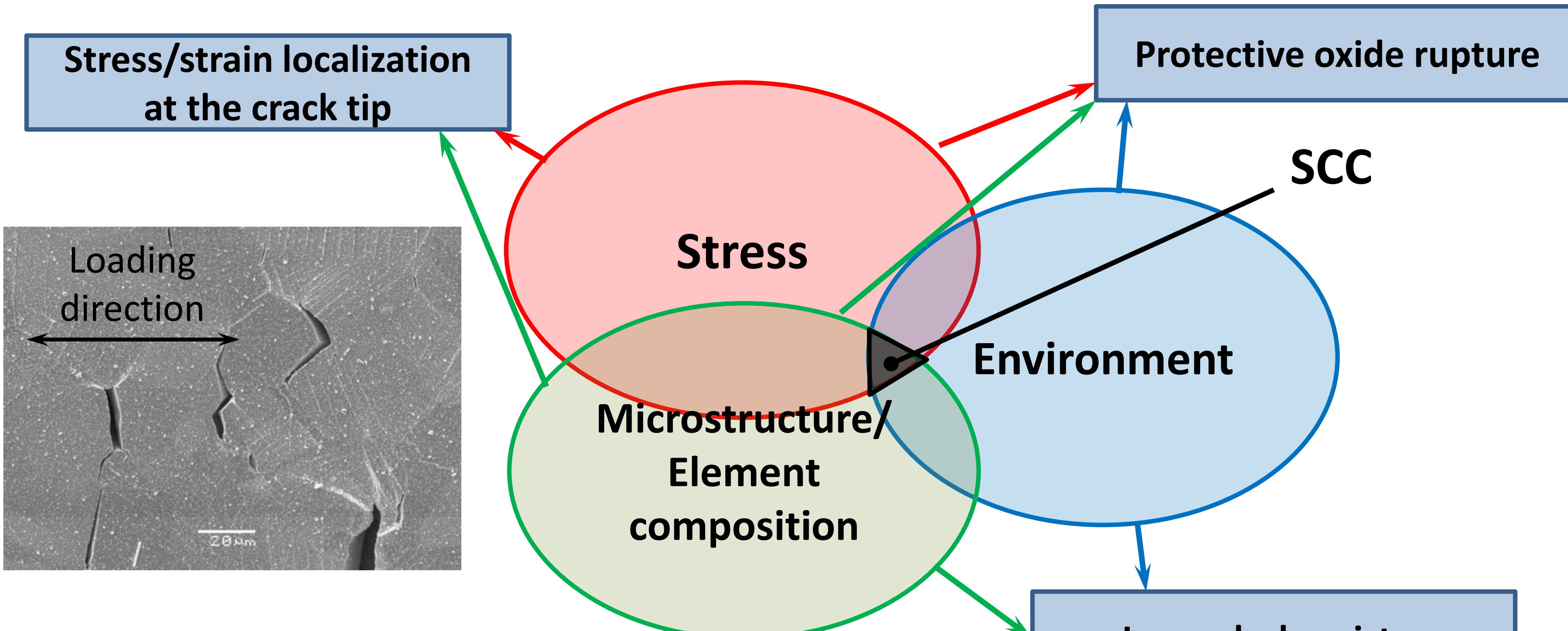
Light Water Reactor Sustainability R&D Program

Presentation outline

- Brief background of irradiation-assisted stress corrosion cracking (IASCC).
- Deformation localization and its role in IASCC.
- Analysis of previous results on deformation localization.
- This FY results.
- Further plans.

Background of stress corrosion cracking

- Stress corrosion cracking (SCC) is often shown schematically as a confluence of stress, material parameters and environment factors.
- SCC is a highly non-linear process with many contributing variables/processes.



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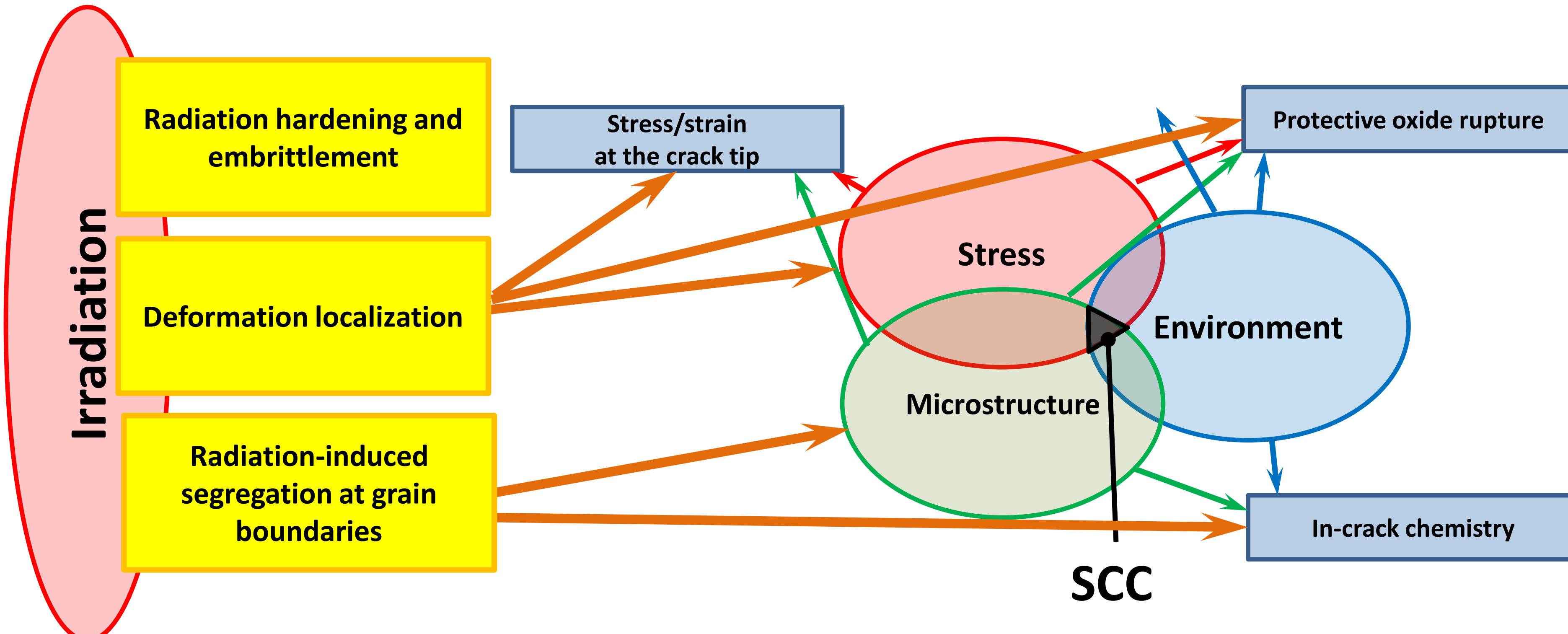
Irradiation assisted stress corrosion cracking

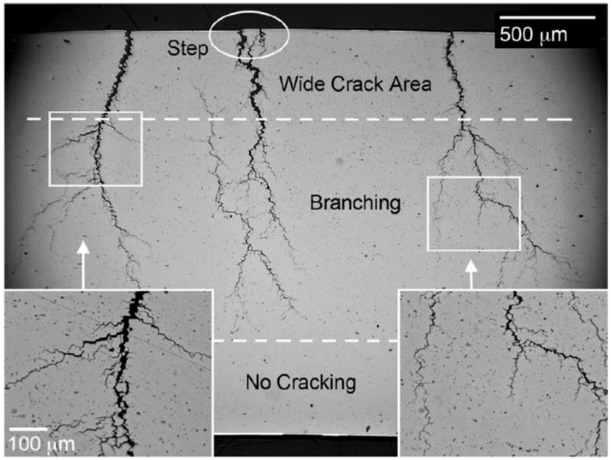
- Nuclear reactor is very harsh environment with a complex combination of stress, temperature, and radiation fields, which vary in time.
- Material structure changes under irradiation producing a number of specific phenomena.

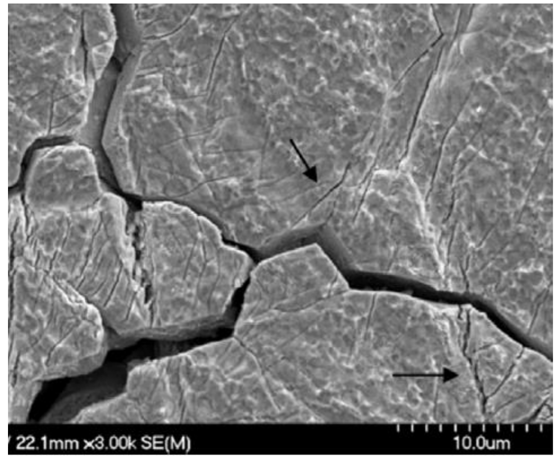


- **At the moment, IASCC is not well understood; no single controlling parameter exists.**

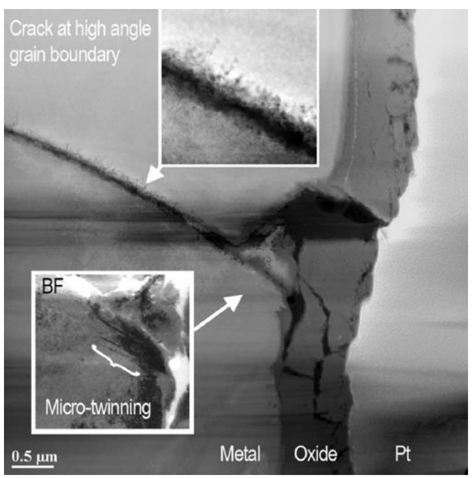
An example of low IASCC predictability

- **Well-known, trusted, and (as was thought) well-understood 718-alloy demonstrated stress-corrosion cracking under LWR conditions.**



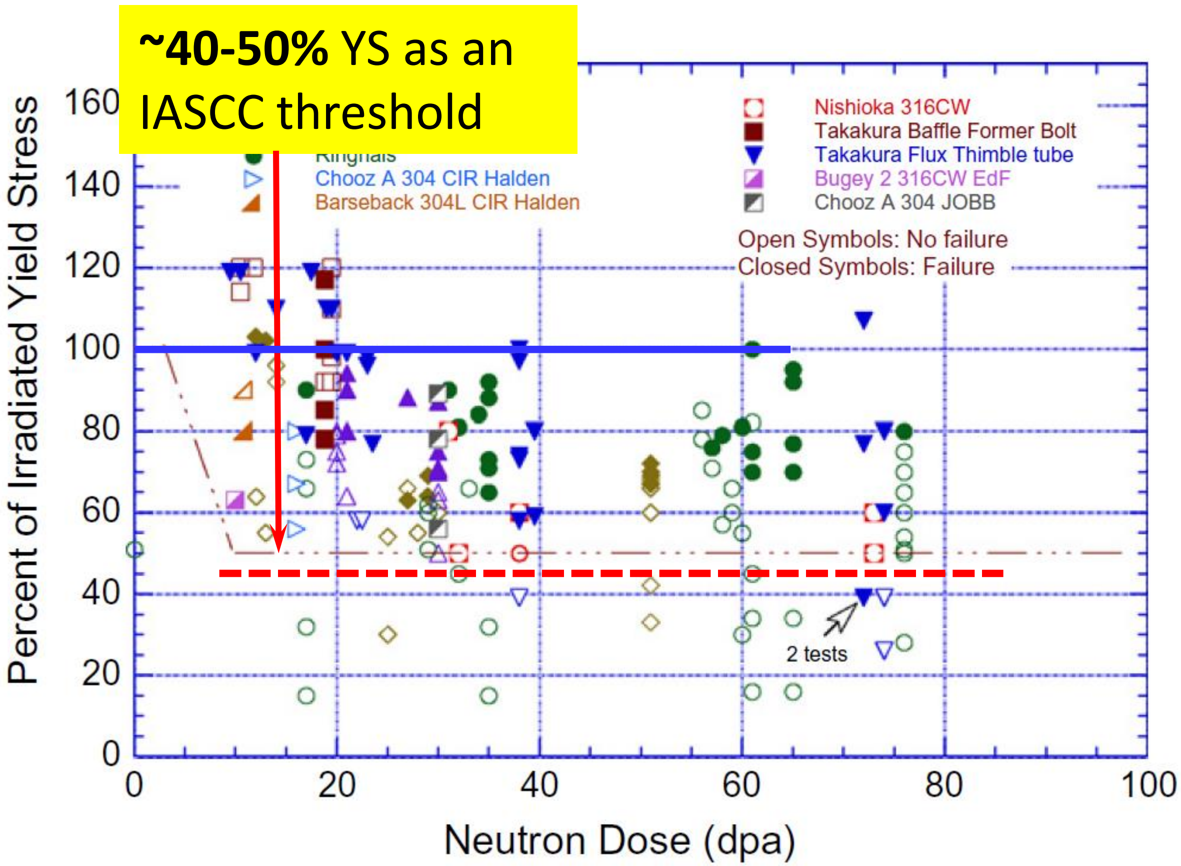


Leonard K.J. et al,
JNM, 2015.



- A full post-irradiation examination performed on irradiated 718-alloy components showed damage following in-service exposure.
- According to the preliminary conclusion, the IASCC was caused by combination of stress level above yield stress (which was not a sole reason) and **slightly decreased amount of delta-phase**.
- It is important that all damaged and non-damaged components were produced within the same specification.

IASCC initiation as a function of stress



Stress as percent of yield stress value for IASCC initiation in a PWR (after Chopra and Rao, JNM, 2011).

According to the literature (for example, Chopra and Rao, JNM, 2011), a threshold stress for the IASCC initiation may exist (roughly, ~40-50% of yield stress, YS).

Not all researchers share the threshold concept. Nevertheless, one may argue that keeping the stress levels below this value will provide a protection against IASCC. However, it is difficult to do because of complex processes in the working LWR: creep, swelling, refueling operations, component replacement etc.

The other argument may be: **why we expect plastic strain below yield stress limit?**

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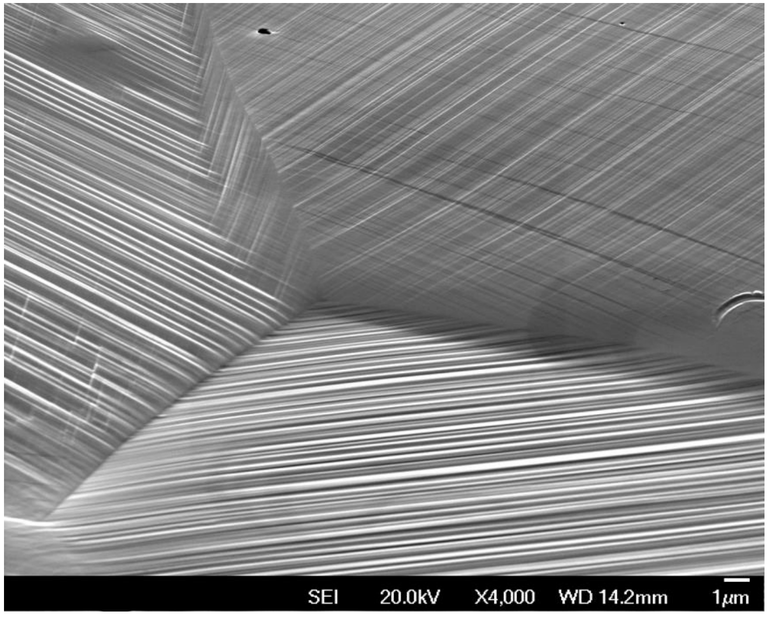
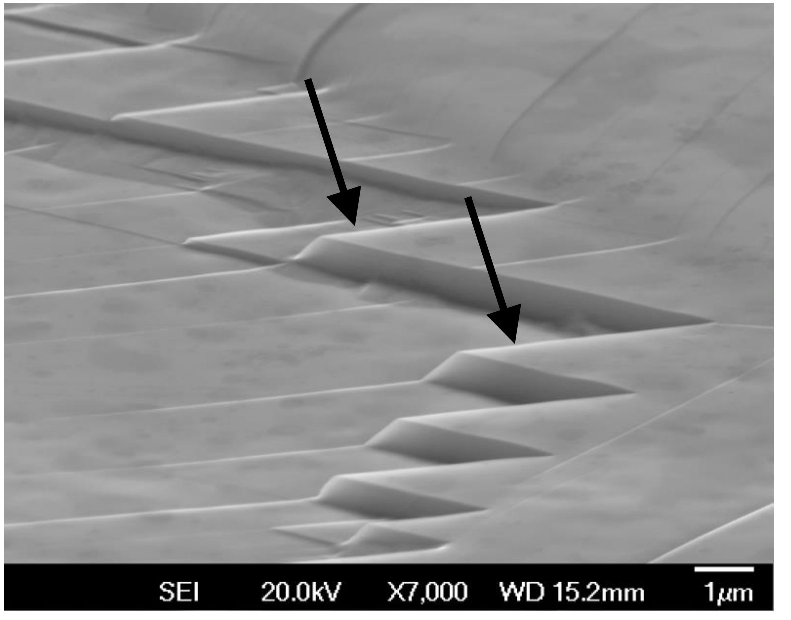
Deformation localization in the irradiated materials

Non-irradiated 304 steel, 6% strain



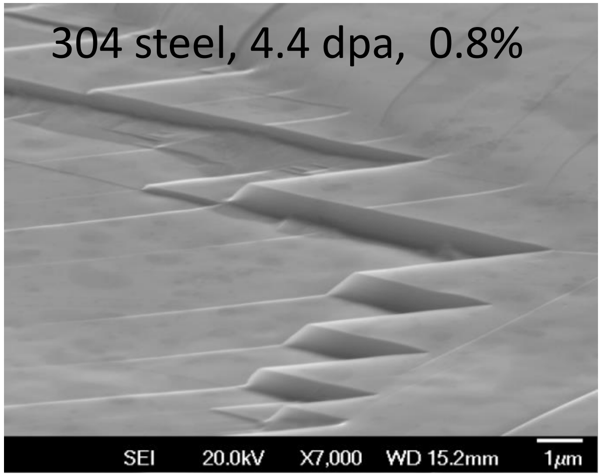
304 steel, 4.4 dpa, 0.8% plastic strain



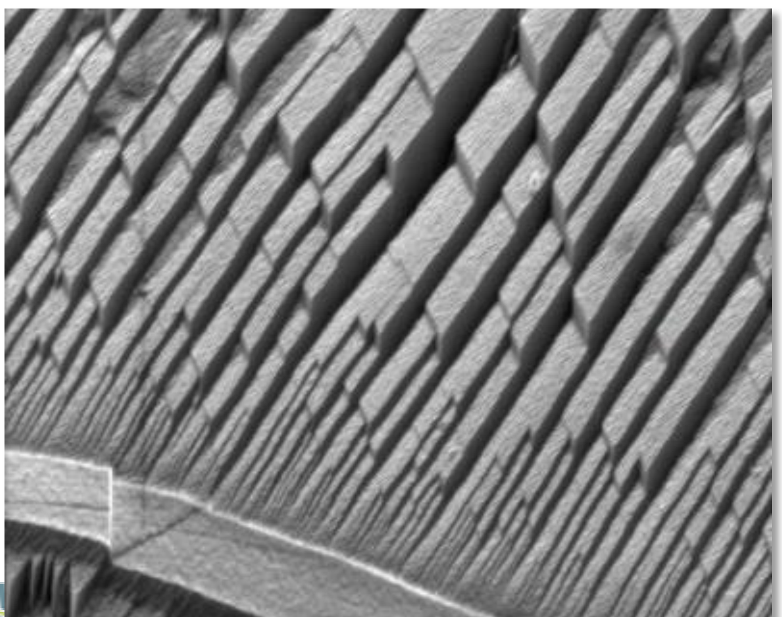
Non-irradiated steel demonstrates numerous, fine slip lines.

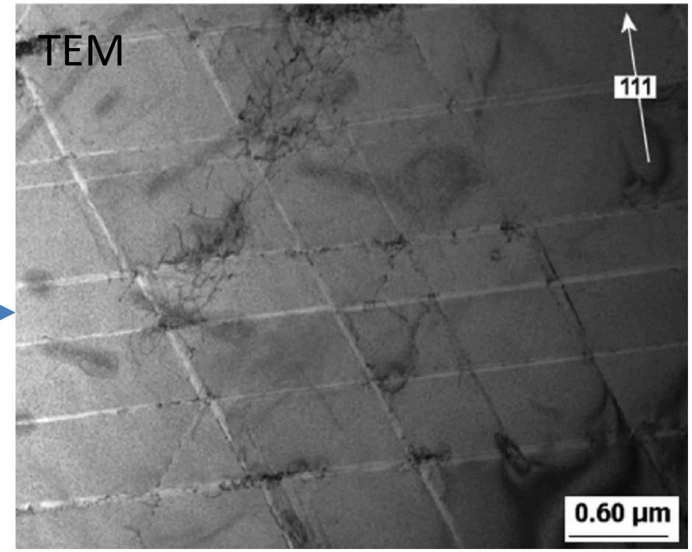
Irradiated and deformed steel has significantly smaller slip line density (orders of magnitude!) and the slip lines are more pronounced; their height at the surface is much higher.

Deformation localization in the irradiated materials (2)



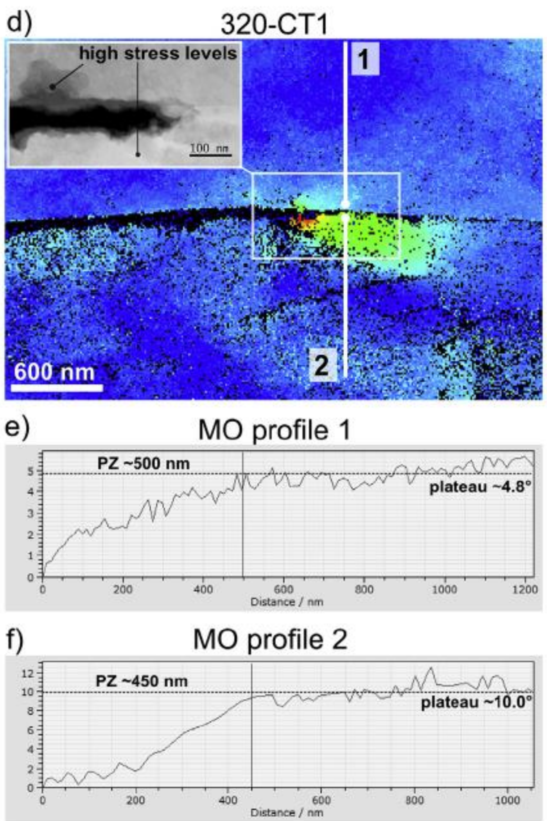
Surface relief in 304 SS, 4.4 dpa, local strain ~0.3





Regular pattern of dislocation channels in irradiated 304L SS (after Sauzay et al., JNM, 2010).

- **Dislocations interact with radiation-induced defects, sweeping them out.**
- **The defect removal process results in a clear, defect-free pathway for the following dislocations (positive feedback loop).**
- **Defect channel interaction demonstrate strong self-organization behavior.**

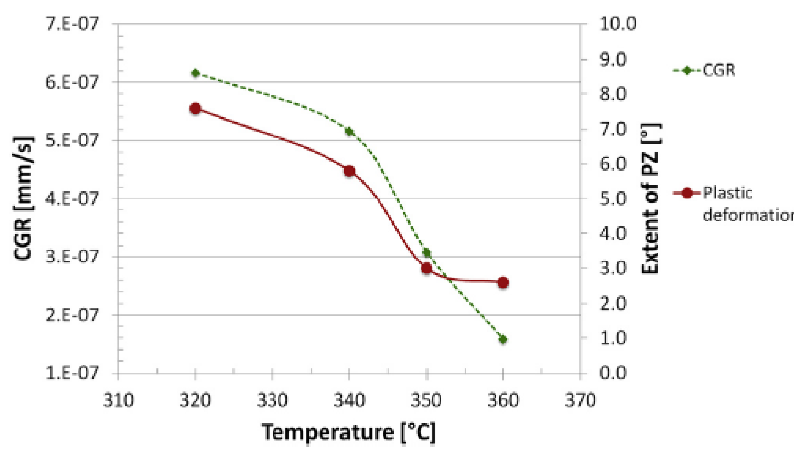


Misorientation map along the crack and near the crack tip (after Meisnar, 2016).

Why deformation localization is important?

Meisnar et al. [Acta Materialia, 2016] investigated crack growth in cold-worked 316L steel; the research was focused on the detailed analysis of crack tip using advanced Transmission Kikuch diffraction (TKD) method allowing for high-resolution analysis of local misorientation level.

In this recently published paper, authors demonstrated **there is a direct correlation between crack growth rate and strain localization** (dislocation density) in the crack tip surrounding area.

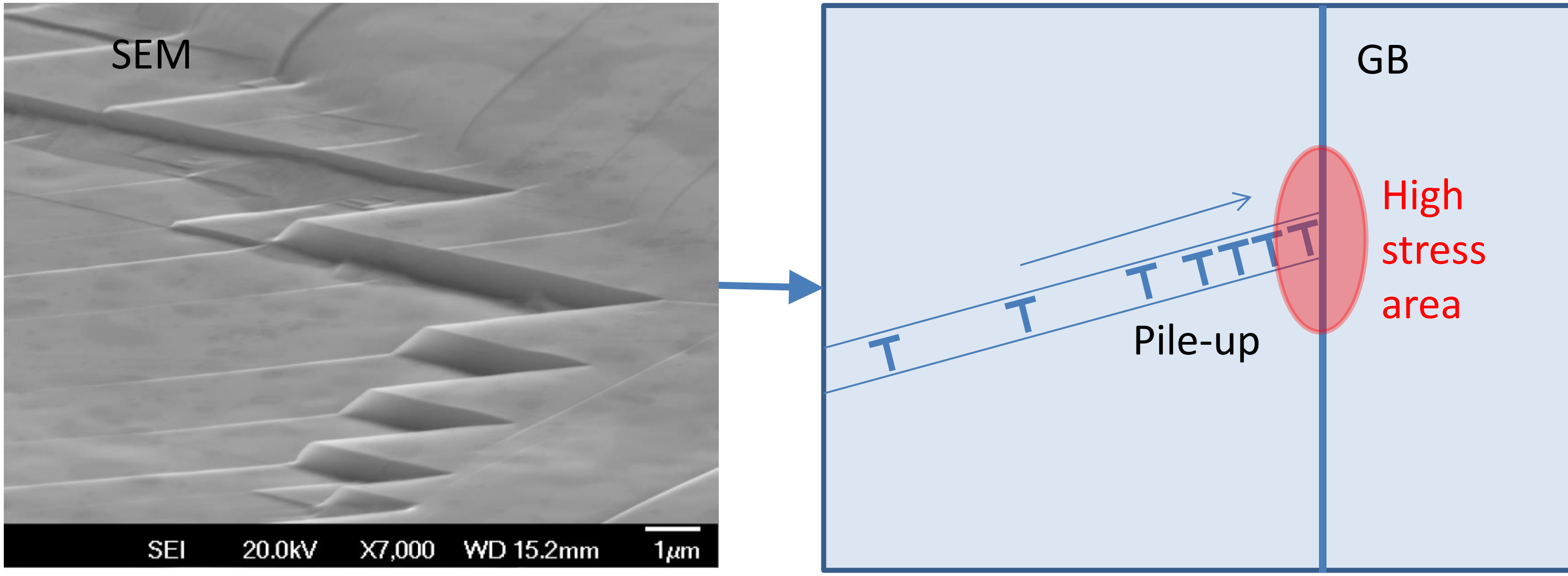


Correlation between deformation localization (expressed in the term of local misorientation) and crack growth rate) (after Meisnar, 2016).

This result raises a number of questions: what about irradiated steel? Is there a way to control the deformation localization at crack tip via alloying? Etc.

Dislocation pile-ups as crack initiation locations

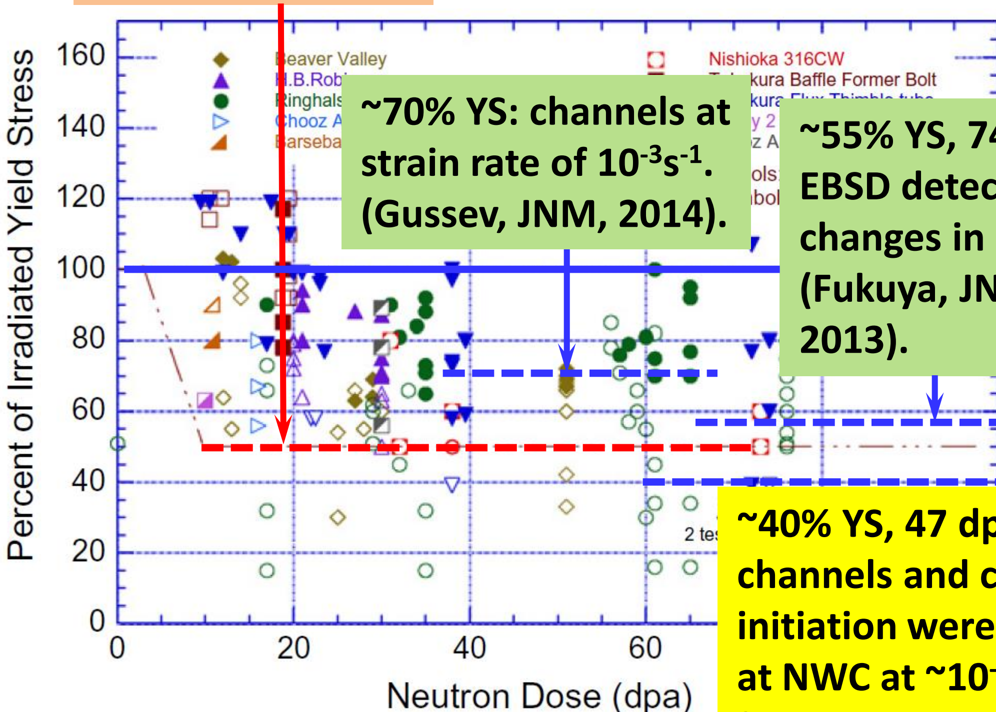
304 steel, 4.4 dpa, 0.8%



- **For bulk material, a simplified classical approach predicts dislocation accumulation at the grain boundary, formation of dislocation pile-up (under certain conditions), and the appearance of area with increased local stress level.**
- **At the surface, channels may lead to protective film rupture and local corrosion and dissolution.**
- **Dislocation pile-up formation leading to the stress concentration is a current working hypothesis for dislocation channel controlled IASCC (G.S. Was et al.).**

An evidence of plastic strain below the formal yield stress

~50% YS as an IASCC threshold



~70% YS: channels at strain rate of $10^{-3}s^{-1}$. (Gussev, JNM, 2014).

~55% YS, 74 dpa: EBSD detected changes in structure (Fukuya, JNM, 2013).

~40% YS, 47 dpa: channels and crack initiation were observed at NWC at $\sim10^{-7}s^{-1}$ (Stephenson, 2015 – Ph.D. thesis).

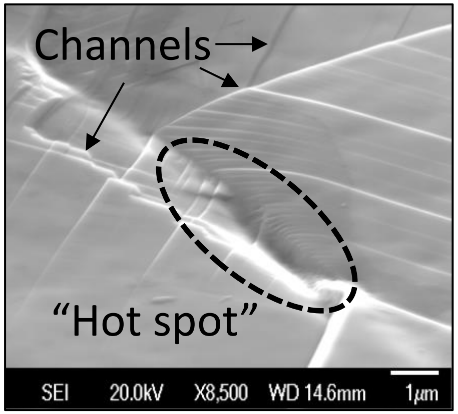
Diagram source: Chopra and Rao, JNM, 2011.

A ~50% of yield stress (YS) is usually accepted as IASCC threshold stress. Such level is usually not associated with the plastic strain presence. However, keeping stress level below the formal engineering YS does not prevent plastic deformation.

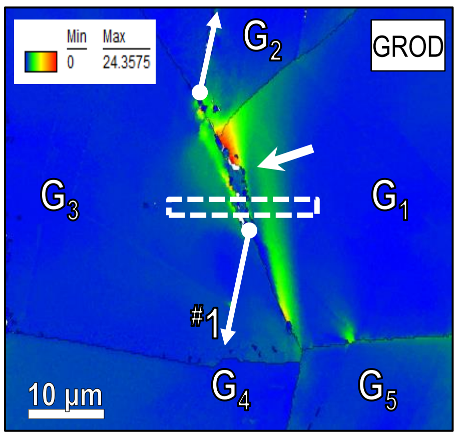
At usual strain rates (**$\sim10^{-3}s^{-1}$**), strain-induced changes in the structure were observed at 55-70% of yield stress.

At low strain rates (**$\sim10^{-7}s^{-1}$**) channels were detected at ~40% YS, and direct correlation between channel and crack initiation site was demonstrated (Was and Stephenson).

Strain localization mechanisms: Deformation "hot spots"

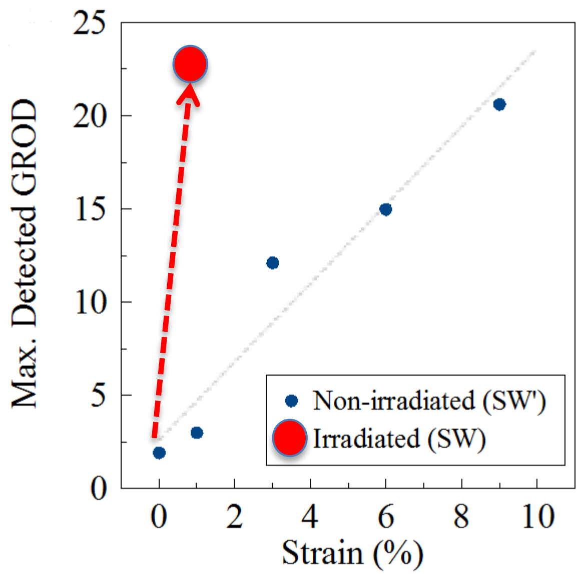


304L steel, 4.4. dpa. SEM image of the area of interest.



GROD map of the area of interest (surface).

Areas of highly-localized deformation were found in 304L SS deformed to **0.8% strain**. The Grain Reference Orientation Deviation (GROD) maps revealed extraordinary high strain-induced misorientation – up to 23°. Thus, these areas were deemed deformation "Hot Spots."



Relationship between the maximum observed GROD value and strain level for non-irradiated and irradiated specimens.

During plastic straining of the irradiated austenitic steel specimens, local in-grain misorientation increased drastically even at small strain level [Field, JNM, 2014]. Thus, "classical defect-free channels" are not a sole contributors to the plastic deformation localization . Other, more complex mechanisms exist.

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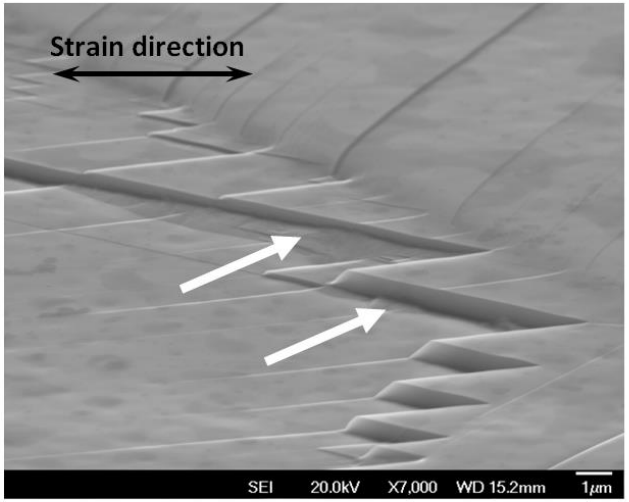
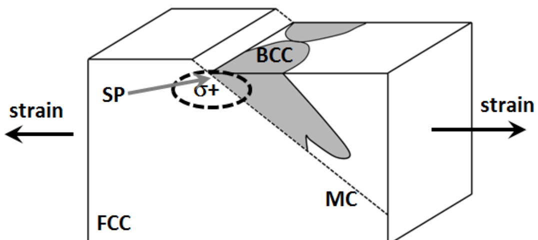
Specific phase instability mechanism

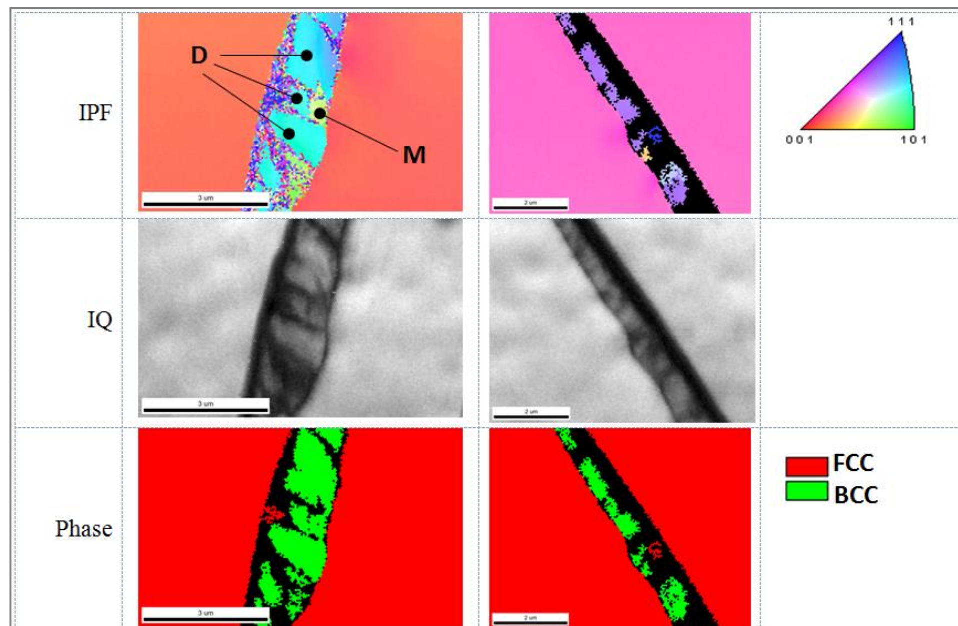


Specific martensitic transformation mechanism was observed at the irradiated specimen surface. Martensitic BCC-domains, identified and confirmed independently by EBSD and TEM, formed along the defect-free channels, if channel heights exceeded ~150 nm.

Dislocation channels at the surface of the deformed specimen, AISI 304 steel (4.4 dpa). The BCC-phase particles are marked with white arrows.



Cross-sectional reconstruction of the BCC-phase domain.



IPF, IQ, and Phase maps for the BCC-domains along the dislocation channel. D,M – martensitic domains.

Shoji et al. Env.Deg.-11, 2003: "…The presence of martensite promoted SCC in both BWR and PWR primary water environments…"

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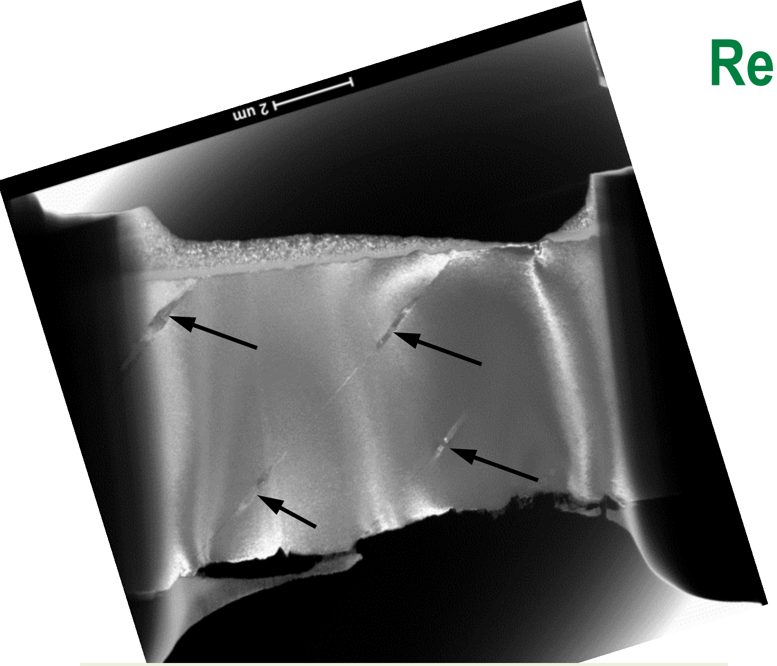
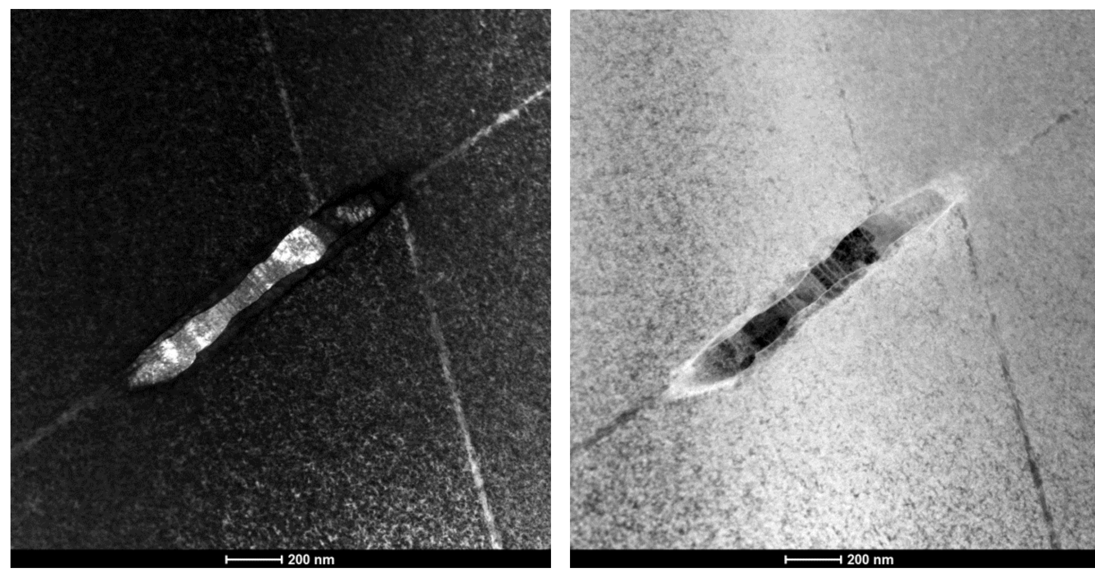
Recent results: deformation localization in 304L steel (10.2 dpa) tested in NWC-environment



General view of the FIB-object structure. One may see several dislocation channels with spacing of 1.5-4 μm. Channel-associated features are marked with arrows.

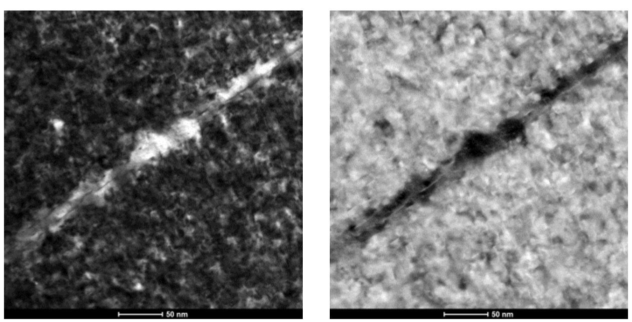
A FIB-liftout was performed at the crack-free location of 10.2 dpa 304L steel specimen. Local strains were small (<1-2%) and the sample was supposed to provide information on the near-surface oxidation processes and the local oxide structure near dislocation channels.

Surprisingly, **specific features were observed at the channels, near the channel intersection points.** The features formed a quasi-regular pattern and some kind of ordered structure.



Specific feature formed at the dislocation channel.

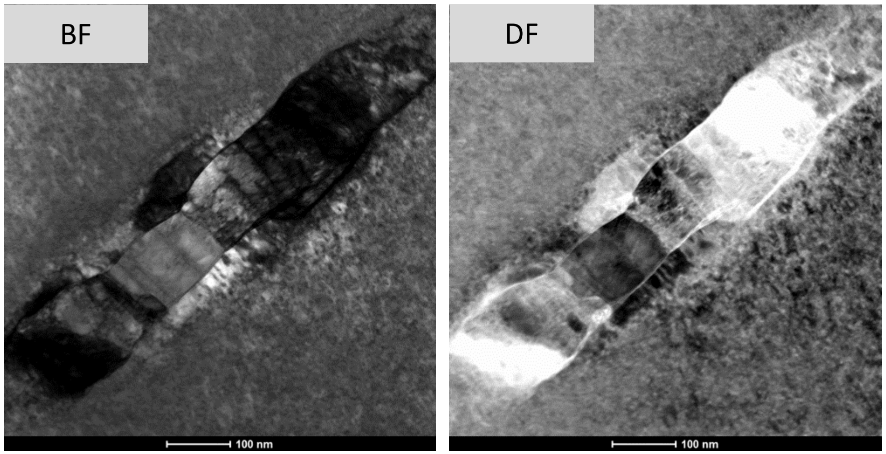
TEM: C. Parish



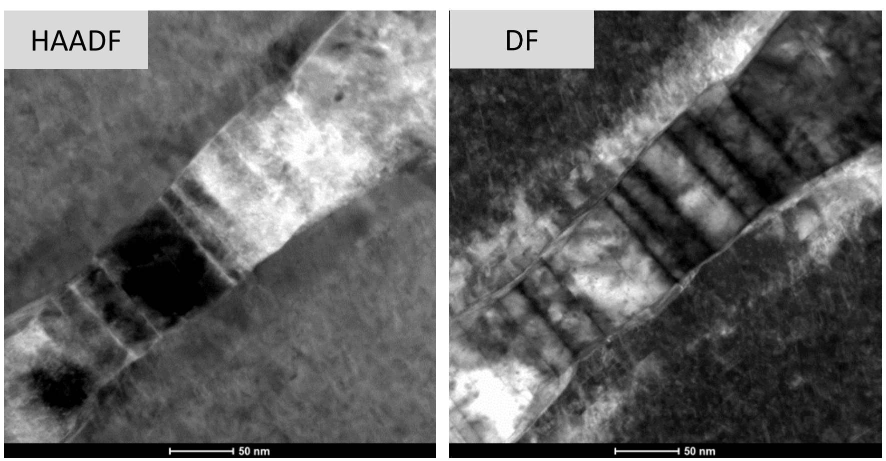
Internal structure of the dislocation channel. No twin was observed in this particular channels.

Specific deformation localization areas formed at small strain in 10.2 dpa specimen

"Inclusion" #1



"Inclusion" #2



Internal structure of two specific features

TEM: C. Parish

The "inclusions" were identified as areas with significant misorientation (several degrees) relative to the parent matrix and high dislocation density.

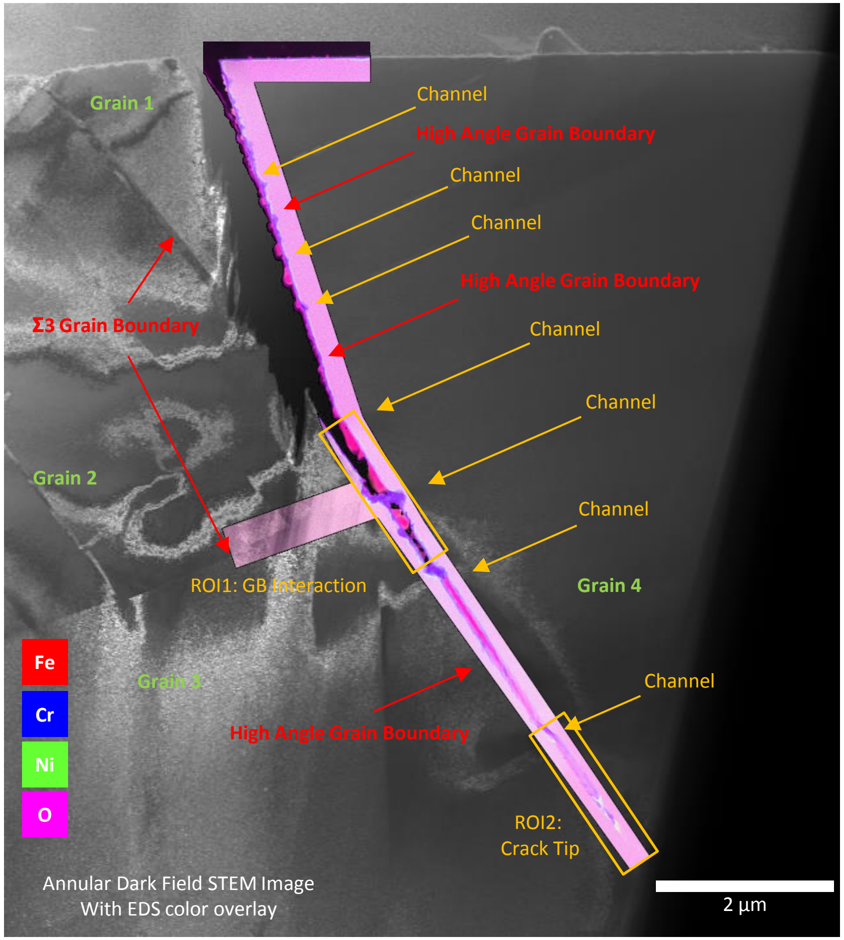
Numerous low-angle dislocation boundaries and complex dislocation structures were observed inside the "inclusions".

It appears, the radiation defect density inside the inclusion was lower compared to the matrix. Also, a shell with decreased defect density existed near the "inclusion".

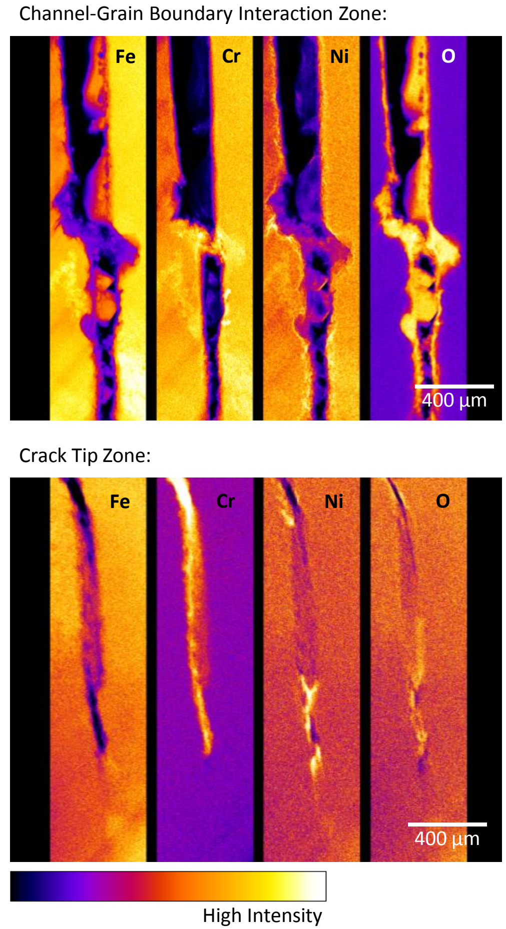
Thus, **instead of small local strain** level (only few channels were observed at the surface), deformation localization processes led to the formation of **high-dislocation density areas** with complex internal structure.

Detailed analysis of oxidation processes inside cracks

Survey Image of Crack-tip



Detailed X-ray Count Maps



- A crack formed in PW-environment was investigated using high-efficiency STEM-EDS.
- Crack was found to propagate along high angle grain boundary pathways.
- Dislocation channels influenced in-crack oxide layer formation and structure.

Outputted data analysis of SW-alloy (commercial AISI 304SS, 4.4 dpa) tested under primary water (PW) conditions using a FEI F200X Talos. Diffraction based contrast is formed using an ADF (annular dark field) image with Fe-Cr-Ni-O color overlay overlapping the crack region, detailed x-ray maps are shown on right. Important features are noted in the image on the left.

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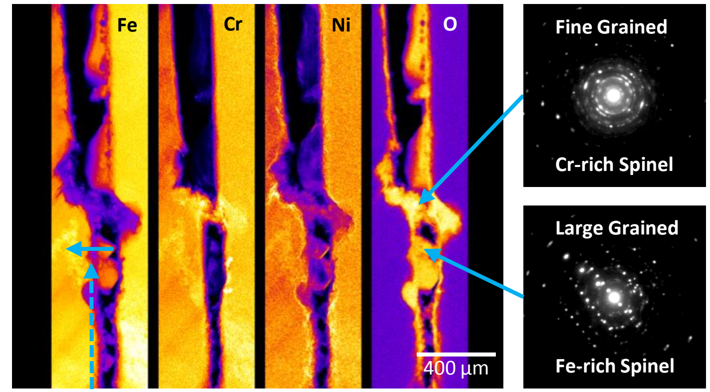
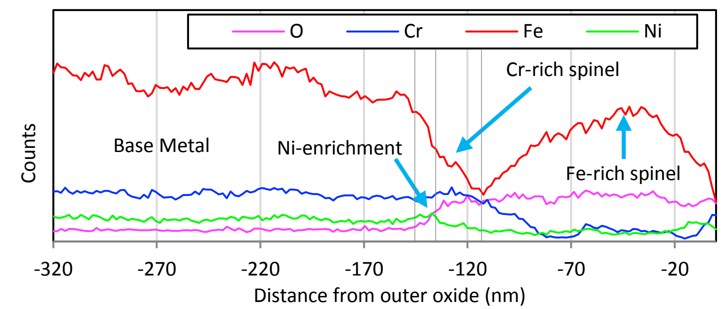
Detailed Oxide Analysis of Crack

- Oxide film had a distinct morphology consistent with literature:
  - Outer layer of larger grained Fe-rich spinel (most likely Fe_3O_4)
  - Fine grained Cr-rich inner spinel layer
  - Ni-enriched metal at base metal-Cr-rich spinel layer
- Oxide grain size and layer thickness larger in highly stressed area including dislocation channels and grain boundary interaction zones
- Crack-tip was primarily filled with Cr-rich oxide
- Ahead of crack-tip was a Ni-metal enriched pocket

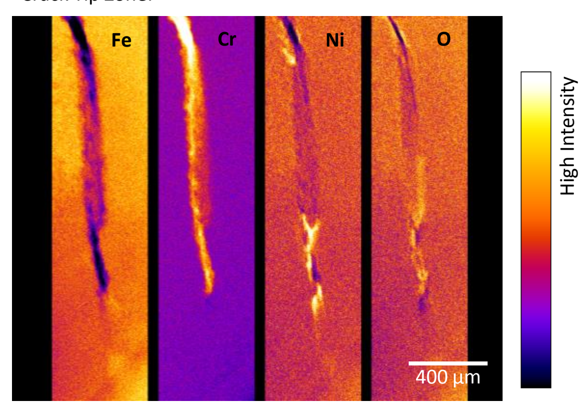
Channel-Grain Boundary Interaction Zone:



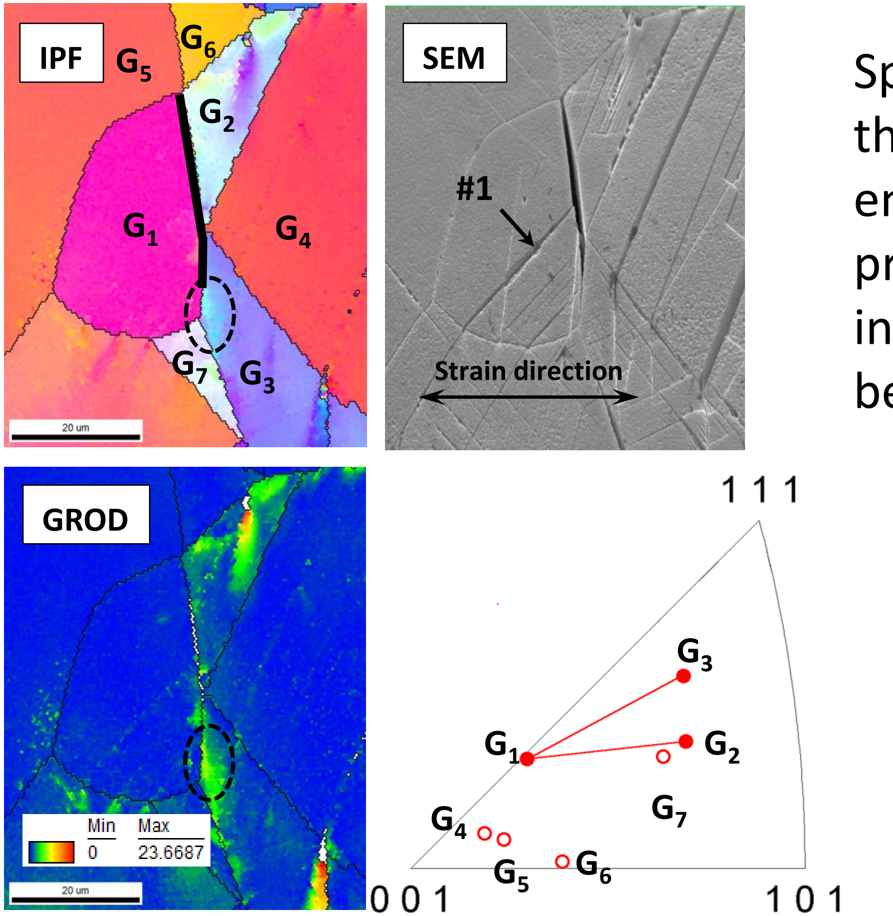
Line Profile in GB Interaction Zone:



Crack Tip Zone:

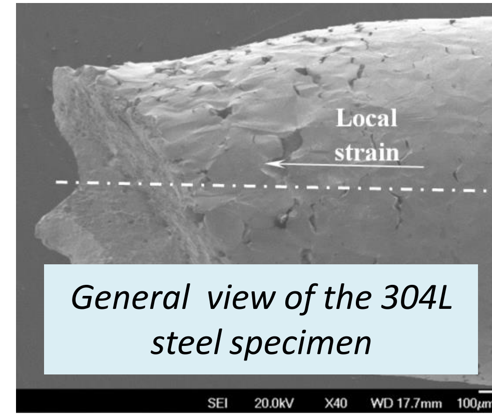


Grain orientation role on crack initiation in 304L steel



EBSD-data for a typical crack. The unit triangle at the right shows the orientation of G_1-G_7 grains in the crack vicinity (closed symbols: grains adjacent the crack; open symbols: grains not involved in cracking).

Specimens, tested in the corrosion environment, may provide further insight on the SCC behavior.



General view of the 304L steel specimen

Cracks were analyzed in detail using SEM-EBSD to retrieve the orientation of crack-adjacent and neighboring grains. It gives an opportunity to investigate crack initiation in detail.

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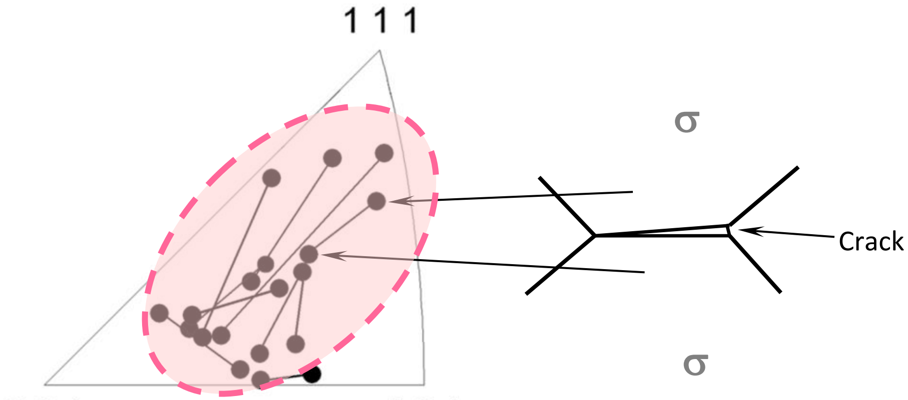
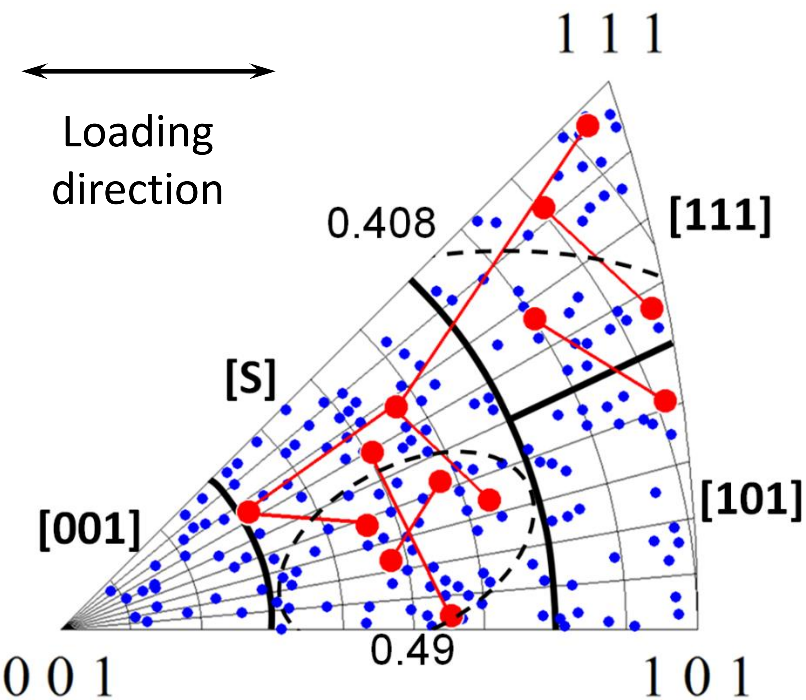
Orientation patterns for the crack-adjacent grains (304L steel)



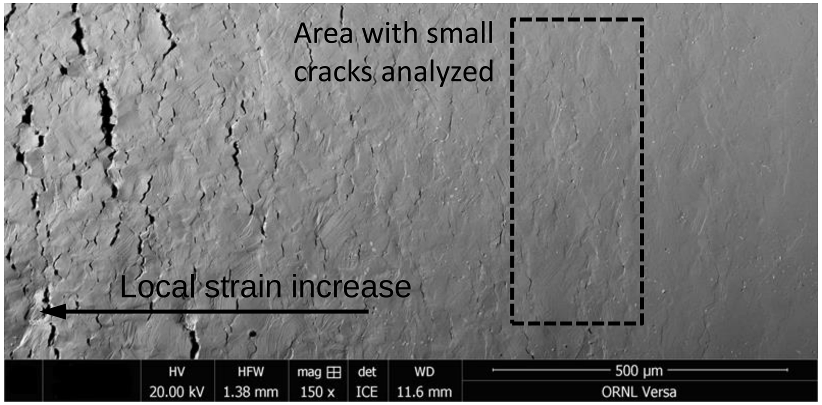
Short cracks (1-2 GBs)



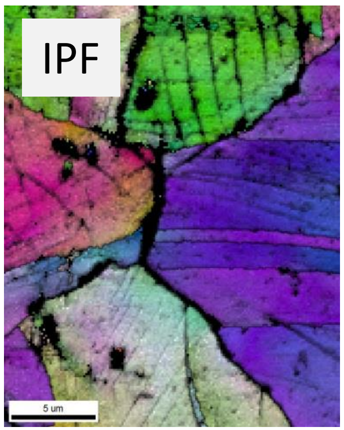
- **Crack initiation is not a random process.** Grain orientation strongly influenced the crack initiation.

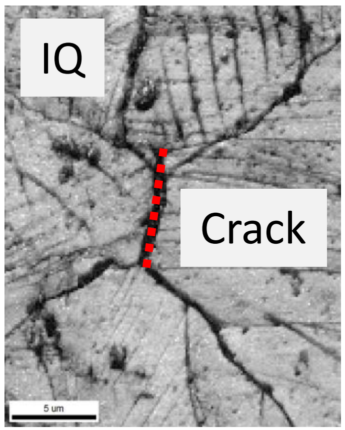
- For short cracks, one or both grains adjacent to the cracked GB were often oriented close to the center of the unit triangle (softest grains) or close to the [111]-corner. Grain orientation role may become less pronounced for longer (or deeper) cracks (this is something we are investigating now).

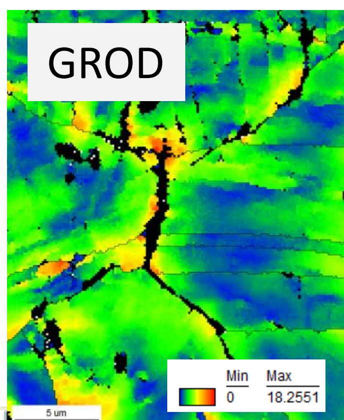
FY2017: Grain orientation role on crack initiation in model alloy

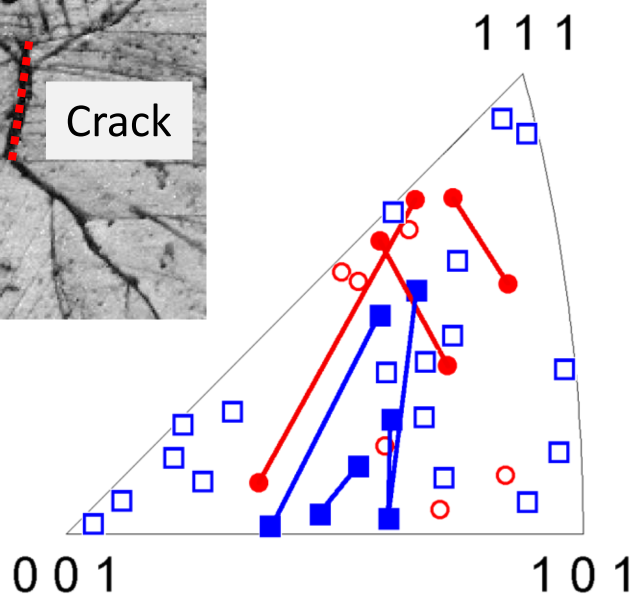


SEM-image showing a gradient in crack density.









Typical EBSD maps (left) and IPF plot showing the orientation of crack-adjacent grains (closed symbols connected by line) and non-cracked (open symbols). The plot shows data for two scanned cracks (circles and squares, respectively).

Crack initiation was investigated in the model alloy (304L+Mo+Hf, ~316L composition, 9.6 dpa) tested in the primary water (PW) environment. It was important to analyze a material with higher stacking fault energy and compare the results to the ordinary 304L steel (A, SW-alloys investigated earlier).

It was shown that most grains involved in the crack initiation were oriented close to the unit triangle (i.e. were the softest grains); many grains had also orientation close to [111]. [001] and [101]-grains were relatively stable.

The observed crack initiation behavior pattern is close to the 304L steel heats investigated earlier (A- and SW-alloys).

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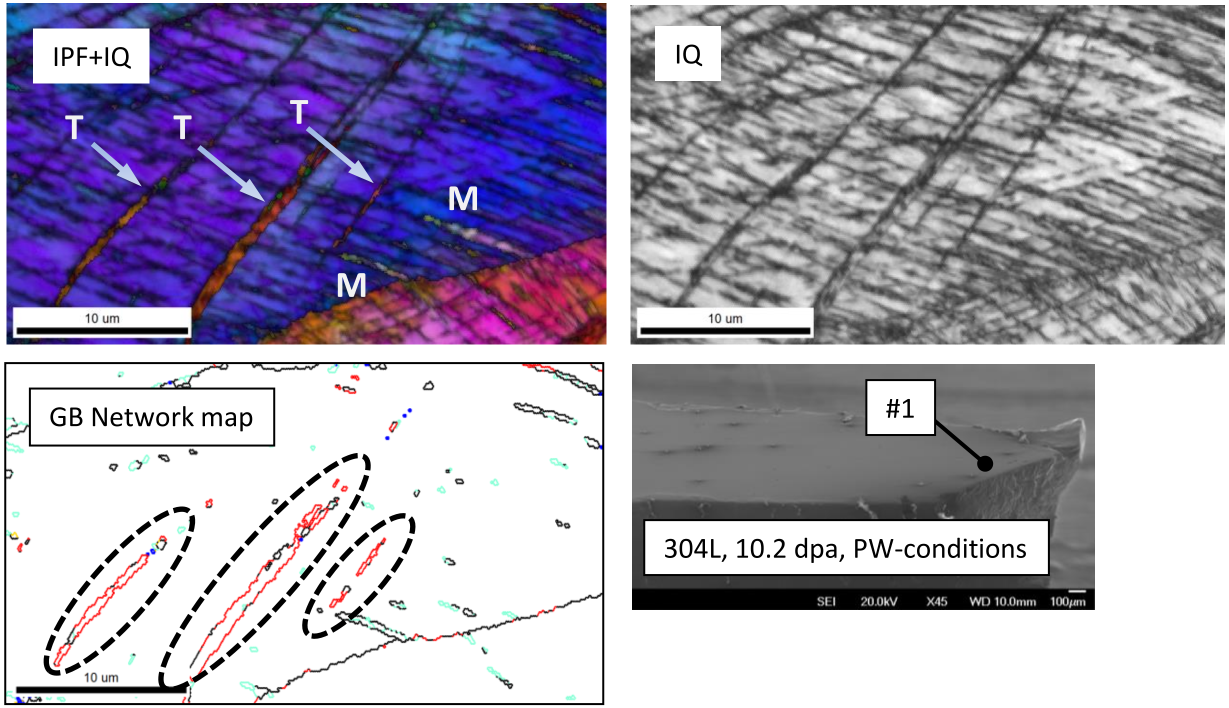
Deformation mechanisms in 304L steel strained in high-temperature water



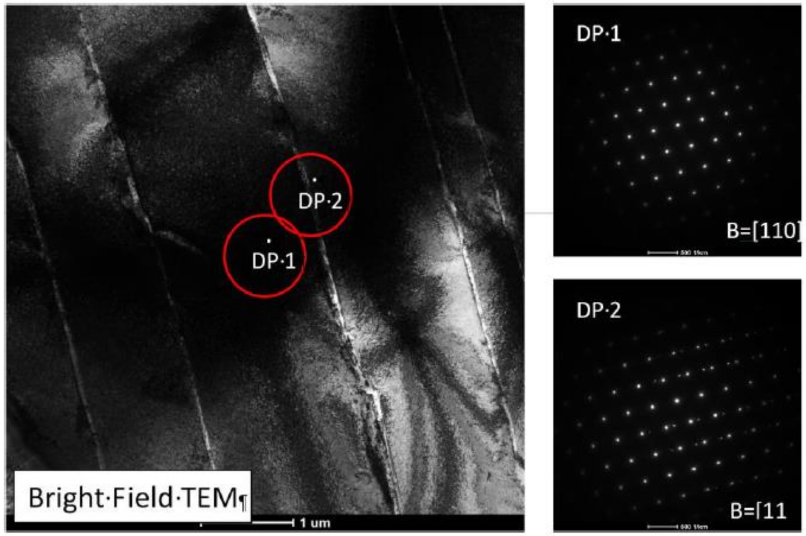
EBSD IPF, IQ, and grain boundary network maps) for the location close to the fracture point (#1). Deformation twins are shown by arrows in the IPF map and dashed ovals in the GB map. M – an area with high local misorientation (not twins). In the GB map, black color represents random high-angle GBs, red – twin boundaries, and teal – random low-angle grain boundaries.

- Early it was shown (G.Was et al.) that deformation localization and channel formation promote stress corrosion cracking.
- Deformation localization in the irradiated steel is an complex process.
- It was established that deformation twinning is an active deformation mechanism during straining in high-temperature water.
- Long (~10-20 μm) and relatively wide (1-3 μm) twins formed close to the fracture point.

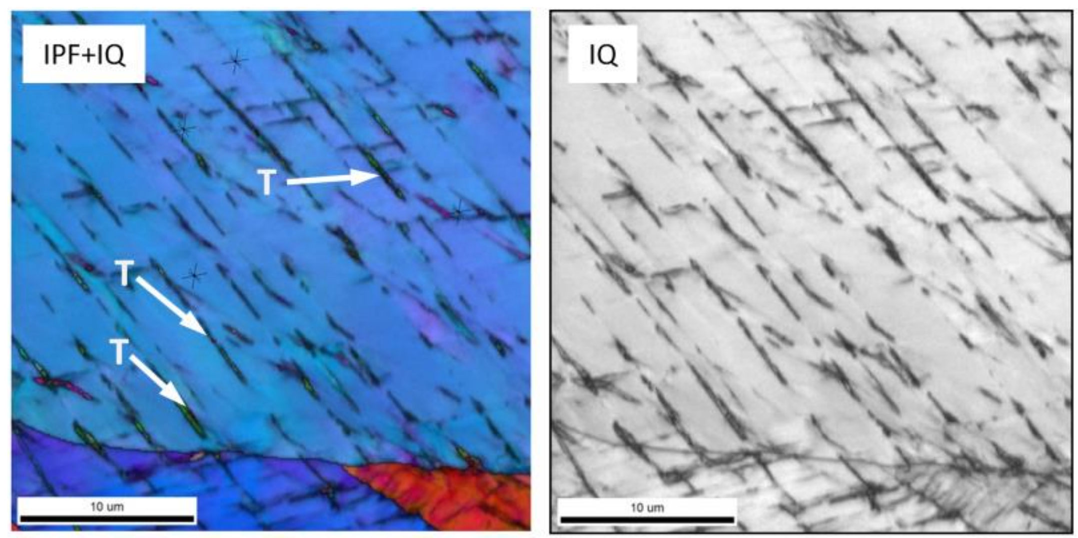
Twinning as an active deformation mechanism under LWR-conditions.

- Unexpectedly, an evidence of deformation twinning in specimens deformed in high-temperature water was found. Fine twins inside the dislocation channels were observed via EBSD and after that by FIB-TEM analysis.

- The presence of twin inside the channel may increase the stress concentration degree at dislocation pile-up and promote cracking.



Signs of deformation twins inside the channel; specimen was CERT-tested in high-temperature water (K.Field & M.Gussev, in preparation).



Fine EBSD-resolvable twins in 304L-specimen deformed in high-temperature water.

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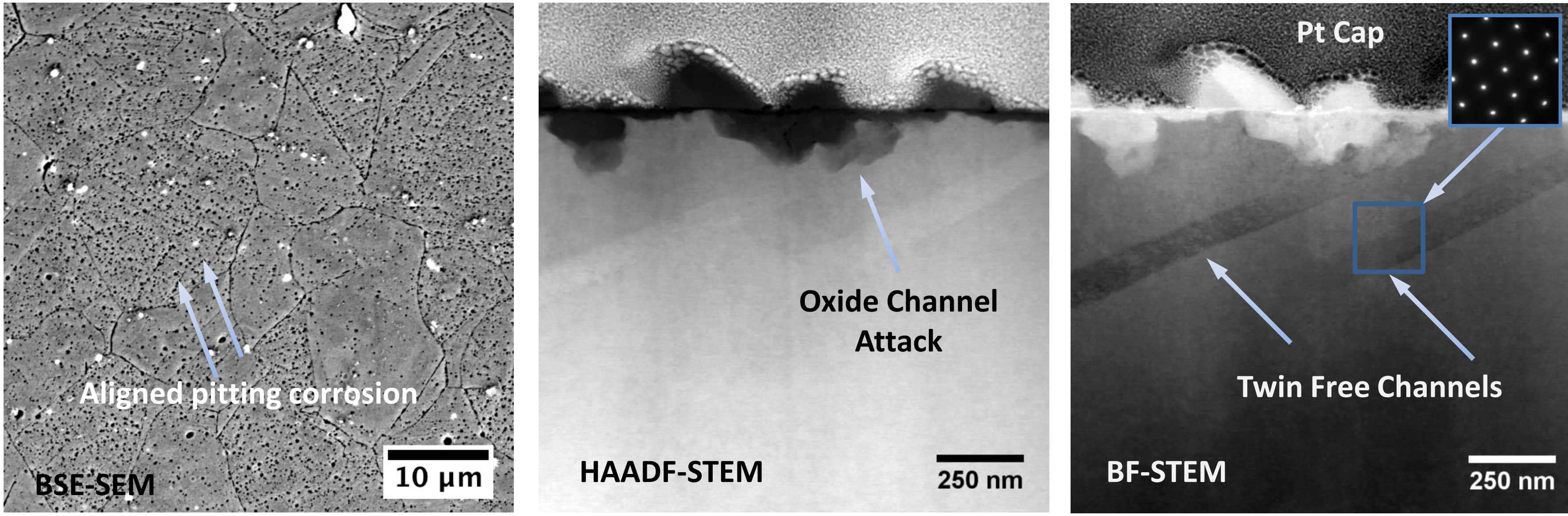
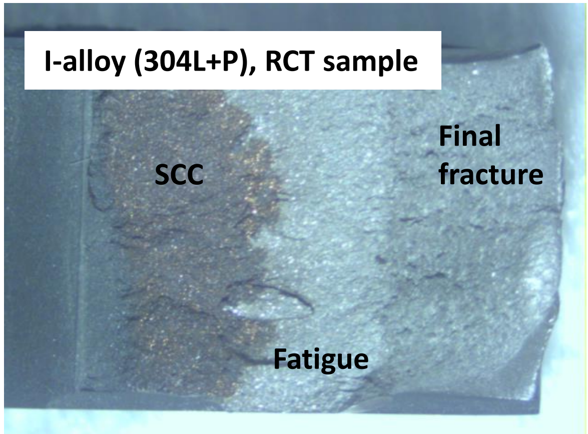
Current activity: localized corrosion might be a non-random process in high-temperature water

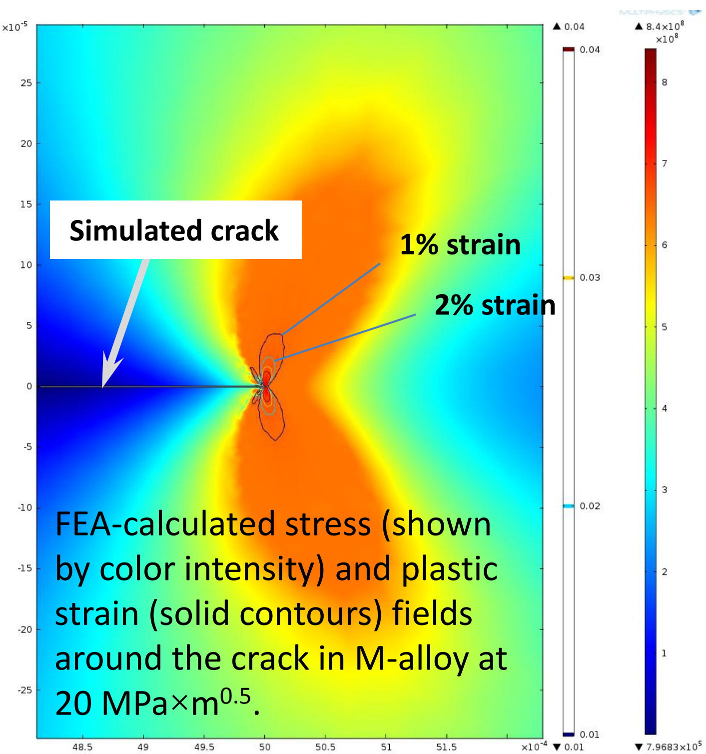


- Oxide attack at the surface and near surface was observed using SEM and TEM on deformed P-alloy exposed to high-temperature water

SEM and TEM show pitting to be non-random in the specimen, oxide attack was observed to be aligned on dislocation channels. Attack occurred to a depth of ~250 nm; micro-pit density varied in different grains.

Current activity: stress and strain analysis in deep stress corrosion cracks

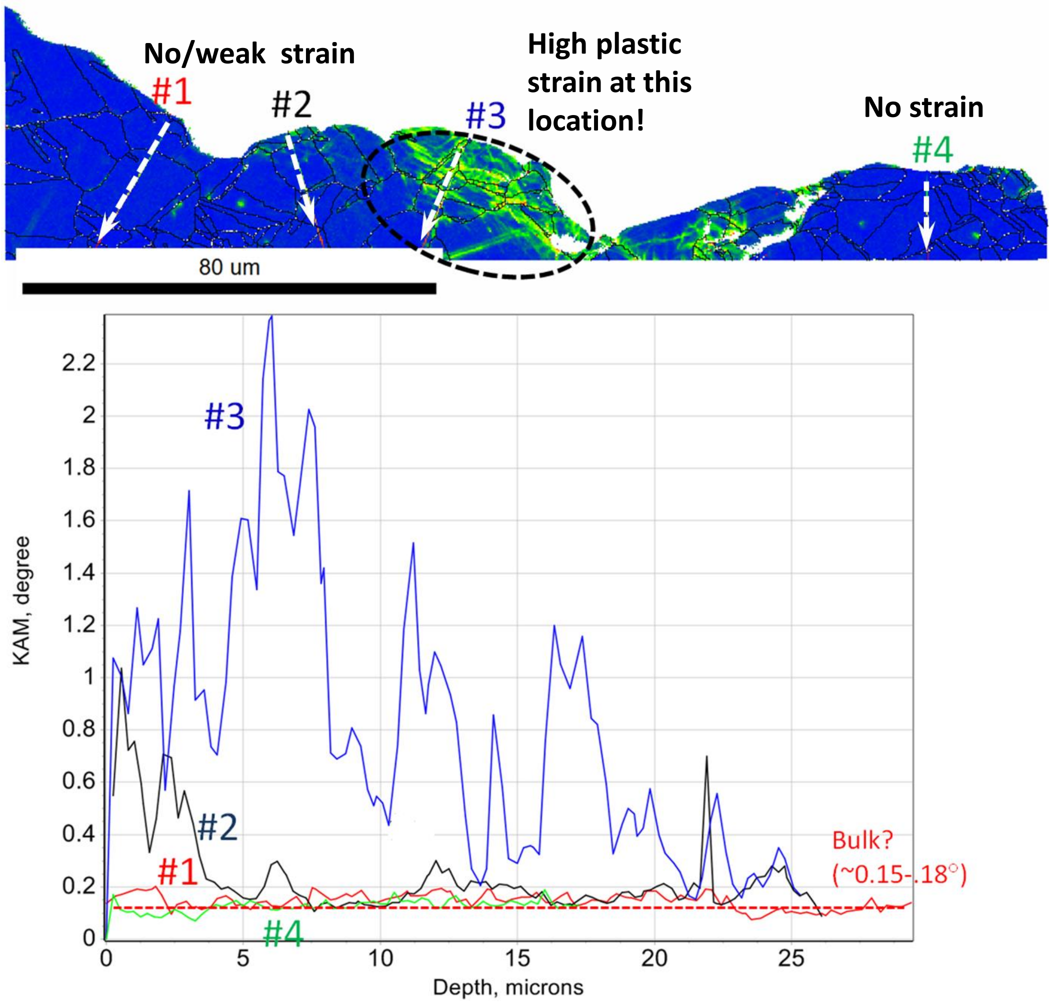




- Round compact tension specimens (RCT) are available for several 304-based model alloys. The crack growth tests were conducted under well-controlled environment parameters and stress intensity factor.
- It was important to investigate plastic strain mechanisms acting during crack propagation.
- Methods: SEM/FIB/EBSD, TEM, Finite Element Analysis (FEA).

As was expected, stress concentration near the growing crack should lead to significant plastic strains in the crack-adjacent grains. The width of the area strained at ~2% (EBSD-detectable strain level) was estimated to be ~20 μm micron on each crack side.

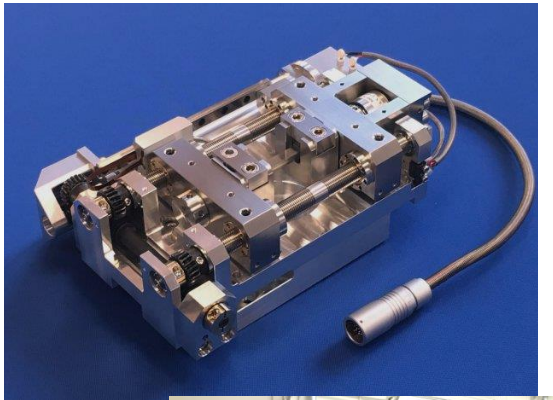
Current activity: EBSD analysis of plastic strain around the deep stress corrosion crack

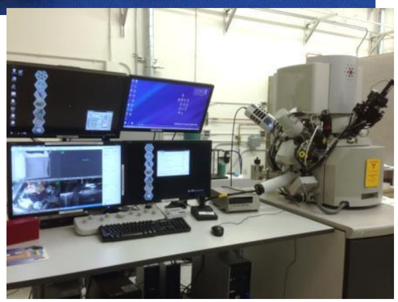


Kernel misorientation distribution at the crack edge. The diagrams show the detailed KAM profiles (#1-#4) taken at different locations.

- Plastic strain distribution was found to be strongly inhomogeneous.
- "Classical" predictive model appeared to have limited capability.
- In the crack vicinity (<20-30 μm), many grains were practically strain-free, some grains experienced strong plastic strain.
- Plastic strain may reflect the nature of grain boundaries passed by crack; most likely, more resistant boundaries led to larger strain in the crack-adjacent grains.

Developing new LWRS-specific techniques/tools: In-situ mechanical testing?





VERSA 3D: Now: SEM+ EDS+ EBSD+ FIB. In the nearest feature: In situ testing capability.

- Plastic strain and deformation localization appeared to be an important contributors to IASCC.
- ORNL currently has an impressive combination of research capabilities: hot cells, LAMDA, FIB-SEMs, High resolution TEM, EDS, EBSD.
- **In-situ testing looks as a powerful addition to the tools and methods we have now.**
- Miniature 5kN tensile frame is being purchased (collaborative efforts with other programs). The device will provide in-SEM mechanical testing (compression, tensile, fracture tests).

Advantages:

- In-situ SEM-EBSD analysis of misorientation evolution and strain localization.
- Employing of the EBSD-based approach to retrieve dislocation density.
- Direct measurement of acting stresses via EBSD pattern analysis.

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Brief discussion of future work

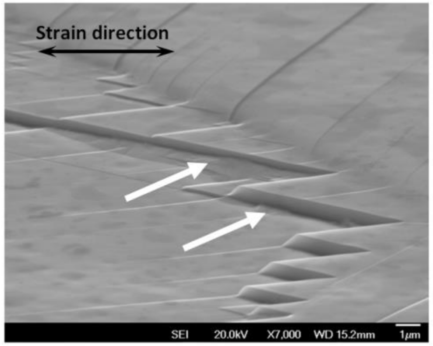
1. **Investigation of deformation processes and mechanisms accompanying stress corrosion crack propagation.** Detailed analysis (EBSD, FIB-TEM) of plastic strain fields around the cracks. Analysis of the available CT-specimens (model austenitic alloys).

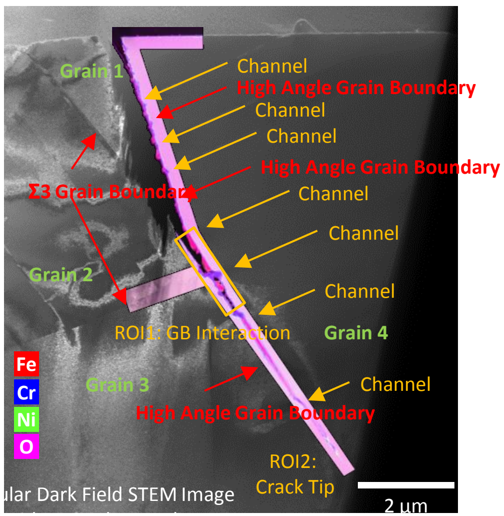
2. **In-situ testing of the irradiated specimens** (in-SEM tensile frame with heating stage option). This research direction will consist of several steps:

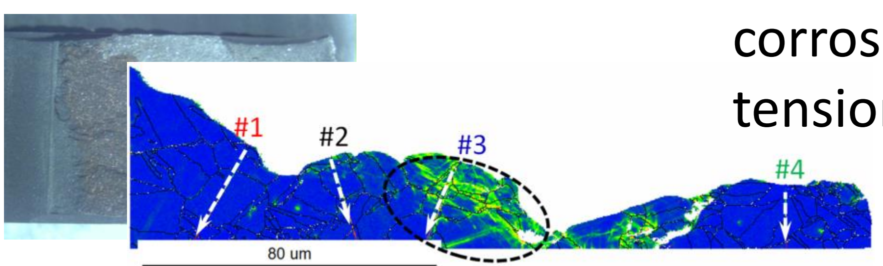
- In-situ SEM-EBSD analysis of misorientation evolution and strain localization.
- Employing of the EBSD-based approach (HR-EBSD) to retrieve dislocation density. Direct measurement of acting stresses via EBSD pattern analysis.
- Additional measurement techniques? (in-situ testing at Spallation Neutron Source (SNS)? TBD).

3. **Harvesting irradiated materials**.

Summary: deformation localization studies in the framework of the LWRS-program.







- ...-FY2013: Mechanical tests at room temperature (RT); dislocation channel dynamics, deformation hot spot observations; phase instability and twinning at RT.
- 2014-2015: Analysis of deformation mechanisms during straining in high-temperature water (at the LWR-relevant conditions); crack initiation analysis; detailed analysis of in-crack processes.
- FY2016-... : Plastic strain accompanying stress corrosion crack propagation (round compact tension specimens).

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