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Microstructural effects on stress corrosion crack initiation in austenitic stainless steels in PWR primary environments

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Background

- Stress Corrosion Cracking (SCC) has significant detrimental implications with respect to nuclear safety and structural integrity of nuclear reactor plant and been one of the main problems affecting plant availability in nuclear power generation
- Service experience of stainless steel in PWR primary circuit has been generally good.
- Limited instances of SCC have been associated with non-free flowing occluded regions.
- However, a small number of occurrences of SCC have been reported in flowing coolant. The majority of the reported cracks were associated with high levels of surface and /or bulk cold work.
  - Laboratory tests indicate that >15% cold work is required to propagate SCC in austenitic stainless steels.
- Cold work can arise from a variety of sources such as fabrication, installation, repair procedures etc.
- Based on previous studies, crack initiation appears to be difficult under constant load conditions.
  - Severe cold working conditions and/or dynamic loading appears to be required for initiation.

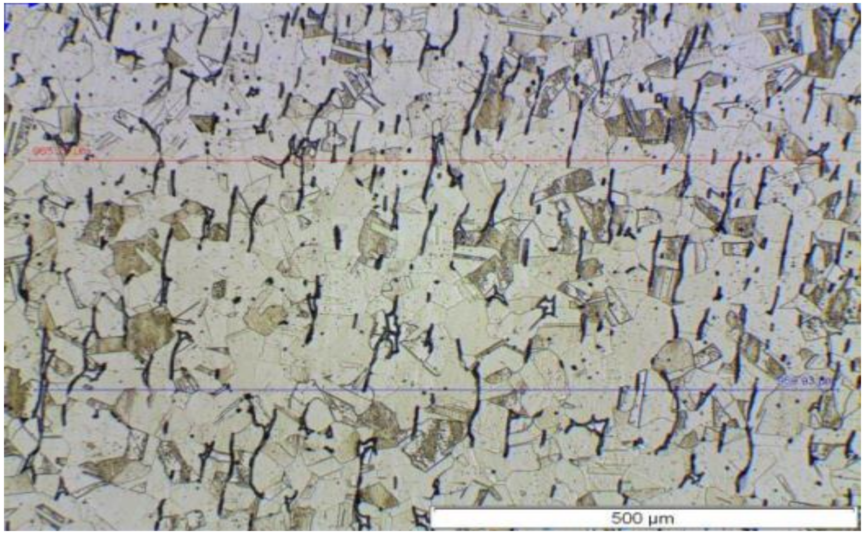
Objectives

- The purpose of the current study is to improve the mechanistic understanding of conditions under which SCC could initiate in austenitic stainless steels, at the microstructure level, in good quality PWR primary coolant, and how small initiated defects could develop into propagating stress corrosion cracks.
- The focus of the project is to address a number of gaps in the current knowledge in SCC initiation and propagation including:
  - Influence of material composition and microstructure.
  - Influence of surface condition.
  - Influence of loading conditions.

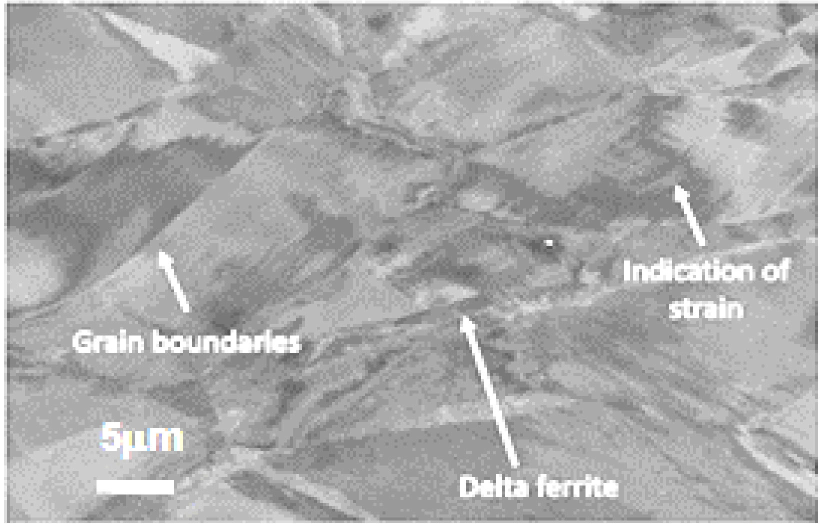
Material

Summary of Material Properties								
Material grade	Type 304L							
As-received condition	Solution annealed plate (70mm thick)							
Chemical Composition	**C**	**Mn**	**P**	**S**	**Si**	**Ni**	**Cr**	**N**
	0.022	1.86	0.023	0.004	0.36	7.98	18.34	0.068
Grain size	20 – 35 µm							
Delta ferrite	4 – 5%							
Inclusion content	Thin : A - 0.5(3.7mm) ; B - 0.5(1.7mm); C - 0.5(1.8mm)							
Hardness	**As received**				**Warm worked (Forged)**			
	148 – 163 HV20				240 – 260 HV30			

Microstructure



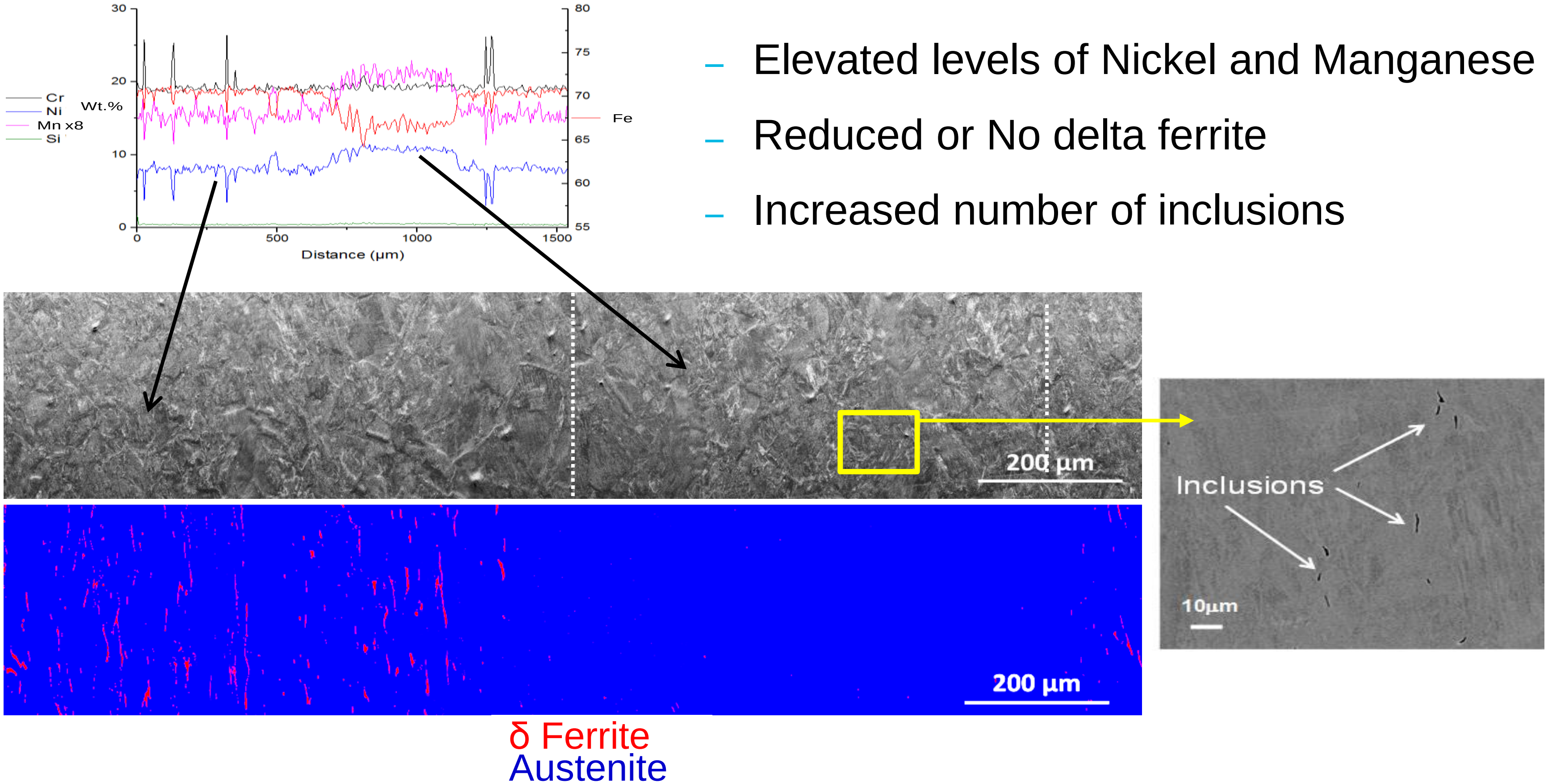
Microstructure: As received
35µm grain size
~4.5% δ-ferrite



Warm forged specimen

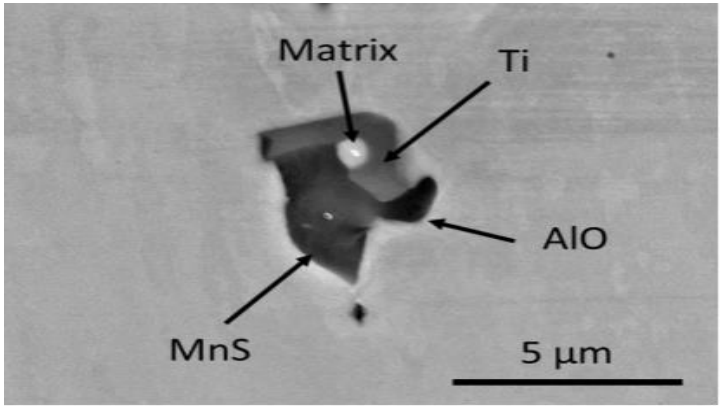
Microstructure Variability (1)

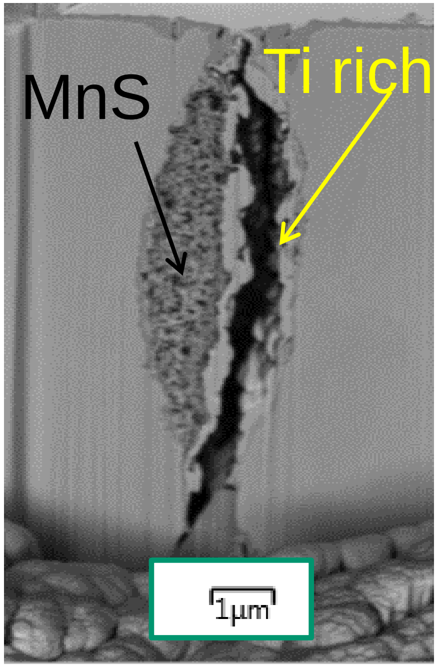
- Distinct bands visible on specimen surface with…
  - Elevated levels of Nickel and Manganese
  - Reduced or No delta ferrite
  - Increased number of inclusions



Microstructure Variability (2)

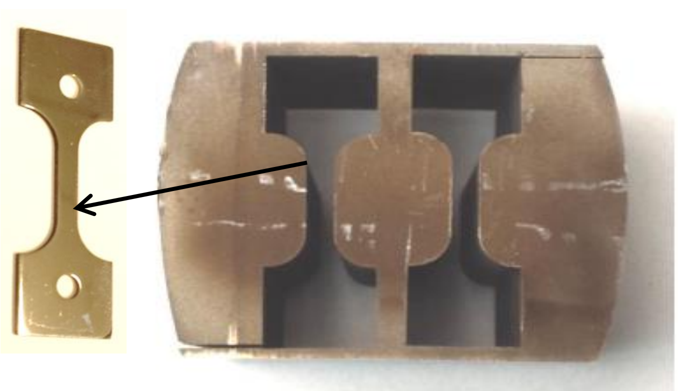
- Inclusions, most numerous in the banded region, are complex in composition.

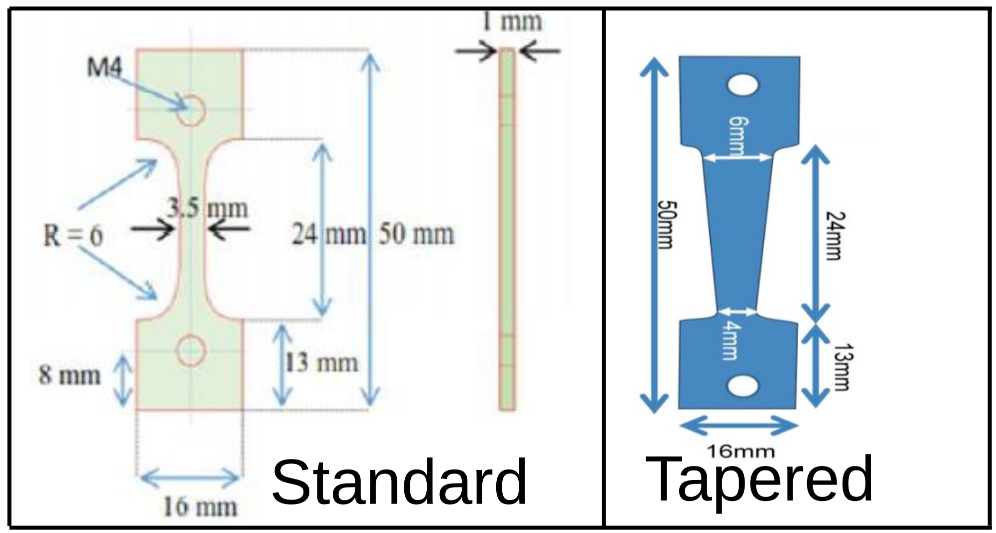




Material Condition and Sample Geometry

- The material was worked to nominal 20% thickness reduction using warm forging at ~300°C to allow testing in the S-L orientation and to simulate weld HAZ conditions.
  - Flat Tensile specimens (standard and tapered in width)
  - OPS (~0.5µm) polished on one side and 600 grit (~25µm) ground finish on the opposite sides.





Test Conditions

- Tests were performed in simulated PWR primary coolant conditions.
- A number of tests performed under mainly slow strain rate loading.
- Supporting program of oxidation exposure tests including some plant relevant constant loading tests addressing possible precursors to SCC.

Testing: Slow Strain Rate Tests (SSRT)

- A number of SSR tests performed with varying parameters such as surface finish, strain rate, total plastic strain, sample geometry, surface condition etc.

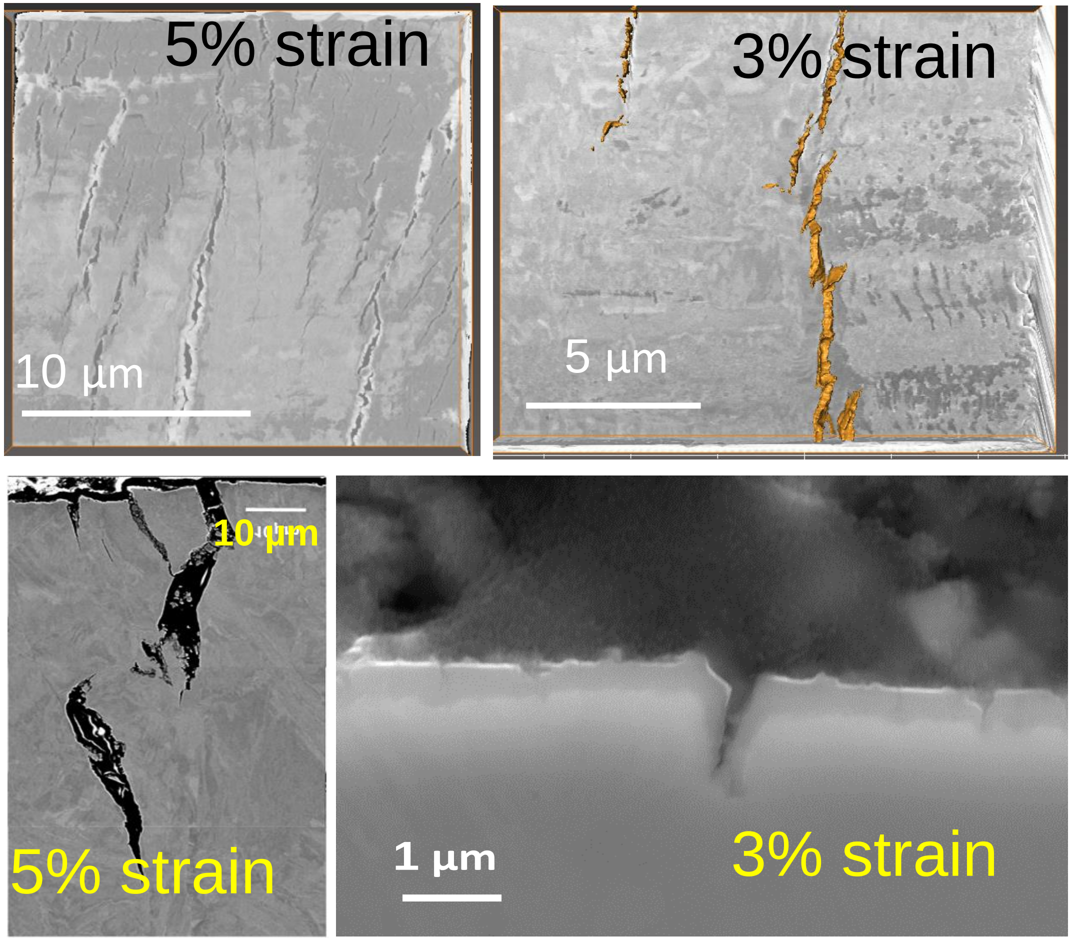
- Samples initially strained at 10^{-6} s^{-1} close to yield for each SSRT test, followed by...

SSRT Test	Additional information	Strain rate	Total strain	Duration
Test 1	Single specimen- standard geometry	2.5×10^{-8} s^{-1}	5% strain	500 hrs.
Test 2	Single specimen- standard geometry	10^{-8} s^{-1}	3% strain	900 hrs.
Test 3	Single specimen- standard geometry	10^{-8} s^{-1}	Rig Failure- test cancelled.	-
Test 4a	Single specimen- standard geometry	10^{-8} s^{-1}	1% pre-strain + 3% strain	525 hrs.
Test 4b	(Retest – Test 4a)	2×10^{-8} s^{-1}	~6% total strain	340 hrs.
Test 5	Single specimen – tapered geometry -2.5mm thick	$2 \times 10^{-8} s^{-1}$	~ max 14% at narrowest point	600 hrs.
Test 6	5 specimens in chain – standard geometry- machined surface condition	$2 \times 10^{-8} s^{-1}$ followed by $1 \times 10^{-7} s^{-1}$	~11% and one sample fractured	470 hrs.
Test 7	Single specimen – standard geometry -Carbon coated	2×10^{-8} s^{-1}	5 % strain	750 hrs.
Test 8	Single specimen- tapered geometry - Higher strain rate	10^{-7} s^{-1}	~ max 14%	400 hrs.
Test 9	Single specimen – standard geometry - Inert test conditions (5%H_2 – 95%N_2)	2×10^{-8} s^{-1}	5% strain	400 hrs.

Key observations

- Effect of Applied Strain
- Crack morphology
- Surface condition
- Oxidation Behaviour
- Microstructure effects
- Effect of Cold work Vs. Warm work

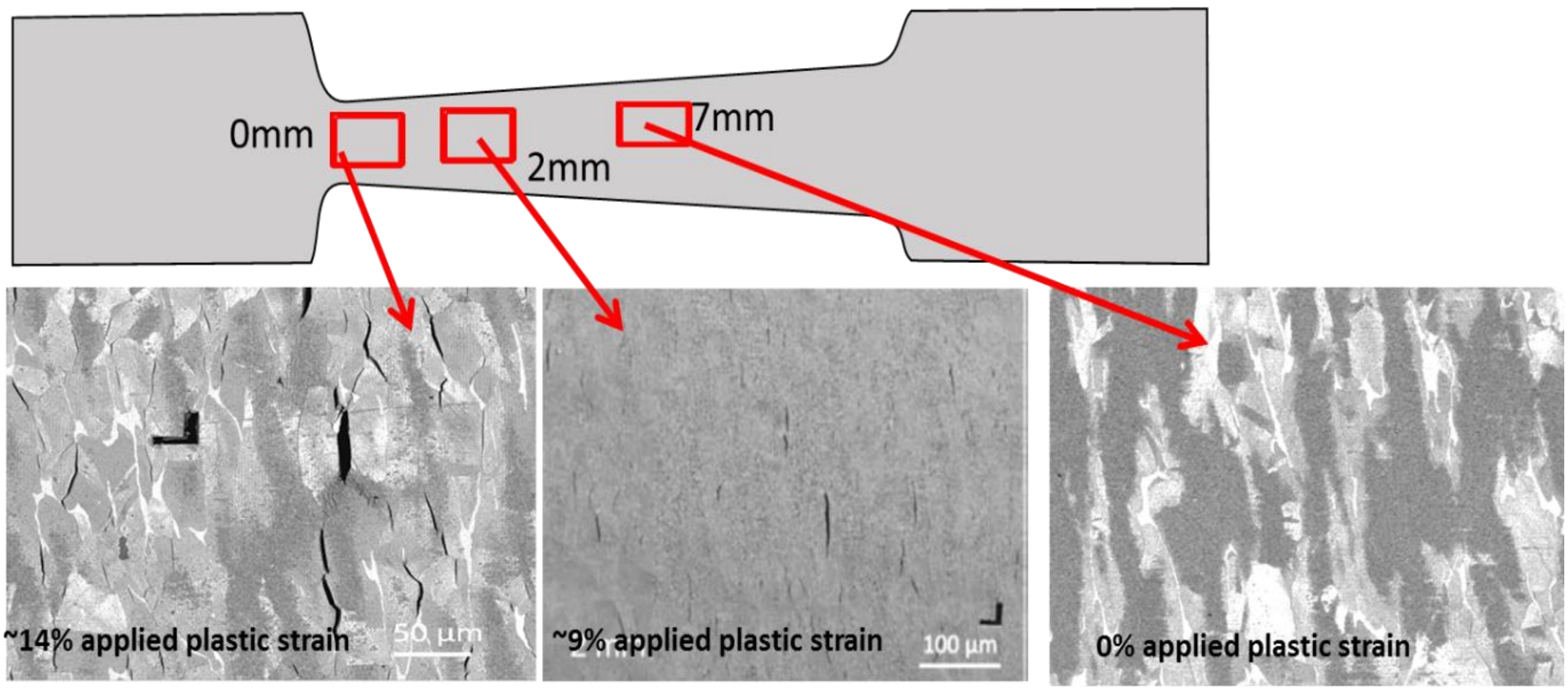
Effect of Applied Strain



- Significant influence of level of applied strain on SCC susceptibility.
- At least 5% plastic strain is required to produce substantial cracking, although some cracking can initiate at ~3% strain (for the strain rates applied).
- The density, length and depth of cracking increases substantially between 3% and 5% applied plastic strain.
- Majority of cracks noted in sample with 3% applied plastic strain were limited to surface oxide. Nevertheless few cracks penetrated into the metal.

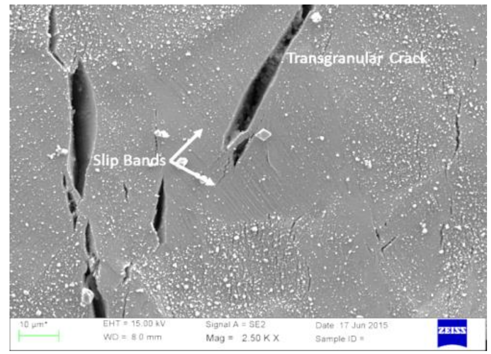
Effect of Applied Strain(2)

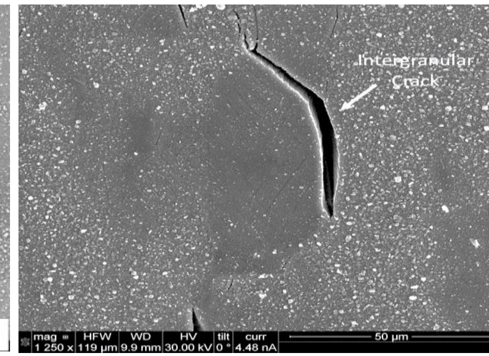
More substantial cracking (>100µm deep) was observed on tapered sample (T5) subject to approx. 14% strain at the narrowest point.

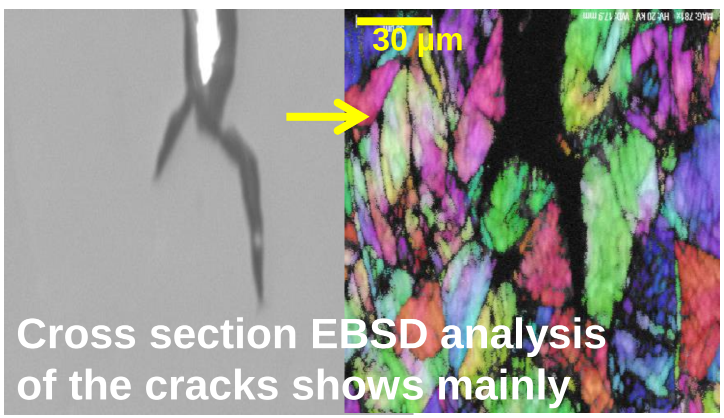


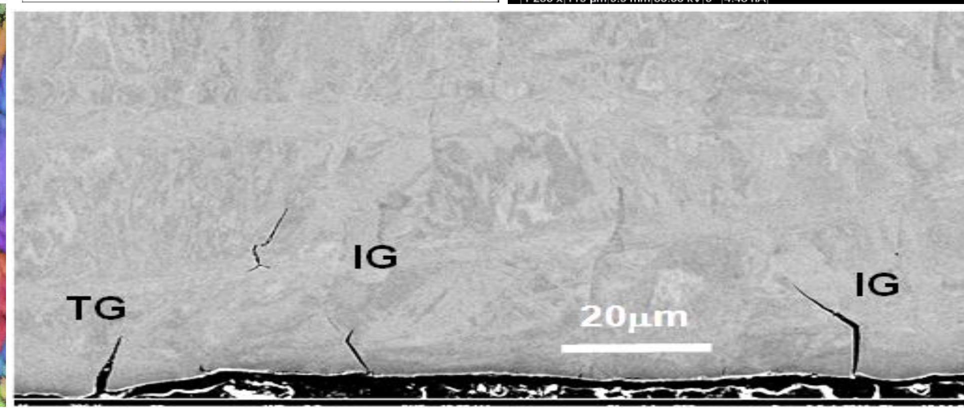
Crack Morphology

- A combination of intergranular and transgranular cracking was noted, although intergranular cracking was dominant, particularly in the higher strain (>5%) samples.



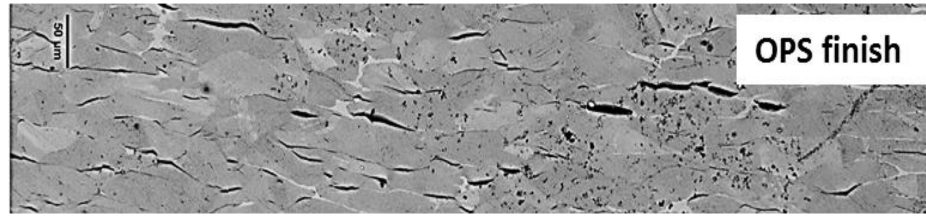


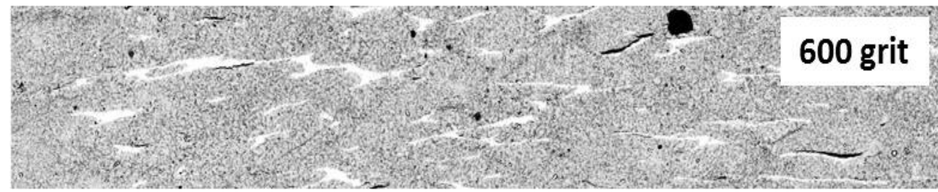


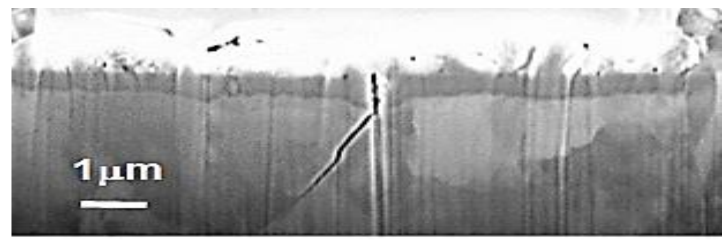


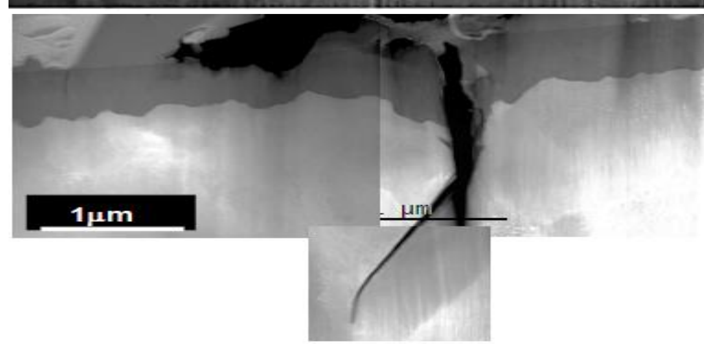
Surface Condition

- Cracks were noted to be relatively widespread on the OPS surface compared to 600 grit surface.
  - This is possibly due to modified surface layer on these ground surfaces, which may resist crack initiation under certain loading conditions
- On OPS surface finish, the majority of SCC cracks initiate at grain boundaries.
- On the 600 grit surface finish, crack initiation appeared to be predominantly transgranular.
  - Tendency for more irregular crack path in ground surface.
  - Cracks appear to develop by enhanced oxidation of deformation bands which subsequently crack.
  - Can develop into IGSCC when the crack intersects a grain boundary.



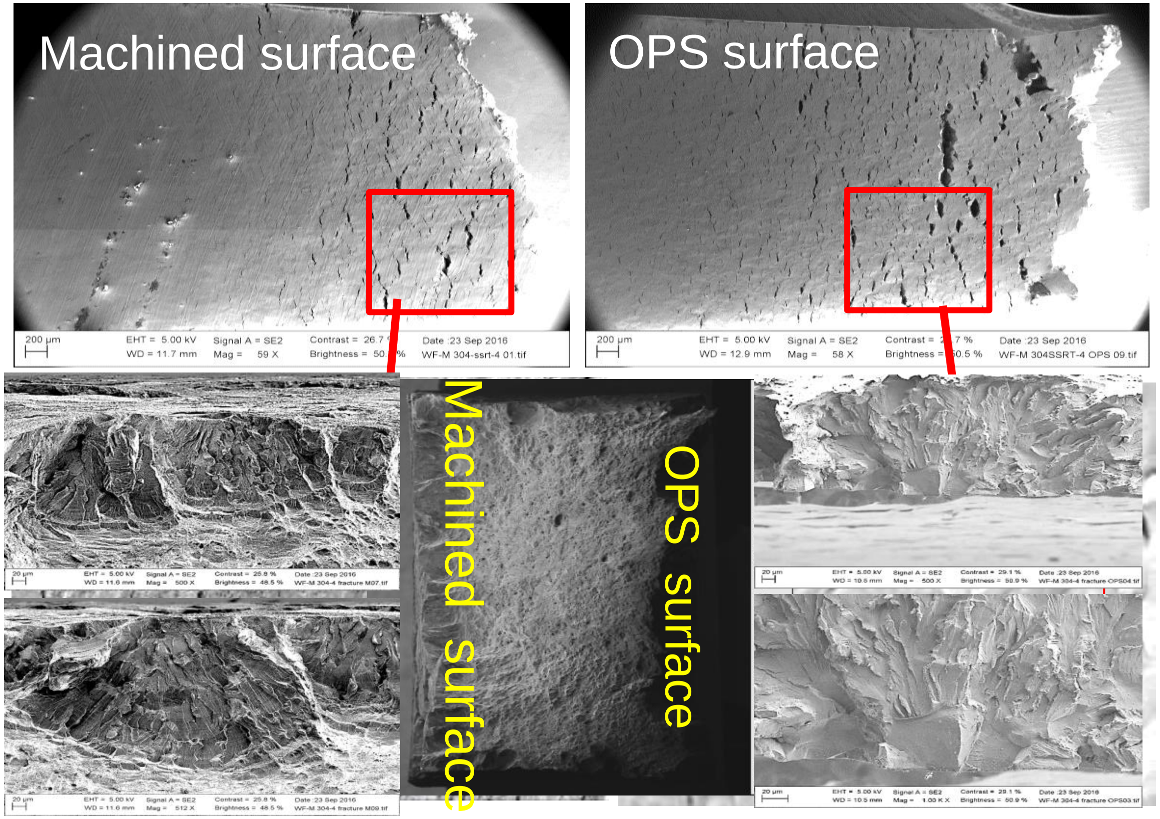






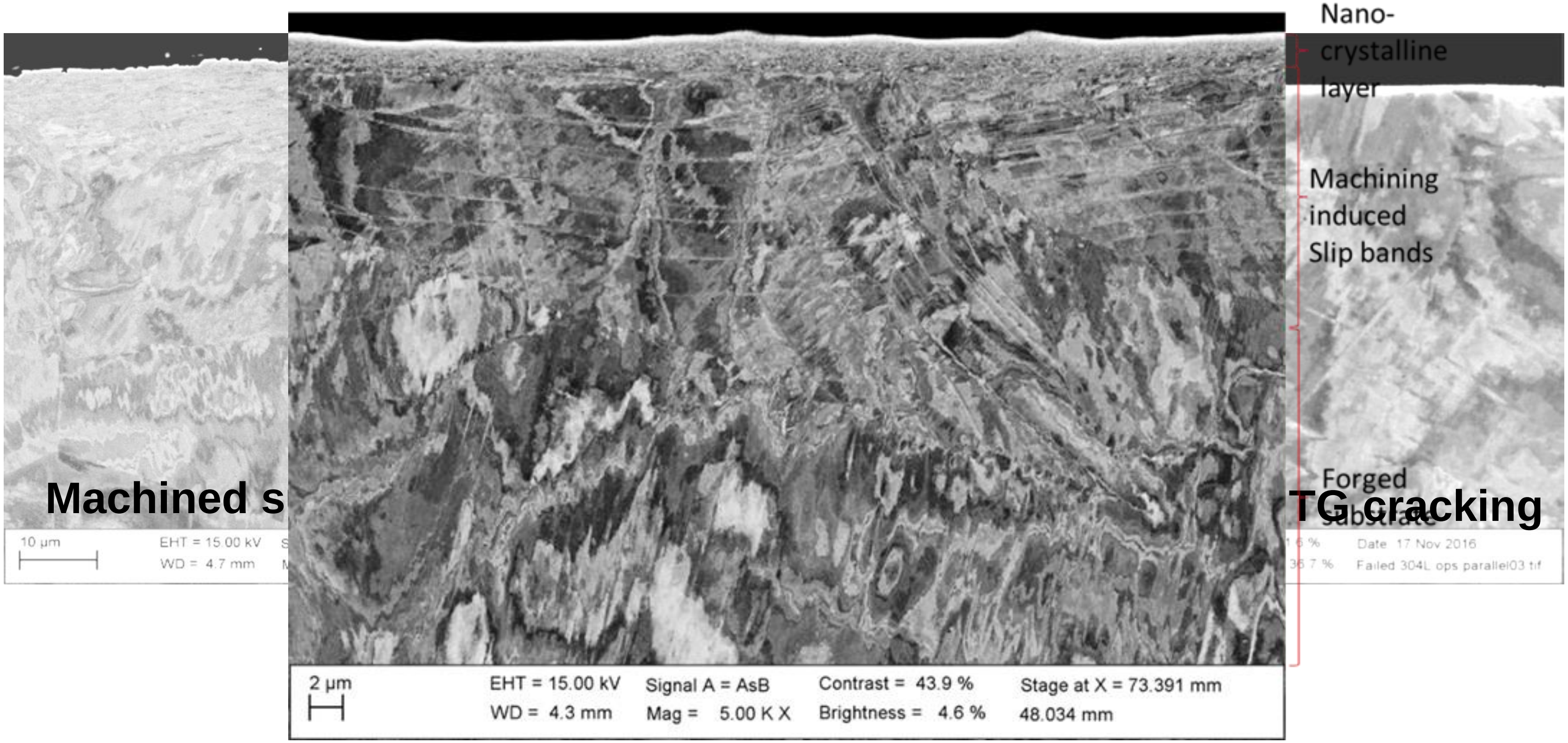
Surface Condition Vs. Crack Morphology(1)

- A sample from one of the SSRT tests, with machined surface on one side and OPS finish on the other side, had fractured.
- TG initiation on machined side along the machining marks whilst mainly IG initiation on polished side.
- Fractography confirmed predominantly TG cracking on machined surface, whilst IG crack initiation was dominant on OPS finish



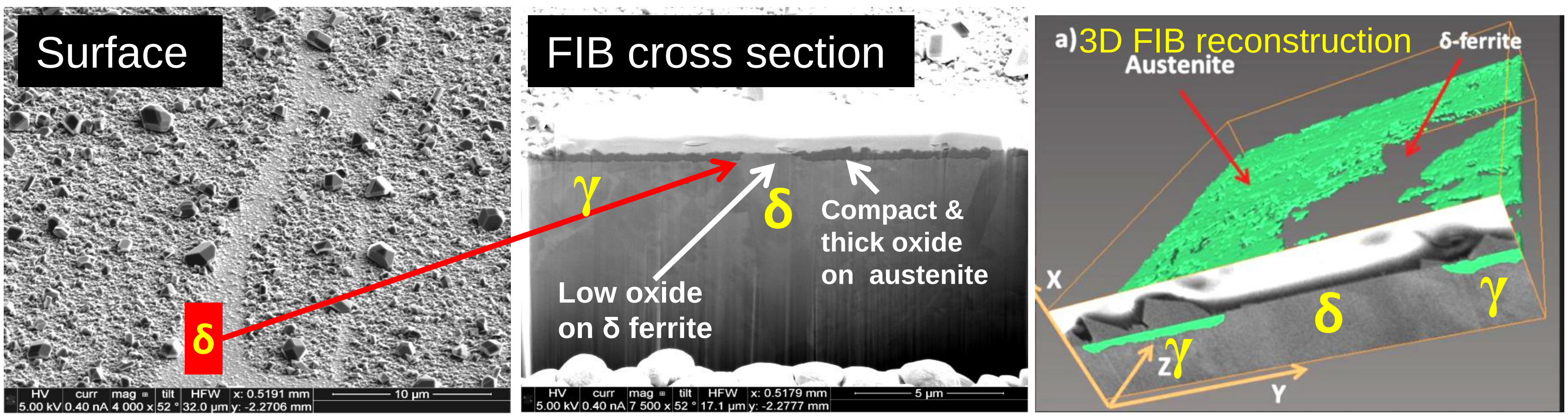
Surface Condition Vs. Crack Morphology(2)

- This observation is further confirmed by cross section analysis.
  - This is possibly due to thin nano-crystalline surface layer (~2µm thick) from the grinding/machining process.



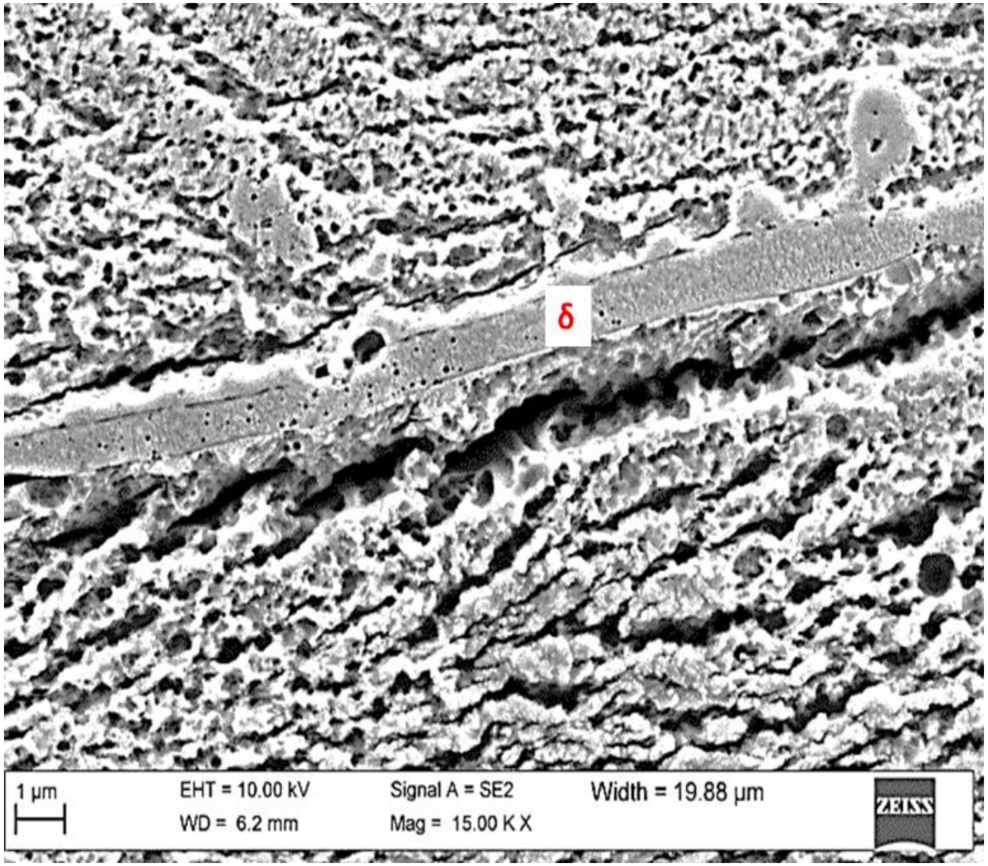
Oxidation Behaviour

- Selective oxidation of austenite vs. δ-ferrite was noted to be consistent across all samples examined.
  - Austenite was noted to have compact and generally uniform oxide whilst delta ferrite had relatively low surface oxide layers.



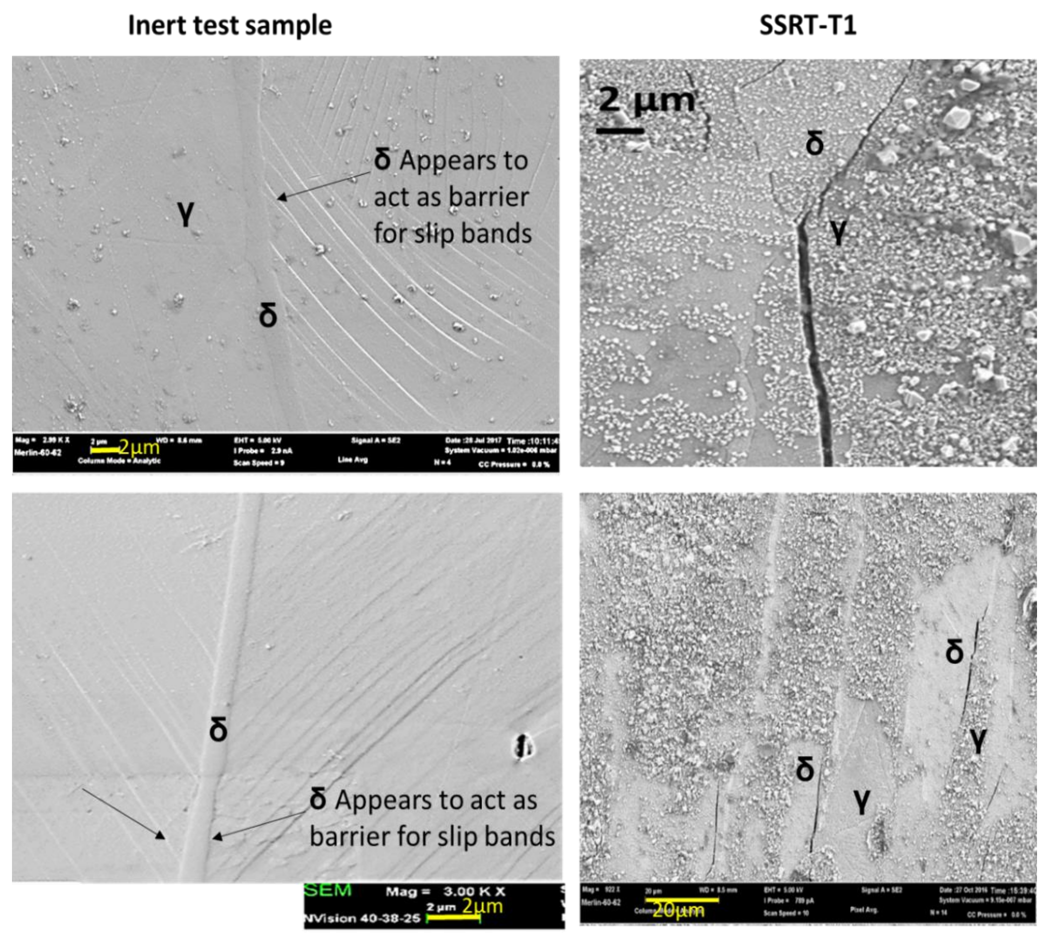
Microstructure Effects: Delta ferrite

- Delta ferrite appears to play a significant role in crack initiation.
  - A significant number (up to 50%) of cracks have initiated at austenite to δ-ferrite interfaces in all the test samples.
  - Although many of these cracks were superficial and limited to the oxide layer, some had extended into the metal substrate.
  - Selective oxidation appears to present a potential stress raiser, where the cracking can initiate at the interface.
  - Detailed examination of these cracks showed that cracking occurred close to the interface (~50nm) in the austenite matrix.



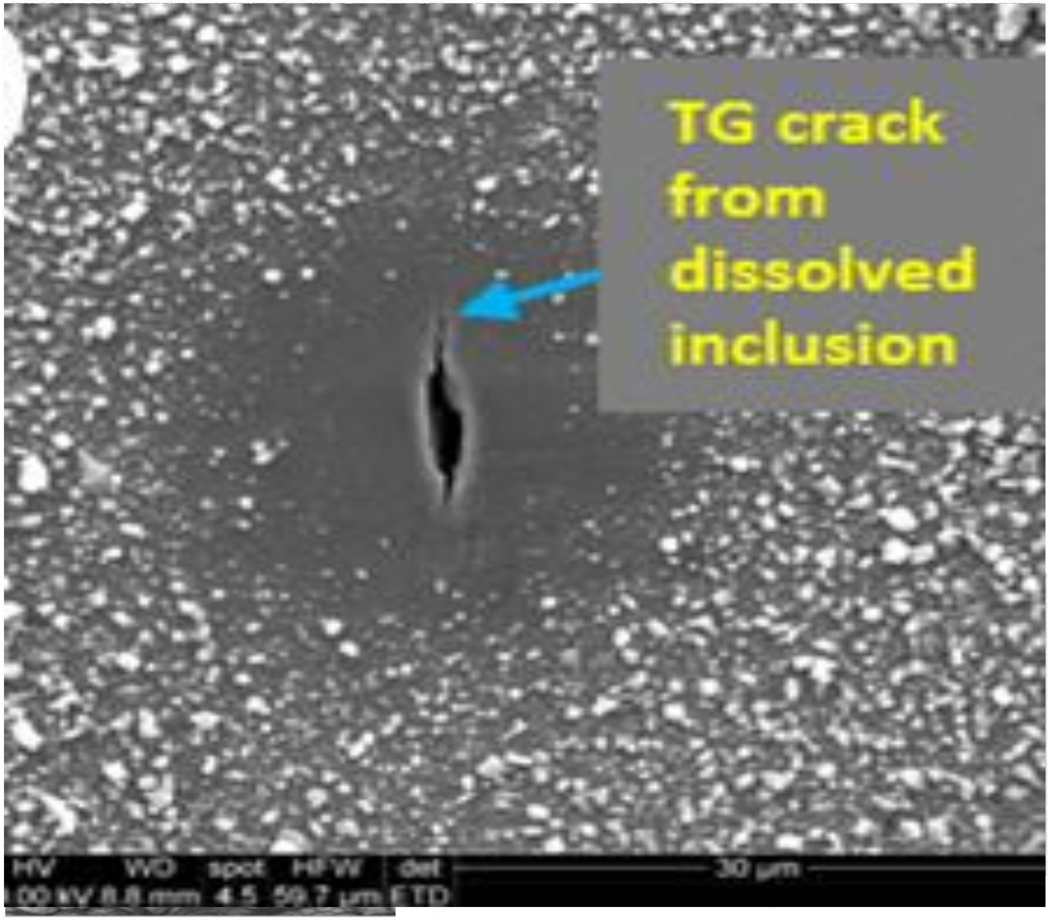
Microstructure Effects: Delta Ferrite (2)

- An SSRT test, subject to ~5% strain, in inert environment has produced no cracks, which indicates that cracking at the interface requires some degree of environmental contribution.
- Delta ferrite appears to act as a barrier for the deformation slip bands thereby producing strain localisation at the interfaces.
- However, more work is required to understand whether, these interfaces act as prominent sites for SCC initiation, in the absence of dynamic straining.



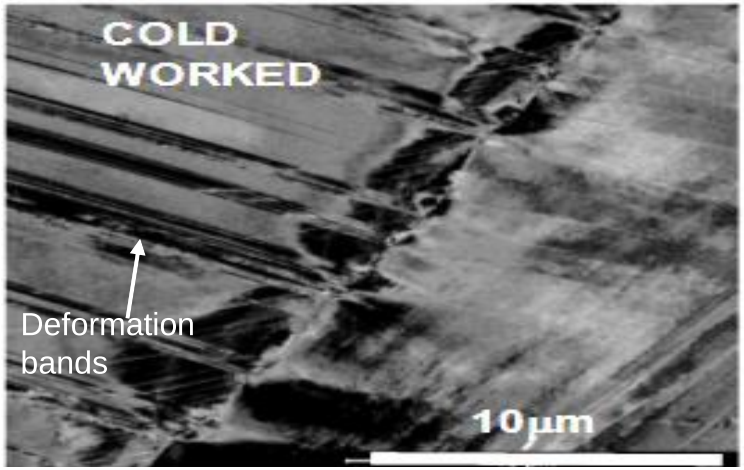
Microstructure effects : Inclusions

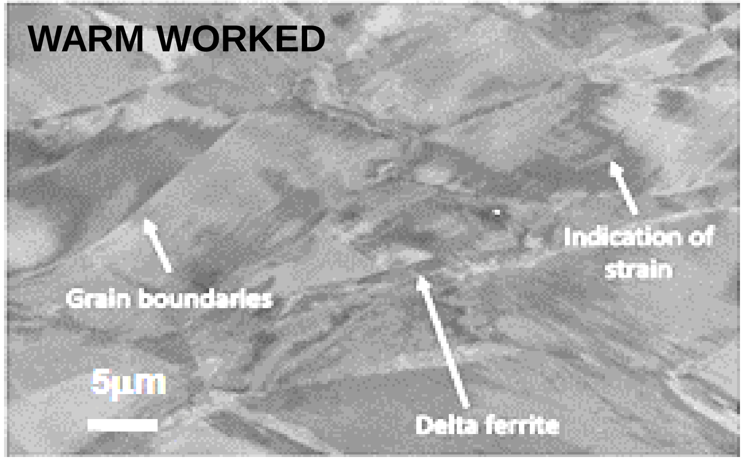
- The material used for this study contained variable levels of inclusions and delta ferrite, segregated into bands.
- EDX analysis confirmed that these inclusions were complex in nature.
  - Many of these inclusions have partially dissolved and produced elongated crack-like features.
  - Some surface cracks also appear to be associated with internal inclusions.
  - Some of these features were observed to have TG crack initiation at the edges, although not confirmed to be SCC.



Cold Work Vs. Warm Work.

- A predecessor study used 20% cold rolled austenitic stainless steel (Type 304) for SCC testing, where maximum applied strain was 3.6%.
- SSRT testing in that study produced significant (several hundred microns deep) and wide spread cracking.
- In comparison the current study used warm forged material subject to >5% plastic strain. However the degree of cracking noted for the same strain was significantly lower.
- The cold working appears to have produced significant deformation features between adjacent grans, where as the warm forging did not generate such deformation between grains.
- This may be the cause for reduced level of cracking in current study even under higher level of applied strain.





Conclusions(1)

- SSRT tests showed that SCC initiation is possible in 20% warm worked austenitic stainless steels in good quality primary water under dynamic loading. Initiated cracks can also propagate under specific conditions.
  - At the strain rates applied, at least 5% plastic strain is required to produce significant SCC initiation.
  - 3% plastic strain did produce some SCC cracking but mainly limited to surface oxide.
- A combination of intergranular and transgranular morphology was noted, although intergranular cracking was found to be dominant.
- Surface condition also influences crack initiation.
  - Smooth polished surfaces produced mainly IG crack initiation.
  - Whilst rougher ground/machined surfaces produced mainly TG crack initiation.
  - Cracking was more widespread on OPS surface finish compared to 600 grit. This is possibly due to modified surface layer on these ground surfaces, which may resist crack initiation under certain loading conditions.
- Type of work hardening (Cold Vs. Warm) also appears to influence the crack initiation with cold worked samples showing more extensive cracking compared to warm worked material at similar strain levels.

Conclusions(2)

- Selective oxidation behaviour between austenite and δ-ferrite. The austenite phase has thicker and more uniform oxide layers compared to δ-ferrite, as expected. The difference in oxide thickness is a potential stress raiser.
- Oxidation behaviour was also noted to be compact and uniform on OPS polished surfaces, whilst non-uniform and banded on 600-grit surfaces.
- Material composition and microstructure appear to play significant roles in SCC initiation.
  - Significant number of SCC cracks have initiated at ferrite/austenite boundaries. Close control of chemical composition is recommended to minimise formation of delta ferrite.
  - Delta ferrite appears to act as a barrier for the deformation slip bands thereby producing strain localisation at the interfaces, with environmental contribution and selective oxidation, the interface may act as potential stress riser for crack initiation.
  - The complex inclusions present in the material have dissolved to form crack-like features. Some TG crack initiation noted near these features but not considered to be SCC.

Questions?

Thank you for your attention!

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