

Environmental Effects on Low-Cycle Fatigue Lives of 316L/316LN Austenitic Stainless Steels in High Temperature Water

Yida Xiong

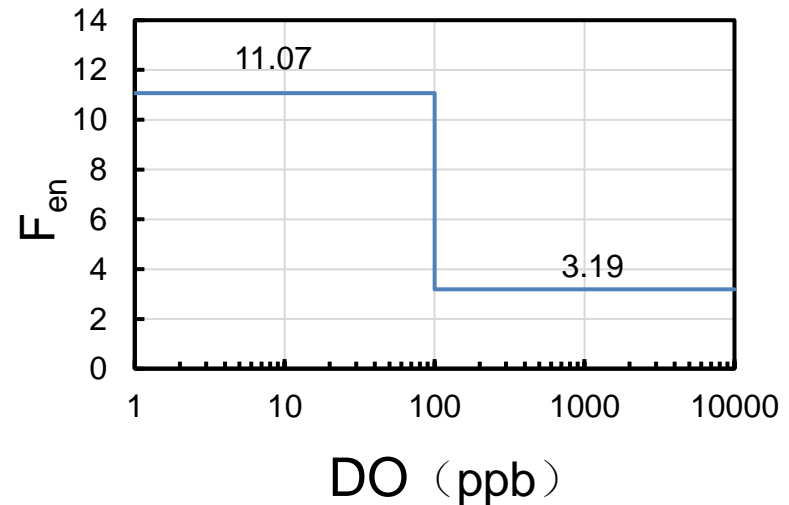
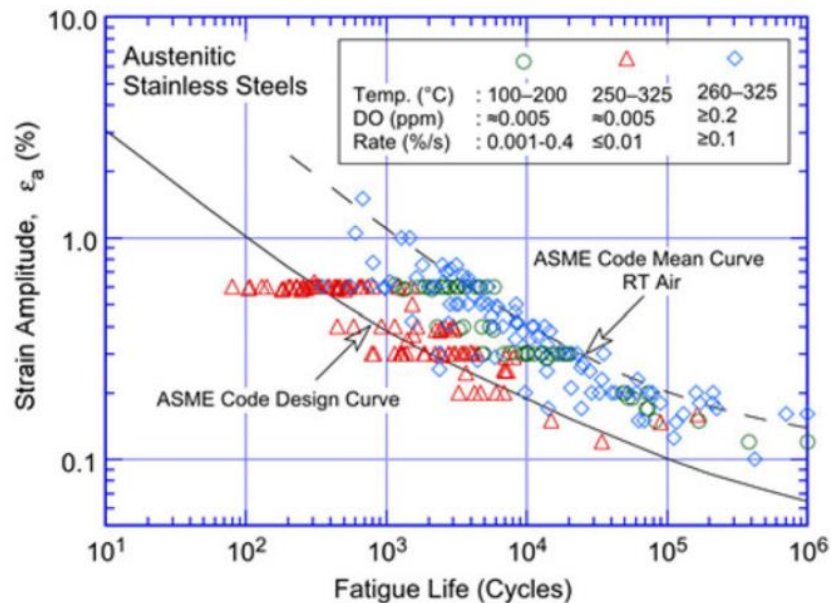
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Background and objectives

DO (dissolved oxygen)

The low-cycle fatigue (LCF) lives of austenitic stainless steels (SSs) were longer at high DO level (≥ 100 ppb) than that at low DO level. The reason remains unclear. Most of the data for high-DO water was obtained by using pure high temperature water, while most of the data for low-DO water was obtained by using borated and lithiated high temperature water. This would be one reason why the phenomenon occurred.



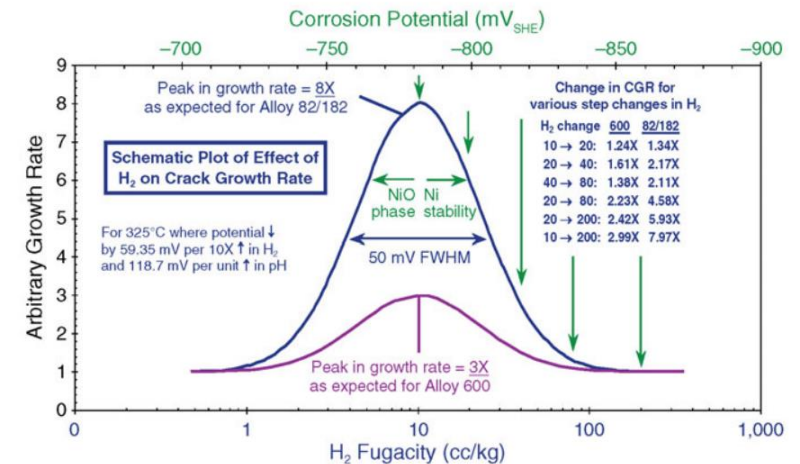
$F_{en} = N_{air}/N_{water}$, N_{air} is the fatigue life in air and N_{water} is the fatigue life in water. The greater the N_{air} , the more deleterious the environment.

Background and objectives

DH(dissolved hydrogen)

1. Currently, around 2.2~3.1 ppm (25 cc/kg to 35 cc/kg) DH is added in the water.
2. Crack growth rate of nickel alloys showed a dependence on DH level.
3. Whether the DH is the main source of the absorbed hydrogen remains unclear.

Conductivity ($\mu\text{S}/\text{cm}$)	1~40
pH	7.2~7.4 (at 285 °C)
B (ppm)	Depends on core reactivity
Li (ppm)	0.2~2.2
Dissolved Oxygen (ppm)	<0.005
Dissolved Hydrogen (cm^3 (STP) $\text{H}_2/\text{kg H}_2\text{O}$)	25~35



Reference : P.L. Andresen, J. Hickling, A. Ahluwalia, and J. Wilson, 2008, Effect of Hydrogen on Stress Corrosion Crack Growth Rate of Nickel Alloys in High-Temperature Water

Reference: 日本原子力学会標準 加圧水型原子炉一次系の水化学管理指針

In corrosion fatigue, change of DH level may affect the crack growth rate of 316 LN, leading to different fatigue lives at different DH level.

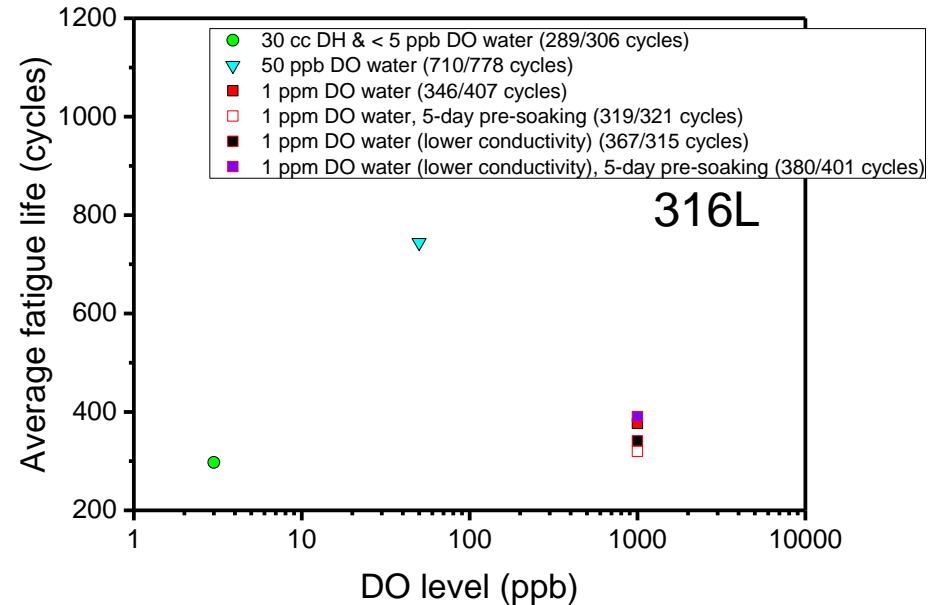
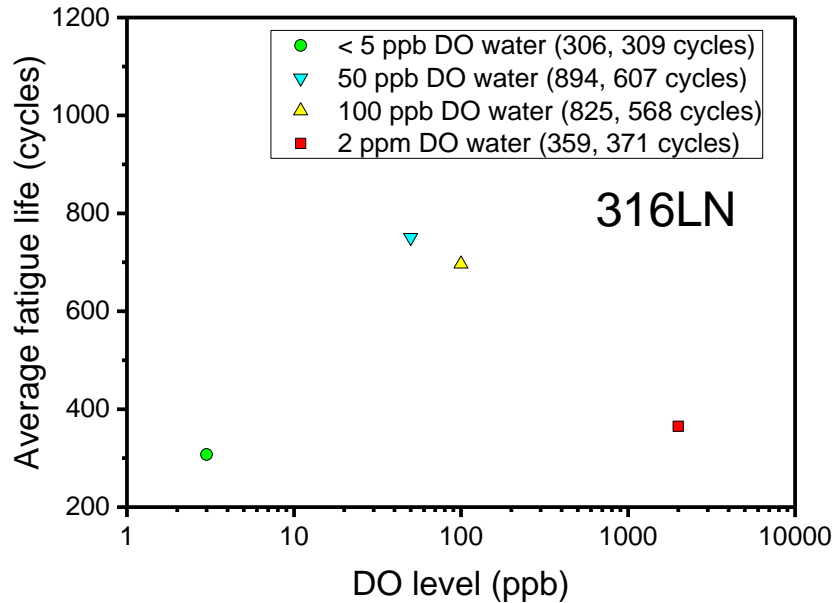
Background and objectives

Objectives:

- Investigating the LCF behaviors of 316L/316LN SSs at different DO levels.
 - Are the previous studies right?
 - Why do the LCF lives change at different DO levels?
- Investigating the LCF behaviors of 316LN SSs at different DH levels.
 - Will the change of the DH level affect the LCF behaviors of 316LN SS?
 - What is the main source of the hydrogen absorbed into the 316LN SS.
- Will the addition of boric acid and lithium hydroxide affect the LCF life of 316L SS in high-DO water?
- Recommendations for the optimization of the water chemistry.

LCF behaviors of 316LN/316L SS at different DO levels

Fatigue life & Cyclic stress response



< 5 ppb, 1 ppm and 2 ppm DO water: $F_{en} > 10$
 50 ppb, 100 ppb DO water: F_{en} was ~ 6

The equation used for predicting the F_{en} in the reference for austenitic stainless steels was not conservative enough at DO levels of < 5 ppb, 100 ppb and 2 ppm and not accurate at DO level of 50 ppb.

Reference:

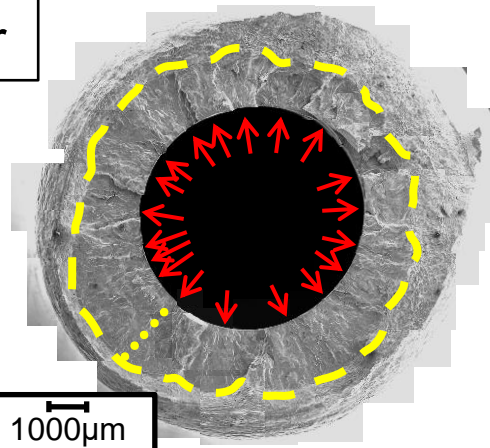
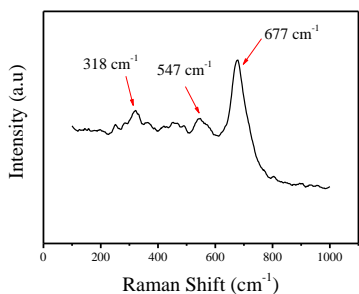
Omesh Chopra and Gary L. Stevens, 2018, "Effect of LWR Water Environments on the Fatigue Life of Reactor Materials," NUREG/CR-6909, Rev. 1

LCF behaviors of 316LN/316L SS at different DO levels

Fracture surface and Raman spectroscopy (316LN)

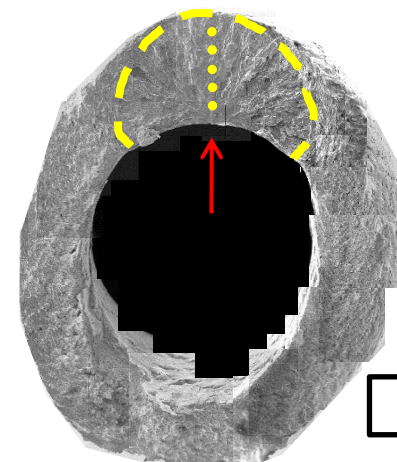
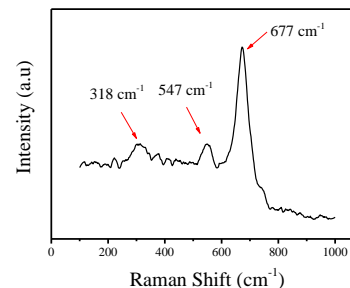
< 5 ppb DO water

Magnetite



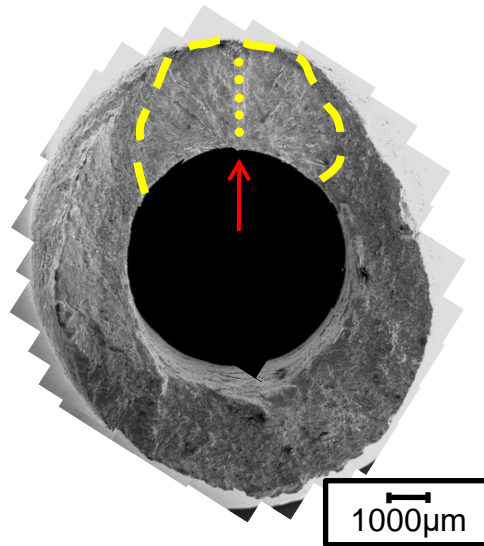
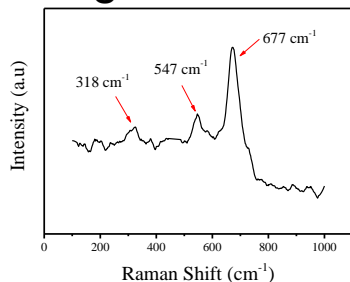
50 ppb DO water

Magnetite



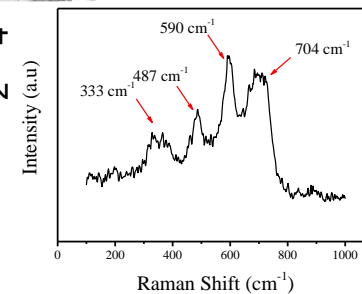
100 ppb DO water

Magnetite

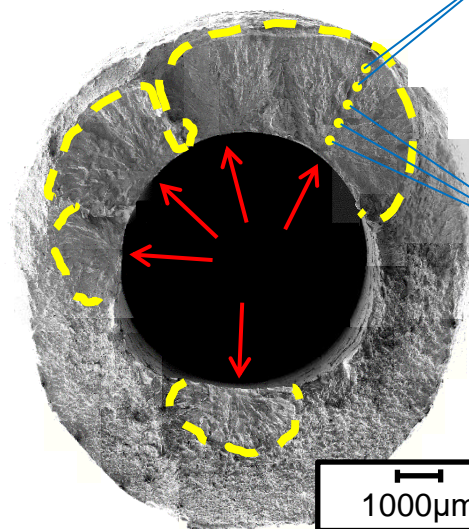
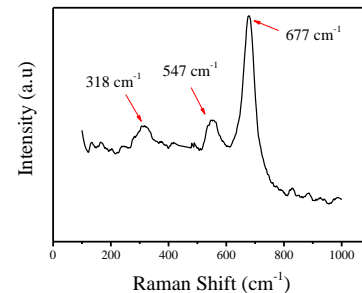


2 ppm DO water

NiFe₂O₄



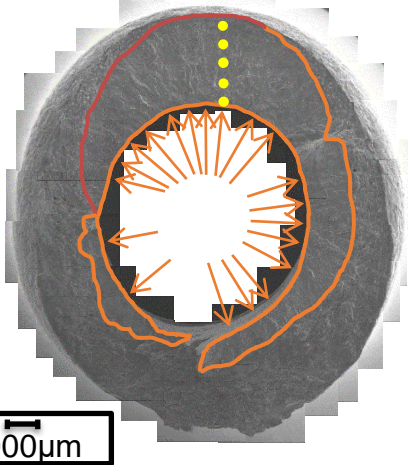
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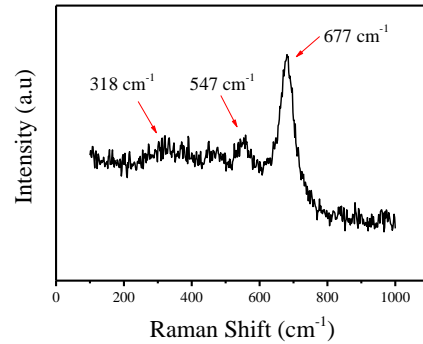
LCF behaviors of 316LN/316L SS at different DO levels

Fracture surface and Raman spectroscopy (316L)

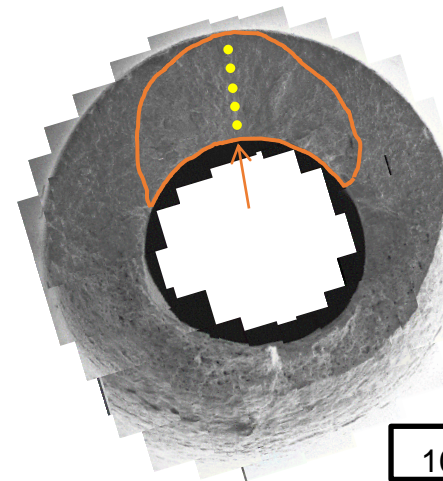
30 cc DH & < 5 ppb DO water



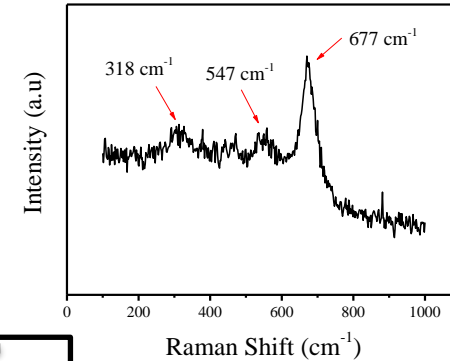
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50 ppb DO water

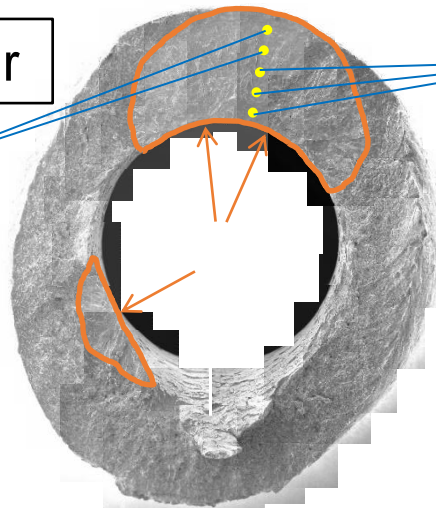
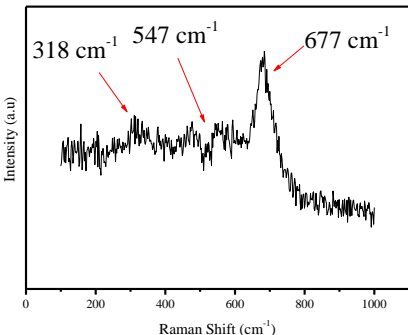


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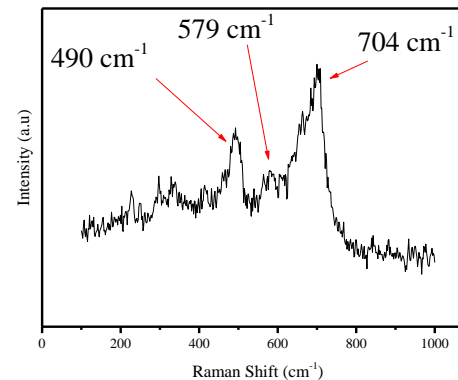


1 ppm DO water

Magnetite

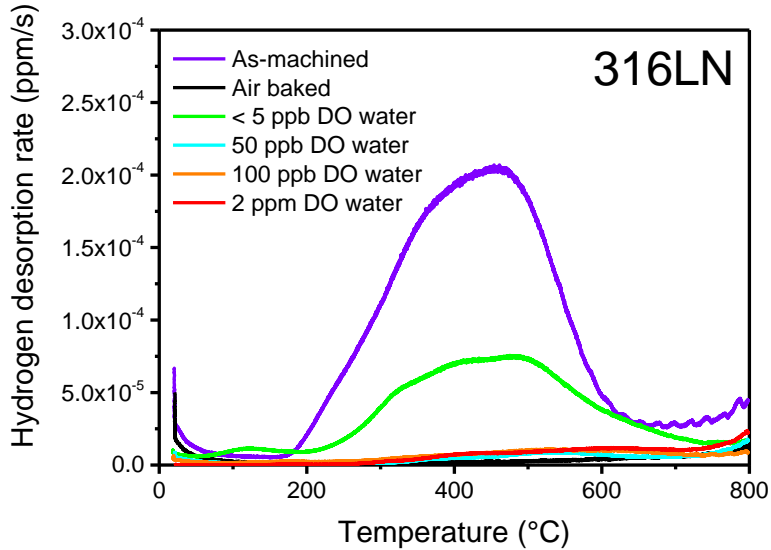


NiFe_2O_4



LCF behaviors of 316LN/316L SS at different DO levels

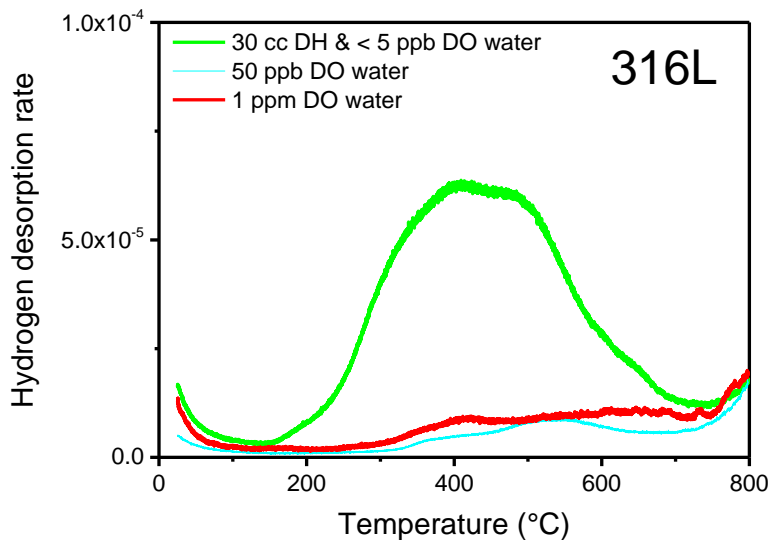
TDS analysis



Condition	< 5 ppb DO water	50 ppb DO water	100 ppb DO water	2 ppm DO water
Hydrogen Content (wppm)	0.96	0.13	0.17	0.18

As-machined: 2.23 ppm

Air baked (325 °C, 3 days): 0.11 ppm

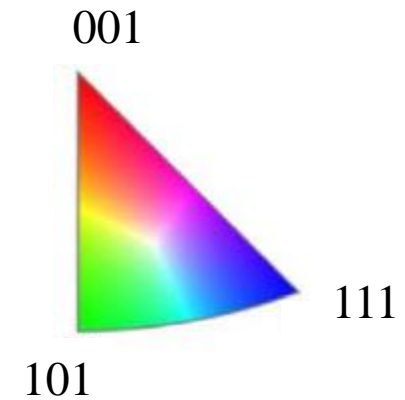
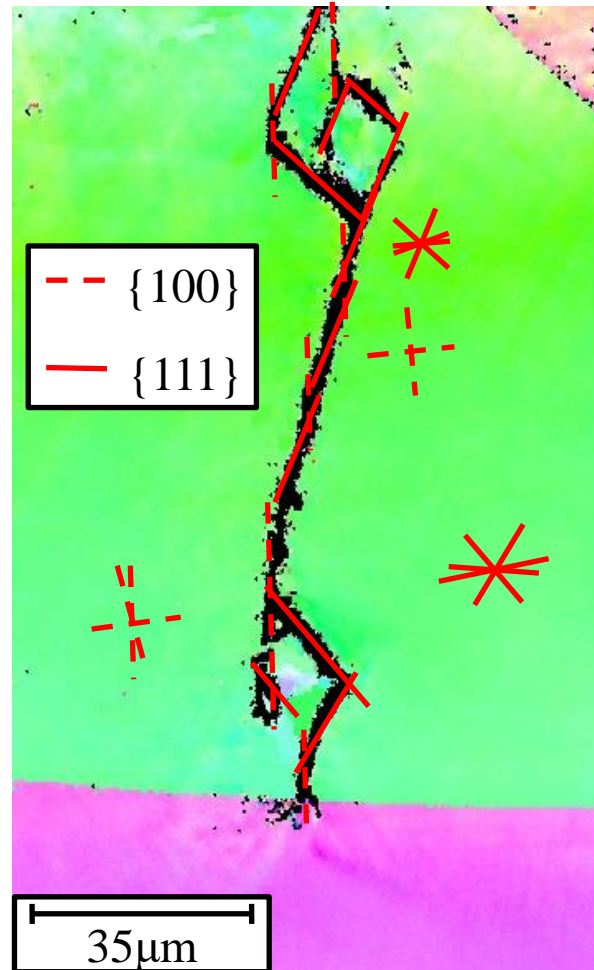


Condition	30 cc DH & < 5 ppb DO water	50 ppb DO water	1 ppm DO water
Hydrogen Content (wppm)	0.80	0.22	0.20

LCF behaviors of 316LN/316L SS at different DO levels

Crack propagation path characterization(316LN)

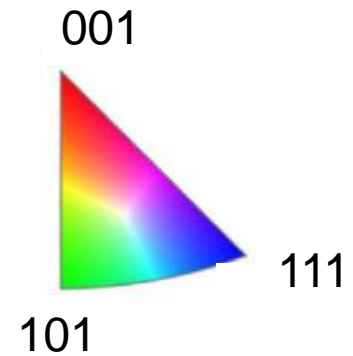
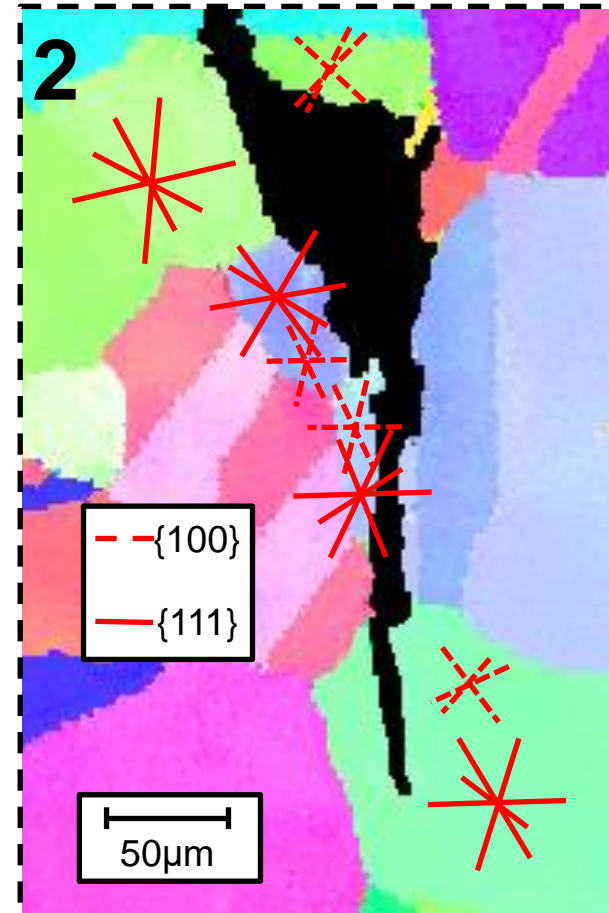
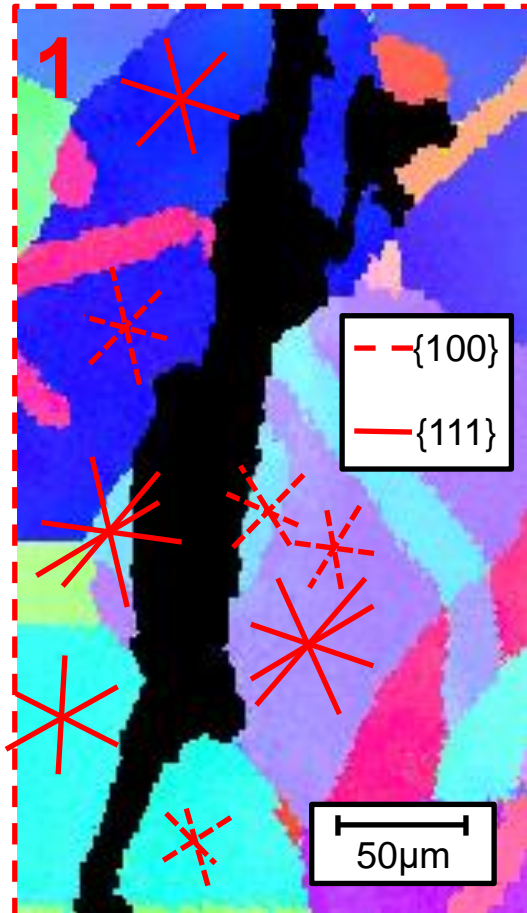
< 5 ppb DO water



LCF behaviors of 316LN/316L SS at different DO levels

Crack propagation path characterization (316LN)

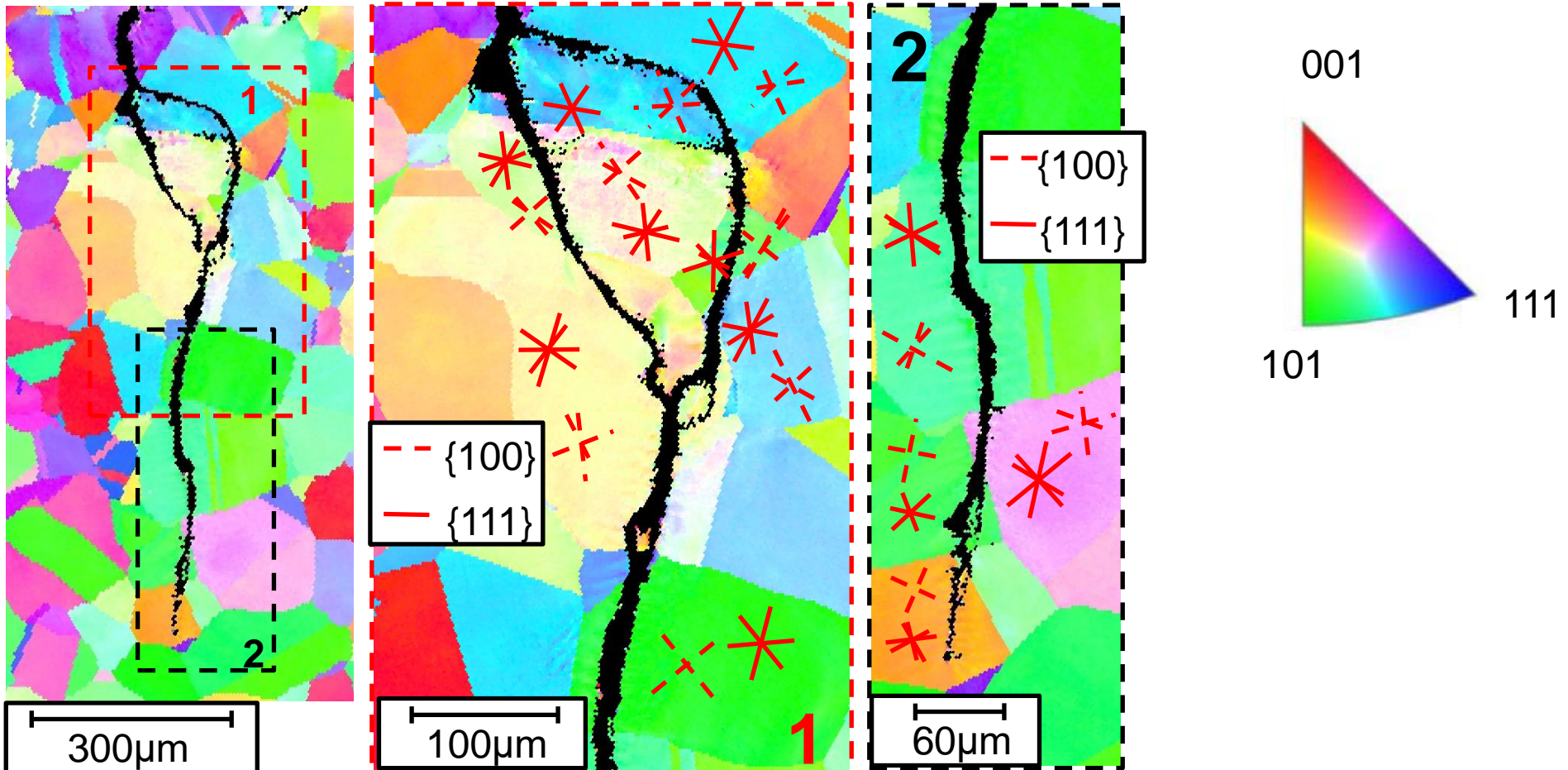
50 ppb DO water



LCF behaviors of 316LN/316L SS at different DO levels

Crack propagation path characterization (316LN)

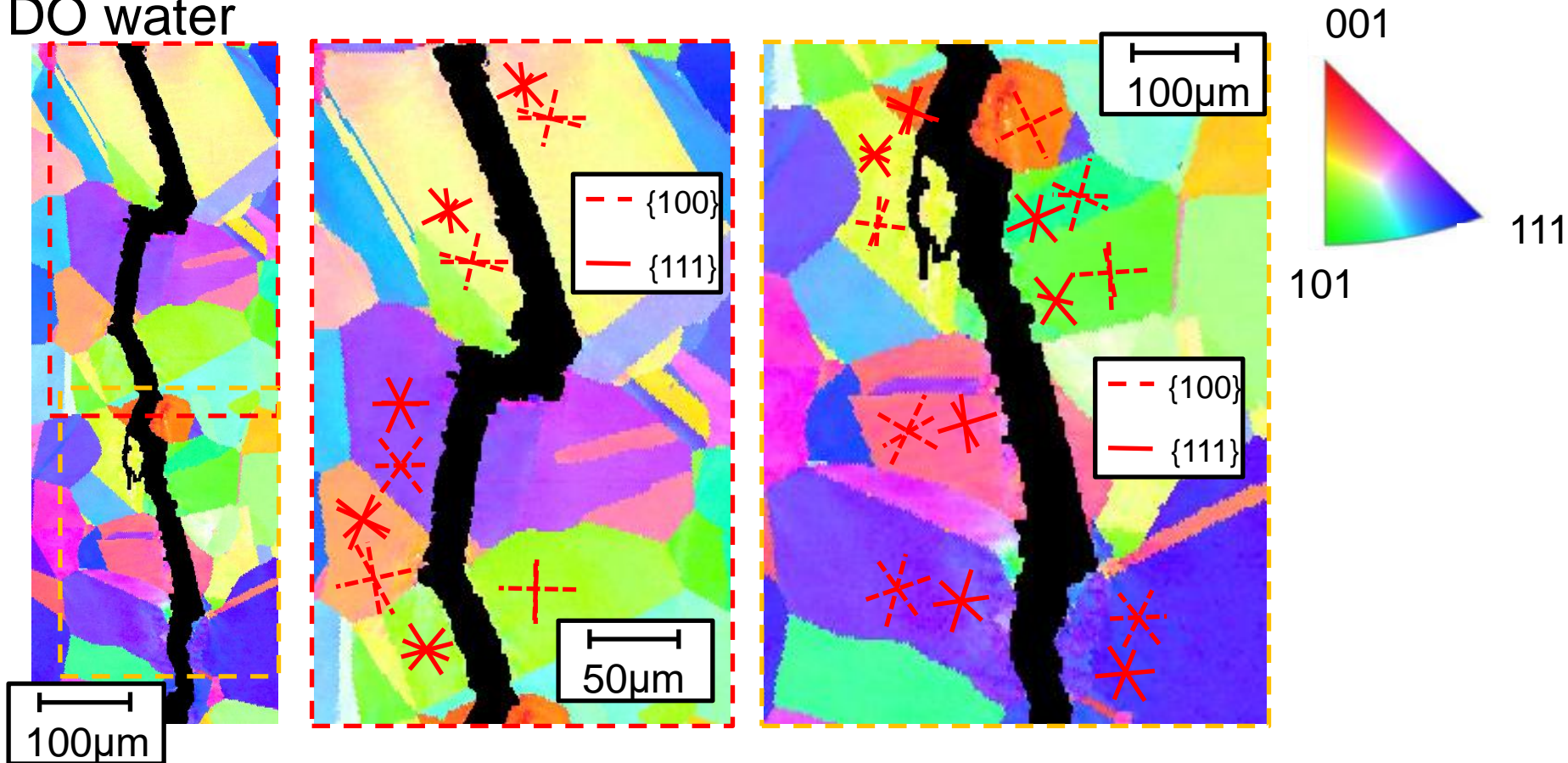
100 ppb DO water



LCF behaviors of 316LN/316L SS at different DO levels

Crack propagation path characterization (316LN)

2 ppm DO water



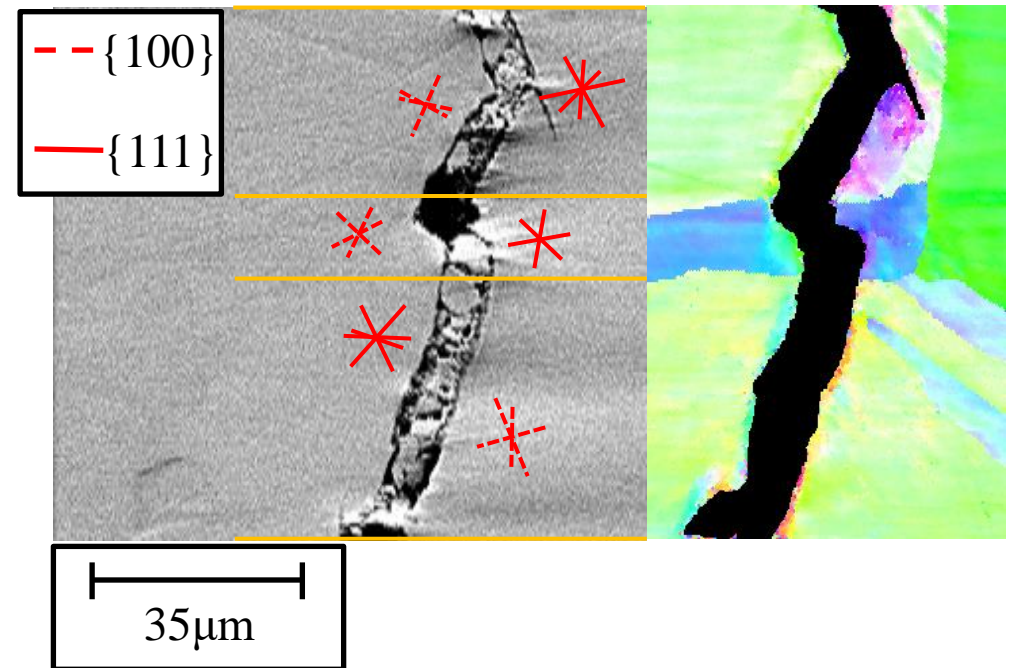
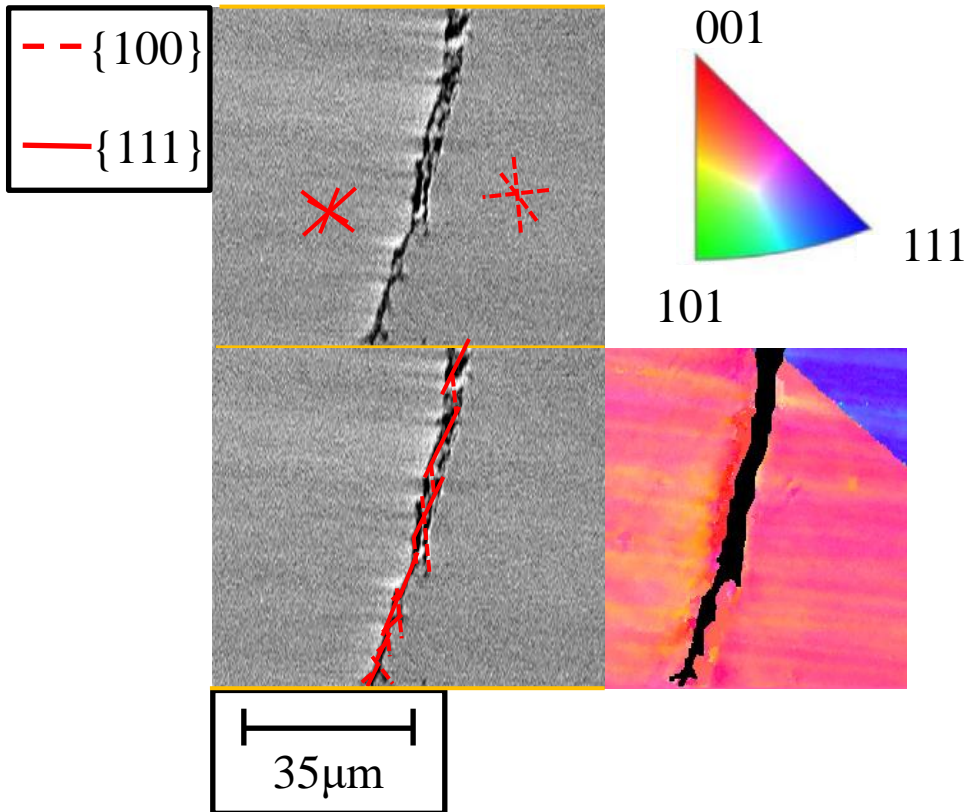
No crystallographic feature was observed at DO levels of 50 ppb, 100 ppb, and 2 ppm, while the crack mainly propagated along {111} planes and sometimes along {100} planes when the DO level was < 5 ppb.

LCF behaviors of 316LN/316L SS at different DO levels

Crack propagation path characterization (316L)

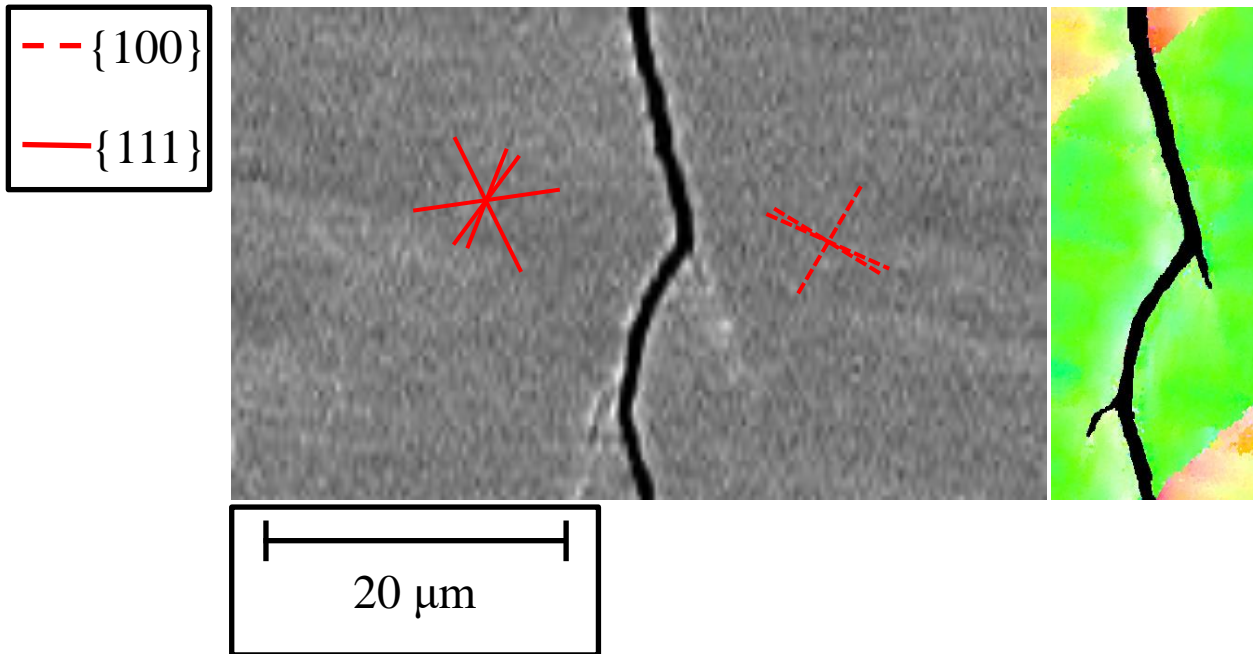
30 cc DH & < 5 ppb DO water

50 ppb DO water



Crack propagation path characterization (316L)

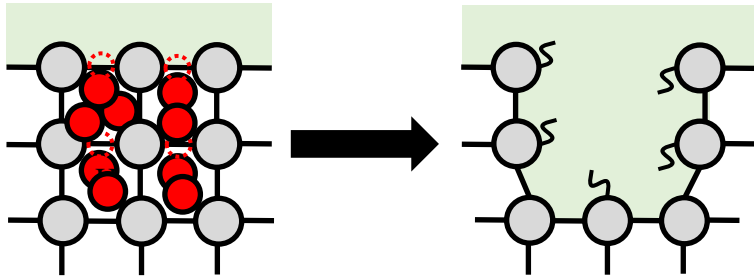
1 ppm DO water



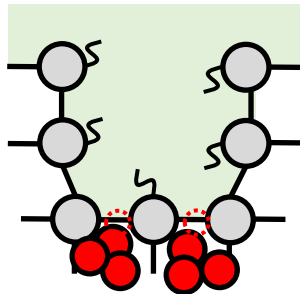
Similar to 316LN, crack of specimen tested in low-DO water showed crystallographic feature while those tested in oxygenated water did not show such a feature. Combining with other results, it is considered that the mechanisms for 316L were the same with those for 316LN

Crack initiation and propagation

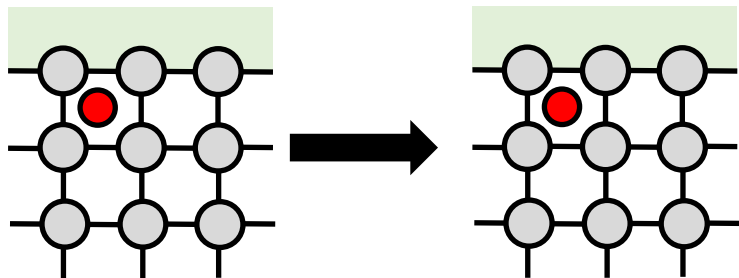
< 5 ppb DO water



○ Metallic element
● H



50, 100 ppb DO water



In the < 5 ppb DO water, more hydrogen was absorbed into the metal. Therefore, more hydrogen-induced decohesion occurred. As the hydrogen usually was trapped by the dislocation, therefore hydrogen accumulated along slip plane. Since the main slip system of metals with fcc structure is $\{111\}\langle 110\rangle$, crack mainly propagated along $\{111\}$.

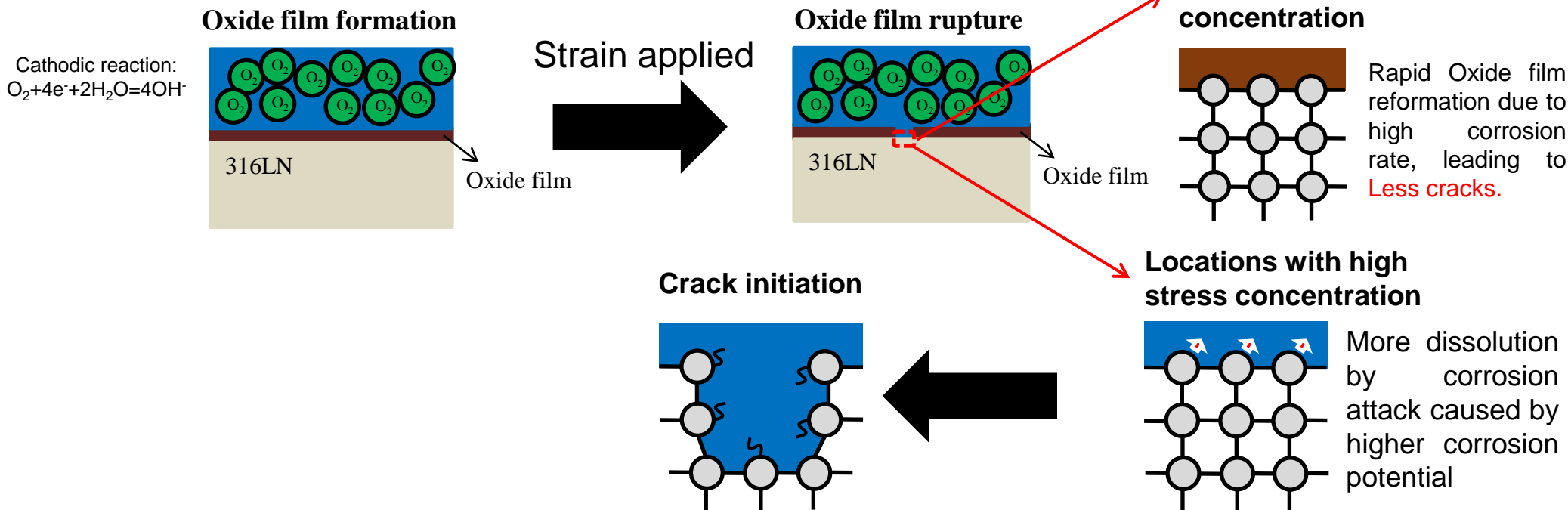
Publication:

1. Yida Xiong, Yutaka Watanabe, Yuki Shibayama, Nicolas Mary, Effects of 100 ppb dissolved oxygen on low-cycle fatigue behaviors of 316LN austenitic stainless steel in borated and lithiated high temperature water and mechanism behind these effects, *Corrosion Science* (2020).
<https://doi.org/10.1016/j.corsci.2020.108567>

Chapter 2: LCF behaviors of 316LN SS at different DO levels

- With regard to the mechanism of the decrease in LCF life at DO level of 2 ppm

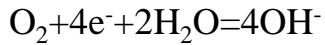
Crack initiation



Chapter 2: LCF behaviors of 316LN SS at different DO levels

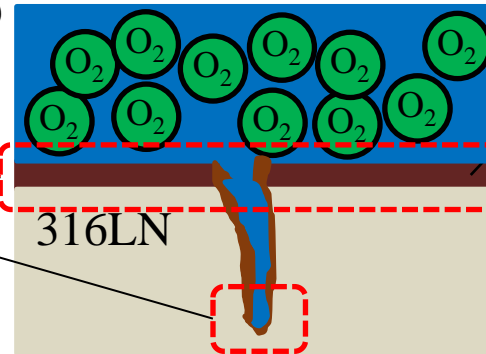
Crack propagation

Cathodic reaction:



Mainly occurred on inner surface or

crack mouth (higher potential)



Oxide film

316LN

Higher potential gradient

REFERENCE:

1. Yida Xiong, Yutaka Watanabe, Yuki Shibayama, Nicolas Mary, Xiangyu Zhong, Low-cycle fatigue behaviors of 316LN austenitic stainless steel in borated and lithiated high temperature water with different levels of dissolved oxygen, Corrosion Science 176 (2020). DOI: <https://doi.org/10.1016/j.corsci.2020.10904>

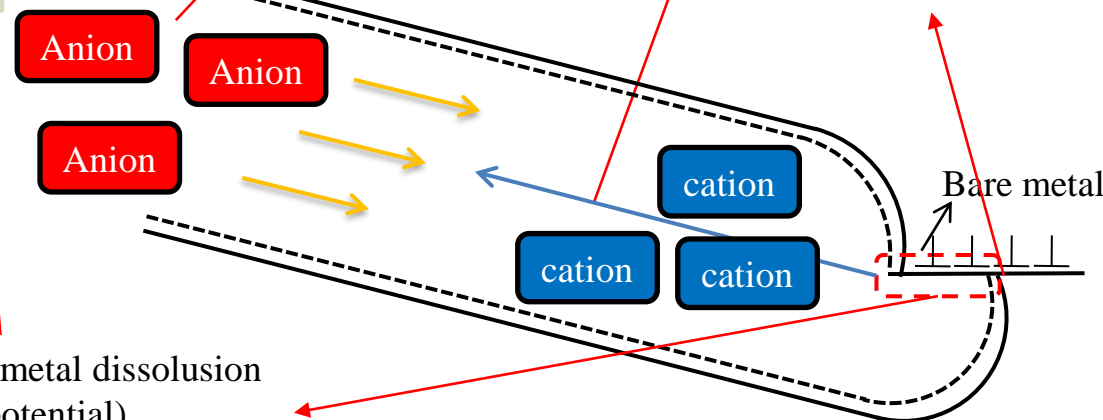
8.

Anodic reaction: metal dissolution (lower potential)

More anions from bulk water (caused by higher potential gradient)

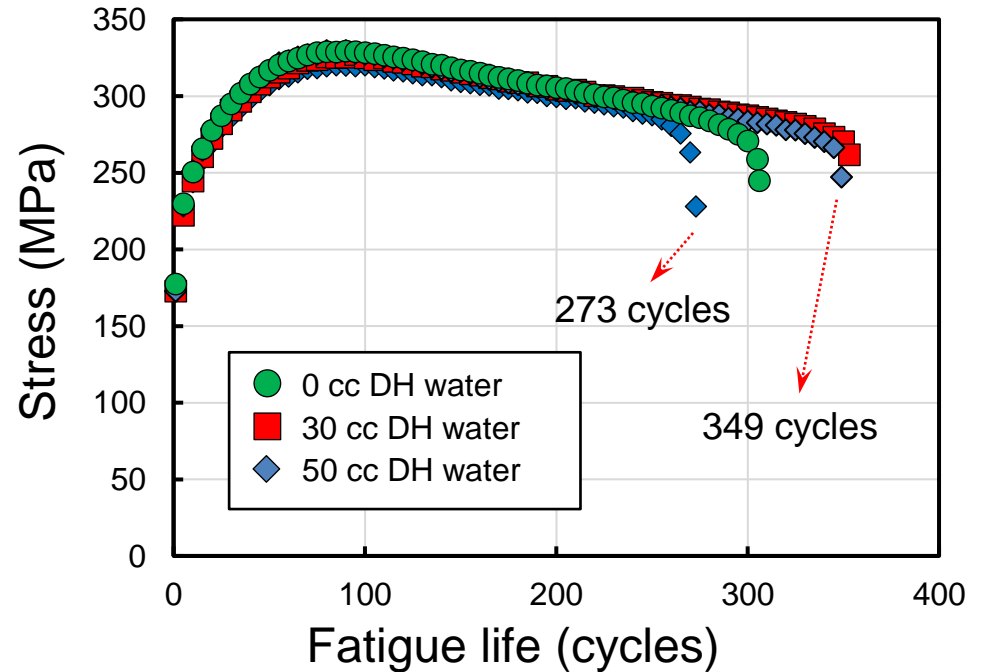
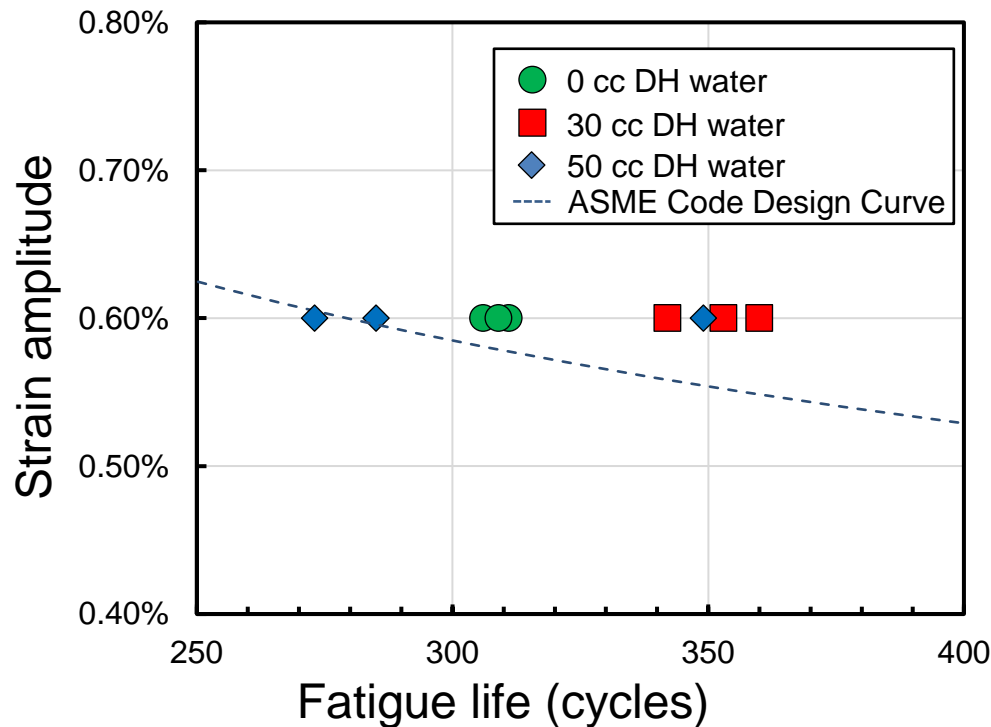
More cations produced by metal dissolution to maintain charge neutrality

More metal dissolution (caused by higher crack tip potential than that in 50 ppb and 100 ppb DO water)



LCF behaviors of 316LN SS at different DH levels

Fatigue life & Cyclic stress response

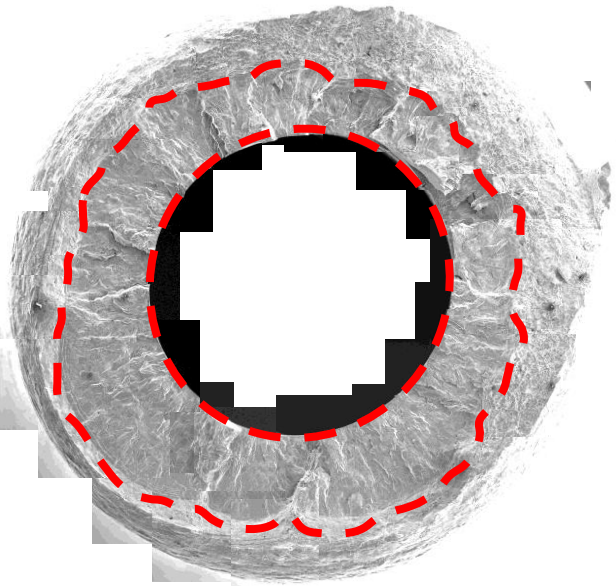


Given the typical scatter of the LCF life in simulated PWR primary water, it is considered that the change of DH level had no effect on the LCF life of 316LN SS. The cyclic stress response at different DH level also did not show a big difference.

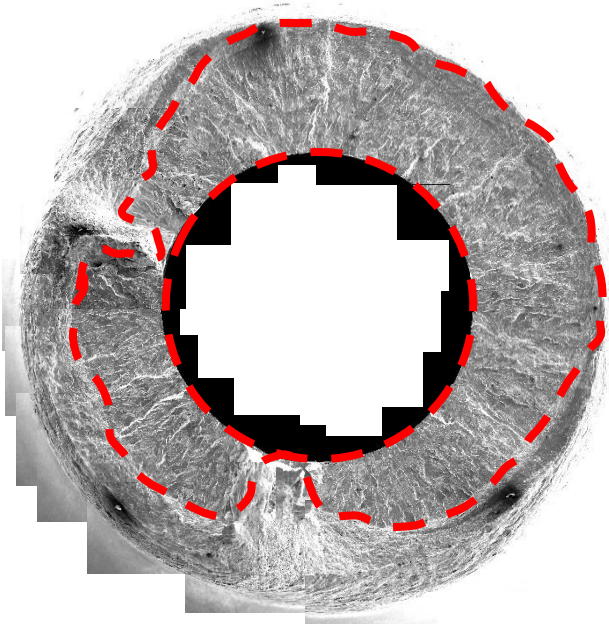
LCF behaviors of 316LN SS at different DH levels

Fracture surface

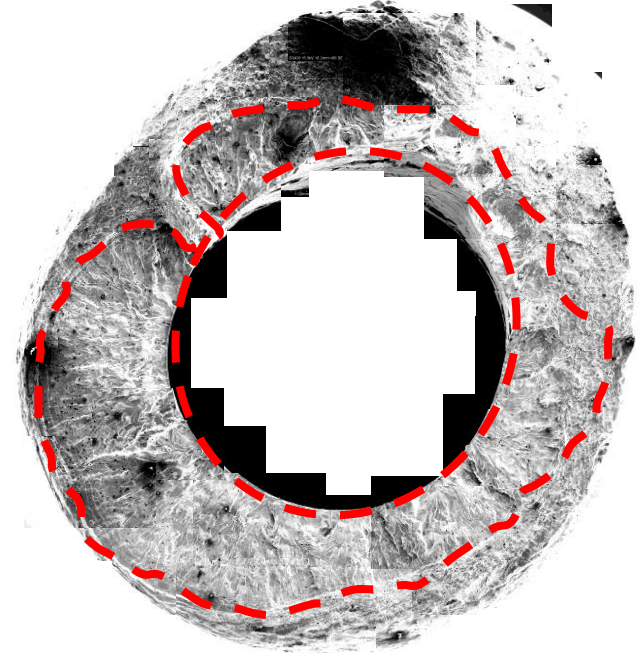
0 cc DH water



30 cc DH water



50 cc DH water



1000μm

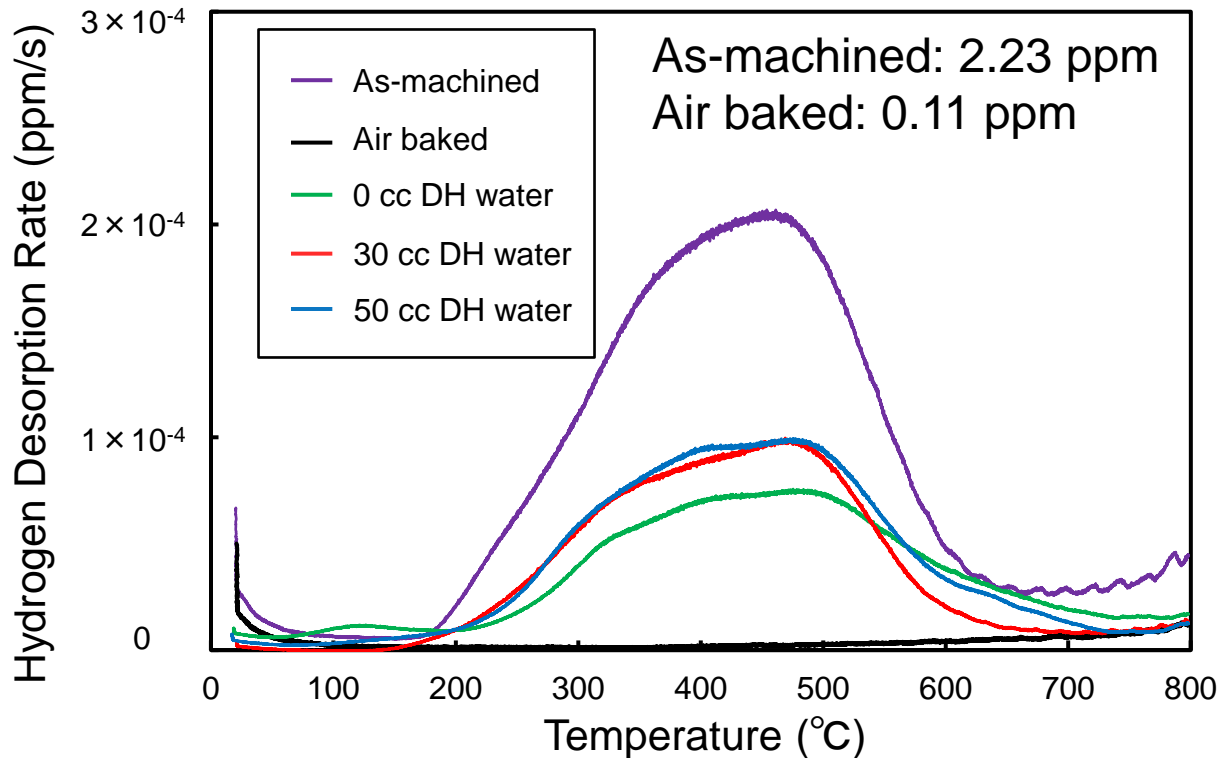
1000μm

1000μm

Fracture surfaces of specimens tested at different DH level did not show a big difference. On these surfaces, striation, second crack and tertiary crack were observed.

LCF behaviors of 316LN SS at different DH levels

TDS analysis (180th cycle)



Even no DH was added, 0.96 wppm hydrogen was detected inside the material.

Hydrogen content increased slightly with increasing DH.

Hydrogen absorbed mainly came from the hydrogen produced by **corrosion reaction.**

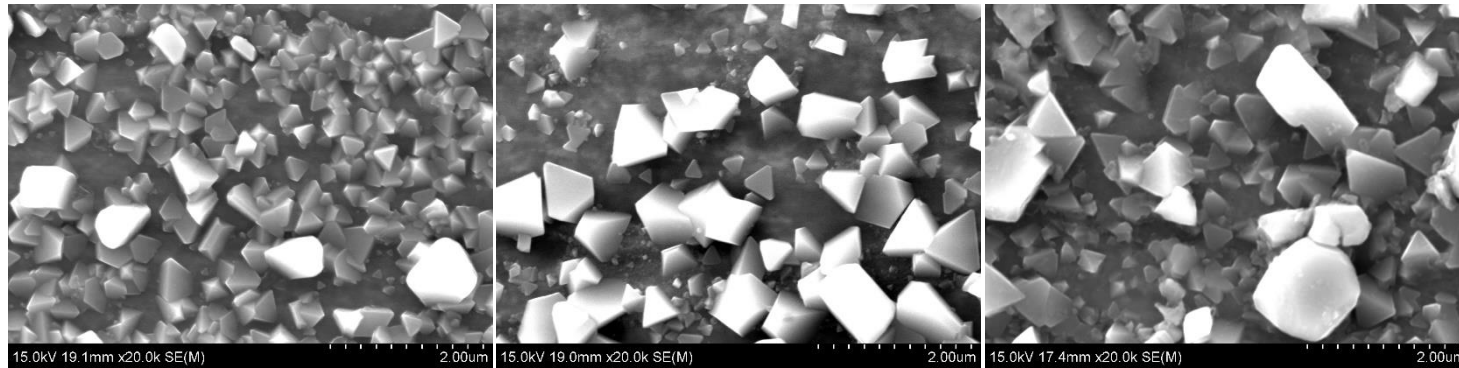
Condition	0 cc DH water	30 cc DH water	50 cc DH water
Hydrogen Content (wppm)	0.96	0.99	1.11

Oxide film characterization

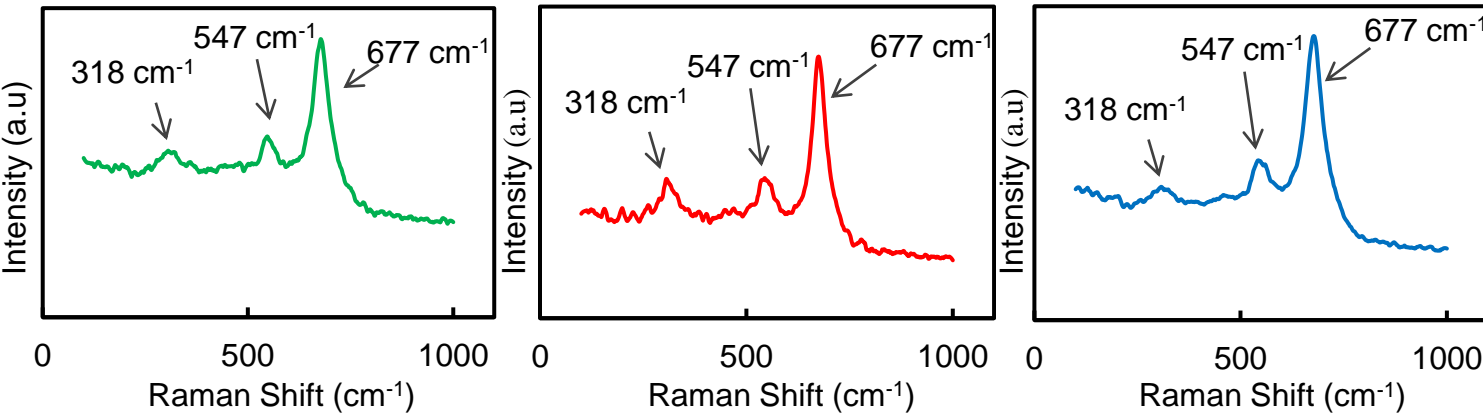
0 cc DH water

30 cc DH water

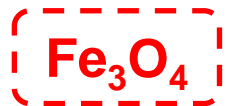
50 cc DH water



Oxide particles formed at different DH level had similar shape. Fe_3O_4 was confirmed by Raman Spectroscopy.

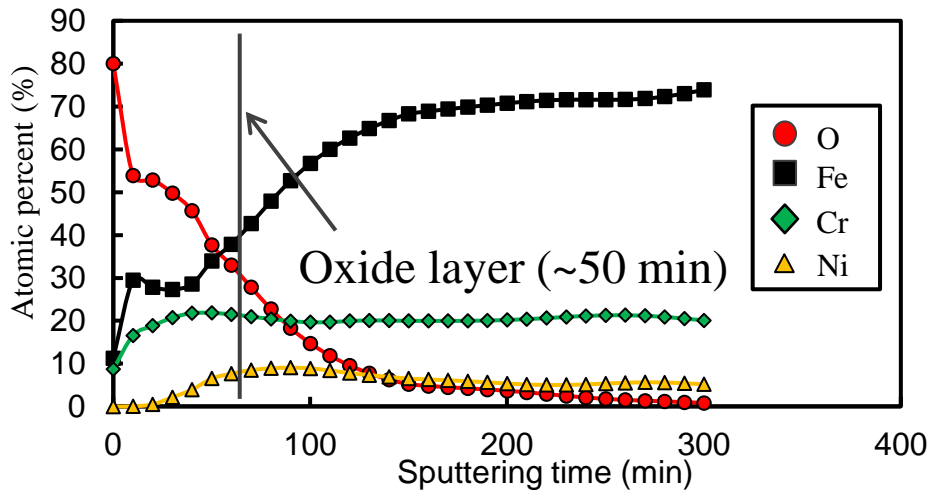


Change of DH level had no significant effect on the oxide morphology.

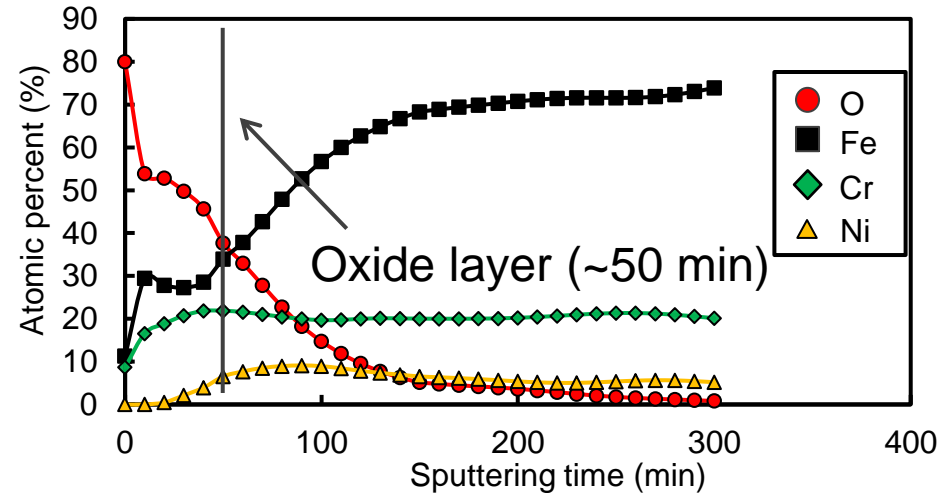


Oxide film characterization

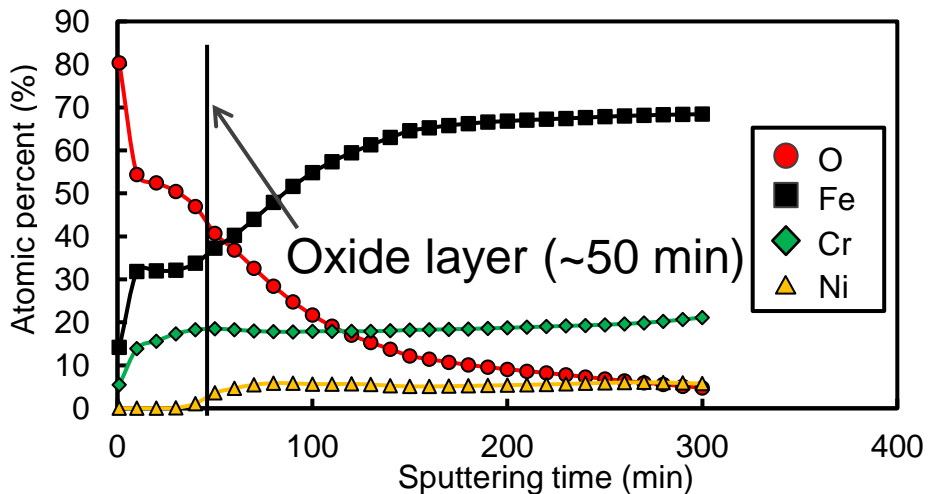
0 cc DH water



30 cc DH water

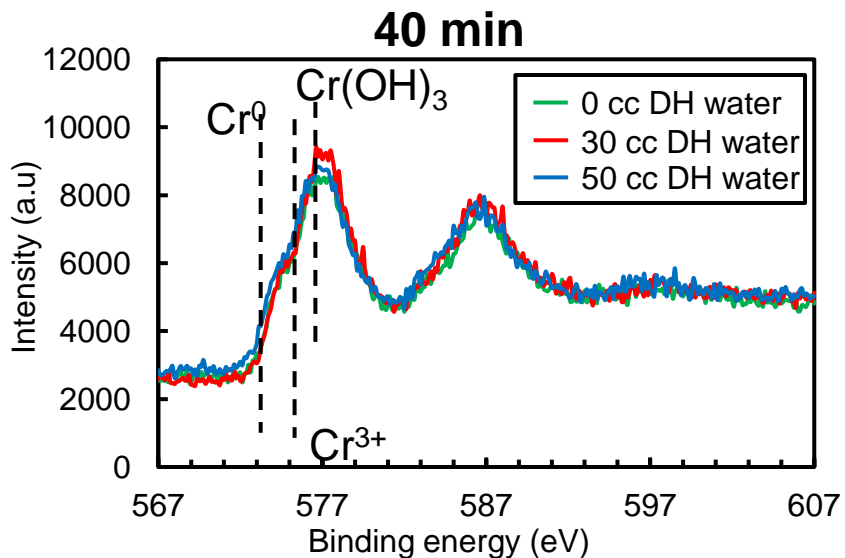
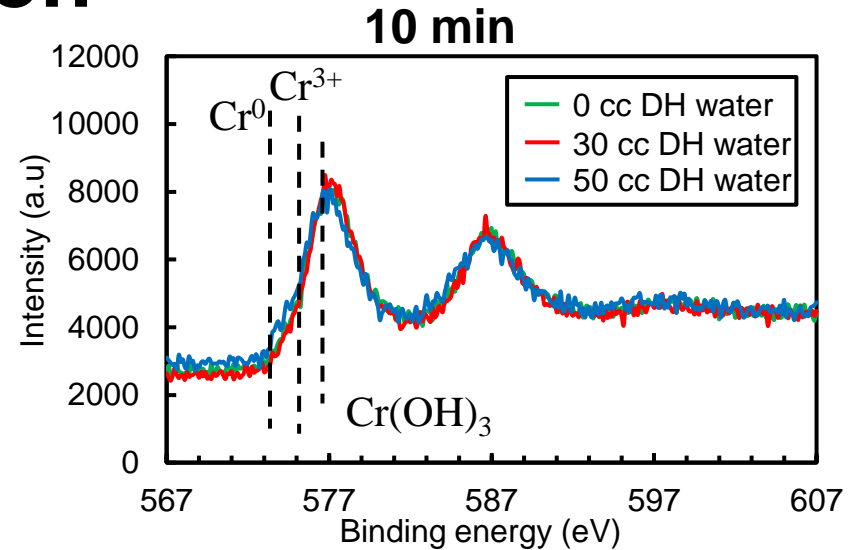
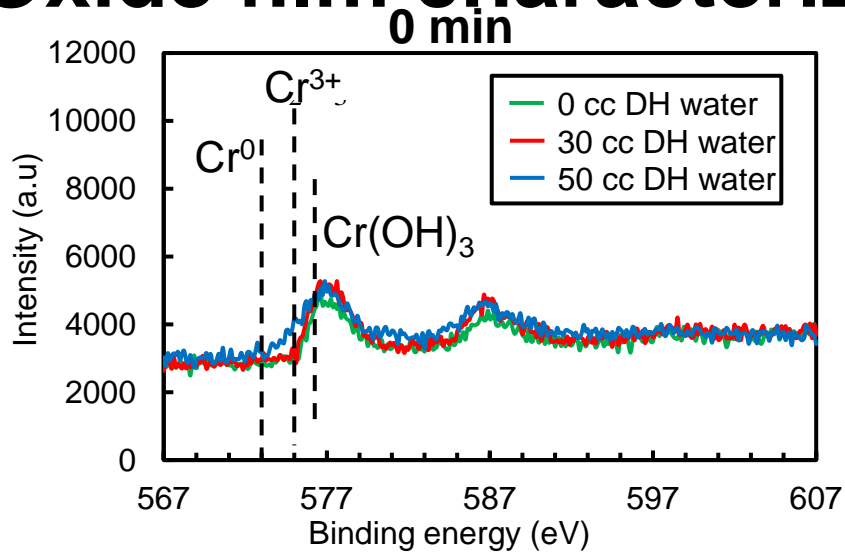


50 cc DH water



Oxide films formed at different DH levels had similar XPS depth profile.

Oxide film characterization



Oxide films formed at different DH levels showed similar Cr 2p spectrums. Combining with the depth profiles, it is considered the change of DH level had no significant effect on the oxide film composition.

Chapter 5: Conclusions

● Key results:

➤ LCF behaviors of 316L/316LN at different DO levels

- The LCF lives of 316L/316LN at different DO levels were different from the data obtained by previous studies. The LCF lives of 316L/316LN in < 5 ppb DO water was shorter than the LCF lives in middle DO water (50 ppb or 100 ppb for 316LN and 50 ppb for 316L) but closed to the LCF lives at high DO levels (2 ppm for 316LN, 1 ppm for 316L). The discrepancy between this study and previous studies was probably caused by the materials used.
- When the DO added was above 5 ppb (50 ppb, 100 ppb and 2 ppm for 316LN; 50 ppb, 1 ppm for 316L), the hydrogen absorption was inhibited, which caused the increase in the LCF lives at these DO levels. However, when the DO added was 2 ppm DO for 316LN or 1 ppm for 316L, even though the hydrogen absorption was inhibited, the crack tip potential, potential gradient between the crack mouth and crack tip were higher, which caused the decrease in the LCF lives at these DO level.

➤ LCF behaviors of 316LN at different DH levels

- Change of DH level did not have significant effects on the LCF life, corrosion behaviors of 316LN SS.
- The amount of hydrogen absorbed into the 316LN slightly increased with increasing DH level. The main source of the hydrogen absorbed into the 316LN is the hydrogen produced by corrosion reaction, instead of the dissolved hydrogen added intentionally.

Chapter 5: Conclusions

● Recommendations:

➤ Recommendations for the optimization of the water chemistry.

- The hydrogen absorbed into the material and the crack tip potential & potential gradient between the crack mouth and the crack tip are considered the factors determining the LCF lives of 316L/316LN SSs. Therefore, adding little DO(50 or 100 ppb in the present case) into the water is beneficial to the 316L/LN SS from the point of view of the LCF, because the adding of the DO can inhibit the hydrogen absorption. However, adding too much DO(2 ppm DO in the present case) will be deleterious to this material as it can increase the potential mentioned.