

Formation of Nanoporous Oxide Layer for SCC Protection on TIG Welded Type 304 Stainless Steel Used in Nuclear Spent Fuel Dry Storage Container

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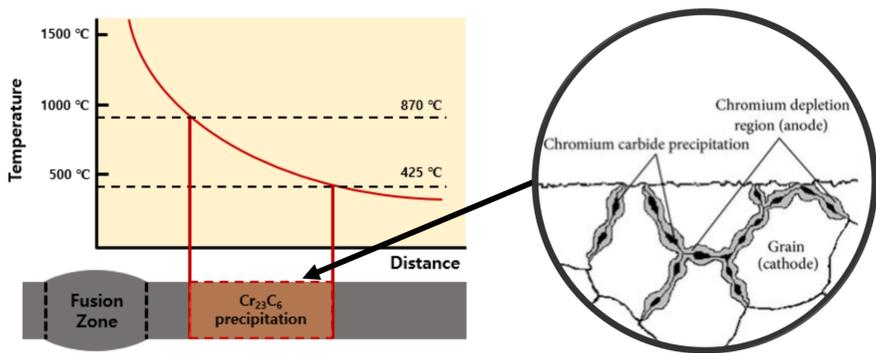
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Introduction

Sensitization of Austenitic stainless steel after welding

- ❖ In specific temperature range (425 ~ 870 °C) chromium carbide precipitates at Heat Affected Zone (HAZ) grain boundary (sensitization)
- ❖ Formation of chromium depleted zone
 - Absence of chromium oriented protective passive film
 - HAZ becomes susceptible of corrosion attack

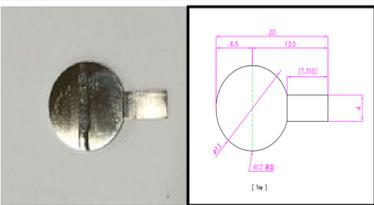


Chloride rich environments & Pitting corrosion

- ❖ Abundant chloride contents at nuclear spent fuel dry storage system which is located near sea coast line
- ❖ Breakdown of protective passive film and chloride rich environments can lead to **pitting corrosion** → **Stress Corrosion Cracking (SCC)** with residual stress of welding

Experimental

Sample Preparation



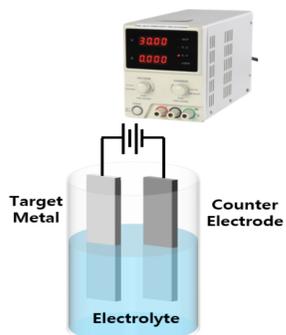
[Electropolished weld 304 SS specimen]

- ❖ Samples were sonicated in acetone followed by deionized water rinsing
- ❖ Dried in oven of 60 °C

Anodization

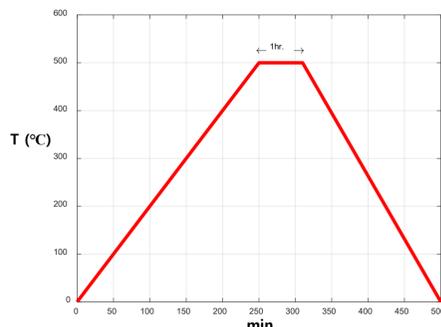
Anodizing condition

- ❖ Cooling bath was used at temperature of 25 °C
- ❖ Target metal: 304 weld SS
- ❖ Counter electrode: Platinum sheet
- ❖ Electrolyte: 0.1 M H₂O + 0.1 M NH₄F in E.G
- ❖ Applied voltage: 50 ~ 60 V
- ❖ Duration: 10 min. ~ 15 min.



Annealing

- ❖ Heat treatment was conducted in order to eliminate fluorine species from anodized sample
- ❖ Heat rate: 2 °C/min.
- ❖ 500 °C for 1 hr.



Electrochemical test

Potentiodynamic control condition

- ❖ Working electrode: 304 SS (surface ~ 1 cm²)
- ❖ Counter Electrode: Graphite rods
- ❖ Reference electrode: Saturated calomel electrode
- ❖ Potential range: -250 ~ 1500 mV
- ❖ Electrolyte: Artificial seawater
- ❖ OCP (open circuit potential) was preconditioned

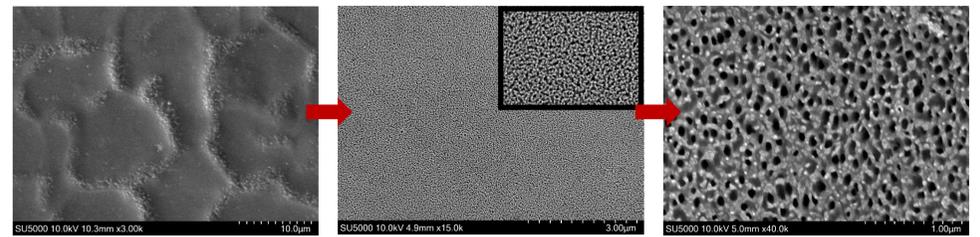


Element	Composition (g/L)
Cl	19.00
Na	9.72
Mg	1.3
S	0.81
Ca	0.40
K	0.35
Sr	0.007
B	0.004

- ❖ Using ISO_15158, samples' pitting potential was averaged through five repetition test
- **Artificial seawater composition**

Results & Analysis

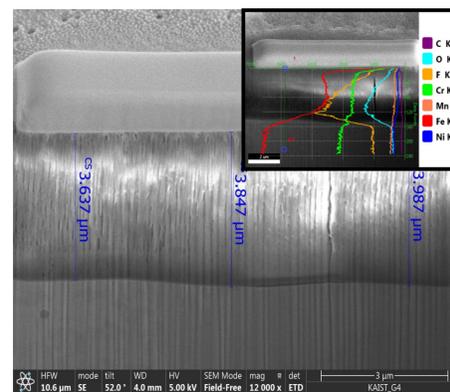
Protective nanoporous oxide layer fabrication



Sensitized weld SS

Anodized weld SS

Anodized weld SS + Heat treated

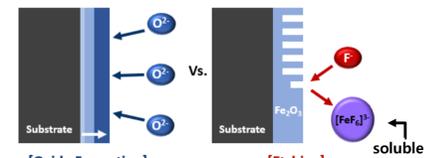


Element	Weight %	Atomic %
C K	1.07	2.28
O K	5.44	8.76
F K	49.72	67.43
Fe L	4.80	2.21
Ni L	0.03	0.01
Cr K	38.94	19.29

Element	Weight %	Atomic %
C K	1.86	4.45
O K	31.72	56.84
F K	3.60	5.43
Fe L	28.41	14.58
Ni L	4.46	2.18
Cr K	29.95	16.51

Fluorine elimination by heat treatment

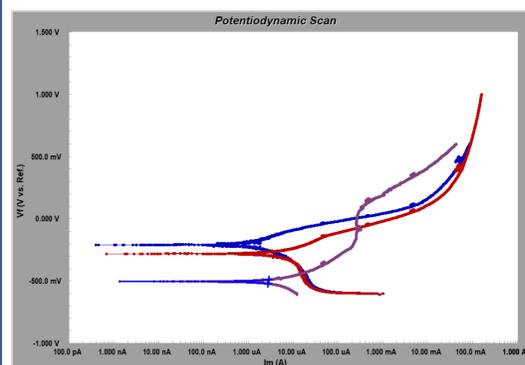
→ To prevent dissolution



Cross sectional image of nanoporous oxide layer

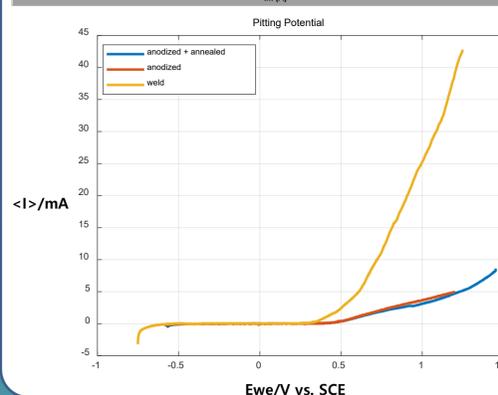
Nanoporous structure formation mechanism

Electrochemical measurements



Specimen	E _{corr} (mV/SCE)	I _{corr} (10 ⁻⁷ A/cm ²)
Weld STS	-504	48.5
Anodized weld STS	-281	8.33
Annealed & Anodized weld STS	-210	4.45

- Corrosion potential and current are improved with anodized and annealed STS
- Overall corrosion resistance improved



Specimen	Average Pitting Potential (mV), V _{c100} (V vs. SCE)
Bare STS	290.3
Weld STS	258.6
Anodized weld STS	397.2
Annealed & Anodized weld STS	373.6

- Anodized samples' pitting potential has been increased about **53%** compared to weld SS
- Pitting resistance improved

Conclusion

- Stainless steel becomes susceptible to corrosion after welding
- Nanoporous protective oxide layer fabrication electrochemical anodization
- Pitting corrosion resistance was improved (pitting potential 53% increased)
- Stress Corrosion Cracking resistance can be improved
- Further corrosion tests should be conducted, various applications expected

References

- ▶ Sourav Kr. Saha _ Self-organized honeycomb-like nanoporous oxide layer for corrosion protection of type 304 stainless steel in an artificial seawater medium
- ▶ ISO15158 _ Method of measuring the pitting potential for stainless steels by potentiodynamic control in sodium chloride solution
- ▶ Yingge Wang _ Fabrication and formation mechanisms of ultra-thick porous anodic oxides film with controllable morphology on type-304 stainless steel