Tensile Properties of Structural Materials Aged in High Temperature S-CO_2 Environment

(Paper #32)

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Sep. 10, 2014

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Contents

1. Introduction

2. Materials & Experimental

3. Results & Discussion

   - Corrosion resistance in S-CO_2
   - Changes in tensile properties after exposure to S-CO_2

4. On-Going Tests

5. Summary

Introduction – Korean PGSFR

❑ Application of supercritical CO_2 power cycle to SFR

❖ Operating condition of Korean PGSFR S-CO_2 cycle

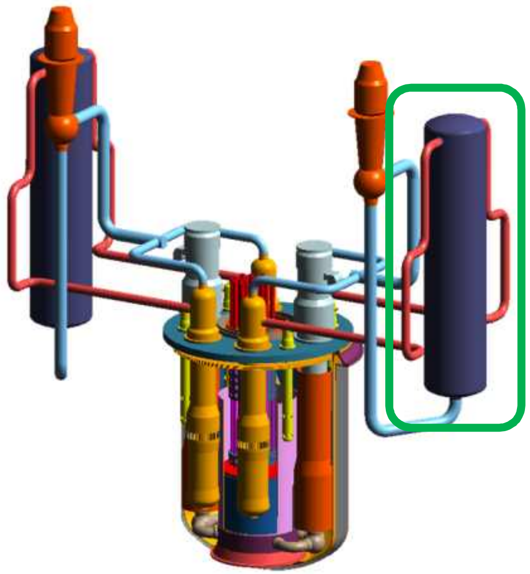
- Brayton cycle using S-CO_2 at 500 - 550 ^oC (200 bar)

❖ Key components

- Intermediate heat exchanger (IHX), Intermediate-loop, and pipes

❖ Materials issues

- Candidate materials: **Fe-base austenitic steels**, **Ni-base alloys**, **FM steels**
- **Corrosion property**, **long-term stability** and **mechanical property in S-CO_2**



▲ Schematic image of PGSFR

▲ SFR IHX by Diffusion Bonding

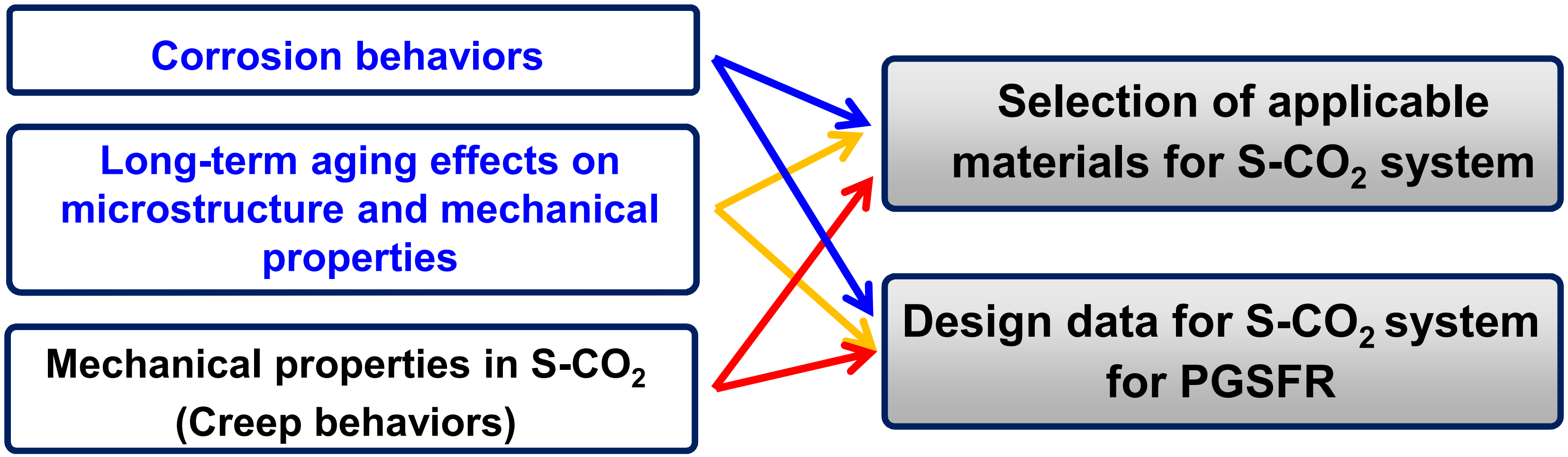
Prototype Gen IV SFR (PGSFR)

- Pool-type reactor
- 150 MWe
- Core in/out T : 390/545 ^oC
- Liquid sodium coolant
- Supreheated steam Rankine cycle

➔ S-CO_2 cycle as an alternative option

Introduction – Material Feasibility of SCO_2 Cycle

❑ Material Compatibility: feasibility of S-CO_2 Cycle to SGR



❖ Evaluation of corrosion property in S-CO_2

- Corrosion test in S-CO_2 environments (550, 600, and 650^{o}C / 200bar for up to 3000h)
- Effects of Cr content, grain size, minor alloying elements at various testing temperatures

❖ Evaluation of aging effects of long-term exposure to S-CO_2

- Exposure of mini-sized tensile specimens to S-CO_2 environment during corrosion tests
- Assessment of microstructural evolution at near the surface and within the matrix
- Assessment of changes in tensile properties compared to the un-aged specimen

Materials & Experimental - Materials

❑ Candidate materials: selected commercial alloys

- Fe-base austenitic steels : 310, 316H, 316LN, 347H, 800HT
- Ni-base alloys : 600, 625, 690
- Ferritic-martensitic steel : G91

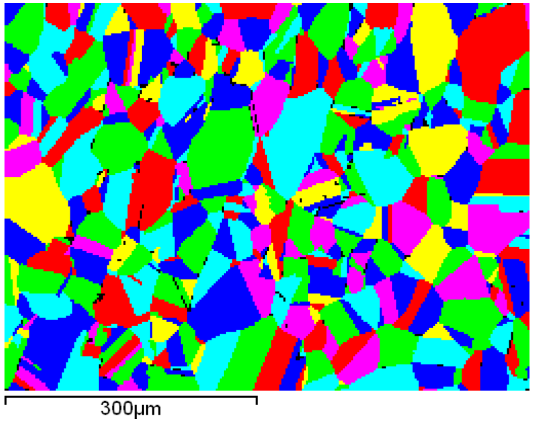
	Fe	Ni	Cr	C	Mo	Mn	Al	Ti	Si	Cu	Others
SS 310S	Bal.	19.1	24.7	0.06	-	0.8	-	-	0.6	-	-
SS 316H	Bal.	10.7	17.3	0.05	2.1	0.6	-	-	0.6	0.2	0.2 Co
SS 316LN	Bal.	13.9	18.9	0.03	2.7	1.9	-	-	0.6	-	0.2 N
SS 347H	Bal.	8.6	18.3	0.07	-	1.2	-	-	0.6	-	0.4 Nb
Incoloy 800HT	Bal.	33.6	21.0	0.06	0.2	0.9	0.48	0.55	0.4	0.1	0.003 B 0.05 Co
Inconel 600	9.3	Bal.	16.1	0.08	-	0.3	0.16	0.20	0.3	0.02	0.002 B
Inconel 625	4.6	Bal.	22.6	0.02	9.8	.04	0.3	0.35	-	-	3.1 Nb 0.15 Ta
Inconel 690	8.3	Bal.	28.4	0.02	-	0.2	0.3	0.26	0.2	0.01	0.002 B < 0.01 Nb
G91	Bal.	0.1	9.3	0.08	0.9	0.3	0.03	-	0.3		0.19 V 0.08 Nb

▲ Measured by Inductively Coupled Plasma (ICP) method

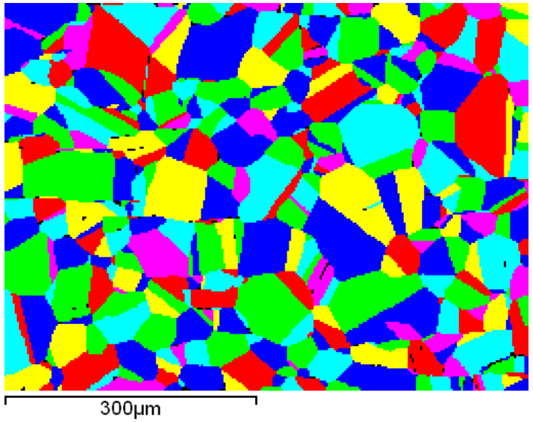
Materials & Experimental - Materials

❑ Average grain size of test materials (EBSD analysis)

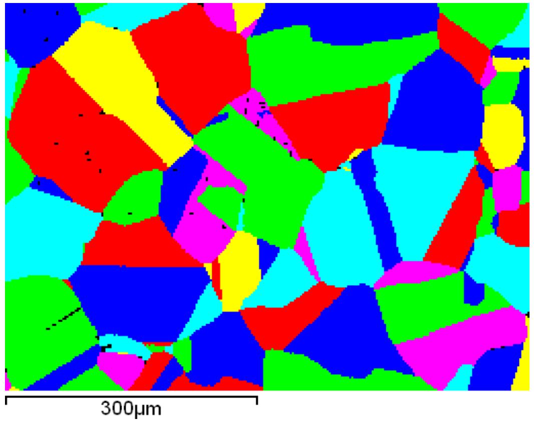
SS 310S (28 μm)



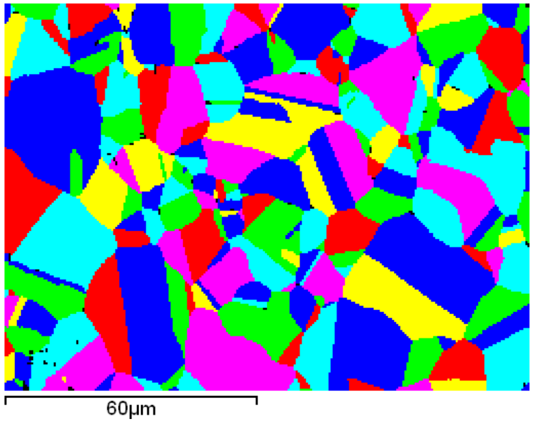
SS 316H (30 μm)



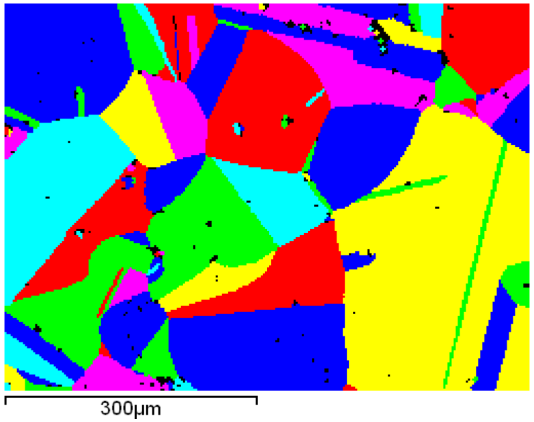
SS 316LN (63 μm)



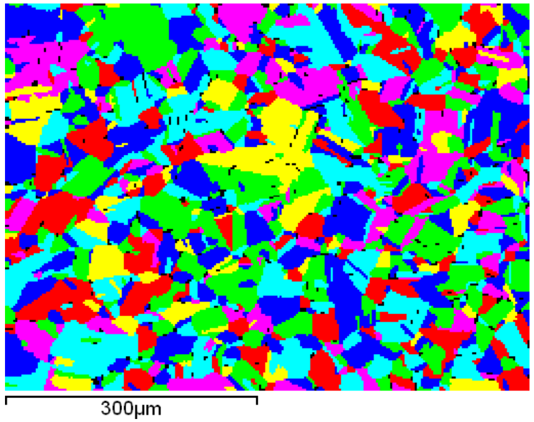
SS 347H (10 μm) →FG



Incoloy 800HT (65 μm)



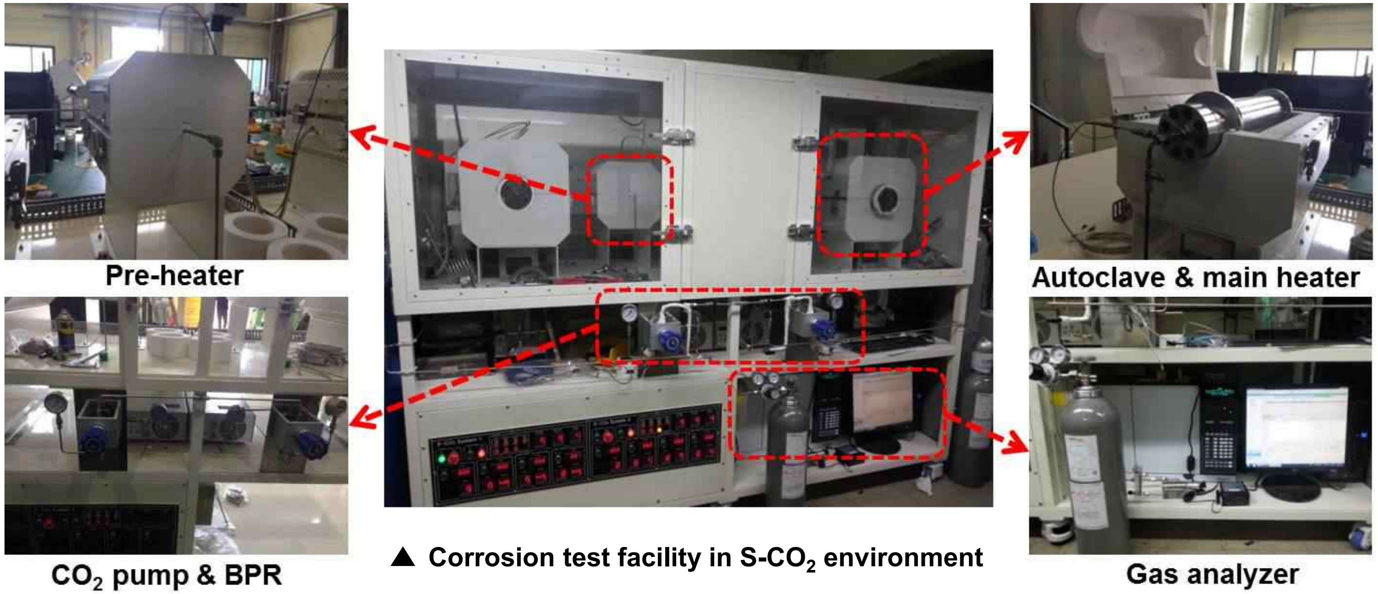
Inconel 600 (21 μm)



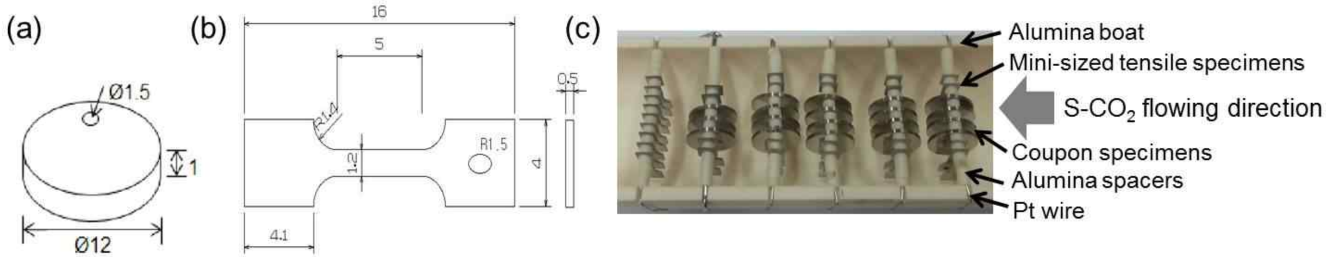
Materials & Experimental – Test Setup

❑ $S\text{-}CO_2$ corrosion test facility

- Once-through, two autoclaves (Alloy 625)
- CO_2: 99.999 % purity liquid phase CO_2 with flow rate of 5 ml/min
- Furnace heating for 5 h → maintain the temperature → furnace cooling



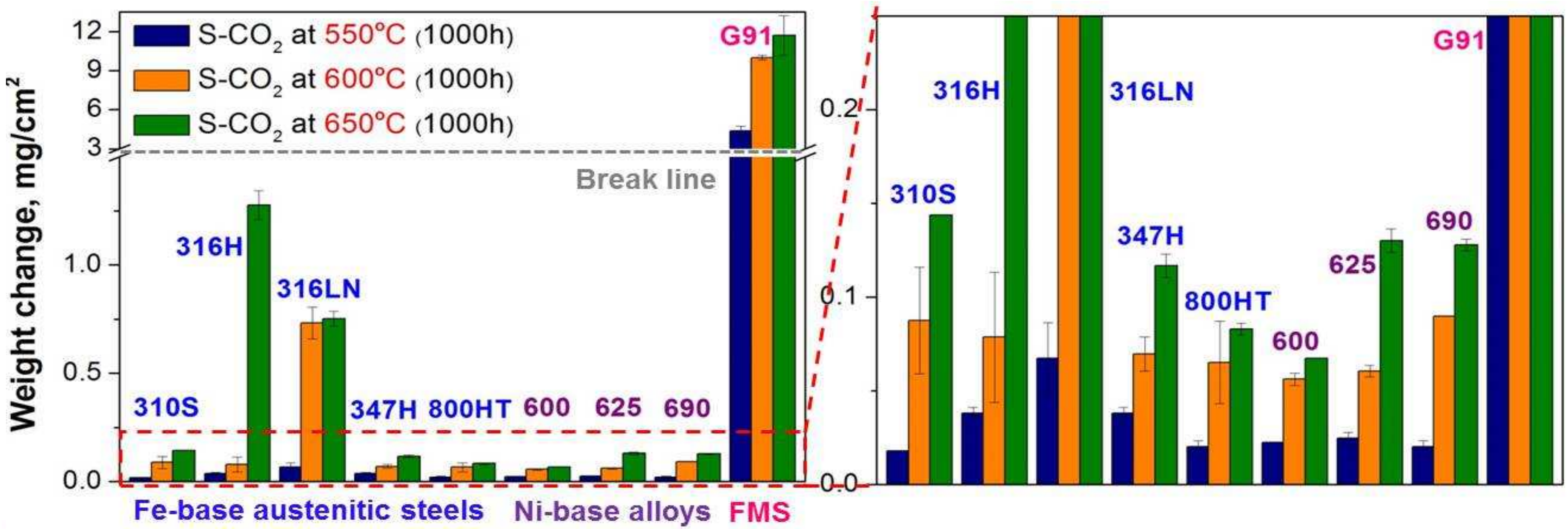
▲ Corrosion test facility in $S\text{-}CO_2$ environment



▲ Geometry and dimensions of (a) coupon and (b) mini-sized tensile specimen (in mm) and the photograph of installed specimens on alumina boat

Results - Corrosion resistance in S-CO_2

- **Corrosion resistance in S-CO_2 : 550, 600 and 650°C (200bar) for 1000h**
  - **Fe-base austenitic steels** : Fe-base with various Cr content (17-25 wt.% Cr)
    - 310S, 347H, and 800HT : relatively good up to 650°C → high Cr (25 Cr in 310S), small grain size (18Cr in FG-347H), high Ni and Cr (33 Ni and 21 Cr in 800HT)
    - 316H and 316LN : depend on exposure temperature → significant weight gain of 316H at 650°C and 316LN above 600°C
  - **Ni-base alloys** : superior corrosion resistance up to 650°C
  - **9Cr FMS** : poor corrosion resistance even at 550°C (Fe-base with low Cr content)

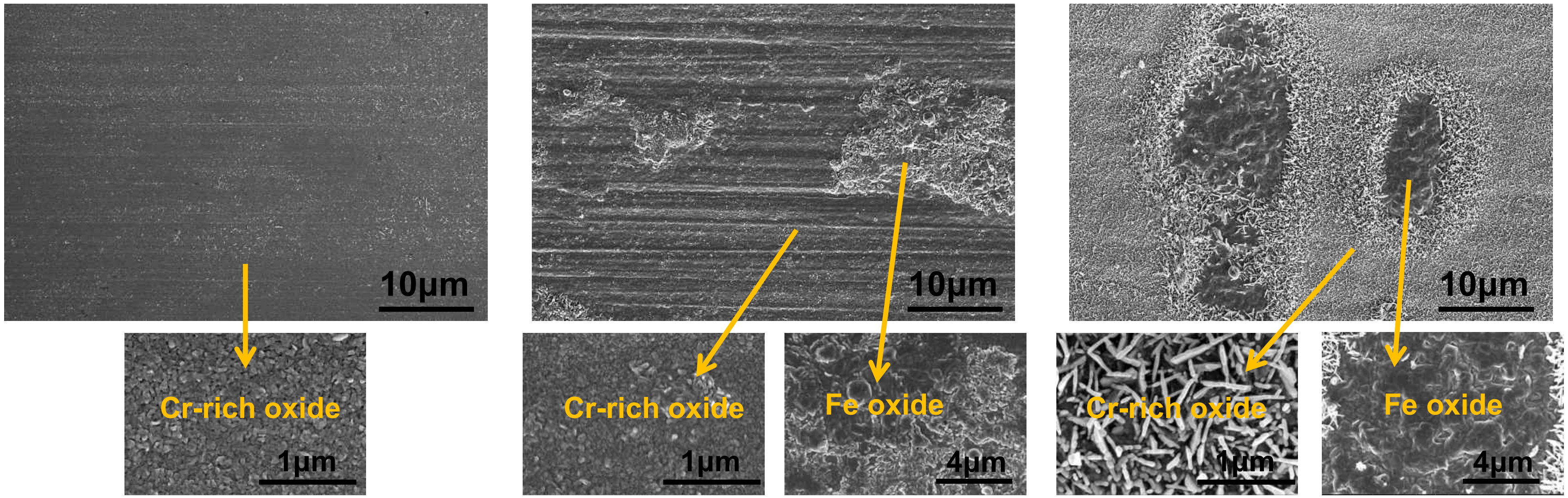


▲ Weight gain for 550, 600 and 650°C (200bar) for 1000h in S-CO_2

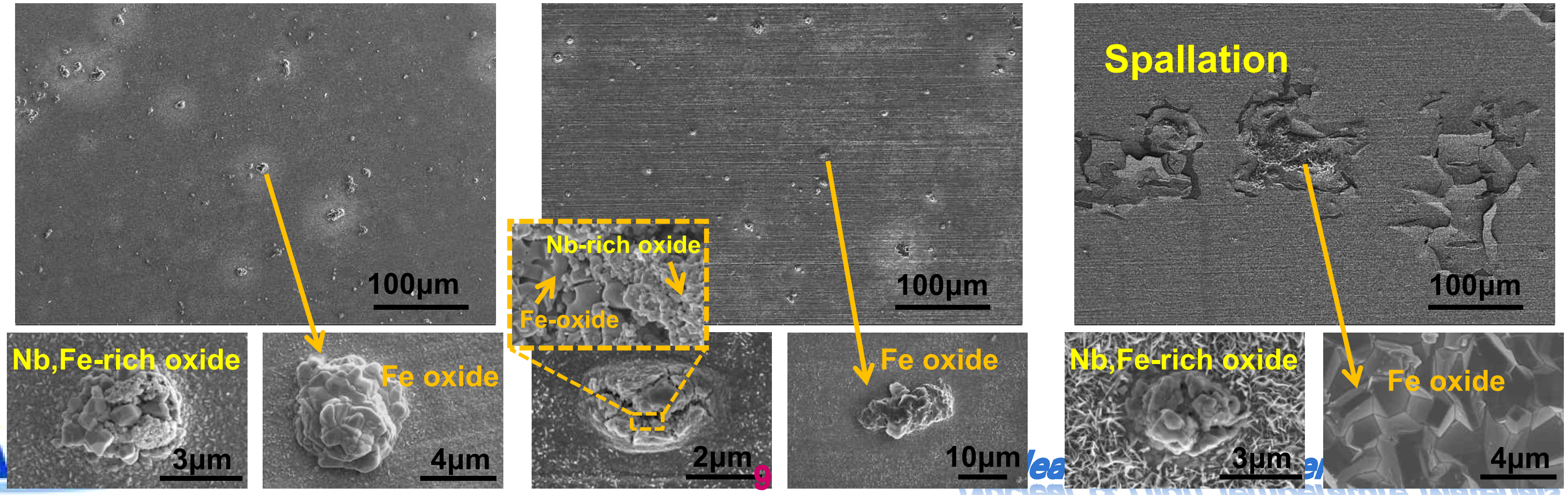
Results – Surface oxides formed in S-CO_2

- Surface oxide morphology exposed in S-CO_2 for 1000h

SS 310S 550°C 600°C 650°C



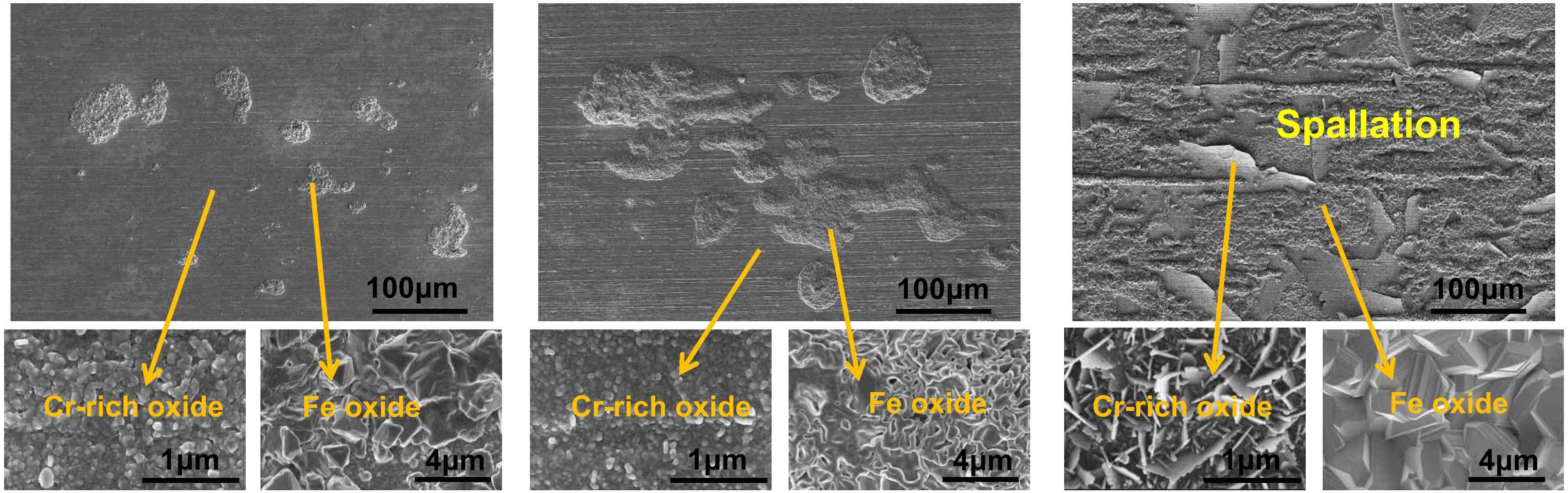
SS 347H 550°C 600°C 650°C



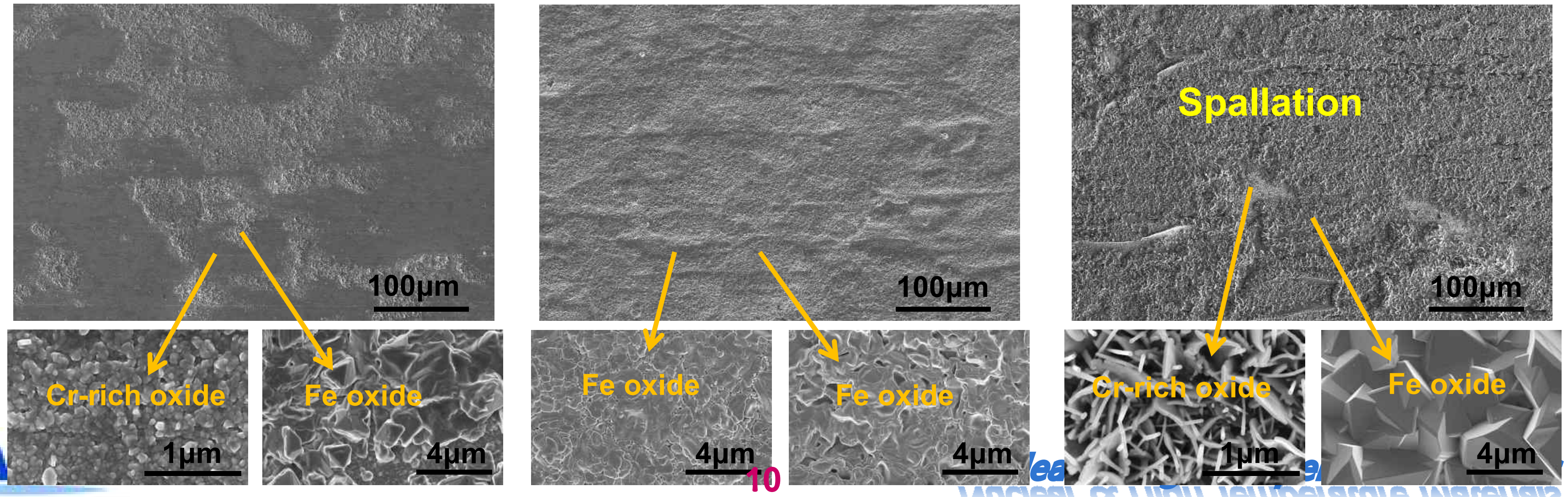
Results – Surface oxides formed in S-CO_2

- Surface oxide morphology exposed in S-CO_2 for 1000h

SS 316H — 550°C, 600°C, 650°C



SS 316LN — 550°C, 600°C, 650°C

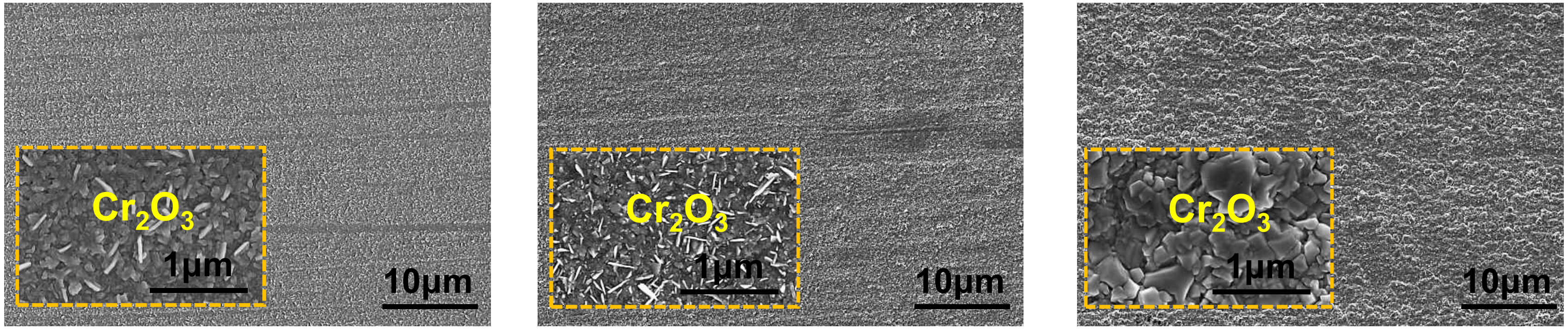


Results – Surface oxides formed in S-CO_2

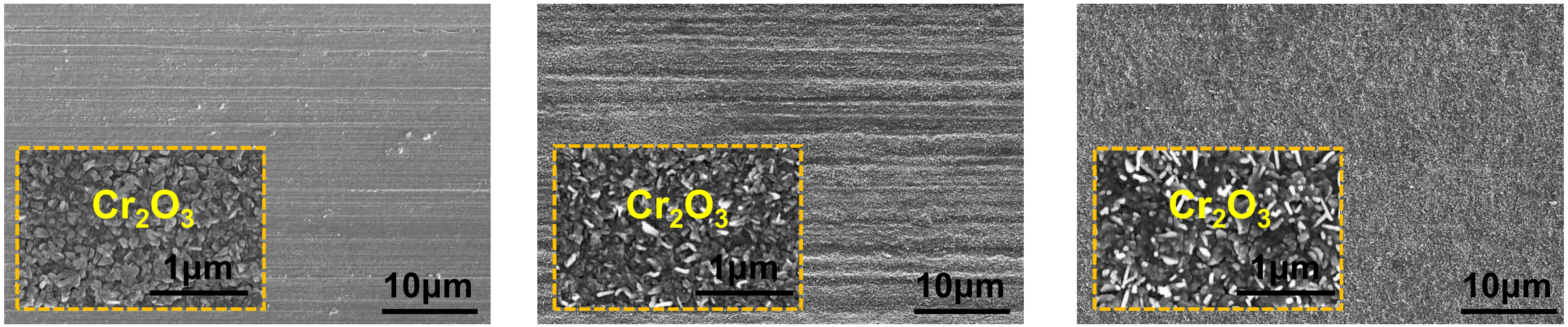
❑ Surface oxide morphology exposed in S-CO_2 for 1000h

- For Incoloy 800HT, Inconel 600, Inconel 625, Inconel 690, Cr_2O_3 is mainly formed in all test conditions → **Superior corrosion resistance**

Incoloy 800HT 550°C 600°C 650°C

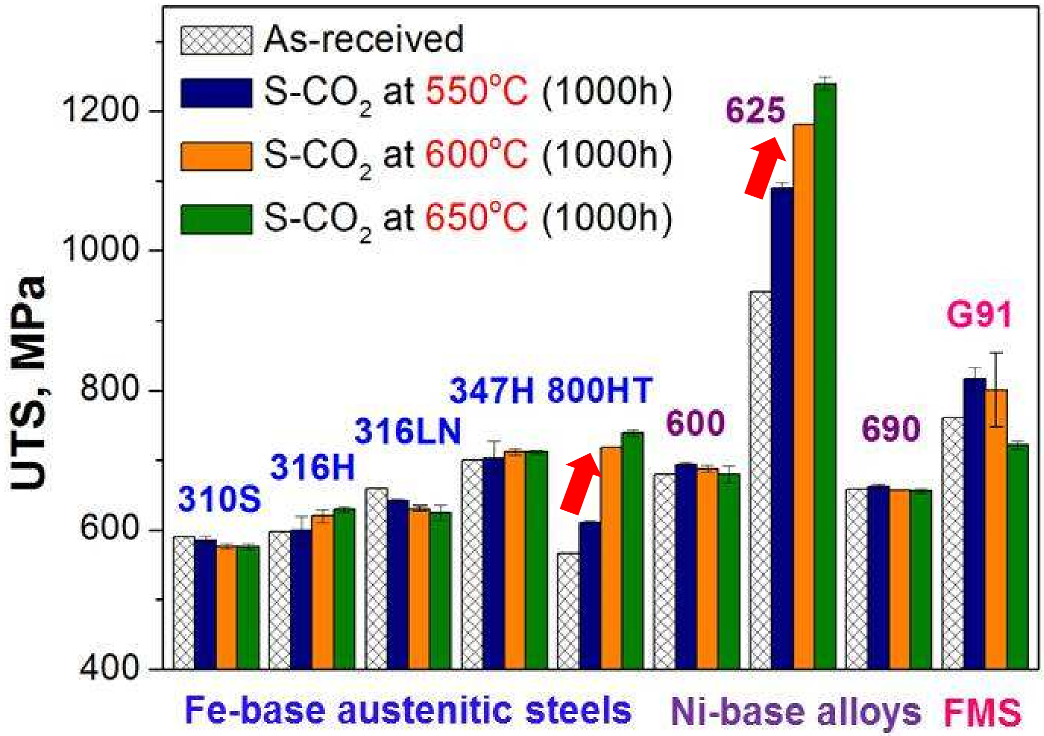


Inconel 600 550°C 600°C 650°C



Results – Tensile Property after exposure in S-CO_2

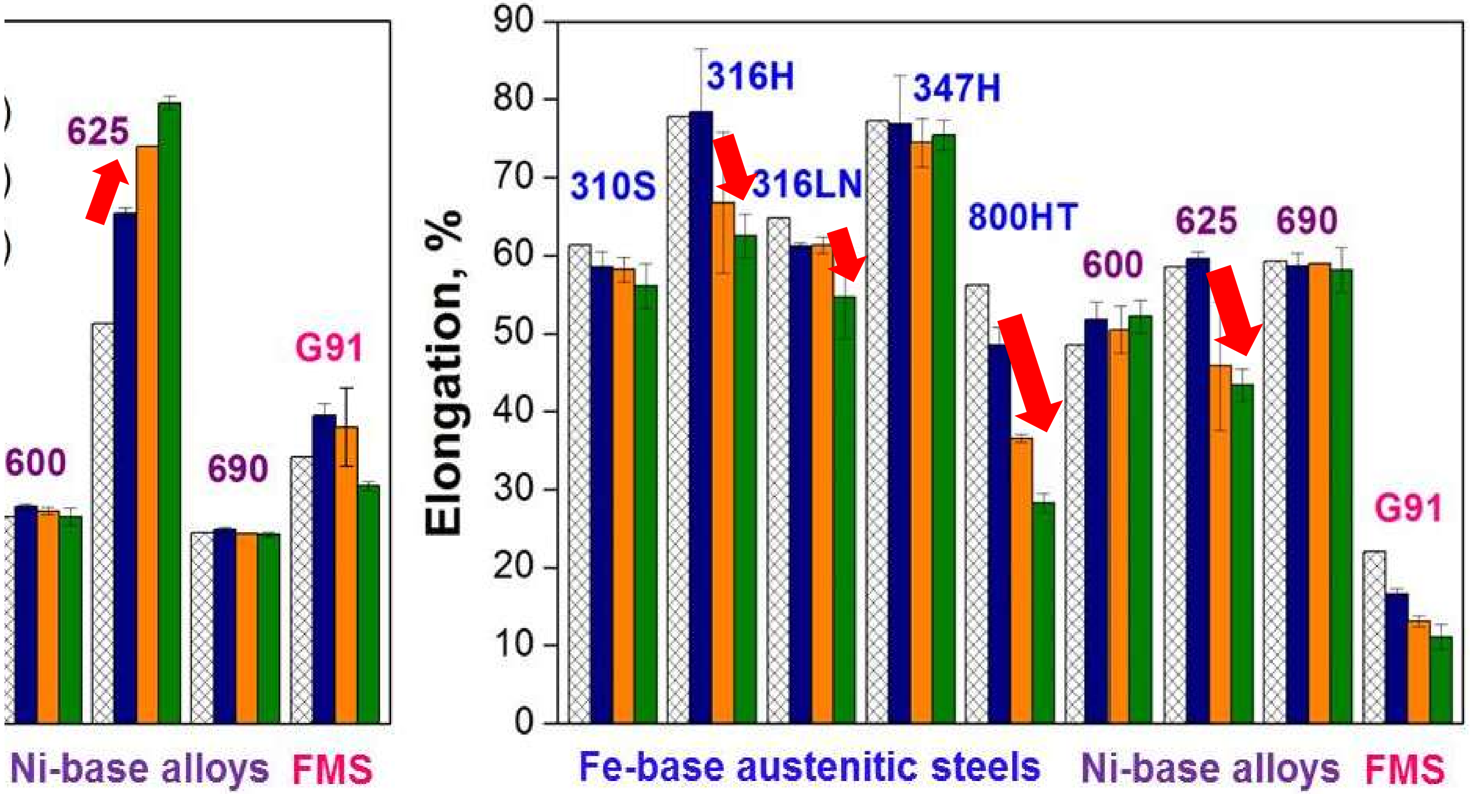
- **Changes in UTS (at RT) after exposure to S-CO_2**
  - **Mini-size tensile specimen (0.5t) contain the corroded surface and aged matrix**
  - **Fe-base austenitic steels : significant** hardening for 800HT, small changes for 316H (+), 347H (+) and 310S (-), 316LN (-)
  - **Ni-base alloys :** Significant hardening for Alloy 625, little change for Alloy 600 & 690
  - **G91 FMS :** loss of strength as exposure Temp. increase



▲ The changes in tensile properties at room temperature after exposure in S-CO_2

Results – Tensile Property after exposure in S-CO_2

- **Changes in Elongation (at RT) after exposure to S-CO_2**
  - **Fe-base austenitic steels :** generally, loss of ductility.
    - ✓ Large decrease for 800HT, 316H, 316LN.
  - **Ni-base alloys :** Little change for Alloy 600 & 690, large decrease for Alloy 625
  - **G91 FMS :** loss of ductility as exposure Temp. increase

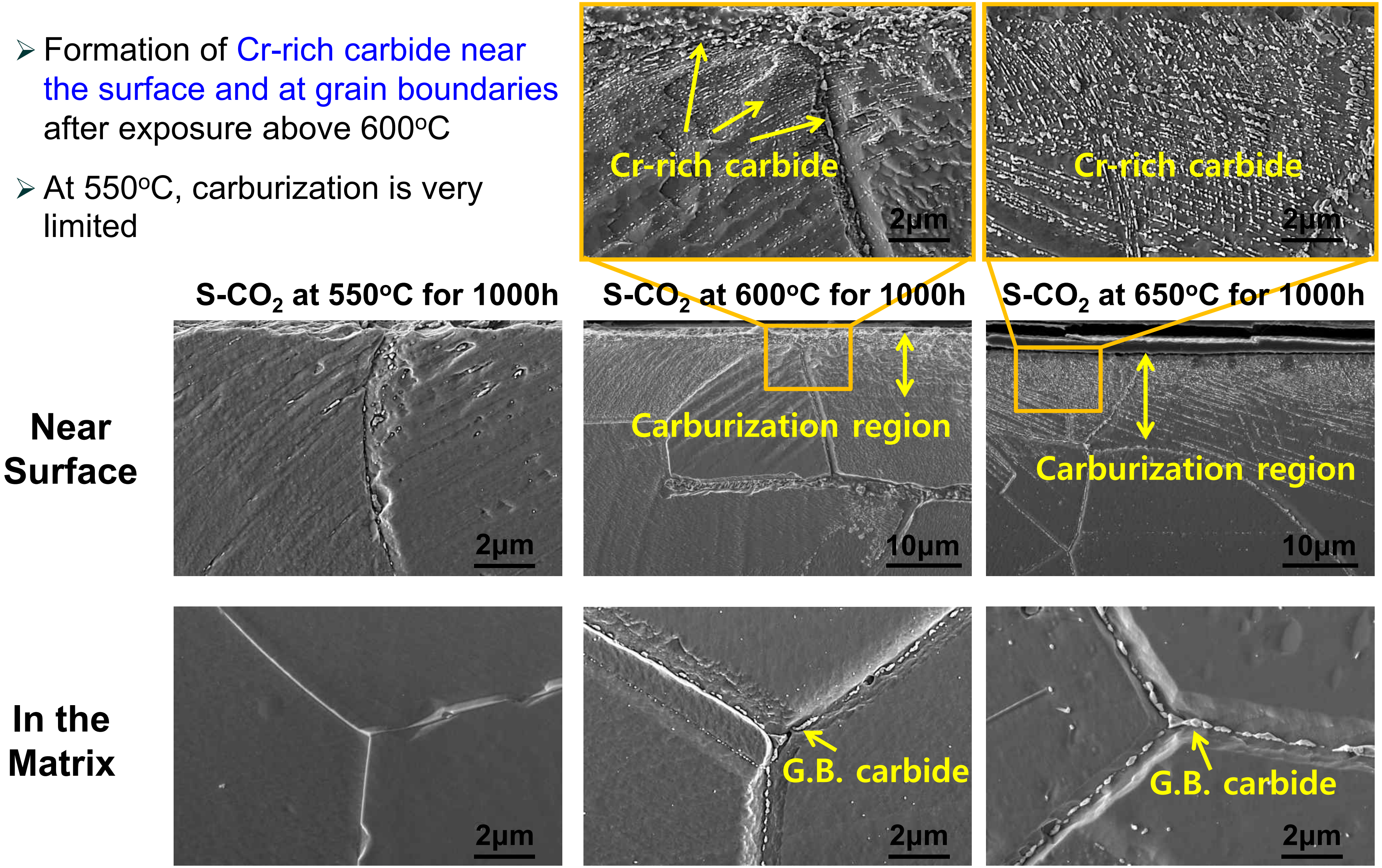


▲ The changes in tensile properties at room temperature after exposure in S-CO_2

Results – Microstructure of Specimen aged in SCO_2

❑ Microstructure of SS 316H after exposure to S-CO_2

- Formation of Cr-rich carbide near the surface and at grain boundaries after exposure above 600°C
- At 550°C, carburization is very limited

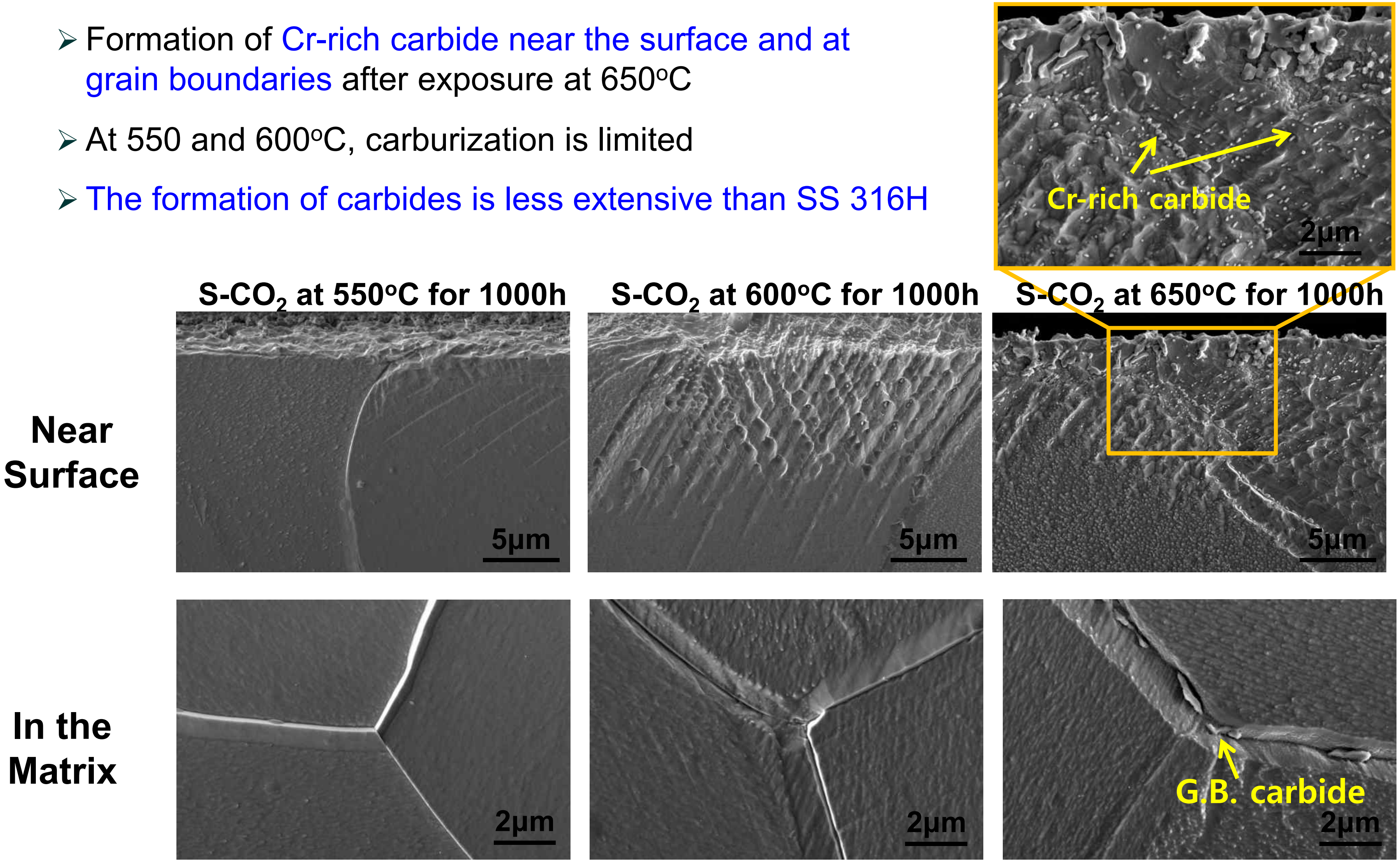


▲ Microstructure evolution of SS 316H after exposure in S-CO_2 at 550, 600 and 650°C (200bar) for 1000h

Results – Microstructure of Specimen aged in SCO_2

Microstructure of SS 316LN after exposure to $S\text{-}CO_2$

- Formation of Cr-rich carbide near the surface and at grain boundaries after exposure at 650°C
- At 550 and 600°C, carburization is limited
- The formation of carbides is less extensive than SS 316H

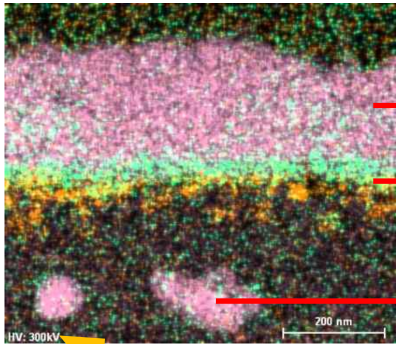


▲ Microstructure evolution of SS 316LN after exposure in $S\text{-}CO_2$ at 550, 600 and 650°C (200bar) for 1000h

Results – Microstructure of Specimen aged in SCO_2

❑ Microstructure of Incoloy 800HT after exposure to $S\text{-}CO_2$

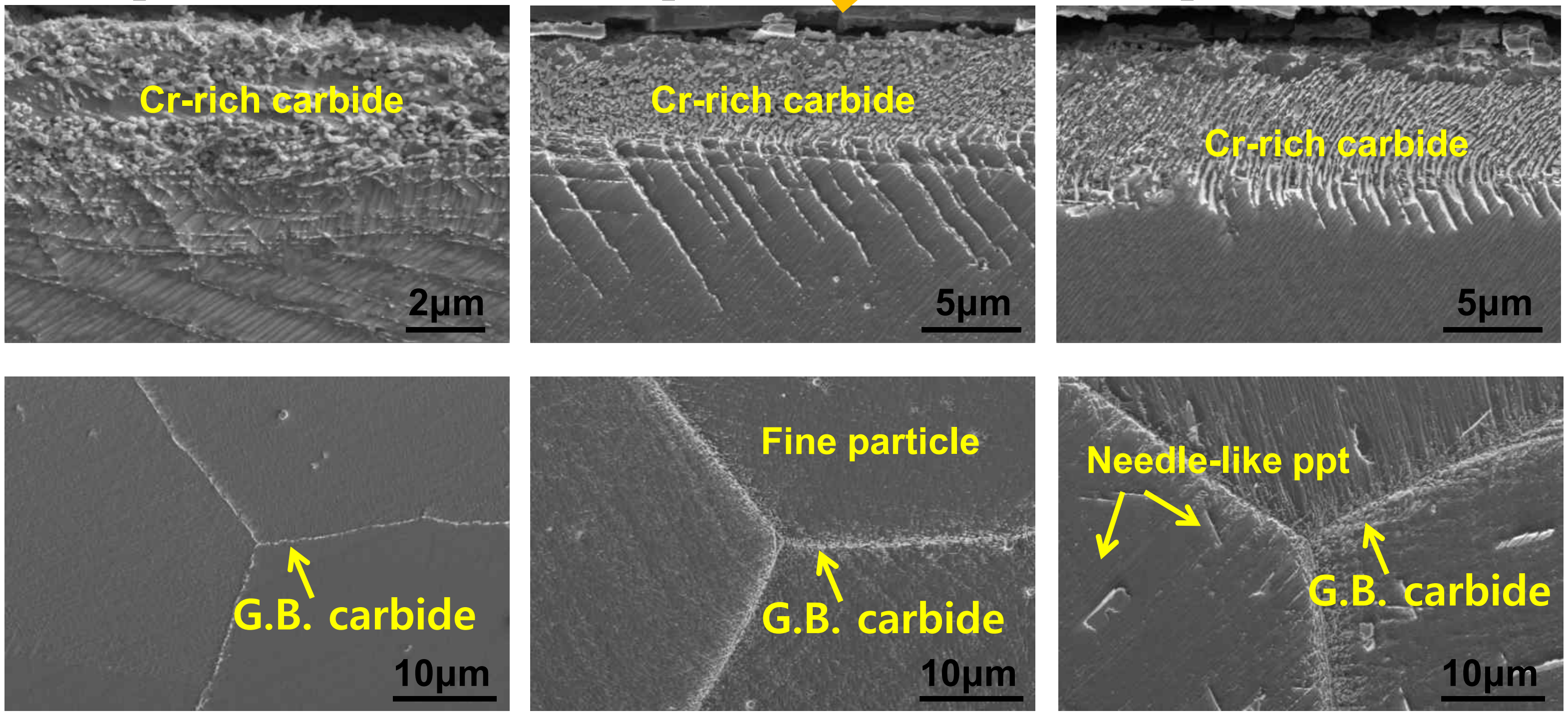
- Significant formation of Cr-rich carbide near the surface and at grain boundaries even after exposure at 550°C
- Fine and needle-like ppt within the grains



S-CO$_2$ at 550°C for 1000h | S-CO$_2$ at 600°C for 1000h | S-CO$_2$ at 650°C for 1000h

Near Surface

In the Matrix

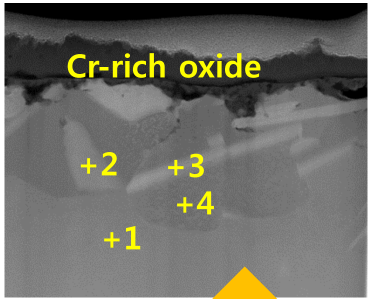


▲ Microstructure evolution of Incoloy 800HT after exposure in S-CO$_2$ at 550, 600 and 650°C (200bar) for 1000h

Results – Microstructure of Specimen aged in SCO_2

❑ Microstructure of Alloy 625 after exposure to $S\text{-}CO_2$

- Formation of Ni, Mo, and Nb-rich phases near the surface after exposure at 600°C
- Grain boundary carbides after exposure to 650°C

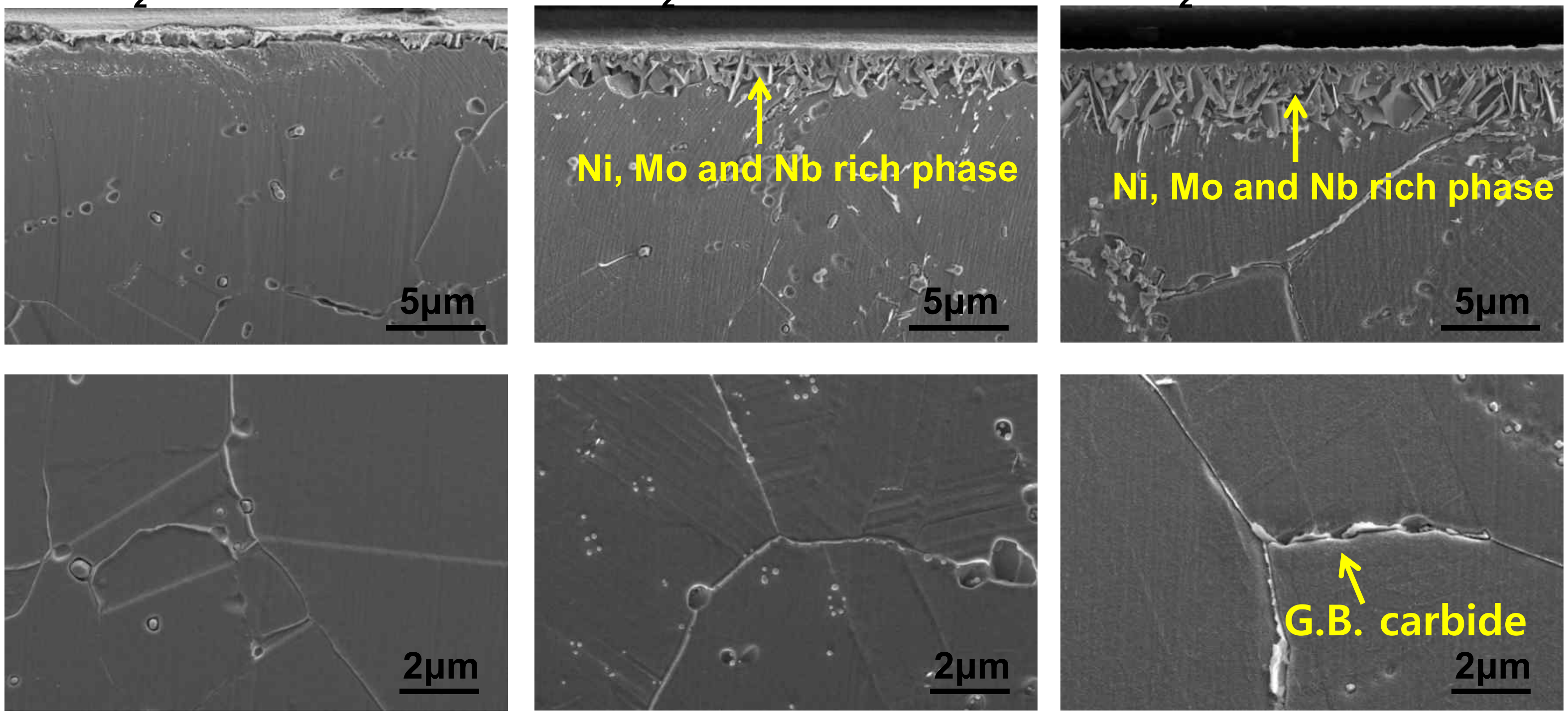


	1	2	3	4
C	1.0	0	0.08	0
Cr	22.3	8.5	5.4	15.9
Fe	5.5	2.9	1.8	6.9
Ni	56.4	61.5	58.7	67.0
Mo	11.2	17.8	21.2	8.3
Nb	3.4	9.2	12.8	1.9

$S\text{-}CO_2$ at 550°C for 1000h | $S\text{-}CO_2$ at 600°C for 1000h | $S\text{-}CO_2$ at 650°C for 1000h

Near Surface

In the Matrix

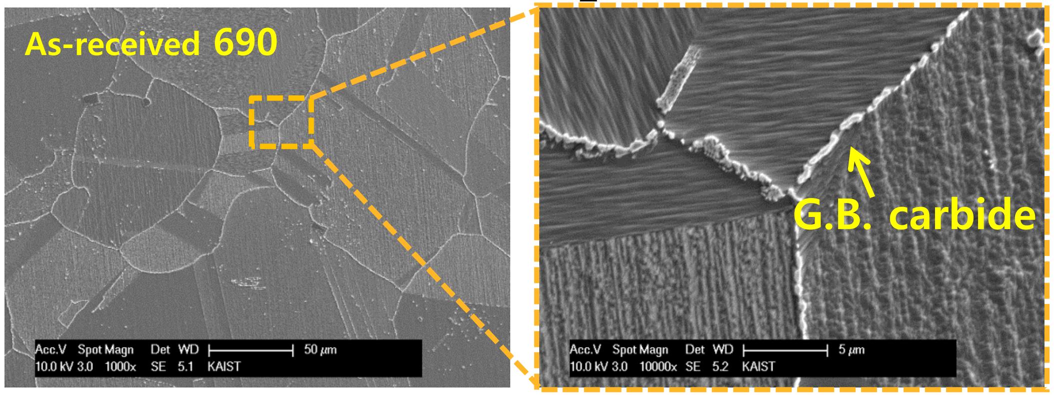


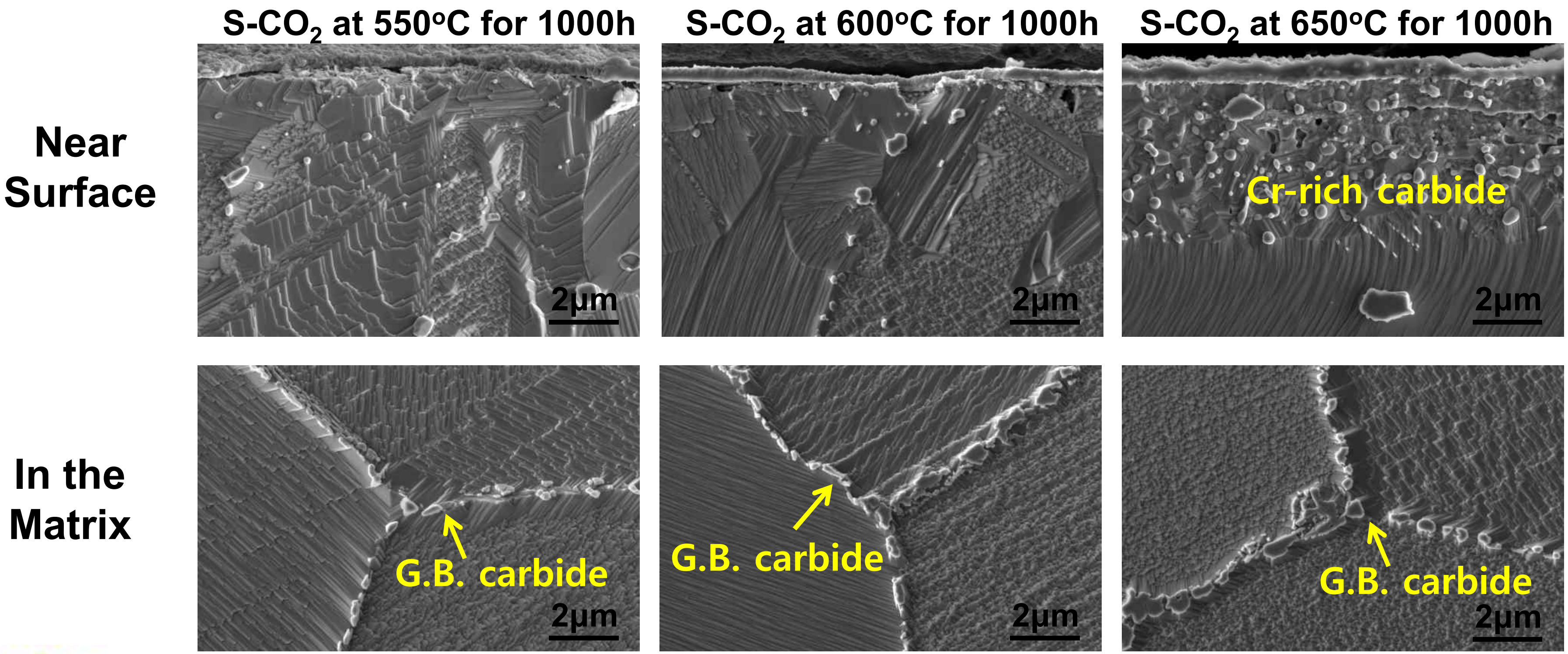
▲ Microstructure evolution of Inconel 625 after exposure in $S\text{-}CO_2$ at 550, 600 and 650°C (200bar) for 1000h

Results – Microstructure of Specimen aged in SCO_2

❑ Microstructure of Alloy 690 after exposure to $S\text{-}CO_2$

- Carbides are present at the grain boundary and within the grains in as-received condition
- Carbide formation is not substantial even after exposure at 650°C





▲ Microstructure evolution of Inconel 690 after exposure in $S\text{-}CO_2$ at 550, 600 and 650°C (200bar) for 1000h

On-Going Tests

- **S-CO_2 corrosion tests up to 3000h (550, 600, and 650°C/200bar)**
  - Evaluate the oxidation kinetics, oxide layer evolution
  - Characterize the carburization behaviors
  - Develop life prediction models

- **Microstructure and tensile property after exposure to S-CO_2**
  - Evaluation of microstructural evolution after long-term exposure to S-CO_2
    → TEM/SAD analysis of 1000h tested specimens is in progress
  - Evaluation of changes in tensile property after 3000h exposure
  - Evaluation of creep properties in S-CO_2 environments (600 °C)

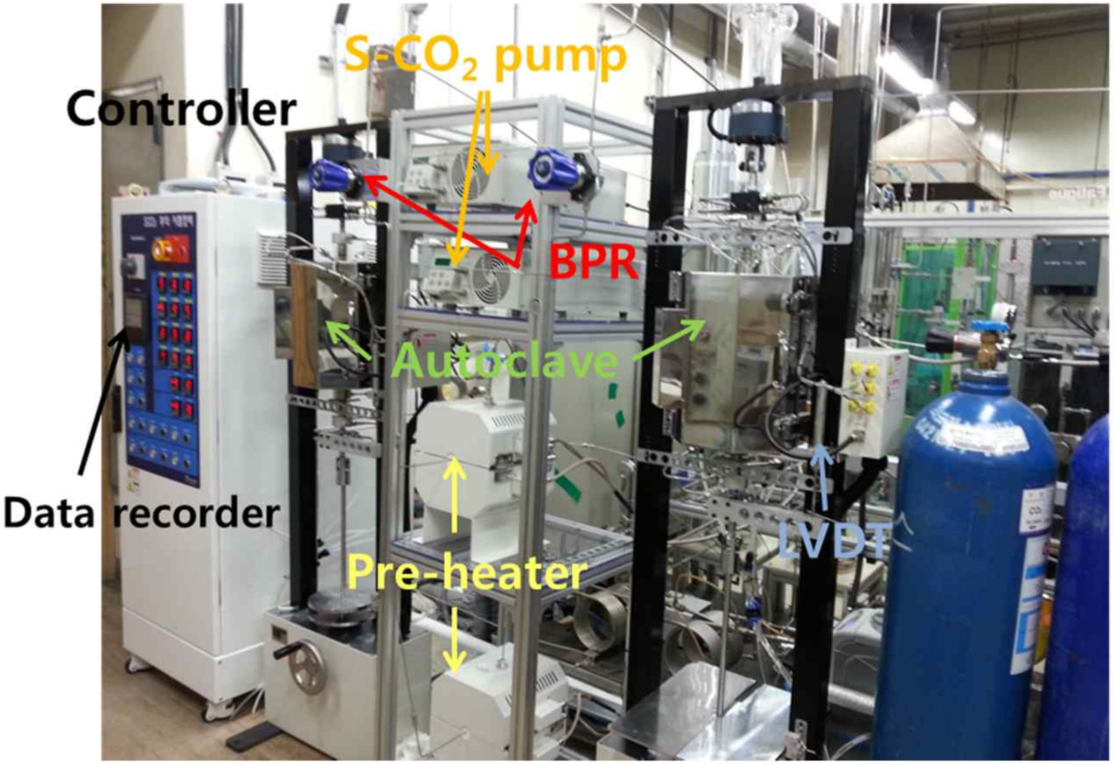
  → Evaluate the effects of aging in S-CO_2 on mechanical properties

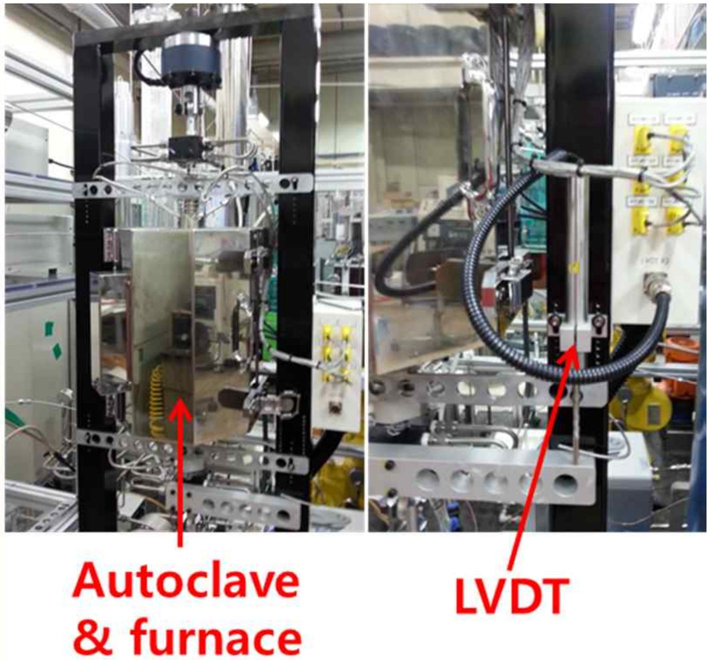
On-Going Tests

❑ Creep in S-CO_2 environment

➢ Evaluation of creep behaviors in S-CO_2

- Dead-weight type creep test setups
- Measure creep strain (LVDT) and creep rupture time at 600 ^oC





Summary

1. The weight gains

- FMS >> Fe-base austenitic steels > Ni-base alloys.
- At 550°C, both Fe-base austenitic steels and Ni-base alloys show a good corrosion resistance => maybe OK for SFR S-CO_2 environment.

2. Surface oxides

- In the case of Fe-base austenitic steels, the surface oxides consist of the mixture of polygonal shaped Fe oxide and nodular (550 and 600°C) or platelet (650°C) shaped Cr-rich oxide.

3. The corrosion resistance of **SS 316H** and **SS 316LN** are strongly dependent on test temperatures resulted from the formation of thick outer Fe oxide.

4. **The tensile properties after exposure to S-CO_2**

- **Incoloy 800HT** and **Alloy 625** → large hardening and loss of ductility probably due to the formation of Cr-rich carbides and Ni, Mo, and Nb-rich phases
- **SS 310S**, **SS 347H**, **Alloy 600**, and **690** → changes in tensile properties such as UTS and elongation are rather small in all test conditions.

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Thank you!