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Corrosion behaviours of austenitic steel and ferritic steel in LBE with oxygen controlled conditions

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Outline

1. Motivations
2. Experiment
3. Experiment Results
4. Analysis and Discussion
5. Summary

Research motivations

- For the development and application of Lead-bismuth cooled fast reactor and ADS system.
- To acquire corrosion data of structural materials in lead-bismuth alloy-for materials selection.
- To illustrate the corrosion behaviours of structural materials in lead-bismuth alloy, which shows the way to conduct materials protection in a LBE system.

Experiment

Experiment sample and facility

- Lead-bismuth alloy: Pb-Bi Eutectic (124°C melting point)
- Structural materials:

  F-M steel: T91

  Austenitic steels:

  CN-1515: Cladding materials for CFR-600
  316Ti: 1st Generation cladding material for CEFR

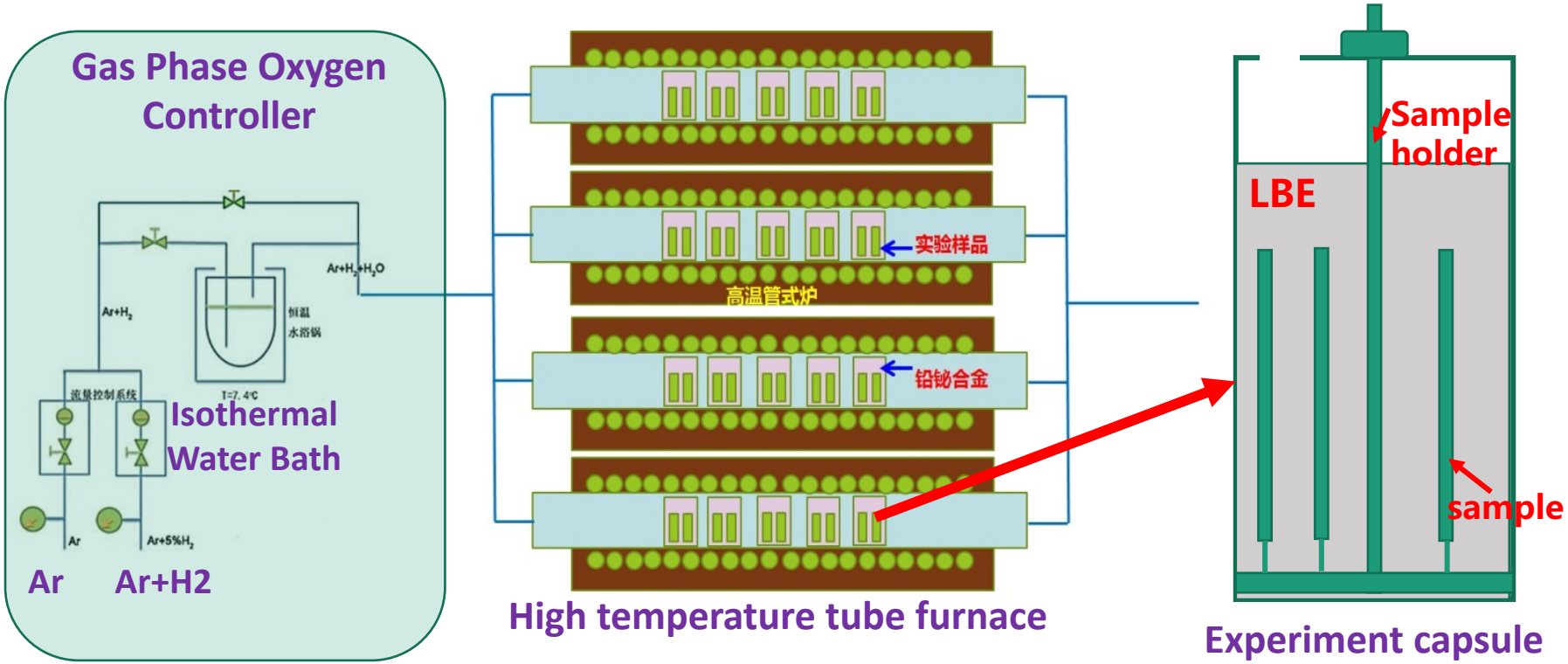
  CFR-600 and CEFR are both sodium cooled reactors
- Sample size:

  20×10×2mm in three direction

Experiment

Experiment samples and facility

- Each capsule contains three specimens and LBE about 240g, and punched holes on the top of capsules.



Experiment

- **Experiment sample and facility**

Experiment

Experiment Condition

- Temperature: 450, 500, 550 and 600℃
- Time: 1000h and 3000h
- Oxygen Concentration: 10^{-6}~10^{-7} wt.%

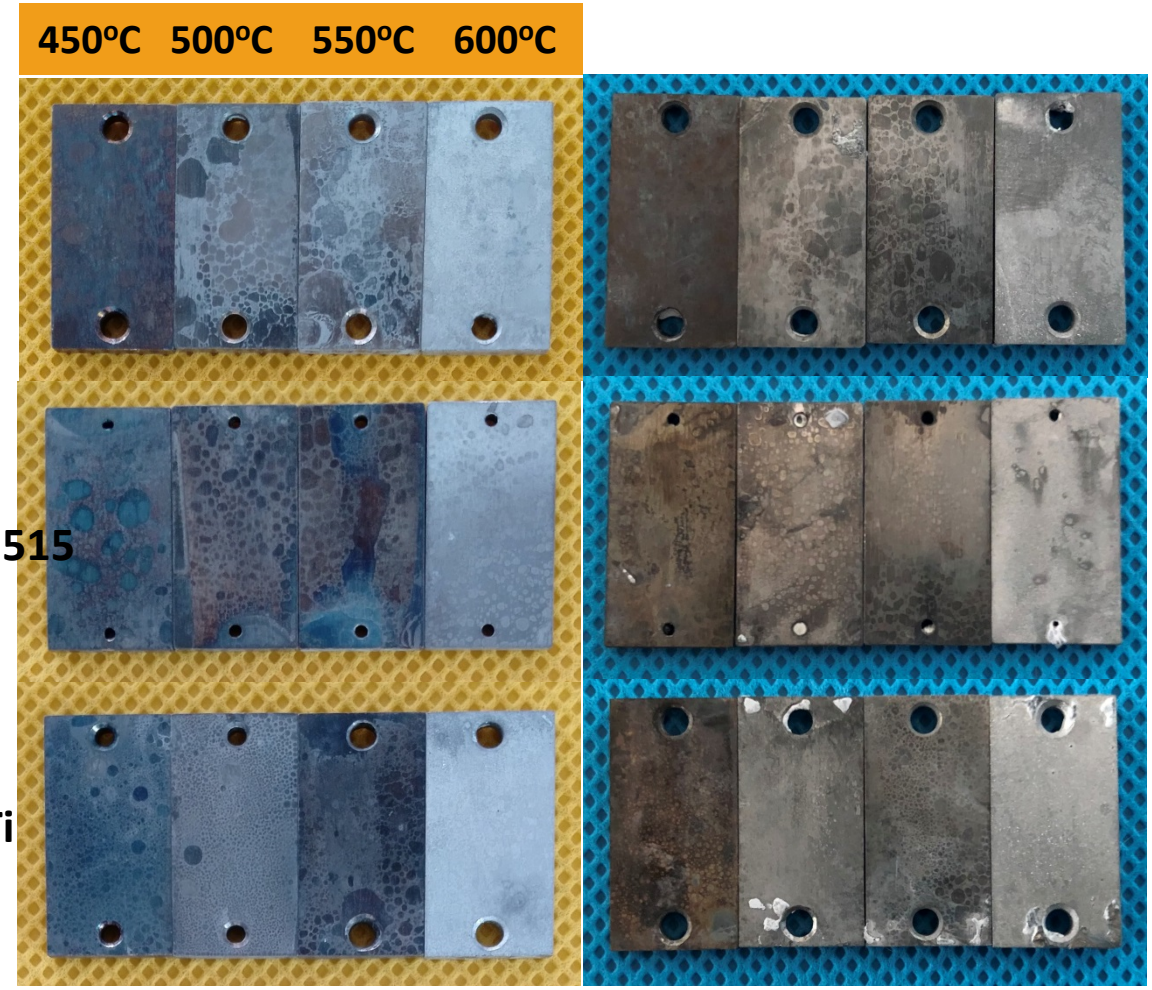
Analysis and Characterization

- Specimen cleaning: C2H5OH: H2O2:CH3COOH=1：1：1
- Scanning electron microscope, SEM: surface and section observation
- Energy disperse spectrometer, EDS: Surface and section composition analysis
- X-ray diffractor: corrosion phase detection

Instruments information

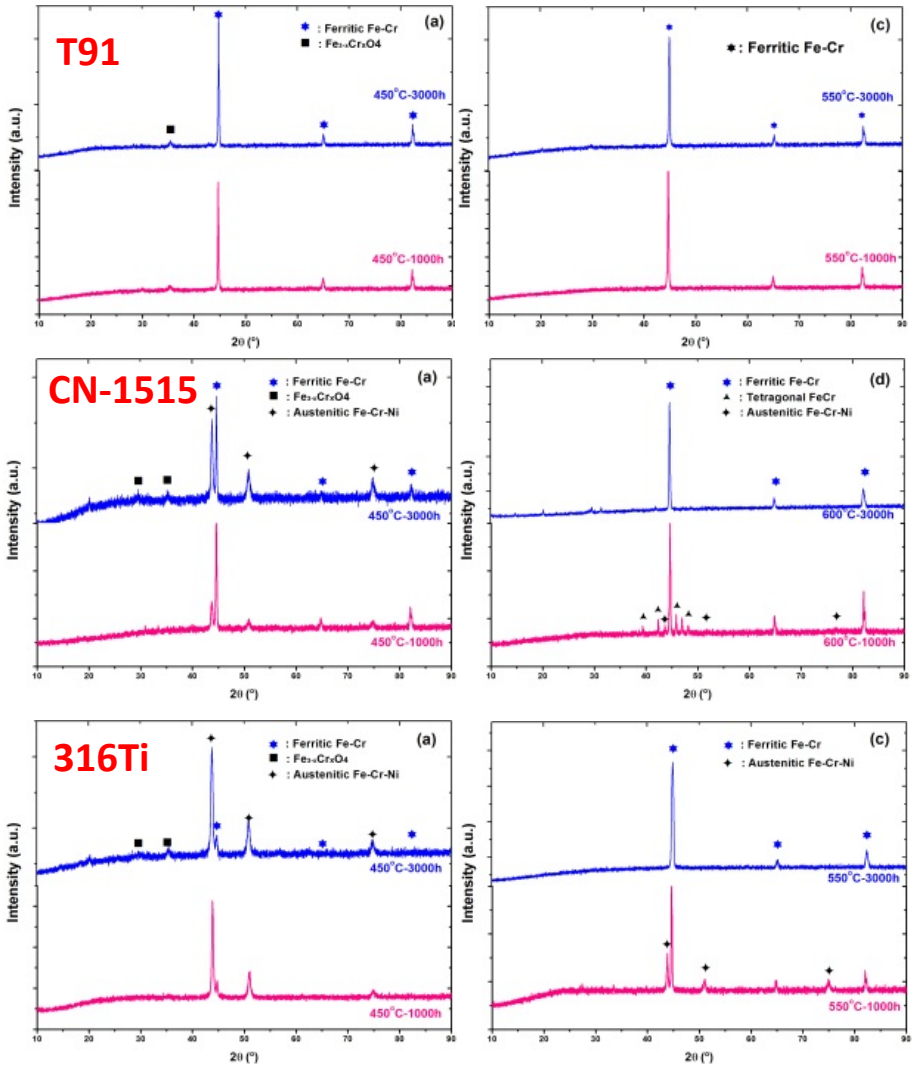
- SEM: ZEISS Supra 55 field emission
- EDS: Bruker E-flash 1510
- X-ray diffractor: Bruker D8 Advance

Macromorphology of specimen after corrosion



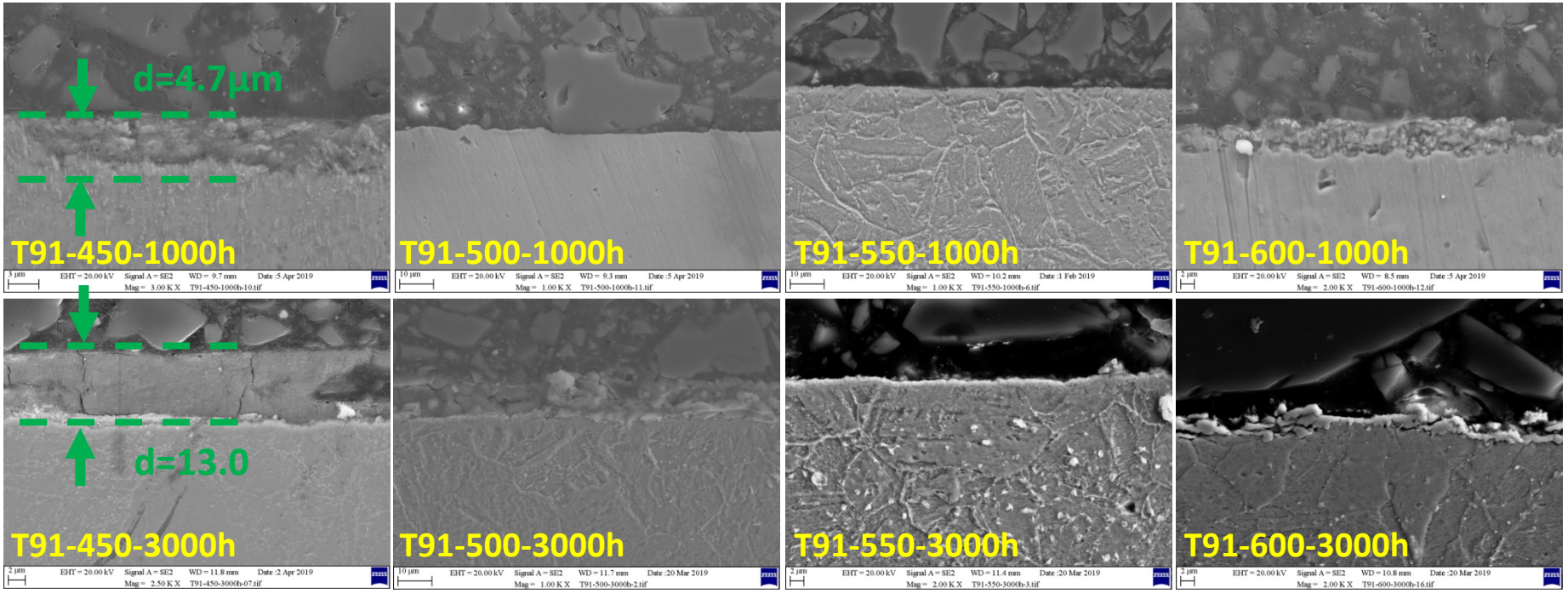
- After LBE exposure experiment, the surface colour of samples changes greatly
- The colour change means a corrosion of materials in LBE
- The difference colour of sample maybe indicates different type of corrosion.

Corrosion phase analysis (XRD)



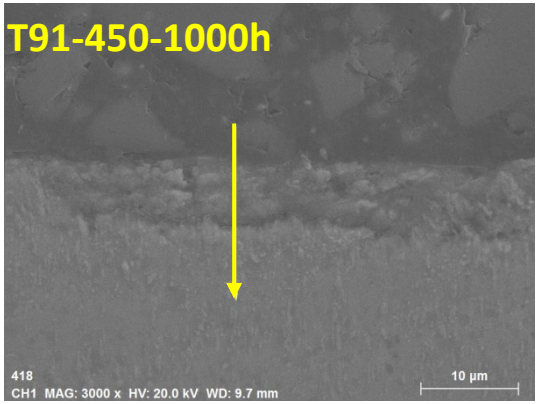
- At 450°C, XRD analysis shows the formation of oxide on the surface of three materials.
- For CN-1515, a ferritic layer is also formed.
- With the increment of temperature, no oxide formation is found for T91, and FCC structure is not stable in LBE, a FCC-BCC phase transformation happened

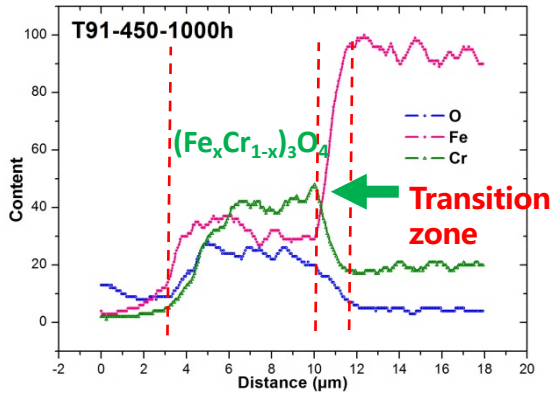
SEM analysis of corrosion section-T91

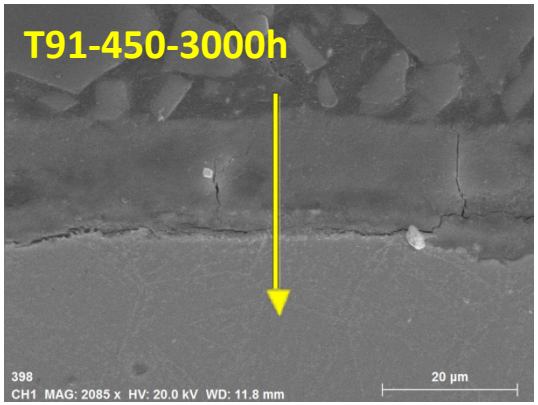


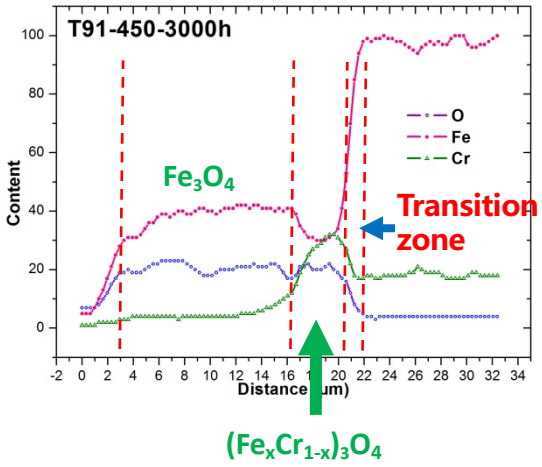
- At 450℃, oxidation corrosion takes place, which is in agreement with the XRD results；
- At 500 and 550℃, only a slight corrosion is observed ；
- With the increase of temperature, at 600ºC, a obvious dissolution corrosion is happened on the surface of T91.

T91: Corrsion product composition analysis



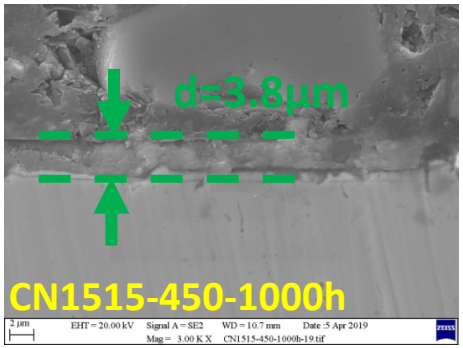


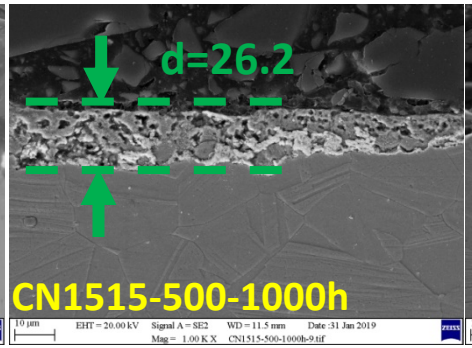


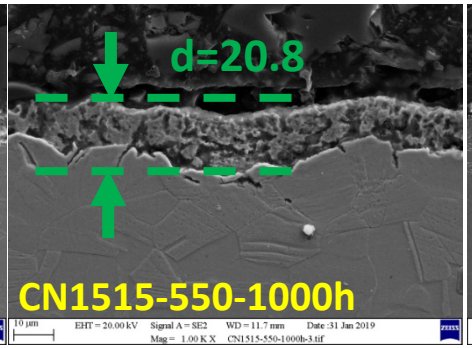


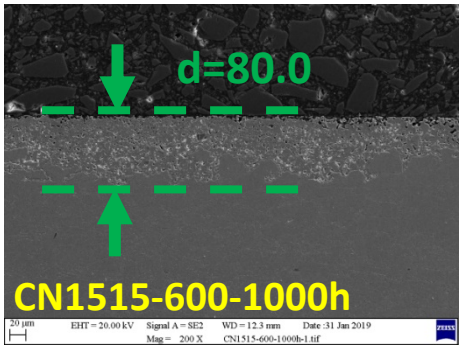
- At 450℃，EDS line scanning shows a single layer of $(Fe_xCr_{1-x})_3O_4$ magnetite is formed after 1000h LBE corrosion.
- At 450℃，after 3000h，a oxide film is composed of two layers.
- Out layer is Fe_3O_4，and inner layer is $(Fe_xCr_{1-x})_3O_4$, respectively.

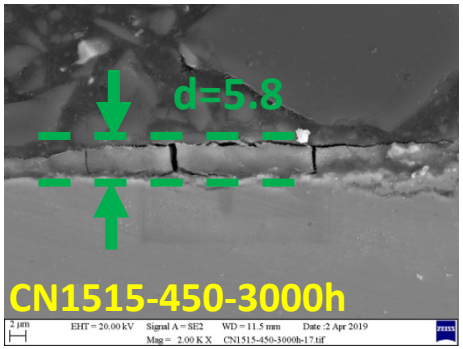
SEM analysis of corrosion section-CN-1515

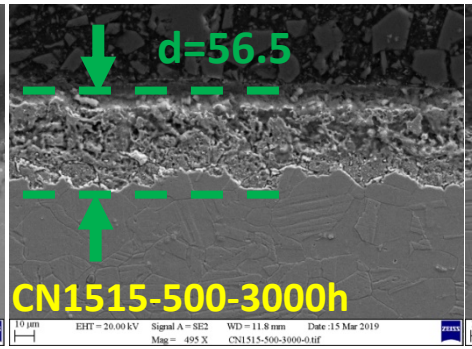


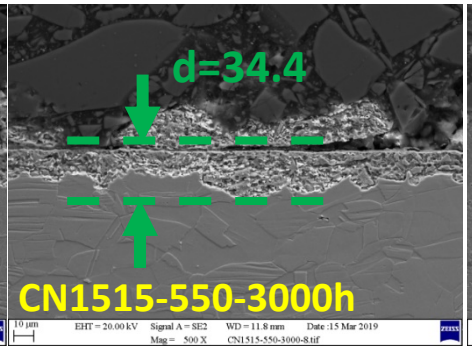


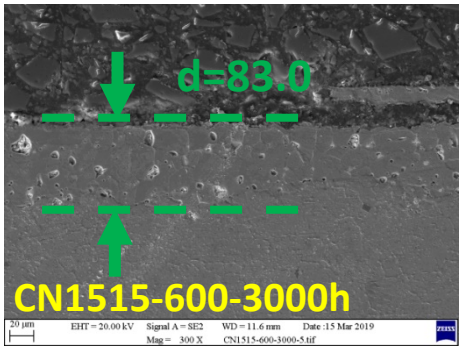








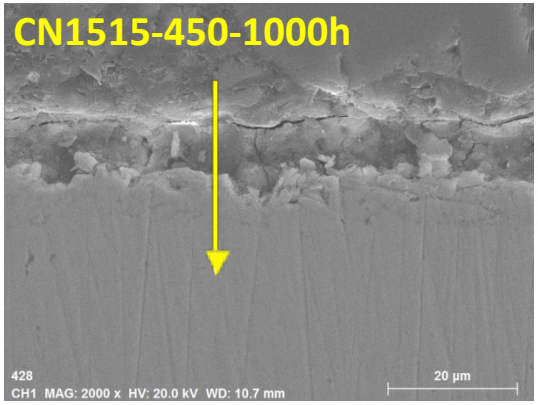


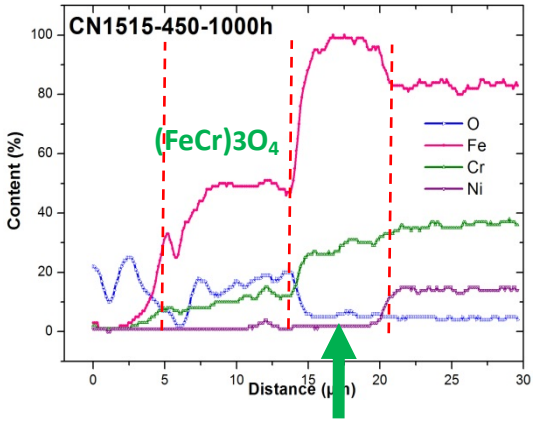


- At 450℃, oxidation corrosion takes place, which is in agreement with the XRD results；
- At 500, 550 and 600℃, an obvious dissolution corrosion is observed ；

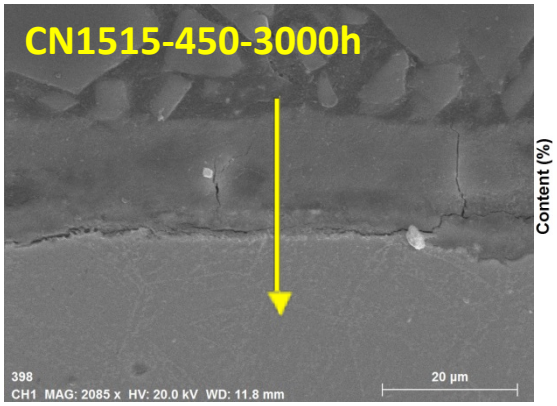
实验结果-腐蚀截面成分分析

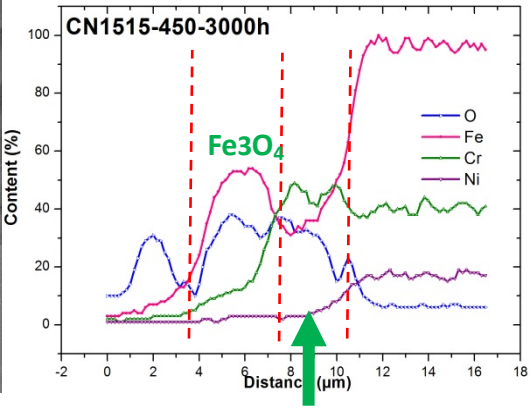
CN-1515: Corrosion product composition analysis





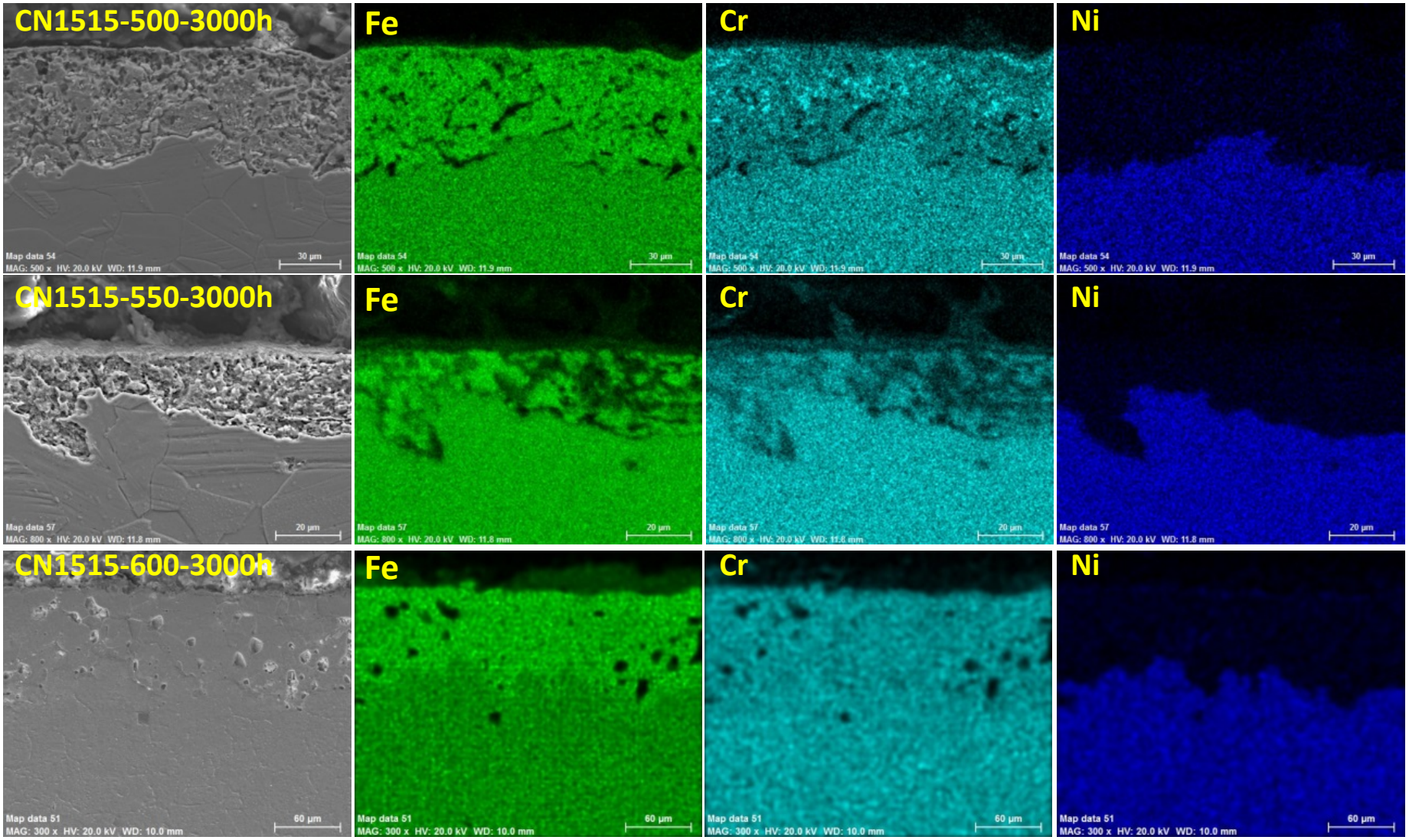
Ni dissolution aeron



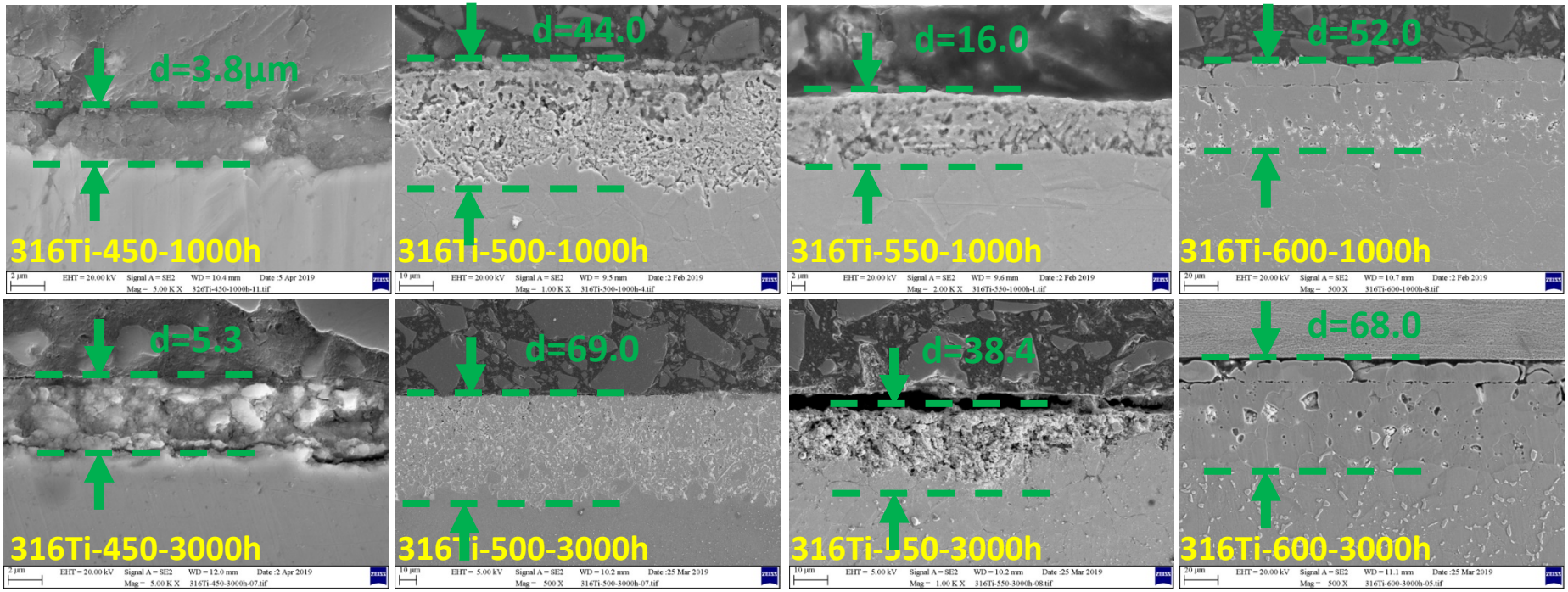


(FeCr)3O4

CN-1515: Corrosion section EDS mapping analysis

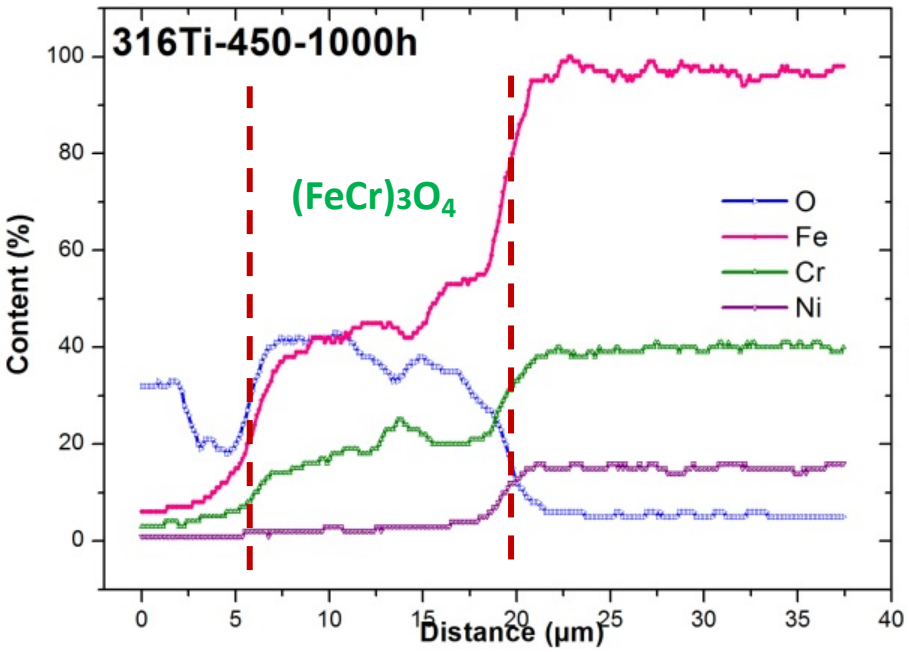


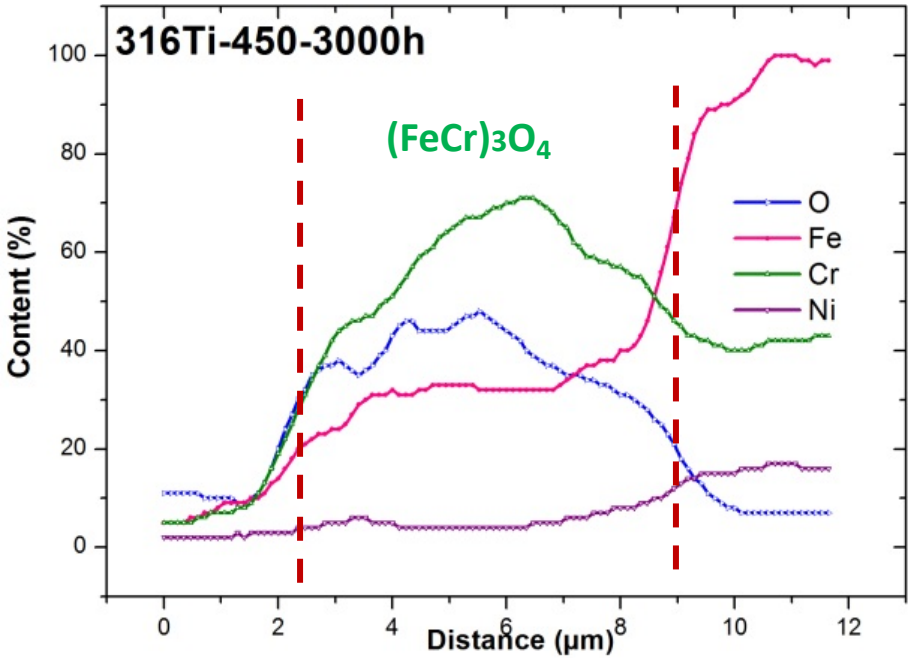
SEM analysis of corrosion section-316Ti



- At 450℃, oxidation corrosion takes place, which is in agreement with the XRD results；
- At 500, 550 and 600℃, an obvious dissolution corrosion is observed ；

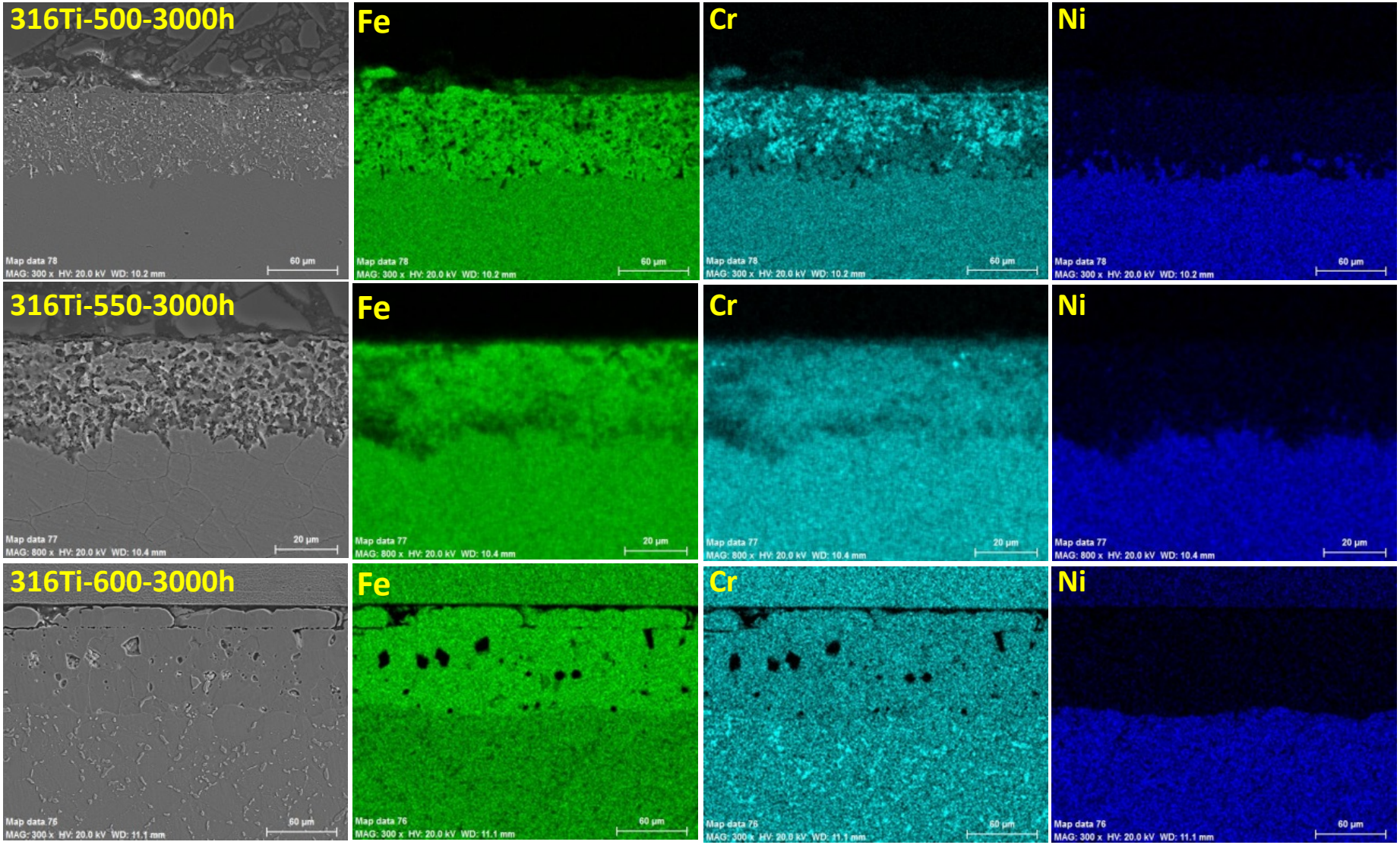
316Ti: Corrosion product composition analysis



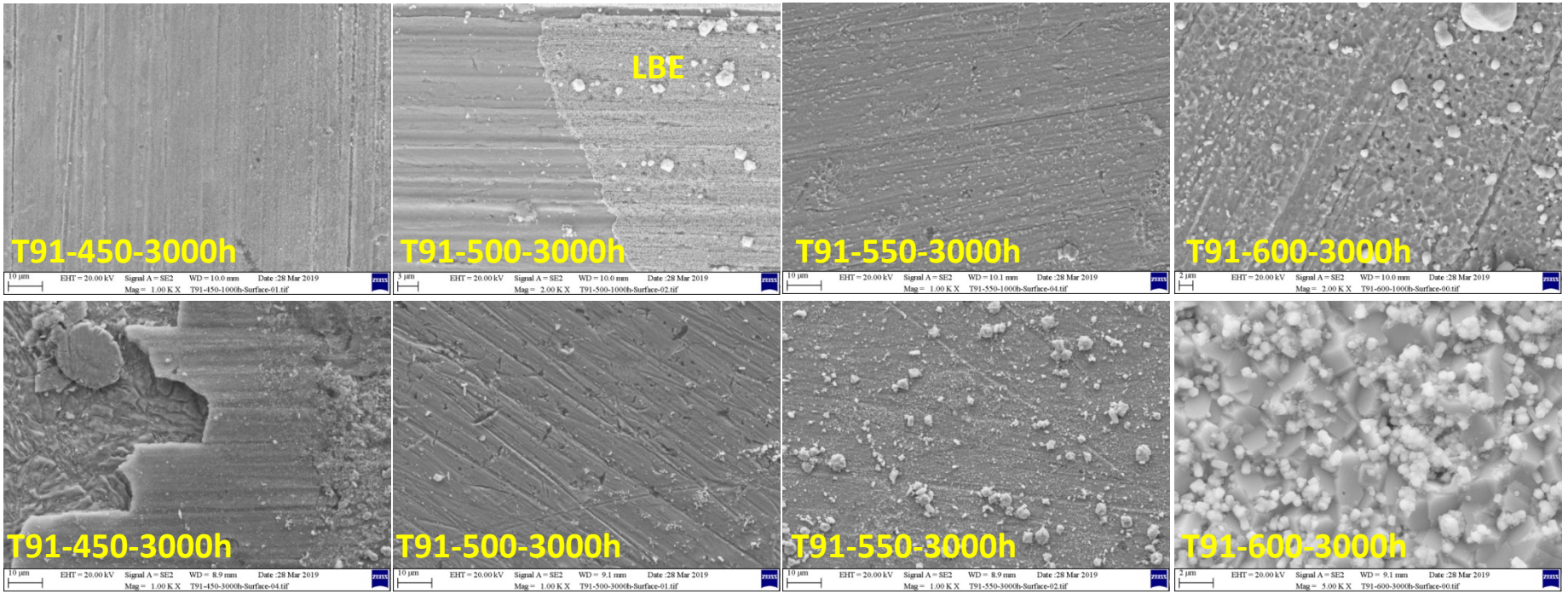


- EDS analysis shows the observed oxidation film is single layer

316Ti: Corrosion section EDS mapping analysis

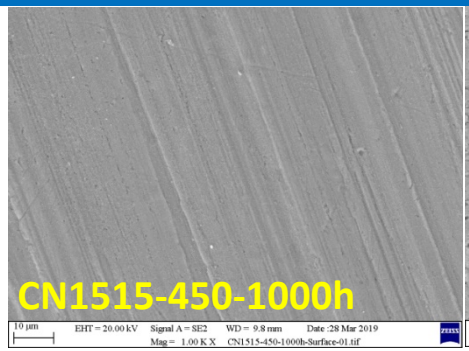


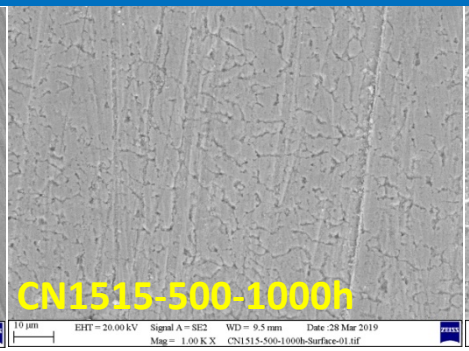
T91-Surface Observation

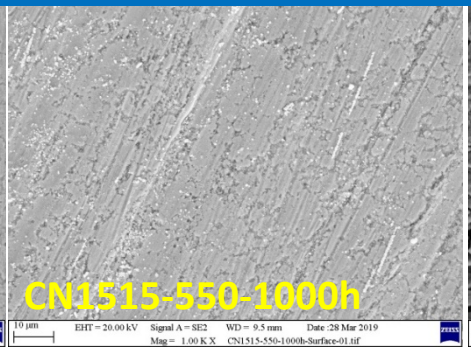


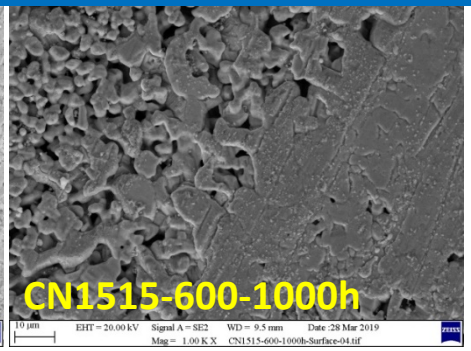
- After 1000h, the original texture of sample on surface can be observed, and at 600℃，an evident corrosion is clearly visible;
- After 3000h, at 450℃, part of the oxide layer is broken and peeled off; at 500 and 550℃,
- But at 600℃, SEM shows a recrystalled morphology.

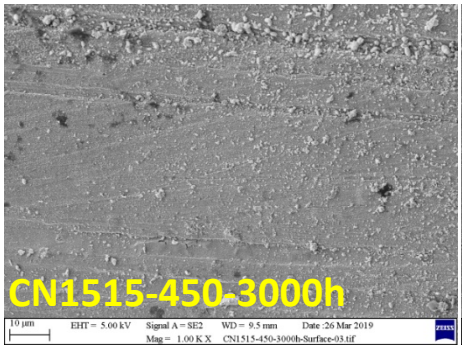
CN-1515 -Surface Observation

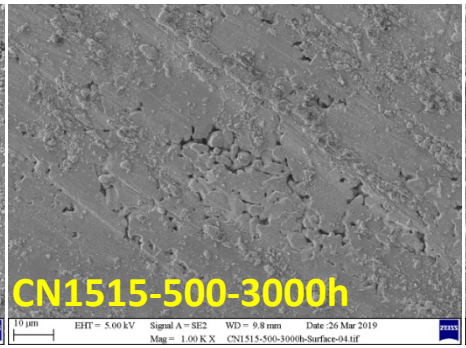


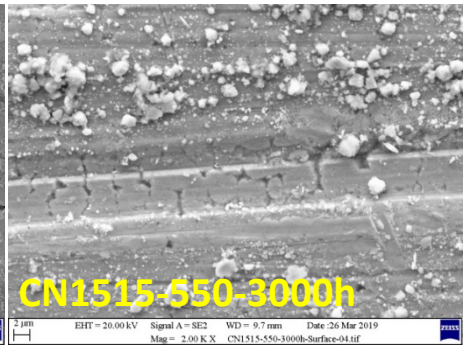


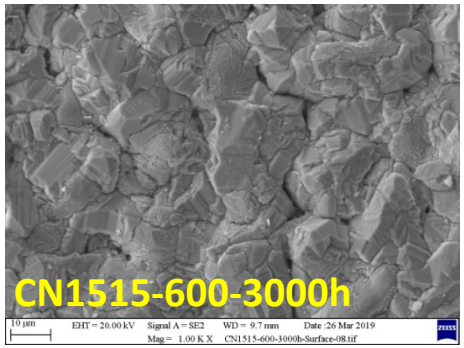






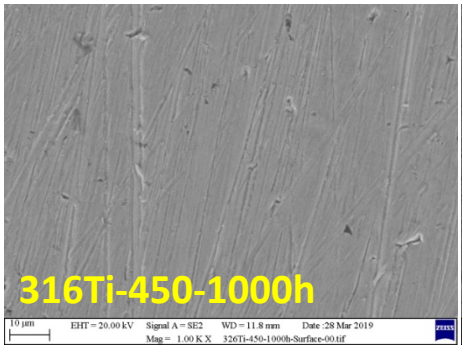


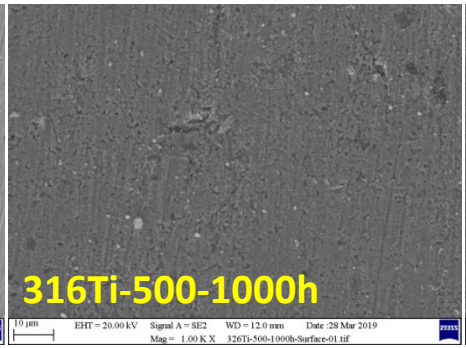


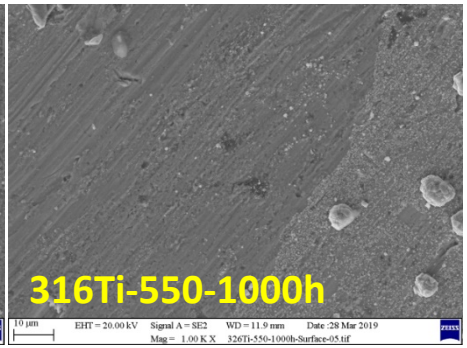


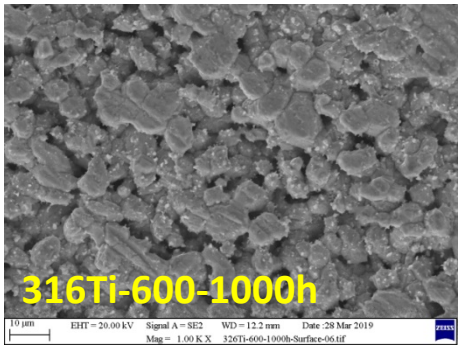
- At 450℃, the surface oxide layer is dense enough to protect matrix ;
- With the increase of temperature, corrosion cracks appear on the surface;
- After 1000h at 600℃, A severe dissolution corrosion occurs, and after 3000h, a recrystallized layer formed on the surface of CN-1515, same as T91.

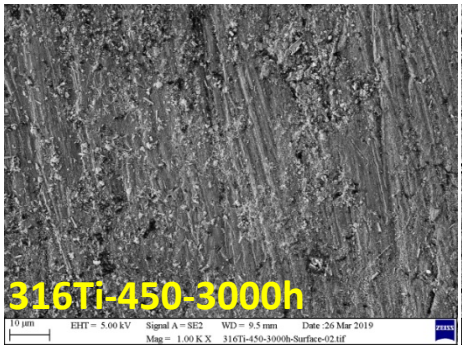
316Ti-Surface Observation

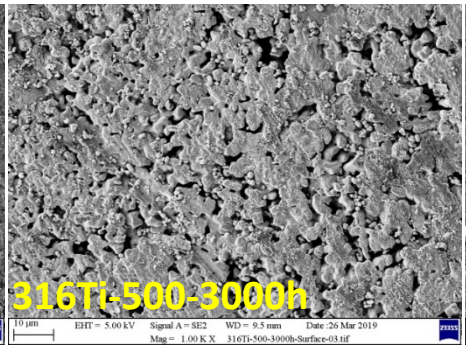


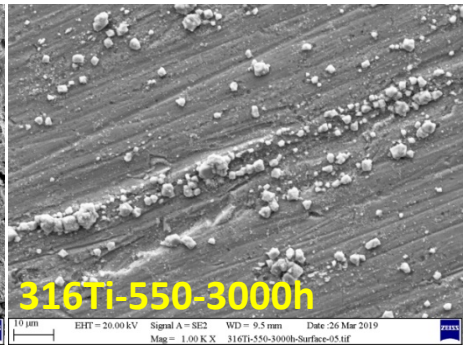


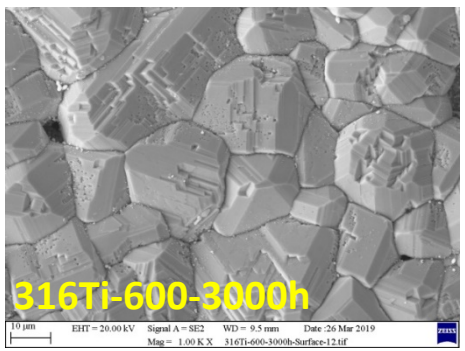






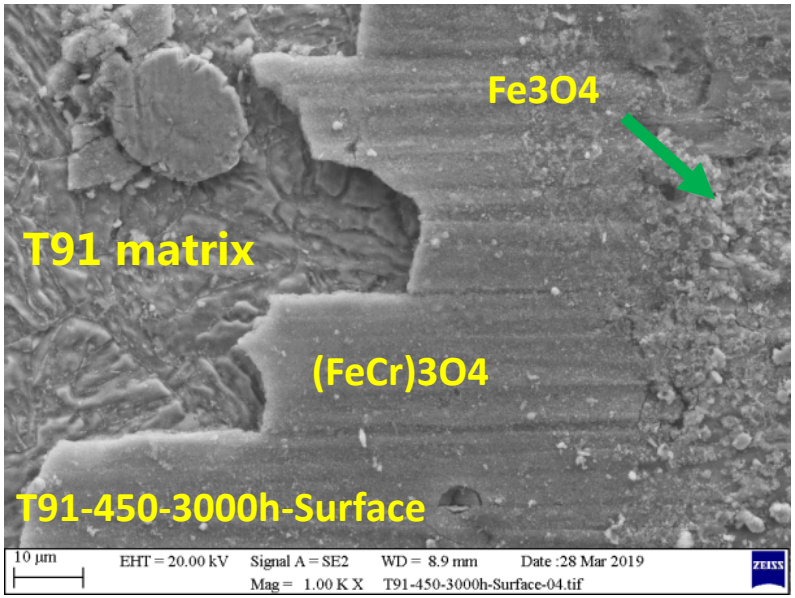


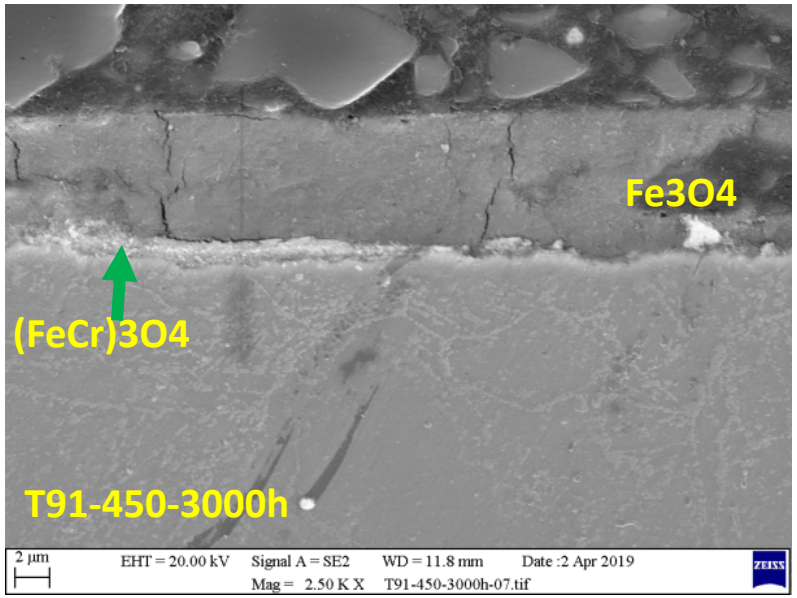




- As same as CN-1515, the 316Ti show a similar corrosion tendency in LBE at different temperatures;

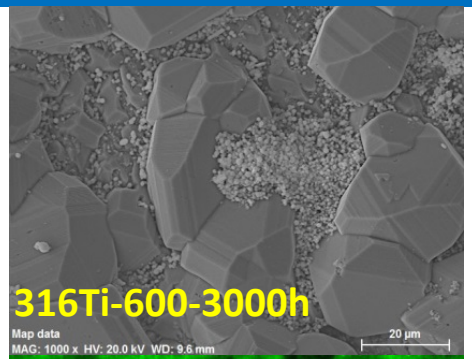
Corrosion time effect

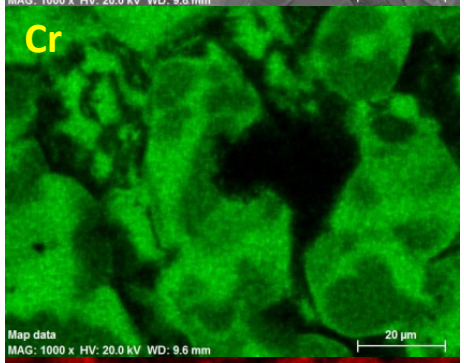


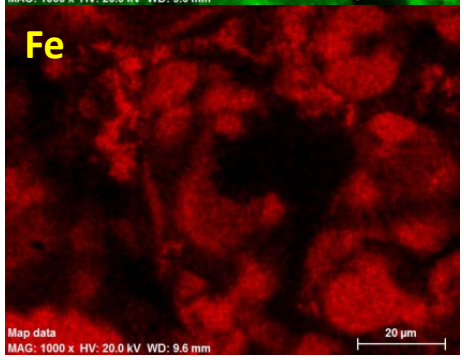


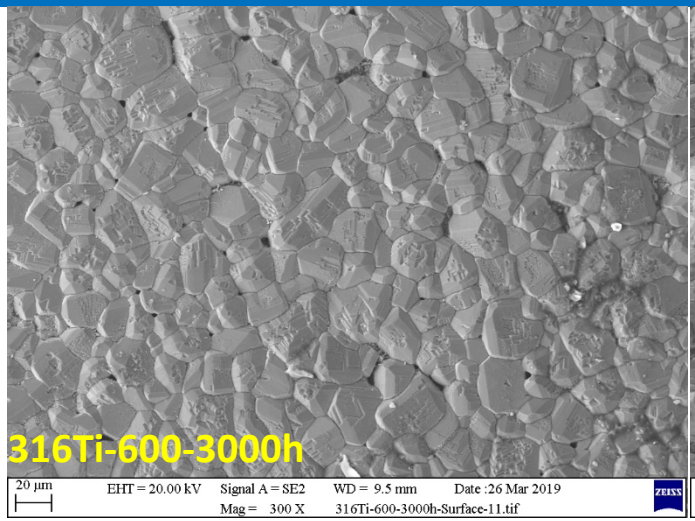
- With increment of corrosion time, the oxide layer is not stable.
- Cracks takes place in the out layers;
- The inner layer is also easy to be broken.

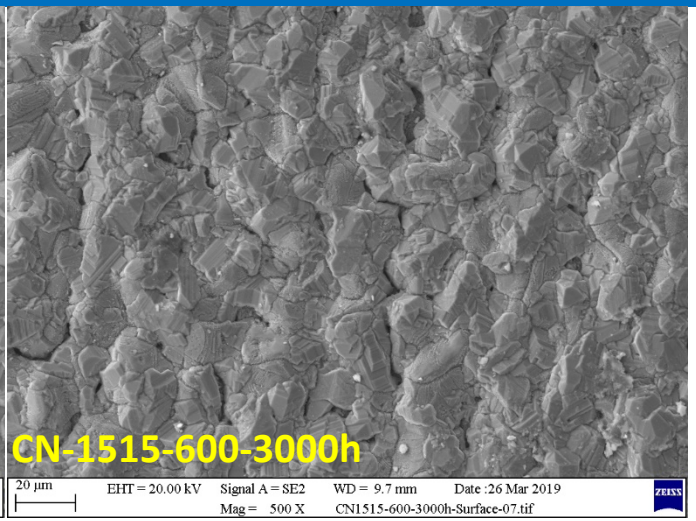
Severe mass transfer effect at high temperature











- At high temperature, LBE has a high solubility of Fe and Cr, and when reduce the experimental temperature, the over-saturated Fe and Cr precipitates on the surface of samples.

Summary

- At 450C°, an oxide film is formed on the surface of T91, CN-1515 and 316Ti, and the layer is not stable when it grows;
- At 500 and 550C°， a slight corrosion occurs on T91ferritic – martensitic steel ；
- For the austenitic steel (CN1515 and 316Ti), the Ni diffuse from corrosion layer to LBE, meanwhile, the Pb and Bi penetrate into materials matrix along the grain boundary;
- Because of Ni diffusion and dissolution, the original FCC structure is not stable, a FCC-BCC phase transformation occurs;
- At 600 C°, dissolution corrosion happened for all three materials;
- A severe mass transfer effect exists at high temperature as result of high solubility of Fe and Cr.

Thanks for your attention!