Impedance Spectroscopy and Microstructural Characterization of the Corrosion Behavior of FeCrAl Steel in Lead-Bismuth Eutectic

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Introduction (1/2)

- Lead and Lead-Bismuth Eutectic (LBE) have been proposed as candidate coolant materials for the next generation nuclear reactors
- LBE at high temperature is very corrosive towards austenitic and ferritic/martensitic (F/M) steels. Currently existing austenitic and F/M steels are restricted to applications at temperatures below 500ºC
- New materials which have improved corrosion resistance in LBE at higher temperature are needed to ensure the high efficiency of the nuclear systems

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Introduction (2/2)

- A few ODS alloys with high Al content, such as PM2000, can withstand the corrosion attack from LBE at temperatures above 500ºC
- An FeCrAl alloy is selected for test because of its similar composition compared with PM2000
- Originally designed for resistance heating, the FeCrAl alloy has superior oxidation resistance due to the formation of protective Al oxide scales on the steel surface
- Surface oxide formation on materials is needed for the implantation of Impedance spectroscopy (IS) to characterize the corrosion behavior of materials in LBE

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Objective

- IS was used to monitor the corrosion kinetics and development of protective oxide layer
- Post-corrosion microanalysis was performed to characterize the corrosion products and corrosion mechanism
  - SEM & EDS
  - XRD
  - AES & XPS

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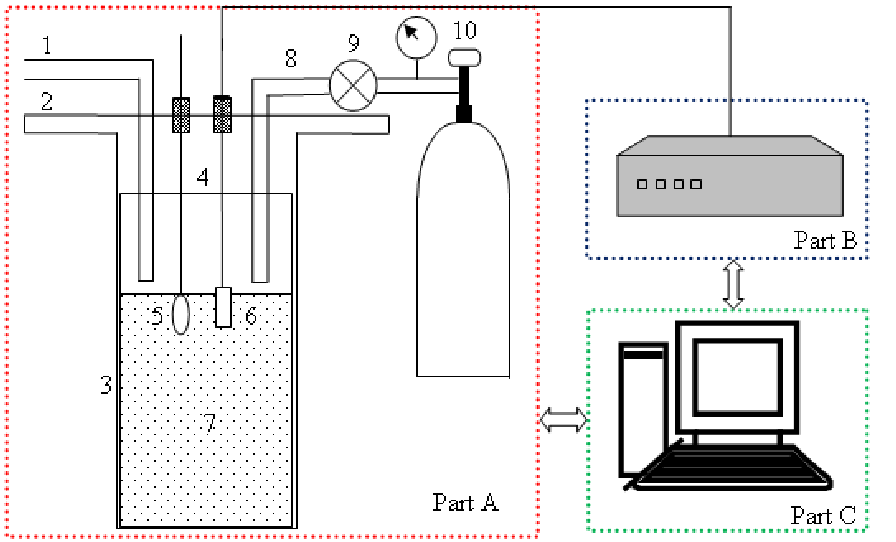
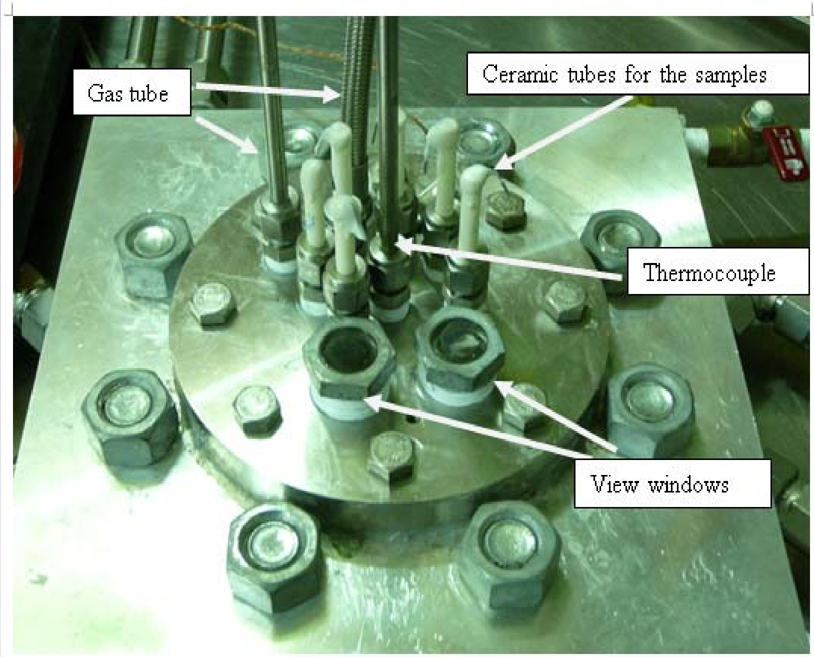
Experimental Setup (1/2)



Part A—corrosion test setup, 1—gas outlet, 2—top lid, 3—stainless steel heating pot, 4—quartz crucible, 5—thermal couple, 6—SS316 sample, 7—LBE, 8—gas inlet, 9—gas flow meter, 10—argon gas tank, Part B—impedance measurement system, Part C—computer controller



Stagnant corrosion facility with real-time IS

Top view of heating pot

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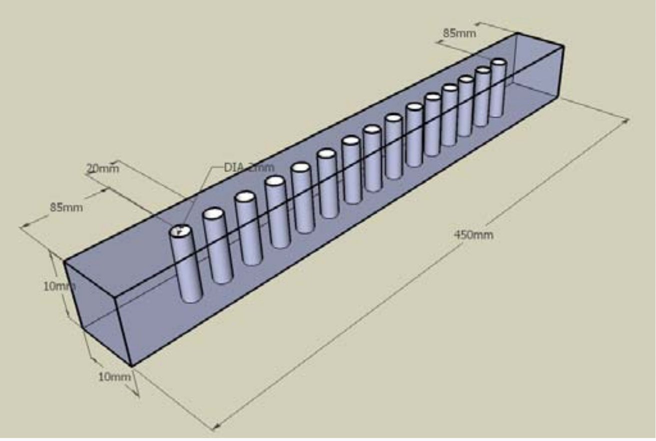
Experimental Setup (2/2)

Stagnant corrosion setup

Inside view of the stagnant corrosion setup



Specimen holder

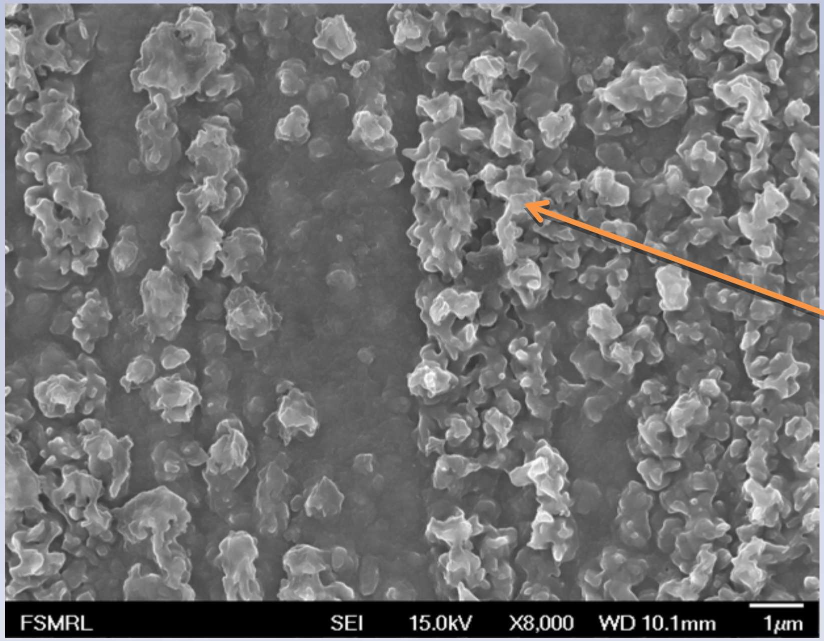
Experimental Conditions

- Sample size: 35mm x 6.35mm x 0.76mm. Polished to grit 600. Pre-oxidized at 1000ºC for 2 hours in air
- Composition of FeCrAl alloy (wt%)

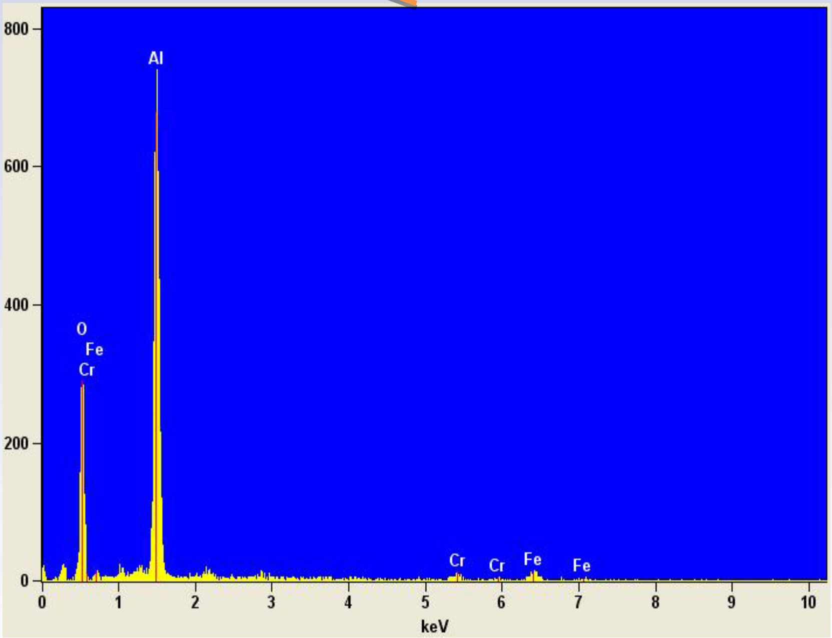
Fe	Cr	Al	Mn	Si	Y	Zr	C
Bal.	22.0	5.0	0.2	0.3	0.1	0.1	0.02

- Test condition: 550ºC static LBE with 1.17×10^{-3} wt% oxygen (oxygen saturated) for up to 3600 hours

Surface SEM and EDS



Surface oxide morphology of 3600-hour exposed specimen is quite different from the oxide formed on SS316.



Al-enriched oxide was found

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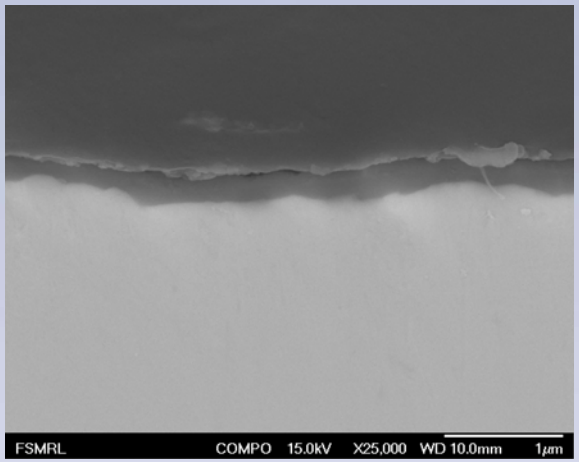
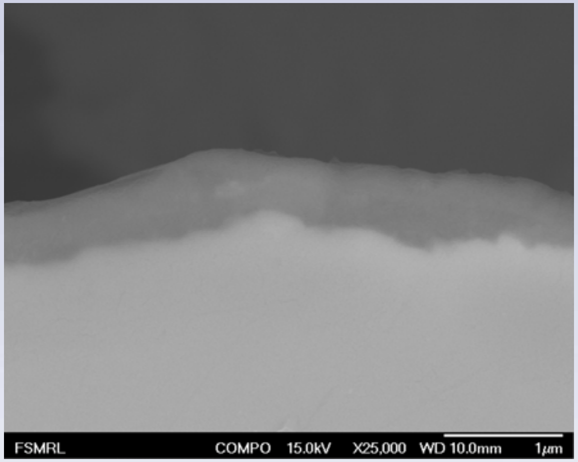
Cross-section SEM Observation

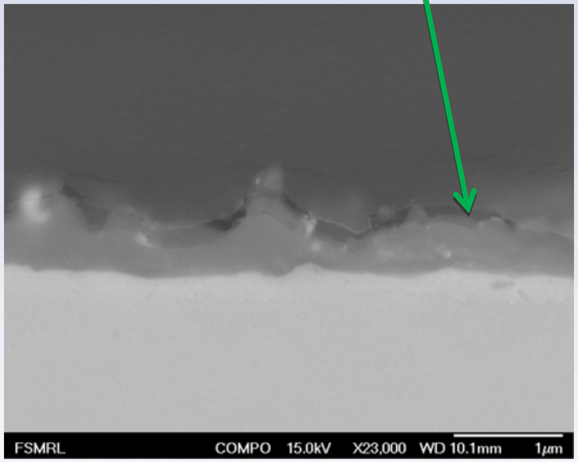


1000 hours

surface oxide



2000 hours



3600 hours

increasing exposure time ↑
oxide thickness ↑

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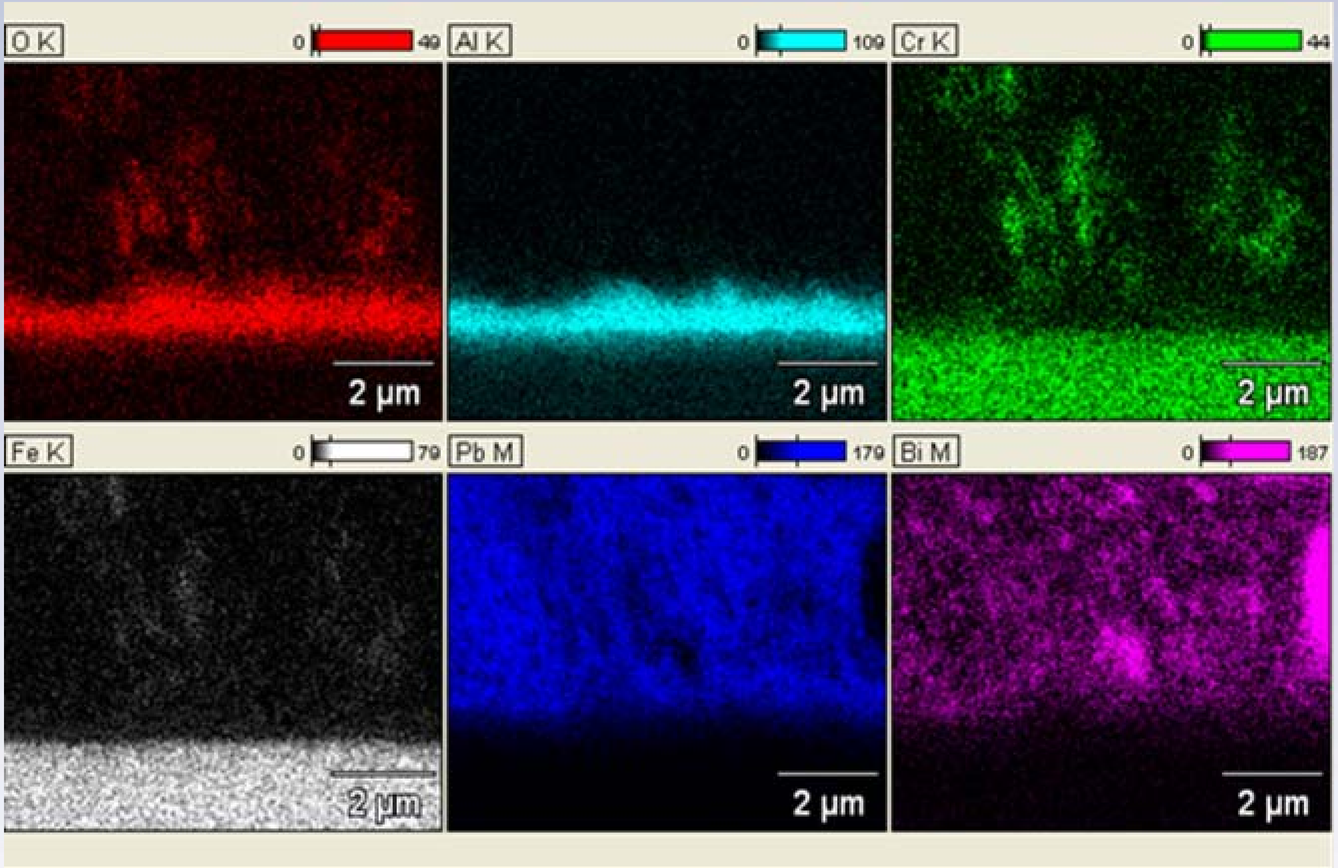
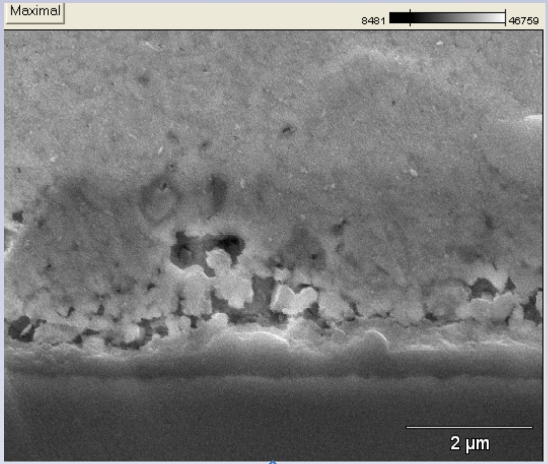
Cross-section SEM figures reveal that a very thin and adherent oxide layer formed on the alloy surface even after 3600-hour exposure in LBE

Cross-section EDS Mapping

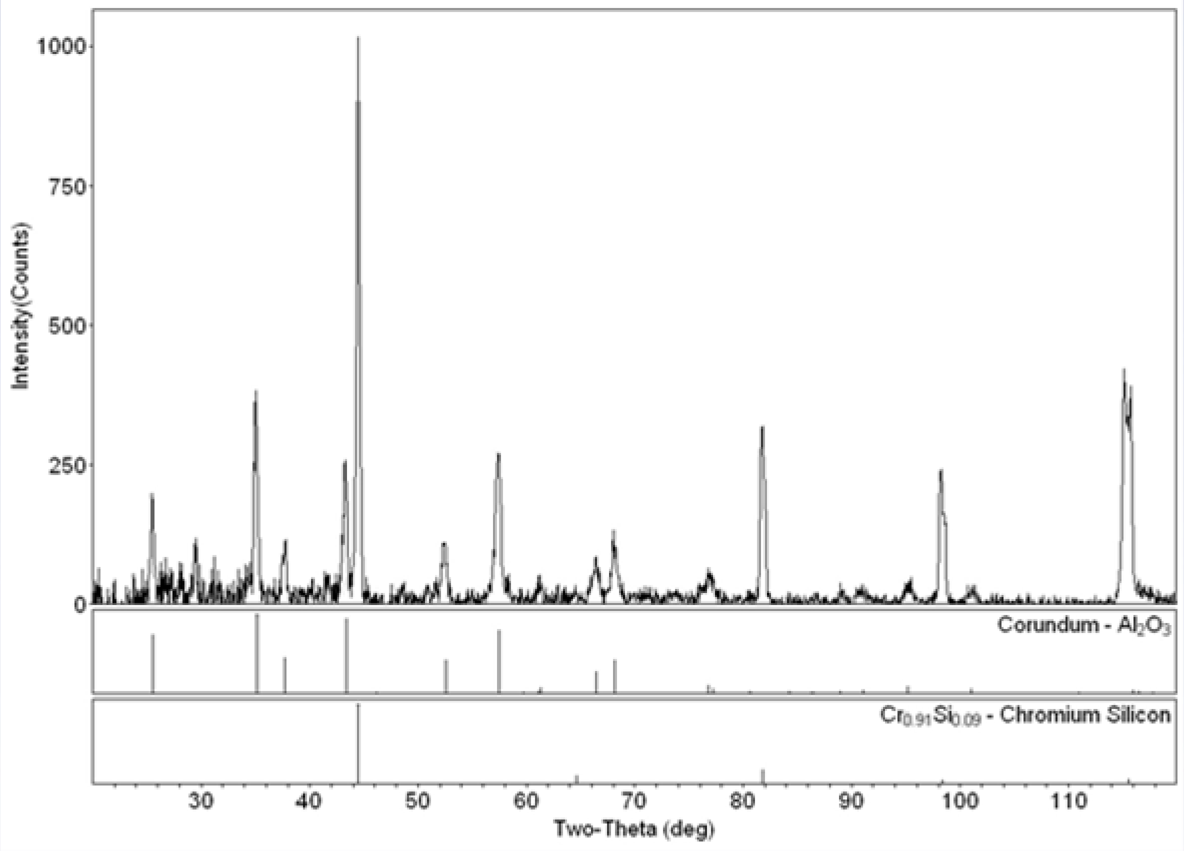




original SEM image for EDS mapping

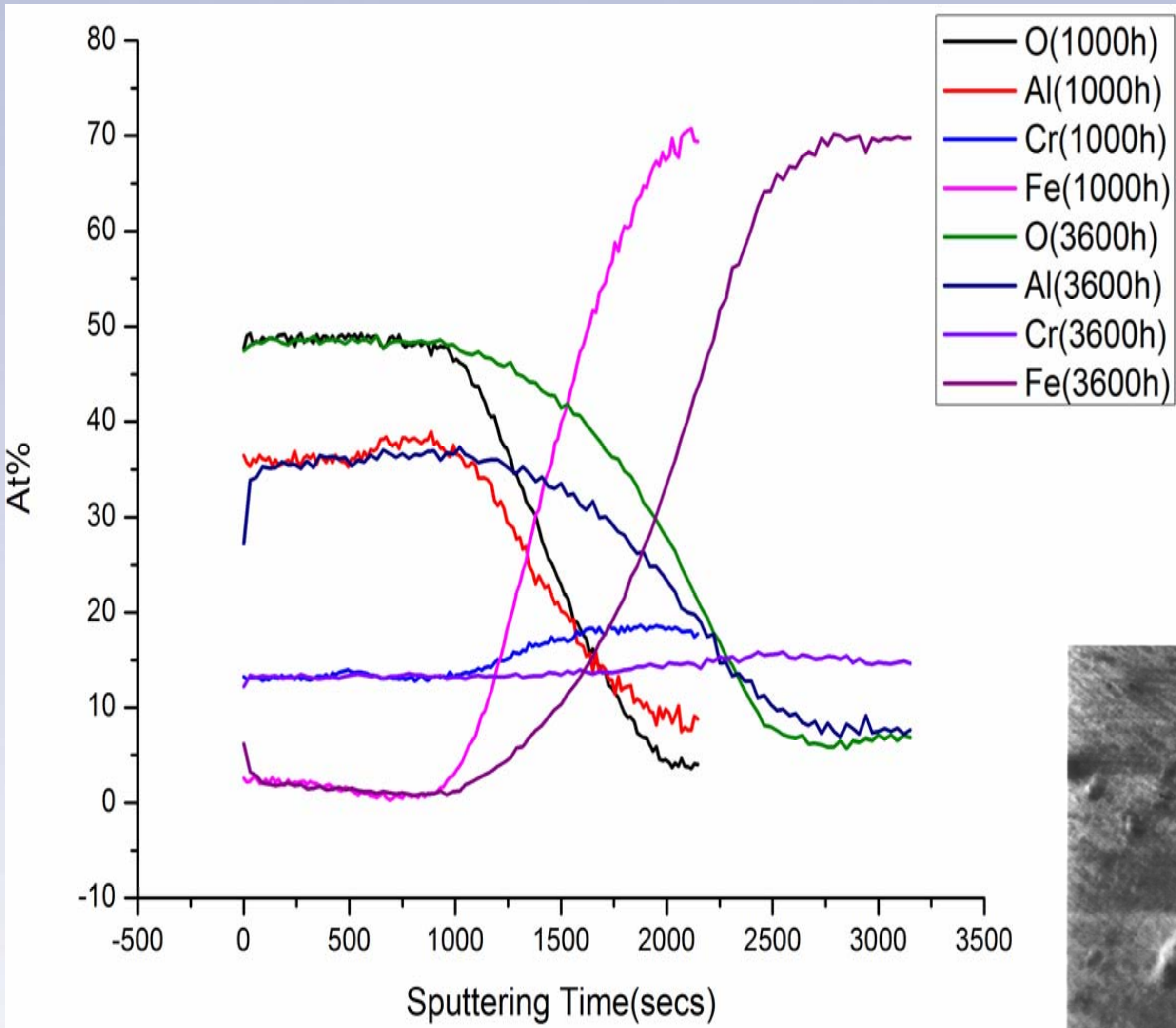
Cross-section EDS mapping shows a predominantly Al-enriched oxide layer after 3600-hour exposure in LBE

XRD Phase Identification

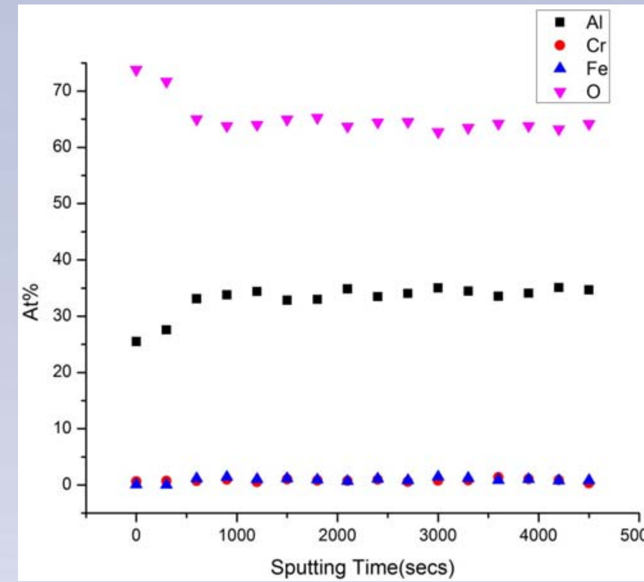


Glancing angle XRD phase identification of surface oxide formed on 3600-hour exposed specimen shows that the major phase of the surface oxide was Al_2O_3

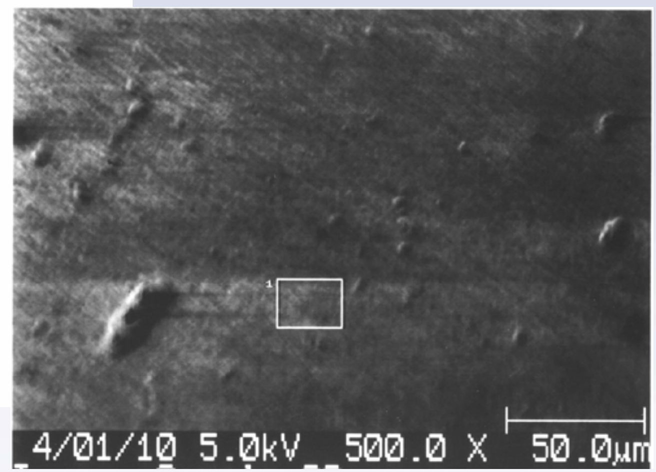
Elemental In-depth Profiling with AES



AES

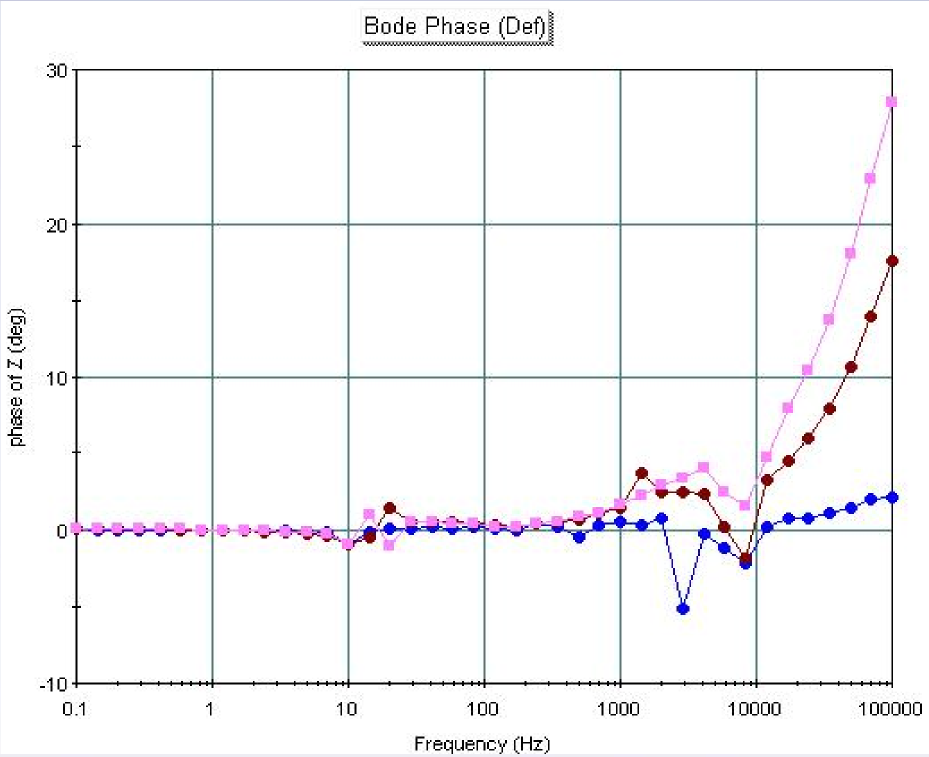


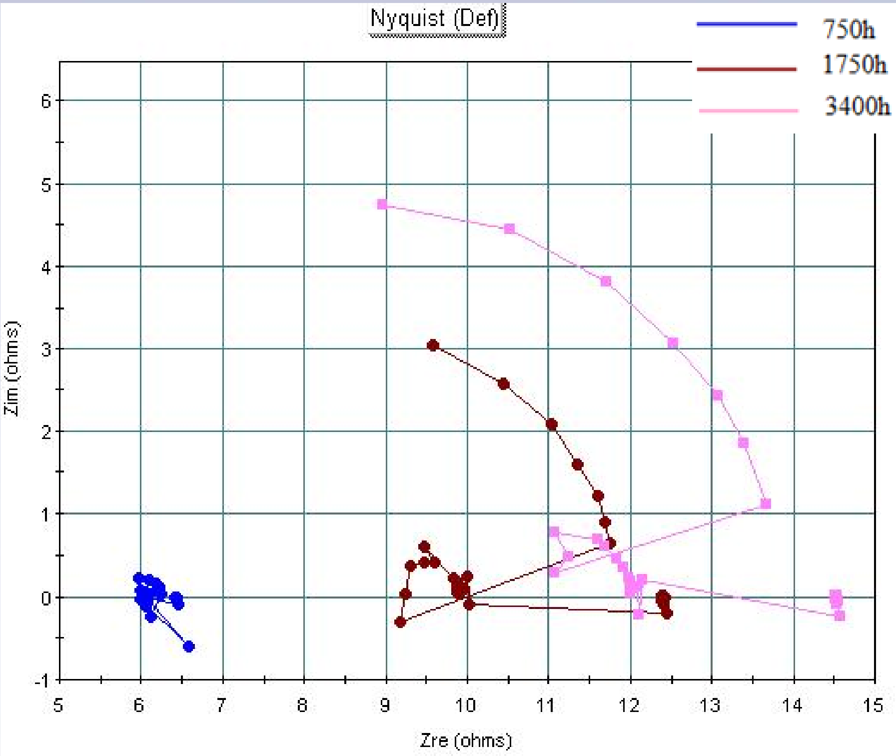
XPS



In-depth profiling with AES finds that the surface oxide is mainly aluminum oxide. As the exposure time in LBE increases, the oxide thickens without substantial composition change

Impedance Spectroscopy (1/3)





The impedance magnitude increased with time and the specimen exhibited similar impedance response

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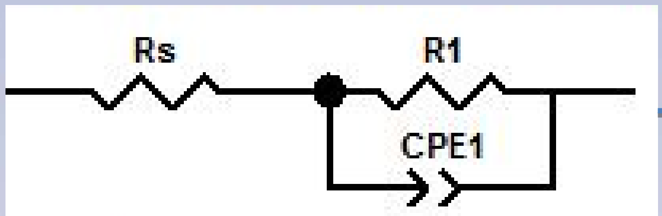
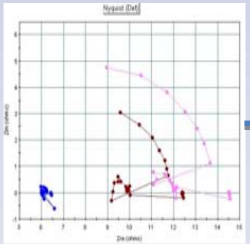
Impedance Spectroscopy (2/3)

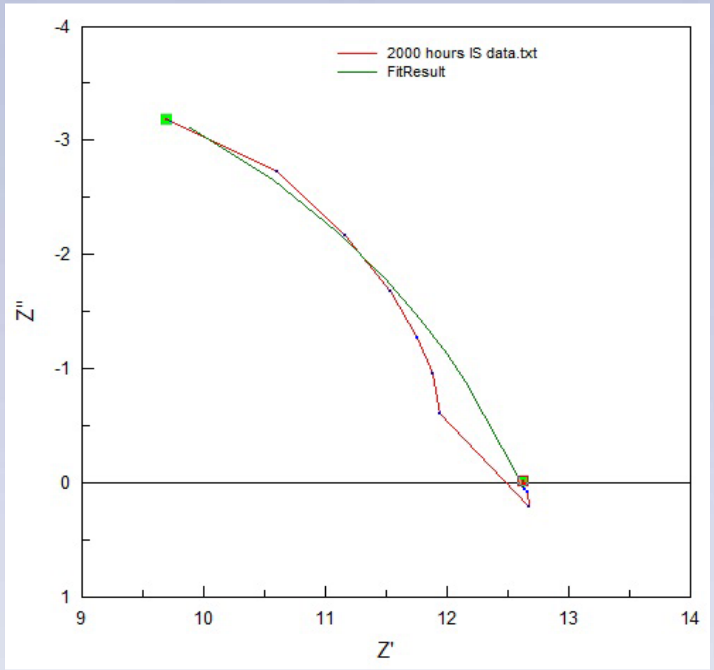


Equivalent circuit based on physical mode of surface oxide

Zview



Nyquist plot



Zview fitting based on experimental Nyquist plot

$$Z_{CPE} = \frac{1}{A(j\omega)^n}$$

$$C = R^{(1-n)/n} A^{1/n}$$

$$d = \frac{\varepsilon \varepsilon_o s}{C}$$

Z_{CPE}--impedance of CPE

A--a fit parameter independent of frequency

n--mathematical facor and has no physical meaning

ω--frequency

C--capacitance

R--resistance

d--oxide thickness

ε--dielectrical constant

ε_o--vacuum permittivity

s--electrode area

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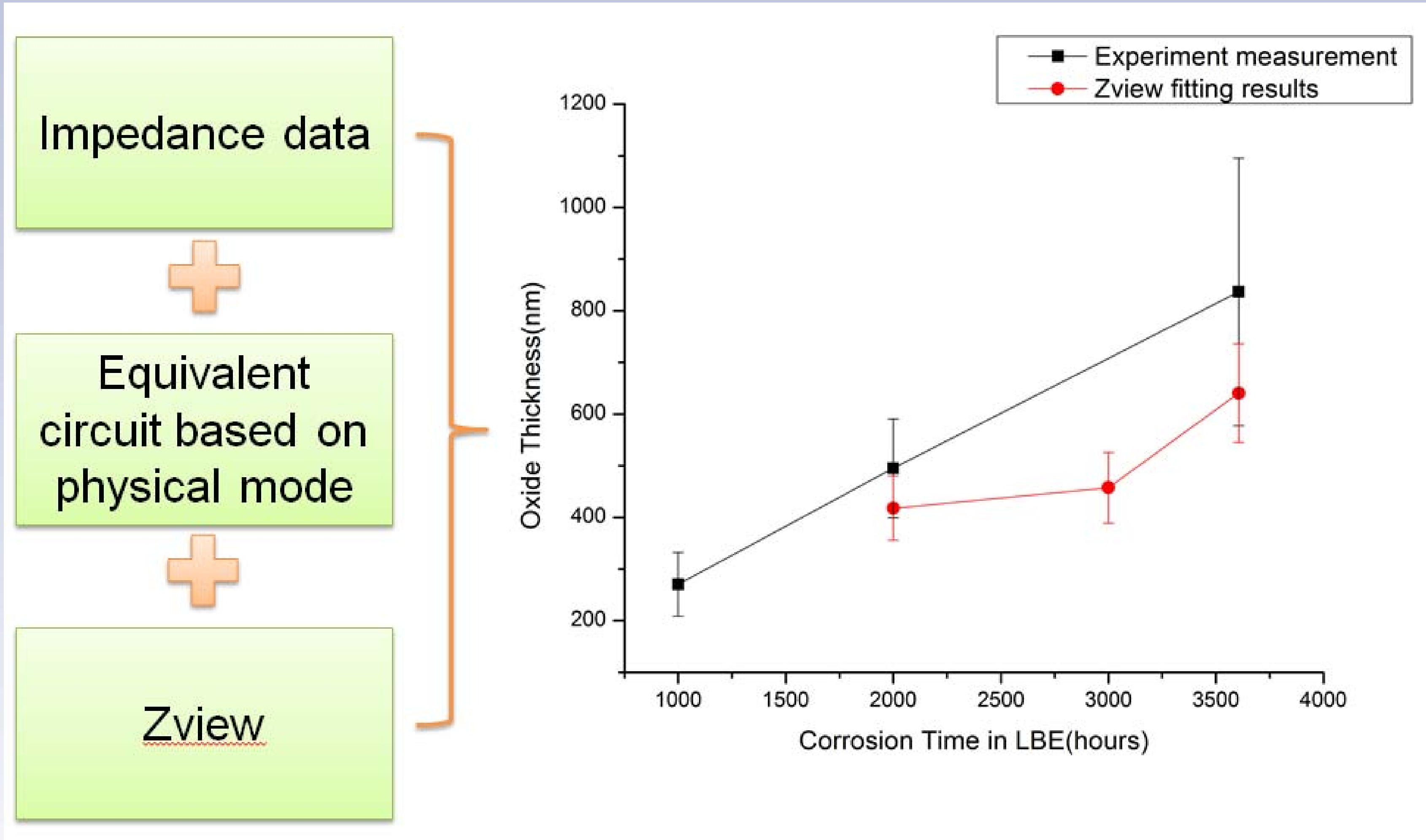
Song et al., J.Mater.Sci., 2003, 38, 3, 499-506

Impedance Spectroscopy (3/3)



Oxide thickness calculated from impedance data is in line with the microanalysis measurement

Conclusions

- After immersion in oxygen-saturated LBE, a very thin and adherent ***alumina oxide*** layer formed on the FeCrAl alloy surface and was able to protect the alloy from corrosion attack of LBE
- The oxide thickness increased very slowly with respect to time and was about ***837nm after 3600-hour*** exposure
- The IS measurements match the microanalysis results which proves the validity of using ***IS technique to monitor the real-time corrosion kinetics*** of steels in LBE

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Future Work

- ***Mechanical testing*** on raw and post-exposure FeCrAl specimens at different temperatures are needed to fully characterize its mechanical performance
- Based on the corrosion and mechanical testing results, the ***composition*** of FeCrAl alloy should be further optimized, especially for Cr and Al content, to ensure **high temperature corrosion resistance, high temperature strength, and resistance to aging embrittlement**. ***Computational thermodynamic*** simulation would be of great help in this process
- The ***shape*** of the specimen used for IS measurement can also influence the IS results, e.g. sharp edges. New specimen designs (e.g. ***disk type*** from Dr. Bolind) should be tested to improve the reliability and stability of IS measurement

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Thank you for your attention!
Comments?

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