Flow Accelerated Corrosion Experiment in LBE for Corrosion Modeling Development Using Impeller Equipped Facility

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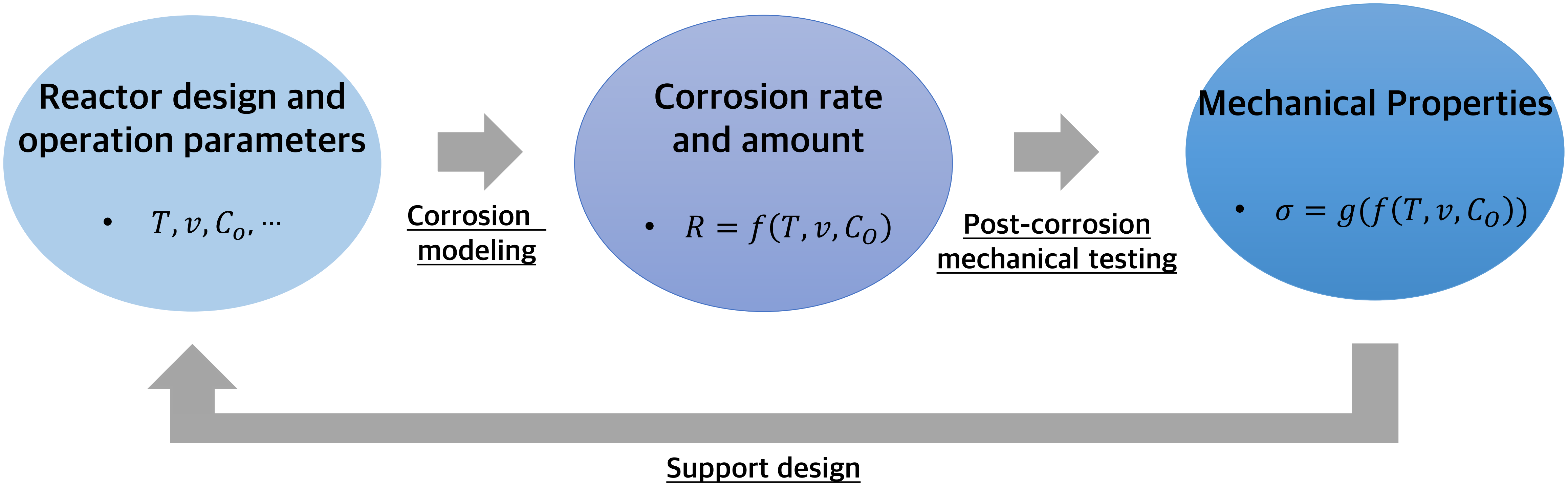
(1) Overview


Reactor design and operation parameters
• $T, v, C_O, \cdots$
Corrosion modeling
Corrosion rate and amount
• $R = f(T, v, C_O)$
Post-corrosion mechanical testing
Mechanical Properties
• $\sigma = g(f(T, v, C_O))$
Support design

• Corrosion theory

- For low oxygen concentration,

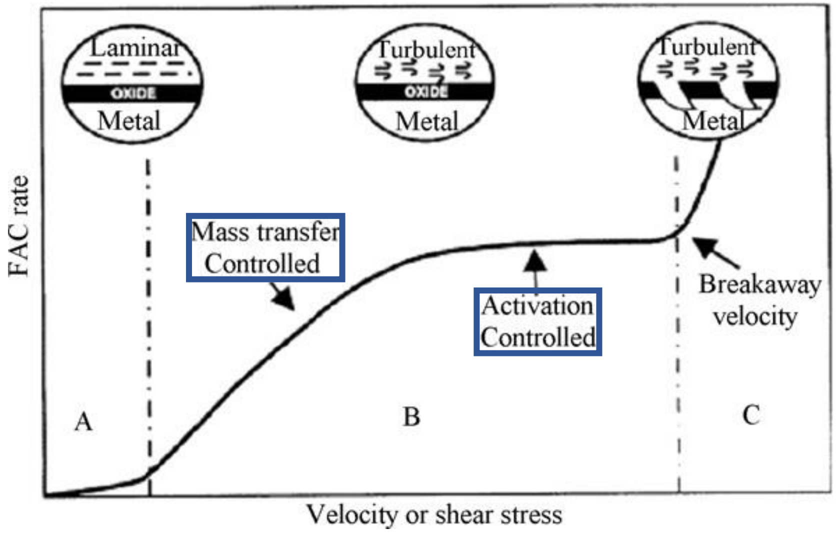
$$M_s \underset{k_{pr}}{\overset{k_d}{\rightleftharpoons}} M_d,$$

dissolution flux of the metal: $\vec{J} = \vec{J}_{diss} = \vec{J}_T = R(S_M - C_b)$, $R = \frac{Kk_{pr}}{K+k_{pr}}$

(S_M: solubility limit of the metal, C_b: the concentration of solute in the bulk solution, K: mass transfer coefficient, k_{pr}: precipitation rate coefficient)

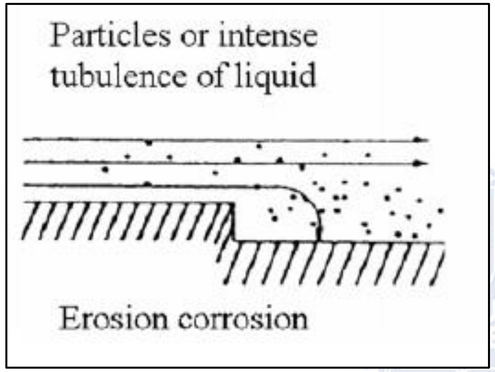
When $v \downarrow$, $K \ll k_{pr} \Rightarrow \mathrm{R} \approx K$: Mass transport($v$) dominant

When $v \uparrow$, $K \gg k_{pr} \Rightarrow \mathrm{R} \approx k_{pr}$: Phase transport (T, activation) dominant



- In high velocity region (C)

Erosion-corrosion take place.



<Jinsuo Zhang, Ning Li, Review of the studies on fundamental issues in LBE corrosion, Journal of Nuclear Materials, Volume 373, Issues 1-3, 2008>

(2) Design concept

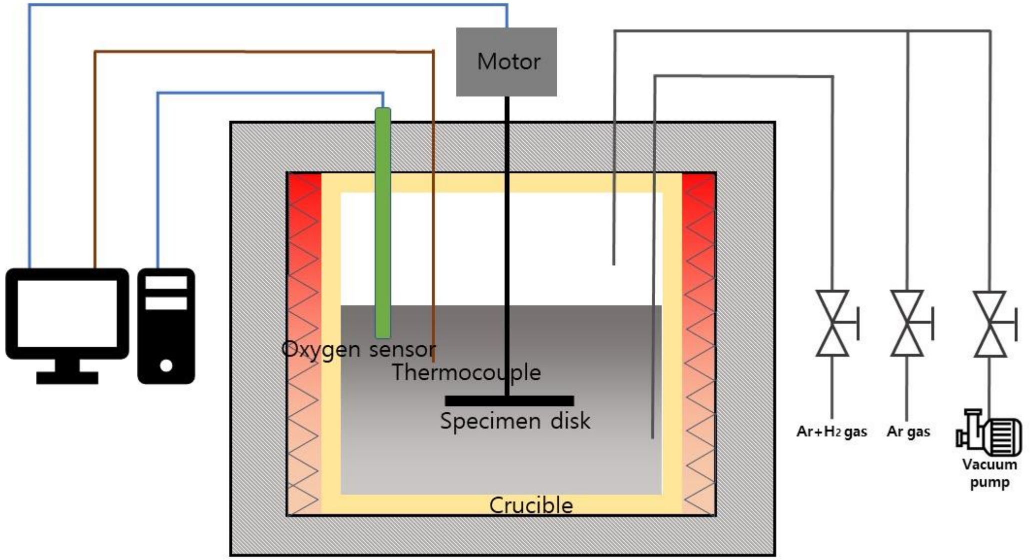
Author	Year	Journal	Type of Device	Type of Pb-alloy	O2 concentration control	Flow accelerated corrosion	Mechanical behavior measurement
Oksana Klo, et al.	2018	Journal of Nuclear Materials	Pool	LBE	O	X	O (tensile test)
Mariya Yurechko, et al.	2018	Journal of Nuclear Materials	Pool	LBE	O	X	O (Creep rupture test)
Anna Hojna, et al.	2018	Corrosion Science	Natural convection loop	LBE	O	O	X
Masatoshi Kondo, et al.	2018	Fusion Engineering and Design	Pool	Pb-Li	X	O	X
Chanho Park, et al.	2018	Fusion Engineering and Design	Pool	Pb-Li	X	O	X
Thomas Lapau, et al.	2019	Journal of Nuclear Materials	Pool/ Forced convection loop	LBE	O	X(Pool)/ O(Loop)	X
Cunfeng Yao, et al.	2019	Journal of Nuclear Materials	Forced convection loop	LBE	X	O	X
Gang Chen, et al.	2019	Progress in Nuclear Energy	Pool	LBE	X	O	X
Donatella Giuranno, et al.	2020	Corrosion	Pool	Pb	O	O	X
Z.Y.Wu, et al.	2020	Journal of Nuclear Materials	Pool	LBE	O	X	X
Haoran Wang, et al.	2020	Corrosion Science	Pool	LBE	O	X	X
L. Martinelli, et al.	2020	Corrosion Science	Pool	LBE	O	O	X
Haiyan Jiang, et al.	2020	Fusion Engineering and Design	Pool	Pb-Li	O	O	X
J.W.Choi	**2021~**	**-**	**Pool**	**LBE**	**O**	**O**	**O**

(2) Design concept

- Pool type vs. Loop type

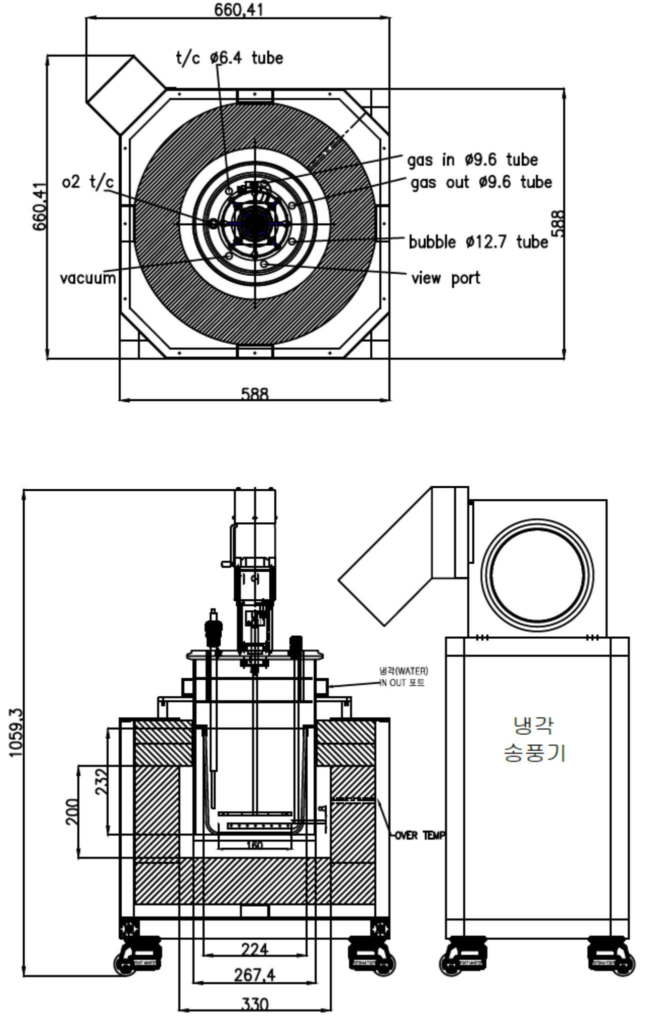
	Pool type	Loop type
Size	• Relatively small	• Relatively large
C_o	• Able to control easily due to relatively small size	• Unfavorable for controlling uniformly
v	• Hardly generate flow by attaching pump	• Favorable for generating flow

<u>Controlling both oxygen concentration and LBE flow by attaching impeller to pool type device</u>



Schematic figure of newly designed FAC experimental device

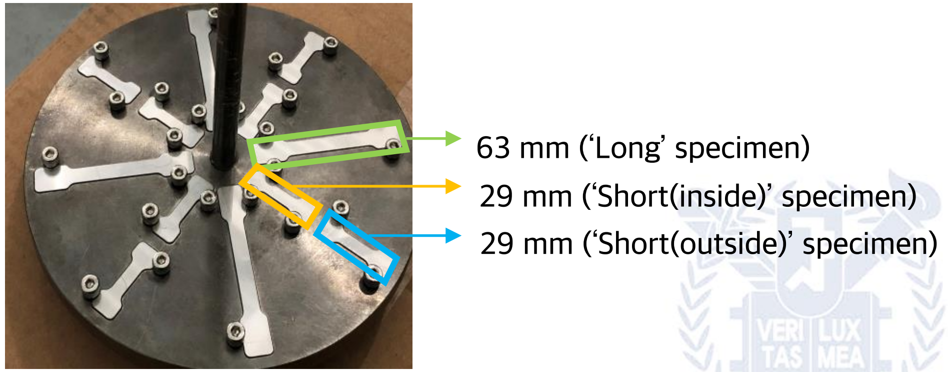
(3) Details of experimental facility



Blueprint of FAC experimental device

→ Magnitude of flow velocity differs continuously depending on a distance from the rotation center even in a same specimen ($v = r\omega$).

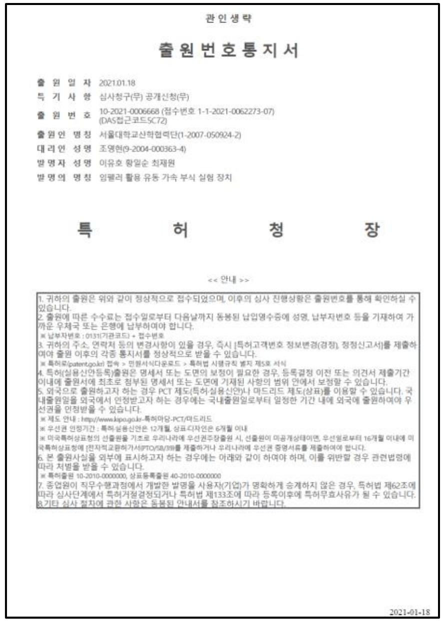
→ Specimens are made in the shape of dog-bone, measuring mechanical properties through tensile test after exposed to LBE.



Actual figure of FAC experimental device and specimen holder

(3) Details of experimental facility

Specification	
Max. temperature	600℃
Max. angular velocity	500 RPM
Specimen holder diameter	180 mm
Max. linear velocity on specimen	4.19 m/s
Crucible diameter	224 mm
Total height	1059.3 mm



관인생략

출원번호통지서

출 원 일 자 2021.01.18
특 기 사 항 심사청구(무) 공개신청(무)
출 원 번 호 10-2021-0006668 (접수번호 1-1-2021-0062273-07)
(DAS접근코드5C72)
출원인 명칭 서울대학교산학협력단(1-2007-050924-2)
대리인 성명 조영현(9-2004-000363-4)
발명자 성명 이유호 황일순 최재원
발명의 명칭 임펠러 활용 유동 가속 부식 실험 장치

특 허 청 장

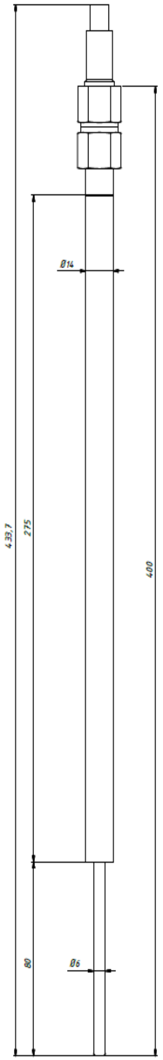
<< 안내 >>

1. 귀하의 출원은 위와 같이 정상적으로 접수되었으며, 이후의 심사 진행상황은 출원번호를 통해 확인하실 수 있습니다.
2. 출원에 따른 수수료는 접수일로부터 다음날까지 동봉된 납입영수증에 성명, 납부자번호 등을 기재하여 가까운 우체국 또는 은행에 납부하여야 합니다.
※ 납부자번호 : 0131(기관코드) + 접수번호
3. 귀하의 주소, 연락처 등의 변경사항이 있을 경우, 즉시 [특허고객번호 정보변경(경정), 정정신고서]를 제출하여야 출원 이후의 각종 통지서를 정상적으로 받을 수 있습니다.
※ 특허로(patent.go.kr) 접속 > 민원서식다운로드 > 특허법 시행규칙 별지 제5호 서식
4. 특허(실용신안등록)출원은 명세서 또는 도면의 보정이 필요한 경우, 등록결정 이전 또는 의견서 제출기간 이내에 출원서에 최초로 첨부된 명세서 또는 도면에 기재된 사항의 범위 안에서 보정할 수 있습니다.
5. 외국으로 출원하고자 하는 경우 PCT 제도(특허·실용신안)나 마드리드 제도(상표)를 이용할 수 있습니다. 국내출원일을 외국에서 인정받고자 하는 경우에는 국내출원일로부터 일정한 기간 내에 외국에 출원하여야 우선권을 인정받을 수 있습니다.
※ 제도 안내 : http://www.kipo.go.kr-특허마당-PCT/마드리드
※ 우선권 인정기간 : 특허·실용신안은 12개월, 상표·디자인은 6개월 이내
※ 미국특허상표청의 선출원을 기초로 우리나라에 우선권주장출원 시, 선출원이 미공개상태이면, 우선일로부터 16개월 이내에 미국특허상표청에 [전자적교환허가서(PTO/SB/39)]를 제출하거나 우리나라에 우선권 증명서류를 제출하여야 합니다.
6. 본 출원사실을 외부에 표시하고자 하는 경우에는 아래와 같이 하여야 하며, 이를 위반할 경우 관련법령에 따라 처벌을 받을 수 있습니다.
※ 특허출원 10-2010-0000000, 상표등록출원 40-2010-0000000
7. 종업원이 직무수행과정에서 개발한 발명을 사용자(기업)가 명확하게 승계하지 않은 경우, 특허법 제62조에 따라 심사단계에서 특허거절결정되거나 특허법 제133조에 따라 등록이후에 특허무효사유가 될 수 있습니다.
8.기타 심사 절차에 관한 사항은 동봉된 안내서를 참조하시기 바랍니다.

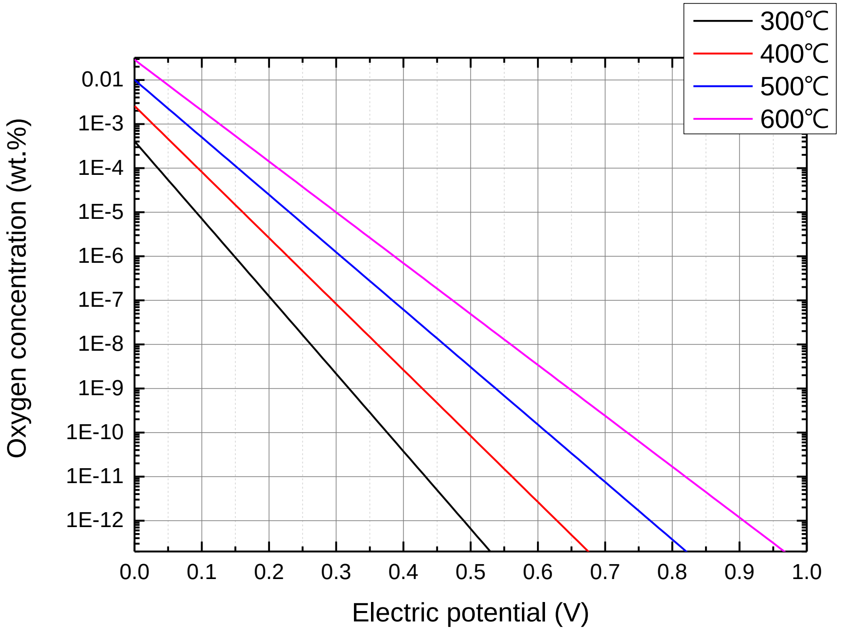
2021-01-18

※ Provisional patent application completed.
(10-2021-0006668, Korea)

(3) Details of experimental facility



Specification	
Manufacturer	SCK-CEN (Belgium)
Type	Bi/Bi2O3 reference electrode
Sensor materials	YSZ and steel sheath
Medium	LBE
Environment	Ar
Operational temperature(℃)	300~



$$C_O = \exp[4.595 - \left(\frac{7104 + 23209\,E}{T}\right)]$$

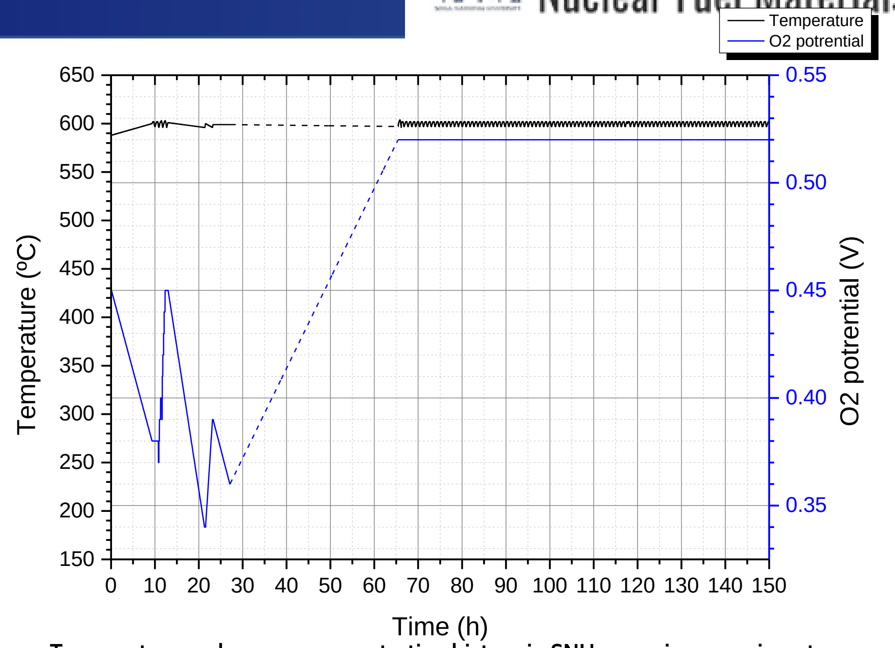
Actual figure and blueprint of Oxygen sensor

Relationship between the electrical potential indicated by the sensor and the dissolved oxygen concentration in LBE

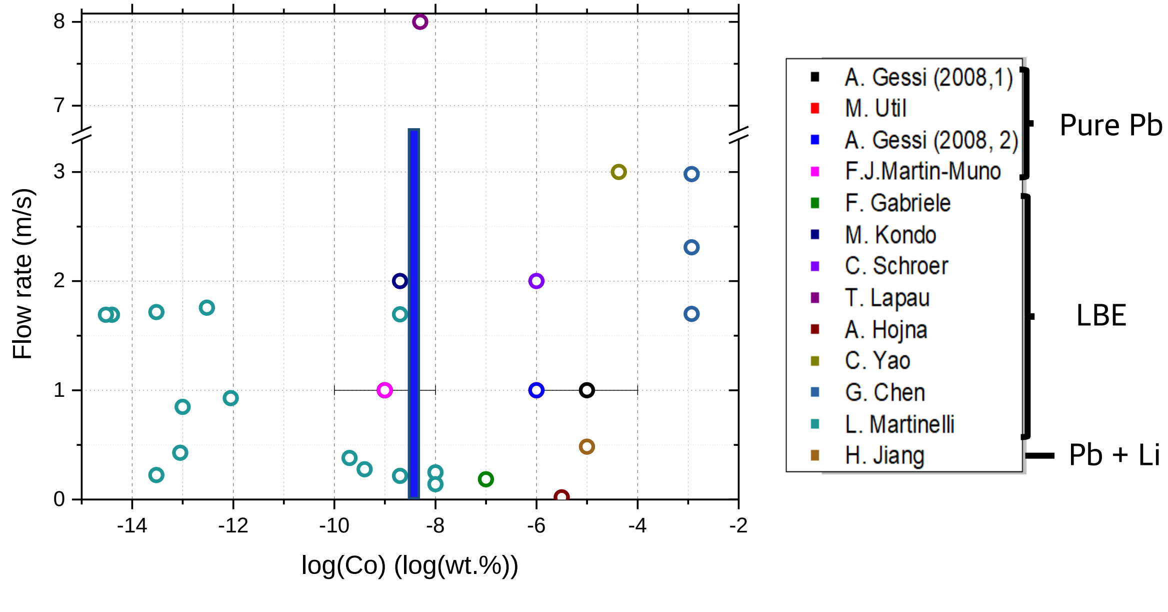
(1) Experiment environment

Lee's Lab
Nuclear Fuel Materials and Safety

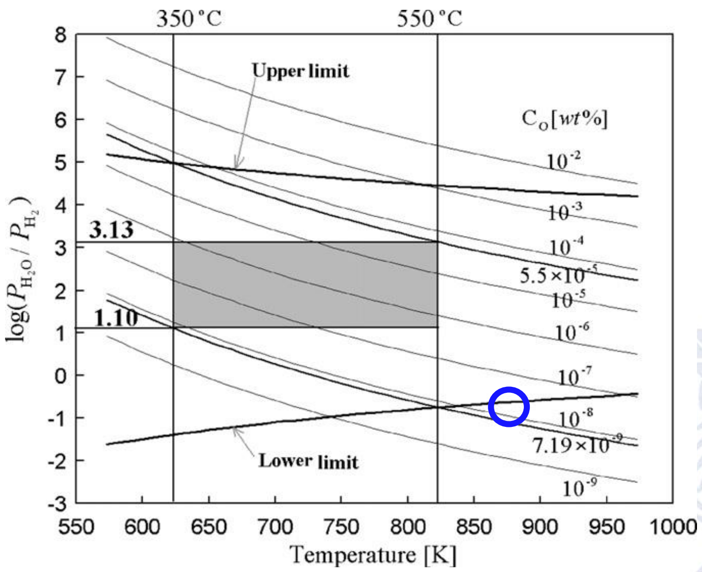
Experiment environment	
Time	~150h
Temperature	600℃
Oxygen concentration	2.87×10^{-8} wt.%
Rotative velocity	400 RPM
Max. linear velocity on specimen	3.35 m/s
Pressure	1.2 bar
Material	SUS 316L



Temperature and oxygen concentration history in SNU corrosion experiment



Flow rate and oxygen concentration conditions of existing researches



H2 and H2O mixture in the cover gas for controlling oxygen level

<Jinsuo Zhang, Ning Li, Review of the studies on fundamental issues in LBE corrosion, Journal of Nuclear Materials, Volume 373, Issues 1-3, 2008>

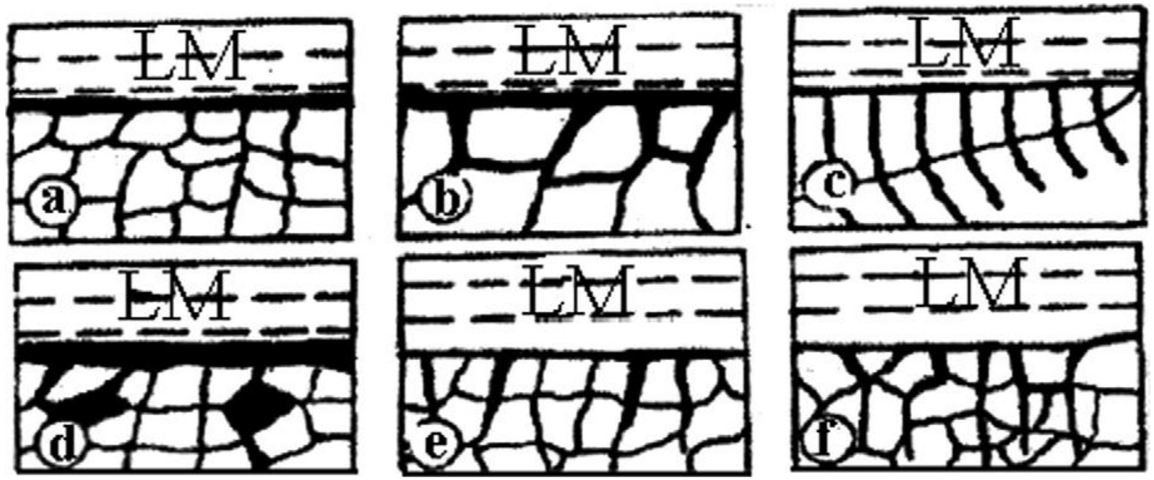
(1) Experiment environment

Specimens after cleaning using 150℃ of glycerin

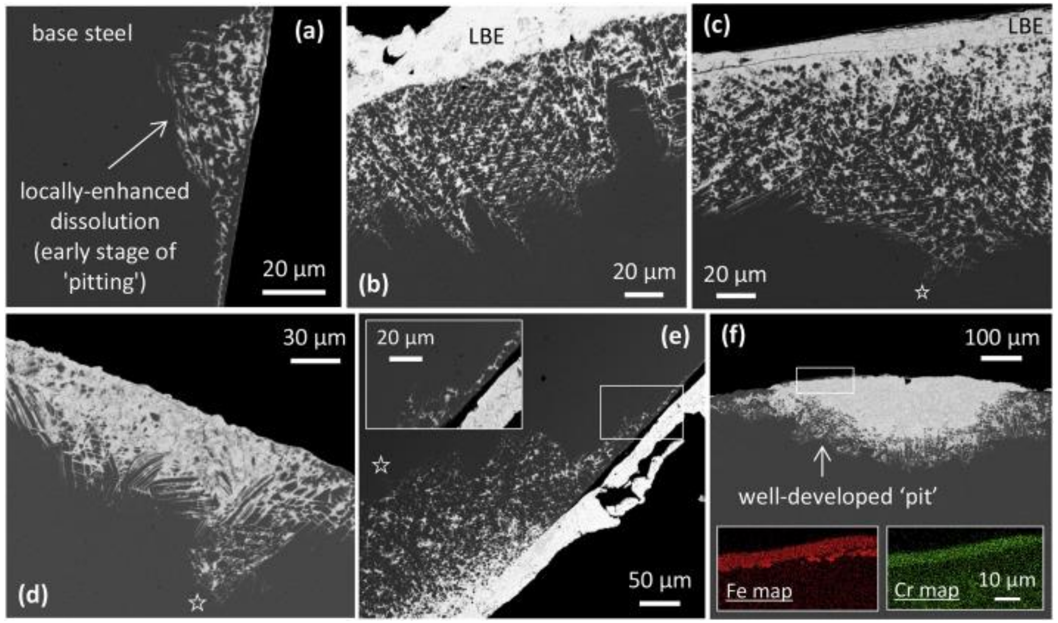
Specimen holder after cleaning using 150℃ of glycerin

- The effects of corrosion that can be observed even by the eyes
- Common color change in specimens on similar locations
- Erosion occurred at the tip of the specimens

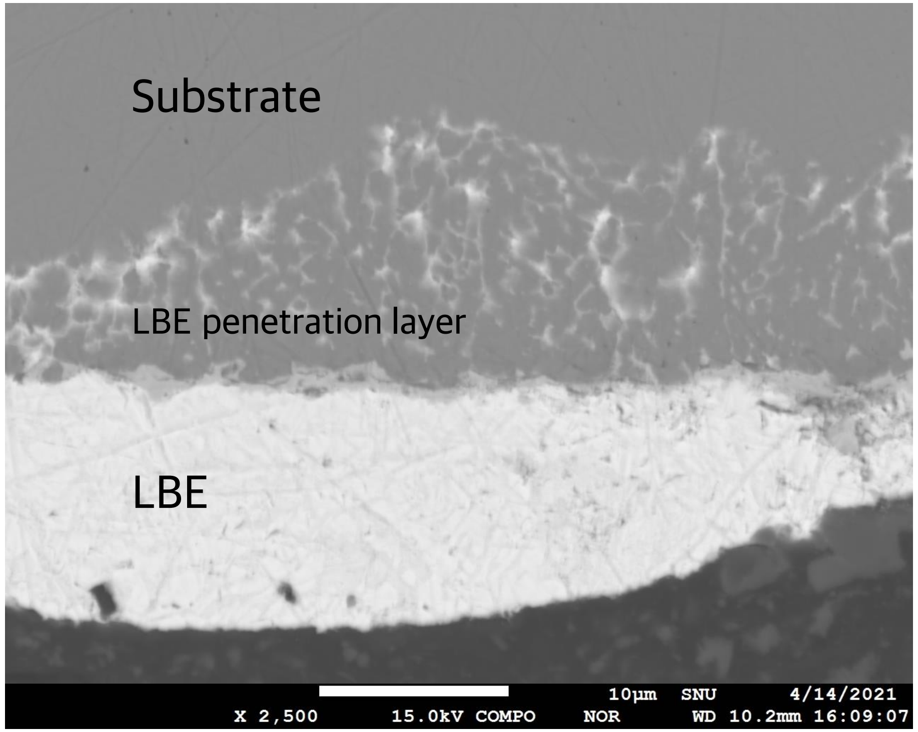
(2) Elemental composition changes according to flow velocities



<Jinsuo Zhang, Ning Li, Review of the studies on fundamental issues in LBE corrosion, Journal of Nuclear Materials, Volume 373, Issues 1-3, 2008>

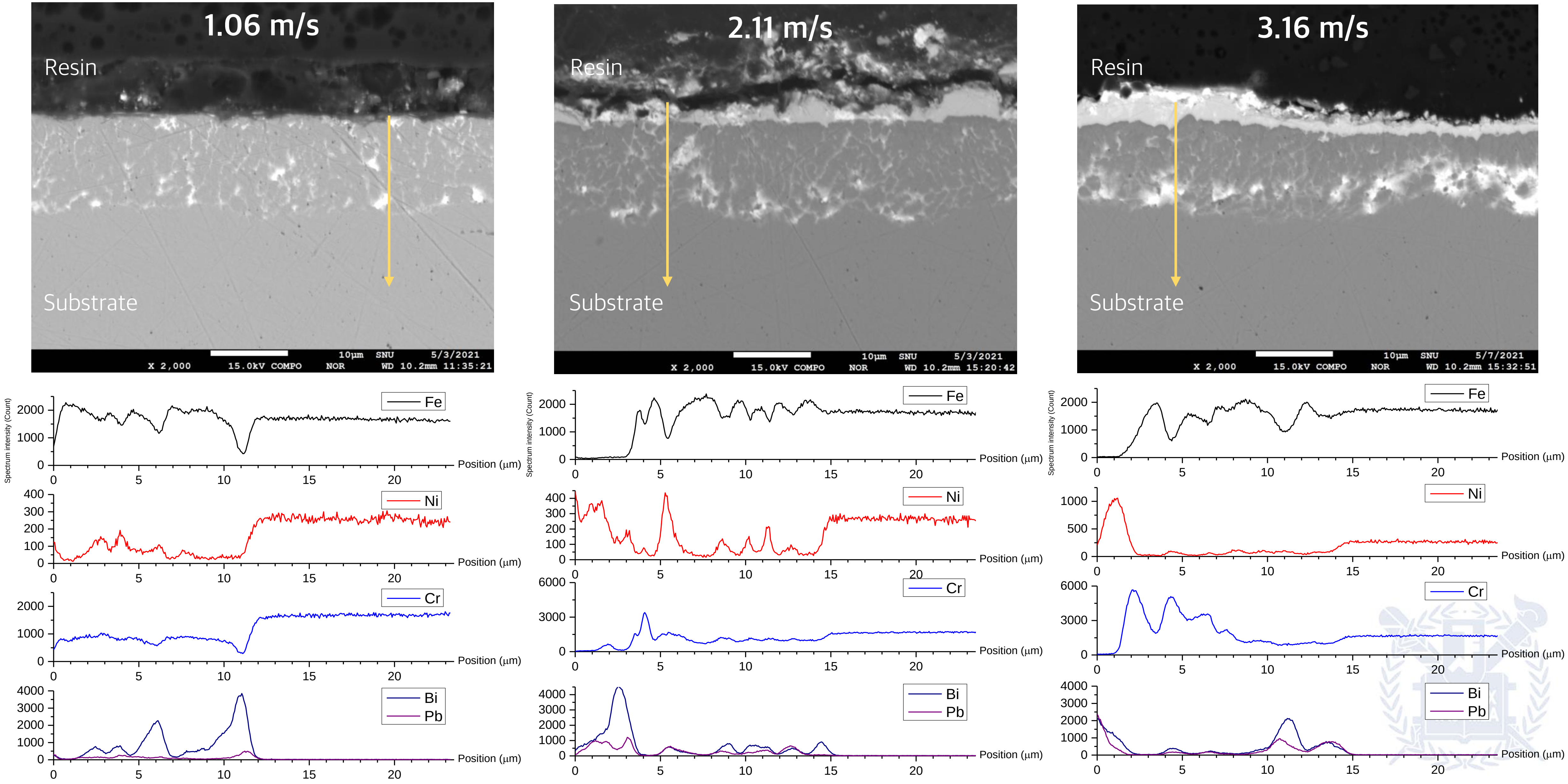


<Konstantina Lambrinou, Evangelia Charalampopoulou, Tom Van der Donck, Rémi Delville, Dominique Schryvers, Dissolution corrosion of 316L austenitic stainless steels in contact with static liquid lead-bismuth eutectic (LBE) at 500 °C, Journal of Nuclear Materials, Volume 490, 2017,>



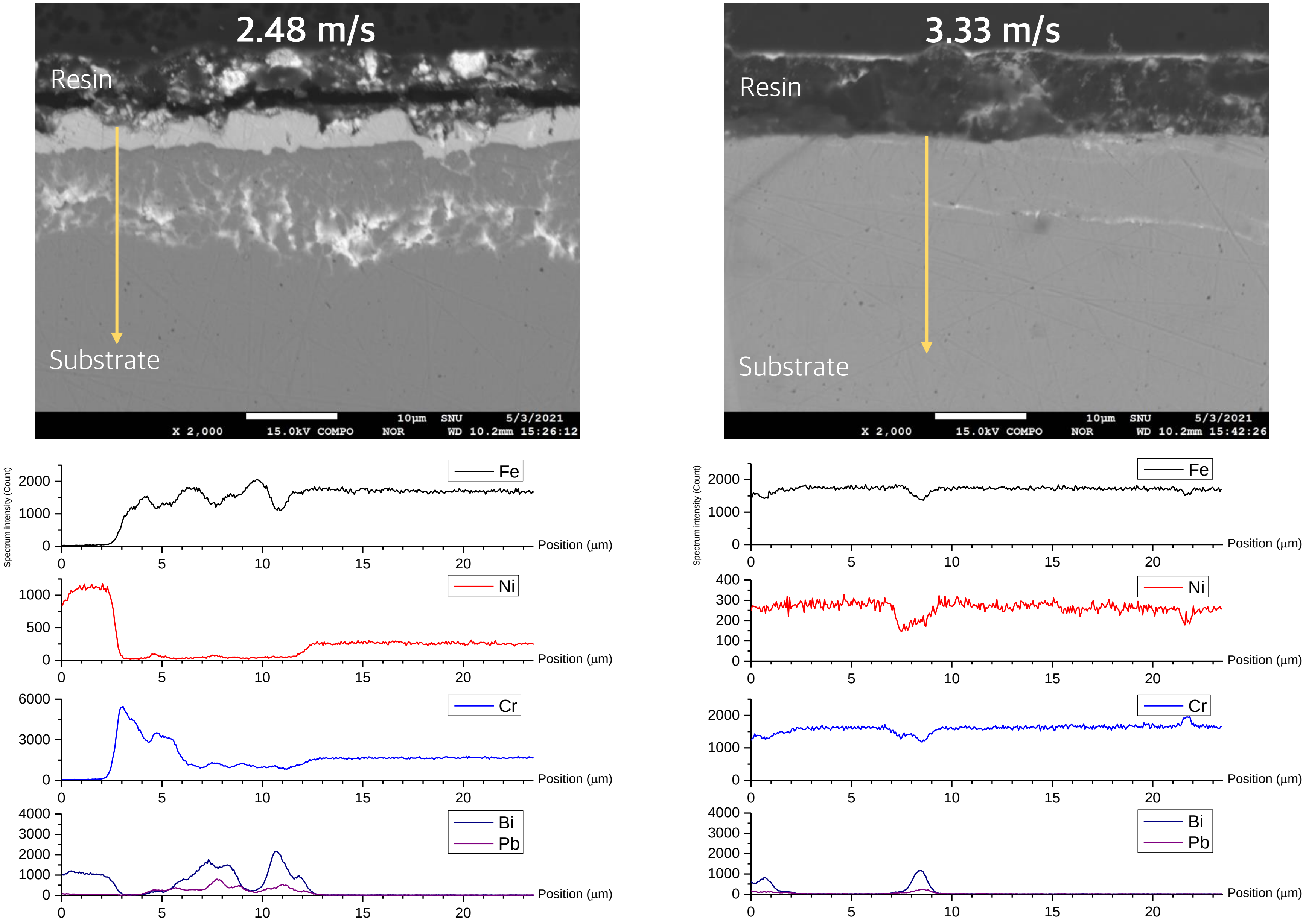
A SEM image of a specimen exposed by LBE in SNU corrosion experiment

(2) Elemental composition changes according to flow velocities



<SEM images and EPMA line-profile of the cross sections>

(2) Elemental composition changes according to flow velocities

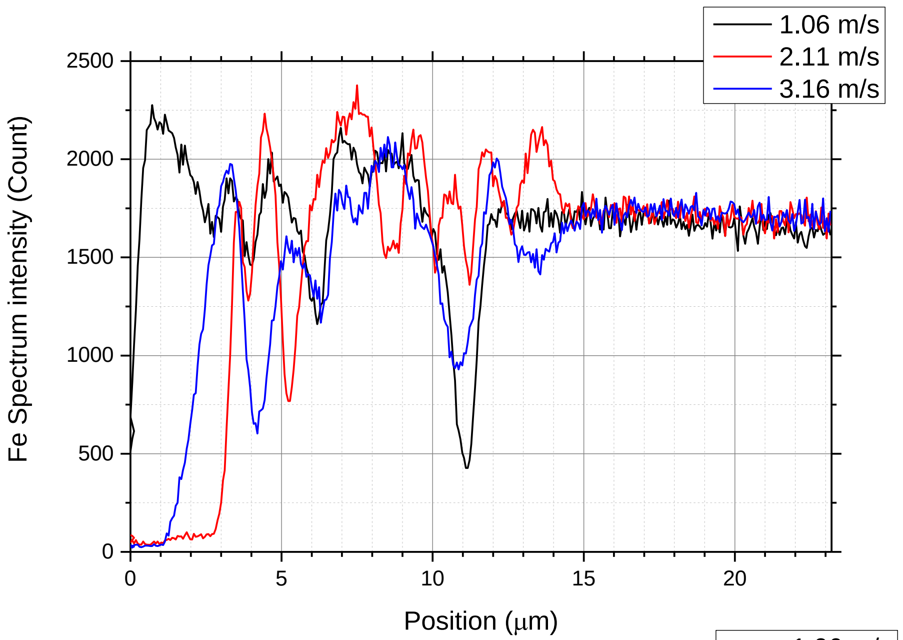


<SEM images and EPMA line-profile of the cross sections>

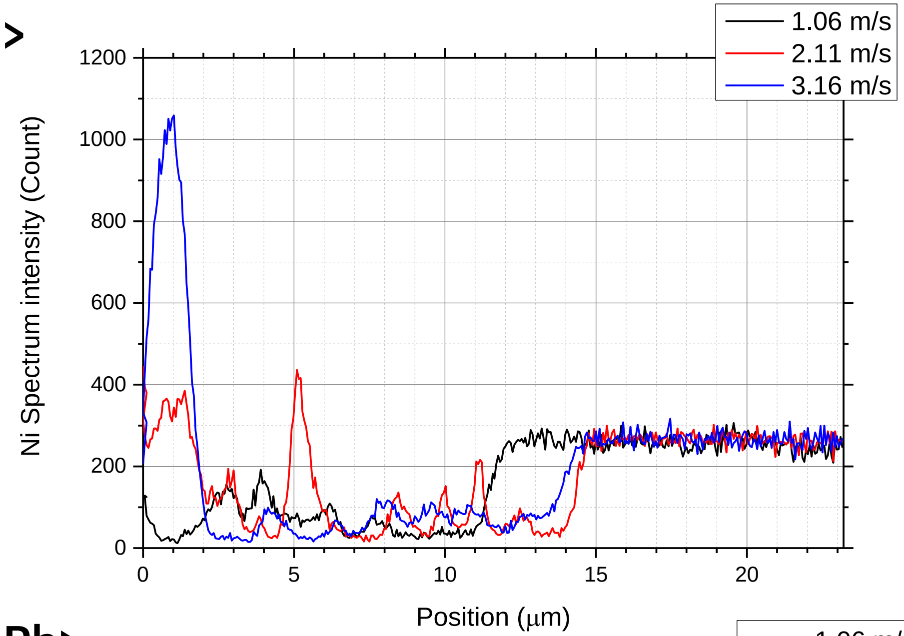
Specimens after cleaning using 150℃ of glycerin

(3) Elemental composition changes according to elements

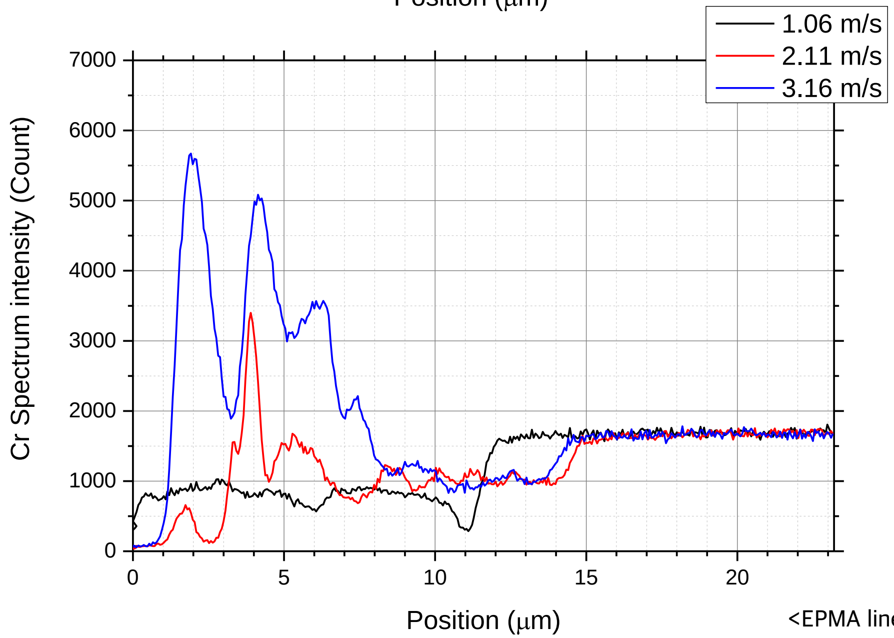
<Fe>



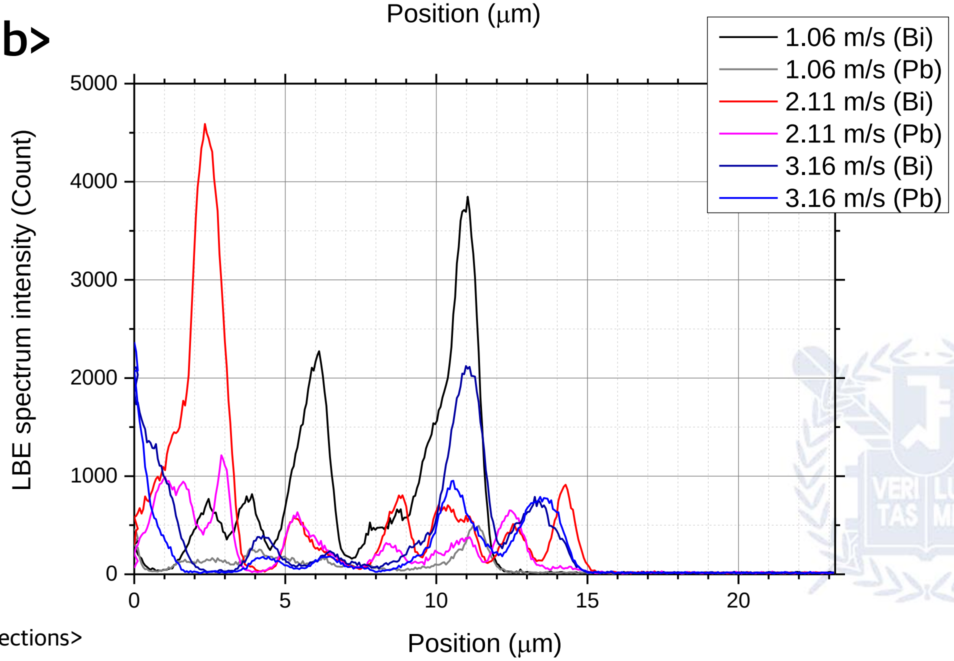
<Ni>



<Cr>

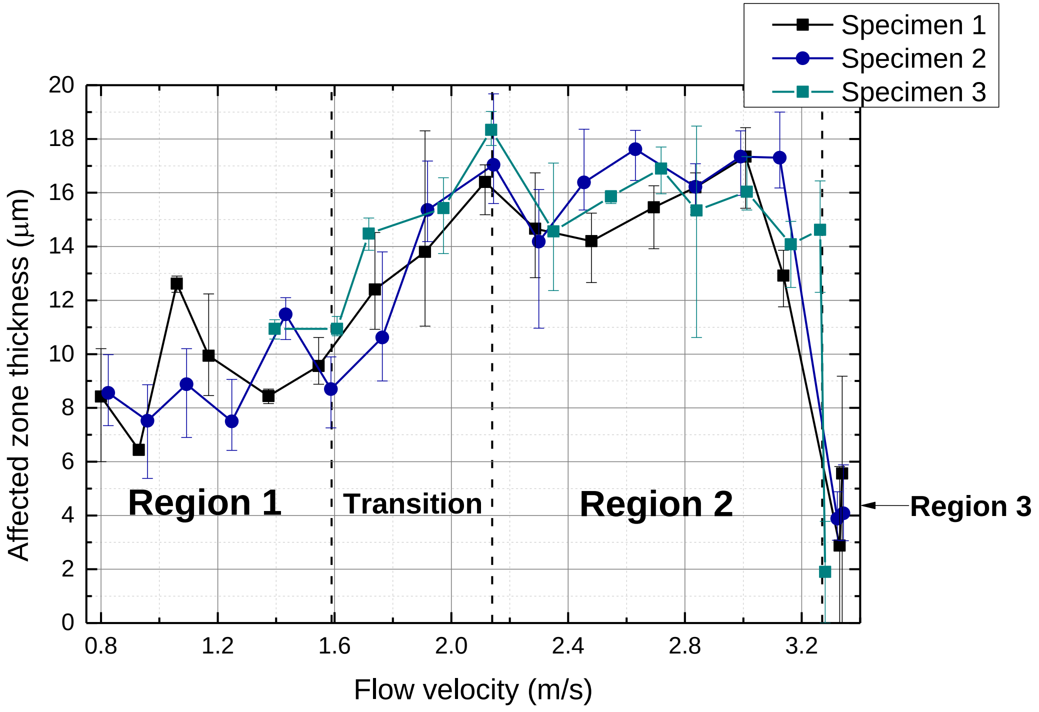


<Bi / Pb>

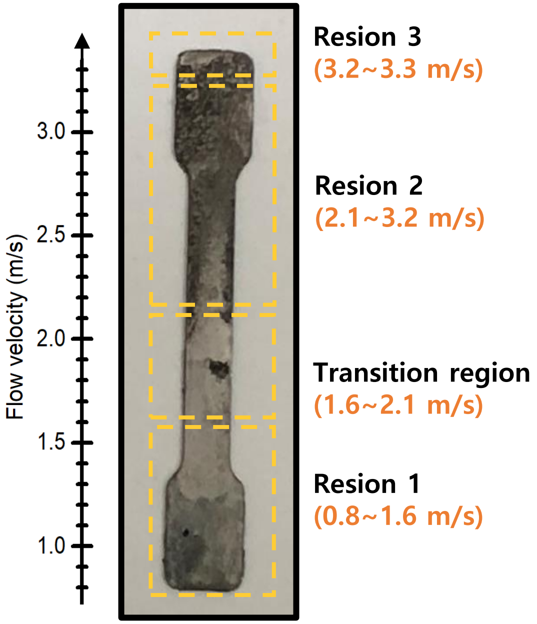


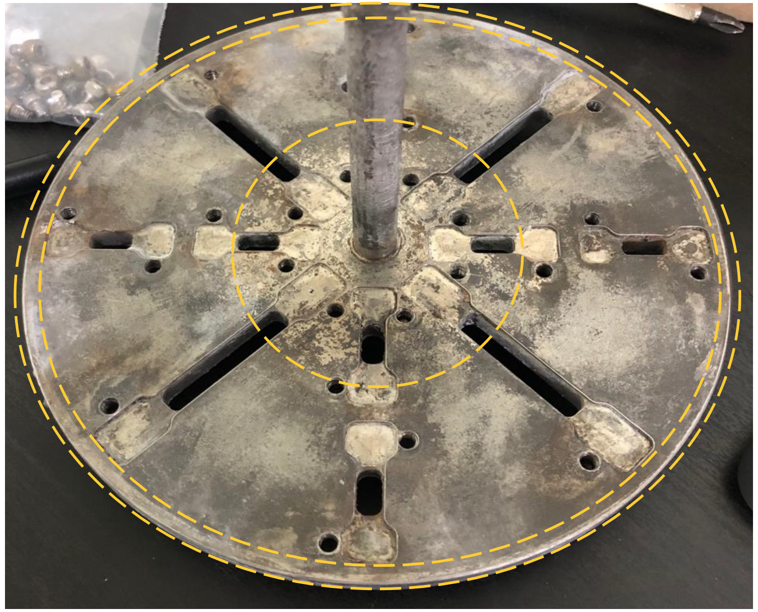
<EPMA line-profile of the cross sections>

(4) Affected zone thickness



Affected zone thickness following flow velocity of LBE

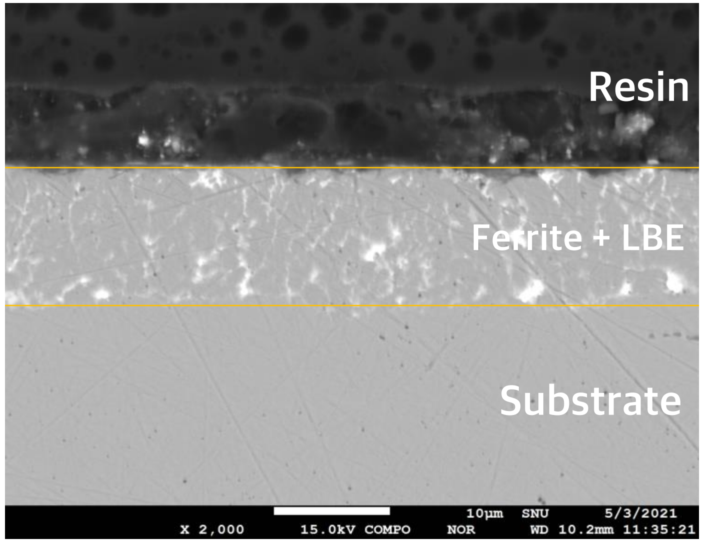


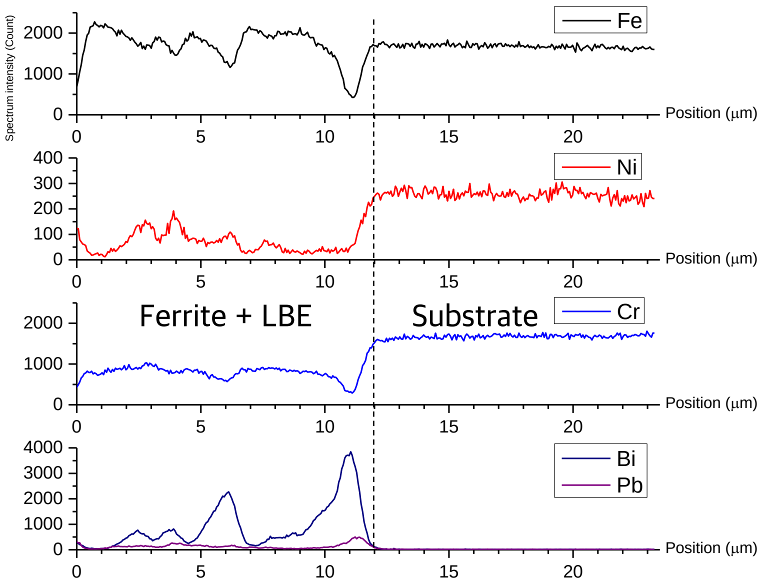


- Ununiform and statistical corrosion behavior
- <u>Affected zone thickness can be a significant factor for mechanical behavior modeling.</u>
- Corrosion modes can be divided 4 regions following rate of change in the thickness and corrosion layer differentiation.

(5) Velocity dependent corrosion modes

(1) Region 1: low velocity region (0.8~1.6 m/s)





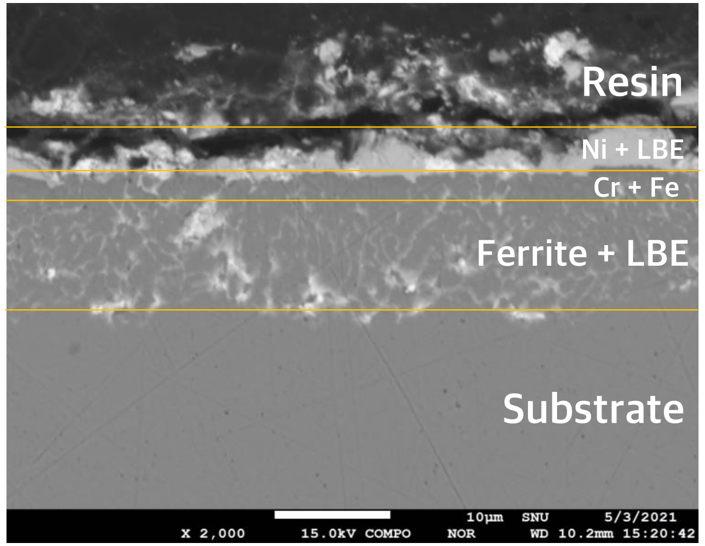
- **Affected zone thickness ~ 10μm**
- **LBE penetration +ferrite layer formation**

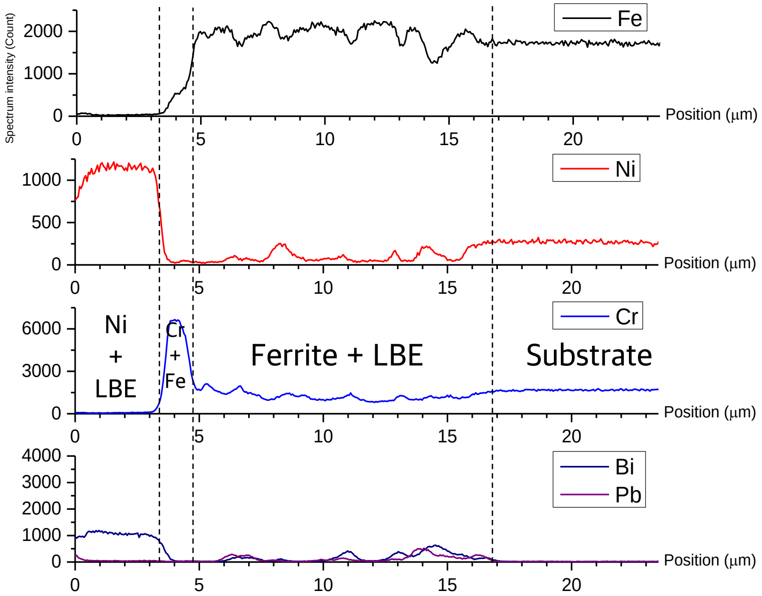
- LBE partly penetrated through the grain boundary or crystal structure defects
- Ni, Cr concentration ($C_{\mathrm{Ni}}, C_{\mathrm{Cr}}$) decreased
- High Fe concentration at the non-penetrated region ($C_{\mathrm{Fe}} > C_{\mathrm{Fe},0}$)

- Corroded region consists of a single layer.

(5) Velocity dependent corrosion modes

(2) Transition region (1.6~2.1 m/s)





- **Affected zone thickness increases from 10μm to 17μm**

- **LBE Penetration + ferrite layer**

- $C_{\mathrm{Ni}}, C_{\mathrm{Cr}} \downarrow$ $C_{\mathrm{Fe}} \uparrow$
- Local corrosion
- Thickness ~ 10μm

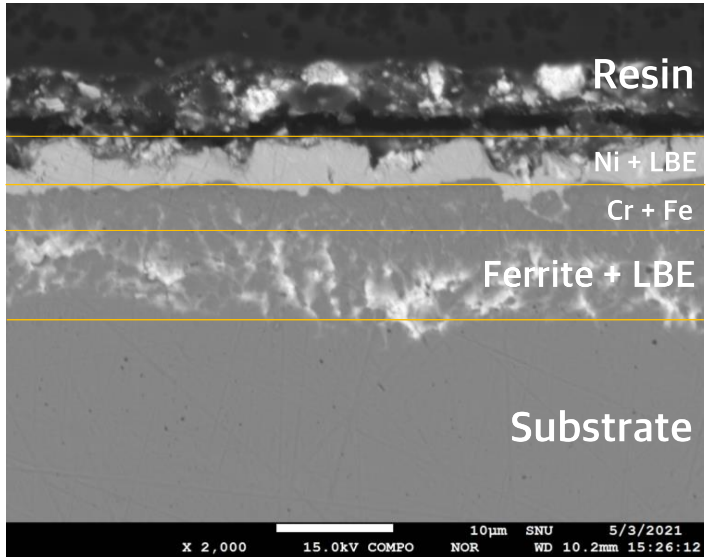
- **Cr+Fe / Ni+LBE layer**

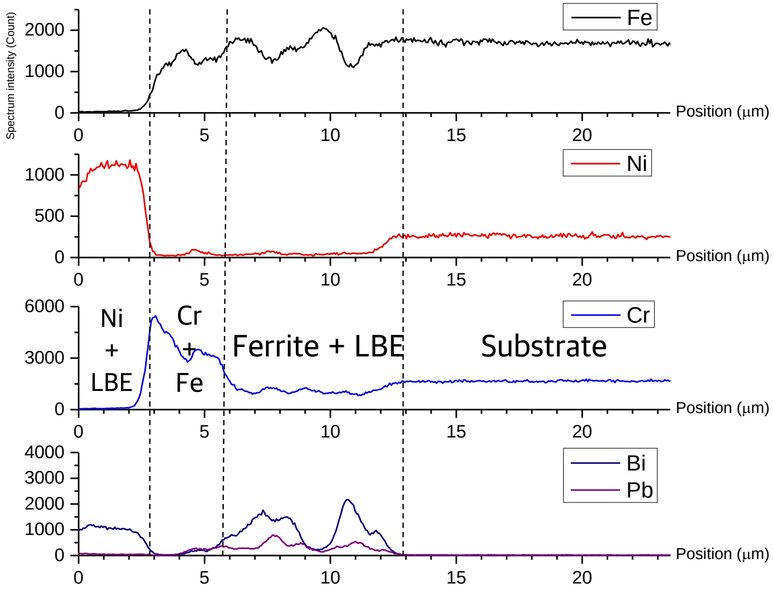
- High flow velocity (2~3 m/s) and low oxygen concentration accelerate diffusion and accumulation of Cr and Ni on the surface
- On each layer, $C_{\mathrm{Cr}} > C_{\mathrm{Cr,0}}$ $C_{\mathrm{Ni}} > C_{\mathrm{Ni,0}}$
- Uniform corrosion

- <u>Cr+Fe layer and Ni layer thickness rapidly increases in the transition area.</u>

(5) Velocity dependent corrosion modes

(3) Region 2: medium velocity region (2.1~3.2 m/s)





- **Affected zone thickness ~ 17μm**
- **LBE Penetration + ferrite layer**

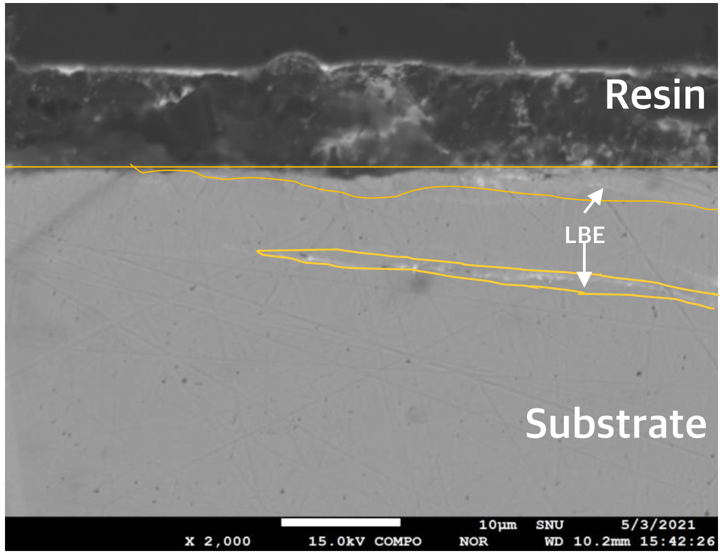
- $C_{\mathrm{Ni}}, C_{\mathrm{Cr}} \downarrow$ $C_{Fe} \uparrow$
- Local corrosion
- Thickness ~ 10μm

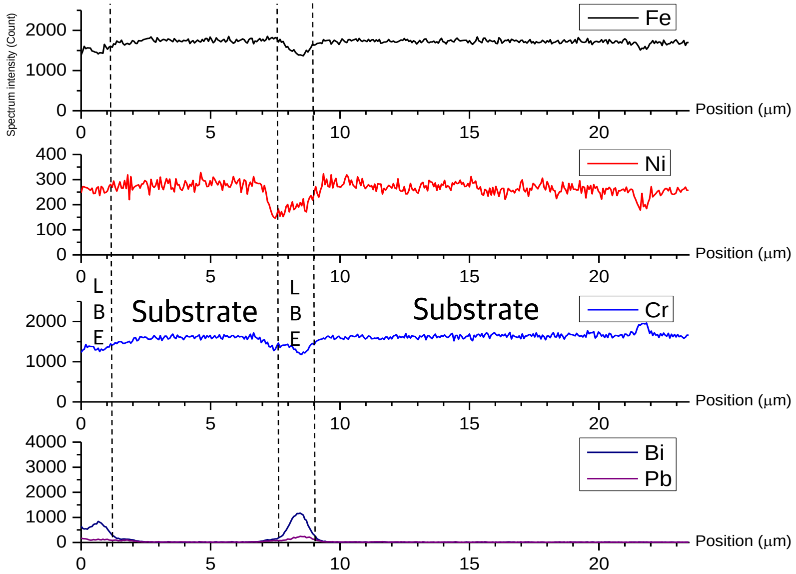
- **Cr+Fe/ Ni+LBE oxide layer**

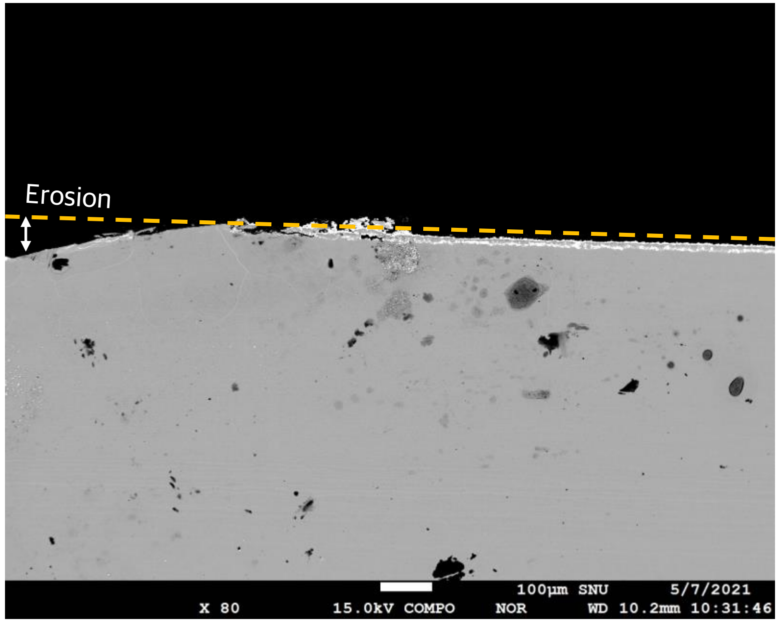
- Both layers are fully developed
- The total corrosion depth stops increasing.

(5) Velocity dependent corrosion modes

(4) Region 3: high velocity region (3.2~3.3 m/s)





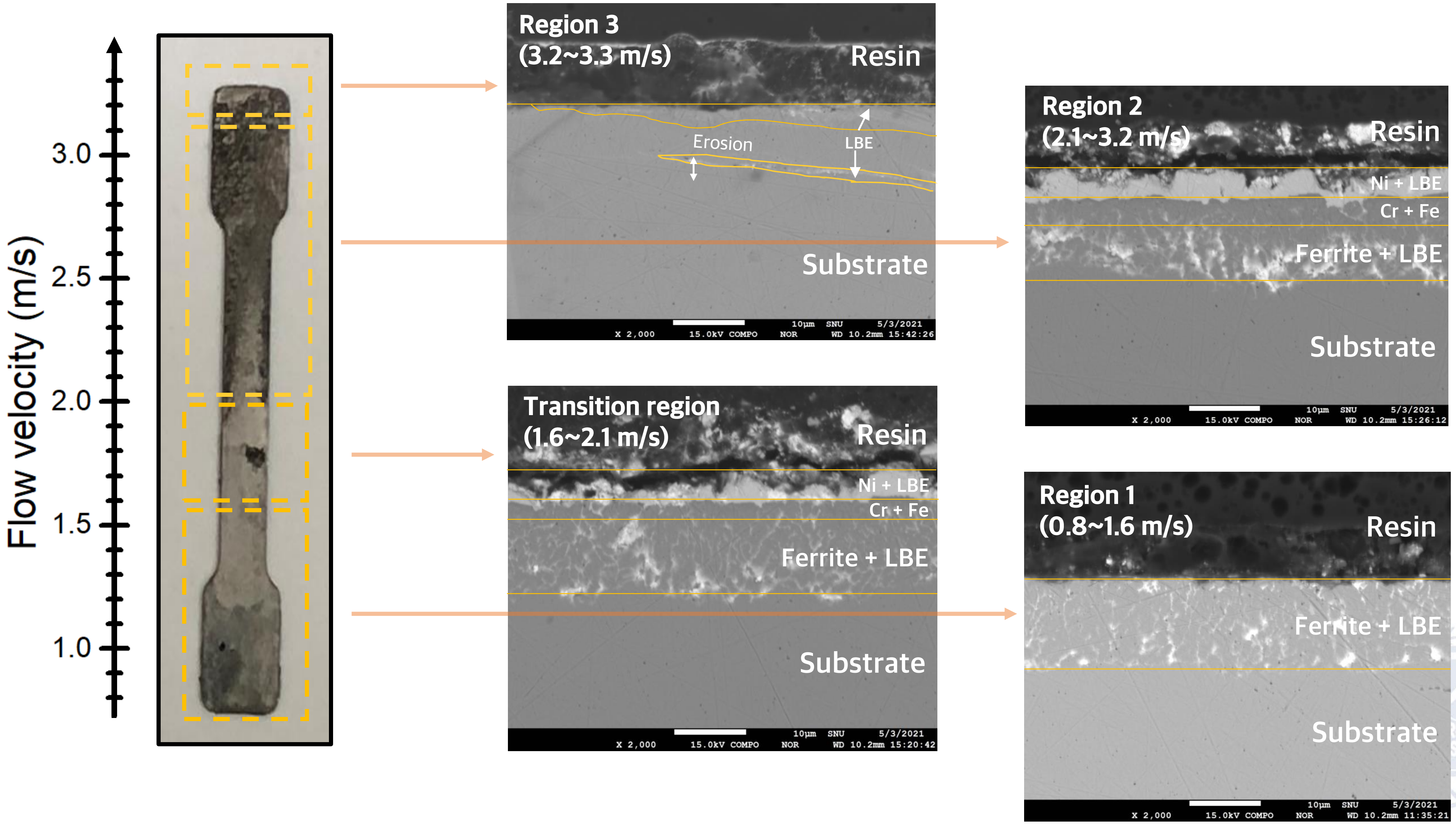


- **Erosion takes place.**
- **Totally different corrosion aspect**

- LBE penetration through grain boundary seldom occurs.
- Affected zone thickness : 0~5 µm

- **Non-continuous layer composition**

(5) Velocity dependent corrosion modes



3. Conclusion

- Experimental device which can control temperature and oxygen concentration and expose continuous flow velocity to specimens was developed.

- As a result of 150h corrosion experiment, it is shown that corrosion on SUS 316L was ununiform and statistical.

- Affected zone thickness(total corrosion depth) would be the best approach to the mechanical behavior modeling from an empirical point of view.

- Corrosion modes can be divided 4 regions following rate of change in the affected zone thickness and corrosion layer differentiation.

3. Conclusion

- Experiment campaigns with more various condition are going to be carried out for developing modeling.

- Tensile test to investigate the correlation with affected zone thickness and mechanical strength is planned

- Accumulation of corrosion data on candidate materials in LFR and ceramics (Al_2O_3, SiO_2, etc.)

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