Corrosion-erosion test of SS316L grain boundary engineering material (GBEM) in lead bismuth flowing loop

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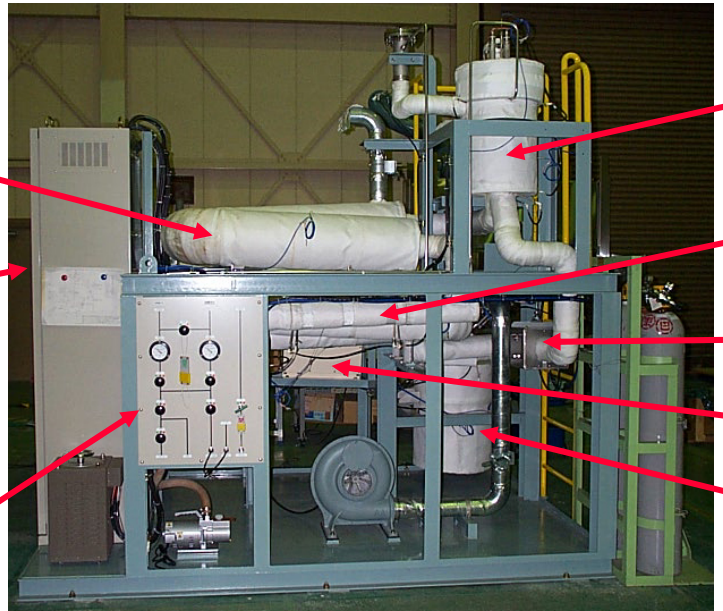
Background

- ADS (Accelerator-driven system) has many advantages
  Higher safety margin, flexibility for fuel composition, and so on.
- Spallation target /Coolant
  LBE/LBE is selected and LBE technology has been developed in many countries.
- J-PARC Project
  ADS target experimental facility (TEF-T) is planned to construct in phase II.
- Lead-bismuth material corrosion loop-1（JLBL-1）
  To evaluate feasibility on structural steels and to obtain LBE handling technique.
  We have done over 20,000 hour operation.

JLBL-1

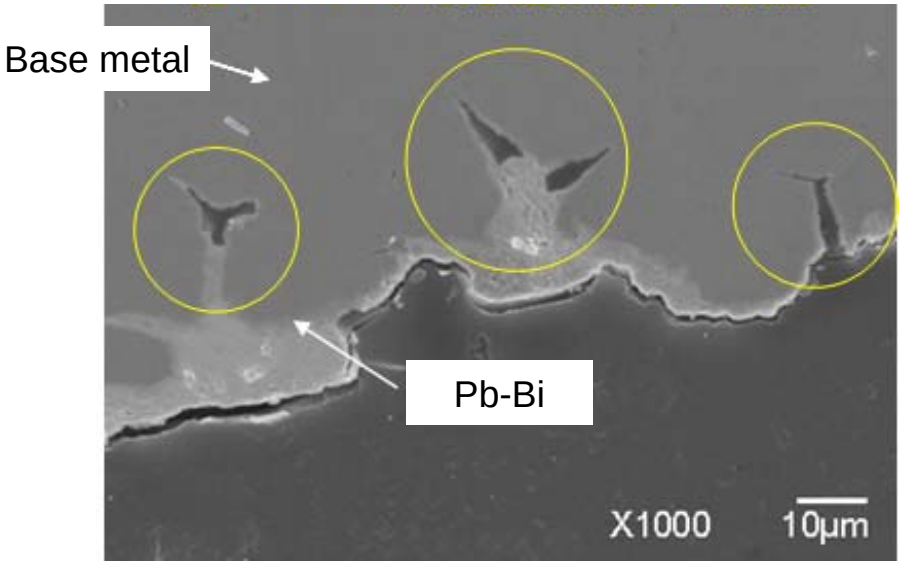


Introduction (1)

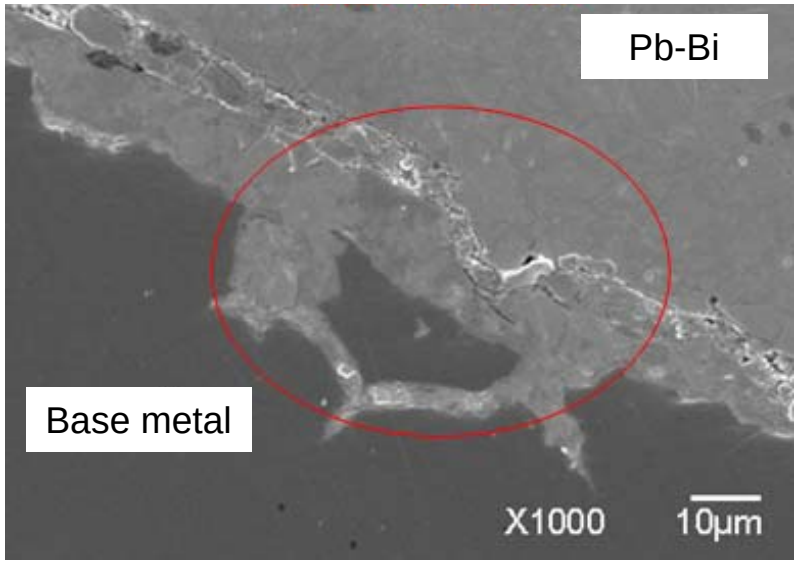
Austenitic steel corrosion in LBE

・At high temperature (450℃), austenitic steel SS316L showed severe corrosion-erosion in LBE, for instance, a depth of corrosion in SS316 tube specimen was 100-300 μ m/3000 hours. LBE penetration through grain boundaries and grain drop were observed for the specimen.

LBE penetration through grain boundaries



Grain drop



SS316L (Phase3)

Introduction (2)

Purpose

・To investigate corrosion behavior of grain boundary engineering material (GBEM) in LBE.

In this study, the results of corrosion test of SS316L-GBE in lead bismuth flowing loop will be reported.

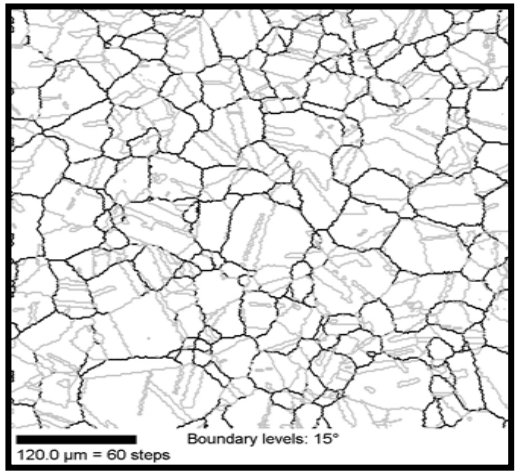
GBE

Grain boundary character distribution by FE-SEM/OIM [1]

Coincidence site lattice (CSL, low energy boundary)
Random boundary (high energy boundary)

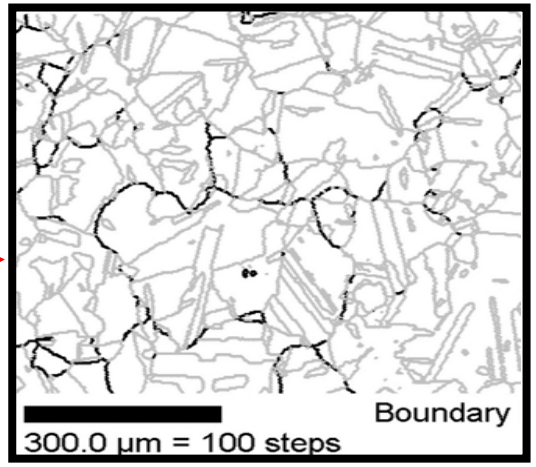
GBE is a thermo-chemically process to transform random boundaries of base metal to coincidence site lattice (CSL). It was reported that grain boundary corrosion resistivity of GBEM was apparently improved[1].

BM



GBE (Thermo-chemically process)

GBEM



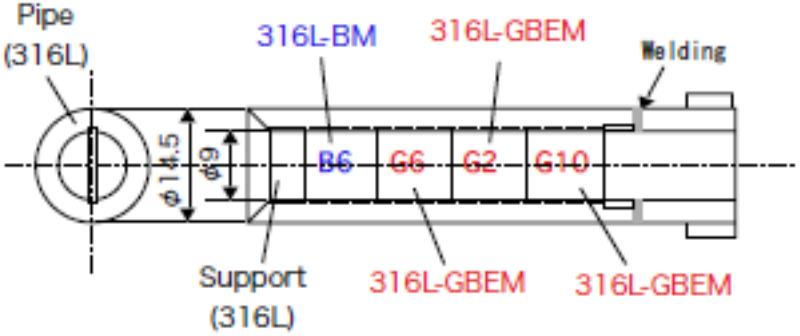
[1] M. Shimada, H. Kokawa, Z.J. Wang, Y.S. Sato, I. Karibe, Acta Materialia, 50 (2002), 2331-2341.

Experiment (1)

- GBEM：
  Tohoku Univ. Kokawa lab.
- Solution annealing : 1100℃, 30 min.
- Thermo-chemically process ：3% pre-strain,967℃,72 hour
- Frequency of CSL : 86% [2]

  [2] M. Michiuchi, H. Kokawa et. al., Acta Materialia, 54(2006)5179-5184.
- Material and specimen
  SS316L-BM and SS316L-GBEM
  （10 mm X10 mm X 1mm^t）

Specimen holder



Experiment（2）

- Test temperature
  Higher（Test section）/Lower : 450℃/ 350℃（ΔT=100℃）
- Flow speed at specimen holder :-0.7m/s
- Covering gas :99.995% Ar
- Oxygen concentration :Not measured
- Test time :-3600 hours
- Test :Metallographical observation,SEM,SIM, TEM/EDX

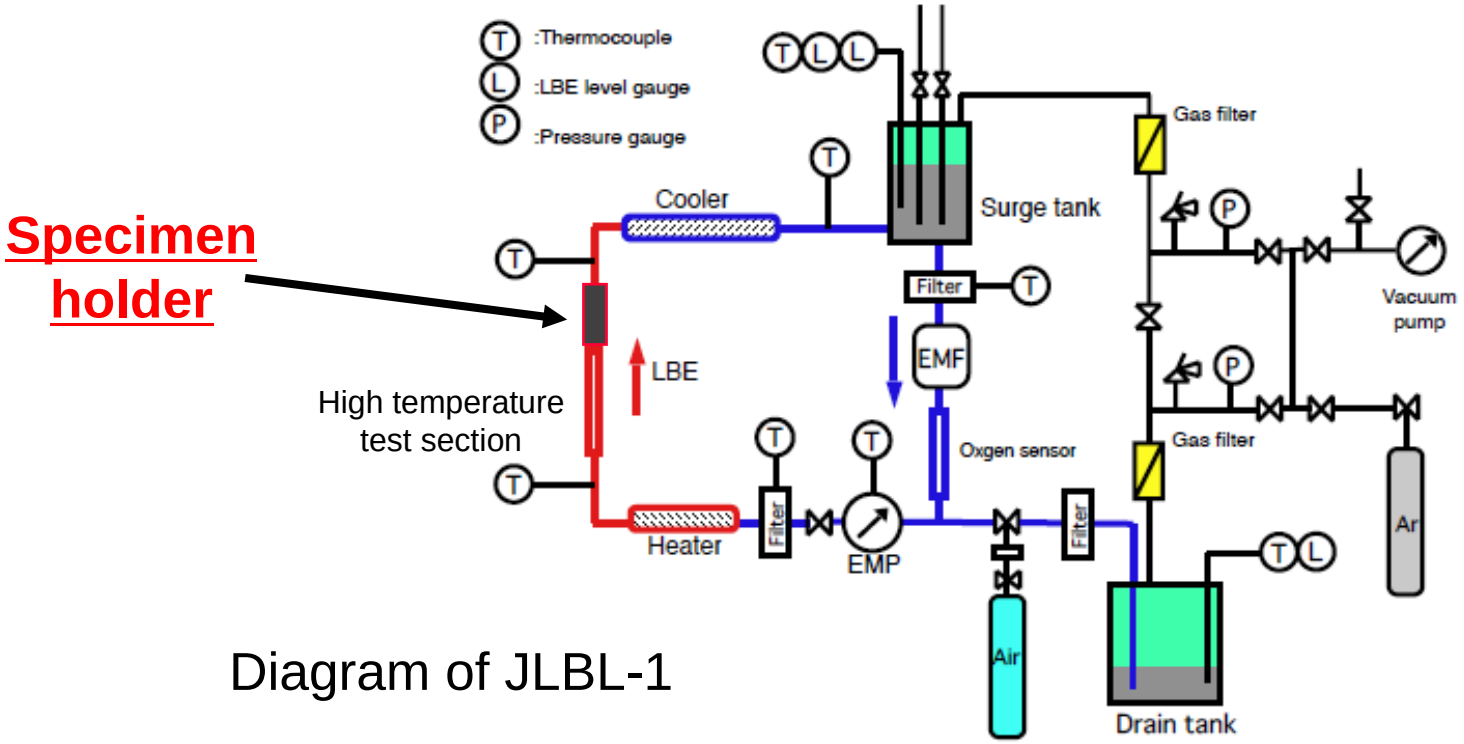
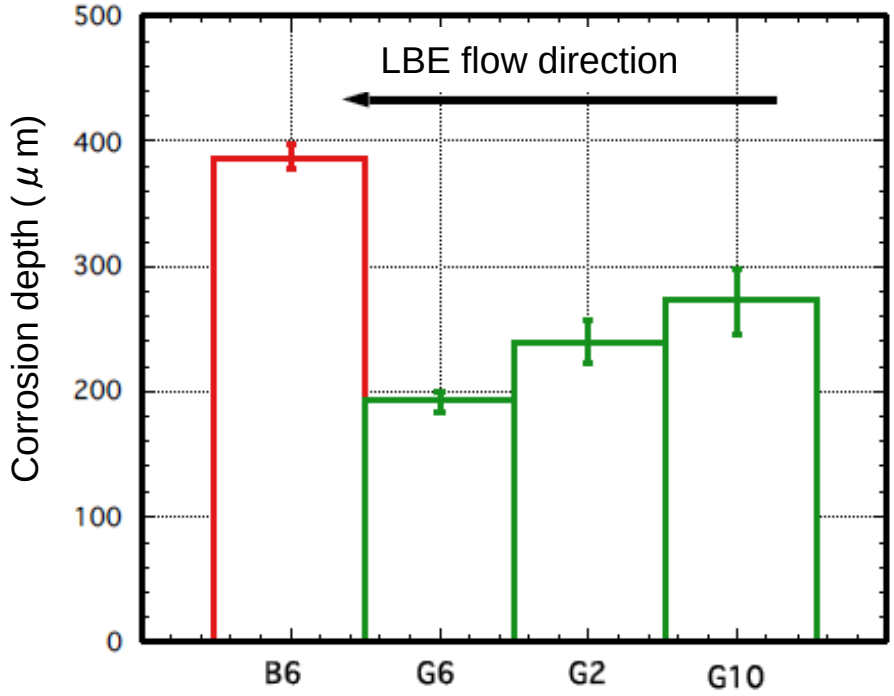


Diagram of JLBL-1

Results -corrosion depth-

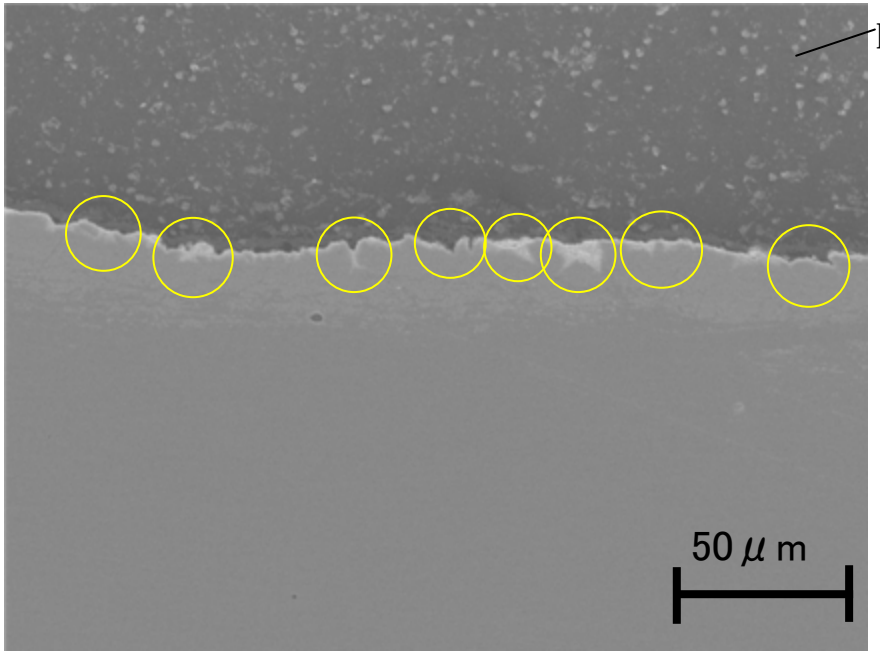


- SS316L-BM specimen (B6) shows the largest corrosion depth. Corrosion depth of BM is about 390 μ m/ one side.
- Corrosion depth of SS316L-GBEM is about 190-270 μ m/ one side (50-70% of BM).

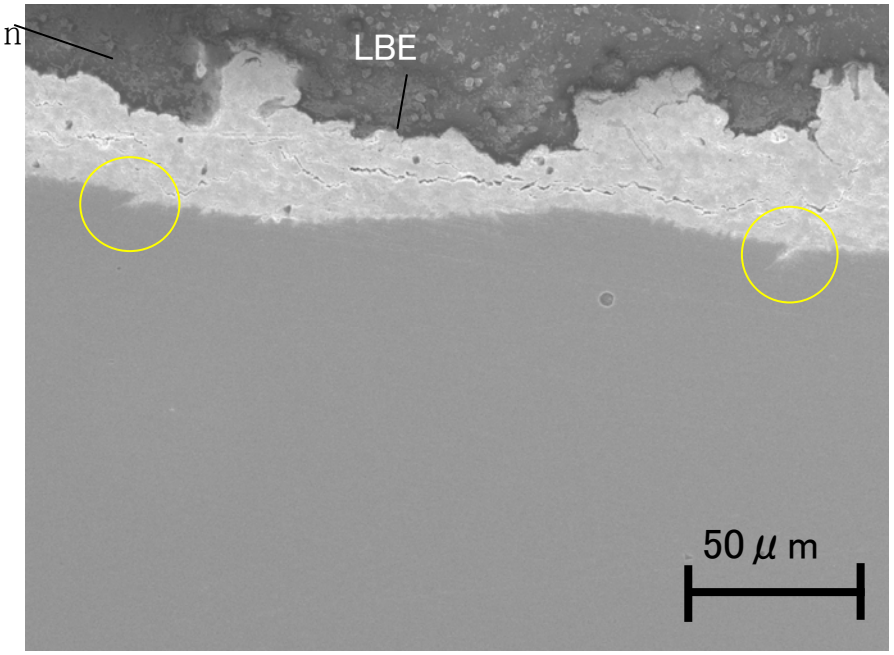
SEM observation

-Cross section-

BM (B6)



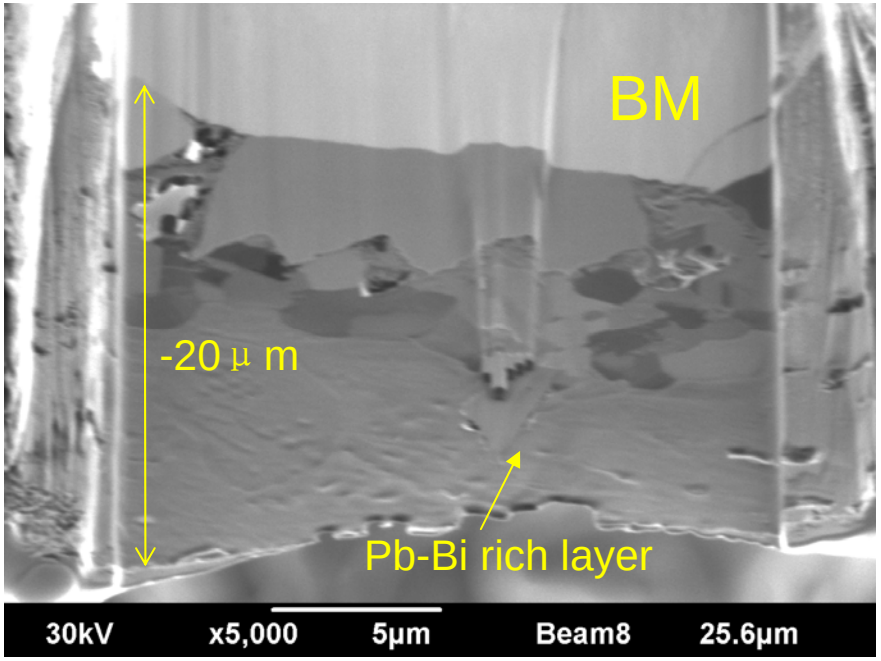
GBEM (G10)



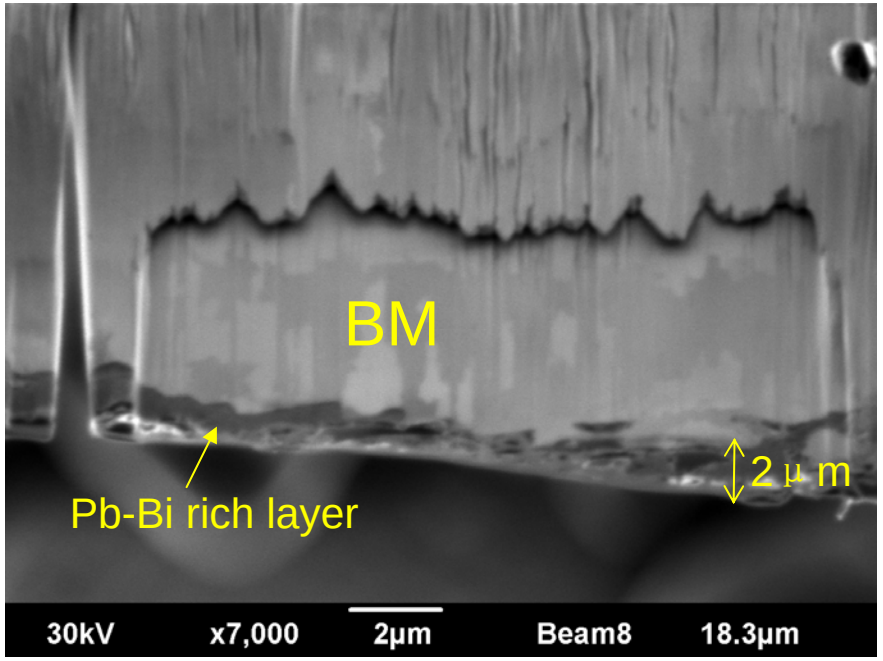
- The number of LBE penetration through grain boundary for GBEM is smaller than that of BM.
- No grain drop is observed for both specimens.

SIM (Scanning Ion Microscopy)

SS316L-BM

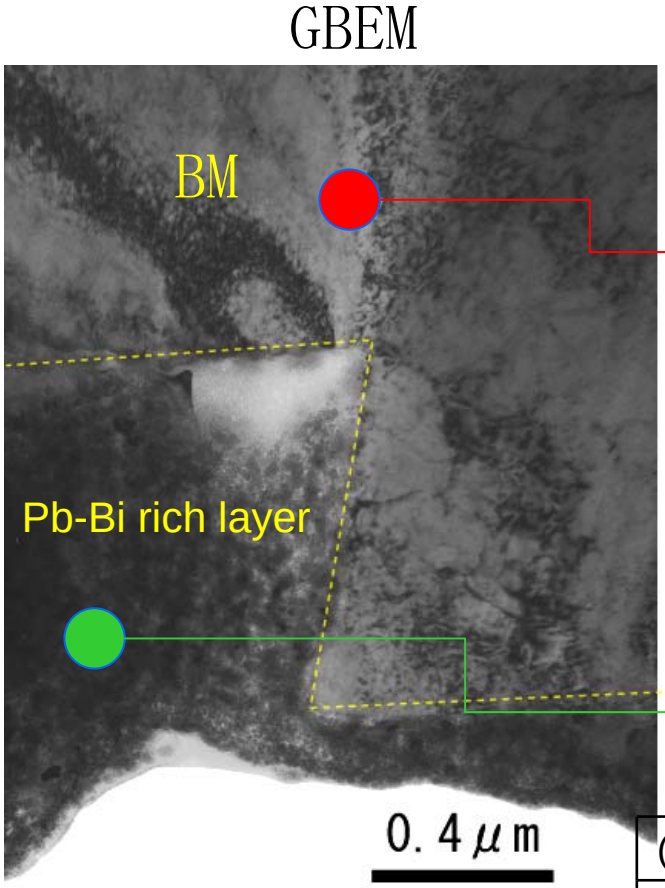


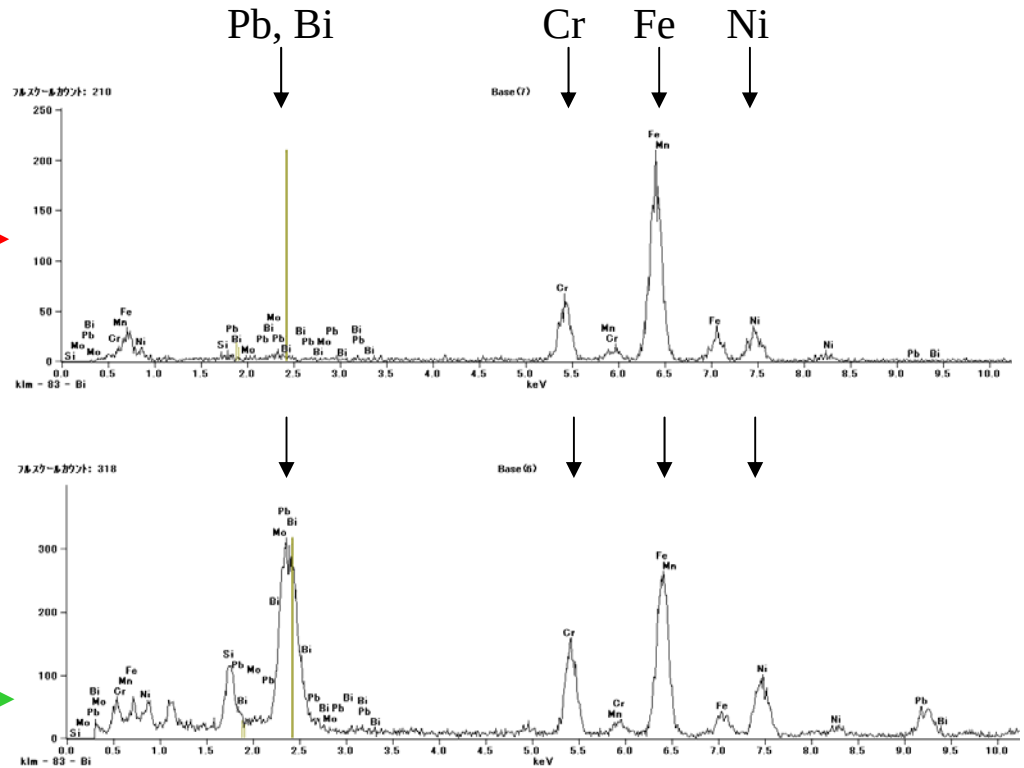
SS316L-GBEM



- The samples were prepared by FIB for TEM observation.
- The width of Pb-Bi rich layer are about 10-20 μ m for BM, about 2 μ m for GBEM.
- In this study, no oxide film is observed for BM and GBEM.

TEM/EDX analysis –GBEM-





(wt%)	Fe	Cr	Ni	Si	Mn	Mo	Pb	Bi
A	64.73	17.99	11.87	0.46	0.60	3.26	0.01	0.29
B	17.81	9.24	7.71	4.32	0.15	15.28	29.71	15.77

•In the Pb-Bi rich layer, dissolution of Fe, Cr, Ni and diffusion of Pb and Bi are observed.

Discussion (1)

- Ni, Fe and Cr dissolution and Pb-Bi penetration into materials are observed.
- （Fe, Cr）oxide layer are not observed.
- These results indicate the oxygen concentration is very low.

Reason of decrease of oxygen concentration

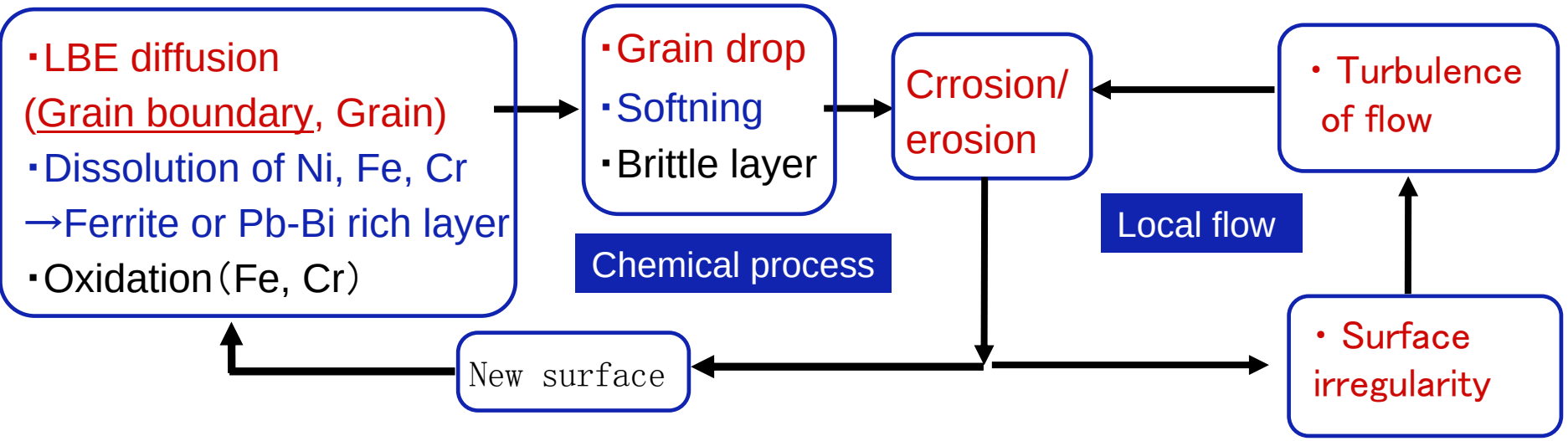
•In beginning of operation, dissolved oxygen is consumed by oxide formation, oxygen supply from cover gas is not enough.

•Oxygen concentration is decreased.

•After that, the oxide layer break away and corrosion will be progressed.

Discussion (2)

- In the condition of low oxygen concentration, corrosion/erosion is accelerated by mutual interaction of chemical process and local flow.
- In spite of low flow rate, corrosion/erosion will be occurred.



●In case of GBEM, grain boundary diffusion is supressed by interrupting continuity of grain boundary.

Summary (1)

Corrosion test was performed for SS316L-BM/GBEM specimens by JLBL-1 at 450℃, ΔT=100℃ for 3600 hours.

- Large corrosion depth by corrosion/erosion are observed.
- Oxide layer is not observed.
- The oxygen concentration during the corrosion test was very low.
- In spite of low flow rate, corrosion/erosion is occurred. Because corrosion/erosion is accelerated by mutual interaction of chemical process and local flow.

Summary (2)

- SS316L-GBEM showed 50-70% of corrosion depth and about 10-20 % of LBE diffusion zone width compared to SS316L-BM

GBE is found to be effective on improvement of corrosion resistivity in LBE.

Because material dissociation is suppressed interrupting continuity of grain boundaries.

Future plans

- TEM/EDX analysis around grain boundary.
- Modification of JLBL-1;
  - Oxygen sensor (position change, countermeasure for LBE leakage)
  - EMP maintenance
  - Filters exchange

  (Oxygen control system was installed.)

↓

Corrosion/erosion test under the oxygen concentration controlled condition.