

Post irradiation examination of the MEGAPIE samples at JAEA(2)

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MEGAPIE

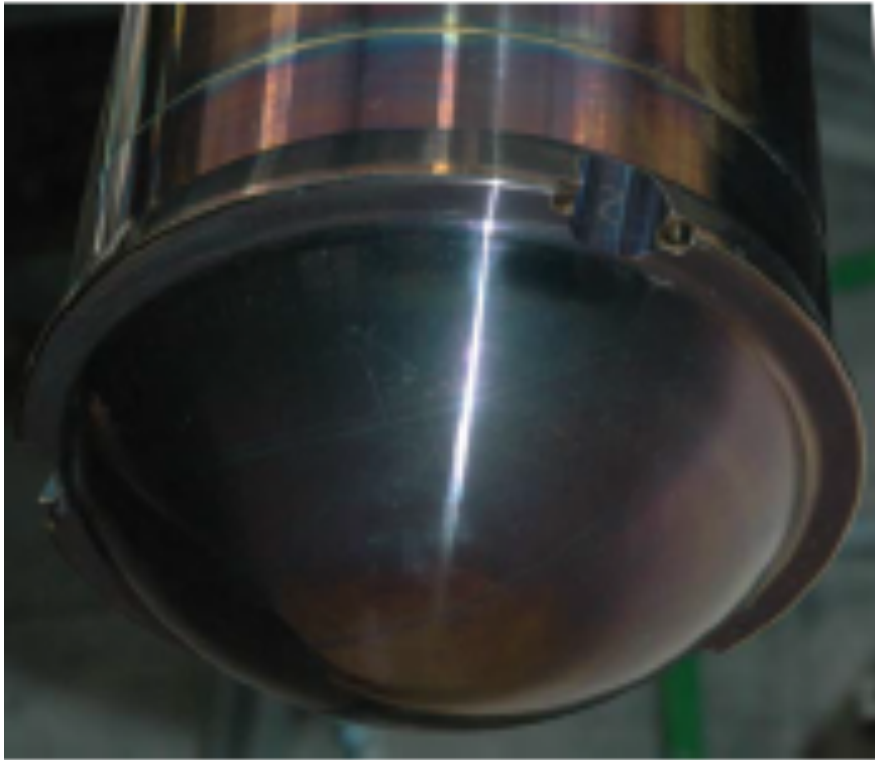
- The world's first megawatt-class lead-bismuth target, MEGAPIE (MEGAWatt Pilot Experiment) was successfully finished in 2006.
- The target was dismantled and post irradiation examination (PIE) samples were prepared at PSI hot-lab.
- The samples were shipped to each institutions including JAEA and PIE works have being performed.



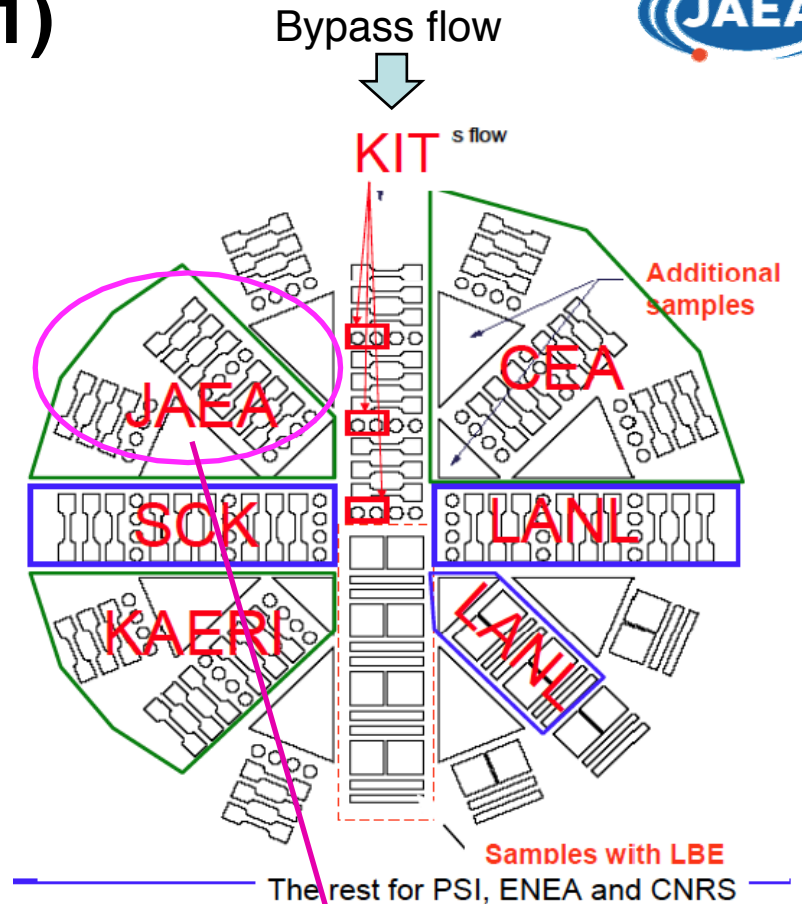
1. JAEA Samples (1)



JAEA samples : Beam window
Flow guide tube (FGT)
All samples were prepared without LBE.



Beam window (H02-1)
Material: T91



H02-1-14-A

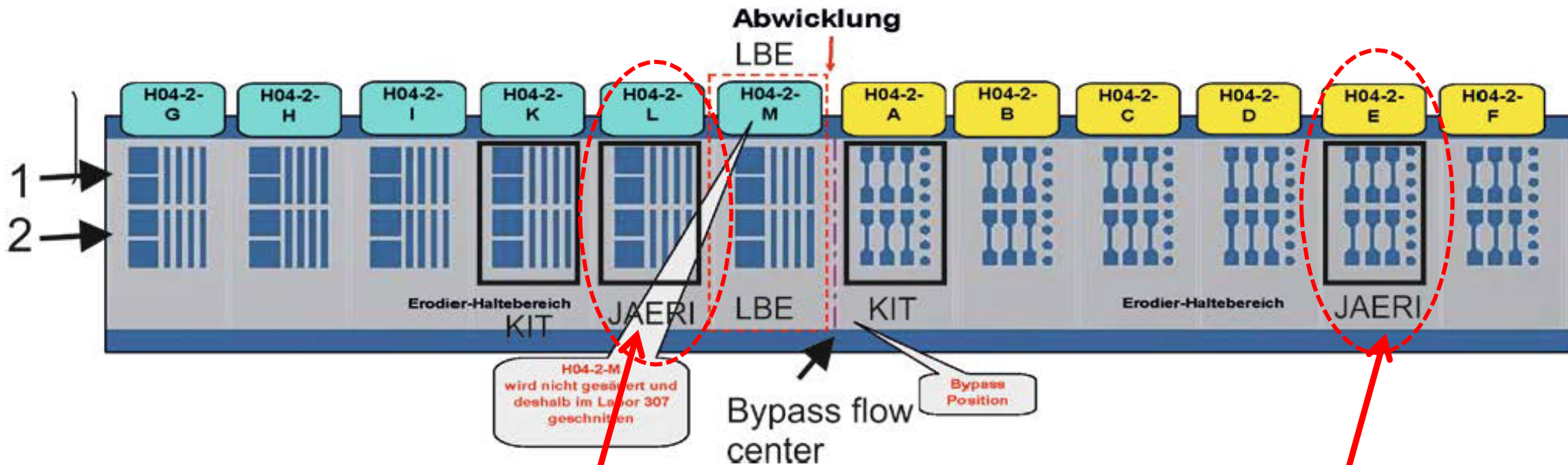
TEM disks



1. JAEA Samples (2)

Flow guide tube (FGT) (H04-2)

Material: SS316L

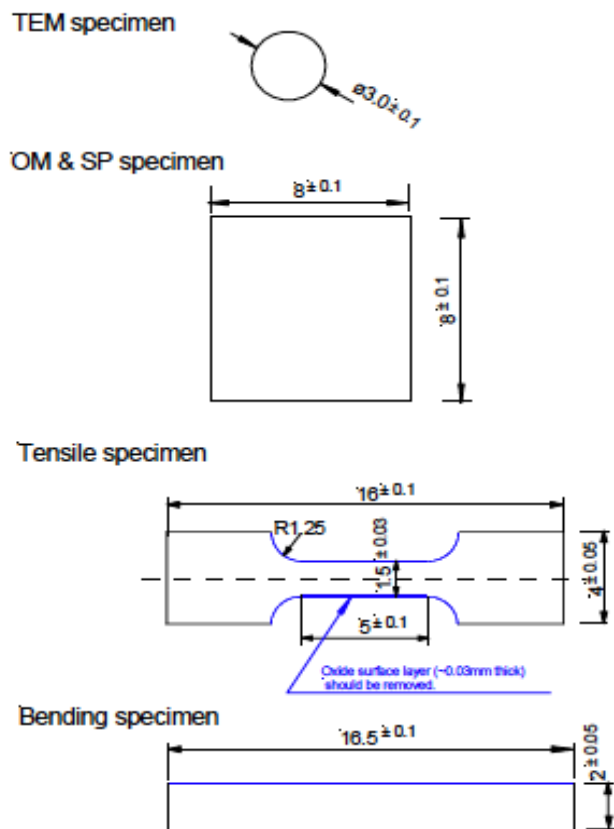


OM/SP specimens and bending specimens

TEM disks

1. JAEA Samples (3)

Dimensions of the specimens



| | Beam window | Flow guide tube | | |
|----------|-------------|-----------------|-------|-----------|
| | H02-1 | H03-2 | H04-2 | |
| | T91 | SS316L | | Total |
| Tensile | 9 | 8 | 6 | 23 |
| TEM | 12 | 10 | 8 | 30 |
| Bend bar | - | - | 4/8 | 8 |
| OM/SP | 2 (Spitze) | 0 | 2/4 | 6 |
| Total | 23 | 18 | 26 | <u>67</u> |

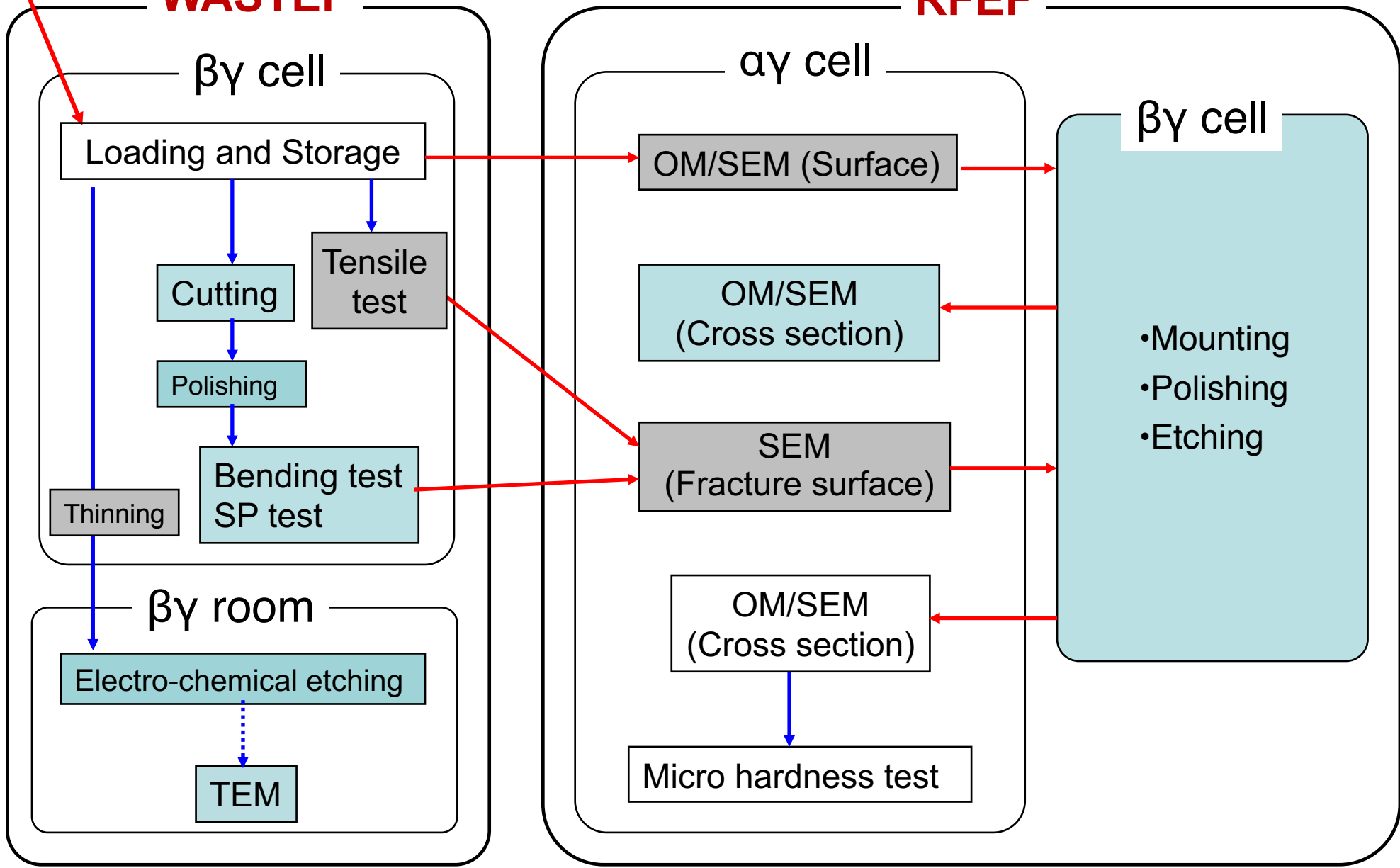
Spitze: triangle part

2. Experimental (1) -PIE flow-

PSI

WASTEF

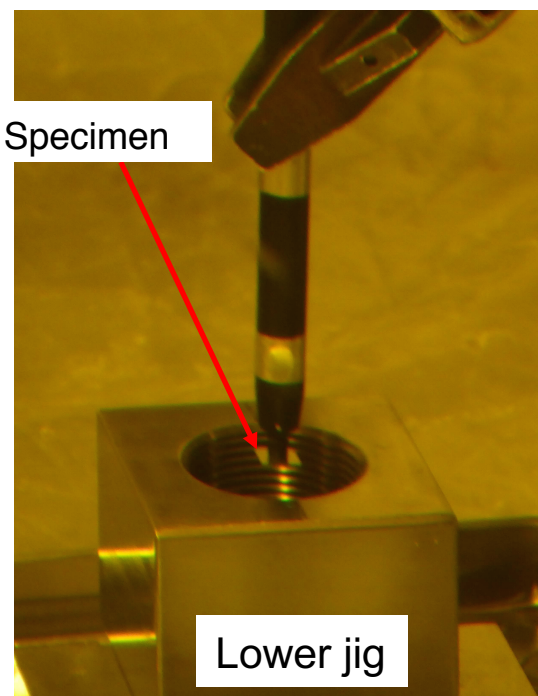
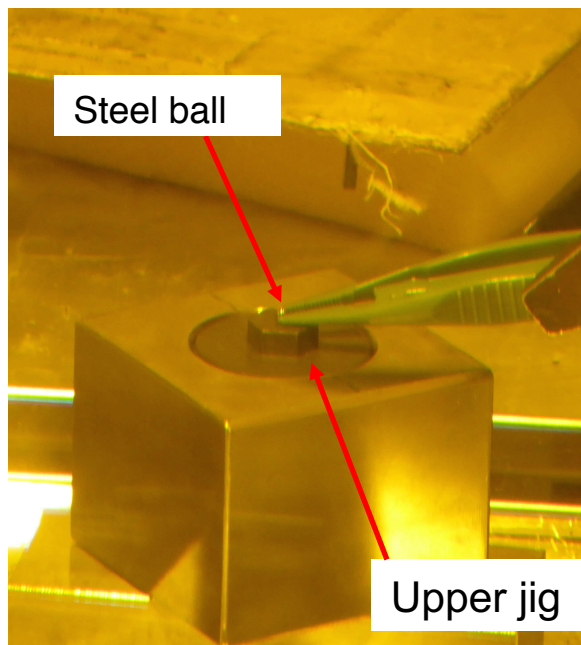
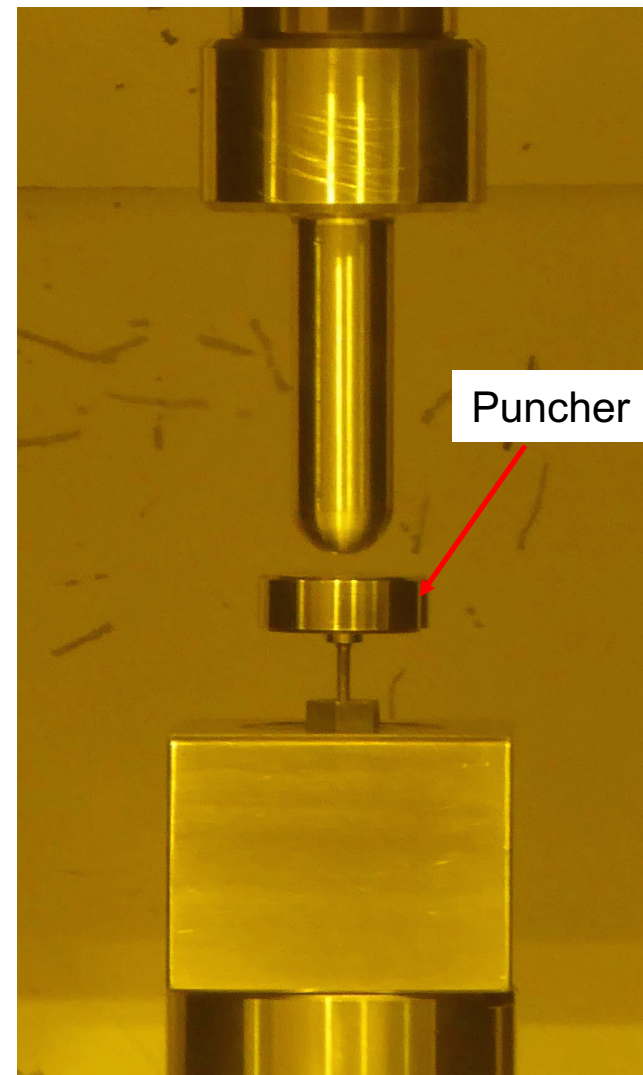
RFEF



Experimental (2) -SP test -

Test conditions of SP test.

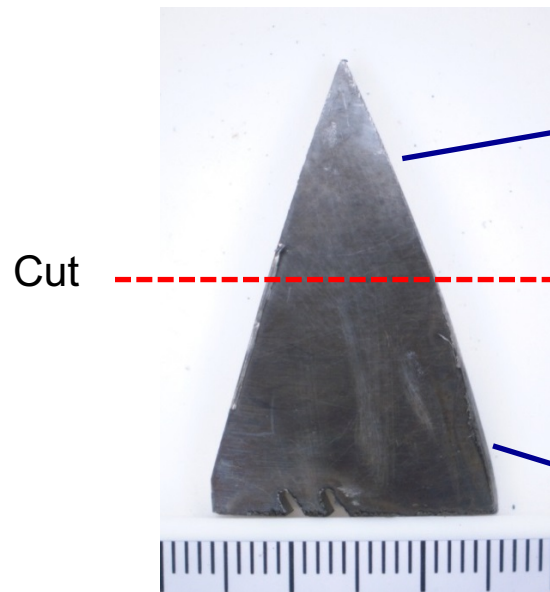
- Materials : T91, SS316L
- Specimen size : 8 mm X 8 mm X 0.5 mm
- Steel ball : $\phi 2.4$ mm (2.381 mm, 3/32inch)
- RT in air
- Cross-head speed: 0.1 mm/min. (6×10^{-4} /s)
- Measurement : Load, Displacement



2. Experimental (3) -SP test-

Sample preparation

Spitze (T91, Beam window)

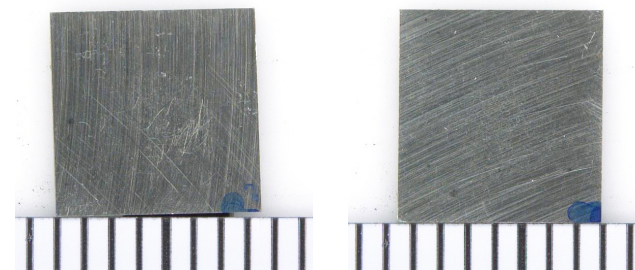


Surface observation

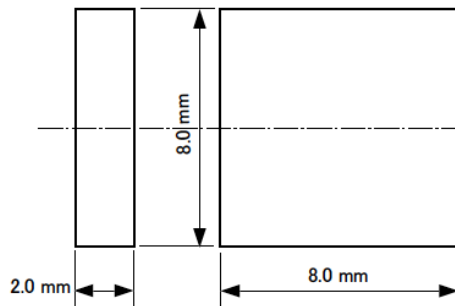
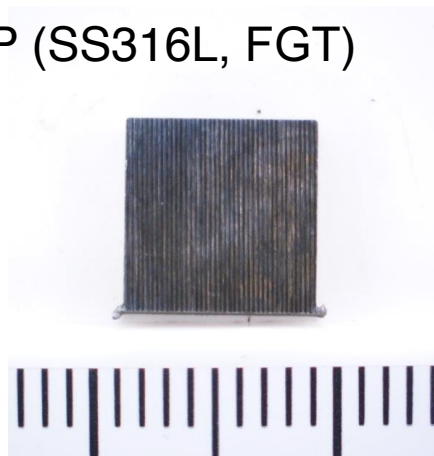
- Mounting
- Polishing
- Etching

Cross sectional observation

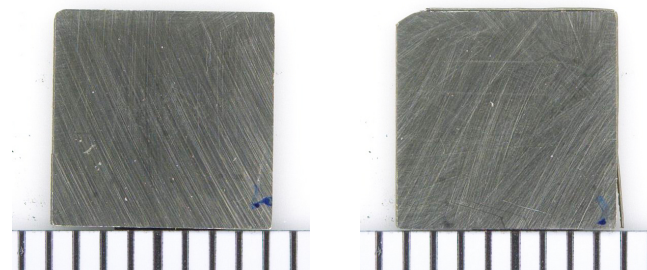
Cutting, Polishing →



OM/SP (SS316L, FGT)



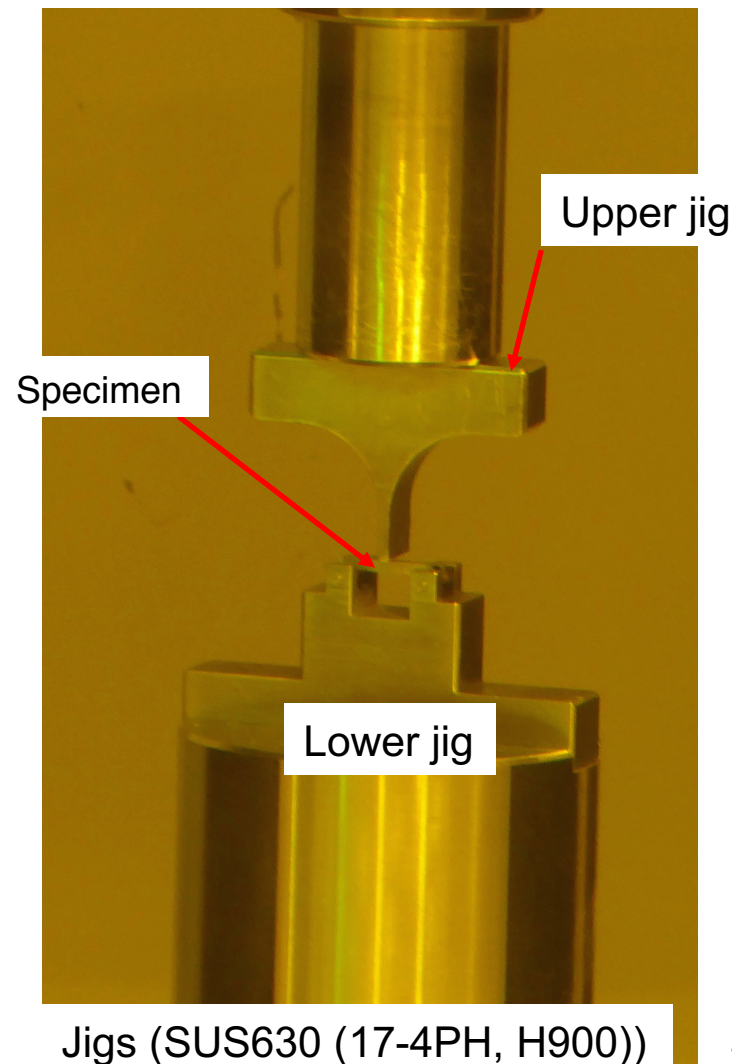
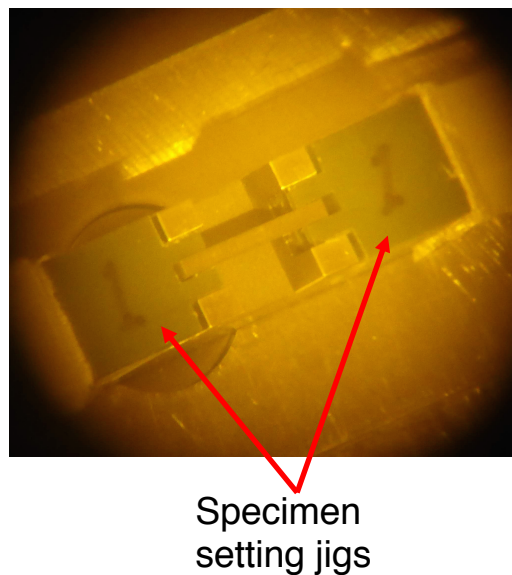
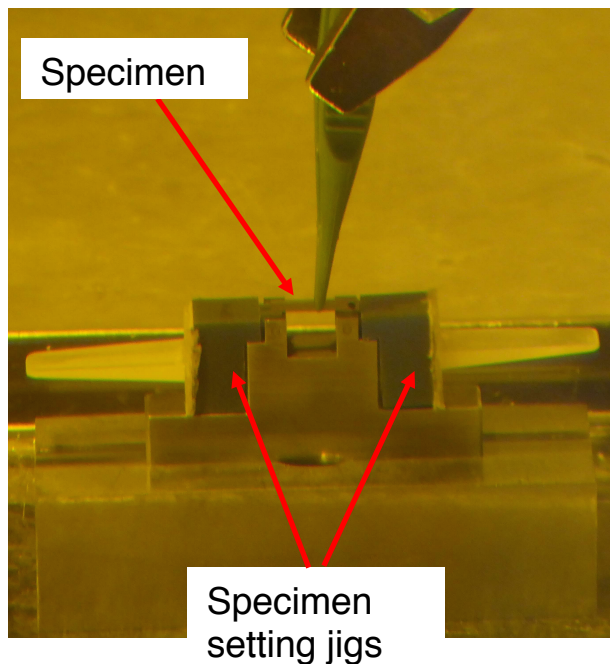
Polishing →



2. Experimental (4) -Three point bending test-

Test conditions of bending test.

- Materials :SS316L
- Specimen size : 1.4 mm X 2.0 mm X 16 mm (without notch)
- Span : 13 mm
- RT in air
- Cross-head speed: 0.5 mm/min. ($3 \times 10^{-3}/s$)
- Measurement : Load, Displacement



2. Experimental (5) -Cross sectional observation-



Sample preparation

- The specimens were cut and mounted in resin.
- After mechanical polishing, the cross section were etched.
- Solution ;
Ethanol 100 ml, Picric acid 2~4 g、Hydrochloric acid 2~5 ml for T91
Hydrochloric acid 25 ml, Nitric acid 10 ml, Glycerin 15 ml for SS316L

SEM/Optical observation

- SEM : X50~X2000
- OM : X50, X400
- Inner side and outer side

SEM



JEOL JSM-5410

2. Experimental (6) - Microstructural observation-



Table. List of MEGAPIE samples polished.

| TEM disk | Group No. | ID | dpa | Irrad. Temp [°C] | Schedule |
|--------------------------|------------|-----------|------|------------------|----------|
| Beam window, T91 | H02-1-14-A | TE04 – 07 | 0.81 | 252 | 2013 |
| | H02-1-15-A | TE14 – 17 | 1.98 | 259 | 2014 |
| | H02-1-15-B | TE24 – 27 | 0.94 | 251 | 2017~ |
| Flow guide tube, 316L SS | H03-2-C1-1 | SE33, 34 | 1.36 | 316 | 2017~ |
| | H03-2-C1-2 | SE44 – 47 | 1.46 | 311 | 2013 |
| | H03-2-C1-3 | SE54 – 57 | 1.57 | 308 | 2014 |
| | H04-2-C1-1 | SE64 – 67 | 0.19 | 341 | 2014 |
| | H04-2-C1-1 | SE74 - 77 | 0.16 | 335 | 2017~ |

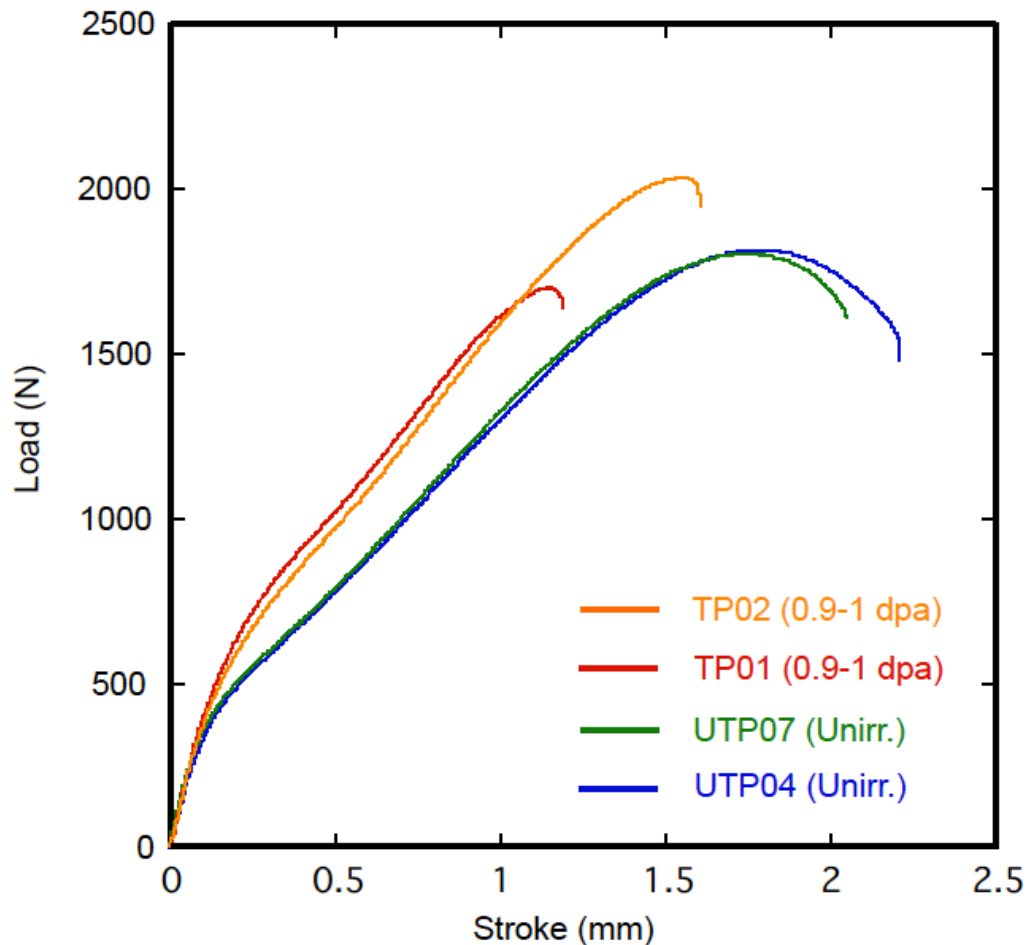
(RED: Observed)

➤ Sample preparation and TEM observation

- At the Waste Safety Testing Facility (WASTEF) of JAEA
- Mechanical polishing and twin jet electro-polishing
- Solution ; 95 vol.% Acetic acid + 5 vol.% Perchloric acid (conc.60%)
- FE-TEM ; Hitachi HF-2000 (replaced with new HF3300 in FY2016)

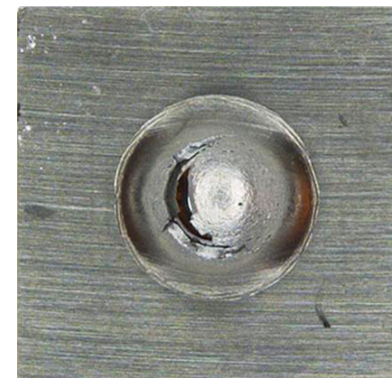
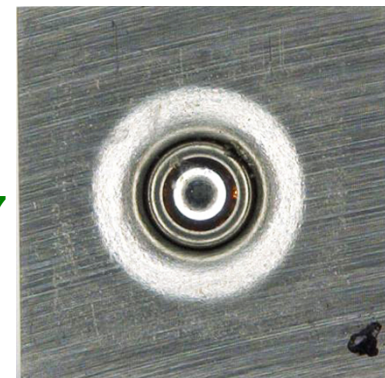
3. PIE results -SP tests (1)-

T91 (Beam window), RT tests

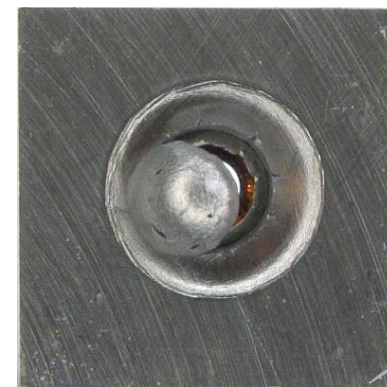
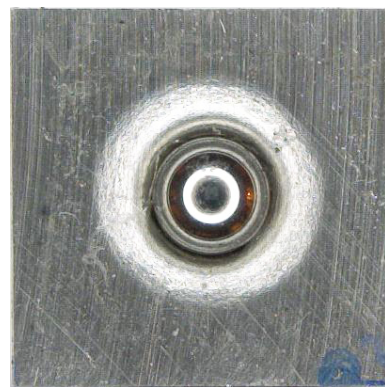


- Irradiation hardening and degradation of ductility.

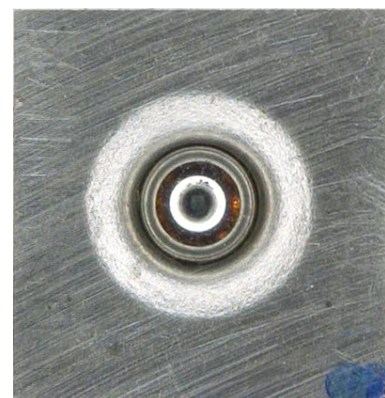
UTP07



TP01



TP02



3. PIE results -SP tests (2)-

T91

| No. | dpa | T _{irr.} (°C) | T _{test} (°C) | Max. load, P _{max} (N) | SP fracture energy (J) | Deflection at fracture, δ* (mm) | Equivalent fracture strain, ε _{qf} | Estimated J _{IC} (kJ/m ²) |
|-------|---------|------------------------|------------------------|---------------------------------------|------------------------------|---------------------------------------|---|---|
| UTP04 | 0 | - | 24 | 1814 | 2.78 | 2.22 | 1.64 | 453 |
| UTP07 | 0 | - | 26 | 1800 | 2.52 | 2.06 | 1.41 | 374 |
| TP01 | (0.9~1) | (260) | 26 | 1692 | 1.30 | 1.19 | 0.47 | 49.6 |
| TP02 | (0.9~1) | (260) | 25 | 2028 | 2.08 | 1.61 | 0.87 | 186 |

Empirical relation; $\epsilon_{qf} = \ln(t/t_0) = \beta (\delta^*/t_0)^2$, $\beta = 0.09$, t_0 = Initial specimen thickness

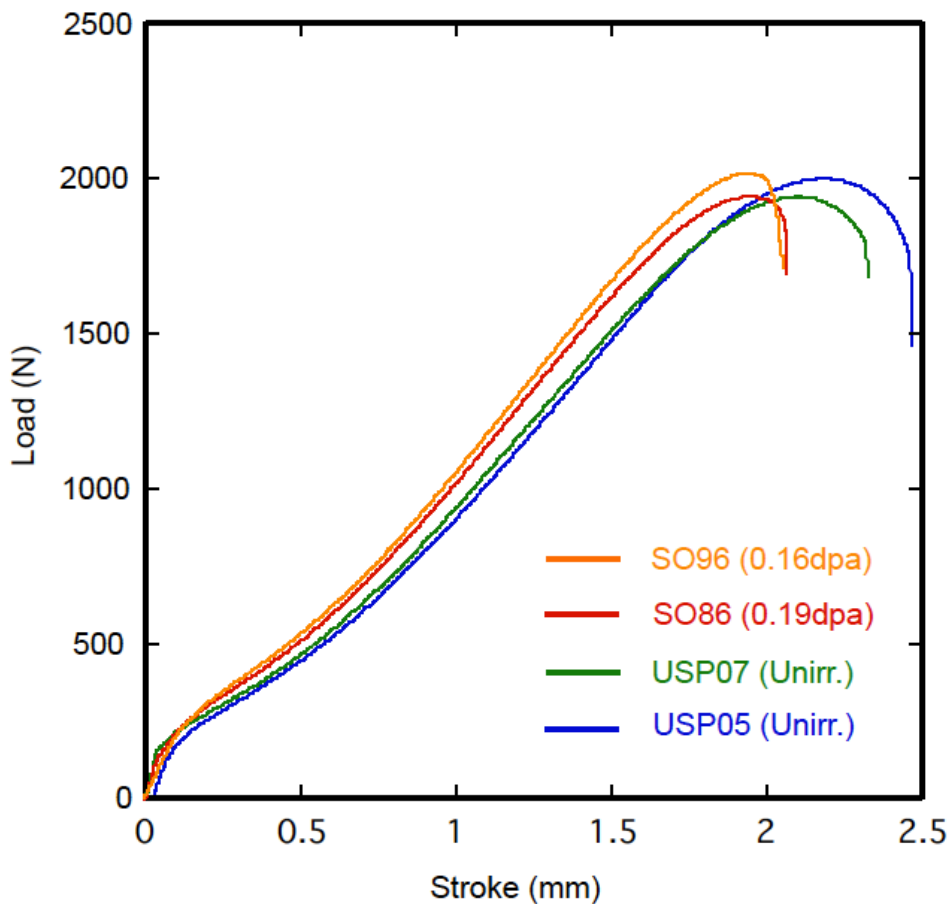
$$\text{Estimated } J_{IC} = 345\epsilon_{qf} - 113$$

(M. R. Bayoumi, et.al. (1983))

- SP fracture energy, deflection at fracture and estimated J_{IC} value decreased.
- Dispersion of the irradiated data.

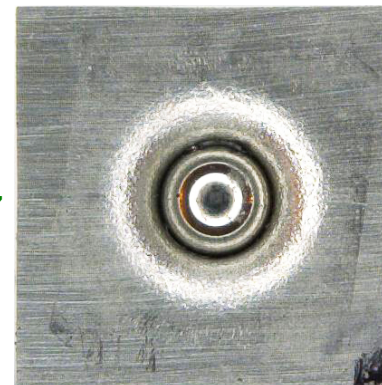
3. PIE results -SP tests (3)-

SS316L (FGT), RT tests



- Irradiation hardening and degradation of ductility
- Max load are almost same.

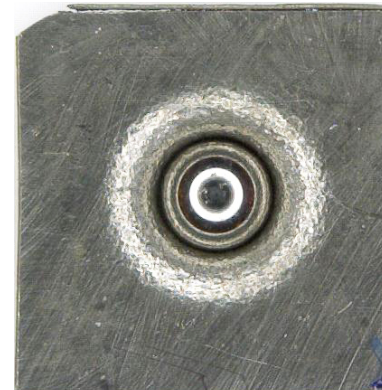
USP07



SO86



SO96



3. PIE results -SP tests (4)-



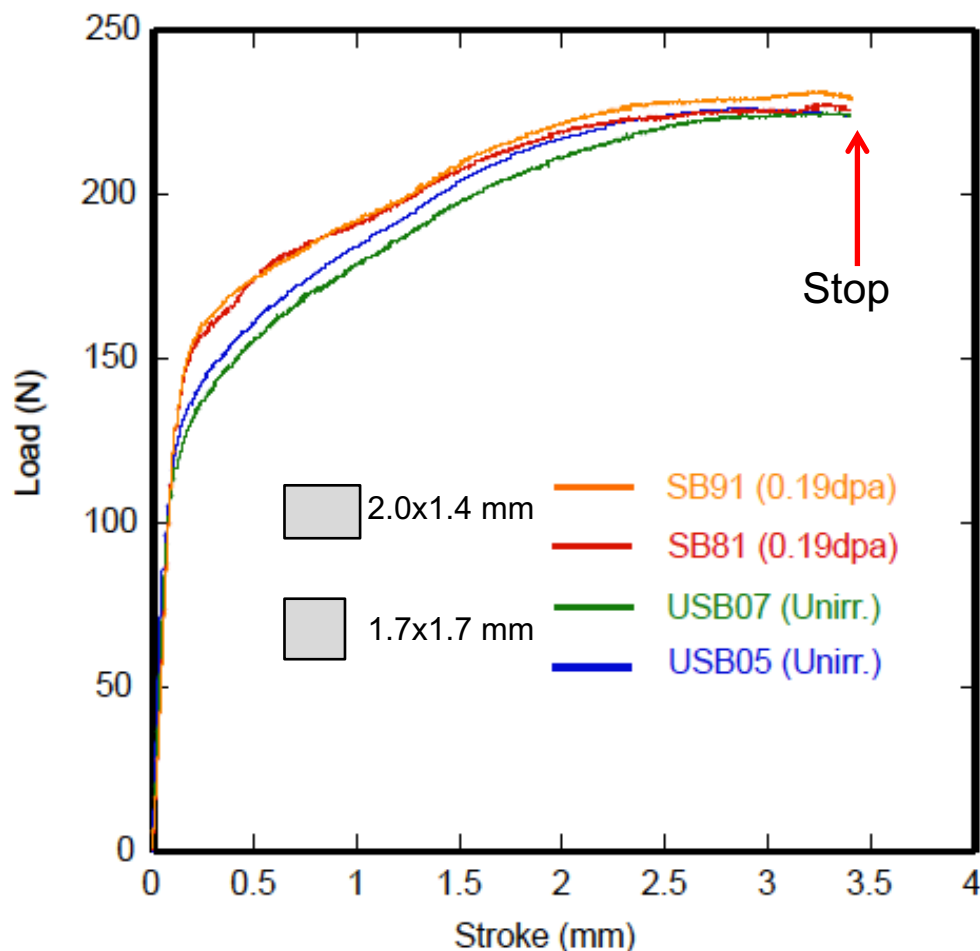
SS316L (FGT), RT tests

| No. | dpa | T _{irr.} (°C) | T _{test} (°C) | Max. load, P _{max} (N) | SP fracture energy (J) | Deflection at fracture, δ^* (mm) | Equivalent fracture strain, ϵ_{qf} | Estimated J _{IC} (kJ/m ²) |
|-------|------|------------------------|------------------------|---------------------------------------|------------------------------|---|---|--|
| USP05 | 0 | - | 21 | 1998 | 2.83 | 2.46 | 2.01 | (582) |
| USP07 | 0 | - | 26 | 1935 | 2.59 | 2.33 | 1.88 | (535) |
| SO86 | 0.19 | 337 | 27 | 1945 | 2.24 | 2.07 | 1.67 | (465) |
| SO96 | 0.16 | 332 | 27 | 2015 | 2.28 | 2.06 | 1.47 | (394) |

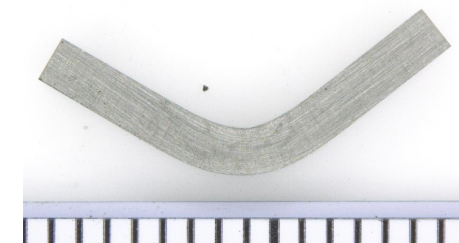
- SP fracture energy, Deflection at fracture decreased.

3. PIE results -Bending tests -

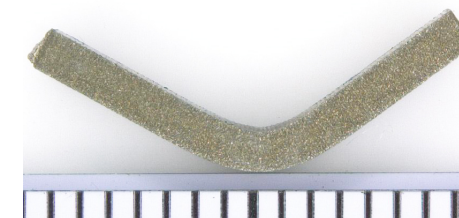
SS316L (FGT), RT tests



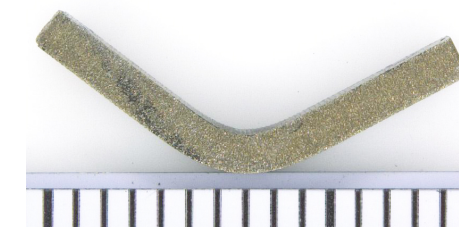
USB07



SB81



SB91

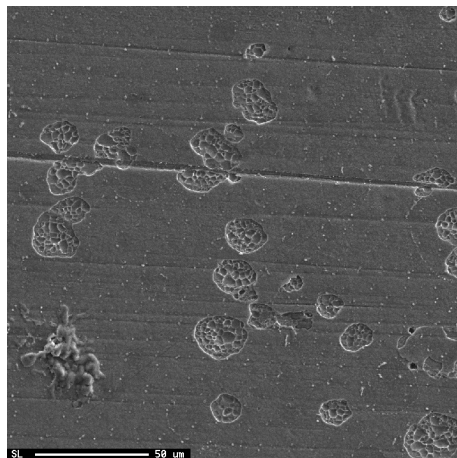


- Size of cross section are different.
- Irradiation hardening are observed.
- Cracks were not observed.

3. PIE results -Cross sectional observation (1)-

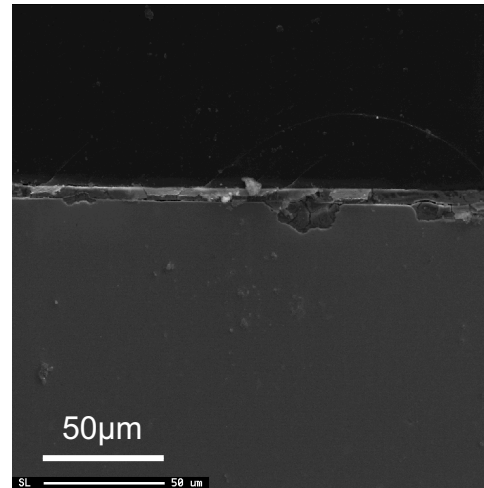
Spitze (T91)

Inner surface (SEM)

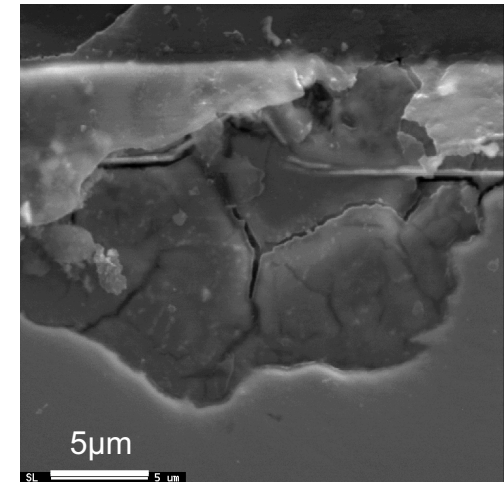


50μm

Inner side (SEM)

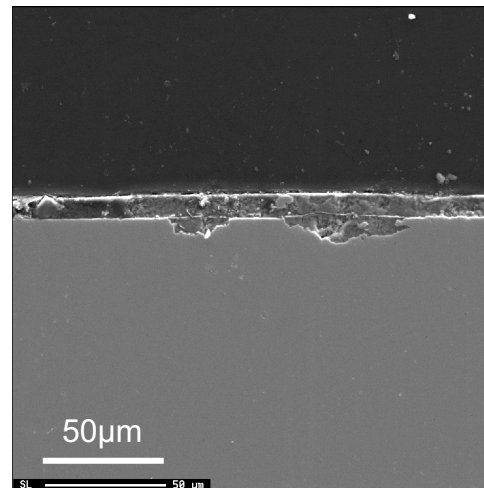


50μm

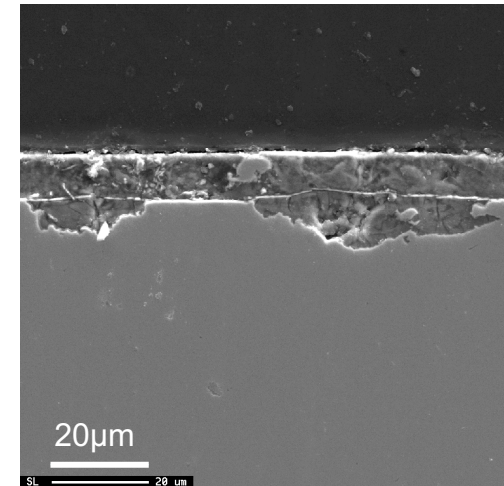


5μm

Inner side (SEM)



50μm



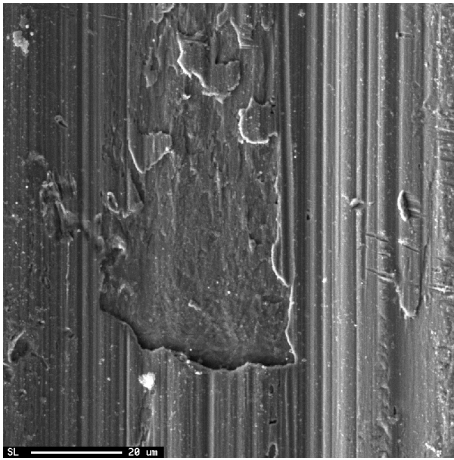
20μm

- Cracks inside of the pits
- No cracks around the pits.

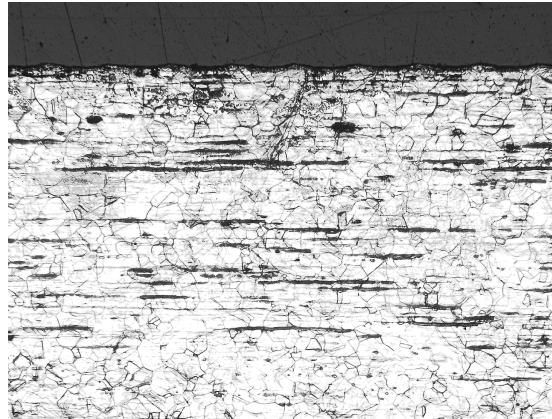
3. PIE results -Cross sectional observation (2)-

FGT (SS316L, SO85)

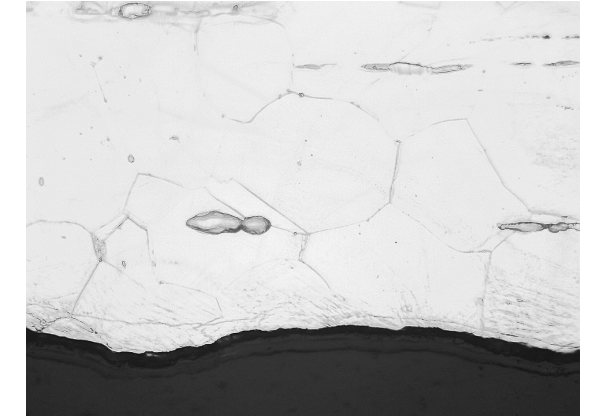
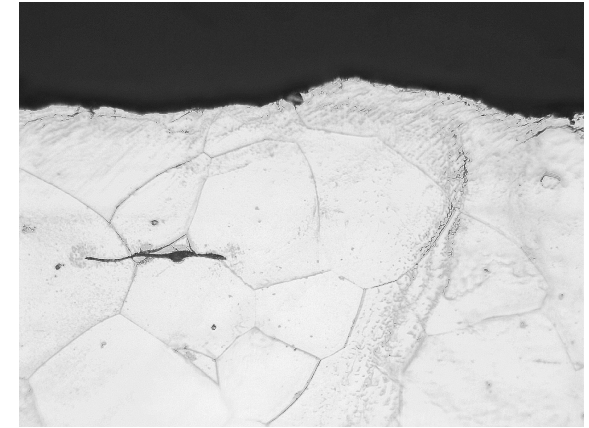
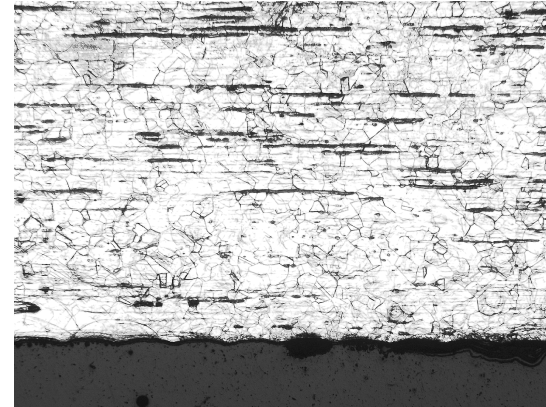
Inner surface (SEM)



Inner side
(OM)



Outer side
(OM)



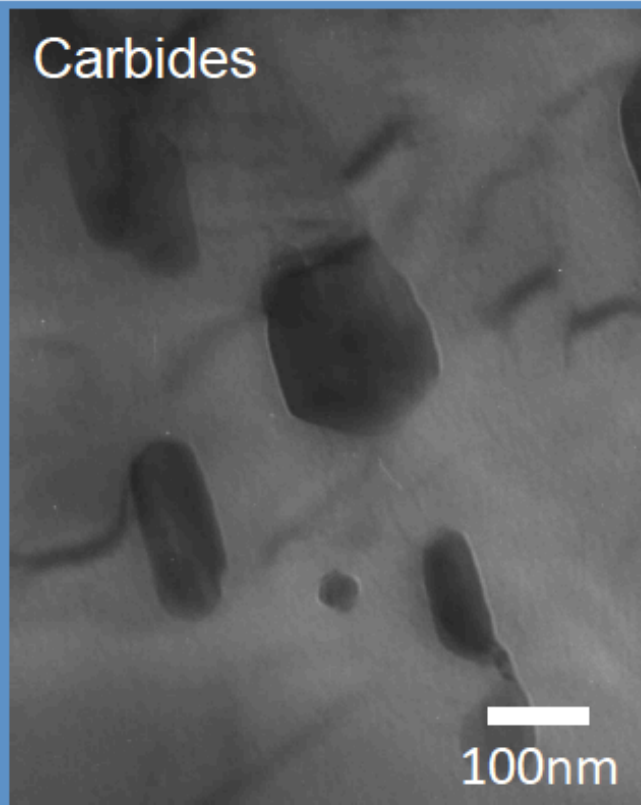
- No evidence of corrosion
- Cracks were not observed for both side.

3. PIE results -Microstructure observation (1)-

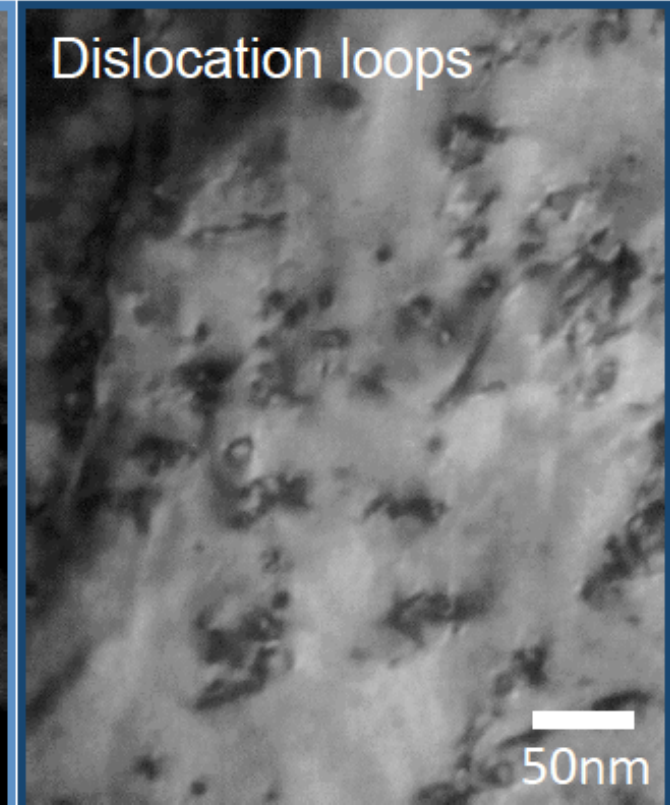
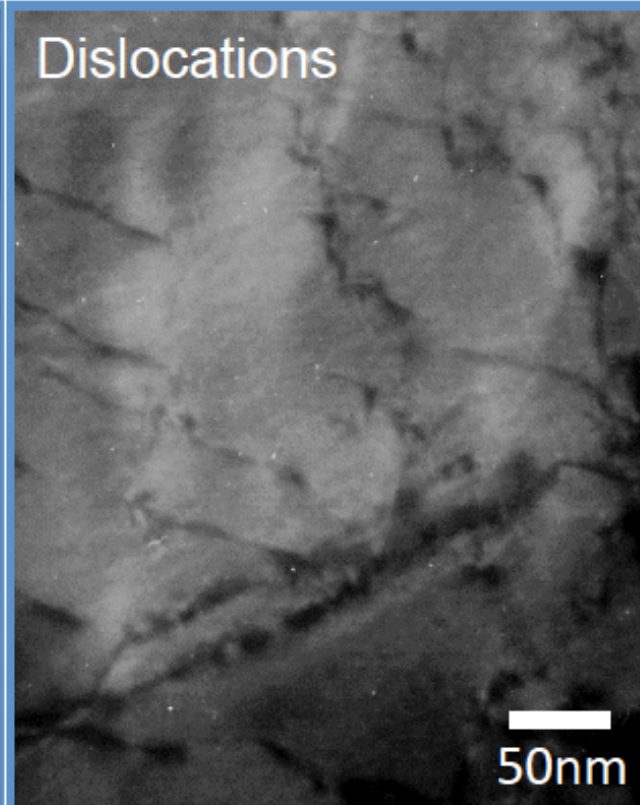
Typical irradiation microstructure of T91

BF image with $g=011$ near $z=111$

0.81 dpa, 252°C
(ID:TE04)



1.98 dpa, 259°C
(ID:TE14)



- 0.81 dpa : carbides and network dislocations
- 1.98 dpa : dislocation loops.

3. PIE results -Microstructure observation (2)-

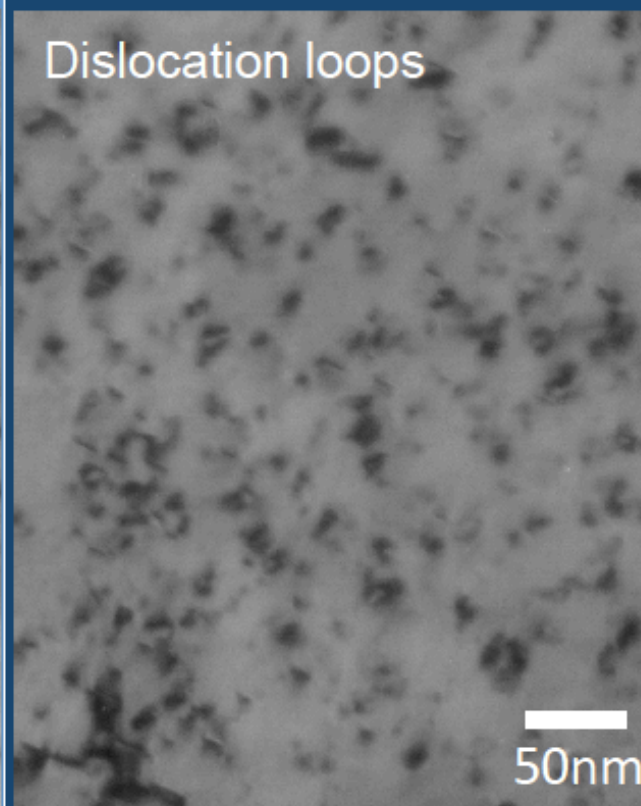
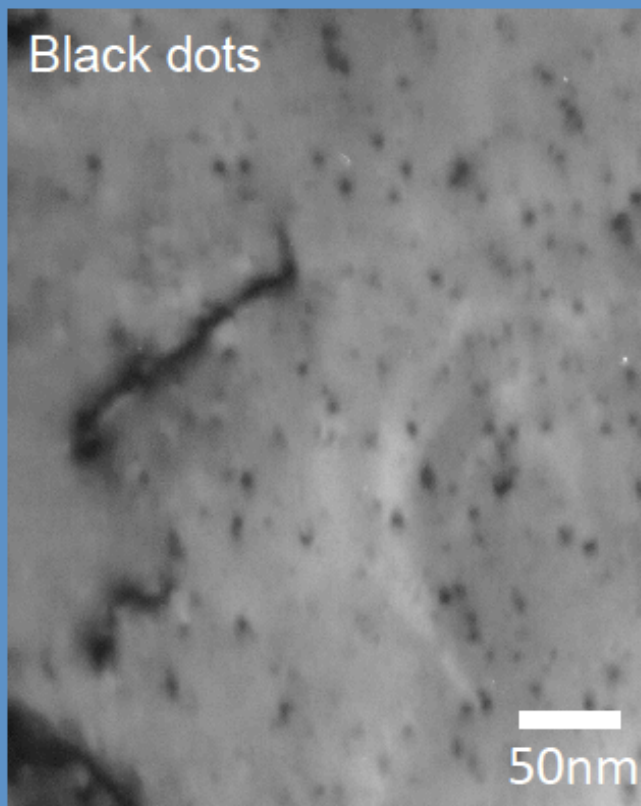
Typical irradiation microstructure of 316L SS

BF image with $g=200$ near $z=011$

Rel-rod streak image

0.19 dpa, 341°C
(ID:SE64)

1.57 dpa, 308°C
(ID:SE54)



- 0.19 dpa : network dislocations and black dot defect clusters
- 1.57 dpa : coexistence of black dots and dislocation loops (Frank loops)

4. Summary



Mechanical property tests

- SP tests ($\phi 2.4$ mm) and the three point bending tests (without notch) on the samples cut from MEGAPIE target were performed and irradiation effects were investigated.
- SP fracture energy, deflection at fracture and estimated J_{IC} value at RT decreased by the irradiation.
- After the bending tests, no cracks were observed for SS316L samples.

Cross sectional observation

- BW (T91); Cracks inside of the pits, no cracks around the pits.
- FGT (SS316L); At surface, corrosion and cracks were not observed.

Microstructure observation

- Irradiation microstructures of T91 and ss316L were investigated by TEM and different microstructures were observed.

Future plan

SP test

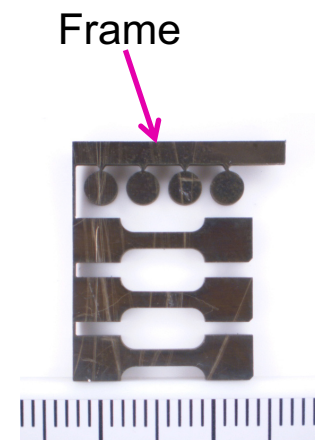
- Cross sectional and fracture surface observation by SEM.
- Development of test technique for SP test with $\Phi 1.0$ mm steel ball to obtain SPDBTT. Specimens will be prepared from $\Phi 3.0$ mm disks and grip part of tensile specimens.
- Temperature control system ($-196^{\circ}\text{C} \sim 250^{\circ}\text{C}$)
- Existing testing machine, JMTR-HL (JNM283-287, T. Ishii et al., (2000))

Three point bending test (with notch)

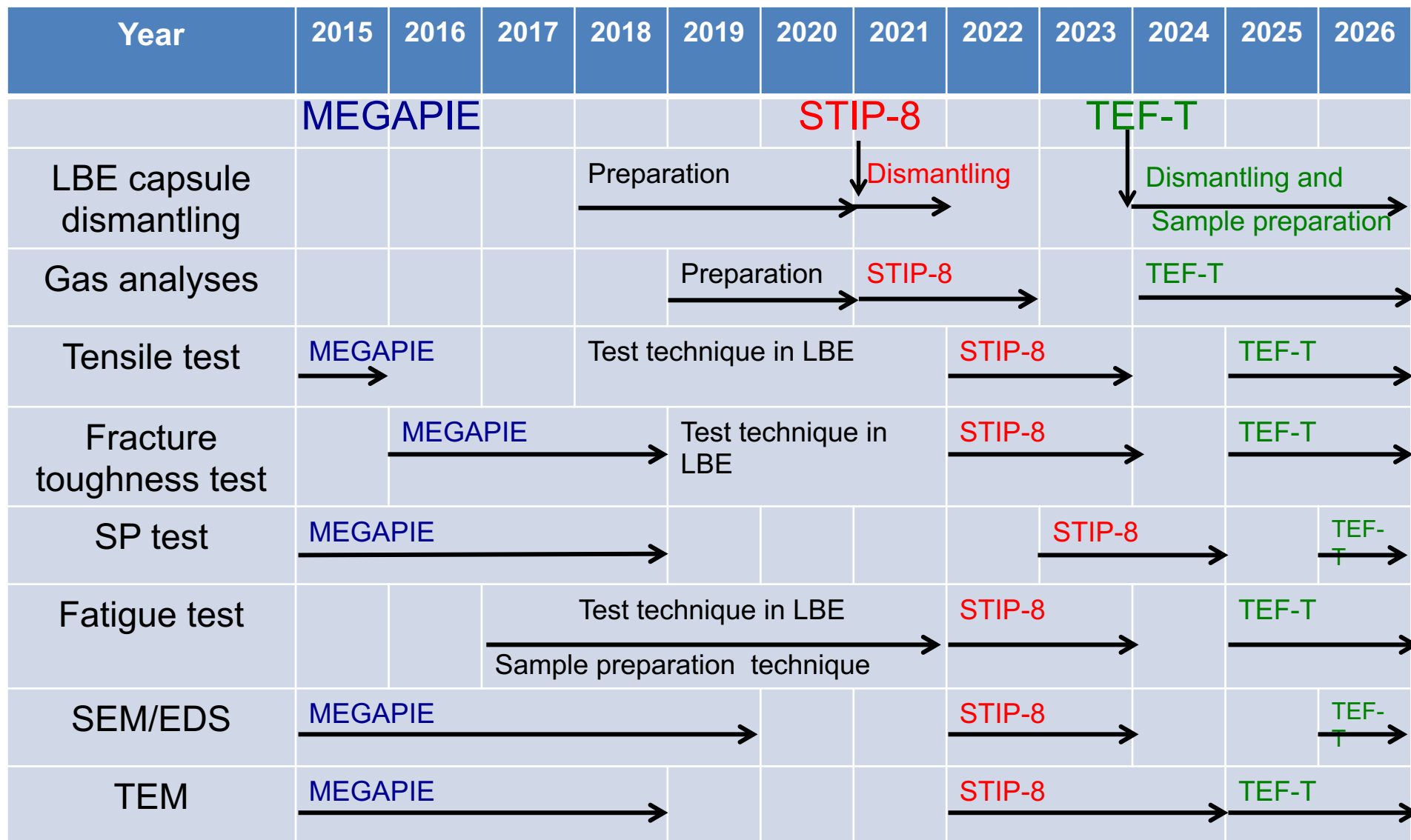
- Development of notching device and test technique for fracture toughness test.
- Specimen; SS316L, T91(Frame)
- Testing machine was installed in WASTE-F.

Microstructure observation

- TEM observation for rest samples and detailed observation will be conducted by brand-new TEM.



PIE -Schedule -



Thank you!