
ETHEL Erosion Tests

	Name	Role/Title
Owner	Per Nilsson	CFD Specialist
Reviewer	Srinivasan Iyengar YongJoong Lee	Senior Scientist Group Leader – Materials Group
Approver	Ulf Odén	Lead Engineer – Target Systems

Document Type	Analysis Report
Document Number	ESS-0051445
Date	Aug 25, 2016
Revision	1
State	Released
Confidentiality Level	Internal

SUMMARY

Due to experienced erosion in other nuclear applications, e.g. gas cooled reactors, the question was raised whether this could occur in the ESS target. ETHEL has therefore been used to investigate erosion of tungsten at conditions similar to the conditions in the ESS target.

The experiments do not show any significant erosion over time. However, some conservative quantitative estimates have nevertheless been made, based on these tests that have been run for 25 hours. A simple estimate of features by looking at the SEM pictures gives 0.04 kg initially for a target wheel. An estimate from weighing the samples gives 0.033 kg initially for a target wheel. There are no indications that it will be a continuous process.

TABLE OF CONTENT	PAGE
1. SETUP AND POSITIONS	4
2. SCANNING ELECTRON MICROSCOPY (SEM).....	7
3. SAMPLE 1.....	9
3.1. Sample 1, Position 1.....	10
3.2. Sample 1, Position 2.....	11
3.3. Sample 1, Position 3.....	12
3.4. Sample 1, Position 4.....	13
3.5. Sample 1, Position 5.....	14
4. SAMPLE 2.....	15
4.1. Sample 2, Position 1.....	16
4.2. Sample 2, Position 2.....	17
4.3. Sample 2, Position 3.....	18
4.4. Sample 2, Position 4.....	19
4.5. Sample 2, Position 5.....	20
5. ESTIMATED FEATURES	21
6. REFERENCES.....	22
DOCUMENT REVISION HISTORY	22

Document Type	Analysis Report
Document Number	ESS-0051445
Date	Aug 25, 2016
Revision	1
State	Released
Confidentiality Level	Internal

1. BACKGROUND

In the ESS target, helium flowing at high velocities, up to about 100 m/s, will cool the spallation target tungsten blocks. In order to learn more, hands on, about cooling with pressurised helium in closed circuit, the ETHEL loop was built. ETHEL stands for ESS Target Helium Experiments at LTH. These experiments are led by Jens Klingmann at the Department of Energy Sciences.

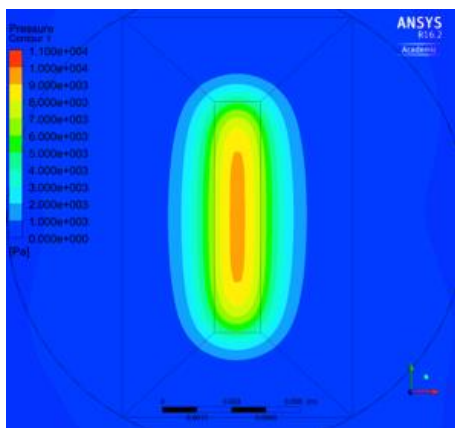
The loop contains a booster compressor which can circulate ~3 g/s helium at 10 bar, an electric heater, a water cooled heat exchanger, I&C and a versatile experiment vessel with windows and exchangeable and adjustable internals which can operate up to 400 C, see [1].

Due to experienced erosion in other nuclear applications, e.g. gas cooled reactors, the question was raised whether this could occur in the ESS target. ETHEL has therefore been used to investigate erosion of tungsten at conditions similar to the conditions in the ESS target. Pressure, temperatures, flow speed and hydraulic dimensions, as well as initial material properties were controlled to matching regimes. The helium quality was conservatively somewhat lower in the experiments, i.e. the oxygen content was not controlled to be quite as low as will be in the target. Also, the tungsten samples were pre-oxidised to a higher degree than is expected in the target.

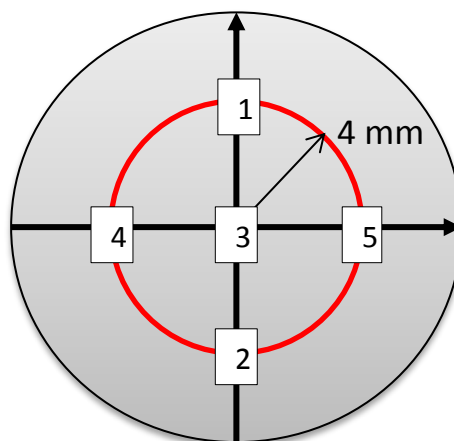
2. SETUP AND POSITIONS

In the ETHEL experiments, a 10 mm by 2 mm planar helium jet of > 100 m/s at 9 bar and > 200 C was blown perpendicularly onto D = 2 cm tungsten samples at above 300 C.

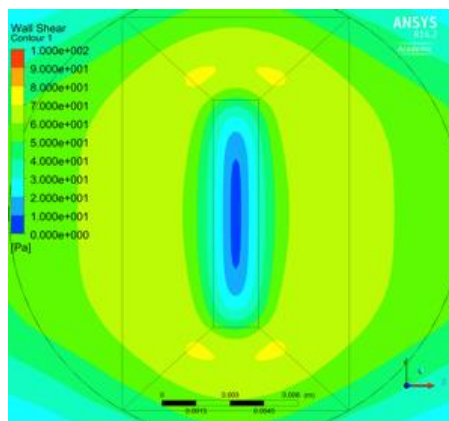
Scanning Electron Microscope (SEM) pictures have been taken at five positions on the samples, one at the centre and the other 4 mm away from the centre.



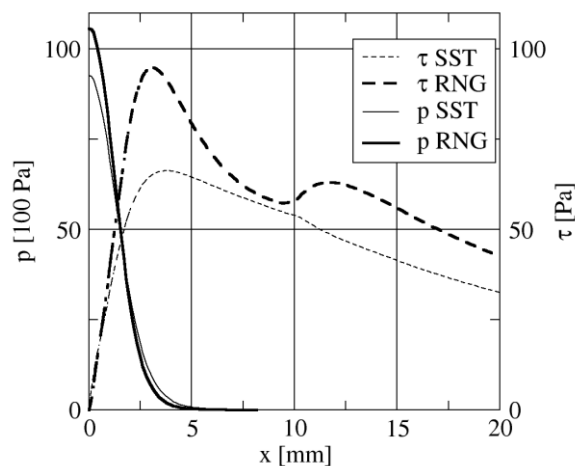
Pressure stress on the sample surface



SEM Positions on the sample



Shear stress on the sample surface.
 The helium nozzle projection can be seen as thin lines.

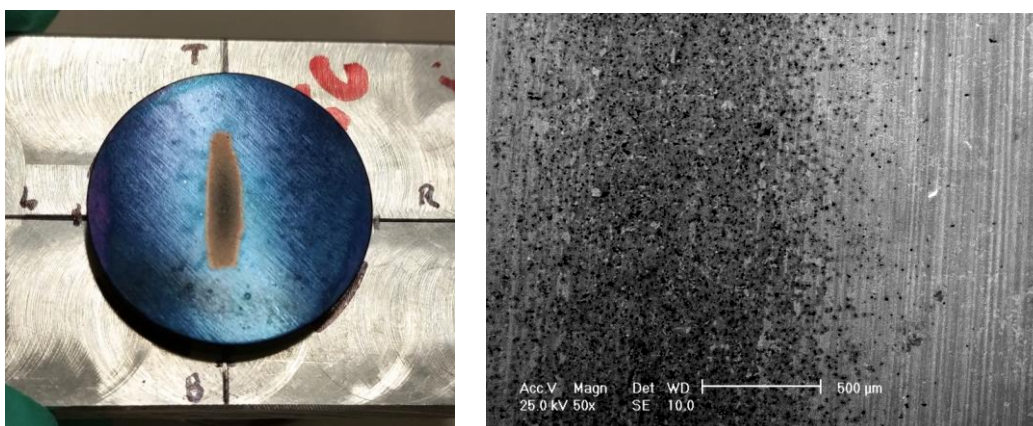


Pressure (p) and shear (τ) stress along a line from the sample centre to the right, with two different turbulence models.

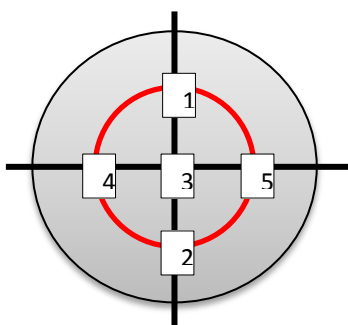
The two colourful contour plots to the left and the diagram to the lower right are results from CFD calculations, which have been validated by comparison to LDA measurements, see ICONE24-60721 [1]. It can be seen that position 3 corresponds to the maximum pressure stress, about 10 kPa and position 4 and 5 are located at about the peak shear stress, less than 100 Pa. See also Section 3 on tests below.

3. LOOP IMPURITIES AND SAMPLE CLEANING

Due to the measurements performed in order to validate the test configuration, see [1], there were some remaining solid impurities in the helium loop. This came from the particle seeding required to perform the LDA measurements. The seeder materials used were titanium dioxide and hydrocarbon polymer. Several attempts were made to clean this out, by circulation and filtration of air and pure helium, but there was still some remaining to end up on the samples, see pictures of sample 1 below.



Elemental mapping confirmed the existence of titanium oxide and carbon on the surface. The agglomeration and adhesion of the particles to the surface is most probably a consequence of the area being subjected to a combination of the highest temperature and impingement velocity. Several attempts were made to remove this coating between the run cycles, e.g. by manually cleaning with ethanol and acetone, and by using an ultrasonic bath. The cleaning was performed in several small steps, all while carefully monitoring the mass. It could be argued that the cleaning would add to the erosion, but as will be seen below, there was no visible erosion anyway.



It should also be noted that the position of the mark on the sample is slightly off the center, less than 1 mm. The reason for this is most likely that the flow tube, from which the jet is blown, is slightly off perpendicular. This has no impact on the validation measurements and calculations in [1], as the angle is negligible and the measurements are aligned with the jet, not the sample. For the erosion experiments it is even beneficial, because SEM position 3 is not in the mark, which would show seeder particles instead of tungsten erosion. The pressure stress is lower at ~1 mm off center, but still not low.

Document Type	Analysis Report
Document Number	ESS-0051445
Date	Aug 25, 2016
Revision	1
State	Released
Confidentiality Level	Internal

4. SAMPLE PRE-OXIDATION

The samples are pre-oxidised at 500 C in He + 0.5 % O₂ for 1 h.

This is at a higher temperature and well above the oxygen content during normal operation in the target system.

Document Type	Analysis Report
Document Number	ESS-0051445
Date	Aug 25, 2016
Revision	1
State	Released
Confidentiality Level	Internal

5. SCANNING ELECTRON MICROSCOPY (SEM)

A large range of SEM pictures have been taken with 200x, 500x and 1000x magnification by Jemila Habainy at Lund University, Division of Materials Engineering.

Only some of the 1000x pictures are shown here.

Each of these shows an area of about 0.01 mm² of the 3.1 cm² top area of the sample.

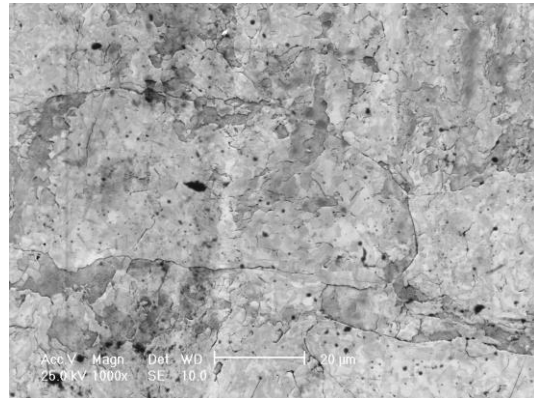
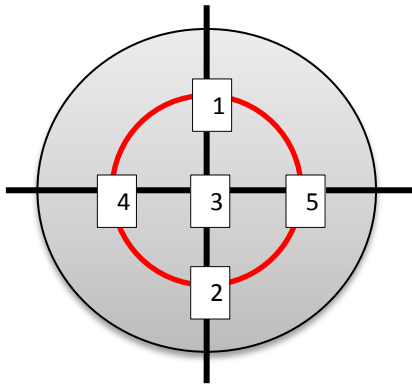
After the pictures, there is a short note with a very rough estimate of protruding features, bumps, to number and size.

Document Type	Analysis Report
Document Number	ESS-0051445
Date	Aug 25, 2016
Revision	1
State	Released
Confidentiality Level	Internal

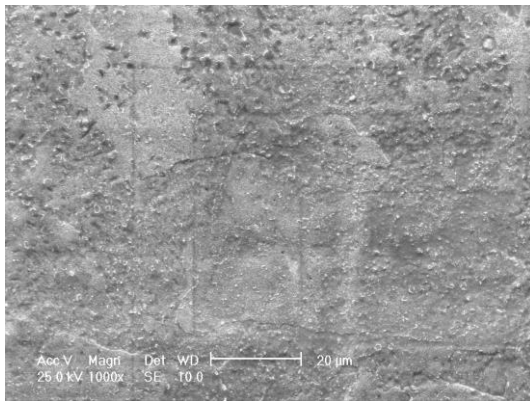
6. SAMPLE 1

Sample 1 has been exposed to two 5 h cycles and one 15 h cycle (in two runs) with impinging helium flow in ETHEL.

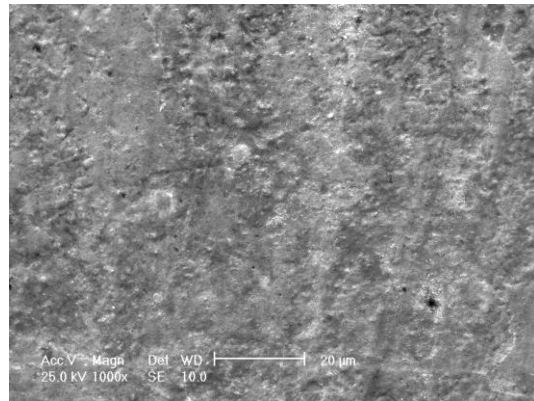
6.1. Sample 1, Position 1



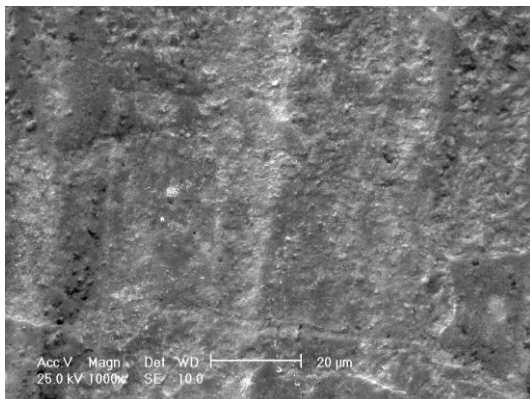
Prior to pre-oxidation



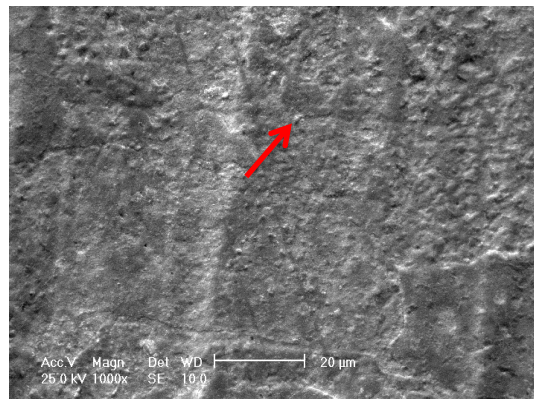
After pre-oxidation



After 5 h helium flow in ETHEL



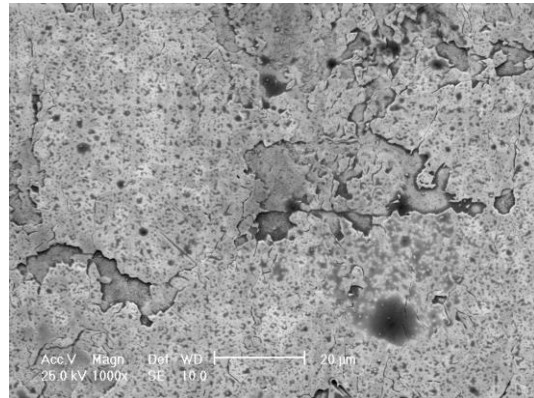
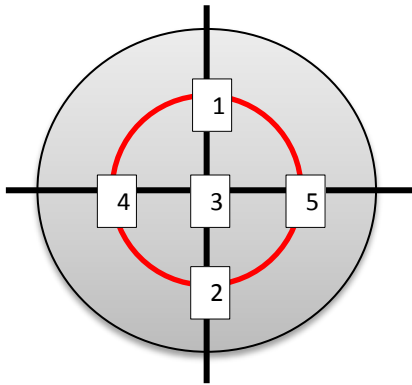
After 5 h + 5 h in ETHEL



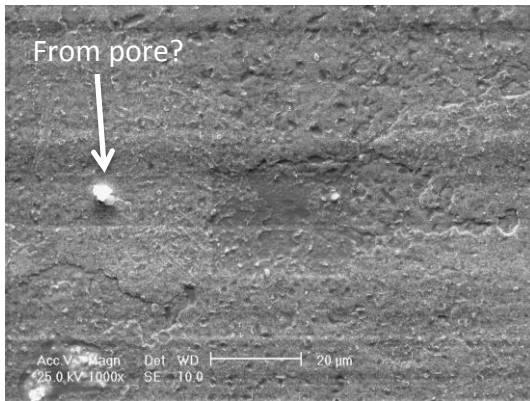
After 5 h + 5 h + 15 h in ETHEL

No erosion over the 10 h was found by visual comparison.
There may be 10 typical features, marked with red arrow, about 2 μm large.

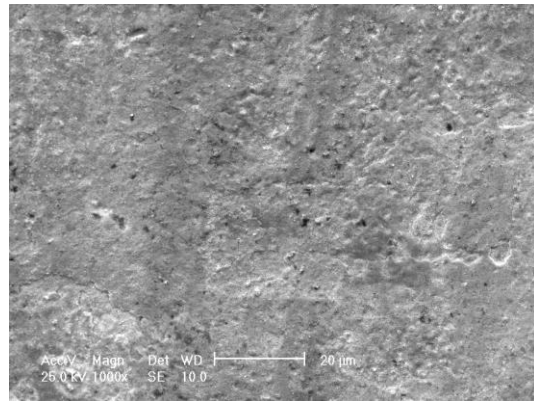
6.2. Sample 1, Position 2



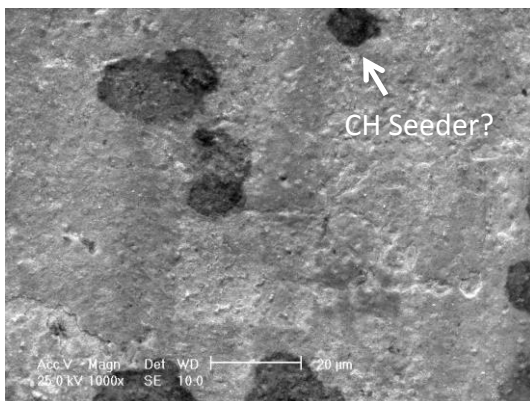
Prior to pre-oxidation



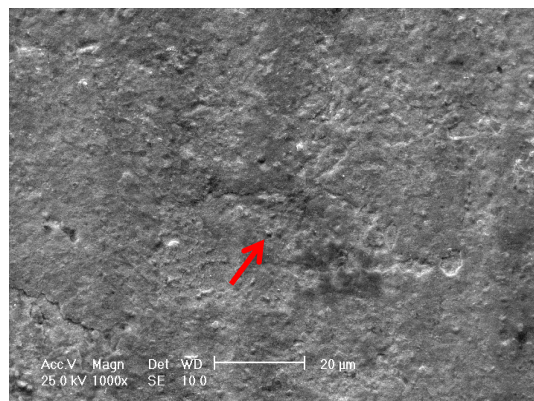
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL

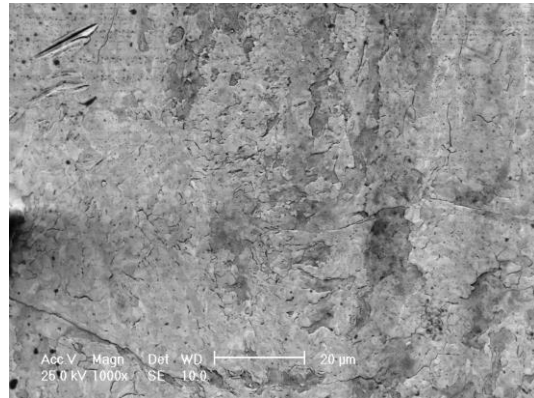
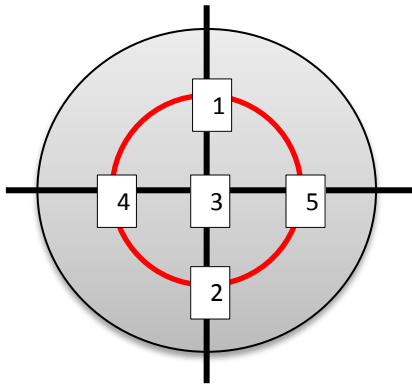


After 5 h + 5 h + 15 h in ETHEL

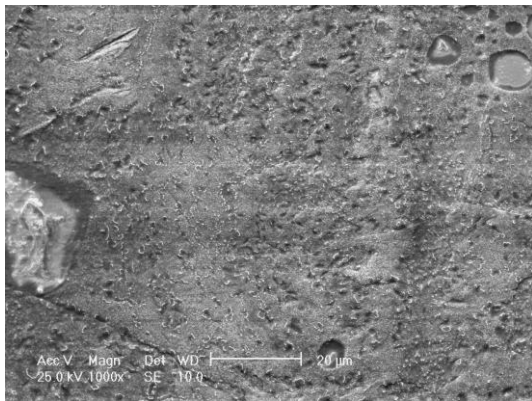
No erosion over the 25 h was found by visual comparison, but some light substance, possibly from pores, was removed.

There may be 10 typical features, marked with red arrow, about 2 μm large.

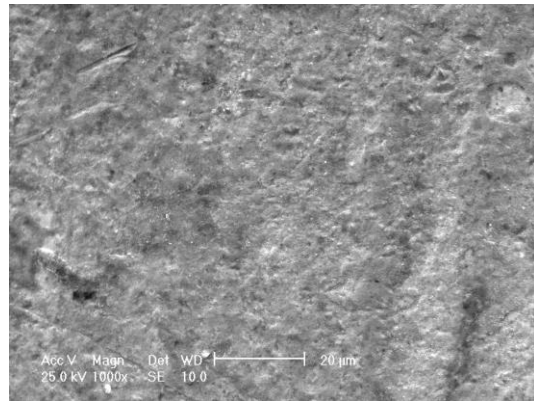
6.3. Sample 1, Position 3



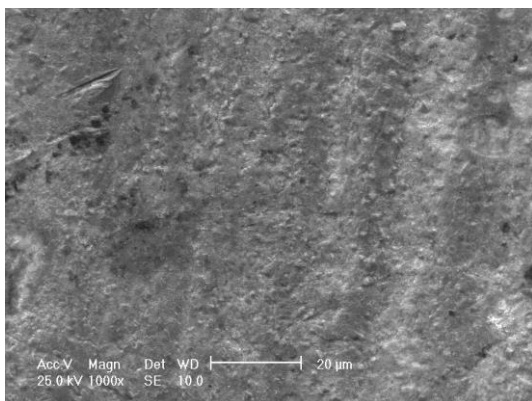
Prior to pre-oxidation



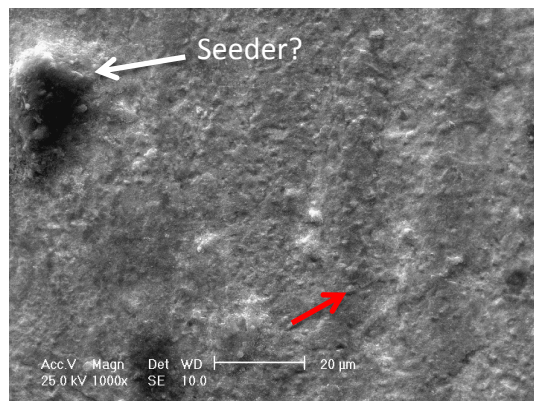
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL

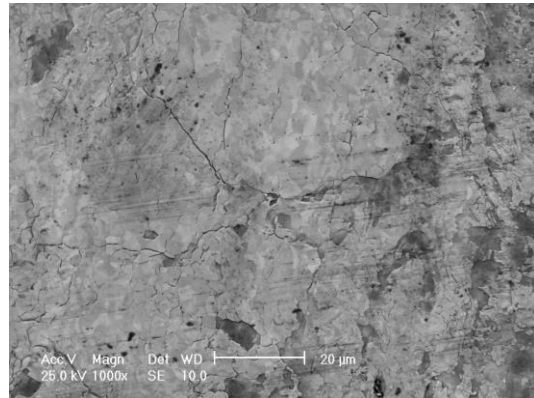
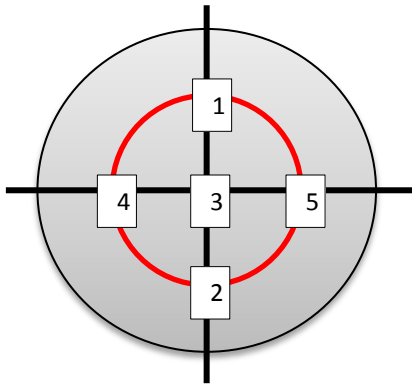


After 5 h + 5 h + 15 h in ETHEL

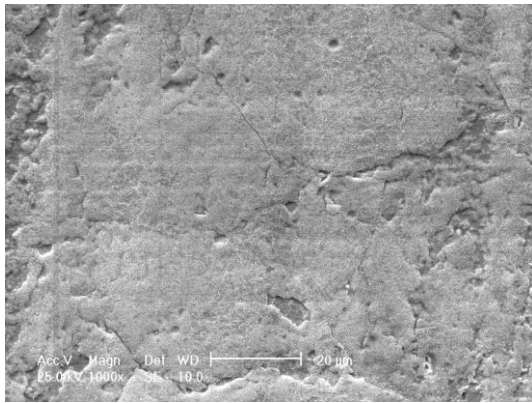
No erosion over the 10 h was found by visual comparison.

There may be 10 typical features, marked with red arrow, about 3 μm large.

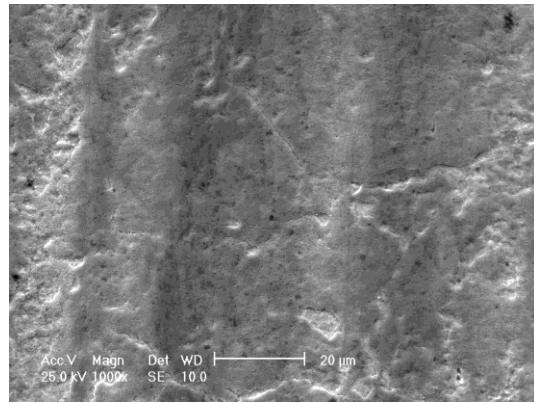
6.4. Sample 1, Position 4



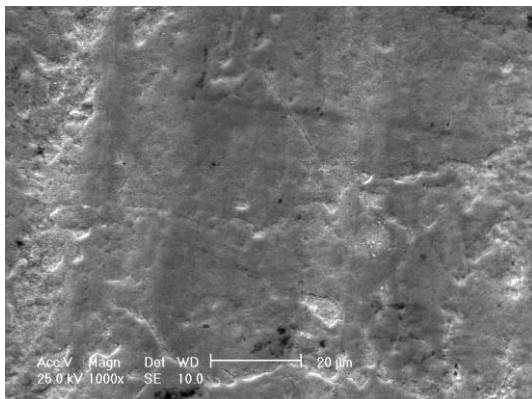
Prior to pre-oxidation



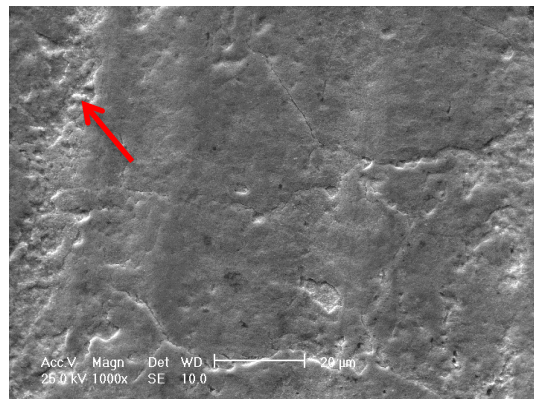
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL

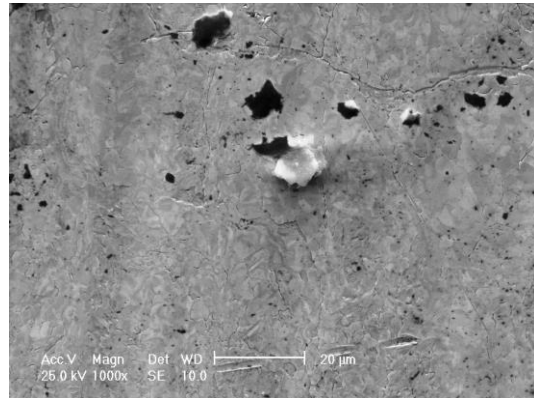
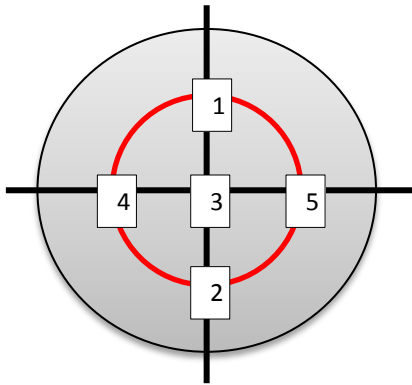


After 5 h + 5 h + 15 h in ETHEL

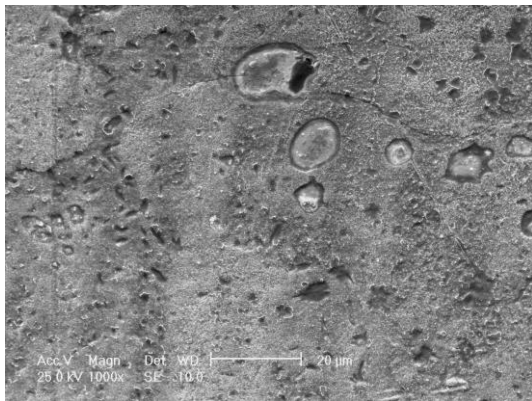
No erosion over the 10 h was found by visual comparison.

There may be 5 typical features, one marked with red arrow, about 5 µm large.

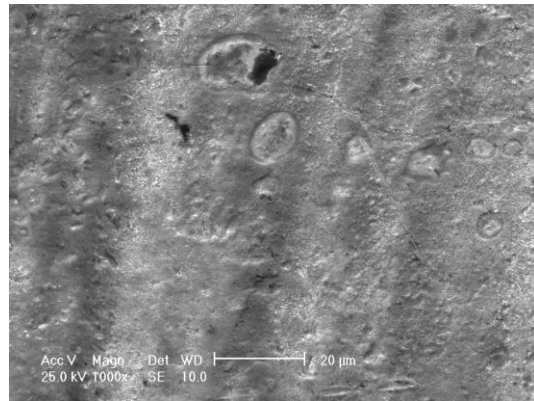
6.5. Sample 1, Position 5



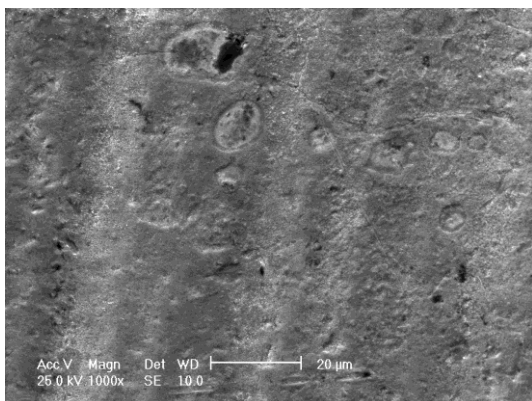
Prior to pre-oxidation



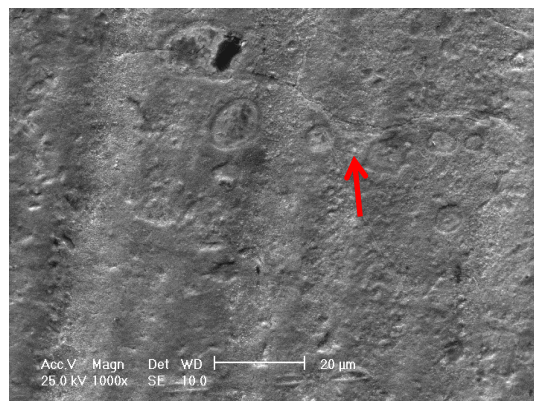
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL



After 5 h + 5 h + 15 h in ETHEL

No erosion over the 10 h was found by visual comparison.

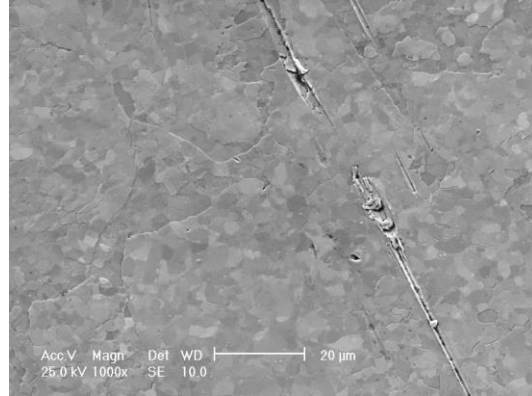
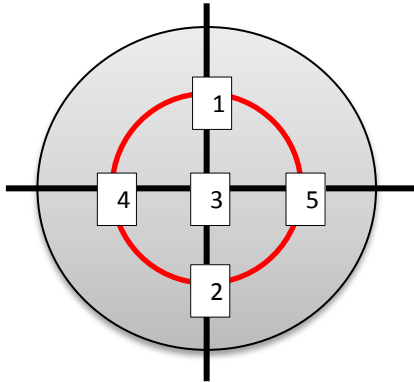
There may be 10 typical features, marked with red arrow, about 2 μm large.

Document Type	Analysis Report
Document Number	ESS-0051445
Date	Aug 25, 2016
Revision	1
State	Released
Confidentiality Level	Internal

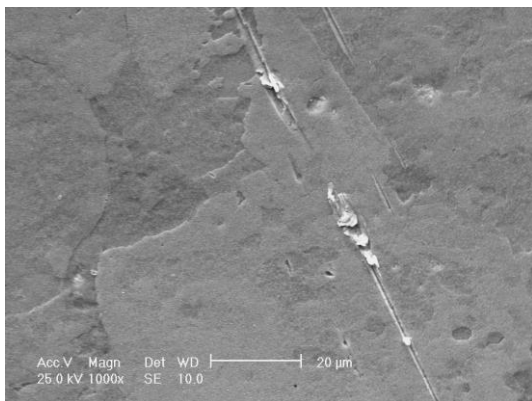
7. SAMPLE 2

Sample 1 has been exposed to two 5 h cycles and one 15 h cycle (in two runs) with impinging helium flow in ETHEL.

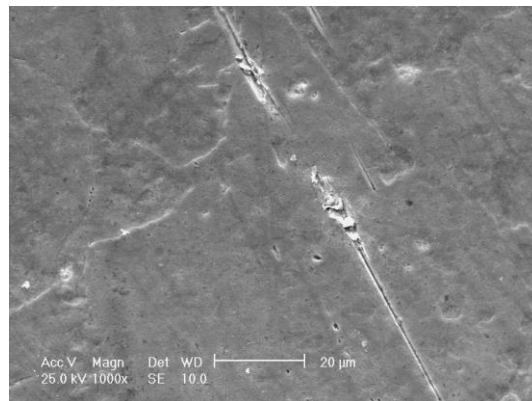
7.1. Sample 2, Position 1



Prior to pre-oxidation



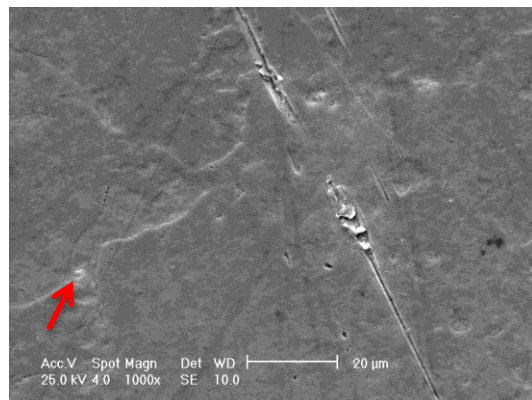
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL

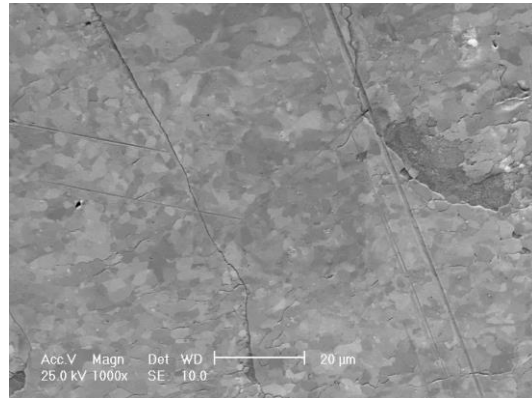
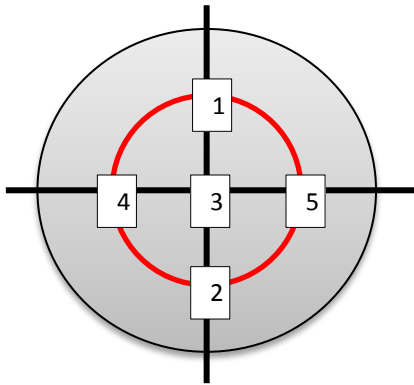


After 5 h + 5 h + 15 h in ETHEL

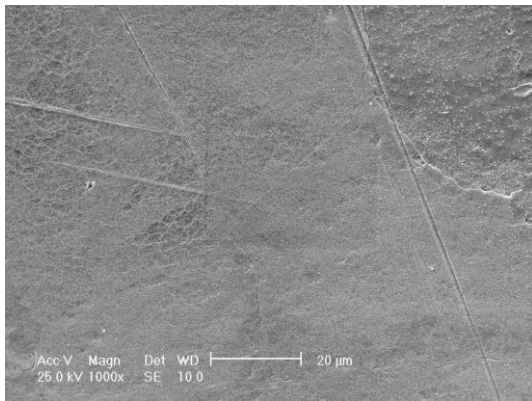
No erosion over the 5 h was found by visual comparison.

There may be 5 typical features, marked with red arrow, about 2 μm large.

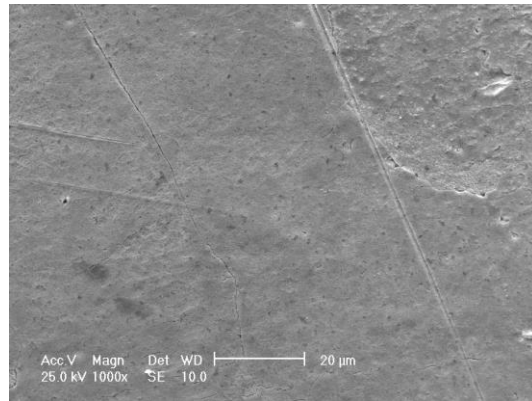
7.2. Sample 2, Position 2



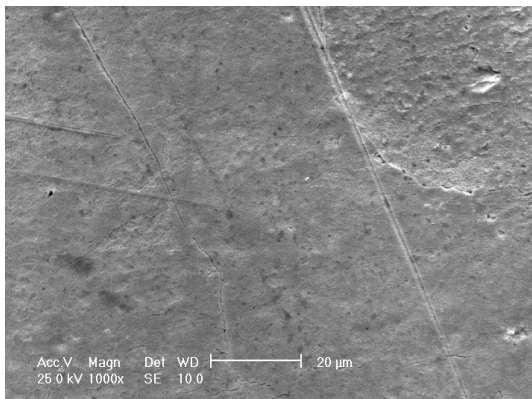
Prior to pre-oxidation



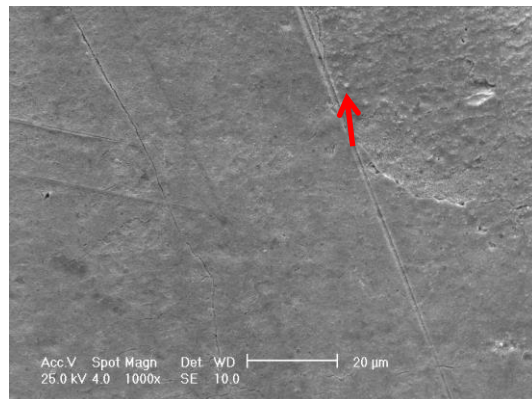
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL

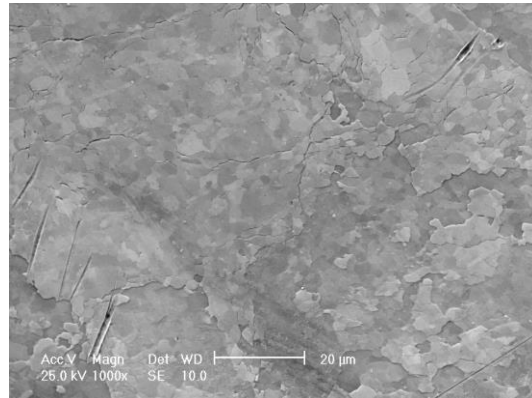
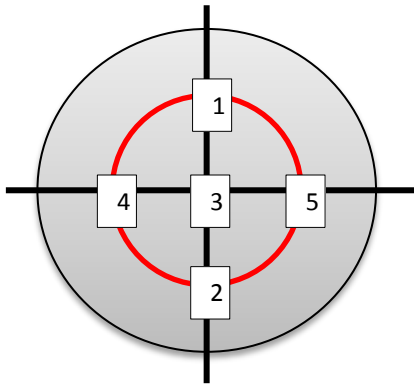


After 5 h + 5 h + 15 h in ETHEL

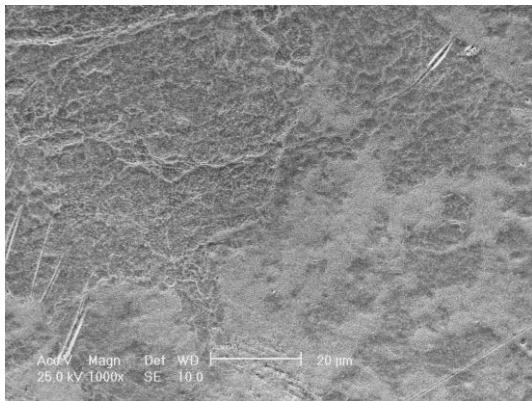
No erosion over the 5 h was found by visual comparison.

There may be 5 typical features, marked with red arrow, about 2 μm large.

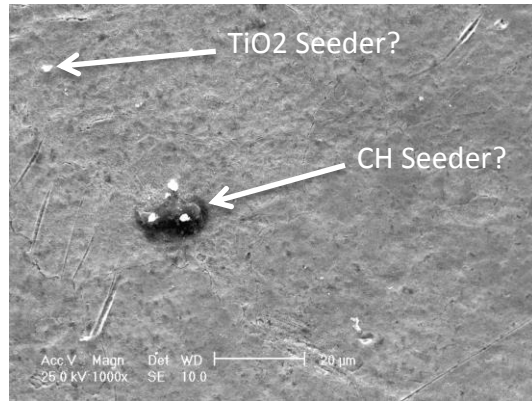
7.3. Sample 2, Position 3



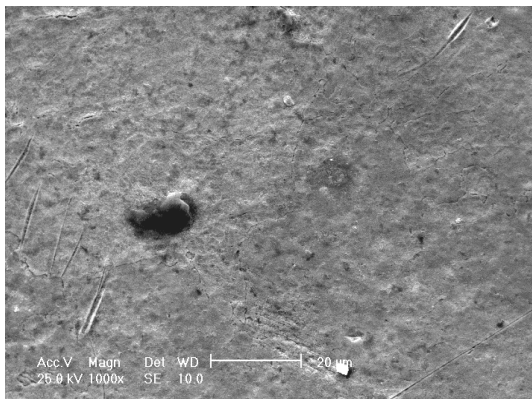
Prior to pre-oxidation



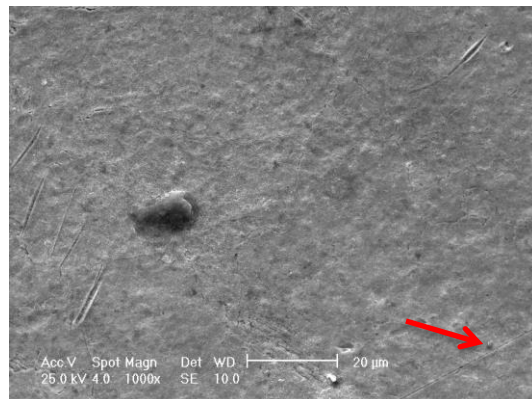
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL



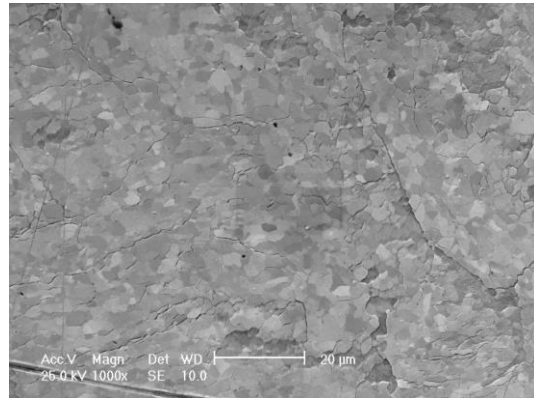
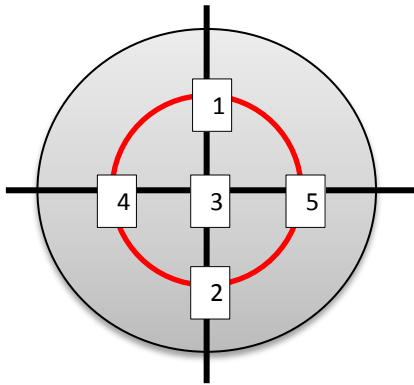
After 5 h + 5 h + 15 h in ETHEL

Titanium and Carbon was confirmed by elemental mapping, see section 3 above.

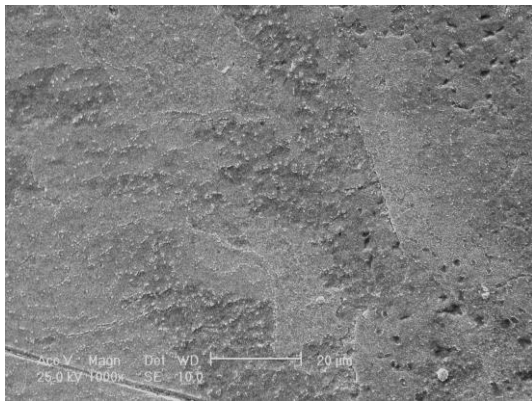
No erosion over the 5 h was found by visual comparison.

There may be 5 typical features, marked with red arrow, about 2 µm large.

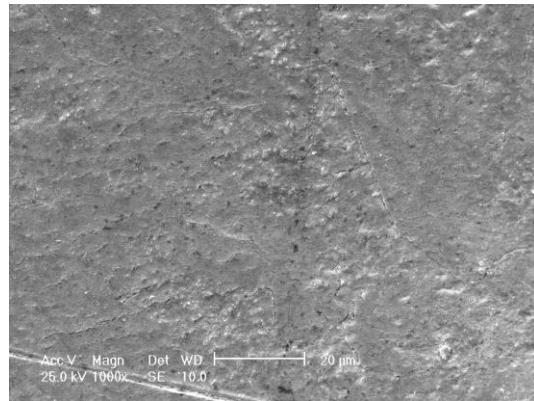
7.4. Sample 2, Position 4



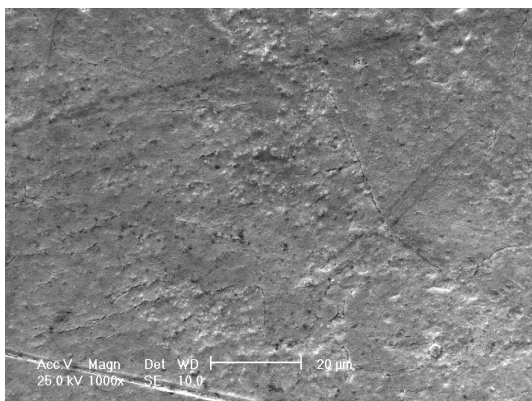
Prior to pre-oxidation



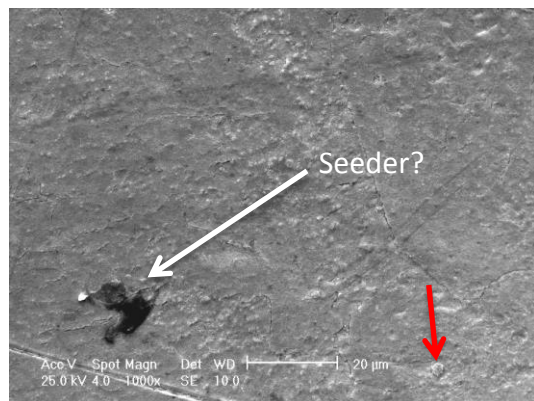
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL

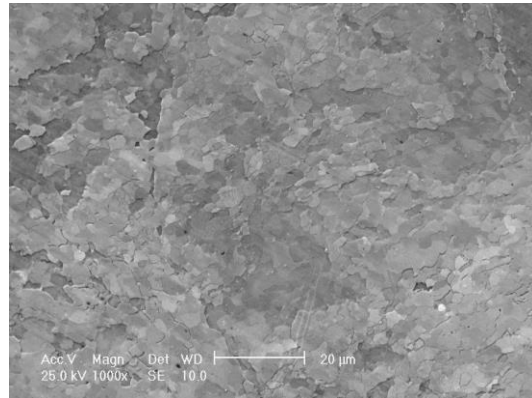
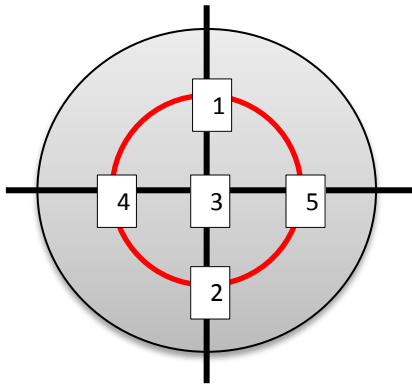


After 5 h + 5 h + 15 h in ETHEL

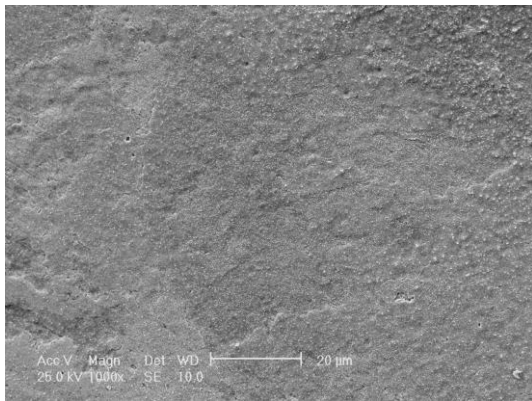
No erosion over the 10 h was found by visual comparison.

There may be 5 typical features, marked with red arrow, about 5 µm large.

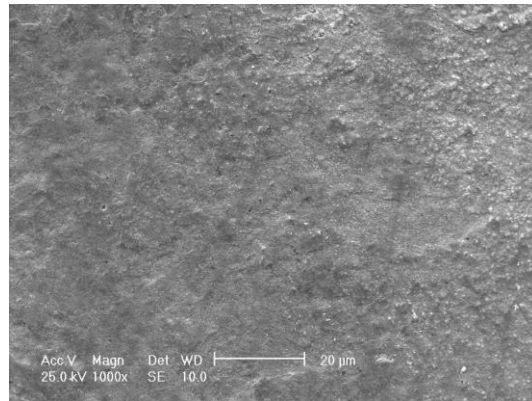
7.5. Sample 2, Position 5



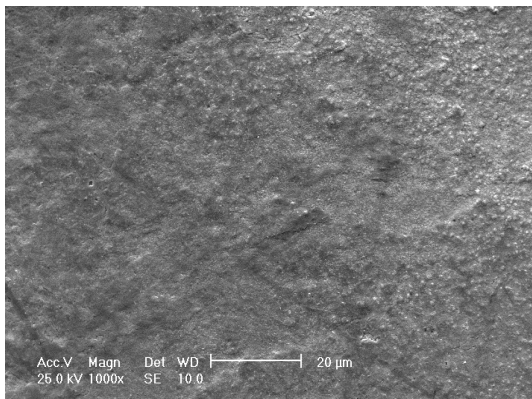
Prior to pre-oxidation



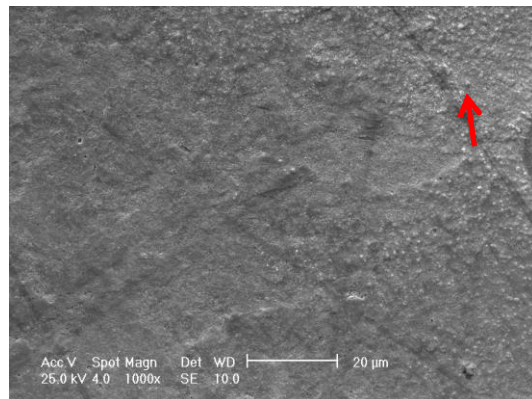
After pre-oxidation



After 5 h helium flow in ETHEL



After 5 h + 5 h in ETHEL



After 5 h + 5 h + 15 h in ETHEL

No erosion over the 10 h was found by visual comparison.

There may be 10 typical features, marked with red arrow, about 2 µm large.

8. ESTIMATED FEATURES

8.1. Volumetric estimate from SEM

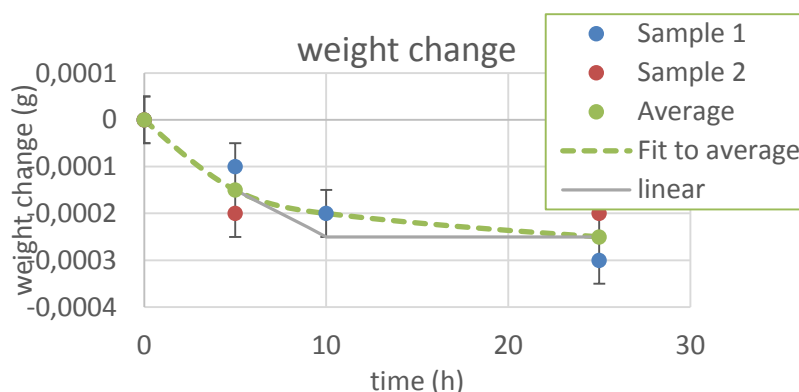
The estimated protruding features are not affected by the flow in these tests, as can be seen in the figures. Nevertheless, the volume of those features could be estimated, to give some kind of estimate of erosion, as if though the helium would blown them all off. Let us estimate that they are cube shaped, the largest ones are $(5 \mu\text{m})^3 = 125 \mu\text{m}^3$. Some pictures have about 10 of them. If we assume that they are blown off in an operation period, scale to the target wheel tungsten area of about 43 m^2 and assume the tungsten oxide density of 7160 kg/m^3 , we get this erosion in the wheel lifetime:

$$1250 * 10^{-18} \text{ m}^3 * 7160 \text{ kg/m}^3 * 43 \text{ m}^2 / 0.01 * 10^{-6} \text{ m}^2 \sim 0.04 \text{ kg.}$$

8.2. Weight estimate

Microbalance weight measurements, before and after every run, confirm that there is no measureable erosion of this type of tungsten oxide, see table:

Weight in g	Sample 1	Sample 2
Prior pre-oxidation	18.2980	18.1154
After pre-oxidation	18.2986	18.1164
After 5 h	18.2985	18.1162
After 5 + 5 h	18.2984	18,1162
After 5 + 5 + 15 h	18.2983	18.1162



Error bars here denote limited resolution only (no inaccuracy). The interpretation that nothing happens after 10 h (linear) is within these error bars, but barely.

The samples lost on average 0.00025 grams in the test. Their area is 0.000314 m^2 and scaling this to the target size yields:

$$43/0.000314 * 0.00025 \sim 0.033 \text{ kg}$$

This would be removal of loose particles from the pre-oxidation, i.e. not continuous.

Document Type Analysis Report
Document Number ESS-0051445
Date Aug 25, 2016
Revision 1
State Released
Confidentiality Level Internal

9. REFERENCES

- [1] Nilsson, P., Schönborn, A, and Klingmann, J., ESS Target Helium Experiments at Lund University, ICONE24-60721, 2016.

DOCUMENT REVISION HISTORY

Revision	Reason for and description of change	Author	Date
0	First issue	Per Nilsson Jemila Habainy Jens Klingmann	160615
1	Incorporating comments from reviewers	Per Nilsson Jemila Habainy Jens Klingmann	160810