

## LA-UR-14-29069

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Title: DELTA Loop Corrosion Study Operations

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Intended for: Work package close-out report to DOE-NE

Issued: 2014-11-24

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# DELTA Loop Corrosion Study Operations

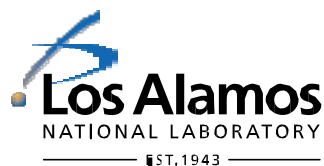
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Close-out Report on Activity SR-14LA2001022

*Author:*

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September 9,  
2014



## Summary

The DELTA (DEvelopment of Lead Alloy Technical Applications) loop, was designed and built as part of the Accelerator Transmutation of Waste program and became operational in 2001. The DELTA loop has supported Lead-Bismuth Eutectic (LBE) corrosion and thermal- hydraulic studies for reactor and accelerator systems and designs [1]. Due to the relatively benign behavior of LBE in contact with air or water, as compared with sodium, makes it an attractive option for high temperature heat transfer applications and reactor cooling. In order to demonstrate the viability of LBE and material candidates, LBE induced corrosion must be understood and controlled. The DELTA loop is a platform with which LBE induced corrosion and LBE oxygen control can be investigated. DELTA is illustrated in Figure 1. During this campaign, a collaboration with SCK-CEN in Belgium on the measurement and control of oxygen content was folded into the schedule. The results of this collaboration are reported in an appendix.

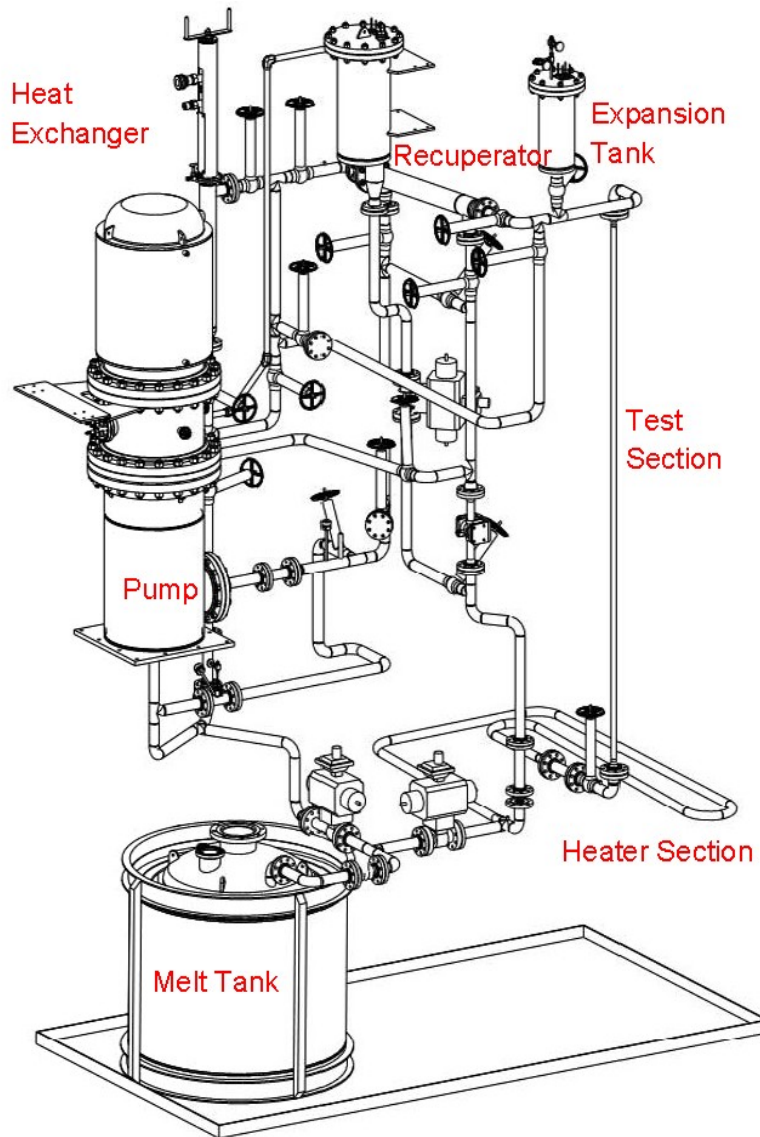


Figure 1: The DELTA Loop.

## Corrosion Test Report, First 1000 Hr

In March 2012, the DELTA planned 3000 hour corrosion test began. The targeted experimental conditions are:

- 500°C
- 3.0 - 3.3 m/s at the canister
- 5 m/s at the teardrop
- $5 \times 10^{-5}$  -  $5 \times 10^{-7}$  %wt oxygen

The test was focused on long term corrosion and erosion on candidate materials for use in new fast LBE reactors. More specifically, the test is highlighting the performance of alloying elements that enhance the corrosion resistance of steels in LBE, namely Si and Al. While many of these materials have been subjected to previous testing, this test was conducted at velocities greater than the generally accepted limit of 2 m/s based on Russian experience, as yet not documented nor collaborated. Table 1 shows the suite of materials included in the experiment. Post-test analysis of the samples is documented in a separate report.

In all, 3 periods of 1000 hrs was planned, with 2 identical sample canisters in at all times. After the first 1000 hrs, a single canister would be removed and replaced with a new canister. The 2 now in place would run for the remaining 2000 hrs, resulting in samples pulled at 1000 hr increments.

The test canister was designed to reach flow speeds of 3 - 3.3 m/s across the samples. The canister is a slight modification of previous canisters used for corrosion tests, as shown Fig 2 and Fig 3. Along with the corrosion canister, a pipe provided by Dr. Ron Ballinger from MIT was tested. The inner surface of this pipe has been weld inlaid with a Si-based coating. The test is to show the viability of this technique for flow velocities up to 5 m/s. This is obtained by using a diffuser like “teardrop” mounted within the pipe as shown in Fig 4. The complete inserted test section is shown in Fig 5. Unfortunately, a failure of the mechanical support of the teardrop invalidated the results of the MIT pipe segment.

The actual average experimental conditions were:

- Temperature - 488°C
- Volumetric Flow - 5.33 m<sup>3</sup>/hr
- Sample Flow Velocity - 3.54 m/s
- Oxygen Concentration -  $2.30 \times 10^{-5}$  %wt oxygen

Running temperature is shown in Fig 7. The heaters were not able to reach the target of 500°C consistently due to heater failures, both in the main heater bank and in the piping trace heaters. The heater system was able to sustain the temperature above the lowest acceptable temperature of 480°C.



Figure 2: Corrosion Test Assembly and Sample Coupons. Top Left: Sample Coupons. Bottom Left: Old style Coupon Holder. Bottom Right: Fully assembled Test Section Insert

The running oxygen concentration is shown in Fig 8. The operational oxygen target was to run to the higher portion of the band at around  $1 \times 10^{-5}\%$  wt. Step changes in oxygen concentration typically correspond to system shut-down and drain, during which the LBE in the loop mixes with stagnant LBE remaining in the melt tank. Fig 9 shows the acceptable oxygen concentration operating regime for LBE as a function of temperature.

The sample canister volume flow rate and flow velocity as shown in Fig 10 and 11, respectively. Actual volume flow rate exceeded our design by a small margin, resulting in average velocity of 3.6 m/s.



Figure 3: Modified canister design with smaller clearances to allow for higher flow velocities of 3 - 3.3 m/s.

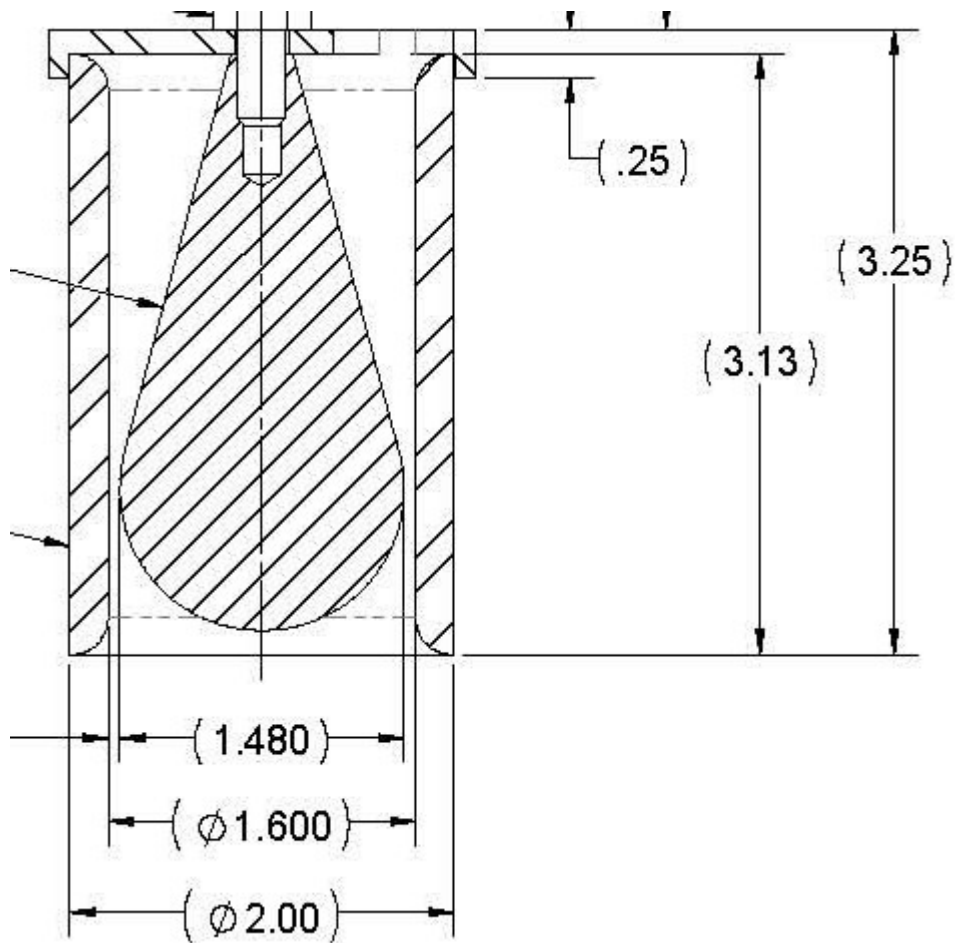


Figure 4: Drawing of the teardrop and pipe portion of the experiment.



Figure 5: The complete test section. Shows the top flange connected to the sample canisters (left) and the pipe/teardrop experiment (right) via a threaded rod.



Figure 6: Corrosion experiment as it looked when removed after 1000 hours. Canisters are still attached to the threaded rod and Si inlaid pipe had been detached from the threaded rod.



Table 1: Chemical Composition of 3000 hr Corrosion Test Specimens

Material	Fe	C	Mn	Si	Cr	Ni	Mo	V	W	Nb	Al	Y203	Ti
(%)													
Ht-9 (2048)	Bal.	0.18	0.4	0.2	12.26	0.49	1	0.3	0.46	—	—	—	—
T91	Bal.	0.105	—	0.43	8.26	0.13	0.95	0.2	—	0.075	—	—	—
EP823 (2054)	Bal.	0.16	0.55	1.09	11.7	0.66	0.74	0.3	0.6	—	—	—	—
MA956	Bal.	0.04	—	—	20	—	—	—	—	—	4.5	0.5	0.4
PM2000	Bal.	0.01	—	—	20	—	—	—	—	—	5.5	0.5	0.5
1.4970	Bal.	0.11	1.4	0.3	15	15	0.48	—	—	—	—	—	0.48
APM	Bal.	< 0.08	< 0.4	< 0.7	20.5-23.5	—	—	—	—	—	5.8	—	—
APMT	Bal.	< 0.08	< 0.4	< 0.7	21	—	3	—	—	—	5	—	—
Alkrothal 14	Bal.	< 0.08	< 0.5	< 0.7	14-16	—	—	—	—	—	4.3	—	—
Alkrothal 720	Bal.	< 0.08	< 0.7	< 0.7	12-14	—	—	—	—	—	4	—	—
Alloy 4	88	—	—	—	12	—	—	—	—	—	—	—	—
Alloy 8	87.5	—	—	0.5	12	—	—	—	—	—	—	—	—
Si-Fe "A"	Bal.	0.01	0.04	1.24	0.09	0.08	0.01	< 0.01	0.03	< 0.01	0.005	—	0.003
Si-Fe "B"	Bal.	0.017	0.12	2.55	0.08	0.15	< 0.01	< 0.01	< 0.02	< 0.01	0.003	—	0.006
316L	Bal.	0.002	1.8	0.46	17.5	12.3	2.3	—	—	—	—	—	—

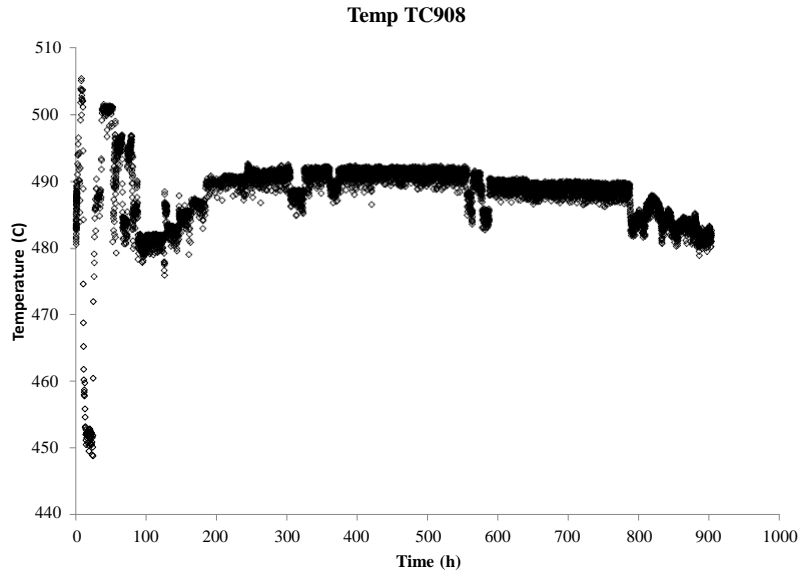


Figure 7: Temperature at the sample canisters (TC908).

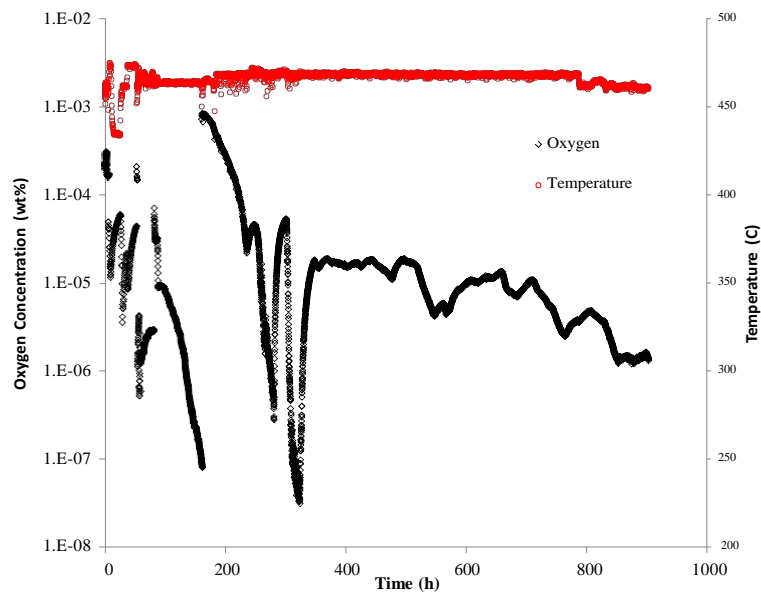


Figure 8: Oxygen concentration from OS104.

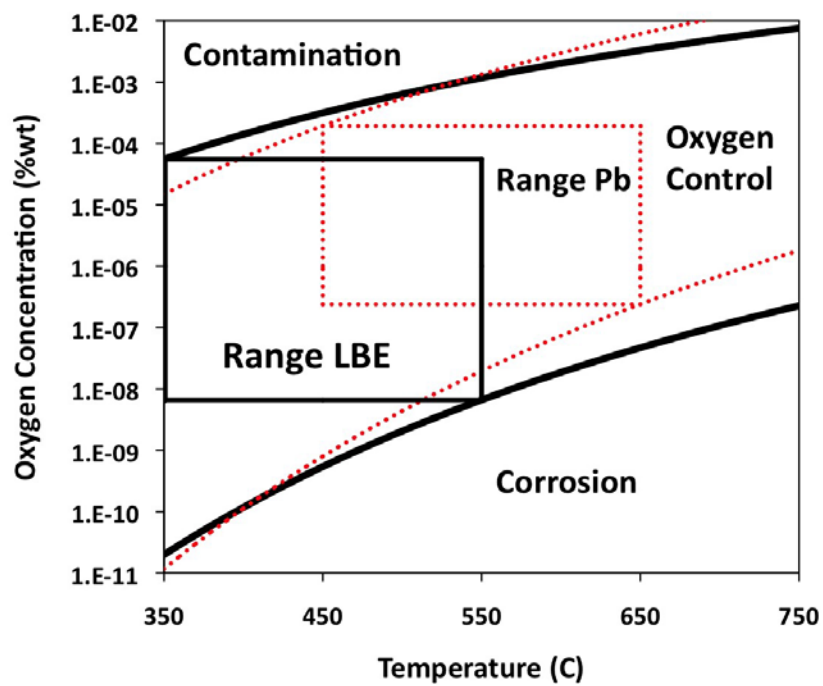


Figure 9: Typical oxygen control band for LBE and lead. Top set of lines are the %  $O_2$  corresponding with saturation for  $PbO$  and the bottom set of lines correspond to the  $Fe_2O_3$  saturation. [2]

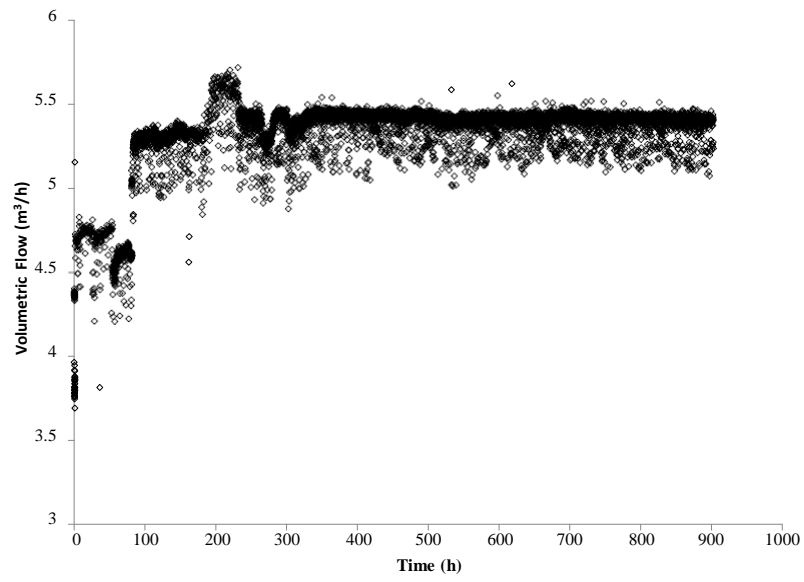


Figure 10: Volumetric flow rate as read from the venturi flow meter.

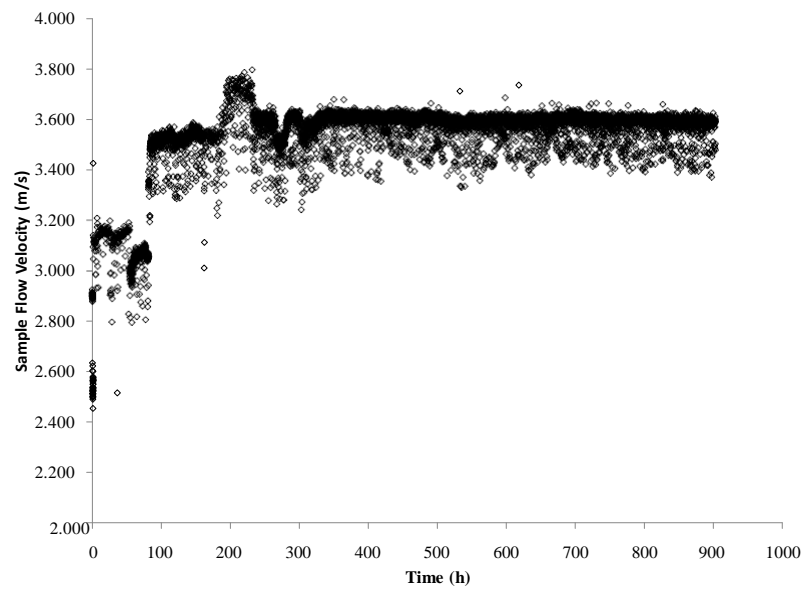


Figure 11: Flow velocity in the test canister.

## **Corrosion Test Continuation**

After removing 1000 hr samples and some maintenance activities, the intended 3000 hr test was resumed. However, operation from that point on was plagued by a series of problems resulting in SCRAM events, followed by maintenance and repair activities, followed by restart. In all, over a 5 month period, the test was interrupted 15 times. Total accumulated time on samples was an additional 400 hrs. The test was prematurely halted because of a flange gasket failure that resulted in a large leak and facility shut-down by ES&H authorities. Clean-up, with the intent of being ready for future programmatic activity, is in progress.

The temperature and flow conditions for this 400 hr run segment were at slightly higher than during the first 1000 hrs, nominally 480°C and 3.6 m/s or higher. Generally however the oxygen concentration was higher, typically at or near saturation. Samples from this 400 hr segment have yet to be removed for analysis.

## Appendix A

### Oxygen Sensors

Oxygen sensors that are used in the DELTA loop are Bi/BiO potentiometric oxygen sensors. They read out a voltage which corresponds to the percent weight of dissolved oxygen using equation 1 [2, 3].

$$O_2(wt\%) = 10^{1.2 - \frac{3400}{T[K]} \times 10^{\frac{10083(0.1381 - V)}{T[K]} - 0.366}} \quad (1)$$

Oxygen sensor design has been altered to make the sensor more streamlined and more efficient to rebuild. As shown in Fig A1, the older style uses a conflat tee piece to connect the BNC to the reference electrode. The current design removes the conflat tee and attaches the BNC connector right to the sensor shell flange as shown in Fig A2. This is possible because the purpose of the conflat tee is affixing the reference electrode wire to the BNC connector. This section is compressed into the top part of the sensor shell. Another design alteration was to use a thick ceramic tube to supply the compression to the bottom gasket seal of the ceramic to the metal holder. This did not work as well as expected. Every sensor using the thick ceramic tube had leaked in and failed. Currently the new oxygen sensors with the old type of internal details are being tested. OS101, OS102 and OS103 are of this design.



Figure A1: Older style oxygen sensors.



Figure A2: New streamlined oxygen sensor without the conflat tee.

The overall goal of the oxygen control experiment was to get a correlation to relate temperature and flow velocity to oxygen dissolution of PbO spheres in support of the MYRRHA project. This is similar to the PbO pebble oxygen mass transfer experiment performed by Alan Bolind [4] previously on the DELTA loop. It was proven that it is possible to increase oxygen concentration by flowing LBE over PbO pellets qualitatively.

The experimental setup was a test basket containing the PbO spheres with SCK-CEN oxygen sensors (Fig. A3) placed at the inlet and outlet of the test section. The top oxygen sensors were designed to detect the small variations in oxygen concentration due to flow variances at the top of the test section. The bottom oxygen sensor was added to replace OS102 at the inlet of the test section. Due to the small diameter of the oxygen sensor, a blank tube was fashioned to house the sensor in order to protect the sensor from flow and the copper conflat gasket from flowing LBE. Despite the difficulties of setting up the experiment, the experiment was performed well and with good data results. Inlet oxygen concentration versus outlet concentration suggests that the PbO spheres are introducing oxygen into the LBE. Also, as shown in Fig 19, the rate of oxygen mass transfer into

the LBE shows a strong relationship. The raw data from the experiment has been analyzed at SCK-CEN by Alessandro Marino, and will soon be submitted for publication.





Figure A3: SCK-CEN oxygen sensors. They work on the same principle as LANL designed oxygen sensor. Internal construction designs are proprietary.



Figure A4: Left: Basket configuration in the test section. Middle: Basket Side View. Left Top: Basket Bottom strainer. Left Bottom: Basket internals with PbO Spheres. Courtesy of SCK-CEN, Belgium



Figure A5: Tube fashioned to protect the SCK oxygen sensor and conflat copper gasket.

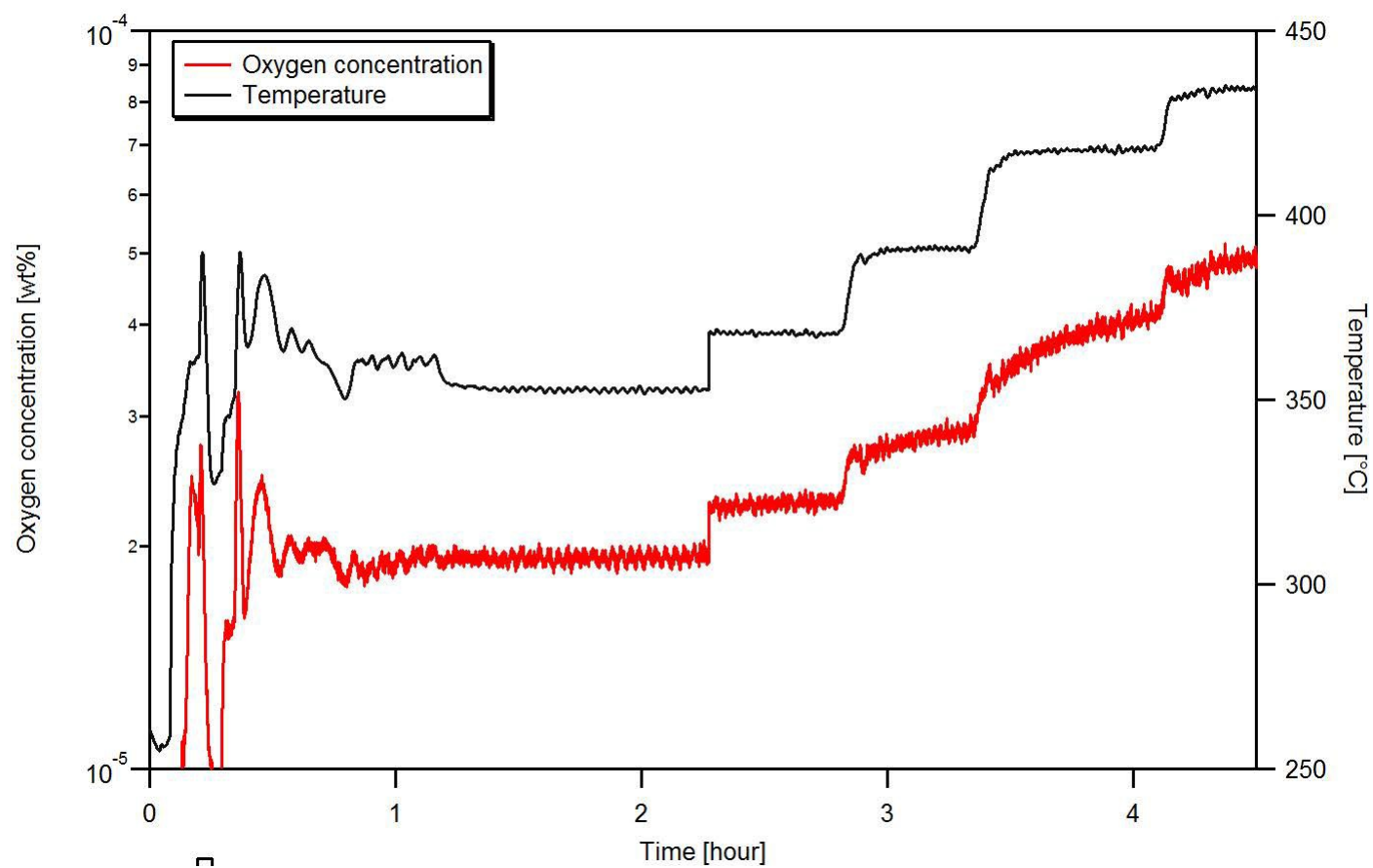


Figure A6: Raw data collected from the direct oxygen mass transfer experiment, courtesy of Alessandro Marino.

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