Detection of hydrogen as impurity in liquid lithium: electrochemical hydrogen-sensor for IFMIF-DONES

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31ST SYMPOSIUM ON FUSION TECHNOLOGY

Development of an H-Sensor system for lithium melts

Non-metal Impurity Measurement in Liquid Lithium :

Motivation: Within the DONES facility to be built in Granada, Spain, a deuteron irradiated Li-flow target generates DEMO-relevant neutrons, by which the forseen fusion power plant materias can be tested and evaluated under DEMO conditions. A critical aspect will be the hydrogen impurity content and its control in the liquid lithium sytems of IFMIF-DONES => ***important issue due to corrosion and safety risks***.

Objective and approach: So far, a consequential EUROFUSION task was the development of an **E**lectro**C**hemical **H**ydrogen **S**ensor for **L**iquid **L**ithium (ECHSLL).

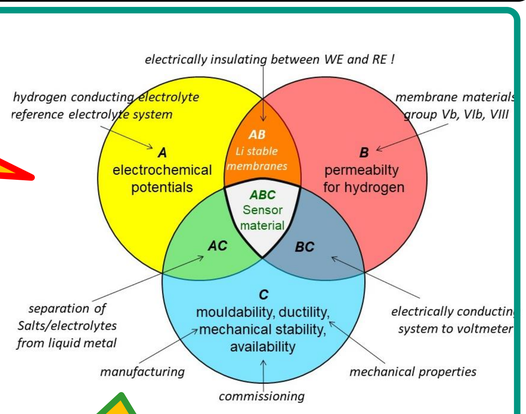
Design of ECHSLL

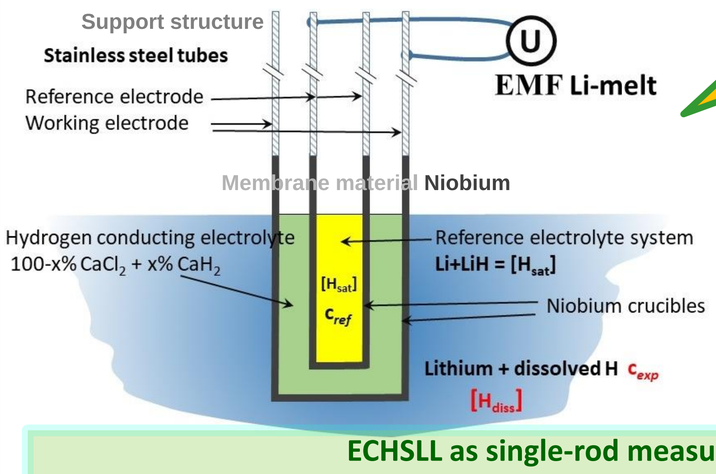
Electrochemical conception of ECHSLL:

Cross-section task requirements:

High permeable for hydrogen
+ stable in liquid lithium +
+ mech stability vs, shear forces
+ insulation for potential measurements
+ conducting voltage contacts,.....

Solution: ECHSLL as "*single-rod measuring cell*"





Inner tube: reference electrode (RE) = Nb + reference electrolyte system (RES)

$c_{ref} = [H_{sat}]$

Outer tube: working electrodes (WE) , supporting H-conducting electrolyte, in contact via Nb with Li-melt and dissolved hydrogen $c_{exp} = [H_{diss}]$.

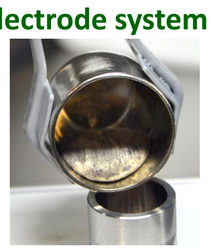
ECHSLL as single-rod measuring cell

EMF as result of voltage between potentials of hydrogen dissolved in Li-melt and the concentration in the RE-system

ECHSLL materials: membranes, electrolyte, reference

Reference electrode system

Li + LiH = 1:1
Supersatured lithium



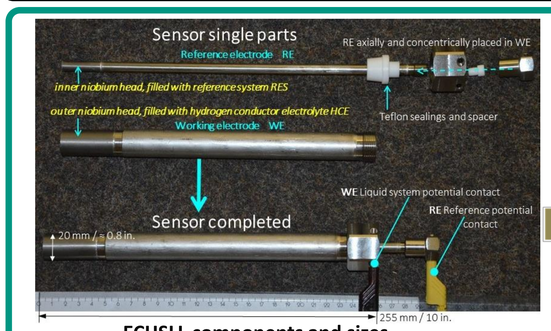
Electrolyte material

$CaCl_2$ +5%CaH_2 + 1073K =>
CaHCl-based hydride conductor electrolyte
Sridharan et.al., J.Nucl. Mat. 223 (1995) 72-79

Niobium membranes

for the double-tube sensor design, thickness 600 µm.
Outer Nb D = 20mm, Inner Nb D = 8mm

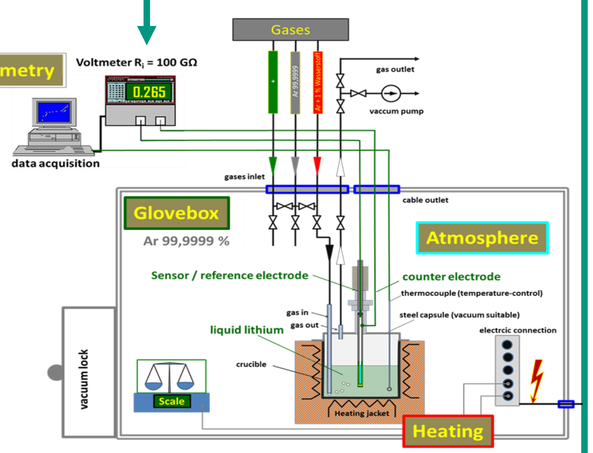
ECHSLL-set-ups



ECHSLL components and sizes

Series 1+2 of ECHSLL sensors

High performance Voltmeter
Keithley K2002 with R_i =100 GΩ: $EMF_{exp} = f(R_i)$

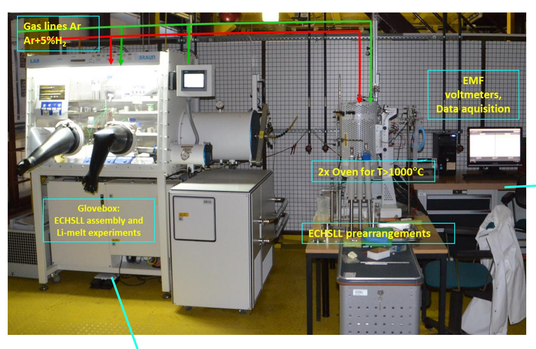


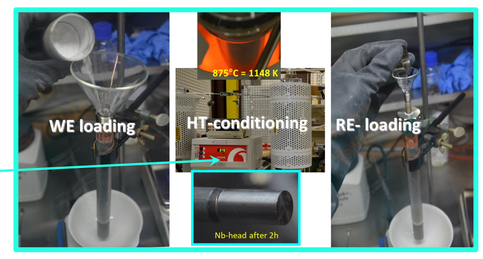
Set-up of sensor and experimental side

Flow diagram: Ar-atmosphere by Glovebox MB20G.
Heating, supporting and monitoring system

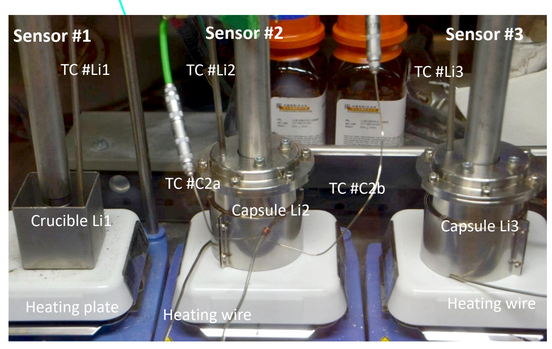
ECHSLL-application and tests

Preparation + Investigation was carried out in inert atmosphere:
ECHSLL chemical systems/substances are sensitive to water/moisture, air, and nitrogen.

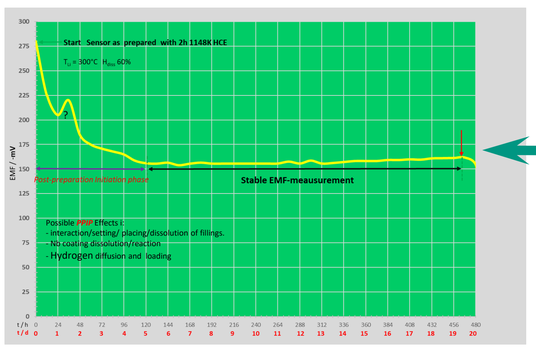




Main steps of sensor preparation:
filing of outer tube (WE) with $CaCl_2/CaH_2$, HCE HT-conditioning, RE filling with Li/LiH



Measurements set-ups :
Besides crucibles for open visible short-term experiments with 1o-5o ml melt, also use of heating capsules with Li melt amounts up to 1oo ml; three independent contemporary systems (containment material: steel).
Due to prevent observed electrical charging effects, each sensor connected to an own voltmeter.



Measurements :

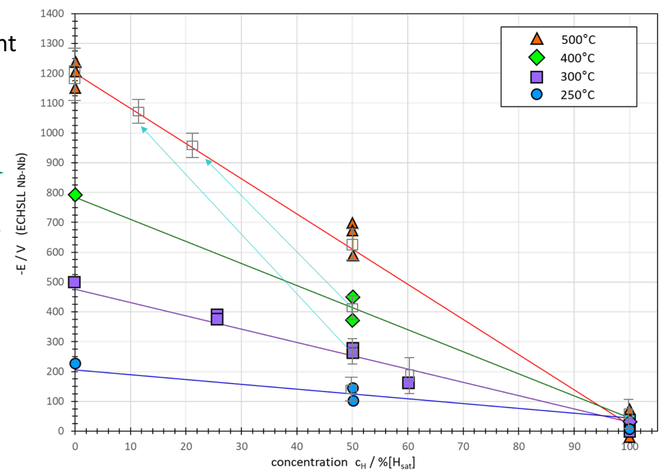
Long-term measurement > 300h with 60% of H_{sat}.
- Observed Initiation phases (PPIP) of sensor, incipiently showing arbitrary parasitic behaviour. To be avoided by post preparation T-t-procedures to reach initial identic balance conditions.
- Stable behaviour for several hundred hours (slight V increase caused by H loss).
- No corrosive affects on Nb-measure heads: functional and structural materials not disabled!

Measurements with comparisons of different temparatues > 300°C, for three c[H]:
Pure Li H ≤ 1ppm, half-saturated, saturated.

First calibration for c(H) as f(EMF).

Observed effects by increasing temperature due to drastic increase of H-solubility in LL.
Higher noise at elevated T.

Forseen DONES temperature range of 250-300C:
Stable and durable sensor technique.



Conclusions and outlook

The developed design of the ECHSLL and the quantification of sensor

- As membrane metal was selected (due to harsh limiting conditions): Niobium
- Selected Ca-chloride based supporting electrolyte acts as solid-electrolyte.
- Internal RE double-tube sensor system: *single-rod measuring cell*.
  Fixed defined electrode distances; practicaly handling under GB conditions.
  Manufacturing of bigger sensor quantities
- Results of measurements in accordance with calculations.
- Very good long-term stability;
- Effects of Y (to reduce H content/measuring time constants) in work;
- Outlook: - thinner membrane materials (300-400 µm)
  - tests of further liq.Li-protection materials (Ti as N_2-getter)

Acknowledgment

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