Deuterium Electrochemical Charging Method for a Desorption Study in Low-alloy Steel and Pure Zr

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Introduction

- The accumulation of the hydrogen isotopes generated from transmutation and diffusion of the plasma isotopes will lead to hydrogen embrittlement. Hydrogen tend to cluster near dislocation and cause small cracks.
- In spite of numerous investigations into the retention and diffusion of Hydrogen in fusion and fission related materials, there is a large scatter in the experimental data and little agreement on the activation energy of H de-trapping.

Characterization Techniques

- Ion Beam Analysis (IBA)
- Secondary Ion Mass Spectrometry (SIMS)
- Image Plate (IP) imaging

Hydrogen retention depends on

- The gap time between introducing H and its analysis
- Temperature
- Pressure
- Complexity to execute the experiment
- Hydrogen sources (natural/ manually introduced)

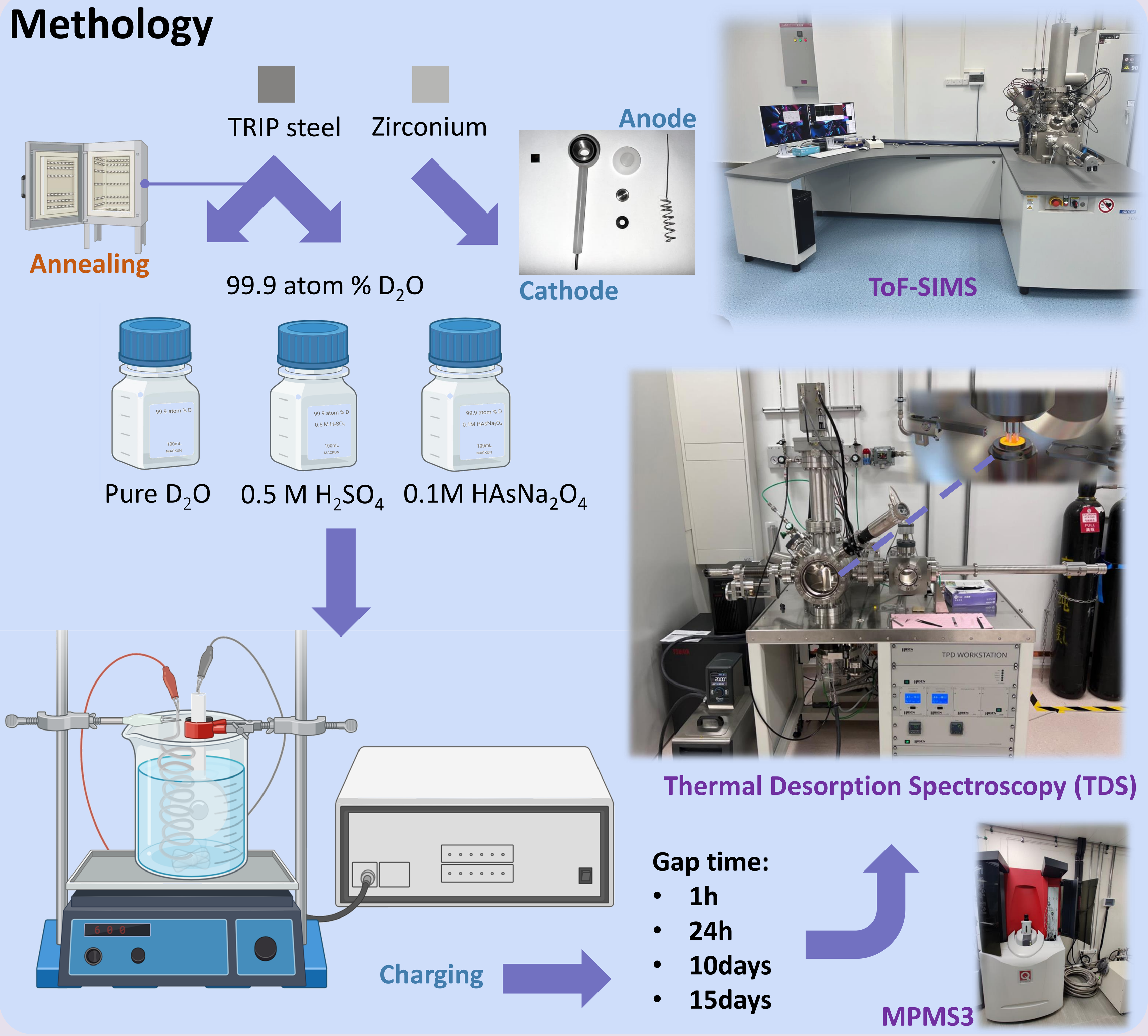
Electrochemical charging of D

Mass Spectrometry

- Simple and reliable route to introduce deuterium (D) into metals
- Clear distinction between the naturally occurring H in the materials and intentionally introduced for the experiment

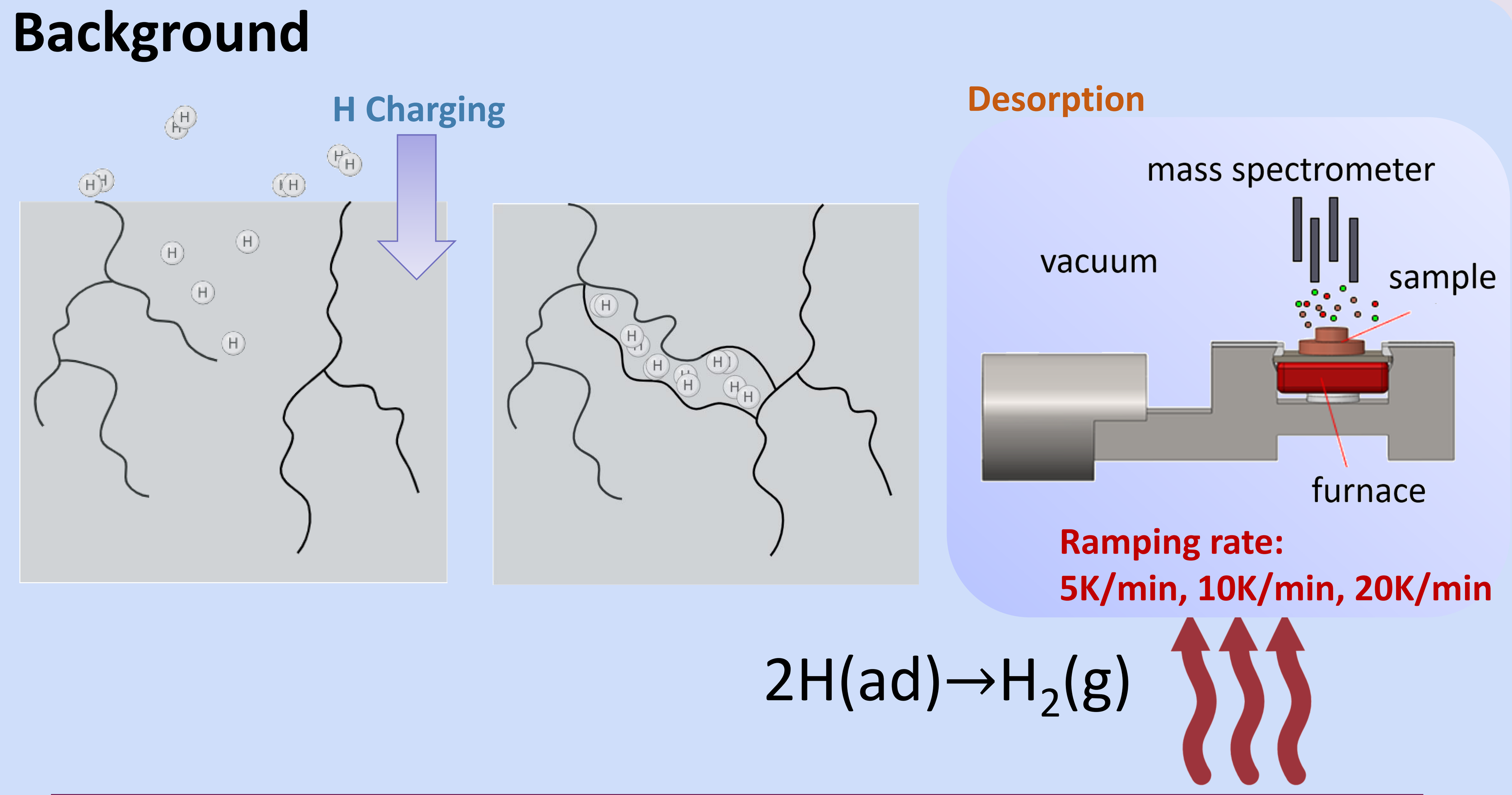
Methods used to detect hydrogen and its isotopes

Methology



Gap time:
- 1h
- 24h
- 10days
- 15days

Background



$2H(ad) \rightarrow H_2(g)$

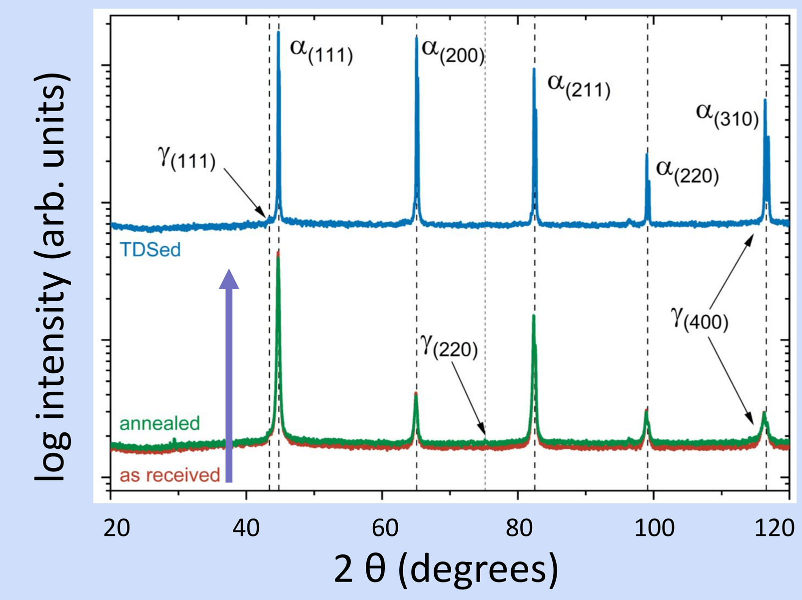
Kissinger Plot

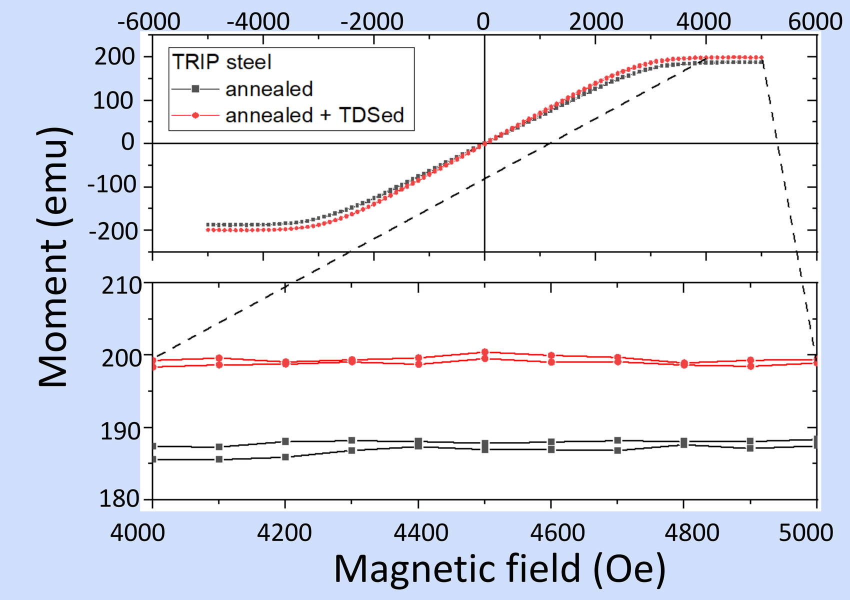
Temperature with constant ramp rate: $T = T_0 + \phi t$

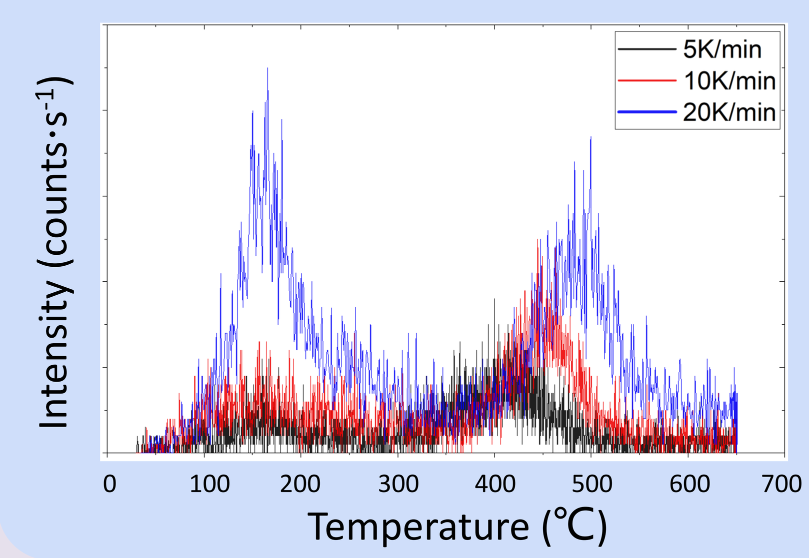
Substituting to Kissinger equation: $\frac{dx}{dt} = A(1-x)^n \exp\left(\frac{E_d}{RT}\right)$

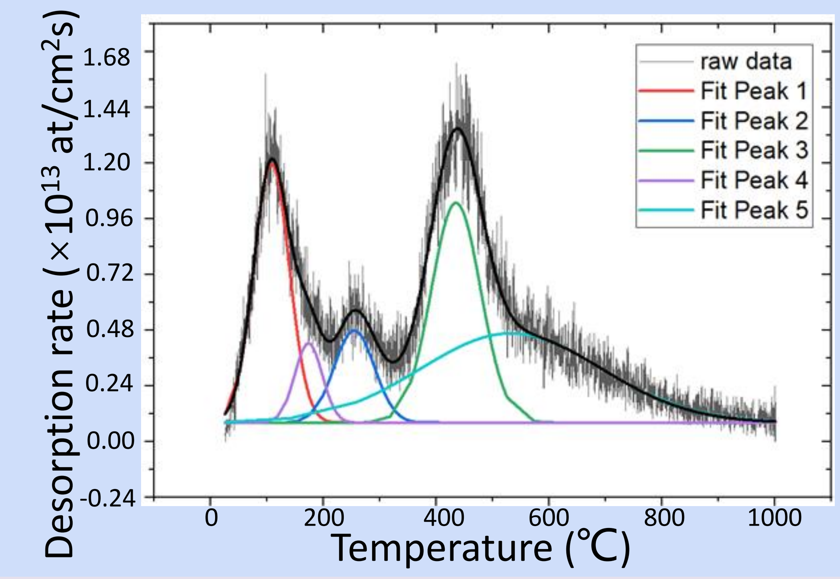
Differentiating→ $\ln\left(\frac{\phi}{T_p^2}\right) = -\frac{E_d}{R}\left(\frac{1}{T_p}\right) + \ln\left(A\frac{R}{E_d}\right)$

Results – TRIP steel

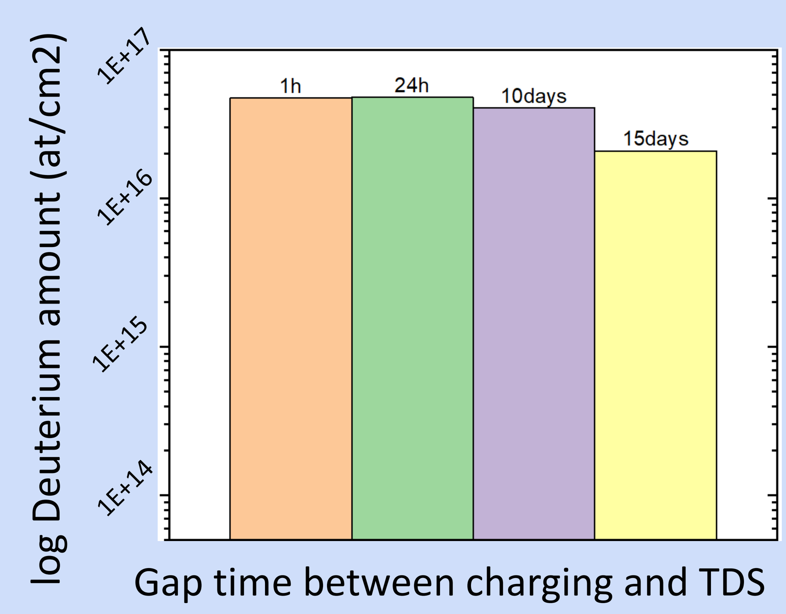


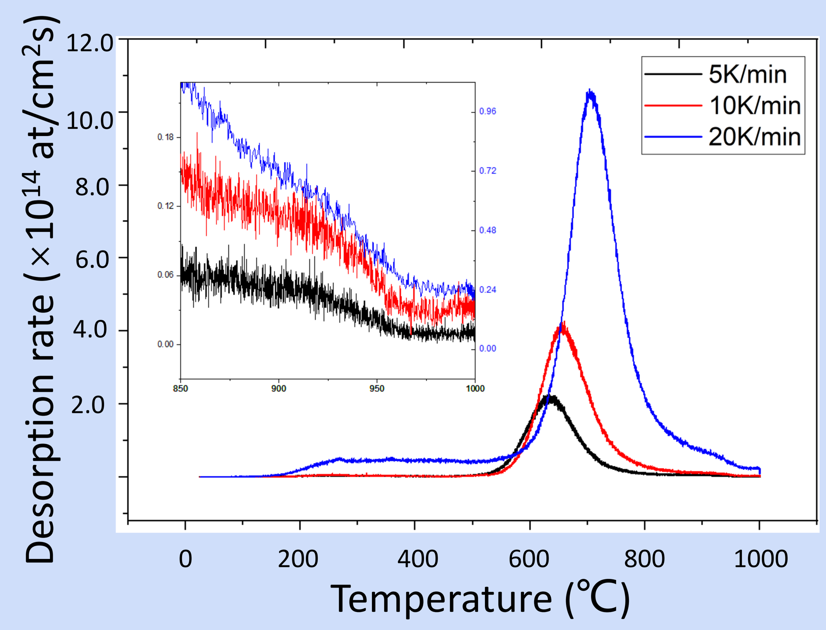


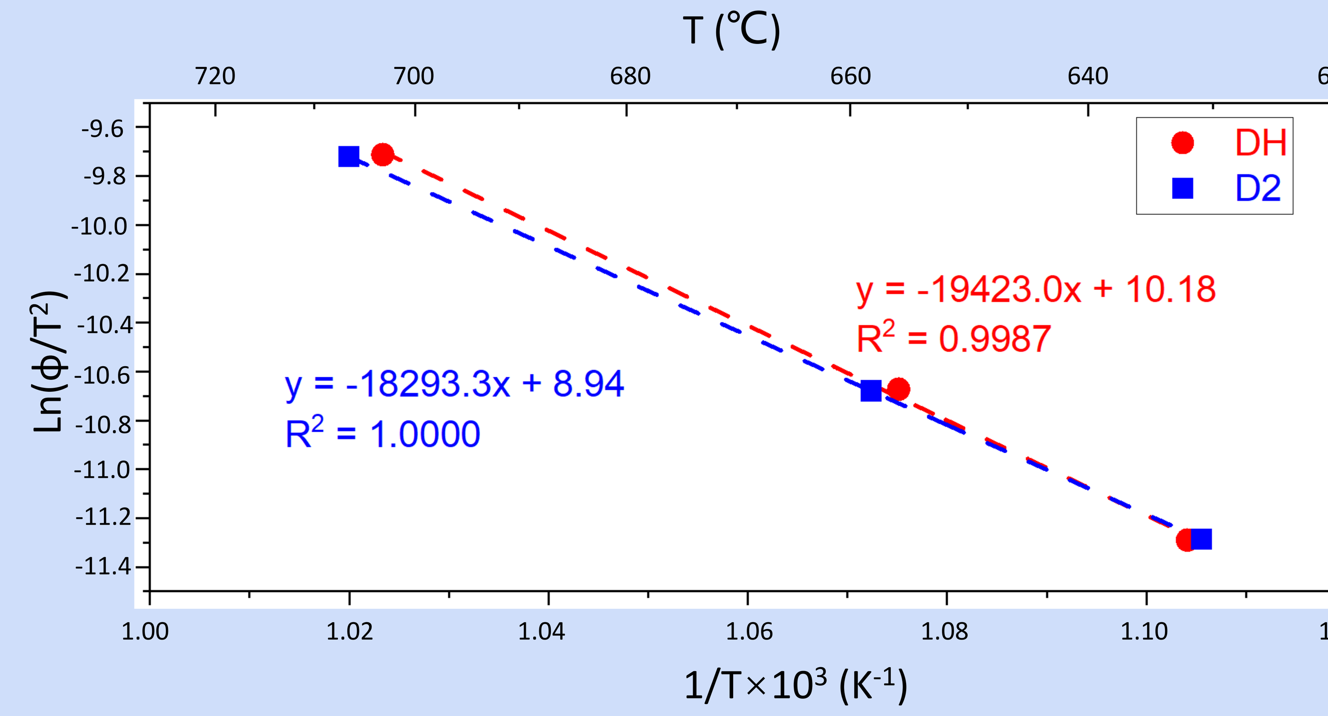




Results - Zr







Desorption activation energy

$E_A = k * R$

slop gas constant

Conclusions

- Surface-related factors, such as gas formation, phase uncertainty, and surface finishing, influence experimental results.
- The inhomogeneity of charging conditions affect the introduction of hydrogen into the samples stronger than the microstructure conditions for low temperatures traps.
- Among tested electrolytes the best reproducibility of results were obtained for pure D_2O and 0.1 M arsenate solutions respectively.

Conducting complementary tests

- **ToF-SIMS measurements:** complimentary test of Deuterium profile after D charging in Zr.
- **SQUID measurements:** investigation on the phase composition change in TRIP steel before and after annealing
- An experimental value is to be established that allows to predict the amount of D introduced to the sample according to time of charging and voltage behavior

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