Describing hydrogen diffusion in ceramic thin films materials

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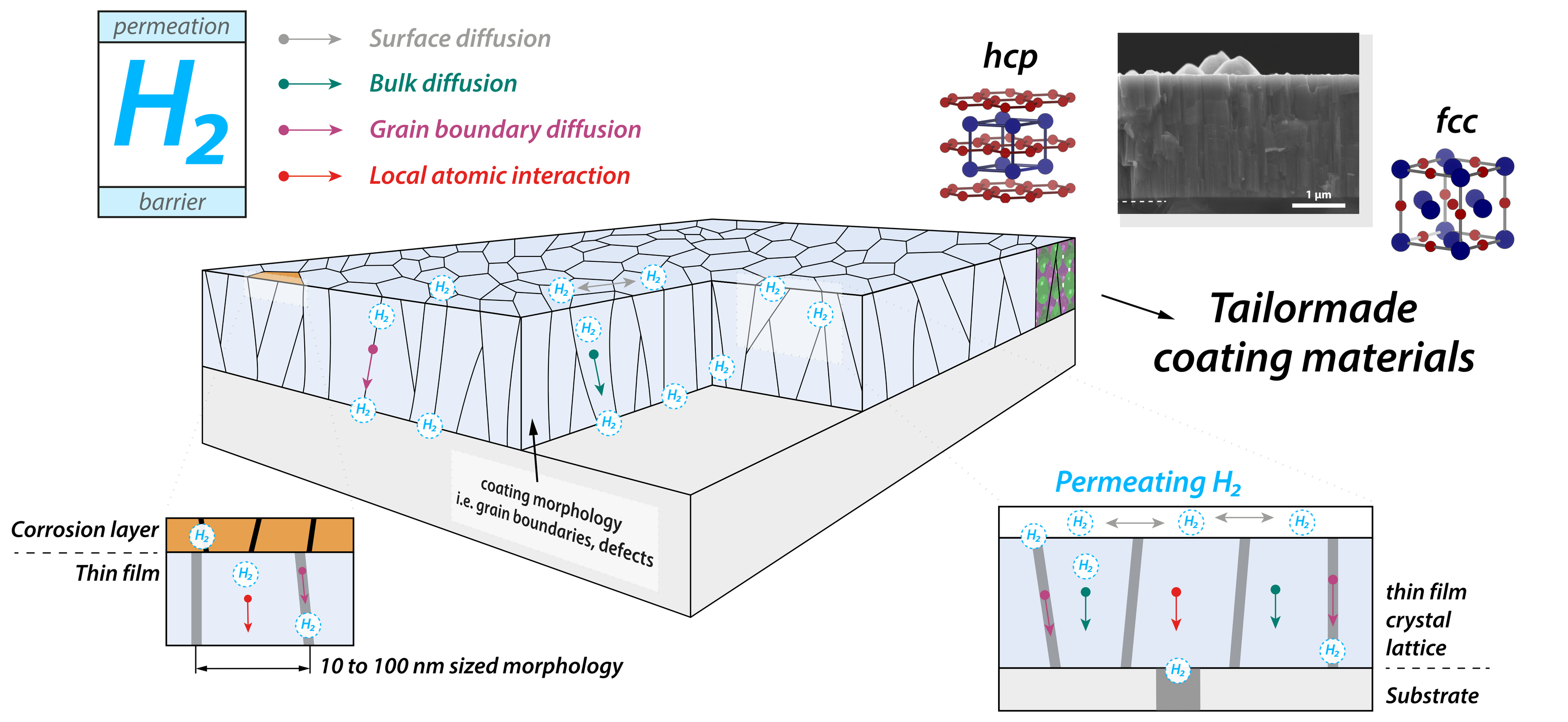
MOTIVATION



- Characterization of hydrogen permeability for bulk materials is currently done via the differential pressure method or in electrochemical setups. For thin film materials, a standard has yet to be established[1,2]
- Within this study, the electrochemical Devanathan-Stachursky setup is tested to describe the hydrogen permeation within CrN and TiN coatings grown by different deposition techniques

[1] V. Nemanic, Hydrogen permeation barriers: Basic requirements, materials selection, deposition methods, and quality evaluation, Nuclear Materials and Energy, 2019
[2] J. Matějíček et al.: Characterization of less common nitrides as potential permeation barriers, Fusion Engineering and Design, 2019

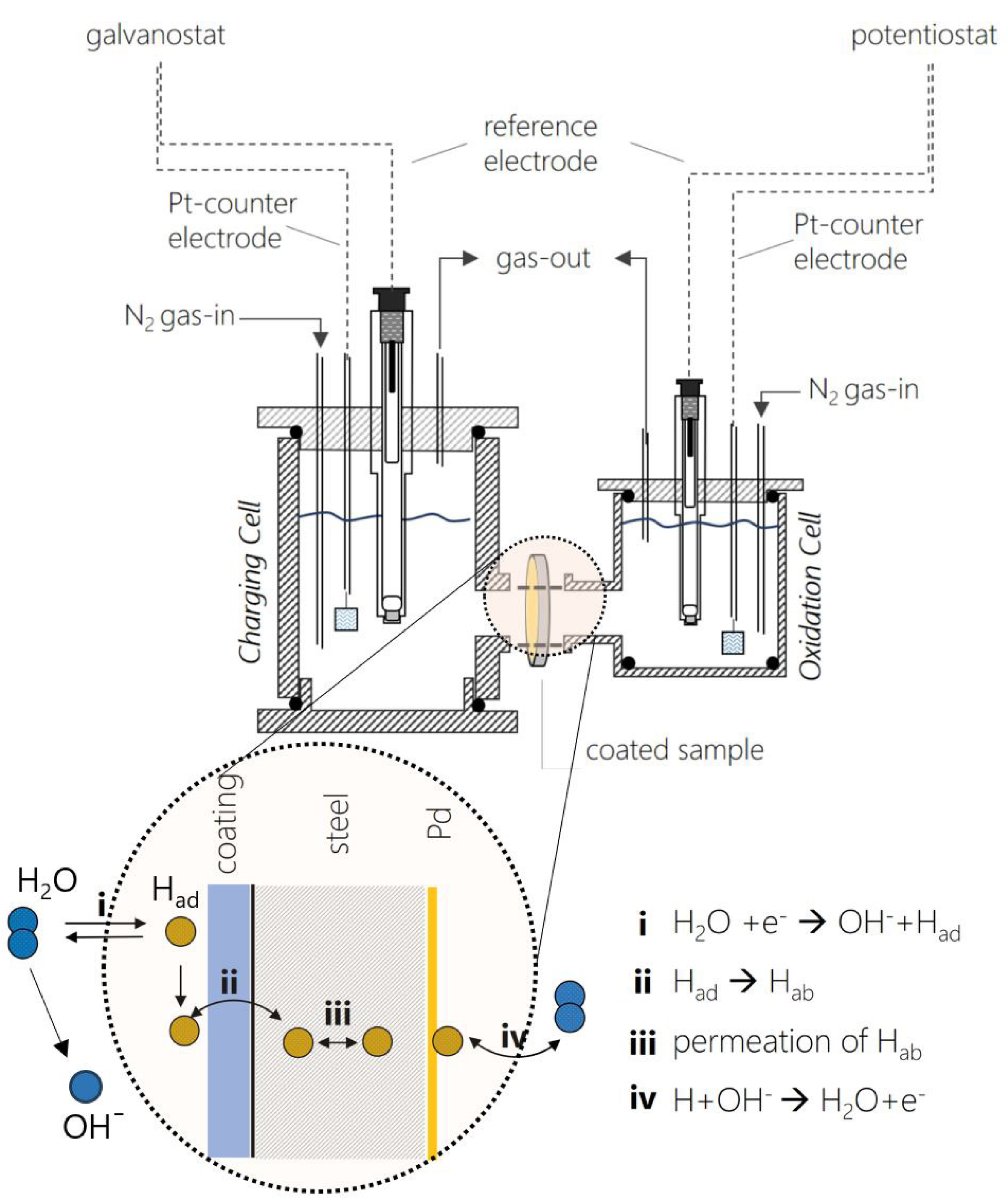
MEASUREMENT SETUP

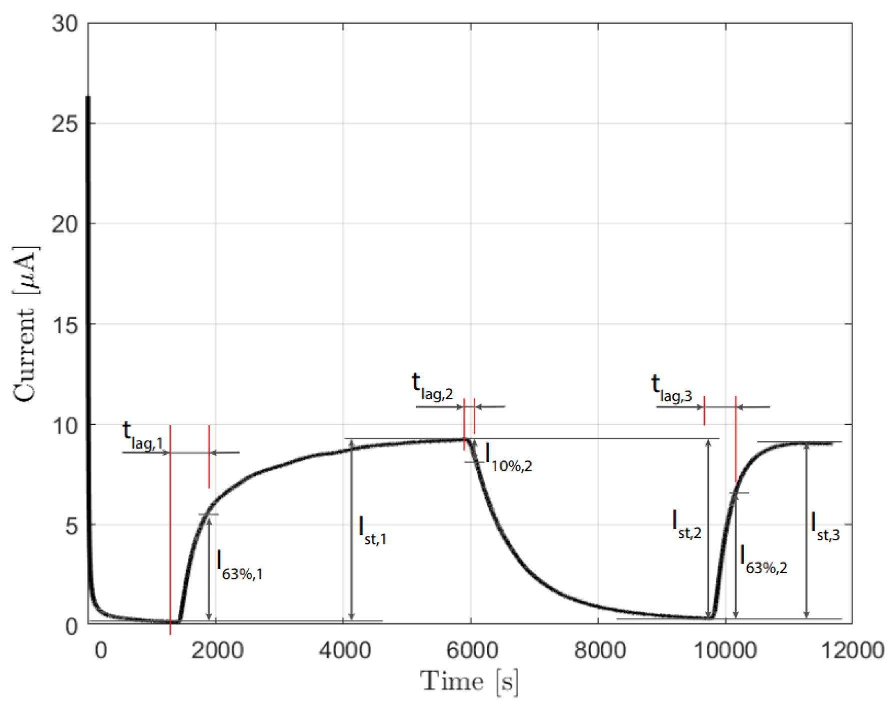
Methodology

- The Devanathan-Stachursky[3] cell consists of two separate compartments, separated by the coated sample
- On the entry side hydrogen is produced galvanostatically by the decomposition of water
- Hydrogen gets adsorbed and atomically permeates through the sample towards the exit side to oxidize
- Generated electrons create a current that can be measured
- Current-over-time data is used for quantification

Limitations

- Inert surface (e.g. Pd) on exit side necessary to hinder recombination





Gained parameters

- Diffusion coefficient, D
- Permeability, φ
- Permeation Reduction Factor, PRF

$$D = \frac{d^2}{Mt} \qquad \frac{L}{D_{eff}} = \frac{l_c}{D_c} + \frac{l_s}{D_s}$$

$$\varphi = \frac{I_p * d}{F * A} \qquad PRF = \frac{J_{substrate}}{J_{compound}}$$

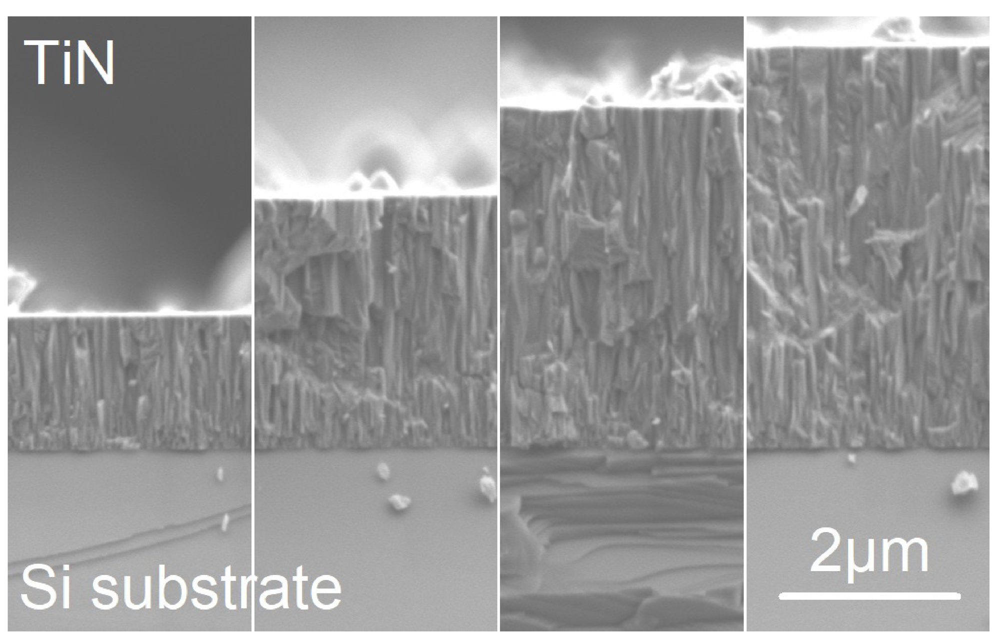
- Time-lag method for calculation of diffusion coefficients[4]
- Max. current for permeability and PRF

[3] ISO 17081, Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique, 2014
[4] S. Frappart et al.: Study of the hydrogen diffusion and segregation into Fe–C–Mo martensitic HSLA steel using electrochemical permeation test, Journal of Physics and Chemistry of Solids, 2010

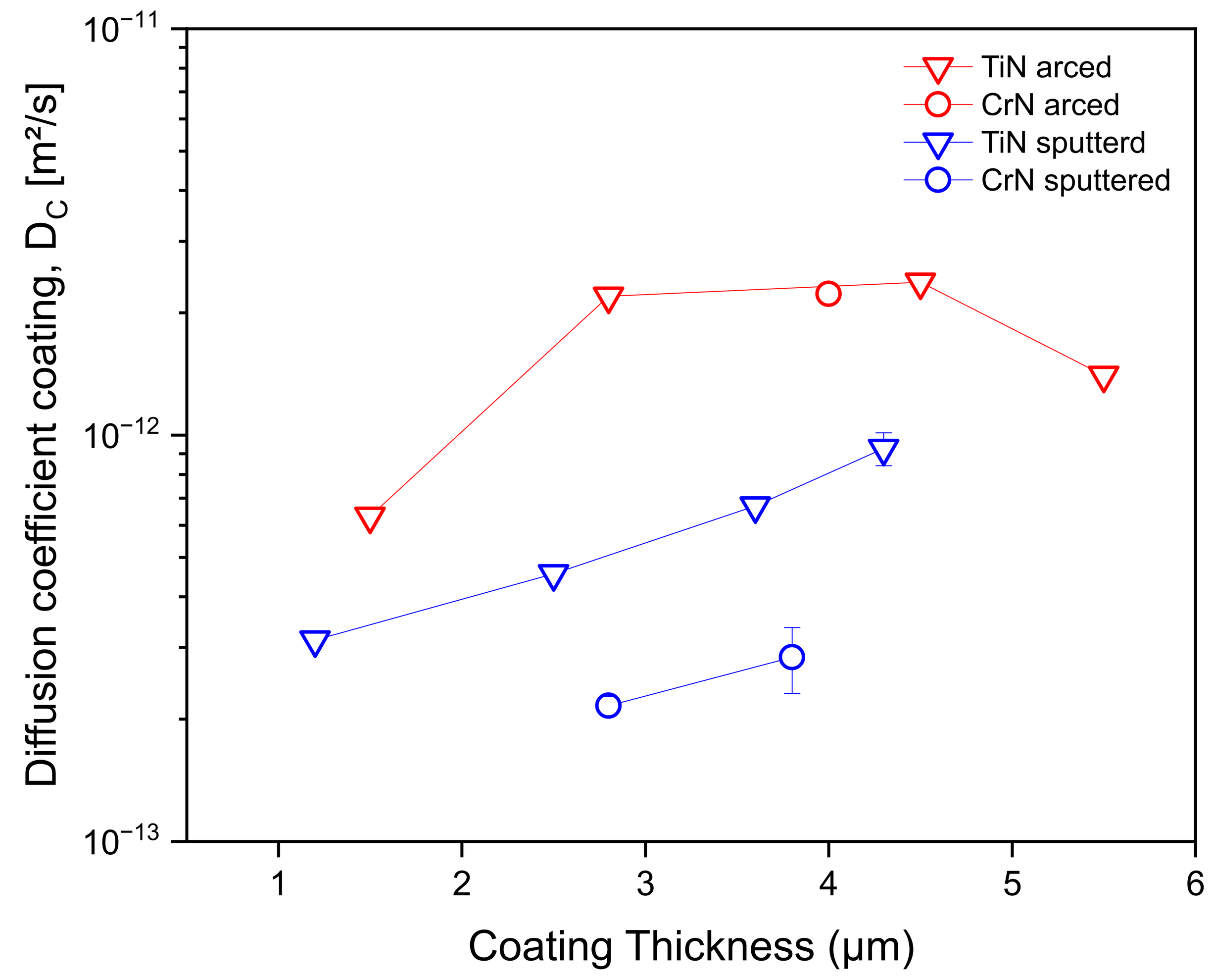
COATING MATERIALS

Comparative study on TiN & CrN

- CAE, Oerlikon Balzers Innova
  - TiN: 1.5 to 5.5 μm
  - CrN: 4.0 μm
- Magnetron Sputtering, Lab scaled deposition systems
  - TiN: 1.2 to 4.5 μm
  - CrN: 2.8 to 3.6 μm

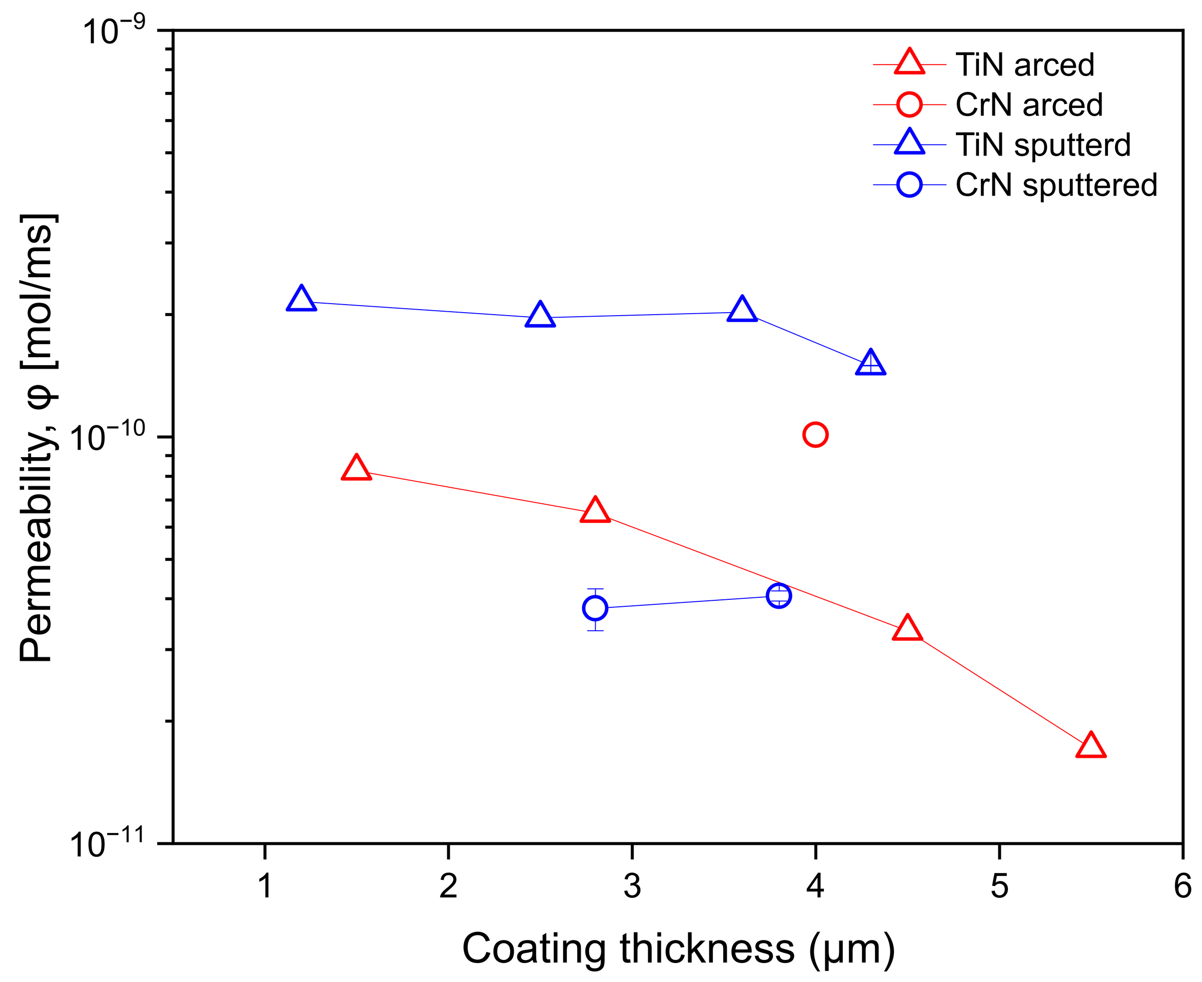


DIFFUSION COEFFICIENT



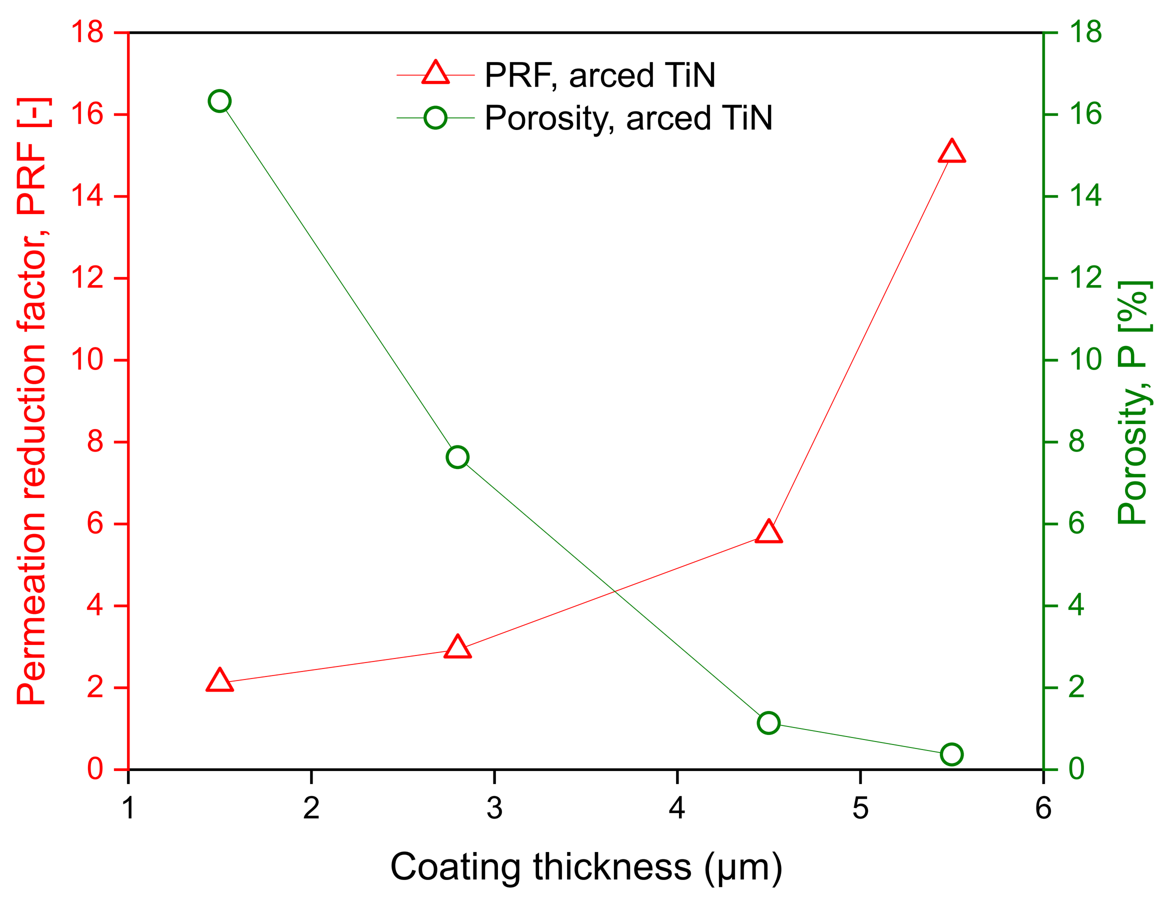
- Variation of coating thickness results in slight influence on diffusion coefficients of sputtered and arced TiN and CrN coatings
- Different morphologies affect hydrogen diffusivity
- Results for 1.0330 steel substrates in good comparison to literature

PERMEABILITY



- TiN (arced and sputtered) shows decreasing hydrogen permeation with increasing thickness, sputtered CrN gives no clear deviation with increasing thickness
- More data points necessary to provide a more detailed insight

OPEN POROSITY ANALYSIS



- Porosity was evaluated via linear sweep voltammetry (LSV) for arced TiN
- There is a clear correlation between decreasing coating porostiy and increasing PRF

CONCLUSION

Electrochemical hydrogen permeation testing is a relatively young and upcoming method concerning hydrogen permeability evaluation of PVD coatings.

- It serves as an **alternative** to the differential pressure method.
- It is highly sensitive and has **direct measures** for hydrogen transportation
- There is a **difference** in hydrogen barrier properties between **arced and sputtered** coatings of comparable thicknesses, that needs to be investigated further
- LSV experiments suggest, that porosities act as pathways for hydrogen diffusion and higher coating density significantly **hinders hydrogen** permeation

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PLANSEE

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