Welding problems – investigations on hydrogen diffusion in steels by neutron radiography

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[2]UVa – Condensed Matter Physics Dept.

Outline

- Motivation
  technical safety, failure analysis
- Neutron radiography
  direct method, visualization
- Results
  $c_H(t)$ profiles
- Conclusion & Outlook
  combinated in-situ diagnostics

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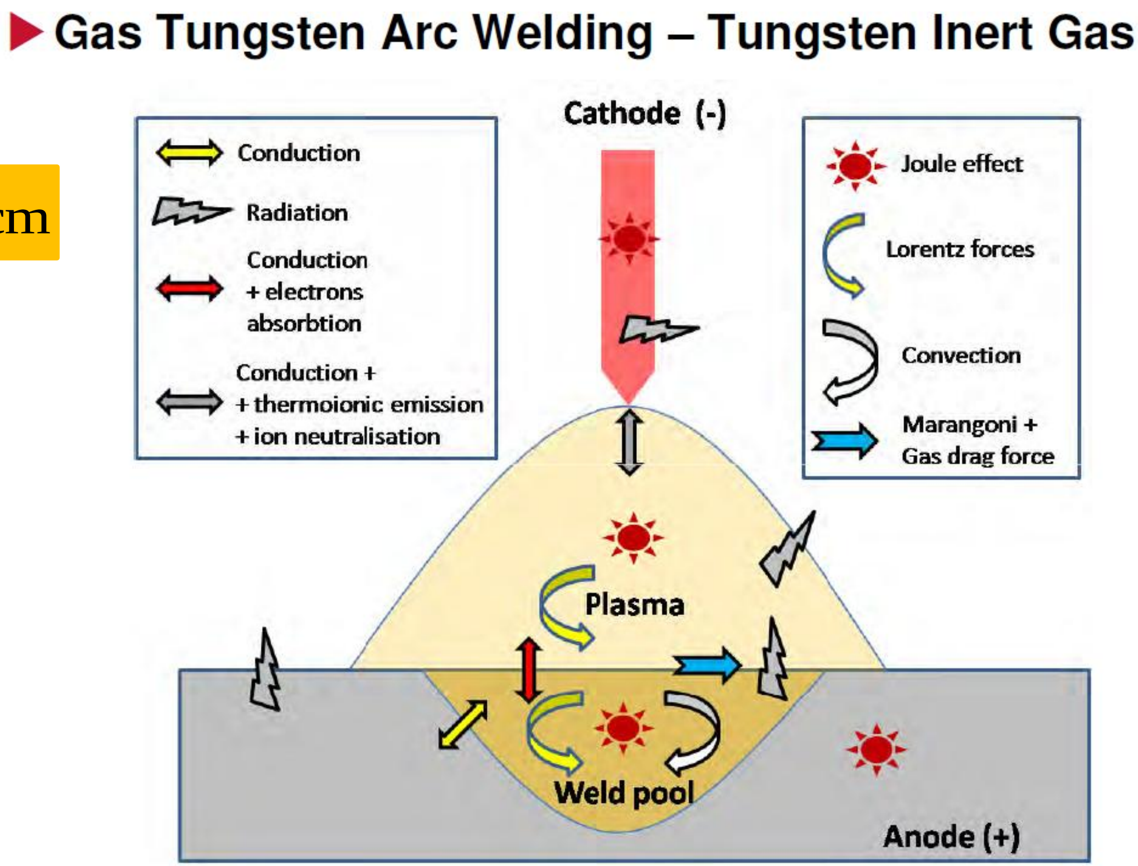
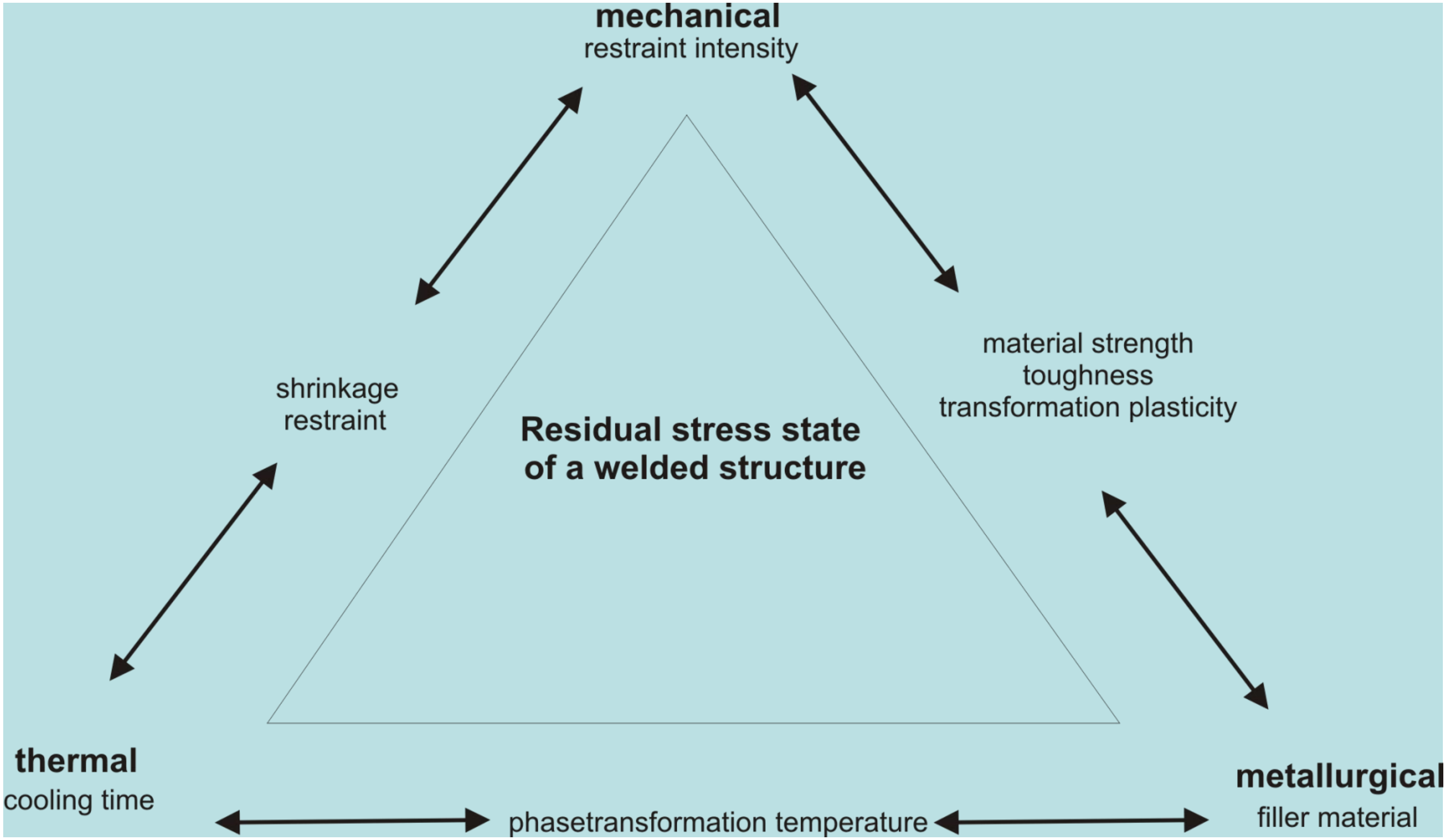
Hydrogen uptake

$\nabla T \sim 10^3$ K/cm

$\dot{T} \sim 10^5$ K/s

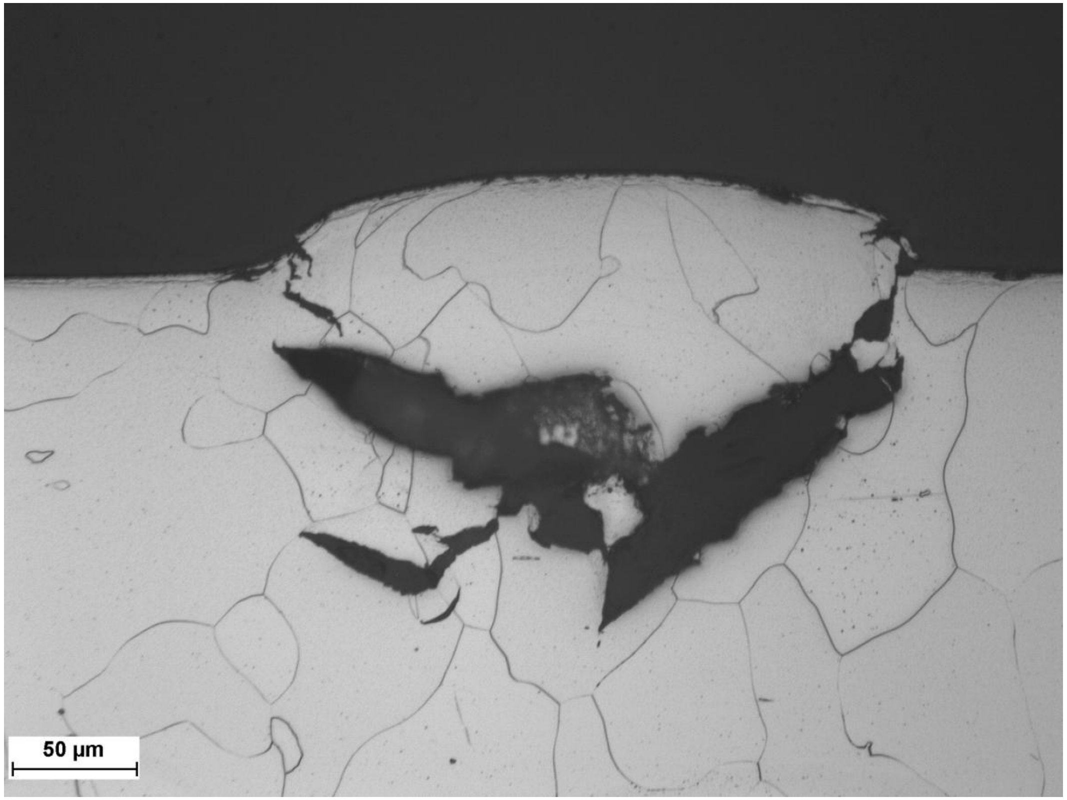


Welding Problems



Post weld treatments do not help always !

Hydrogen-assisted cracking



LM image of blistering in ARMCO (technical iron)

Information necessary to understand damage mechanism

- triaxial residual stress state of the bulk material
- surface stress state (crack propagation)
- effect of stress & strain on hydrogen diffusion
- diffusion mechanism of hydrogen
- stress-induced phase transformation
- hydrogen-induced phase transformation

Some considerations with respect to NR

- more information accessible?

  concentration profile below surface, hydrogen distribution in microstructure etc.

- easy and well defined neutron experiments possible ?

  key experiments, full use of detector’s spatial and temporal resolution and its dynamic range possible

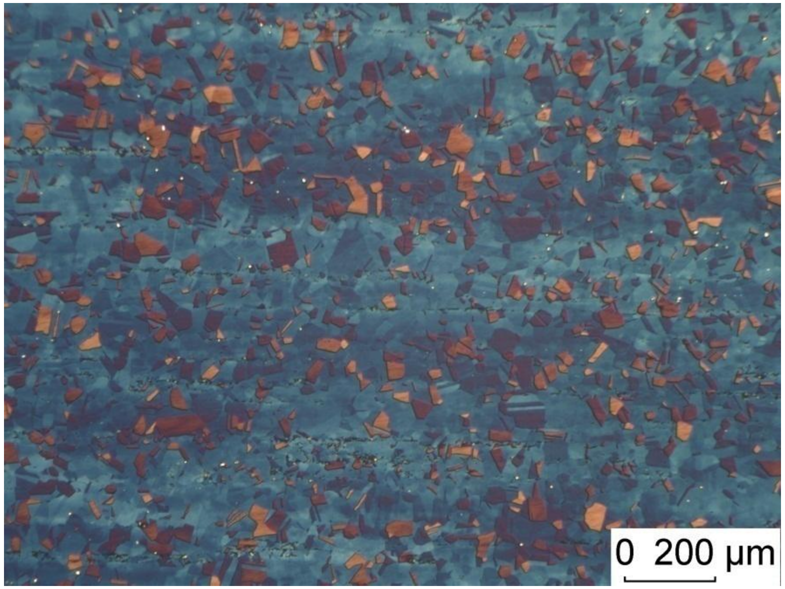
- in-situ analysis of hydrogen transport during welding ?

  additionally to XRD@synchrotron

Experimental Part

Austenitic Stainless Steel (1.4301)

Fe	C	Cr	Ni	Mn	Si	N
balance / wt%	**0.07**	**18.3**	**9.3**	**1.9**	**0.09**	**0.01**



LM image of microstructure

- H reduces tensile stress[1]

- H induces phase transformations (to ε and/or α‘ martensite)[2]

Mechanisms?

Role of H diffusion?

***D*???**

[1] G. Schuster, C.J. Altstatter, Met. Trans 14A (1983) 2085

[2] N. Narita et al., Met. Trans. 13A (1982) 1355

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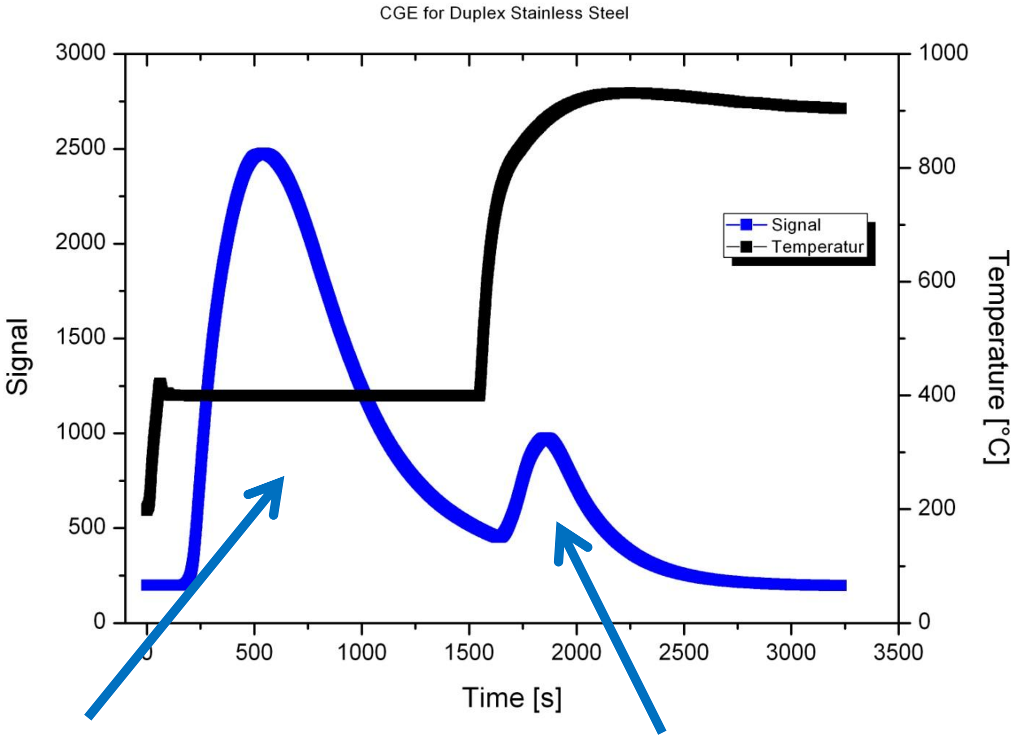
Carrier gas hot extraction



low energy traps

austenite/precipitations

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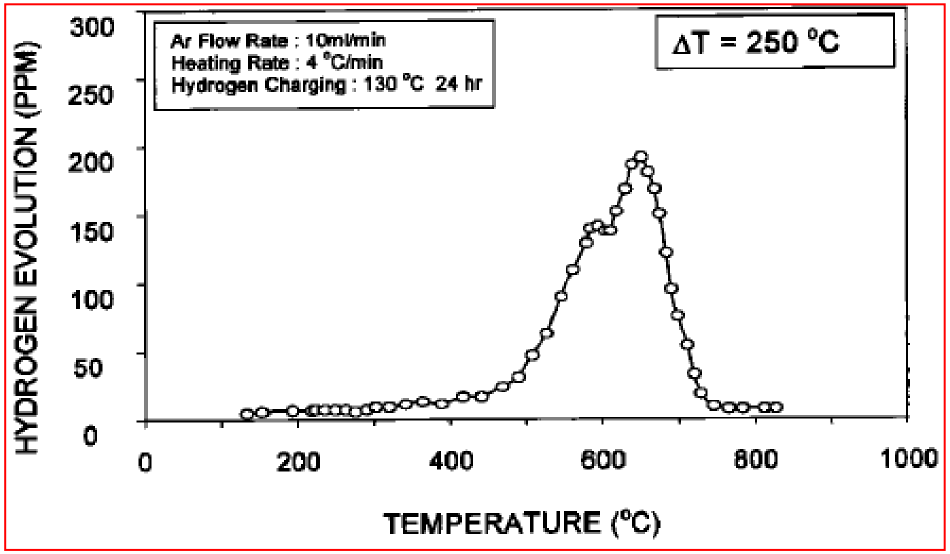
Carrier gas hot extraction II

- quantitative gas concentration analysis
- Kissinger analysis ⇒ activation energy of “traps”



dual-phase steel: retained austenite, ferrite, martensite

Traps:

- strain regions around dislocations (20kJ/mol)
- grain boundaries (22.5-28.5kJ/mol)
- dislocations (34.8–40.3 kJ/mol)
- vacancies / austenite–ferrite interfaces (50.2–57.4 kJ/mol)

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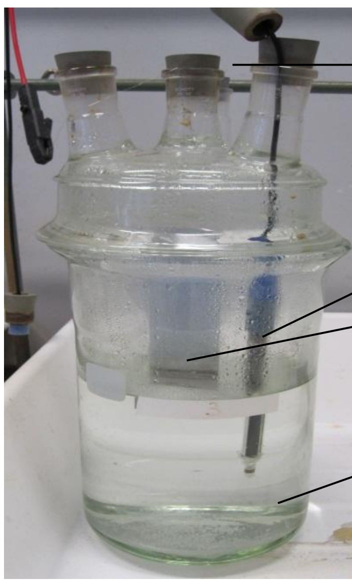
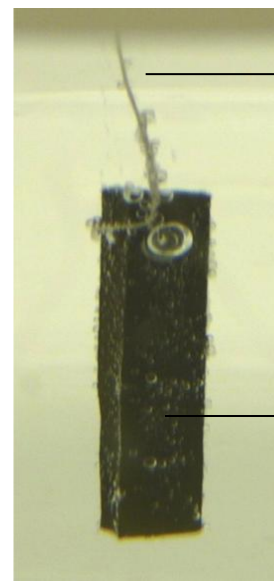
Park et al., Welding J (2002)27
Dabah et al., MSE A 527(2010)4851

Hydrogen charging

- **Cathodic hydrogen charging**
  - **I_A = 10 mA/cm^2 for 48 h**
  - **Electrolyte: 0.05 M H_2SO_4 + 0.1 M $NaAsO_2$ (ratio 1:500)**



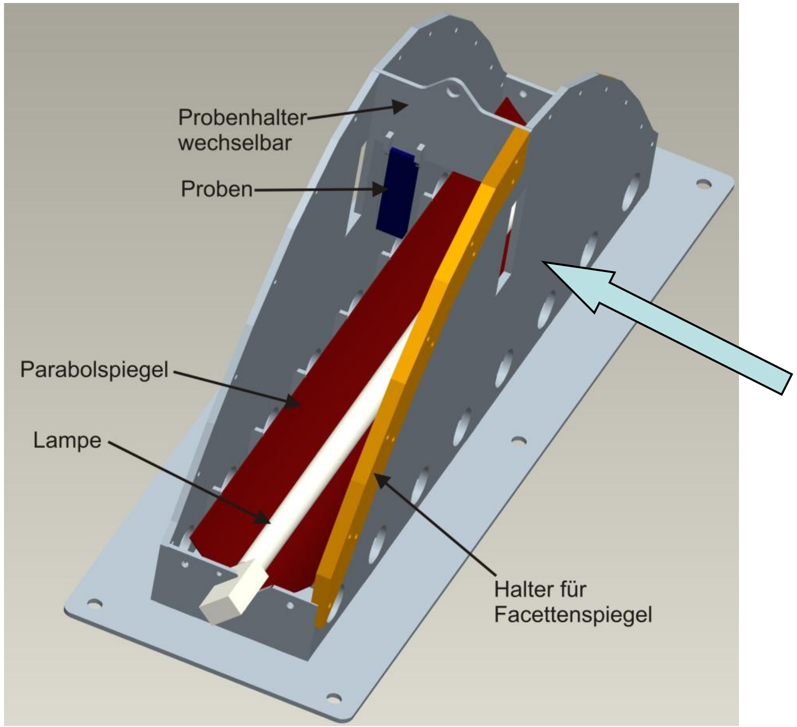


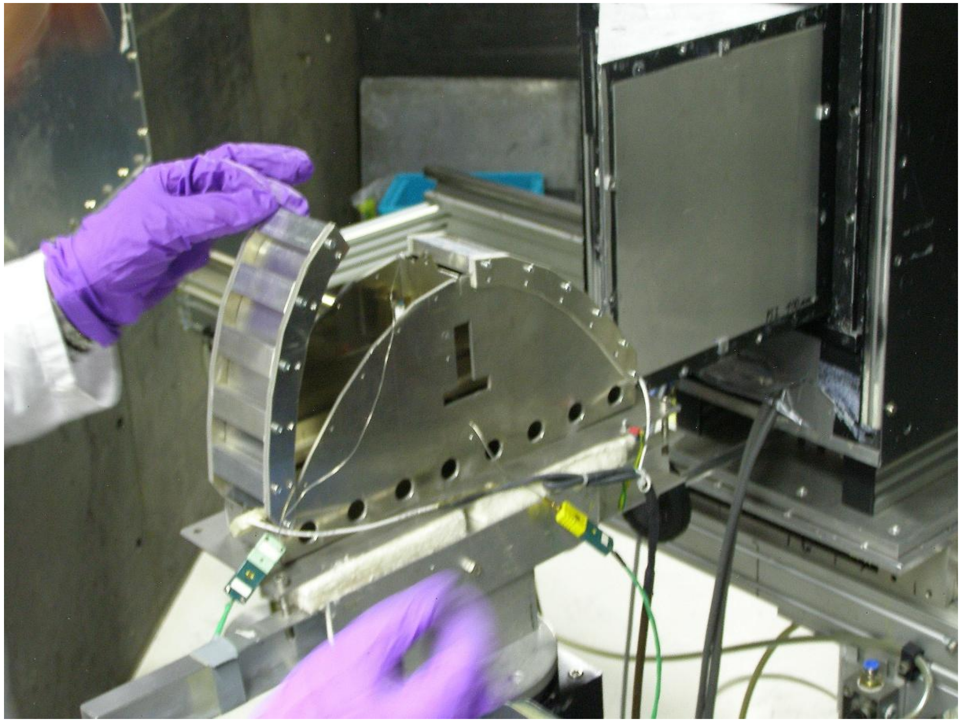
Neutron radiography@ANTARES (FRM II)

+ total scattering cross section: H^1 = **82,02** barn Fe = **11,62** barn

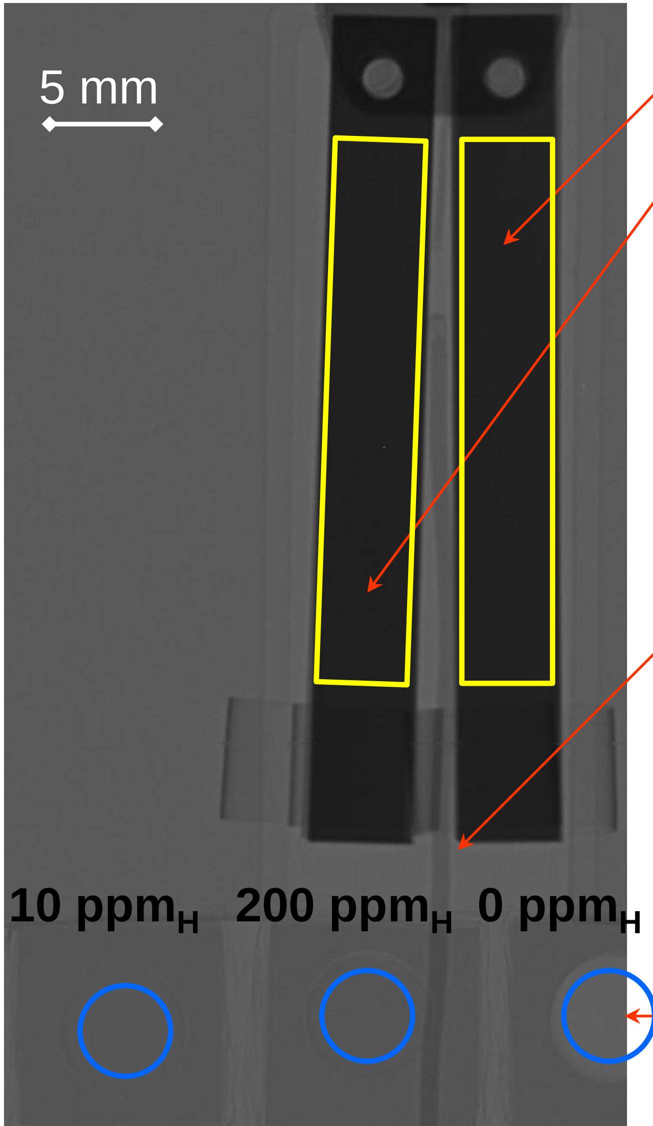
+ **$I=I_0\exp(-\mu d)$**

+ $\Delta x < 100\mu m$, $\Delta t = 20$ s, 16-bit gray value resolution





Results



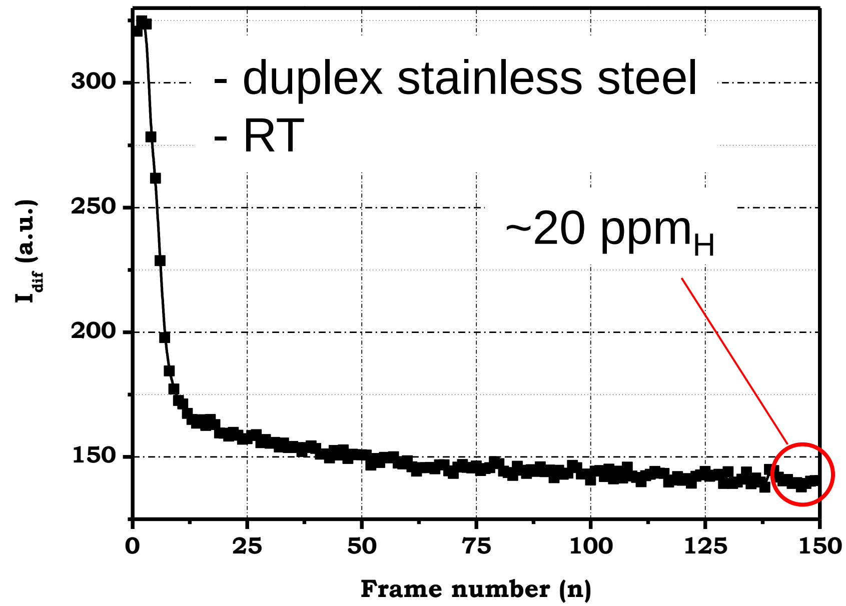
reference

sample

TC

calibration standards (TiH_2 + SiC)

→ standard image corrections
→ take mean values (yellow)
→ normalize with standards (blue)



Analysis

- conversion of $\Delta I(t)$ into $C_H(t)$ for all images
- fitting solution of Fick's equation (surface evaporation conditions) to isothermal data

assumptions

gross transport is a single diffusion mechanism

diffusion-controlled transport

$C_{H,\ atmosphere} = 0$

$C_{H,bulk} = C_{H,surface}$

$\partial M / \partial t \propto (C_{H,surface} - C_{H,\ atmosphere})$

$D(C_H) = \text{const.}$

$$M_H = \left(\frac{C_0 - C_1}{h}\right)\left\{\exp(h^2 Dt)\, erfc(h\sqrt{Dt}) - 1 + \frac{2}{\sqrt{\pi}} h\sqrt{Dt}\right\} \qquad h = \alpha / D$$

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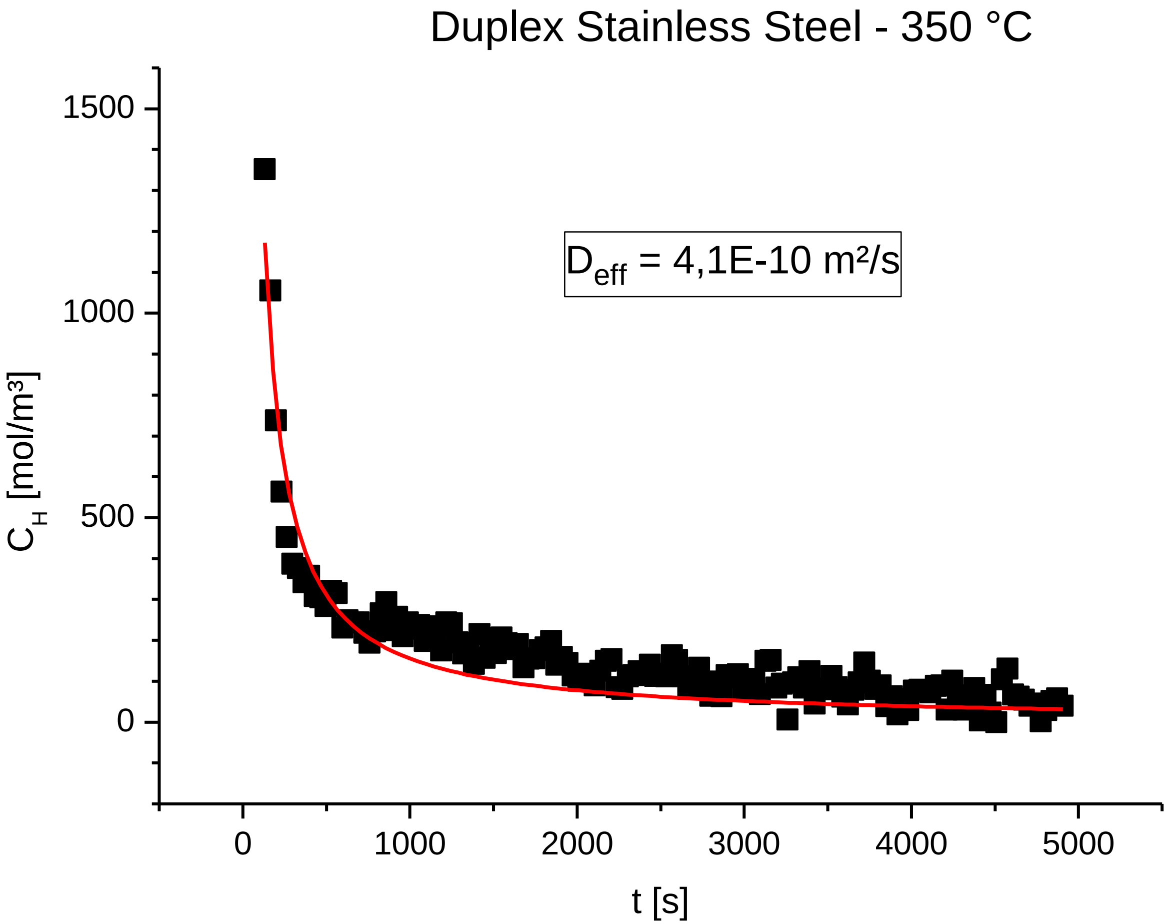
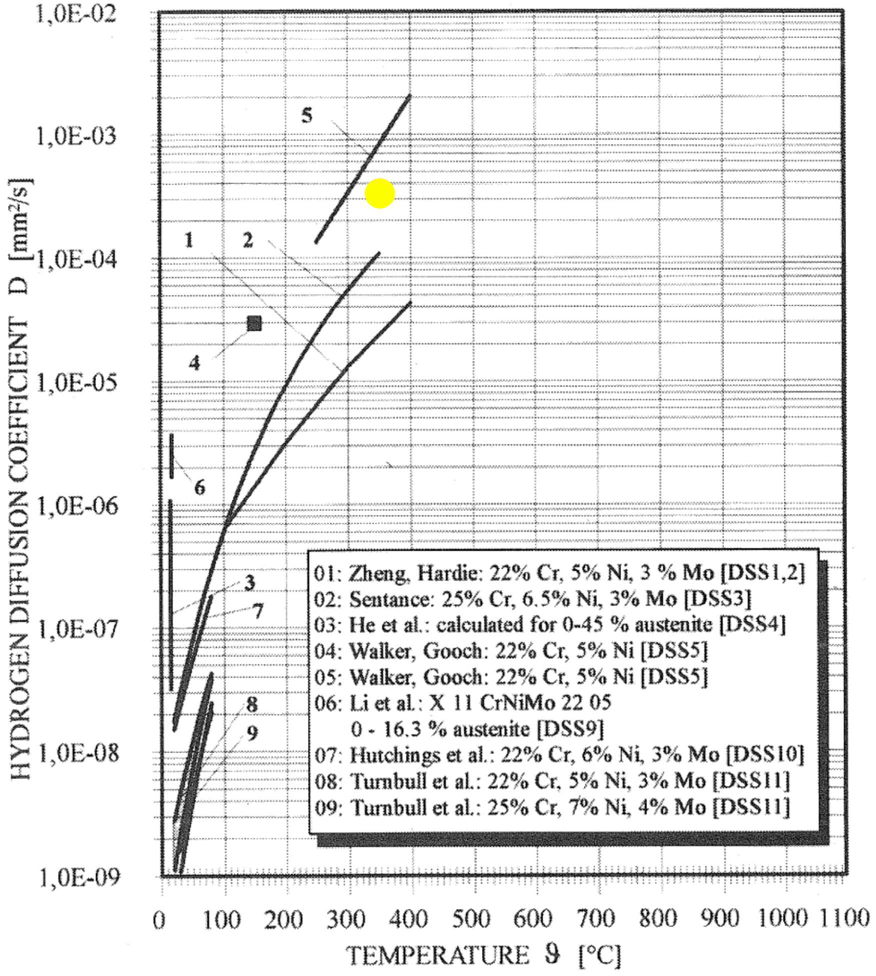
Crank, The Mathematics of Diffusion, Oxford 1975

Concentration profiles



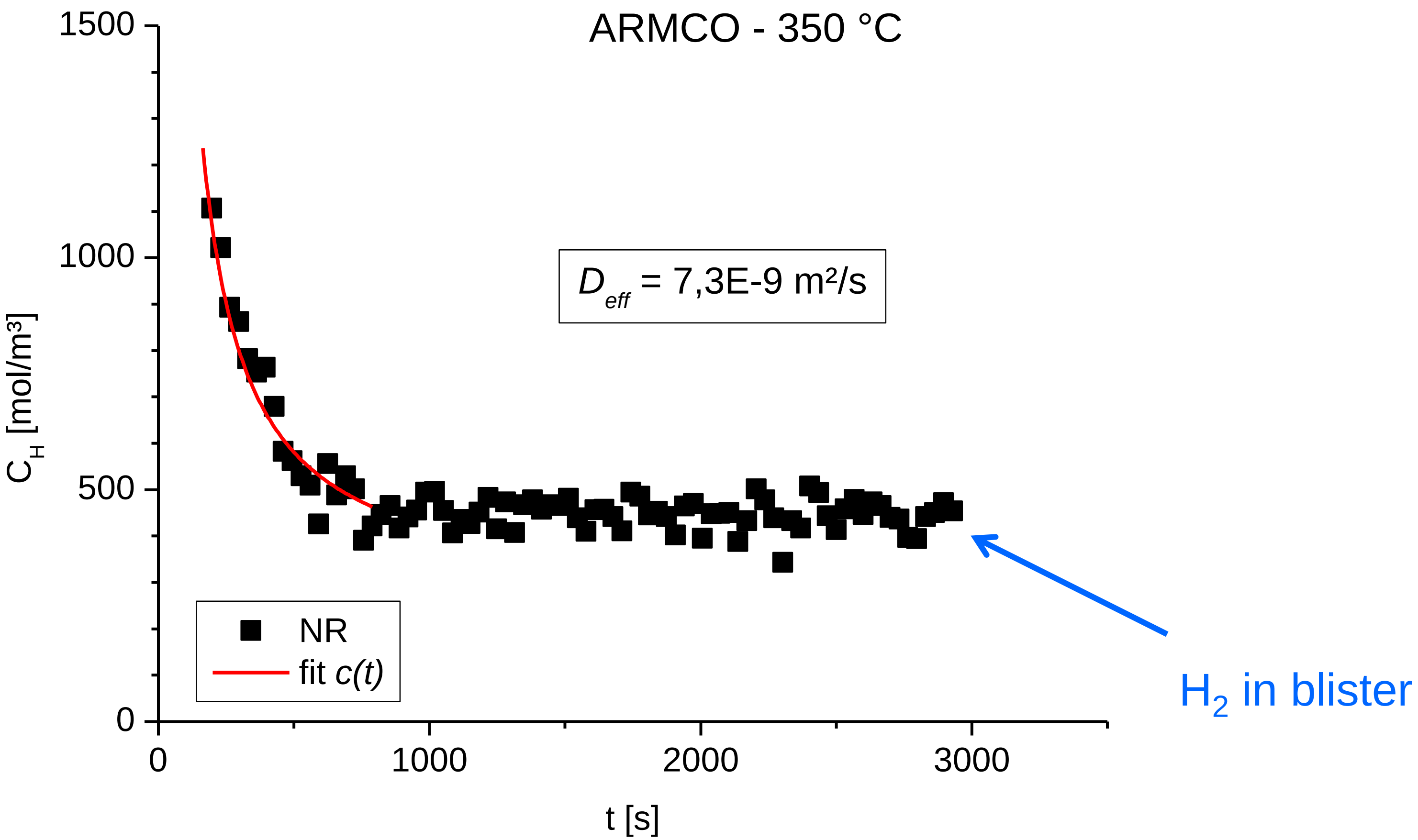
Diffusion coefficients in DSS



NR: D_{eff} = 4,1E-10 m²/s

Böllinghaus et al. WiW37(1996)16

Concentration profiles

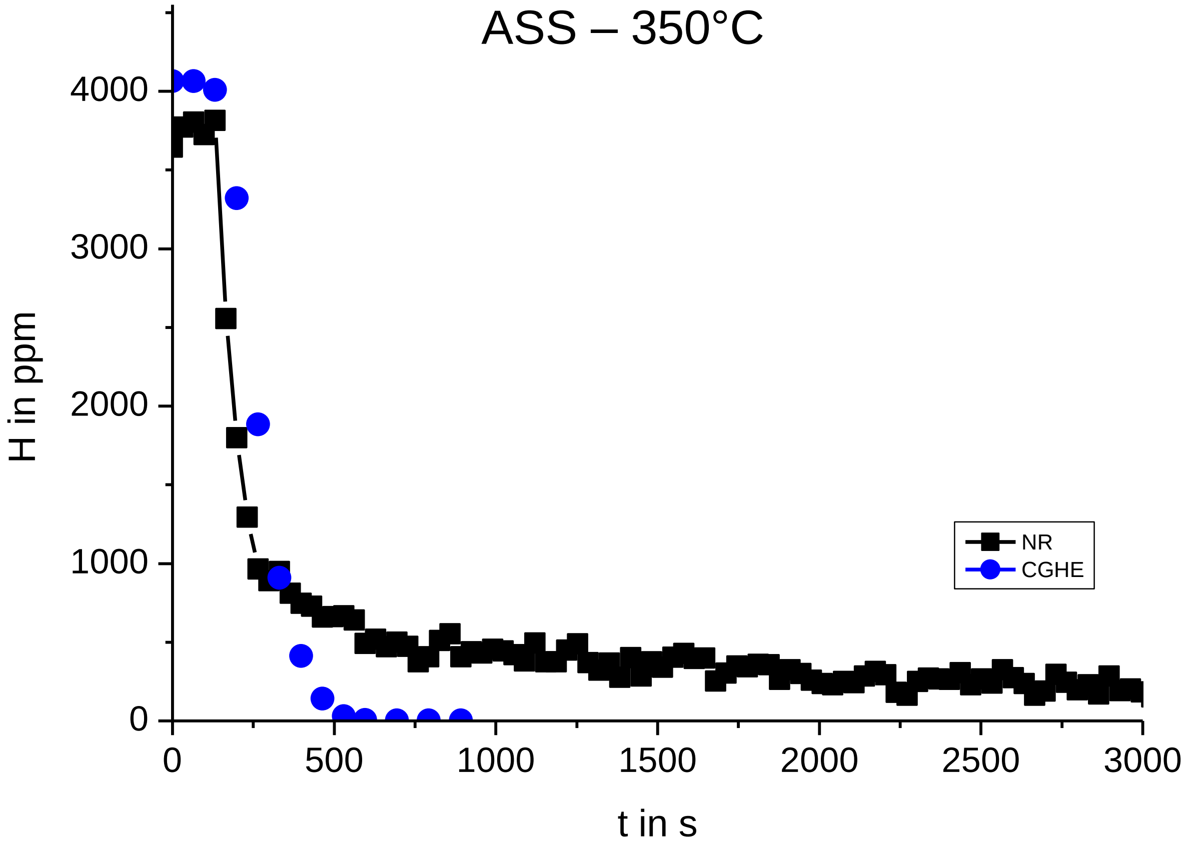


• good agreement with permeation experiments*: D_{eff} = (5E-8 - 5E-9) m²/s

Hydrogen Blister

LM image of blistering in ARMCO (technical iron)

Concentration profiles



• good agreement between NR and CGHE

Conclusion

- In-situ measurement of hydrogen effusion in steels using neutron radiography possible
- Determination of effective diffusion coefficients
- Direct measurement / visualization
- Accurate measurement of $C_{hydrogen} < 20\ ppm_H$
- Development of calibration standards

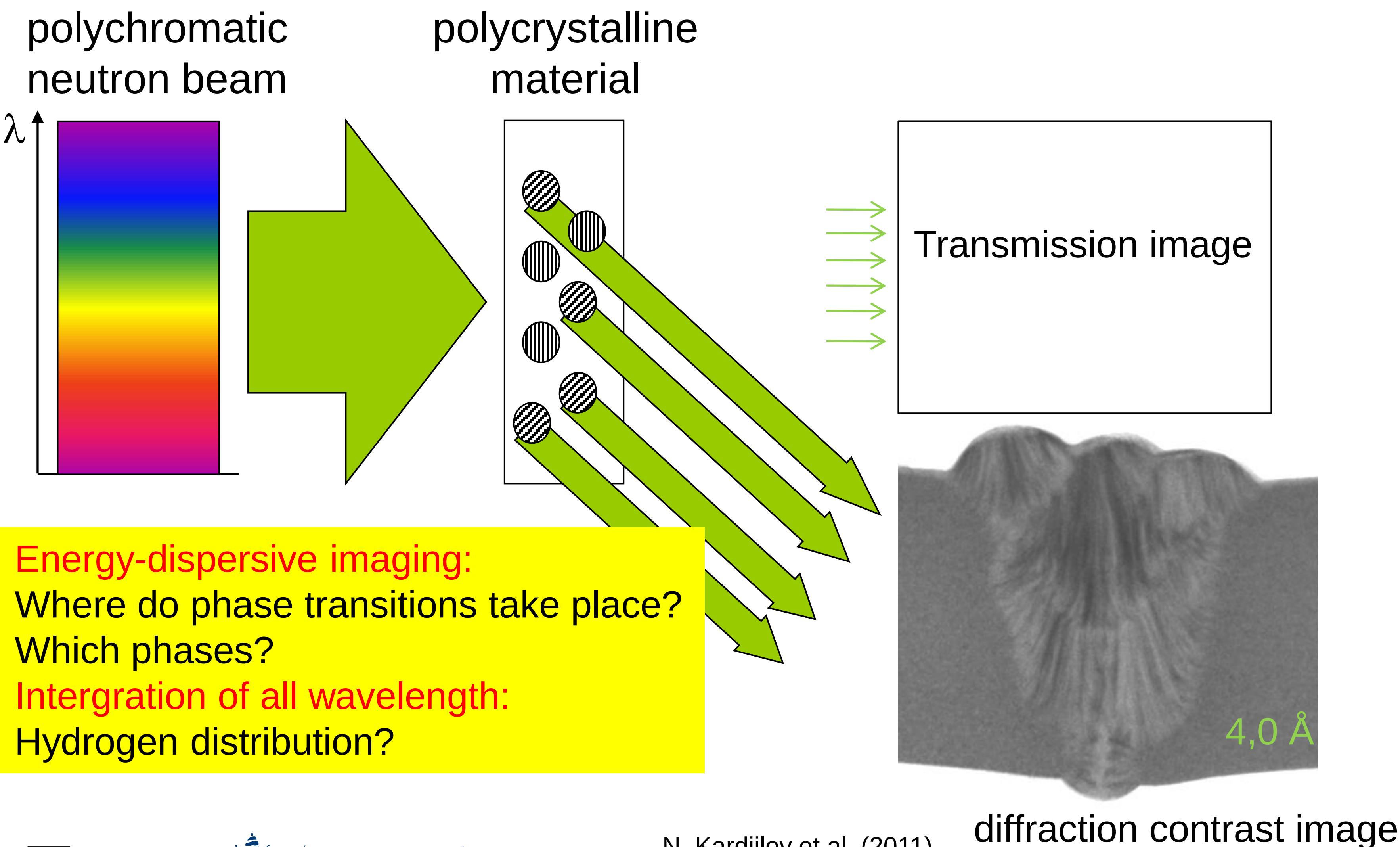
Outlook

- Simultaneous mapping (with good spatial and temporal resolution) during welding of

1) triaxial residual stresses ε in the weld material
2) 3D-distribution of T & C_H
3) mapping of all phases (equilibrium and non-equilibrium)
4) lattice deformation (due to H, thermal treatment, external forces)

- Δx < 1µm, Δt < 1s, ΔC_H < 1ppm

Bragg-Edge- + Transmission Radiography



Energy-dispersive imaging:
Where do phase transitions take place?
Which phases?
Intergration of all wavelength:
Hydrogen distribution?

N. Kardjilov et al. (2011)

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Wish List

- Tensile/compression/bending apparatus with heating option and temperature diagnostics
- Sample manipulator with tilting/rotation during residual stress measurement
- High sample through-put
- Complementary analytic methods on-site: XRD, µ-focus x-ray tube
- Express-line for industry

Acknowledgement:

- Burkhard Schillinger – ANTARES team
- Andreas Hannemann – BAM 9.4

Literature:

- Beyer et al., Nuc Instrum Meth A, DOI:10.1016/j.nima.2011.02.010
- Beyer et al., J Mat Sci 46(2011)5171, DOI:10.1007/s10853-011-5450-7
- Kargl, Griesche et al., *In-situ studies of mass transport in liquid alloys by means of neutron radiography*, J. Phys Condens Matter 23 (2011) 254201

Thank you for your attention!