AGH UNIVERSITY OF SCIENCE
AND TECHNOLOGY

Rozpraszanie neutronów w badaniach wodorków i rezerwuarów na wodór

Łukasz Gondek

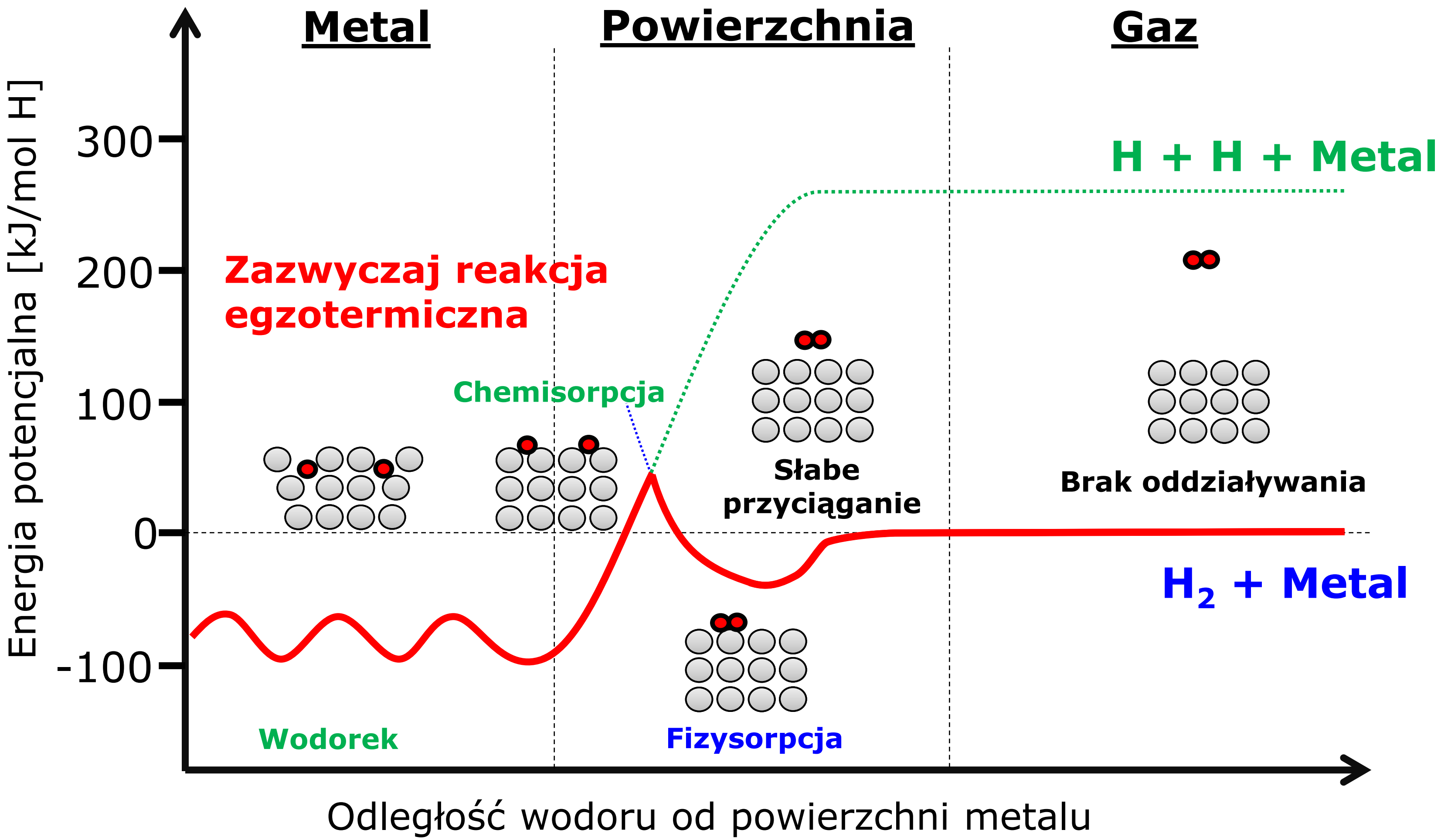
WFiIS, AGH

AGH

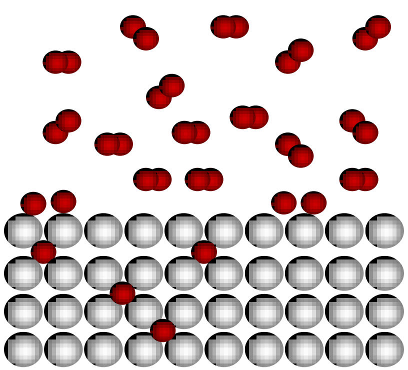
Plan referatu

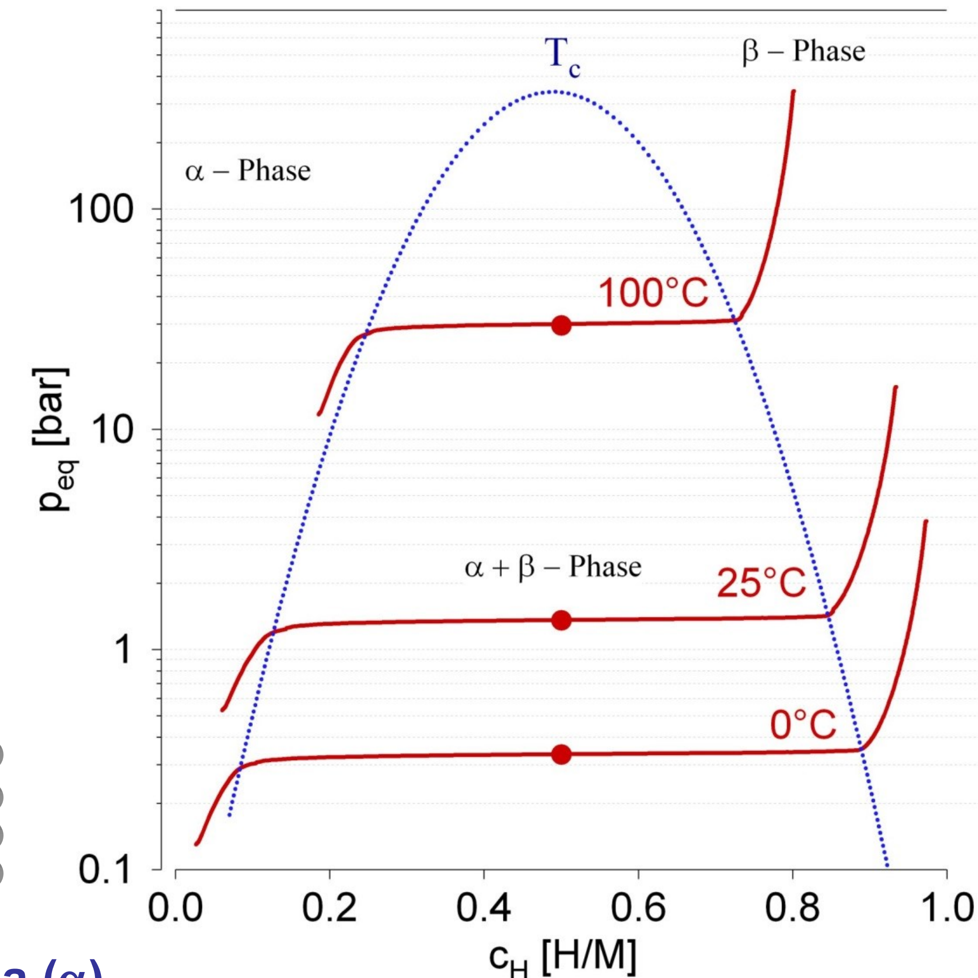
- Słów kilka o preferencjach strukturalnych dla wodoru.
- Przykłady badań strukturalnych z wykorzystaniem neutronów (RTX, RT_5).
- Magnetyzm wodorków (RPdIn).
- Przemiany fazowe w stopach Ti-Zr-Ni wyzwalane wodorem (szkliste kwazikryształy).
- Magazynowanie wodoru, czyli „neutron TV”.
- Perspektywy…

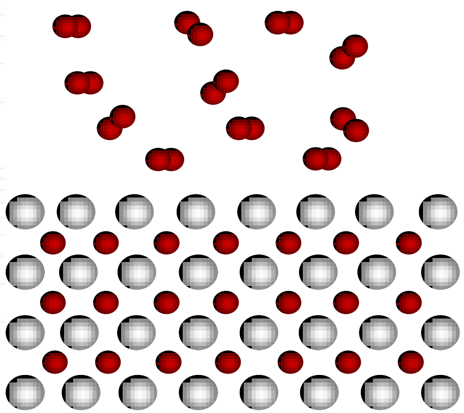
Słów kilka o preferencjach…



Słów kilka o preferencjach…



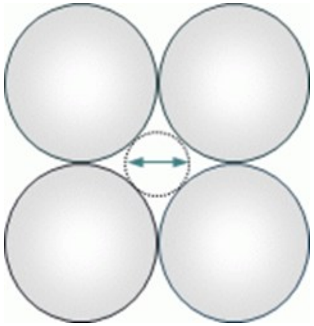




Faza niskonasycona (α)

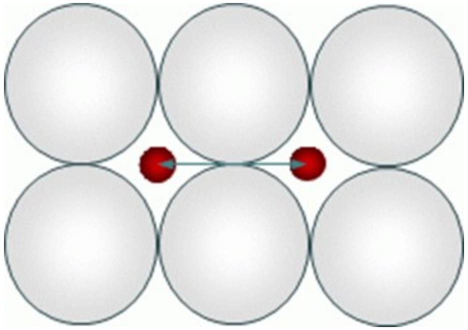
Faza wodorku (β)

Słów kilka o preferencjach…



Kryterium Westlake'a:

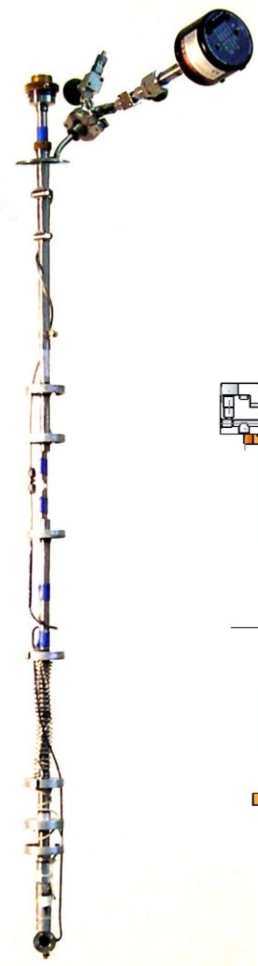
Wodór może obsadzać pozycje, gdzie zmieści się kulka o promieniu 0.37 Å (kowalencyjny promień atomu wodoru)

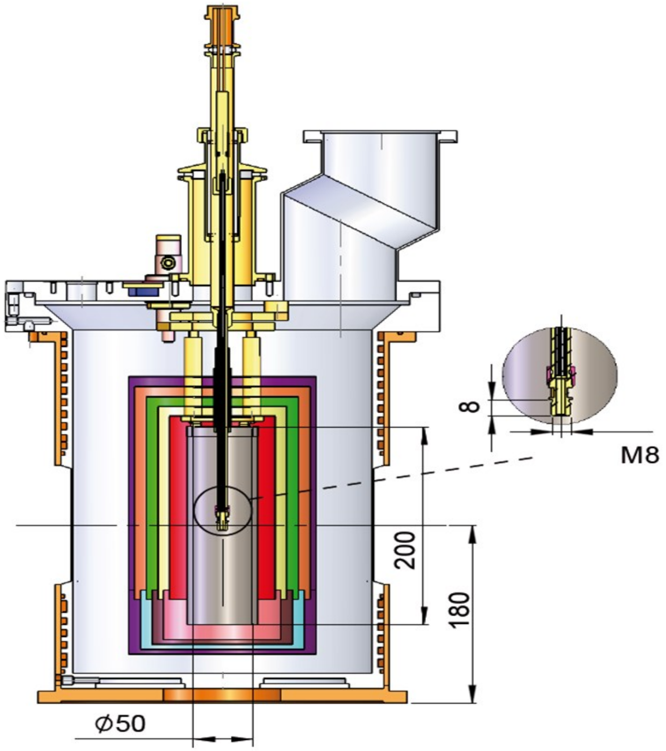


Kryterium Switendicka:

W strukturze krystalicznej dwa atomy wodoru nie zbliżają się do siebie poniżej 2.1 Å.

Badania z wykorzystaniem neutronów



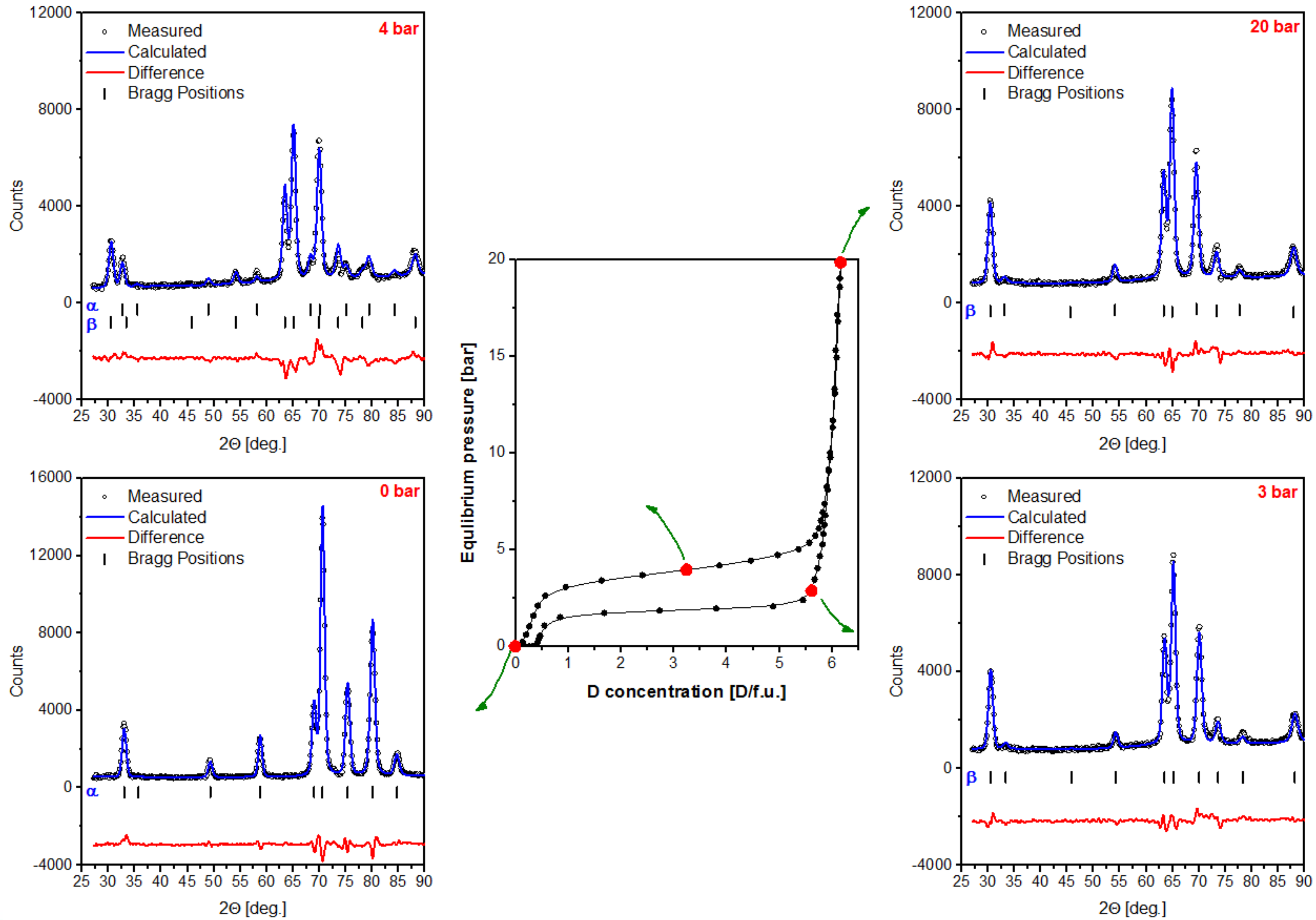


Badania in-situ:

Ciśnienia: 0-80 bar H_2 lub D_2
Temperatury: 1 – 1000 K

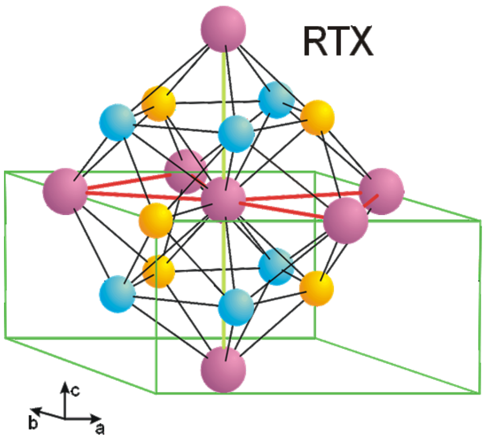
Możliwość pomiarów przy użyciu większości dostępnych technik.

Badania strukturalne dla pochodnych $LaNi_5$

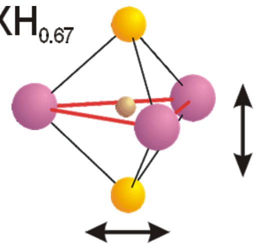


Badania strukturalne dla związków RPdIn

RTX



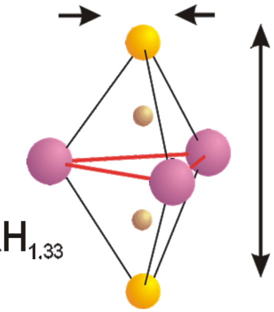
$RTXH_{0.67}$



PHYSICAL REVIEW B 67, 014101 (2003)

Short hydrogen-hydrogen separation in $R\mathrm{NiInH}_{1.333}$ (R = La, Ce, Nd)

P. Vajeeston,[1,*] P. Ravindran,[1] R. Vidya,[1] A. Kjekshus,[1] H. Fjellvåg,[1,2] and V. A. Yartys[2]
[1]*Department of Chemistry, University of Oslo, Box 1033, Blindern, N-0315, Oslo, Norway*
[2]*Institute for Energy Technology, N-2027 Kjeller, Norway*
(Received 2 July 2002; revised manuscript received 19 September 2002; published 15 January 2003)



$RTXH_{1.33}$

PHYSICAL REVIEW B 70, 014107 (2004)

Search for metal hydrides with short hydrogen–hydrogen separation: *Ab initio* calculations

P. Vajeeston,* P. Ravindran, H. Fjellvåg, and A. Kjekshus
Department of Chemistry, University of Oslo, Box 1033 Blindern, N-0315 Oslo, Norway
(Received 7 August 2003; revised manuscript received 24 March 2004; published 21 July 2004)

Badania strukturalne dla związków RPdIn

Acta Materialia 81 (2014) 161–172

Do the RPdIn (R = rare earth) deuterides break the Switendick rule?

Ł. Gondek [a,*], K. Koźlak [a], J. Czub [a], J. Przewoźnik [a], A. Kupczak [a], W. Sikora [a], A. Hoser [b], O. Prokhnenko [b], N. Tsapatsaris [b]

	a (Å)	$\Delta a/a$ (%)	c (Å)	$\Delta c/c$ (%)
LaPdIn	7.7379(2)		4.1470(4)	
$LaPdInD_{1.2}$	7.7583(4)	0.26	4.2700(2)	2.97
CePdIn	7.7024(2)		4.0735(2)	
$CePdInD_{1.1}$	7.7076(1)	0.07	4.2172(1)	3.53
PrPdIn	7.6894(2)		4.0409(4)	
$PrPdInD_{1.0}$	7.6208(4)	−0.89	4.1818(1)	3.49
NdPdIn	7.6830(3)		3.9996(2)	
$NdPdInD_{1.2}$	7.6552(2)	−0.36	4.1612(1)	4.04
HoPdIn	7.6404(5)		3.7857(4)	
$HoPdInD_{1.2}$	7.5034(9)	−1.79	3.9962(9)	5.56
ErPdIn	7.6377(3)		3.7576(3)	
$ErPdInD_{1.2}$	7.5615(2)	−1.00	3.9229(2)	4.40

Badania strukturalne dla związków RPdIn

	x_R
LaPdIn	0.5861(8)
$LaPdInD_{1.2}$	0.5949(7)
CePdIn	0.5793(7)
$CePdInD_{1.1}$	0.5831(6)
PrPdIn	0.5845(6)
$PrPdInD_{1.0}$	0.5891(7)
NdPdIn	0.5882(7)
$NdPdInD_{1.2}$	0.6018(7)
HoPdIn	0.5897(7)
$HoPdInD_{1.2}$	0.5978(9)
ErPdIn	0.5942(7)
$ErPdInD_{1.2}$	0.6002(7)

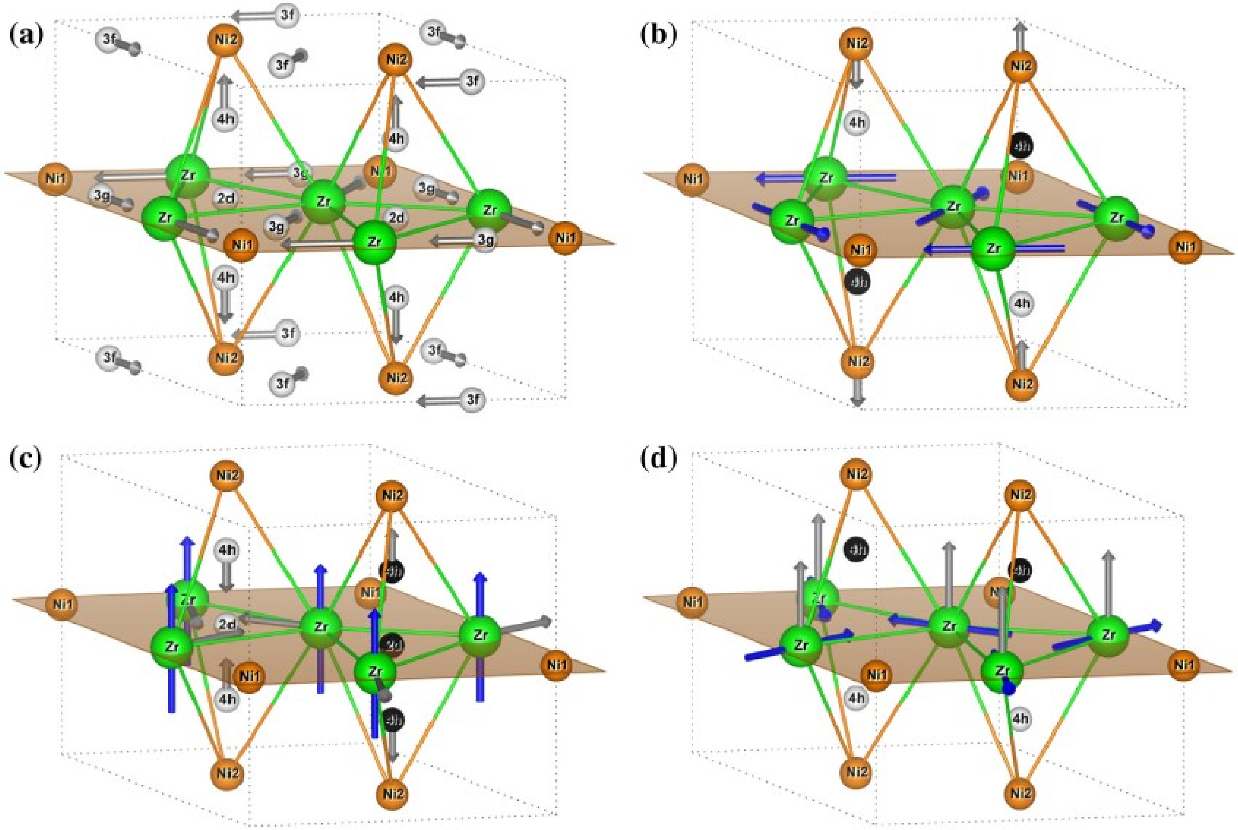
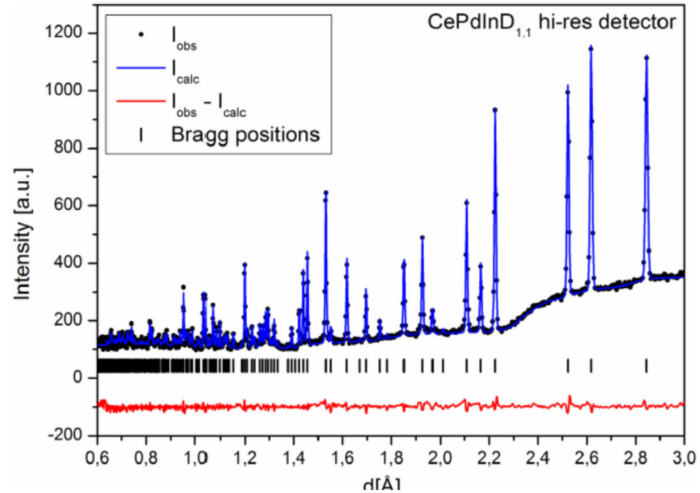


Fig. 1. The possible positions of hydrogen atoms within the ZrNiAl-type structure with the related distortions and the magnetic moment orientations derived from the symmetry analysis. The subsequent diagrams show the results for the different representations: τ_1 (a), τ_2 (b), τ_3 (c) and τ_4 (d).

Badania strukturalne dla związków RPdIn



The structural parameters of the RPdIn deuterides derived from the TOF neutron scattering at room temperature. The space group is P-62 m (No. 189). As well as the lattice parameters (a, c), the deuterium occupied sites are listed.

	a (Å)	c (Å)	Site	Positional parameter	Site occupancy (%)
$LaPdInD_{1.2}$	7.75828(6)	4.26997(4)	4h	$z_D = 0.0685(2)$	50
			3g	$x_D = 0.8045(2)$	41
			3f	$x_D = 0.2881(2)$	12
$CePdInD_{1.1}$	7.70763(5)	4.21717(3)	4h	$z_D = 0.0671(2)$	50
			3g	$x_D = 0.8041(2)$	35
			3f	$x_D = 0.2878(2)$	8
$NdPdInD_{1.2}$	7.65528(5)	4.16121(3)	4h	$z_D = 0.0655(2)$	45
			3g	$x_D = 0.8039(2)$	42
			3f	$x_D = 0.2872(2)$	17
$ErPdInD_{1.2}$	7.56148(7)	3.92304(4)	4h	$z_D = 0.0646(2)$	47
			3g	$x_D = 0.8052(2)$	40
			3f	$x_D = 0.2875(2)$	15

Badania strukturalne dla związków RPdIn

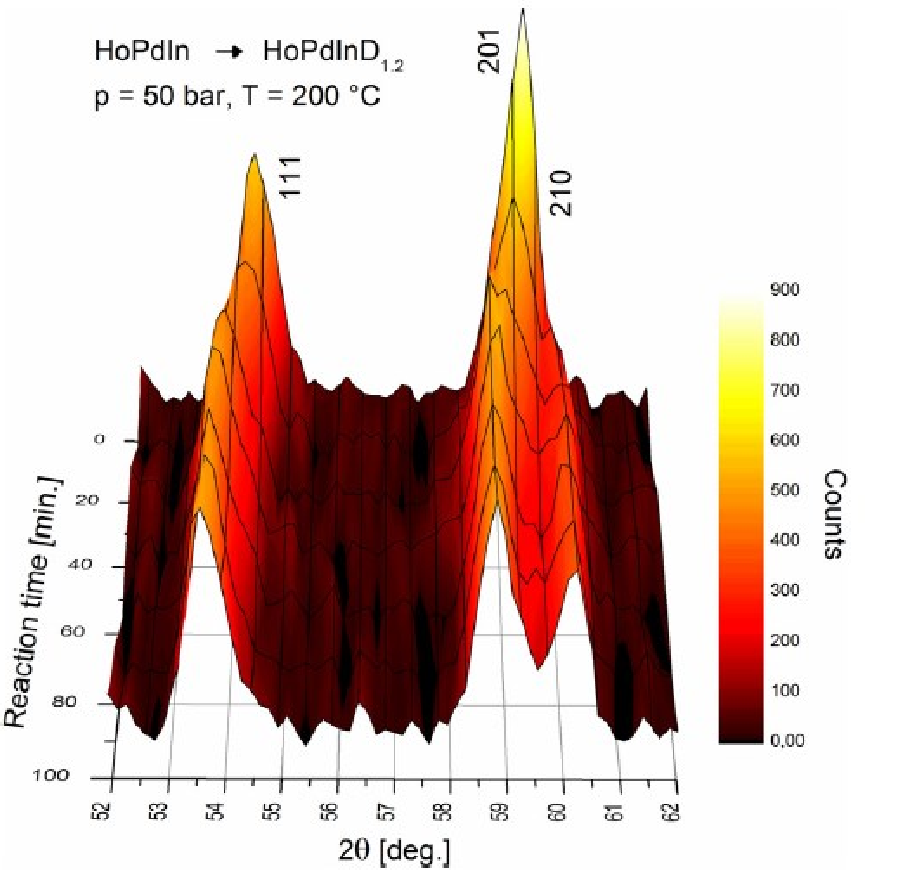
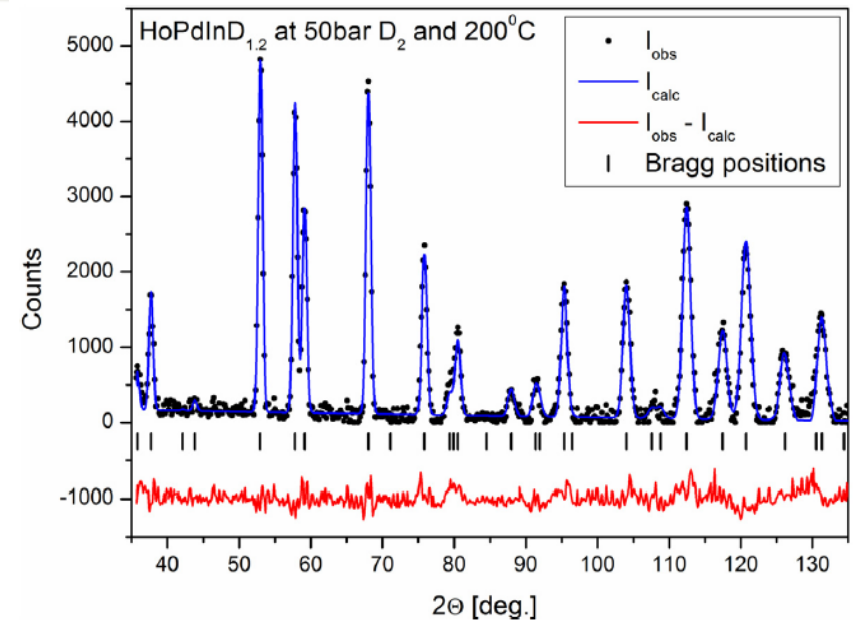
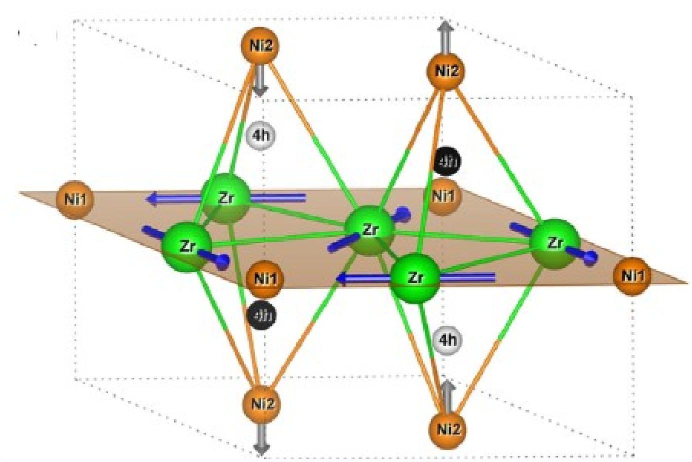
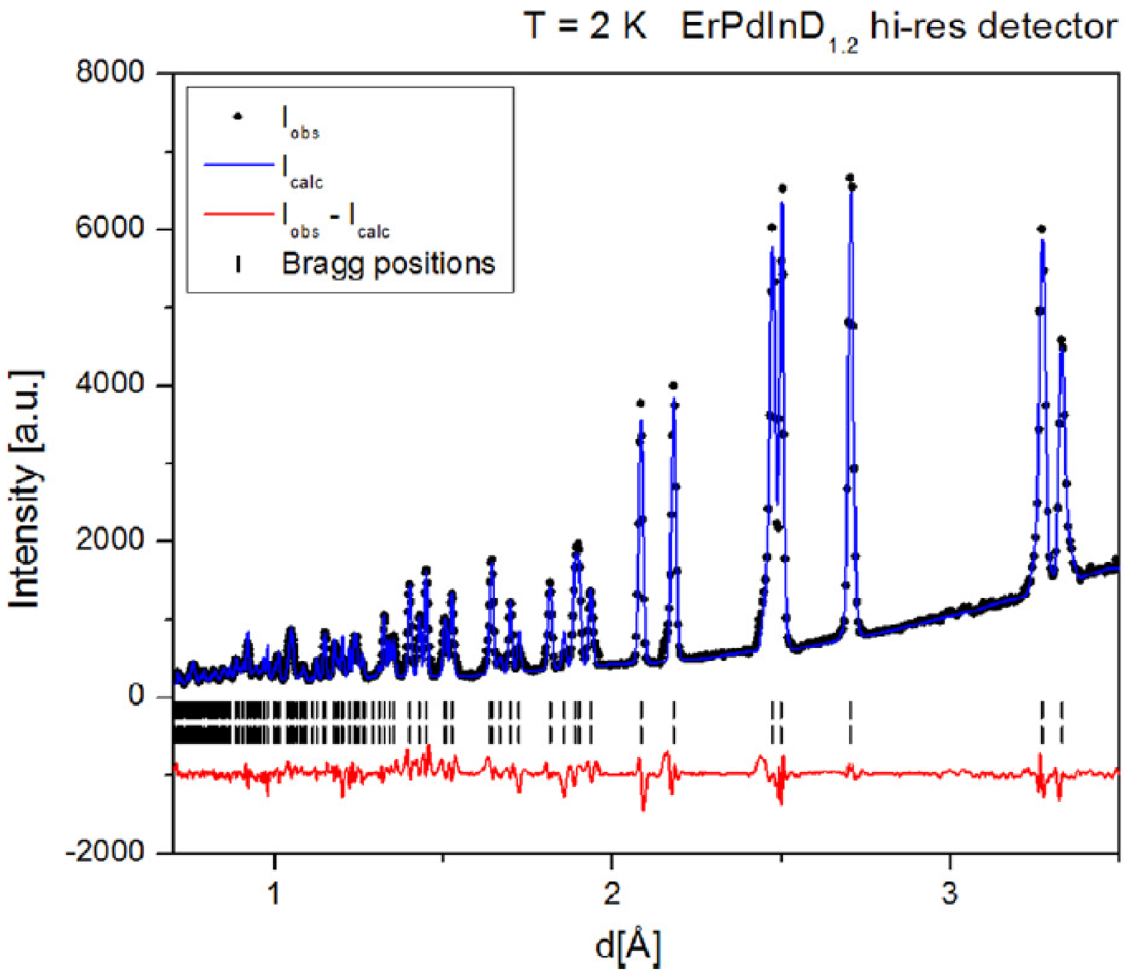


Fig. 3. In situ deuterization of the HoPdIn sample as seen by the neutron diffraction. The region with the most prominent reflections is presented. The deuterization was carried under 50 bar of D_2 pressure at a temperature of 200 °C.

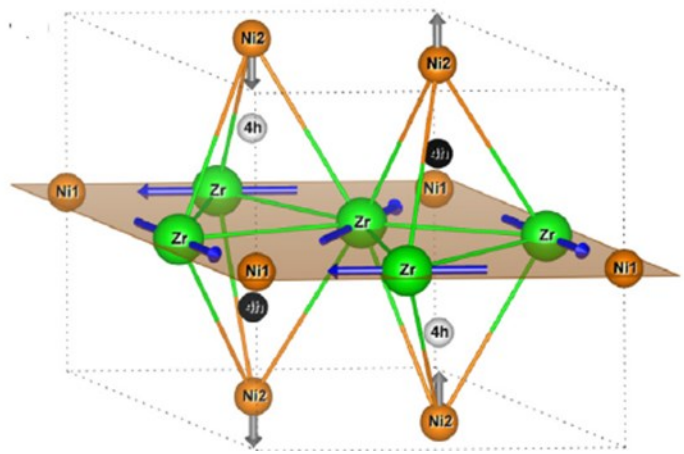




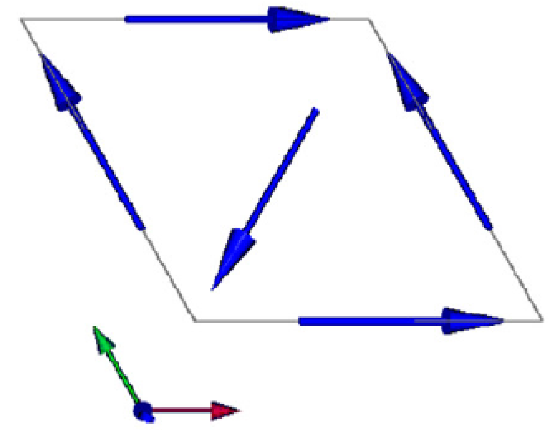
A magnetyzm?



T = 2 K ErPdInD$_{1.2}$ hi-res detector
I_{obs}
I_{calc}
$I_{obs} - I_{calc}$
Bragg positions
Intensity [a.u.]
d[Å]
8000
6000
4000
2000
0
-2000
1
2
3

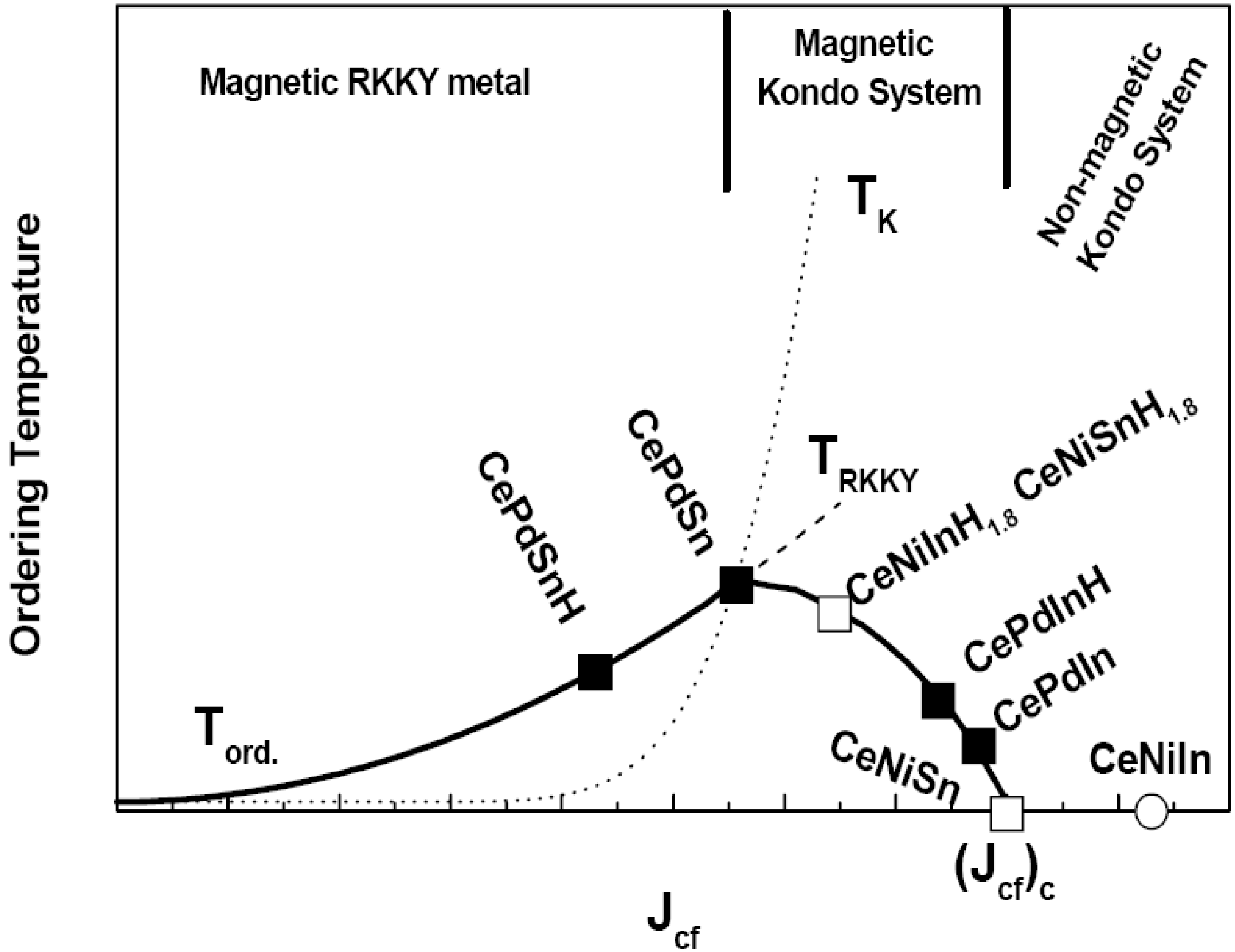


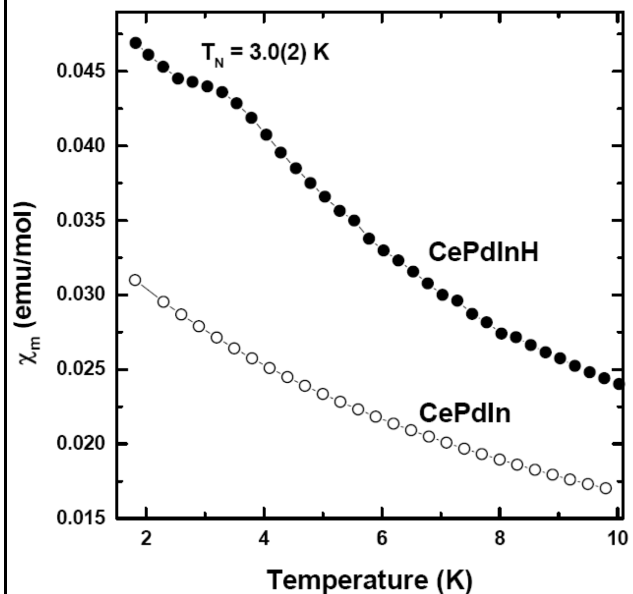
Ni2
Ni1
Zr
4h



Wpływ wodoru na magnetyzm (RPdIn)

J. Phys.: Condens. Matter **18** (2006) 1743





Wpływ wodoru na magnetyzm (CePdIn)

Vol. **126** (2014) *ACTA PHYSICA POLONICA A* No. 1

Proceedings of the 15th Czech and Slovak Conference on Magnetism, Košice, Slovakia, June 17–21 2013

Solution Growth and Investigation of the Single Crystals from the CePdIn System

M. Kratochvílová*, J. Custers, V. Sechovský

Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5, 121 16 Praha 2, Czech Republic

We report on the investigation of recently discovered heavy fermion compounds within the Ce-Pd-In system. Single crystals of $Ce_nPd_mIn_{3n+2m}$ ($n = 2, 3, 5$; $m = 1, 2$) and $Ce_4Pd_{10}In_{21}$ were synthesized from In-flux. Specific heat measurements of the multiphase $Ce_nPd_mIn_{3n+2m}$ system revealed a superconducting transition at $T_c = 0.69$ K arising from Ce_2PdIn_8 and another, magnetic transition at $T_m = 1.67$ K arising from either Ce_3PdIn_{11} or $Ce_5Pd_2In_{19}$. Low-temperature data of $Ce_4Pd_{10}In_{21}$ display ferromagnetic long-range order below 0.8 K.

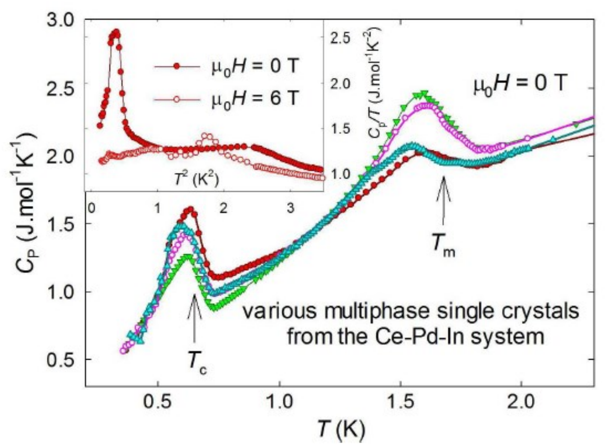
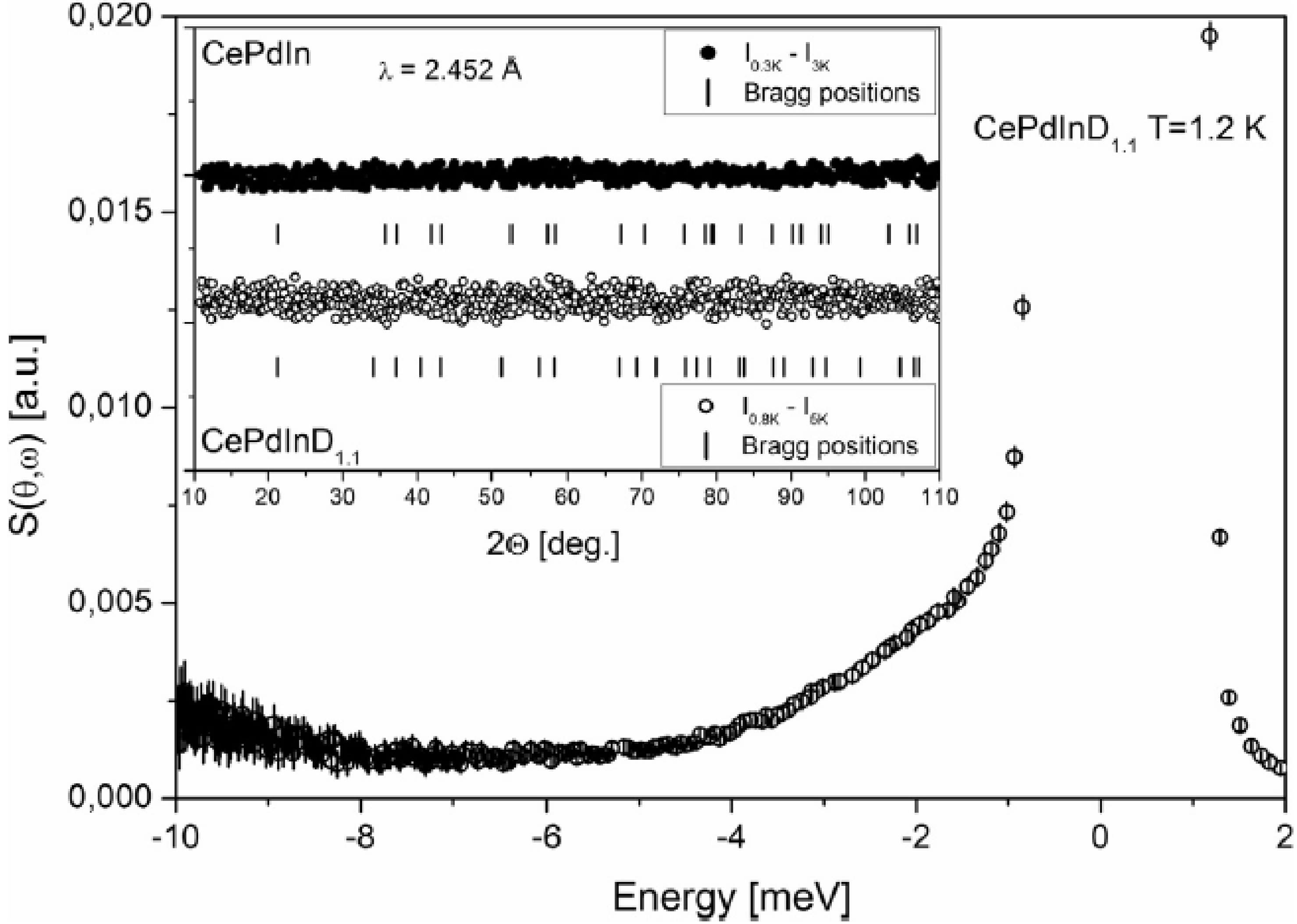


Fig. 2. Specific heat of multiphase samples in zero magnetic field. The arrows mark the superconducting transition at T_c and the magnetic transition at T_m.

Wpływ wodoru na magnetyzm (RPdIn)

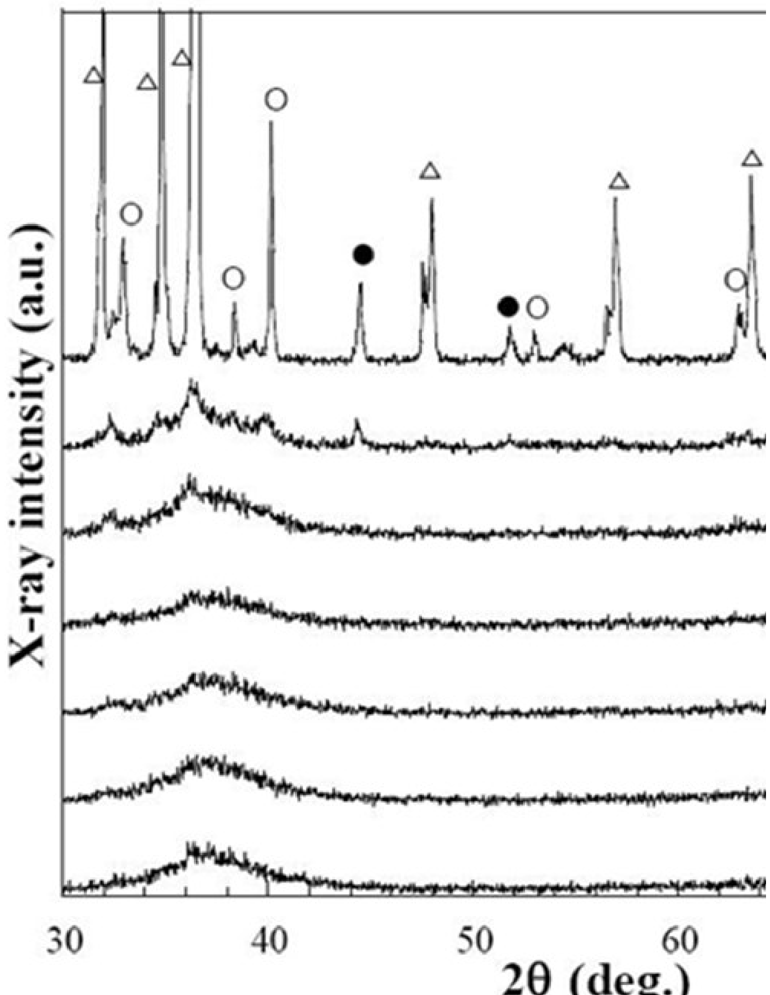


Wpływ wodoru na magnetyzm (RPdIn)

The magnetic properties of the RPdIn deuterides obtained from the magnetometric measurements. T_C is the Curie temperature, T_N Neél temperatures, T_R the reorientation temperature, Θ_P the paramagnetic Curie temperature, μ_{eff} the effective magnetic moment and χ_o the temperature-independent susceptibility.

Compound	T_C, T_N	T_R	Θ_P	μ_{eff}	χ_o
CePdIn [16]	?	–	−41	2.64	–
$CePdInD_{1.1}$	?	–	−32.8(4)	2.59(3)	–
PrPdIn [21]	–	–	−8.8	3.57	0.0019
$PrPdInD_{1.2}$	–	–	−7.0(1)	3.61(2)	0.0002
NdPdIn [29]	30	–	2	3.59	–
$NdPdInD_{1.2}$	5.7	–	0.6(4)	3.45(3)	0.0022
HoPdIn [29]	25	6	7	10.8	–
$HoPdInD_{1.2}$	3.0	–	0.5(3)	10.56(5)	0.0010
ErPdIn [29]	12.3	–	1.6	9.7	–
$ErPdInD_{1.2}$	6.9	–	−1.2(2)	6.20(3)	0.0021

Przemiany fazowe w stopach Ti-Zr-Ni



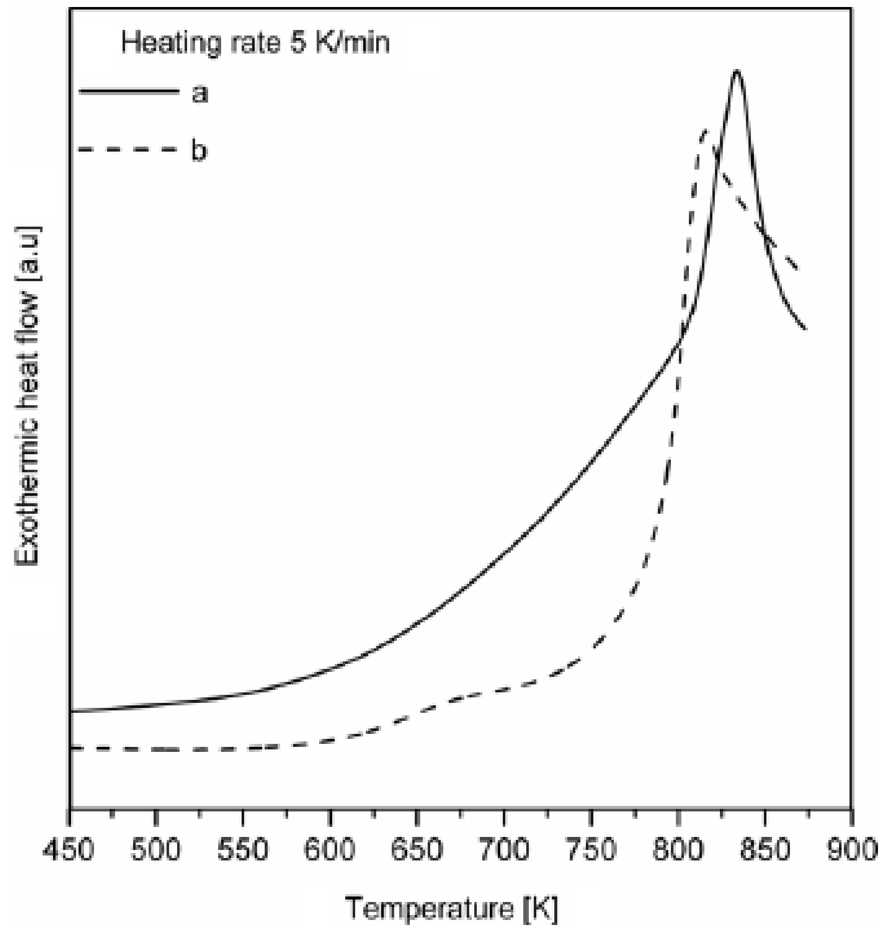


Fig. 3. DSC curves for the $Ti_{45}Zr_{38}Ni_{17}$ amorphous powder obtained after MA for 40 h (a) first heating, (b) second heating.

Przemiany fazowe w stopach Ti-Zr-Ni

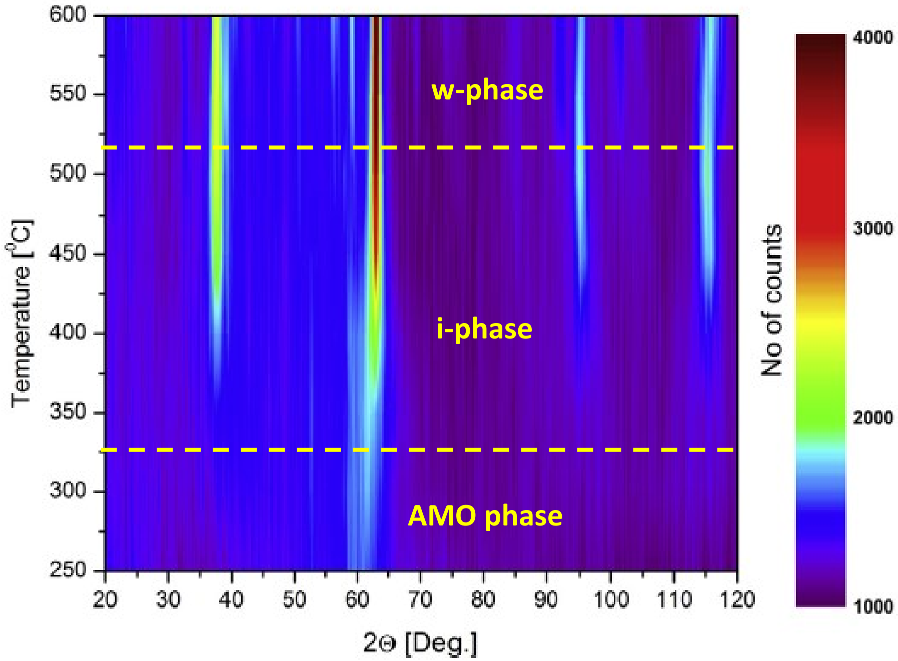


Fig. 2. The neutron diffraction patterns of the initially amorphous $Ti_{45}Zr_{38}Ni_{17}$ at elevated temperatures.

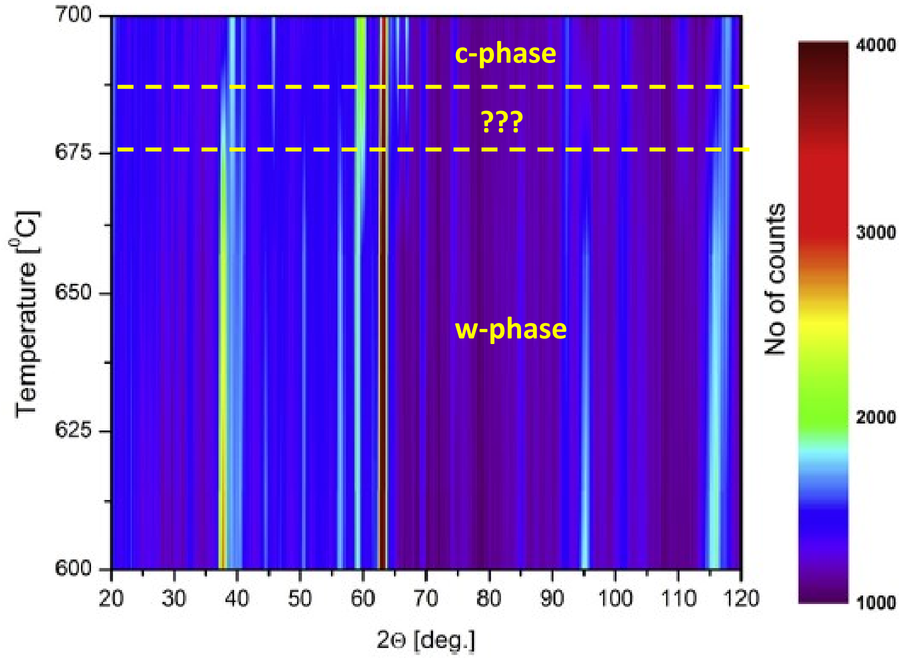
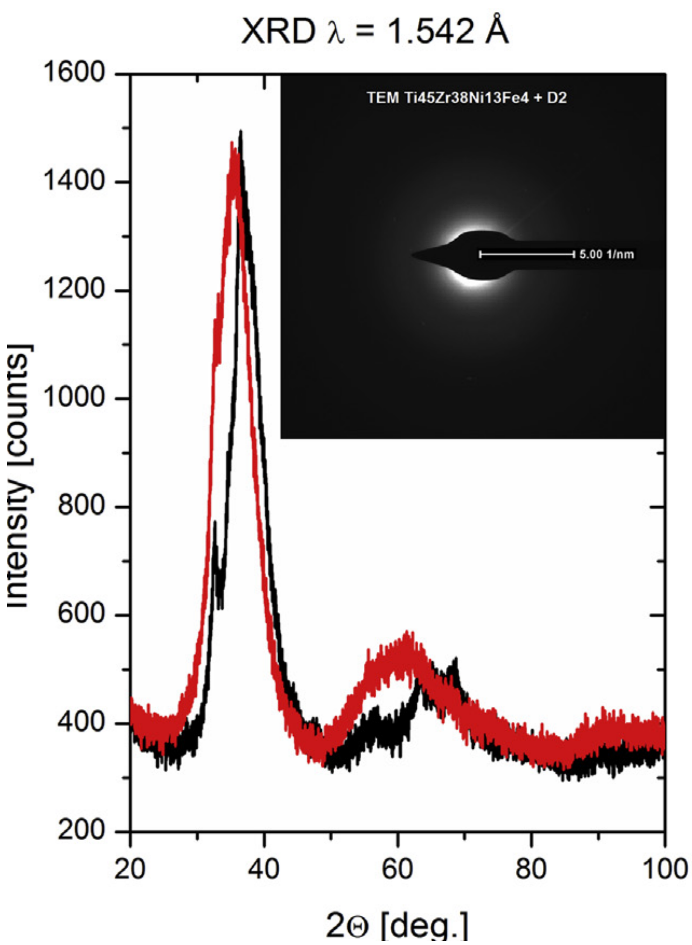
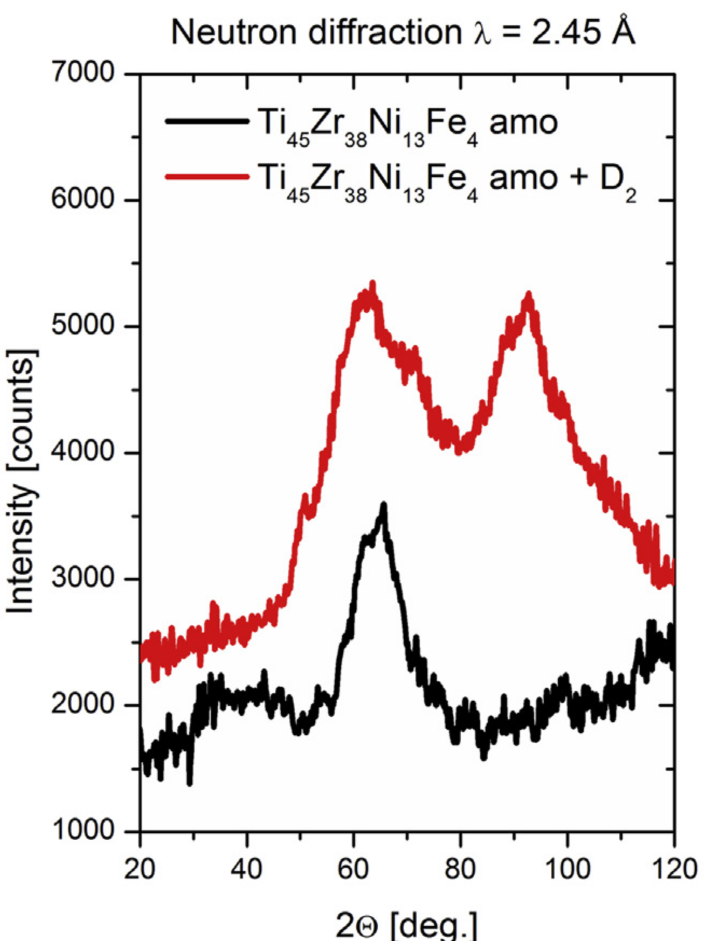
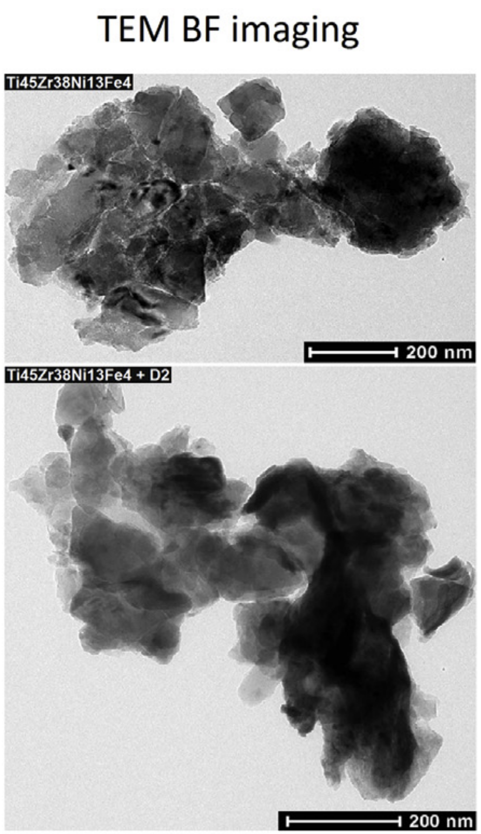


Fig. 3. The thermal evolution of the *w-phase* of the $Ti_{45}Zr_{38}Ni_{17}$ as seen by ND.

Amorficzne wodorki w układzie Ti-Zr-Ni







Przemiany fazowe w stopach Ti-Zr-Ni-D

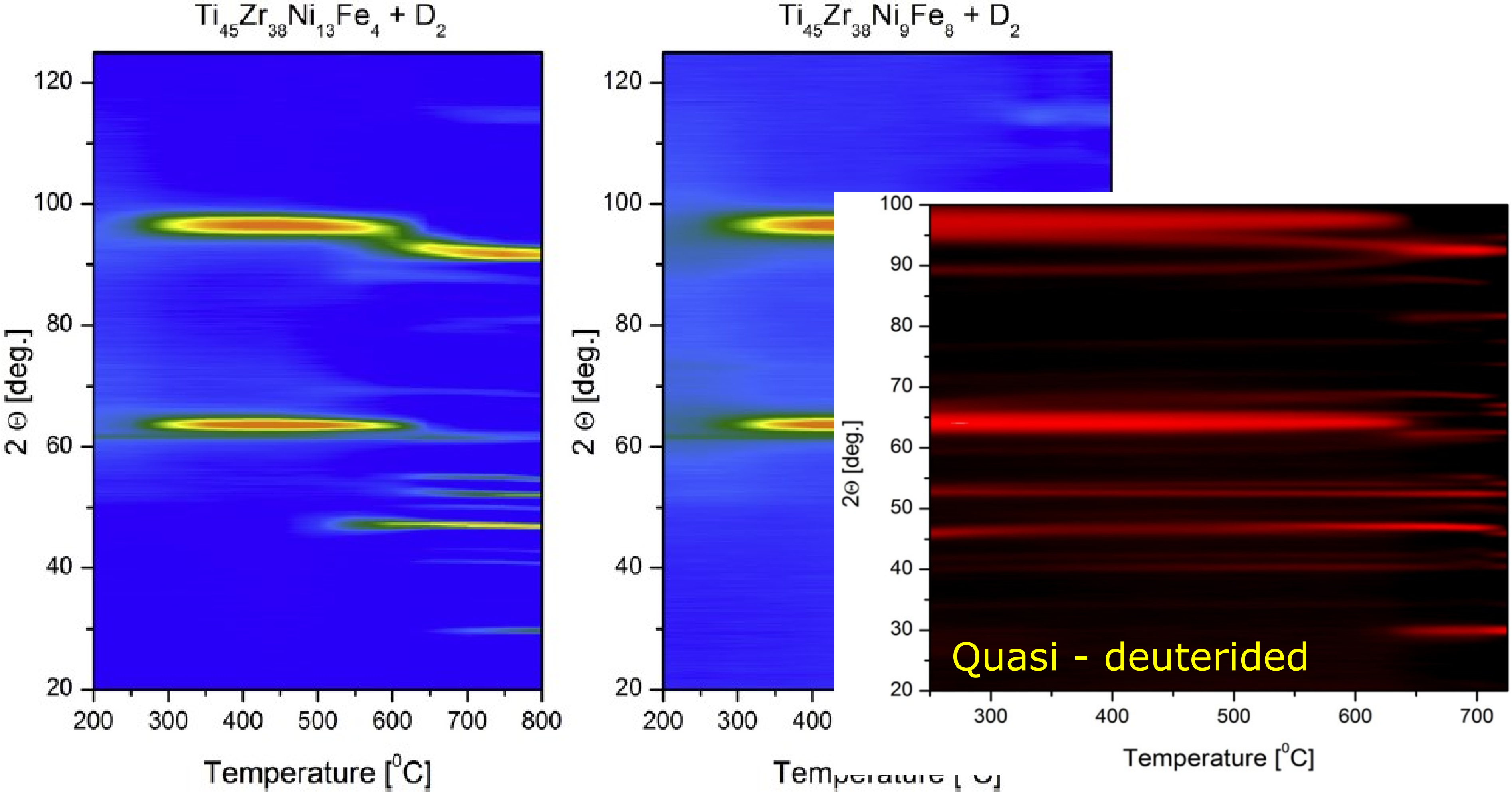
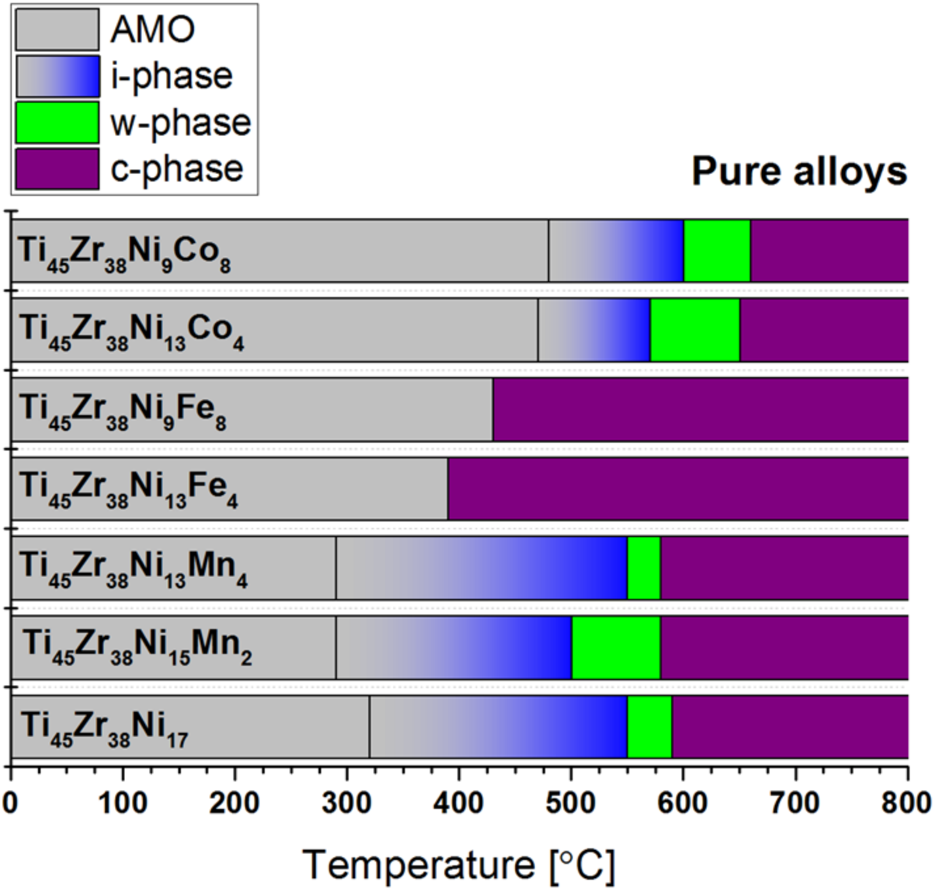
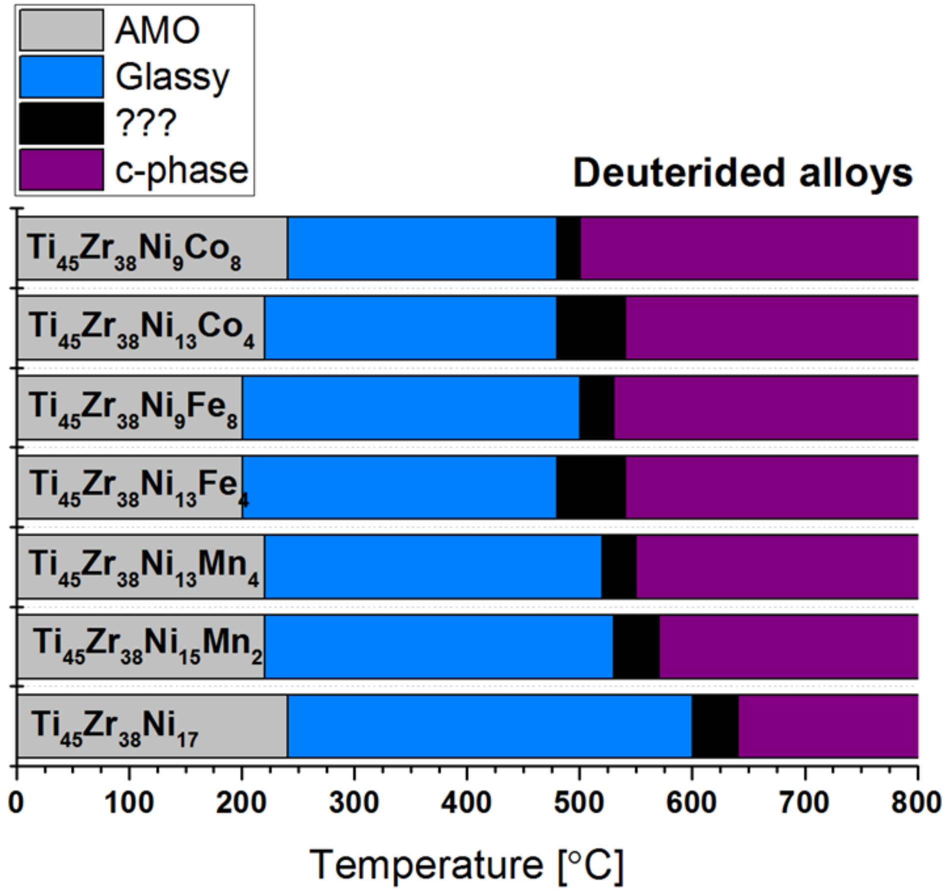


Fig. 6. Thermal evolution of the deuterided amorphous $Ti_{45}Zr_{38}Ni_{13-x}Fe_x$ (x = 4 and x = 8) specimens.

Przemiany fazowe w stopach Ti-Zr-Ni



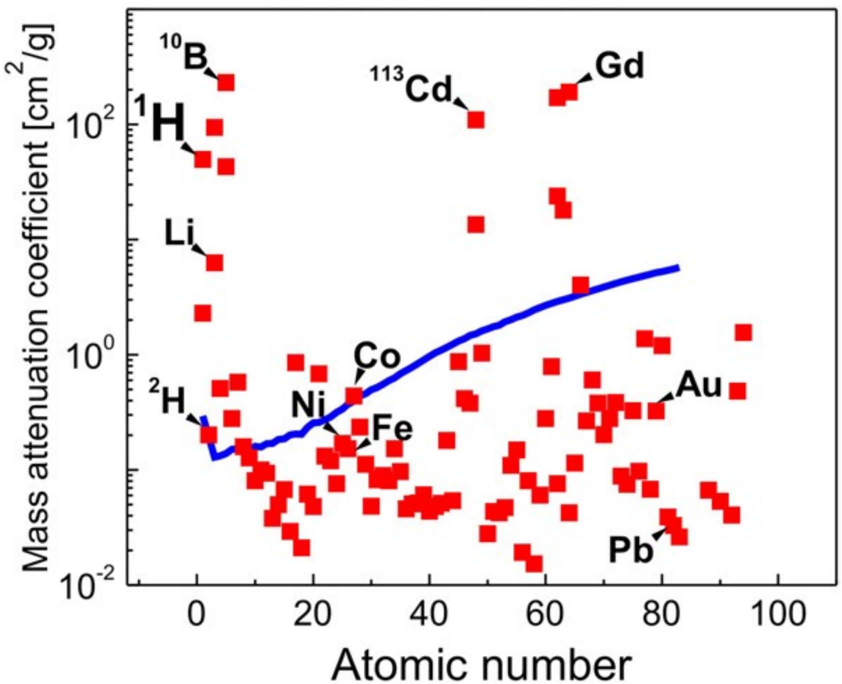


AGH

Neutron TV

HZB Helmholtz Zentrum Berlin

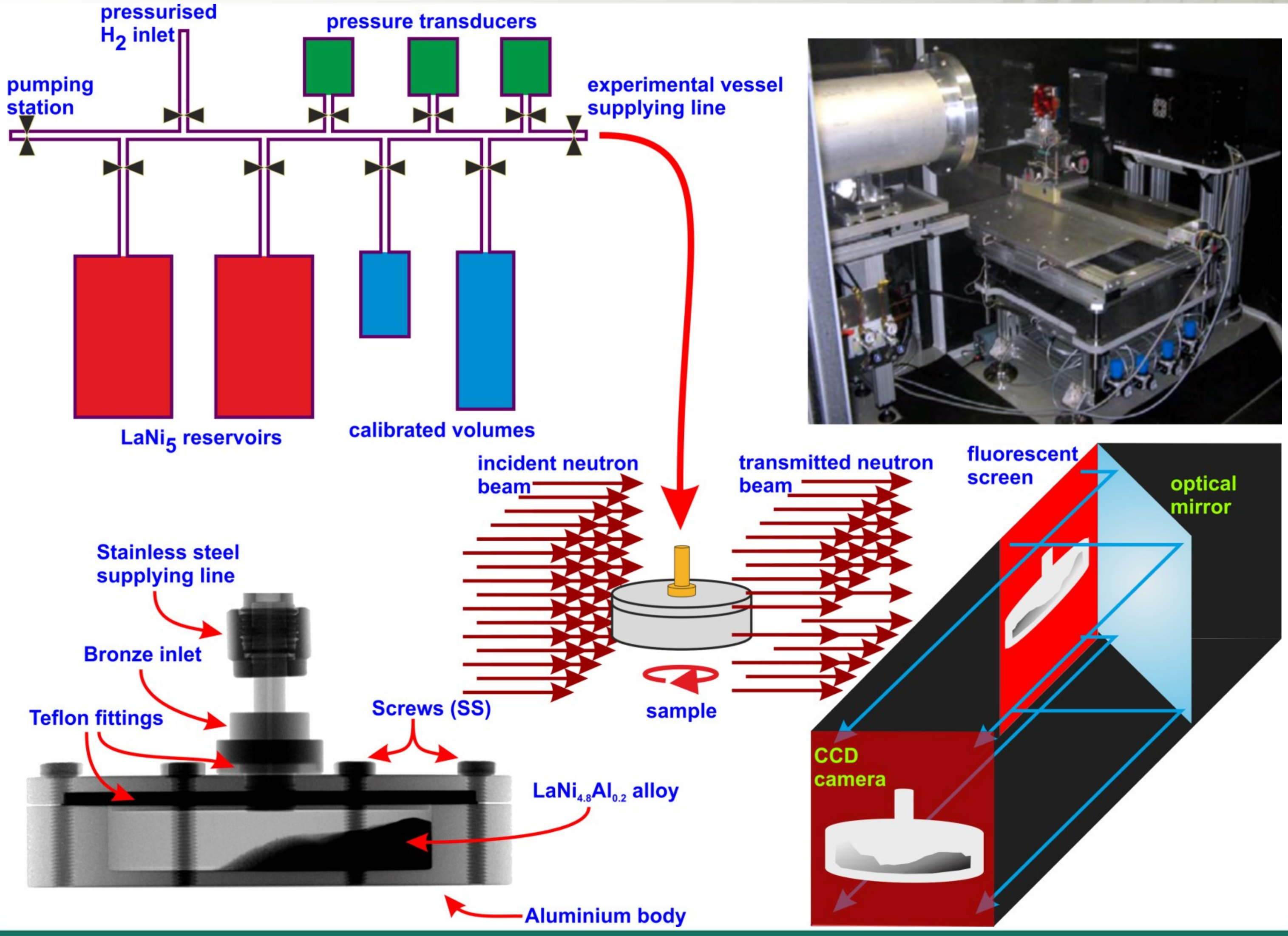
V7 Cold Neutron Imaging (CONRAD)



Spectrum	0.2 – 1.2 nm (Maxwellian distribution with a maximum at 0.35 nm)
Sample environment	Cooling water, air pressure, N-, He- and H- gas lines
Measuring positions	Two (the parameters are listed below)
Detectors	CMOS camera (Position I) – PCO 1200hs - 1280 x 1024 pixel CCD camera (Andor DW436N-BV) 2048x2048 pixel (Position II)
Instrument responsible	N. Kardjilov, kardjilov@hmi.de direct dialling -2298

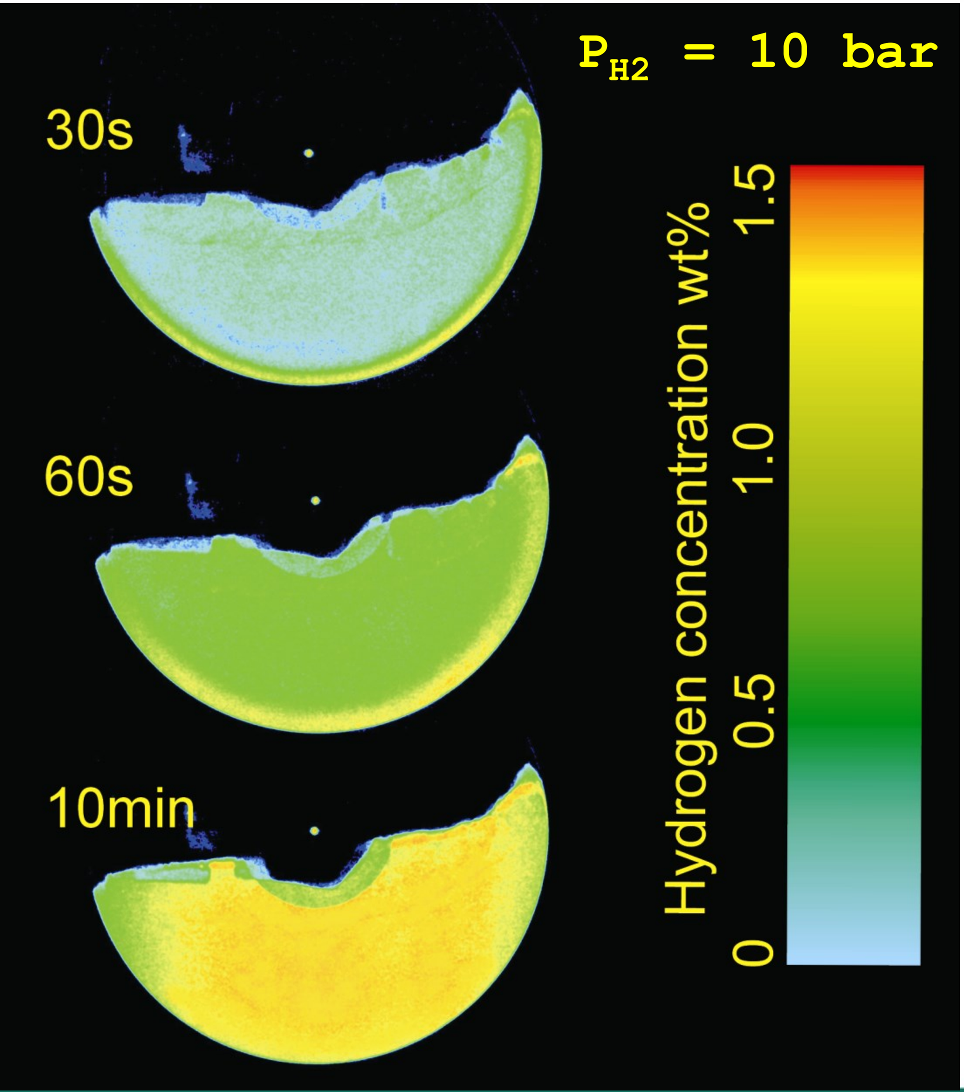
	L/D	Resolution [μm]	Max neutron flux at sample [n/cm^{2}s]	Exposure time [s]	Beam size [cm^2]
Position I	~70	300-500	2×10^{8}	0.01-0.5	3x5
Position II					
- 3 cm pinhole	167	~300	$\sim2\times10^{7}$	1-5	12x12
- 2 cm pinhole	250	~200	$\sim1\times10^{7}$	5-15	11x11
- 1 cm pinhole	500	~100	$\sim6\times10^{6}$	10-25	10x10

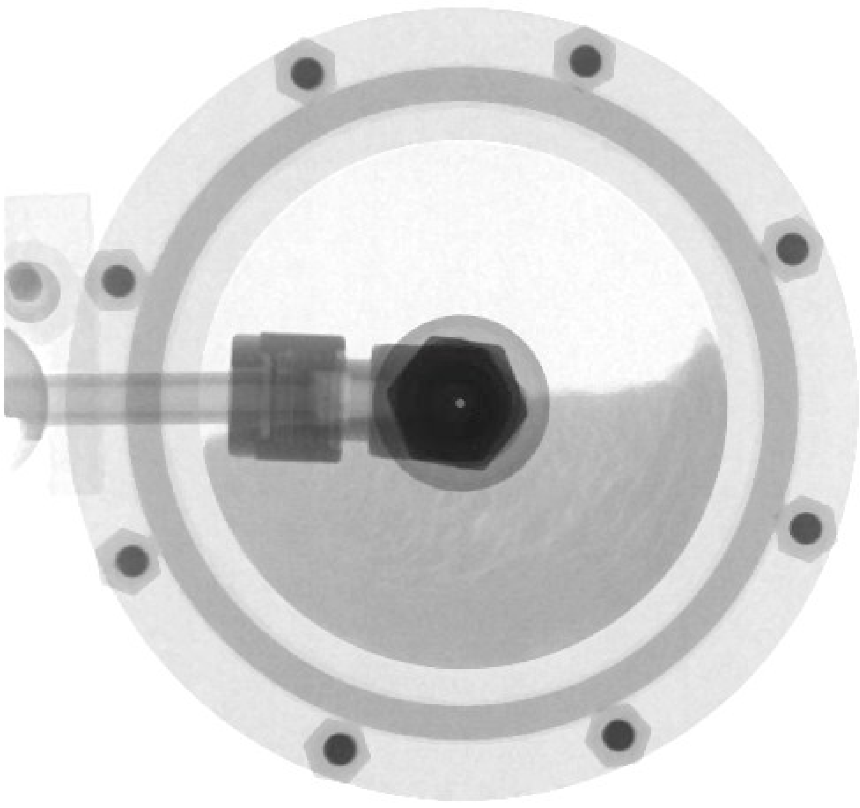
Neutron TV



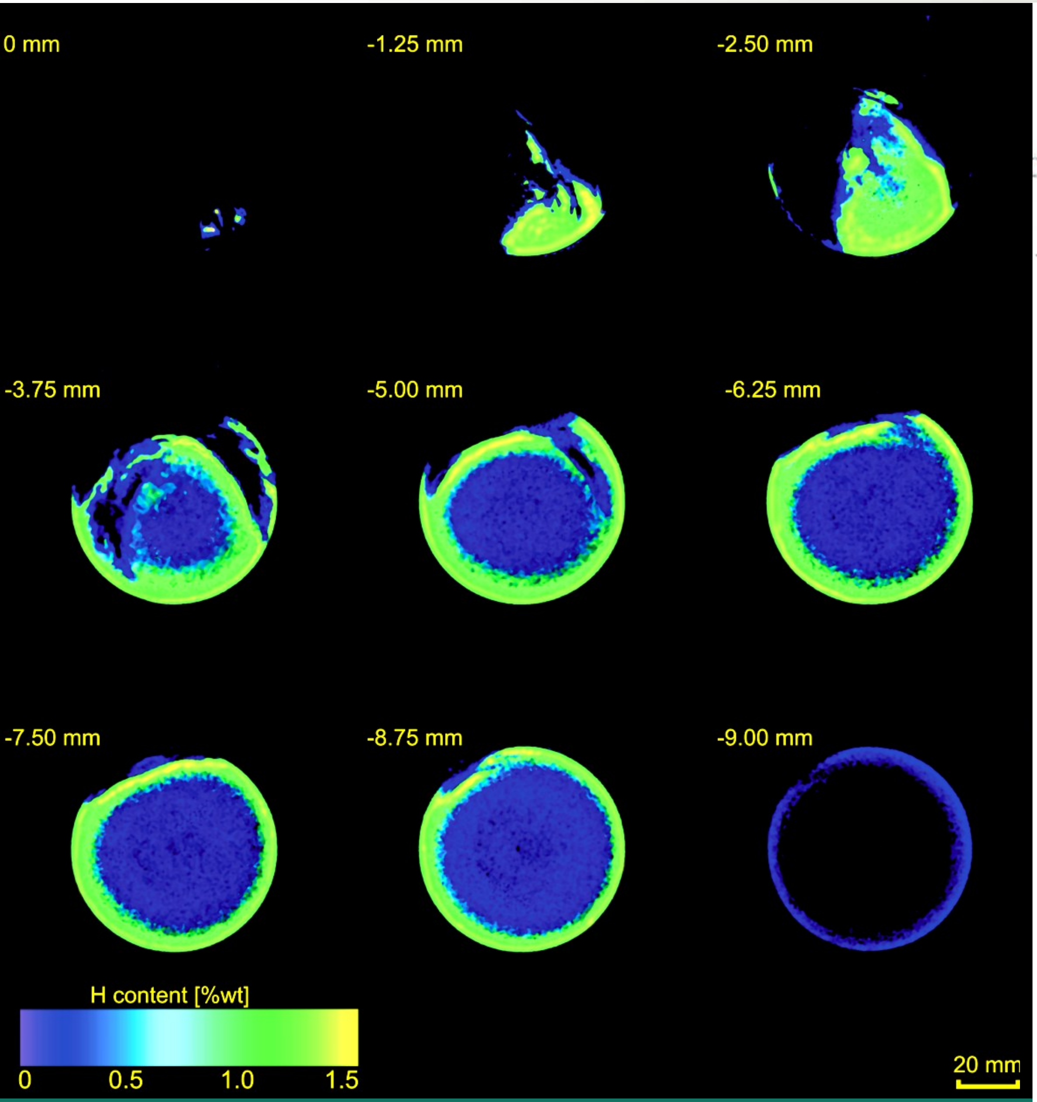
pressurised H_2 inlet
pressure transducers
pumping station
experimental vessel supplying line
$LaNi_5$ reservoirs
calibrated volumes
incident neutron beam
transmitted neutron beam
fluorescent screen
optical mirror
Stainless steel supplying line
Bronze inlet
Teflon fittings
Screws (SS)
sample
CCD camera
$LaNi_{4.8}Al_{0.2}$ alloy
Aluminium body

Neutron TV





Neutron TV



0 mm
-1.25 mm
-2.50 mm
-3.75 mm
-5.00 mm
-6.25 mm
-7.50 mm
-8.75 mm
-9.00 mm
H content [%wt]
0
0.5
1.0
1.5
20 mm

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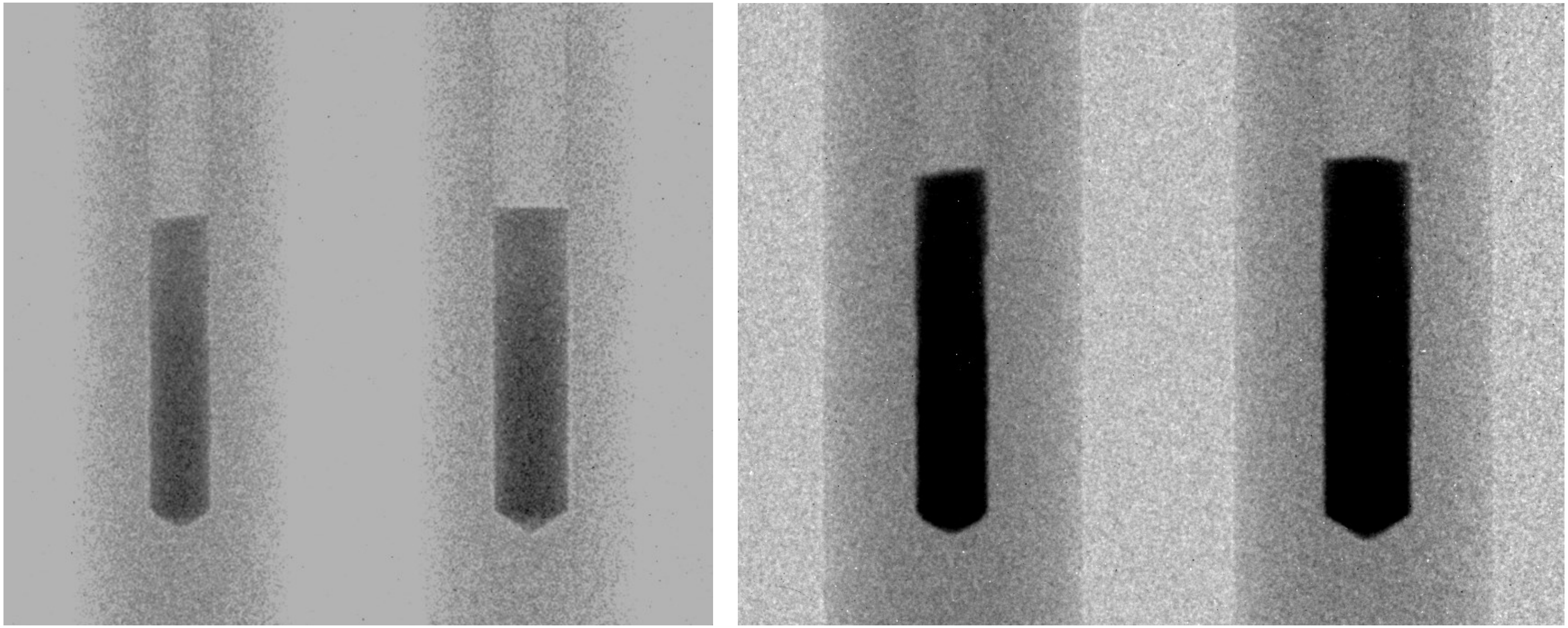
Nuclear materials
Fuels and materials challenges for nuclear systems

Neutron TV

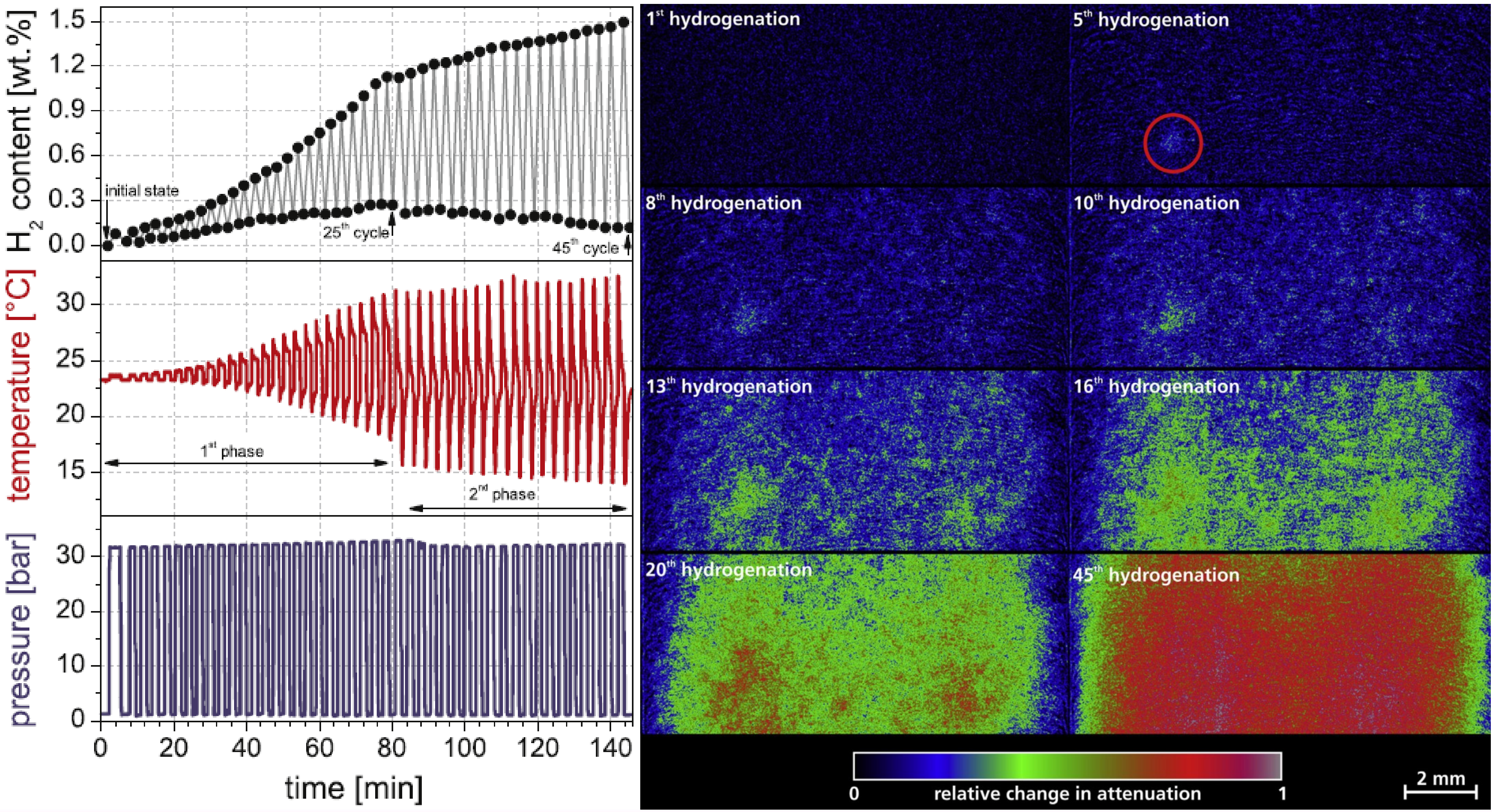


Neutron TV

Journal of Power Sources 277 (2015) 360–369

In operando visualization of hydride-graphite composites during cyclic hydrogenation by high-resolution neutron imaging

Carsten Pohlmann [a], Kai Herbrig [b], Łukasz Gondek [c], Nikolay Kardjilov [d], André Hilger [d], Henryk Figiel [c], John Banhart [d,e], Bernd Kieback [a,b], Ingo Manke [d], Lars Röntzsch [a,*]



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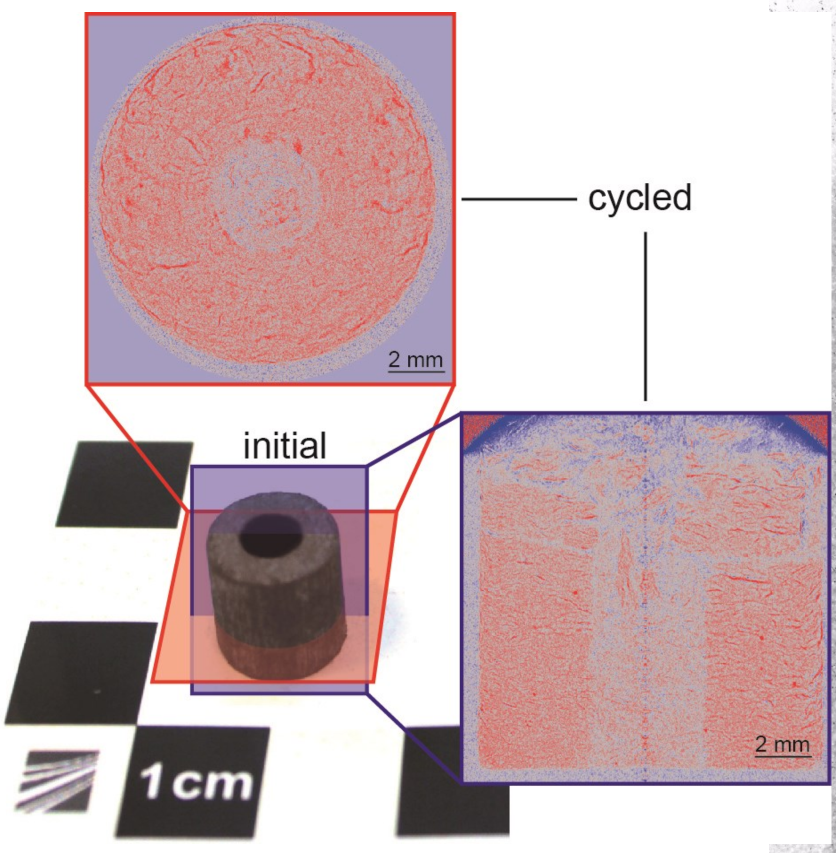

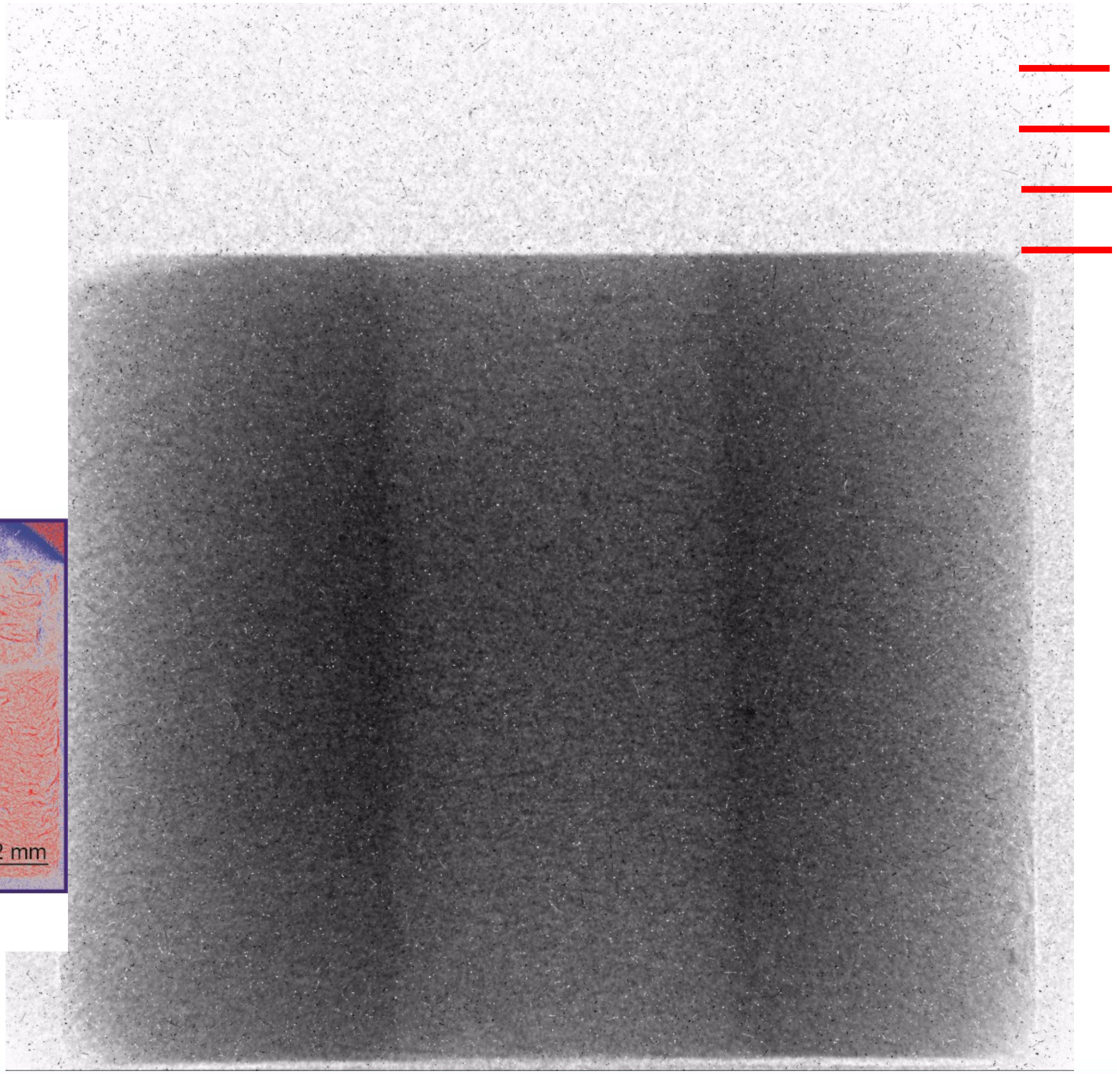

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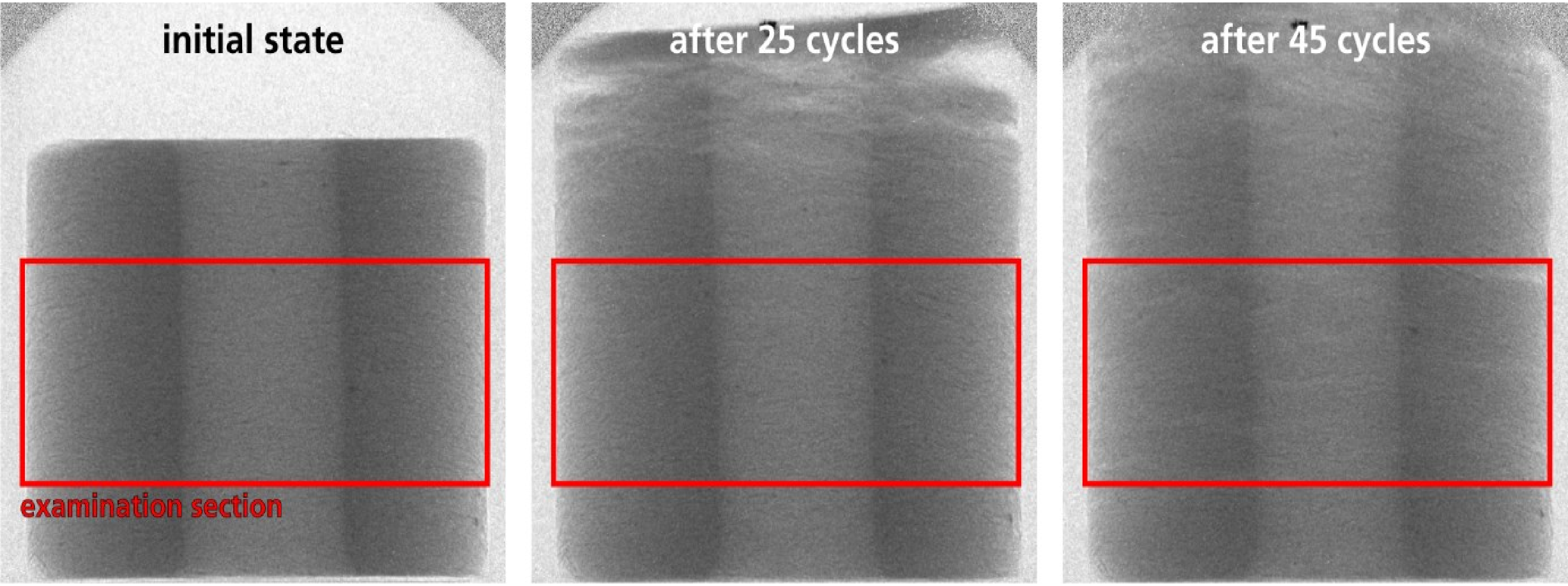
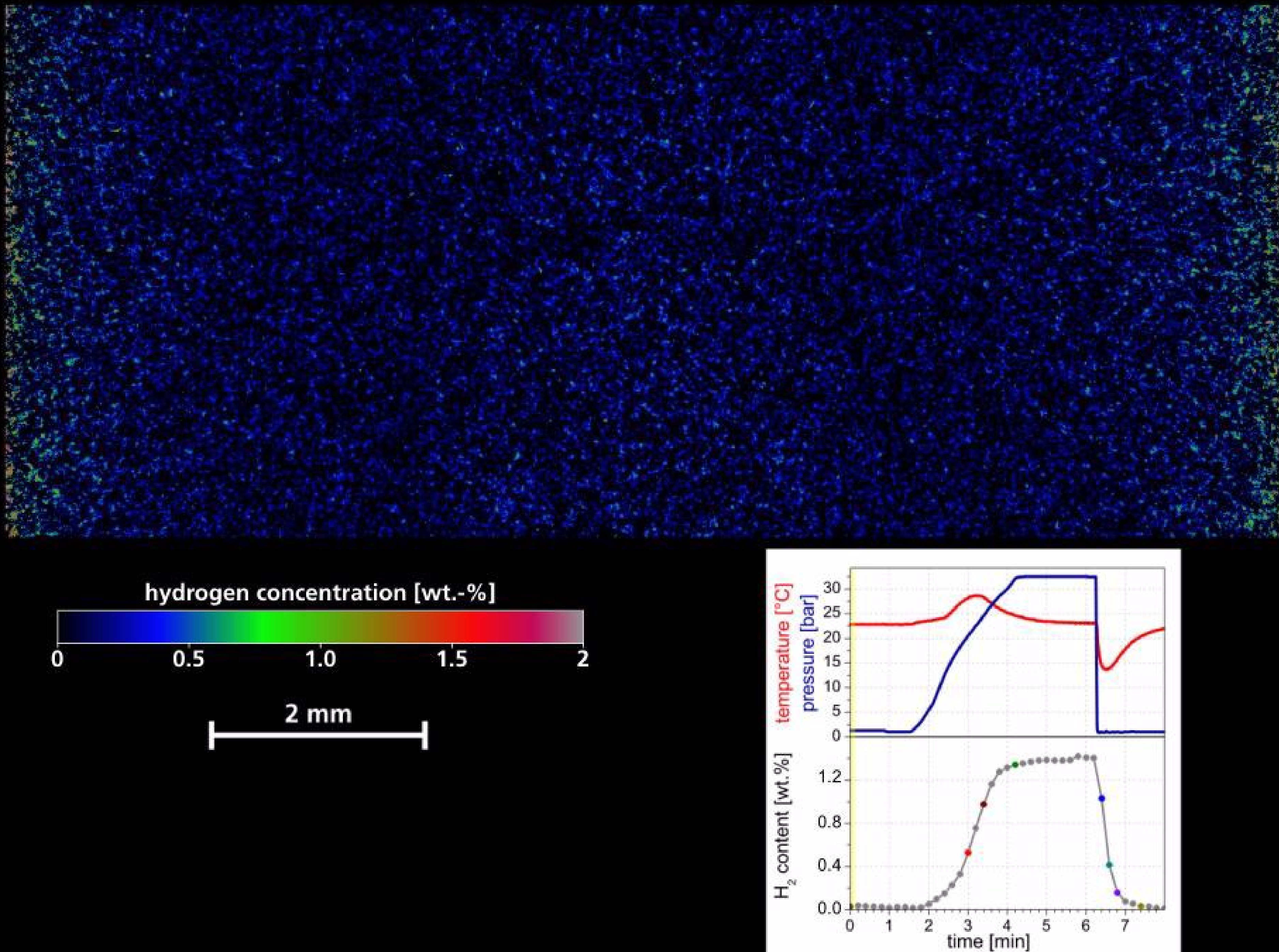


initial state
examination section
after 25 cycles
after 45 cycles



hydrogen concentration [wt.-%]
0
0.5
1.0
1.5
2
2 mm
temperature [°C]
pressure [bar]
0
5
10
15
20
25
30
H2 content [wt.%]
0.0
0.4
0.8
1.2
time [min]
0
1
2
3
4
5
6
7

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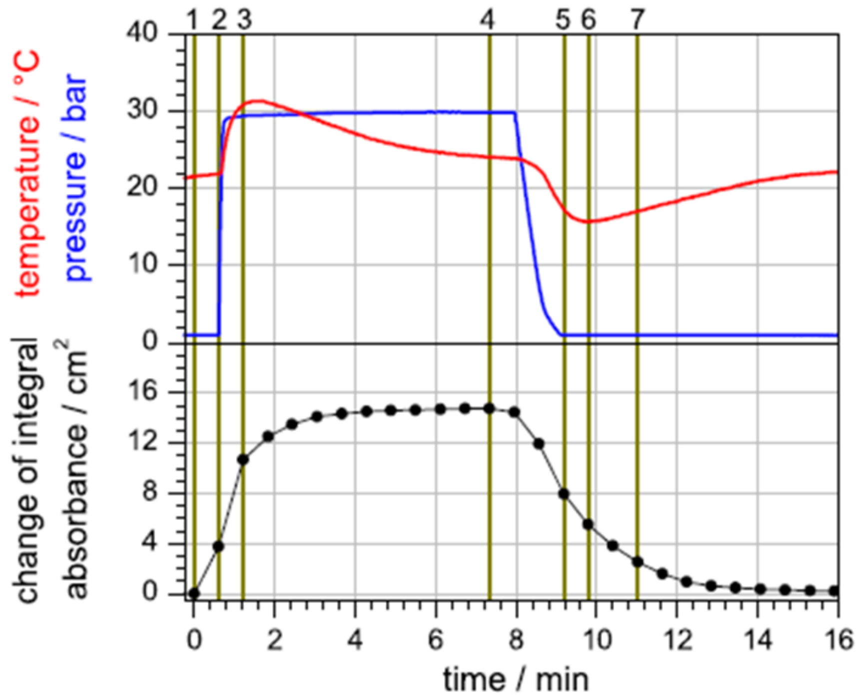
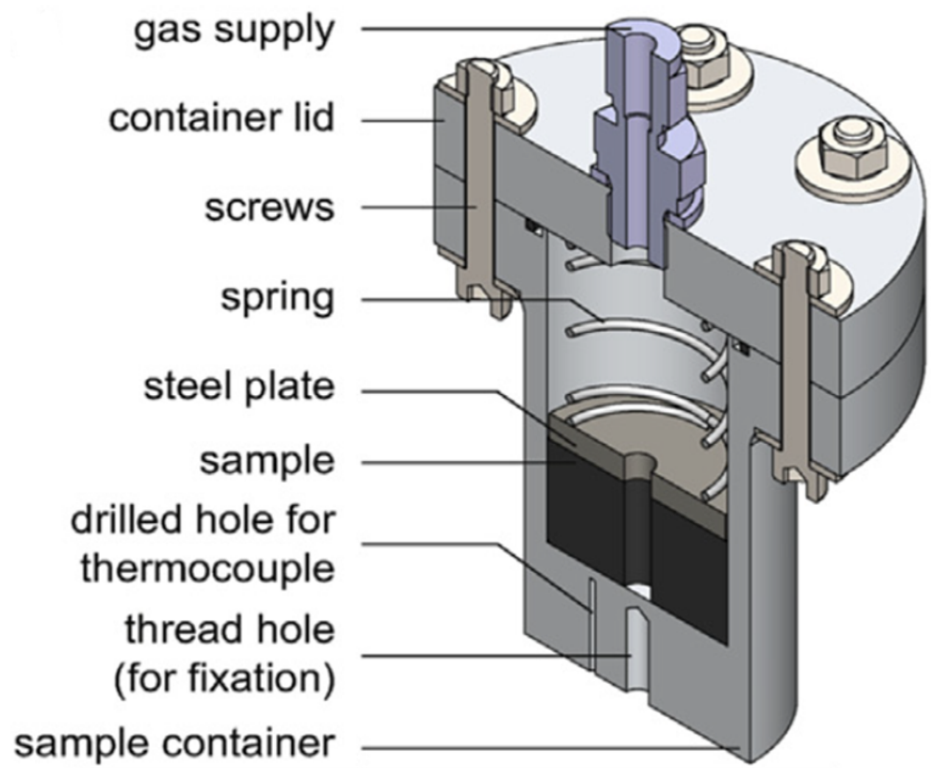
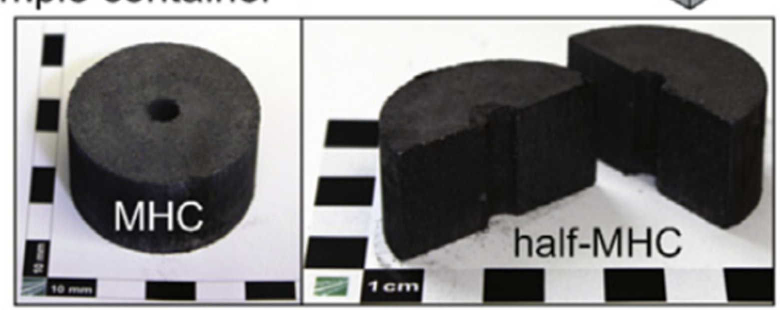
Investigations of the structural stability of metal hydride composites by in-situ neutron imaging

Kai Herbrig [a, b], Carsten Pohlmann [a], Łukasz Gondek [c], Henryk Figiel [c], Nikolay Kardjilov [d], André Hilger [d], Ingo Manke [d], John Banhart [d, e], Bernd Kieback [a, b], Lars Röntzsch [a, *]







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