

Experimental methods of Neutron Physics Laboratory in NPI Řež for Testing of Metallic Samples

Experimentální metody Laboratoře neutronové fyziky v ÚJF Řež pro testování kovových vzorků

RNDr. Pavol Mikula, DrSc., RNDr. Pavel Strunz, CSc., Mgr. Vasyl Ryukhtin, Ph.D.

LNF, ÚJF AV ČR Řež, Husinec-Řež čp. 130, PSČ: 250 68, Česká republika, korespondenční email: mikula@ujf.cas.cz

The use of neutron scattering methods for structural and property studies of technologically interesting materials as well as solution of industrial problems has been expanding in the last decades. Correspondingly, demands for the beam time at neutron sources have been strongly increased in all neutron research laboratories in the world. Several methods related to materials science has been effectively also used at a medium-power research-reactor LVR-15 operated by the Research Centre Řež. Several methods with the examples of unique experimental results which could be attractive and useful for people employed in metal production and procession are presented. Moreover, the access to the research experiments targeting new experimental results is free.

Key words: *Neutron scattering methods, Neutron Physics Laboratory, experimental equipment, user access*

Využití neutronového rozptylu pro studium struktur a vlastností technologicky zajímavých materiálů a také při řešení technologických problémů s materiály v průmyslu se v posledních dvou desetiletích velice rozmáhá. Současně s tím požadavky na získání odpovídajícího experimentálního času silně narostly ve všech neutronových laboratořích ve světě. Několik experimentálních metod využitelných v materiálovém výzkumu je také k dispozici na experimentálních zařízeních vybudovaných Laboratoří neutronové fyziky ÚJF AV ČR u výkumného reaktoru středního výkonu LVR-15, který provozuje Centrum výzkumu s.r.o. v Řeži. V článku je uvedeno několik příkladů unikátních experimentálních výsledků, které by mohly zaujmout pracovníky zabývající se produkcí a obráběním kovových materiálů a případně i přípravou technologie výroby výstupních vzorků. V této souvislosti je nutné poznamenat, že nekomerční měření na experimentálních zařízeních (včetně obsluhy), která jsou zaměřená na získání nových experimentálních výsledků, jsou poskytována bezplatně.

Klíčová slova *Metody neutronového rozptylu, Laboratoř neutronové fyziky, experimentální vybavení, vnější uživatelé*

1. Řež Neutron Physics Laboratory (NPL)

First, let us shortly introduce all research activities and facilities existing in NPL. NPL is a part of Nuclear Physics Institute ASCR, enabling us to perform neutron physics experiments as well as to provide experimental facilities and research experience for external users. Research activities of NPL are concentrated at the medium power 10 MW (mean power) reactor LVR-15 operated by Research Centre Řež (<https://cz.linkedin.com/company/research-centre-rez>) [1] on a commercial basis. In total, NPL operates 8 instruments installed at 5 radial horizontal beam tubes (for experiments in nuclear physics, solid state physics and materials research) and two vertical irradiation channels (for neutron activation analysis). Fig. 1 shows a schematic picture of the arrangement of the instruments installed at the reactor LVR-15. For details see the web

page <https://www.ujf.cas.cz/cs/oddeleni/oddeleni-neutronove-fyziky/> [2]. The individual instruments are offered for measurements to external users free by means of the CANAM project [14,15]. After carrying out the experiment an entire support including a high-quality software for data analysis, preliminary raw data elaboration and a permanent assistance of the responsible researcher are provided.

2. Provided experimental basis

A short description of the several operating experimental facilities and activities carried out with neutrons in NPL is as follows:



Fig. 1 Schematic picture of the experimental facilities of NPL at the reactor LVR-15

Obr. 1 Schematický obrázek experimentálních zařízení Laboratoře neutronové fyziky instalovaných u reaktoru LVR-15

TKSN-400 high-resolution diffractometer (Fig. 2, Fig. 3) is equipped with a bent Si single crystal monochromator and ^3He position sensitive detector. Due to its equipment, the instrument is dedicated especially to thermo-mechanical testing of materials, i.e. to study the deformation and transformation mechanisms of modern types of newly developed materials. Neutron diffraction performed in situ upon external loads, brings a wide range of valuable structural and sub-structural parameters (e.g. dislocation density and average grain size) of the studied material which is easy to correlate with parameters of external loads. The diffractometer yields very good resolution and luminosity. It is mainly used for mapping residual macrostrains, microstrain analysis and in situ tension/compression tests of samples in a wide range of temperatures above the room value. The instrument is equipped with the tension/compression rig (with Joule heating) for measurements under a thermo-mechanical load. The diffractometer operates in the angular range of the scattering angles $25^\circ < 2\theta_s < 90^\circ$ with the resolution $2 \times 10^{-3} \leq \Delta d/d \leq 3 \times 10^{-3}$ (d is the lattice spacing). It is equipped with a variety pieces of the sample environment:

- Deformation machine for uni-axial loading (tension, pressure) ± 20 kN
- Resistant heating ($T < 1200^\circ\text{C}$)
- Hot-air heating ($T < 300^\circ\text{C}$)

- Eulerian cradle: inner diameter of 400 mm, $0^\circ < \chi < 160^\circ$, $0^\circ < \varphi < 360^\circ$
- Miniature deformation machine for uni-axial loading (tension, compression) ± 10 kN, suitable for installation on the Eulerian cradle to measure lattice strain in all directions
- Deformation machine for bending loading, maximum cycling frequency of 27 Hz
- In-situ acoustic emission measurement during loading test. Physical Acoustics PCI-2 board. AE sensor frequency range: 100–1200 kHz.

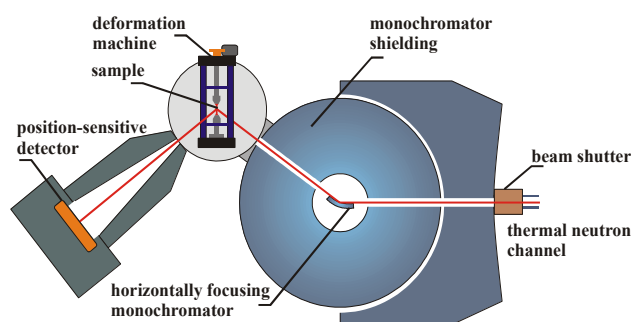


Fig. 2 Schematic picture of the diffractometer TKS-400

Obr. 2 Schematický obrázek difraktometru TKS-400

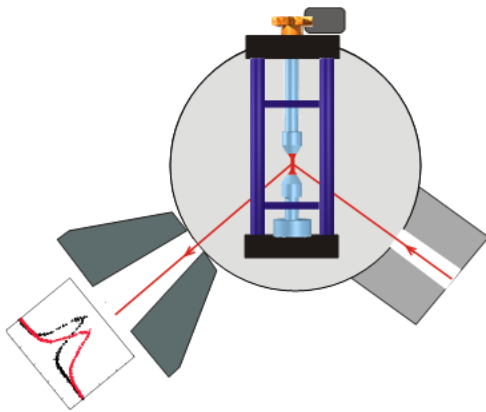


Fig. 3 Detail of loading in tension or compression resulting in a shift and change of the diffraction profile

Obr. 3 Detail zařízení pro zatěžování vzorků v tahu a tlaku, které způsobuje úhlový posun a tvar (šířku) difrakčního profilu

SPN-100 multipurpose diffractometer (Fig.4) equipped with a bent Si single crystal monochromator and ^3He position sensitive detector. The diffractometer uses advantages coming from focusing both in real and momentum space. It is dedicated to mapping of residual macrostrains inside polycrystalline materials. Typical examples are residual strains/stresses developed in the course of welding or accumulated during processing and/or usage of engineering components. Furthermore, it can be

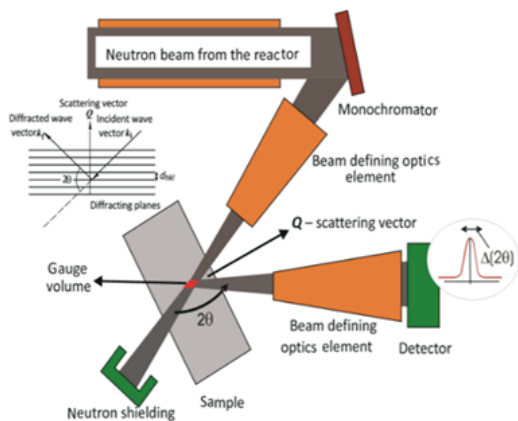


Fig. 4 Schematic picture of the strain/stress device SPN-100 and the sketch of the sample setting for determination of three strain components with a diagram of neutron scattering by a gauge volume

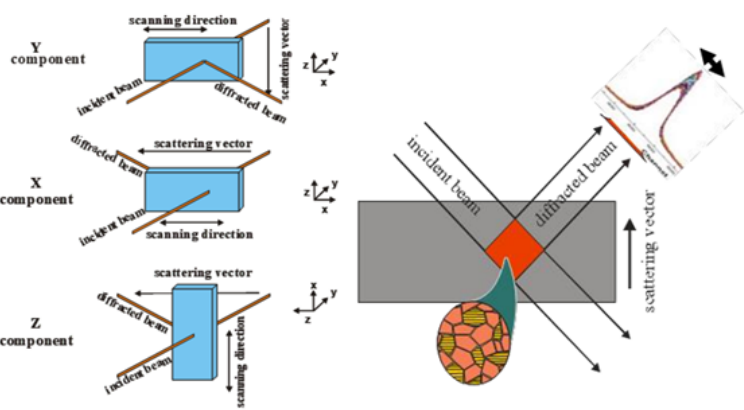
Obr. 4 Schematický obrázek zařízení SPN-100 pro měření deformace/napětí vzorků a náčrt poloh vzorku pro určení třech komponent elastické deformace s detailem rozptylu neutronů vymezeným objemovým elementem

The device measures strains which are then used for calculation of the stresses. Strain is defined as $\varepsilon = \Delta d / d_{0,hkl}$. Diffraction obeys the Bragg condition $2d_{hkl} \sin \theta_{hkl} = \lambda$, (d_{hkl} - lattice spacing, θ_{hkl} - Bragg angle, λ - the wavelength). The differentiation of the Bragg condition provides the strain as $\varepsilon = -\cot \theta_{hkl} \times \Delta \theta_{hkl}$. Q is the scattering vector perpendicular to the lattice planes. Conversion of the strains to stresses is given by

$$\sigma_x = \frac{E_{hkl}}{(1 - 2\nu_{hkl})(1 + \nu_{hkl})} \left[(1 - \nu_{hkl})\varepsilon_x^{hkl} + \nu_{hkl}(\varepsilon_y^{hkl} + \varepsilon_z^{hkl}) \right]$$

used for neutron topography and some special tasks of powder diffractometry in a short Q -range. The instrument is equipped with a robot-arm system for manipulation with the sample. The diffractometer has a changeable monochromator take-off angle and can be set and operated at a suitably chosen wavelength in the range 0.1 nm to 0.235 nm. In the case of α -Fe and γ -Fe samples, it usually operates at the neutron wavelength of 0.23 nm, providing a good resolution after diffraction on the α -Fe(110) and/or γ -Fe(111) lattices planes. Parameters of the instrument are:

- Monochromator: Bent Si(220) single crystal
- The range of the monochromator take-off angle: $30^\circ - 60^\circ$
- Neutron wavelength: 0.1 – 0.235 nm
- Neutron detection: 2-D Position sensitive detector with the spatial resolution $2 \times 2 \text{ mm}^2$
- Sample – detector distance: 1000 mm
- Beam defining optics: Cd slits in the incident and diffracted beams defining the gauge volume
- Specimen weight capacity: XYZ stage: up to 50 kg, robotic arm: up to 7 kg.



where $\varepsilon_{x,y,z}^{hkl}$ is the x,y,z-component of the lattice strain measured at the hkl crystal lattice planes, E_{hkl} and ν_{hkl} are the diffraction elastic Young modulus and diffraction Poisson ratio, respectively.

MEREDIT (Fig.5) medium-resolution powder diffractometer is designed for determination crystal and/or magnetic structure of powder or solid polycrystalline materials, analyzes their phase composition, phase transitions *in-situ* etc. The related measurements are complementary to the X-ray powder diffraction ones. The advantages of using neutrons consists in a deep sample penetration. Furthermore, neutrons can

"see" the light elements as a hydrogen and distinguish elements standing beside in the periodic table and permit possibility of the magnetic structure studies. Samples can be studied at the room temperature or at low and high temperature when situating it in the cryostat or vacuum furnace. The instrument is equipped with the multi-detector bank containing 35 ^3He point counters with corresponding 10' Soller collimators. They are all individually set at angular intervals of 4.00° covering the range of the scattering angles 2θ of 150 deg. There are several sample environments to enhance the measurement ability of the instrument: vacuum furnace able to cover the measurement from the room temperature up to $\sim 1000^\circ\text{C}$; light furnace covering the same temperature range (RT up to 1000°C) but in addition, it is possible to heat up the sample in different atmospheres, including open air, close cycle cryostat operating from room temperature down to $\sim 10\text{K}$; sample changer (carousel) - for up to six samples measured at room temperature. A special deformation rig permits *in-situ* measurements under uni-axial stress/pressure or fatigue cycles. For textured samples, it is also possible to mount up the Euler goniometer. For the powder samples, the vanadium containers with different

diameters - 6, 10 and 13 mm can be used. These containers can be used only for measurement in the carousel, vacuum furnace and the close cycle cryostat. Possible applications of the instrument are:

- Phase transformation in the cast iron during the tensile load
- Conduction path way in the oxygen conductors for fuel cell membranes
- Doping level of the hydrogen storage material
- In-situ chemistry during charging in the lithium batteries
- Magnetic structure of magneto-caloric compounds
- Magnetic structure of antiferromagnetic semiconductors for spintronic devices
- Base and corrosion materials in archaeological artifacts
- Twinning model in the magnesium alloys during tension and compression
- and many others

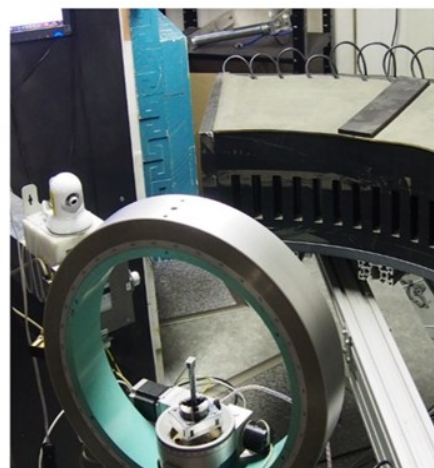
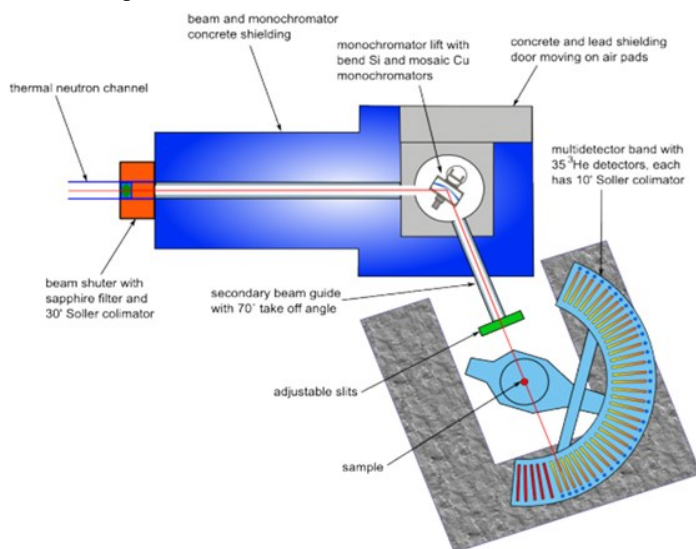


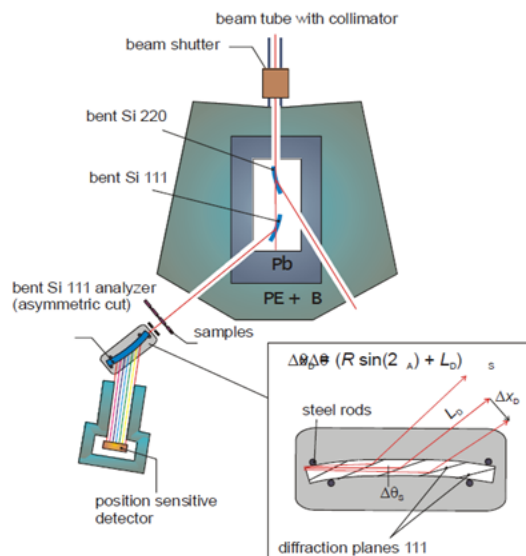
Fig. 5 Schematic picture and the photo of the sample and detector parts of the powder diffractometer MEREDIT

Obr. 5 Schematický obrázek a foto-detail pohyblivých částí kolem vzorku a části detektoru práškového difraktometru MEREDIT

MAUD (Fig.6) small-angle-scattering double-crystal diffractometer is designed for the measurements of small-angle neutron scattering (SANS) with a high momentum-transfer Q -resolution. The fully asymmetric diffraction geometry of the bent Si analyser is employed to transfer the angular distribution of the scattered neutrons to the spatial distribution and to analyse the scattering curve by the 2-d PSD. The remote control of the curvatures of the monochromator and analyser crystals makes possible to tune the instrument resolution easily in the δQ range from 10^{-4} to $10^{-3} \text{ \AA}^{-1}$, according to the expected size of structural or compositional investigated inhomogeneities. It is mainly used for investigations of the inhomogeneities (e.g. in alloys, porous media), cavities, fractal structures, precipitates etc. in the size range from 20-50 nm to several

μm . The instrument is equipped with the vacuum furnace capable to heat the sample up to 1400°C , automatic sample exchanger (up to 9 samples), deformation rig with maximum tensile/pressure load up to 20 kN, rotation table and electrical magnet up to 2 Tesla. Magnetic field can be applied to sample in horizontal as well as vertical direction with respect to the beam.

SANS is generally used for investigation of rather large objects of the average dimension from 1 nm to 10 nm: Complex fluids, alloys, precipitates, biological assemblies, ceramics, glasses, flux lattices, long-wavelength CDWs and SDWs, critical scattering, porous media, fractal structures etc. Bulk samples can be used (1 mm - 2 mm thick). SANS is sensitive to the light elements (H, C and N) as well as isotopes such as H and D. Overview of applications related to usually used methods – Fig. 7.



3 types of SANS techniques in neutron laboratories

	size range (R)
Pin-hole (conventional) instrument	1 nm ... 100 nm
<u>DBC diffractometer with bent crystals</u>	<u>10 nm ... 1 μm</u>
Ultra DC-SANS setting with perfect crystals	1 μ m ... 10 μ m

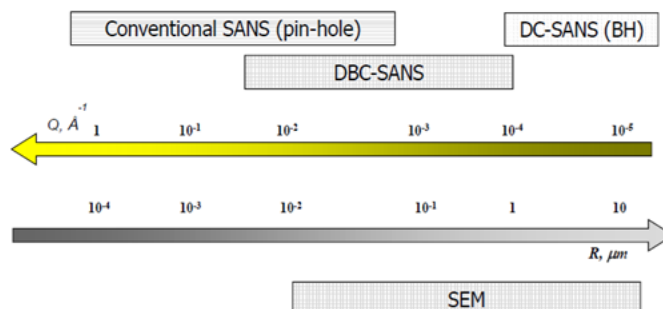


Fig. 6 Left: Schematic diagram of the DBC-SANS diffractometer MAUD. Inserted part shows the transformation of the scattering angular deviation on the position coordinate. Right: Q or R range comparison of the individual instruments

Obr. 6 Vlevo: Schematický obrázek maléhoúhlového difraktometru DBC-SANS. Vložená část ukazuje převod úhlových posunutí vlivem rozptylu na lineární prostorový rozměr. Vpravo: Porovnání oblastí měření parametrů Q nebo R pro jednotlivá zařízení

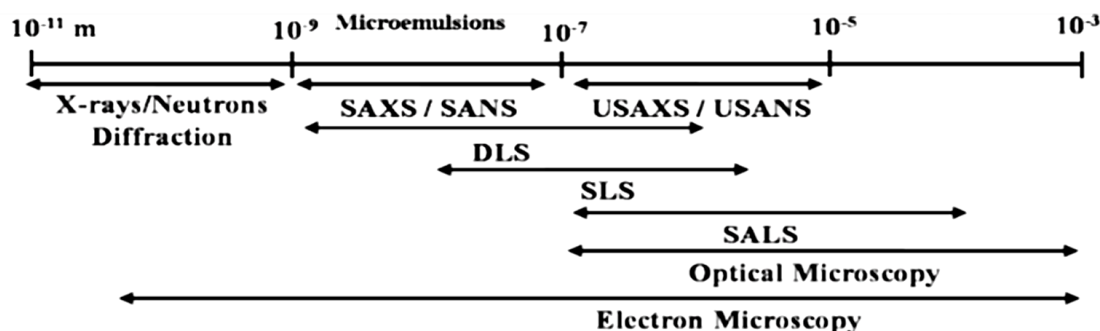


Fig. 7 Overview of applications related to usually used methods

Obr. 7 Přehled aplikací a oblastí jejich uplatnění při studiu částic různých rozměrů

3. Several related experimental results

TKSN-400 DIFFRACTOMETER (Fig. 8 – Fig. 11): As an example, we show the experimental results obtained on this instrument from in-situ studies of pearlitic steel wires (chemical composition 0.85 C, 0.24 Si, 0.82 Mn, 0.011 P, 0.01 S, 0.02 Al and 0.05 Cr (mass %)). Several samples were at a disposal: P1 - patenting by industrial process (pearlitic lamellar structure), P2 - drawing to wires (reduction area 75%), P3 - ageing at 423 K / 12.6 ks, P4 - 698 K / 0.6 ks (Zn-plating heat treatment), P5 - 963 K / 18 ks (cementite spherical particles). Other experimental examples can be found in ref. 3-5.

SPN-10 - RESIDUAL STRAIN/STRESS SCANNER (Fig. 12 – Fig.14): It can be said, that the strain/stress scanners can be found practically in every neutron scattering laboratory working, at least, at the medium-power research reactor. First, as an example the strain scanning through the 15 mm thick ferrite plate covered by the 5 mm thick austenitic protection plate welded on the

ferrite one. Other experimental examples can be found in ref. 6-8.

As an example of the use of the **MEREDIT - POWDER DIFFRACTOMETER** (Fig. 15, Fig. 16). **Fig. 15** shows powder diffraction spectra of several porous samples of High-Entropy Carbides (HEC): Two binary alloys HfC and TaC and then HEC1 - ((Hf,Nb,Ta,Ti,Zr)C and HEC2 - (Mo,Nb,Ta,V,W)C. It has been found that the structure of all of them is single phase Fm-3m. However, the lattice parameter of the individual samples is slightly different which results in the mutual 2 θ -shift of the peaks related to measured reflections. Similarly, Fig. 16 displays an example of the powder diffraction spectrum of the single γ phase of the TiAl alloy. Other experimental examples can be found in ref. 9-11.

MAUD – SANS DIFFRACTOMETER: As an example of the exploitation of the MAUD instrument, **Fig. 17** shows the result of the measurements of the porosity of earlier investigated HEC samples. The resolution of the SANS instrument MAUD allowed in this case to study microstructures (pores) in the size range R from 20 nm to

2 μm and thus the pores which are not within this range could not be characterized. The SANS measurements are treated in absolute scale and therefore, the volume fraction

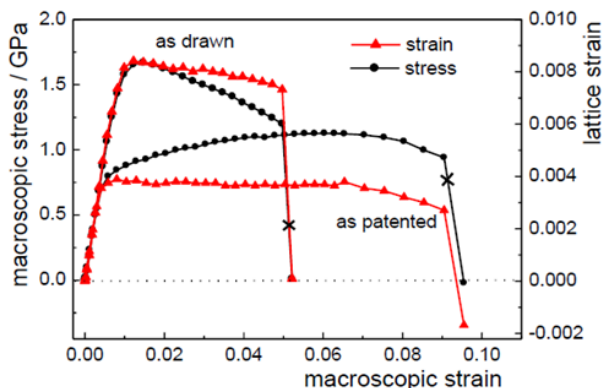


Fig. 8 Conventional strain/stress curves for two samples
Obr. 8 Typické křivky makrodeformace/napětí pro dva vzorky

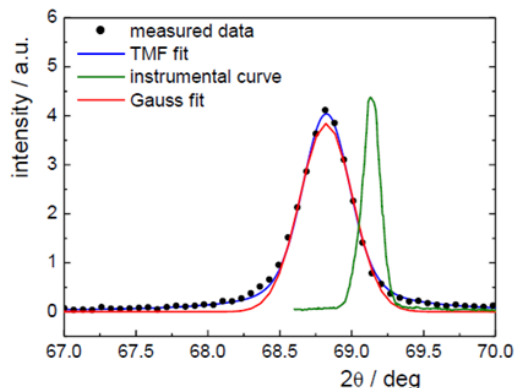


Fig. 9 Examples of the basic curves as used for the evaluation procedure
Obr. 9 Příklad základních profilů používaných k vyhodnocení

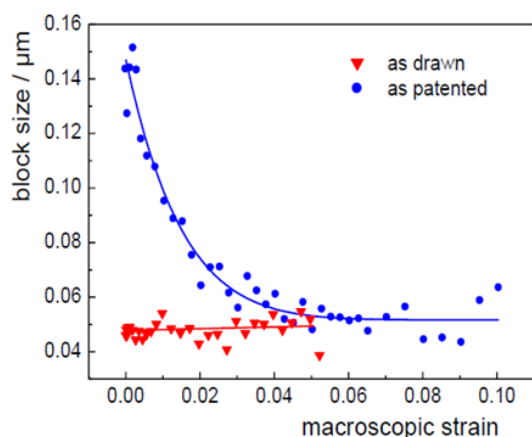


Fig. 10 Block size dependence on the macroscopic strain
Obr. 10 Závislost rozměrů zm od makrodeformace

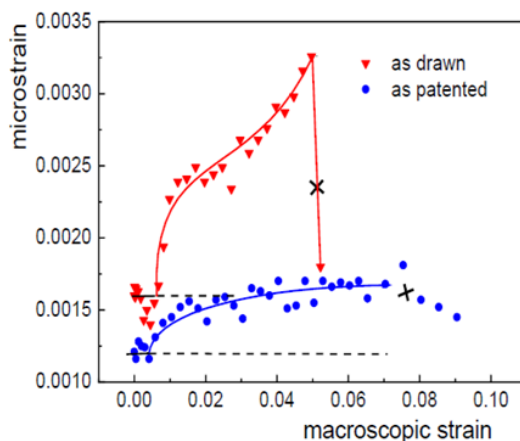


Fig. 11 Microstrain dependence on the macroscopic strain
Obr. 11 Závislost mikrodeformace od makrodeformace

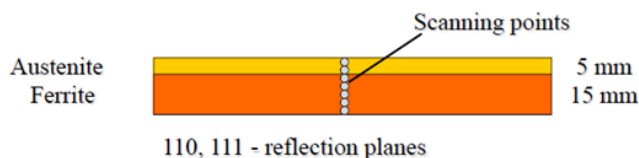


Fig. 12 Sketch of the austenite protection welded on the ferrite
Obr. 12 Náčrt austenitické ochrany navažené feritickou ocel

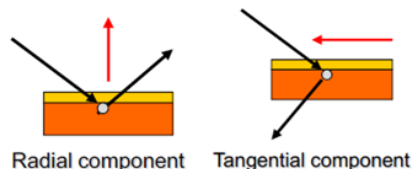


Fig. 13 Sketch of the measured strain components
Obr. 13 Náčrt měřených komponent elastické deformace

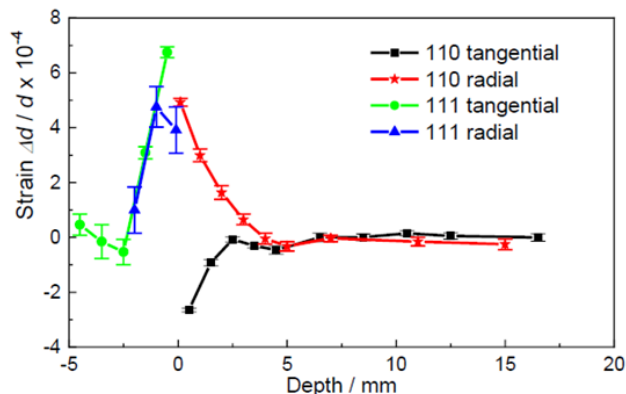


Fig. 14 Depth distribution of the strains in the vicinity of the weld
Obr. 14 Hloubkový profil elastické deformace v okolí sváru

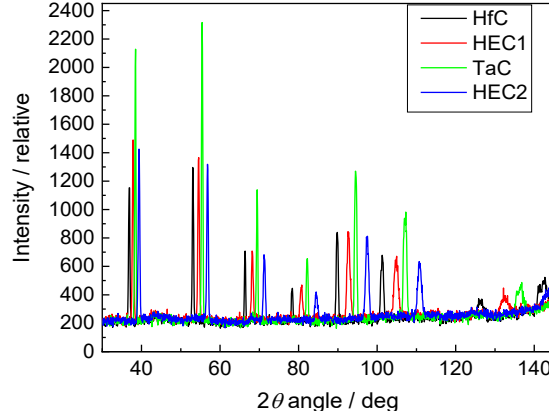


Fig. 15 Powder diffraction spectra related to the HECA samples
Obr. 15 Difrakční spektra (práškové) několika vzorků HECA

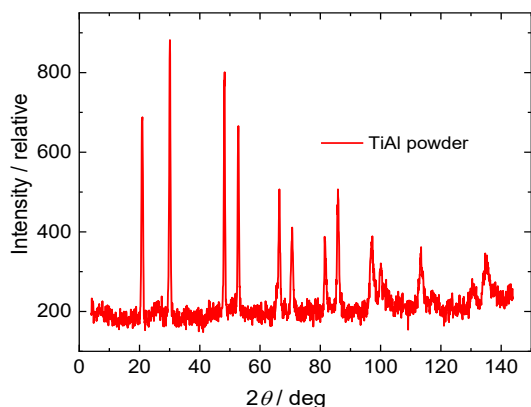


Fig. 16 Powder diffraction spectrum of the TiAl single γ phase
Obr. 16 Difrakční spektrum (práškové) u γ fáze slitiny TiAl

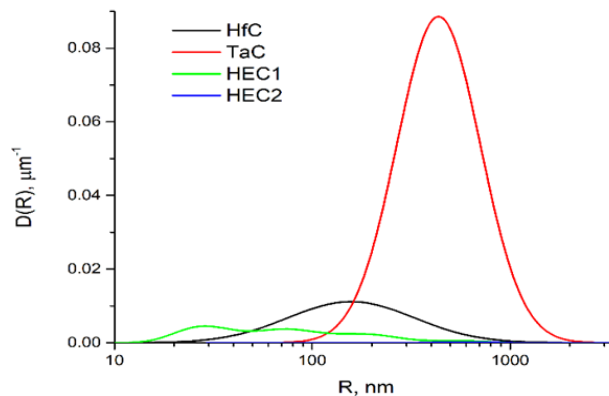


Fig. 17 Fitted volume weighed size-distribution of the pores for individual HEC samples for the SANS measurements
Obr. 17 Distribuce velikostí pórů pro jednotlivé vzorky HEC z měření SANS

Table 1: Summary the results of the evaluation procedure.
Tabulka 1: Souhrn výsledků hodnocené procedury

	HfC	TaC	HEC1	HEC2
Volume fraction, %	$0.425 \pm 0.019 \%$	$5.296 \pm 0.03 \%$	$0.103 \pm 0.011 \%$	$0.009 \pm 0.014 \%$
Mean radius of pores, nm	$355 \pm 28 \text{ nm}$	$621 \pm 3 \text{ nm}$	$261 \pm 35 \text{ nm}$	$720 \pm 71 \text{ nm}$
Model used	LogNormal distribution (w=0.49) of spherical pores	LogNormal distribution (w=0.49) of spherical pores	Spherical particles with size distribution of 7 splines	Spherical particles with size distribution of 7 splines

and size distribution of the pores can be fitted. The **Table 1** summarizes the results of the evaluation procedure. Other experimental examples can be found in ref. 12-13

4. Conclusion

Comparison of neutrons and synchrotron radiation as probes is follows:

1. Neutrons

- Particle beam (neutral)
- Interactions with the nuclei and the magnetic moment of the atoms
- Scattered by all elements, also the light ones like the hydrogen isotopes
- Deep penetration depth (bulk samples)
- Less intense beam measuring larger samples

Neutrons Applications:

- Magnetic structures & excitations
- Organic structures (the H-D isotope effect)
- Bulk studies (strains, excitations)
- Low-energy spectroscopy e.g. molecular vibrations

2. Synchrotron radiation (SR)

- Light beam (electromagnetic wave)

- Interactions with the electrons surrounding the nuclei (in the sample)
- Mainly scattered by heavy elements
- Small penetration depth (surface studies of samples)
- Very intense beam measuring small or ultra-dilute samples

SR Applications:

- Protein-crystal structures
- Fast chemical reactions
- Surface studies (defects, corrosion)
- High-energy spectroscopy e.g. measurements of electron energy-levels

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INFORMATIVNÍ ČLÁNEK

JFE Steel zadala společnosti Danieli zakázku na nový „Digimeltshop“ v Japonsku

3. července 2025

Společnost JFE Steel si vybrala firmu Danieli jako technologického partnera pro svou ekologickou transformaci a plán snižování emisí skleníkových plynů.

Danieli dodá novou, digitálně řízenou elektrickou obloukovou pec (EAF) a stanice sekundárního zpracování, které budou instalovány v závodě JFE Steel West Japan Works v Kurashiki. Zahájení provozu je plánováno na první čtvrtletí roku 2028. Půjde o jednu z největších elektrických obloukových pecí na světě z hlediska kapacity odpichu.

Celý článek na webu [Danieli.com](https://danieli.com)

Nový „Digimeltshop“ bude zahrnovat elektrickou obloukovou pec Digimelter® Zerobucket a zařízení Digirefiner, obě vybavené pokročilým napájecím systémem Q-One pro výrobu vysoce kvalitní tekuté oceli.

Primární odsávací vedení kouřových plynů zajistí účinné odprašení elektrické pece.

Napájecí systém Q-One zvyšuje efektivitu provozu snížením spotřeby energie a elektrod, minimalizací negativních dopadů na elektrickou síť a zvýšením provozní flexibility díky redundantní architektuře.

REDAKCE

Váš názor nás zajímá

Do rukou se vám dostává naše vůbec první online číslo. Jsme zvědaví, jak na vás působí – co vás zaujalo, co byste si rádi přečetli příště, a co vám případně chybí.

Budeme rádi, když se s námi podělíte o své dojmy a nápady. Jak se vám líbí první číslo? Co byste uvítali do budoucna? Pište nám o svých pocitech i podnětech, každý váš názor je pro nás důležitý.

Své připomínky, pochvaly i kritiku posílejte na adresu redakce: redakce@hutnickelisty.cz

Děkujeme, že jste u zrodu našeho časopisu a že nám pomáháte ho společně posouvat dál.