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The Use of Thermal Neutron Beams at Medium Power Reactor LVR-15 in Řež for Competetive Neutron Research

P. Mikula[1], P. Strunz[1]

[1]Nuclear Physics Institute ASCR, v.v.i. 25068 Řež, Czech Republic

E-mail contact of main author: mikula@ujf.cas.cz

Abstract. Neutron Physics Laboratory of NPI ASCR, v.v.i. operates several neutron instruments installed at the medium power reactor LVR-15 having a nominal thermal power of 10 MW and administrated by Research Centre Rez, Ltd. The following instruments are succesfully operated at the reactor: High resolution SANS diffractometer, Strain/stress scanner, Instrument for thermomechanical testing of materials, Thermal neutron depth profiling, Medium resolution powder diffractometer, Neutron optics diffractometer and Neutron activation analysis. On the dedicated instruments there are carried out research investigations on a competitive international level. Description of the most important recent research activities accompanied by several highlight results are introduced in the paper. As the reactor operates on average about 170 days per year with a pattern of operating cycles of three weeks, each followed by one week for maintenance and instrumentation development, it provides a sufficiently high number of experimental days.

Key Words: Medium power reactor, research with thermal neutrons

1. Introduction

After the last reconstruction and upgrading finished at the end of the year 1990, the light water medium-power research reactor LVR-15 achieves the parameters equivalent to similar research reactors in the world. This reactor having a nominal thermal power of 10 MW, a neutron flux rate of about 1 x 10^{14} n.cm^{-2}s^{-1} in the core, and several horizontal and vertical irradiation channels, is at present one of the several neutron sources in Central Europe providing attractive research possibilities for the basic as well as applied neutron research [1,2]. In total, Neutron Physics Laboratotry 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. For details see the Web page http://neutron.ujf.cas.cz/cs/instruments/lvr15. Thermal neutrons from the reactor are well suited for interactions with investigated samples, and also are ideal tools for the study of the structure and physical properties of condensed matter and particular problems of nuclear and fundamental physics and applied research. Photo of the reactor and a schematic layout of neutron scattering instruments of NPI are in Fig. 1, where only the diffractometer installed at the channel HK-2 belongs to another institution. A level of the instruments of NPI and the carried out research investigations have opened the door for joining the access programs within EU projects. The corresponding instruments are in other laboratories either overloaded by user demands (e.g. strain diffractometer and powder diffractometer) or are rarely provided (high-resolution SANS, T-NDP and NAA). The following instruments and examples of the experimental studies document high quality experiments of basic, interdisciplinary as well as applied research which can be effectively carried out.

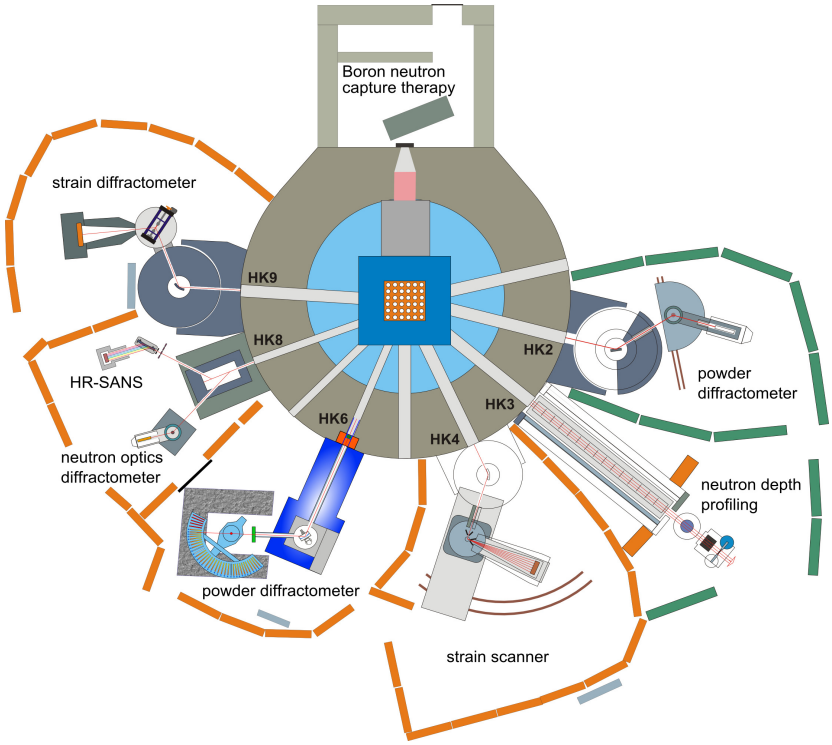

FIG. 1. Photo of the LVR-15 reactor operated by Research Centre Řež, Ltd. and schematic sketch of neutron scattering instruments installed at the reactor.

2. Experimental activities at the reactor LVR-15

2.1. Thermal Depth Pofiling (TNDP)

Thermal Neutron Depth Profiling is a nondestructive technique for determining depth profiles of selected light elements (i.e., ^{3}He, ^{6}Li, ^{10}B, ^{14}N, etc.). The technique is based on specific nuclear reactions induced by thermal neutrons, and accompanied by the emission of charged particles as reaction products. The technique was first introduced in 1972 by Ziegler et al. [1] and later used in several laboratories. In NPI Rez the technique was established at the reactor in the 80s. Since that time, the method has been further developed from the 1D set-up mode with a single Si-detector to the 2D mode with two Si-detectors in a sandwich arrangement [2]. Similarly to NIST, recently, the 3D technique has been also tested when using spectroscopic multipixel detectors (TimePix). In general, the sensitivity of the TNDP method depends on the cross-section of the nuclear reactions in use, intensity and purity of the thermal neutron beam, and the efficiency of the spectrometric system. The quality of the neutron beam (assessed by the Cd ratio) can be improved using neutron guides and neutron filters, which can substantially reduce the background level. The efficiency of the system can also be improved electronically, e.g., by pulse-shape discrimination [3], or by detection of both reaction products using large angle coincidence spectroscopy [2,4].

In general, TNDP is a non-destructive method that leaves only trace amount of residual radioactivity, and examined samples can thus be measured repeatedly. Concentrations down to a ppm (with a 1D mode) or even ppb (with a 2D mode) level can be determined, depending on the element and the matrix. Profiling to depths of about 15 μm (e.g. Li in metals) or even 60 μm (Li in polymers) can be obtained, with a depth resolution to a few nano-meters only (for glancing angle geometry). The examined samples have to be solid (or liquid with very low volatility), flat with a smooth surface (with roughness of few nm only) and minimum area

of at least a few mm^2. Depending on the nuclides and the used substrates the analysis takes a few tens of minutes to a few tens of hours. The TNDP technique is applicable only to the elements with a relevant cross-sections and energy of reactions [5].
The TNDP method is routinely and effectively utilized for the study of relevant elements and their spatial distributions in various materials (polymers, metals, diamonds, etc.) and technological instruments (Li batteries, biosensors, bio-implants, etc.). As a novel approach for study of nanostructures, spectroscopic, position-sensitive detectors have been applied for the first time. The study was performed on Li structures deposited on a thin polymeric foil (PET, 1.5 μm) through a mask an ion beam sputtering procedure. Fig. 2 shows the TNDP distribution of Li atoms measured by two TimePix detectors in a sandwich arrangement with the sample. The observed Li contours represent the reconstruction of the Li nanostructure on the PET foil that was obtained by evaluation of coincidence events.

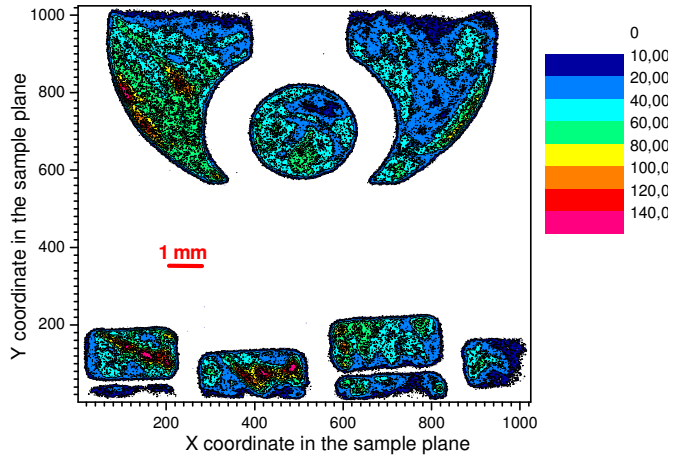

FIG. 2. Distribution of Li atoms (sputtered on a 1.5 μm thick PET foil through a mask) measured by 2 TimePix detectors in a sandwich arrangement. The Li contour reconstruction was obtained by the evaluation of coincidence events.

2.2. Neutron Activation Analysis

At NPI, several modes of neutron activation analysis are maintained, namely instrumental neutron activation analysis (INAA), epithermal neutron activation analysis (ENAA), and radiochemical neutron activation analysis (RNAA). Irradiations are carried out in vertical channels of the LVR-15 reactor, in which thermal neutron fluence rates $2 – 4 \cdot 10^{13}$ cm^{-2} s^{-1} are available. One of these channels is equipped with a pneumatic transfer facility with a transport time of 3.5 s to the gamma-spectrometric laboratory. NAA procedures are available for determination of up to 60 elements using both relative and k_0 standardization in various matrices. The NAA methods can be complemented by photon activation analysis (IPAA) using irradiation by bremstrahlung with energies 8 – 25 MeV obtained from a MT-25 microtron, if needed. Recently, the NAA methods have been further developed and applied in various fields of science and technology.

INAA with k_0 standardization was validated by analysis of a set of synthetic multi-element standards SMELS and matrix standard reference materials of the US NIST SRMs [6]. Quantification of the results was performed with Kayzero for Windows programme showing surprisingly different uncertainties with respect to k0–IAEA. Therefore, a novel Excel spreadsheet technique according to Kragten to evaluate uncertainty of k_0-NAA results has been devised [7]. In order to match specific conditions of irradiation in the LVR-15 reactor in Řež (irradiation channels located within the Be reflector), new sets of neutron flux monitors for both short- and long-time irradiation that consist of the elements Au+Mn+Rb and Au+Mo+Rb, respectively, have been designed [8,9]. In the field of geo- and cosmochemical research, a representative set of 160 samples of the Central European tektites - moldavites - covering the main parts of the Central European tektite strew field, supplemented with samples of tektites and impact glasses from the strewn fields in other parts of the world, was characterized by various analytical methods dominated by methods of NAA. Supporting evidence for our new theory, which assumes participation of plant biomass in the source

materials for moldavites, was obtained [10]. We also used INAA to determine contents of more than 30 elements in meteorites Morávka [11] and Jesenice [12]. Environmental research was focused on the determination of ^{129}I and the $^{129}I/^{127}I$ ratio in biomonitors, namely in bovine thyroid and moss, collected in the vicinity of the Temelín nuclear power plant (NPP) in south Bohemia using NAA in several modes (NAA with pre-irradiation separation followed by RNAA, and ENAA). No significant differences of ^{129}I levels and the $^{129}I/^{127}I$ ratios in the thyroids collected prior to the start and after several years of the NPP operation have been indicated [13]. In another environmental study, the accuracy of Hg determination in polluted soil samples by atomic absorption spectrometry with an AMA-254 spectrometer using small sample masses was confirmed by assay of much larger, otherwise identical samples by our RNAA procedure [14]. For agricultural and nutritional research, we used a RNAA procedure to study the Se-transfer from soil or seed to wheat plants [15] and the ability of bread and durum wheat to accumulate Se via a soil-addition procedure at sowing time [16] to increase the desired uptake of the element in the Portuguese population. Silicon is an important trace element in humans, because it reduces the absorption of aluminium in human gastrointestinal tract. The daily intake of silicon should be about 10–25 mg, and its most readily absorbable form is H_4SiO_4, which is contained in beer. Using INAA, we found that Si-concentrations in Czech lager beer(s) varied in the range of 13.7 – 44.2 mg L^{-1} [17]. Concerning the cultural heritage, in 2010, the grave of the famous astronomer Tycho Brahe was opened by a Czech–Danish research consortium and samples of his bones, hair, and teeth were procured for scientific investigation. We carried out mercury determination in segmented hair samples by RNAA. The results showed that in the last 2 months of Brahe´s life, he was not exposed to lethal (or fatal) doses of mercury, as was previously speculated [18]. Furthermore, graphene is another example of a material difficult to assay by classical analytical techniques. Therefore, elemental impurities were determined by INAA in graphene samples prepared by various oxidation procedures of graphite to graphite oxide followed by various reduction processes [19]. On the corresponding webside one can find many other NAA results usually taken in international collaboration.

2.3. Strain/stress scanning in polycrystalline materials

Neutron diffraction permits to measure very accurately the distances between atoms and their changes due to elastic deformation. Neutrons are therefore very well suited as a probe for non-invasive determination of residual stresses in large material depths up to several centimetres. This is very important for the assessment of structural integrity and lifetime of critical engineering components, by evaluation of stresses developed during material processing (e.g. welding, rolling or case hardening) or long term use. The dedicated two-axis diffractometer installed at the channel HK4 is equipped with bent Si and Ge perfect single crystal monochromators which are easily changeable according to the experimental requirements. The diffractometer is usually used for macro/micro strain scanning of polycrys talline materials. The diffractometer uses advantages coming from focusing both in real and momentum space and yields good resolution and luminosity especially for samples of small dimensions [20]. The resolution properties of the device are reached in a limited range of momentum transfer for which the focusing conditions are optimized. The optimization can be done easily by using a remote control of the curvature of the monochromator. A new six-axis robotic arm has been recently installed which allows more flexible manipulation of complex samples and automatisation of the strain/stress measurements. The diffractometer has a changeable monochromator take-off angle and can be set and operate at a suitably chosen - neutron wavelength in the thermal neutron range from 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 0.235 nm, when providing a maximum detector signal and good resolution after diffraction on α-Fe(110)

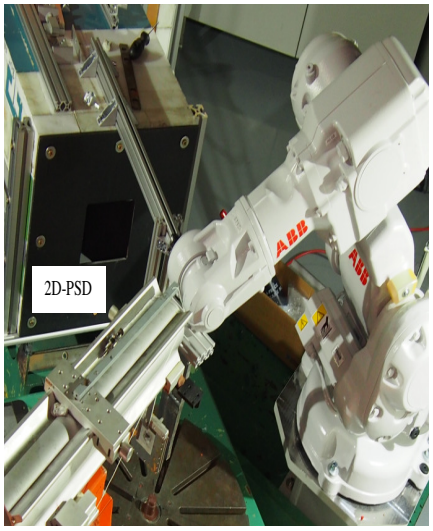

FIG. 3. Photo showing the ABB robot system and 2D-PSD detector.

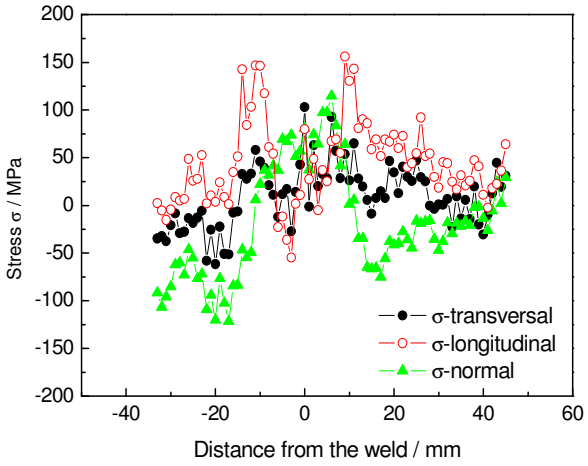

FIG. 4.. The experimental results of stresses versus the distance from the weld as measured on the 12 mm thick welded α-Fe plate.

and/or γ-Fe(111) lattice planes. By recent installation of the robot system and the 2D-PSD (20x20 cm^2, 2 mm spatial resolution), the acquisition of the data has been increased by a factor of 4. For the strain scanning of the sample, the gauge volume is determined by two fixed Cd slits (2-5) mm x (3-30) mm in the incident and diffracted beams and a *x-y-z* stage is used. The instrument is remotely controlled by PC. The photos of the basic components of the instrument as well as the first experimental results obtained on the welded test-sample are shown in the following figures. As a sample we used a 12 mm thick α-Fe welded plate of the dimensions 234 mm (length) x 50 mm (width) x 12 mm (thickness). The thickness at the welding point was 15 mm. The weld area extended to approx. 10 mm on one side and 20 mm on the other side. As recent highlights one could mention the following experimental studies:

- ***High Strength Steels Welds***, when residual stresses were studied with the aim to find optimum composition of the additive material in order to decrease residual stresses in the vicinity of the foot of the welding joint and consequently to increase the fatigue strength [21].
- ***Near-surface residual stresses steel coated by tungsten carbide,*** when residual stresses were studied in inoxidizable martensitic steel 13Cr4Ni with the surface hardened either by tungsten carbide coating. The achieved results gave indications which can be used in the monitoring of the coating characteristics, in particular the adhesion [22].
- ***Correlation of magnetic properties and residual stress distribution in welded steel,*** when residual stress distribution in the welded AISI 1008 steel sheets was monitored by the X-ray and neutron diffraction technique. Alternatively, surface and bulk magnetic properties, namely Barkhausen noise and quasi-dc permeability, were measured in the same material. The study showed good agreement between the diffraction and magnetic measurements, which is important as an independent validation of the magnetic methods [23].

2.4. High Resolution Diffraction for Materials Research

In addition to the diffractometer described in paragraph 2.3, another high-resolution two-axis diffractometer optimized for investigation of elastic and plastic deformation studies in polycrystalline materials is installed at the channel HK-9. The instrument is used especially for 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 of the studied material which is easy to correlate with the parameters of external loads. The obtained microstructural parameters of the examined material can be directly

compared with the parameters of micromechanical models. This approach brings a deeper understanding of processes ongoing in materials upon deformations, thermal treatments or phase transformations. The instrumental parameters are as follows: Horizontally and vertically focusing monochromator employing elastically bent Si single crystals, neutron wavelength 1 Å $\leq \lambda \leq$ 2.7 Å, neutron flux at the sample position 10^5 n.cm^2.s^{-1} at λ=2.3 Å, angular range of scattering angles $25° < 2\theta < 90°$ and resolution $2.10^{-3} \leq \Delta d/d \leq 3.10^{-3}$. The following sample environments are at a disposal: Deformation machine for uni-axial loading (tension, pressure) ± 20 kN, resistance heating (T < 1200°C) or hot-air heating (T < 300°C), miniature deformation machine for uni-axial loading (tension, pressure) ± 10 kN, Eulerian cradle: inner diameter of 400 mm, $0° < \chi < 160°$, $0° < \omega < 360°$ and deformation machine for bending loading, maximum cycling frequency of 27 Hz. The neutron signal is recorded by 2-dimensional position sensitive detector. The above described methods have been recently mainly applied to the investigation of deformation mechanisms of *magnesium alloys*, including the innovative application of acoustic emission method simultaneously with neutron diffraction. Complementary dataset about the loading mode dependence of twinning was obtained, since acoustic emission is sensitive to twin nucleation whereas diffraction to twin growth. Theoretical calculations of Schmid-factor dependence of twin nucleation complete the experimental results which clearly explain the different behaviour of this material in tension and compression [24].

2.5. Neutron Powder Diffraction

The medium resolution powder diffractometer (MEREDIT) installed at the channel HK-6 consists basically of 3 changeable monochromators placed in a massive shielding, two large HUBER goniometer circles and a multi-detector bank which is mounted in a moulded neutron shielding made from boron carbide powder in epoxy resin. The bank contains 35 ^{3}He counters with corresponding 10' Soller collimators. The detector bank moves on air pads, which provides together with the stepping motor smooth positioning of this heavy loaded bank. Diffraction patterns can be collected in the angular range from 2° to 148° in $2\theta_S$ with step size down to 0.02° and step delay controlled by strict time or neutron flux read by monitor. Monochromator and beam parameters are shown in Table III. The diffractometer is mainly used for non destructive structure phase identification, crystalline structure determination, magnetic structure determination, temperature dependent phase transition, quantitative multi-phase analysis and also for in-situ internal stress-strain evolution. The following sample environments are at a disposal: close cycle cryostat for 4 K → 300 K, vacuum furnace for 300 K → 1300 K, light furnace for 300 K → 1300 K, Euler goniometer, automatic 6 samples echanger for RT and a deformation rig (uni-axial tension/compression, ~20 kN) and Euler

TABLE 1 – PARAMETERS OF SECONDARY NEUTRON BEAMS AVAILABLE FOR MEASUREMENT ON MEREDIT INSTRUMENT.

Monochromator	**Reflection**	**Wavelength (Å)**	**Minimum Δd/d (x10^{-3})**	**Neutron Flux (n.s^{-1}.cm^{-2})**	**Beam size (cm^2)**
bent Si single crystal	422-asym	1.2691	3.8 (at 57° 2θ)	≈ 7 × 10^5	2.5x4.5
	311-sym	1.8762	4.0 (at 61° 2θ)	≈ 8 × 10^5	2.5x4.5
bent Ge single crystal	422-sym	1.9525	5.1 (at 67° 2θ)	≈ 9 × 10^5	2.5x4.5
	311-asym	1.3216	4.2 (at 62° 2θ)	≈ 8 × 10^5	2.5x4.5
3 mosaic Cu crystals	220-sym	1.460	4.9 (at 71° 2θ)	≈ 4 × 10^6	4.5x4.5

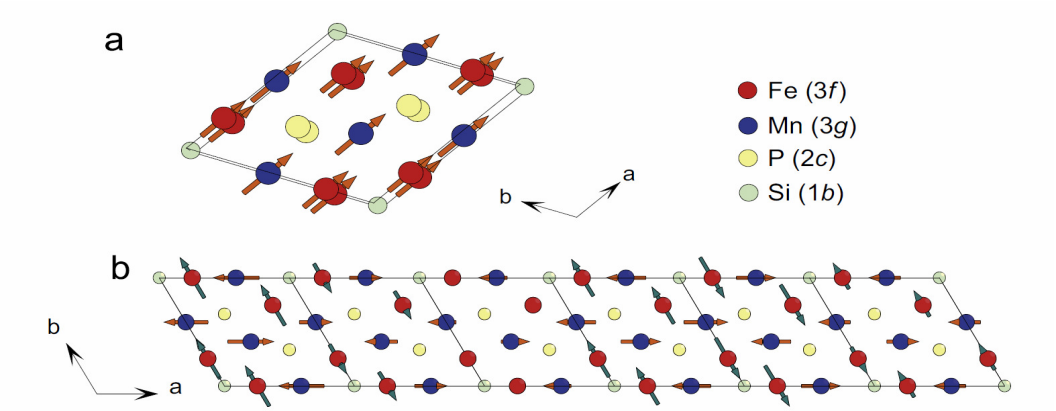

FIG. 5. Two very similar structural phases were found in the sample $FeMnP_{0.75}Si_{0.25}$ with completely different magnetic structure upon cooling – the ferromagnetic structure (a) and the antiferromagnetic and incommensurate (q_x=0.363(b)). The magnetic moments of the Mn and Fe atoms in (b) are aligned in the basal plane along the a- and the b-axis, respectively, and the amplitude of the moments propagates sinusoidally along the a-axis.

goniometer (texture measurement, crystal orientation). As an application example, Fig. 5 shows the result of the determination of magnetic arrangements when in special cases of the phase diagram in $FeMnP_{1-x}Si_x$ system and following structural evolution with temperatures was only possible by neutron powder diffraction. Such results play an important role in materials science and help to develop, optimize and tailor material properties for application needs. The diffraction methods can reveal not only crystallographic structure, but also bulk microstructure characteristics such as the size and preferred orientation of constituent single crystal domains. Other examples of the research carried out by powder diffractometer are: Lithium ion battery test cell for in-situ neutron diffraction measurements when real-time monitoring the structural changes in the battery material during the charge-discharge cycle [25]. Other attractive studies of structural properties were done on the samples of graphene, when the thickness and lateral parameters of the graphene sheets were determined [26]. Neutrons are particularly well suited for studies of magnetically ordered materials. Recently, magnetic structure of magneto-caloric material FeMnP0.5Si0.5 with enhanced magneto-caloric properties and contributions of different individual mixed sites (Fe/Mn) to overall magnetic structure have been determined [27]. The neutron powder diffraction experiments are often complemented by the large-scale microstructure SANS measurements, namely in the case of Co-Re-based alloys.

2.6. Small-Angle Neutron Scattering (SANS)

SANS investigations are carried out on the double-crystal diffractometer (MAUD) designed for the measurements in the high Q-resolution range. In contrast to conventional double-crystal arrangements, the fully asymmetric diffraction geometry on the elastically bent Si analyzer is employed to transfer the angular distribution of the scattered neutrons to the spatial distribution and to analyze the whole scattering curve by a one-dimensional position sensitive detector (see Fig. 6) [28]. It reduces the exposition time per sample typically to 0.5-5 hours (depending on the Q-resolution and sample cross-section). The remote control of the curvatures of the monochromator and analyzer crystals makes possible to tune the instrument resolution in the ΔQ range from 10^{-4} to 10^{-3} Ă$^{-1}$, according to the expected size of investigated inhomogeneities. An absolute calibration of scattering cross-sections is possible by measuring the intensity of the direct beam (no calibration samples are required). The instrument operates in fully automatic mode, including sample exchange.

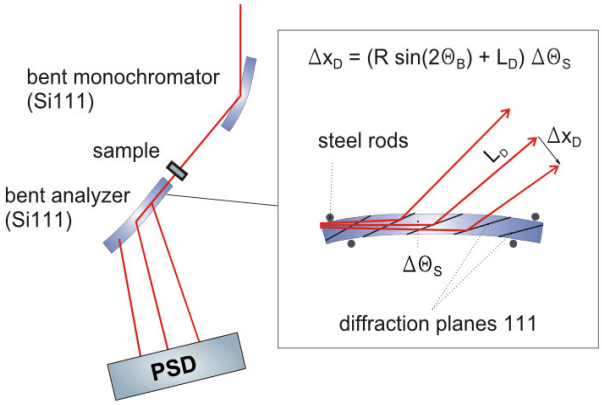

FIG. 6. Schematic sketch of the double-crystal SANS diffractometer operating in combination with PSD.

The SANS method provides valuable information about material microstructure in a wide range of size scales ranging from units of nanometres to several microns. Covering this broad range requires the combination of measurements at various types of SANS instruments. Our SANS diffractometer is unique in covering the gap between about 50 nm and 1 μm, which is not efficiently accessible by other existing SANS instruments. As to the investigations, namely, large precipitates in alloys (superalloys), porous materials (superplastic ceramics, ceramic thermal barrier coatings), nano-particles in ceramic-intermetallic compounds (MoSi2 with Si3N4 and SiC particles) and large inhomogeneities in polymers/microemulsions, etc. are investigated. As application examples let us introduce the following chosen materials: Precipitate studies of Co–Re alloys, which are good candidates for new high-temperature materials for future gas turbines. For the first time, in-situ neutron diffraction at high temperatures was used to investigate phase transformation in a Co–Re base alloy developed for the application at high temperatures [29,30]. Further important results were achieved in the research of Ni-base superalloys where an additional γ′ precipitation with slow kinetics was detected and characterized (formation, dissolution, kinetics) in IN738LC high-temperature superalloy for the first time [31,32]. The Al-Pb binary system as a metal-matrix composite is a suitable model system for testing liquid phase dispersion strengthening in bulk materials for structural applications. SANS measurements during the subsequent in-situ thermal cycling enabled to monitor changes of morphology of the Pb particles and their solid-liquid phase transition [33].

Using a selective phase dissolution technique, nano-porous membranes can be produced from simple two-phase metallic CMSX4 superalloys, which contain through-thickness elongated channel-like pores of only a few hundred nanometre in diameter. For the optimization of the production of porous membranes, knowledge of the pore-depth dependence on the etching time was desirable (see Fig. 7 and 8). Complementary SANS experiments carried out together with HZB Berlin enabled us to determine microstructural parameters of the membrane (pore-to-pore distance, raft thickness, pore volume fraction,

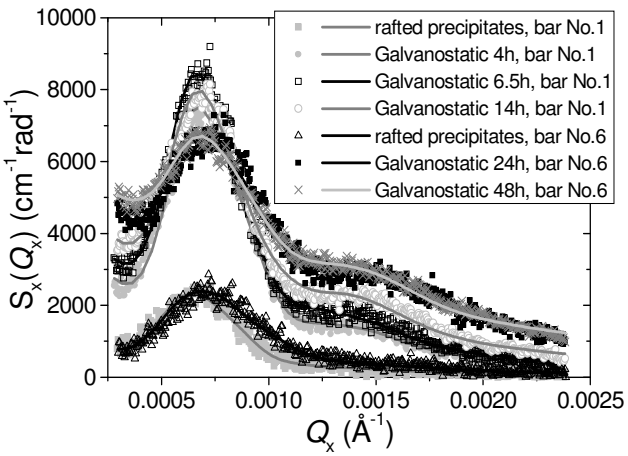

FIG. 7. Measured and fitted scattering curves of CMSX4 samples (SANS diffractometer).

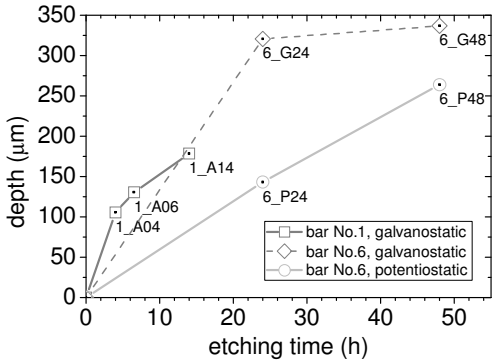

FIG. 8. Dependence of the pore depth on the etching time.

specific interface, pore depth) and, through contrast variation, to measure kinetics of pore filling by liquid and their subsequent emptying by evaporation [34].

3. Summary

It has been demonstrated on a few examples that on medium power research reactor LWR-15 in Řež high quality experiments of basic, interdisciplinary as well as applied research can be effectively carried out on a international competetive level. The research can cover a large scale of experimental investigations related to structure studies of new materials, structure phase transformations, chemistry, material testing, industrial product qualification, tracing of elements (e.g. in enviromental, chemical, geological and biological samples), cultural heritage etc. Some more details about the research in NPL can be found on the web page http://neutron.ujf.cas.cz/en/npl/research. Finally, it should be pointed out that all instruments of NPI are opened to external users and the measurements can be free when a new proposal is submitted through CANAM-ACCESS project (see http://neutron.ujf.cas.cz/en/instruments/user-access/.

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