

# Recenzované výzkumné a vědecké články

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## On the Measurements of Residual Stresses in Poly-crystalline Metallic Materials by Neutron Diffraction

### Měření zbytkových napětí v polykrystalických kovových materiálech neutronovou difrakcí

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#### Abstract

*A neutron diffraction method for mapping of residual strains/stresses inside poly-crystalline materials which is not well known in some sectors of industry, is introduced. The method routinely used on the dedicated instrument of NPI ASCR installed at the 10 MW research reactor and operated by the Research Centre Řež, is offered for external users free. Typical examples are measurements of residual strains/stresses developed e.g. in the course of welding or strains accumulated during processing and/or by fatigue of instrument components after usage after some operation. Recommended thickness of the samples is in the range from 3 mm to 20 mm and the specimen weight up to 50 kg. The experimental data are elaborated by the responsible person and then basic figures and graphs are prepared for the user. In addition, thanks to variety instrument environment, for specially prepared samples, in-situ measurements on samples subjected to external thermo-mechanical load can be carried out and thus, e.g. to study the deformation and transformation mechanisms. Principles of this enhanced method and an example of internal strain/stress measurements in the vicinity of welds are demonstrated on two welded samples.*

**Keywords:** residual strain/stress, neutron diffraction, polycrystalline metals, welds

#### Abstrakt

*Príspevek uvádza metodu neutronovej difrakcie pro mapování zbytkových deformací a napětí v polykrystalických kovových materiálech, která v některých průmyslových odvětvích není příliš známá. Tato metoda, běžně používaná na specializovaném přístroji v NPI ASCR instalovaném u 10 MW výzkumného reaktoru a provozovaném Centrem výzkumu s.r.o. v Řeži, je externím uživatelům nabízena bezplatně. Mezi typické příklady patří měření zbytkových deformací a napětí vzniklých například při svařování, nebo deformací nahromaděných během zpracování a/nebo v důsledku únavy součástí přístrojů po určité době provozu. Doporučená tloušťka studovaných vzorků se pohybuje v rozmezí 3 mm až 20 mm a hmotnost vzorku může činit až 50 kg. Získaná experimentální data jsou analyzována odpovědným pracovníkem a následně jsou pro uživatele připraveny základní tabulky a grafy. Kromě toho lze díky širokému přístrojovému vybavení provádět měření in-situ na speciálně připravených vzorcích vystavených vnějším termomechanickým zatížením, což umožňuje například studium mechanismů deformace a transformace. Principy této pokročilé metody a příklad měření vnitřních deformací a napětí v blízkosti svarů jsou demonstrovány na dvou svařovaných vzorcích.*

**Klíčová slova:** zbytkové elastické deformace a napětí, neutronová difrakce, kovové polykrystaly, svary

## 1. Introduction

Residual stresses and distortions are important factors influencing microstructure of the material and thus, the mechanical and life properties of manufactured component. We can often meet with them in welds and their vicinity, after a thermo-mechanical load, inhomogeneous distribution of alloying elements in the material, time of a working operation etc. Many investigations have been performed in order to determine the nature and also the consequences on the properties of individual polycrystalline structures. It has been recognized that in the case of welded materials the level of residual stresses can be affected by the welding process and welding procedure. It is clear that the estimation of residual stress levels in material components is very demanding. There is a number of experimental methods that can be used in dependence on the particular experimental conditions [1,2]. When focusing on the welds, several of them, including the hole drilling method using special strain gauges, ultrasonic measurements as well as X-ray and neutron diffraction can be used to measure the stress level after welding. Both diffraction methods (X-ray or neutron) can provide information about the stress distribution within the studied material and offer the unique way for advanced (complementary) nondestructive investigation of engineering materials. Contrary to X-rays usually used for near surface examinations, the advantage of using neutrons as a probe lies in the fact that the neutrons can penetrate deeply (several millimeters) into most of materials and thus provide a bulk information about their properties [1,2]. Therefore, the neutron diffraction method is very well suited to non-destructive 3D mapping of residual stresses. There has been already found a powerful application in texture studies and radiography/tomography. Moreover, the method can be used for the in-situ investigations of the material strain/stress behavior under external thermo-mechanical load and in many cases this method provides information which cannot be obtained by other methods [3,4]. It appears attractive in the case of multiphase materials when looking at the crystalline lattice, one can study structural details of individual phases. Conventional neutron strain scanners are in fact powder diffractometers optimized for such measurements and provide  $FWHM(\Delta d/d)$  – resolution related to the diffraction profiles (see the insert in **fig. 1**) of about  $(5-10) \times 10^{-3}$  which is sufficient for macrostrain studies. If the diffractometer resolution is higher, even microstrain studies of materials e.g. in the plastic deformation region could be carried out, when the presence of microstrains and the grain size affect the diffraction line profiles.

## 2. Principle of the neutron diffraction method

When illuminated by radiation of wavelength similar to the interplanar spacing ( $0.5-3 \text{ \AA}$ ), crystalline materials elastically and coherently scatter this radiation as distinctive Bragg peaks imaged usually by a position sensitive detector (PSD). In neutron and X-ray diffraction the angular positions of the diffraction maxima are directly related to the values of the lattice spacing through the Bragg equation  $2d_{hkl} \cdot \sin \theta_{hkl} = n \cdot \lambda$  ( $d_{hkl}$  - lattice spacing,  $\theta_{hkl}$  - Bragg angle,  $\lambda$  - the neutron wavelength) (see **fig. 1** and its insert). The diffraction peak related to the chosen gauge volume is observed at the angle of  $2\theta_{hkl}$  from the direction of the incident beam. When a specimen is strained elastically, the lattice spacing changes and this elastic strain results in a shift in the value of  $2\theta_{hkl}$  (for a particular reflecting plane illuminated by a fixed wavelength).

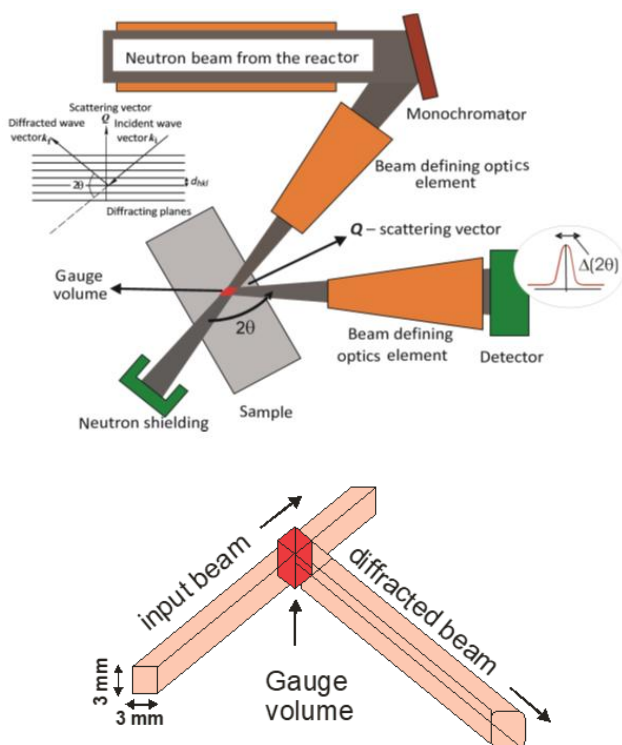
The principle of the neutron diffraction method is quite simple. It consists in the precise determination of the  $d_{hkl}$ - spacing of particularly oriented crystal planes [1,2].

However, for determination of the strain tensor, the particular diffraction geometry provides only one strain component perpendicular to the reflecting planes (i.e. parallel to the scattering vector  $\mathbf{Q}$ ). When defining the strain  $\varepsilon$  as  $\varepsilon = \Delta d / d_{0,hkl}$  ( $d_{0,hkl}$  is the lattice spacing of the strain-free material), thus it is related to a relative change  $\Delta d$  in the lattice spacing with respect to  $d_{0,hkl}$ , i.e. to a component parallel to the scattering vector  $\mathbf{Q}$  which is perpendicular to the reflecting set of planes. Therefore, the knowledge of the  $d_{0,hkl}$  value is a crucial task [3]. It should be pointed out that the shift in the Bragg angle (relative to that of the stress-free material) permits the determination of the average lattice macrostrain over the irradiated gauge volume.

For determination of the stress tensor several independent strain measurements for different orientations (usually three) of the sample with respect to the scattering vector should be carried out (see **fig. 2**). As the diffraction techniques measure components of the lattice strains, these have to be then converted to stress components  $\sigma_{x,y,z}$  using appropriate material constants according to the formula

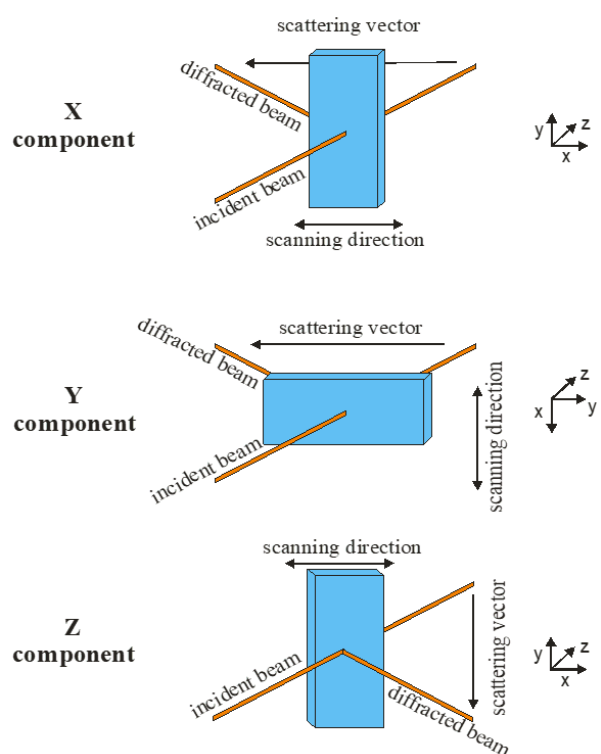
$$\sigma_x = \frac{E_{hkl}}{(1-2\nu_{hkl})(1+\nu_{hkl})} \left[ (1-\nu_{hkl})\varepsilon_x^{hkl} + \nu_{hkl}(\varepsilon_y^{hkl} + \varepsilon_z^{hkl}) \right] \quad (1)$$

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



**Fig. 1** Schematic illustration of a diffractometer for strain measurement and the scheme of the gauge volume

**Obr. 1** Schéma difraktometru pro měření zbytkových napětí a schéma ozařovaného elementu ve vzorku



**Fig. 2** Sketch of the sample orientation for measurement of 3 strain components  $\varepsilon$

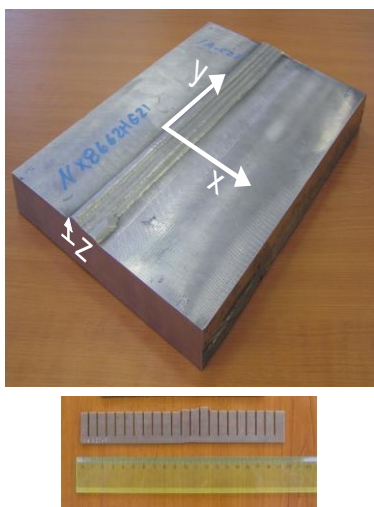
**Obr. 2** Schéma orientace vzorku ve svazku pro měření 3 komponent elastické deformace  $\varepsilon$

Corresponding relations for other  $y$  and  $z$  stress components are obtained by simple permutations of  $x$ ,  $y$  and  $z$  indexes. As the strains are usually of the order of  $10^{-3}$  -  $10^{-4}$  and the sample gauge volumes of several cubic millimeters, a good luminosity and a measurement sensitivity of the device with respect to  $\varepsilon$  between  $10^{-4}$  and  $10^{-5}$  is required.

It should be pointed out that the detector signal is proportional to the irradiated volume of the sample (gauge volume) and its reflectivity, however, it is decreased by neutron beam attenuation in the material. Therefore, the characteristics of the strain/stress diffractometers are practically always a compromise between luminosity and  $(\Delta d/d)$ -resolution.

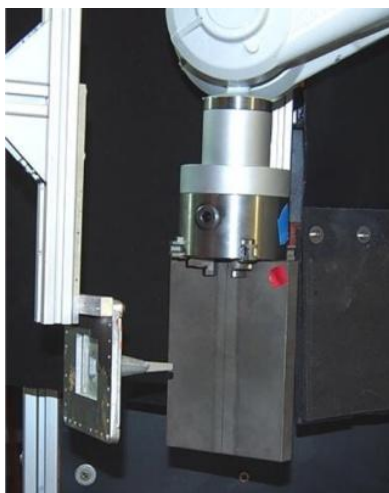
### 3. Stress field determination around welds

Experimental measurements were carried out on the dedicated neutron strain scanner [5] installed at the medium-power (10 MW) research reactor LWR-15 in Řež, Czech Republic. As an example, the C-Mn unalloyed steel specimen of the dimension of  $150 \times 200 \times 15 \text{ mm}^3$  which was cut off the circumferential weld of the storage tank, was chosen for demonstration of the strain measurement method (see **fig. 3** and **fig. 5**). During the measurement, the chosen sample was held by the robotic arm (see **fig. 4**), which made it possible to move the sample in any direction for virtual choice of the gauge volume whose dimension is determined by slits situated in the incident and diffraction beams. **Fig. 5** shows the system of coordinates related to the measurement points (gauge volumes points).



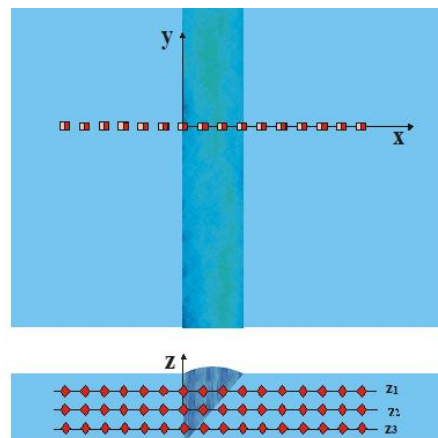
**Fig. 3** Photo of the welded plate with the coordinates and a calibration stress free specimen

**Obr. 3** Snímek desky se svárem a souřadnicemi a kalibrační vzorek bez napětí



**Fig. 4** Photo of the sample attached to a robotic arm of the strain/stress scanner

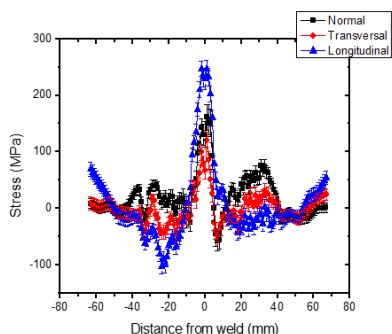
**Obr. 4** Snímek vzorku uchycen robotickým ramenem neutronového skeneru



**Fig. 5** System of coordinates for the experimental measurement and the measurement points

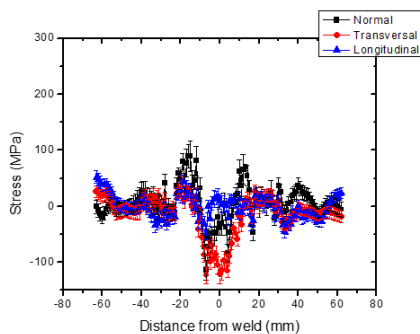
**Obr. 5** Souřadnicový systém použitý pro měření a schéma vybraných měřených bodů

When a virgin stress-free sample is not at the disposal for strain calibration a specimen in the form of a comb having the cross-section of the tooth about  $2 \times 2 \text{ mm}^2$  can be used for this purpose. Normal, transversal and longitudinal strain components have been estimated for three through thickness positions 3.5 mm, 7.5 mm and 11.5 mm below the weld root surface of the tested specimen. Figures **fig. 6a, b, c** show the obtained results.



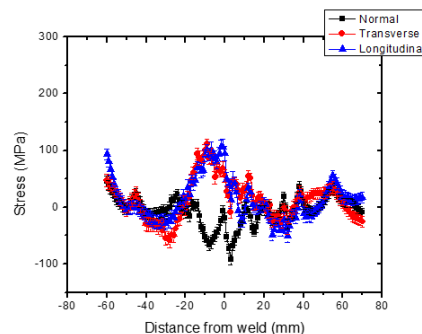
**Fig. 6a** Experimental results taken at the depth of 3.5 mm

**Obr. 6a** Experimentální výsledky získané v hloubce 3,5 mm



**Fig. 6b** Experimental results taken at the depth of 7.5 mm

**Obr. 6b** Experimentální výsledky získané v hloubce 7,5 mm



**Fig. 6c** Experimental results taken at the depth of 11.5 mm

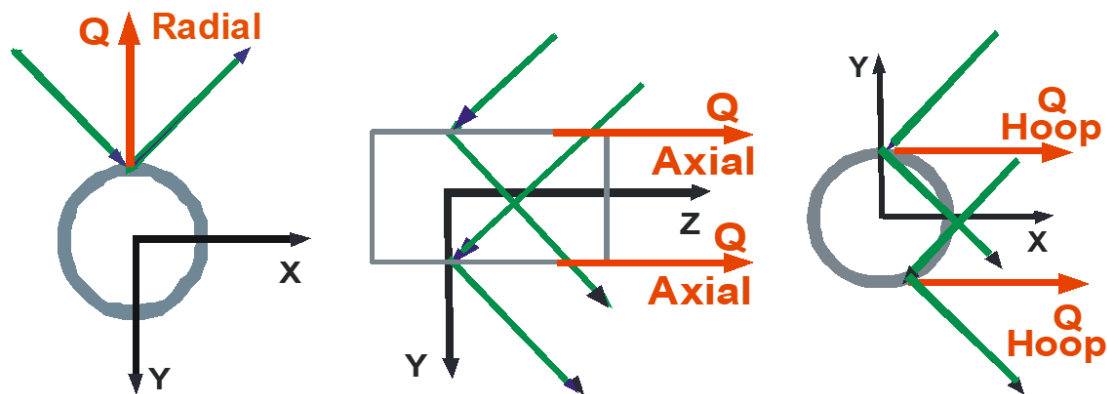
**Obr. 6c** Experimentální výsledky získané v hloubce 11,5 mm

Next example of strain/stress measurement is documented on the weld on the T24  $\alpha$ -Fe pipe of the length of 180 mm and the diameter of  $\varnothing$  33.7 x 6.3 mm (see photo on **fig. 7**), where the measurement points were situated at the depth of 3.15 mm. **fig. 8** shows the sketch of the tube orientation for measurement of three strain components.



**Fig. 7** Photo of the welded on the T24  $\alpha$ -Fe pipe

**Obr. 7** Snímek svařované trubky z materiálu T24  $\alpha$ -Fe

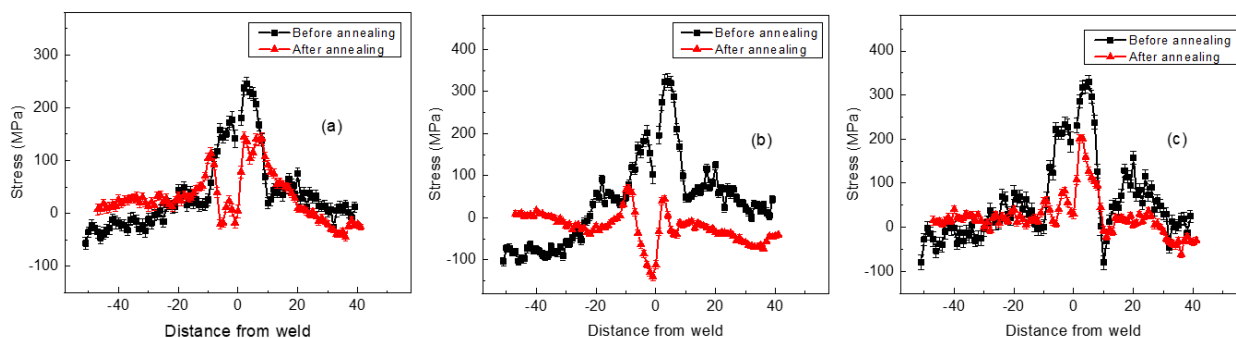


**Fig. 8** The sketch of the tube orientation for the measurement of the strain components  $\varepsilon$

**Obr. 8** Schematické znázornění orientace trubky pro měření komponent elastické deformace  $\varepsilon$

Then, **Fig. 9** shows the experimental comparison of the stress components for the sample before and after annealing. Clear drop of individual stress components after low temperature long term (48 hours) annealing is evident though not remarkable.

According to these results we assume that low temperature heat treatment can influence final behavior of circumferential welds in service.



**Fig. 9** Radial, axial (parallel to X-axis) and hoop (tangential) stresses in the vicinity of the weld measured before and after annealing.

**Obr. 9** Radiální, axiální a tangenciální zbytková napětí naměřená v okolí sváru před a po žhání.

#### 4. Summary

The paper, as an example, demonstrates the experimental results of two neutron diffraction applications of measurements of residual stress distribution in the vicinity of the welds. In the second case, the obtained results show that even the low temperature long term heat treatment can influence the level of residual stresses and one can suppose that this effect can play an important role in the behavior of weld during initial period of power station start up. It can be stated that a large penetration depth (in our case of about 20 mm for steels) and selective absorption of neutrons make the neutron diffraction technique a powerful tool in non-destructive testing of materials. In fact, this technique is one of few nondestructive methods that can facilitate 3-D mapping of residual stresses in bulk components. The strain/stress instrument is offered for measurements to external users free by means of the CANAM project [6].

#### Acknowledgement

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