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HIGH-RESOLUTION NEUTRON DIFFRACTION - A NOVEL TECHNIQUE FOR NONDESTRUCTIVE SCANNING OF MACRO- AND/OR MICROSTRAINS IN POLYCRYSTALLINE MATERIALS

NEUTRONOVÁ DIFRAKTOMETRIE S VYSOKÝM ROZLIŠENÍM - NETRADIČNÍ METODA NEDESTRUKTIVNÍHO MĚŘENÍ NAPĚTÍ V POLYKRYSTALICKÝCH MATERIÁLECH

Pavel Mikula[1], Petr Lukáš, Miroslav Vrána

Neutron Physics Department of NPI CAS routinely operates at the medium-power reactor LVR-15 in Řež two high-resolution neutron diffractometers dedicated to strain/stress investigations in polycrystalline materials. Good luminosity of the diffractometers and a sufficiently high-resolution (FWHM of the instrumental Δd/d - profile can be about $1x10^{-3}$ at d_{hkl}=0.2 nm) permit investigations of both the macro- and microstrains in the sample gauge volumes of several cubic millimetres with a strain sensitivity of about 10^{-4}. Both instruments are equipped with a tension/compression rig (up to ±20 kN) and heating system for samples (up to 1000 °C) and the response of the lattice of the polycrystalline samples under thermomechanical load can be investigated in situ. Excellent properties and abilities of the neutron diffraction strain/stress scanners are proved by some results of experimental measurements. The measurements on the strain/stress scanners are opened for external users.

Oddělení neutronové fyziky ÚJF AV ČR u reaktoru LVR-15 v Řeži provozuje dva neutronové difraktometry s vysokým rozlišením pro měření relativních změn mřížkového parametru ε=Δd/d (přístrojové rozlišení vyjádřené pomocí FWHM(Δd/d) je přibližně $2x10^{-3}$ pro d_{hkl}=0,2 nm; d_{hkl} je mezirovinná vzdálenost). Difraktometry jsou využívány zejména ke zjišťování a proměřování vnitřních deformací v polykrystalických materiálech. Velmi dobrá světelnost a dostatečně vysoké rozlišení difrakčních zařízení nám dovoluje kromě známého proměřování makrodeformací, také studovat a proměřovat mikrodeformace v objemových elementech několika kubických milimetrů s přesností10^{-4}. Difraktometry jsou navíc vybaveny trhacím strojem pro tahové a tlakové zkoušky v rozsahu ±20 kN pro in-situ měření odezvy struktury studovaného materiálu v oblasti elastické a plastické deformace. Výborné vlastnosti měřících zařízení a jejich experimentální možnosti jsou doloženy několika experimentálními výsledky. Měření na difraktometrech je přístupné také po vnější uživatele

Keywords: Strains, neutron diffraction, polycrystalline materials, nondestructive testing.

Klíčová slova: Mřížková pnutí, neutronová difrakce, polykrystalické materiály, nedestruktivní analýza.

1. Introduction

Residual stresses or their development under applied external force are difficult to predict in engineering materials and can have a strong influence on their basic mechanical properties. The stresses displace atoms from their original positions in a crystalline material that in fact results in a change of the interatomic distances that vary from those in a stress-free case. Neutron dif-

[1] Pavel Mikula: Nuclear Physics Institute CAS and Research Centre, Ltd.; 250 68 Řež near Prague, Czech Republic, tel.: +42066173553; e-mail: mikula@ujf.cas.cz

fraction along with X-ray diffraction where angular positions of diffraction maxima are directly bound with the values of lattice constants through the Bragg equation $2d_{hkl} \cdot \sin \theta_{hkl} = \lambda$ (d_{hkl} - lattice distance, θ_{hkl} - Bragg angle, λ - the neutron wavelength) offers an unique non-destructive technique for investigation of stress fields [1-6]. The large penetration depth and selective absorption of neutrons make them a powerful tool in non-destructive testing of materials. In fact, neutron diffraction is the only NDT method that can facilitate 3-D mapping of residual stress in a bulk component. In principle, the substructural changes in engineering components can be investigated while in use or in-situ, under loading by an external force. The stresses are not measured directly by diffraction techniques, but one measures residual strains, which are then converted to stresses using appropriate moduli. Consequently, when defining strain ε as $\varepsilon = \Delta d/d_{o,hkl} = -\cot \theta_{hkl} \cdot \Delta\theta_{hkl}$ ($d_{o,hkl}$ is the lattice spacing of the strain-free material) the residual stresses or the ones applied by an external force recall a change in the lattice spacing just by Δd. Consequently, neutron diffraction together with X-ray diffraction provide unique possibilities of a precise determination of the lattice spacing changes Δd through the Bragg diffraction on the corresponding lattice planes (*hkl*). Thus, the relation for the strain $\varepsilon = -\cot \theta_{hkl} \cdot \Delta\theta_{hkl}$ (in fact a component parallel to the scattering vector $\boldsymbol{Q}$) indicates that it gives rise to a change in the scattering angle $2\theta_{hkl}$ resulting in an angular shift $\Delta(2\theta_{hkl})$ of the peak position. In such a way, the shift in the Bragg angle (relative to that of the stress-free material) permits one to determine the average lattice macrostrain over the irradiated gauge volume. Information on the lattice microstrain present in this gauge volume can be determined from a change of the width and the form of the diffraction peak profile. Conventional neutron strain scanners using the Bragg Diffraction Angle Analysis method are in fact powder diffractometers optimised for high resolution measurements at large scattering angles. As neutron diffraction requires neutrons of sufficiently high current such measurements are carried out at the powerful neutron sources, namely, nuclear research reactors or neutron spallation sources. At present, two high-resolution focusing neutron-diffraction strain diffractometers are routinely operated at the medium-power reactor LVR-15 in Řež which are opened for external users. By using focusing principles of the Bragg diffraction optics, they have good luminosity and a sufficiently high-resolution (*FWHM* of the instrumental $\Delta d/d$ - profile can be less than $2\text{x}10^{-3}$ at d_{hkl}=0.2 nm) and permit us investigations of both the macro- and microstrains (by a peak profile analysis) in the gauge volumes of several mm^3 with the sensitivity to $\Delta d/d$ changes of about 10^{-5}. These dedicated neutron strain diffractometers are equipped with tension/compression rig and heating system for samples (up to 1000 ^{o}C) and the response of the lattice of the polycrystalline samples under thermomechanical load can be investigated in situ. Excellent resolution properties and abilities of the focusing neutron diffraction strain scanners will be documented by the results of experimental measurements.

2. Experimental performance and results

When a crystalline material is illuminated with a neutron beam of wavelength λ, comparable with the inter-planer spacing d_{hkl}, a diffraction pattern is observed in which the position of each plane (*hkl*) is defined by the Bragg relation:

$$2d_{hkl} \sin \theta_{hkl} = \lambda. \qquad (1)$$

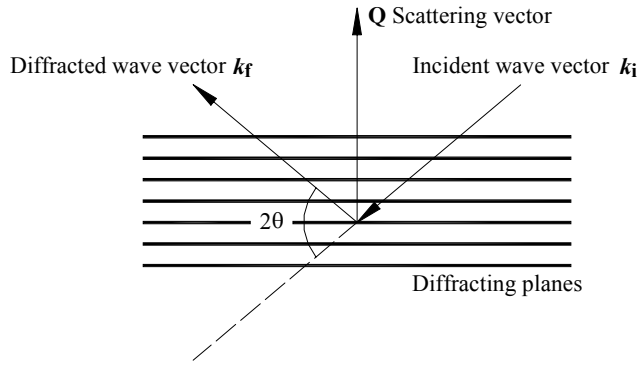

Fig.1 Schematic illustration of Bragg scattering geometry.

In the nuclear reactor the produced neutrons are moderated to bring their energies to the thermal range, i.e. $\lambda \geq 0.05$ nm. Then, a monochromatic beam of neutrons is produced by using a monochromating device to select a given neutron wavelength from the polychromatic beam. The entire diffraction pattern is recorded at any particular scattering angle.

The strain is measured in the direction of the scattering vector, $\boldsymbol{Q} = \boldsymbol{k_f} - \boldsymbol{k_i}$, which bisects the angle between incident and diffracted beams and is perpendicular to the diffracting planes as shown in Fig. 1. The value of the lattice spacing is determined from the measured angular position of the diffraction peak (Bragg reflection) by illuminating the specimen with a monochromatic collimated beam of neutrons. If the specimen contains no strain, the lattice spacing has the strain free (stress free) value for the material and is denoted by $d_{0,hkl}$. In a stressed specimen, lattice spacing is altered and a shift in each related Bragg peak position occurs and the elastic strain then is given by

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} = \frac{\Delta d_{hkl}}{d_{0,hkl}} = \frac{sin\theta_{0,hkl}}{sin\theta_{hkl}} - 1. \qquad (2)$$

The angle $\theta_{0,hkl}$ is the angle at which Bragg peak is observed from the strain free reference. Stress σ_{ij} and strain ε_{kl} are second rank tensors and are related through elastic constants, C_{ijkl} as

$$\sigma_{ij} = C_{ijkl}\,\varepsilon_{kl}\,. \qquad (3)$$

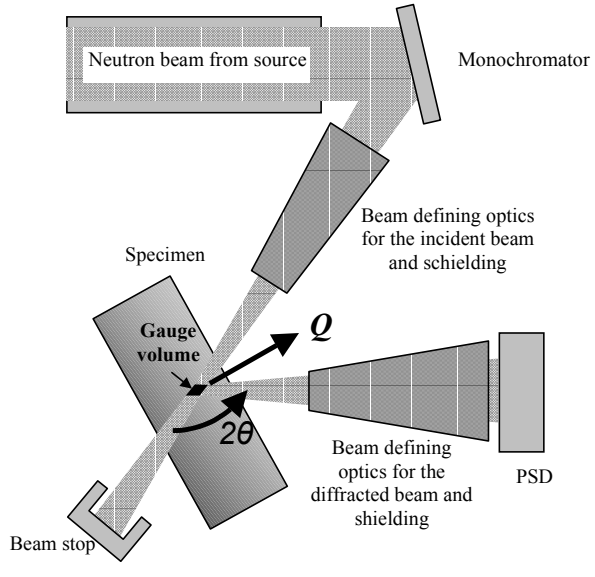

Fig.2 Schematic illustration of a reactor source based diffractometer for strain measurement.

An instrument used for strain measurement at a reactor source is shown schematically in Fig. 2. The polychromatic neutron beam is first monochromated to a chosen wavelength by diffraction from a suitable monochromator. The divergence and size of the monochromatic beam is suitably adjusted using appropriate neutron optical devices and is then diffracted from the specimen. In a similar way, the diffracted beam is shaped using suitable optical devices, before it is captured by the neutron position sensitive detector (PSD). The gauge volume, over which the strain measurement is made, is given by the intersection of the incident and diffracted beams (Fig. 2). An example of diffraction line profiles measured by PSD of such an instrument is shown in Fig. 3. Since

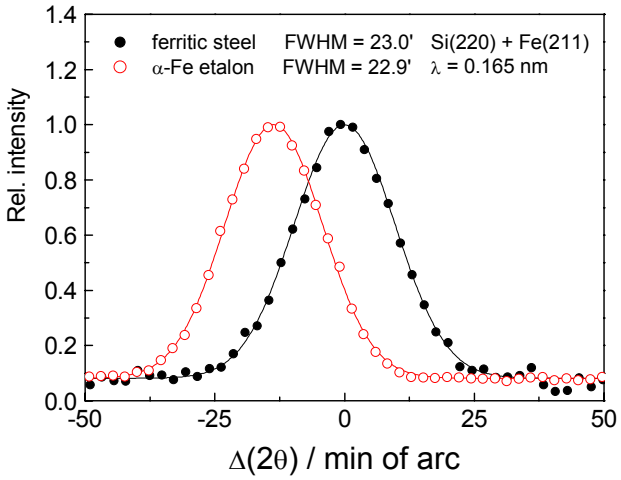

Fig.3 Examples of line profiles of α-Fe(211) etalon and a steel sample taken in combination with Si(220) monochromator.

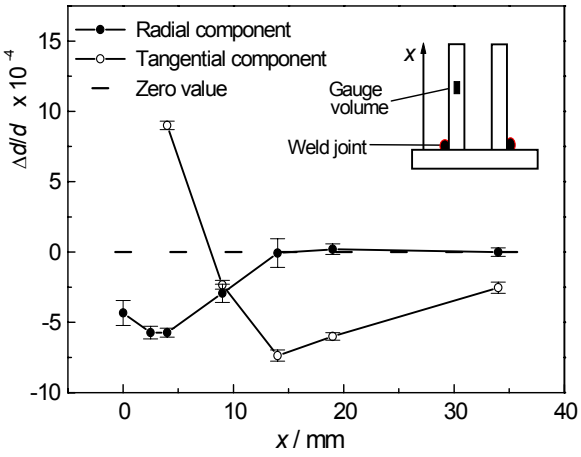

Fig.4 The dependence of the strain components on the distance from the weld.

neutron diffraction can measure the elastic strain within a defined volume in a crystalline solid, it is possible to calculate the mean stress in that volume provided the relevant elastic constants for the material are known. Full determination of the strain tensor requires measurements of the elastic strain in at least six independent directions. If the principal strain directions within the specimen are known, measurements along these three directions are sufficient. For plane stress or plane strain conditions, a further reduction to two directions is possible. Measurement along one direction only is needed in the case of uni-axial loading. As stresses and strains in a specimen are usually direction and position dependent, it leads to the need to measure strains at a number of locations and in more than one direction. This in turn requires accurate positioning of the specimen with respect to the collimated neutron beam and the detectors with linear translation and rotation tables, on which the specimen is mounted. Then, by sequentially moving the specimen through the gauge volume the spatial variation in stress can be mapped e.g. in the vicinity of small fixing elements (rivets), surfaces, barriers, coatings, welds etc. As an example, Fig. 4 shows several experimental points of the radial and tangential component of macrostrain in the dependence of the distance of the weld joint connecting the tube of ferritic steel (Φ=85 mm, thickness of the wall of 8 mm) with a flat steel plate (thickness of 12 mm). The value of the lattice spacing at a large distance from the weld joint was taken as a strain free value d_o (5x2x2 mm^3 gauge volume). Similarly, Fig. 5 displays another example of the strain components (see scanning line) in the

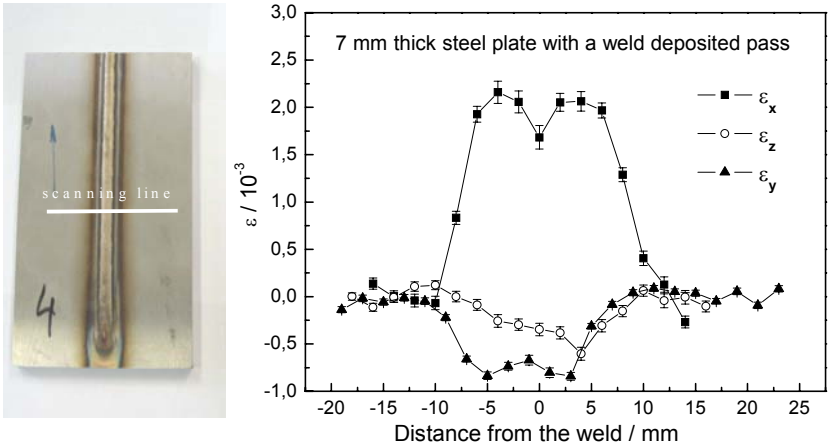

Fig.5 Strain components in the plate of 15Ch2MFA versus the distance from the weld deposited pass (welding material - Inconel 52).

vicinity of the 10 mm wide and 3.5 mm high weld deposited pass. The gauge volume of 3x2x2 mm^3 was situated in the middle of the plate and the scanning was carried out perpendicularly to the pass. The axes x, y and z are parallel to the longest edge of the plate, the medium wide edge and the shortest edge, respectively.

3. Conclusion

Residual strain/stress formed in a material during manufacturing, welding, utilisation or repairs can be measured by means of neutron diffraction. Such studies can help to improve the manufacturing quality of engineering components, to optimise their design criteria in applications and to predict their operational life. Exciting new industrial applications of neutron scattering are expected in so called in-situ investigations, namely for kinetics studies, fatigue behaviour studies, thermal cycling stress evolution, heat treated experiments permitting optimisation of thermal treatment, texture measurements of samples under the load of the extension force or during recrystallization at elevated temperature, phase measurements in multiphase alloys under thermal and/or tension/compression loading, investigations of an early stage precipitation, early stage of microcracking, early stage of dislocation evolution, early stage of radiation damage resulting from the effect of irradiation etc. Anisotropies in thermal and electrical conductivity, for instance of fuel elements, and mechanical properties of materials depend on the textures developed during their preparation or thermal treatment. Textures can also be studied using neutron diffraction techniques. The exploitation of neutron difraction techniques for material research is in NPI Řež opened for external users (http://omega.ujf.cas.cz).

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