Characterisation of residual stresses by neutron diffraction at the research reactor IR-8 of NRC “Kurchatov Institute”

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Abstract. The main principles of the neutron diffraction method for residual strain/stress measurements in polycrystalline materials and components at the continuous research reactor are described. The main technical characteristics of neutron diffractometer for residual stress measurements STRESS, installed at the horizontal beam port of research reactor IR-8 at NRC “Kurchatov Institute” are presented. Examples of stress measurements in the welding joint and the component of additive manufacturing using the diffractometer STRESS are reported.

1. Introduction
Residual stresses are introduced into engineering components during manufacturing (rolling, forging, bending, welding, additive manufacturing etc.) or can be developed in a component during service. They can be very detrimental to the performance of a material or life of a component. Therefore, reliable information about residual stresses in a component is very important. Thanks to high penetration ability of neutrons in most industrial materials, the neutron diffraction method is the only method at this moment that allows measuring all three components of strain/stress tensor in bulk materials non-destructively [1]. Therefore, in the past 15-20 years this method was actively developed in many neutron research centers [2-5] to solve scientific and applied problems related to the internal stresses.

2. The neutron diffraction method for stress measurements
The neutron diffraction method for stress measurements is based on measurements of the stressed crystal lattice plane spacing \(d\) and an unstressed (stress-free) lattice spacing \(d_0\). According to Bragg’s law, \(2dsin\theta = n\lambda\) (\(n\) – integer), at a neutron diffractometer with constant wavelength \(\lambda\), the \(d\) spacing can be determined by an accurate measurement of the angular position of a diffraction peak \(2\theta\) (angle between incident and reflected neutron beams) as shown in Fig. 1. A small change \(Ad\) will result in a shift of a diffraction peak \(\Delta2\theta\), so that the lattice strain \(\varepsilon\) can be determined by measuring the angular shift of the diffraction peak from the position, corresponding to the unstrained crystal lattice:

\[
\varepsilon = \frac{d - d_0}{d_0} = -(\theta - \theta_0)ctg\theta_0, \quad (1)
\]
where $\theta$ and $\theta_0$ are correspondingly the angular positions of diffraction peak for stressed and stress-free samples. The diffraction is measured experimentally from a small diffracting volume (the gauge volume) inside a massive sample. The gauge volume is well defined by slits placed in incident and scattered neutron beams (Fig. 1). As a rule, slits are cut in cadmium plates that actively absorb thermal neutrons. By superimposing the points of the sample, at which stresses must be measured with the center of the gauge volume, one can measure the distribution of stresses over the sample.

**Figure 1.** Schematic of the neutron diffraction method for residual stress measurements

Strain, like stress, is a second rank tensor. Therefore, in a general case, three strain components $\varepsilon_x$, $\varepsilon_y$, $\varepsilon_z$ along three mutually perpendicular “principal directions” $x$, $y$, $z$ in the sample should be measured to calculate principal stress components $\sigma_x$, $\sigma_y$, $\sigma_z$. So strains are measured at three sample orientations with axes $x$, $y$, $z$ directed along the scattering vector $Q$ (normal to the reflecting planes) as shown in Fig. 2.

**Figure 2.** Principal directions $x,y,z$ in a sample and orientation of sample for measurement of strain components $\varepsilon_x$, $\varepsilon_y$, $\varepsilon_z$ along principal directions $x$, $y$, $z$.

The strains in three orthogonal directions in the sample are measured and the residual stresses in these directions can be calculated by generalized Hooke’s law [1]:

![Diagram of neutron diffraction method](image-url)
\[ \sigma_i = E[(1-2\nu)\varepsilon_i + \nu(\varepsilon_x + \varepsilon_y + \varepsilon_z)]/(1 + \nu)(1 - 2\nu), \quad (2) \]

where \( i = x, y, \) or \( z; \) \( E \) and \( \nu \) are the elastic modulus and Poisson’s ratio, respectively. Elastic constants are different for different planes \( (hkl) \) in crystal structure. Therefore, diffraction elastic constants, \( E_{hkl} \) and \( \nu_{hkl}, \) corresponding to the specific reflecting plane \( (hkl) \) should be used for the stress calculation.

3. Diffractometer for stress measurement STRESS
Neutron diffractometer STRESS is installed at beam port #3 of the research reactor IR-8 (maximal power 8MW) at NRC “Kurchatov institute” (Fig. 3). Thanks to optimized double-crystal monochromator and diffractometer optics [6-7], the instrument ability for stress measurements is comparable to other instruments at more powerful reactors. The double-crystal monochromator PG(002)/Si(220) provides a fixed wavelength of \( \lambda = 0.156 \text{ nm}, \) which is optimal for stress measurements at depth in ferritic steel [8]. The diffractometer is equipped with a 2-D position sensitive detector with a 250×150 mm\(^2\) (height × width) active area and a 2 mm spatial resolution in the horizontal direction. Three positioning translators X, Y, Z, mounted on the sample table allow strain scan over the sample volume. The maximal load on the sample platform is 100 kg. The maximum size and thickness of a sample are 500 mm and 50 mm, respectively. The diffractometer’s resolution \( (\Delta d/d) \) is approximately 0.003. Typical gauge volume is 1-100 mm\(^3\) and typical uncertainty in strain measurements is ±100 \( \mu \varepsilon \) (1\( \mu \varepsilon \) (microstrain) = \( 10^{-6} \)).

![Figure 3. General view of the STRESS diffractometer on beam port #3 of the reactor IR-8.](image)

4. Measurements of residual stresses in additively manufactured stainless steel prism.
The distribution of residual stresses in an additively manufactured CL 20ES steel prism with 20×20 mm\(^2\) base and 69 mm height (Fig. 4) was measured. The prism was built of CL 20ES metal powder with chemical composition corresponding to the 316L austenite grade steel via selective laser melting (SLM) method using Concept Laser M2 Cusing machine. The layers were deposited in the x-y plane and the sample was grown along the z-axis. After manufacturing, the sample was removed from the base plate and mesh sub-structure using a cut off wheel in the as-built condition, i.e., without undergoing any stress-relief heat treatment.
The strain components in x, y, z directions were measured at points, located along the 7 lines parallel to the z-axis as shown in Fig.4a. Along each line the measurements were conducted with a 2.5 mm step. The coordinates x, y of the lines are shown in Fig.4b. The cadmium slits defined the gauge volume about 2×2×2 mm³. In the case of austenitic stainless steel, (311) diffraction peak has shown to have little sensitivity to intergranular stresses [1]. Thus, this diffraction peak measured at a scattering angle 2θ about 91° was used for residual stress measurements. Residual stress calculations from the neutron diffraction measurements depend strongly on the determination of the stress-free lattice spacing d₀. To determine stress-free lattice spacing d₀ after measurement of d spacing in the sample had been finished, a small cube 4×4×4 mm³ was cut from the top of the prism using wire electric discharge machining. We assumed that the cube is free from macro stresses and d₀ spacings in x, y, z directions were measured with 2×2×2 mm³ gauge volume. These values of d₀ were used for strain calculations at all measured points in the sample. In general, d₀ depends on chemical composition and can be location-dependent. Measurements of d-spacing in the sample showed low variation of d-spacing, corresponding to ±2⋅10⁻⁴ variation in the strain, for all lines in the comparatively large (30 mm) height interval (20 ≤ z ≤ 50 mm). Since such low variation is caused by combined variation of stress and chemical composition, we could assume the chemical composition of sampling volume (2×2×2 mm³) is the same throughout the sample and at all measured points we can use the same values of d₀.

Principal stresses σₓ, σᵧ, σ₀ were calculated by using generalized form of Hooke’s law (2), where the elastic constants for (311) planes, E₃₁₁ and ν₃₁₁, were assumed to be 184 Gpa and 0.294 respectively [9]. The maps of 2D distributions of stress along the height/length of the prism (σ₀) in vertical z-x and z-y sections are shown in Fig.4. One can see that in the middle part of the prism height (18 ≤ z ≤ 55 mm) the tensile stresses (≈ 320 MPa) near the prism edges are compensated by the compressive stresses (≈ - 300 MPa) near the prism centerline. When approaching the bottom and top faces of the prism, both tensile and compressive stresses decrease and at a depth of 5 mm from the bottom and top faces.

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**Figure 4.** a) design of the prism and the lines of the points in which the stresses were measured; b) coordinates x, y of the lines (x, y coordinates of the measured points).
faces they are less than 80 MPa. The measured stress distribution was symmetrical with respect to the vertical planes passing through the prism centerline perpendicular to the vertical prism faces.

\[
\text{Stress, MPa} \quad \text{Z, mm}
\]

![Stress Distribution](image)

**Figure 5.** 2D maps of distribution of stress along z direction ($\sigma_z$) in vertical z-x and z-y sections.

5. **Stresses and low transformation temperature (LTT) welding consumable.** Application of low transformation temperature (LTT) consumables is an innovative approach to decrease the residual stresses in welding joints. The idea is to use volumetric expansion associated with phase transformation from austenite ($\gamma$) to martensite ($\alpha'$) at a relatively low temperature to compensate the thermal shrinkage of a weld metal during cooling.

Two samples of V-groove butt welded plates with conventional (HTT) and LTT welding consumable were studied [10] using diffractometer STRESS. The samples were prepared from 25 mm thick plates of high-strength low-carbon ferritic steel. The schematic of the samples with dimensions of 300×300×25 mm$^3$ is shown in Fig. 6. One sample (HTT) was prepared using conventional ferritic consumable alloy. The second sample (LTT) was prepared using two different welding metals: conventional consumable alloy was utilized for the first three welding passes, and the 10.2Ni-9.6Cr based austenitic alloy with comparatively low (~ 100°C) transformation temperature was used for the other nine passes. The diffraction peak (211) at the scattering angle 2$\theta$ of about 82.8° was measured with gauge volume of 4×4×4 mm$^3$. A total of 45 different locations were selected for measurement at 0, ±1, ±3, ±5, ±10, 15, 25, 40, 60, 90, and 130 mm from the weld centerline at three depths of 5 mm,
12.5 mm (mid-thickness) and 20 mm. The measurement time was about 20 min for each strain component and a typical strain uncertainty was about ±100 με. The comb-like “stress-free” reference sample was extracted and prepared by electric discharge machining. It has a “tooth” with dimensions of 5 mm long (x), 4 mm wide (y), and 20 mm deep (z) at each identical location to that of strain scanning. The stress-free lattice spacing (d0) were carefully measured with the gauge volume 2×2×2 mm³ at these locations. The utilized elastic constant (E211) was 210.0 GPa, and the Poisson's ratio (ν211) was 0.3.

![Figure 7. 2D maps of the longitudinal residual stresses (σx) in welded plates with conventional (HTT) and low transformation temperature (LTT) welding consumables.](image)

Fig. 7 shows two-dimensional maps of longitudinal stresses (σx) measured in the cross-section (z-y) of the HTT and LTT samples. Overall, the maps clearly show different through-thickness stress distributions between HTT and LTT samples. HTT sample shows high residual stresses (up to 530 MPa) in the upper area. Such high tensile stresses are typical for multi-pass butt welds. LTT sample shows significant compressive stresses (~510 MPa) near the top surface of the weld metal while high tensile stresses were observed in corresponding region of LTT sample. In addition, it should be mentioned that significant tensile stresses (up to 590 MPa, about 95% of yield strength) were observed close to the bottom interface between the HTT and LTT regions.

6. Conclusion
Nowadays, the neutron method of stress measurement has become a powerful tool for investigation internal stresses in various polycrystalline materials. Experiments have shown that despite the relatively low power of research reactor IR-8 (8MW), the technical characteristics of the STRESS diffractometer installed on this reactor allow the research of stresses in massive metal products, including welded joints and products of additive manufacturing.

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