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# Nondestructive Neutron Diffraction Residual Stress Measurements in a Large Depth in Steels

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**Abstract:** Residual stresses can be effectively and nondestructively measured through a large thickness of e.g. welded plates, by means of a new design of a reactor-based neutron diffractometer. Most of the engineering neutron diffractometers have a difficulty in increasing neutron penetration capability over 25 mm total thickness in steels. However, it can be significantly enhanced up to 70 mm with 4-mm spatial resolution along the depth by using the wavelength selection in combination with neutron focusing by cylindrically bent perfect monochromator. Present paper presents details of neutron diffractometer performance and some results.

**Keywords:** Stress; Analysis; Neutron Diffraction

## 1. Introduction

Residual stresses are stresses that are "locked-in" within a material, and exist without any external load. They are caused by incompatible internal permanent strains. They may be generated or modified at every stage in the component life cycle, from original material production to final disposal or can be formed in a material during repairs. Welding is e.g. one of the most significant causes of residual stresses and typically produces large tensile stresses balanced by lower compressive ones elsewhere in the component. Residual stresses may be measured by non-destructive techniques, including X-ray diffraction, neutron diffraction and magnetic and ultrasonic methods; by locally destructive techniques, including hole drilling and the ring core and deep hole methods; and by sectioning methods including block removal, splitting, slicing, layering and the contour method. The large penetration depth and selective absorption of neutrons make them a powerful tool in nondestructive testing of materials. Thanks to these favourable properties of neutrons, among all these measurement techniques neutron diffraction appears as the most powerful when permitting stress determination in bulk material non-destructively, in a rather large depth under the surface with the error of about 10-30 MPa. Moreover, neutron diffraction is phase sensitive. Neutron diffraction studies can thus significantly help one to improve the manufacturing quality of engineering components, to optimize their design criteria in applications and to predict their operational life.

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## 2. Residual strain/stress measurement by neutron diffraction

The stresses displace atoms from their original positions in a crystalline material, which in fact results in a change of the interatomic distances, which vary from those in a stress-free case. The stresses are not measured directly by diffraction techniques, but one measures residual strains, which are then converted to stresses using appropriate moduli. Neutron diffraction along with X-ray diffraction where angular positions of diffraction maxims are directly bound with the values of lattice constants through the Bragg equation offers a unique non-destructive technique for investigation of stress fields [1-3]. Thus, the elastic strains are derived from the change in the lattice spacing of the crystalline material. As the strains in the material are of the order of $10^{-4}$-$10^{-3}$ the sensitivity of neutron diffraction instrument for strain detemination should be $\leq 10^{-4}$. By translating the specimen through a neutron beam, stresses at different locations can be determined. In fact, neutron diffraction is the only non-destructive and highly accurate method which can facilitate 3-D mapping of residual stress in bulk components.The neutron strain/stress scanner evaluates the variations of lattice spacing within a sample with a spatial resolution of the order of mm given by the dimensions of the gauge volume. Typical neutron diffractometer dedicated to strain/stress measurements at a reactor source is shown schematically in Fig. 1. The polychromatic neutron beam is first monochromated to a chosen wavelength by diffraction from a suitable monochromator. This beam of a suitable cross-section and divergence is given by the use of appropriate beam defining elements and is then diffracted from the specimen. In a similar way, the geometry of the diffracted beam is shaped by additional beam limitation devices, before it is captured by a neutron detector. The gauge volume to which the strain measurement is related is given by the intersection of the incident and diffracted beams. Typically, neutron strain scanner is established on existing powder diffractometer. First generation instruments usually used mosaic monochromators (Cu, PG or Ge) without or with a poor beam focusing and monochromatic beam spreads its cross-section at the place of the sample position. Then, the beam defining elements determining the gauge volume bring about a strong decrease of the final detector signal while the resolution (*FWHM* of the diffraction profile) is still strongly influenced by the mosaicity of the monochromator $\beta$ being tens of minutes of arc. Even in the

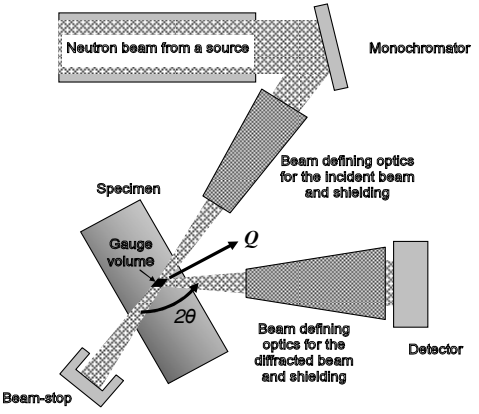


**Fig. 1.** Schematic illustration of a conventional reactor source based diffractometer for strain measurement.

optimum case, when the diffraction angles at the monochromator ($\theta_M$), and the sample ($\theta_S$) fulfil the relation $\tan\theta_S / \tan\theta_M = +1/2$, *FWHM* has a minimum value but still it is larger than $\beta$ [4]. As can be seen from Fig. 1, the best geometrical choice for residual strain/stress measurements corresponds to $2\theta$=90° when the gauge volume is in the form of a cube or rectangular prism. Consequently, it is required to have an intense Bragg peak at $2\theta \approx 90°$. However, in the conventional case, due to the required resolution a large diffraction angle at the monochromator should be set. The current of the monochromatic neutrons impinging the sample is proportional to the wavelength spread $\delta\lambda=\lambda\cdot\Delta\theta\cdot\cot\theta_M$, where $\Delta\theta$ is the angular divergence of the beam. Therefore, the larger diffraction angle at the monochromator means the smaller neutron beam current and consequently, the smaller detector signal. However, this problem can be avoided by employing focusing bent perfect crystal (BPC) monochromators [5,6].

### 3. Focusing monochromator in the powder diffraction case

A great advantage of the strain diffractometer equipped with the focusing BPC-monochromator is very good predictability of its focusing properties as well as resolution and reflectivity parameters which has been proved many times. Usually used focusing diffraction performance (see Fig. 2a) consists of the following steps: Monochromatic neutrons selected by the BPC monochromator from a white spectrum are focused on a sample (real space focusing). As each neutron fulfills the Bragg condition $2d_{hkl}\sin\theta = \lambda$, due to the crystal curvature there is a strong correlation between the divergence of the incoming and outgoing beams with respect to the monochromator and the sample. This correlation can be easily manipulated by changing the radius $R$. Then, by setting a radius of curvature of $R = (2L_{MS}/\sin\theta_M)/(2\text{-}1/a_{SM})$, the diffracted beam from the sample is (quasi)-parallel and not dependent on $\Delta\alpha_1$ ($L_{MS}$ is monochromator/sample distance) and $a_{SM} = -\tan\theta_S / \tan\theta_M$) [5,6]. Therefore, a large width $W$ of the polychromatic beam inpinging on the monochromator can be successfully used. This is called focusing in scattering. In practice, it means that for some optimum crystal curvature, the peak intensity and *FWHM* of the diffraction line achieve their maximum and minimum,

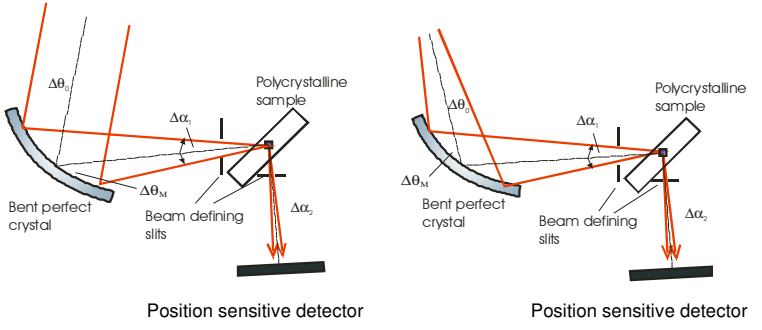


**Fig. 2.** Schematic geometry of the diffractometer performance with a BPC monochromator; usually used performance (left) and a new one tested in KAERI (right).

simultaneously. The quasi-parallel diffracted beam is then directly analyzed by using a position sensitive detector (PSD). Of course, there are small resolution uncertainties influencing the instrumental resolution which make the (quasi-) parallel diffracted beam slightly divergent [5,6]. They come from a non-negligible thickness $t_M$ of the monochromator (3-6 mm) and from the finite width $w$ of the irradiated volume of the sample determined by the input and output slits (1-2 mm). It is clear from Fig. 2 that contrary to the conventional diffractometer performance with the mosaic monochromator no Soller collimators which always cut the neutron current are required.

As schematically shown in Fig. 2a, the most usual diffractometer performance uses the monochromator take-off angle $2\theta_M$ and if possible also the scattering angle $2\theta_S$ equal to $90^o$; in the former case because of resolution and in the latter one because of the cuboidal gauge volume. Also in this case the general diffractometer property saying that the larger monochromator take-off angle, the smaller neutron beam current and consequently, the smaller detector signal is obtained. However, a question related to a possible use of smaller monochromator take-off angle and thus to increase the beam current impinging the sample has remained opened. An advantage of the BPC monochromator consists in the fact that its employment offers exploiting several free parameters in optimisation of the diffractometer performance. One of them is the thickness of the crystal considerably contributing to the resolution uncertainty. Thus, when using smaller monochromator take-off angle and increasing the monochromatic neutron current, the worsening of the diffractometer resolution can be compensated by the employment of a thinner BPC crystal. This idea has been experimentally tested and contrary to the conventional conservative meaning it has appeared that by using this new diffractometry geometry the luminosity of the instrument with a acceptable resolution can be substantially increased [7].

## 4. Experimental results

First, it was necessary to test the intensity and resolution behaviour of the diffractometer performance when using a small irradiated volume of a polycrystalline sample as is usually used in the strain/stress measurements. For simplicity, instead of a bulk sample and beam defining slits we used an α-Fe(211) steel pin of 2 mm diameter and 40 mm height. By using a focusing Si(111) monochromator (cylindrically bent perfect crystal) set at $2\theta_M = 30^o$, for $\lambda = 0.162$ nm the scattering angle on the sample was $2\theta_S = 87.8^o$. After the optimisation of the crystal curvature shown in Fig. 3 there has been found an excellent luminosity with the acceptable resolution of the instrument. Moreover, the resolution can be also adjusted by a suitable choice of the thickness of the monochromator crystal slab (compare Fig. 3a and 3b) followed of course, by a decrease of the detector signal. Fig. 4 displays the diffraction profiles of α-Fe(112) pin of 1 mm diameter and 40 mm height taken for different measurement times by the PSD at the distance of 120 cm from the sample. Smaller diameter of the sample had practically no influence on the *FWHM* of the diffraction profile, because the spatial resolution of the PSD was 2.5 mm (one channel corresponds to $0.0095^o$) was much worse. We used as a sample

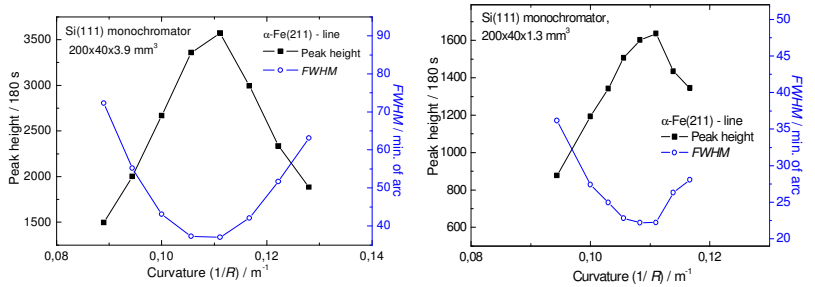


**Fig. 3.** The luminosity and resolution characteristics of the strain/stress diffractometer performance for the focusing Si(111) monochromator of different thickness of 3.9 mm (left) and 1.3 mm (right).

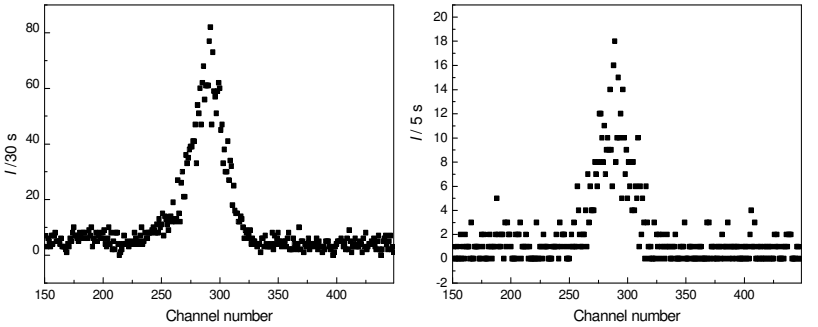


**Fig. 4.** Diffraction profiles of the sample of α-Fe(112) pin of 1 mm diameter and 40 mm height for different measurement times.

deliberately only 1 mm diameter pin and followed the detector signal and the error in determination of the peak position. The evaluation showed that even at the measurement time of 5 s the peak position can be determined with a relative error of about $10^{-4}$ which is sufficient in most cases of residual strain/stress measurements. Thanks to very good luminosity and resolution the new instrument performance permits also studies of some kinetic processes related to macro- and/or micro-strain/stress distribution. In such cases much larger gauge volume is usually used resulting in a stronger detector signal.

## 5. Effect of wavelength-dependent attenuation on neutron diffraction stress measurements at depth in steels

After using Bragg diffraction optics for improvement of luminosity and resolution of the dedicated diffractometer for residual strain/stress scanning the effectivity of the measurement can be further increased by a suitable decrease of the neutron attenuation in the investigated material. One of the possibilities is to exploit the irregular wavelength dependence of the total neutron cross-section in the thermal energy range where the so called Bragg diffraction edges occur (see Fig. 5).

**Table 1. Maximum penetration depths for different crystals and wavelengths**[a]

***Ferritic steel (low-carbon steel, b.c.c.).***

| Monochromator | $2\theta_M$ (°) | $\lambda$ (Å) | Reflection plane | $2\theta_S$ (°) | FoM | l (mm) | $D_{ref}$ (mm) | $D_{tr}$ (mm) |
|---|---|---|---|---|---|---|---|---|
| Si(220) | 42 | 1.36 | (211) | 71.2 | 73 | 71 | 21 | 58 |
| Si(220) | 45 | 1.46 | (211) | 77.1 | 82 | 68 | 21 | 53 |
| Si(220) | 48 | 1.55 | (211) | 82.9 | 105 | 77 | 26 | 58 |
| Si(220) | 51 | 1.65 | (211) | 90.1 | 119 | 68 | 24 | 48 |
| Si(111) | 43 | 2.28 | (110) | 68.5 | 90 | 64 | 18 | 53 |
| Si(111) | 45 | 2.39 | (110) | 72.1 | 100 | 83 | 24 | 67 |
| Si(111) | 46 | 2.44 | (110) | 73.8 | 84.5 | 80 | 24 | 64 |

***Austenitic steel (stainless steel 304L, f.c.c.).***

| Monochromator | $2\theta_M$ (°) | $\lambda$ (Å) | Reflection plane | $2\theta_S$ (°) | FoM | l (mm) | $D_{ref}$ (mm) | $D_{tr}$ (mm) |
|---|---|---|---|---|---|---|---|---|
| Si(220) | 38 | 1.24 | (311) | 69.6 | 54 | 70 | 20 | 57 |
| Si(220) | 45 | 1.46 | (311) | 85.6 | 93 | 72 | 24 | 53 |
| Si(220) | 46.5 | 1.5 | (311) | 87.8 | 120 | 75 | 26 | 54 |
| Si(220) | 53.2 | 1.71 | (311) | 104 | 148 | 76 | 30 | 47 |
| Si(111) | 41.2 | 2.19 | (111) | 63.8 | 97 | 87 | 23 | 74 |
| Si(111) | 45 | 2.39 | (111) | 70.1 | 91 | 79 | 23 | 65 |
| Si(111) | 49.5 | 2.61 | (111) | 78.0 | 100 | 83 | 26 | 65 |

[a]Maximum penetration depths in reflection ($D_{ref}$) and transmission ($D_{tr}$) geometries for different wavelengths in ferritic steel and austenitic steel. The maximum path lengths (l) were determined from the depth scans (80 $mm^3$ gauge volume, 1 h measurement time) for $10^{-4}$ accuracy in strain.The figure of merit (FoM) was calculated by using the integral intensity (I) and peak width (*FWHM*) of the diffraction peak from the powder samples.

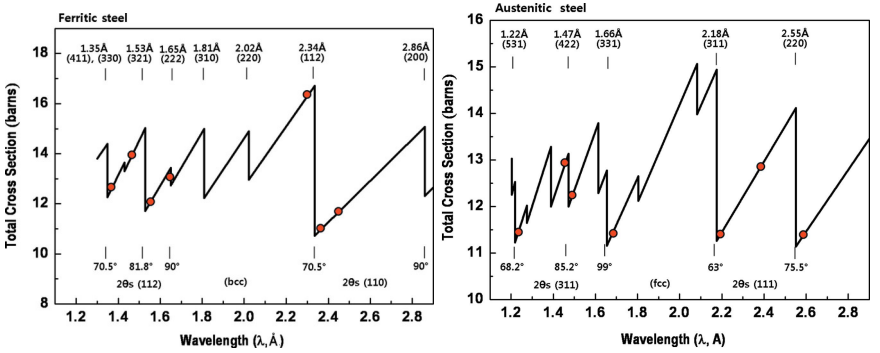


**Fig. 5.** Total neutron cross section of the ferritic and astenitic steel as a function of wavelength. Indices (hkl) of several Bragg edges and corresponding wavelengths are shown at the top. Scattering angles of reflections corresponding to the wavelength at Bragg edges are shown at the bottom. The wavelengths marked with filled circles were tested in the current study.

Consequently, a proper choice of the neutron wavelength can have a big influence on the neutron attenuation and maximum feasible penetration depth. This possibility was examined by using neutron wavelengths in the close vicinity of the Bragg

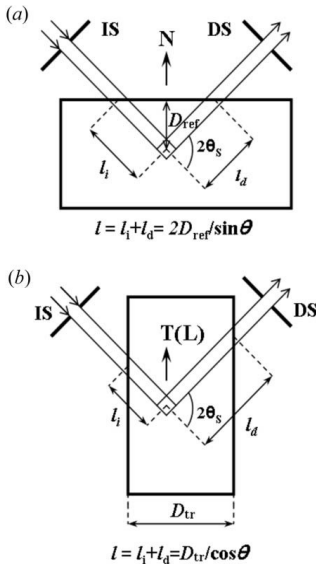


**Fig. 6.** The reflection (a) and the transmission (b) geometries of a sample.

edges, namely, for the wavelengths convenient for stress measurements. The geometry of the experiment for strain /stress measurements is shown in Fig. 6. The reflection geometry permits the measurements of the normal (N) component while the transmission one is used for the measurements of the transverse (T) or longitudinal (L) components. The gauge volume is defined by slits in the incident (IS) and diffracted (DS) beams. The neutron path length (l) is the sum of the incident ($l_i$) and diffracted ($l_d$) beam path lengths. l increases with depth in the case of (a), while it is the same for all depths in (b). Table 1 shows the obtained results. It can be seen from it that by using suitable wavelength of 2.39 Å or 2.19 Å for ferritic or austenitic steel, respectively, the diffractometer employing a focusing BPC-Si(111) monochromator is able to scan the strains effectively even for the total beam path length of about 85 mm in both ferritic and austenitic steels. It means that the normal N-component in the reflection mode can be effectively measured at the depth of about 25 mm. If we take into account that by simple rotation of the sample by 180° the N-component can be measured from the other side of the sample, the total thicknes which can be scanned with the 2 mm spatial resolution could be 2x25 mm [8]. Of course, that by relaxing the spatial resolution or by using a longer measurement time, the depth in the material where the measurement could be carried out can be enhanced up to about 35 mm and thus, the total thickness of the sample would achieve 70 mm. This value is considerably larger than estimations caried out on conventional instruments [9] Fig. 7 shows an example of stress investigation in the vicinity of low-carbon steel weld on 50 mm thick steel plate [10].

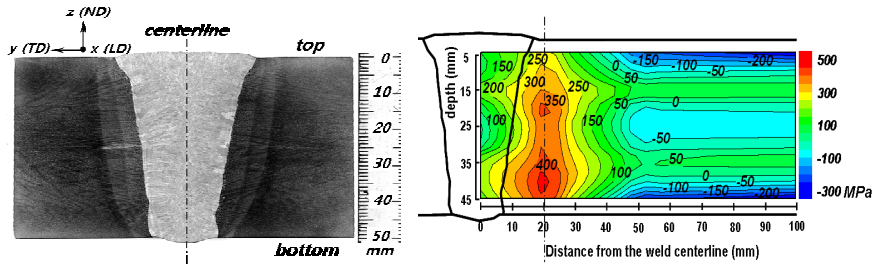


**Fig. 7.** Macrostructure of the 50-mm thick low-carbon steel weld (noted the LD -longitudinal - x), TD - transverse - y) and ND - normal - z directions of the weld plate) and the two-dimensional map of the longitudinal residual stress ($\sigma_x$).

## 6. Conclusion

The basic properties of unconventional strain/stress diffractometer performance employing Bragg diffraction optics are presented. It has been demonstrated that even at the medium power research reactor one can achieve very good luminosity and resolution of the dedicated instrument, which permit to carry out effectively macro-strain/stress scanning but also micro-strain/stress studies and even to study some kinetic processes in polycrystalline materials running within a few seconds. Some improvements are still possible e.g. by installing horizontally and vertically focusing monochromator and a position sensitive detector od a better spatial resolution. As the vertical focusing has no influence on the resolution of the instrument, its future installation could improve the luminosity by a factor of 2-3.


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