# On the reliability of neutron diffraction for residual stress measurement in cold-drawn steels

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**Abstract**. Residual strains were measured in the ferrite phase of pearlitic steel rods along the radial, axial and hoop directions. Two samples with different initial diameters were subjected to one drawing pass (using same drawing parameters) with 20% section reduction and measured in two different neutron diffraction instruments. The results show that the residual strain state is very similar in both cases, regardless of the diameter of the initial rod. This means that the final residual strain-stress state is unique and it is related to the cold-drawing process parameters. In addition, the results show the reliability of strain scanning with different neutron instruments and experimental conditions.

## 1. Introduction

Wire drawing is a manufacturing process employed to improve mechanical properties by coldworking. The process consists of reducing the wire section by successive passes through a set of conical dies. This process is widely employed in industry for producing high tensile strength pearlitic steel wires that find widespread applications as structural reinforcements, such as prestressed concrete, mining and fishing cables and tire reinforcements [1]. Inhomogeneous plastic deformations associated with the fabrication process involve the development of residual stress and texture gradients across the section of the wire. The residual stress pattern developed for deep drawing consists of tensile stresses at the wire surface and compressive ones in the interior [2-5].

It is well known that residual stresses may influence both the mechanical behaviour of the wires and their durability. In cold-drawn eutectoid steel wires, the presence of residual stresses decreases the elastic limit and increases the stress relaxation losses [6,7]. In addition, time to rupture in stress corrosion tests is clearly reduced with tensile residual stresses at the wire surface [8]. Consequently, it is important to control the residual stress profile resulting from cold-drawing in order to optimize wire performance.

In this work, samples of pearlitic steel rods were studied. Two samples with different initial diameters were subjected to one drawing pass (using same drawing parameters) with 20% section reduction and measured in two different neutron diffraction instruments. Through-thickness residual strain profiles were measured by neutron diffraction in the ferrite phase along the three principal directions of the rods, namely radial, hoop and axial. The main objective of this work is to assess the effect of the cold-drawing process with different initial rods. In addition, the reliability of neutron

diffraction measurements was checked. The results show that the residual strain-stress state is very similar in both cases, regardless of the diameter of the initial rod and the instrument employed.

## 2. Experimental

## 2.1. Material

Straight rods (ø20 and ø16 mm diameter and 6 m length) of pearlitic steel (eutectoid steel) were specially produced for this research by Saarstahl AG (Völklingen, Germany). The rods were produced by hot rolling and aged to reduce residual stresses to a minimum. The chemical composition is given in Table 1.

Table 1. Chemical composition of pearlitic steel rods (weight %)					
C (%)	Si (%)	Mn (%)	P (%)	S (%)	Al (%)
0.78	0.15-0.35	0.60-0.90	<0.025	<0.025	0.02-0.06

Samples were cold-drawn in one pass (in precisely controlled conditions) with a 20% section reduction (final diameter of 18 mm and 14.3 mm respectively). Die geometry was precisely measured in both cases. Details are given elsewhere [9]. The rods were kept straight during the whole process in order to avoid any change in the residual stress pattern generated by drawing.

#### 2.2. Neutron diffraction measurements

Measurements were performed with the REST diffractometer (NFL-Studsvik, Sweden) for the ø18 mm sample and with Stress-Spec Diffractometer (FRMII-Garching, Germany) for the ø14.3 mm sample.

REST is a two-axis high-resolution diffractometer, equipped with a double focusing monochromator that provides a neutron beam with wavelength between 1.66 and 1.76 Å, and a position sensitive detector. In this case the 110  $\alpha$ -Fe reflection was chosen for the investigation, which produces a Bragg peak at about  $2\theta = 49.7^{\circ}$  for the above-mentioned wavelength. Strain scanning was carried out along one diameter, by measuring one point every 1.5 mm. The specimen was reoriented to measure strains in the three principal rod directions: axial, radial and hoop. The slits connected to the diffractometer defined gauge volumes different for each component, which are depicted in Fig. 1. For the axial measurements (rod axis parallel to scattering vector) typical slits were 1.5 x 3 mm<sup>2</sup> (incident) and 1.5 mm (final). For radial and hoop measurements (rod axis perpendicular to scattering vector), the beam could be enlarged vertically without loss in spatial resolution (due to the symmetry of the sample). So, in this case the slit dimensions were 1.5 x 6 mm<sup>2</sup> (incident) and 1.5 mm (final). In all cases the resulting scattering volume provides reasonably good spatial resolution.

Stress-Spec is located at the thermal beam port SR3 of FRM II. The setup employed in the measurements on ferrite consists of a bent silicon monochromator Si(400) at a take-off angle of 82°, which corresponds to a wavelength of approximately 1.67 Angstrom. The monochromatic beam is diffracted by the specimen and detected by a position sensitive detector with an area of 20x20 cm2.

In this case ferrite strains were measured using the (211) diffraction line with  $\lambda = 1.6714$  Å. This combination produces a Bragg peak around  $2\theta = 91^{\circ}$ , very close to the optimum (90°). Again, ferrite lattice spacing was collected in the axial, radial and hoop directions of the samples, by measuring one point every mm. The strain scanning was carried out along the rod diameter. The slits connected to the diffractometer defined different gauge volumes for each component (with the aim of achieving the same spatial resolution). A nominal gauge volume of  $2x2x2 \text{ mm}^3$  was used in the axial measurements. For radial and hoop measurements, slit dimensions were  $2x10 \text{ mm}^2$  (incident) and 2 mm (receiving).



**Figure 1.** Gauge volumes employed in the neutron diffraction experiments at NFL-Studsvik for the three components: axial, radial and hoop (dimensions are scaled)

Stress-strain analysis can be carried out by using Bragg's law:

$$2d_{hkl}\sin\theta = n\lambda\tag{1}$$

where  $\lambda$  is the wavelength, 2 $\theta$  is the scattering angle and  $d_{hkl}$  is the lattice spacing of the hkl reflection. The strain for every {hkl} set of planes can be obtained from the variation in d-spacing:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \tag{2}$$

where  $\varepsilon_{hkl}$  is the longitudinal strain in the direction of the scattering vector, and  $d_{hkl}^0$  is the unstressed lattice spacing of the hkl reflection. In REST measurements, the stress-free lattice spacing for the 110 reflection was measured from iron filings of the original rod before drawing (annealed for two hours at 500 °C to relieve stresses). On the other hand, in Stress-Spec experiments d<sub>0</sub> was computed for the 211 reflection from "comb-like" samples taken from the initial rods (before cold-drawing).

## 3. Experimental results

The experimental results corresponding to both samples are shown in Fig. 2. The error bars for the strains are equal or slightly larger than the symbols employed in the plots. The residual strains in the ferrite phase of the pearlitic rod are considerable (minimum around  $-3 \cdot 10^{-3}$  for the axial component at the rod centre) after cold-drawing. The residual strain field in both samples is remarkably similar in the three directions studied. It seems that any residual stress in the original rod is erased by the cold-drawing process, because the results are almost the same for two different initial rods. In addition, the repeatability of the results obtained in two different instruments and with different experimental conditions is striking. This proves the suitability of this technique to measure in-depth residual strain fields in materials and structures as a tool to validate finite element models.

# 4. Conclusions

Residual strain scanning was performed in the ferrite phase of two pearlitic steel rods along the radial, axial and hoop directions in two different neutron diffraction instruments. The results show that the residual strain state mainly depends on the deformation that the material suffers in the cold-drawing process (resulting from the section reduction and the die geometry), irrespective of the diameter of the initial rod. In addition, the results validate the reliability of the neutron diffraction technique for residual strain scanning by comparing results from two different instruments and experimental

conditions. It is remarkable that the experimental results agree very well although different methods were employed to measure  $d_0$  in both cases. It is known that this is one the main issues in residual stress measurements by neutron diffraction. These results provide very valuable information regarding the cold-drawing process in steel rods.



**Figure 2.** Residual strains in the radial, hoop and axial directions (ferrite phase) as a function of the relative radius (r/R) for both pearlitic rods (ø18 mm and ø14.3 mm)

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