Determination of crystallographic young’s modulus for sheet metals by in situ neutron diffraction

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Abstract. Elastic recovery is an important issue in sheet metal forming, especially in the context of the upcoming use of high strength steels due to shifted relations between Young's modulus and strength. One important factor when it comes to elastic recovery prediction is a deep understanding for the elasto-plastic characteristics of the material. Today in general simple elastic behavior with constant Young's modulus and Poisson's ratio is assumed. Macroscopic analysis in standard tests shows that these assumptions are insufficient for an appropriate prediction of elastic recovery in sheet metal forming, which is why different phenomenological correlation models are derived. An experimental setup and microscopic investigation to further prove these models and to verify the approaches on another scale for sheet metals is presented within this paper. In the study microscopic deformation behavior of loading and unloading of a HC260LA sheet metal is analysed using in-situ neutron diffraction. Based on the lattice plane strains an orientation specific crystallographic Young's modulus for different rolling directions is determined.

1. Introduction
The demand for affordable and safer vehicles with complex body shells due to modern designs and lightweight requirements issues a challenge for the sector of metal forming. Deep material understanding is essential to ensure the preferably, high accuracy. Although the amount of elastic deformation is small compared with the plastic deformation, the elastic recovery of sheet metals, the so called springback, can lead to serious problems in press-forming processes [1, 2]. The extent of elastic recovery principally depends on Young’s modulus and yield strength [3]. Hence, for high quality forming products it is necessary to have suitable material models [1, 4]. Generally, it is assumed that the elastic recovery is equivalent to the elastic deformation whereby the elastic portion can be determined with the Young’s modulus, yield strength and the Poisson’s ratio [5]. Among these parameters the influence of the Young’s modulus is the most significant one. Hence, valid knowledge of the characteristics of the Young’s modulus is necessary [6]. Prior studies have shown, that the Young’s modulus of metallic materials changes with increasing plastic deformation. Furthermore, unloading and reloading tests have shown characteristics, which differ significantly from standard assumed linear to a non-linear hysteresis behavior [1-9]. Moresin and Boivin [6] performed cyclic tensile-compression tests to evaluate Young’s modulus variation. The results show a decrease of Young’s modulus of 20% in relation to work hardening for several steel types. Cleveland and Ghosh [7] investigated the characteristics of the
Young’s modulus with a detailed consideration of the linearity of the loading and unloading phase. Therefore, cyclic tensile tests have been performed for a high strength steel and an aluminum material. For both materials the occurring recovery was higher than the expected elastic recovery. Furthermore, the results show that loading as well as unloading is non-linear. Also Mendiguren et al. [4] showed for a TRIP steel, that the tangent modulus (dε/da) for the loading phase decreases significantly as stress grows. In the experiments of Luo and Ghosh [10] the influence of different strain rates on the non-linear behavior has been considered determining no considerable effect. Similar investigations like Cleveland and Ghosh [7] were taken by Andar et al. [1], who also analysed Young’s modulus variation for two different steel alloys under cyclic loading and unloading. Subsequent Young’s moduli were lower compared to the initial Young’s modulus. Consequently, there occurs not only elastic but anelastic straining during loading and unloading [7]. Sun and Wagoner [11] describe the anelastic deformation as recoverable like elastic strain and energy-dissipating like plastic strain. In several studies explanations and assumptions for this anelastic behavior can be found [1, 4, 5, 7, 11-13]. Doege et al. [12] assumes, that the anelastic behavior occurs because of phase transformations. Thus, characteristics of the different phases lead to the decrease of the Young’s modulus. Cleveland and Ghosh [7] consider the tangent modulus for loading and unloading and divide the non-linear curve in three stages. The characteristics of the stages depend on the movement and behavior of mobile dislocations. Also Sun and Wagoner [11] explain the anelastic behavior through pile-ups of dislocations at grain boundaries. All studies above investigated the macroscopic elasto-plastic behavior of metallic materials. For a precise prediction of the macroscopic characteristics of sheet metals and proof of taken assumptions for anelastic behavior a detailed investigation of the microscopic behavior is essential [13]. Microstructure analysis models of Hart [14] and Ghosh [15] confirm the assumption that anelastic behavior is caused by dislocation movement and pile-ups at barriers. Yang et al. [2] investigated microscopic Young’s modulus with nano-indentation. A decrease of the modulus near to the grain boundaries could be observed. But there is a difference between outer and inner grain behavior [16]. Neutron diffraction is more suitable for measuring microscopic characteristics because the penetration depth of neutrons is higher than the one of nano-indentation. Hence, grains can be considered, which are entirely embedded in the matrix [17]. Using the neutron diffraction measurement, Tomota et al. [18] showed for interstitial-free and ultra low-carbon steel that while elastic deformation the lattice plane strains of different (hkl) are equal and approximately linear. After elastic deformation a clear deviation of lattice strains for different orientations (hkl) can be determined. Consequently, there is a dependence between the lattice strains and elastic-plastic deformation. Stebner et al. [19] investigated monoclinic NiTi with neutron diffraction. His results indicate, that material behavior until yielding is not predominantly elastic. The purpose of this work is the microscopic investigation of the crystallographic Young’s modulus $E_{hk}$ for the micro alloyed steel grade HC260LA. In order to do this, the applicability of neutron diffraction for analyzing sheet metals has to be proven. Neutron diffraction with thin sheet metals issues a challenge for the experimental setup, because sufficient measurement volume is necessary. The experimental approach and settings for this kind of measurement are described in detail. Further, in-situ investigation during loading and unloading allow for determining microscopic loading and recovery Young’s modulus with regard to lattice orientation.

2. Material and experimental methods

2.1. Material
For the investigations within this study the sheet metal HC260LA with a thickness of 1 mm is used. The chemical composition of the chosen material HC260LA is shown in table 1. The mechanical characteristics derived from standard tensile tests are summarized in Table 2. No heat- or any other pretreatment before the tests was performed.
Table 1. Chemical composition of HC260LA in mass%.

| Steel   | C  | Si | Mn  | P   | S   | Al  | Ti  |
|---------|----|----|-----|-----|-----|-----|-----|
| HC260LA | 0.100 | 0.500 | 0.600  | 0.025 | 0.025 | 0.015 | 0.150 |

Table 2. Mechanical properties of HC260LA.

| Steel   | Yield strength [MPa] | Tensile strength [MPa] | Fracture elongation [%] |
|---------|----------------------|------------------------|-------------------------|
| HC260LA | 295                  | 372                    | 27                      |

2.2. Experimental setup and neutron diffraction

Standard uniaxial tensile tests are performed in this study. DIN-Standard tensile flat specimens with a length and width of 165 mm and 20 mm and a measuring length and width of 50 mm and 12.5 mm are used for the experiments [20]. The specimen thickness was 1 mm. The research neutron source Heinz Maier-Leibnitz (FRM II) in Garching near Munich is used to measure microstructure behavior. The diffractometer STRESS-SPEC was used for in-situ measurement of the tensile tests [21]. Figure 1 simplifies the setup of the described experiment. Figure 2 shows the experimental setup.

![Figure 1. Schematic setup for neutron diffraction measurement.](image1)

![Figure 2. Experimental setup of the tensile test machine at the research neutron source FRM II.](image2)

The wavelength is dependent on the monochromator and can be adjusted between 1.0 Å and 2.4 Å [22]. For the experiments within this study a wavelength of 1.67 Å was chosen and for the detection of the diffracted neutrons, a two dimensional $^3$He-PSD detector was used. With the detector’s covering angular range of about 10°, the diffraction profiles of the lattice planes (200), (211) and (220) are
collected during tensile tests. Tensile loading was applied stepwise. During stops neutron diffraction measurement is performed. A holding time of two minutes ensure a high neutron intensity. To get results with constant stress values, the sample needs to be stabilized for the neutron diffraction measurement [23]. Therefore, the stop was force controlled by the test machine. Tomota et al. [18] showed, that the results of a conventional tensile test differ from the results of a tensile test with arresting stops for the neutron diffraction measurement, which further has to be considered.

2.3. Measurement method and modulus calculation

A Digital Image Correlation (DIC) system is used to determine the macroscopic strain providing the effective cross sectional area for true stress calculation (Figure 2). The DIC system is using an applied pattern for the strain determination. The occurring force is given by a load cell, which is connected to the tensile test machine.

The diffraction measurement method provides, that lattice orientations of the crystallites are measured separately and successively with respect to Bragg’s law [23]. With Bragg’s law the lattice distance \( d \) of different \((hkl)\) lattice planes can be calculated according to the following equation [24]

\[
n \cdot \lambda = 2d \cdot \sin(\theta).
\]

The equation is fulfilled, if the path difference of neutrons, which are scattered by neighboured lattice planes with an angle of \( 2\theta \), is a whole number multiple \( n \) of the wavelength \( \lambda \). Figure 3 shows the physical principal of Bragg’s law. Hence, the angular settings of the test arrangement (Figure 1) have to be adjusted to detect the particular diffraction spectra profiles \((211),(220)\) and \((200)\). Furthermore, through specimen alignment in general, the micro strain in longitudinal \( \varepsilon_1 \), width \( \varepsilon_2 \) or thickness \( \varepsilon_3 \) direction can be determined.

With this condition fulfilled micro strains respectively lattice strains of the different \((hkl)\) planes can be determined. The lattice plane strain \( \varepsilon_{hkl} \) is the relative change of the lattice distance \( d_{hkl} \) at the measurement points during the tensile test with respect to the initial lattice distance \( d_{hkl}^0 \):

\[
\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0}.
\]

With neutron diffraction particular grains are measured. With an appropriate test setting of the diffractometer and tensile test machine lattice plane strains in loading direction can be detected. The lattice strain \( \varepsilon_{hkl} \) isn’t equal to the global macroscopic strain. Thus, microscopic Young’s modulus \( E_{hkl} \) is a crystallographic orientation modulus and not directly comparable with the macroscopic Young’s modulus \( E \). With the definition of the lattice strain \( \varepsilon_{hkl} \) in equation (2), \( \varepsilon_{hkl} \) is the value of the average elastic engineering strain with the orientation of the particular \((hkl)\) plane in a polycrystalline specimen [19]. Qiu et al. [25] made assumptions for a NiTi steel to compare microscopic with macroscopic characteristics. The first assumption states, that macroscopic stress in all monoclinic lattice orientations is equal and uniformly distributed. The second assumption says, that volume fractions of the lattice orientations are constant. Following these assumptions the microscopic Young’s modulus \( E_{hkl} \) can be calculated with the lattice strain \( \varepsilon_{hkl} \) and the macroscopic true stress \( \sigma \) [19]. As a sufficient measurement volume is necessary to perform neutron diffraction, investigating sheet metal specimens with low
thickness issues a challenge. Specimen positioning in the neutron beam is crucial to ensure valid measurement results. The intensities of the diffracted beam have to be examined before the tensile test. Therefore, several measurements with different specimen positions using 0.1 mm steps were performed. Schematically the left side of Figure 4 shows specimen positioning in relation to the neutron beam. For every measurement point the integral intensity for the crystallographic orientation (211) was calculated to identify the position with maximum intensity (right side of Figure 4). With this methodology the focus in x-direction can be determined.

In addition to the definition of the focus in x-direction it is essential to proof that the measurement results are independent of the beam position in the sheet metal plane. Hence, measurement results of five different spots on the specimen have been compared to proof the homogeneity of the crystallographic strains. The specimen was moved along the y-axis as shown in Figure 5 with regard to the focus point on the x-axis. Figure 5 illustrates stress-micro strain curves of five different spots M1 – M5 for the crystallographic orientation (211). The experiment consists of two cycles with plastic transition during loading. This type of loading and unloading was chosen in order to cope three deformation modes: Elastic loading, plastic deformation and elastic unloading. Regarding the five measurement curves in Figure 5 no significant difference in the elastic as well as plastic range could be found. Hence, lattice strain homogeneity is to be assumed. It can be assumed that the deviation of the flow curve around 300 MPa occurs because of the Lüders strain.

**Figure 4.** Specimen positioning in the neutron beam to get maximum intensity.

**Figure 5.** Measurement at five different spots (M1-5) in the specimen to validate strain homogeneity for (211).
3. Results
First of all, the conducted study shows the possibility to analyze the microscopic characteristics of sheet metals. This was achieved using a suitable experimental setup including a routine for specimen positioning and the assurance of microscopic strain homogeneity in the utilized measurement volume. The results of Tomota et al. [18] showed, that the lattice plane strains of different orientations (hkl) are equal and almost linear during elastic deformation, whereas during plastic deformation a clear deviation of lattice strains with different orientations (hkl) is determined. This results could be confirmed with the conducted experiments in this study. Even the macroscopic pronounced elastic limit of HC260LA is visible in every microscopic stress-strain curve dividing the almost linear elastic loading from the plastic portion (Figure 5).

![Graph of Microscopic (211)](image)

![Graph of Microscopic (220)](image)

![Graph of Microscopic (200)](image)

**Figure 6.** Loading (without prestraining) and unloading (prestrained) curve for the lattice planes (211), (220) and (211) distinguished in three rolling directions.

The elastic behavior in $\varepsilon_1$-direction of different lattice planes for three rolling directions (0°, 45° and 90°) are considered. Figure 6 shows the stress-microstrain curve for initial loading and unloading after prestraining. The loading as well as the unloading measurement points can be approximated utilizing linear curves. The stress values for loading are calculated using the initial cross sectional area of the specimen. For the unloading phase the current cross sectional area derived from optical measurement was used to calculate the true stress at the point of time the unloading starts.
Table 3 shows the numerical values for the crystallographic Young’s moduli derived from the linear approximation of the measurement points for different rolling directions and lattice plane orientations. The results show that the rolling direction does not have a significant impact on the microscopic characteristics. The derived crystallographic Young’s moduli for initial loading are in good agreement with the literature value of 210 GPa for the macroscopic Young’s modulus of steels. Regarding unloading after prestraining steeper curves compared to the initial loading can be observed for all crystallographic orientations (211), (220) and (200) (Figure 6). Hence, the derived crystallographic Young’s moduli increase with growing prestraining for the micro alloyed steel grade HC260LA.

**Table 3.** Orientation specific crystallographic Young’s moduli of loading and unloading curves of different lattice planes.

| Lattice direction \((hkl)\) | (211) | (211) | (211) | (220) | (220) | (200) | (200) |
|---------------------------|-------|-------|-------|-------|-------|-------|-------|
| Rolling direction [°]     | 0     | 45    | 90    | 0     | 45    | 90    | 0     |
| Loading Young’s Modulus \(E_{hkl}\) (without prestrain) [GPa] | 212   | 208   | 206   | 194   | 198   | 169   | 189   | 194   | 165   |
| Unloading Young’s Modulus \(E_{hkl}\) (prestrained) [GPa]       | 249   | 257   | 242   | 226   | 238   | 219   | 218   | 209   | 219   |

**4. Conclusion**

This study deals with microscopic material behavior of the micro alloyed steel grade HC260LA. For the experimental investigation in-situ measurements during tensile tests utilizing neutron diffraction were performed with sheet metal of 1 mm thickness. The analysis within the study shows the possibility of using neutron diffraction for in-situ evaluation of microscopic behavior of thin sheet metals.

Therefore, a methodology is proposed for the accurate positioning of sheet metal specimens in relation to the neutron beam to achieve maximum measurement intensity. Furthermore, neutron diffraction was performed at five different measurement spots to show independence of the measurement position in the sheet metal plane. The homogeneity of the microscopic strain distribution in the tensile specimen was proven for elastic loading, plastic deformation as well as for elastic unloading. The measurement method enables the determination of orientation specific crystallographic Young’s moduli.

With the proposed experimental setup crystallographic Young’s moduli for the lattice planes (211), (220) and (200) in three rolling directions \((0°, 45° \text{ and } 90°)\) have been calculated for the loading and unloading phase with prestraining. From the obtained results no significant influence of the rolling direction is recognizable. The derived values of microscopic Young’s modulus for elastic loading show good agreement with the macroscopic values assumed for steel. The unloading crystallographic Young’s moduli after prestraining show higher values compared to the crystallographic Young’s moduli for the initial elastic loading (without prestraining). Hence, an increase of the microscopic Young’s moduli with growing prestrains can be observed.

In general the proposed measurement setup provides the basis to investigate the microscopic behavior of sheet metals especially with regard to microscopic elasticity. For neutron diffraction experiments a discontinuous tensile testing is necessary to receive sufficient intensity. Thus, temporal behavior of the conducted material and mechanical characteristics of the testing machine should be taken into account for increased accuracy. In order to expand the microscopic analysis of this study to nonlinear elastic phenomena, the accuracy of the proposed experimental setup has to be enhanced with regard to the abovementioned experimental constraints.

The possibility to analyze the microscopic elastic behavior of sheet metals with in-situ neutron diffraction offers potential connecting microscopic and macroscopic material characterization especially with regard to elastic recovery prediction. Therefore, in consideration of microscopic elastic effects, existing phenomenological models could be tested and enhanced.
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