Effect of crystallographic orientations of grains on the global mechanical properties of steel sheets by depth sensing indentation

P Burik\textsuperscript{1}, L Pesek\textsuperscript{2}, P Kejzlar\textsuperscript{3}, Z Andrsova\textsuperscript{3} and P Zubko\textsuperscript{2}

\textsuperscript{1}Technical University of Liberec, Faculty of Mechanical Engineering, Department of Material Science, Liberec, Czech Republic
\textsuperscript{2}Technical University of Kosice, Faculty of Metallurgy, Department of Materials Science, Kosice, Slovakia
\textsuperscript{3}Technical University of Liberec, Institute for Nanomaterials, Advanced Technology and Innovation, Liberec, Czech Republic

E-mail: peter.burik@tul.cz

Abstract. The main idea of this work is using a physical model to prepare a virtual material with required properties. The model is based on the relationship between the microstructure and mechanical properties. The macroscopic (global) mechanical properties of steel are highly dependent upon microstructure, crystallographic orientation of grains, distribution of each phase present, etc... We need to know the local mechanical properties of each phase separately in multiphase materials. The grain size is a scale, where local mechanical properties are responsible for the behavior. Nanomechanical testing using depth sensing indentation (DSI) provides a straightforward solution for quantitatively characterizing each of phases in microstructure because it is very powerful technique for characterization of materials in small volumes.

The aim of this experimental investigation is: (i) to prove how the mixing rule works for local mechanical properties (indentation hardness H\textsubscript{IT}) in microstructure scale using the DSI technique on steel sheets with different microstructure; (ii) to compare measured global properties with properties achieved by mixing rule; (iii) to analyze the effect of crystallographic orientations of grains on the mixing rule.

1. Introduction
Real materials such as multi-phase steels exhibit on the microscopic scale (microstructure) a heterogeneous material. Multiphase steels combine a high strength and a high formability, which is a condition for deep drawing parts in the automotive industry [1]. The overall behaviour of microheterogeneous steels depends strongly on the size, shape, spatial distribution and properties of the microstructural constituents and their respective interfaces [2]. As the properties of these materials are determined by the contributions from the constituent phases, the properties of separate phases and their volume fraction are the key factors for product design. Accordingly, there is a need for a reliable tool for phase analysis. Electron backscatter diffraction (EBSD), which is principally a microtexture determination technique, is a popular tool for microstructure characterization. EBSD is a technique based on the analysis of the Kikuchi pattern by the excitation of the electron beam on the surface of the sample in a scanning electron microscope (SEM) [3].
A micromechanical approach based on volume fraction components enables to describe the heterogeneities with the help of continuum mechanical quantities and by this to link mechanical properties at different scales. Thereby it is the goal to derive effective material properties for the component from microstructure quantities and to quantify the effect of microstructural features. The rule of mixture means - macroscopic properties of aggregates or composites consisting of two or more constituents can be obtained as the sum of the volume fraction of the components times their individual properties [1].

The composite property is estimated as the sum of the responses of the composite components weighted by the component volume fractions as shown in the equation below:

\[ P = \sum_i^n V_i P_i \]  

(1)

where \( P \) - some property and \( V \) - the volume fraction of the components, \( i = 1, 2, 3, \ldots n \) [4].

Mechanical properties of individual components can be determined by depth sensing indentation derived from the indentation load-displacement data used a micromechanical model developed by Oliver & Pharr (O&P) [5].

In the O&P analysis, the hardness, \( H_{IT}^{O&P} \), is determined from the load-displacement curve as:

\[ H_{IT}^{O&P} = \frac{P_{\text{max}}}{A_p^{O&P}} \]  

(2)

where \( P_{\text{max}} \) - the maximum indentation load and \( A_p^{O&P} \) - the projected contact area at \( P_{\text{max}} \).

The projected contact area \( A_p^{O&P} \) is calculated by evaluating an empirically determined indenter shape function \( A_p^{O&P} = f(h_c^{O&P}) \). For an ideal Berkovich indenter, it is given by:

\[ A_p^{O&P} = 24.56 h_c^{O&P} \]  

(3)

where \( h_c^{O&P} \) - the contact depth between material and indenter at the \( P_{\text{max}} \) [6].

2. Experimental materials and methods

2.1 Materials

The characteristic microstructure of the investigated material:

(i) XSG steel - interstitial free steel (ferrite microstructure), Figure 1a. The mean size of the ferrite grain is 19 \( \mu \text{m} \).

(ii) HR 45 steel - microalloyed steel (ferrite-pearlite microstructure), Figure 1b. The mean size of the ferrite and pearlite grain is 7.9 \( \mu \text{m} \) and 3.9 \( \mu \text{m} \), respectively. The volume fraction of pearlite is 14%.

(iii) DP 600 steel - dual phase steel (ferrite-martensite microstructure), Figure 1c. The mean size of the ferrite and martensite grain size is 12 \( \mu \text{m} \) and 2.7 \( \mu \text{m} \), respectively. The volume fraction of martensite is 27 %.

Chemical composition, mechanical properties and thickness \( t \) of investigated steel sheets are in Table 1.

![Figure 1. Microstructure of investigated steels, a) XSG, b) HR 45, c) DP 600](image-url)
Table 1. Chemical composition (wt. %), mechanical properties (Rp0.2 - yield stress, Rm - tensile strength, A80 - elongation, n - strain hardening exponent) and thickness t of investigated steel sheets

| Steel | C   | Mn  | V   | S   | P   | Rp0.2 (MPa) | Rm  (MPa) | A80 (%) | n (-) | t (mm) |
|-------|-----|-----|-----|-----|-----|-------------|-----------|---------|-------|--------|
| XSG   | 0.0013 | 0.082 | 0.002 | 0.0105 | 0.011 | 177         | 286       | 47.2    | 0.211 | 1.95   |
| HR 45 | 0.156 | 0.654 | 0.002 | 0.004 | 0.013 | 360 R_e     | 449       | 27      | 0.139 | 1.80   |
| DP 600| 0.072 | 1.807 | 0.003 | 0.006 | 0.017 | 388         | 581       | 26.1    | 0.160 | 1.60   |

2.2 Methods
The nanoindentation tests were carried out at room temperature using a CSM instrument equipped with a Berkovich tip at a constant loading rate of 400 mN/min from 0 to the maximum force 5 mN and 500 mN, respectively, with 10 s hold period and constant unloading, the distances between the indentations were 10 μm (P_{max} = 5 mN) in the 11 x 10 matrix and 100 μm (P_{max} = 500 mN) in 3 x 1 matrix. The force-displacement curves were analyzed using the O&P analysis. After indentations, the indent location was observed via scanning electron microscope and the crystallographic orientation of grains with individual orientations was examined by means of EBSD method. High resolution EBSD images were prepared on an area ~ 120 x 110 μm using a step size of 300 nm and applying a tilt angle of 70°. The obtained EBSD data were analyzed by HKL Channel 5 software. Pairing of the measured mechanical properties to the corresponding crystallographic orientation of each indent was carried out so that the indents located inside the grains were taken into account, those which corresponded to the close grain boundary area of crystals (closer than the diameter of an indent) were neglected from the evaluation.

3. Results and discussion
Figure 2 shows inverse pole figure (IPF) map which describes the crystallographic orientation of detected grains of XSG steel. IPF map of the investigated area on which the nanoindentation tests were performed, practically completely covers the whole possible grain crystallographic orientations. The indentation hardness is dependent on the crystallographic orientation, Table 2. The indentation hardness is substantially lower in (001) plane than in both (101) and (111) planes in all steels. XSG steel has the lowest difference between indentation hardness of (001) plane and indentation hardness of both (101) and (111) planes (about 7%). DP 600 steel has the greatest difference between indentation hardness of (001) plane and indentation hardness of both (101) and (111) planes (about 11%).

Effect of crystallographic orientation on the indentation hardness of pearlite component in the HR 45 steel and of martensite component in the DP 600 steel are not determined due to the fine grain microstructure of these components; indent size is equal to the grain size of the component and therefore it is not possible to accurately determine the hardness for the given crystallographic orientation of the grains.

The values of mechanical characteristics of ferrite in different crystallographic planes have a high standard deviation due to:
- High surface roughness: for example - HR 45 steel R_a = 0.053 ± 0.006 μm, the maximum depth in (001) plane h_{max} = 0.359 ± 0.017 μm; in (101) plane h_{max} = 0.346 ± 0.019 μm and in (111) plane h_{max} = 0.351 ± 0.013 μm.
- Different indentation distance from the grain boundary.
- Roughness of the soft phase and that of hard phase is not equal, slip of the indenter can occur.
- Depth of the indented area is unknown; it is likely that measured hardness value of hard component is affected by the soft component which is under indented hard grain and opposite the soft component may be affected by the hard one lying under that. Therefore the hardness value of hard component may be shifted to lower and that of soft component to higher value.
Effect of crystallographic orientation on the global indentation hardness of ferrite was determined using various combinations of indentation hardness of individual planes to the volume fraction of individual planes according to the following models for indentation hardness of ferrite:

1. Based on relative ratio of basic planes volume fraction:

\[ H_{IT}^{ferrite} = H_{IT}^{001}V_{001} + H_{IT}^{101}V_{101} + H_{IT}^{111}V_{111} \]  

where the values of \( V_{001} \), \( V_{101} \), \( V_{111} \) are calculated as:

\[ V_{001} = \frac{100}{V_{real(001)}+V_{real(101)}+V_{real(111)}} + V_{real(001)} \]  

\[ V_{101} = \frac{100}{V_{real(001)}+V_{real(101)}+V_{real(111)}} + V_{real(101)} \]  

\[ V_{111} = \frac{100}{V_{real(001)}+V_{real(101)}+V_{real(111)}} + V_{real(111)} \]
2. Based on real ratio of the basic planes volume fraction and the arithmetic average of the other planes volume fraction:

\[
\frac{H_{ITferrite}}{3} = \frac{H_{ITferrite}(001)V_{real(001)} + H_{ITferrite}(101)V_{real(101)} + H_{ITferrite}(111)V_{real(111)}}{(H_{ITferrite}(001)/101) + H_{ITferrite}(101)/111} + \frac{V_{real(001)/101)}{111)} + V_{real(001)/111)} + V_{real(111)/101)}
\]  

(8)

3. Based on a real ratio of all planes volume fraction:

\[
H_{ITferrite} = H_{ITferrite}(001)V_{real(001)} + H_{ITferrite}(101)V_{real(101)} + H_{ITferrite}(111)V_{real(111)} + \\
H_{ITferrite}(001)/101) + V_{real(001)/1(111)} + V_{real(001)/111)} + V_{real(111)/101)}
\]  

(9)

where the values of \(V_{real(001)}\), \(V_{real(101)}\), \(V_{real(111)}\), \(V_{real(001)/1(101)}\), \(V_{real(001)/1(111)}\), \(V_{real(111)/1(101)}\) for model 2 and model 3 are from the Table 3.

Using a mixture rule we can obtain the model data. Indentation hardness of ferrite in mixture rule varies according to the individual model and the indentation hardness of other components (pearlite component in HR 45 steel, martensite component in DP 600 steel) in the mixture rule is constant.

Mixture rule can be rewritten as following equation:

\[
XSG steel \quad H_{ITglobal} = H_{ITferrite}
\]

\[
HR 45 steel \quad H_{ITglobal} = H_{ITferrite}V_{ferrite} + H_{ITpearlite}V_{pearlite}
\]

\[
DP 600 steel \quad H_{ITglobal} = H_{ITferrite}V_{ferrite} + H_{ITmartensite}V_{martensite}
\]

where \(H_{ITglobal}\) - model for global indentation hardness, \(H_{ITferrite}\) - indentation hardness of ferrite calculated according to the model 1, 2, 3 (equations (4), (8), (9)), \(H_{ITpearlite}\) indentation hardness of component (pearlite, martensite), Table 4, \(V\) - the volume fraction of the component.

Bulk indentation hardness - means data for series of large indents (\(P_{max} = 500\) mN). Model for \(H_{IT}\) - means data calculated using mixture rule (equation (10) - (12)) with corresponding ferrite, ferrite / pearlite and ferrite / martensite content. Modeling parameter \(X = \) model for \(H_{IT}\) / bulk \(H_{IT}\). The more the \(X\) is close to 1, the better works the mixture rule model.

**Table 4.** Indentation hardness \(H_{IT}\) (MPa) (\(P_{max} = 5\) mN) for different structural components

| Steel Model for \(H_m\) of ferrite (calculated) | XSG | HR 45 | DP 600 |
|-----------------------------------------------|-----|-------|--------|
| 1. model                                      | 1665| 1980  | 2231   |
| 2. model                                      | 1670| 2042  | 2372   |
| 3. model                                      | 1687| 2042  | 2389   |
| \(H_m\) pearlite (P), martensite (M) (measured) | 2423(P) | 2582(M) |

Model 1, 2, 3 for indentation hardness \(H_m\) of ferrite calculated according to the model 1, 2, 3 (equation (4), (8), (9)), \(H_m\) pearlite and martensite - indentation hardness \(H_{IT}\) of components of small indents (\(P_{max} = 5\) mN) positioned into pearlite and martensite grains.
Global indentation hardness of ferrite is dependent on the individual model for indentation hardness of ferrite, Table 4. Models for global indentation hardness of ferrite contains “between planes” (model 2 and model 3) have greater indentation hardness than model based on relative ratio of basic planes volume fraction (model 1). Table 5 compares the measured and calculated (model) global mechanical properties. It can be observed the difference between the measured mechanical properties and calculated properties. Bulk indentation hardness gives greater value than model value for global indentation hardness because in bulk materials are included grain boundaries and precipitates which increase the indentation hardness. Model for global indentation hardness based on a real ratio of all planes volume fraction (model 3) is closer to bulk indentation hardness than model for global indentation hardness based on relative ratio of basic planes (model 1) volume fraction. XSG steel with coarse-grained microstructure of ferrite has the difference between the model based on real ratio of all plane volume fractions and the model based on relative ratio of basic planes volume fraction only 1.3%. DP 600 steel has the greatest difference between model based on real ratio of all plane volume fractions and model based on relative ratio of basic planes volume fraction (~ 4.1 %) because DP 600 steel has fine grained microstructure of ferrite and DP 600 steel has the greater difference between individual planes between basic planes ((001), (101), (111)) and “between planes” ((001) (111) and (101) (111)).

Table 5. Bulk (measured) and model (calculated 1, 2, 3) global indentation hardness $H_{IT}$ (MPa)

| Steel  | Bulk $H_{IT}$ (Pmax= 500 mN) | Model for $H_{IT}$ (Pmax = 5 mN). | X= Model for $H_{IT}$ / Bulk $H_{IT}$ (1) |
|--------|-----------------------------|----------------------------------|------------------------------------------|
|        |                             | 1. 2. 3.                         | 1. 2. 3.                                  |
| XSG    | 1763                        | 1665 1670 1687                    | 0.944 0.947 0.957                         |
| HR 45  | 2425                        | 1980 2042 2042                    | 0.842 0.864 0.864                         |
| DP 600 | 2765                        | 2231 2372 2389                    | 0.841 0.878 0.882                         |

4. Summary
Depth sensing indentation method is suitable for measuring the mechanical properties of each crystallographic orientation of ferrite grain in steel sheets with the aim to obtain the global mechanical properties from the particular mechanical properties using the mixture rule. The main limitation of the DSI method is measuring the mechanical properties of each crystallographic orientation of components with fine-grained microstructure.

Indentation hardness of ferrite in the steel sheets is dependent on the crystallographic orientation. Slip planes ((101), (111)) in body centred cubic crystal have higher hardness than the (001) plane. Investigated steel sheets contain approximately 50% of basic planes ((001), (101), (111)) oriented parallel to the surface.

Crystallographic orientations of ferrite have a significant effect on the global mechanical properties and therefore crystallographic orientation has to be included into the mixture rules in steel with ferrite. Model for indentation hardness calculated from the real ratio of crystallographic planes is closer to the bulk indentation hardness than model for indentation hardness calculated from the basic planes ((001), (101), (111)). Effect of crystallographic orientation of the ferrite on the mixing rules decreases with increasing size of the ferrite.

Acknowledgment
The paper was supported partly by Grant Agency of the Czech Republic thought the SGS project "Studium a hodnocení struktury a vlastností materiálů", partly by the project LO1201 were obtained with co-funding from the Ministry of Education, Youth and Sports as part of targeted support from the "Národní program udržitelnosti I" programme and partly by the VEGA project No. 1/0582/13 at the Technical University of Kosice, Slovakia.
References

[1] Prahl U Ramazani A Quade H and Twardowski R 2012 Forming Technology Forum Damage modeling of multiphase steel using microstructure based RVE technique

[2] Fereiduni E and Ghasemi Banadkouki S S 2013 Journal of Alloys and Compounds 577 Reliability / unreliability of mixture rule in a low alloy ferrite-martensite dual phase steel p 351-359

[3] Chen Z Yang Y and Jiao H 2012 Scanning Electron Microscopy Some applications of electron back scattering, diffraction (EBSD) in materials research

[4] Alabbasi F and Nemes J A 2003 International Journal of Mechanical Sciences 45 Micromechanical modeling of dual phase steels p 1449-1465

[5] Hay J L 2000 ASM Handbook 8, Mechanical testing and evaluation 236 Instrumented indentation testing p 232-244

[6] Zhou X Jiang Z Wang H and Yu R 2008 Materials Science and Engineering A 488 Investigation on methods for dealing with pile-up errors in evaluating the mechanical properties of thin metal films at sub-micron scale on hard substrates by nanoindentation technique p 318-332