

# Estimation of Strength Properties from Microhardness Results in Dual Phase Steels with Different Martensite Volume Fraction

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## Abstract

*This study is about the effect of the martensite volume fraction and indentation load on microhardness profiles of dissimilar types Dual Phase steels and DC04 mild steel. Experimental investigations were performed by mickrovickers method with using of eight different indentation loads from 0.01 kp up to 1 kp. Besides, microscope and tensile tests were carried out to complete the estimation.*

*The hardness profiles show similar characteristics in case of all examined steels independent from the microstructure. In the lowest load ranges at 0.01 and 0.025 kp ( $HV_{0.01}$  and  $HV_{0.025}$ ), there are no appropriate approximations with the martensite volume fraction, due to the high deviation of the hardness results which caused by the little indentation geometry. In higher ranges, above 0.05 kp ( $HV_{0.05}$ ), linear evaluations could be applicable. With the utilization of the fitted parameters, a definite relationship is reported in the hardness values and even in the strength and elongation properties with the martensite content. Based on these correlations such contexts are added which make contact between microhardness and strength values for the practice. The discrepancy between the measured and calculated results stay under 10 HV which is less than 5%.*

## Keywords

*indentation load, martensite volume fraction, dual phase steels*

## 1 Introduction

In order to achieving the necessary reduction of the greenhouse gas emission in the next few decades, the automotive industry continuously develops the light weight technologies and materials. To satisfy the global regulations, new high strength steels are applied to thinning the body-in-white elements. These steels are called as Advanced High Strength Steels (AHSS) which group includes the Dual Phase (DP) steels with others (Keeler and Kimchi, 2015; Malen and Hughes, 2015; Kuziak et al., 2008).

Dual Phase steels are widely used in the automotive industry thanks to those properly high uniform elongation and strain hardening rate, beside relatively high strength. Based on experimental results (Paul, 2013; Sodjit and Uthaisangsuk, 2012; Uthaisangsuk et al., 2011) the true stress of these steels can exceed the 600 MPa while those total true strain remains over 0.1. These mechanical properties are provided by the presence of the soft ferrite and hard martensite particles in the microstructure. Thereby the amount and the distribution of the martensite basically determines the strength and ductility attributions. The martensite volume fraction (MVF) is formed in the intercritical temperature range, depending on the holding temperature and the holding time ( Hoydick, 2004).

For this reason, there are more researches about the relationship between the MVF and mechanical properties. Rosenberg et al. (2013) investigated the effect of the MVF for the capacity of DP steel to absorb energy in the presence of stress concentrators. The absorb energy was expressed by the ultimate tensile strength and uniform elongation. They pronounced that with the increasing of MVF, the absorbed energy remained stable or decreased in the case of smooth tensile specimens, while it always increased in case of tensile specimens with stress concentrators. In the aspects of elongation and MVF, there are varied opinions. Researches of Sun et al. (2009), and Sodjit and Uthaisangsuk, (2012) show that the elongation decreases as the MVF increases, while other research of Movahed et al. (2009) presented that the elongation is decreased over 50 % MVF only.

Hardness test results were investigated in the function of MVF in the paper of de la Concepción et al. (2015). They identified linear correlation between  $HV_1$  hardness values and MVF. The microscopic properties of ferrite and martensite

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phases in DP steels were investigated by more authors (Zhang et al., 2016; Ghatei Kalashami, 2016) also. They used nanoindentation tests to complete their microstructure examinations, and gave a complex evaluation of such properties like dislocation structure or fracture mechanism.

Present paper differs from the previous researches that they did not take into consideration the effect of the indentation load. In our research, microvickers tests were used with different indentation loads on DP steels with different MVF, and on DC04 mild steel. The results showed that clear correlation exists between the hardness values and the MVF in more indentation load ranges.

## 2 Materials

Three types of conventional DP steels as DP600, DP800 and DP1000, and a commercial mild steel - DC04 - with the chemical composition shown in Table 1 were applied in this study. The chemical compositions were defined by Foundry Master Pro optical spectrometry. The DP steels are certificated and delivered by SSAB Swedish steel company, while the DC steel comes from a home supplier.

Small pieces were grinded and polished by using standard metallographic sample preparation techniques and then etched in 2 % nital. The microstructures of different DP steels are shown in Fig. 1.

**Table 1** Chemical compound of the investigated steels

	Fe (wt%)	C (wt%)	Si (wt%)
DP600	98.6	0.085	0.171
DP800	97.9	0.161	0.187
DP1000	97.8	0.148	0.181
DC04	99.5	0.050	0.023
	Mn (wt%)	P (wt%)	S (wt%)
DP600	0.87	0.013	0.005
DP800	1.52	0.012	0.003
DP1000	1.50	0.012	0.004
DC04	0.23	0.012	0.009

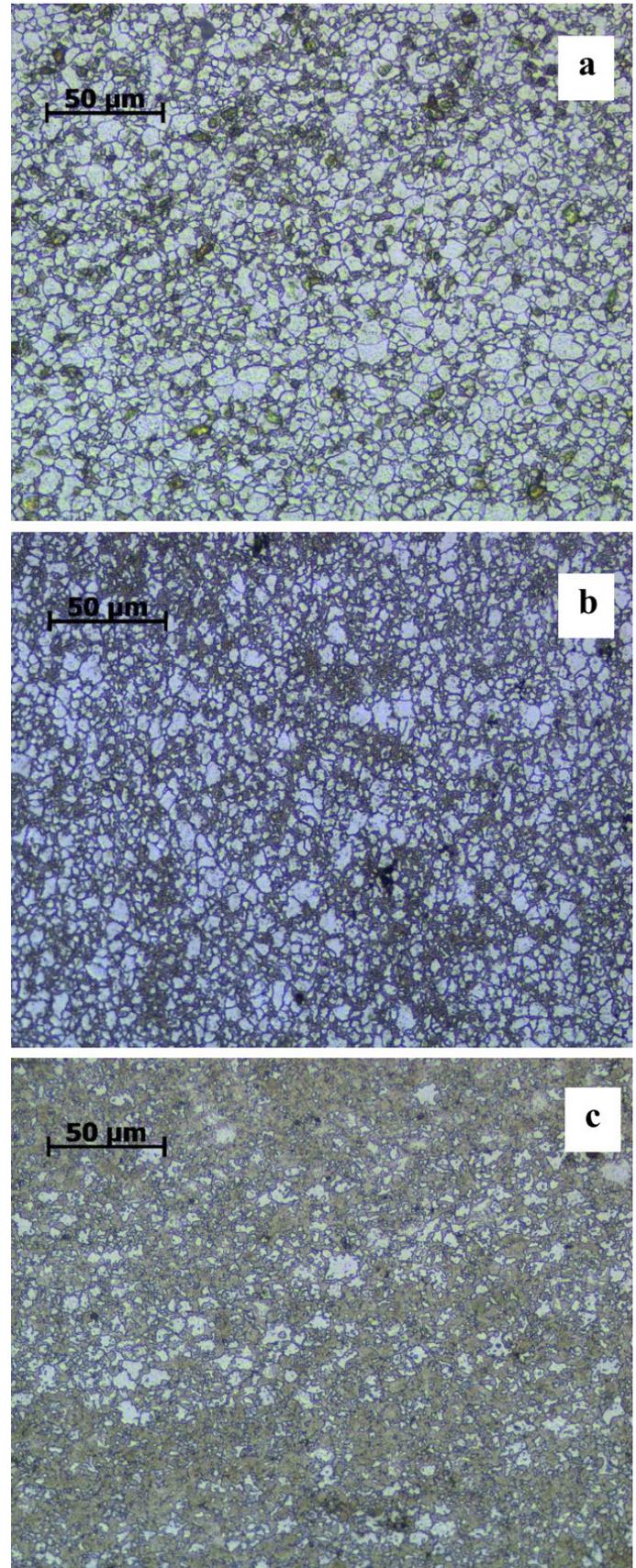
## 3 Experimental details

### 3.1 Microstructure characterization

The microstructures of DP steels were examined by the built-in automatic measurement program of Zeiss Imager M2m optical stereo microscope. The area percent module detected each phases and defined those area fraction in the examined plane. With the investigations of more planes, the volume fraction of the constitutive phases could be calculated.

The phase proportions i.e. the ferrite and martensite volume fractions are 0.734-0.266 for DP600, 0.579-0.421 for DP800 and 0.350-0.650 for DP1000 steels. The first numbers match with the amount of the ferrite.

It is worthy to note that DP800 has a bit higher carbon content than DP1000 as shown by Table 1. It is contrary to the general experiences that higher carbon content belongs to higher MVF.



**Fig. 1** Microstructure of a) DP600, b) DP800 and c) DP1000 materials, etched in 2% nital

DC04 mild steels has homogenous ferritic microstructure (Fig. 2) with low carbon content (Table 1). These two features are responsible for it's good formability and low strength.

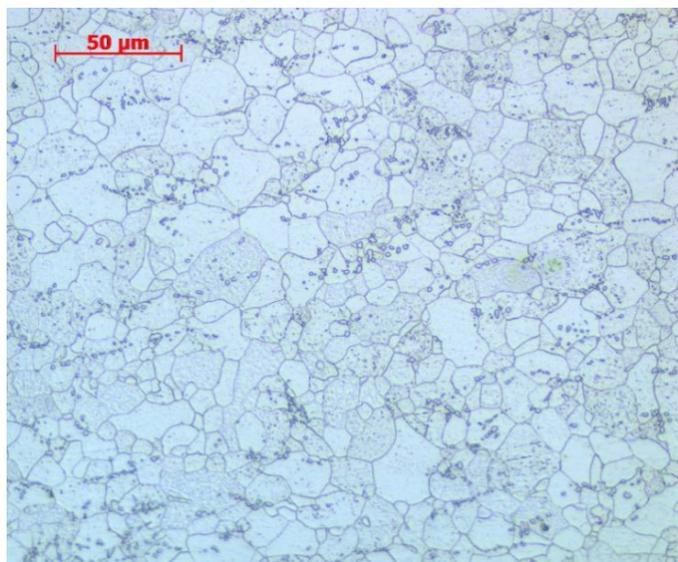


Fig. 2 Microstructure of DC04 ferritic mild steel

Seeing the microstructure of DP600 it can be observed that the martensite islands are consistently distributed in the ferrite matrix. This is different from DP800 where concentrated ferrite and martensite regions are visible, moreover coherent martensite zones appear at DP1000. In this way the properly definition of the grain size of the martensite particles is not feasible at the latter two materials. Otherwise, more literatures (Paul, 2013; Sodjit and Uthaisangsuk, 2012; Uthaisangsuk et al. 2011) highlight only the ferrite grain size i.e. the free dislocation path in the ferrite as an influencing factor of the mechanical properties. These papers contain the microstructure/dislocation based strain hardening theory developed especially for DP steels in details.

In Fig. 3 the most common ferrite grain size dispersion moves around 2-5 μm at all types of DP steels, resulted by more than fifty randomly measured diameters, in different cross sections. Steady grain size dispersion characterizes the DP600, and with the increasing of MVF the average ferrite diameter reduction become typical for grains. Especially on Fig. 1 c) where concave grain boundaries occurred also as a result of partial solution, the average ferrite grain diameters stay under 3 μm.

### 3.2 Mechanical testing

Microhardness measurements were performed by Wilson Wolpert 401 MVD test machine in Vickers method. The eight applied indentation loads were 0.01, 0.025, 0.05, 0.1, 0.2, 0.3, 0.5 and 1 kp respectively. Five measurements were performed by the usage of all indentation loads, and the average hardness results with the deviations are indicated in Fig. 4. The load-holding time was 12 second in all cases. The tensile tests were performed by Instron 4482 universal material tester.

## 4 Results and discussion

### 4.1 Effect of the indentation load

It is well known that the hardness values are depending on the indentation load, although in case of Vickers method the

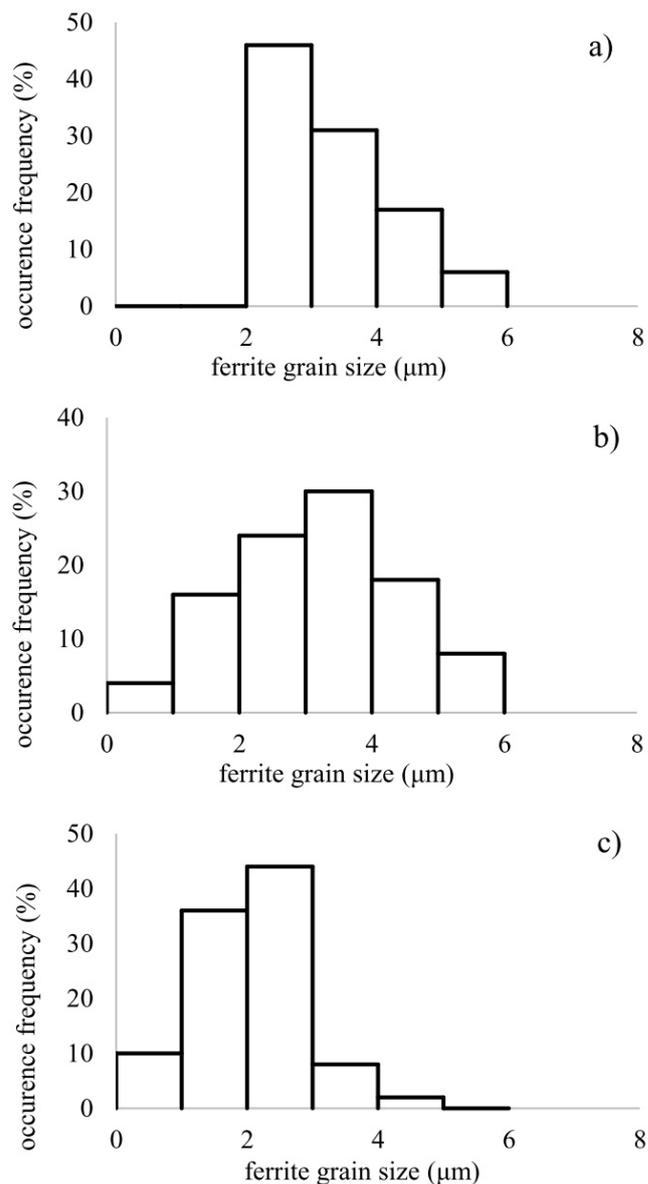


Fig. 3 Ferrite grain size distribution in a) DP600, b) DP800 and c) DP1000 steels

external circumstances have less influencing effect for the results than at Brinell measurements. It can be attributed to the deformation of the Brinell-type indentation ball. Refers to Vickers test, this influencing effect can be perceived stronger in the micro-hardness ranges, mainly for inhomogeneous materials. The extent of the deviation in the function of the indentation load is defined by the following factors: the elastic and plastic deformation tendency of the sample, the geometry and the hardness of the indentation tool and the friction between the tool and the sample (Tisza, 2001). Assuming that the friction and the tool hardness remained constant during the tests, the reason of the deviation can be specified as the elastic and plastic deformation of the investigated phases and the indentation geometry.

Since the volume fraction of the ferrite and martensite phases were varied in each samples, the average plastic and elastic properties could show different load-dependence characteristics. Seeing the diagrams of the average hardness values

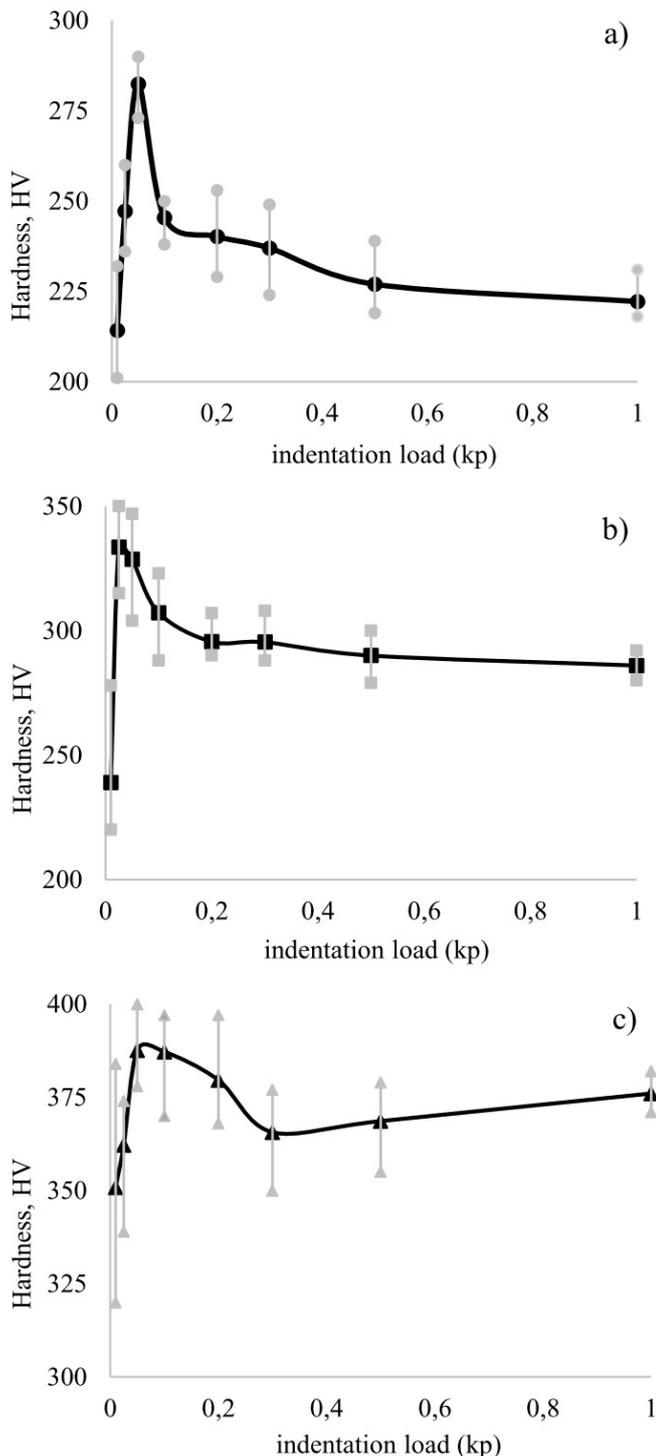


Fig. 4 Hardness profiles of a) DP600, b) DP800 and c) DP1000 steels for different indentation loads

in Fig. 4, similar hardness profiles can be observed independently from the MVF.

Primary concluded that in low loading ranges, the hardness increases roughly as the indentation load increases for all types of DP steels. The initial increasing is at around 100 HV for samples of lower MVF than 50 % (DP600 and DP800), while it reduces to 50 HV only for DP1000. It is still considerable at all materials that the hardness values start to decrease as the loading value exceeds the 0.05 kp. After continuously reducing up to

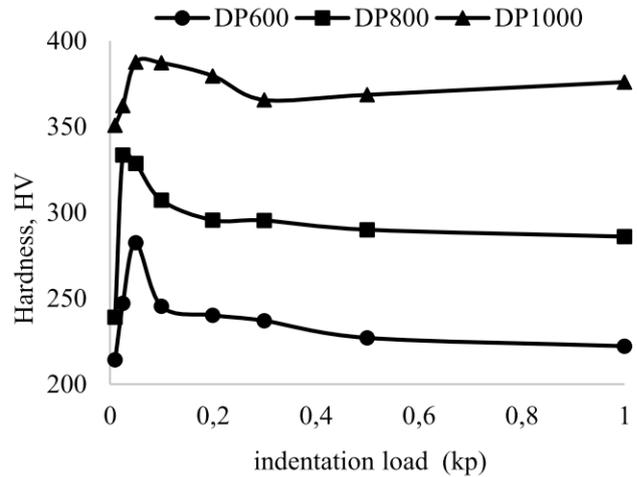


Fig. 5 Comparing of the hardness profiles in the function of indentation load for DP steels with different MVF

0.2 kp, the hardness sets a nearly constant value. This constant value corresponds to the macrohardness results, and higher for DP1000 than for DP800 and DP600 obviously. The summarized hardness profiles are indicated in the diagram of Fig. 5.

The deviation shows that the lower discrepancy can be registered at higher indentation loads, especially at 1 kp ( $HV_1$ ) for all materials. In lower load ranges, the indentation of a few grain sized ferrite or martensite island can be occurred easily, due to the smaller indentation sizes. For example, in the case of DP1000, the indentation diameters formed between 0.007-0.009 mm with applying 0.01 kp ( $HV_{0.01}$ ), while with the using of 0.3 kp ( $HV_{0.03}$ ) this value lifted up to 0.038-0.042 mm. In this way the hardness values refer to lots of particles (seeing the average grain diameters in Fig. 3) at larger indentation geometries, so provide more balanced measurement results and more close to the average value.

To study the effect of the dual-phase – inhomogeneous -microstructure we compared this results to the homogenous ferritic DC04's indentation load sensitivity. The hardness profile of this mild steel shown by Fig. 6. The nature of the curve is really similar to DP steels. Here also quick raising happens below 0.5 kp, and then the results change to slope the curve. The nearly constant values also appear first at around 0.2 kp, then keep it until the last highest load.

In terms of the discrepancy of DC04, high deviation seems at lower load values also, so the deviation rather depends on the indentation diameters than the microstructure of the investigated sample. Note that the tool geometry and hardness, furthermore the friction assumed constant during all measurements.

#### 4.2 Effect of the MVF

The average hardness values for each steel types were chosen to reveal the relation between the MVF and the hardness results, but take into consideration the indentation load effect also. Previously mentioned study (de la Concepción et al., 2015) stated linear correlation between  $HV_1$  microhardness and MVF in case of DP steels with different carbon content.

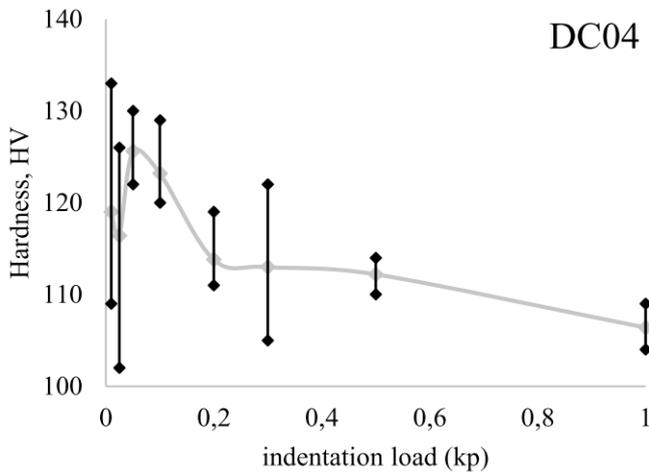


Fig. 6 Hardness profile of DC04 steels for different indentation loads

Furthermore, ISO 18265:2003 European standard also assumes linearity between the macrohardness ( $HV_{30}$ ) and the tensile strength for steels in general. For the reason of linear relationship between MVF and tensile strength (TS) reflected by Fig. 7, these properties can be regarded equivalent in this comparison. Fig. 7 illustrates the changing of the yield strength (YS) also, in the function of the MVF. These values respond a little slower but almost linear growing with the amount of the martensite in the microstructure.

Here is to mentioned that the MVF relates inversely but similarly with the elongation than with the strength. This follows from a previous research work (Béres and Tisza, 2017) about the strength and formability properties (dome height, elongation) in case of the same DP steels. That paper estimate linearity between strength and formability parameters, so the effect of the strength can be substituted with the MVF, for formability estimation also. Taking care of the visualization of the formability-MVF relationship, Fig. 8 is applied.

In this figure tensile elongation (TE) and ultimate elongation (UE) are interpreted as engineering strain ( $\varepsilon$ ). Former concept expresses the elongation in the moment of the failure, while

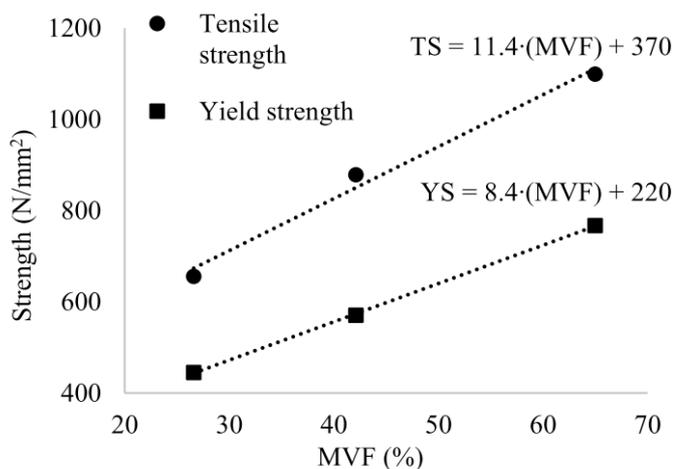


Fig. 7 Changing of the tensile (TS) and the yield (YS) strength with the growing of MVF

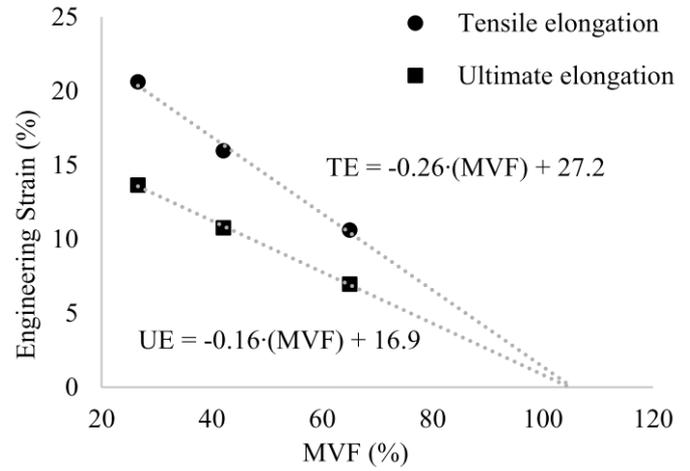


Fig. 8 Changing of the tensile (TE) and the ultimate (UE) elongation with the growing of MVF

latter belongs to the occurrence of the plastic instability. The way those are calculated is reflected by Eq. (1), where  $l_0$  is the initial base length, and  $l_1$  is the current examined length:

$$\varepsilon = \frac{l_1 - l_0}{l_0} (\%). \quad (1)$$

TE has stronger steepness and approaches to UE as the material becomes more brittle with the increasing of MVF. The approximated values meet over than 100 (%) MVF. This confirms the fact that the low carbon martensite has a little formability in contrast with the theoretically absolutely rigid hardenable steels' martensite.

Accepting the linear relations between the tensile strength and MVF and hardness based on Fig. 7 and referred literatures, linearity should be supposed between the microhardness values and MVF also. This relation represented by Fig. 9 and Fig. 10, in the function of the indentation load. The first two highlighted hardness values, which do not follow this linear assumption (black pointed lines) belong to 0.01 kp ( $HV_{0.01}$ ) and 0.025 kp ( $HV_{0.025}$ ) indentation loads. Although, the linear regression  $R^2$  in case of  $HV_{0.01}$  exceeds the value of 0.9, but still stays under 0.86 at  $HV_{0.025}$  if approximating linearity. Using exponential approximation (grey permanent line),  $R^2$  does not even reach 0.97 as well at  $HV_{0.01}$  and do not improve at  $HV_{0.025}$ . In this way neither of these two methods can be said really proper evaluation. Taking into consideration the regularly occurring of high deviation at these cases, the low load applied hardness values could not be recommended for conversion to macroscopic mechanical properties.

The linear correlation obviously exists in higher load ranges after 0.05 kp up to 1 kp in Fig. 10. At  $HV_{0.05}$  the steepness of the assumed straight differs from the others but can be estimated well by linear relation.

For this reason, with ignoring the fitted parameters of  $HV_{0.05}$ , nearly constant parameters reflect the correlation between hardness and MVF and thus between hardness and mechanical

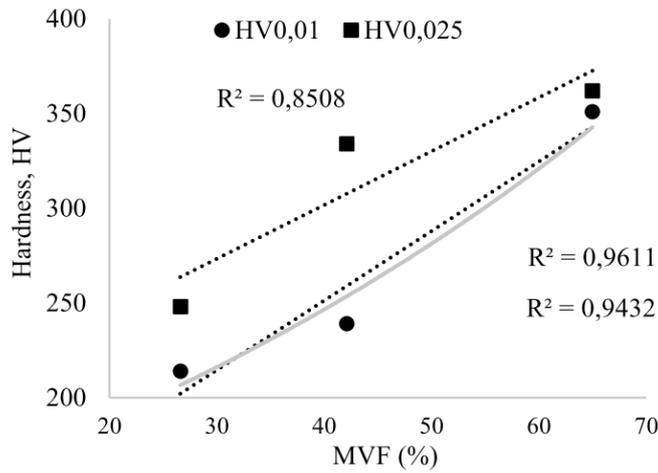


Fig. 9 Correlation between the microhardness and MVF with using different indentation loads: 0.01 and 0.025 kp

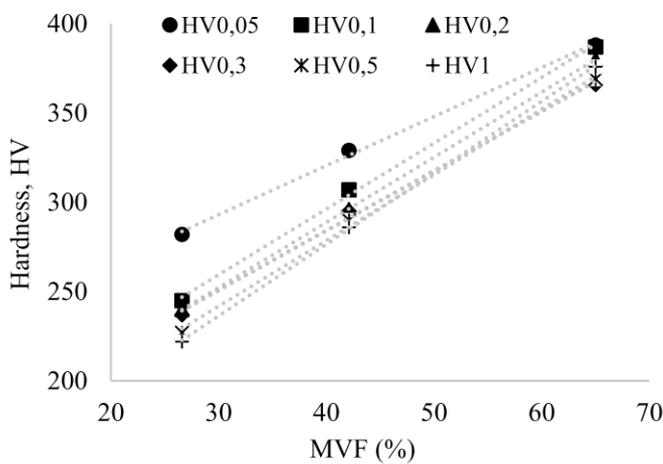


Fig. 10 Correlation between the microhardness and MVF with using different indentation loads: 0.1-1 kp

properties like TS, US and TE, UE according to Fig. 7, Fig. 8 and Fig. 10, from  $HV_{0,1}$  up to  $HV_1$ .

The fitted parameters are summarized in Table 2, where  $HV_0$  refers to the starting point of the approximated function.

Table 2 Fitted parameters at different indentation loads

	$HV_{0,01}$	$HV_{0,025}$	$HV_{0,05}$	$HV_{0,1}$
steepness	3.7	2.8	2.8	3.7
$HV_0$	105	188	211	149
	$HV_{0,2}$	$HV_{0,3}$	$HV_{0,5}$	$HV_1$
steepness	3.7	3.3	3.7	4.0
$HV_0$	144	151	131	116

The correlation between the MVF and hardness can be expressed by Eq. (2) with taking the mean value of the parameters.

$$HV = 138 + 3.7 \cdot MVF. \quad (2)$$

The given approximated function is similar than the suggested relationship between nanohardness and yield strength reported by Tiryakioglu (2015). Knowing the connection

between the MVF and mechanical properties, Eq. (2) can be extended to yield strength and tensile strength also. With the substituting of the equations from Fig. 7 to Eq. (2) the

$$HV = 18 + \frac{1}{3.1} \cdot TS \quad (3)$$

$$HV = 38 + \frac{1}{2.3} \cdot YS \quad (4)$$

contexts succeed between the mechanical properties.

Using Eq. (3) and Eq. (4), the calculated results are represented by Table 3 and by Fig. 11, as those do not exceed the 10 HV (< 5%) deviation from the measured mean values. It is important to note again that it refers to indentation loads between 0.1 kp up to 1 kp, which give the expected linear correlation.

Table 3 Measured and calculated hardness values

	Measured (HV)	Calc_YS (HV)	Calc_TS (HV)
DP600	234	230	232
DP800	295	302	286
DP1000	376	373	372

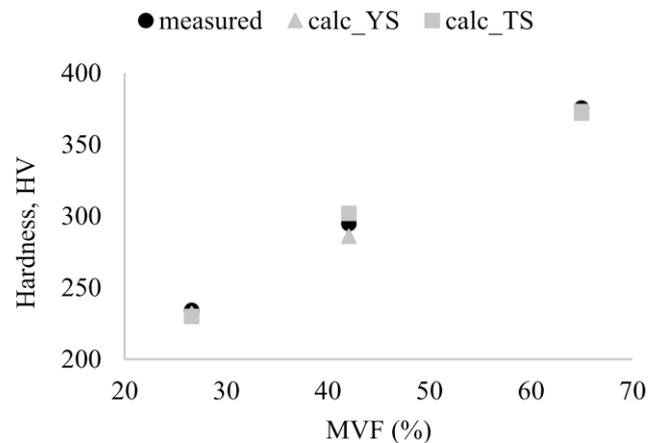


Fig. 11 Mean hardness from 0.1 to 1 kp and calculated values of the three DP steels in the function of MVF

In the figure and in the table Calc\_YS hardness means the results got by Eq. (4) while Calc\_TS belongs to Eq. (3).

## 5 Conclusion

Experimental investigation of mechanical tests results of DC04 ferritic mild steel and different types of Dual Phase steels with different martensite volume fraction is performed in this study. Vickers measurements were executed in wide range of indentation load, from 0.01 kp ( $HV_{0,01}$ ) up to 1 kp ( $HV_1$ ), and the evaluation of the correlation between the mechanical properties was confirmed by tensile tests' results. With the observation of the influencing effect of the indentation load, and then comparing the results to the martensite volume fraction, the next points can be stated:

- In low load ranges, the hardness increases for all types of investigated steels, as the indentation load increases.
- The hardness values start to decrease as the applied load exceed the 0.05 kp, then become permanent over 0.2 kp.
- The lowest discrepancy can be registered at the highest indentation load (1 kp - HV<sub>1</sub>). Lower indentation loads result higher deviation, as the smaller indentation geometries become comparable to the few grain sized martensite and ferrite islands in the microstructure. Although, the discrepancy and the shape of the hardness profile less depend on the ferrite-martensite proportion than the indentation load as the main influencing factor. This observation was justified by the comparison of DP-DC steels' results.
- Linear correlation exists between the MVF and mechanical properties like strength and elongation, in case of the DP steels. TE strongly decreases with the growing of MVF, but the material is still not completely brittle, even in the presence of 100 % MVF.
- HV<sub>0.01</sub> and HV<sub>0.025</sub> microhardness values do not follow linear correlation with the MVF, but the exponential presumption is also a poor approximation, due to the high deviation. At higher load ranges (from 0.1 to 1 kp) proper relationship can be reported between the microhardness and the MVF, and so the tensile and yield strengths. The difference between the measured and calculated values is less than 10 HV (5%).

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