

Reverse indentation size effect of a duplex steel

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Abstract

In this investigation, duplex steel 1.4462 was subjected to the microstructural analysis and Vickers hardness measurements. The tests were performed on three characteristic surfaces, considering the rolling direction. Obtained results showed that this duplex steel exhibited an increase in measured hardness with the increase of the applied load, known as the *reverse indentation size effect (RISE)*. The following mathematical models were used for the phenomenon explanation: *Meyer's law*, *proportional specimen resistance (PSR) model* and *modified proportional specimen resistance (MPSR) model*. The regression analysis showed that all models could be used for the RISE analysis in duplex steel. “True” Vickers hardness was determined by the PSR and MPSR models. The MPSR model was more suitable in this particular case, because it considered the effect of the finishing process on the hardness results and gave the “true” hardness closest to the measured value.

Key words: duplex steel, Vickers hardness, reverse indentation size effect

1. Introduction

Duplex stainless steels consist of two-phase microstructure – ferritic and austenitic grains, in approximately equal amounts. Their usage has been more interesting in the last few decades, because of their extraordinary properties, especially resistance to different types of corrosion, as well as good mechanical properties and weldability. For successful integration of this material into design process, its mechanical properties, including hardness, must be well known.

Hardness represents a measure of material resistance to permanent plastic deformation and is a very frequently investigated mechanical property. One of the most common indentation methods for the hardness determination is the Vickers hardness measurement, due to its simplicity and minimum of machining required for the specimen preparation. It has been experimentally confirmed that the hardness usually depends on the load applied by the hardness tester. Some materials endure a decrease in hardness with an increasing load, which is called indentation size effect (ISE), while others manifest an increase in hardness with the increasing load, called reverse indentation size effect (RISE) [1–10]. Both phenomena are illustrated schematically in Fig. 1.

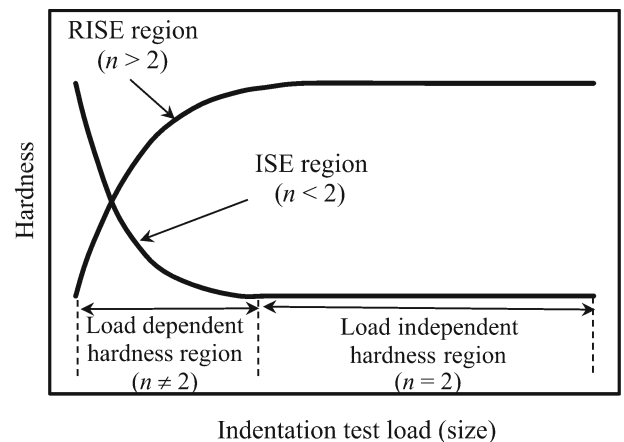


Fig. 1. Schematic plot of hardness variation with test load, showing the indentation size effect (ISE) and reverse indentation size effect (RISE) [11].

With lower indentation loads, a greater possibility appears that the indenter falls into one grain, which usually does not result in representative hardness value. This hardness, which depends on the applied load, is called load dependent or “apparent” hardness. The constant hardness value is called the

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Table 1. Chemical composition of the investigated material (wt.%)

C	Si	Mn	Cr	Mo	Ni	N	Fe
0.024	0.26	1.62	22.78	3.00	5.27	0.156	balance

“true” hardness and usually appears at the high indentation loads [5–8].

The objective of this work is to measure Vickers hardness of a rolled duplex stainless steel in a wide range of loads, in three routes, considering the rolling direction, and to observe the influence of the applied test loads and microstructure on the measured hardness. Subsequently, results will be described by three known mathematical models, which give the correlation between the applied load, F , and the resulting indentation size, d . These models are: *the Meyer’s law*, *the proportional specimen resistance (PSR) model* and *the modified proportional specimen resistance (MPSR) model*.

2. Experimental

The objective of this work is to determine how is the Vickers hardness of rolled duplex steel influenced by the sample microstructure, as well as the applied indentation load.

Investigated material was a duplex stainless steel 1.4462 (X2CrNiMoN 22 5 3), rolled in the form of a 10 mm thick sheet. All specimens were cut out of the same sheet. Chemical composition analysis was conducted on the glow discharge optical emission spectrometer by Leco (GDS850A), while the nitrogen content was analysed on the Leco TC-436, by burning in the helium atmosphere. Obtained chemical composition is given in Table 1.

The rolling process results in directed microstructure, which means that different surfaces have different microstructure. In this paper, the Vickers hardness was measured on three characteristic surfaces, considering the rolling direction, in order to establish how the directed microstructure of the rolled duplex steel influenced the obtained hardness values. Chosen surfaces are schematically shown in Fig. 2. Sample 1 is parallel to the sheet surface and the rolling direction. Sample 2 is perpendicular to the sheet surface, but parallel to the rolling direction, while the sample 3 is perpendicular to both, sheet surface and rolling direction.

Another objective of this work was to investigate whether the applied indentation load had the effect on the hardness measurement results. Therefore, a wide range of loads was applied: 0.4905 N (HV0.05),

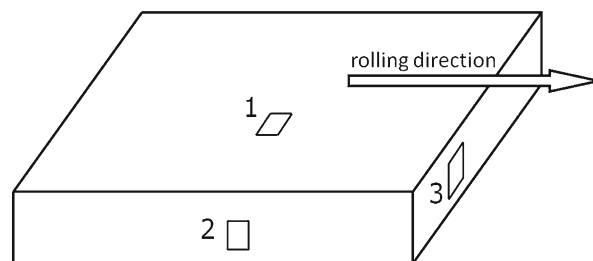


Fig. 2. Schematic plot of chosen samples, considering the rolling direction: surface 1 / sample 1: parallel to the sheet surface and the rolling direction; surface 2 / sample 2: perpendicular to the sheet surface and parallel to the rolling direction; surface 3 / sample 3: perpendicular to both, sheet surface and rolling direction.

0.981 N (HV0.1), 1.962 N (HV0.2), 4.905 N (HV0.5) and 9.81 N (HV1). In order to obtain statistical relevant data, 20 measurements of each indentation were made on each tested surface. All indentations lasted for 15 s. Prior to the indentation, all surfaces were grinded and polished adequately, according to the standard metallographic procedure. Indentations were made by a conventional Vickers hardness tester (Instron, Wilson-Wolpert Tukon 2100 B). The length of the indentation diagonals was measured immediately after unloading and the hardness was calculated by the built-in software.

After indentation, the samples were etched in LB I solution and their microstructure was observed by the optical microscope (Olympus BH).

Statistical analysis of measured hardness indicated that sample 2 (Fig. 2) showed the most equable hardness values, that is, the dissipation was the lowest. Therefore, this sample was chosen for further mathematical analysis. The measured hardness values were fitted satisfactorily to three chosen mathematical models for the indentation size effect evaluation. These models were: *the Meyer’s law*, *the proportional specimen resistance model (PSR)* and *the modified proportional specimen resistance model (MPSR)*.

3. Results and discussion

Three different specimens, considering the rolling direction, were cut from the sheet of the chosen rolled duplex steel. They were subjected to the microstructural analysis and the Vickers hardness measurement.

The microstructural analysis showed expected variations in the grain shape. The grains were somewhat elongated in the rolling direction. However, the amount of the austenitic and ferritic phase was approximately equal in all three samples. Figure 3 shows microstructure of all three samples.

The Vickers indentations were made on polished

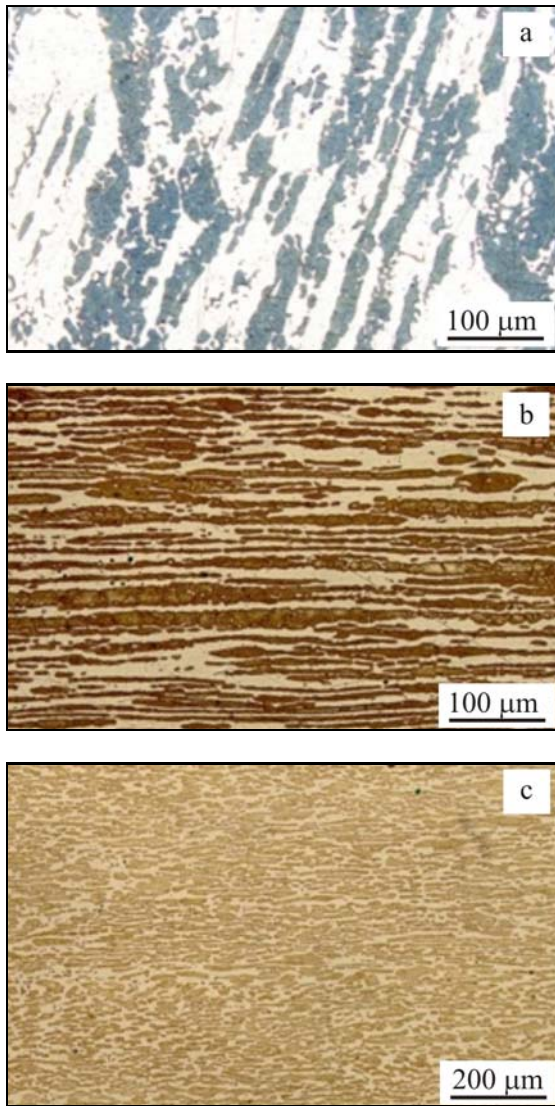


Fig. 3. Microstructure of the investigated duplex steel samples. Dark (etched) grains represent austenitic phase, while the ferritic grains remain unetched (lighter colour): sample 1: surface parallel to the sheet surface and the rolling direction (a), sample 2: surface perpendicular to the sheet surface and parallel to the rolling direction (b), sample 3: surface perpendicular to both sheet surface and rolling direction (c).

samples. Immediately after unloading, the size of the indentation diagonals was measured. The Vickers hardness, HV, is defined as the ratio of the applied load, F (N), to the pyramidal contact area, A (mm^2), of the indentation, giving:

$$\text{HV} = \frac{F}{A} = \alpha \frac{F}{d^2}, \quad (1)$$

where d (mm) stands for the indentation mean diagonal and α is the geometrical constant of the indenter, which equals 0.1891 for the Vickers indenter.

The mean diagonal d corresponds to the arithmetical mean value of two measured diagonals for each indentation $\left(d = \frac{d_1 + d_2}{2}\right)$.

After twenty Vickers hardness measurements for each indentation load and chosen surface, the mean values and standard deviations were calculated. Obtained results indicated a hardness variation dependent on the applied load – specifically, an increase in hardness with increasing indentation load. This phenomenon is known as the *reverse indentation size effect (RISE)* [5–9]. Sample 2 (Fig. 2) showed the most typical duplex structure and the hardness measurements results gave the least dissipation for it. Therefore, this sample was chosen for further investigation of the RISE and was described by the chosen mathematical models. Figure 4 presents the mean Vickers hardness values and error bars, depending on the applied indentation load, for the sample 2.

In order to describe the ISE and RISE, several mathematical models can be found in literature, showing the indentation diagonal length, d , dependence on the applied test load, F . In this paper three models will be used.

The simplest way to describe this phenomenon is the *Meyer's law* [1–10]:

$$F = Kd^n, \quad (2)$$

where n stands for the Meyer's number (index), while K (N mm^{-n}) represents the standard hardness constant for a given material. If the Meyer's index n equals 2, the applied indentation load has no effect on the measured hardness, but the much more common case is that n is unequal to 2, that is, the measured hardness depends on the applied load. This is schematically shown in Fig. 1. For $n < 2$, the hardness decreases with increasing applied load, which is called the normal ISE. For $n > 2$, the hardness increases with the applied load increase, known as the reverse ISE (RISE). When $n = 2$, the applied indentation load has no effect on the measured hardness.

The values of the coefficients K and n (Eq. (2)) depend on the size of the applied indentation load. These coefficients can be calculated by the linear regression analysis of the $\log F$ versus $\log d$ plot (Fig. 5), where the slope of the obtained straight line gives the Meyer's index n , while the intercept presents the value of the $\log K$. The resulting Meyer's index n equals 2.424, which indicates that the Vickers hardness of the investigated duplex steel depends on the applied load and exhibits the reverse indentation size effect. Regression analysis data are given in Table 2.

The *proportional specimen resistance (PSR) model* [1] is another way to mathematically describe the ISE. It explains the relationship between the applied load, F , and the mean indentation diagonal, d , by means

Table 2. Regression results for all applied mathematical models

Meyer's law				
n	$\log K$	K (N mm ⁻ⁿ)	R^2	
2.424 ± 0.123	3.6132 ± 0.169	4104	0.9923	
Proportional specimen resistance (PSR) model				
a_1 (N mm ⁻¹)	a_2 (N mm ⁻²)	R^2	"true" hardness, HV _T	
-18.245 ± 2.618	1574.1 ± 48.833	0.9971	297.66	
Modified proportional specimen resistance (MPSR) model				
a_0 (N)	a_1 (N mm ⁻¹)	a_2 (N mm ⁻²)	R^2	"true" hardness, HV _T
0.4573 ± 0.218	2.7999 ± 9.089	1375.2 ± 81.167	0.9999	260.05

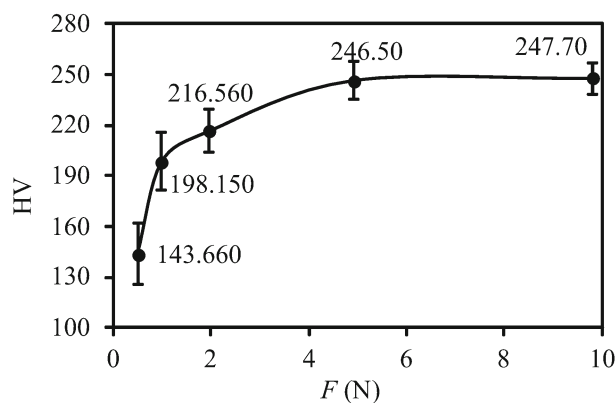


Fig. 4. The dependence of the measured Vickers hardness on the applied load for the duplex steel (mean values and standard deviation).

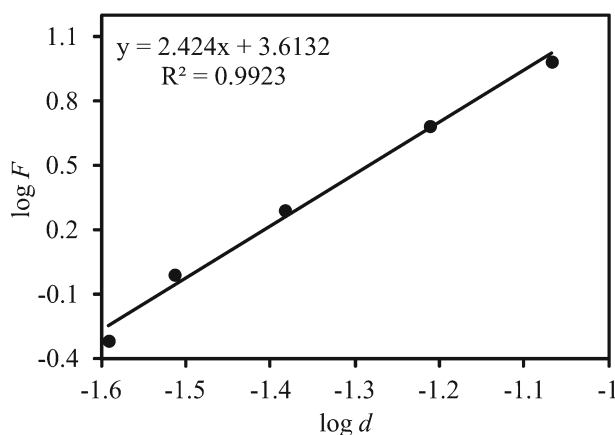


Fig. 5. The Vickers hardness data on duplex steel according to the Meyer's law, Eq. (2).

of energy balance analysis, resulting in the following expression:

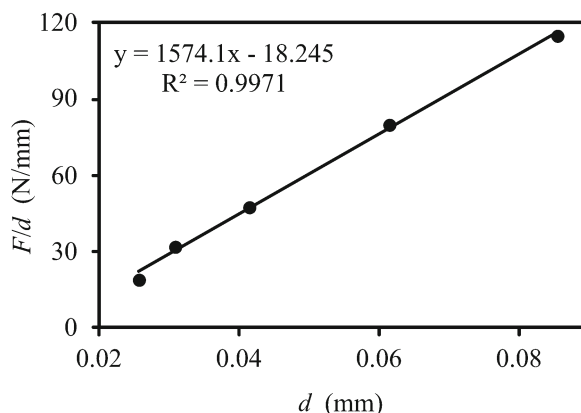


Fig. 6. The Vickers hardness data on duplex steel according to the proportional specimen resistance (PSR) model, Eq. (3).

$$F = a_1 d + a_2 d^2, \quad (3)$$

where a_1 (N mm⁻¹) is a constant related to the specimen proportional resistance, while a_2 (N mm⁻²) is related to the load independent ("true") hardness. Some authors assume that a_1 shows the energy consumed in generating new surfaces, that is, indentations and microfractures, while the others consider this coefficient is related with friction and elasticity. It is assumed that the a_2 coefficient is related to the resulted permanent deformation [1–10].

These coefficients are evaluated through the linear regression of F/d versus d plot, shown in Fig. 6. The slope gives the a_2 value, while the intercept shows the associated a_1 value. The results are presented in Table 2.

Gong et al. [2] proposed an alteration of the PSR model by introducing the *modified proportional specimen resistance (MPSR) model*, given by:

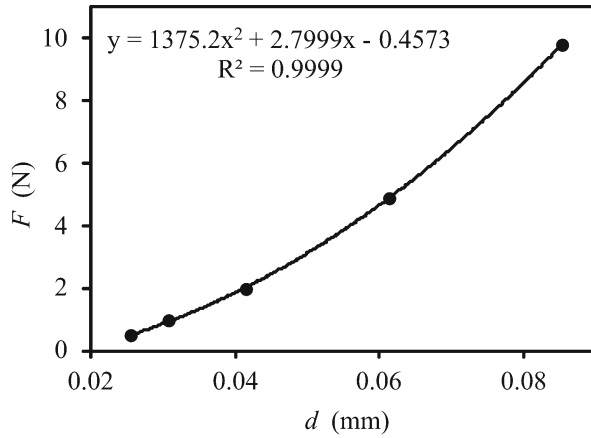


Fig. 7. The Vickers hardness data on duplex steel according to the modified proportional specimen resistance (MPSR) model, Eq. (4).

$$F = a_0 + a_1d + a_2d^2. \quad (4)$$

As in the previously used PSR model, F is the indentation load, d stands for the mean diagonal of the Vickers indentation, while a_0 , a_1 and a_2 are constants. As in the previous example (the PSR model), a_1 is a coefficient related to the specimen proportional resistance, while a_2 is related to the load independent (“true”) hardness. The a_0 (N) coefficient depends on the material properties and the applied surface finishing process (machining and polishing). All three constants can be calculated by means of conventional polynomial regression from the plot of the F versus d (Fig. 7). All obtained results are summarized in Table 2.

It is known that the test specimen resistance, W , is a minimal level of the applied indentation load that causes permanent deformation [12]. Li and Bradt [1] suggested a linear relationship between the specimen resistance, W , and the size of the corresponding mean indentation diagonal, d :

$$W = a_1d, \quad (5)$$

where the a_1 constant is the coefficient found in the PSR and MPSR models (Eqs. (3, 4)), related to the specimen proportional resistance. The load that causes permanent deformation is known as the effective indentation load, F_{eff} [2, 4, 10]. It is defined as the difference between the applied load, F , and the specimen resistance, W :

$$F_{\text{eff}} = F - W. \quad (6)$$

If Eqs. (3) and (5) are substituted into Eq. (6), the following relationship between the effective indentation load F_{eff} and indentation diagonal d is given:

$$F_{\text{eff}} = a_2d^2. \quad (7)$$

The substitution of Eq. (7) into Eq. (1) results with the following expression, used for the calculation of the “true” hardness, HV_T :

$$HV_T = \alpha a_2. \quad (8)$$

It is clear that the “true” hardness HV_T depends only on the coefficient a_2 , because α is the constant of the Vickers hardness indenter. Indirectly, this means that the “true” Vickers hardness depends on the effective load, F_{eff} . Equation (8) was used to determine the “true” Vickers hardness, HV_T , for the duplex steel specimen, according to the PSR and MPSR models. The results are given in Table 2.

4. Conclusion

Analysed material, that is, rolled duplex stainless steel 1.4462 (X2CrNiMoN 22 5 3), showed a variation in measured hardness with the applied indentation load. Specifically, the specimen showed an increase in measured hardness with the increasing indentation load, which is called the reverse indentation size effect (RISE). This is confirmed by the Meyer’s index, which was greater than 2 ($n = 2.424$).

The hardness was measured on three characteristic surfaces, considering the rolling direction (Fig. 2). All three samples showed the RISE. Sample 2 (surface perpendicular to the sheet surface and parallel to the rolling direction) showed the least dissipation and was therefore selected for further investigation by chosen mathematical models. Observed phenomenon can be explained by the duplex microstructure, composed of ferritic and austenitic phase. When applying the lower indentation loads, there is a greater possibility that only one phase, ferritic or austenitic, will be affected by measuring. Therefore, the dissipation will be greater and the mean value can be somewhat lower. In contrary, the application of higher indentation loads can usually lead to measurement that affects both phases, which results in somewhat higher hardness with less dissipation.

All mathematical models used in this study (the Meyer’s law, the proportional specimen resistance (PSR) model and the modified proportional specimen resistance (MPSR) model) can successfully describe the reverse indentation size effect in investigated duplex steel. The obtained correlation coefficients, R^2 , are: 0.9923 for Meyer’s law, 0.9971 for the PSR model and 0.9999 for the MPSR model.

The “true” Vickers hardness, HV_T , determined by the PSR model, equals 297.66 and 260.05 for the MPSR model. The difference between these models is acceptable, so it can be established that they explain

the RISE in duplex steel equally well. However, the MPSR model is recommended for further use, because it considers the effect of the finishing processes on the hardness results, and the “true” hardness value obtained by this model is closest to the measured value.

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