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T. KAŹMIERSKI[1*], J. KRAWCZYK[2], Ł. FROCISZ[2], P. MATUSIEWICZ[2]

A STUDY OF CORRELATIONS BETWEEN CHEMICAL COMPOSITION, HOT ROLLING PROCESS PARAMETERS, MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HOT ROLLED DP600 STEEL

DP600 steel is widely used in the automotive industry due to its exceptionally favourable combination of high tensile strength and good ductility. In the hot-rolling process, DP600 steel is produced by use of controlled cooling of the rolled strip from temperature above A_{r3} to temperature below M_s. In this process, it is quite difficult to control key process parameters such as the time or temperature, and the final microstructure of the material is also affected by the degree of deformation of the material at various stages of the process. This paper presents a statistical analysis of the effect of chemical composition and selected hot rolling process parameters on the microstructure of DP600 sheet with respect to its mechanical properties. Based on industrial data from hot rolling mill combined with extended microstructure analysis, it was possible to find correlations between some of the analysed parameters and material properties. Among many correlations discussed in this work, most notable are those between martensite morphology and mechanical properties, between Mn and Si concentration and martensite morphology and between rolling speed, strain, cooling rate and mechanical properties.

Keywords: Dual phase steels; hot rolling process; mechanical properties; microstructure

1. Introduction

DP600 steel consists of two different phases. Approximately 10-15% of its volume is martensite, occurring mostly as islands dispersed in a ferritic matrix. The hard martensitic phase contributes to the high strength properties of this steel, while ferrite provides good ductility [1-4]. The combination of high tensile strength and good ductility is the main advantage of DP steels. In addition, these steels are characterized by a high coefficient of strain hardening and a lack of specific yield point. All of this makes these steels widely used in the automotive industry, where they are used for the production of stamped car parts, such as the disks of car wheels [5-8].The share of steel wheels in the automotive wheels market is currently at about 35%, and forecasts for the coming years predict that the steel wheels will keep its high share in the market. It is also expected, that the net value of this market will increase by almost $400MM from year 2020 to 2028 in Europe alone [9-10]. At the same time, car wheels manufacturers are constantly striving to reduce their weight, and in order to ensure the best possible performance and visual properties of the steel wheels, their shape is gradually becoming more and more complex. All this makes the fracture resistance of input material, such as DP600 steel, even more important than before. For cold forming operations, not only mechanical properties, but also local formability and crack resistance play a key role in manufacturing process design [11-12]. The problem of DP600 steel cracking at high deformation is related to its inhomogeneous structure, which leads to uneven deformation of the two phases. The large strain gradient created in the material during its deformation is one of the main causes of early cracking during the stamping process [13-15]. Four basic mechanisms of DP steels cracking can be named: decohesion at the ferrite-martensite interface, martensite cracking, ferrite cracking in close proximity to martensite islands, and nucleation of voids on non-metallic inclusions [16].

Attempts are being made to determine the relationship between microstructure characteristics, such as the volume fraction of martensite, the presence of other phases such as pearlite or bainite, the size, shape and degree of dispersion of martensite islands, the grain size of ferrite, and mechanical properties of DP steel [17-19]. It is currently considered that the most favourable microstructure of DP steel in terms of mechanical properties and crack resistance is the microstructure characterized by equiaxial, evenly dispersed martensite islands. Some studies have reported that the presence of martensite bands, present

1 ARCELORMITTAL POLAND S.A. UNIT IN KRAKOW, TADEUSZA SENDZIMIRA 1 STREET, 31-752 KRAKOW, POLAND; AGH DOCTORAL SCHOOL, AL. MICKIEWICZA 30, 30-059 KRAKOW, POLAND
2 AGH UNIVERSITY OF KRAKOW, FACULTY OF METALS ENGINEERING AND COMPUTER SCIENCE, AL. MICKIEWICZA 30, 30-059 KRAKOW, POLAND

* Corresponding author: tkazmierski@agh.edu.pl

in the form of chains of islands, negatively affects the properties of DP steels. The majority of published studies addressing these issues concern DP steels obtained on a laboratory scale by annealing and subsequent controlled cooling of cold rolled ferritic-pearlitic steel. In such studies, it is relatively easy to control both the degree of deformation of the input material and the time-temperature parameters at the various steps of the annealing process. In the case of DP600 steel produced in the hot rolling process, in order to obtain the desired microstructure, in addition to strict control of the temperature parameters, it is also necessary to apply the appropriate strain and strain rate in each pass [20-22]. The influence of hot rolling process parameters on microstructure and mechanical properties of DP600 steel is not well known. Publications referring to this method of production are scarce, and despite decades long history of dual phase steel production, there is very little information available about the hot rolling process of DP600. This work aims to present the results of an analysis carried out on material obtained by an industrial hot-rolling process. The study attempts to determine the effect of the chemical composition and the hot rolling process parameters on the microstructure of DP600 steel and the relationship between the microstructure and mechanical properties of this steel grade. First, authors investigate if the mechanical properties of material taken from different locations along the hot rolled coils are different. The key objectives of this work are: to find a correlations between the martensite morphology and mechanical properties, to examine how Mn, C and Si concentration influences material microstructure, to determine the influence of selected hot rolling parameters on both microstructure and mechanical properties of investigated DP600 steel.

2. Material and experiment

The tests were carried out on material taken from 23 hot rolled coils of DP600 steel produced in industrial hot rolling mill. The dimensions of the rolled strips were 4.2 × 1441 mm and 4.5 × 1315 mm. The coils were rolled from 220 mm thick slabs weighing about 24 tons each. After reheating in a walking beam furnace for 160 minutes to a temperature of 1250°C the slabs were first rolled in 7 passes on a reversing mill to a thickness of 35 mm. The steel bars was then rolled in a group of 6 finishing stands to a thickness of 4.2 or 4.5 mm. The final rolling temperature measured after last finishing stand was about 830°C. After finishing rolling, the strip was cooled to a temperature of about 150°C in a laminar cooling section. The cooling of DP600 steel is carried out using a pause in water cooling at the intercritical temperature. Cooling is a key stage in the production of DP600 steel grade on a hot rolling mill, since it is at this stage that the dual-phase microstructure is obtained. Fig. 1 schematically shows the two-stage water cooling process after finishing rolling. Once the first stage of water cooling is completed, the overcooled austenite undergoes a transformation into ferrite, but due to relatively high temperature at which this cooling stage ends, only about 80-90% of the austenite is transformed into ferrite. The remaining austenite is quenched and undergoes martensitic transformation in the second cooling stage [23-24].

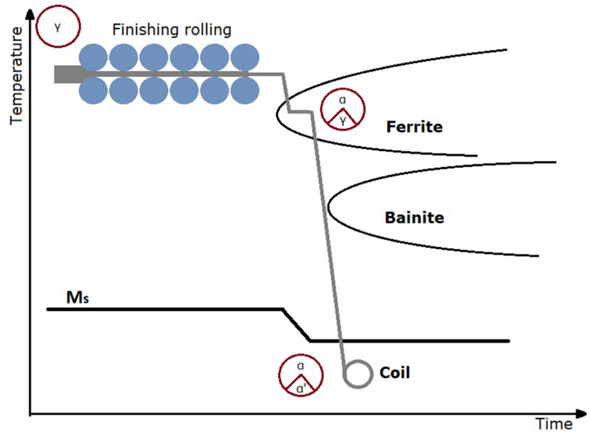

Fig. 1. Schematic TTT diagram of strip cooling after finishing rolling. Reference: T. Kaźmierski, J. Krawczyk, Ł. Frocisz, Archives of Metallurgy and Materials, Characteristic od DP600 steel produced in hot rolling process, In print

Samples were taken from the head and tail ends of each hot rolled coil after cutting off about 10 m of the strip. The samples were used for the tensile tests in the longitudinal and transverse direction to the rolling direction, as well as for microstructure analysis. Same as in the case of tensile testing, microstructure analysis was carried out on samples taken at the head and tail ends of each hot rolled coil. Samples, after polishing, were etched with Klemm's reagent, which provides a good contrast between ferrite and martensite. The ferrite takes on a dark brown colour on the microsections etched with Klemm's reagent, while the martensite takes on a light golden colour. In order to statistically analyse the morphology of the martensite islands on an optical microscope, three images of each sample were taken at 100× magnification. Then, using MetIlo software, parameters such as the size and aspect ratio of the martensite islands, as well as their number, were calculated.

These data were used to calculate the average size of martensite islands (*Sm*), their average aspect ratio (Shape) and the volume fraction of martensite in the material (*Vm*). Fig. 2 shows the results of the binarization performed in MetIlo software. To determine the average size and aspect ratio of martensite islands, martensite islands cut by the edge of the image were discarded from the analysis, as including incomplete martensite islands in the analysis would have distorted the results. Eqs. (1)-(3) shows the formulas for calculation of *Sm*, Shape (*S*) and *Vm* respectively.

$$Sm = \frac{\sum_{i=1}^{k} Sm_i}{k} \quad (1)$$

$$S = \frac{\sum_{i=1}^{k} S_i}{k} \quad (2)$$

$$Vm = \frac{\sum_{i=1}^{k} Vm_i}{k} \quad (3)$$

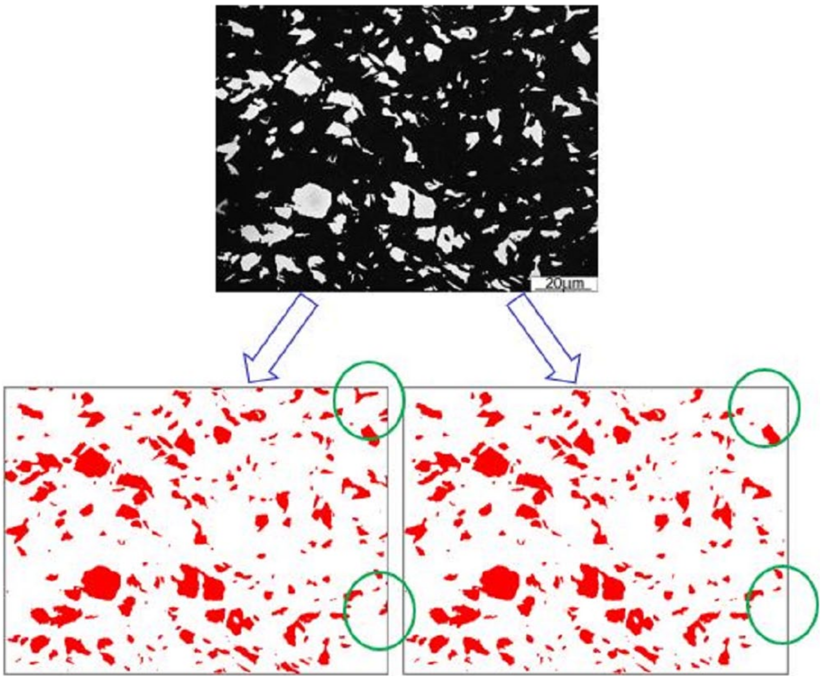

Fig. 2. Example of image binarization in MetIlo application; red – martensite, white – ferrite

The chemical composition of the rolled material was determined by spark optical emission spectrometer. The chemical composition analysis was performed on slabs directly after the continuous casting process. The process data on the hot rolling mill were gathered through the control and measurement systems of the mill. The data are recorded continuously for the entire length of the rolled strip, and the measured values used for the analysis refer to the location of the samples for tensile testing and microstructure analysis.

3. Results and discussion

Data analysis was carried out in Minitab. TABLE 1 shows the results of chemical composition analysis of 46 samples taken from coils after the hot rolling process.

TABLE 1

Concentration of C, Mn and Si in investigated steel

%wt.	Mean	StDev	Minimum	Median	Maximum
C	0.074	0.0022	0.069	0.074	0.077
Mn	1.073	0.0135	1.056	1.070	1.106
Si	0.142	0.0049	0.132	0.142	0.150

The results of tensile tests (YS – yield stress, TS – tensile strength, E – elongation) are presented in Table 2. The data shown were calculated from the results of 23 tensile tests of samples taken from the heads of the coils (sample location – head) and 23 tests for samples taken from the tails of the coils (sample location – tail). Tensile tests were carried out in the longitudinal direction (L) and transverse direction (T) to the rolling direction. The table also shows the average values (avg), calculated as average from both directions. Two Sample T-Tests were carried out for all three quantities: YS, TS, E. In case of YS and E, the P-value was significantly below 0.05, meaning that the differences in mean values between the samples are statistically significant. The P-value calculated for TS was 0.320, which means that there is no statistically significant difference in the TS between the two samples.

TABLE 2

Mechanical properties measured on investigated samples

Property	s. loc.	Mean	StDev	Minimum	Median	Maximum
YS_L [MPa]	head	366	12.7	345	365	389
	tail	380	19.5	346	381	425
YS_T [MPa]	head	371	19.7	346	363	419
	tail	394	22.2	357	389	443
TS_L [MPa]	head	602	6.2	590	601	613
	tail	597	9.7	573	599	614
TS_T [MPa]	head	600	7.2	586	601	612
	tail	599	11.1	561	601	613
E_L [%]	head	30.4	0.7	28.8	30.6	31.5
	tail	30.1	1.3	28.4	30.0	33.2
E_T [%]	head	29.1	1.6	26.0	28.7	31.8
	tail	27.6	1.4	25.1	27.3	30.5
YS_avg [MPa]	head	368	14.9	345	366	404
	tail	387	19.5	353	384	420
TS_avg [MPa]	head	601	5.8	588	600	611
	tail	598	9.8	567	599	613
E_avg [%]	head	29.8	1.0	27.6	29.9	31.4
	tail	28.9	1.1	27.0	28.4	30.9

Figs. 3-5 show a graphical representation of the results of tensile tests.

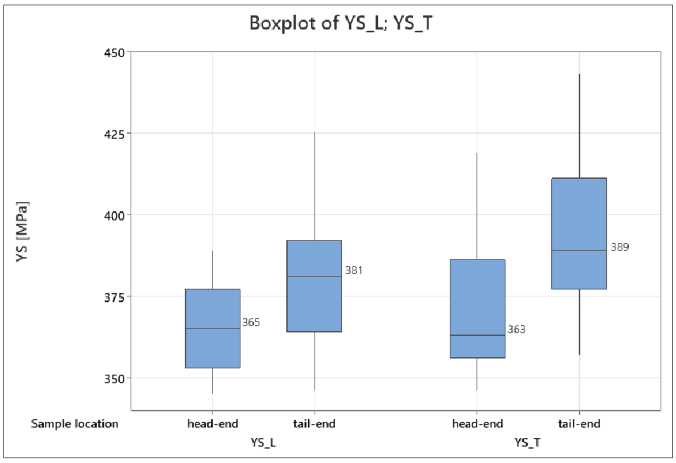

Fig. 3. Boxplot of yield stress

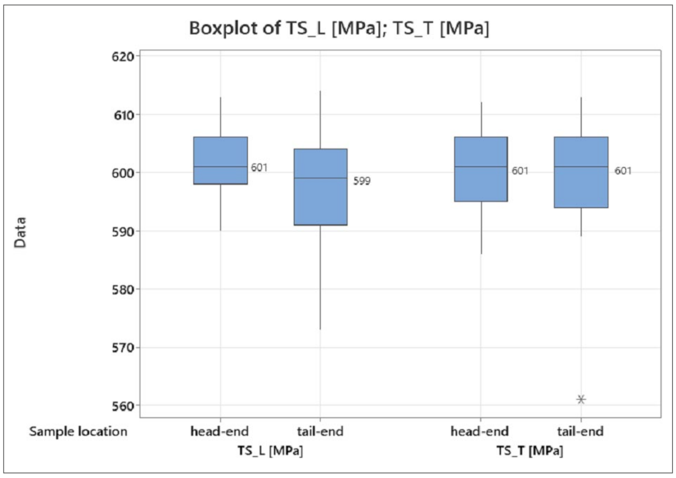

Fig. 4. Boxplot of tensile strength

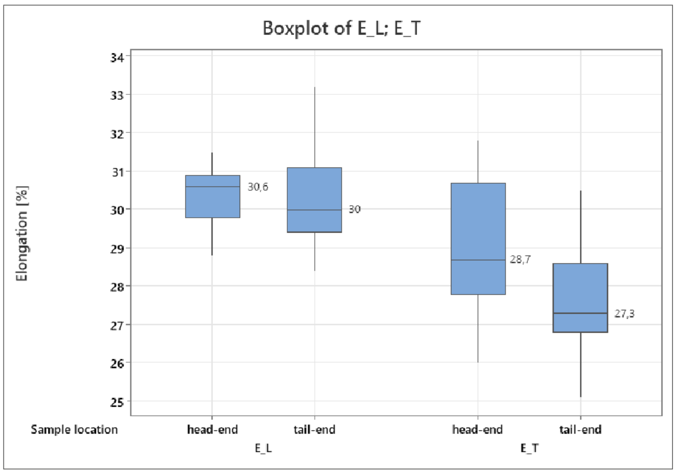

Fig. 5. Boxplot of elongation

The results of the tensile tests indicate that there are clear differences in yield stress and elongation depending on the direction and location of sampling. Material taken from the heads of the coils is characterized by a lower YS than material taken from the tails of the coils. Samples tested in transversal direction are characterized by significantly lower elongation compared to samples tested in longitudinal direction. In the case of tensile strength, no relationship is observed between the location and direction of sampling. The results indicate that hot rolled DP600 steel is characterized by a certain degree of anisotropy of plastic properties. Differences in the properties of material taken from different locations along the length of the rolled strip may be due to the differences in the process parameters occurring during the rolling of a single coil [20,21].

Fig. 6 shows the correlations between the *Vm* and *Sm*, while Fig. 7 shows correlation between Shape and *Sm*.

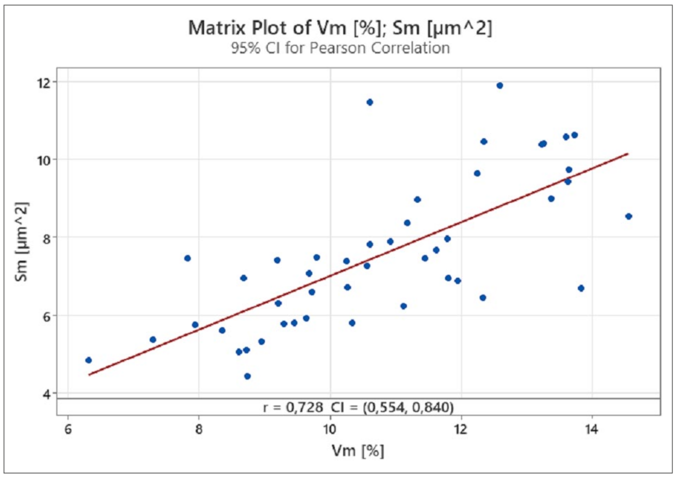

Fig. 6. Correlation between the martensite volume fraction and average martensite islands size

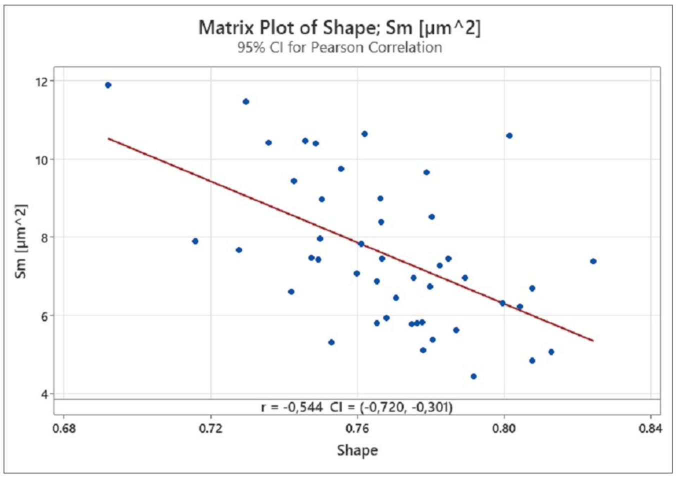

Fig. 7. Correlation between the average martensite islands shape and average martensite islands size

A strong positive correlation ($r > 0.6$) can be seen between the *Vm* and *Sm*. Based on this, it can be concluded that the increase in the *Vm* in DP600 steel is mostly due to increase in the martensite islands size. Another possibility for an increase in the *Vm* could be an increase in the number of martensite islands without affecting their average size. However, as the results presented in Fig. 6 show, in case of hot rolled DP600 steel the *Vm* increases by increase in the *Sm*. A moderate negative correlation ($-0.6 < r < -0.3$) is also observed between the shape of martensite islands and their size. It can be seen that as the *Sm* decreases, their shape factor moves closer to 1.0, which means that the martensite islands are more equiaxial.

Figs. 8-10 show the correlations between the *Vm* and the mechanical properties of DP600 steel.

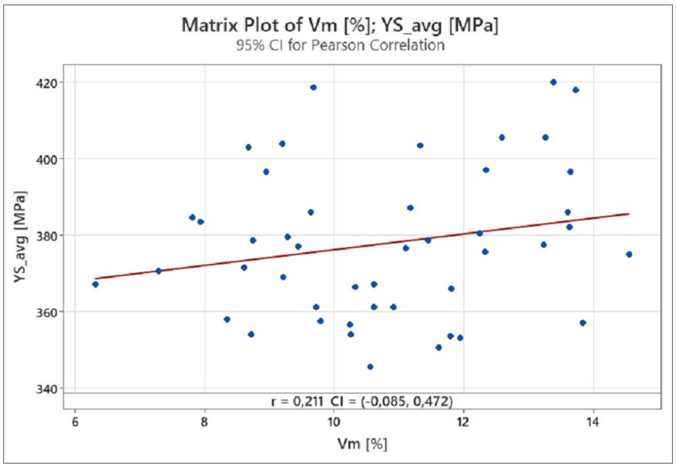

Fig. 8. Correlation between the martensite volume fraction and average yield stress

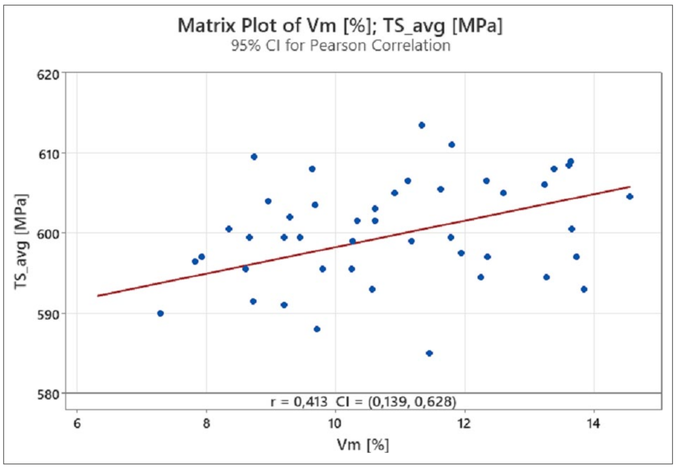

Fig. 9. Correlation between the martensite volume fraction and average tensile strength

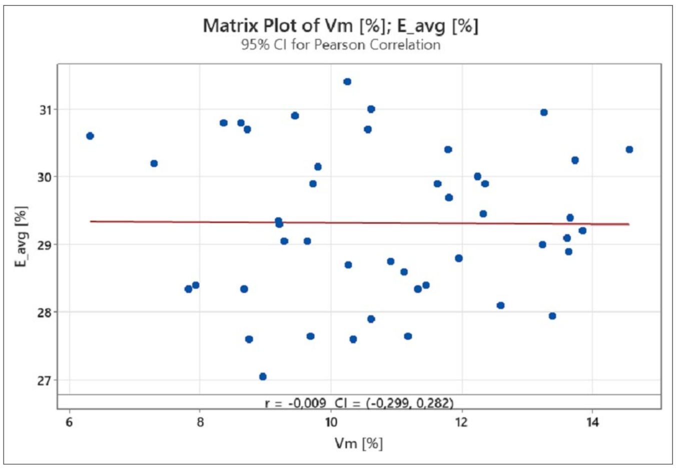

Fig. 10. Correlation between the martensite volume fraction and average elongation

There is a weak positive correlation ($r < 0.3$) between *Vm* and YS and a moderate positive correlation ($0.3 < r < 0.6$) between *Vm* and TS. No correlation ($r = -0{,}009$) was observed between *Vm* and E. These results can be explained by the strengthening effect of martensite in DP600 steels. Due to its high toughness, martensite is hardly deformable and increases strength of dual phase steel [6,7]. In terms of cold formability it is beneficial that in the studied range of 8% to 14% *Vm*, an increase in the *Vm* does not correlate to a decrease in ductility. Fig. 11 shows that there is a moderate positive correlation ($0.3 < r < 0.6$) between the *Sm* and the YS. It can be observed that as the *Sm* increases, the YS increases as well. This can be explained by the fact that the finer, more dispersed martensite islands increase the dislocation density in the adjacent ferrite to a greater extent, which facilitates the onset of plastic deformation [4].

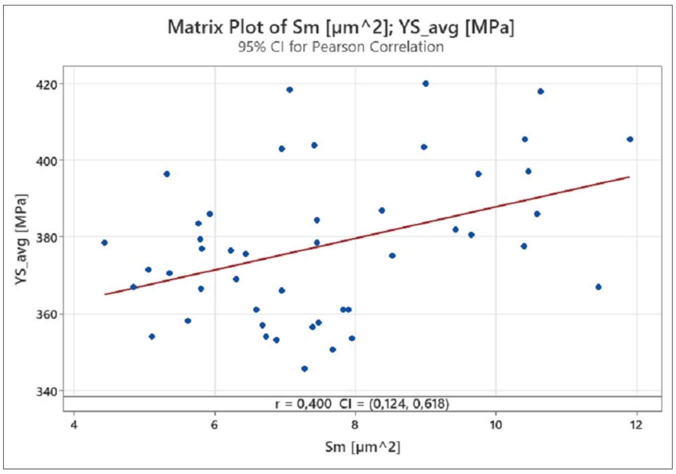

Fig. 11. Correlation between the average martensite islands size and average yield stress

Fig. 12 shows the correlation between the shape of martensite islands and the elongation. The observed relationship is weak ($r < 0.3$), but the results are in accordance with literature data, which also suggest that more equiaxial martensite islands results in better ductility in DP steels, while the presence of ir-

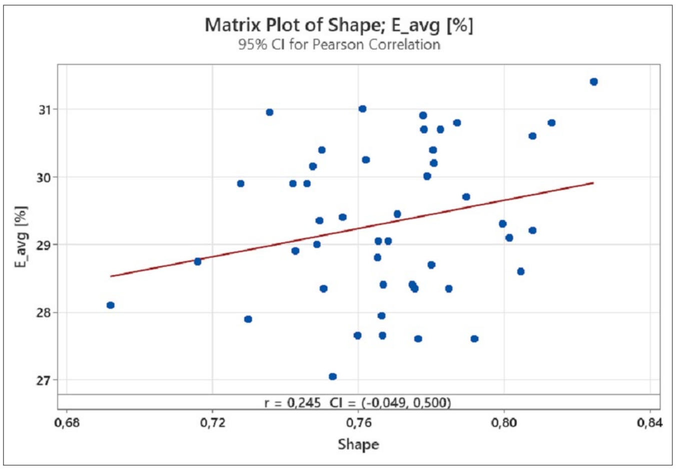

Fig. 12. Correlation between the average martensite islands shape and average elongation

regular martensite islands generally decreases elongation and may be the cause of early cracking during the cold deformation [2,25].

The two following graphs show the effect of chemical composition and some of the hot-rolling process parameters on the *Vm*. Figs. 13-14 show the correlations between *Vm* and the concentration of Mn and Si in the investigated steel.

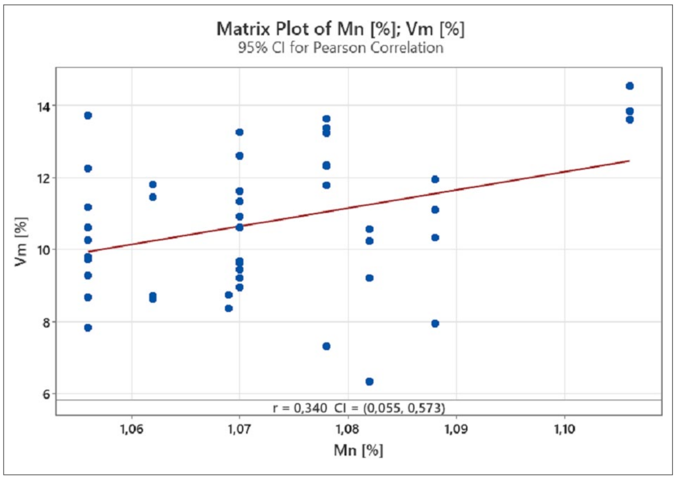

Fig. 13. Correlation between the martensite volume fraction and Mn concentration

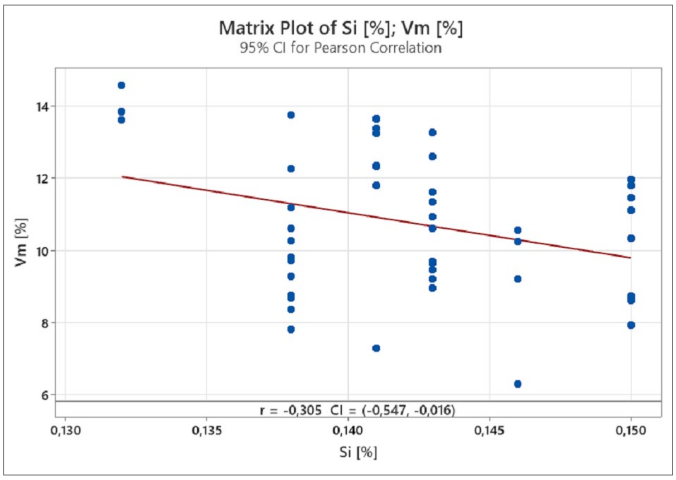

Fig. 14. Correlation between the martensite volume fraction and Si concentration

There is a moderate positive correlation ($0.3 < r < 0.6$) between *Vm* and Mn concentration, and a moderate negative correlation ($-0.6 < r < -0.3$) between *Vm* and Si concentration. This observation can be explained by the effect of both alloying elements on the stability of ferrite and austenite [15]. Mn lowers the transformation temperature of austenite to ferrite and increases austenite hardenability, while Si stabilizes ferrite. Therefore, at the end of first stage of water cooling, material with higher Mn concentration combined with lower Si concentration will have higher volume fraction of austenite, so that in the next intensive water cooling stage, a larger volume of martensite can be formed from the larger volume of austenite. Fig. 15 shows moderate negative correlation ($-0.6 < r < -0.3$) between the strain in the last pass of the roughing mill (RedRM) and *Vm*. It can be observed that higher values of strain result in a decrease in *Vm*.

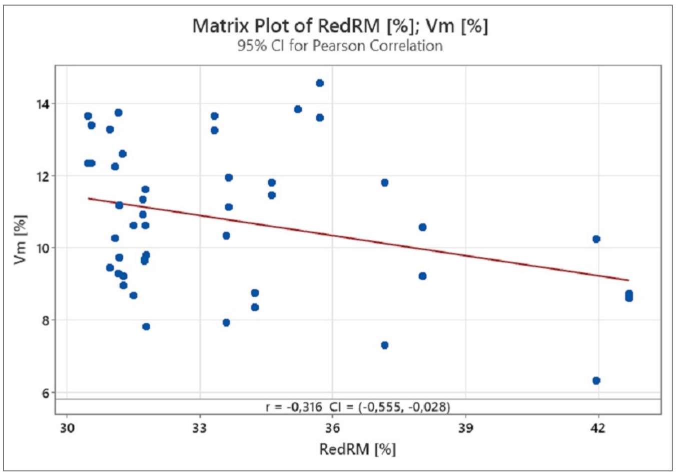

Fig. 15. Correlation between the martensite volume fraction and strain in last pass of roughing mill

This phenomenon can be explained by the effect of plastic deformation on the last pass of the roughing rolling on the austenite grains refinement [12]. A higher strain increases the degree of austenite gains refinement, thus increasing the number of preferential nucleation sites of ferrite during cooling. As a result, after the first stage of water cooling, austenite volume fraction in material is lower and the grains of austenite are smaller, which results in formation of finer martensite islands in the second cooling stage. The contour plot Fig. 16 shows the relationship between the Sm and the intensity of strip cooling in two sections of laminar cooling. The values of water flow in the first stage of strip cooling (avgFlow1) are shown on the y-axis, while the values of water flow in the second stage of strip cooling (avgFlow2) are shown on the x-axis. It can be clearly seen that the material cooled with lower intensity in both cooling sections (low flow values) is characterized by lower *Sm* than the material cooled with higher intensity (high flow values).

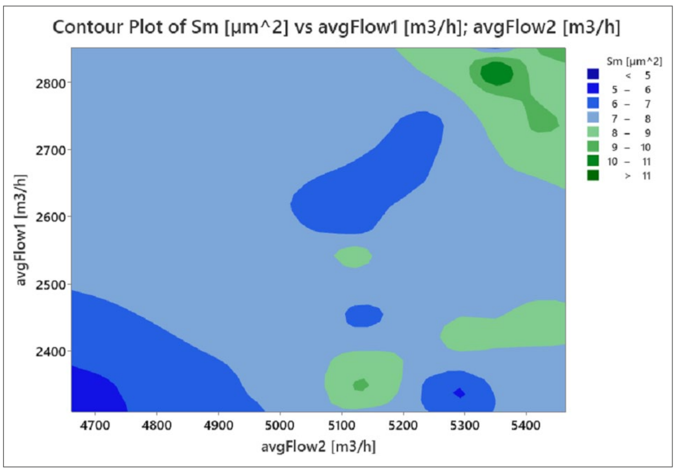

Fig. 16. Correlation between average size of martensite islands and water flow in laminar cooling sections

The large size of martensite islands in the material cooled with high intensity is due to the fact that with intensive cooling, relatively large areas of austenite undergo martensitic transformation throughout their entire volume. On the other hand, low cooling intensity means that more austenite will transform into ferrite or bainite before the temperature of material reach the M_s temperature. The contour plot Fig. 17 shows the relationship between Vm and the rolling speed in the last finishing stand (VF6) and the temperature after the first stage of water cooling (Tint).

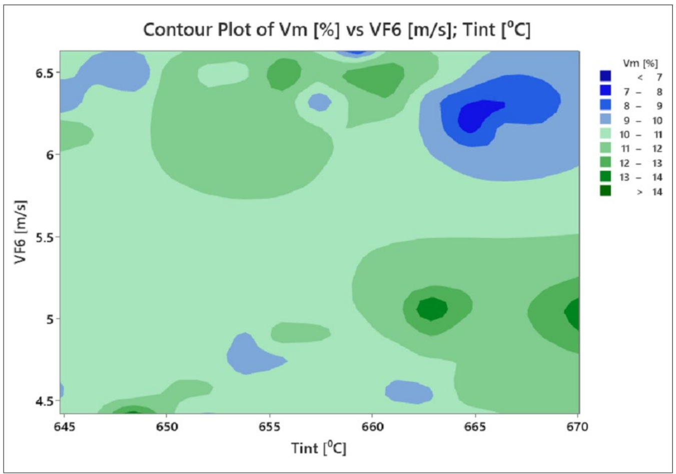

Fig. 17. Correlation between the martensite volume fraction, finishing rolling speed and intermediate temperature

It can be seen that the high rolling speed combined with the high intermediate temperature results in the formation of microstructure with a low Vm. This can be explained by the process of carbon diffusion from ferrite to austenite that occurs during the interval between the first and second stages of water cooling [20]. If the intermediate temperature is too high, then the extent of phase transformation of austenite into ferrite is smaller, which reduces the amount of carbon that diffuses from ferrite to austenite. At the same time, the high rolling speed makes the strip quickly cover the distance between the first and second cooling sections. As a result, the time for carbon diffusion is shorter. Both of these factors limit the diffusion of carbon into austenite, so that its hardenability is reduced. As a result, the Vm in a material produced with a high finishing rolling speed and high intermediate temperature is significantly lower. The last two graphs show the relationship between the yield stress, finishing rolling speed and carbon concentration (Fig. 18) and between the elongation, finishing rolling speed and carbon concentration (Fig. 19).

It can be seen that a low finishing rolling speed has the effect of lowering the yield stress and simultaneously increasing the elongation of the material. Contrary, increasing the carbon content has the opposite effect, that is, it increases yield strength and lowers elongation. The effect of rolling speed on mechanical properties may be due to the recrystallization process of the material that starts before the cooling process begins, once the

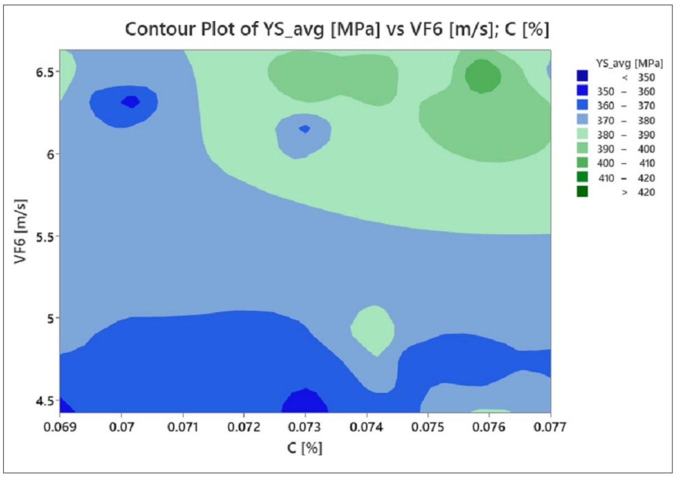

Fig. 18. Correlation between the yield stress, finishing rolling speed and carbon concentration

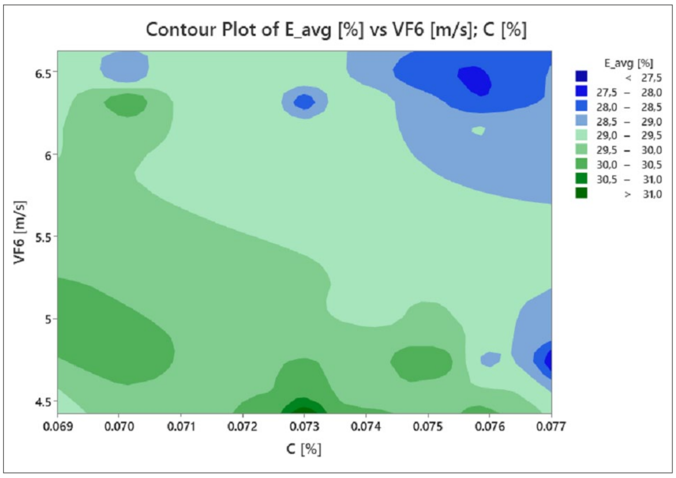

Fig. 19. Correlation between elongation, finishing rolling speed and carbon concentration

deformation in the last finishing stand is completed. The strain in last finishing stand is constant and equals approximately 20%, while the rolling speed gradually increases during rolling of a single strip from about 4,5 m/s to about 6,5 m/s, which is shown on y-axis of contour plot Fig. 19. If the rolling speed is low, then the time between end of deformation and start of cooling is long enough for recrystallization to complete, so that larger ferrite grains are formed during the cooling process, resulting in increased ductility of the material. At high rolling speeds, the material enters the cooling zone with a structure that is not fully recrystallized, leading to greater strengthening of the material due to the ferrite grains refinement during the cooling process, which results in lower elongation and higher yield strength. The effect of carbon content on mechanical properties can be explained by the effect of solution strengthening of ferrite, as well as the effect of increase in the hardness difference between the ferrite and martensite. The carbon rich martensite has a higher hardness, thus increasing the hardness difference between the two phases, which is known to be unfavourable in terms of the plastic properties of DP steel [7].

4. Conclusions

The presented analysis of the relationships between the chemical composition, process parameters, microstructure and mechanical properties of DP600 steel leads to the following conclusions:

1. The mechanical properties of DP600 steel obtained in the hot rolling process vary depending on whether the material comes from the head-end or the tail-end of the strip. These differences are due to the variations of some of the hot rolling process parameters along the length of the rolled strip. These differences, combined with the anisotropy of mechanical properties, results in that the difference in yield stress of material taken from the same hot rolled coil can be up to 25 MPa, and the difference in elongation can be up to 3%.
2. There are clear relationships between *Vm* and *Sm* and between *Sm* and average shape of the islands. As *Vm* increases *Sm* increases too, while as Sm increases, the shape of martensite islands decreases. While an increase in *Sm* alone is beneficial and leads to an increase in yield stress, the accompanying decrease in the martensite islands aspect ratio has an adverse effect and slightly lowers E.
3. An increase in *Vm* results in increase of both YS and TS, while correlation between *Vm* and TS is stronger than between *Vm* and YS. In the studied range of *Vm* between 8% and 14%, no effect of *Vm* on elongation was found. The highest *Vm* values are achieved on material with high Mn content and low Si content. This is due to the effect of these alloying elements on the stability of austenite and ferrite as well as austenite hardenability.
4. In order to increase *Vm* in the hot rolling process, it seems to be more advantageous to use low strain in the last pass of the roughing mill. High intensity of strip cooling at both cooling stages results in the formation of a microstructure with higher *Sm*. High rolling speed combined with high intermediate temperature results in microstructure with lower *Vm*. Low rolling speed results in lower YS and higher elongation of DP600 steel, while increasing carbon has an opposite effect on YS and elongation.

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