THE EFFECT OF ANNEALING TEMPERATURE, INTERCRITICAL PAUSE AND INITIAL MICROSTRUCTURE OF LOW CARBON STEEL ON DP MICROSTRUCTURE FORMATION

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Abstract

The ferritic-martensitic microstructure is typical for the dual-phase steels such as DP600. This microstructure can be obtained during the hot rolling process followed by interrupted water cooling with short isothermal pause in the intercritical region. In manufacturing of the DP steel by hot rolling process the final deformation takes place slightly above A_{r3}, where the material is composed of austenite alone. Upon completion of plastic deformation, the hot strip is rapidly cooled to the temperature between A_{r3} and A_{r1}, where supercooled austenite undergoes phase transformation into ferrite. Since both the temperature and time spent in this temperature region are not sufficient for complete phase transformation, remaining austenite is quenched during the second step of cooling process, which leads to the formation of a martensite. By knowing the relation between the temperatures used in the process and resulting microstructure - especially martensite volume fraction, it is possible to design industrial process so that the requirements regarding mechanical properties of a material can be obtained. Two experiments were carried out to analyze the mentioned process parameters. First, the quenching experiment, where samples with two different initial microstructures, ferritic-pearlitic and ferritic-martensitic, were annealed in wide range of temperatures and then quenched. Second, the two stage cooling from austenite region, where samples were first annealed at different temperatures just above A_{r3}, then cooled and held for a few seconds at different intercritical temperatures and finally quenched. Resulting microstructures allowed for evaluation of phase composition and its morphology in relation to temperatures used in experiments.

Keywords: dual phase, annealing, quenching, microstructure.

1. INTRODUCTION

The dual-phase microstructure found in DP600 steel results in its properties that are highly desirable in the automotive industry. Car manufacturers particularly value the combination of high strength and good ductility that characterizes this type of steel. For the passengers safety, an important feature of this material is its high work hardenability. On the other hand, in cold forming operations used in manufacturing of complex body components, the lack of pronounced yield point and cracking resistance make this steel highly appreciated [1-3]. The mechanical properties of DP steels are mostly influenced by the volume fraction and morphology of the hard structural constituent islands. Islands of this second phase (hard structural constituent) usually consist of martensite or bainite [4-7]. While analyzing the formation of a dual-phase microstructure in the hot rolling process, it is necessary to take into account factors that significantly affect the kinetics of phase transformations. Among the factors of greatest importance are: the degree of plastic deformation, the segregation of alloying elements, the cooling rate and the temperatures at various stages of the process. By knowing the A_{r1}, A_{r3}, and M_s temperatures it is possible to define the hot rolling process parameters that will ensure satisfactory quality of the material and repeatability of the achieved results [8-13]. Currently, the most widely published research on DP steels concerns mainly microstructures obtained by annealing and controlled cooling processes. This paper presents the results of research that can be helpful in designing or modifying the hot rolling process of DP600 steel. The purpose of the study was to determine the effect of carbon segregation, that may result from different processing parameters in hot rolling process, on the phase transformations kinetics and the effect of annealing temperature and intercritical pause on the formation of the dual-phase microstructure.

2. MATERIAL AND METHODOLOGY

In order to determine the effect of carbon segregation on the phase transformation kinetics, an experiment was conducted where steel samples with the same chemical composition but two different initial microstructures resulting from different processing parameters were annealed for 20 minutes at different temperatures in the range of 740ºC-880ºC, followed by quenching in water. The dimensions of the samples were 30 mm x 20 mm x 4 mm. The chemical composition of the investigated steel is shown in **Table 1**.

Table 1 Chemical composition of the investigated steel (mass %).

C	Mn	Si	Cr	Fe
0.065 - 0.075	1.00 – 1.20	0.10 – 0.30	0.40 – 0.60	bal.

One set of samples had a ferritic-martensitic (FM) microstructure. These samples were taken from a hot rolled coil of DP600 steel coiled at 130ºC. Second set of samples with a ferritic-pearlitic (FP) microstructure was taken from a hot rolled coil of E295 steel coiled at 620ºC. **Figure 1** shows images of the initial microstructure of these two steels before annealing and quenching.

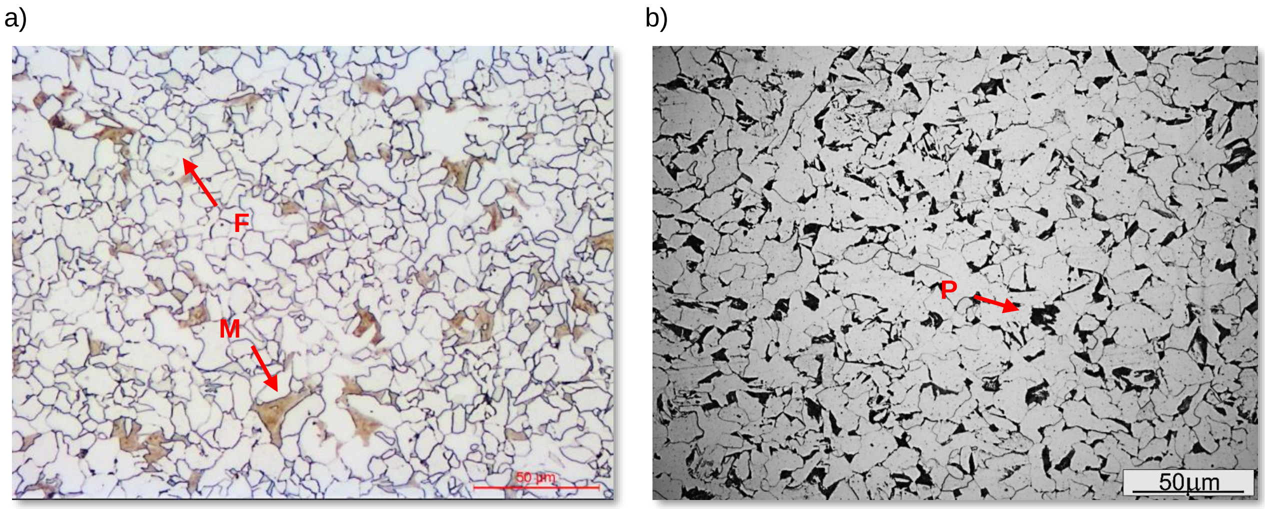

Figure 1 Initial microstructures before quenching series: a) ferritic-martensitic (DP600); b) ferritic-pearlitic (E295).

Researchers report, that the degree of C segregation in FM steel is expected to be greater than in FP steel [3, 10]. After quenching, specimens were polished and etched with nital 2%. Images from light microscope were then processed by point count technique to calculate the ferrite volume fraction in each sample. Hardness tests by the Vickers method with load 50 N were also carried out on the prepared specimens. In the second experiment, steel with the same chemical composition as in quenching series and FP structure was tested. During the experiment 16 samples were annealed for 20 minutes at various temperatures in the range of 810ºC-870ºC. After this time, the samples were immersed for about 10 seconds into a liquid zinc bath heated to various temperatures in the range of 610ºC -670ºC. The samples were then quenched in water. **Figure 2** shows the schematic view of the experiment setup.

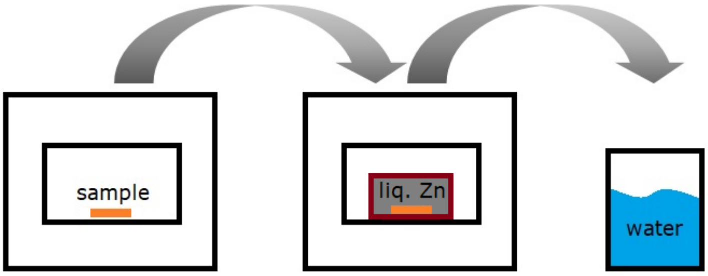

Figure 2 Schematic view of the experiment setup.

The idea behind the experiment was to simulate the two stage cooling at the end of hot rolling process. The annealing temperatures in this experiment correspond to the feasible final rolling temperatures, while the temperatures of the zinc bath in which the samples were immersed correspond to the feasible temperatures of pause in water cooling. All 16 heat treatment variants performed in this experiment are shown in **Table 2**.

Table 2 Temperature variants used in the experiment.

Intercritical temperature / Temperature of Zn bath	Final rolling temperature / Annealing temperature			
	870ºC	850ºC	**830ºC**	810ºC
670ºC	S1	S8	S9	S16
650ºC	S2	S7	S10	S15
630ºC	S3	S6	**S11**	S13
610ºC	S4	S5	S12	S14

The sample S11 was annealed at the temperature corresponding to the final rolling temperature used in the process and immersed in the zinc bath heated to the temperature at which the pause in water cooling takes place during hot rolling. **Figure 3** shows dilatometric curve obtained during the slow heating of ferritic-pearlitic sample. At the temperature 840ºC the phase transformation kinetic is the highest, which is in accordance with the quenching series results presented later on.

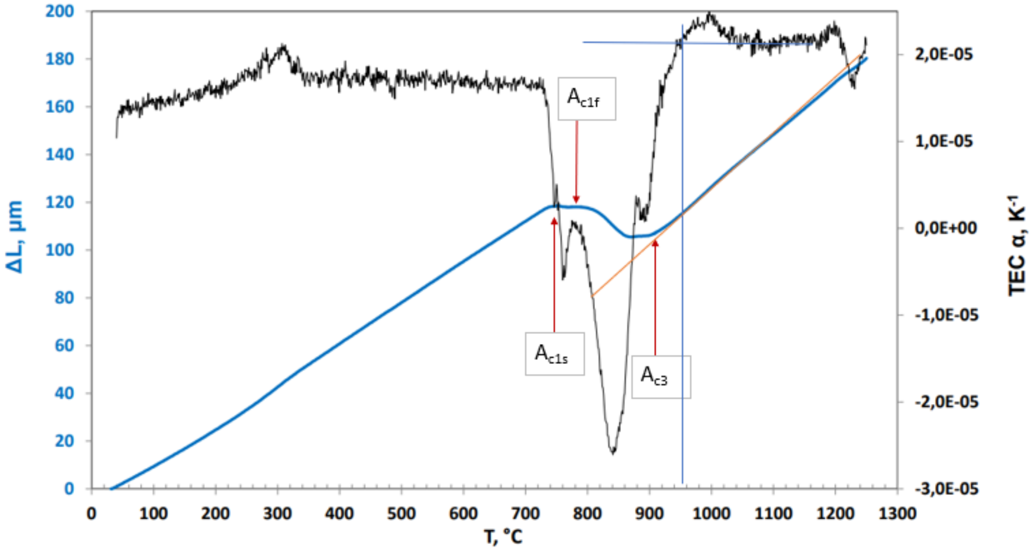

Figure 3 Dilatometric curve during slow heating of FP sample.

3. DISCUSSION

The results of ferrite volume fraction (V_f) and hardness measurements of samples quenched from different annealing temperatures are shown in **Figure 4**. In this experiment, the V_f should be interpreted as a sample volume that at a given temperature did not transform into austenite during annealing.

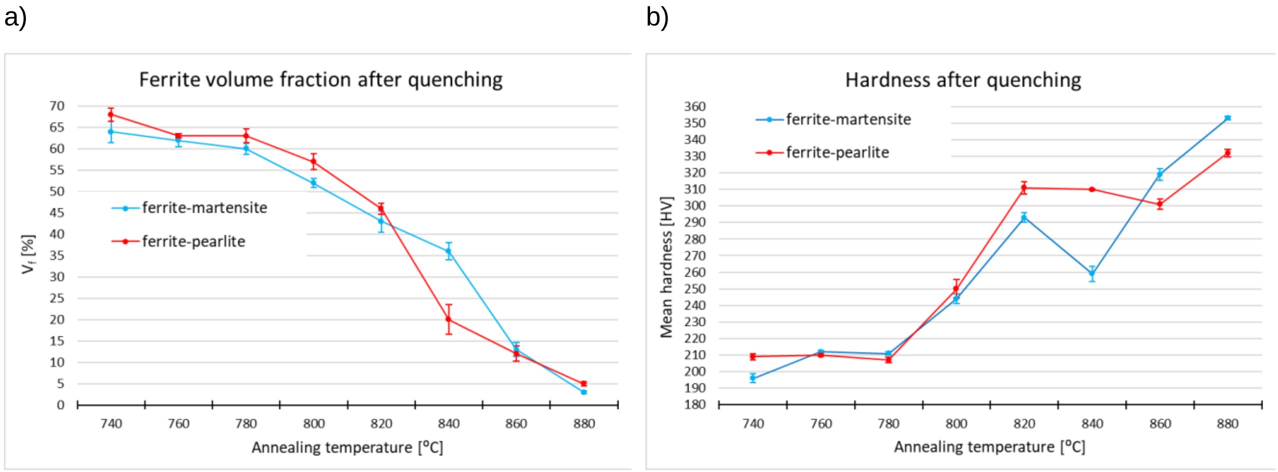

Figure 4 Results of ferrite volume fraction measurements (a) and Vickers hardness (b).

With increasing annealing temperature, V_f decreases in both sets of samples, because increasing annealing temperature leads to a greater austenite volume fraction formation in material microstructure. Initially, in temperature range from 740ºC to 820ºC the decrease of V_f progresses slowly. Sample with ferritic-pearlitic initial microstructure shows highest phase transformation kinetic at temperature 840ºC, where the V_f drops from 46% at 820ºC to 20% at 840ºC. In case of ferritic-martensitic initial microstructure, this temperature seems to be shifted to 860ºC, where V_f drops from 36% at 840ºC to 13% at 860ºC. The difference between the results for both sets of samples may come from different carbon concentration in initial ferrite. The ferrite in FM sample may be depleted of C, as during the production of DP600 at hot strip mill, C diffuses from ferrite to austenite during the pause in water cooling. There is a good correlation between increasing hardness and decreasing V_f, as with decrease in V_f, volume fraction of bainite or martensite increases, which results in higher hardness. Hardness difference between the two sets of samples is the biggest at 840ºC, which can be partially explained by the biggest difference in V_f at that temperature. **Figure 5** shows samples microstructures after quenching from temperature 840ºC.

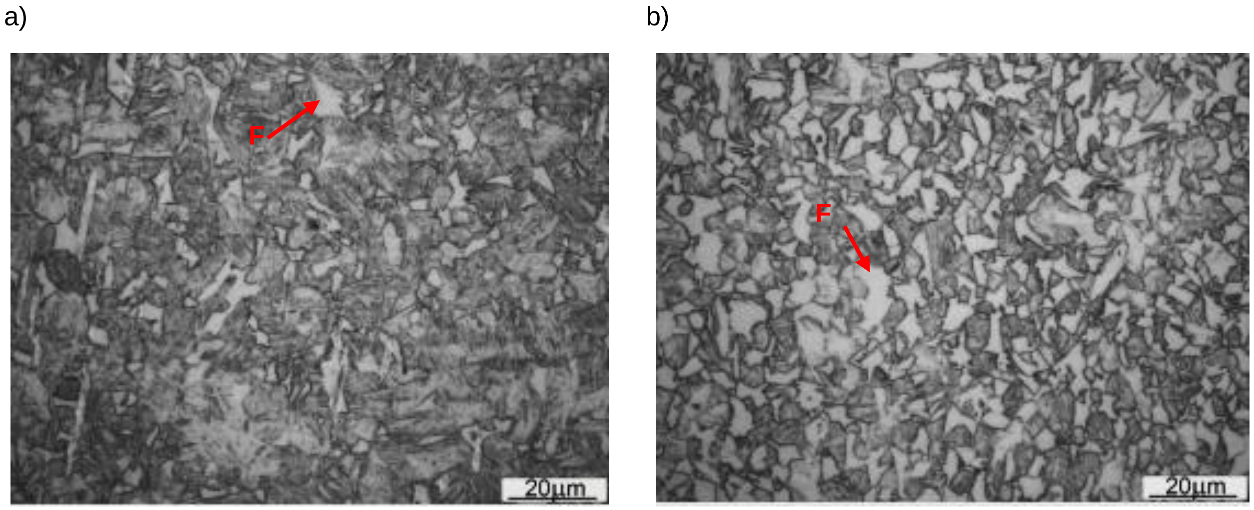

Figure 5 Microstructures of FP sample (a) and FM sample (b) as quenched from 840ºC.

The results of phase composition analysis of samples investigated in the second experiment are presented in **Figure 6**. These results were also obtained by point count technique.

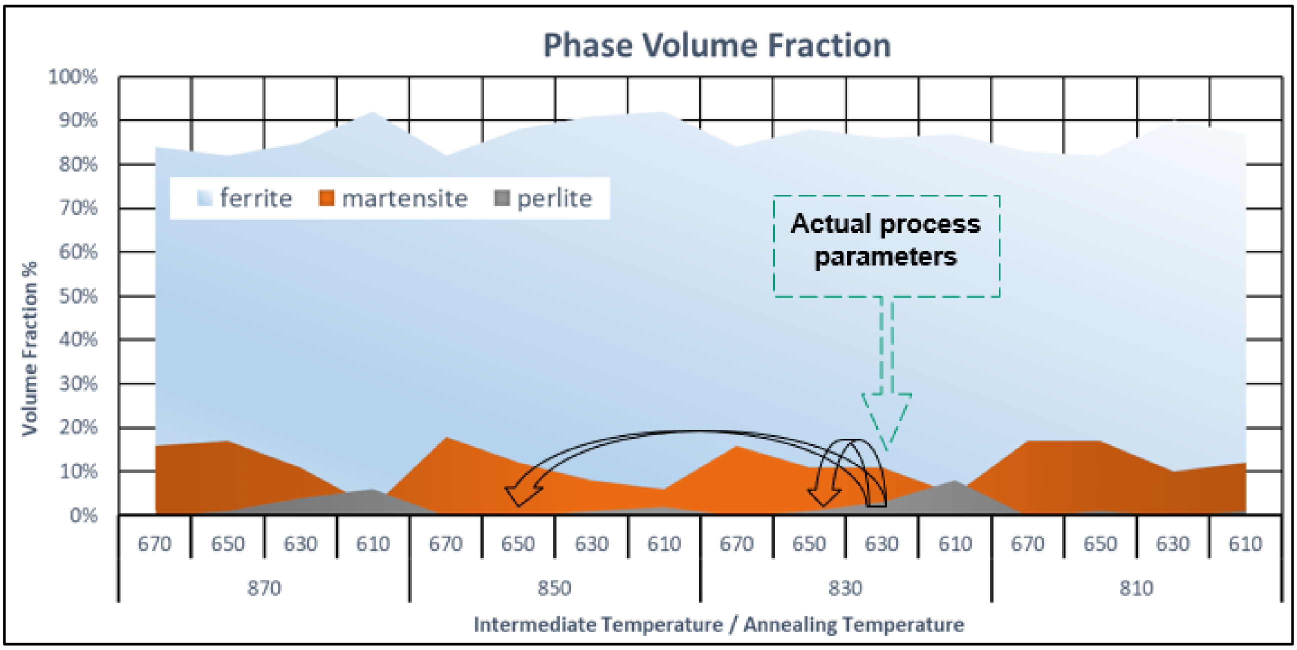

Figure 6 Phase composition of samples treated with different temperatures in the second experiment.

Green arrow on the graph points to the actual process parameters used during the industrial hot rolling process (final rolling temperature of 830ºC and intermediate temperature of 630ºC). The results shows, that lower intermediate temperatures promotes pearlite formation, which should normally be avoided as pearlite can cause brittleness and lead to early cracking during the cold forming. In order to produce DP steel with higher martensite volume fraction one should consider using higher intermediate temperatures. Annealing temperatures does not seem to have strong effect on phase composition, but it should be further investigated weather or not it can influence the morphology of the hard phase. Black arrows indicate possible variants of process parameters that could possibly lower the risk of pearlite formation while keeping the same martensite volume fraction as for current process parameters.

4. CONLCUSION

- The highest ferrite to austenite transformation rate is shifted to 860ºC in FM steel compared to 840ºC in FP steel. It may be explained by lower carbon concentration in ferrite matrix of DP steel comparing to conventional ferritic-pearlitic steel.
- Hardness increases with decrease of V_f for both FM and FP initial microstructures except for FM sample quenched from 840ºC. The drop of hardness at this temperature might be explained by tempering of initial martensite from DP microstructure.
- By changing both annealing and intermediate temperatures, it is possible to reduce the risk of pearlite formation while keeping the same martensite volume fraction.
- It seems that annealing temperature does not affect the martensite volume fraction measured in the second experiment, however it should be further investigated if it has an influence on the morphology of the hard phase.

Based on the results from the two experiments it is evident that carbon segregation in DP steels needs to be accounted for in the future dilatometric tests planned for M_s temperature determination.

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