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# MICROSTRUCTURE EVOLUTION AND MECHANICAL PROPERTIES OF C-Mn COLD ROLLED DUAL PHASE STEEL AFTER CONTINUOUS ANNEALING PROCESS IN LABORATORY CONDITIONS

# ZMIANY MIKROSTRUKTURY I WŁAŚCIWOŚCI MECHANICZNE WALCOWANEJ NA ZIMNO, DWUFAZOWEJ STALI C-Mn PO PROCESIE CIĄGŁEGO WYŻARZANIA W WARUNKACH LABORATORYJNYCH

The article deals with the influence of annealing parameters on evolution of microstructure and mechanical properties of dual phase steel. Dual phase steel was annealed in laboratory conditions according to the three chosen cycles of annealing: into intercritical region (780°C), into austenite region (920°C) and into austenite region (920°C) by subsequently cooling into intercritical region (780°C) with the hold at the temperature of 495°C. Simulation of annealing regimes by thermo-mechanical simulator Gleeble was done. The obtained microstructure consists from three phases: ferritic matrix, martensite and martensite/bainite grains. For the microstructure identification the TEM and nanoindentation experiments were performed.

*Keywords*: dual phase steel, microstructure, annealing simulation, TEM observations, nanoindentation method

Praca dotyczy wpływu parametrów wyżarzania na zmiany mikrostruktury i właściwości mechaniczne stali dwufazowej C-Mn. Stal dwufazową poddano wyżarzaniu w warunkach laboratoryjnych według trzech wybranych cykli: w zakresie międzykrytycznym (780°C), w obszarze austenitu (920°C) i w obszarze austenitu (920°C) ze schładzaniem do zakresu międzykrytycznego (780°C) przy temperaturze wytrzymania 495°C. Przeprowadzono symulację schematów wyżarzania przy użyciu symulatora obróbki cieplno-plastycznej Gleeble. Uzyskana mikrostruktura składa się z trzech faz: osnowy ferrytycznej, martenzytu oraz ziaren martenzytu/bainitu. W celu identyfikacji mikrostruktury wykonano badania metodą TEM oraz nanoindentacji.

## 1. Introduction

At present, an increasing interest of automotive companies in using high strength steels, including dual phase steels (DP), is observed. The DP steels are characterized by a good combination of strength and elongation, relatively low yield strength and a favourable ratio of yield strength to the ultimate tensile strength (YS/UTS) [1-3].

Cold rolled DP steels are mainly produced in the process of continuous annealing in the dual phase (intercritical) region of ferrite and austenite. A typical continuous annealing regime consists of the stage of heating up to the required temperature, hold time, followed by rapid cooling, and ageing. Modifications of parameters of continuous annealing, especially of temperature, have pronounced the influence on the microstructure development and thereby also on properties of steel sheets [4-6].

The microstructure of DP steels consists of dispersive islands of hard martensitic phase, soft ferritic matrix and a small amount of retained austenite. The influence of retained austenite, the morphology and distribution of martensite and ferrite grains, are still objects of continued research. During the production process, final properties of the DP steels are influenced by many factors, such as a temperature of intercritical annealing, temperature of zinc coating process and the cooling rate during the heat treatment process. With changes in parameters of continuous annealing process, the volume fraction of martensite, its morphology and distribution, the amount of ferrite and residual austenite, and also the grain size of ferrite [7-9] are changed.

## 2. Material and experimental procedure

The experimental material was produced in a laboratory resistance furnace in an inert atmosphere of argon. Subsequently, the hot rolling under laboratory conditions followed by cold rolling with the final reduction of 70% was performed. The chemical composition of studied material is presented in Table 1. To obtain the desired properties, the Cr and Mo content ratio was 1:1.

TABLE 1

Chemical composition of the DP steel

| C | Mn | Cr + Mo | P | S | Si | $Al_{total}$ | V |
|---|---|---|---|---|---|---|---|
| 0.10 | 1.90 | 1.00 | 0.020 | 0.005 | 0.80 | $\leqslant$2.00 | 0.002 |

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Simulation of annealing regimes using the thermo-mechanical simulator Gleeble was performed. Three annealing regimes were selected. The first annealing cycle consisted of heating into the intercritical region (780°C) and subsequent cooling with the hold at 495°C (1); the second cycle involved heating into the austenite region (920°C) (2) and subsequent cooling with the hold at 495°C; and the third cycle consisted of heating into the austenite region (920°C) and subsequent cooling into intercritical region (780°C) with the hold at 495°C (3), as shown in Fig. 1. For calculations of transformation temperatures of the intercritical region $A_{c1}$ and $A_{c3}$, the following equations were used [10]:

$$\mathbf{A_{c1}} = 723-10.7\mathrm{Mn}-16.9\mathrm{Ni}+29.1\mathrm{Si}+16.9\mathrm{Cr}+290\mathrm{As}+6.8\mathrm{W} \quad (1)$$

$$\mathbf{A_{c3}} = 910 - 203\mathrm{C}^{1/2} - 15.2\mathrm{Ni} + 44.7\mathrm{Si} + 10.4\mathrm{V} + 31.5\mathrm{Mo} + 13.1\mathrm{W} - (30\mathrm{Mn} + 11\mathrm{Cr} + 20\mathrm{Cu} - 700\mathrm{P} - 400\mathrm{Al} - 120\mathrm{As} - 400\mathrm{Ti}) \quad (2)$$

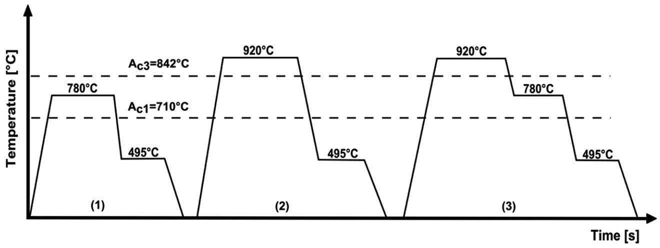

Fig. 1. Annealing regimes for DP steels

The microstructure of dual phase steel was documented by the optical microscopy and scanning electron microscopy. To obtain colour contrast, two etchants were used, starting with the Nital and thereafter by Klemm. The grain boundaries were well exposed by using the Nital etchant. The grain bodies were contrasted by Klemm colour etching. As a result of colour etching, a favourable contrast of different phases was obtained. The ferrite grains are in brown or blue tones, the martensite is brown and the austenite phase is white [11]. For the SEM observations, only Nital was used as an etchant.

In order to identify the individual phases in steel microstructure, the transmission electron microscopy observations were performed for all annealing regimes. The discs were electro-polished using a standard double-jet procedure. For the electro-polishing process, the $CH_3OH$:$HClO_4$ electrolyte, in the 9:1 ratio, was used. The polishing process was conducted in the temperature range of -35°C to -25°C at 18 V.

For determination of nanohardness (H) of individual phases, the nanoindentation measurements were performed by a Nano Indenter G200 fitted with the Berkovich tip. All annealing regimes were studied and 25 measurements in total for each phase and regime were done. The measurements of hardness of ferritic grains were performed by maximal loading force of $F_{max}$ = 19.62 mN, for hardness measurements of martensitic and martensitic/bainitic grains, the maximal loading force of $F_{max}$ = 0.981 mN was utilised. With relation to the size of martensitic and martensitic/bainitic grains in comparison with ferritic grains, the lower loading force has to be used for avoiding of hardness distortion by surrounding ferritic grains [12].

## 3. Results and discussion

The values of yield stress and the ultimate tensile stress showed a local maximum at the second chosen annealing cycle. No change of elongation was observed and this was kept at the value of 19%. The strain hardening exponent *n* values reached the same value for the first and the second cycles and for the third cycle, the higher value was obtained. The ratio YS/UTS with the changeable annealing cycle decreased and this drop ranged from 0.70 to 0.61, Fig. 2.

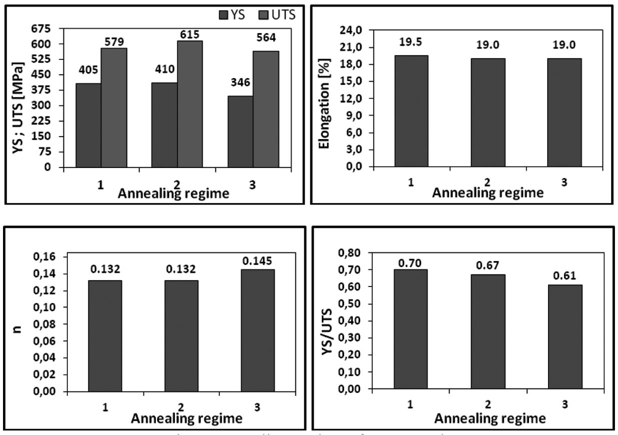

Fig. 2. Mechanical properties of DP steels

Figure 3 showed the microstructures of all studied regimes of annealing. For the first selected regime, the microstructure consists of ferritic matrix, austenite and areas suggested to be martensite. No typical martensite was observed in the microstructure. After the second selected regime of annealing, the microstructure formed by the ferritic matrix, austenite and martensite was observed. In this case, the partly martensite grains were found whereas the austenite was around. Moreover, grains of the needle-like structure were observed. After the third annealing regime, the microstructure composed also of ferritic matrix, austenite and martensite, observed in the case 2, was found; however, in comparison with the regime 2, higher occurrence of martensitic grains, was observed.

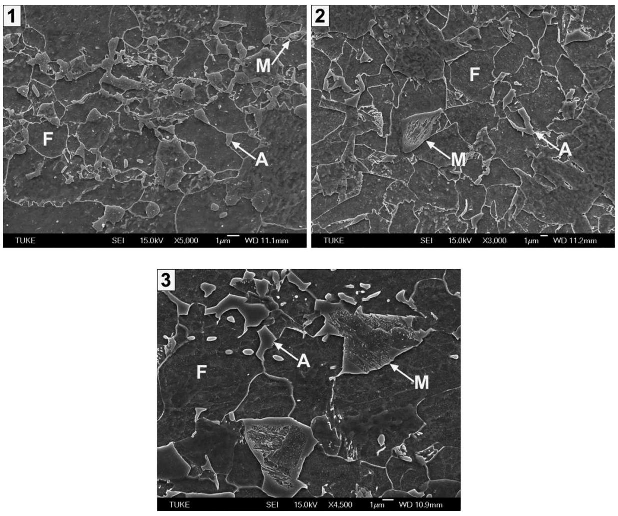

Fig. 3. Microstructure of DP steel: 1. annealing regime 780°C-495°C; 2. annealing regime 920°C-495°C; 3. annealing regime 920°C-780°C-495°C

The microstructure of dual phase steel was documented by the optical microscopy. To obtain colour contrast, two etchants were used, starting with the Nital followed by Klemm. The microstructure of DP steel consists of all regimes of annealing, from the ferritic matrix (tones of brown colour) to austenite (white colour). It seems, based on the SEM observations, that the white grains with brown colour in the middle are martensitic grains surrounded by austenite (regimes 2 and 3).

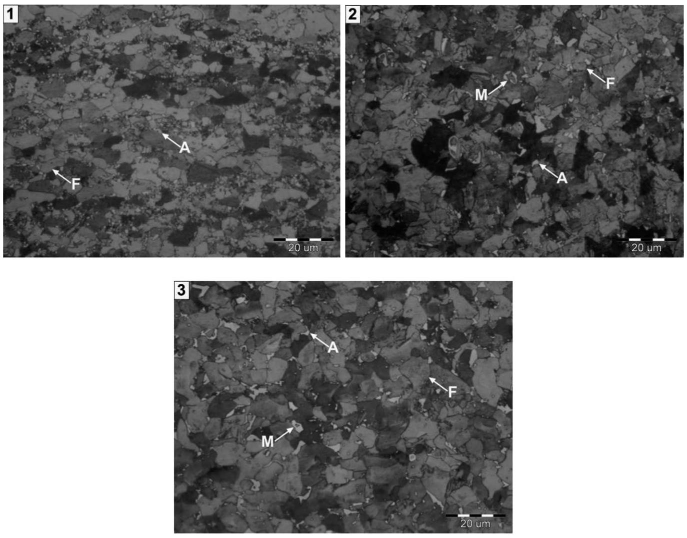

Fig. 4. Microstructure of DP steel: 1. annealing regime 780°C-495°C; 2. annealing regime 920°C-495°C; 3. annealing regime 920°C-780°C-495°C

When the martensitic grains were not surrounded by the austenite phase and the transformation occurred within the whole grain, no remarkable contrast between the ferrite and martensite by colour etching was obtained. For this reason, a specimen etched by Nital was used and SEM observations were performed. Then, the colour etching by Klemm etchant was performed and observations by optical microscopy were done. Results of martensitic grain identification are given in Fig. 5 where the red arrows showed the martensitic grains.

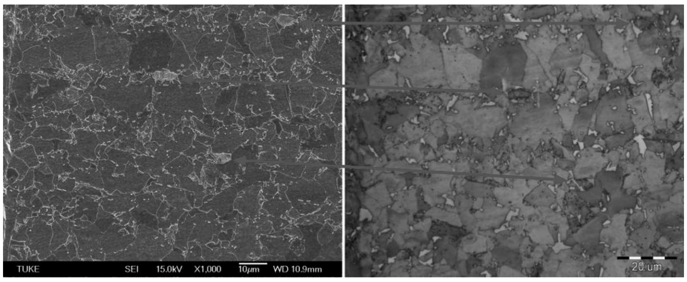

Fig. 5. Identification of martensitic phase

The TEM analysis was performed for two annealing regimes: 780°C-495°C and 920°C-780°C-495°C. In the case of 780°C-495°C regime, the microstructure consists of the ferritic grains with the islands of martensitic grains, Fig. 6 a). Fig. 6 b) shows the detail view of martensitic grain with the high density of dislocations in individual martensitic plates.

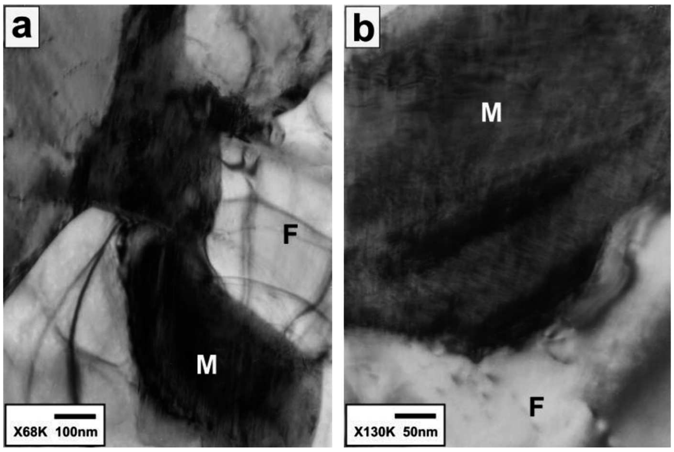

Fig. 6. Annealing regime 780°C-495°C: a) characteristic structure b) details of martensitic grain

Figure 7 shows the final characteristic microstructure of specimen after the 920°C-780°C-495°C regime. The microstructure consists of bainitic grains, Fig. 7 a), and bainitic grains surrounded by the martensitic phase, Fig. 7 b). Figure 7 c) shows details of martensitic grain next to bainite structure.

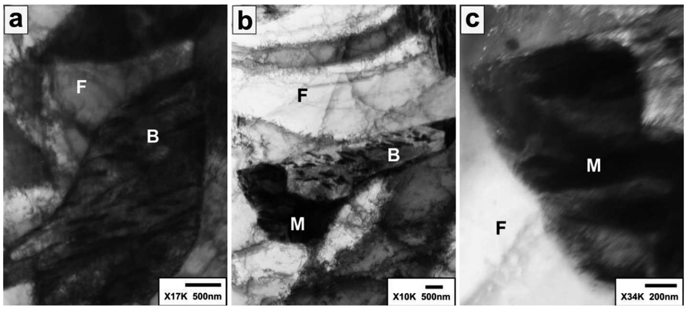

Fig. 7. Annealing regime 920°C-780°C-495°C, a) a bainitic grain, b) a partly transformed grain of bainite surrounded by martensite c) details of martensitic grain

Figure 8 shows the correlation of individual phases and structures observed by means of the optical microscopy, SEM and TEM. Figure 8 a) shows the microstructure observed by SEM and Figure 8 b) shows the same area observed by the optical microscopy after colour etching. The TEM was used to find the partly transformed grains analyzed using optical microscopy and SEM. With respect to the SEM observations, the grains seemed to be martensitic. The TEM observations showed that the partly transformed grains were, in fact, bainite. The TEM observations also confirmed that the phase which totally or partly surrounded bainitic grains and seemed to be untransformed austenite was actually martensite. The evidence of that is fragmentation into the smaller plates and the high dislocation density. In the case of colour etching (Klemm etchant), it can be concluded that the white grains represent martensite, bainite is brown and ferrite is of the brown colour; in the case of bainite, it is darker.

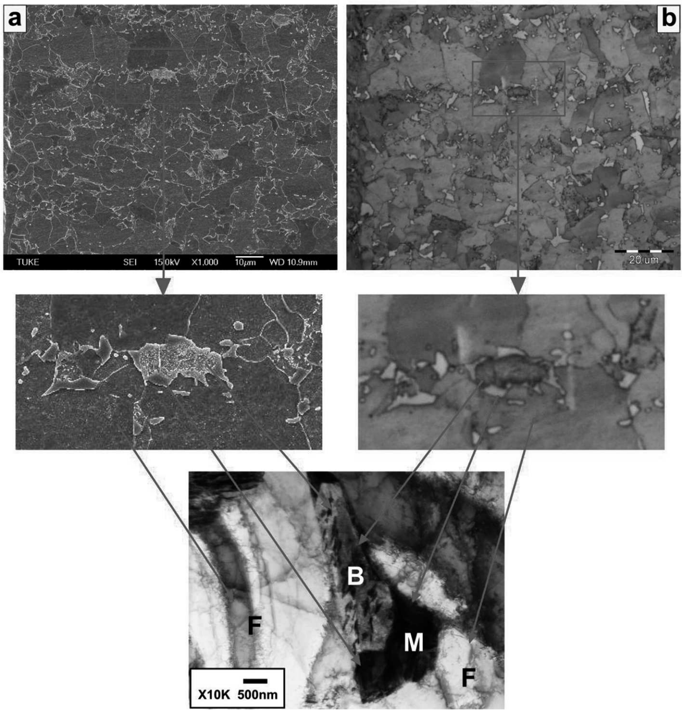

Fig. 8. Identification of individual phases and structures observed by means of the optical microscopy, SEM and TEM

Table 2 summarizes the results of nanoindentation measurements $H_{IT}$. Three different values of hardness were obtained. The hardness for ferrite was 2.5±1 GPa. By simulation of annealing regime at temperature 495°C, martensite is tempered and, therefore, the lower values of hardness were obtained (4.1 to 4.5 GPa). The hardness of mixed grains martensite/bainite (bainite surrounded by martensite), for the 920°C-495°C and 920°C-780°C-495°C annealing regimes, were 7.1 and 7.2 GPa, respectively.

TABLE 2

Summary of the nanoindentation measurements

| **Specimen** | **$H_{IT}$ [GPa]** | **$H_{IT}$ [GPa]** | **$H_{IT}$ [GPa]** |
|---|---|---|---|
| **1** | 2.4 | 4.2 | - |
| **2** | 2.5 | 4.1 | 7.1 |
| **3** | 2.6 | 4.5 | 7.2 |
| **1. Annealing regime:** 780°C-495°C | | | |
| **2. Annealing regime:** 920°C-495°C | | | |
| **3. Annealing regime:** 920°C-780°C-495°C | | | |

## 4. Summary

1. Applying of the annealing regime which consists of heating into the austenite region, results in the uniform structure. The regime with the heating into austenite region and subsequent cooling and holding in the intercritical region ensures more stable mechanical properties with good strength and plastic characteristics of the studied dual phase steel.
2. The microstructure consists of the ferritic matrix, the islands of martensite and a mixed structure consisting of bainite and martensite.
3. The TEM observations markedly defined the individual phases of studied steel after simulated conditions of annealing.
4. Nanoindentation measurements confirmed three different values of nanohardness for three phases in the microstructure of DP steel.

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