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Heat treatment and mechanical properties of low-carbon steel with dual-phase microstructure

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<u>ABSTRACT</u>

Purpose: The aim of the paper is to design heat treatment conditions of dual-phase steel and to determine their influence on the structure and mechanical properties of steel.

Design/methodology/approach: The heat treatment of the C-Mn steel in order to obtain a dual-phase ferritic – martensitic structure of desirable phase fractions was realized. To investigate the influence of heat treatment parameters on the structure light and transmission electron microscopy methods were used. Mechanical properties were determined by means of tensile test. A strain hardening exponent as a function of true strain was evaluated, too.

Findings: It was found that an initial structure influences essentially the morphology of martensite in an obtained dual-phase structure. It can occur as a network, fine fibres or islands in a ferritic matrix of high dislocation density in the vicinity of diffusionless transformation products of austenite. The best combination of strength and ductile properties has a steel with the martensite in a form of fine fibres.

Research limitations/implications: Investigations concerning using the thermomechanical treatment to obtain a ferritic – martensitic structure of steel are required.

Practical implications: The established heat treatment conditions can be useful for manufacturing dualphase structure sheets characterized by high strength and ductile properties as well as a good suitability for metalforming operations.

Originality/value: The relationship between the initial structure and martensite morphology in dual-phase steels was specified.

Keywords: Heat treatment; DP-type steel; Martensite; Mechanical properties

1. Introduction

The important factors taking into account by the automotive industry in a manufacturing process of modern cars are: the high ratio of material strength to its density, reduced fuel consumption, safety improvement and limitation of the harmful exhaust gases. It can be achieved by an optimization of well known materials and using new groups of materials with the good formability. The growing significance have metallic materials with a high value of strain hardening exponent and absorbing the large amount of the energy under conditions of high strain rate. The microalloyed structural steels are an example of materials fulfilling requirements of the automotive industry. Their application together with suitable metallurgical technologies enable to manufacture products with the fine-grained structure of transformation products of plastically deformed undercooled austenite [1÷3].

A variety of produced vehicles decides about the necessity of manufacturing weldable plates and sheets, characterized by the various tensile strength, formability and work hardening depending on the structure. In the modern automotive industry the hot-rolled plates of microalloyed steels are often used. They are manufactured in integrated lines connecting the continuous casting, rolling and accelerated cooling from a finishing rolling temperature. Depending on the specific application and localization of an element in the structure of a vehicle different steels are selected. They are characterized by the various ratio of strength to ductility and ability to energy absorbing during crash events. For elements subjected to deep drawing cold-rold steel sheets of IF-type (Interstitial Free) or BH-type (Bake Hardening) structure can be used [4-7]. Reinforced elements can be made of steels with a multiphase structure. They are characterized by a good strength-ductility balance compared to both conventional and microalloyed steels. The DP-type (Dual Phase) steels have a structure of a ferritic matrix with islands of martensite [8, 9] and TRIP-aided (Transformation Induced Plasticity) steels contain ferrite, bainite and some fraction of retained austenite transforming to martensite during strain-induced martensitic transformation [10-13]. The TRIP-effect can be used in highmanganese austenitic steels, too. They are intensively strengthened by the mechanical twinning occurring during technological deformation of elements [14, 15]. Elements subjected to a risk of impact or absorbing the high values of energy should be produced of CP-type (Complex Phase) steels containing ferrite, bainite, martensite, retained austenite and dispersive particles of carbides and nitrides [2, 11].



Fig. 1. Schematic drawing of undercooled austenite transformations of DP-, TRIP-, CP- and TMS-type steels thermo-mechanically processed and underhardened or isothermally quenched from a temperature slightly higher than A_{c1} of the steel

The best opportunities of the application for different elements of cars have the steels with a ferritic – martensitic structure. Their potential share in a steel structure of a car can reach even over 50% [16]. The dual-phase steels are low-carbon steels containing manganese, silicon and microadditions with a

high chemical affinity to carbon and nitrogen. Sheets of dual phase steels are characterized by YS_{0.2}~300÷500MPa, UTS~500÷800MPa, UEI to 26% and TEI to 30%, dependent on the chemical composition and martensite fraction, located in a ferritic matrix [1, 2, 17]. Sheets with a thickness below 2 mm are produced by underhardening after the cold-rolling [18-21]. During an intercritical annealing the recrystallization of ferrite and forming of austenite are proceeded. After annealing the steel is water-quenched. The austenite with the share and carbon concentration dependent on a holding temperature transforms into martensite. A more energy-saving technological route includes hot-rolling in the austenite range [22-24] or two-phase region with a direct controlled cooling from a finishing rolling temperature [25, 26]. The indispensable condition to manufacture sheets of desirable dual-phase structure, and especially with a sufficient ferrite fraction is to adjust quenching conditions to the kinetics of undercooled austenite transformations. It can be achieved by a selection of the chemical composition of steel, ensuring a leftdisplaced region of the $\gamma \rightarrow \alpha$ transformation, while pearlitic and bainitic transformations in the opposite direction (Fig. 1). The displacement of the $\gamma \rightarrow \alpha$ transformation on the left proceeds with increasing concentration of Si, Al and P in the steel [2, 11, 27].

The aim of the paper is to design various routes of a heat treatment in order to obtain a DP-type structure with required fractions of ferrite and martensite and optimum mechanical properties of the investigated low-carbon steel.

2. Experimental procedure

2.1. Material and heat treatment

The chemical composition of the investigated low-carbon structural steel is given in Table 1. The steel contains increased concentrations of manganese, silicon, molybdenum, titanium and a boron microaddition to improve the hardenability of steel and a small concentration of impurities. After continuous casting the slabs of a section 100x100mm were hot-rolled and then forged for rods with a section of 24x24mm.

Table 1.

Chemical composition of the investigated steel

Mass contents in %											
С	Mn	Si	Ni	Cr	Mo	Ti	Р	S	В	Al	Ν
0.09	1.50	0.26	0.07	0.06	0.14	0.113	0.014	0.009	0.003	0.029	00112

The established heat treatment conditions are schematically shown in Fig. 2. In order to properly design parameters of the heat treatment, the knowledge of the characteristic temperatures of the investigated steel is needed. The A_{c3} and A_{c1} temperatures calculated on the basis of the chemical composition of the investigated steel [28] equal 865°C and 714°C – respectively. The M_s temperature for the steel equals 453°C. However, it should be noted that both during the isothermal annealing and the $\gamma \rightarrow \alpha$ transformation the carbon concentration in the γ phase is increased. It improves the hardenability of the steel and decreases the M_s temperature for the austenite, calculated from Andrews' relationship [28]:

$$M_{s} = 539 - 423 \cdot C_{v} - 30 \cdot Mn - 17.7 \cdot Ni - 12.1 \cdot Cr - 7.5 \cdot Mo \quad (1)$$

where: Mn, Ni, Cr, Mo – mass contents of the elements in the investigated steel, C_{γ} – carbon concentration in the austenite.

The specimens subjected to the heat treatment routes shown in Fig. 2 have various initial structures. In two of three cases the steel was annealed in a $\gamma+\alpha$ range and water quenched. The soaking temperature 36°C higher than A_{c1} of the steel was selected on the basis of initial investigations concerning the influence of austenitizing temperature on a martensite fraction of the quenched steel. The authors of the works [18, 19] suggest, that DP-type steels obtain the best mechanical properties for the α ' fraction from 20 to 25%. In the third variant the specimens were initially air cooled for 45s to a temperature of 750°C and then water quenched. The cooling time in air of the specimens was determined experimentally based on thermocouple measurements.



Fig. 2. Schematic representation of the heat treatment of DP-type steel according to routes I, II and III

2.2. Methodology

The specimens for structure investigations were conventionally prepared and etched using nital and a saturated aqueous solution of the picric acid with the CuCl₂ addition. The Leica MEF4a light microscope was used to the observations of obtained structures after the heat treatment and to evaluate fractions of ferrite and martensite using the automatic image analyzer. Thin foils were investigated in the JEM-200CX transmission electron microscope at an accelerating voltage of 120kV. Thin foils destinated to transmission electron microscopy investigations were prepared by cutting the disks of 3mm in diameter and a thickness of 0.4mm and grinding to a thickness of about 0.08mm. In a final stage of foil preparation the disks were electrolytically polished by the use of the Struers Tenupol 5 polishing machine at the voltage of 35V and current intensity of 350mA. The solution of the 12°C temperature consisting of 900ml of acetic acid and 100ml of perchloric acid was used.

The specimens with a diameter of 6mm and a gauge length of 30mm were subjected to the tensile test using the Zwick Z/100 machine. Strain hardening of the steel on the basis of true strain exponent n was evaluated. The exponent changes with an increase in the amount of plastic deformation can be described using Ludwik-Hollomon's equation [29]:

$$\sigma = k\epsilon^n \tag{2}$$

where: σ - true stress, k – constant, ϵ - true strain in a range of uniform elongation of the samples.

3. Results and discussion

A various initial structure of the investigated steel aimed at the determination its influence on a morphology of structural constituents in a final dual phase structure. The initial structures of the specimens subjected to the heat treatment according to three routes in Fig. 2 were: ferrite with some fraction of pearlite, low-carbon martensite and primary austenite with a grain size of about 12µm (Fig. 3) - respectively for the routes I, II and III. It was found that the martensite morphology exhibits substantial differences dependent on the initial structure of the steel. The specimens heat-treated according to the route I have a ferritic matrix and the martensite is located on boundaries of the α phase as an irregular envelope (Fig. 4). The location of martensite is strongly dependent on a distribution of the austenite formed due to a carbon enrichment of the boundary-zones of ferrite connected with a decomposition of pearlite grains. During annealing of the investigated steel at a temperature of 750°C the privileged diffusion of carbon on the boundaries of the α phase is occurred. The underhardening from an increasing annealing temperature leads to the increase of martensite volume fraction, keeping a network distribution of this phase on grain boundaries of the α phase (Fig. 5).

The martensite fraction in the steel quenched from a temperature of 750°C equals about 22% and increases to 40% after quenching from a temperature of 810°C. A result of the increase of martensite fraction together with increasing the underhardening temperature is unfavourable decreasing the carbon concentration in austenite and increasing the M_s temperature of this phase. For 22% volume fraction of martensite, the carbon concentration in the γ phase equals about 0.47% and the M_s temperature of this phase about 290°C. The austenite enriched in carbon increases the hardenability of steel, too. The additional influence in improving the hardenability of steel have alloying elements dissolved in the austenite, i.e. manganese, silicon, molybdenum, nickel, chromium and especially a boron microaddition. Its positive influence is guaranteed due to titanium fixed the nitrogen entirely. It is a very important element of manufacturing the DP-type steels. The underrate hardenability of austenite and the smaller precipitation hardening by martensite islands are obtained after the quenching of steel from a temperature of 810°C. The carbon concentration in the austenite is about 0.2% and Ms increases to 390°C, for this temperature.

The different morphology of the structure have the specimens quenched twice, i.e. from a temperature of 910°C and 750°C. In this case, during heating the steel to an underhardening temperature of 750°C, the nucleation of austenite mainly occurs on the boundaries of martensite laths formed after primary quenching of the investigated steel from a temperature of 910°C. Here, the martensite fraction in a form of the network on grain boundaries of the α phase is not large. The predominated martensite fraction occurs mainly as thin fibres located in a surroundings of grain boundaries and inside ferrite grains (Fig. 6). Moreover, in a surroundings of martensite, especially at a boundary zone of large grains of the α phase, small grains of the recrystallized ferrite can be identified. They are a result of plastic deformation connected with volume changes accompaning the martensite transformation.



Fig. 3. Boundaries of primary austenite grains of the steel quenched from a temperature of $910^{\circ}C$

Fig. 4. Ferritic – martensitic structure of the steel quenched from a temperature of $750^{\circ}C$

Fig. 5. Ferritic – martensitic structure of the steel quenched from a temperature of 810°C

Fig. 6. Ferritic – martensitic structure of the steel twice quenched from the temperatures of 910°C and 750°C

Fig. 7. Ferritic – martensitic structure of the steel quenched from a temperature of 750° C after previous air cooling for 45s from 910° C

Apart from the morphology differences after the heat treatment of steel according to the routes I and II the difference in a grain size of ferrite is observed (Table 2). The more fine-grained structure has a steel heat-treated according to the path II. The average grain size of the α phase in this case is about 7µm, while for the route I is about 3µm larger. This is due to the increased number of places convenient for the nucleation of ferrite and also a partially course of the recrystallization of the specimens with an initial structure of martensite. The martensite fraction after underhardening for both variants of the heat treatment is near the same. For the route I equals about 22% and for the route II is about 2% higher. Such a fraction of martensite and ferrite being a complement to 100% has the influence on the enrichment of austenite in carbon and also causes lowering the M_s temperature of austenite to Ms= 286°C and 297°C, respectively for the routes I and II.

Fig. 8. Ferritic – martensitic structure of the steel quenched from a temperature of 785°C after previous air cooling for 35s from 910°C

A different type of the structure specimens was found after a realization of the heat treatment according to the route III (Fig. 7). In this case the specimens after austenitizing at a temperature higher than A_{c3} for a steel were initially air cooled for 45s or 35s to a temperature of 750°C and 785°C - respectively, and next water quenched in order to transform the austenite fraction of various carbon concentration to martensite. This variant of the heat treatment can simulate cooling after rolling, e.g. during thermomechanical processing. As distinct from another heat treatment routes, the martensite does not occur as a network or thin fibres but in a form of islands of diversified size. They are located on grain boundaries of the α phase. The optimum fraction of martensite averaging 20% occurs after air cooling of the specimens for 45s (Fig. 7). A reduction of the cooling time to 35s increases the martensite fraction to 32% (Fig. 8). The grain size of the α phase is about 8.5µm and is average between the specimens cooled according to the routes I and II.

Table 2.

Structural parameters of the obtained structures

	Ferrite fraction.	Martensite fraction,	C content	Msγ, °C	Ferrite grain
Route I	78.3	21.7	0.450	286	9.7
Route II	76.1	23.9	0.412	297	7.0
Route III	79.1	20.9	0.500	265	8.5

The optimum cooling time of specimens in air equaling 45s (route III) was not selected only based on the comparable martensite fraction to another variants. A reduction of the cooling time increases a fraction of diffusionless products of undercooled austenite, but at the lower carbon enrichment of the austenite. It causes lowering the hardenability of steel (austenite depleted in carbon) and can lead to a transformation of the part of austenite to undesirable bainite.

Fig. 9. A part of the martensitic island in the ferrite matrix of high dislocation density

Fig. 10. Martensitic island on the boundaries of three ferrite grains of high dislocation density

In order to investigate in detail the structure of DP-type steels the investigations of the thin foil structure by the use of the transmission electron microscopy were carried out. Figures 9 and 10 are transmission electron micrographs showing the structure of steel water quenched following austenitizing at a temperature of 750°C. According to the prediction, the martensite has a lath morphology and occurs as regular islands, surrounded by ferrite of high dislocation density (Fig. 9). The dislocation density decreases with increasing distance from the islands (Fig. 10). The increased dislocation density in ferrite is a result of increasing the volume of diffusionless products of austenite transformations for the carbon concentration in the γ phase about 0.47%. A lack of the presence of bainite in the structure of the steel proves the good hardenability of the steel. Apart from the enrichment of the austenite in carbon, a substantial effect on the increase of the hardenability of steel have Mn, Si, Cr, Mo and first of all boron

dissolved in the solid solution. A positive influence of boron is possible due to the effective shield by titanium, fixing the total nitrogen in the steel as TiN. It was also found that between martensite laths the films of retained austenite can locally occur (Fig. 11a). This phase is a desirable constituent of the structure of DP-type steels. Its transformation into martensite proceeding during technological forming of the elements can positively increase the strengthening of steel, like in case of the steels showing the TRIP effect (Transformation Induced Plasticity).

Fig. 11. Structure of lath martensite with the retained austenite located between martensite laths, a - light field, b - diffraction pattern

On the basis of the tensile test, it was found that the morphology of heat-treated DP-type specimens has a fundamental effect on mechanical properties of steel (Table 3). The comparison of ductile and strength properties of the investigated steel for used variants of the heat treatment is purposeful taking into account comparable fractions of martensite for all the steels (Table 2). The best connection of strength and ductile properties has the steel heat-treated according to the route II, i.e. quenched from a temperature of 750°C following primary quenching from a temperature of 910°C. For this variant of the heat treatment, the vield point is 516MPa, tensile strength 800MPa, total elongation about 20% and uniform elongation 16%. The steel quenched from a temperature of 750°C with an initial ferritic – pearlitic structure has lower values of the yield point and tensile strength by about 50MPa as well as total and uniform elongations by about 4%, i.e. TEI = 15% and UEI = 12%. The highest strength properties has the steel air cooled for 45s from a temperature higher than Ac3 to a temperature of 750°C. It leads to a transformation of about 79% austenite into ferrite and a residual fraction of the austenite enriched in carbon to C_{γ} = 0.5% into martensite. The yield point of the specimens cooled according to this route achieves 635MPa and tensile strength about 1000MPa. However, the high strength properties have the influence on ductile properties, which are slightly lower. They are comparable to the properties obtained for the specimens cooled according to the route I (TEl = 15%, UEl = 12%). The shortening of the time for a realization of the $\gamma \rightarrow \alpha$ transformation from 45s to 35s leads to an increase of the

martensite fraction to 32%. As a result of that, the strength properties ($YS_{0,2} = 690MPa$, UTS = 1180MPa) increase but ductile properties decrease.

Table 3.	
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Mechanical	properties	of the	investigated	DP-type steel
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	Value	UTS, MPa	YS, MPa	UEl, %	TEl, %	YS/ UTS	UTS · UEl, MPa · %
Route I	Mean	751	501	12.3	15.0	0.66	9282
	±	12	64	3.5	3.0	0.08	1202
Route II	Mean	798	516	16.0	19.3	0.65	12781
	±	14	54	1.1	1.2	0.06	1009
Route III	Mean	998	635	11.7	14.6	0.63	11625
	±	35	25	1.1	1.1	0.02	1180

The DP-type structure produced under investigated conditions of the heat treatment ensures a favourable value of the $YS_{0.2}/UTS$ ratio, equaling 0.58 to 0.67. It indicates an intensive course of strain hardening of steel after exceeding the yield point. The high strain strengthening of the steel caused by precipitation hardening of the matrix by martensite islands is characterized by a value of true strain exponent, described by the equation (2). The exponent value intensively risis initially. It is connected with the strong hardening of steel caused by martensite islands. The n value decreases after attaining a maximum for a value of true strain, dependent on a heat treatment route. The maximum value n~0.18 at a true strain equaling ε~0.06 shows the steel of DP-type structure produced after the heat treatment route I, i.e. after underhardening of the steel with a ferriticpearlitic initial structure (Fig. 12). The highest maximum value $n\sim0.25$ at a true strain $\varepsilon\sim0.08$ shows the steel with a DP-type structure produced in the second variant of the heat treatment, i.e. after twice quenching from the temperatures of 910°C and 750°C - respectively (Fig. 13). The comparable value n~0.23 achieves the steel obtained after the heat treatment consisting in the underhardening after the partial $\gamma \rightarrow \alpha$ transformation (Fig. 14). However, the exponent n increases only to a true strain about $\varepsilon \sim 0.05$.

The carried out investigations showed that mechanical properties of DP-type steels are dependent on many factors. They are: the ferrite and martensite morphology, properties of these phases and their volume fractions. The temperature-time conditions of the heat treatment were chosen in order to get a comparable fraction of martensite for all the variants. Taking it into account, the carbon supersaturation for all of the variants is comparable too. In this case, the dominant factors affecting properties of the steel are: the martensite morphology and ferrite properties. To determine the properties of the α phase, its behaviour during plastic deformation caused by the martensitic transformation has to be taken into account. The difference in the strength of ferrite is most probably a main reason of the difference in the strength of specimens with different structures. The network structure (route I) was produced by the martensitic transformation of the austenite located along grain boundaries of coarse-grained ferrite. The limitation of the transformation under these conditions is much weaker. It results in much smaller density of dislocations in the α phase and the lowest strength properties.

Fig. 12. True stress - true strain curve and the work hardening exponent n as a function of true strain of the steel heat-treated according to the route I

Fig. 13. True stress - true strain curve and the work hardening exponent n as a function of true strain of the steel heat-treated according to the route II

Fig. 14. True stress - true strain curve and the work hardening exponent n as a function of true strain of the steel heat-treated according to the route III

Authors of the works [18, 19] suggest, that the deformation of ferrite in the DP-type structure with the martensite in a form of fine fibres is much larger. There is a dislocation substructure in ferrite, increasing strength properties of this phase. Additionally, a factor deciding about the highest strength properties of the specimens heat-treated according to the routes I and III is a finegrained structure of ferrite. The grain size of this phase is about 3μ m (route II) and 1.5μ m (route III) smaller in comparison to the ferrite grain size of specimens heat-treated according to the route I (Table 2).

Mechanical properties of steels with a various kind of DP-type structure indicate on their different suitability to metalforming operations, especially to sheet-metal forming. It results from a various value of the UTS \cdot UEl coefficient (Table 3) and a course of the work-hardening exponent n (Fig. 12-14). The highest drawability shows the steel heat-treated according to the route II. The steel produced in a variant of the heat treatment consisting in the underhardening after the partial $\gamma \rightarrow \alpha$ transformation is characterized by the high value of the hardening exponent but has the limited deformability (Fig. 14). It results in lowering the sheet-metal forming of the steel with this kind of DP-type structure.

4.Conclusions

The chemical composition of the investigated steel enables to manufacture the products with the ferritic - martensitic structure by underhardening from the $\gamma+\alpha$ range. The various initial structure influences a morphology of martensite in a final DP-type structure of the heat-treated steel. After underhardening of the steel with a ferritic - pearlitic initial structure from a temperature of 750°C the martensite in a form of the network surrounding ferrite grains was obtained. Both twice quenching of the steel from the temperatures 910°C and 750°C and quenching after the partial $\gamma \rightarrow \alpha$ transformation results in a various shape of martensite. It was found, that in the first case the martensite occurs as fine fibres and in the second one as islands in a matrix of the α phase. The used conditions of the heat treatment led in obtaining the DP-type steels of comparable fractions of ferrite and martensite for all of the variants. The obtained martensite has a similar supersaturation in carbon. The optimum fraction of the martensite is from 21% to 24%, and a grain size of the α phase equals from 7µm to 10µm. The most fine-grained structure has the steel heat-treated according to the route II.

The various structure of heat-treated specimens influences substantially the mechanical properties and formability of the steel. The best strength-ductility balance has the steel after underhardening from a temperature of 750°C with an initial lowcarbon martensite structure. The steel shows a high value of the UTS UEl coefficient and a beneficial course of the workhardening exponent n. However, the heat treatment consisting of the underhardening following quenching from a temperature of the austenite stability is not an energy-saving solution. The highest economy and possibilities of forming mechanical properties of the products are connected with the steel heat-treated according to the route III. This steel can obtain the tensile strength from 1000MPa to 1200MPa dependent on various fractions of martensite and ferrite. It is predicted, that cooling of the steel in case of the thermomechanical processing after finishing of hotrolling should contribute to an additional increase of mechanical properties of the products with a DP-type structure.

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