INFLUENCE OF THE LABORATORY EQUIPMENT ON RESEARCH RESULTS ON A LOW MANGANESE DUAL-PHASE STEEL

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Abstract: *This article presents the influence of research equipments used in studies conducted at the University "Stefan cel Mare" from Suceava to characterize a dual-phase steel with 0.094% C and 0.53% Mn (Si, Cr, Ni, Mo, Al, Cu, P and S below 0.1%). The research was conducted in two stages, namely: the study of the influence of intercritical quenching on the structure and mechanical properties (2014 - 2015) and the study of the influence of structural succession on the ferrite-martensite structure and mechanical characteristics (2019 - 2020). Spectrophotometric, metallographic analyses and mechanical tests were performed with different equipments from one research stage to another.*

Keywords: *dual-phase steel, intercritical quenching, ferrite, martensite, mechanical properties.*

1. Introduction

Due to the superior mechanical characteristics, dual-phase steels are used in an increasing percentage in the modern mechanical industry. These materials have the structure formed by a soft and tenacious ferrite matrix in which martensite (10 - 35%) and a small amount of residual austenite (1 - 2%) are homogeneously dispersed. They generally have a carbon content of less than 0.12%, a manganese content of 1.0% to 3.5%, and elements such as V, Cr, Mo, Si, Nb, Ti are found. in the chemical composition in proportions below 1.0%; in recent years, dual-phase steels have been made in which the manganese content was below 1% (0,5 - 1,0 % Mn). The main method of producing these steels is quenching from temperatures in the range ($\alpha + \gamma$), called intercritical quencing, the structure resulting from the application of this method being influenced by both the chemical composition of the steel and the heat treatment technology applied. At the same time, the mechanical properties depend in a decisive way on the quantitative ratio and the morphology of the structural components that arise as a result of the thermal processing process, [Dulucheanu et al., 2011; Rashid, 1981; Golovanenko and Fonshteyn, 1986; Dulucheanu et al., 2015; Dulucheanu et al., 2019].

2. Experimental details

The chemical composition of the alloy under investigation (Table 1) was determined using POLYVAC 2000 spectrophotometers. (Hilger Analytical Limited, United Kingdom) in 2014 and FOUNDRY-MASTER Xpert (Oxford Instruments Analytical GmbH, Germany) in 2019, [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014; Besleaga, 2014; Grosu, 2019; Dulucheanu et al., 2020; Bazga, 2020]. Even if the two spectrophotometers are part of different generations (2000 year for POLYVAC 2000 and 2015 for FOUNDRY-MASTER Xpert), the results of the analyses were very close; only small differences to the third or fourth decimal to some accompanying elements.

Table 1: Chemical composition of the studied alloy.

Chemical elements, [%]				
C	Mn	Si	Cr	Ni
0.094	0.53	0.085	0.029	0.042
Mo	Al	Cu	P	S
0.005	0.003	0.065	0.003	0.004

The research was carried out in two stages, namely:

- study of the influence of heating temperature (quenching) in the intercritical range (Ac_1 - Ac_3) on the ferrite-martensite structure and mechanical properties (2014 - 2015);

- the study of the influence of structural succession on the ferrite-martensite structure and mechanical characteristics (2019 - 2020).

For the design and development of a technology for the production of a dual-phase steel it is necessary to know the temperatures of the critical points Ac_1 and Ac_3. During the studies carried out at the University "Stefan cel Mare" of Suceava until 2015, the mathematical model elaborated by G. Kaszatkin and B. Vinokur was used to determine the two critical points; after 2015, the temperatures of these critical points were determined by dilatometric analyzes performed with a DIL 402 Expedis-SUPREME Dilatometer (Netzsch Gerätebau GmbH, Germany), [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014; Besleaga, 2014; Grosu, 2019; Dulucheanu et al., 2020; Bazga, 2020]. The values obtained were:

- by calculation (with mathematical model): Ac_1 = 719.97 ˚C, Ac_3 = 869.40 ˚C;

- by dilatometric analyses: Ac_1 = 725.50 ˚C, Ac_3 = 900.40 ˚C.

Heating to intercritical quenching was performed in electric laboratory furnaces, and rapid cooling was done in water with temperature (T_{water}) of 20 ˚C. If in the studies from 2014 to 2015 artisanal cooling tanks were used without a precise and efficient control of the temperature of the cooling environment, in those from 2019 - 2020 was used LBS 2 bath (FALC INTRUMENTS S.R.L., Italy). It has a capacity of 22.5 liters and ensures the regulation of the temperature of the cooling medium between 20 and 80 ˚C; in addition, it allows the activation of the ultrasonic cooling medium with the frequency of 40 kHz or 59 kHz, [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014; Besleaga, 2014; Grosu, 2019; Dulucheanu et al., 2020; Bazga, 2020].

During the research stage from 2014 - 2015, the metallographic analyses were performed with an Optech optical metallographic microscope, model IM/IMT (Exacta + Optech GmbH, Germany), equipped with a digital camera, images processing being made with the Optika Vision Pro Software, and in the second stage 2019 - 2020, with a LEXT OLS4100 Laser Microscope (Olympus Corporation, Japan), helped by specialized software (OLYMPUS Stream MOTION Image Analysis Software) [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014; Besleaga, 2014; Grosu, 2019; Dulucheanu et al., 2020; Bazga, 2020]. The ferrite-pearlite and martensite structures were highlighted with 2% nital, and the ferrite-martensite ones with the following metallographic etchant: picric acid 4 % solution in alcohol (etching time - 60 seconds) and then nital 2% (etching time - 5 seconds), [Burikova and Rosenberg, 2009].

To determine the mechanical properties of dual-phase steel (ultimate tensile strength and total elongation) were used, in 2014 - 2015, a machine for tensile testing of sheets (modified for cylindrical specimens), made by teachers and students from Faculty of Mechanical Engineering, Mechatronics and Management of the University "Stefan cel Mare" of Suceava, and in 2019 - 2020, a QUASAR 600 universal testing machine (Cesare GALDABINI SpA, Italy). Tensile tests were supplemented with Vickers hardness tests which were performed with hardness testers: CV-700, CV Instruments (2014 – 2015) and DuraScan 70, Emco-Test Prüfmaschinen GmbH (2019 – 2020), [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014;

Besleaga, 2014; Grosu, 2019; Dulucheanu et al., 2020; Bazga, 2020].

3. Results and discussions

3.1. Influence of quenching temperature on ferrite-martensite structure and mechanical properties (2014 - 2015)

The steel was subjected to intercritical quenching with heating at 750, 770, 790, 810 and 830 ˚C (temperatures between critical points Ac_1 and Ac_3, calculated with the mathematical model achieved by G. Kaszatkin and B. Vinokur), maintaining these temperatures for 30 minutes and cooling in water (T_{water} = 20 ˚C). Determination of the influence of quenching temperature (T_Q) on the structure was made on samples with a diameter of 5 mm (10 samples for each quenching temperature) on which metallographic analyses were performed, the quantitative determinations of the metallographic constituents being made on micrographs by the points count method (five micrographs for each sample). The results obtained are presented in Table 2, [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014; Besleaga, 2014].

Table 2: Results of metallographic analyses (average values).

T_Q [˚C]	750	770	790	810	830
V_M [%]	19.45	21.06	26.68	32.35	41.73
V_F [%]	80.55	78.94	73.32	67.65	58.24

Note: V_M – volume fraction of martensite; V_F – volume fraction of ferrite.

Tensile tests were performed on cylindrical specimens with a diameter of 5 mm and a initial length between markers of 25 mm (10 specimens for each quenching temperature). With the data obtained from these tests the ultimate tensile strength and total elongation were determined. In addition, Vickers hardness was determined (with CV-700 hardness tester) from samples used in metallographic analyses. The results obtained are shown in Table 3, [Dulucheanu et al., 2015; Dulucheanu, Bancescu et al., 2015; Munteanu, 2014; Besleaga, 2014].

Table 3: Mechanical properties of the dual-phase steel (average values).

T_Q [˚C]	V_M [%]	R_m [MPa]	A_5 [%]	HV
750	19.45	591	19.15	159
770	21.06	609	18.12	165
790	26.68	631	15.83	189
810	32.35	684	13.84	197
830	41.73	738	11.02	264

Note: R_m – ultimate tensile strength; A_5 – total elongation; HV –Vickers hardness.

3.2. The influence of structural succession on the ferrite-martensite structure and mechanical characteristics (2019 - 2020)

Samples made of the studied steel were subjected to the following heat treatments: full annealing (noted FA – heating to 950 ˚C for 60 minutes and cooling in furnace), normalizing (noted N – heating to 950 ˚C for 60 minutes and cooling in air), subcritical annealing (noted SA – heating to 650 ˚C for 120 minutes and cooling in furnace), full quenching (noted FQ – heating to 950 ˚C for 30 minutes and cooling in water), intercritical quenching (noted IQ – heating to 790 ˚C for 30 minutes and cooling in water), with which the following cycles of heat treatments were made: FA + IQ, N + IQ, N + SA + IQ, FQ + IQ, [Dulucheanu et al., 2020; Grosu, 2019; Bazga, 2020].

On the metallographic samples (with a diameter of 5 mm) that were subjected to the heat treatments described above, qualitative and quantitative metallographic analyzes were performed; for each heat treatment seven metallographic samples were made, and for each sample five micrographs were made; three quantitative determinations of the constituents were performed on each micrograph (OLYMPUS Stream MOTION Image Analysis Software), the results obtained are presented in Tables 4 and 5, [Dulucheanu et al., 2020; Grosu, 2019; Bazga, 2020].

The mechanical properties (ultimate tensile strength and total elongation) were determined by tensile tests performed on cylindrical specimens with a diameter of 5 mm and an initial length between markers of 25 mm (in the calibrated portion); batches of 15 specimens were tested for each cycle of heat treatments. Tensile tests (with the QUASAR 600 universal testing machine) and data processing (with GraphWork 6 software) were made in accordance with the provisions of the standard EN ISO 6892-1:2015, "Metallic material - Tensile testing - Part 1: Method of test at room temperature". In addition, on the samples from the metallographic analyses, the Vickers hardness was determined (with DuraScan 70 hardness tester). The results obtained are presented in Table 6, [Dulucheanu et al., 2020; Grosu, 2019; Bazga, 2020].

Table 4: Results of metallographic analyses (for the samples to which intercritical quenching was not applied).

Volume fraction of constituents [%]	Heat treatments			
	FA	N	N + SA	FQ
V_F	92.45	87.10	76.74	-
V_P	7.55	12.90	13.26	-

Note: V_F - volume fraction of ferrite; V_P - volume fraction of pearlite.

Table 5: Results of metallographic analyses (for samples to which intercritical quenching was also applied).

Volume fraction of constituents [%]	Cycles of heat treatments			
	FA + IQ	N + IQ	N + SA + IQ	FQ + IQ
V_M	19.75	23.49	17.44	26.16
V_F	80.25	76.51	82.56	73.84

Note: V_M - volume fraction of martensite; V_F - volume fraction of ferrite.

Table 6: Mechanical properties of the dual-phase steel (average values).

Mechanical property	Cycles of heat treatments			
	FA + IQ	N + IQ	N + SA + IQ	FQ + IQ
R_m [MPa]	552	570	556	580
A_5 [%]	22.26	21.13	20.30	20.18
HV	180	182	174	187

Note: R_m - ultimate tensile strength; A_5 - total elongation; HV - Vickers hardness

3.3. Comparison of the results obtained

The common element of the two research stages is represented by the following variant of intercritical quenching: T_Q = 790 °C, t_m = 30 minutes, cooling in water (T_{water} = 20 °C). In 2014 - 2015, the initial structure was that of delivery from the metallurgical company, i.e. a ferrite-pearlite structure, made by normalizing, and in 2019 - 2020, the initial structure was also ferrite-pearlite, obtained by a normalizing (T_i = 950 °C, t_m = 60 minutes, cooling in air).

The results established by metallographic analyses and mechanical tests performed on samples with ferrite-martensite structures (characteristic of dual-phase steels), are presented in Table 7. [Bazga, 2020].

Table 7: Results and differences obtained (average values).

Measured characteristic	Research stage		Distinction	
	2014 - 2015	2019 - 2020	in measurement units	in percentages
V_M, [%]	26.68	23.49	– 3.19 %	– 11.95 %
R_m, [MPa]	631	570	– 61 MPa	– 9.67 %
A_5, [%]	15,83	21,13	+ 5.3 %	+ 33,.48 %
HV	189	182	– 7 HV	– 3.70 %

Analyzing the obtained results, we find the following [Bazga, 2020]:

1. The LEXT OLS4100 Laser Microscope allowed higher quality micrographs to be obtained than those obtained with the Optech microscope, and OLYMPUS Stream MOTION Image Analysis Software allowed a much more accurate metallographic analyses (Figures 1 and 2).

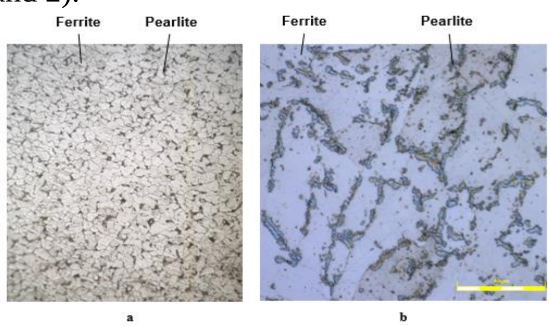

Figure 1: Ferrite-pearlite structures
a) 2014 - 2015; b) 2019 - 2020.

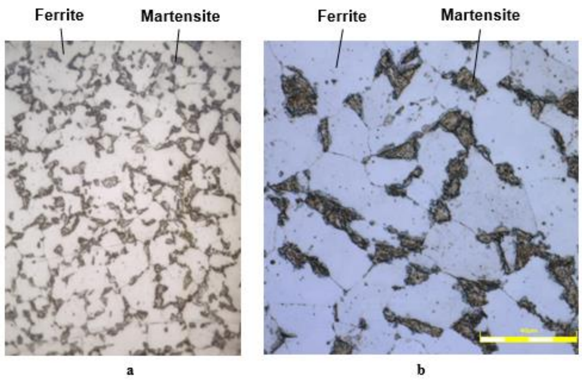

Figure 2: Ferrite-martensite structures
a) 2014 - 2015; b) 2019 - 2020

The volume fraction of martensite (V_M), the constituent with major influence on the properties of a dual-phase steel, determined on the samples from 2019 - 2020 (with OLYMPUS Stream MOTION Image Analysis Software) was smaller with 11.95% than that determined by the points count method for the samples in 2014 - 2015 (Figure 3.).

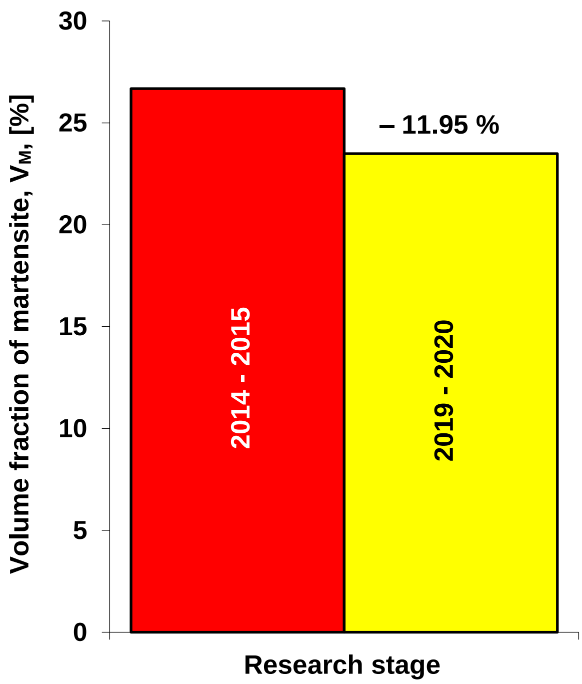

Figure 3: The volume fraction of martensite (V_M) in the structure of dual-phase steel according to the research stage.

2. The results of the tensile tests carried out with the QUASAR 600 universal testing machine (2019 - 2020) were different from those determined on the sheet metal testing machine made at the University "Stefan cel Mare" of Suceava and modified for cylindrical specimens. Thus, the ultimate tensile strength (R_m) was 61 MPa the smallest, i.e. with 9.67%; in 2014 - 2015 an average value of it was obtained R_m of 631 MPa, and in 2019 - 2020, of 570 MPa (Figure 4). A much larger difference resulted in the case of total elongation (A_5); its average value A_5 obtained in 2019 - 2020 was with 33.48% higher than that determined in 2014 – 2015 (Figure 5).

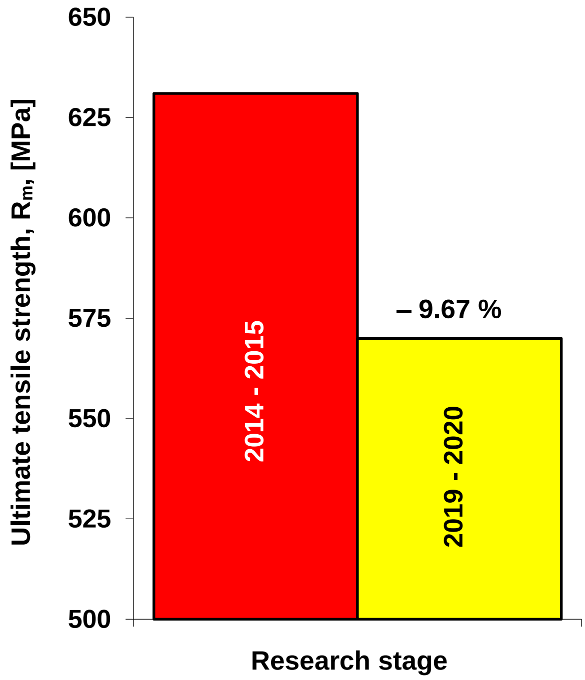

Figure 4: The ultimate tensile strength (R_m) of dual-phase steel depending on the research stage.

3. Because in both research stages close hardness testers were used as performance, the average Vickers hardness values were close, the differences between them being only seven Vickers units (7 HV), i.e. only 3.70% (Figure 6).

There is a correspondence between the hardness values and the ultimate tensile strength of the material, a correspondence which is presented in the literature and which is not valid in the case of austenitic steels and cold-formed plastics. The results obtained for the dual-phase steel studied by transforming the Vickers hardness values into ultimate tensile strength values and comparing them with the values obtained by the tensile test, are given in Table 8. [Bazga, 2020].

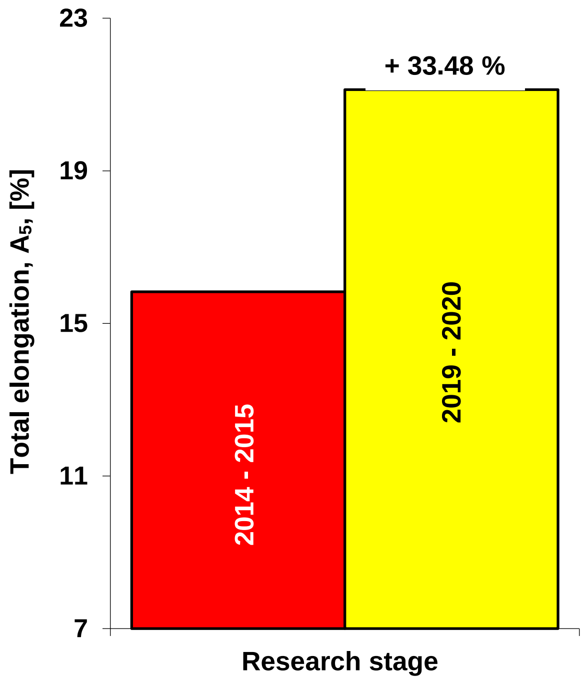

Figure 5: The total elongation (A_5) of dual-phase steel depending on the research stage.

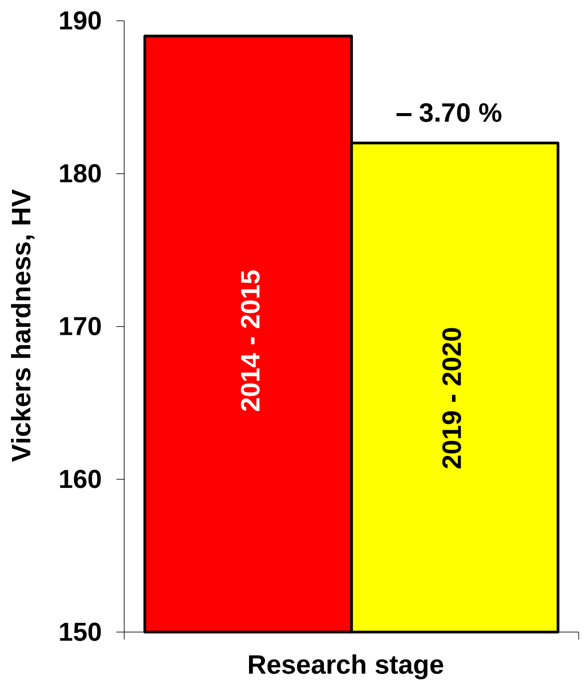

Figure 6: Vickers hardness (HV) of dual-phase steel according to the research stage.

Table 8: Correspondence between hardness and ultimate tensile strength.

Research stage	Vickers hardness	R_m (from Vickers hardness)	R_m (from tensile tests)	Distinction	
				in measurement units	in percentages
2014 - 2015	189 HV	597 MPa	631 MPa	– 34 MPa	– 5.38%
2019 - 2020	182 HV	576 MPa	572 MPa	+ 4 MPa	+ 1.01%

Table 8 shows that between the values from 2014 - 2015 of R_m there is a difference of -5.38% (- 34 MPa), while between the values from 2019 - 2020, the difference is only + 1.01% (+ 4 MPa).

4. Conclusions

- The temperatures of the critical points obtained by dilatometric analyses were higher by 5.53 ºC for Ac_1 and with 31.0 °C for Ac_3 compared to those determined using the mathematical model; intercritical temperature range (Ac_1 – Ac_3) established by dilatometric analyses was with 25.47 °C higher than that obtained by calculation (174.90 °C to 149.43 °C).
- The modern microscopic analysis equipment used in 2019 - 2020 led to different results than those obtained before 2015. Thus, in the 2019 - 2020 stage, the volume fraction of martensite (V_M) was 11.95% lower than in ferrite-martensite structures analyzed in the 2014 - 2015 stage (23.49% compared to 26.68%).
- The results of the tensile tests performed in 2019 - 2020 were also different from those of 2014 - 2015. The ultimate tensile strength (R_m) was lower by 9.67% (570 MPa compared to 631 MPa), and the total elongation (A_5) was higher by 33.48% (21.13% compared to 15.83%).
- Regarding the Vickers hardness (HV), a characteristic that was determined with hardness testers close in performance, the difference between the values obtained in the two research stages was only 3.70% (189 HV in 2014 - 2015 and 182 HV in 2019 - 2020).

Acknowledgements

This work was partially supported from contract no. 18 PFE/16.10.2018 funded by Ministry of Research and Innovation within Program 1 - Development of national research and development system, Subprogram 1.2 - Institutional Performance - RDI excellence funding projects, The infrastructure used for this work was partially supported from the projects "Integrated Center for research, development and innovation in Advanced Materials, Nanotechnologies, and Distributed Systems for fabrication and control (MANSiD)", Contract No. 671/2015.

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