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Influence of the initial structure on the critical points in solid-state phase transformation of some hypoeutectoid steels

C. Dulucheanu[1], T.L.Severin[1,*], A. Potorac[1], L. Irimescu[1], J. Javorova[2]

[1]*University "Stefan cel Mare" of Suceava, Faculty of Mechanical Engineering, Mechatronics and Management & Research Center MANSiD, 13 University Street, 720229 Suceava, Romania*
[2]*University of Chemical Technology and Metallurgy, Bulevard Sveti Kliment Ohridski 8, 1756 Studentski Kompleks, Sofia, Bulgaria*

This study allowed, by dilatometric analyses, both to highlight the solid state transformations that occurred during the continuous heating of three hypoeutectoid steels (with 0.087% C - 0.511% Mn, 0.101% C - 0.529% Mn and 0.093% C - 1.922% Mn), as well as to investigate the effect of the initial ferrite-pearlite structure on the critical points at which these transformations occurred. Dilatometric analyses were performed on specimens of steels with unmodified structures (as-delivered structures) and on specimens with structures obtained from full annealing (heating at 980 °C for 60 minutes and slow cooling in the furnace). These structures (with different grain size indices, mean diameters and mean area of grain) have influenced both the aspect of the dilatometric and the first derivative curves, as well as the values of the critical points in solid-state phase transformation.

Keywords: critical point, dilatometric analysis, structure, grain size index, dual-phase steel

INTRODUCTION

The behaviour of a steel in the basic operations of the various heat treatment technologies, the structures and the properties obtained therefrom, are influenced both by the homogeneity of the austenite grains and by their dimensions. The size of the austenitic grains formed by heating depends not only on the parameters of the heating regime (temperature, time), but also on the initial structure of the steel. According to the Fe-C equilibrium diagram, when heating a hypoeutectoid Fe-C binary alloy, results austenite (γ) through two transformations, namely, the pearlite (P) dissolution which represents an eutectoid transformation ($P \rightarrow \gamma$) and the transformation of proeutectoide ferrite (α) which is an allotropic transformation ($\alpha \rightarrow \gamma$); the temperatures at which these transformations take place are considered critical points, being noted as Ac_1, respectively Ac_3. In conditions of thermodynamic equilibrium, in the Fe-C binary alloys, the pearlite dissolution into austenite (eutectoid transformation) occurs at a constant temperature of 727 °C [1-5]. Commercial steels, unlike Fe-C binary alloys, are metallic materials which have in their composition also other chemical elements (such as impurities or alloying elements), elements which modify the position of the critical points; in addition, in the commercial steels, under industrial heating conditions (different from thermodynamic equilibrium), the pearlite dissolution into austenite (eutectoid transformation) no longer occurs at 727 °C (at constant temperature), but in a range of temperatures, namely, between Ac_1 temperature (pearlite dissolution start temperature) and pearlite dissolution finish temperature, denoted Ac_{fp}. The temperature Ac_{fp} indicates the beginning of ferrite and austenite coexistence range during heating (beginning of the allotropic transformation of ferrite into austenite, $\alpha \rightarrow \gamma$), range that stretches up to the temperature of the Ac_3 point, the determination of this range of coexistence of the ferrite and austenite (Ac_{fp} - Ac_3) being of great importance for the technologies of manufacturing dual-phase steels [1-7]. These steels have a low carbon content and a structure formed of a soft and ductile ferrite matrix in which are homogeneously dispersed, martensite (10 to 35 %) and a small amount of residual austenite (1 to 2 %); the main technology for producing such steels consists of the intercritical quenching. The structure obtained, for a given chemical composition, is the result of combined action of the technological parameters of intercritical heat treatment (heating temperature, cooling rate etc.), their influence on the structure of the material being directly reflected on its properties. Consequently, for designing and developing a dual-phase steel production technology, it is necessary to know both the temperatures of the critical points Ac_1, Ac_{fp} and Ac_3,

* To whom all correspondence should be sent:
severin.traian@usm.ro

as well as the influence of certain technological parameters (heating rate, grain size of ferrite and austenite etc.) on these points [4, 5, 8]. A quick and convenient method for determining the temperature of critical points in solid-state phase transformation when heating a steel lies in the use of mathematical models that take into account the chemical composition (equations obtained by statistical processing of experimental results), but these equations can generate large errors [1, 2, 4, 5]. A high precision in determining these temperatures is obtained by dilatometric analyses; modern dilatometers, connected to computerised systems, collect the signals of change in the length of a specimen as a function of temperature, plot a dilatometric curve, $\Delta L/L_o = f(T)$, calculate and generate its corresponding derivative, $d(\Delta L/L_o)/dT = f(T)$, and allow the identification of both critical points Ac_1 and Ac_3, as well as the pearlite dissolution finish temperature (the Ac_{fp} point) and therefore, the determination of the temperatures range (Ac_{fp} - Ac_3) [2, 7, 9, 10]. Unfortunately, the literature is relatively poor in information on the results of dilatometric analyses performed in order to identify the temperatures of the critical points in solid-state phase transformation of dual-phase steels or to establish the influence of some technological parameters (heating speed, initial structure, grain size etc.) on these critical temperatures.

This article describes some of the researches carried out at University "Stefan cel Mare" of Suceava, Romania, in order to obtain and characterise dual-phase steels; the results of the dilatometric analyses performed on specimens made from three of the alloys used in the research are presented. As a result of these researches, the temperatures of the critical points in solid-state phase transformation were identified and the influence of the initial structures on these temperatures was established.

EXPERIMENTAL DETAILS

The chemical compositions of the investigated three Fe-C alloys (denoted A_Steel, B_Steel and C_Steel in this article) were determined with a FOUNDRY-MASTER Xpert spectrometer (Oxford Instruments Analytical GmbH, Germany), and led to the next values (weight %):

- A_Steel: Fe, 0.087 C, 0.511 Mn, 0.091 Si, 0.0036 P, 0.0039 S, 0.029 Cr, 0.005 Mo, 0.049 Ni, 0.003 Al, 0.082 Cu, 0.003 V, 0.003 W;
- B_Steel: Fe, 0.101C, 0.529 Mn, 0.091 Si, 0.0032 P, 0.0037 S, 0.036 Cr, 0.005 Mo, 0.015 Ni, 0.003 Al, 0.015 Cu, 0.003 V, 0.003 W, 0.011 Pb;
- C_Steel: Fe, 0.093 C, 1.922 Mn, 0.065 Si, 0.0191 P, 0.0116 S, 0.111 Cr, 0.036 Mo, 0.092 Ni, 0.012 Al, 0.154 Cu.

The influence of the initial structure on the critical points in solid-state phase transformation was studied on:

- specimens with as-delivered structure, denoted D_A for A_Steel, D_B for B_Steel and D_C for C_Steel;
- specimens with structure obtained by full annealing, denoted FA_A for A_Steel, FA_B for B_Steel and FA_C for C_Steel.

The heat treatments (full annealing) were performed in a Nabertherm LT 40/11/P330 electrical laboratory furnace (Nabertherm GmbH, Germany) and consisted of heating to 980 °C (for 60 minutes), followed by cooling slowly, in furnace.

The metallographic analyses, which allowed the identification and characterization of the structures, were performed with a LEXT OLS4100 Laser Microscope (Olympus Corporation, Japan). The surfaces needed metallographic analyses (three samples for each structure and each steel) were obtained by processing with Hot Mounting Press OPAL 410 and Grinding/Polishing Machine SAPHIR 530 (ATM GmbH, Germany). The quantitative characterization of the structures was achieved by the planimetric method (in compliance with EN ISO 643:2003 [11]), with OLYMPUS Stream MOTION Image Analysis Software.

The dilatometric analyses, aimed to reveal the critical points in the solid-state phase transformation of the three steels, were made with a DIL 402 Expedis-SUPREME Dilatometer (NETZSCH Gerätebau GmbH, Germany), on cylindrical specimens with a diameter of 5 mm and a length of 25 mm, continuous heated in (30 ÷ 980) °C temperature range, with a heating rate of 3 °C/min, in nitrogen atmosphere (100 ml/min N_2) and with a load at the specimen of 200 mN; for each initial structure and each steel, three analyses were made (three specimens for each initial structure and each steel). Finally, the signals provided by the dilatometer were processed with NETZSCH Proteus® Software 7.1.0.

RESULTS AND DISCUSSION

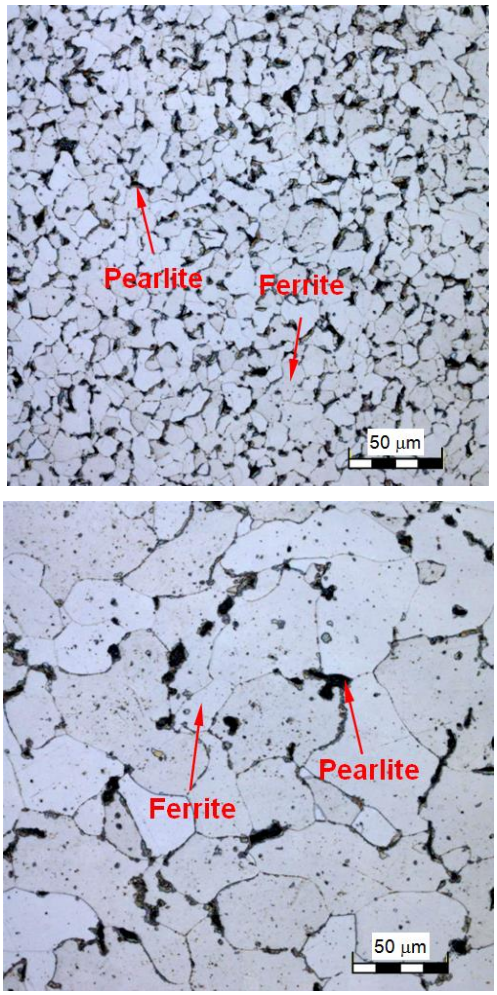
Micrographic determination of grain size

After highlighting the structures, five micrographs were achieved (in different regions) on

each metallographic sample (Figs.1-3); all the structures of the three steels were made of ferrite and pearlite (ferrite-pearlite structures). Finally, on each micrograph the grain size index (G), the mean diameter of grain (đ) and the mean area of grain (ā) were determined; for each parameter (G, đ and ā), three determinations were made. The results obtained (average values) are presented in Tab.1.

Table 1. Characterization of grain size

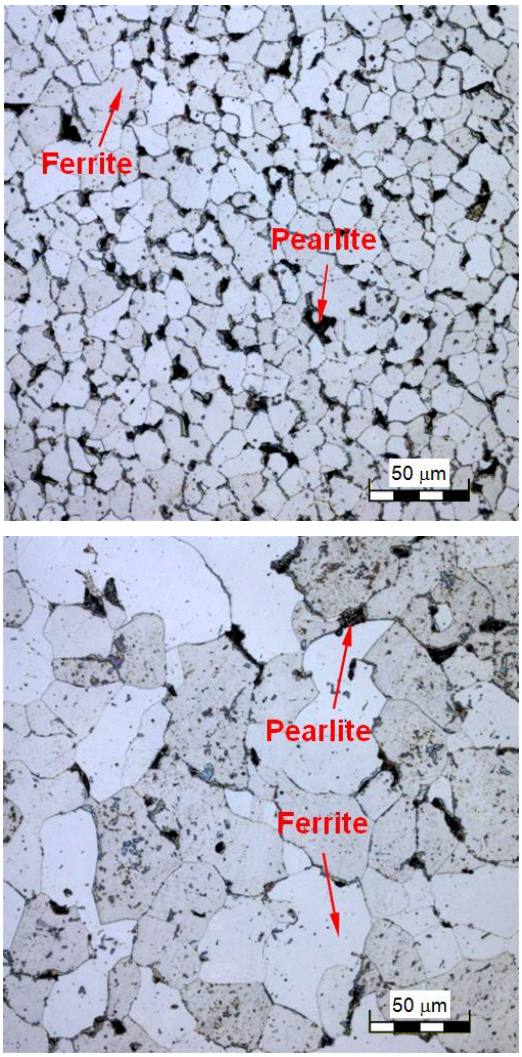
Steel	As-delivered structure		
	G	đ	ā
		μm	μm^2
A_Steel	10.16	10.49	109.44
B_Steel	9.77	12.06	143.87
C_Steel	8.69	17.62	304.43
Steel	Full annealing structure		
	G	đ	ā
		μm	μm^2
A_Steel	7.48	26.83	702.98
B_Steel	7.28	28.65	809.06
C_Steel	8.39	19.57	373.02



a

b

Fig.1. Structures of the A_Steel (etchant 2% Nital): a) D_A; b) FA_A



a

b

Fig.2. Structures of the B_Steel (etchant 2% Nital): a) D_B; b) FA_B

The data in Tab.1 shows that, for each steel, the full annealing structures had grains larger than those in the as-received structures, the biggest differences being highlighted at structures of the A_Steel and B_Steel; for example, the mean diameter of grain (đ) was 2.56 times greater for A_ Steel, 2.38 times for B_Steel and 1.11 times for C_Steel.

The grain size indice (G) diminished with 26.38% (from 10.16 for as-received structure to 7.48 for full annealing structure) for A_Steel, with 25.49% (from 9.77 for as-received structure to 7.28 for full annealing structure) for B_Steel and with only 3.45% (from 8.69 for as-received structure to 8.39 for full annealing structure) for C_Steel (Fig.4).

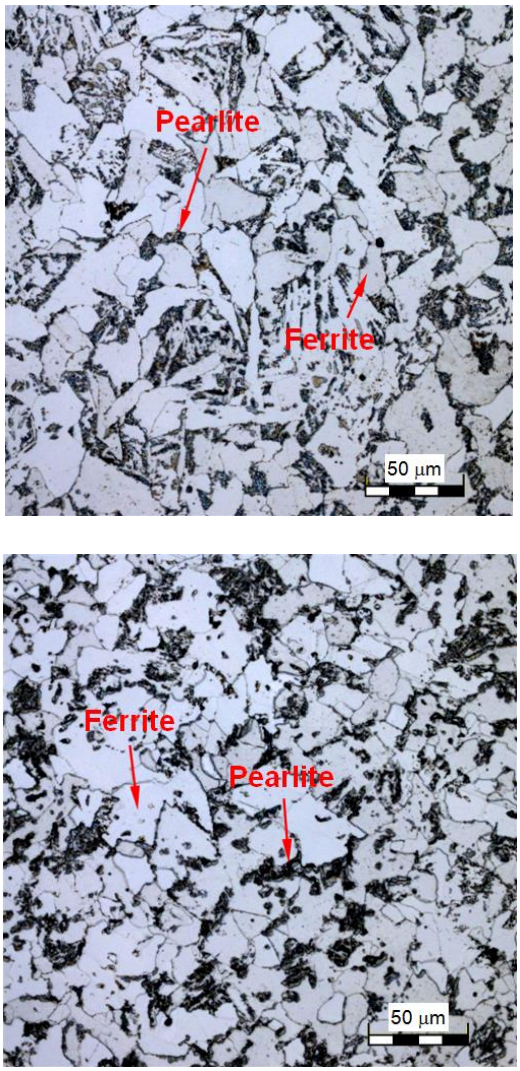


Fig.3. Structures of the C_Steel (etchant 2% Nital): a) D_C; b) FA_C

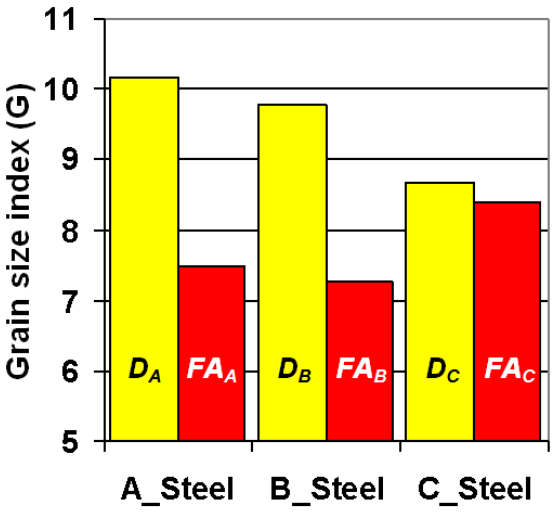


Fig.4. The grain size index (G) of the structures

Influence of the initial structure on the critical points in solid-state phase transformation

The signals of change in the length of the specimens as a function of temperature, collected during dilatometric analyses with the computerised systems of the DIL 402 Expedis-SUPREME Dilatometer, have led to the plotting of the dilatometric curves, as well as to the calculation and graphic representation of their derivatives (Figs.5-7).

Normally, no difference between the eutectoid transformation (the pearlite dissolution in austenite, (P $\rightarrow \gamma$) and the allotropic transformation of ferrite into austenite ($\alpha \rightarrow \gamma$) is detected on the continuous heating dilatometric curve obtained for a hypoeutectoid Fe-C binary alloy. However, all the dilatometric curves drawn for the specimens of the three steels with as-delivered structures (D_A, D_B and D_C) present an unusual anomaly at the onset of the austenitization; this obvious anomaly is a contraction associated to the pearlite dissolution (Figs.5.a, 6.a, 7a) [2-7, 9, 10, 12, 13].

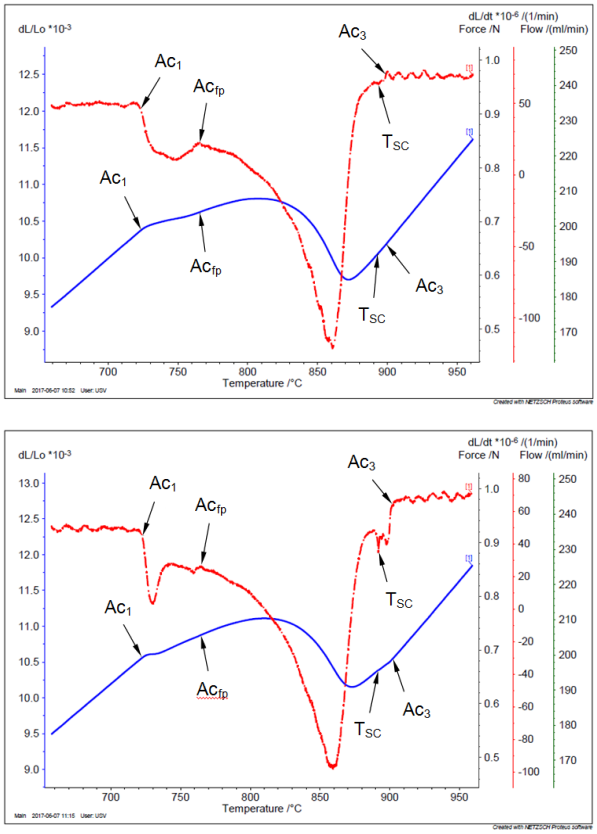


Fig.5. Dilatometric curve (continuous line) and its corresponding derivative (dashed line) of the A_Steel: a) D_A; b) FA_A

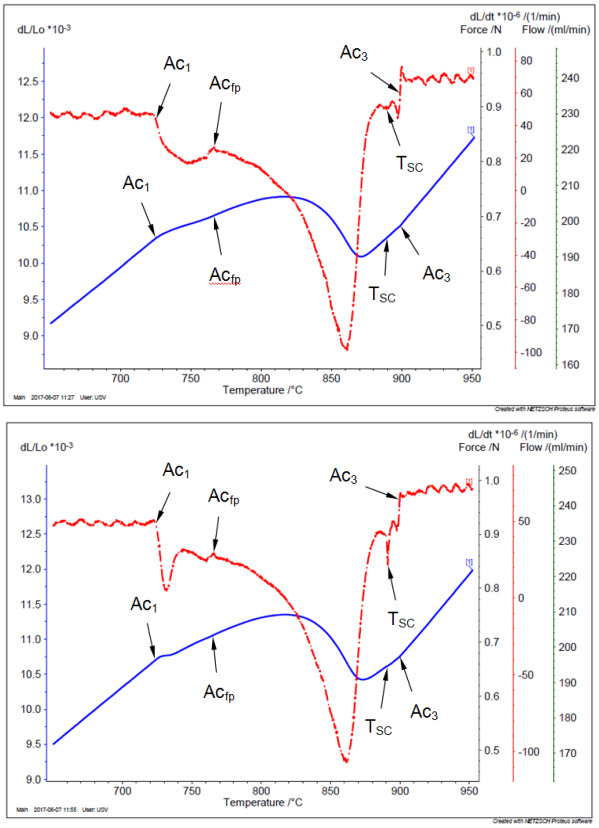


Fig.6. Dilatometric curve (continuous line) and its corresponding derivative (dashed line) of the B_Steel: a) D_B; b) FA_B

The high resolution dilatometry, which highlights precisely the two processes that take place when heating a hypoeutectoid steel (the pearlite dissolution into austenite and the allotropic transformation of ferrite into austenite), permits the determination of the Ac_{fp} temperature (the pearlite dissolution finish temperature). Ac_{fp} is the temperature at which the first contraction on the dilatometric curve, due to pearlite to austenite transformation, finishes, being also a transition temperature between the two transformations, (P $\rightarrow$ γ) and ($\alpha \rightarrow \gamma$); this temperature is much easier to highlight on the first derivative curve, curve that has two peaks which indicates the contractions caused by (P $\rightarrow$ γ) and ($\alpha \rightarrow \gamma$) transformations (Figs.5.a, 6.a, 7.a) [2, 4, 5, 9, 10]. Immediately after finishing pearlite dissolution (P $\rightarrow$ γ) the allotropic transformation of ferrite in austenite ($\alpha \rightarrow \gamma$) begins, transformation that determine also a decrease in the specific volume (a contraction). After achieving a minimum in the variation of the relative length of the specimen, on the dilatometric curves drawn for A_Steel and B_Steel with as-delivered structures (D_A and D_B), at a temperature noted T_{SC}, there is a small contraction which is better highlighted on the first derivative curves, which have a small peak at T_{SC} temperature (Figs.5.a, 6.a) [4, 5]. This modification of volume is difficult to explain, being determined, probably, either through the formation of austenite from a ferrite that remains untransformed in structure (which transforms almost instantaneously due to the change in ferrite-to-austenite transformation kinetics) [3-5, 13], either through a diffusion of carbon from rich-carbon austenite (formed of pearlite) in less-carbon austenite (formed of ferrite) [4, 5, 14]. The modification of volume of the specimens highlighted for A_Steel and B_Steel, at T_{SC} temperature, does not appear on the dilatometric curves and their corresponding derivatives obtained for C_Steel (specimens with as-delivered structures, D_C), Fig.7.a [5].

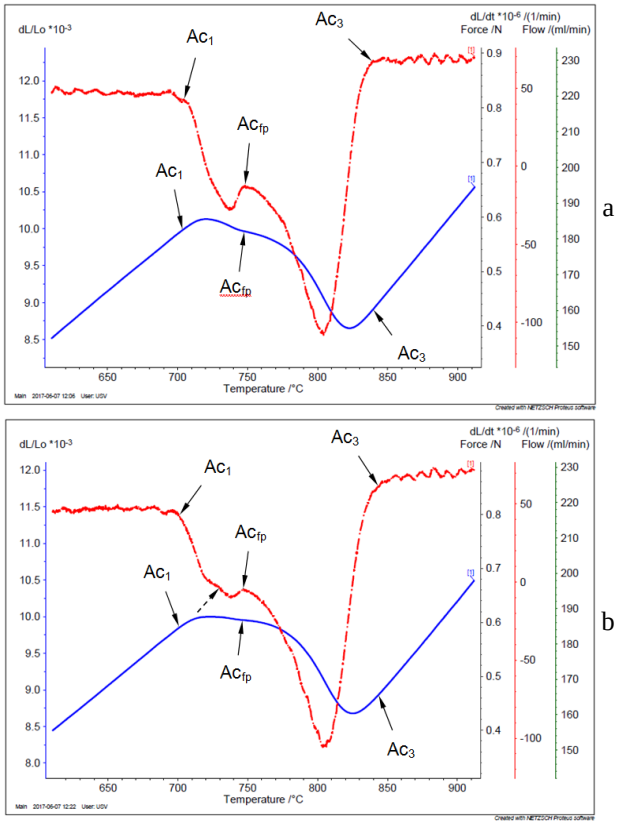


Fig.7. Dilatometric curve (continuous line) and its corresponding derivative (dashed line) of the C_Steel: a) D_C; b) FA_C

The dilatometric curves and its corresponding derivative for the specimens with full annealing structures (FA_A, FA_B and FA_C) have modified shapes as compared to those designed for as-received structures (D_A, D_B and D_C), Figs.5.b, 6.b., 7.b. Thus, in the case of A_Steel and B_Steel, on the dilatometric curves the contraction to the onset

of austenitisation appears better outlined (Figs.5.b, 6.b), and on the first derivative curve, between the temperatures Ac_1 and Ac_{fp}, there are two peaks. The first peak (higher than the second one) corresponds to the eutectoid transformation (the pearlite dissolution in austenite), but the presence of the second peak is difficult to explain; either in the austenite formed by the pearlite dissolution would remain a small amount of undissolved cementite particles which then dissolved, either homogenization of the austenite (which was formed by the pearlite dissolution) was produced. In the case of C_Steel, there are no highlighted the modifications or aspects noticed for the other two steels; only a small anomaly appears on the first derivative curve, marked with an dashed arrow in Fig.7.b. The contraction observed at T_{SC} temperature on the dilatometric curves plotted for the A_Steel and B_Steel specimens with as-received structures also appears on the curves plotted for specimens with full annealing structures (Figs.5.b, 6.b); it is better emphasized on both the dilatometric curves and the first derivative curve. In the case of C_Steel, this contraction from T_{SC} temperature, does not even occur in the specimens with full annealing structures (Fig.7.b).

Tab.2 shows, depending on the initial structure, the results of the dilatometric analyses, which were obtained through processing with NETZSCH Proteus® Software 7.1.0. of the signals of variation in the length of the specimens, provided by the dilatometer, during tests.

Table 2. The results of the dilatometric analyses

Steel	Specimens with as-received structures			
	Ac_1	Ac_{fp}	Ac_3	$(Ac_{fp} - Ac_3)$
	°C	°C	°C	°C
A_Steel	724.00	765.60	899.40	133.80
B_Steel	725.10	766.30	898.90	132.60
C_Steel	703.80	747.70	839.70	92.00
Steel	Specimens with full annealing structures			
	Ac_1	Ac_{fp}	Ac_3	$(Ac_{fp} - Ac_3)$
	°C	°C	°C	°C
A_Steel	722.70	764.70	901.90	137.20
B_Steel	723.80	765.20	899.70	134.50
C_Steel	700.90	746.60	843.60	97.00

Data in Tab.2 show that the temperatures of the critical points in solid-state phase transformation determined for the specimens with full annealing structures have lower values for Ac_1 (with 1.30 °C for A_Steel and B_Steel, 2,90 °C for C_Steel) and Ac_{fp} (with 0,90 °C for A_Steel, 1,10 °C for B_Steel and C_Steel) and higher for Ac_3 (with 2,50 °C for A_Steel, 0,80 °C for B_Steel and 3,90 °C for C_Steel) than those established for the specimens with as-received structures (Figs.8-10). This has led to a slight increase in the temperature range (Ac_{fp} - Ac_3), with 3,40 °C for A_Steel, 1,90 °C for B_Steel and 5,0 °C for C_Steel (Fig.11), range which is important for the dual-phase steels production technologies.

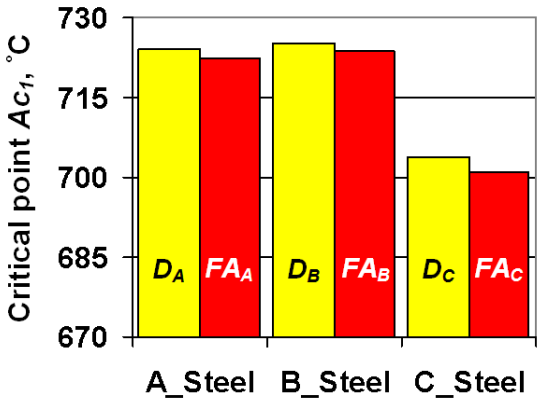


Fig.8. Influence of the initial structure on critical point Ac_1

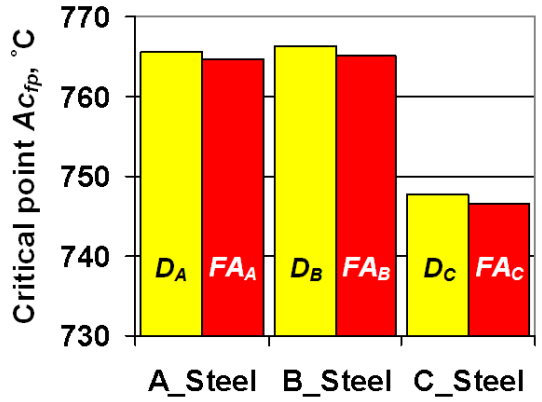


Fig.9. Influence of the initial structure on critical point Ac_{fp}

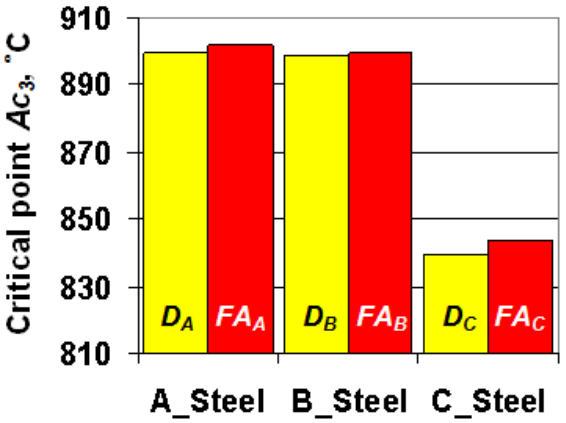


Fig.10. Influence of the initial structure on critical point Ac_3

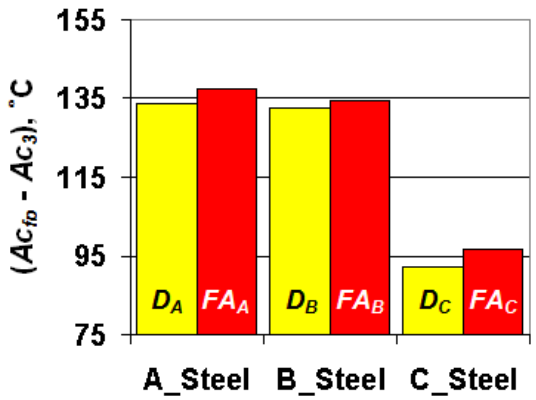


Fig.11. Influence of the initial structure on the temperature range (Ac_{fp} - Ac_3)

CONCLUSIONS

Dilatometric analyses performed on a DIL 402 Expedis-SUPREME dilatometer, allowed to highlight the solid-state phase transformations that occurred during the continuous heating of three hypoeutectoid steels, 0.087% C - 0.511% Mn, 0.101% C - 0.529% Mn and 0.093% C - 1.922% Mn (the dissolution of the pearlite in austenite and the allotropic transformation of proeutectoide ferrite into austenite), as well as to determine the critical temperatures (points) at which these transformations occurred.

All the specimens used had ferrite-pearlite structures, but with the grain size index (G) different: those with as-received structures had grain size index (G) between 8.69 and 10.16, and those with a full annealing structures between 7.28 and 8.39. The pearlite dissolution into austenite (eutectoid transformation) was carried out in a temperature interval ranging between pearlite dissolution start temperature (critical point Ac_1) and pearlite dissolution finish temperature (denoted Ac_{fp}); allotropic transformation of ferrite into austenite occurred between the Ac_{fp} temperature and the ferrite to austenite transformation finish temperature (critical point Ac_3).

The dilatometric curves and its corresponding derivative for the specimens with full annealing structures have modified shapes as compared to those designed for as-received structures, both in the region of the pearlite dissolution into austenite (eutectoid transformation), and in the region of the allotropic transformation of ferrite into austenite.

The temperatures of the critical points in solid-state phase transformation determined for the specimens with full annealing structures had lower values for Ac_1 and Ac_{fp} and higher for Ac_3 than those established for the specimens with as-received structures. This has led to a slight increase in the temperature range (Ac_{fp} - Ac_3), range which is important for the dual-phase steels production technologies.

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NOMENCLATURE

Ac_1 - pearlite dissolution start temperature (lower temperature of the ferrite-austenite field during heating), °C;

Ac_3 - ferrite to austenite transformation finish temperature (upper temperature of the ferrite-austenite field during heating), °C;

Ac_{fp} - pearlite dissolution finish temperature (ferrite to austenite transformation start temperature), °C;

T_{SC} - small contraction temperature, °C;

α - proeutectoide ferrite;

γ - austenite;

P - pearlite;

đ - mean diameter of grain;

ā - mean area of grain;

G - grain size index.

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