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Characteristics of Phase Transformations in Medium-Carbon Nanobainitic Steel of High Hardenability

Abstract: The article presents the results of phase transformations taking place in steels having a carbon content of 0.55 % and characterised by high hardenability (NBH), intended for the industrial fabrication of sheets. Phase transformations were examined using the dilatometric method under continuous cooling conditions (CCT) and during isothermal holding (TTT), applying results of microstructural observations and those of hardness measurements of dilatometric specimens. The research work involved developing the diagram of phase transformations under continuous cooling conditions, using the cooling rate range of 0.025 °C/s to 50 °C/s and a fragment of a diagram of isothermal phase transformations within the temperature range of 200 °C to 275 °C. The Authors proposed a new type of isothermal diagram of phase transformations developed for a specific cooling rate from austenitisation temperature equal to v (TTTv), enabling its application in the precise design of industrial isothermal specimens. The analysis of dilatograms as well as that of microstructural observations and dilatometric specimen hardness test results enabled the identification of experimental parameters applied in the heat treatment of specimens made of the NBH steel used in tensile tests. The research-related tests confirmed information contained in scientific reference publications stating that the presence of retained austenite of blocky morphology did not necessarily result in the low plasticity of nanobainitic steels. After the isothermal transformation into nanobainite at a temperature of 225 °C and that of 235 °C, the steel (NBH) contained approximately 20 % of retained austenite, including blocky austenite transformed into martensite during deformation and characterised by favourable plasticity restricted within the range of 13.6 % to 15.5 % of ultimate elongation in relation to a strength of 1.9 GPa.

Key words: isothermal and continuous phase transformations, medium-carbon steel, nanobainite, dilatometric analysis, microstructure, mechanical properties

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1. Introduction

Nanobainitic steels are new structural materials currently implemented in industrial production, characterised by a tensile strength of up to approximately 2.2 GPa, favourable plasticity and acceptable impact strength making them usable in many applications. The conventional term of “nanostructure” is used for cases where the average size of matrix grains separated by high-angle boundaries is less than 100 nm. The definition of nanostructure refers to both equiaxial grains and morphological elements of the matrix having the form of lamellae or laths (as is the case with low-temperature bainite). In terms of the lamellar or lath structure, the dimension defining the degree of matrix fragmentation is the average thickness of lamellae or laths. The term of submicrometric (or submicron) structure is related to the average size of grain or lamella ranging from 100 nm to several hundred nm.

The invention (at the beginning of the 21st century) of the first grades of nanobainitic steels characterised by high carbon contents (usually restricted within the range of 0.8 % to 0.9 %) (contents of chemical elements referred to in the article are expressed in wt %) [1–4] was followed by the period of intense research concerning the development of these steel grades, including grades characterised by lower carbon contents than those in the first nanobainitic steel grades. Nanobainite is a dual-phase microstructure (duplex type) composed of lamellae or laths of carbide-free bainitic ferrite of high dislocation density forming bundles made up of parallel lamellae or laths as well as of non-transformed (retained) austenite. Retained austenite exists in two morphological forms, i.e. as layers between lamellae of bainitic ferrite having an average thickness of less than 100 nm and grains (blocks) sized between tenths of a millimetre and several micrometres. The thickness of carbide-free bainite lamellae is predominantly affected by the yield point of austenite during the transformation into bainite as well as on free transformation energy and the bainite lamellae nucleation rate, which in turn, depends primarily on the degree of austenite supercooling [5]. The strength of austenite is the function of the amount and type of chemical elements dissolved in austenite and of transformation temperature – the higher the alloyability of austenite and the lower the transformation temperature, the higher the strength of austenite [5]. An increase in the yield point of bainitic ferrite by reducing the thickness of ferrite lamellae could be obtained by increasing the alloyability of steel (particularly the content of carbon) and/or by reducing the bainitising temperature. The yield point of nanobainitic steel depends directly on the thickness of bainitic ferrite lamellae and the fraction of bainitic ferrite in steel – the thinner the lamellae of bainitic ferrite and the greater the fraction of ferrite volume, the higher the yield point of nanobainitic steel [6]. Tests discussed

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in [7] revealed that the average thickness of bainitic ferrite lamellae was not constant during the bainitic transformation. It was observed that after exceeding a specific degree of transformation, newly formed lamellae of bainitic ferrite were characterised by greater thickness than those formed in the previous stages of transformation. The above-presented phenomenon was not obvious with its reasons remaining unknown. Mechanical properties of nanobainitic steels also depend on the content (volume fraction) of retained austenite and its morphological form, the degree of refinement, enrichment with carbon and mechanical stability (being the function of, among other things, the above-presented physical features). Works by Caballero, Morales-Rivas, Garcia-Mateo, Sourmaila et al. [7–11] point to the following primary conclusions concerning the effect of retained austenite on the mechanical properties of nanobainitic steels:

- lack of direct correlation between the content (volume fraction) of retained austenite before deformation and steel plasticity expressed by the value of elongation in the tensile stress;
- lack of confirmation of a previously formulated thesis that there is an optimum level of retained austenite mechanical stability ensuring the maximum obtainable plasticity. However, the Authors point out that the higher the mechanical stability of retained austenite, the higher the plasticity of nanobainitic steels;
- lack of evidence confirming previous findings according to which the obtainment of the acceptable ductility of nanobainitic steels required that the content of retained austenite should not exceed approximately 10 %;
- related test results revealed that a factor increasing the ductility (plasticity) of nanobainitic steels was the reduction of the difference between the strength of retained austenite and the strength of bainitic ferrite (i.e. the phase characterised by greater strength than that of retained austenite) by increasing the strength of retained austenite, obtained primarily by increasing the content of carbon dissolved in retained austenite.

Designed chemical compositions of nanobainitic steels constitute a compromise selection dictated by the effect of individual chemical elements on technological and service properties required for specific applications. Research conducted since 2008 at the Łukasiewicz Research Network – Upper-Silesian Institute of Technology (previously known as the Institute for Ferrous Metallurgy and Łukasiewicz Research Network – Institute for Ferrous Metallurgy) on nanobainitic steels was concerned with the design and fabrication of steel grades with a carbon content of approximately 0.6 % and characterised by higher plasticity if compared to that of steels grades having a carbon content restricted within the range of 0.8 % to 0.9 %. The above-named project led to the development of a technology enabling the production of sheets readily implementable industrially [12–16]. However, further research works are needed to develop nanobainitic steel grades characterised by improved technological properties enabling the economically efficient fabrication of a wide range of products in comparison with that used presently in the manufacturing of steel products. A particularly significant issue was concerned with the selection of the carbon content so that, on one hand, it could be sufficient (when combined with the effect of alloying elements contained in the steel) to ensure the obtainment of sufficiently low temperature MS and high carbon content in retained austenite and, on the other, be low enough to minimise technological risks such as the low plasticity of air-cooled products after hot plastic working as well as to ensure high ductility during the plastic deformation of finished products. The reduction of carbon content in steels combined with the simultaneous obtainment of high hardenability requires increasing the content of alloying elements significantly increasing hardenability (Mo, Mn, Cr), which, in turn, leads to the segregation of chemical elements during the solidification of ingots, resulting in the structural banding in finished products. For the above-presented reasons, the chemical composition of nanobainitic steels should be designed to ensure the required level of service properties of finished products and take into account characteristics of technological operations during the manufacturing of such products.

The primary objective of the tests, the results of which are presented in the remainder of the article, was to characterise phase transformations taking place in nanobainitic steel having a carbon content of 0.55 % and characterised by high hardenability, intended for the industrial-scale production of sheets/plates having a thickness of up to approximately 10 mm. Phase transformations were tested using the dilatometric method under continuous cooling and isothermal holding conditions. The results of dilatometric tests, microstructural observations and hardness measurements of dilatometric specimens were used to identify parameters of experimental heat treatment and perform tensile tests of specimens.

2. Test materials and methodology

2.1. Preparation of test materials

The solidification of alloy steels is accompanied by the interdendritic and zonal segregation of substitute elements such as Mo, Mn, Cr and Si. The consequence of chemical heterogeneity is the formation of banded microstructure in finished products. In terms of nanobainitic steels, the bands characterised by the higher concentration of chemical elements contain higher amounts of retained austenite (responsible for the reduction of hardness), whereas the bands characterised by the lower concentration of chemical elements contain lower amounts of retained austenite, translating into higher hardness. In industrial processes, it is not possible to entirely homogenise substitute elements through comparative diffusion in thermal operations, typical of transforming ingots into products, including the homogenising procedure involving the application of

Table 1. Result of check chemical analysis of NBH steel, weight %; CE_{IIW}= (%C) + (%Mn)/6 + (%Cr + %Mo + %V)/5 + (%Ni + %Cu)/15 – formula for the calculation of carbon equivalent adopted by the International Institute of Welding – IIW)

C	Mn	Si	P	S	Cr	Mo	V	Ti	Al	CE_{IIW}
0.55	2.00	1.86	0.010	0.005	1.30	0.73	0.098	0.010	0.018	1.31

parameters which are both technically possible and economically rational (typically 1200 °C – 24 hours).

The investigation of the effect of homogenising treatment of semi-finished products made of nanobainitic steel, using the highest technically obtainable hold temperature involved the holding of a slab (60 mm × 250 mm × 760 mm) made of nanobainitic steel (from heat designated NBH) at a temperature of 1300 °C for 72 hours. After homogenising, the slab made of the NBH steel was subjected to surface milling aimed to remove a decarbonised and internally oxidised layer followed by hot rolling aimed to obtain flat bars which, directly after rolling, underwent softening at a temperature of 675 °C for 5 hours. A fragment of a flat bar in the softened state, having a thickness restricted within the range of 9.3 mm to 9.4 mm and a width of 227 mm, was sampled for test specimens used in the research work discussed in the article. Table 1 contains the results of a check chemical analysis involving the specimens made of NBH steel. The contents of chemical elements were identified using the OES method and a Magellan Q8 spark emission spectrometer (Bruker). The determination of the carbon content involved additional analyses performed using a CS-600 analyser (Leco).

The homogenising treatment performed at a temperature of 1300 °C (higher than that used in standard processing) for 72 hours led to the homogenised arrangement of chemical elements on a micro scale and the disappearance of microstructural banding, characterised by the average distance between areas of increased contents of alloying elements below approximately 100 µm. The use of significantly increased homogenising temperature and extended hold time (at the homogenising temperature) did not trigger the homogenising of the arrangement of alloying elements (and the resultant disappearance of structural banding) on a macro scale and on a scale between a micro and a macro scale, i.e. in relation to a distance in excess of 0.1 mm between zones characterised by maximum contents of chemical elements.

The comparison of the hardenability of NBH steel and selected nanobainitic steels of various chemical compositions necessitated the adoption of a critical cooling rate as a hardenability measure. The critical cooling rate was defined by the minimum value of cooling rate not triggering diffusive (i.e. into pearlite or ferrite) or para-diffusive transformations (i.e. into upper bainite) – the lower the critical cooling rate, the higher the hardenability. The value of the critical cooling rate affects the maximum thickness (diameter) of the cross-section of a product which can be subjected to cooling in the air between the austenitising temperature and the temperature of isothermal holding without the initiation diffusive transformations. The comparison of continuous TTT diagrams of nanobainitic steels of various chemical compositions, found in reference publications [6, 9, 17–20] and generated using the JMatPro8adv software programme, revealed that the NBH steel subjected to the tests was characterised by the lowest critical rate amounting to 0.5 °C/s and, consequently, by the highest hardenability. The beginning of the bainitic transformation at a temperature of less than 300 °C requires the cooling of NBH steel at a rate of not less than 0.5 °C/s, whereas the transformation of austenite into ferrite is initiated at a lower cooling rate. The above-presented analysis reveals that the NBH steel grade belongs to medium-carbon nanobainitic steels characterised by high hardenability and enabling the production of sheets/plates having thicknesses of up to approximately 10 mm, using the technology including uninterrupted cooling in the air from the temperature of the end of the rolling process or the re-austenitising temperature to the temperature of isothermal holding directly above temperature MS.

2.2. Methodology of dilatometric tests

The specimens used in the dilatometric tests in the form of tubes having an external diameter of 4 mm, an internal diameter of 2 mm and a length of 10 mm were made from a flat bar (made of the NBH steel) in the softened state, maintaining the parallelism of the tube axis in relation to the flat bar rolling direction. The tests, performed using DIL805 A/D and DIL805A/D/T dilatometers, included the determination of characteristic temperatures of the test steels (A_{c1}, A_{c3}, M_S), the development of a phase transformation diagram for continuous cooling conditions (continuous TTT diagram) and a fragment of a diagram of transformations under isothermal conditions (TTT). Both heating and holding at the preset temperature were performed in a vacuum $<5 \cdot 10^{-4}$ mbar. Temporary temperature deviations from the preset value did not exceed +/-1.0°C. The determination of critical temperature values and the development of diagrams of phase transformations were performed using the method which involved placing a tangent to the dilatation curve near the beginning and the end of the phase transformation [21, 22]. In terms of the analysis of inseparable (integral) transformations (taking place directly one after another or overlapping one another), it was necessary to apply the numerical differentiation of dilatation curves. The determination of the temperatures at the beginning and the end of phase transformations characterised by weak dilatation effects involved the methodology based on the linear transformation of a segment of the dilatation curve subjected to analysis [23]. Critical temperatures A_{c1} (A_{c1p}, A_{c1k}) and A_{c3} were determined by heating the specimens at a rate of 2.5 °C/min. to a temperature of 1000 °C. The austenitisation of the specimens before time-temperature cycles, used to develop diagrams of phase transformations, was performed at a temperature of 950 °C for 30 minutes. The cooling rates applied when developing the CCT diagram were constant within the entire range of temperature and amounted to 50 °C/s, 20 °C/s, 10 °C/s, 5 °C/s, 2 °C/s, °C/s, 0.5 °C/s, 01 °C/s, 0.05 °C/s and 0.025 °C/s. The specimens used to prepare the TTT diagram were cooled from the austenitising temperature at a rate of 20 °C/s to the temperature of isothermal transformation and held at the temperature to the cessation of transformation, not longer, however, than for 160 hours. The monitoring of the progress of isothermal transformation ultimately led to the use of the following hold parameters: 200 °C – 160 hours, 225 °C – 160 hours, 235 °C – 113 hours, 245 °C – 120 hours, 255 °C – 67 hours and 275 °C – 48 hours.

2.3. Methodology of microstructural tests and measurements of mechanical properties

The microstructural tests and measurements of mechanical properties were performed in accordance with certified procedures. Microstructures of the specimens etched with Nital of various HNO_3 concentrations were observed and imaged using a DSX500 light microscope (LM) (OLYMPUS) as well as an Inspect F (FEI) and JSM-7200F (JEOL) scanning electron microscopes (SEM). The measurements

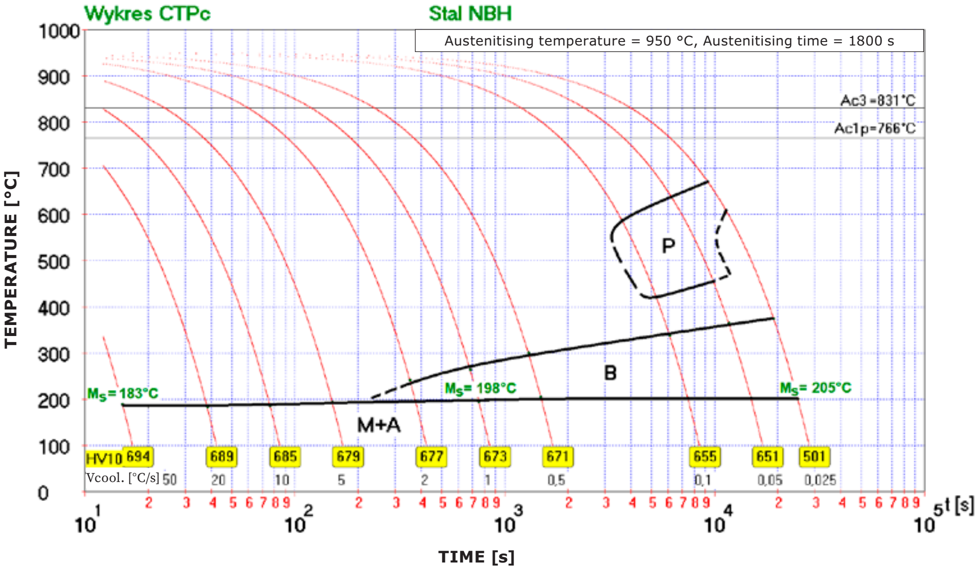


Fig. 1. Experimentally determined continuous cooling transformation diagram (CCT) for the NBH steel; A – austenite, M – martensite, B – bainite and P – pearlite

of retained austenite contents were performed using an Empyrean X-ray diffractometer (PANalytical) and filtered copper radiation in configuration with a Pixcel detector applying the X-radiation microbeam formed by using a capillary having a diameter of 300 µm. The identification of phase composition was performed in accordance with an accredited procedure, using the database of the International Centre for Diffraction Data PDF-4+, version 2023. The content of retained austenite was identified using the Averbach-Cohen method. The error of measurements, performed using the XRD method and determined by the parameter of extended uncertainty, amounted to a maximum of ±1 % of austenite content in the specimen [24]. Hardness measurements involved the use of the Vickers test method, a Swiss Max 300 hardness tester and a load of 10 kG (HV10). Tensile tests were performed using a Z250 testing machine (Zwick/Roell) and flat specimens with the cross-section of the part subjected to deformation having dimensions of 4.5 mm × 10 mm × 60 mm (length), cut out in parallel to the flat bar rolling direction. The value of elongation was measured (using an extensometer) in the central segment of the specimens between points located 40 mm away from each other. The deformation rate used in the test was characteristic of quasi-static deformation, had the same value in relation to all the test specimens and amounted to $4.0 \cdot 10^{-3}$ s^{-1}.

3. Test results

3.1. Phase transformations during continuous cooling

Figure 1 presents the diagram of phase transformations taking place during continuous cooling (CCT) in relation to the NBH steel, developed on the basis of dilatograms of the specimens cooled after austenitisation at constant cooling rates, in accordance with lines mapped on the diagram. The diagram also contains the temperature of the beginning and that of the end of the transformation of stable phases into austenite (at ambient temperature), i.e. – A_{c1p} and A_{c3} respectively, as well as the average hardness values of the dilatometric specimens after austenitisation and cooling performed using related parameters. The use of a cooling rate of above 5 °C/s led to the formation of martensite in the specimens (which also contained untransformed, i.e. retained austenite). The reduction of the cooling rate to approximately 2 °C/s initiated the low-temperature bainitic transformation, yet the primary phase formed from austenite was martensite. The further reduction of the cooling rate to approximately 0.1 °C/s initiated transformation into pearlite. Further cooling led to the transformation of austenite into bainite and, subsequently, martensite. The microstructure formed as a result of the above-named cooling process is presented in Figures 2a and b. Cooling at a rate of 0.025 °C/s led to an increase in the contents of pearlite and bainite in the microstructure (Figures 2c–e). After the end of pearlitic transformation at cooling rates restricted within the range of 0.1 °C/s to 0.025 °C/s, in the CCT diagram (Fig. 1), it was possible to observe a field between the pearlitic and bainitic transformations, characterised by the increased stability of austenite, in which phase transformations had not taken place. The initiation of bainitic transformation required the further supercooling of austenite, aimed at obtaining the critical energy of bainite nucleation. The final microstructure at ambient temperature, formed as a result of cooling at the lowest rate amounting to 0.025 °C/s, was composed of pearlite, bainite, martensite and untransformed austenite (Fig. 2c–e).

The critical cooling rate (read out of the experimental CCT diagram) ensuring the obtainment of initial temperature BS of 300 °C or lower amounted to 0.5 °C/s and was the same as the cooling rate read out of the CCT diagram for the NBH steel, generated using the JMatPro8adv software programme. Measurements of free air cooling rates in relation to the centre of plates having thicknesses restricted within the range of 5 mm to 10 mm revealed that the maximum thickness of the plate cooled from the austenitisation temperature amounting to 950 °C to the temperature of isothermal transformation (without initiating transformation into bainite at a temperature of 300 °C or higher) amounted to approximately 10 mm.

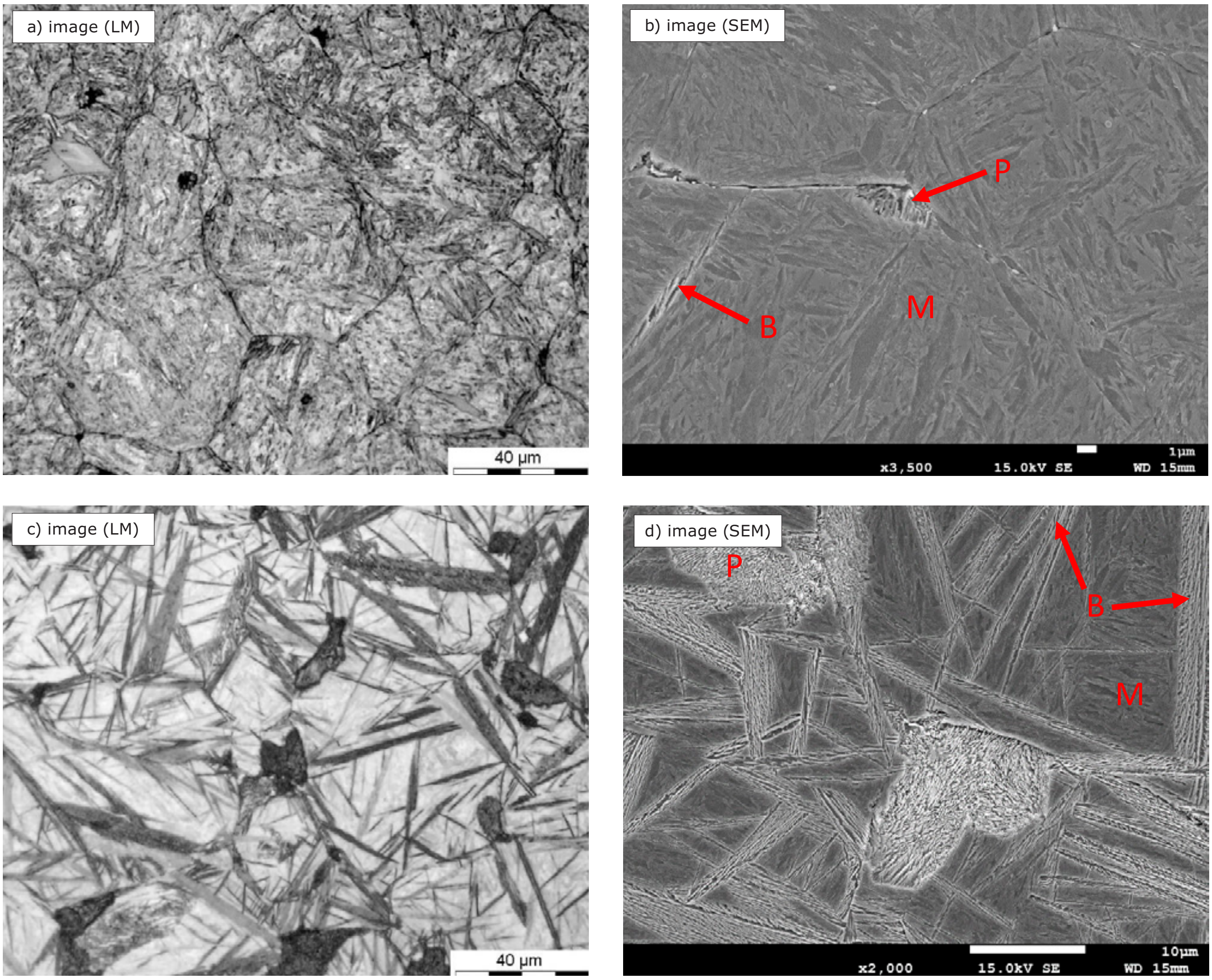
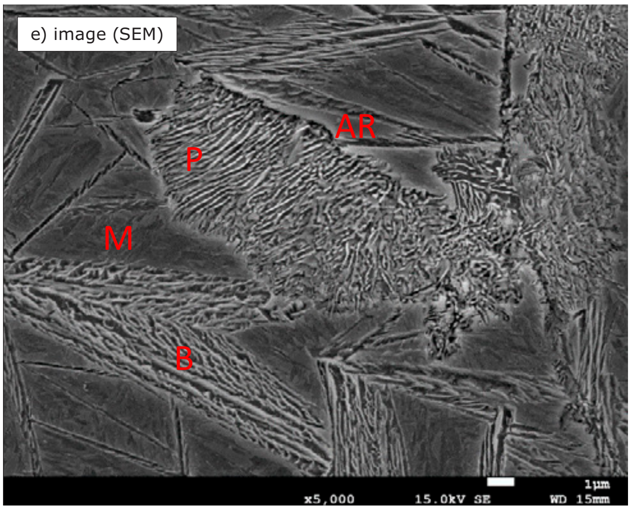


Fig. 2. Microstructures of dilatometric specimens after austenitising and cooling with rates: a), (b) 0.1 °C/s and (c), (d), (e) 0.025 °C/s; M – martensite, B – bainite, P – pearlite and AR – retained austenite



3.2. Phase transformations during isothermal holding

A fragment of a diagram of phase transformations taking place in the NBH steel during isothermal holding (TTT) was prepared for the temperature range of 275 °C to 200 °C, i.e. within which nanobainite can be formed in the test steel (Fig. 3). The diagram contains lines indicating the beginning (initiation) and the end (cessation) of bainitic transformation (B_S and B_W respectively) as well as line M_S, identified under conditions of the continuous cooling of the specimens. After the cessation of bainitic transformation, it was possible to observe the presence of untransformed austenite, indicated in the diagram in Figure 3 as A_R – retained austenite, which could undergo partial transformation into martensite as a result of cooling to ambient temperature. The line in the diagram (Fig. 3) indicating temperature M_S, being a fragment of line M_S determined for conditions of continuous cooling (Fig. 1), reveals that, in relation to the test steel, the temperature of the beginning of martensitic transformation depends on the cooling rate. The reason for an increase in temperature M_S along with a decrease in the cooling rate could be short-range diffusive processes in supercooled austenite, increasing the density of areas where the nucleation of martensite lamellae took place.

Table 2 presents times indicating the initiation and cessation of bainitic transformation read out of dilatograms in relation to individual temperature values, contents of retained austenite in the specimens after the transformation at a temperature 235 °C and that of 225 °C as well as specimen hardness measurement results after transformation. The times preceding the initiation and cessation of transformation were measured from the moment at which the cooling process was initiated from the austenitising

Specimen designation	Temperature [°C] / time [h] of isothermal holding	Time for initiation of transformation – t_i [s], time for cessation of transformation – t_c [s]	Content of retained austenite, XRD measurement, vol. % nm – not measured		Average hardness, HV10
			Single measurements	Average value	
a	275 / 48	t_i = 2270; t_c = 128605 (35.7 h)	nm	–	510
b	255 / 67	t_i = 2628; t_c = 208697 (58.0 h)	nm	–	518
c	245 / 120	t_i = 3030; t_c = 321714 (89.4 h)	nm	–	542
d	235 / 113	t_i = 3386; t_c = 392985 (109.2 h)	23; 10; 23	19	551
e	225 / 160	t_i = 4490; t_c = 562870 (156.4 h)	13; 24; 18	18	556
f	200 / 160	t_i = 40471; t_c = continued after 160 h	nm	–	572

Table 2. Times for the initiation and cessation of bainite transformation read out of the dilatograms recorded during the isothermal holding of specimens of the NBH steel at temperatures specified in the Table, contents of retained austenite in the specimens transformed at temperatures of 235 °C and 225 °C as well as the hardness of dilatometric specimens after transformation; nm – not measured

temperature. The time preceding the cessation of bainitic transformation was determined graphically, i.e. by identifying the point on the dilatometric curve in the area of *plateau*, where the diagram of the change in the length of the dilatometric specimen as a function of time became tangent to the horizontal line [25]. The average values of the total content of retained austenite identified in relation to the specimens after isothermal transformation at a temperature of 235 °C and that of 225 °C were similar and amounted to 19 % and 18 % respectively. A probable reason for the scatter of results of single measurements of retained austenite on the cross-sectional areas of the dilatometric specimens was the small cross-sectional area of the measurement beam (°C approximately 300 µm), applied due to the limited accessible wmeasurement surface (which increased sensitivity to microsegregations of chemical elements observed in the material and the resultant inhomogenous presence of retained austenite). Another possible reason could be the crystallographic texture of the material.

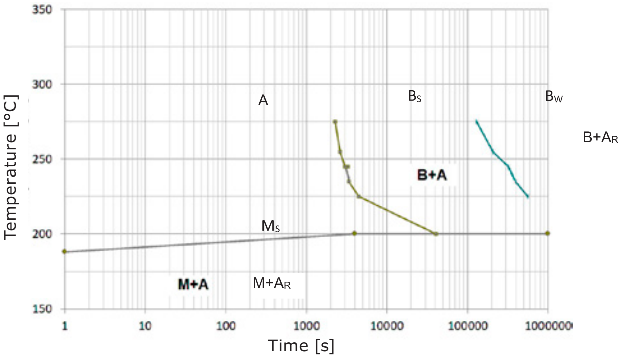


Fig. 3. Experimentally determined part of isothermal transformation diagram (TTT) within the temperature range of 275 °C to 200 °C in relation to the NBH steel; A – austenite, B – bainite (nanobainite), M – martensite, A_R – retained austenite, M_S – temperature at the initiation of martensitic transformation during continuous cooling, B_S – temperature at the start of bainitic transformation during isothermal holding and B_W – temperature at the cessation of bainitic transformation during isothermal holding

Figure 4 presents LM and SEM images of the microstructure of the dilatometric specimens after austenitising and isothermal holding at a temperature of 275 °C for 48 hours and at 225 °C for 160 hours. The transformation led to the formation of a structure composed of bundles and packets of laths of bainite and untransformed (retained) austenite, visible in the LM images as light polyhedral (polygonal) grains. In the SEM microphotographs, exemplary grains of blocky retained austenite are designated as ARb. Nanolaths of retained austenite present between the bainite laths or lamellae are not distinguishable in the SEM at small magnifications (see Figures 4b, 4d). The comparison of the microstructural images revealed that the specimen after transformation at a temperature of 225 °C contained fewer grains of blocky retained austenite in comparison with the amount and morphology of retained austenite which remained after transformation at the higher temperature (275 °C). As a result of the isothermal transformation, part of previously untransformed austenite might have been transformed into martensite during cooling to ambient temperature, forming austenitic-martensitic islands. A detailed description of the final microstructure requires additional tests involving the use of high-resolution scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The above-named tasks are intended to be performed at the subsequent stage of the project.

3.3. Determination of the parameters of the heat treatment of the NBH steel aimed to obtain nanobainite

The parameters of the heat treatment of the specimens used in the tensile tests were identified on the basis of the analysis of dilatograms recorded for isothermal holding conditions within the range of temperature including the transformation of supercooled austenite into nanobainite as well as on the basis of results of microstructural tests and hardness measurements concerning the specimens after cycles of processing in the dilatometer. The primary criteria affecting the adjustment of experimental heat treatment parameters were adopted in relation to mechanical properties, i.e. a minimum hardness of approximately 550 HV, as well as in relation to the advancement of the

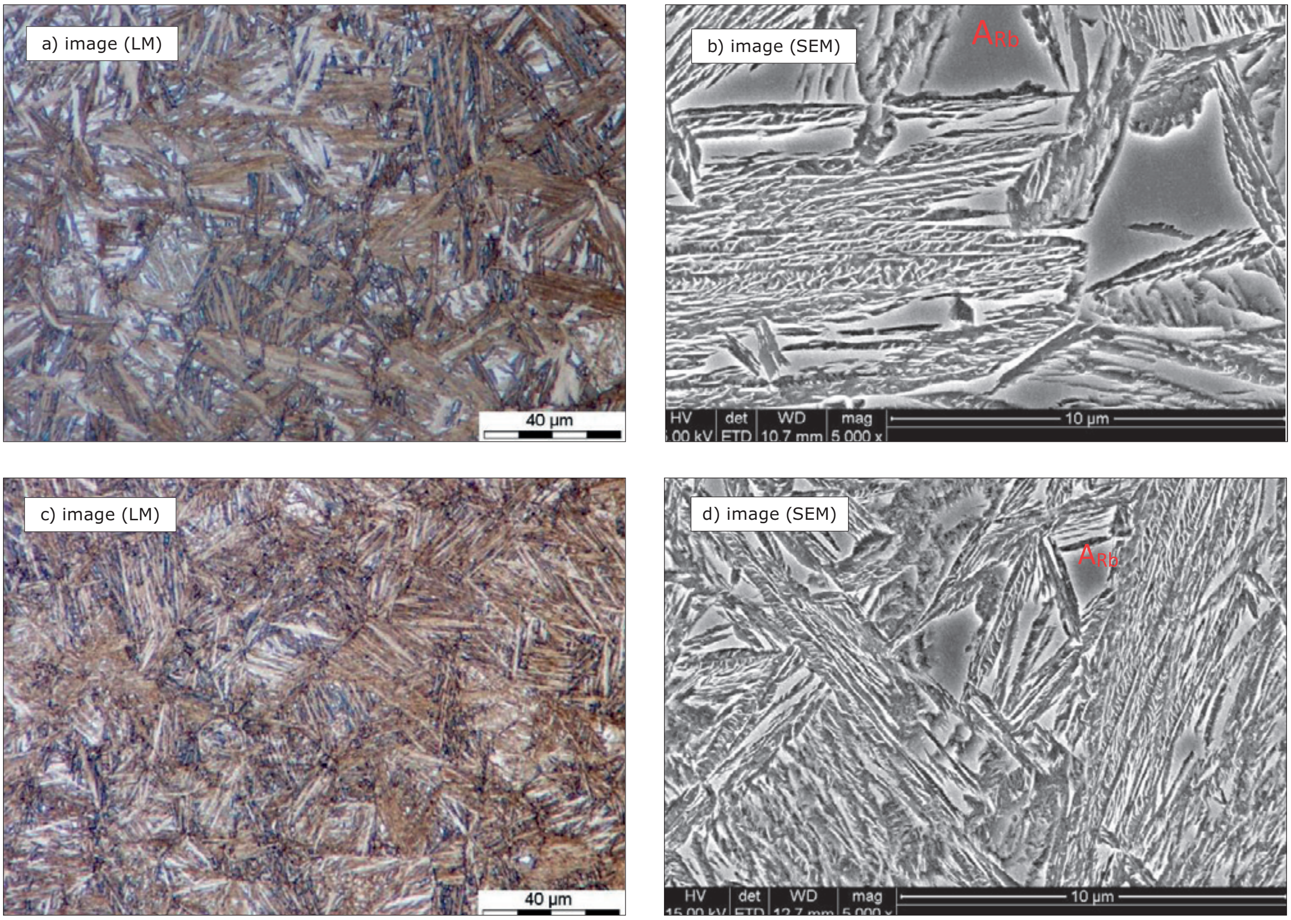


Fig. 4. Microstructures of dilatometric specimens after austenitising and isothermal holding performed using the following parameters: (a), (b) 275 °C / 48 h and (c) and (d) 225 °C / 160 h; A$_{Rb}$ – blocky retained austenite

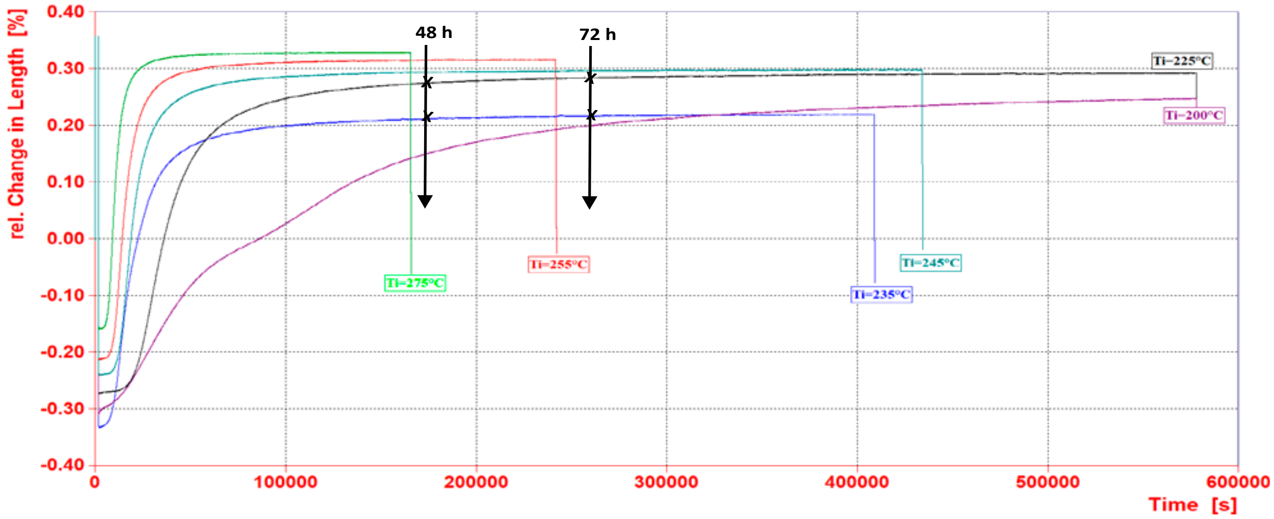


Fig. 5. Correlations between the relative change in the length of the dilatometric specimens and the time of holding at specific temperatures with points (marked with ×) on dilatograms recorded at temperatures of 235 °C and 225 °C, designating holding times of 48 hours and 72 hours

transformation of austenite into nanobainite, i.e. near the cessation of transformation. Figure 5 presents correlations between the relative change in the length of dilatometric specimens and the time of holding at test temperatures. The analysis of the kinetics of bainitic transformation (within the temperature range subjected to tests) (Fig. 5) as well as of the microstructure and hardness of the dilatometric specimens (Table 2) led to the determination of the following parameters (marked with crosses on the dilatograms presented in Figure 5) of the heat treatment of the specimens used in the tensile tests: HT1/235 °C – 48 h, HT2/235 °C – 72 h, HT3/225 °C – 48 h and HT4/225 °C – 72 h.

3.4. Mechanical properties and the microstructure of the NBH steel after isothermal transformation at temperatures of 225 °C and 235 °C

The mechanical properties identified in the tensile tests and hardness measurements of the specimens subjected to heat treatment including austenitising at a temperature of 950 °C for 30 minutes, cooling in still air to the temperature of isothermal holding and holding performed using parameters HT1 through HT4 are presented in Table 3. Table 4 presents the contents of retained austenite in the specimens after strength tests in the gripped (undeformed) part and in the part subjected to the tensile test, under the surface of rupture. The more favourable set of parameters determined during the tensile test was obtained for the specimens subjected to isothermal holding for the longer time amounting to 72 hours (in relation to both temperatures of isothermal transformations used in the tests). The heat treatment involving the use of a temperature 225 °C for 72 hours led to the obtainment of the following average values: $R_{p0.2}$ = 1308 MPa, R_m = 1950 MPa and A = 14.2 %. In turn, the treatment performed using a temperature of 235 °C for 72 hours resulted in the obtainment of $Rp_{0.2}$ = 1351 MPa, R_m = 1944 MPa and A = 15.5 %. Slightly lower values, i.e. $Rp_{0.2}$ = 1283 MPa, R_m = 1934 MPa and A = 14.4 %, were obtained in relation to holding at a temperature of 235 °C for a shorter time of 48 hours. In comparison with the other variants of heat treatment, the use of a shorter holding time of 48 hours and a lower temperature of 225 °C significantly decreased the value of the yield point to $R_{p0.2}$ = 1162 MPa, increased strength up to R_m = 1988 MPa and decreased total elongation to A = 13.6 %. The post-heat treatment hardness values of the specimens varied only slightly and were restricted within the range of 529 HV10 to 533 HV10. Similar to the results of retained austenite-related measurements performed using the dilatometric specimens, also the results of measurements involving the undeformed parts of the specimens were characterised by significant scatter (Table 4), precluding the analysis of the correlations between the contents of retained austenite and the values of the yield point, strength and elongation (plasticity). Probable reasons for the scatter of the results of single measurements concerning retained austenite in the cross-section of the gripped parts of the specimens subjected to the strength-related tests included the irregular arrangement of retained austenite in the volume of material induced by chemical inhomogeneity and, at the same time, by the small cross-sectional area of the measurement beam (°C approximately 300 μm), applied due to the very limited availability of the measurement area, as well as by the crystallographic texture of the material. The measurements concerning the content of austenite directly beneath the rupture in the specimens subjected to the strength tests preceded by HT1 and HT4 revealed that retained austenite transformed entirely into martensite (Table 4). Figure 6 presents exemplary SEM images of the microstructure of the ruptured specimen subjected to the strength test preceded by HT4 in the gripped (undeformed) part (Fig. 6a) and in the area directly beneath the rupture surface, with visible martensite M_D formed from the block of retained austenite (Fig. 6b).

4. Discussion

The procedure concerning the experimental development of standard TTT diagrams of steels using dilatometric measurements involves the very fast cooling of specimens from austenitising temperature to the temperature of isothermal transformation, aimed to prevent changes in the location/arrangement and the chemical state of atoms of chemical elements dissolved in austenite. For instance, ASTM standard [26] in relation to conditions concerning the development of diagrams of phase transformations for low-alloy

Table 3. Mechanical properties determined during the tensile tests and hardness measurements of the specimens made of the NBH steel subjected to heat treatment including austenitising at a temperature of 950 °C for 30 minutes, cooling in still air to the temperature of isothermal holding and isothermal holding performed using parameters of HT1–HT4

Variant of hea treatment	Specimen designation	HV10	Mean HV10	$R_{p0,2}$ [MPa]	Mean $R_{p0,2}$ [MPa]	R_m [MPa]	Mean R_m [MPa]	A/A_{gt} [%]	Średnia A/A_{gt} [%]
HT1: 235°C/48 h	1A	526	529	1300	1283	1941	1934	16.0/9.1	14,4/8,4
	2B	531		1254		1959		13.0/9.1	
	3C	531		1294		1902		14.2/7.0	
HT2: 235°C/72 h	2A	530	530	1324	1351	1929	1944	15.3/9.4	15,5/9,3
	3B	531		1378		1959		15.7/9.2	
HT3: 225°C/48 h	1B*	528	532	1216	1162	1952	1988	12.5/9.4	13,6/9,0
	2C*	531		1137		1960		14.5/8.8	
	3A*	533		1186		1964		15.0/8.6	
	1C	534		1107		2074		12.5/9.2	
HT4: 225°C/72 h	4A*	533	533	1355	1308	1923	1950	14.0/8.2	14,2/8,7
	4B*	535		1270		1961		14.0/8.6	
	4C	530		1299		1965		14.7/9.2	

* Specimens subjected to re-austenitising and isothermal holding because of furnace failure during the original cycle of heat treatment

Table 4. Contents of retained austenite in the specimens subjected to the tensile test: (a) in the undeformed (gripped) part and (b) directly beneath the fracture surface; nm – not measured

Variant of heat treatment	Specimen designation	Temperature [°C]/ time [h] of isothermal holding	Content of retained austenite, XRD measurement, vol. %		
			Undeformed gripped part		Underneath rupture surface
			Single measurements	Mean – without (*)	
OC1	3C	235 / 48	30; 20; (55)*	25	lack of austenite XRD line
OC2	3B	235 / 72	21; 30; 16	22	nm
OC3	1C	225 / 48	21; 15; (49)*	18	nm
OC4	4C	225 / 72	21; 18; 15	18	nm
	4A	225 / 72	nm	-	lack of austenite XRD line

(*) Very strong second line of austenite of hkl indices (200), probably triggered by the crystallographic texture

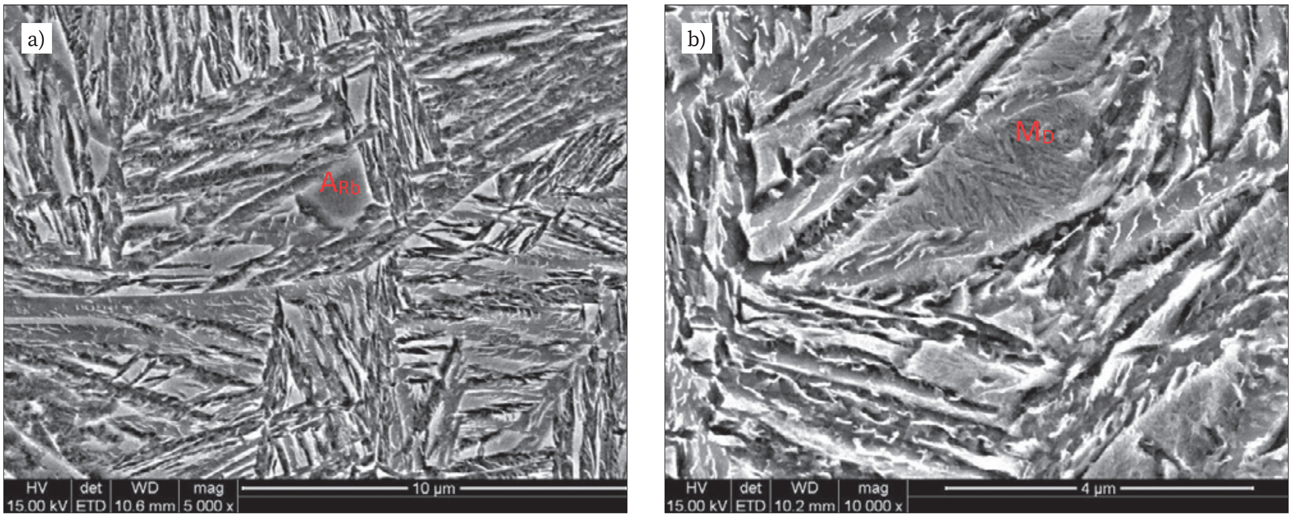


Fig. 6. Microstructure of specimen 4A after the tensile test: (a) in undeformed gripped part), ARb – blocky retained austenite and (b) – directly beneath fracture surface, MD – martensite formed as a result of deformation or stress; SEM images

hypoeutectoid steels recommends the use of a cooling rate of not less than 175 °C/s. Publications containing results of the development of bainitic transformation-related TTT diagrams refer to the use of various cooling rates to the temperature of isothermal transformation such as 50 °C/s [27], 100 °C/s [28], 150 °C/s [29] and 20 °C/s (in the tests discussed in this article). The comparative analysis revealed (e.g. [30] and the test results discussed in the article) that the time of the incubation of bainitic phase nucleation and the kinetics of bainitic transformation depended on the rate of cooling from austenitising temperature to the temperature of transformation – due to the effect of the cooling rate in the thermodynamic state of austenite (caused particularly by the segregation of atoms to austenite grain boundaries). The isothermal diagram presented in Fig. 3 reveals that after cooling at a rate of 20 °C/s from austenitising temperature, the incubation time of isothermal bainitic transformation at a temperature 275 °C along with the time of cooling amounted to 2270 s (Table 2). In turn, the CCT diagram presented in Fig. 1 reveals that the initiation of bainitic transformation at a temperature of approximately 275 °C (as a result of cooling at a rate of 1 °C/s from the austenitising temperature) took place after 700 s from the start of the cooling process, i.e. after an approximately three-fold shorter time. In order to enable the use of TTT-type diagrams of phase transformations to design industrial isothermal treatment including cooling from austenitising temperature at various cooling rates (including those of values decreasing along with temperature drops) it is necessary to develop modified isothermal diagrams for cooling rates used in industrial processes. In the symbol of modified isothermal diagram proposed by the Authors, i.e. TTTv, v stands for the constant cooling rate from austenitisation temperature expressed in /s (e.g. TTT20). In turn, in relation to cooling at a variable (changeable) rate (typical of industrial conditions), the symbol could be TTTvc. Applied cooling parameters would be added to a given TTTvc diagram.

Recent years have seen many research works concerning the development of nanobainitic steels involving the design of chemical composition and the determination of heat treatment parameters leading to the reduction of isothermal holding time and the improvement of technological properties (e.g. weldability). The subject of research included, among other things, steel grades with reduced contents of carbon and alloying elements, characterised by improved weldability and shorter treatment time, which, however, led to a decrease in hardenability, whose high level is indispensable in many steelmaking processes. In the formation of the duplex-type structure, composed of carbide-free nanobainite and retained austenite, an important role is played by carbon, which has the greatest effect on the reduction of temperature MS, is responsible for the solid solution hardening of carbide-free bainite and retained austenite and has the greatest effect on the thermal and mechanical stability of retained austenite. The question which arises is concerned with the lowest content of carbon in low and medium-alloy steels enabling the obtainment of nanobainite, i.e. carbide-free ferrite lamellae having a thickness of less than approximately 100 nm and ultra-high-strength steel. Previous tests have confirmed that a carbon content of 0.6 % in medium-alloy steel enables the obtainment of ultra-high-strength nanobainitic products [12–16]. Research aimed to develop nanobainitic steel grades with a carbon content restricted within the range of 0.4 % to 0.5 % has not yet yielded decisive results [31]. The results of research performed within this work revealed that the lower limit of carbon content enabling the formation of nanobainite in medium-alloy steels as a result of the isothermal transformation of recrystallised austenite was close to 0.50 %. The formation of lamellae or laths of bainitic ferrite having a thickness comparable to nanobainite in steel with a carbon content reduced to less than 0.5 % could be achieved by deforming austenite before transformation [32] or by using isothermal multi-stage treatment [33]. Lowering the carbon content below 0.4 % enabled the obtainment of classic lower bainite morphology as a result of isothermal treatment or continuous cooling, which, depending on the type and amount of added alloying elements, could be characterised by the high level of mechanical properties, yet not comparable with those of nanobainite [34].

The description of the mechanism concerning the effect of the morphology and content of retained austenite on mechanical properties (in particular plasticity) of nanobainitic steels, presented briefly in Chapter 1, changed significantly along with the collection of experimental data, but it has not yet been finalised in the form of a theory explaining related phenomena. Particularly noteworthy is the well-established view that the classic TRIP mechanism does not have a decisive influence on the plasticity of nanobainitic steels and that the dominant mechanism is more complex, involving the mechanical effect of adjacent areas of bainitic ferrite and retained austenite [9, 10]. Numerous experimental tests revealed that the presence of blocky retained austenite did not automatically translate into the reduction of steel plasticity. The above-named observation has been confirmed by the results of the research work discussed in the article. After transformation into nanobainite at a temperature of 225 °C that of 235 °C, the microstructure of the test NBH steel contained approximately 20 % austenite, including blocky austenite (with blocks being several micrometres in size – Fig. 4), transformed into martensite during deformation (Fig. 6 b). The plasticity measured on the basis of total elongation obtained in tensile tests was restricted within the range of 13.6 % to 15.5 % with the strength amounting to 1.9 GPa.

5. Conclusions

The use of dilatometric tests, microstructural observations and hardness measurements enabled the identification of phase transformations taking place under isothermal conditions (TTT) and during continuous cooling (CCT) in nanobainitic steel having a carbon content of 0.55 % and characterised by high hardenability, intended for the industrial production of metal sheets/plates. The analysis of the above-presented test results justified the formulation of the conclusions presented below.

The critical cooling rate read out of a related CCT diagram and ensuring the obtainment of the initial value of temperature BS (300 °C or lower) of the test NBH steel amounted to 0.5 °C/s.

Because of the effect of the cooling rate on the thermodynamic state of austenite, triggered, particularly, by the segregation of atoms to austenite grain boundaries, the time of incubation of the bainite nucleation process and the kinetics of the bainitic transformation depended on the cooling rate from the austenitisation temperature to the temperature of transformation. In order to enable the use of TTT-type phase transformation diagrams in the design of industrial isothermal treatment processes, including cooling (at different rates) from austenitisation temperature, it is necessary to develop modified isothermal diagrams adjusted to cooling rates applied in industrial processes. In the symbol of the modified isothermal diagram proposed by the Authors, i.e. TTTv, v stands for the constant cooling rate from austenitisation temperature expressed in °C/s (e.g. TTT20).

The tests conducted within the research work confirmed information found in reference publications stating that the presence of blocky retained austenite does not necessarily trigger the reduction of steel plasticity. After transformation into nanobainite at a temperature of 225 and that of 235 , the microstructure of the test NBH steel contained approximately 20 % of austenite, including blocky austenite (with blocks being several micrometres in size – Fig. 4), transformed into martensite during deformation (Fig. 6 b). The plasticity measured on the basis of total elongation obtained in tensile tests was relatively high and restricted within the range of 13.6 % to 15.5 %; the strength being 1.9 GPa.

The Authors formulated a thesis, the confirmation of which requires further experimentation, that the lower limit of the carbon content enabling the formation of nanobainite in medium-alloy steels as a result of isothermal transformation of recrystallised austenite was close to 0.50 %.

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